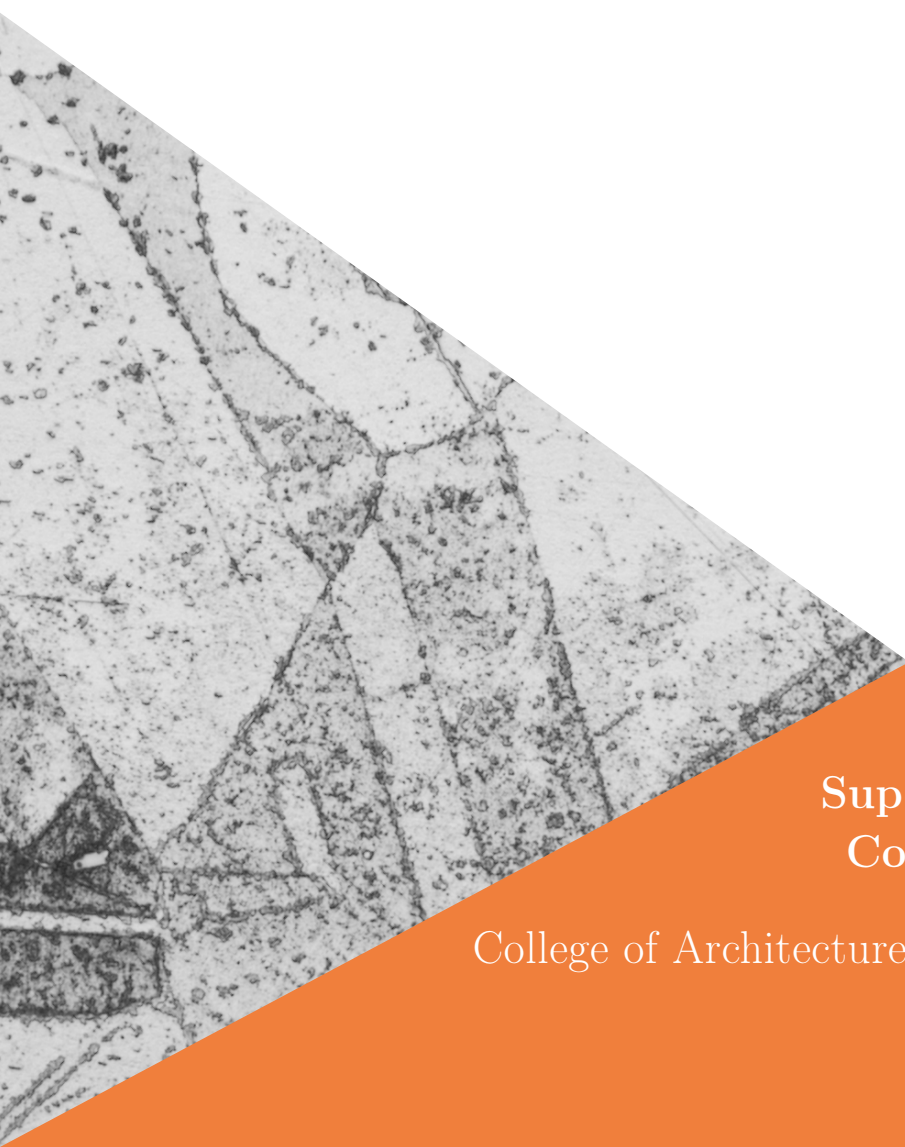


Development of a Multi-Scale creep modeling approach

A thesis submitted in partial fulfillment of the requirements for the degree
of Doctor of Philosophy (PhD) in Engineering Science

by

Carlos Eduardo Rojas Ulloa



Supervisor: Habraken, Anne Marie

Co-supervisor: Duchêne, Laurent

College of Architecture, Geology and Civil Engineering

August 2026

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Jury members

Prof.Dr. Anne Marie Habraken (Supervisor)	anne.habraken@uliege.be Department of Urban & Environmental Engineering (UEE), University of Liège 9 Allée de la Découverte, Parking B52/3 4000 Liège, Belgium
Prof.Dr. Laurent Duchêne (Co-supervisor)	l.duchene@uliege.be Department of Urban & Environmental Engineering (UEE), University of Liège 9 Allée de la Découverte, Parking B52/3 4000 Liège, Belgium
Prof.Dr. Víctor Tuninetti Vásquez	victor.tuninetti@ufrontera.cl Department of Mechanical Engineering, Universidad de La Frontera Av. Francisco Salazar #01145 4811230 Temuco, Chile
Dr. Jérôme Tchoufang Tchuindjang	jerome.thoufang@uliege.be Aerospace & Mechanics (A&M) department, University of Liège 9 Allée de la Découverte, Parking B52/3 4000 Liège, Belgium
Prof.Dr. Anne Mertens	anne.mertens@uliege.be Aerospace & Mechanics (A&M) department, University of Liège 9 Allée de la Découverte, Parking B52/3 4000 Liège, Belgium
Dr. Florian Riedlsperger	florian.riedlsperger@jku.at Institut für Metallische Konstruktionswerkstoffe, Johannes Kepler Universität Altenberger Straße 69 4040 Linz, Austria

MScEng. Alexandre Vendramini

alexandre.vendramini@drever.be

R&D Department, Drever International,
SMS Group GmbH

Allée des Noisetiers 15

B-4031 Angleur (Liège), Belgium

*To my family,
my father Hugo Eduardo Rojas Montesino,
my mother Carmen Isabel Ulloa González,
and for the memory of those who could not stay with us to the end*

Abstract

The high-temperature and low-stress creep deformation response of Incoloy 800H is characterized by two consecutive creep strain rate minima. The first minimum creep rate peak is attributed to a solid-solution hardening phenomenon, whereas the second one is associated to the steady-state creep rate of the material.

In this thesis, the accurate prediction of the complex microstructure-dependent creep behavior of 800H alloy is achieved through a multi-scale creep modeling approach involving a micromechanical mean-field model (MFC) and a macromechanical finite element constitutive law (VMVP).

A thorough experimental campaign addressing creep tests and microstructural characterization techniques was first organized to understand the high-temperature creep deformation response of 800H alloy. Microstructural characterization tests of specimens in as-received, furnace-aged and creep-tested conditions revealed a complex microstructure evolution involving aging, oxidation and nitridation. Additionally, curves showing non-classical characteristics were observed during low-stress creep tests. Given the limited number of experimental creep curves, these were complemented with virtual creep curves generated using the MFC model developed during this PhD.

MFC computes the creep strain rate by solving a non-linear equation system where dependency on the mean dislocation density and precipitate kinetics is established. The latter was introduced from ex-situ thermokinetic simulations conducted in Thermo-Calc[®] PRISMA. The 1st creep strain rate peak was assimilated as an internal stress contribution slowing down dislocation glide, whereas the 2nd peak was captured by an improved stress sensitivity formulation of the mechanical energy threshold in the mean dislocation glide velocity equation. The virtual tests generated with MFC demonstrated remarkable physical consistency and accuracy within the stress-temperature loadings of interest for this work, improving our understanding of the non-classical creep deformation response at creep loadings found between our creep experiments (interpolation) and at very-low stresses (extrapolation, where conventional tensile creep tests would take years to complete).

The database comprised of experimental and virtual creep curves was then used for the identification and validation of VMVP in Lagamine. To predict the non-classical creep behavior of 800H, VMVP incorporates a new viscosity function: a classical Norton law recovering the steady-state creep strain rate of the material modified by an activation function factor which reduces the initial creep strain rate to capture the 1st solid-solution hardening creep strain peak. The activation function Norton (*afN*) equation demonstrated good numerical stability while capturing the 2-peak creep behavior of Incoloy 800H.

The creep deformation response predicted by VMVP and identified through the MSCreep approach (VMVP-MSCreep) was found in good agreement with ULiège experiments and MFC virtual creep curves associated to high-temperature and low-stress creep tests. Post-assessment tests recommended by the European Creep Collaborative Committee (ECCC) show that the log.-log. prediction error for the steady-state creep rate and the time-to-1% creep deformation are found within the $\pm \ln(2)$. According to the ECCC standard, the predictability of the VMVP-MSCreep model is considered admissible. Additional experimental validation for complex loadings was ascertained by modeling the creep deflection of 6 cantilever beams. Numerical results achieved with VMVP-MSCreep were found in reasonable agreement with experiments. Additionally, the cantilever bending configuration allowed to establish a mesh size dependency on the stress gradient, thereby ensuring the consistency of the creep prediction of VMVP-MSCreep for realistic industrial applications.

Acknowledgments

This project has been funded by the Fonds de la Recherche Scientifique F.R.S.-F.N.R.S. through the PhD. FRIA grants (Fonds prour la Recerche dans l'Industrie et l'Agriculture) B1 4000-8987 and B2 4002-1907. Additionally, the project has received economical and scientific support from the European Project AID4Greenest HORIZON-CL4-2022-RESILIENCE-01-19. The author acknowledges the scientific support from the international research cooperation agreement WBI-Belgium/AGCID-Chile 2023-2025 RI-02 (DIE23-001), the technical and economical support by Drever International on the experimental campaign, and researchers Dr. Leo A.I. Kestens and Dr. Tuan Nguyen-Minh for providing expert guidance on the dislocation density characterization of metals via EBSD.

To our industry partners and my future colleagues: MScEng. Amedeo di Giovanni, MScEng. Olivier Pensis, Dr. Van Long, and MScEng. Alexandre Vendramini,

to our M&S laboratory: Mr. Carl Vrooman and Mr. Mario Bustos Hermosilla,

to our fellow MMS researchers: Dr. Jérôme Tchoufang Tchuindjang, Dr. Olivier Dedry and Mme. Sylvie Salieri,

to our dear mean field modeling enthusiasts: PhD. candidate Laura Witzmann, Dr. Florian Riedlsperger and Dr. Surya D. Yadav,

to the brave souls of the MS²F division in B52/3:

my supervisors Prof. Dr. Laurent Duchêne and Prof. Dr. Anne Marie Habraken, for pursuing excellence in every review

my fellow MSM team members: Dr. Ehssen Betaieb, Dr. Fan Chen[♥], Dr. Hélène Morch, and PhD. candidates[♣] Guillian Bryndza, Rayane Eloudrhiri[♥], Calogero Gallo and Héctor Sepúlveda

our lunchtime president Dr. Benjamin Dewalls, our secretary Laurence Defrere, and all my coffee break enthusiasts in B52/3 +1:[♣] Kawthar Achary, Pratik Chakraborty, Alexandre De Becker, Cristophe Dessers[♥], Utashi Ciraane Docile[♥], Joris Hardy, Verónica Herrera Gómez[♥], Tohid Jamali[♥], Hiba Khaif[♥], Luca Santoro[♥], Vincent Schmitz and Shuyue Yu[♥]

to my Professors in Universidad de La Frontera (Temuco, Chile), for being the spark of to my scientific curiosity:[♣] Fabiola Arévalo, Leonardo Balart, Pablo I.R. Pincheira, Mauricio Santibañez, Fabian A. Torres-Ruiz, Víctor Tuninetti and Asticio Vargas,

and to the greatest gifts this life has given me:[♣] MScEng. Anne-Lou Borgna, Lt. Kevin Carvajal, MEd. María Redondo Plaza, Dr. Daniela Rodríguez Castro, MPT. Javier Vera Ojeda, and my девушка, Валерия Павлиенко.

Thank you all for your unwavering support throughout these years, and for walking beside me along this path of beautiful struggle. Each one of you a beacon that guided me to become a better human, a better friend, and a PhD.

[♣] : names organized alphabetically, so no one gets mad!

List of Acronyms

afN	activation-function Norton
AGS	Average grain size
AR	As-Received (material)
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
BCC	Body centered cube
BF-TEM	Bright Field Transmission Electron Microscopy
BMD	Bird-Mukherjee-Dorn (creep model)
BSE	Back-scattered electron
CALPHAD	CALculation of PHase Diagrams
CAL	Continuous annealing line
Cc	Coble creep
CDD	Continuum dislocation dynamics
CDM	Continuum damage mechanics
CGL	Continuous galvanizing line
COLAW	Constitutive law (Finite Element)
CP	Crystal Plasticity
CSL	Coincident Side Lattice
DDD	Discrete Dislocation Dynamics
DIN	Deutsches Institut für Normung
DMM	Deformation Mechanism Map
DRX	Dynamic recrystallization
DSA	Dynamic strain aging
DW	Dislocation Wall

EBSD	Electron BackScattered Diffraction
ECCC	European Creep Collaborative Committee
ECISS	European Committee for Iron and Steel Standardization
EDM	Electric discharge machine
EDS	Energy-Dispersive X-ray Spectroscopy
FCC	Face centered cube
FE	Finite element
FESEM	field Emission Scanning Electron Microscope
GA	genetic algorithm
GAC	General Atomic Company
Gb	Grain boundary
GBE	Grain Boundary Engineering
GBS	Grain boundary sliding
GIT	Górniośląski Instytut Technologiczny
GW	Graham-Walles
HAZ	Heat Affected Zone
HAGB	High-Angle Grain Boundary
HCP	Hexagonal close-packed
HCF	High Cycle Fatigue
HTc	High-temperature creep
HTR	High Temperature Reactor
IHX	Intermediate Heat Exchangers
IU	International Units
KAM	Kernel average misorientation
KME	Kocks-Mecking-Estrin (model)
LCF	Low Cycle Fatigue
LFR	Life fraction rule
LMP	Larson-Miller parameter
LOM	Light Optical Microscopy

LPBF	Laser Powder Bed Fusion
LTc	Low-temperature creep
LVDT	Linear variable differential transducer
MBP	Manson-Brown parameter
mcr	minimum creep strain rate
MFC	Mean Field Creep
MF	Mean-Field
MGR	Monckman-Grant relationship
mGW	modified Graham-Walles (creep model)
MHP	Manson-Haferd parameter
MMELO Online	Metallic Microstructures European Lectures
MMS (research team, ULiège)	Metallurgic and Materials Science
M&S	Materials and Structures
MSCreep	Multi-Scale Creep
MSM (research team, ULiège)	Materials and Solid mechanics
NDA	non-disclosure agreement
NHc	Nabarro-Herring creep
NINMS	National Institute of Materials Science
NPP	Nuclear power plant
OAZ	Oxidation affected zone
OPM	One parameter model
OPS	Oxide polishing suspension
OSR	Objective Stress Range
OTR	Objective Temperature Range
PAN	Plasma-assisted nitridation
PAT	Post-Assessment Test
PLM	Polarized light microscopy
RGB	Red-Green-Blue (light)

RMSPE	Root-Mean Squared Percentile Error
RT	Room Temperature (RT, 20°C)
SA	Solution Annealing
SA+WQ Quenching	Solution Annealing followed by Water Quenching
SCW	Super-critical water
SCD	Statistical continuum dislocation
SCT	Standard Creep Test
SE	Secondary electron
SEM	Scanning Electron Microscopy
SGb	Subgrain boundary
SHS	Super-heated water
SSH	Solid-solution hardening
SSM	Stepped iso-Stress Method
TEM	Transmission electron microscopy
TIG	Tungsten Inert arc Gas
TMP	Thermo-Mechanical Processing
TTT	Time-Temperature Transformation (diagram)
UNS	Unified Number System
UTS	Ultimate Tensile Strength
UVCN	Unified viscoplastic constitutive model
VHTR	Very-High Temperature Reactor
VMVP	von-Mises Visco-Plastic (Lagamine COLAW)
VT	virtual test
WQ	Water Quenching
Z/R	Zwick/Roell (creep testing equipment manufacturer)

List of Latin-style symbols

b		Burgers' vector
t		Dislocation line vector first
E		Elastic modulus
G		shear modulus
k_B		Boltzmann constant ($= 1.38065 \times 10^{-23} \text{ JK}^{-1}$)
MV		macro-indentation Vickers
R_{SGb}		Mean subgrain radius (R_{SGb})
$t_{1\%}$		Time-to-1% creep deformation
T_m		Melting point temperature
t_r		Time-to-rupture

List of Greek-style symbols

α_T		Thermal dilatation coefficient
ϵ		strain
$\dot{\epsilon}_{ss}$		Steady-state creep strain rate
μV		micro-indentation Vickers (μV)
ρ_b		Boundary dislocation density (m^2)
ρ_{GND}		Geometrically necessary dislocations density
ρ_m		Mobile dislocation density (m^2)
ρ_s		Static (or dipole) dislocation density (m^2)
σ_0		Initial (engineering) stress (MPa)
σ_a		Applied stress stress
σ_y		Yield stress
Θ_{ave}		Average local misorientation
ν		Poisson ratio

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Introduction

Creep is defined as the slow viscous-like inelastic deformation of metallic materials produced by a combination of sufficiently high temperatures (usually above 0.5 times their melting point temperature) and mechanical loadings found well within their elastic regime. The existence of this deformation mechanism went unnoticed for centuries, perhaps because of the limited number of high temperature applications and the counterintuitive idea of permanent deformations coexisting with elastic stresses. The discovery of creep is linked to the advent of the industrial revolution in the XIX century. The rapid development of more efficient steam-powered technologies lead to a rampant increment in the service temperatures of critical components, as illustrated in Figure 1. Unbeknownst to engineers at the time, the creep deformation induced by high thermomechanical loadings and favored by the rather lackluster performance of popular C-steels often resulted in the catastrophic failure of critical components. Records describing incidents likely associated with the accumulation of creep damage (e.g., boiler explosions in vapor boats, locomotives and power plants) account for thousands of fatalities in the United States alone [1].

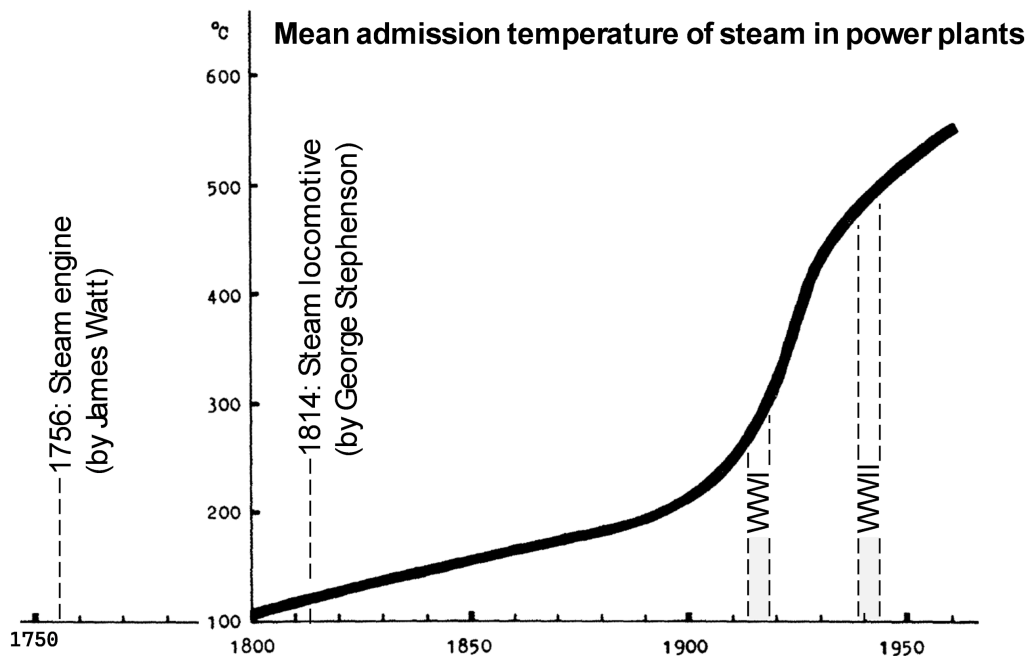


Figure 1: Mean operational temperature of pressure vessels across the years associated with the industrial revolution of the XIX century. After [2].

*Creep is inevitable. We can either fear it, or learn how to control it.
This thesis deals with the latter.*

Context

Much like during the XIX century, the increase in the mean service temperature of critical components subjected to creep in strategic industry sectors is still an inherent consequence of scientific innovation. To ensure the safety and reliability of these technological developments, engineers are required to perform accurate creep life assessments. This means an optimal material selection and a realistic service life projection.

Material: Incoloy 800H

Incoloy 800H is a well known austenitic Fe-Ni-Cr alloy developed during the 1960s that provides good mechanical performance at high temperatures. It combines good weldability, improved creep and corrosion resistance compared to other stainless steel alloys, and a lower cost/weight ratio compared to more expensive Ni-based alloys. Following American Society of Mechanical Engineers (ASME) approval, it has seen widespread use in high temperature gas-cooled reactor components reaching temperatures of 760°C [3], however the limited information available on creep-fatigue and environmental effects has prevented its extension toward very-high temperature reactors (gen.IV, reaching temperatures beyond 850°C, see [4]). Nevertheless, it is still a popular choice for the manufacturing of high temperature components required to withstand decades in corrosive environments. Such is the case of pigtail lines in petrochemical industries where temperatures up to 1000°C have been recorded [5].

Studies conducted since the 1980s have consistently revealed that the deformation response of Incoloy 800H is strongly microstructure-dependent [6–12]. In particular, high-temperature low-stress creep loading regimes were found to induce a non-classical creep behavior characterized by two consecutive minimum creep strain rate peaks. The 1st peak was attributed to a solid-solution hardening (SSH) phenomenon [13, 14] caused by the interstitial diffusion of atoms originating from the dissolution of the intragranular Cr_{23}C_6 carbides that nucleated during the SA+WQ heat treatment of the alloy. The 2nd peak was correlated to the conventional steady-state creep strain rate ($\dot{\epsilon}_{ss}$) of the material as a consequence of the delicate balance reached between dislocation production and annihilation. Additionally, the diffusion of N into the matrix material exposed to high- N_2 atmospheres (e.g., air) in high temperature environments at atmospheric pressure induces the precipitation of Cr- and Al nitrides (often observed in the vicinity of cracks [8, 12]). These nitrides have recently been associated with a creep strengthening mechanism by A.M. Young et al.[15] in a perfectly controlled laboratory environment; however, the consistency of N diffusion and its strengthening effect in a realistic industrial atmosphere are scientific inquiries we aim to address over the course of this thesis.

Creep modeling

The first formal description of creep as a new time-dependent inelastic deformation mechanism is found in the doctoral studies by Dr. *Edward Neville da Costa Andrade*[16, 17] in 1910. The empirical time-dependent power law expression proposed by *da Costa Andrade* is shown in Eq.(1a), where the creep strain (p) is computed as a summation of both primary (p_I) and secondary (p_{II}) equations with α_A , β_A and m_A phenomenological constants. The validation of this first creep modeling approach was limited because of the experimental constraints of the time. Nevertheless, it has been recognized as the milestone marking the beginning of the long road of creep models. Later, the studies by F.H. Norton [18] and J.E. Bailey & P.B. Hirsch [19] built what is currently known as the popular Norton-Bailey-Hirsch (or simply Norton) law. It establishes a generalized steady-state creep strain rate ($\dot{\epsilon}_{ss}$) dependency on stress for a given temperature through the power-law expression shown in Eq.(1b), where σ is the applied stress,

and K & n are material constants.

$$p = p_I + p_{II} = \alpha_A t^{m_A} + \beta_A t \quad (1a)$$

$$\dot{\epsilon}_{ss} = \left(\frac{\sigma}{K} \right)^n \quad (1b)$$

The Norton power law is perhaps the most popular creep model of all time. The expression $\dot{\epsilon}_{ss} = f(\sigma)$ is a simple yet accurate phenomenological law that is easy to identify and validate for multiple materials. It is, however, only one of the possibly hundredths of creep modeling approaches developed to date. An outstanding rundown of creep models intended for full creep curve modeling, or extrapolation of steady-state creep rate, time-to-1% creep deformation or time-to-rupture is found in the review paper of M. Sattar et al.[20]. A more modest literature review on this subject, addressing creep models considered fundamental in the context of this PhD., is given in chapter 1, section 1.1.

Creep modeling of 800H: a scientific challenge

Creep modeling of 800H alloy is often addressed from an empirical standpoint where the steady-state creep strain rate ($\dot{\epsilon}_{ss}$) or the time-to-rupture dependency on stress and temperature is captured using diagrams or classical extrapolation models such as the Larson-Miller parameter [21, 22] or the Manson-Haferd parameter [23]. The design-to-failure approach prioritized in ASME standards and industrial data sheets is useful for analytical calculations where the stress states are largely tensile (e.g. pressure vessels) or where deformations can just be found within allowable ranges (e.g. pigtail lines). This PhD. project diverges from simple analytical calculations as it aims to provide an accurate prediction of the creep deformation response of complex geometries of 800H using the finite element method. To this end, the in-house finite element software Lagamine [24] was used.

A remarkable precedent on the modeling of creep in Lagamine is the work of H. Morch [25]. It consists of a unified viscoplastic constitutive model (UVCN) that combines a temperature-sensitive Norton law formulation with complex Armstrong-Frederik [26] backstress formulations and creep-fatigue damage formulations[27, 28]. The viscosity function coupled with complex backstress formulations predict the creep-fatigue behavior of Hastelloy A230 components with good experimental validation [29].

Attempts to use the original Chaboche-Morch UVCN to predict the 2-peak creep deformation response of Incoloy 800H were unsuccessful. The Norton viscosity function implemented in this UVCN attains a simple $\dot{\epsilon}_{ss}$ relationship, which is equivalent to the 2nd creep strain rate minimum of Incoloy 800H. The 1st peak, attributed to SSH, could not be recovered. This observation led us to develop a new viscosity function that incorporates these microstructure dependencies in the form of empirical parametric functions. The latter is a common practice among the creep engineering community, as it provides simplified expressions with acceptable results.

*Make creep models as complicated as they must.
But no more.*

Objectives

The main objective of this thesis is **to develop of a Multi-Scale creep model to predict the high-temperature & low-stress creep deformation response of Incoloy 800H**. To this end, the following secondary objectives must be achieved:

- To organize and conduct a thorough experimental campaign on the creep behavior of Incoloy 800H relying on macromechanical and microstructural characterization techniques.
- To develop a micromechanics-based mean-field creep model to be used as a physics-based inter- and extrapolation tool.
- To exploit this mean-field creep model as a tool to generate virtual creep curves to complement those obtained during the standard creep tests conducted during our experimental campaign.
- To develop a numerically reliable finite element viscosity constitutive law capable of capturing the complex 2-peak creep deformation response of Incoloy 800H.
- To define a multi-scale modeling framework to identify this constitutive law and validate the final finite element model for the high-temperature low-stress creep regime of 800H alloy.

Thesis summary

Methodology

1. **Literature review:** An organized compilation of all the scientific contributions relevant for this PhD. project. It was subject to continuous updates and structural modifications following the discovery of new interesting information.
2. **Experimental campaign:** A comprehensive experimental methodology organized in cooperation with our industry partners at Drever International, comprised of:
 - Macroscopic tests: standard (tensile) creep tests conducted in cooperation with our technicians and machine operators in Materials and Structures (M&S) laboratory (ULiège).
 - Microscopic test: Vickers indentation, light optical microscopy and scanning electron microscopy (SEM) experiments were conducted under the expert supervision of Metallurgic and Materials Science (MMS) researchers in ULiège and with the help of UGent materials research team of Prof. Leo A.I. Kestens.
3. **Model development:**
 - Macromechanical von-Mises viscoplastic (VMVP) finite element constitutive model. The ability to model the non-classical creep deformation response of 800H alloy is incorporated by means of a modified viscosity function.
 - Micromechanical mean-field creep (MFC) model. This dislocation density-based model is used to produce virtual tests to complement the macromechanical finite element model.
 - Multi-scale creep modeling approach. The extended material creep behavior knowledge bank, comprised of experimental and virtual (MFC) tests, is used to identify the parameters of the macromechanical (VMVP) finite element law.
4. **Validation** of the VMVP constitutive model identified following the multi-scale creep modeling approach. The validation process addresses the following:

- Experimental creep curves benchmark addressing high-temperature low-stress creep tests conducted in ULiège.
- Post-Assessment Tests (PATs) (standard testing methodology by the European Creep Collaborative Committee (ECCC)).
- Industrial benchmark addressing material manufacturer data sheets provided by our industry partners at Drever International.
- Prediction of the cantilever bending of 800H beams under creep. This validation allowed us to establish a mesh size dependency on the stress gradient, thus providing a simple rule to maintain the accuracy and consistency of creep predictability across different finite element geometries.

Structure

- Chapter 1, “State of the Art”, is an organized compilation of the literature findings deemed relevant for the progress of this research project. Section 1.1 addresses the physics and phenomenology of creep, addressing modeling techniques across different length scales. Section 1.2 describes the mechanical properties and high-temperature performance of Incoloy 800H. Emphasis is placed on microstructure evolution following long-term high-temperature exposure (e.g., aging, oxidation and nitridation) and how these factors affect the creep behavior of the alloy.
- Chapter 2, “Experimental campaign”, describes the experiments conducted over the course of this project. The experimental methodology and testing equipment are presented in section 2.1, whereas section 2.2 presents the experimental results. Note that the experimental curves as well as exact stress-temperature loadings addressed during our campaign are not disclosed. This chapter contains the following annexes:
 - Annex 2A: Preprint version of the scientific contribution by C. Rojas-Ulloa et al.[30] on the microstructural characterization of Incoloy 800H specimens following years of exposure to a realistic industrial environment.
 - Annex 2B (confidential): Brief semi-quantitative study on the oxidation/nitridation competition for Cr observed in 800H alloy.
 - Annex 2C: In-house written user-manual describing the series of steps required to perform a creep tests in the Zwick/Roell Kappa 100DS creep machine located at the M&S laboratory in ULiège.
 - Annex 2D (confidential): Experimental creep curves with no normalization.
- Chapter 3, “Mean-Field Creep (MFC)”, deals with the micromechanics-based numerical model developed to produce physically-grounded virtual creep curves. MFC is presented through the article preprint by C. Rojas-Ulloa et al.[14]. The manuscript, published in *Journal of Materials Science*, places particular emphasis in the two key numerical contributions that enabled MFC to capture the complex two-peak creep behavior of 800H alloy: the incorporation of the 1st (SSH) peak as an internal stress contribution, and an improved stress-sensitivity of the activation volume capturing the 2nd ($\dot{\epsilon}_{ss}$) creep rate peak. Validation of the model was established for high-temperature and low-stress creep regimes.
This chapter contains a single annex:
 - Annex 3A (confidential): Non-normalized creep curves and implicit numerical implementation algorithm

- Chapter 4, “Macromechanical creep model: VMVP”, follows the development of a UVCM that predicts the non-classical two-peak creep deformation response observed in Incoloy 800H. To this end, the classical Norton viscosity function (used for instance by the Chaboche-Morch constitutive model [25]) is replaced by an activation-function Norton (afN). The parameter identification procedure offered in section 4.2 relies exclusively on experimental creep curves obtained at the M&S laboratory in ULiège (i.e., no MFC virtual tests are addressed).
 - Annex 4A: Preprint version of the scientific contribution by C. Rojas-Ulloa et al.[31] on the implementation of a Graham-Walles viscosity function to predict the non-classical creep behavior of alloy 800H.
- Chapter 5, “Multi-scale creep modeling approach: MSCreep”, establishes the multi-scale modeling framework and applies it to predict the high-temperature low-stress creep behavior of Incoloy 800H. The multi-scale creep modeling methodology, described in section 5.1, consists of the identification of VMVP fitting parameters by means of a comprehensive creep curve knowledge bank comprised of ULiège experimental tests and MFC virtual tests. Section 5.2 describes the applications of the multi-scale creep modeling methodology on 800H alloy. An extensive validation involving ULiège experimental creep tests, Post-Assessment Tests (PATs), and creep deflection predictability in a cantilever beam configuration is provided.

This chapter contains a single annex:

 - Annex 5A (confidential) provides all the identified parameters and discloses all curves in a non-normalized form.

Original contributions

Over the course of this PhD. project, which lasted ≈ 4.5 years from January 2021 until June 2026, the following scientific contributions were made:

Peer-reviewed scientific publications

- **Carlos Rojas-Ulloa**, Hélène Morch, Víctor Tuninetti, Jérôme Tchoufang Tchuindjang, Olivier Pensis, Amedeo di Giovanni, Anne Mertens, Laurent Duchêne and Anne Marie Habraken. “Microstructure evolution of Incoloy 800H in industrial environment and correlation with creep mechanisms from literature”. *Materials at High Temperatures*, Vol.41(2), pg.311-321. ISSN: 0960-3409 (Feb. 2024). doi: 10.1080/09603409.2024.2342602
- **Carlos Rojas-Ulloa**, Hélène Morch, Víctor Tuninetti, Laurent Duchêne and Anne Marie Habraken. “Implementation of a modified Graham-Walles viscosity function within a Chaboche viscoplastic constitutive model”. *Computers & Mathematics with Applications*, Vol.155, pg.165-175. ISSN: 0898-1221 (Mar. 2024). doi: 10.1016/j.camwa.2023.12.002
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deformation response in Fe-Ni-Cr alloy". *Journal of Materials Science*. ISSN: 0022-2461, 1573-4803 (June 2026). doi: 10.1007/s10853-026-13121-z

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- **Carlos Rojas-Ulloa**, Hélène Morch, Ehssen Betaieb, Víctor Tuninetti, Laurent Duchêne and Anne Marie Habraken. *Assessment of the influence of creep transition and nitridation in the creep-life prediction of Incoloy 800H*, 8th international conference on Advanced Computational Methods in ENgineering (ACOMEN), August 31st–Septembet 2nd 2022, Liège, Belgium.
- **Carlos Rojas-Ulloa**, Hélène Morch, Víctor Tuninetti, Jérôme Tchoufang Tchuindjang, Anne Mertens, Olivier Pensis, Amedeo di Giovanni, Laurent Duchêne and Anne Marie Habraken *Accurate very-high temperature creep-life prediction of Incoloy 800H addressing effects of creep mechanism transition and nitridation*, 6th international Creep & Fracture conference by the European Creep Collaborative Committee (ECCC), 22nd–25th May 2023, Edimburgh, United Kingdom.
- **Carlos Rojas-Ulloa**, Fan Chen, Víctor Tuninetti, Amedeo di Giovanni, Olivier Pensis, Alexandre Vendramini, Laurent Duchêne and Anne Marie Habraken. *Application of Activation-Function Norton law (AFN) to predict the two-step minima creep deformation observed in Incoloy 800H*, 13th international conference on processing and manufacturing of advanced materials (THERMEC), June 30th–July 4th 2025, Tours, France.
- Fan Chen, **Carlos Rojas-Ulloa**, Jérôme Tchoufang Tchuindjang, Anne Mertens and Anne Marie Habraken. *Multiscale Modeling of Creep Deformation in 30CrMoNiV11-5 Steel: Integrating Mean-Field Theory, Dislocation Dynamics, and Experimental Validation*. 18th European Congress and Exhibition on Advanced Materials and Processes – FEMS (EUROMAT), 14th–18th September 2025, Granada, Spain.
- **Carlos Rojas-Ulloa**, Fan Chen, Jérôme Tchoufang Tchuindjang, Olivier Dedry, Tuan Nguyen-Minh, Anne Mertens, Alexandre Vendramini, Amedeo di Giovanni, Víctor Tuninetti, Laurent Duchêne and Anne Marie Habraken. *Mean-field modeling of the creep deformation response of alloy 1.4959 (800H) addressing precipitate kinetics*. 7th international Creep & Fracture conference by the European Creep Collaborative Committee (ECCC), 18th–20th May 2026, Aix-en-Provence, France.

Poster presentations in international conferences

- **Carlos Rojas-Ulloa**, Fan Chen, Víctor Tuninetti, Amedeo di Giovanni, Olivier Pensis, Laurent Duchêne and Anne Marie Habraken. *Mathematical framework for a semi-physical model for the prediction of creep life of Fe-Ni-Cr alloys addressing solid-solution hardening*, 19th European Mechanics of Materials Conference (EMMC19), 29th–31st May 2025, Madrid, Spain.

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State of the art

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1.1 Creep

Creep in metallic materials is defined as an incremental plastic deformation taking place under a constant mechanical stress in combination with temperatures near or above 0.5 times the melting point temperature T_m of the material in study [1]. The experimental study of creep is conventionally conducted by means of a standard creep test (SCT), where the uniaxial deformation of the material induced by a constant tensile axial load and constant medium or high temperature is continuously monitored. In this controlled experimental environment, the large majority of metallic materials exhibit a classical creep deformation response characterized by 3 consecutive creep stages. From a macromechanical perspective:

- I. **Primary stage:** The initial creep stage is characterized by a continuous hardening behavior. The hardening rate will slowly decrease until a steady-state is reached.
- II. **Secondary stage:** Also known as steady-state creep, it is the stage where the creep deformation rate reaches a constant value held over time. In this generic example, the constant creep rate reached at this state is known as $\dot{\epsilon}_{ss}$ or minimum creep strain rate (**mcr**).
- III. **Tertiary stage:** In this final stage, a continuous creep softening behavior is observed. The consequent loss of the load carrying capacity of the material precedes the fracture of the material.

The visual identification of the classical creep stages in generic creep strain and creep strain rate curves is illustrated in Figure 1.1.

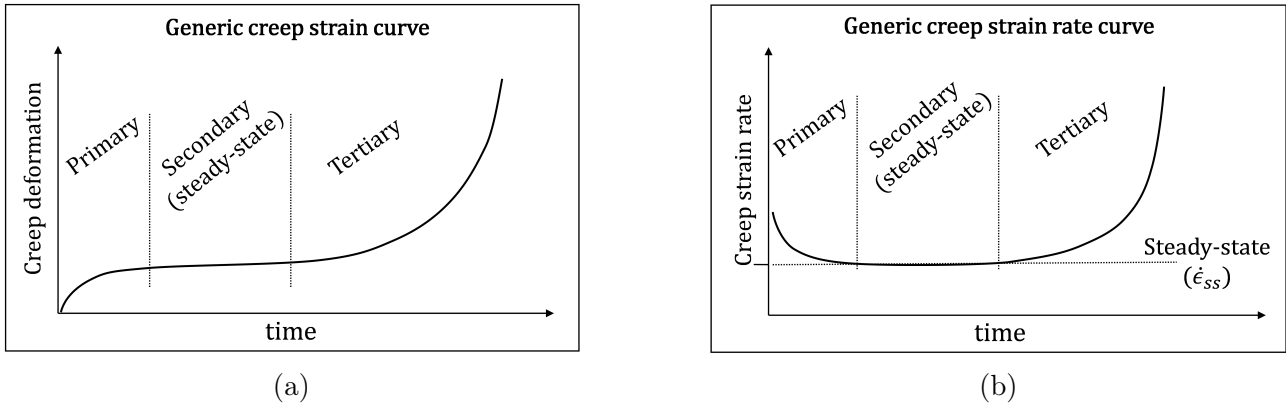


Figure 1.1: Standard (a) creep strain vs. time and (b) creep strain rate vs. time curves. A visual identification of the primary, secondary and tertiary creep stages is given in both cases.

Creep is a consequence of the rearrangement of crystal defects and instabilities induced by external loadings. Here, we provide a brief but comprehensive physical background to better understand the physics of creep.

1.1.1 Physics of creep

The rheological stress-strain relationships describing elasto-plasticity are regarded as the foundation of mechanical engineering [2]. To date, they still provide a reliable mathematical framework and source of inspiration for the development of new material models. Despite their timeless success, their shortcomings became apparent during the early 1900s; they were deemed

unsuitable for the prediction of deformations aided by thermal effects (e.g., creep) and unreliable when modeling plastic deformation of single crystals. The first was approached by developing empirical models (see section 1.1.2), whereas the latter entailed a deeper scientific challenge.

Plastic deformation of single crystals occurs by means of shear throughout preexisting slip planes [3]. In 1905, the Italian scientist Vito Volterra first theorized that such a phenomenon would occur via dislocation movement. According to Frenkel's [4] theory, this crystal shearing would take place in a linear fashion (see Figure 1.2). Compared with experimental observations, the theoretical shear stress calculated under this assumption was orders of magnitude higher [5, 6]. During the 1930s the works of G.I. Taylor [7], supported by those of Polanyi [8] and Orowan [2], challenged Frenkel's theory of crystal shearing, and a reliable physical framework for plasticity was provided. According to Taylor's theory, plastic deformation of crystals takes place in a nonlinear fashion. The rearrangement of atoms throughout the shear plane of the crystal can be interpreted as a wave of crystal defects. The shearing of a crystal by the wave-like displacement for a crystal defect is illustrated in Figure 1.2.

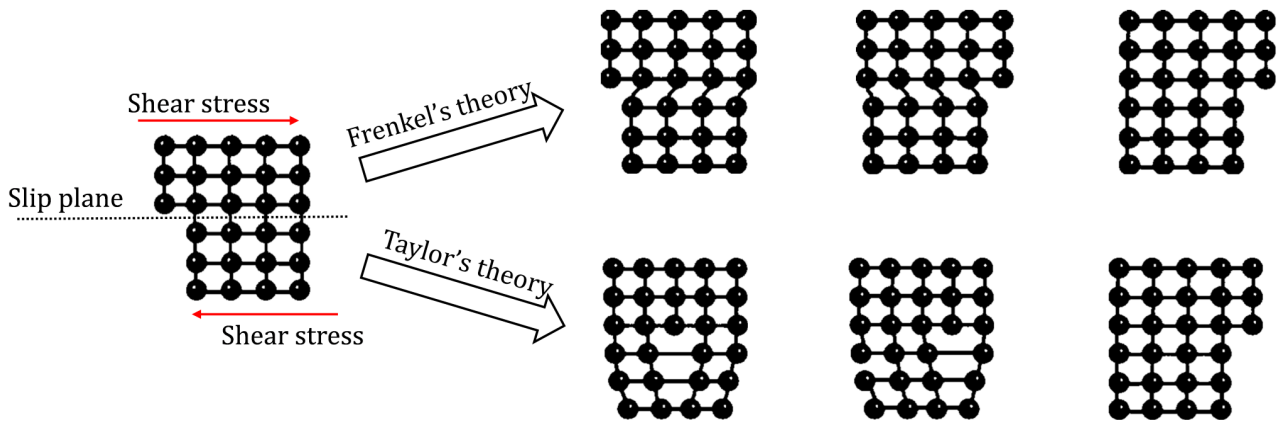


Figure 1.2: Shearing process of a crystal-like atomic arrangement showcasing the differentiation between the outdated linear Frenkel's theory and the currently accepted non-linear Taylor's theory. Figure after [5].

Experimental studies, conducted later on, not only confirmed the deformation mechanism proposed by Taylor but also showed that crystals are inherently imperfect. In other words: despite crystal defects being the mechanism through which materials permanently deform, the existence of these defects is not entitled to the application of an external load. To achieve a deeper understanding of plastic deformation, researchers shifted toward the study of the nature and behavior of crystal defects. Crystal defects can be classified into two main types: vacancies and dislocations.

- **Vacancies** are point-defects found in a bi-dimensional section of a crystallographic atomic arrangement, where the otherwise perfect structure is interrupted by a single missing atom. This forces the rest of the atoms in the plane to accommodate accordingly. In Taylor's crystal shearing model of Figure 1.2, the wave-like moving crystal defect corresponds to a vacancy.
- **Dislocations** are linear defects; projections of point defects where the whole tridimensional crystallographic structure is rearranged to accommodate the defect. To better understand dislocation motion and creep, some important topological definitions are hereafter introduced:

- The plane upon which the atoms are shifted to accommodate the defect is known as the *dislocation plane*.
- The line direction of the dislocation in the plane is defined by the vector $\mathbf{t}$. It is represented by a blue arrow in Figure 1.3.
- The total displacement induced by the dislocation on the projected plane is indicated by the Burger's vector $\mathbf{b}$. It is calculated as the residual vector necessary to close the atom-to-atom circuit around a dislocation, following a methodology similar to that of Ampere's law. Note that in the absence of a dislocation (i.e., no Burger's vector), the atom-to-atom circuit loops illustrated in Figure 1.3 would close.

Dislocations can be classified into two types:

- **Edge dislocations**, where the Burger's vector is perpendicular to the dislocation line vector (i.e., $\mathbf{b} \cdot \mathbf{t} = 0$). As a result, atoms shear in a direction perpendicular to that of the dislocation edge.
- **Screw dislocations**, where the Burger's vector is perpendicular to the dislocation line vector (i.e., $|\mathbf{b} \otimes \mathbf{t}| = 0$). Here, the atoms shear in a direction parallel to that of the dislocation edge.

A graphical representation of an edge and a screw dislocation is provided in Figure 1.3.

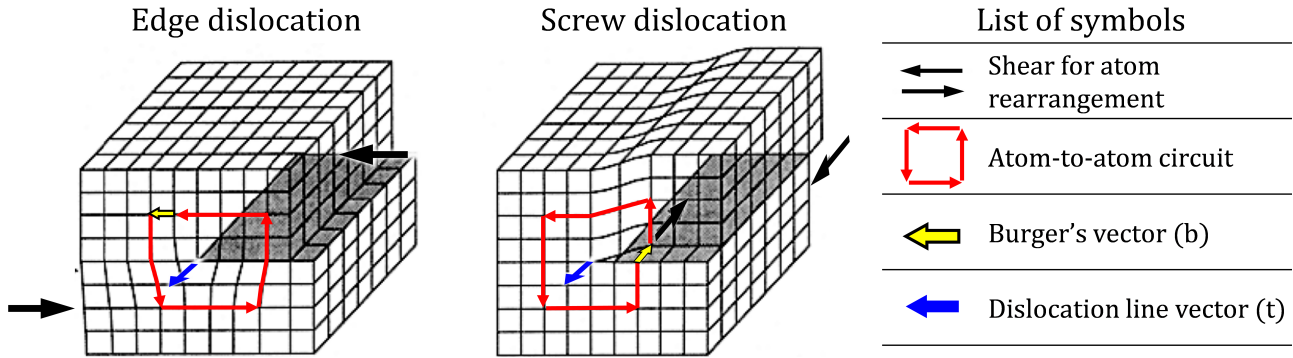


Figure 1.3: Explanatory sketch of an edge (left) and screw (right) dislocation showing their corresponding topological characteristics. Modified from [9].

In conventional plasticity, the motion of dislocations in the system is initiated and driven by the introduction of an external mechanical energy that surpasses the elastic work threshold entitled to the yield stress σ_y of the material. Conversely, creep takes place when the temperature of the system increases to a point where the mechanical energy threshold necessary to initiate the movement of crystal defects (vacancies and/or dislocations) decreases to levels found within or below the elastic limit of the material. To better understand creep, one must understand the process of motion and interaction of the key crystal defects involved in this phenomenon [10–12]: dislocations, vacancies, and (to a lesser extent) grain boundary sliding.

Micromechanical aspects of creep deformation

The influence of temperature and stress on the creep deformation response and fracture of metallic materials was thoroughly reported (see, for instance, the review articles of [13, 14]). To address complex non-linear relationship between creep deformation and stress-temperature loading, the concept of a creep mechanism was adopted. It defines the predominant combination of microscopic deformation phenomena driving creep deformation for a given thermomechanical loading. While some authors were adamant in identifying several co-existing

creep mechanisms (see for instance [15]), four main creep mechanisms are generally accepted in the field: Low-temperature creep (LTc), High-temperature creep (HTc), Coble creep (Cc) and Nabarro-Herring creep (NHc). A brief summary of the theoretical aspects of each mechanism is provided hereafter:

- LTc takes place at high stress and low temperatures. Given that the thermal energy of the system is not sufficiently high to activate dislocation motion, creep is attributed to the diffusion of atom vacancies through dislocation lines (i.e., pipe diffusion).
- HTc occurs at high stress and high temperatures. Under this high-energy configuration, creep deformation is the result of dislocation gliding through their respective slip planes. In addition, dislocations are able to surmount obstacles (e.g., particles) by climbing to a nearby slip plane. The process of dislocation climbing is attributed to vacancy diffusion (i.e., transmission of atoms in exchange for vacancies) occurring between both slip planes [14, 16].
- Cc is observed under high stresses and low temperatures. The deformation of crystals here is attributed to a predominantly diffusional process, where the atom-vacancy current originates in the bulk of the grain [17].
- NHc occurs under low-stress and low temperature loadings. At this low-energy state, creep deformation is purely diffusional. However, in contrast with Cc, diffusion takes place predominantly at the grain boundary (Gb) level [18, 19].

An illustrative representation of each creep mechanism is provided in Figure 1.4.

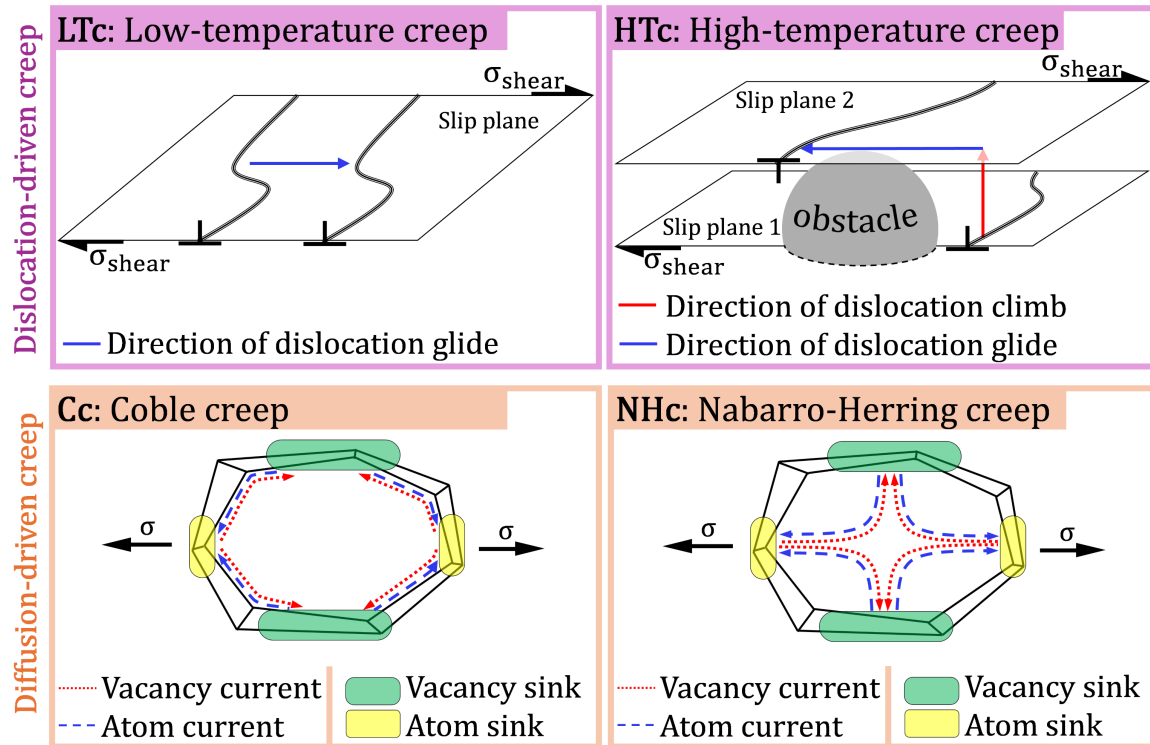


Figure 1.4: Graphical scheme showing the underlying physical aspects involved in each creep deformation mechanism. Reproduced from [20].

Depending on the stress and temperature, the creep deformation of a material can be predominantly driven by any of the four creep mechanisms described in Figure 1.4. Under this

premise, the stress-temperature dependent area of dominion of each mechanism can be analytically mapped for a given material. The resulting diagram is known as a Deformation Mechanism Map (DMM) [21–24], or "Ashby Map" (despite the work being officially presented in the context of Harold J. Frost's PhD. thesis [25]). The original book [23] is difficult to obtain, however an online version summarizing the content of the book in a nicely organized manner has been made publicly available in [26]. A generic DMM is shown here in Figure 1.5a. The area is defined as function of the shear stress σ_{shear} normalized by the G , and the homologous temperature (i.e., temperature normalized by T_m).

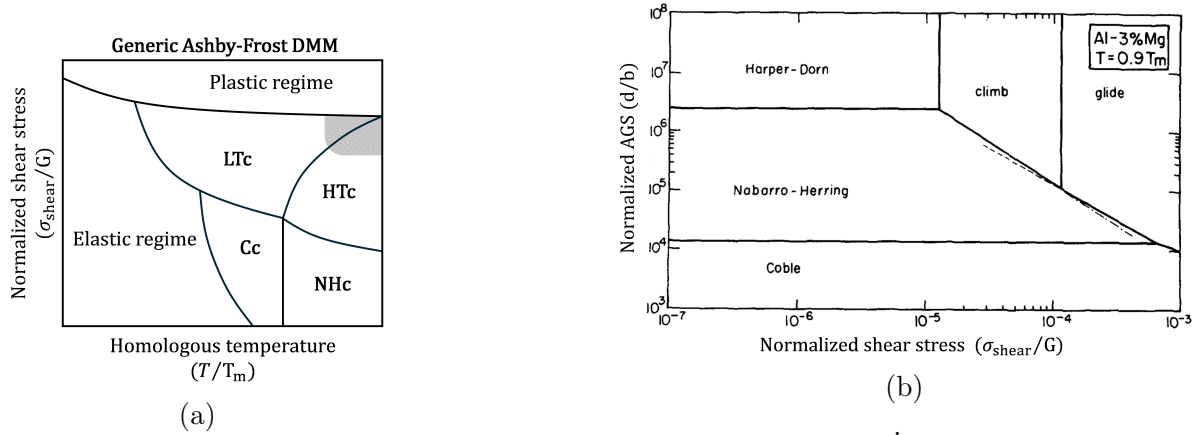


Figure 1.5: (a) Generic DMM according to H.J. Frost & M.F. Ashby [23]. The darkened upper right corner indicates Dynamic Recrystallization (DRX). (b) Alternative DMM depicting the predominant creep mechanisms for Al-3%Mg alloy for $T=0.9T_m$. After F.A. Mohammed & L.T. Langdon [27].

The DMM approach is ultimately defined by the material properties, microstructural features, and the numerical model chosen to describe each deformation mechanism [28]. An interesting application of the DMM approach is shown in [29], where the parameters of the semi-physical equations chosen to describe the $\dot{\epsilon}_{ss}$ of several alloy 800H experimental curves are identified by a genetic algorithm (GA). Given the huge influence of the average grain size (AGS) on the creep response of the alloy, the authors provide one DMM for each different AGS. In a similar fashion, [27] provides an alternative DMM intended for use in solid-solution alloys. It differs from Frost and Ashby's version by mapping the deformation mechanisms onto a normalized AGS v/s normalized shear stress diagram for a single temperature. An example of the latter is provided in Figure 1.5b.

According to [30], the creep behavior of materials for a given thermomechanical loading is a consequence of the combined effects of three main types of internal properties: the *initial creep resistance* (referring to the as-received properties of the material), the *latent creep resistance* (grouping all non-permanent properties), and *overaging or spheroidization of precipitates* (addressing the precipitate coarsening effects of long-term high-temperature exposure). This vision was the basis to further understand the strong influence of the microstructure of a material on the creep deformation response. A brief summary of the main microstructural features and their influence on the motion of dislocations and diffusion of vacancies during creep is listed hereafter:

- **Crystal lattice:** The crystal lattice is considered a permanent property of the material, and is not included within numerical analyses. Nevertheless, it should be noted that the crystal lattice plays an important role in creep resistance. For instance, crystal lattices such as face centered cube (FCC) and hexagonal close-packed (HCP) materials are known to provide high resistance to vacancy diffusion due to the dense packing of their

crystal lattice. In general, austenitic (FCC) and HCP alloys often exhibit a greater creep resistance than body centered cube (BCC) alloys.

- **Average grain size (AGS):** Grains are the largest group of neighboring crystals with a common orientation, and their boundaries act as both dislocation/vacancy sinks or sources. Therefore, the dislocation motion and vacancy diffusion are conditioned by the AGS of the material. From a broader perspective, the AGS can be considered an indicator of the average glide distance of dislocations (i.e., large AGS entail larger creep ductility and thus longer creep life).
- **Grain boundaries:** As indicated in the illustration of Figure 1.4, Gbs can act as sinks, sources, or channels for the motion of dislocations or diffusion of vacancies, thus greatly influencing creep deformation. Additionally, creep damage and fracture take place at the Gb level, further highlighting the role of Gbs.
- **Precipitates:** The role of precipitates during creep is greatly determined by their hardness (e.g., composition) and size. Small precipitates (~ 50 nm) tend to act as dislocation obstacles [31, 32], whereas large precipitates (> 1 μm) act as dislocation sources due to the non-coherent precipitate-matrix boundary. Due to their inherent presence in as-received condition, and their susceptibility to coarsening or diffusion as long-term high temperature exposure occurs, they play an important role in both the permanent and latent creep properties of materials. When found at the Gb, they can facilitate the onset of damage via grain boundary sliding.
- **Solute atoms:** The vacancy current generated during diffusion-driven deformation mechanisms entails an equivalent and opposite current of solute atoms. Therefore, interstitial (small, often C, N) and substitutional (larger, such as V, Ti, Mo, etc.) atoms embedded into the material matrix play an essential role in creep. In addition, materials with a high density of dissolved atoms (known in the literature as solid-solution alloys) are reported to exhibit a strengthening effect known as solid-solution hardening (SSH). In physical terms, the latter is explained as the presence of the atom cloud (Cottrell atmosphere [33]) hindering the motion of dislocations. This dislocation dragging effect can also be observed at the initiation of the plastic regime during high-temperature tensile tests, where the stress-strain curves exhibit a somewhat erratic hardening/softening response known as Dynamic Strain Aging (DSA) [34].
- **Dislocation walls:** A DW is any type of boundary that hinders the normal flow of dislocations. While Gbs are known to act as dislocation barriers, a single grain can be subdivided into progressively smaller sections, each having smaller misorientation angles with its neighbors (see Figure 1.6). The smallest section known to act as a barrier to dislocation motion is the subgrain, exhibiting misorientation angles in the vicinity of $\leq 5^\circ$, and their size is often found in the range of a < 1 μm . Regardless of their small size and low misorientation angle, they are a key factor determining the creep ductility of the material.

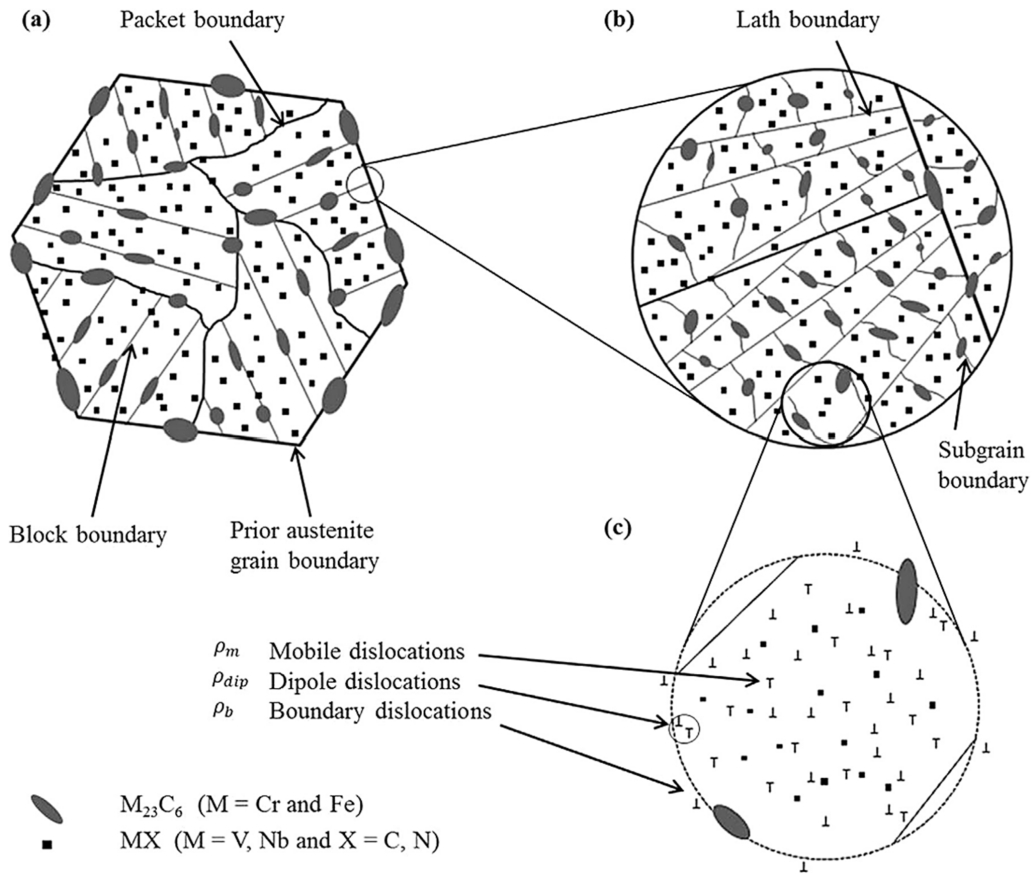
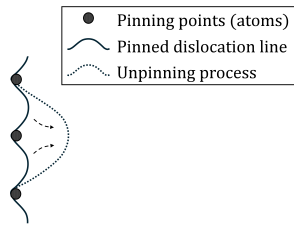
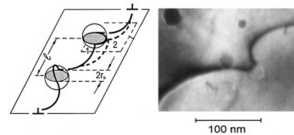
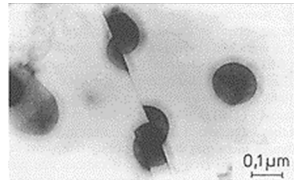
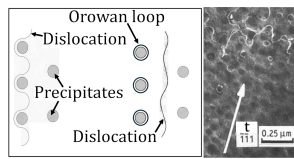
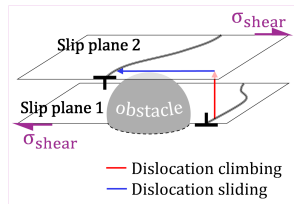


Figure 1.6: Identification of the different types of dislocation walls encountered in metallic materials at different length scales. From largest to smallest: grain boundary (Gb) > packet > block > lath > subgrain. Illustration reproduced from [35].

Ultimately, creep deformation is largely determined by the interaction between crystal defects and the material microstructure. A brief summary of the main interactions occurring between dislocations, precipitates, particles (i.e., solute atoms), and dislocation walls is provided in Table 1.1.

Table 1.1: Summary of dislocation-dislocation (**DD**), dislocation-atom (**DA**) and dislocation-precipitate (**DP**) interaction phenomena contributing to the deformation response of crystalline materials.

Phenomena	Description	Figure
DD: Dislocation annihilation	Dislocations topologically opposite to each other (i.e., same t and different b , or vice versa) attract each other because of their associated stress fields (see [36]). Upon meeting as they glide in the same slip plane, segments of each dislocation will merge and mutually annihilate. The climbing of dislocations (changing slip planes) raises the probability of annihilation.	Figure after [36].

Phenomena	Description	Figure
DA: Dislocation pinning (by atoms)	The atoms found in solid-solution state within the crystal lattice of the material hinder the normal glide of a dislocation if both are found in the same slip plane. This phenomenon is known as SSH. A pinned dislocation will remain immobilized until the system reaches a certain thermomechanical energy threshold that allows the dislocation to surmount the obstacle (atom).	
DP: Dislocation pinning (by precipitates)	Small precipitates act as obstacles that hinder the otherwise normal glide of dislocations. In contrast to the atom-pinning process reviewed here, precipitates are surmounted via precipitate cutting, dislocation loop formation, or climbing. These mechanisms are briefly reviewed below.	 Adapted from [37].
DP: Cutting of precipitates	In the presence of low-strength precipitates (such as γ' phase precipitates, $\text{Ni}_3(\text{Al}, \text{Ti})$), and in addition to high mechanical loadings, dislocations can get free from the precipitates by cutting through the particle.	 Figure after [11].
DP: Dislocation loops (Orowan loops)	When precipitates are of considerable hardness, and in combination with high thermomechanical energy in the system, dislocations can glide around the obstacle. In such cases, a section of the dislocation becomes static as a dipole encircling the precipitate, forming what is known as an <i>Orowan loop</i> . The rest of the dislocation line is granted free movement afterwards.	 Sketch after [36]. Transmission electron microscopy (TEM) micrograph after [38].
DP/DD: Dislocation climb	Let us picture a dislocation being pinned by a hard precipitate located on its slip plane. Under high-temperature conditions, dislocations can surmount this obstacle by climbing to a neighboring slip plane from which it can continue gliding. The dislocation climbs towards the objective slip plane by means of vacancy diffusion, interacting with neighboring crystal defects by either giving or incorporating vacancies. In the event of two neighboring dislocations attempting to climb their respective slip planes, they can climb by generating a vacancy current between each other; this phenomenon is known as cross-slip.	 Cross-slip process involving dislocation climb during high-temperature creep. Sketch reproduced after [20].

The above physical explanation of creep deformation allows for a deeper understanding of

the macroscopic deformation response illustrated in Figure 1.1. It is an entangled process of movement (glide + climb), generation, pinning and annihilation of dislocations. Within Figure 1.1, from a micromechanical perspective:

- I. **Primary stage:** The high initial creep rate is attributed to the massive initiation of dislocation movement as they are generated at dislocation sources (e.g., large precipitates or grain boundaries). The creep hardening process observed herein is a consequence of the slowly decreasing number of free dislocations as these begin to reach obstacles in their path (e.g., particles, dislocation walls, and other dislocations).
- II. **Secondary stage:** The dislocation-obstacle interaction continues until the rate of dislocation generation is roughly matched by the rate of dislocations being stopped by surrounding obstacles.
- III. **Tertiary stage:** After a certain level of deformation, the material will eventually exhibit a loss of load-carrying capacity attributed to the onset, growth and coalescence of voids. The creep damage process consists in the generation of intergranular cavities aided by the accumulation of vacancies, spheroidization of precipitates and grain boundary sliding (GBS). The progressive increment and coalescence of such cavities are known to lead to the fracture of the material.

Creep damage and fracture

As mentioned above, the sustained softening behavior observed within the tertiary creep stage is consequence of the initiation and accumulation of damage in the material. This causes a progressive loss of load carrying capacity leading towards the fracture of the material. In contrast to conventional plastic deformation, creep damage is initiated predominantly at Gbs. Other cases include damage initiation by material decohesion at large particles, triple joints, or a combination of them. An explanatory illustration of the different damage initiation mechanisms on a polycrystalline material is given in Figure 1.7.

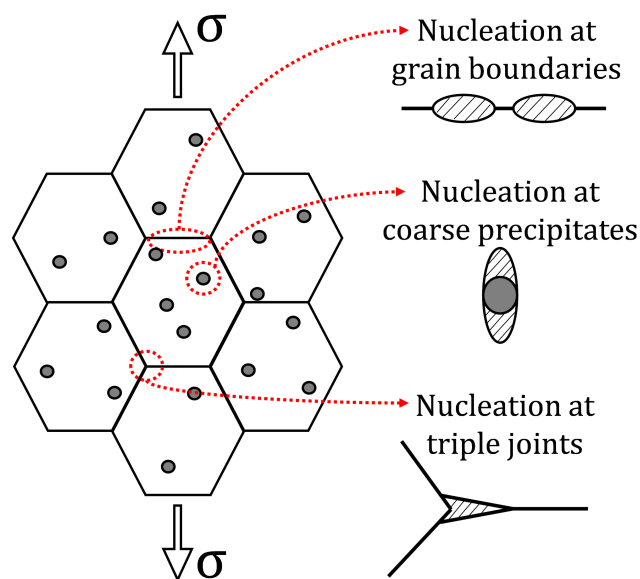


Figure 1.7: Damage initiation or void nucleation mechanisms during creep in polycrystalline materials.

1.1.2 Empirical modeling of creep

The energy level and time-scale at which creep deformations occur differ drastically from those of conventional plasticity. Knowledge of the stress and temperature for creep to take place was soon deemed insufficient for the prediction of the complex non-linear deformation response associated with the phenomenon. The lack of physical understanding during the early 1900s led researchers to consider creep as a time-dependent type of deformation (hence the use of *viscoplasticity* as a synonym). A first empirical model was provided by [39], where the creep strain p is calculated as the summation of time-dependent primary (p_I) and secondary (p_{II}) creep strain functions. The model is presented in Eq. 1.1, where α_A , m_A and β_A are material constants ($m_A < 1$).

$$p = p_I + p_{II} = \alpha_A t^{m_A} + \beta_A t \quad (1.1)$$

Approaches addressing time, temperature and stress dependency were thereafter explored. Good compilations of the most well-known creep models from a historical perspective are given in [40, 41].

Norton law

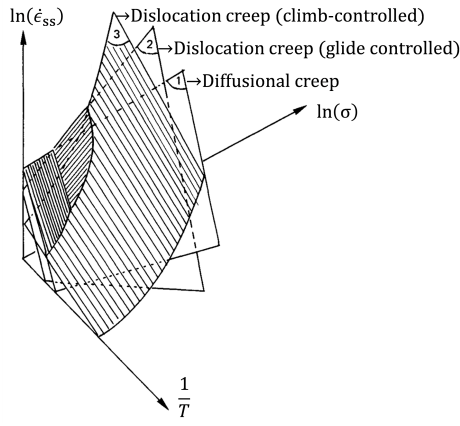
The first well-established creep model was presented by H.F. Norton in his book of 1929 [42]. The Norton power law (or simply *Norton law*) provides a well-substantiated empirical relationship where temperature-dependency is given by an Arrhenius equation (i.e., a temperature-dependent activation energy is imposed for creep to occur). The formula is presented here in Eq. 1.2, where R is the gas constant, T is the temperature (Kelvin), and A_N , n_N , m_N and Q are material constants.

$$p = A_N \sigma^{n_N} \exp\left(\frac{-Q_c}{RT}\right) t^{m_N} \quad (1.2)$$

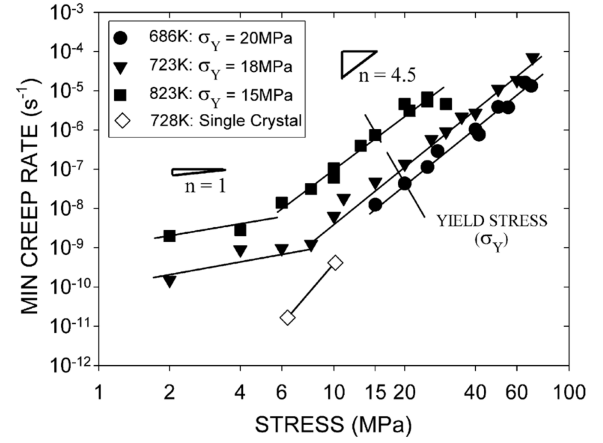
A more generalized version of the Norton law can be obtained by rearranging the terms of the time derivative of Eq.1.2 to recover the empirical power-law stress- $\dot{\epsilon}_{ss}$ relationship observed for crystalline materials in experimental conditions (primary and secondary creep stages, see Figure 1.1). The equation showcasing this relationship is presented in Eq.1.3, where $K(T)$ is the temperature-dependent drag stress (usually an Arrhenius-type function) and n is the power-law exponent.

$$\dot{\epsilon}_{ss} = \left(\frac{\sigma}{K(T)}\right)^n \quad (1.3)$$

The inherent simplicity of Eq.1.3 has made it a popular choice for creep characterization in metallic materials. To take into account the sudden slope change in the σ - $\dot{\epsilon}_{ss}$ relationship observed in some materials after a certain stress threshold is reached, different n_n exponents were identified for different temperature ranges. The latter assumes the existence of a creep mechanism change (a concept introduced in Figure 1.5). A tri-dimensional illustration showing the σ - T - $\dot{\epsilon}_{ss}$ relationship on which the Norton power law is based is provided in Figure 1.8a. The experimental σ - $\dot{\epsilon}_{ss}$ dependency of polycrystalline copper for different temperatures together with their respective Norton exponents fitted is shown in Figure 1.8b.



(a) Generic illustration of the $\sigma - T - \dot{\epsilon}_{ss}$ observed in metallic materials. After [43].



(b) Stress-dependency of $\dot{\epsilon}_{ss}$ for polycrystalline copper. After [44].

Figure 1.8: Empirical experimental temperature-stress- $\dot{\epsilon}_{ss}$ supporting the empirical relationship proposed in the Norton creep law.

Bird-Mukherjee-Dorn (BMD) model

The creep models visited up to this point consider that macromechanical creep deformation is entitled to macro-scale physical variables, used to determine the creep deformation response of a given material. However, with consideration of the creep micromechanics facts presented previously in section 1.1.1, it is clear that accurate creep deformation response calculations entail knowledge and consideration of the physical features involved. Following this line of inquiry, the first well-established semi-physical creep model was developed by [14]. The BMD model is presented in Eq.(1.4), where D_0 is a diffusion coefficient, G is the shear modulus, k_B is the Boltzmann constant, d is the mean grain diameter, ΔQ is an activation energy, and A , p and n are fitting parameters.

$$\dot{\epsilon}_{ss} = \frac{AD_0Gb}{k_BT} \left(\frac{b}{d}\right)^p \left(\frac{\sigma}{G}\right)^n \exp\left(\frac{-\Delta Q}{k_BT}\right) \quad (1.4)$$

The BMD creep model was regarded as a good compromise between physicality and phenomenology. It established a link between the $\dot{\epsilon}_{ss}$ of a material and key deterministic physical features on the macro (e.g., G or σ) and micro-scale (e.g., b , d), with consideration of few fitting parameters. It was the reinterpretation of the known creep mechanisms (see Figure 1.4) within the BMD model framework that allowed researchers H.J. Frost & M.F. Ashby [23] to develop their DMM theory (see Figure 1.5).

Graham-Walles equation

Calculation of the creep deformation of metallic components requires a reliable model capable of providing an accurate representation of the total creep curve (addressing all three creep stages until fracture). In 1953, A. Graham and K.F. Wallis [45] proposed a simple phenomenological approach where the total creep curve is calculated as a sum of different functions, each function addressing a different hardening or softening creep behavior. A graphical representation of this method applied on a classic 3-stage creep curve is shown in Figure 1.9. The original form of the Graham-Walles creep model is presented here in Eq.1.4, where K_i , α_i and β_i are material constants, and nVP is the total number of functions.

$$p = \sum_{i=1}^{nVP} K_i \sigma^{\alpha_i} t^{\beta_i} \quad (1.5)$$

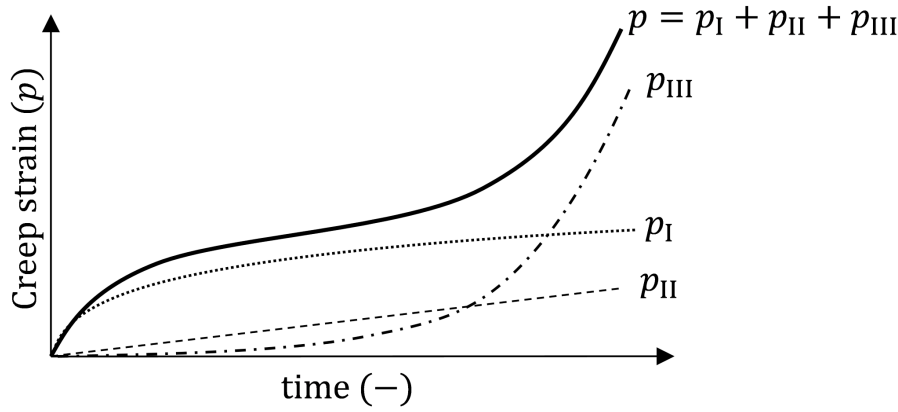


Figure 1.9: Graphical description of the Graham-Walles method for calculating the total creep curve p as the result of the sum of different functions (p_I , p_{II} and p_{III})

The creep function-summation principle from Figure 1.9, currently known as the Graham-Walles method [46], served as the foundation for the development of other popular creep models such as the Barch model [47], the Garofalo model [48], or the Theta model [49]. Of particular interest for this work is the modified Graham-Walles (mGW) creep model, where the empirical time-dependency of the original model (see Eq.1.5) is substituted by physical terms. The mGW creep model, presented in Eq.1.6, consists of a non-linear expression where the creep strain rate $\dot{p}$ is calculated as function of the creep strain p , and the temperature dependency is given by an Arrhenius-type expression. K_i , α_i , β_i and C_i are material constants.

$$\dot{p} = \sum_{i=1}^{nVP} K_i \exp\left(\frac{T}{C_i}\right) \sigma^{\alpha_i} p^{\beta_i} \quad (1.6)$$

However, to predict the creep deformation response of materials, the prevailing objective of creep modeling is to provide reliable numerical tools to evaluate the creep performance of materials. In the context of mechanical design, the creep resistance of materials is often evaluated in the form of bidimensional diagrams where the time to reach a certain level of creep deformation under constant temperature and stress is mapped. The most popular of such diagrams is the time-to-rupture (t_r) diagram.

Creep time-to-rupture extrapolation

t_r diagrams are often regarded as a reliable indicator of the creep resistance of materials. Given the large time-scale whereupon creep is developed, the prediction of the t_r is a challenge by itself. A pioneering approach was proposed by [50] based on the assumption that damage across different creep loading combinations (stress and temperature) could be simply added proportionally to the t_r at that load. This model, known as the Life fraction rule (LFR), is presented in Eq.1.7, where t_i is the time spent under the i^{th} loading, and $t_{r,i}$ is the t_r of the material at the i^{th} loading.

$$\sum_i \frac{t_i}{t_{r,i}} = 1 \quad (1.7)$$

The reliability of the proportional damage addition approach shown in Eq.1.7 is reportedly conditioned to the loading levels of each case. Damage accumulation prediction when different stress levels (i.e., different creep mechanisms) are considered was deemed by [51]. In addition, the remaining creep life of cast pipe steel components calculated using the LFR was consistently overestimated in cases where no prior damage (pure aging) was present, whereas that of components with previous damage was greatly overestimated [52].

The Monckman-Grant relationship

An empirical rule relating the t_r and the $\dot{\epsilon}_{ss}$ of a material was first discovered by F.C. Monckmann and N.J. Grant [53]. The original approach, popularly known as the Monckman-Grant relationship (MGR), consists of a simple inverse proportional relationship to fit the mapping of both quantities obtained under experimental conditions for a single material at different temperatures and stress loadings. The approach was successful in that it allowed researchers to explore more physical t_r extrapolation methods. Additionally, the original MGR has since been generalized by [54], thus allowing its use for a large number of materials. The form of the relationship in its original (left) and generalized (right) forms is given in Eq.(1.8), where α and β are material constants.

$$\dot{\epsilon}_{ss} \cdot t_r = Constant \quad \text{or} \quad t_r = \alpha \cdot (\dot{\epsilon}_{ss})^\beta \quad (1.8)$$

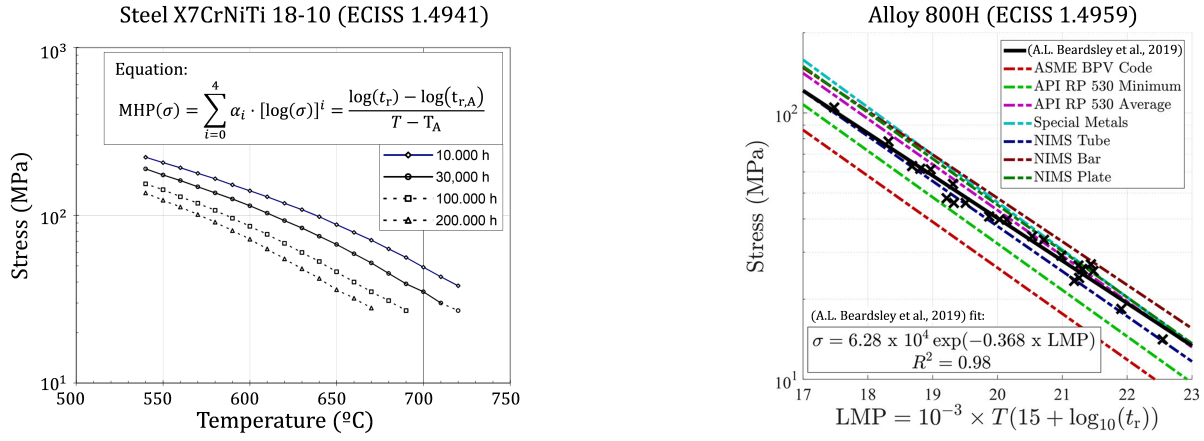
Time-to-rupture (t_r) diagrams

The mathematical framework for developing a t_r model consists in finding a function of the form $f(\sigma, T, t_r) = Constant$. The t_r of the material can be reliably mapped with knowledge of the thermal T and mechanical σ crepe loadings. The Larson-Miller Parameter (LMP) [55], the Manson-Haferd Parameter (MHP) [56], and the Manson-Brown Parameter (MBP) [57] are found among the most popular approaches. The original form of the equations describing the LMP and the MHP are introduced in Eq.(1.9) and Eq.(1.10) respectively.

$$f_{LMP}(\sigma) = 10^{-3} \cdot T_{abs} \cdot [C + \log_{10}(t_r)] \quad (1.9)$$

$$f_{MHP}(\sigma) = \frac{\log(t_r) - \log(t_{r,A})}{T - T_A} \quad (1.10)$$

In Eq.(1.9), $f_{LMP}(\sigma)$ is a stress function, C is a material constant, and T_{abs} is the absolute temperature. In Eq.(1.10), $f_{MHP}(\sigma)$ is a stress-dependent function, whereas $t_{r,A}$ and T_A are the t_r and the temperature for a given linear intercept A , respectively. In summary, the LMP represents an inversely proportional logarithmic relationship, whereas the MHP represents a simpler linear relationship. The form of the stress-dependent function is often regarded as arbitrarily chosen, taking care of fitting the experimental behavior of the material observed. Common examples of functions are power laws [58], exponential [29], or multiple degree polynomials [59]. Examples of t_r diagrams built using the MHP and the LMP are given in Figure 1.10a and Figure 1.10b respectively.



(a) Application of MHP to build a t_r diagram with a polynomial stress function. After [59].

(b) Application of LMP to build a t_r diagram with an exponential stress function. After [29].

Figure 1.10: Time-to-rupture diagrams built using (a) the Manson-Haferd Parameter (MHP) and (b) the Larson-Miller Parameter (LMP).

1.1.3 Semi-physical modeling of creep

According to [12, 60], numerical creep models can be classified into three main categories according to the modeling approach:

- **Empirical models**, rely on empirical experimental correlations found between two or more state variables. They attain a very simplified but effective way to understand creep deformation from a phenomenological perspective, with minimal assessment of physics. Such is the case of the MGR, the Andrade creep model [39], or the Norton creep law seen in Eq.(1.3).
- **Semi-physical models** incorporate functions and parameters representing physical phenomena. They often rely on statistically representative quantities founded in thermodynamic principles. For example, models such as Bergström's [61] or Ghoniem's [62] consider inelastic deformations to be proportional to variations in dislocation forest densities in the material. Equations representing mechanistic interpretations of physical phenomena often include fitting parameters to improve the model accuracy.
- **Physical models** focus on the prediction of phenomena from a purely deterministic standpoint. In the context of material modeling, these models reduce plasticity to the smallest scale possible; dislocation motion (climb, glide and interaction with surrounding media). This scale is studied in atomistic simulations as seen in the PhD. thesis/book of Dr. Thomas D. Swinburne [5].

The discrete classification provided here above illustrates the existence of semi-physical models within a simplified context. In practice, numerical models form a rather continuum distribution across all length scales as seen in Figure 1.11a. There is no perfect model, and choosing one or another is a matter of balancing aspects such as computational efficiency, parameter uncertainty and model accuracy. An illustrative overview of the uncertainty level of the experiment, model form and model parameters across different length scales is offered in Figure 1.11b.

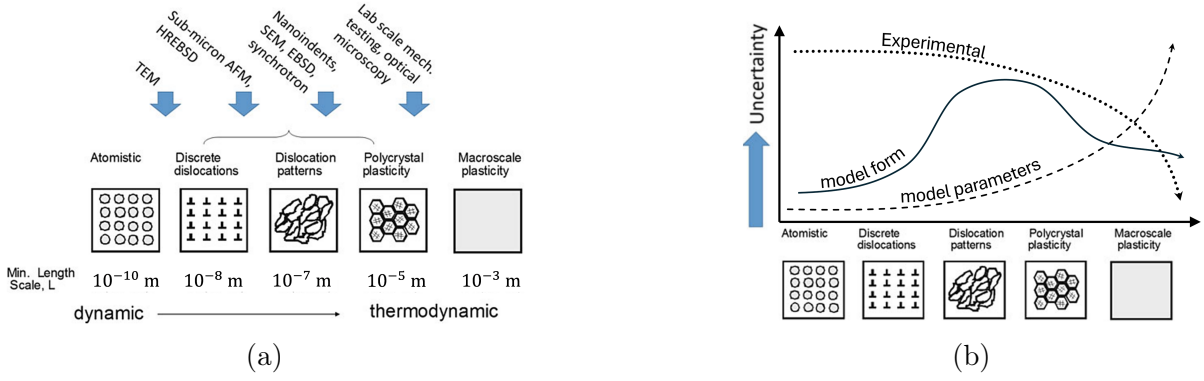


Figure 1.11: (a) Length scale hierarchy of metal plasticity models and (b) uncertainty level of experiment, model form and model parameters in relation to the length scale. After [63].

Given the high relevance of dislocation density-based semi-physical creep models in the context of this PhD. project, this family of models is hereafter introduced following a logical and historical perspective. A general theoretical background is first provided, summarizing the building blocks of this family of models. Later, two models relevant for this thesis are introduced: the Kocks-Mecking-Estrin models, and the Ghoniem-type models.

General theoretical background

Semi-physical creep models stem from a mechanistic interpretation of the concept of hardening: the stress-strain relationship described by a material during inelastic deformation regimes. Conventional hardening models such as Voce [64] and Swift [65] laws recover the isothermal quasi-static stress-strain relationship of materials based on phenomenology. The hardening definition is expressed in Eq. (1.11). Formulations incorporating strain rate and temperature dependency were for instance adopted within the Johnson-Cook hardening law family [66]. Hardening models incorporating Continuum Damage Mechanics (CDM) approaches have also been explored [67, 68].

$$\Theta = \left. \frac{\partial \sigma}{\partial \epsilon} \right|_{\dot{\epsilon}, T} \quad (1.11)$$

In contrast, dislocation density-based hardening models describe the stress-strain relationship as function of the movement and accumulation of crystal defects following Flick's theory (see section 1.1.1). Two key formulations stem from this principle: the strain-hardening Orowan equation and the stress-hardening Taylor equation. The Orowan equation [2], shown in Eq. (1.12a), describes the local resolved shear strain rate ($\dot{\gamma}$) as a function of the mean dislocation density (ρ), the Burgers' vector (b), and the mean glide velocity of dislocations (v). The Bailey-Hirsch law [69], better known as the "Taylor equation", describes the directly proportional relationship between the critical resolved shear stress (τ) and the square root of ρ . The mathematical formula is presented in Eq.(1.12b), where α is considered a geometric constant in athermal conditions (i.e., $\dot{\epsilon} = \text{constant}$ and $\dot{T} = 0$).

$$\dot{\gamma} = \rho b v \quad (1.12a)$$

$$\tau = \alpha G b \sqrt{\rho} \quad (1.12b)$$

The hardening formulations presented in Eq.(1.12) showcase explicit dependence on a statistically representative physical variable. However, in this state, they only provide a snapshot of

the mechanical response of the material. For instance, Eq.(1.12a) computes the $\dot{\epsilon}_{ss}$ for a given ρ and v , whereas Eq. (1.12b) recovers the yielding point of a material for a given ρ . A full semi-physical model, capable of predicting the time-dependent deformation response of a material, requires at least one equation describing the evolution of ρ . Inspired by the theoretical works of A.H. Cottrell [33, 70] on dislocation-driven plasticity, an expression describing ρ dependency on inelastic deformations was first proposed by Y. Bergström [61]. The generalized formula is shown in Eq.(1.13), where ρ_0 is the initial dislocation density and $\Delta\rho$ represents the variation in ρ as a consequence of positive ($\rho^{(+)}$, such as dislocation production and multiplication) and negative ($\rho^{(-)}$, such as recovery/annihilation and immobilization) contributions.

$$\rho = \rho_0 + \Delta\rho \quad \text{where} \quad \Delta\rho = \sum \Delta\rho^{(+)} - \sum \Delta\rho^{(-)} \quad (1.13)$$

The equation system formed by combining any of Eq.(1.12) with Eq.(1.13) is the working principle of a ρ -based mean field model. Together, these equations compute the inelastic stress-strain response of materials based on mathematical expressions representing physical interactions between dislocations and the material microstructure.

To consolidate a ρ -based mean-field (MF) model, one must establish the physical characteristics of the ρ system, namely (but not limited to), the physical dislocation-microstructure interaction phenomena, and the ρ types. A brief description of these two model features is provided below.

- **Physical phenomena** involving the increment (+) or decrement (-) of ρ are incorporated into the dislocation-density equation in a similar fashion to that described in Eq.(1.13). These terms affect the stress-strain response depending on their magnitude, sign, and ρ type. Formulae addressing phenomena such as dislocation multiplication (+), annihilation (-), or others (see for instance DD, DA and DP in Table 1.1), are presented later in the context of two fundamental model types.
- **Dislocation density types** allows MF models to better capture the complexity of dislocation interactions. For instance, the differentiation between mobile and immobile dislocations allows MF models to recover the physical effects entitled to the interactions between both dislocation types [71], as opposed to single ρ models where immobile dislocations are just subtracted from the mobile type. The following list presents the most popular dislocation density types used in MF models.
 - **Mean mobile dislocation density** (ρ_m): Inelastic deformations are actively driven by this ρ type. The ρ magnitude in both the stress and strain hardening models presented in Eq.(1.12) are in FCC materials function of ρ_m (according to [72], BCC materials exhibit an additional dependency on the static dislocations).
 - **Mean static dislocation density** (ρ_s): Also known as immobile dislocations, these crystal defects are found in a stationary state forming a closed loop with itself (i.e., a stable configuration). They are formed, for instance, from ρ_m becoming immobilized during gliding (e.g., after encountering a precipitate, another dislocation of equal b and t , or after the over-extension of a jog belonging to a screw dislocation). An in-situ TEM analysis capturing the formation of a static dislocation from a screw dislocation is shown in Figure 1.12.

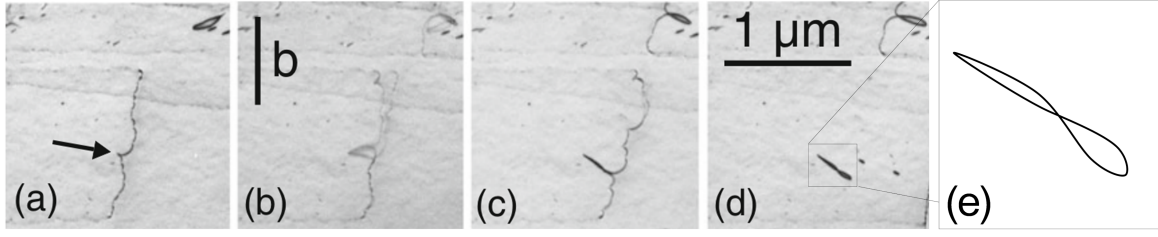


Figure 1.12: Sequence (a→b→c→d) of TEM images capturing the formation of a static dislocation from the overextended jog of a screw dislocation in a MgO crystal. After [71, 73]. (e) Illustration sketch to better visualize the dislocation loop.

While ρ_s do not participate actively from the inelastic deformation process, they do interact with ρ_m . A summary of the possible outcomes from the encountering of these two dislocation types is provided in section 3.1 (see Figure 3.1) of this thesis.

- **Mean boundary dislocation density (ρ_b):** They are found embedded within the crystal defects inherent to the misorientation found between substructure units; in other words, they are the structural component of DWs, from grain to subgrain boundaries, see Figure 1.6. Given the energy minimization process entitled to the formation of DWs, ρ_b are considered stationary and do not participate in the inelastic deformation process. However, their quantization can be relevant to better estimate the total number of dislocations, to compute the DW size variation in the form of subgrain boundary growth [62], or to estimate the conditions for a DRX process [74–76].

Each new ρ type included in the model requires a new equation describing their evolution and interdependency to other state variables. The combination of ρ types and equations is defined based on the material properties and the modeling target. In continuum models (i.e., intended for the prediction of full curves), this often leads to a non linear system of equations. This mathematical problem is analogous to a system of consecutive water tanks or predator-prey cycles [77]. An illustrative example is shown in the European Creep Collaborative Committee (ECCC) conference presentation by C. Rojas-Ulloa et al.[78].

The high versatility of the MF approach has allowed scientists to implement ρ -model solutions for the prediction of inelastic deformations in multiple material systems and conditions; a creative process limited only by physics and computational aspects. In the context of creep modeling, two MF model families stand out as capable of providing microstructure-dependent creep curves: the Kocks-Mecking-Estrin models and the Ghoniem-type models. While they are presented hereafter in chronological order, both are equally relevant among the scientific community.

The Kocks-Mecking-Estrin models

The Kocks-Mecking-Estrin (KME) family of models describes the continuum inelastic response of materials using a single ρ equation. The general form of the model, named after the individual contributions of U.F. Kocks, H. Mecking and Y. Estrin, is presented in Eq.(1.14), where h is a hardening constant, r_d is a dynamic recovery constant, and r_s is a static recovery constant.

$$\dot{\epsilon} = f(\sigma, \rho) \quad (1.14a)$$

$$\frac{d\rho}{d\epsilon} = h\sqrt{\rho} - r_d\rho - r_s\dot{\epsilon} \quad (1.14b)$$

The first contribution to the KME is attributed to the works of U.F. Kocks published during the mid 1970s [79, 80]. Seeking improvement over the predictability limitations observed in conventional hardening models, the author developed a mechanistic interpretation of the stress-strain hardening of materials with a single ρ equation considering dislocation production and dynamic recovery (h and r_s in Eq.(1.14b)).

The KME formalism of Eq.(1.14) continued its development with the subsequent works of H. Mecking and Y. Estrin [81–84]. Extended ρ formulations addressing particle strengthening or static recovery (r_s in Eq.(1.14b)) were developed therein.

Application to Incoloy 800H: A noteworthy application of this modeling approach, also known as the "one-parameter model" (OPM) given its dependence on a single ρ equation, was that by B. Reichert et al.[32, 85, 86]. The authors elaborated a MF model to predict the hot tensile and creep deformation response of Incoloy 800H addressing particle hardening. The hardening equation stems from the phenomenological hardening interpretation shown in Eq.(1.15), where the logarithmic homologous stress-strain curves follow an inversely proportional slope m , with the reference strain rate ($\dot{\epsilon}_0$) and reference stress (σ_0) as fitting parameters. The slope is often found to fit a temperature-dependent formulation where k_B is Boltzmann's constant, and A is a fitting parameter.

$$\ln \left| \frac{\dot{\epsilon}}{\dot{\epsilon}_0} \right| = \ln \left| \left(\frac{\sigma}{\sigma_0} \right)^m \right| \quad \text{where} \quad m = -\frac{k_B T}{A} \quad (1.15)$$

The reference stress σ_0 in Eq.(1.15) is then replaced by the resolved critical shear stress from the Taylor equation from Eq.(1.12a); a physical stress threshold. The reference strain rate $\dot{\epsilon}_0$ is however left as a fitting parameter. This mathematical work provides the first equation of the system, shown in Eq.(1.16a).

The ρ equation shown in Eq.(1.16b) represents the rate of change of the dislocation density calculated from three different contributions. A brief description of the physical meaning of each term is provided hereafter. Note that positive (+) contributions are entitled to material hardening, whereas negative (-) contributions add to material softening.

- (+) dislocation obstacle interaction: the gliding of dislocations throughout the lattice is damped by the presence of several types of obstacles such as DWs (grain and subgrain boundaries), precipitates or solute atoms. L_i represents the mean free gliding path of dislocations limited by the i^{th} obstacle.
- (+) dislocation forest immobilization: another obstacle damping the otherwise unopposed gliding of dislocations are dislocations themselves. This effect is captured in the second term of the equation, where $\rho^{1/2}$ represents the average dislocation separation distance, and k_1 is a fitting parameter.
- (-) dynamic recovery: this last term addresses the material strengthening effects entitled to incremental plastic deformations. The constant k_2 is defined in [32] as a strain rate-dependent power function.

$$\frac{\Delta \epsilon}{\Delta t} = \dot{\epsilon}_0 \left(\frac{\sigma}{\alpha M G b \sqrt{\rho}} \right)^m \quad (1.16a)$$

$$\frac{\Delta \rho}{\Delta t} = \frac{\Delta \epsilon}{\Delta t} \left(\frac{1}{b L_i} + k_1 \sqrt{\rho} - k_2 \rho \right) \quad (1.16b)$$

In principle, the differential equation system presented in Eq.(1.16) allows us to evaluate the mechanical response during different loading regimes depending on the chosen boundary conditions. For instance, work hardening or relaxation problems are solved by imposing $\dot{\epsilon} = \text{constant}$, whereas creep requires $\Delta\sigma = 0$.

Their model was solved via explicit integration, and boundary conditions were imposed accordingly. Among the most important choices of this work was the particle-hardening expression. To calculate L_i in Eq.(1.16b), the authors considered a comprehensive list of obstacles, namely grain boundaries (L_{Gb}), subgrain boundaries (L_{SGb}), stable carbides (L_{sC}), and unstable carbides (L_{nC}) as seen in Eq.(1.17). Their first article [32] addresses the effect of three different model formulations: with only stable carbides ($1/L_{sC}$), with only non-stable carbides ($1/L_{nC}$), and combined ($1/L_{sC} + 1/L_{nC}$). Their second article [85] improved upon their previous article by including an Ostwald-ripening formulation to account for carbide coarsening and dissolution during creep as a function of temperature and time. The model was therein validated for creep tests at 900°C and a variety of low-stress levels with different aging times (e.g., different initial carbide size distributions). Lastly, the third piece of research [86] presented a complete version of the model where validation was provided for an extended hot-tensile and creep tests campaign; the size distribution of carbides and its evolution was obtained after a thorough Scanning Electron Microscopy (SEM) material characterization campaign where as-received and aged material samples were assessed. The simulation results are compared to their corresponding experimental hot tensile and creep curves in Figure 1.13a and Figure 1.13b respectively.

$$\frac{1}{L_i} = \frac{1}{L_{Gb}} + \frac{1}{L_{SGb}} + \frac{1}{L_{sC}} + \frac{1}{L_{nC}} \quad (1.17)$$

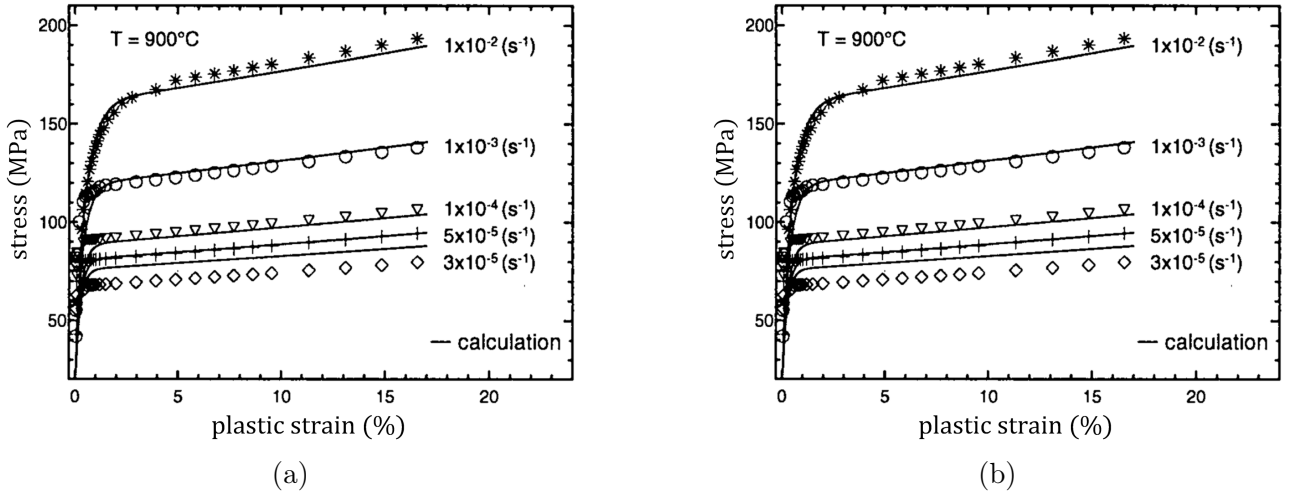


Figure 1.13: Experimental validation of OPM adapted to predict the (a) hot-tensile and (b) creep deformation response of Inocloy 800H at 900°C. Figures after the work of B. Reichert et al.[86].

Today, the KME model stands as a well-regarded mechanistic approach. The inherent physicality of the model provides good predictability of hardening/softening trends in relation to the thermal and mechanical loading conditions and its initial microstructure. However, a more strict comparison to the experimental curves (see for instance Figure 1.13b) shows limited accuracy. The authors attributed the lack of accuracy to the generic stress-strain approach used in the model.

According to Y. Estrin & H. Mecking, [82] the main advantage of the KME and its variants was that they allowed us to compute the stress-strain response of materials by solving an

equation system where a single state variable (ρ) was involved. However, the addition of an essentially phenomenological stress-strain relationship in Eq.(1.16a) and the consideration of fitting parameters k_1 and k_2 ruling the recovery processes in Eq.(1.16b) were simplifications that limited its applicability. This was addressed in variants of the original KME model, considering for instance multiple dislocation-density types [87], adding new microstructural phenomena, or replacing the first hardening equation with a more physically meaningful term. A comprehensive rundown of the mechanistic interpretations of creep arising from these formulations is provided by [77].

The Ghoniem-type models

Aware of the influence of micromechanical and microstructural features on the macromechanical deformation response of steel alloy HT-9, N.M. Ghoniem et al.[62] developed a semi-physical numerical model to predict the creep deformation response of a steel alloy (HT-9). The mathematical framework supporting the dislocation forest plasticity equations of the model is found in the theoretical studies by H.P. Hirth and J. Lothe (lately revisited by P.M. Anderson in [10]) in addition to the Orowan equation [2]. The model proposed by N.M. Ghoniem consisted of 5 differential equations describing the time-dependent evolution of 5 codependent and dimensionless state variables. These are: the strain ϵ , the mobile dislocation density ρ_m , the static (or dipole) dislocation density ρ_s , the boundary dislocation density ρ_b , and the mean subgrain radius R_{SGb} . The differential equation system showcasing the functional interdependency of the equations with regard to the state variables is given in Eq.(1.18).

$$\dot{\epsilon} = f(\epsilon, \rho_m) \quad (1.18a)$$

$$\dot{\rho}_m = f(\epsilon, \rho_m, \rho_s, \rho_b, R_{\text{sb}}) \quad (1.18b)$$

$$\dot{\rho}_s = f(\epsilon, \rho_m, \rho_s, \rho_b, R_{\text{sb}}) \quad (1.18c)$$

$$\dot{\rho}_b = f(\epsilon, \rho_s, \rho_b, R_{\text{sb}}) \quad (1.18d)$$

$$\dot{R}_{\text{sb}} = f(\rho_m, \rho_s, \rho_b, R_{\text{sb}}) \quad (1.18e)$$

The physical concept of N.M. Ghoniem et al.[62] consisted in the modeling of the creep deformation response driven by Eq.(1.18a) by means of solving the equation system Eq.(1.18). Phenomena such as dislocation forest production, annihilation, and interaction with other dislocations, precipitates and material substructure were addressed (a comprehensive description of the creep micromechanics involving these concepts is provided in section 1.1.1). The identification of fitting (i.e., non-physical) parameters and validation for HT-9 steel alloy is only partially provided. The numerical implementation, computational efficiency or convergence of the model were not addressed. Nevertheless, the numerical results of the model are consistent with the experimental results. Mechanical and state variable predictions provide physically accurate results consistent with theoretical knowledge.

The physical consistency of this modeling approach is demonstrated from a thermodynamic perspective in Discrete Dislocation Dynamics (DDD) simulations [88], later corroborated by I. Groma [89] and A. El-Azab [90] using statistical continuum dislocation (SCD) simulations. Altogether, these studies would drive the development of more complex Continuum Dislocation Dynamics (CDD) models [63, 91–93].

MF & creep modeling: a historical perspective

In summary, MF mechanical modeling of materials entails a good compromise between physical predictability and computational efficiency. However, the identification procedure entails a deep understanding and knowledge of the initial and latent microstructural features of the material. In addition to conventional macromechanical experiments (e.g., hot-tensile or creep tests), microstructural characterization techniques such as SEM, TEM or Energy-Dispersive X-ray Spectroscopy (EDS) are necessary to identify precipitates, observe substructure formation, and quantify the dislocation density. Evaluation of the initial material microstructure (As-Received (AR) condition) allows the identification of key material parameters and state variables, whereas characterization tests performed on deformed or aged material microstructures are necessary to validate the physical prediction capabilities of the model.

The research interest in mean field modeling of materials has increased in response to the promising prediction capabilities it offers. New sets of equations have been developed to improve the model's suitability for specific materials or to explore its applicability to different mechanical problems. In 2002, Dr. Christof Sommitsch et al., [94] published on the development and implementation of a mean field model to predict the recrystallized grain structure during hot deformation of steel alloys. To this end, the authors included an equation to compute the nucleation and coarsening of recrystallized grains during hot forming as both a strain-dependent and thermally activated process. The resulting equation system was embedded into the commercial finite element (FE) method software DEFORM [95]. Such a study was motivated by the huge influence of microstructure on the macromechanical properties of materials [96, 97]. The resulting model allowed engineers to improve control over grain size and recrystallized grains during material processing, thus improving the efficiency of heat treatments [98, 99]. While far from creep, these studies showcase the versatility of this type of model and the amount of coupling possibilities in multiscale analysis.

In the same year (2002), R. Sedláček and W. Blum [100] developed a generic numerical framework for microstructure-informed macromechanical constitutive law suitable for tensorial calculations (e.g., a unified viscoplastic constitutive model (UVCM)). To this end, the authors included the microstructural variables within the kinematic hardening equation of the model. The framework was later used to build a proper numerical model intended for the prediction of the creep behavior of a 9-12% Cr steel in [101]. Microstructural variables such as R_{sb} and dislocation density forest (ρ_m , ρ_s and ρ_b) were included. Additionally, particle (precipitate), Orowan-loop and dislocation pile-up hardening contributions were introduced by means of their internal stress contribution. The deformation response predicted with the model was determined to be in good agreement with experimental results.

Around 2010, the development of simpler mean-field models (i.e., following a framework similar to that of its predecessor, the KME) saw widespread popularity. For instance, a generalized dislocation forest based energy density formulation was proposed by N. Ohno et al. [102] and later expanded for hardening [103] in Crystal Plasticity (CP) formulations [104, 105]. Another study improving over the original KME model was conducted by G. Engberg and L. Lissel in 2008 [106], where dynamic recrystallization and grain growth were included. Notably, it was recently adapted and applied to predict the hot deformation response of a martensitic steel by N. Safara et al. [107]. The model presented in the latter resorts to thermokinetic simulations in Thermo-Calc® [108] to recover information on the kinetics of precipitates involved in the substructure evolution of the martensitic alloy in study. In his recent book of 2024, [12], Dr. Rolf Sandstörn described in detail the process of development and application of dislocation density based formulations in the context of creep-life assessment of metallic components for the nuclear energy sector. Despite working in similar KME-style models for the prediction of stress-strain hardening [109] and creep curves [110], the author also deviated from the conventional formulations in an attempt to explore more "fundamental modeling" techniques where

the dislocation-density formulations (for instance, the KME equations shown in Eq.(1.16)) are condensed into a single expression. With the Orowan-equation (see Eq.(1.18)), suitable physical formulations for each case study and material, and a well-defined set of boundary conditions, the author was able to recover physically-founded mathematical expressions for extrapolation of creep behavior. The resulting diagrams (e.g., time, stress, creep deformation and damage relationships) provide powerful analytical engineering tools to rely upon the design of metallic components. The book [12] contains a thorough breakdown of these approaches, addressing complex creep micromechanics phenomena such as particle-strengthening, solid-solution hardening, grain boundary sliding and damage accumulation.

Around 2015, mean-field microstructure-dependent creep modeling was revisited in the context of the PhD. of Surya Deo Yadav on the modeling of the creep deformation response of a new 9-12% martensitic steel. Independent and thorough microstructural characterization analyses conducted in material samples in both as-received and creep-affected conditions revealed a continuously evolving microstructure [35]. A complex arrangement of DWs (from Gbs to subgrain boundary (SGB)s) was consistently observed in all specimens. These factors were deemed to affect the creep performance of the material, limiting the glide distance to the smallest boundary type (e.g., SGB). In addition, ripening and coarsening of precipitates was observed in the numerous creep-affected samples, further confirming the complexity of the problem as these features affected dislocation motion and SGB evolution. In view of the high microstructural impact, the researchers opted for the application of a mean-field type of model similar to that of N. M. Ghoniem et al.[62]. Their numerical model corrected mathematical typos found within the original equation terms, improved over the particle-hardening terms, and included a creep damage formulation [111]. Additionally, a clear methodology for the identification of the physical parameters, in particular the quantification of the initial state variables (i.e., the initial dislocation densities and the mean SGB radius) was provided [112]. The research, conducted in cooperation with teams at TU Graz and Johannes Kepler University (JKU) in Austria, led to the training of multiple research teams focused on mean-field models for creep and hot forming of metallic alloys. As part of the Indian Institute of Technology (BHU), Dr. S.D. Yadav continued his work on the development and characterization of new metallic alloys by means of mean-field modeling techniques applied to hot deformation [76] and creep response [113]. Into the 2020s, the Austrian team, led by Dr. Bernhard Sonderegger, has reported successful applications of mean-field creep models for P91 martensitic steel [60, 114–116] and A617 Ni-based superalloy [114, 117]. Additionally, the team has developed a mean-field software [118] envisioning industrial applications. At the moment of writing this thesis, a first version of the software CreeSo has been uploaded in [119] and recently presented at the 7th Creep & Fracture Conference organized by the ECCC. Cooperation between both research teams at BHU and JKU is still ongoing.

1.2 Material: Incoloy 800H

Incoloy 800H is an austenitic Fe-Ni-Cr developed during the 1950s to provide a material solution for applications where materials are expected to withstand high temperatures under corrosive environments. Different denominations include trademark names (such as Nicrofer 2022H [120] and Sandvik Sanicro 31 [121]) as well as standard nomenclatures (such as Unified Number System UNS N08810 [122], European Committee for Iron and Steel Standardization (ECISS) Steel X8NiCrAlTi 32-31 or Deutsches Institut für Normung DIN 1.4959 [59]). It combines good creep and corrosion resistance with a low cost/weight ratio, making it an attractive material for a variety of industrial applications. The alloy has seen extensive use in the metallurgical, petrochemical and power-generation industries.

1.2.1 Standard characteristics

The Incoloy 800 family is composed of three alloys: 800, 800H and 800HT. The required content (wt%) of Ni, Cr and Fe is consistent across all three alloys. Alloy 800H differs from its predecessor alloy 800 by having a specific C content within the range of 0.05–0.10 (wt%) and a higher solution annealing temperature within 1100–1200°C followed by water quenching [123]. The latter is performed to achieve a fully austenitic FCC main phase with an AGS of American Society for Testing and Materials (ASTM) Gr.5 or coarser, further enhancing its mechanical performance during high-temperature applications. The specific (wt.%) ranges for all alloying elements of Incoloy 800H are specified in Figure 1.14

Condition of alloy to which the properties apply						
	Details of materials tested				Specified ranges*	
		Units	Min	Max	Min	Max
Chemical composition	C	wt%	0.05	0.10	0.05	0.10
	Si	wt%	0.32	0.74	-	0.70
	Mn	wt%	0.50	1.34	-	1.50
	P	wt%	0.007	0.019	-	0.015
	S	wt%	0.001	0.017	-	0.010
	Ti	wt%	0.21	0.59	0.25	0.65
	Al	wt%	0.19	0.58	0.25	0.65
	(Al+Ti)	wt%	0.46	1.07	-	-
	Cr	wt%	19.0	21.5	19.0	22.0
	Ni	wt%	29.4	34.45	30.0	34.0
	Co	wt%	0.028	0.76	-	0.50
	(Ni+Co)	wt%	30.09	34.78	30.0	34.0
	N	wt%	-	-	-	0.03
	Cu	wt%	0.006	0.70	-	0.50
Product	Form		Tubes ($\leq 50\text{mm}$), plate and sheet, cold and hot rolled strip, bars and forgings			
	Section size	mm	-	-	-	75
Heat treatment	Solution Annealed	°C	1120	1190	1100	1200
		ASTM grain size 5 or coarser				
Tensile Properties	R _{P,0.2}	N/mm ²	-	-	170	-
	R _{P,1.0}	N/mm ²	-	-	200	-
	R _M	N/mm ²	-	-	500	750

* Specified ranges according to specification in DIN 17459 and DIN 17460 (Sept. 1992).

Figure 1.14: Standard chemical composition and average grain size requirements for Alloy 800H. After [59].

According to ASTM, the minimum AGS required for Incoloy 800H is grade 5. The equivalency between ASTM standard AGS grades and its equivalent on the IU system is provided in Table 1.2.

Table 1.2: ASTM grain size scale equivalency to the IU system. Adapted from [124].

ASTM grain size	Number of grains at 100X	AGS μm
1	1.00	254.00
3	4.00	127.00
5	16.00	63.50

1.2.2 Overview of microstructural features

As an austenitic alloy, the as-received crystallographic structure of Incoloy 800H is composed of a predominant γ -phase (a Fe-Ni-Cr based FCC lattice). This phase, together with a minimum

AGS, is achieved throughout a solution annealing (SA) process carried out at a temperature of $1120 \leq T \leq 1200^\circ\text{C}$ for $(10 \leq t \leq 120)$ min. followed by water quenching (WQ) to preserve the newly formed microstructure. Increments in the annealing temperature or the time length of the process generally result in a larger AGS [125–127]. To better visualize the microstructural changes of 800H alloy, one must refer to its phase diagram, where the phase fraction of all phases are mapped as a function of temperature. It allows for a better understanding of the phase transformations (occurring for example during the SA+WQ heat treatment or during service life), while also enabling a quantitative context from which one can make an educated guess about the phases that may nucleate or dissolve after long-term exposure to a certain temperature. A phase diagram calculated on Pandat software with the PanFe database is presented in Figure 1.15a [128], whereas Figure 1.15b showcases a phase diagram obtained at the University of Liège on Thermo-Calc[®] software using the TCFE13 database. The latter was performed using the material composition provided by (A.M Young’s PhD. thesis [129] and scientific work [130–132]).

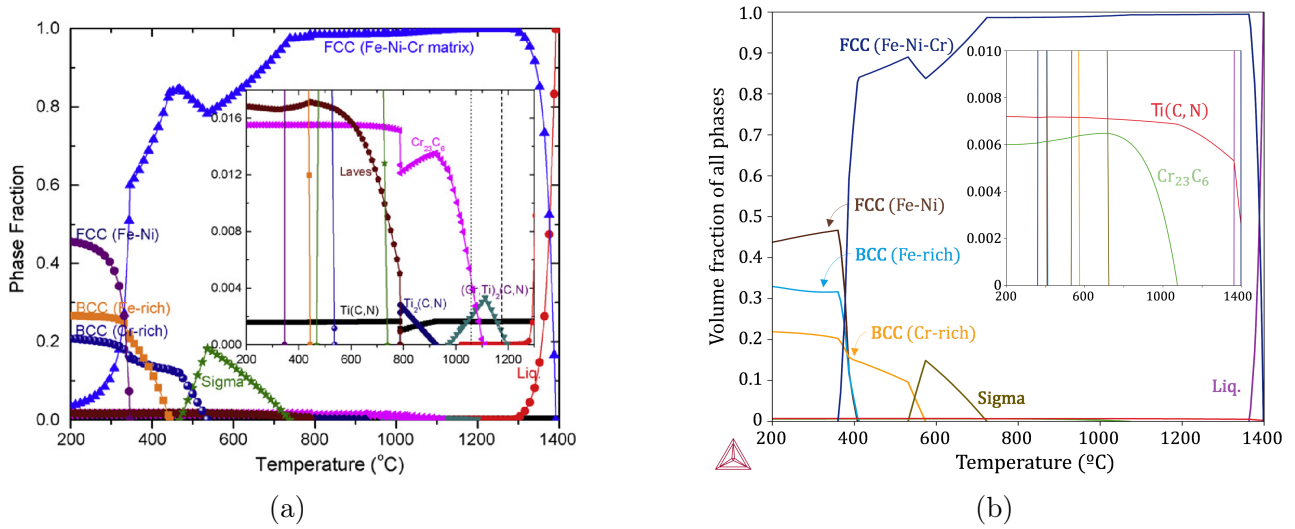


Figure 1.15: Phase fraction diagrams calculated for alloy 800H using two different CALPHAD software programs: (a) Pandat with the PanFe database (after [128]); (b) Thermo-Calc[®] with the TCFE13 database in ULiège.

During the SA+WQ of 800H, the annealing and grain coarsening process is accompanied by the nucleation of primary precipitates: titanium carbonitrides ($\text{Ti}_x(\text{C}, \text{N})$) and Cr-based M_{23}C_6 [125, 127–129, 133, 134]. The resulting microstructure consists of a predominantly intragranular network of primary precipitates embedded within a γ FCC lattice exhibiting abundant grain twinning.

During service life in high-temperature environments, the material may exhibit microstructural changes as precipitates evolve towards reaching thermodynamic stability. For instance, small intragranular chromium carbides of the type (Cr_{23}C_6) nucleating at dislocations tend to dissolve, whereas those nucleating at high-angle grain boundaries HAGBs exhibit a progressive coarsening [20, 127, 130, 131]. Conversely, $\text{Ti}_x(\text{C}, \text{N})$ possess high thermodynamical stability. Long-term high temperature exposure (within the ranges of approximately 450 to 670°C) induces the precipitation of the γ' or Sigma phase (distorted FCC $\text{Ni}_3(\text{Al}, \text{Ti})$ phase [135], reported to increase strength and decrease creep ductility [136]) and G phase ($\text{Ti}_6\text{Ni}_{16}\text{Si}_7$) [134]. Given our objective temperature range (OTR) however, the precipitation of Sigma and G phases is rarely reported. Two time-temperature-transformation (TTT) diagrams for 800H alloy are presented in Figure 1.16, where $\text{Ti}_x(\text{C}, \text{N})$ and Cr_{23}C_6 are the only precipitates involved.

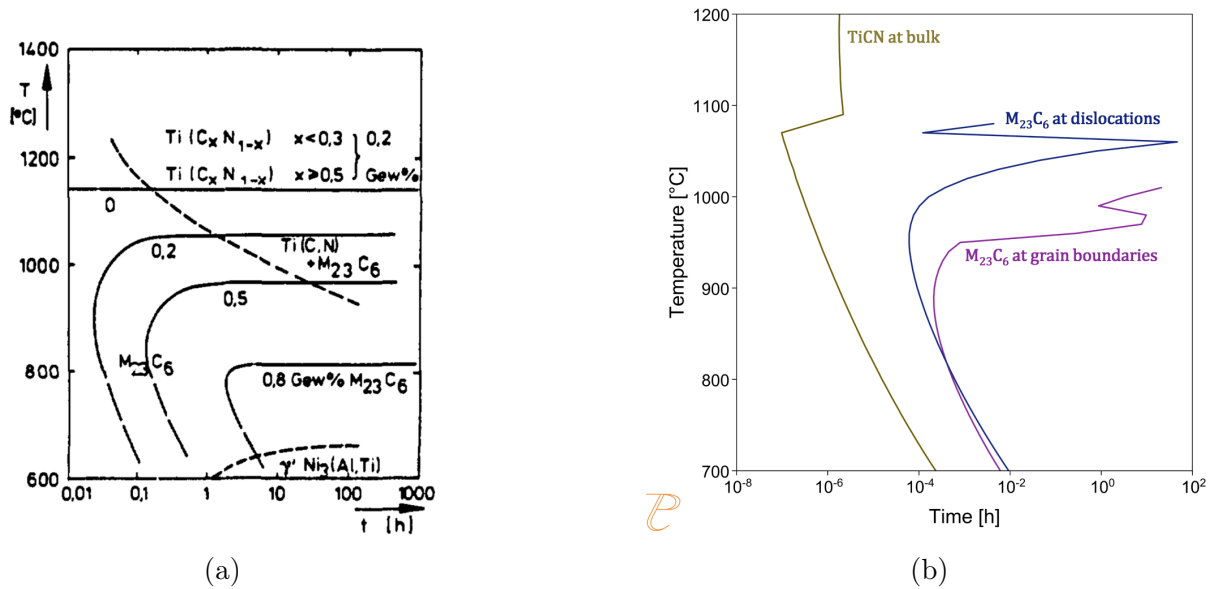


Figure 1.16: TTT diagrams for Incoloy 800H: (a) after [137], and calculated in ULiège using Thermo-Calc® TC-PRISMA with a material composition given by [131].

Primary precipitates

Primary precipitates are those formed during the first solidification process of the alloy. In other words, their presence is inherent to the material itself. Further processes of nucleation, growth or ripening will later depend primarily on the thermal history of the alloy. In the case of Incoloy 800H, three main types of primary precipitates are identified: MC, $M_{23}C_6$, and titanium nitrides (TiN). A brief description of the aforementioned precipitates alongside an overview of their kinetics under high temperature exposure is provided hereafter.

- MC precipitates:** These small carbides are initially found embedded within the crystallographic structure of 800H in both intra- and intergranular configurations. In the case of 800H alloy, “M” (nomenclature used to group a number of possible alloying elements) stands for titanium (Ti), niobium (Nb), tungsten (W) and tantalum (Ta). Titanium carbides (TiC) are the most abundant and well-studied among all MC-type precipitates. The observation and kinetics of niobium carbides (NbC), tungsten carbides (WC) and tantalum carbides (TaC) are not well documented. The size and shape, segregation and literature sources found on these carbides for 800H alloy is provided in Table 1.3. While the minor alloying elements Nb, W and Ta are known to form these carbide precipitates, no literature information has yet provided any details on the size, shape, distribution or kinetics of these precipitates on Incoloy 800H. This might be due to the small content of these alloying elements and the negligible influence of these precipitates on the viscoplastic behavior of the alloy.
- $M_{23}C_6$ carbides:** These are by far the most common primary precipitates found in Incoloy 800H. Their geometry is cubo-octahedral [138], but they can also be found in the form of fine square-shaped precipitates of <100 nm [131, 139]. “M” comprises iron and chromium based carbides (i.e., $(Fe, Cr)_{23}C_6$). However only chromium carbides of the type $M_{23}C_6$ are reported in 800H alloy. In AR 800H material, they precipitate at dislocations and at grain boundaries. Those nucleating at dislocations are reported to induce a precipitate strengthening effect.
- Ti_xN nitrides:** Titanium nitrides are the most stable of all precipitates found in 800H alloy. The size of the precipitates ranges from large ($\leq 10\mu m$), see Figure 1.17A, B

& **C**) to very small sub-micron precipitates (see Figure 1.17D). The larger precipitates distribute randomly in both intra- and intergranular configurations, and are easily identifiable due to their angular cubic-like shape (see Figure 1.17A) and golden coloration under Red-Green-Blue (RGB) Light Optical Microscopy (LOM). Although not exclusively (see Figure 1.17B), TiN is often observed to nucleate around oxides (such as Al_2O_3 , MgO) and sulfides (MgS) [140, 141], as shown in Figure 1.17C). Furthermore, larger TiN precipitates are often found decorated by fine particles such as TiC or NbC (the latter being the rarest) as seen in Figure 1.17C. Given the size and proximity of these precipitates, EDS spectroscopy analyses often do not effectively discriminate between the signals detected from Ti, C or N atoms. Therefore, these precipitates are often treated as a single family of titanium carbonitrides: $\text{Ti}_x(\text{C}, \text{N})$.

Table 1.3: Description of the main MC-type primary precipitates of Incoloy 800H.

Precipitate	Size & shape	Nucleation sites	References
TiC	Fine carbides of cubo-octahedral structure, also described as spherical or rhomboidal. Their average size ranges from 0.1 to 0.5 μm .	- Dispersed throughout the matrix material. - Conglomerates of TiC can often be found around larger TiN or Cr_{23}C_6 precipitates.	[125, 126, 128, 129, 131, 136, 138]
NbC	Fine ($< 100 \text{ nm}$) carbides of cubo-octahedral structure (Nb_2C carbides have a hexagonal structure [142]). No studies addressing NbC in 800H have been conducted. In a similar austenitic steel 25Cr20Ni-Nb-N, they reach $< 300 \text{ nm}$ [143].	Observed in dispersion throughout the bulk material in Fe-Ni-Cr-Nb alloys; uncertain for 800H.	[126]
WC & TaC	Mentioned, but no further information is given.		[126]

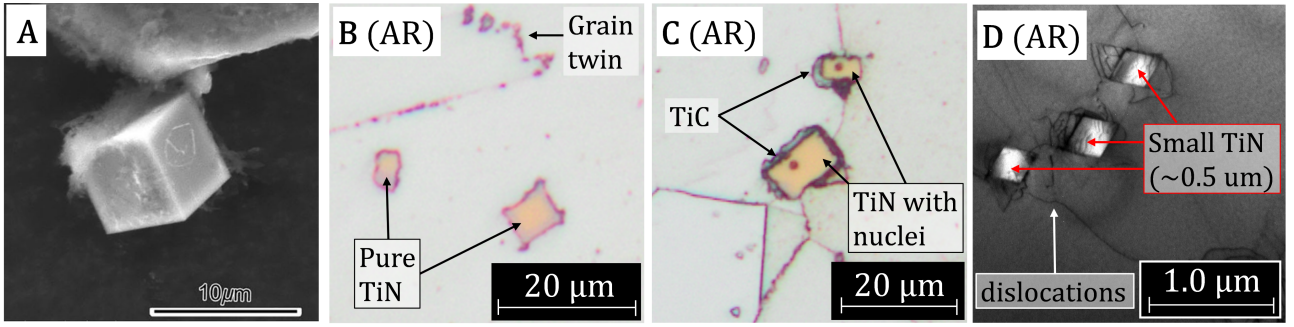


Figure 1.17: (A) 3D morphology of TiN precipitate (after [141]). (B) pure-TiN precipitates observed under LOM on AR 800H alloy sample. (C) TiN precipitates decorated by TiC in AR 800H sample observed under LOM. (D) Bright Field Transmission Electron Microscopy (BF-TEM) image of AR Incoloy 800H showcasing sub-micron size TiN precipitates decorated with dislocations (after [131]).

1.2.3 Mechanical properties

Elastic modulus, shear modulus and Poisson ratio

Given the industrial applications where Incoloy 800H is commonly found, studies on the elastic properties of this alloy often consider experiments at constant temperatures. These range from Room Temperature (RT, 20°C) up to 1000°C. An example of such a study is Hammond et al.[144], where a hot tensile test campaign was conducted in an air atmosphere and at a quasi-static strain rate within the range of $1.0 \times 10^{-6} \leq \dot{\epsilon} \leq 1.0 \times 10^{-5} \text{ s}^{-1}$. No assessment of strain rate influence on the mechanical response of 800H is provided. Measurement of mechanical properties from true stress-true strain curves (Poisson's ratio, elastic and shear moduli) was performed within a very low strain of $\epsilon = 1 \times 10^{-4}$ in an attempt to minimize the possible effects of creep and other high temperature phenomena that could affect the measurements (e.g., recovery, precipitate strengthening/softening). In addition, two samples were tested per temperature. The elastic properties of 800H (i.e., the elastic modulus (E), the shear modulus (G) and the Poisson ratio (ν)) measured at different temperatures are presented in Figure 1.18.

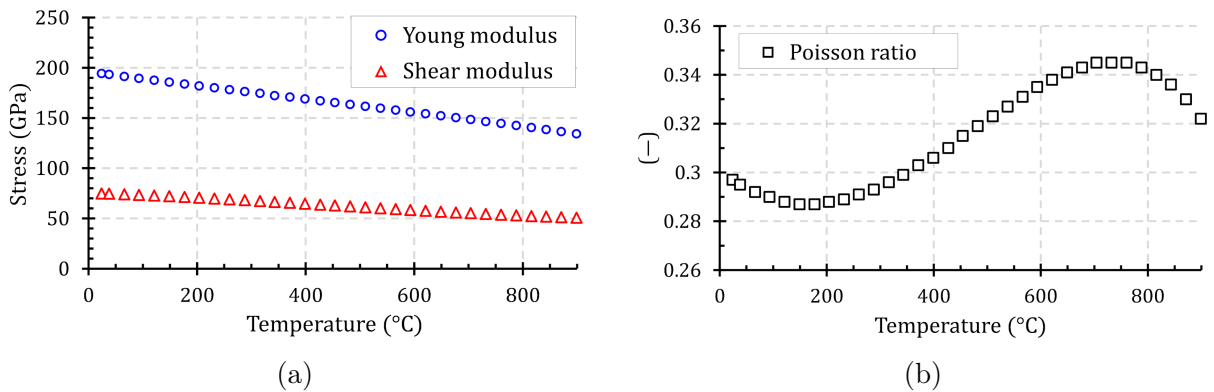


Figure 1.18: Experimental mapping of the temperature dependency of (a) elastic and shear modulus, and (b) Poisson ratio, measured for Incoloy 800H under quasi-static hot tensile tests. After [144].

Along with the raw experimental data, the authors from [144] provide a temperature-dependent polynomial curve fit for the curves presented in Figure 1.18. It allows for an easy interpolation of results within the tested temperature range. The polynomial function $f(T)$ intended to describe

these properties is presented in Eq.(1.19), where A, B, C and D are material parameters, and T_0 is RT (20°C).

$$f(T) = A \cdot [1 + B \cdot (T - T_0) + C \cdot (T - T_0)^2 + D \cdot (T - T_0)^3] \quad (1.19)$$

The parameters A, B, C and D for Eq.(1.19) to describe the three aforementioned elastic mechanical properties of 800H alloy are given in Table 1.4.

Table 1.4: Parameters for the polynomial function $f(T)$ from Eq.(1.19) to fit the experimental elastic constants of Incoloy 800H at temperatures ranging from $20 \leq T \leq 900^\circ\text{C}$. Identification performed by Hammond et al.[144].

Property	A	B ($^\circ\text{C}^{-1}$)	C ($^\circ\text{C}^{-2}$)	D ($^\circ\text{C}^{-3}$)
E	194.21 ± 0.85 (GPa)	-3.65×10^{-4}	7.06×10^{-8}	-6.53×10^{-11}
G	76.74 ± 0.16 (GPa)	-2.50×10^{-4}	-4.32×10^{-7}	3.40×10^{-10}
ν	0.297 ± 0.0022 (-)	-6.05×10^{-4}	2.66×10^{-6}	-2.11×10^{-9}

Yield stress

The 0.2 % offset yield stress of Incoloy 800H has been presented in many recent studies [138, 145–148] for temperatures ranging from 20 to 900°C. The experimental yield stress datapoints shared therein appear to have been obtained following a methodology founded in the latest extension on the standards for high-temperature applications and mechanical performance of 800H alloy provided in ASME report STP-NU-035 [149]. These experimental datapoints recovered from literature are presented in Table1.5.

Table 1.5: Experimental 0.2% offset yield stress for Incoloy 800H at various temperatures. Experimental data after [146, 147, 150].

Temperature ($^\circ\text{C}$)	Yield stress (MPa)	Temperature ($^\circ\text{C}$)	Yield stress (MPa)
20	247.37	700	148.68
200	203.95	750	148.68
300	186.84	800	140.79
400	181.58	850	135.53
500	161.84	900	105.26
600	160.53		

The experimental information provided in Table 1.5 allows to provide an empirical temperature-dependent yield stress model for 800H. In a similar fashion to that of, [144], a 4th order polynomial function is used. The function is presented in Eq.(1.20), where T is the current temperature of the material, T_0 is 20°C, and the coefficients α_i are determined via numerical optimization by minimizing the root-mean squared percentile error (RMSPE). The parameters obtained following this procedure are summarized in Table 1.6.

$$\sigma_y(T) = \sum_{i=0}^4 \alpha_i \cdot (T - T_0)^i \quad (1.20)$$

Table 1.6: Optimized parameters for the polynomial function of Eq.(1.20) describing the evolution of the 0.2% offset yield stress of Incoloy 800H as a function of temperature.

α_0 (MPa)	α_1 (MPa °C ⁻¹)	α_2 (MPa °C ⁻²)	α_3 (MPa °C ⁻³)	α_4 (MPa °C ⁻⁴)
2.43×10^2	-3.14×10^{-1}	3.88×10^{-4}	5.69×10^{-12}	-2.60×10^{-10}

The experimental data points along with the proposed empirical analytical model describing the temperature dependency of the 0.2% offset yield stress of 800H are graphically presented in Figure 1.19.

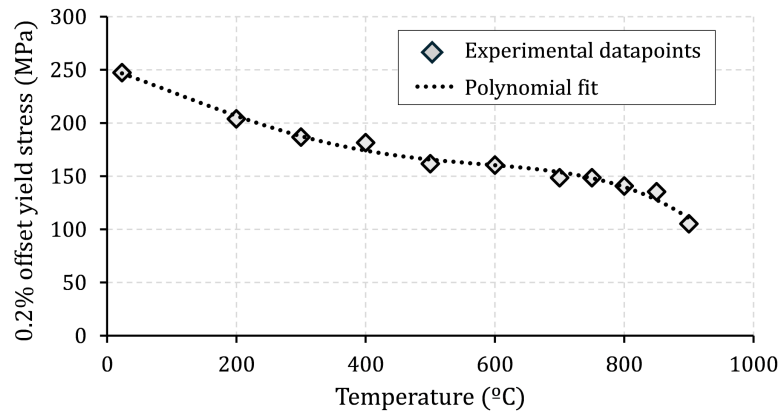


Figure 1.19: Experimental data points of the 0.2% offset yield stress of Incoloy 800H and 4th order polynomial function optimized to fit the temperature-dependent yield behavior. Experimental data after [146, 147, 150].

Temperature-dependent plastic response

The ultimate tensile strength (UTS), σ_y (also known as "tensile strength" and "yield strength") are shown for a limited temperature range from RT to 760°C in Figure 1.20a. The same properties in addition with the maximum elongation under quasi-static tensile load are shown for temperatures ranging from RT up to +1000°C in Figure 1.20b. According to Figure 1.20, the evolution of the UTS and σ_y follows an inverse and non-linear relationship with the temperature increment. The non-linearity of this relationship is exacerbated in Figure 1.20b, where the temperature dependency of these properties is shown for an extended temperature range. Within temperature ranges close to our OTR, the UTS shows an abrupt decrement after 550°C. A similar trend is observed for the yield strength after 800°C. In contrast, a notable increment in the elongation to fracture is observed beyond 750°C, indicating a loss of strength in favor of ductility within our OTR. This behavior can be observed in the stress-strain plot in Figure 1.21a, where hot tensile tests were performed by [147] at a quasi-static strain rate of $1.6 \times 10^{-4} \text{ s}^{-1}$. A different hot-tensile experimental campaign on Incoloy 800H was performed by [148], with a strain rate of $1 \times 10^{-3} \text{ s}^{-1}$ and under a pure N₂ atmosphere. This combination of high strain rate and N₂ atmosphere appears to have a large effect on the material response within our OTR, exhibiting a more brittle behavior (i.e., lower ductility). The engineering stress-strain curves at different temperatures for both experimental campaigns are shown in Figure 1.21a and Figure 1.21b.

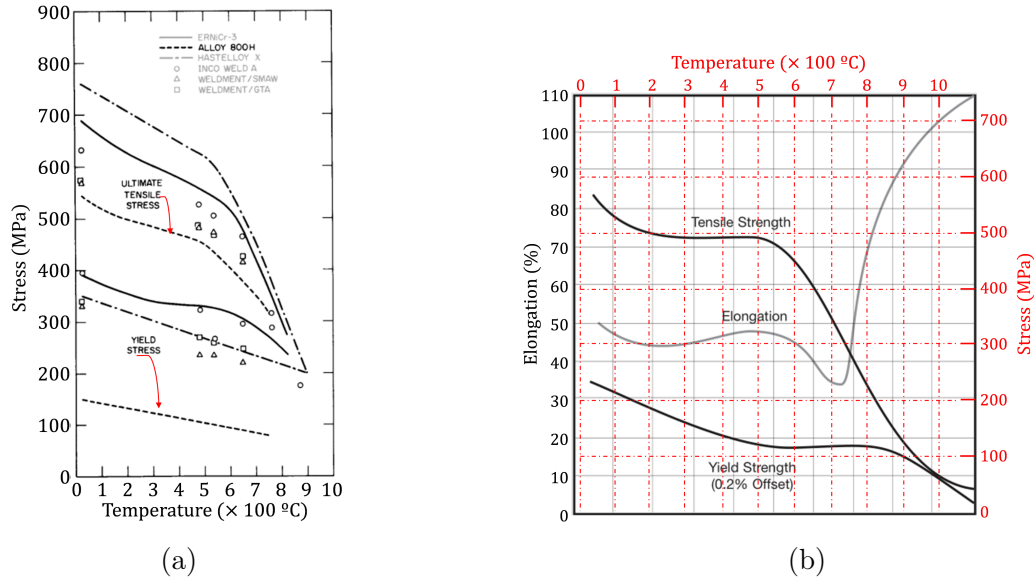


Figure 1.20: Temperature dependency of yield strength (σ_y), ultimate tensile strength UTS and maximum elongation measured for Incoloy 800H under quasi-static tensile test conditions. (a) by [151] and (b) by [152]. A similar diagram can also be found in industrial datasheets such as [122].

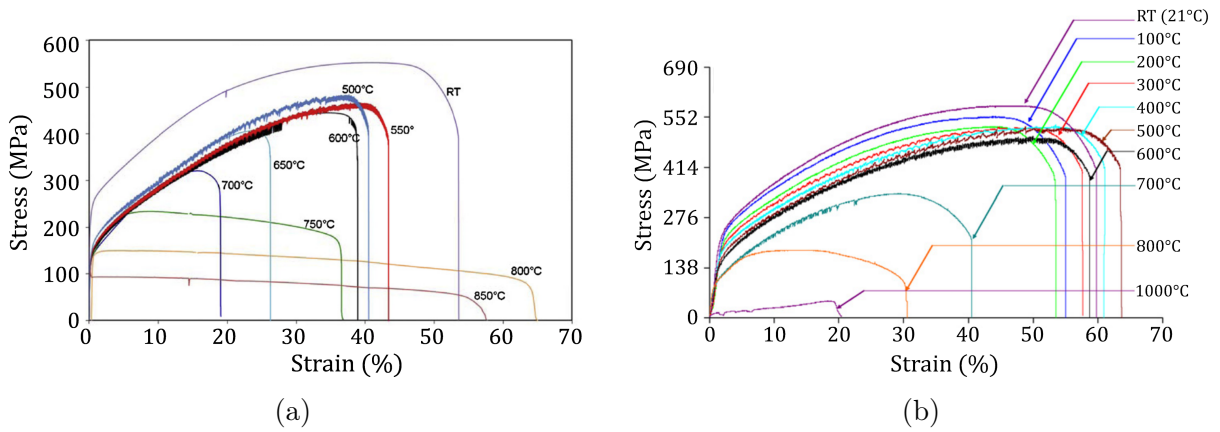


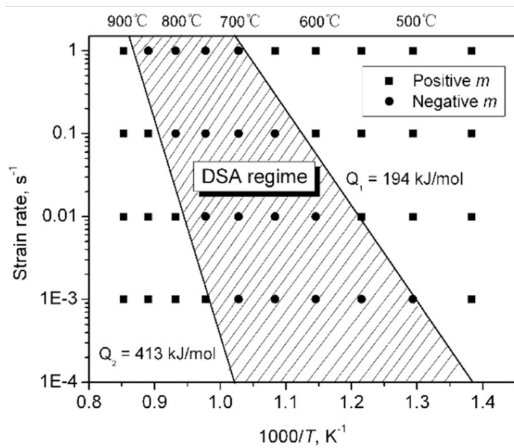
Figure 1.21: Engineering stress-strain curves of Incoloy 800H under hot-tensile conditions at various temperatures. (a) Tests conducted under a quasi-static strain rate of $\dot{\epsilon} = 1.6 \times 10^{-4} \text{ s}^{-1}$ under an undisclosed environment; after [147, 153]. (b) Tests conducted with an initial strain rate of $\dot{\epsilon} = 1.6 \times 10^{-4} \text{ s}^{-1}$ in N_2 atmosphere; after [148].

Studies on the stress-strain response of Incoloy 800H under high temperature deformations falling within mid-to-high strain rates (roughly higher than $\dot{\epsilon} = 1 \times 10^{-3} \text{ (s}^{-1}\text{)}$) are often addressed in the context of thermo-mechanical processing (TMP) (e.g., hot rolling) and part production. This is the case of the research conducted by Cao et al.[139, 145, 154, 155] and Luo et al.[156], where samples of as-received Incoloy 800H were tested in compression at high and very high-temperatures, assessing the effect of the strain rate on the elastoplastic response and on the resultant microstructure. Hardening law identification addressing the strain rate effect was conducted within annealing temperatures (beyond our OTR). These studies, however, address two important phenomena: DSA and DRX.

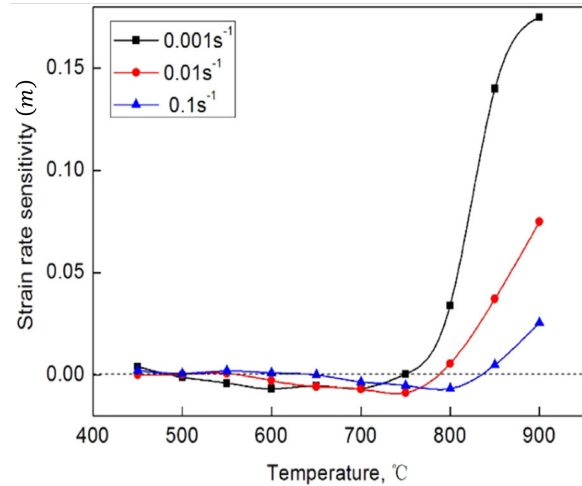
- DSA is only observed during plastic deformation regimes falling within certain ranges of strain rate and temperature. In-situ observations of this phenomenon report the onset of

serrated stress-strain curve sections localized within the first half of the plastic regime during either tensile, compressive or cyclic loadings at high temperature. This phenomenon is attributed to the continuous pinning and unpinning of dislocations as they interact with atoms in solid-solution state (such as C, Nb, Mo, etc.). Pinned dislocations tend to form intragranular arrangements around slip-planes, forming sub-grain structures that lead to a decrement in the material ductility in favor of tensile strength [155, 157, 158]. In Cao et al.[155], the critical strain for the onset of the DSA regime in 800H alloy is modeled by the Arrhenius-type function, where the strain rate sensitivity of the phenomenon is related to the parameter m . A 2D map describing the temperature and strain rate sensitivity of DSA for Incoly 800H is shown in Figure 1.22a, whereas the strain rate sensitivity of the DSA regime for $m < 0$ is shown in Figure 1.22b.

- DRX consists of the nucleation and coarsening of new grains. The recrystallized microstructure is characterized by having a larger Gb coherency compared to the original. DRX is therefore used as a tool to improve the stability of the microstructure of materials; the foundation of grain boundary engineering (GBE). It is induced with this purpose in the production of certain alloys. For Incoloy 800H, DRX takes place at temperatures above 1000°C and affects the flow stress exhibited by the material [155].



(a) 2D mapping of the strain rate & temperature dependency for the onset of a DSA regime in Incoloy 800H. After [155].



(b) Strain rate sensitivity for the onset of DSA regime in Incoloy 800H, given by the parameter $m < 0$ in (a). After [155].

Figure 1.22: Strain rate and thermal activation energy dependency for the onset of a DSA regime in Incoloy 800H during high temperature deformation. Figures after [155].

Modeling of the elastoplastic stress-strain response

The actualization of ASTM standards of Incoloy 800H [149] has provided the engineering community with a reliable empirical model to predict the stress-strain response of 800H alloy over the full elastoplastic range. The model, attributed to the General Atomic Company (hence named GAC in [149]) defines the natural logarithm of the elastoplastic stress response as a function of the strain as seen in Eq.(1.21), where the constants A_i (with $i = 0, 1, 2, 3$) are defined as temperature-dependent parameters of the equation. A reliable identification procedure is provided in a case study on the hot tensile behavior of Incoloy 800H published by Kim et al.[146]. To this end, a total of 11 high temperature tensile tests were conducted at a quasi-static strain rate of $5.55 \times 10^{-4} \text{ s}^{-1}$ and temperatures ranging from RT up to 900°C. The curve fitting procedure on each material stress-strain curve allowed to map the evolution of the

A_i parameters at each temperature. The temperature dependency of A_i parameters was found to follow the trend of a 4th grade polynomial function of the form shown in Eq.(1.22), where A_j ($j = 0, 1, 2, 3, 4$) are material constants. In total, this approach entails the identification of 20 parameters for the material. These are identified via a simple optimization algorithm consisting of minimizing the RMSPE of the polynomial function to recover the temperature-dependency observed for each A_j parameter as shown in [146]. The optimization procedure was likely conducted in OriginLab® software. The optimized parameters found by Kim et al.[146] for 800H alloy are given in Table 1.7.

$$\ln(\sigma) = \sum_{i=0}^3 A_i \cdot (\ln|\epsilon|)^i \quad (1.21)$$

$$A_i = \sum_{j=0}^4 B_j \cdot T^j \quad (1.22)$$

Table 1.7: Optimized values of polynomial constants from Eq.(1.22) defining the temperature dependency of B_i parameters from Eq.(1.21). Identification and validation after [146].

	B_0	B_1	B_2	B_3	B_4
A_0	$6.83 - 2.44 \times 10^{-3}$	1.40×10^{-5}	-3.26×10^{-8}	2.36×10^{-11}	
A_1	4.94×10^{-1}	-9.45×10^{-4}	9.08×10^{-6}	-2.58×10^{-8}	2.10×10^{-11}
A_2	7.37×10^{-2}	2.30×10^{-4}	3.94×10^{-7}	-2.84×10^{-9}	3.18×10^{-12}
A_3	5.74×10^{-3}	1.52×10^{-5}	-6.92×10^{-8}	-1.18×10^{-11}	1.15×10^{-13}

The application of the analytical stress function model provided herein enables the analytical modeling of the temperature and strain dependent stress response of the alloy under a uniaxial tensile quasi-static deformation rate regime (i.e., $\sigma = f(T, \epsilon)$). An example of the application of the model compared with experimental hot tensile curves of Incoloy 800H at 600°C and 800°C provided by [146] is shown in Figure 1.23.

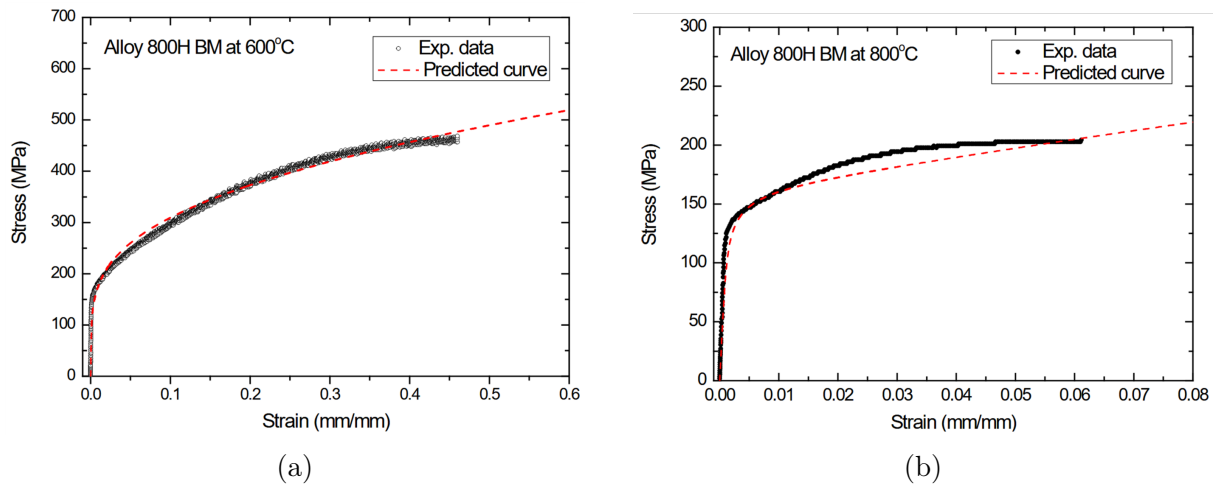


Figure 1.23: High temperature elastoplastic stress-strain response prediction of GAC model applied on Incoloy 800H hot tensile tests at (a) 600°C and (b) 800°C under a quasi-static strain rate of $5.55 \times 10^{-4} \text{ s}^{-1}$. After [146].

Mechanical properties of welded 800H alloy

Manufacturing of tubes can be either performed via centrifugal cast [159] or welding [160]. The high-efficiency and overall uniform behavior achieved with the first technique comes at a high monetary cost. The second method has been recognized for decades due to its reliability, and is currently regarded as a popular technique. The behavior of 800H alloy after welding is out of the scope of the current research project, and experiments on such conditions are therefore out of our experimental campaign. It is however briefly addressed in the following paragraphs given the relevance of welding in the manufacturing of 800H components for industrial applications. Tungsten inert arc gas (TIG) welding is reportedly the preferred welding method for 800H and other steel, Mg and Al alloys. The protective inert-gas atmosphere generated around the molten weld pool prevents atmospheric contamination of the fusion zone. Two main welding techniques are reported in research: filler-welding and filler-less (or autogenous) welding. The first consists of filling the gap between the 800H pieces with high-Ni content alloys, such as Incoloy 82,[160, 161] and NicoFer S 7020 [135]. Conversely, the second does not consider the use of a filler material; it is recommended when welding thin metal sheets [151]. The effect of the heat affected zone (HAZ) on the mechanical properties of the material is reported to depend on the welding heat input. High heat input levels induce the formation of a coarse grain size, reduced mechanical properties and sensitization issues within the HAZ (precipitation of intergranular Cr-carbides and generation of Cr depleted zones). In contrast, lower heat inputs favor the formation of finer grain sizes with increased mechanical properties [161]. An interesting experimental procedure on TIG welded of 800H specimens using NicoFer S 7020 as filler is addressed by Kolluri et al.,[135] with the aim of characterizing the fatigue-life of 800H under high stress cyclic loadings. Although these loadings are not found within our objective stress range (OSR) and OTR, the results provide an idea on the particularly high-hardness of weldments compared to 800H alloy. The observed fracture modes of pure 800H alloy and welded 800H material under quasi-static hot tensile loading is qualitatively analyzed and compared in Figure 1.24a and Figure 1.24b respectively.

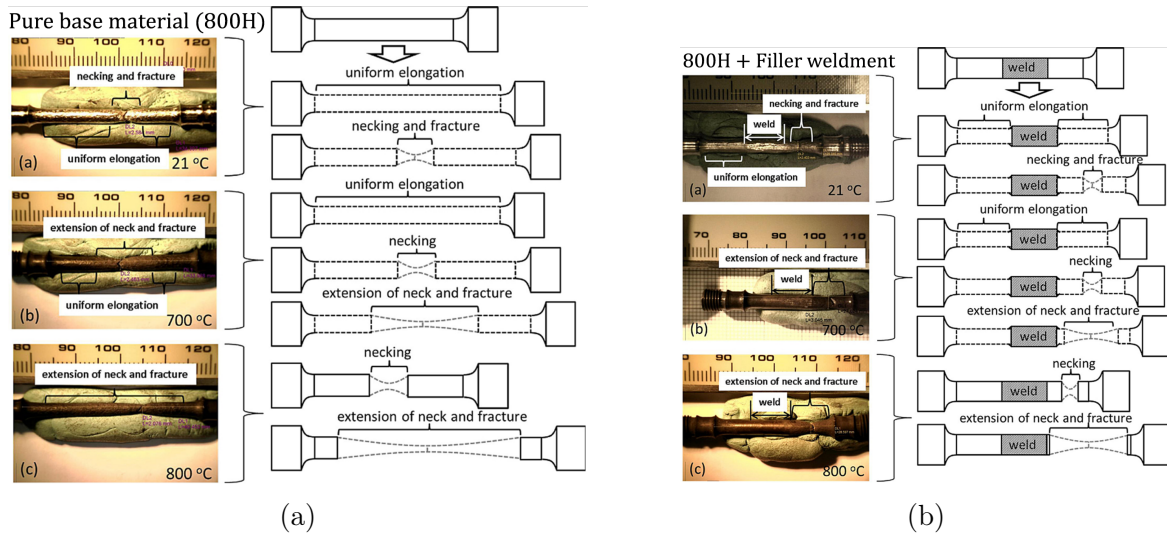


Figure 1.24: Qualitative comparison of fracture modes of (a) pure 800H alloy and (b) 800H + filler weldments under quasi-static hot tensile tests at 21, 700 and 800°C. After [135].

Thermal dilatation coefficient (α_T)

A temperature variation in a closed system is a phenomenological interpretation of the statistically significant increment in the vibration rate of molecules. This increment causes the average distance between molecules to increase, which leads to an overall expansion of the

system: a thermal expansion. The α_T is for solid materials the ratio of spatial length (volume) change with temperature. Reliable experimental information on the temperature dependent α_T of Incoloy 800H is provided in industrial material datasheets such as [122, 152, 162]. A brief list of α_T α measured within the range from 100°C and 1000°C retrieved from literature is provided in Table 1.8.

Table 1.8: Experimental measurements of α_T of Incoloy 800H at different temperatures. Data recovered from industrial datasheet documents [122, 152, 162]; no information on measurement techniques, measurement error or standard deviation is provided.

Temperature (°C)	100	200	300	400	500	600	700	800	900	1000
$\alpha_T (\times 10^{-6} \text{ } ^\circ\text{C}^{-1})$	14.6	15.9	16.4	16.8	17.0	17.3	17.7	18.1	18.3	18.6

The data points given in Table 1.8 can be used to fit a simple empirical analytical model that depicts the variation in α_T as function of temperature $\alpha(T)$. To this end, an Arrhenius-type logarithmic curve of the form of Eq. (1.23) is used, where $T_0 = 20^\circ\text{C}$, and A_α , B_α and C_α are material constants. The latter were optimized by a simple standard deviation minimization algorithm. The optimized material constants for 800H alloy are summarized in Table 1.9.

$$\alpha(T) = A_\alpha + B_\alpha \cdot \ln \left(\frac{T - T_0}{C_\alpha} \right) \quad (1.23)$$

Table 1.9: Optimized parameters for the natural logarithmic Arrhenius-type equation describing the evolution of the α_T of Incoloy 800H. St.Dev.=0.09.

$A_\alpha (\times 10^{-6} \text{ } ^\circ\text{C}^{-1})$	$B_\alpha (\times 10^{-6} \text{ } ^\circ\text{C}^{-1})$	$C_\alpha (^\circ\text{C})$
1.78×10^1	1.50	6.57×10^2

The resulting empirical model from Eq.(1.23) with parameters from Table 1.9 is compared with the experimental data points in Figure 1.25.

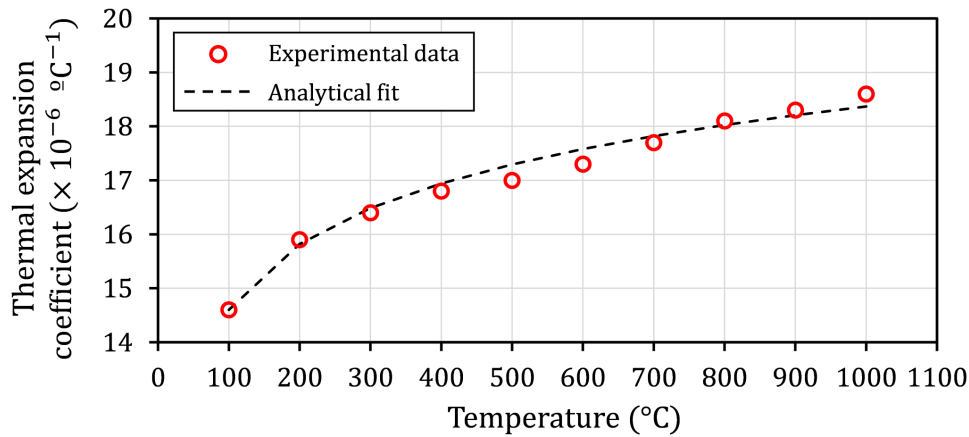


Figure 1.25: Evolution of the average coefficient of thermal expansion of Incoloy 800H as function of temperature. Experimental data extracted from the following industrial datasheets [122, 152, 162].

1.2.4 High-temperature creep behavior

A chronological retrospective on creep in 800H

Conceived to withstand high temperature environments, studies on the creep performance of 800H alloy were adamant in characterizing its mechanical performance in accordance with contemporaneous technological and industrial requirements. This research was accompanied by the continuous process of establishing and improving manufacturing standards under the supervision of entities such as ASME in the U.S., DIN in Germany, and National Institute of Materials Science (NIMS) in Japan. In particular, a comprehensive chronological summary of the evolution of ASME codes for 800H is given in [163]. Scientific publications or reports on the testing campaigns upon which these standards rely are hard to find and often not publicly available given the nature of the industrial application (nuclear energy sector). The oldest public record on the study of the creep behavior of 800H alloy dates back to a project report from Wilson et al. [121] where the analytical extrapolation of the rupture strength of the alloy at stresses of $41 \leq \sigma \leq 82$ MPa and temperatures between $760 \leq T \leq 871^\circ\text{C}$ is analytically explored by means of LMP and MHP t_r extrapolation methods. In 1978, a technical report by M. K. Booker [58] presented the results of a thorough analytical study on the phenomenology of creep curves of 800H alloy within $41 \leq \sigma \leq 82$ MPa and $538 \leq T \leq 760^\circ\text{C}$. It provides multiple phenomenological analytical models fitted to interpolate key creep stages, including time-to-1% creep and t_r . The latter was followed by a more comprehensive technical report, where M. K. Booker and V. K. Sikka [164] extended the previously described analytical methodology to multiple austenitic steels. Notably, the authors highlight the "non-classical" behavior exhibited by 2 1/4 Cr-1 Mo steel under the studied thermomechanical conditions; based on the more in-depth studies conducted on that and similar alloys by Dr. Ronald Klueh[†] [165, 166]. The sudden increment in the creep rate during the secondary stage is attributed to a creep mechanism transition caused by microstructural instabilities occurring during creep. An illustrative example of non-classical creep strain rate v/s time and creep strain v/s time curves is given in Figure 1.26a. No further discussion on the creep behavior of 800H is given in this study.

Across the Atlantic, a research team based in the Czechoslovak Academy of Sciences led by Dr. Alena Orlová[†] studied the dispersion-strengthening effect (i.e., particle-hardening, a dislocation-precipitate (DP) phenomenon described previously in Table 1.1) exhibited during creep in austenitic steels. In their first work, published in 1977 [167], experimental data gathered from creep tests conducted on an 800-type austenitic steel alloy at $24 \leq \sigma \leq 344$ MPa and $650 \leq T \leq 750^\circ\text{C}$ were used to identify a semi-physical dislocation density based hardening model to describe the internal stresses caused by dislocation interactions with primary precipitates (in particular, M_{23}C_6 and $\gamma' \text{Ni}_3(\text{Al}, \text{Ti})$ particles). In their work published in 1981 [168], TEM was used to observe and quantify the dislocation density and particle size distribution of a similar alloy. The results allowed the authors to find a physical correlation between the distribution of primary precipitates (i.e., number density and mean diameter of particles) and the free and pinned dislocation density at different stages of creep. The study included stresses within $35 \leq \sigma \leq 441$ MPa and temperatures between $550 \leq T \leq 900^\circ\text{C}$. The correlation between the applied and internal stresses is presented in a different article [169]. The latter elaborates on the application of the dislocation-density based internal stress model and provides a semi-physical relation between the real dislocation density and the geometrically necessary dislocations density (ρ_{GND}) measured during the experiment. Further studies [170–172] would focus thoroughly on the micromechanical aspect of dislocation-particle interactions and the internal stresses induced during dislocation pinning. Nevertheless, these studies present solid pieces of evidence on the particle-strengthening effect of austenitic alloys during creep, and their internal stress formulations and calculations prove fundamental for the application of

dislocation density based models.

The first observation and assessment of a non-classical deformation response in Incoloy 800H is mentioned in the article by V. Guttman and R. Bürgel [133]. The study explores the creep behavior of 800H alloy in air within $8 \leq \sigma \leq 60$ MPa and $900 \leq T \leq 1000^\circ\text{C}$. The combination of low-stress and very-high temperature loadings resulted in the alloy exhibiting a non-classical creep response, as seen in Figure 1.26b. The authors apply analytical methods to describe the temperature and stress dependency of both creep rate minima, resulting in a not unique set of parameters. TEM images captured from purely-aged specimens show precipitation of small ($<1\mu\text{m}$) within dislocations, whereas those captured from interrupted creep tests showcase dislocation stacking at grain boundaries and the formation of subgrains. SEM technique was used to investigate the fracture mode of the specimens. Crack propagation was found to be intergranular, in agreement with creep theory. However, material exposed to atmosphere during creep (including the vicinity of cracks) was found to be heavily decorated by corrosion-related precipitates identified as nitrides. Based on these observations, the authors attribute the first peak to a dislocation-precipitate interaction phenomenon, whereas the second creep rate minimum is attributed to a nitridation-induced hardening.

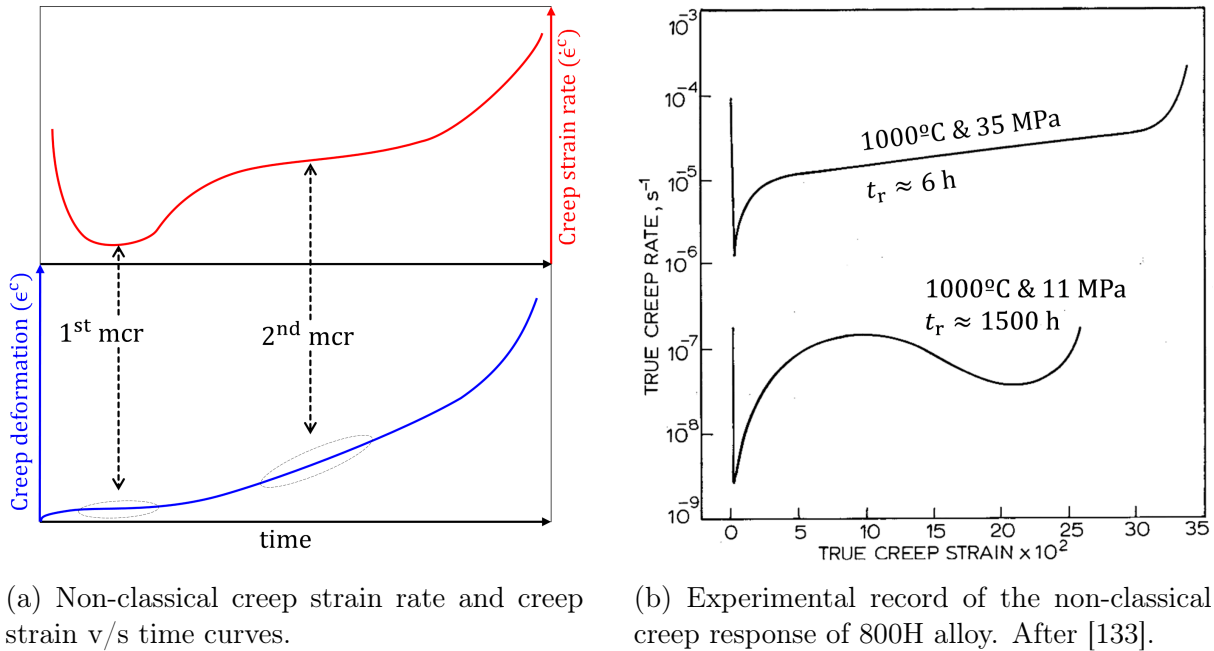
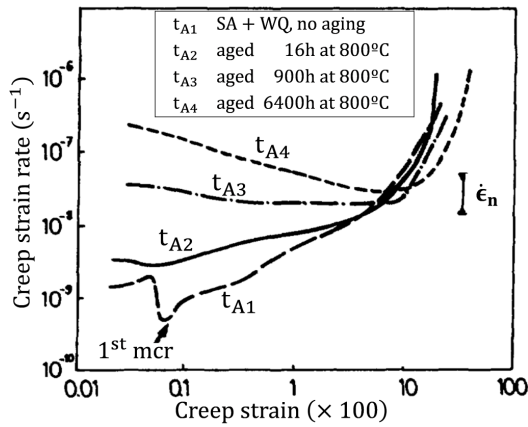


Figure 1.26: (a) Schematic and (b) experimental creep curves describing a non-classical creep behavior.

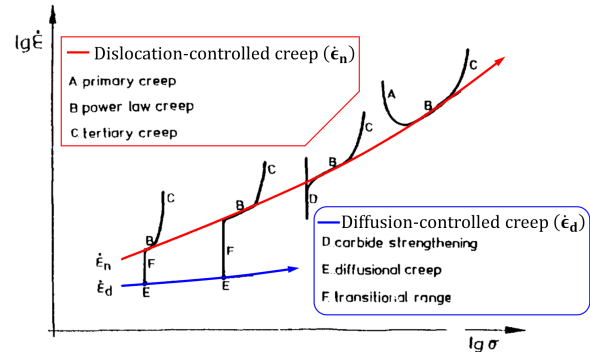
A thorough study attempting to uncover the physicality of the non-classical creep response of 800H alloy was published by Degischer et al. [137] and Spiradek et al.[173]. The authors explored the creep behavior and microstructure evolution of Incoloy 800H samples at 800°C under different initial conditions. Samples with different solution annealing temperatures ($980 \leq T \leq 1250^\circ\text{C}$) different aging times ($0 \leq t_{aging} \leq 6400$ h), and different cold working levels (up to 10%). A summary of the main conclusions driven by the researchers' findings is listed hereafter:

- Based on TEM observations, they conclude that the first **mcr** is caused by M_{23}C_6 precipitates nucleating at dislocations, thus inducing a dislocation-pinning effect. This conclusion is in agreement with the observations of the previously referenced study of V. Guttman & R. Bürgel [133].

- The second **mcr** is established to be the actual steady-state creep rate of the material. This is merely the result of a delicate balance between dislocation generation, pinning and annihilation.
- The authors show how ordinary SA processing of 800H alloy induces precipitation of sub-micron size $M_{23}C_6$ carbides at dislocations. However, overaging of the material leads to the ripening of these carbides, thus neglecting the particle strengthening effect. The superposition of creep curves belonging to different aging times is shown here in Figure 1.27a. This evidence shows how incremental aging times lead to the progressive vanishing of the first **mcr** from the creep curve, thus resembling that of classical behavior. Such as-received v/s overaged material comparison methodology would be later followed by other researchers such as B. Reichert et al.[32, 85, 86] and V. Guttman & J. Timm [174] in 800H, or El-Magd et al.[175] in 800HT.
- Upon superposing the creep curves gathered throughout the investigations (see Figure 1.27b), a qualitative relationship between the two **mcr** and the creep stress is established in [173]. In consideration of the pinned state of dislocations during the first **mcr**, the authors theorize that deformations occurring therein are diffusion-controlled (i.e., diffusion climb, HTc). In contrast, the second **mcr** is attributed to dislocation glide (LTc). The creep mechanism transition theorized by the researchers responds to a phenomenological simplification of the physics of creep, and allows for a better understanding of the process within a creep mechanism map. however, no quantitative extrapolation of results is given.



(a) Superposition of creep strain rate v/s creep strain curves obtained at 800°C and 45 MPa from Incoloy 800H specimens with different initial aging times. Adapted from [173].



(b) Qualitative mapping of creep strain rate curves showcasing the transitional creep behavior of 800H alloy. Adapted from [173].

Figure 1.27: Experimental creep curves describing the non-classical creep response of Incoloy 800H.

With the observations from Degischer et al. [137] and Spiradek et al.[173] presented hereabove, V. Guttman & J. Timm [174] studied the effect of different cooling procedures applied after a SA treatment on the creep deformation response of Incoloy 800H at 800°C. Following a similar curve superposition approach to that shown in Figure 1.27a in addition to TEM images, the authors corroborate the direct relationship between the initial amount of $M_{23}C_6$ carbides nucleating at dislocations and the magnitude of the first **mcr** reached during creep (referred to as pseudo-minimum creep rate in [174]). However, considering that carbide nucleation and

dissolution occur both during heat-treatment, the authors provide a more comprehensive perspective on the matter: Compared to slower cooling methods, specimens subjected to direct water quenching (e.g., ordinary as-received 800H material) after the mandatory SA exhibit more dislocations. This entails more nucleation sites for precipitates involved in dislocation pinning. Conversely, a different cooling regime (e.g., air or furnace cooling applied after SA for a certain period of time, later followed by conventional WQ) is a process where not only carbide dissolution can be observed, but also a lower amount of dislocations given the stress relaxation, thus providing fewer nucleation sites for precipitates. Additionally, the authors point out that dislocation-decorated subgrains are more clearly observed during the first **mcr** in samples undergoing conventional SA+WQ.

The observations of the works summarized here above provide a framework to understand the physics of creep in 800H alloy at stress and temperature levels where a non-classical response is observed. Studies performed later on would build upon this knowledge basis seeking to ascertain the effect of multiple internal and external factors. Information on the main microstructural features (e.g., AGS or Gbs) and environmental factors involved in the creep behavior of 800H is presented hereafter.

Average grain size (AGS):

The role of the AGS in the plastic deformation of materials is entitled to the inherent inverse relationship between the grain size and the amount of Gbs. In conventional plasticity [176], where permanent deformations are driven by dislocation glide, large AGS are associated with low mechanical performance. This occurs because Gbs act as barriers for dislocation movement, preventing them from gliding from one grain to the next. Therefore, materials with small AGS exhibit lower ductility, a higher yield limit, and hardening due to the higher amount of Gbs preventing free dislocation movement. This well-studied phenomenon is known as the Hall-Petch effect [177]. Conversely, in the context of creep, a large AGS is commonly associated with lower creep ductility, a higher creep resistance and an overall longer creep life [12]. This can be attributed to the active role of Gbs in creep deformation, not behaving as barriers but rather as pathways for some creep mechanisms (see for instance Figure 1.7 in Section 1.1.1) or creep damage (e.g., GBS) to occur.

As discussed in section 1.2.1, the AGS in Incoloy 800H is largely determined by the mandatory SA+WQ heat treatment conducted at the end of the manufacturing process. To study the effect of the AGS on the creep behavior of 800H, researchers obtain material samples with different AGS by performing new SA+WQ treatments adjusting the temperature and/or time of the SA accordingly. The creep responses of the samples are later analyzed in addition to pertinent microstructural characterization techniques. Following this experimental methodology, a research study published in two parts by E. Hamzah et al.[125] and M. Mudang et al.[126] compared the creep behavior of groups of 800H samples. With the exception of the AR samples, other sample groups were subjected to different SA+WQ heat treatments where different SA times were applied. The heat treatments alongside the resulting AGS and Vickers hardness are provided in Table 1.10. Representative SEM images showcasing the microstructure of each sample group after their respective heat treatments were applied are provided in Figure 1.28.

Table 1.10: Summary of microstructural characteristics of Incoloy 800H samples after undergoing specific solution annealing treatments at different temperatures [125, 126]. The AGS were measured via LOM using the line-interception method, whereas Vickers hardness was calculated by RT indentations with a force of 30 kg.

Tag	Heat treatment description	AGS (μm)	AGS (ASTM Gr.)	Vickers hardness
AR	none	96 ± 50	4	—
ST900°C	SA 900°C for 3h + WQ	96 ± 50	4	183 ± 10
ST950°C	SA 950°C for 3h + WQ	97 ± 50	4	146 ± 10
ST1000°C	SA 1000°C for 3h + WQ	113 ± 50	3	138 ± 10
ST1050°C	SA 1050°C for 3h + WQ	123 ± 50	3	131 ± 10
ST1100°C	SA 1100°C for 3h + WQ	143 ± 50	2	115 ± 11
ST1150°C	SA 1150°C for 3h + WQ	221 ± 50	1	105 ± 11
ST1200°C	SA 1200°C for 3h + WQ	381 ± 50	0	104 ± 11

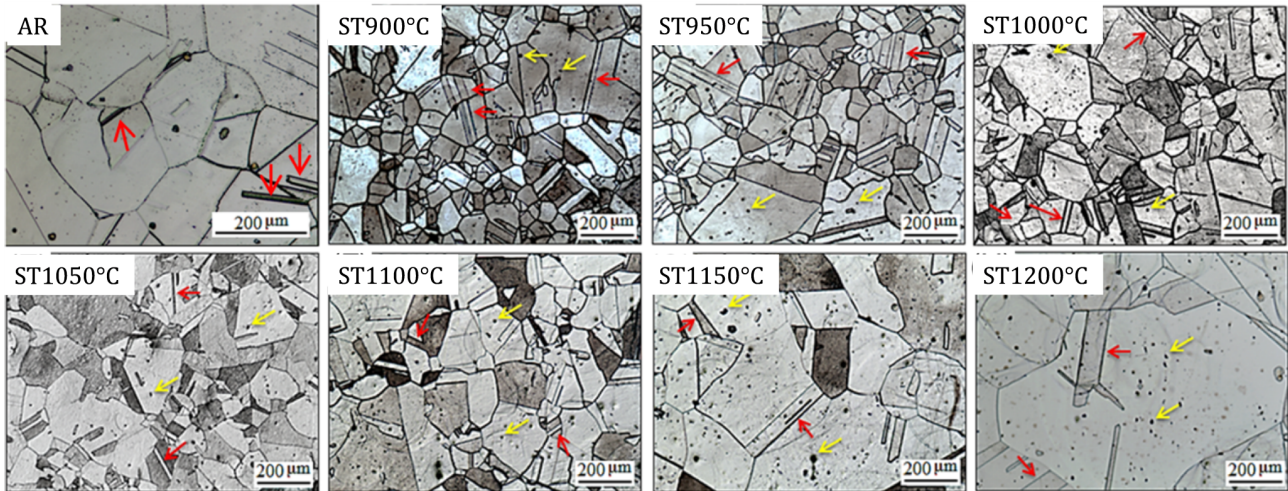


Figure 1.28: SEM images showing the different microstructures of Incoloy 800H samples after undergoing the SA+WQ heat treatments described in Table 1.10. Red arrows indicate the presence of twins, whereas yellow arrows point towards precipitates. Adapted from [125]

The article from E. Hamzah et al.[125] studies the creep performance of samples from the AR, ST1050°C. and ST1200°C. The tests were conducted at 90 and 100 MPa. According to ASTM E139 standard. The results are assessed in terms of the AGS, while barely addressing other microstructural changes that can be observed in the microstructural SEM/EDS analysis, such as a notorious increment in grain twinning and the ripening of large precipitates. It is overall a low-quality article containing errors in terms of presentation and analysis of results. The “creep curves” (elongation % v/s time) obtained therein are presented in Figure 1.29.

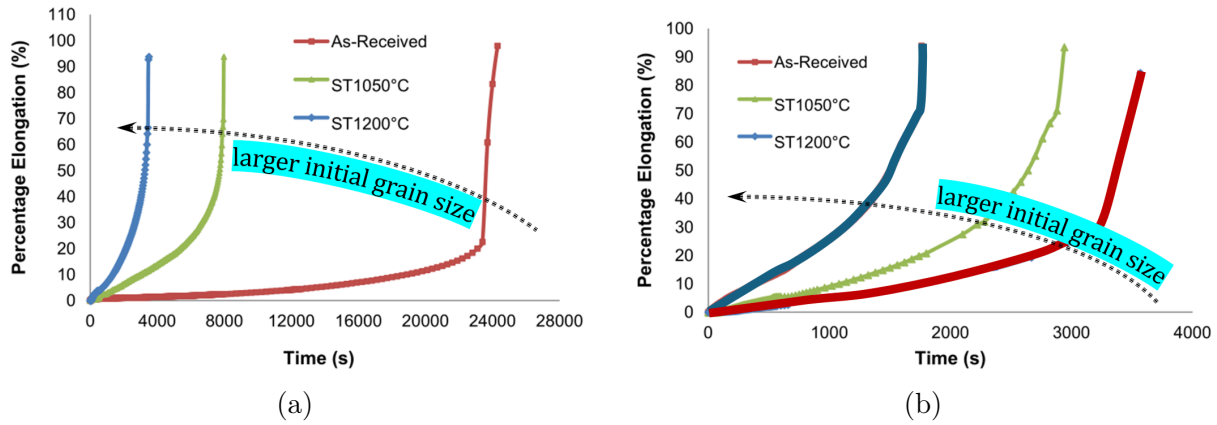


Figure 1.29: Creep behavior of Incoloy 800H samples expressed in elongation (%) v/s time in AR, ST1050°C and ST1200°C samples at 900°C and (a) 90 MPa and (b) 100 MPa (b). Adapted from [125]. Please note that red (AR) and blue (ST1200°C) colors of plot (b) have been manually edited in order to be consistent with the color code of the data across both graphs. This does not occur in the original source. Additionally, black arrows and light-blue-highlighted text labels are added to indicate the effect of the initial grain size on the creep performance.

The second part of this research was presented by M. Mudang et al.[126], where the creep behavior of samples belonging to AR, ST900°C, ST1050°C, ST1150°C and ST1200°C batches is compared. Creep tests were performed at temperatures of 800°C and 900°C and a stress of 100 MPa. Additionally, a comprehensive microstructural characterization of the initial and final state (before and after creep) of 800H samples is provided, including LOM, SEM, EDS and Electron Backscattered Diffraction (EBSD) analyses. The superposition of the creep strain v/s time curves, and the **mcr** and t_r dependency on the AGS are provided in Figure 1.30.

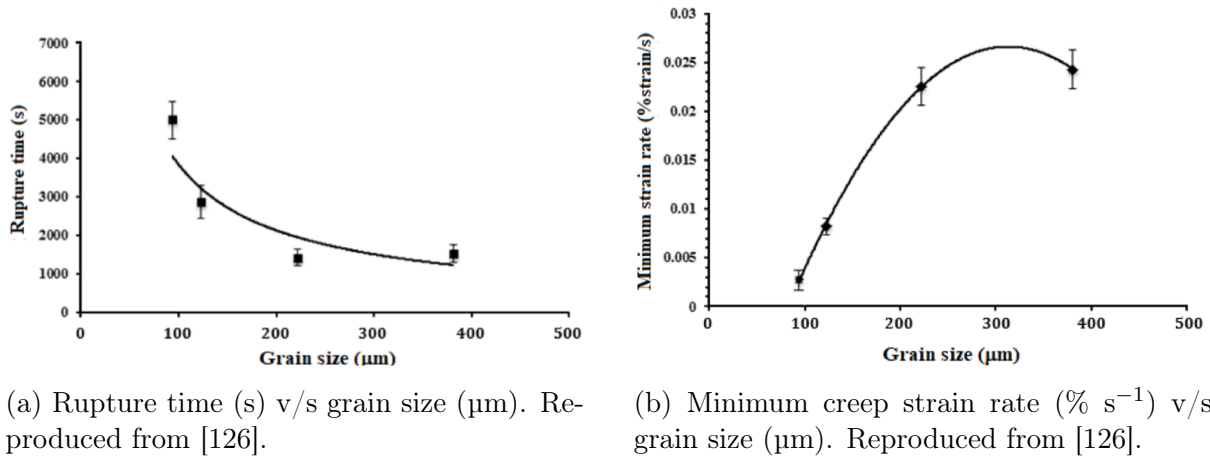


Figure 1.30: Creep response at 900°C and 100 MPa for 800H samples with different grain sizes.

The creep behavior of 800H in all creep scenarios tested by [125, 126] is of the classical type. The AGS-dependent analysis provided by the authors shows that increasing the AGS results in a detrimental effect on the creep properties of the alloy. Samples with larger AGS were observed to exhibit higher **mcr**, lower elongation to fracture and lower t_r . In addition to the AGS evolution, other effects accompanying the incremental temperatures of the second SA heat treatment in each batch are discussed. For instance, a correlation between higher SA temperatures and higher amounts of DRX grains and coarsening of precipitates at Gbs was found. The authors attribute the detriment in the mechanical properties of the material to the

Hall-Petch effect.

In the context of a PhD. thesis of Dr. Benjamin Gardiner from the University of Canterbury [127], a thorough study on the very high-temperature ($980 \leq T \leq 1200^\circ\text{C}$) and low-stress (13 MPa) creep on Incoloy 800H was conducted. The objective of this study was to characterize the creep behavior and microstructure of 800H alloy for applications in pigtail lines (tubular component used in petrochemical industry; for more information on the creep behavior of Incoloy 800H under such applications, please refer to [134, 178]). To analyze the effect of the initial microstructure on the creep deformation response of the alloy, samples were subjected to a variety of thermomechanical processing techniques (including hot and cold working deformations followed by conventional SA+WQ). The microstructural characterization of each batch of samples comprised the identification of primary precipitates and measurements of the statistical distribution of AGS, Gb coherency, and misorientation. The **mcr** tendency of both hot and cold-worked specimens as function of their initial AGS is shown in Figure 1.31a, whereas the creep ductility and rupture time of representative specimens is shown as a function of the AGS in Figure 1.31b.

Towards the end of the study, the author develops a semi-physical approach for the calculation of the **mcr** based on physical parameters (e.g., diffusion coefficients, AGS and other material parameters) and a combination of 4 creep mechanisms. This is described in more detail in [29].

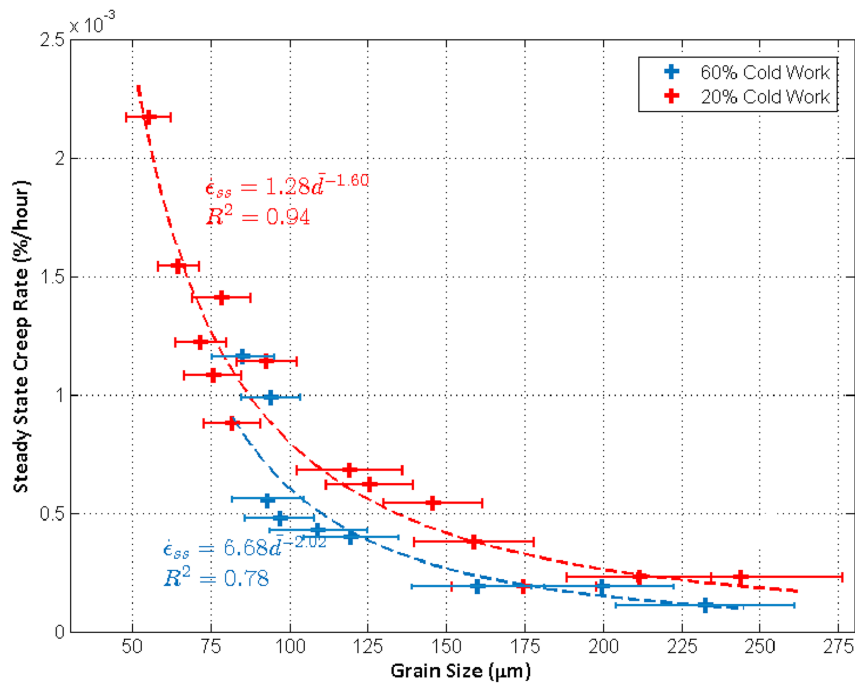
Grain size conclusion: A correlation between the initial AGS and the creep behavior of Incoloy 800H can be established in both empirical and physical terms:

- For **high-stresses**, the works of [125, 126] show that a larger AGS is correlated with a decrement in the mechanical performance of the material. This results in a higher creep rate, higher creep ductility, and lower creep resistance. From a physical perspective, this can be explained by referencing the Hall-Petch effect: the lower amount of Gbs (associated to a larger AGS) entails a lower amount of barriers for dislocation glide, thus leading to lower mechanical strength and higher ductility. This explanation assumes that creep under high-stresses in 800H alloy is predominantly driven by dislocation glide, a theory corroborated by [29]. In contrast, low-stress creep deformation of 800H alloy is driven by a combination of dislocation and diffusion-driven creep mechanisms.
- For **low stresses**, the work from [127] shows that larger AGSs can be associated with an enhanced creep performance. At this stress, the creep regime of Incoloy 800H is not exclusively driven by dislocation glide; other creep mechanisms such as climb-assisted glide are present. In addition, the lower amount of Gbs (associated with a larger AGS) restrains the progression of intergranular diffusion (i.e., Cc) and GBS, thus further improving creep performance.

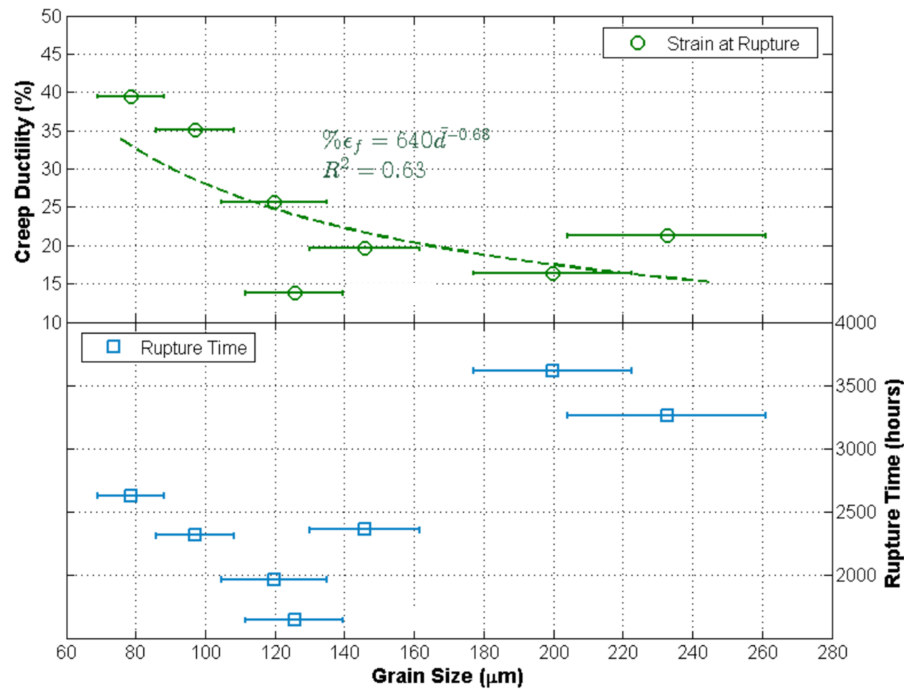
Nevertheless, the effects of particle coarsening or ripening during the secondary heat treatments in the experiments addressed in this section (see for instance [31, 32, 85, 86, 174]) must not be fully disregarded. Longer annealing times or higher temperatures often lead to a decrement in the initial **mcr** of the alloy, which could result in a lower creep life.

Grain boundaries (Gbs):

The SA+WQ heat treatment is applied for Incoloy 800H to achieve a minimum admissible AGS, following industrial requirements and standards (see section 1.2.1). Other microstructural features such as Gb coherency, texture, and the presence of annealing twins and triple joints, are however much harder to control as they do not depend exclusively on the SA conditions and are rather a consequence of the TMP (such as hot or cold rolling, forging, etc.) chosen to produce the material. The combination of deformation, temperature, and other environmental variables



(a) Steady-state creep rate (%/hour) relation to the grain size (μm) of specimens subjected to cold and hot work prior to SA+WQ. After [127].



(b) Creep ductility (%) and t_r of representative samples with respect to the grain size (μm). After [127].

Figure 1.31: Visual summary of the creep deformation response measured at 980°C and 13 MPa on Incoloy 800H samples with different initial grain sizes. Reproduced from PhD. thesis of B. Gardiner [127].

Note: It appears that the author calculated the steady-state creep rate as the first **mcr**, regardless of the presence of a secondary **mcr**.

(e.g., atmosphere) can greatly affect the microstructure. A brief diagram representing the main microstructural changes during an ordinary annealing process is presented in Figure 1.32.

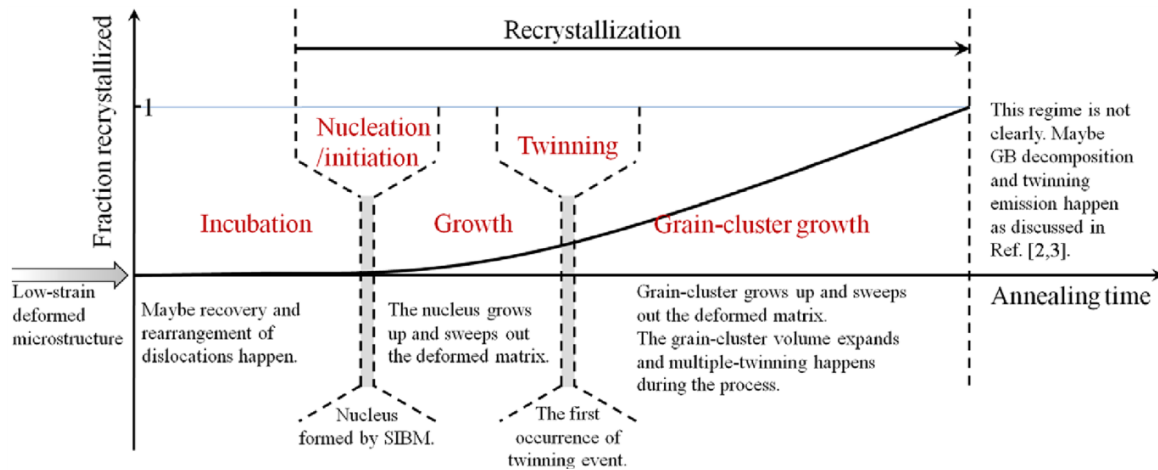


Figure 1.32: Flowchart depicting the different microstructural evolution stages associated with a standard annealing process for austenitic Ni-alloys. After [179].

Gbs are classified based on their misorientation angle and coherency. The first deals with the angular difference between the orientation of the crystals of two adjacent grains, whereas the latter entails the quantification of the Gb energy. To this end, the proportion of atoms falling in place if the lattices of both adjacent grains are superposed is calculated; a methodology known as the coincident side lattice (CSL). The calculated Gb energy indicates that Gbs offer less resistance to the movement of crystal defects across or throughout itself. A low Gb energy entails a high Gb coherency, which implies a low occurrence of Gb phenomena [180]. Gbs of the type $\Sigma 1$ or $\Sigma 3n$ are known to have the higher coherency of all. Three main Gb features, known to influence the creep behavior of Incoloy 800H, are hereafter addressed: Gb coherency, grain twinning and the presence of triple joints.

- **Gb coherency:** The study of B. Gardiner [127] reports a $\Sigma 3$ -type Gb percentage of 50.9%, whereas the analyses conducted by C. Bishop et al.[181] give a $\Sigma 3$ -type Gb percentage range between 46% and 53%. According to [128, 182], a high number of $\Sigma 3$ -type Gbs is associated with improved intergranular corrosion resistance and a longer creep and fatigue life. However, M.Y. Li et al.[138] observed via TEM that, for dislocation-driven creep of 800H alloy (a high-stress creep mechanism), $\Sigma 3$ -type boundaries would act as dislocation sinks, thus favoring the onset of cracking.
- **Grain twinning:** Grain twins are a special type of $\Sigma 3$ -type Gb. These entail a crystallographic rearrangement of a segment of a grain as a consequence of mechanical (strain twin) or thermal (annealing twin) energy in the system. While strain twins have been classified as incoherent and unstable (i.e., they can easily migrate), annealing twins are coherent in nature as they form stable low-energy $\Sigma 3$ -type configuration barriers [183]. The latter are therefore desirable characteristics for an alloy to improve its mechanical performance.
- **Triple joints:** As discussed in section 1.1.1, the presence of triple joints is detrimental for creep performance as cavities are prone to nucleate there. An interesting observation shown for instance in [127] points out that the increment of grain twins entails an inherent increment in triple joints. As a matter of fact, the great majority of triple joints in 800H alloy are the result of the intersection of a $\Sigma 3$ -type grain boundary with a HAGB.

Grain boundary conclusion: A link between the Gb coherency and the mechanical performance of Incoloy 800H could be physically and theoretically established. Large amounts of annealing twins ($\Sigma 3n$ -type Gbs) produced during the SA+WQ favor corrosion resistance and mechanical strength.

The interest in studying Gb properties in Incoloy 800H is mostly founded by their involvement in the yield strength and corrosion performance of the material. Improving the Gb quality could potentially enhance such mechanical properties. Following this line of inquiry, numerous GBE seeking to explore the effects of different TMP techniques [128, 138, 139, 145, 181, 184–187] have been conducted. The specific effects of Gb coherency on the creep performance have not been studied.

Primary precipitates:

According to J. Glen [30], the creep properties of materials are a consequence of a combination of three main factors: the *initial creep resistance* (AR properties), the *latent creep resistance* (non-permanent properties), and the *overaging* or *spheroidization of precipitates* (accounting for the reduced creep resistance after long-term high temperature exposure). This groundbreaking concept at the time motivated deep physical studies that confirmed creep as a phenomenon where permanent and latent factors are involved. These studies on the physics and micromechanics of creep addressing materials depicting non-classical creep responses (such as for the 2.25Cr-1Mo steel alloy [165, 166]) evidenced the strong relation between microstructure and creep properties. Microstructural characteristics of as-received Incoloy 800H such as its predominant austenitic FCC phase, AGS, TiN precipitates and Gb related features (coherency and twinning) are reported to exhibit little to no change during long-term high temperature exposure occurring within our OTR and OSR. Creep properties related to these microstructural features are therefore characterized as permanent. The transient creep properties of this material are attributed to two as-received state microstructural features known to evolve over time:

- A substantial presence of low-alloying atoms (primarily Cr) dissolved within the crystallographic structure of the main FCC phase; hence, it was classified as a solid-solution alloy [125, 163, 188].
- A number of micro- and nano-sized $M_{23}C_6$ precipitates nucleating during the WQ phase of the mandatory SA+WQ heat treatment of 800H alloy [131]. These precipitates have been observed to nucleate at Gb and dislocations [127, 129, 131, 137, 138, 173]. A curious case where no precipitates of such kind were found in as-received specimens of 800H is reported in [189].

The SA+WQ heat treatment conditions for 800H are designed to optimize its microstructure to improve its mechanical properties for creep and corrosion resistance. The DRX induced during this process allows a large AGS of ASTM Gr.5 or coarser to be achieved. Alloying C atoms dissolved within the microstructure either remain embedded therein or precipitate in the nucleation of small $M_{23}C_6$ carbides. The microstructure reached after SA is metallurgically unstable, and a WQ process is applied to ensure its stability after cooling. This WQ and other rapid cooling techniques (e.g., oil quenching) are common practices in metallurgy as they allow optimization of the mechanical properties of materials. Consequences of these process and/or post-process operations, such as the inherent thermodynamic instability of the microstructure (due to the incompleteness of the carbide precipitation or ripening) or possible residual stresses (e.g. work hardening after hot rolling), are not necessarily detrimental to the performance of the alloy. In our case however, the long-term high temperature exposure of 800H alloy (for instance, during creep within our OTR and OSR) creates the conditions that allow the microstructure

to evolve towards a higher level of metallurgical stability. This affects the creep deformation response in such way that a non-classical two-step creep strain rate minima is observed.

1. The first **mcr** takes place during the very initial moments of creep. Gbs and large precipitates such as TiN nitrides and coarse $M_{23}C_6$ carbides act as dislocation sources. Cr atoms diffuse from Gbs in an attempt to pursue the precipitation of $M_{23}C_6$ carbides. Together with the C atoms found in a solid-solution state, these carbides act as dislocation pinning points (inhibiting the free movement of dislocations). The progressive dislocation immobilization leads to the onset of this first **mcr**, while the diffusion of C and Cr atoms leads to the onset of a predominantly diffusional creep stage.
2. As small ($< 1 \mu m$) $M_{23}C_6$ carbides nucleate within dislocations, the latter become progressively freed from pinning atoms, however their mobility is instead impeded by new small $M_{23}C_6$ precipitates. The interaction between precipitates and dislocations is interpreted as a thermodynamic process. It is conditioned by the phases and precipitates involved, and ruled by the stress and temperature of the material. In this case, dislocations are reported to overcome $M_{23}C_6$ obstacles by a combination of low- and high temperature creep mechanisms. Dislocation multiplication and glide/climb mechanisms have been reported, whereas other phenomena such as the formation of Orowan loops or particle slicing have not been observed.
3. With time and heat, the progressive dissolution of $M_{23}C_6$ carbides enables dislocations to move with relative freedom. The slow transition towards a dislocation-driven creep deformation is observed as a damped softening in the creep curve, as dislocation glide is limited only by the remaining precipitates and the pre-existing Gbs and SGbs. The latter are formed from slip planes decorated with atoms and small precipitates that impede the further movement of dislocations. TEM pictures show massive dislocation pile-up at Gbs and SGbs in 800H specimens during advanced creep stages [133, 137, 173, 174].
4. During the dislocation-driven creep, the viscoplastic deformation is ruled by dislocation generation, annihilation and interaction with precipitates and barriers. Once a balance is reached between these events, a secondary **mcr** is observed (see curve at $1000^\circ C$ & 35 MPa in Figure 1.26b or other curves from K. Spiradek et al.[173], V. Guttman & J. Timm [174], K. Tachibana et al.[190] and B. Gardiner's PhD. thesis [127]). Following the findings from [137, 173], this **mcr** is the actual steady-state creep rate ($\dot{\epsilon}_{ss}$) of the alloy for the given stress and temperature level. At this point:
 - the intragranular carbide dissolution is accompanied by a massive nucleation and coarsening of these precipitates at incoherent Gbs [128].
 - in **special cases**, under very long-term high temperature (see, for instance, curves at $1000^\circ C$ and 11 MPa in Figure 1.26b), a secondary hardening is observed after the secondary **mcr** has been reached. Microstructural studies conducted on austenitic Fe-Ni-Cr alloys exhibiting a similar creep response [143, 165, 166, 191–193] have concluded that this secondary creep rate hardening is caused by a new dislocation-precipitate interaction phenomenon occurring at the Gb level.

Secondary precipitates (corrosion):

Corrosion is a natural thermodynamic process where a material composition affected by an external agent diffusing inwards (namely, a non-inert atmosphere or environment, often accompanied by high temperature) leads to the formation of new compounds as the system (material

+ environment) seeks to minimize its free energy [194]. The progressive penetration of the corrosive agent into the material is accompanied by the precipitation of corrosion-related phases. The corroded material layer formed in the process exhibits different mechanical properties than that of the non-corroded material, thus affecting the mechanical properties of the entire mechanical component. Without regard to it being beneficial or detrimental to the material, the change in the local mechanical properties of a component is considered a risk factor that must be taken into consideration for engineering purposes.

A variety of corrosion resistant alloys have been designed to fulfill specific roles in hazardous environmental conditions. These are not invulnerable but resistant enough to ensure long service life in combination with a good safety factor. This is the case for Fe-Ni-Cr alloys, whose tight austenitic γ -phase (FCC) provides a higher opposition to diffusion [123]. In addition, the presence of chromium (and aluminum, in the case of Incoloy 800H) provides the alloy with the capacity to form a thin protective oxide layer against agents such as oxygen, hydrogen and molten salts [195]. It is composed of Cr, Fe and Mn oxide phases. In an oxidation experiment conducted by N. Parimin et al.[196], two 800H samples annealed at 1000°C and 1100°C (see Figure 1.33) were submitted to an oxidation regime of 150 cycles and 700°C. The authors show that the higher annealing temperature led to a thickened oxide layer, resulting in lower Oxygen diffusion inwards the material.

Different external conditions may induce other corrosion mechanisms. For instance, sulfur and oxide corrosion has been reported in 800H components (e.g., engine parts and tubes) working at high-temperatures under hazardous environments containing large quantities of sulfides (such as different types of salts) or oxygen (such as the case of super-critical water (SCW) or super-heated steam (SHS) in the power generation industry) respectively [197, 198]. While some of these oxide phases formed under these conditions (such as Cr_2O_3 , NiO , Fe_2O_3 , and NiCr_2O_4) have been associated with improvements in corrosion resistance [199], unfavorable phases such as Fe_3O_4 formed after high oxygen concentrations favor oxygen diffusion and lower the load carrying capacity of the material [200].

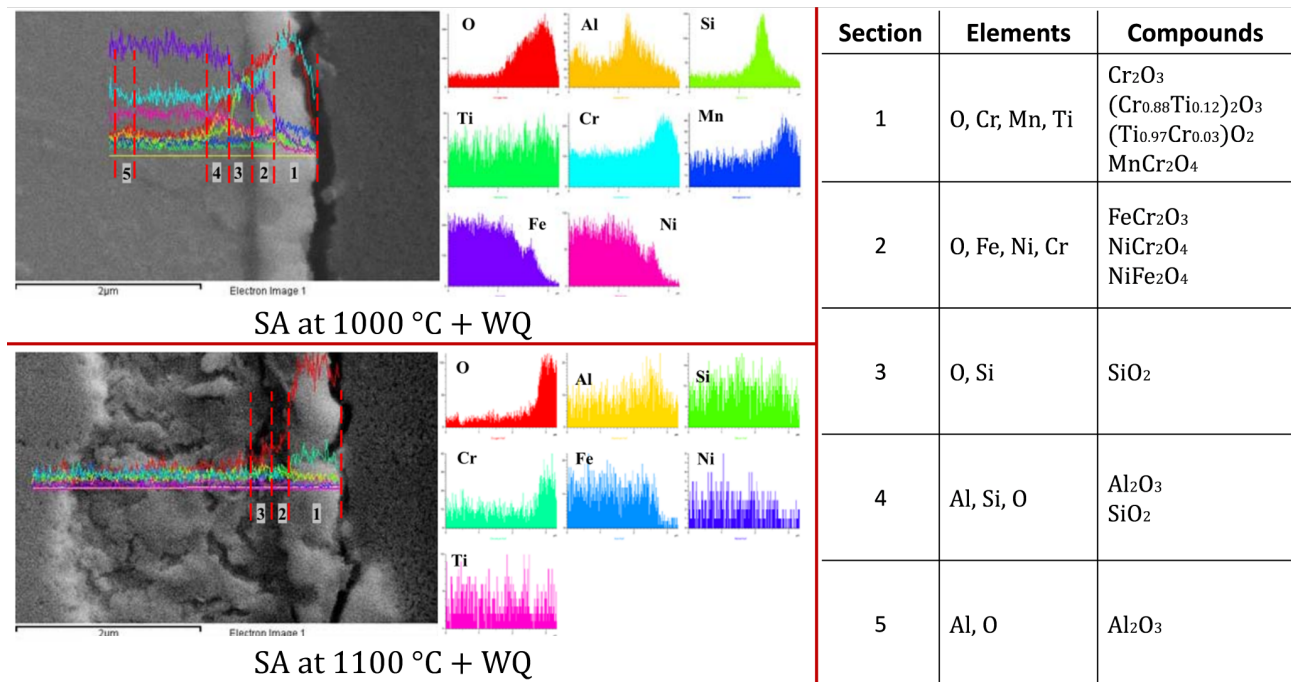
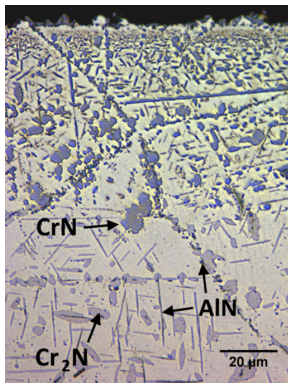
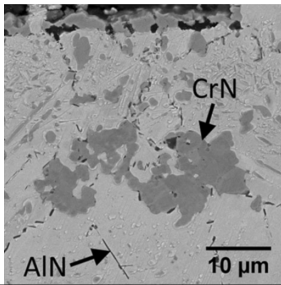
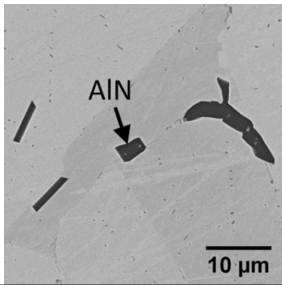
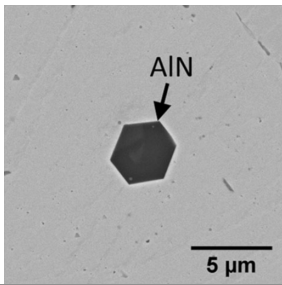


Figure 1.33: Surface Field Emission Scanning Electron Microscope (FESEM) superficial analysis of two Incoloy 800H samples after undergoing an oxidation regime consisting of 150 cycles (roughly equivalent to a 3000 hour exposure to an O-rich atmosphere at 700°C), specifying the elements forming the main protective thickened oxide layer. Adapted [201]

As seen in Figure 1.33, oxygen penetration under cyclic loading at 700°C and 3000 hours barely reached over 2 μm into the surface. Nitrogen however, is likely to penetrate deeper into 800H during long term high temperature exposure to a N-rich environment. Within our particular OTR and under air atmosphere, N diffusion from the exposed surface occurs ubiquitously in both intra- and intergranular modes (see subsection **Experiments A** in section 2.1.3, or the article by C. Rojas-Ulloa et al.[20] provided in Annex 2A). The progressive diffusion of nitrogen into the material (nitridation) leads to the nucleation of aluminum and chromium nitrides. The presence of nitrides precipitating around cracks has been reported in creep tests to rupture in 800H at 1000°C & 11 MPa [133] and 980°C & 13.8 MPa [127]. The phenomenon was recently studied in depth during the PhD. of A.M. Young [129]. The first part of their peer-reviewed research publications [130, 131] dealt with the experimental observations and characterization of nitridation in 800H under laboratory conditions. Nitridation of coupon-like samples of 800H alloy in AR condition was conducted using a Lindberg M-series furnace (see manufacturer webpage [202]) under a 95%N₂ – 5%H₂ environment and temperatures ranging from 800°C to 1000°C at 1 atm. Coupons were held inside the furnace for up to 750 hours. Microstructural characterization techniques consisting of LOM, SEM and EDS allowed to correctly identify primary and secondary precipitates embedded within the FCC material matrix at different times: after 50, 150, 350, and 750 hours in furnace. The experimental observations provide a very comprehensive picture of aluminum and chromium nitrides precipitate into the matrix material as consequence of nitrogen diffusion. An overview of the chromium (CrN & Cr₂N) and aluminum (AlN) nitride phases observed by the researchers is given in Table 1.11.

Table 1.11: Summary of N-based secondary precipitates found in nitrided 800H alloy specimens. SEM images after [130].

Cr-based nitrides		
CrN	Cubic (FCC) shaped precipitate	
Cr ₂ N	Trigonal (HCP) shaped precipitate	
AlN nitrides		
Needle-like		
		
Cubic shaped		
		
Hexagonal shaped		
		

The presence and phase fraction of primary and secondary precipitates are affected by the nitrogen concentration in the matrix material. A process of nucleation, growth and dissolution of some of these precipitates is induced as nitrogen diffuses into the material. The nitrogen

concentration gradient can therefore be interpreted as the formation of different material layers, each containing a characteristic combination of primary and secondary phases. The authors highlight this finding by comparing SEM micrographs combined with EDS analyses, thus identifying the different precipitates present alongside the nitridation gradient of the 800H specimens at different nitridation times. The comparative analysis together with the identified "layers" and their corresponding precipitates is provided here in Figure 1.34.

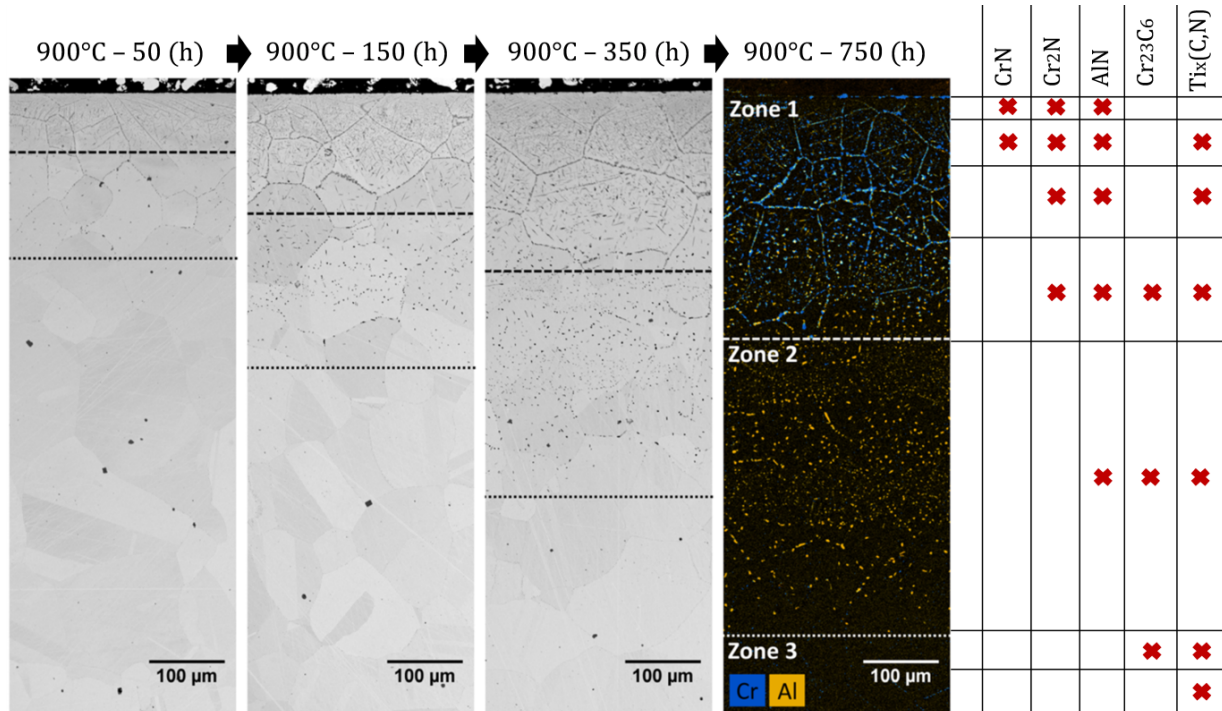


Figure 1.34: Side-by-side comparison of SEM images captured on coupon samples of 800H alloy after different times in nitridation furnace. The precipitates accompanying the nitrogen diffusion gradient into the matrix material for the last picture (750 h) are identified in the right columns. Adapted from [129].

As observed in Figure 1.34, the nucleation of nitridation-related phases (i.e., Cr- and Al nitrides) is only observed after a certain nitrogen concentration threshold is reached. The kinetics of primary $M_{23}C_6$ and secondary (CrN , Cr_2N & AlN) precipitates observed during nitridation in 800H are thoroughly discussed in [131] and [130] respectively. In both cases, the authors perform a comprehensive numerical study where the diffusivity of nitrogen into the matrix is validated with the precipitation of each phase of interest. Simulations are performed using Thermo-Calc[®] DICTRA (for calculations of phase transformations with diffusion) using databases TCFE8 and MOBFE3 for steel alloys. A phase fraction diagram calculated for 800H as a function of the N (wt.%) concentration at 1000°C indicates that the threshold for the precipitation of AlN is indeed lower than that of Cr-based nitrides. The diagram, calculated using TC-DICTRA, is shown here in Figure 1.36a. The AlN precipitates seem to penetrate deeper into the material, and are characterized as the most stable nitridation precipitates. In contrast, Cr-based nitrides are less stable. As observed in the diagram of Figure 1.36a, a dissolution of Cr_2N is observed to occur in favor of the formation of CrN . This transformation is explained from a thermodynamic perspective as the system seeks to decrease its free energy as the N concentration in the matrix increases. This continuous process of nitridation driven by chemical stabilization is showcased in N-concentration (wt.%) equilibrium phase diagrams in Figure 1.35.

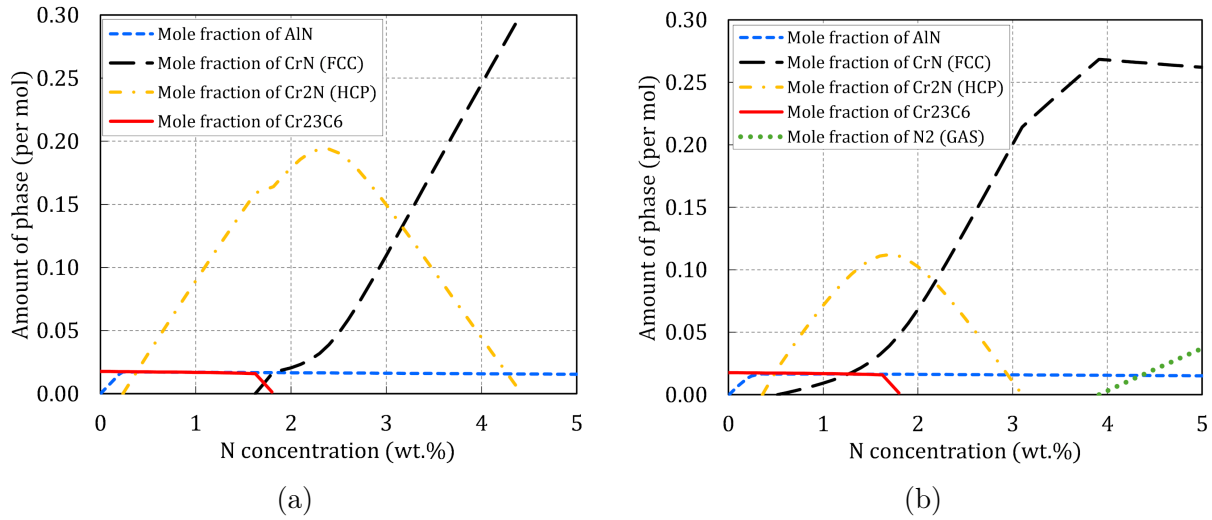
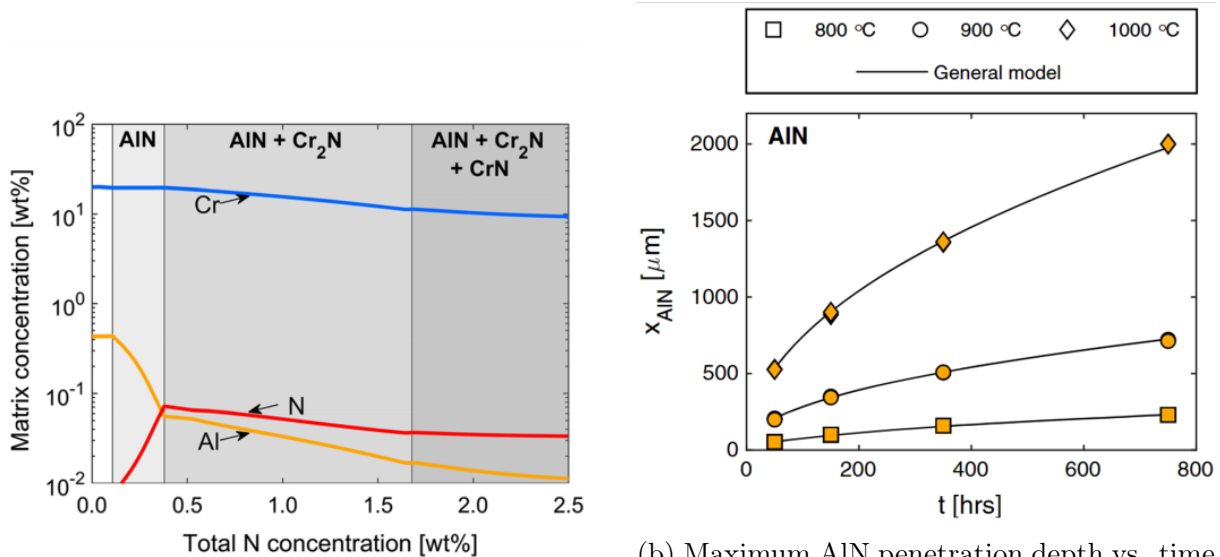


Figure 1.35: Thermo-Calc® PRISMA equilibrium calculations showcasing the progressive phase transformations occurring during progressive nitridation of 800H alloy at (a) 700°C and (b) 1000°C. Calculations conducted in ULiège using the TCFE13 and MOBFE8 databases.

Consideration of the limit of the AlN and Cr₂N layers (each representing approximately 100% and 50% of the total nitrogen penetration depth during nitridation), the researchers provide a parabolic diffusion model for both precipitates. The model is fitted for each of the studied cases, and is compared and validated with literature information and Thermo-Calc® predictions. The relationship established between the precipitation of AlN v/s time for each of the studied cases (i.e., 800°C, 900°C and 1000°C) is provided in Figure 1.36b.



(a) Matrix concentration diagram for 800H alloy showing nitrogen diffusion. Calculated using TC-DICTRA, by [130].

(b) Maximum AlN penetration depth vs. time (hours) diagram in 95% N₂ environment at different temperatures. Figure and parabolic diffusion model by [130].

Figure 1.36: Thermodynamical and diffusion kinetics analyses of nitridation in 800H alloy. After [130].

The experimental EDS measurements summarized in Figure 1.34 show that during advanced nitridation stages M₂₃C₆ carbides (mostly Cr-based) are not observed within the outer layer of the material. According to the N-concentration phase fraction diagram of Figure 1.36a, this

can be attributed to the dissolution of such carbides in favor of the formation of more stable Cr-based nitrides.

Creep and nitridation in 800H The interest in ascertaining the effects of nitridation on the creep response of Incoloy 800H is founded in the following facts: the fast N diffusion occurring upon exposure to long-term high temperatures in air [127, 133], and the local strength increment associated with the precipitation of Cr and Al nitrides [129, 132]. The experimental observations of nitridation building in the vicinity of cracks during very-high temperature and low stress creep tests in [127, 133] is pointed out by the authors as a potential factor affecting the creep response of the alloy. In particular, [133] theorizes that the secondary creep strain hardening taking place after the second **mcr** is reached (registered for instance during a 1000°C and 11 MPa creep test, see Figure 1.26b) can be attributed to a nitridation-induced creep hardening. A similar hypothesis is pointed out by [127], referencing that of [133].

Alloy design and selection entails reaching a balance of properties (e.g., creep, corrosion, hardness or wear resistance, cost, material availability); reaching a balance in this context entails a compromise of some of these properties in favor of others [203]. In this context, artificial (or industrial) nitridation is one of the many techniques available to improve the mechanical properties of these alloys. Four main artificial nitridation techniques can be found in literature:

- Nitridation by heat-treatment. This technique requires steels with particularly high nitrogen content (e.g., 0.7 (wt.%); a high concentration compared with the 0.03 max. (wt.%) established for 800H). It consists of inducing the precipitation of nitrides (mainly Ti- and Cr- based nitrides) during conventional heat-treatments, thus achieving uniform internal (bulk) nitridation. Compared to conventional Ti-based nitrides, researchers note that the additional precipitation of Cr-based nitrides leads to marginal improvements in the yield and ultimate tensile strength. However, the improved material hardness is associated with a major material embrittlement. Moreover, massive precipitation of Cr-nitrides induces material sensitization: a condition where the decrement of the Cr concentration in matrix reduces corrosion resistance [204, 205].
- Solution nitridation (gas nitridation). It can be described as an accelerated form of natural nitridation where the material is subjected to a combination of heat pressure and time in a high-N₂ atmosphere. Applied previously by A.M. Young et al., [129–132], it results in the rapid diffusion of nitrogen with no formation of oxides (given the absence of O₂ or other contaminants in the gas). It represents a middle-ground between the first and the following nitridation techniques, where the resulting surface material acquires improved hardness and wear resistance. Please note that full (through whole depth) solution nitridation can be achieved in cases where a sufficiently thin material plate is placed in a high-N₂ environment for a sufficiently long period of time. However, continuous high-temperature exposure of the nitrided material component (e.g., in operational conditions) may eventually lead Cr-nitrides to dissolve back into the matrix. Therefore, the reliability of this technique for long service life at hot temperatures has yet to be investigated.
- Plasma-assisted nitridation (PAN). This technique is recognized as the most efficient and reliable of the three. It consists in the controlled discharge of a plasma ion beam onto the surface of a material while contained in a vacuum chamber (pressure between 100-1000 Pa) filled with a high-N₂ gas and at a temperature of approximately 400°C (lower than that needed in the techniques described above) [203, 206]. In contrast with the previous two techniques, PAN induces the formation of a highly-saturated nitrided surface layer consisting of γ -Fe-N (or S-phase) [207].

- Pack nitridation. A recently studied technique that can be regarded as the simplest of them all. The material component to be nitrided is placed into a furnace and surrounded by a nitrogen compound (e.g., particles of Cr_2N , AlN , Si_3N_4 ,...). With no assessment of any special pressure or inert-gas chamber, K.Y. Li & X.D. Xiang [208] achieved nitridation in the form of precipitation of Cr-based nitrides.

Nitridation and oxidation conclusion: Subjected to high-temperatures in air-like atmospheres, Incoloy 800H is observed to exhibit a combination of oxygen and nitrogen corrosion. Of the two, nitridation is the most aggressive corrosion mechanism. Conversely to oxidation, it induces a surface hardness increment trend associated with nitrogen penetration and the corresponding precipitation of AlN , CrN and Cr_2N . The strengthening effect may however be impermanent, as continuous exposure to high temperatures leads Cr-nitrides to dissolve back into the matrix material (see for instance [134]). The latter may be attributed to the low thermodynamic stability of these precipitates (see Annex 2B), or the matrix instability caused by the complex environmental conditions the material undergoes during service life (see subsection **Experiments A** in section 2.1.3, or the article by C. Rojas-Ulloa et al.[20] provided in Annex 2A).

1.2.5 Fatigue and creep-fatigue

Fatigue is the loss of load-carrying capacity of a material as a consequence of thermal-, stress-, or strain-dependent cyclic deformations. The permanent deformations induced by the loading cycles are accompanied by the initiation and accumulation of fatigue damage. Combined with environmental factors (e.g., corrosion or high-temperatures) or creep, the accurate prediction of the number of cycles to failure of a component in study can turn into a difficult engineering challenge. A brief illustration of the key attributes used to characterize a fatigue cycle is provided in Figure 1.37a.

To achieve accurate predictions of the number of cycles to failure of a material under given cyclic loading conditions, the individual contributions and interaction between creep and fatigue mechanisms must be ascertained. To this end, a series of comprehensive experimental studies on the effects of different loading cycles on the deformation response and damage initiation of Incoloy 800H at 750°C was conducted by Z. Mu et al.[209–211]. The experimental observations provided by the authors establish a correlation between the fatigue curve shape and the dominant damage mechanism. Nevertheless, interaction and competition between intergranular crack initiation (e.g., cavitation, creep) and transgranular crack propagation (fatigue) is almost ubiquitous. A study conducted by B.S. Rao et al.[157] assessing the effect of the loading and unloading strain rate of symmetric cycles at 850°C would further improve the understanding on the subject. A qualitative summary of the main findings of these studies is listed here below.

- **Symmetric cycles effect**
 - Fatigue is the predominant deformation and damage mechanism when:
 - the mean stress is null (0)
 - absence of dwell-times
 - sufficiently high strain rate
 - equal strain rates in both loading and unloading regimes

In such scenarios, creep contributions can be neglected without sacrificing accuracy during analytical calculations of the number of cycles to failure. Optical micrographs

showcasing both intragranular (creep) and transgranular (fatigue) types of crack propagation after low cycle fatigue (LCF) at 850°C is given in Figure 1.37b

- In all other symmetric cycle cases, the material will exhibit a combined creep-fatigue damage interaction. As a general rule, the onset of creep is favored by positive mean-stresses, the presence of dwell-times in a tensile state, or slow tensile strain rates within the loading ($\dot{\epsilon} > 0$) regime. Other loading combinations may induce different damage initiation mechanisms; for instance, high compressive strain rates are associated with decohesion damage mechanisms.

- **Dwell times effect**

- Tensile dwell times favor the onset of creep deformations. Damage initiation is observed to occur at Gbs (cavitation). The creep-fatigue interaction leads to a mixed crack propagation mode where both intragranular and transgranular modes are observed.
- Compressive dwell times are signaled as favorable as they allow for cavity shrinkage. However, large compressive deformations may induce the formation of cavities by decohesion. It occurs at Gbs parallel to the loading direction or at interfaces with large precipitates (see Figure 1.7).

- **Strain rate effect** (see Table 1.12).

- At temperatures of 850°C, fast strain rates ($\dot{\epsilon} \geq 1 \times 10^{-3} \text{ s}^{-1}$, "F" in Table 1.12) during a loading regime are observed to favor fatigue damage mechanisms, whereas slow strain rates ("S" in Table 1.12) favor cavitation and intergranular crack propagation. Additionally, DSA is observed in both loading and unloading slow-strain rate regimes.

In an unloading regime, high strain rates have been observed to induce decohesion at the Gb level or at particle interfaces.

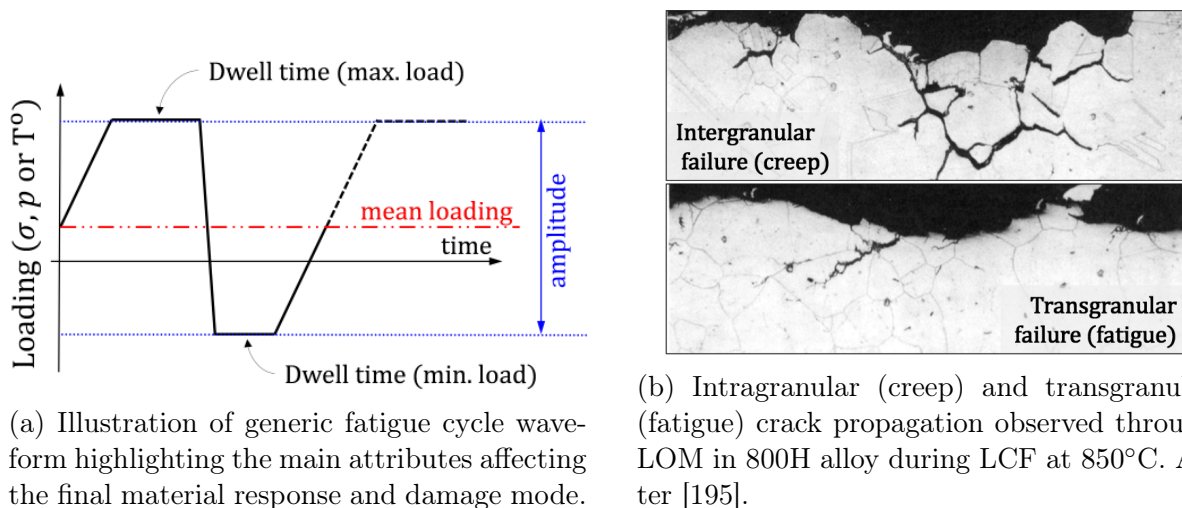
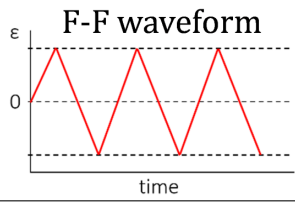
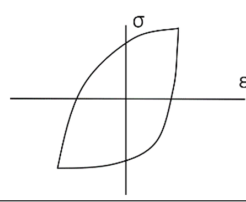
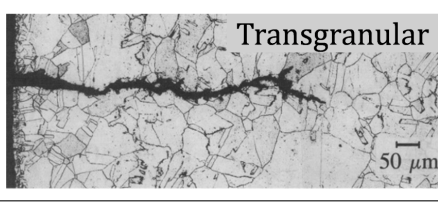
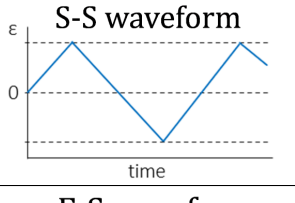
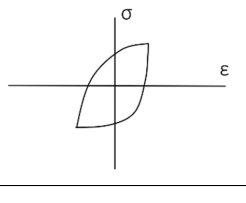
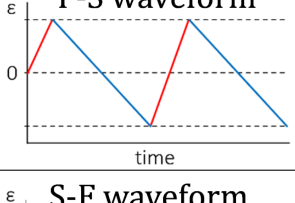
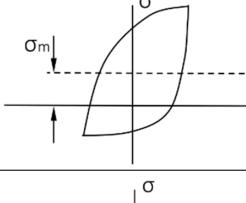
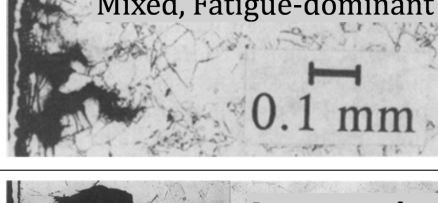
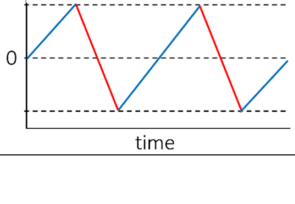
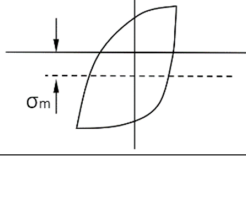
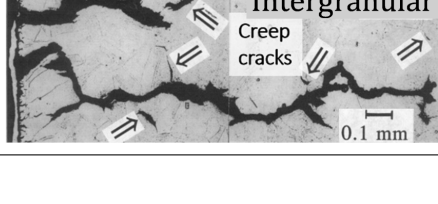


Figure 1.37: Features of a standard fatigue cycle waveform and micrographs showing the two crack initiation and crack propagation modes observed in 800H alloy depending on the dominant loading and damage type.

The relations between fatigue loading features and damage mechanisms established in the list here above provide guidelines for a basic understanding of the mechanical response of the

Table 1.12: Strain fatigue waveform, stress-strain hysteresis loop response, and damage initiation/fracture propagation mode observed for 800H samples undergoing LCF tests at 850°C in air. LOM images are captured on specimens after a 50% loss of load carrying capacity during fatigue is detected. Adapted from B.S. Rao et al.[158].

Strain waveform F (fast) & S (slow)	Stress-strain hysteresis loop	Damage mode
F-F waveform 		
S-S waveform 		
F-S waveform 		
S-F waveform 		

material. However, accurate prediction of the mechanical and damage response induced by complex waveforms combining two or more of these features is not as straightforward. Additionally, external factors such as temperature and atmosphere conditions (causing for instance corrosion) further complicate calculations of the number of cycles to failure of metallic components. To address specific scenarios (e.g. in heat exchangers in a very-high temperature reactor (VHTR) or HTR for next generation NPPs), researchers often develop their own experimental campaigns. The mechanical response of material specimens is thereby investigated under a controlled laboratory environment where the expected operational conditions are recreated. Such is the case of the study conducted by P. Soo et al.[195], where the mechanical response of 800H specimens under High cycle fatigue (HCF) loadings ranging from 20 to 200 MPa under a He-rich (emulating the environment of a HTR in a NPP is studied. The report describes a clear oxidation phenomenon taking place as impurities contained in the He-rich atmosphere (such as CO, CO₂, CH₄ and H₂O)) entered in contact with the tested specimens. The thin protective Mn-Cr oxide layer characteristic of 800H alloy did not allow oxygen to diffuse into the material tested in air, but was clearly depleted and replaced by nonprotective Fe oxides in an impure He-rich environment. In summary, 800H samples tested in a He-rich environment exhibited advanced oxidation in the vicinity of cracks and in crack tips; thus, oxidation is also observed to promote the transgranular propagation of cracks, as seen in Figure 1.37b. Although He-rich tested specimens exhibited a slightly longer fatigue life, this observation is not consis-

tent across all temperatures in the report. The authors state that, oxidation results in a more ductile behavior in comparison to the rather brittle fatigue behavior of Air-tested 800H samples. Moreover, as long-term fatigue results seem to be quite similar among Air and He tested specimens, the material performance under an impure He-rich environment is concluded to be similar to that in air for macroscale tests and applications. J.K. Wright et al.[147, 153] and M. Kolluri et al.[135] investigated the mechanical response of as-received and TIG welded 800H alloy during hot tensile, creep and LCF loadings at temperatures up to 800°C for applications in intermediate heat exchanger (IHX)s. Of particular interest in this context are the results of LCF tests presented in [135]. To assess the effects of pure fatigue and creep-fatigue, two main types of strain-controlled fatigue waveforms were assessed: without dwell-time, and with 1000 s dwell time in the loading (positive) regime. The fatigue cycles were conducted at strain rates of $1 \times 10^{-3} \text{ s}^{-1}$ and total strain ranges from 0.6% to 1.5% with a null mean strain (ratio $\epsilon_{min}/\epsilon_{max} = -1$). Tests were conducted until 50% of the load carrying capacity of the material was lost (N_{50}). The results show a consistent decrease in the number of cycles to failure in the presence of dwell times compared to pure-fatigue cycles. The strain level v/s number of cycles to failure diagram in Figure 1.38a shows that the effect on creep in the deformation and damage mechanisms is inversely proportional to the strain level. Additionally, the authors assess the damage and final performance of the alloy to further evaluate the mechanical performance of 800H alloy in accordance with ASME code case N-47. The cumulative creep/fatigue fraction diagram obtained, shown here in Figure 1.38b illustrates that the material may be suitable for such dwell-LCF operational conditions. Nevertheless, the authors highlight the need for more extensive experimental campaigns to verify the results from a statistically representative standpoint.

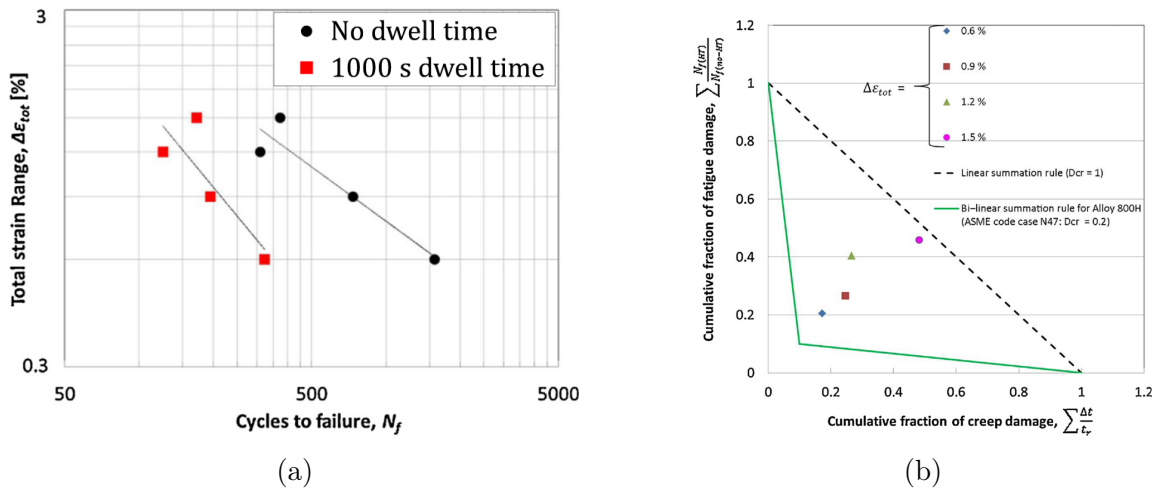


Figure 1.38: (a) Number of cycles to failure and (b) cumulative creep-fatigue damage for 1000 s dwell time diagrams by M. Kolluri et al.[135]. LCF tests conducted on 800H alloy specimens at 800°C.

Creep-fatigue conclusion: During high temperature HCF loadings, Incoloy 800H exhibits a relatively regular fatigue damage behavior, where damage accumulation is related to the accumulated plastic deformations and the crack initiation and propagation is mostly transgranular, aided by oxidation (in air-like atmospheres). The serrated stress peaks resulting from these fast tension-compression stress-strain cycles likely indicate the presence of DSA, thus indicating a stress concentration at the crack tips. Although this phenomenon has proven to affect the mechanical behavior of materials, recent studies demonstrate that DSA regimes on Incoloy 800H are related to high strain rate deformations (such as those induced by HCF) and the presence of dissolved atoms within grains microstructure. Thus, it not observed under slow deformation

rates. LCF damage in 800H is often accompanied by creep. Creep damage mechanisms include intergranular crack initiation and propagation, and grain boundary and precipitate decohesion. The dominant mechanism is ultimately determined by a combination of all waveform features. Cases where both (creep and fatigue) mechanisms are involved often result in a mixed crack propagation mode. The onset of oxidation-related precipitates is observed to promote intergranular crack initiation. Additionally, oxides nucleating in the vicinity of cracks are known to favor crack propagation as they act as crack widening agents. This material has proven to be highly strain-rate sensitive. The presence of particularly slow tensile strain rates (in the order of $1 \times 10^{-5} \text{ s}^{-1}$), as well as the eventual dwell-times between cycles, favors the onset of creep damage. This leads to a combined creep-fatigue damage accumulation. Depending on particular fatigue wavelength features, different levels of severity for creep-fatigue damage accumulation are observed: For instance, unbalanced load cycles of the S-F strain rate type (see Table 1.12) are associated with the highest levels of creep-fatigue deformation.

1.3 Outlook and remarks

The consideration of creep deformations is paramount for the accurate estimation of the service life of metallic components undergoing long-term high temperature loading regimes under any constant load. Once understood as a time-dependent deformation and described in its simplest form by an empirical power law, viscoplasticity is currently conceived as a complex deformation mechanism. The high service temperatures decrease the mechanical threshold to initiate the propagation of crystal defects (i.e., vacancies and dislocations), allowing slow creeping permanent deformations to occur even within low mechanical loadings. The mechanical response of the material is thereafter determined by the interaction between those defects and the material microstructure. Dislocation multiplication, dislocation pinning, solid-solution hardening, or the formation of a dislocation substructure, are examples of those interactions. Considering that some materials exhibit substantial microstructural changes (e.g., precipitate dissolution, nucleation or coarsening) after long term exposure to high temperatures adds an additional layer of complexity to this problem. Such is the case of Incoloy 800H, an austenitic steel alloy well regarded for its good mechanical properties and cost/weight ratio. According to the information presented throughout section 1.2, its creep behavior at temperatures roughly above 760°C and stresses $\lesssim 0.5 \times \sigma_y$ is characterized by a two-step creep strain rate minima, involving features such as:

- Dislocation-precipitate interaction
- Subgrain formation
- Dissolution of sub-micron intragranular Cr-rich M_{23}C_6 carbides (leading to solid-solution atoms)
- Nucleation and coarsening of precipitates at grain boundaries (aging)
- Precipitation of secondary phases: nitridation and oxidation
- Precipitate coarsening and cavitation damage

The following chapters address the development of a multi-scale creep model adapted to predict the service life of 800H components under creep loadings where such complex behavior is reported. Chapter 2 showcases the planning and results of an experimental campaign involving macro- and microstructural characterization techniques. Chapter 3 deals with the development of a mean-field creep model adapted to predict the complex creep behavior of the alloy. This

semi-physical model, inspired by approaches presented within section 1.1.3, includes formulations entitled to solid-solution hardening and improved stress sensitivity as to recover the creep deformation of 800H alloy within a physical framework. Chapter 4 summarizes the numerical improvements over a macromechanical finite element unified viscoplastic constitutive model. Modifications on the Norton power law respond to the specific creep phenomenology of 800H alloy. Finally, Chapter 5 deals with the multi-scale creep modeling aspect of this thesis, consisting in the exploitation of the mean-field model as a physically-founded inter- and extrapolation tool to complement creep results to feed into the creep deformation bank knowledge to identify the macromechanical finite element law.

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Experimental campaign

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This section describes the experimental campaign performed to characterize macro- and micro-mechanical aspects related to the high temperature creep deformation response of Incoloy 800H. Since information on the temperature dependency of elastoplastic and thermal constants of 800H was found in literature (see section 1.2.3), the macro-mechanical stage of this campaign is comprised exclusively of creep tests. These were conducted at temperatures falling within well defined stress and temperature range of interest. However, in compliance with the NDA signed between ULiège and Drever International (industrial partners sponsoring our experimental campaign), stresses, temperatures and non-normalized curves cannot be disclosed. All experimental curves recovered in the context of this experimental campaign are hereafter normalized by undisclosed arbitrary factors.

The material source and experimental methods, techniques and equipment used throughout this campaign is described in section 2.1, whereas the experimental results are presented in section 2.2.

2.1 Material and methods

2.1.1 Creep testing equipment

The creep machine used in this campaign is a Zwick/Roell (Z/R) Kappa 100DS property of the University of Liège and owned by the Materials and Structures (M&S) laboratory. It is suited with room- and high-temperature testing components. The description and technical specifications of the creep machine and its main components (furnace, load cell, extensometer, and pulling rods) listed hereafter are intended to provide the lector with the necessary context to comprehend the methodological decisions that shaped this experimental campaign. Note that the maximum allowable temperature and load during creep testing is often limited by the combination of these components.

- **Machine.** With the objective of conducting reliable high-temperature creep tests, the Materials and Solid mechanics (MSM) team in ULiège acquired a creep machine model Kappa 100DS from the Austrian company Z/R suited for high-temperature creep testing. Room temperature testing equipment is available; however such tests are considered inappropriate due to the machine loading limitations and concerns about the extensometer components being unsuitable to withstand the impact of such a test. The most important technical specifications of the machine are provided in Table 2.1.

Table 2.1: Main technical characteristics of Zwick/Roell Kappa 100DS creep machine.

Characteristic	Technical specification	Photograph
Load capacity	100 kN	
Test area depth	Unlimited	
Test area-width between drive screws	610 mm	
Test area-height	max. 1350 mm	
Crosshead strokes	1350 mm	
Lateral support of moving crosshead	Precision sliding bearing on four chromium plated columns (40 mm diameter)	
Test speed range	0.001 mmh^{-1} to 100 mmmin^{-1}	
Return speed	100 mm min^{-1}	
Crosshead speed accuracy	$\pm 0.1\%$ of setting (no load or constant load averaged over 10 mm or 5 s)	
Return stroke-encoder	0.068 mm	
Frame Dimensions	$860 \times 655 \times 2310 \text{ mm}^3$ (width $\times$ depth $\times$ height)	
Weight	840 kg	
Power requirements	230 VAC, 1 kV A	

- **Furnace.** Acquired together with the creep machine, the Z/R MP00850 furnace is engineered for creep testing. It is a resistance-based furnace, and combines high thermal inertia with excellent temperature control during long tests. The main technical characteristics of the furnace are provided in Table 2.2. The maximum heating rate advised by the manufacturer as a function of the temperature of the furnace is provided in Table 2.3.

Table 2.2: Summary of the main characteristics of the furnace used in combination with the creep machine.

Characteristic	Technical specification	Photograph
Furnace model	MP00850	
Maximum furnace temperature	1200°C	
Maximum specimen temperature	1150°C	
Maximum heating rate*	Up to 30 Kmin ⁻¹	
Cooling rate	Natural cooling	
Connection flange	60 mm	
*: limited by the current temperature of the furnace, see Table 2.3		

Table 2.3: Maximum heating rate achievable by the Z/R MP00850 furnace according to the manufacturer.

Temperature (°C)							100	150	200	250	300	350	400	450	500
Max. heating rate (°C min ⁻¹)							3	3	3	3	5	5	6	8	10
550	600	650	700	750	800	850	900	950	1000	1050	1100	1150	1200		
12	12	15	15	17	20	20	25	25	30	30	30	30	30	30	

- **Load cell.** The load cell of the machine is the device in charge of constantly monitoring the axial force. The machine was initially acquired with a 100 kN load cell, in accordance with the maximum load capacity of the machine (see Table 2.1). This load cell provides reliable force measurements (1% error) down to 200 N. This was deemed insufficient in creep tests involving forces down to 100 N. A new 10 kN load cell was therefore acquired, rated with 1% error force measurements down to 20 N. The main technical characteristics of both load cells are summarized in Table 2.4.

Table 2.4: Summary of the main technical characteristics of the available load cells. The accuracy class classification is given by the manufacturer (Z/R) according to ISO 376 standard. An accuracy class 0.5 means that the absolute measurement error is found within 0.011%-0.020% of the minimum resolution, whereas class 1 entails an error between 0.021%-0.050%.

Load cell 100 kN		Photograph
Load cell model	Xforce-K 100 kN (item N° 318932)	
Nominal force	100 kN	
Accuracy Class* 1	200 N - 100 kN	
Accuracy Class* 0.5	1000 N – 100 kN	
Installation height	106 mm	
Connection flange	60 mm	
Load cell 10 kN		
Nominal force	10 kN	
Minimum resolution	0.034 N (297×103 steps)	
Accuracy Class* 1	20 N - 10 kN	
Accuracy Class* 0.5	100 N – 10 kN	
Connection flange	60 mm	
Temperature influence	at 0-point: $\pm 0.001\%/K$ at measured value: $\pm 0.004\%/K$	

- **Load strings.** Upon testing, the specimen is mounted into the machine in connection with 2 or 3 pulling rods (2 for cylindrical specimens; 3 in case of using flat specimens). These are manufactured from a corrosion-resistant Ni-alloy, and the alignment is engineered following ASTM E1012. They are rated to withstand temperatures of up to 1200°C. No maximum service time as a function of the axial load or temperature is indicated.
- **High temperature adapters.** The connection between the pulling rods and the specimens is achieved through the use of two high temperature adapters specific to the specimen geometry. These are manufactured from MAR-M 246 corrosion resistant Ni-alloy. The maximum admissible thermal and mechanical loadings these components can withstand is given by the manufacturer in relation to the length of the creep test. The maximum applicable load according to the test temperature and time-length is given according to the geometry of compatible specimens in Table 2.5. The geometry of the compatible specimens (P58, D06 and D10) is discussed in Table 2.7.
- **Extensometer.** To allow reliable recording of axial displacements during a test inside the furnace, a specialized extensometer is used. It has long ceramic arms that connect the linear variable differential transducers (LVDTs)s to the specimen gauge length section. The extensometer is attached to the specimen by a combination of ceramic pieces that match the geometry of the specimen collar. The main technical characteristics of the extensometer are given in Table 2.6. A photograph showing the extensometer, the LVDTs with a specimen mounted on is presented in Figure 2.1b.

Table 2.5: Manufacturer instructions on the maximum admissible axial force that can be applied on the high-temperature adapters according to the temperature and time span. The specimen geometries referenced in labels "D06", "D10" and "P58" are described in Table 2.7.

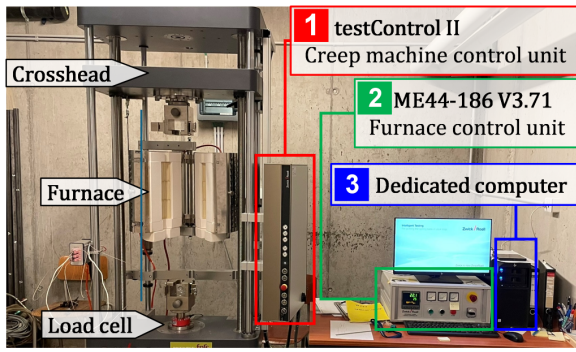
D06 adapters	Maximum axial force (kN) according to creep test duration		
Temperature (°C)	10 hours	100 hours	1000 hours
600	56.3	34.8	32.7
700	45.0	27.4	23.3
800	35.3	19.2	14.3
900	24.0	11.5	6.9
1000	9.7	3.2	2.4

D10 adapters	Maximum axial force (kN) according to creep test duration		
Temperature (°C)	10 hours	100 hours	1000 hours
600	110.0	85.0	80.0
700	88.0	67.0	57.0
800	66.0	47.0	35.0
900	47.0	28.0	17.0
1000	19.0	8.0	6.0

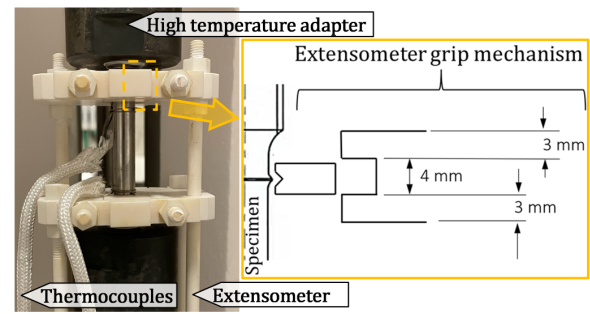
P58 adapters	Maximum axial force (kN) according to creep test duration		
Temperature (°C)	10 hours	100 hours	1000 hours
20	8.0	8.0	8.0
800	8.0	4.9	4.0
900	7.2	2.8	1.9
1000	4.7	1.3	0.9

Table 2.6: Main technical characteristics of our high temperature extensometer.

Characteristic	Technical specifications
Material (ceramic)	Main structure Al_2O_3 Replaceable inserts: ZrO_2
Maximum temperature	1200°C
Resolution	<1 μm
Maximum displacement	50 mm, regulated by adjusting the LVDTs screws (see Figure 2.1b)



(a) Creep machine layout.



(b) Illustration of the extensometer mounting mechanism.

Figure 2.1: Photographs of the creep machine Zwick/Roell Kappa 100DS in the Materials & Structures laboratory at the University of Liège. The main machine components and peripherals are highlighted.

2.1.2 Creep testing methodology

The SCTs performed in this experimental campaign followed the latest creep test standard ISO-204 [1]. It provides guidelines for test preparation, progressive creep (thermal and mechanical) loading application, and data postprocessing. The sequence of steps describing the temperature and axial force application methodology during a SCT can be disassembled into 5 steps (A to E) presented hereafter. A graphical description of these steps is provided in Figure 2.2.

- A While remaining at room temperature (T_0), apply and hold a tensile pre-load equivalent to 10% of the total axial load of the test (F). Hold the pre-load until step (D).
- B Activate the furnace and heat up the specimen until the target temperature (T_1) is reached.
- C Hold the target temperature for 60 minutes. This time span is specified in ISO-204 [1] as a way to ensure a stable temperature control during the experiment. In this particular case however, given the relevance of observing the first minimum creep rate entitled to a solid-solution hardening phenomenon, a modification of this time is imposed. To capture the creep hardening effect of solute atoms into the material matrix before these diffuse into Gbs, **this time is reduced to 15 minutes**. A similar approach has already been implemented by A.M. Young et al.[2] in a different context.
- D Increase the tensile axial load from pre-load ($0.1F$) until the total creep test force (F) is reached. The load increment is performed while imposing a maximum crosshead displacement velocity of 0.1 mm/min.
- E From this point onward, the creep test is started. The temperature and axial force are held constant until a user-defined maximum (displacement or test time) is reached, or until the specimen breaks.

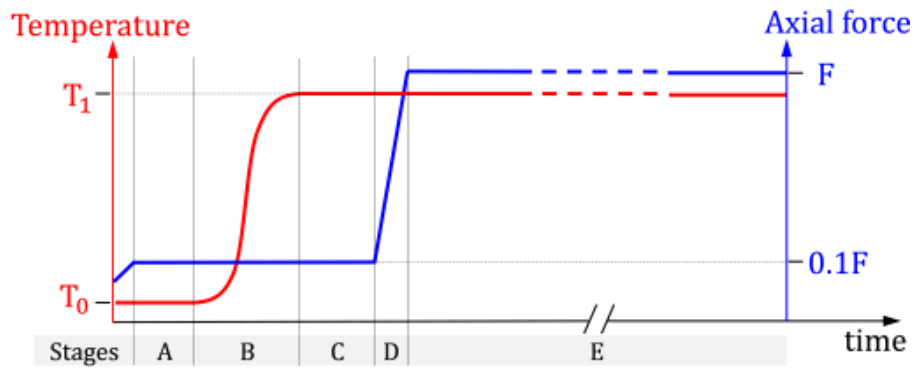


Figure 2.2: Standard loading history of temperature and axial force for a conventional SCT according to ISO-204 [1].

To conduct such a test, the sequence (A) to (E) is efficiently grouped into three different programming blocks into the Z/R software testXpert III. A detailed description of the machine operation addressing creep test programming and specimen mounting is provided in Annex 2C.

Creep specimen geometry

The machine components acquired by ULiège allow for a total of three different specimen geometries to be tested for creep. These are hereafter labeled "D06" (cylindrical specimen with 6 mm diameter), "D10" (cylindrical specimen with 10 mm diameter) and "P58" (flat specimen with $5 \times 8 \text{ mm}^2$ cross section). Geometry dimensions for machining purposes provided by the machine manufacturer are presented here in Table 2.7.

Table 2.7: Dimensions and manufacturing tolerances of specimens compatible with Zwick/Roell creep testing equipment in ULiège.

D06: Cylindrical specimen of 6 mm diameter	Detailed collar view
D10: Cylindrical specimen of 10 mm diameter	Detailed collar view
P58: Flat specimen with $5 \times 8 \text{ mm}^2$ cross section	Detailed collar view

2.1.3 Microstructural characterization techniques

The creep campaign comprises a series of microstructural characterization experiments intended to observe and/or quantify microstructural features in as-received, furnace-aged and creep tested material samples. These aim to improve the understanding of the physical relationship between the material microstructure and the macromechanical creep behavior of Incoloy 800H. Aspects such as grain size, primary (aging) and secondary (oxidation and nitridation) precipitates, are some of the key features addressed in this context. A brief summary of the experimental methods applied throughout this thesis is given hereafter:

Light Optical Microscopy (LOM).

It consists of the observation and qualitative characterization of the material microstructure through a microscope lens. The device belongs to the Metallurgic and Materials Science (MMS) laboratory, and corresponds to an Olympus BX60 LOM coupled with an OLYMPUS UC30 CCD digital camera and equipped with a motorized stage together with the OLYMPUS STREAM Motion® software.

Polishing technique: All material samples destined for microstructural characterization were first ground and polished to a surface tolerance of $1\mu\text{m}$. Samples destined for SEM analyses underwent a second polishing process consisting of a super finishing with oxide polishing suspension (OPS) down to a surface tolerance of $0.3\mu\text{m}$, while using a Struers Tegramin 30 automatic device.

Different light source modulation, surface treatments and lens arrangements were used to improve the accuracy of our observations:

- **Surface etching:** It consists of attacking the polished specimen surface with a chemical agent that reacts with certain microstructural compounds (e.g., precipitates or phases) thus highlighting them during LOM observations. The chemical agent is chosen depending on the chemical composition of the microstructure. In the case of Incoloy 800H, A.M. Young [3] recommends the use of Glyceregia (2 parts glycerol + 4 parts hydrochloric acid (HCl) + 1 part nitric acid (HNO_3)). For alloy 800HT (a variant of 800H with different Al, Ti and C contents), A. El-Magd et al.[4] opted for the use of a solution of 10 parts H_2O , 10 parts HCl and 1 part HNO_3 , whereas L.A. Spyrou et al.[5] used a mixture of HCl and H_2O . In this work, etching of 800H specimens destined for microstructural characterization was conducted using Glyceregia.
- **Polarized light microscopy (PLM):** The use of polarizing lenses allows to highlight certain phases during LOM observations without the need for an etching agent. To this end, the microscope detector must be allowed to detect RGB (color) light, and a set of polarized lenses must be properly arranged in series: one between the light source and the material specimen, and another between the material and the optical lens. During observation, the user should manipulate one of the polarizing lenses (preferentially the one close to the optical lens) until the surface elements sensitive to polarized light appear highlighted. Phases will react to the polarized light depending on their crystal structure and composition. For instance, titanium nitrides appear golden, whereas carbides reflect bright light. In this case, higher surface quality polishing leads to clearer observations. In this context, this technique is recommended as a complementary analysis of super-polished specimens intended for SEM analyses.

Scanning Electron Microscopy (SEM)

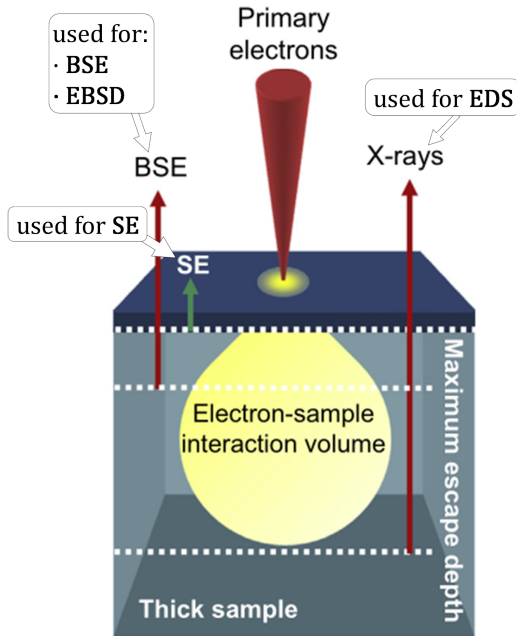
In electron microscopy, a coherent electron beam is shot onto the surface of a specimen. This is conducted within a confined vacuum-sealed chamber to avoid specimen contamination by external agents. Additionally, the specimen surface must be clean and polished to a high tolerance (generally $\leq 0.5\mu\text{m}$). The latter often requires the implementation of polishing techniques such as vibration polishing using OPS, or electrochemical polishing.

In this work, SEM analyses were carried out using a Tescan Clara SEM associated with Back-Scattered electron (BSE) for the images and a 129 eV BRUKER EDS with a silicon drift detector. The system was connected to ESPRIT® software for pre- and post-treatment of data. The accelerating voltage for SEM analyses was set at 15 kV, with a minimum scanning

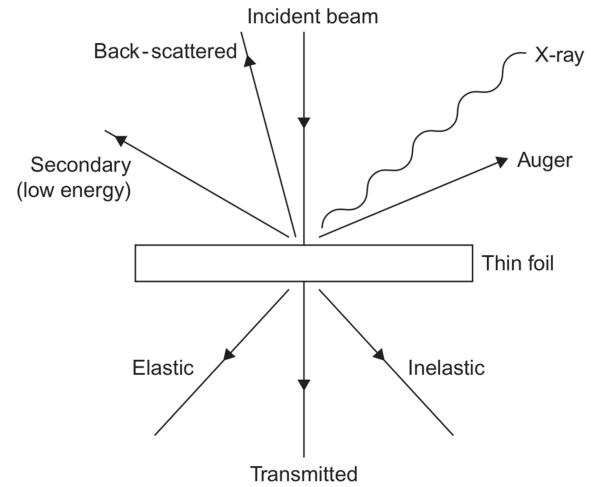
step size of electron beam of 1 μm , thus limiting the definition of particle observations to 1 μm as well.

The electrons contained within the incident beam react with those belonging to the specimen in analysis, generating electron emission (reflection and transmission) and plasmon emission (X-ray, Auger beams, heat) responses, as shown in Figure 2.3a. Different detectors are required to measure each of the output signals mentioned above. The postprocessing of this information allows the observation and quantification of microstructural features such as phase density, phase orientation, or chemical composition.

- Secondary electron (SE): this response beam is the result of the direct reflection of incident beam electrons. The information contained within is therefore interpreted as surface features, similar in nature to that of an optical microscope. The grayscale color modulation does not provide further information.
- Back-Scattered electron (BSE): the scattering phenomenon is defined as the dispersion of particles as they travel and interact with a surrounding energy field. In this context, a BSE beam is composed of those electrons emitted by slightly deep layers of material surface and scattered after interacting with upper surfaces as seen in Figure 2.3b. In the context of direct Back-Scattered electron (BSE) observation, the information contained within the beam can be interpreted in a similar manner to Secondary electron (SE), thus visualizing the 2D surface. The difference lies in the backscattering effect: the fact that certain electrons experienced scattering due to interactions with deeper material can be correlated to their density around the electron emission zones. The electron brightness level of the pixels in the post-treated grayscale image can indeed be correlated to the material density in the observed zone.
- Electron Backscattered Diffraction (EBSD): taking advantage of the scattering concept, this technique consists in analyzing the backscattered electrons and extracting information related to the local crystal orientation. Contrary to the techniques described here above, an EBSD analysis requires mapping the specimen surface using a thorough array of highly-focused incident electron beams. The information gathered upon completion of this lengthy process (often taking hours) is later post-processed and presented in a data file listing the local misorientation per beam shot. This information can be used to construct bi-dimensional texture maps, mean average misorientation maps, or calculate ρ_{GNDs} .
- Energy-Dispersive X-ray Spectroscopy (EDS): it consists of the semi-quantitative stoichiometric analysis of the specimen based on the wavelength-based analysis of the X-ray response emitted by the specimen. Similar to EBSD, the application of this technique entails a local analysis of the X-ray response using high-focused electron beam. The zone of interest in the specimen, designated by the user (e.g., single point(s), line or surface), is then mapped automatically using a series of focused electron beam shots. These follow the user-defined zone of interest, arranged in either a point-based (1 shot per point), linear-based (1D array) or surface (2D array) configuration. Given the beam definition limitations and method inaccuracies, the resulting information is regarded as semi-quantitative in nature. The scientific validity of the information recovered from EDS observations is attained only if consistent with prior knowledge on the microstructural characteristics and chemical composition of the material.



(a) Scheme showcasing all electromagnetic beam responses by a thin material specimen. After [6].



(b) Fundamental electron beam responses of a thick specimen under SEM. After [7].

Figure 2.3: Graphical representation of the electron- and X-ray output signals coming from a material specimen after excitation in SEM.

Indentation

Indentation is considered a non-destructive type of test. It consists of pressing a diamond tip onto the surface of a specimen and measuring the elastoplastic force-displacement-time response using a special integrated device. The post-processing of these curves allows the extraction of elastic material properties such as elastic modulus and surface hardness [8]. Studies on the extraction of creep properties have also been conducted [9, 10]. The geometry of the diamond tip is standardized (e.g., Vickers, Brinell, or conical, to name a few), and is chosen according to the length scale of the experiment, the force level, and material.

Indentations for this project were conducted at the University of Liège on a Universal EMCO M1C 010 hardness testing machine belonging to the MMS laboratory. It was configured with a Vickers tip. Two indentation techniques were employed: micro-indentation Vickers (μV , with an axial force of 0.05 kg) and macro-indentation Vickers (MV, with an indentation force of 10 kg).

2.1.4 Material samples

Heat-treated specimens: first material acquisition

In preparation for this PhD. project, a total of 4 Incoloy 800H hot-rolled metal sheets were acquired during 2018 by Drever in the form of “scrap metal” (i.e., second-hand material, not acquired from the alloy manufacturer). Two of these sheets, of 12 and 18 mm thickness, respectively, were used to extract cylindrical specimens of 5 and 10 mm diameter. The geometry of these specimens is hereafter referred to by D05* and D10*, where the “*” symbol is used to indicate that the dimensions and the manufacturing process of these specimens date back to 2018. Given that the ULiège creep machine was at the time still in the process of acquisition, the manufacturing process of the specimens was conducted with only basic notions of the

required dimensions for specimen compatibility to the creep machine components. The original geometrical dimensions of specimens D05* and D10* are given in Figure 2.4.

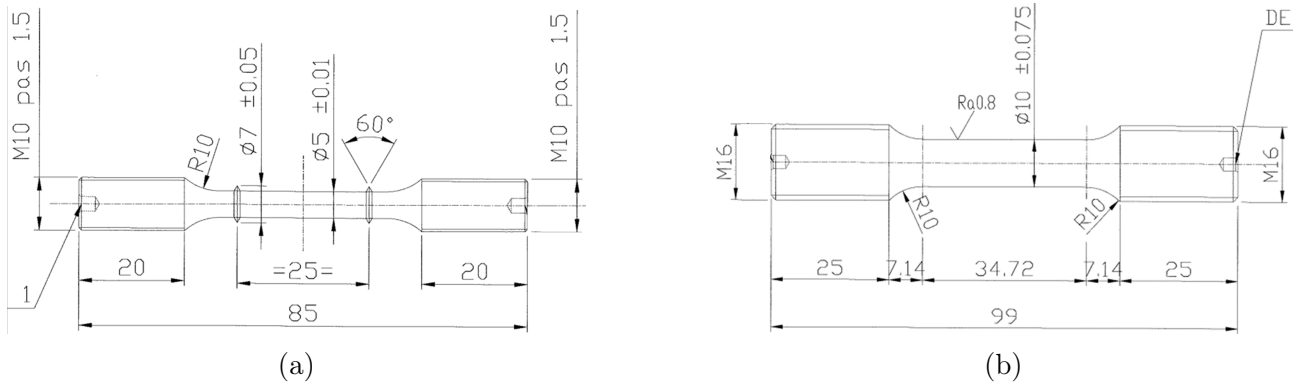


Figure 2.4: Geometrical dimensions of Incoloy 800H specimens (a) D05* and (b) D10*, manufactured during 2018 by a subcontractor of Drever International. Note that the collar geometry in (a) is not compatible with the one required by the Z/R Kappa 100 DS machine (compare to Table 2.7), whereas for (b) it is simply not present.

The last 2 sheets acquired at the time were 6 mm thick. No specimens were cut from them. A summary of the initially acquired 800H material sheets and their use during this project is provided in Table 2.8. These are identified with a tag where the first number indicates the sheet number of this type, "S" indicates it is a sheet, and the last 2 digits indicate the sheet thickness (e.g., 1S12 is the 1st sheet acquired of 12 mm thickness).

Table 2.8: Summary of different sheet thicknesses and their relation to the D10* and D05* geometries available since the beginning of this research project (2018), preceding the start of the PhD. of the student (January 2021).

Sheet	Dimensions (mm ³)	Destination
1S06 & 2S06	300 × 420 × 6	· Sheets have remained in furnace ever since. · A single piece of approximately 300 × 100 × 6 mm³ was retrieved from 1S06 in July/2023 for microstructural characterization. The results of such tests are provided in section 2.1.3.
1S12	650 × 400 × 12	Sheet used to manufacture D05* specimens in 2018
1S18	800 × 400 × 18	Sheet used to manufacture D10* specimens in 2018

According to literature [2, 3, 11–13], exposure of 800H to a high N₂ gas concentration combined with high temperatures promotes nitrogen corrosion. The precipitation of Cr and Al nitrides under high-temperature and high-N₂ atmosphere in 800H alloy was independently studied by A.M. Young et al. in [3, 14]. These studies were not available when this project was first conceived, and regardless, microstructure evolution during long term exposure in a realistic industrial environment is not addressed in the academic research of A.M. Young. The two 6 mm sheets (1S06 and 2S06), all D05*, and all but one of the D10* specimens were introduced into an industrial furnace under the supervision of Drever industrial partners and engineers. The objective of this treatment was to enable the material to age and acquire the microstructural

and mechanical characteristics of a component after service in a realistic industrial environment. While the specific thermomechanical and environmental conditions cannot be disclosed, they correspond to a combination of high- and low-frequency thermal cycles reaching up to 1000°C within a N₂-rich environment at sea-level atmospheric pressure. For reference, a similar environment corresponding to that of a CAL or a CGL as is described in [15]. To study the microstructure evolution of the material under this particular environment, a number of specimens have been retrieved from the furnace on a yearly basis since 2019. Along this project, a total of 5 batches of furnace-aged samples were recovered. These are hereafter identified as "Nx", where "x" stands for the number of years in-furnace (i.e., $0 \leq x \leq 5$). A summary of all specimens counted up until the moment where the last N5 batch was retrieved is given in Table 2.9. These specimens were exclusively destined for microstructural characterization.

Table 2.9: Description of the Incoloy 800H specimens retrieved from the furnace by Drever engineers. All specimens were placed in the furnace since August-2018.

N-batch	Date of retrieval	Retrieved 800H specimens
N0	2018	1 × D10* (control sample)
N1	August-2019	10 × D10* + 10 × D06*
N2	31-July-2020	10 × D10* + 10 × D06*
N3	17-August-2021	20 × D10* + 20 × D06*
N4	19-August-2022	12 × D10* + 10 × D06*
N5	27-July-2023	6 × D10* + 6 × D06* + 1 × sheet piece from 1S06
still in-furnace, to 09/2025		89 × D10* + 113 × D06* + 1S06 & 2S06 sheets

The nomenclature described in Figure 2.5 is used to identify each specimen. It contains information on the source (sheet, cutting direction with respect to the sheet rolling direction), a number for the specimen in-sheet layout, and the corresponding in-furnace N-batch. The example provided in Figure 2.5 describes a specimen of geometry D06, obtained from sheet number 2 of 12 mm thickness alongside the longitudinal (rolling) direction, with the number tag 011 within the in-sheet layout, and retrieved from the industrial furnace, after 4 years.

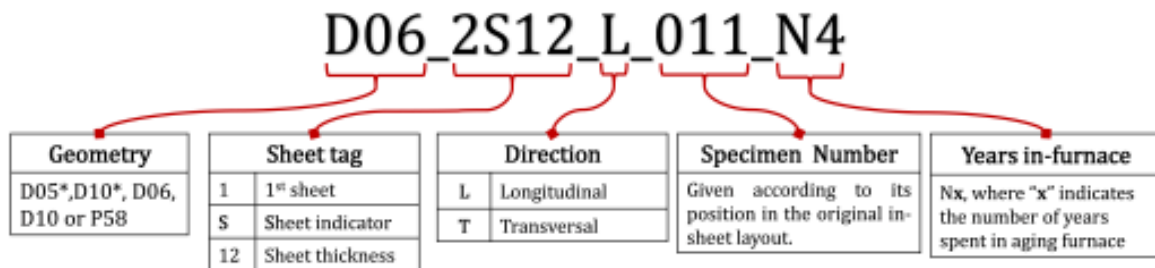


Figure 2.5: Nomenclature for all Incoloy 800H specimens used to identify specimens during this project.

Creep specimens: second material acquisition

Samples of geometries D06, D10 and P58 (see Table 2.7) were cut from new 800H sheets with thicknesses of 12, 18 and 5 mm respectively. All sheets were acquired in the form of "scrap metal", and were confirmed to be in AR condition (e.g., SA+WQ + hot rolling). The inventory of all sheets acquired in 2023 is given in Table 2.10.

Table 2.10: Summary of hot-rolled sheet 800H material acquired for the fabrication of creep specimens.

Tag	Dimensions mm ³	Destination
1S05 2S05 3S05 4S05	300 × 420 × 5	Sheets received during July-2023 for the manufacturing of specimens of flat geometry P58
3S06 4S06 5S06 6S06	300 × 420 × 6	Sheets received during August-2023
2S12 3S12 4S23 5S12	300 × 420 × 12	Sheets received during August-2023 for the manufacturing of specimens of cylindrical geometry D06
1S20 2S20 3S20 4S20	300 × 420 × 20	Sheets received during August-2023 for the manufacturing of specimens of cylindrical geometry D10

2.2 Experimental campaign

In this work, the characterization of the creep deformation response of Incoloy 800H in relation to its initial (AR) and transient microstructural features was achieved by means of a thorough experimental campaign consisting of numerous high-temperature creep and microstructural characterization tests. This section provides the reader with a comprehensive summary of all tests and results obtained over the 4+ years of this PhD. project. The campaign is described following a logical order, thus maintaining coherency throughout the chapter. Experiments entitled to the characterization of the material microstructure in AR, heat-treated and creep-affected (deformed or damaged after testing) material are described in section 2.2.1. Creep experiments are later described in section 2.2.2. In the public version of this thesis, all creep curves are normalized by an undisclosed factor for confidentiality reasons.

2.2.1 Microstructural characterization

All microstructural experiments were conducted in cooperation and under supervision of researchers from MMS team from the Aerospace & Mechanics Department of University of Liège. All specimen samples were extracted using a Charmilles Robofil Electric Discharge Machine (EDM) model 310. Once extracted, samples were placed into individual 30 mm diameter Bakelite molds compatible with all polishing machines and microscopes. A summary of the experimental methods applied during our microstructural campaign is provided in section 2.1.3. In addition, a more detailed description of the procedures with illustrations is provided in section **methods** of the article of C. Rojas-Ulloa et al.[16], (the pre-print version of which is shared here in Annex 2A).

Table 2.11: List of all microstructural characterization experiments conducted on different types of Incoloy 800H source material at the University of Liège.

Material source	Experiment	Objective of the experiment	Tag
A) First acquisition: heat-treated material	LOM - etching	Measurement of AGS and grain aspect ratio	A1
	MV indentation	Microstructure homogeneity at the macro-scale	A2
	μ V indentation	Microstructure homogeneity at the micro-scale, nitridation-induced hardening	A3
	SEM - BSE/EDS	Identification of primary and secondary precipitates	A4
B) Second acquisition: new AR material	LOM - etching	Measurement of AGS and grain aspect ratio	B1
	LOM - PLM	Microstructure observation and identification of primary precipitates	B2
	SEM - BSE/EDS	Identification of primary and secondary precipitates	B3
C) Second acquisition: Creep tested specimens	LOM etching + PLM	Measurement of AGS and grain aspect ratio after creep	C1
	SEM - BSE/EDS	Identification of primary and secondary precipitates	C2
	SEM - EBSD	Quantization of ρ_{GND}	C3

Methodology

Microstructural characterization tests were conducted on 3 material samples from 3 different sources. These are:

- A First acquisition material: this material source corresponds to the heat-affected specimens described in section 2.1.4. The specimen NX-batches are described in Table 2.9.
- B Second acquisition material, AR condition: it corresponds to sheet specimens extracted from some of the 800H sheets acquired in AR condition described in Table 2.10.
- C Second acquisition material, creep-tested condition: material samples extracted from creep-tested specimens, manufactured at the University of Liège from sheets 2S12 and 1S20 from Table 2.10.

A detailed description of the tests involved in each of the microstructural characterization instances, designated here above by the letters A, B, and C, is provided in Table 2.11.

Experiments A

The objective of these experiments was **to investigate the microstructure evolution** in Incoloy 800H samples exposed to an industrial environment consisting of a combined high- N_2 gas at atmospheric pressure and high temperatures consistent with those found in a CAL. The experiments, results and conclusions derived from the experimental observations were all published in C. Rojas-Ulloa et al.[16]. The peer-reviewed pre-print version of the manuscript, describing in detail the microstructural characterization techniques, results and conclusions driven from experimental observations, is given in Annex 2A of this thesis chapter. Nevertheless, a brief summary of the experimental outcomes of these experiments is given hereafter.

A1: Samples from batches N0 (AR condition) up to N5 (5 years time in industrial furnace) were extracted, mounted in bakelite molds, polished to a $1\text{ }\mu\text{m}$ tolerance and etched in Glycergia. The etching process lasted no more than 60 seconds per sample. Given the high presence of intergranular carbides, the etching process allowed Gbs to stand out during LOM observation. The AGS of each sample across their longitudinal and transversal directions were measured by means of the line intercept procedure following ASTM E112 standards [17]. The intersecting lines were added digitally to the images using ImageJ [18] (see Figure 2.6a), however the counting of intersections and calculations were conducted manually. The AGS measurements for all N-batches are given in Figure 2.6b.

Finally:

- The AGS of the 800H material is found within $148.25 \pm 8\text{ }\mu\text{m}$. The magnitude is approximately ASTM Gr. 2.5 in ASTM units. The study shows negligible grain size variation throughout samples across batches N0 to N5. No correlation was found between the AGS variations and the years spent in a high temperature industrial environment.
- The grain aspect ratio was found at 1.3 ± 0.1 . It was calculated automatically as the ratio between the major and minor axes of the ellipses drawn around 20 grains across all samples, using the same ImageJ software. The grain aspect ratio was not found to exhibit any particular orientation preference.

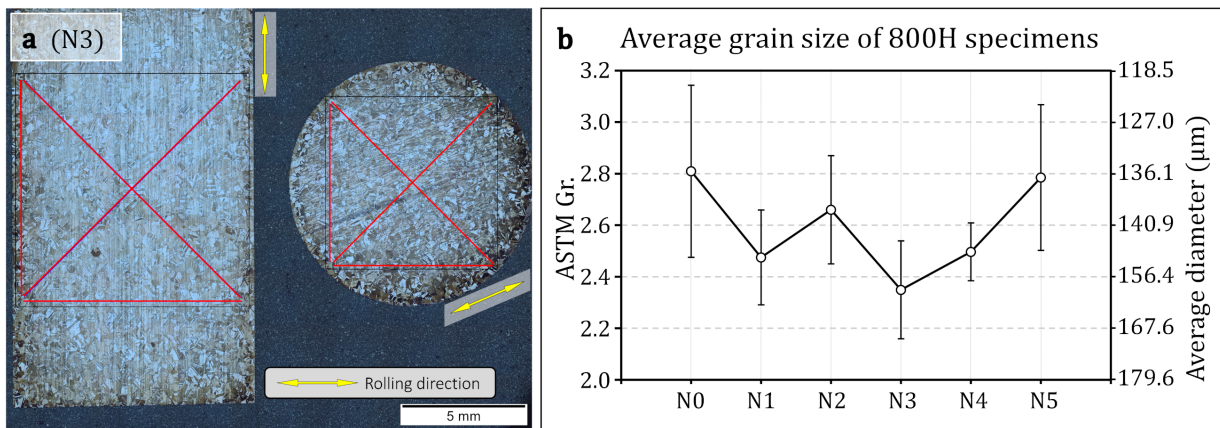


Figure 2.6: (a) Assembled LOM image of an in-bakelite sample belonging to a specimen of batch N3. The red lines are used for calculating the AGS of the alloy via the linear interception method. (b) AGS measured for each N-batch specimen addressing both ASTM Gr. and the equivalent average grain diameter (μm). Figure after [16].

A2: The source material used to manufacture the specimens (cylindrical geometries D05* and D10*, see Figure 2.4) corresponds to hot-rolled sheets. Because these were acquired in the

form of scrap metal, no information on the hot rolling process was provided. To investigate the possible microstructural inhomogeneities induced by the hot-rolling process, the surface hardness homogeneity of the material samples in both transversal and longitudinal directions was studied by means of MV experiments. These tests were conducted in an EMCO M1C 010 indentation machine, with an axial force of 10 kg. The cross-like pattern drawn with the indented is showcased in Figure 2.7a of the manuscript [16], whereas the results for both transversal and longitudinal sections of the cylindrical specimens of batched N0 to N5 are shown in Figure 2.7b. The analysis of results leads to the following conclusions:

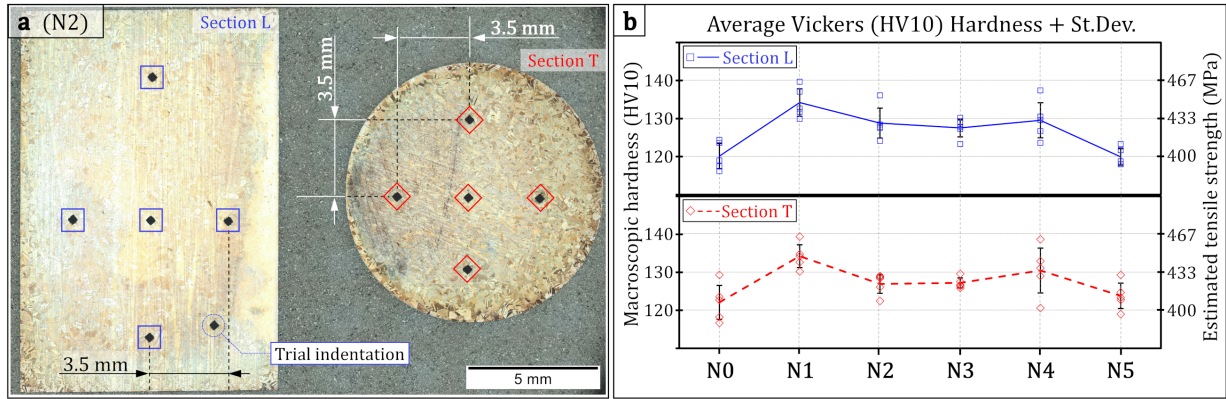


Figure 2.7: (a) LOM image showcasing the spatial distribution of MV indentations for specimen of batch N2. (b) HV10 MV hardness and estimated tensile strength of all N-batch specimens of Incoloy 800H belonging to the "first acquisition". Figure after [16].

- The surface hardness difference between the central indentation and those close to the surface of each sample was deemed negligible. From a macromechanical perspective, the material is thereafter considered homogeneous.
- The MV hardness of all material specimens from N0 to N5 batches give a MV hardness (HV10) of $120 \leq \text{HV10} \leq 140$. With consideration of the equivalent yield stress variation range being $400 \leq \sigma_y \leq 435$ MPa, this difference is considered negligible.

A3: The possibility of secondary precipitates (corrosion) induced by the long-term high-temperature exposure of these specimens under a high- N_2 concentration environment with occasional water vapor was investigated by means of a μV indentation campaign. The indentations with a force of 0.5 kg were performed at 13 different depths in arranges of 5×13 on each longitudinal sample, as seen in Figure 2.8a in [16]. The μV indentation maps are plotted in Figure 2.8b of [16] for all samples N0 to N5.

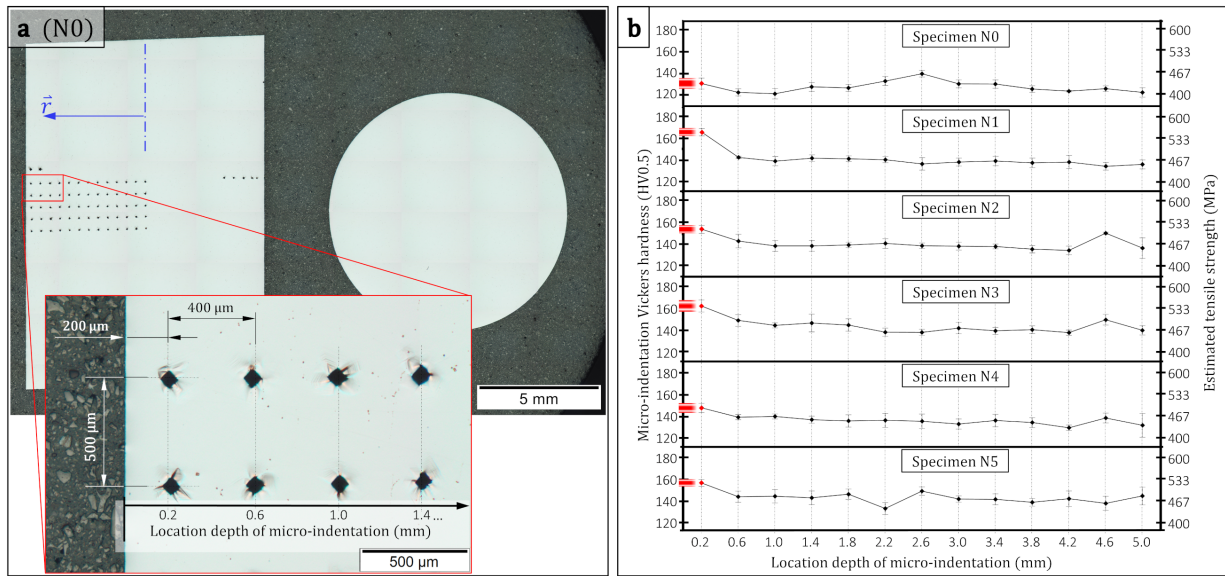


Figure 2.8: (a) Spatial distribution of μV hardness measurements. The blue arrow indicates the radial direction from the core of the originally cylindrical specimen. b) HV0.5 μV hardness and estimated tensile strength of all N-batch specimens of Incoloy 800H belonging to the "first acquisition". Figure after [16].

- Compared to N0 (in AR condition), the μV hardness (HV0.5) at the zone closest to the surface in contact with the environment exhibit an increment throughout samples N1 to N5. In particular, the maximum HV0.5 hardness of 170 is reached at N1. Thereafter, the μV hardness at the surface decreases to an HV0.5 of approximately 140. The hardness increment is deemed consistent with nitridation, however more studies are needed to comprehend the reason behind the decrement observed from N2 to N5.
- Disregarding the μV indentation close to the surface material, the inner (core) material hardness mapping exhibits variations consistent with the presence of a grain structure with occasional precipitates. Variations in magnitude are deemed consistent with that of a homogeneous material core.

A4: The investigation of the possible precipitation of corrosion-related secondary phases was followed by SEM observations. Given the cylindrical shape of the samples in the furnace, the analysis was conducted on the transversal section of each specimen. A total of 4 zones per section were analyzed: one central and 4 distributed around 4 equidistant edges, as seen in Figure 2.9a. Primary observations of each zone were conducted using SE and BSE detectors. In cases where microstructural features of interest were observed (e.g., precipitates), these were identified via EDS analyses. The latter is named "EDX" in C. Rojas-Ulloa et al.[16] article (see peer-reviewed pre-print in Annex 2A); this nomenclature discrepancy is also common in literature. The combination of EDS (semi-quantitative stoichiometric characterization) and BSE (material density-based grayscale coloring) allowed us to rapidly and reliably identify all primary and secondary precipitates larger than $1\text{ }\mu\text{m}$ in diameter. Finally:

- The core material, unaffected by the diffusion of foreign agents but exposed to high temperatures, exhibited a progressive nucleation and coarsening of Cr-based M_{23}C_6 precipitates exclusively at Gbs. Such an event is consistent with **aging**, a common occurrence for stainless steels, including 800H [13].
- SEM Observations conducted at the edges of the transversal specimen sections showed evidence of progressive **internal oxidation**. Despite the structural integrity of the pro-

protective Cr-based oxide (see Figure 2.9b, after 5 years in furnace), internal oxygen diffusion is evidenced by the nucleation of oxides such as Al_2O_3 , SiO_2 , and Fe-based oxides. According to Figure 2.10a, the maximum oxide penetration depth reached after 5 years (N5) is of approximately 50 μm .

- The presence of nitrides nucleating within the edge of all furnace-aged specimens (N1 to N5) was ascertained via combined BSE/EDS analyses. Compared to oxidation, nitridation appears to be a much more aggressive corrosion mechanism. After 5 years, it reached up to 500 μm from the surface (a distance 10 $\times$ deeper than that of oxidation, see Figure 2.10b). At this point however, a distinction between the nucleation of Al and Cr based nitrides must be made:
 - AlN: The high thermodynamic stability of these precipitates, first revealed in CALPHAD simulations (see Figure 1.35) and observed in-situ during these analyses, means that these will be the first nitrides to nucleate upon increasing the N concentration in-matrix. In other words, new (Cr) nitrides will not form until all free Al atoms are depleted in favor of AlN nucleation. With consideration of this fact, **AlN were used as indicators to locate the maximum nitridation penetration depth. Nucleation of AlN was predominantly intergranular.**
 - CrN & Cr_2N : These nitrides were observed to nucleate into much coarser precipitates in both intra- and intergranular configurations. However, conversely to what was calculated in equilibrium diagrams in Figure 1.35, a high surface density of Cr-based nitrides was first observed throughout the edges of sample N1. **This peak surface density of Cr nitrides in N1 was followed by their progressive dissolution from N2 towards N5.** Such a behavior can be empirically correlated to the surface hardness variation detected throughout the μV experiments described here above in A3.

At first glance, it can be argued that the findings described herein contradict those reported by A.M. Young et al.[2, 3, 13, 14]. The authors study solution (gas) nitridation in 800H within a controlled environment where oxides penetration is not observed. In our case, nitridation is studied within a non-controlled environment: the internal diffusion of oxygen into the material matrix affects the precipitation of nitrides. A thermodynamical study inquiring on the oxidation vs. nitridation competition in Inocloy 800H is presented in Annex 2B.

- With few exceptions, the maximum penetration depth of nitrides and oxides around each transversal section was not homogeneous. Discrepancies of up to 3 times in the maximum penetration depth were observed (see for instance the maximum oxidation depth in transversal section N3, ranging from 20 μm up to 60 μm in Figure 2.10a). This is attributed to a non-uniform gas and heat flux in combination with spontaneous leakages and impurities contaminating the otherwise sterile atmosphere. For comparison, a perfectly controlled atmosphere (e.g., consistent throughout the entirety of the experiment) leads to a clear parabolic diffusion curve of the type $x = at^b$, where x is the penetration depth, and a and b are phenomenological constants [14]. In this PhD. context, the divergence from a conventional parabolic trend is explained by a water leak occurring around the 3rd year of samples in-furnace.

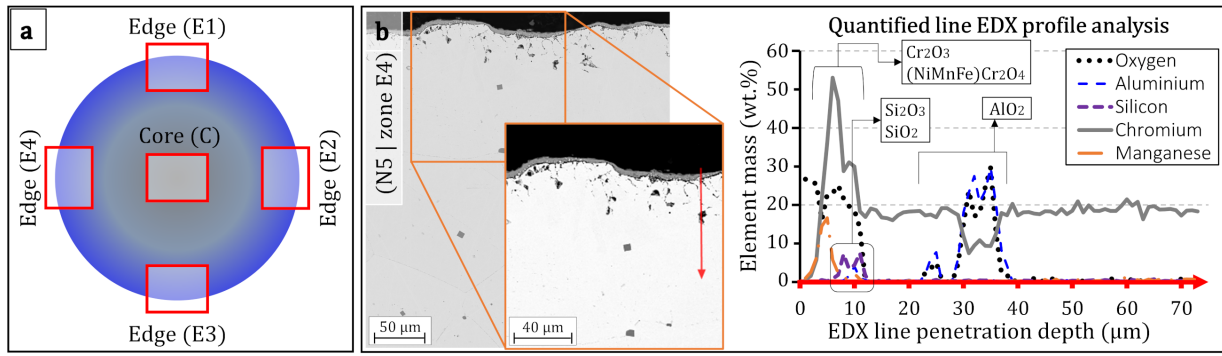


Figure 2.9: (a) Illustration of the spatial distribution and nomenclature of zones of interest in each transversal (T) section when conducting SEM analyses. (b) Identification of oxidation-related precipitates in zone E4 of specimen N5 (5 years in industrial furnace) throughout a line EDS (named "EDX" in the figure) profile analysis. Figure after [16].

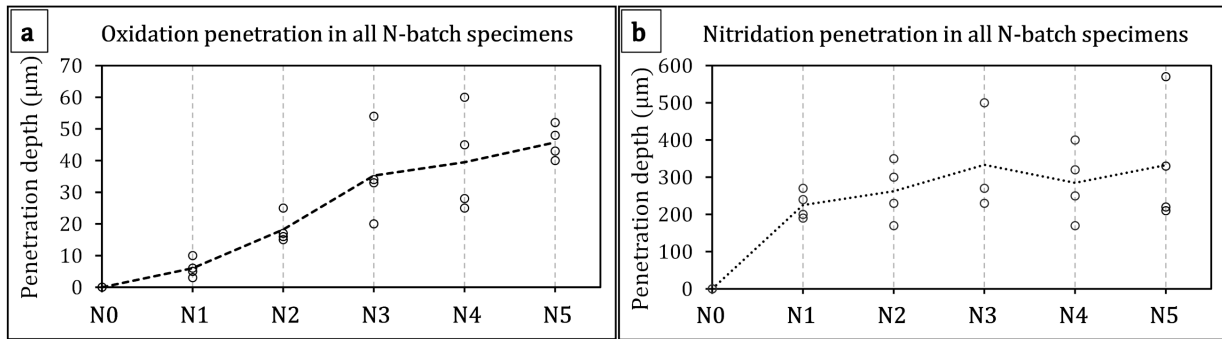


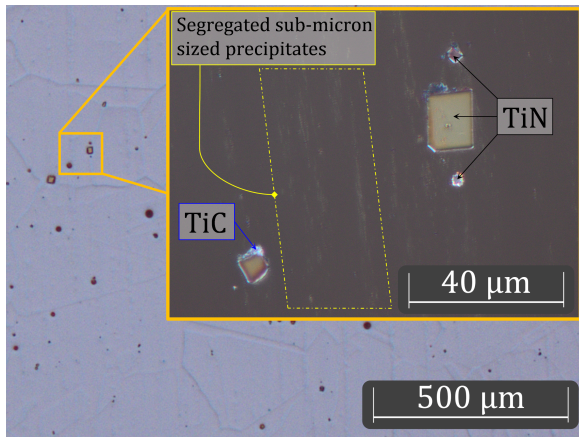
Figure 2.10: (a) Oxidation and (b) nitridation penetration depth based on microstructural observations of the deepest precipitate found. The hollow circular points indicate the maximum depth measured at each edge zone of interest (zones E1 to E4, see Figure 2.9a). Figure after [16].

Experiments B

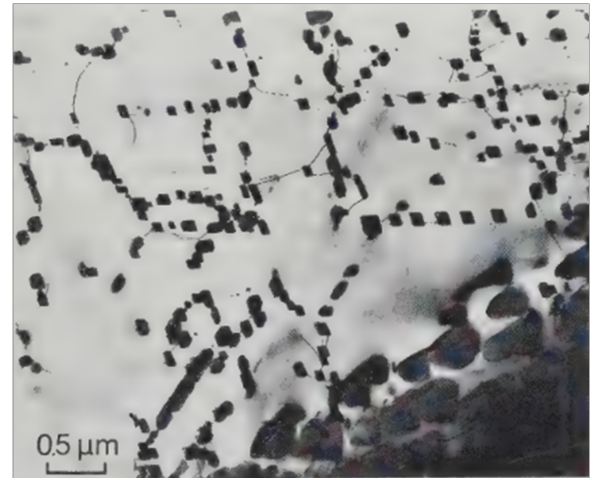
B1 The AGS of new AR material was measured on coupons extracted directly from sheet 2S12 (see Table 2.10). The procedure, consisting of etching-LOM observations and application of the linear interception method, was similar to that followed for stage A1. An average grain size (diameter) of $136.6 \pm 13\mu\text{m}$ was obtained. The results were found well within the ASTM Gr. 2.5 size range previously calculated for heat-affected material batches in AR condition (see Experiments A).

B2 The use of PLM allowed the correct identification of the following key primary precipitates:

- Intragranular sub-micron sized carbides (Cr_{23}C_6 and TiC) nucleating during the WQ process. As seen in Figure 2.11a, they appear to be arranged into segregation lines, following the rolling direction of the source material sheet. A TEM micrograph captured from a similar AR 800H material is provided in Figure 2.11a for comparison.
- Massive agglomerates of sub-micron sized TiC precipitates nucleating around larger TiN , evidenced as a bright light reflection around the TiN in Figure 2.11a.



(a) Bright light reflected by sub-micron carbides during PLM in ULiège. Figure after CITE ROJASULLOA SPRINGER, NO DOI YET.



(b) Submicron carbides nucleating at dislocations observed via TEM. After [19].

Figure 2.11: Sub-micron sized $M_{23}C_6$ and TiC carbides observed in 800H in AR condition.

B3 SEM observations using SE and BSE detectors revealed a completely standard AR Incoloy 800H microstructure. As observed during B2, primary precipitates correspond to standard TiN decorated by TiC, whereas sub-micron $M_{23}C_6$ and Ti(C,N) (see Figure 2.11a) could not be identified due to SEM electro beam precision.

Experiments C

C1 Microstructural observations of the material samples tested under creep until failure (i.e., reaching very high deformations or until fracture) were performed exclusively under LOM. Due to the risk of depressurization explosions occurring during the vacuum process at the SEM chamber, SEM analyses could not be conducted. The goal of these experiments was to observe and ascertain microstructural changes during creep. These include grain deformation, void nucleation and coalescence, and fracture propagation mode. To this end, three creep tested specimens were destined for this analysis:

- C1a: Figure 2.12a characterizes the sample at the lowest temperature tested during the creep experimental campaign of this work. At 40 MPa, it reached the smallest creep deformation to rupture of all our experiments (≈ 0.20).
- C1b: Figure 2.12b characterizes the sample at the highest creep temperature tested in this PhD. project. The analysis of this sample was motivated by the unusually large creep deformation reached during the test. The creep elongation reached $\tilde{70}\%$, and the test was stopped automatically before fracture due to safety concerns.
- C1c: Figure 2.12c characterizes the sample after creep rupture following the longest creep test performed in the context of this PhD. project.

The samples analyzed under LOM revealed a grain structure decorated with intergranular voids and cracks consistent with creep damage. The density and size of cracks were evidently higher at the fracture (or necking, in the case of sample C1b, see Figure 2.12b) zone of the specimens. Void formation mechanisms such as grain boundary decohesion, grain boundary-precipitate decohesion (see in Figure 2.12b), and void nucleation at triple joints were observed.

Reliable identification of microstructural features within highly damaged zones (e.g., fracture or necking) could not be attained due to the high density of voids and surface scratches. According to MMS metallurgists, the pre-existing voids act as sinks, where polishing and etching compound particles are trapped during the sample preparation process. Numerous surface scratches produced by SiC and oxide particles trapped and released during polishing, in combination with highly darkened surface areas due to etching compound particles trapped at voids, severely affected the quality of the samples.

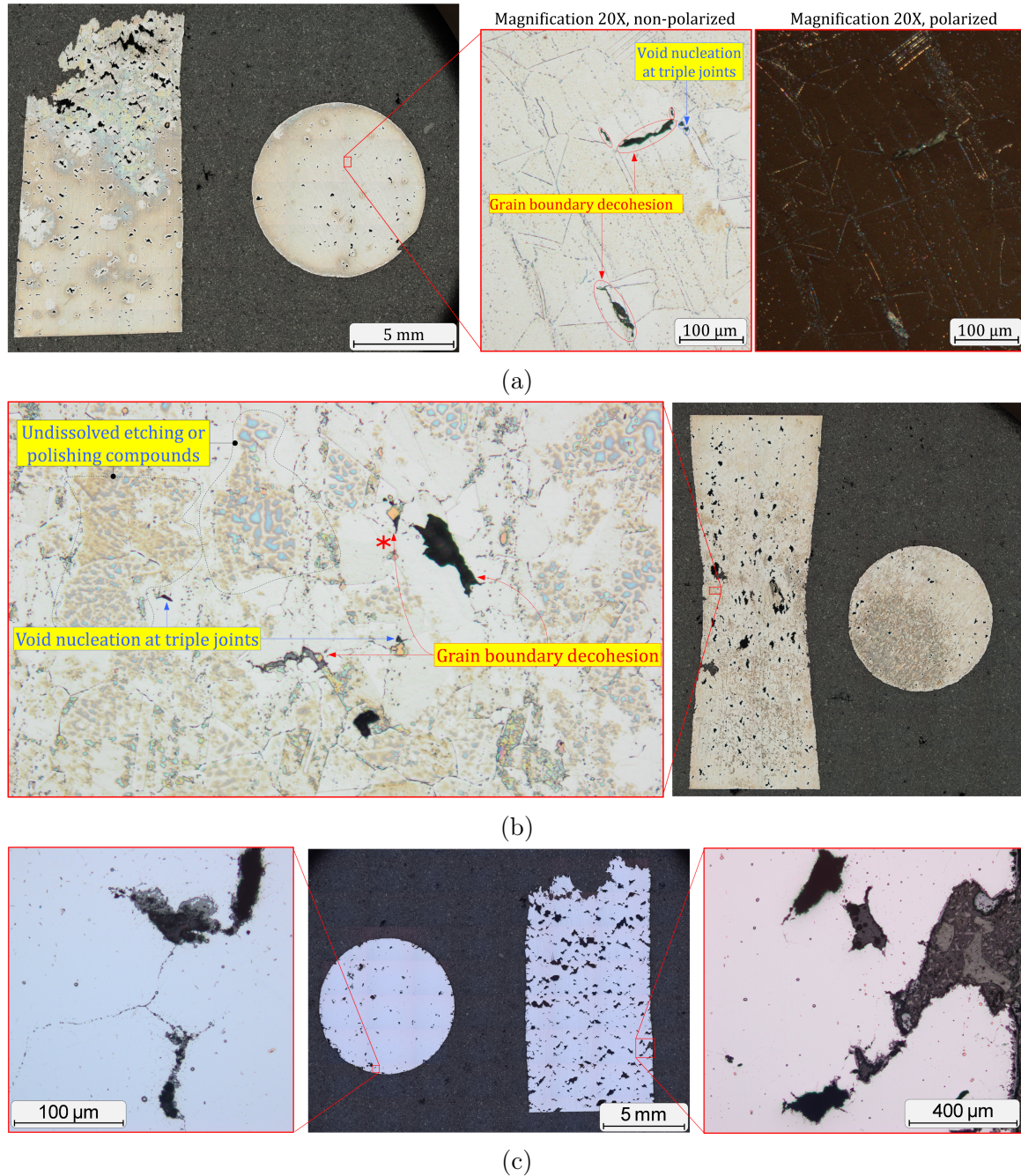


Figure 2.12: LOM and detailed PLM observations of creep tested specimens. Notable void formation and coalescence mechanisms are identified throughout.

C2 Microstructural changes induced during high-temperature creep, excluding damage, were identified by ex-situ SEM observations conducted for a single stress-temperature creep regime. To exclude damage from the analysis, all samples (transversal sections) were extracted from

specimen zones exhibiting low or null creep damage. A total of 3 samples were extracted from 3 tests stopped at different times. These were:

- C2a. Creep test stopped to analyze the material microstructure after reaching the first solid-solution hardening creep rate peak.
- C2b. Creep test stopped to analyze the material microstructure after reaching the steady-state creep rate, i.e., the second creep strain rate plateau.
- C2c. Creep test carried out to rupture. To observe microstructural changes under SEM while avoiding the vacuum pressure problems caused by the high density of voids in highly damaged material, sample C2c was extracted from the zone near the collar of the creep-tested specimen.

Two zones of interest were defined for each transversal specimen section: core material (center, presumed to be affected by aging) and surface material (edges, reportedly affected by corrosion). The findings are as follows:

- **Core material:** Observations revealed a slightly larger amount of intergranular Cr-based $M_{23}C_6$ carbides throughout the test, consistent with aging. Because of the accuracy limitations of the equipment, observation of the dissolution of sub-micron sized $Cr_{23}C_6$ precipitates was not possible.
- **Surface material:** The BSE and EDS analyses of all C2 samples provided a valuable insight on the formation and evolution of the protective Cr_2O_3 alongside with the progressive corrosion in air. This oxide layer is not observed in specimens after manufacturing (see BSE image of surface material in AR condition in Figure 2.13). However, the BSE image corresponding to specimen C2a specimen in Figure 2.13 shows a well-formed Cr_2O_3 layer after approximately 72h high temperature exposure under creep, indicating rapid precipitation of oxides. The BSE images corresponding to samples C2b and C2c showcase the progressive onset of localized cracks of this layer. These occur because of the internal stresses generated after the nucleation and coarsening of Al and Si oxides in the vicinity of the Cr_2O_3 oxide layer. These localized cracks turn into spots where oxygen diffusion is now prevented only by the FCC matrix diffusivity. Given the low diffusivity of oxygen in FCC, oxygen diffusion is a predominantly intergranular phenomenon. Eventually, the increased oxygen content in-matrix leads to the precipitation of Al, Si and Fe oxides.

No additional forms of corrosion (e.g., nitridation) were observed.

C3 The quantization of the geometrically necessary dislocations density (ρ_{GND}) was required for the validation of the mean-field creep model presented in Chapter 3. Measurements were conducted on a total of 3 samples: 2 in AR condition and a single coupon extracted from a creep test stopped after the $\dot{\epsilon}_{ss}$ was reached. The sample preparation procedure follows that of conventional SEM technique with an extra final step: to maximize the electrical conductivity between the material and the aluminum bakelite holder, all bakelite surfaces surrounding the sample were covered with an electrical conducting silver coating.

The ρ_{GND} was computed from the postprocessing of the local misorientation angles measured via EBSD. After constructing the corresponding kernel average misorientation (KAM) based on the Nye-tensor approximation methodology for local misorientation of neighboring points, the ρ_{GND} is estimated as $\rho_{GND} = \Theta_s \cdot b^{-1}$, where b is the Burgers' vector and Θ_s is the slope of the linear fitting of the accumulated frequency of angular local misorientations (Θ_{ave}) in radians per meter [20]. An illustration of this procedure is showcased in Figure 2.14.

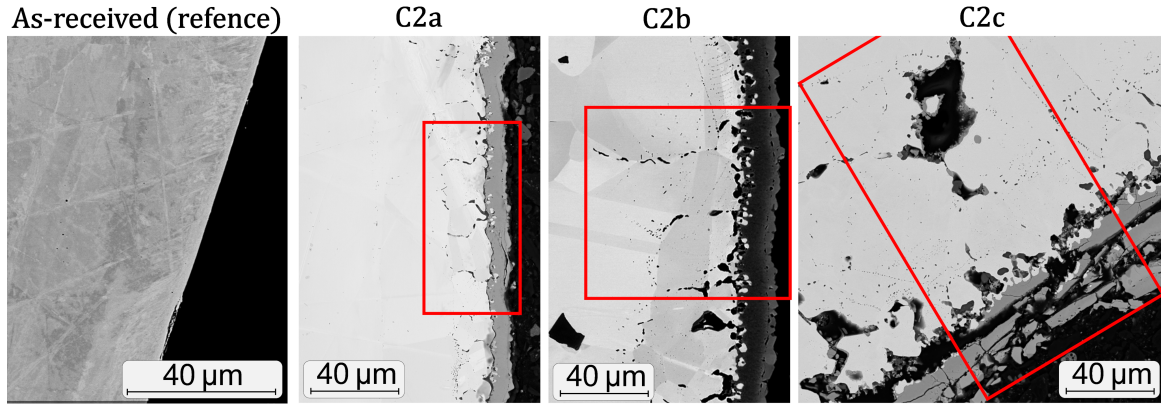


Figure 2.13: BSE micrographs showcasing the absence (AR sample, for reference), rapid formation (after ≈ 72 h in C2a), and progressive loss of the protective Cr_2O_3 oxide layer (throughout C2b and C2c) creep in air. The oxidation affected zone (OAZ) in samples C2a to C2c are highlighted in red squares. The inverted grayscale of the micrograph belonging to experiment C2b makes it difficult to highlight the OAZ.

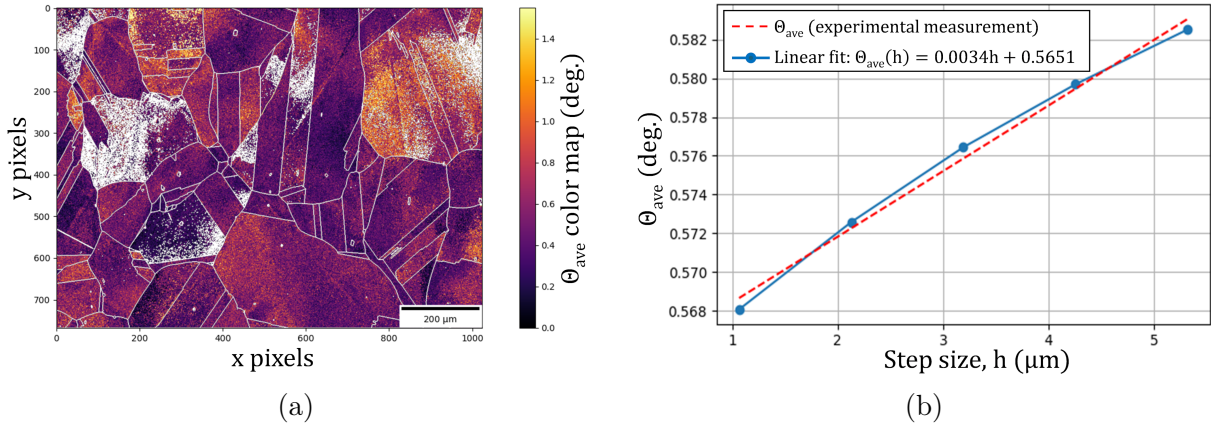


Figure 2.14: EBSD data postprocessing methodology to calculate the ρ_{GND} . (a) KAM calculated for a maximum neighboring misorientation of 5° , and (b) Θ_{ave} (deg.) vs. step size (m) showcasing a linear fit with a slope of $0.0034 \text{ deg m}^{-1}$. The data belongs to an AR specimen of 800H alloy.

The source of both AR (C3a & C3b) and the single creep-tested (C3c) sample are displayed on an illustrative normalized creep curve in Figure 2.15. The ρ_{GND} estimations for the three specimens alongside with the corresponding misorientation gradient, background noise and R^2 error are all provided.

Concluding remarks

- The core material of all heat-affected samples (either after furnace aging or creep) showed signs of **aging**. This is, intragranular sub-micron Cr_{23}C_6 carbides dissolve in favor of the nucleation and coarsening of those located at grain boundaries.
- Titanium carbo-nitrides ($\text{Ti}_x(\text{C},\text{N})$) appear unaffected by aging.
- Given that temperatures were below the recrystallization point ($\approx 1100^\circ\text{C}$, see for instance [12, 21, 22]), the grain structure (i.e., grain size, twinning, and grain aspect ratio) of the samples was not affected by aging.
- Oxidation was the only corrosion mechanism observed during the creep tests conducted

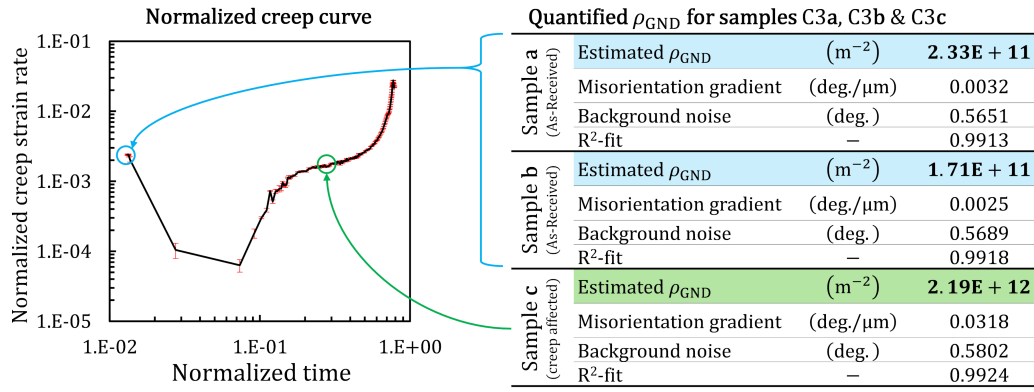


Figure 2.15: Calculated ρ_{GND} and associated misorientation gradient, background noise (intersection of linear fit) and R²-fit error for both AR (C3a & C3b) and creep-tested (C3c) samples analyzed in the context of microstructural characterization experiments C3. The source of each sample (AR or creep-tested) is illustrated onto a normalized creep strain rate vs. creep strain plot.

at ULiège. A protective chromia (Cr_2O_3) layer was observed to form at the surface of all heat-affected samples (furnace-aged and creep tested). This layer hinders the penetration of corrosive elements due to its low diffusivity (see Annex 2B), however does not prevent it. Localized chromia layer breakage and oxide penetration was observed in creep tests conducted in air after mere days of testing.

- **Nitridation was only observed in those high-temperature aged specimens under a realistic industrial environment combining high N_2 concentration and occasional water vapor.** The maximum penetration of AlN, considered the most stable nitridation-related precipitate, reaches higher penetration depths compared to those entitled to oxidation in every sample (N1 to N5). Chromium-based nitrides (CrN and Cr_2N) on the other hand, do not show the same consistency. These appear with higher N concentration in matrix. However they are more predominant in sample N1, and appear to progressively dissolve throughout the years. A more thermodynamical oriented study of this phenomenon is given in Annex 2B.

2.2.2 Creep behavior characterization

Creep characterization was exclusively conducted on specimens belonging to the second acquisition batch. These were of cylindrical geometries D06 and D10 (see Table 2.7), extracted from sheets 2S12 and 1S20 (see Table 2.10) respectively. All samples were manufactured in University of Liège under the supervision of Mr. Samuel Rondia, following strictly all the geometrical constraints and tolerances advised by Z/R. In particular, threading of specimens ($\text{M10} \times 1.5 - 6\text{g}$ and $\text{M16} \times 2 - 6\text{g}$ for samples D06 and D10, respectively) was carefully verified to ensure compatibility (see Annex 2C).

A total of 14 successful creep tests were conducted. To capture the non-classical two-step creep strain rate, high temperature and low stresses were prioritized. The exact test loadings and duration were defined following to maximize the efficiency of this campaign, seeking to acquire the maximum amount of information in the shortest amount of time.

Postprocessing of raw creep test data was performed on a short Matlab[®] script, which provides a smaller time - creep strain - creep strain rate dataset with the respective standard deviations. The postprocessed creep test results are provided from Figure 2.16 to Figure 2.22 for each temperature T_i with $i = 1, 2, \dots, 6$ ($T_1 < T_2 < \dots < T_6$). Creep strain vs. time and creep strain

rate vs. creep strain curves are normalized by different undisclosed factors due to confidentiality reasons.

T_1

A single test was conducted at temperature T_1 , using a specimen of geometry D10. It was the lowest creep temperature addressed during this experimental campaign. A relatively high stress was imposed in order to achieve rupture within a reasonable amount of time. Results, shown in Figure 2.16, correspond to a classical creep behavior where no solid-solution induced creep strain hardening is involved.

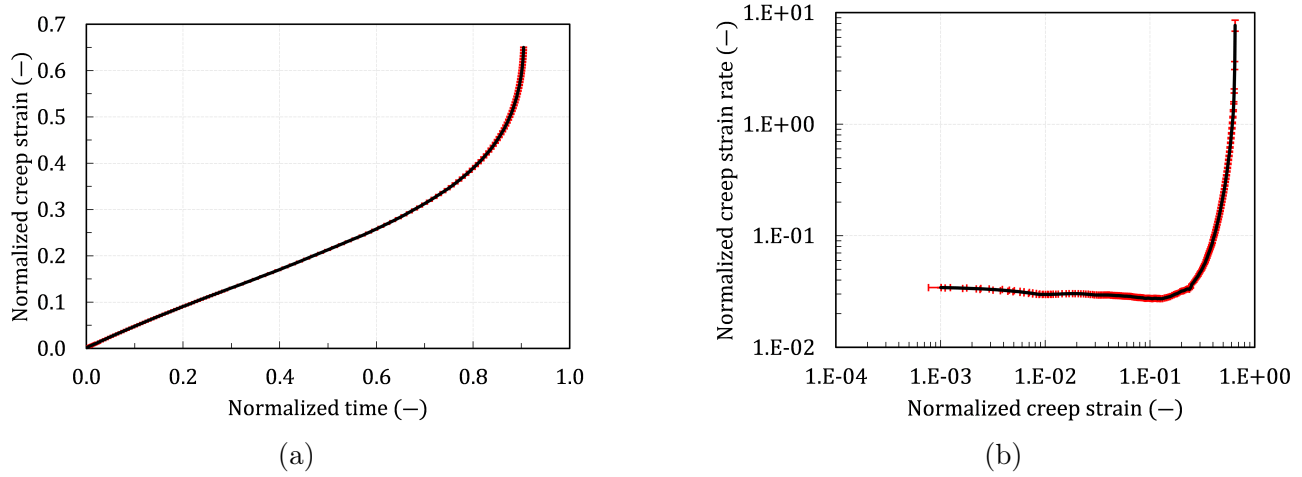


Figure 2.16: Normalized (a) creep strain rate vs. time, and (b) creep strain rate vs. creep strain experimental curves obtained for 800H alloy at T_1 .

T_2

A single test was conducted at temperature T_2 , using a specimen of geometry D10. This test was planned until rupture, however an unclear machine issue triggered an automatic test stop at around 0.8 normalized time in Figure 2.17a. After cooling down, the creep test was continued on the same specimen. This procedure allowed us to recover the entirety of the creep behavior at T_2 until rupture. Figure 2.17 is built by merging the results of these two separate tests into a single curve.

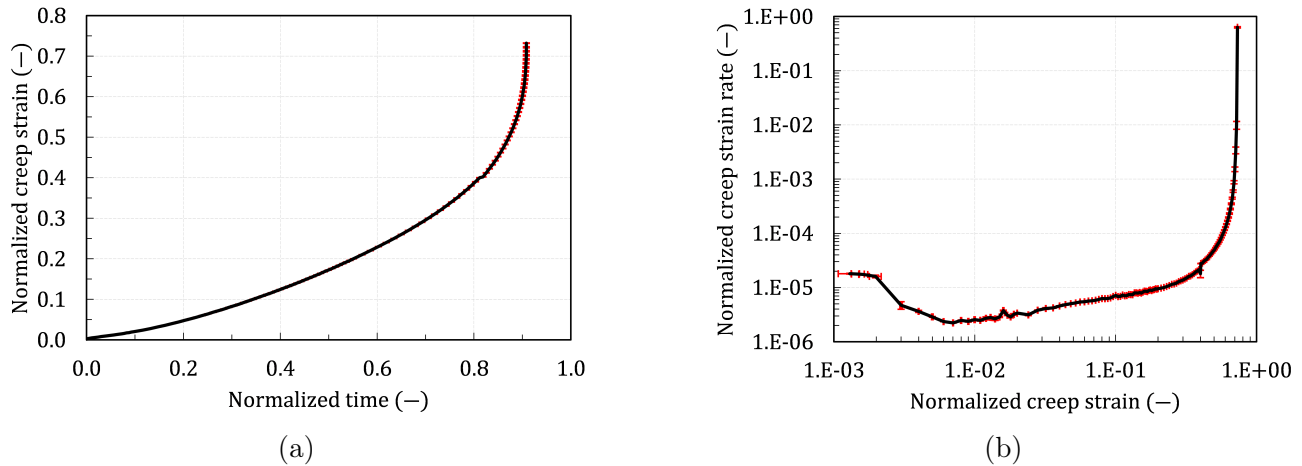


Figure 2.17: Normalized (a) creep strain rate vs. time, and (c) creep strain rate vs. creep strain experimental curves obtained for 800H alloy at T_2 .

T₃

Three creep tests were conducted at temperature T_3 , using specimens of geometry D06. Two of the tests, one at σ_A and one at σ_B , were conducted until rupture. A third test was conducted at σ_B until the steady-state creep rate ($\dot{\epsilon}_{ss}$) was reached in order to verify the results of the previous test. Figure 2.18 showcases results for all three tests. In particular, the third test (σ_B conducted until $\dot{\epsilon}_{ss}$) results are consistent with those for σ_B until rupture.

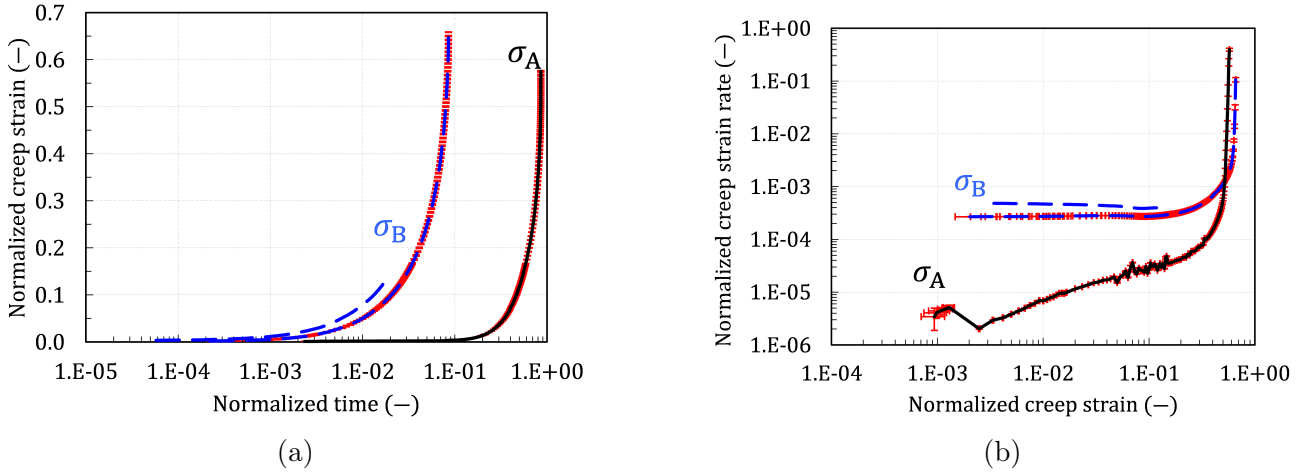


Figure 2.18: Normalized (a) creep strain rate vs. time, and (c) creep strain rate vs. creep strain experimental curves obtained for 800H alloy at T_3 at 2 stress levels: $\sigma_A < \sigma_B$.

T₄

A single creep test was conducted until rupture at temperature T_4 , using a D10 specimen. Results shown in Figure 2.19 are consistent with a non-classical creep behavior.

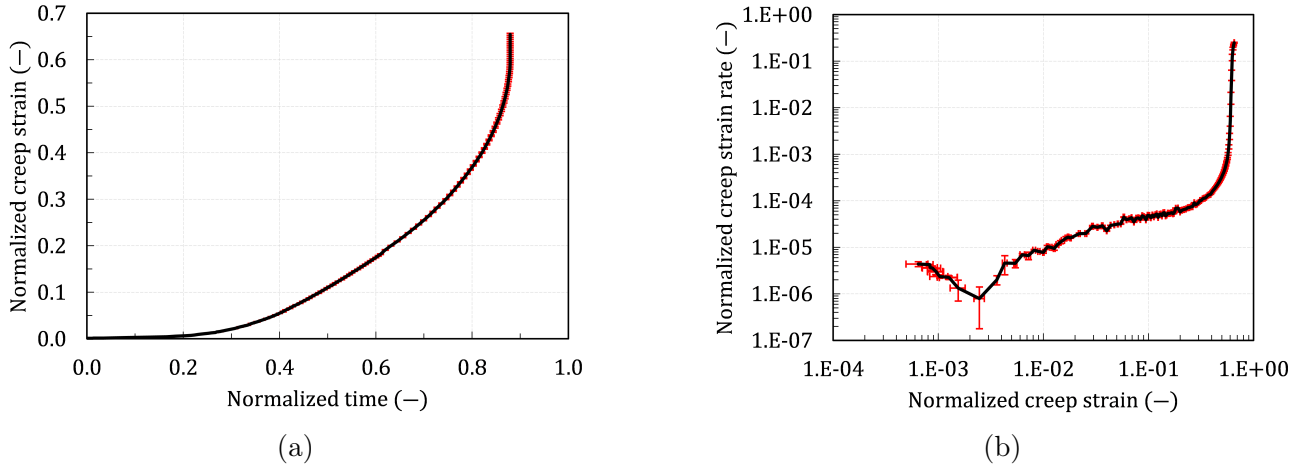


Figure 2.19: Normalized (a) creep strain rate vs. time, and (c) creep strain rate vs. creep strain experimental curves obtained for 800H alloy at T_4 .

T₅

A total of 5 creep tests were conducted at temperature T_5 . 4 creep tests were firstly conducted at 4 different stress levels 4, labeled σ_C , σ_D , σ_E and σ_F in Figure 2.20 ($\sigma_C < \sigma_D < \sigma_E < \sigma_F$). Note that creep test σ_D could not be conducted until rupture due to an automatic stop triggered by a thermocouple failure. All other tests showcased in Figure 2.20 were conducted until fracture of the specimen was achieved.

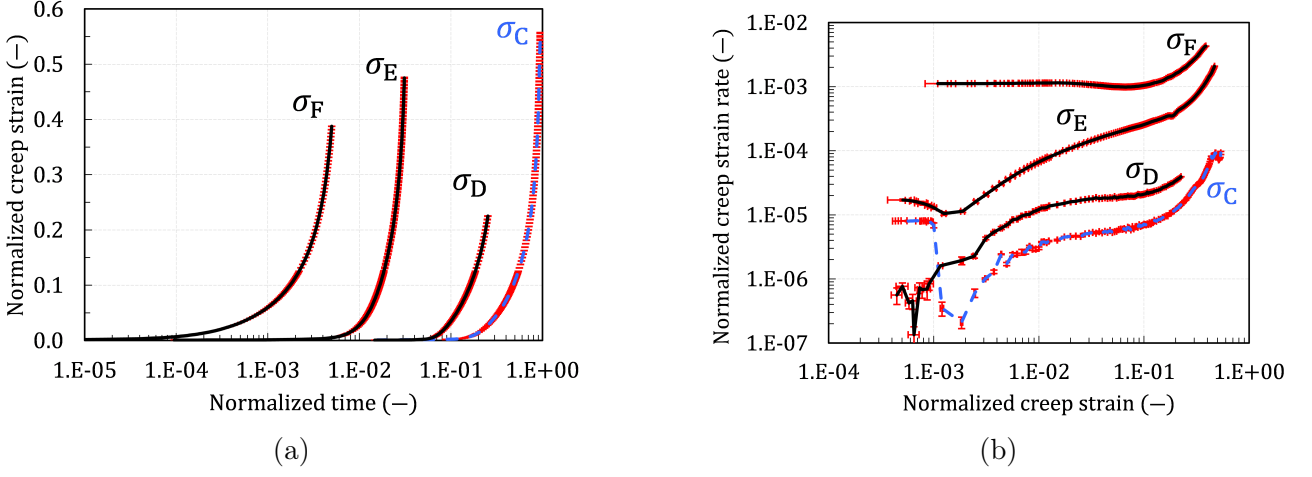


Figure 2.20: Normalized (a) creep strain rate vs. time, and (c) creep strain rate vs. creep strain experimental curves obtained for 800H alloy at T_5 and 4 different stress levels: $\sigma_C < \sigma_D < \sigma_E < \sigma_F$.

An additional test at T_5 and σ_C interrupted after reaching the $\dot{\epsilon}_{ss}$ was conducted to provide a creep-affected specimen for the quantification of ρ_{GND} . The experimental creep strain and creep strain rate curves obtained for this fifth test at T_5 are compared to the previous σ_C to rupture in Figure 2.21.

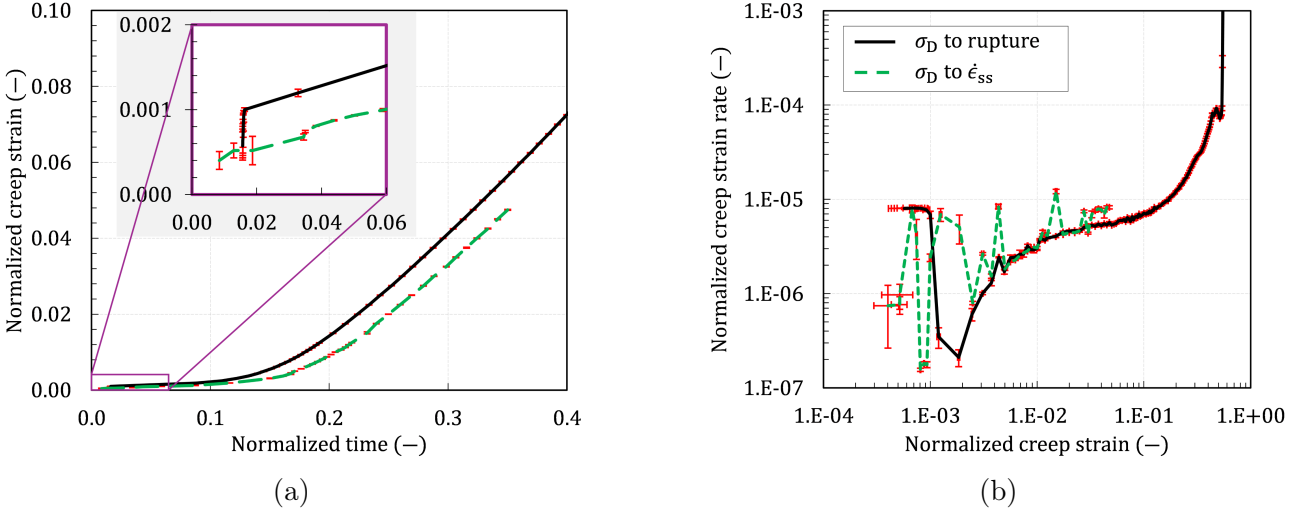


Figure 2.21: Normalized (a) creep strain rate vs. time, and (c) creep strain rate vs. creep strain experimental curves obtained for 800H alloy at T_5 and σ_C .

T_6

A single creep test was conducted at temperature T_6 using a specimen D10. It was the highest temperature addressed during this campaign. Results are shown in Figure 2.22.

The test was planned until rupture, however the specimen exhibited an unusually ductile behavior. During testing, it reached strains of almost double of that recorded at rupture for other temperatures. The large deformation triggered the mechanical safety switch of our creep machine, stopping the test before sample breakage. Observations of the necking zone via LOM, presented in Figure 2.12b), showcase extremely high damage accumulation consistent with creep damage.

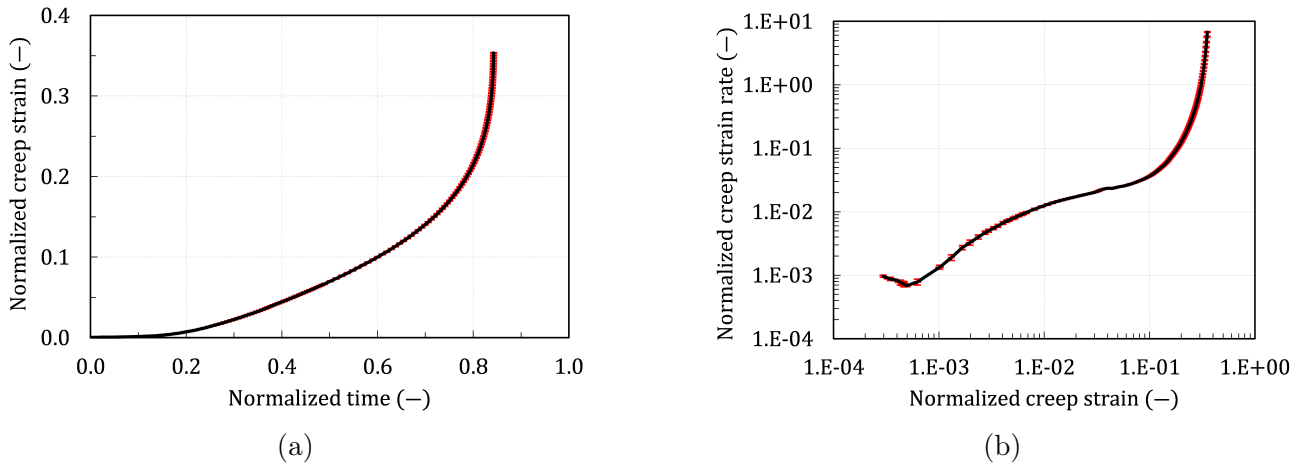


Figure 2.22: Normalized (a) creep strain rate vs. time and (b) creep strain rate vs. creep strain experimental curves obtained for 800H alloy at T_6 .

Concluding remarks

- The creep experimental campaign conducted in ULiège attained a comprehensive characterization of the creep deformation response of Incoloy 800H at high temperature and mid-low stress loadings. The geometrical constraints of the specimens allowed us to measure creep strain in a perfectly uniaxial deformation state, whereas the optimized creep testing methodology enabled us to detect the SSH stage. Exploitation of the reliable Incoloy 800H creep database obtained herein is envisioned to aid during the identification and validation of micro- and macromechanical creep models.
- The peculiar microstructural features entitled to the hot rolled sheet form of our source material, in addition to the perfectly uniaxial gauge zone in our specimens and the optimized creep experimental methodology, make our results not comparable to those found in literature.
- Microstructural characterization of creep affected specimens shows microstructural changes consistent with:
 - aging (nucleation and coarsening of $M_{23}C_6$ carbides at grain boundaries.
 - creep damage (intergranular void formation and crack propagation)
 - oxidation (formation of chromia layer and occasional penetration of spinel Al,Si,Mg oxides)

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Micromechanical creep model: MFC

Mean-Field Creep

Contents

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3.1.1	Mean Field Creep article:	116

The present chapter deals with the development, implementation and validation of a mean-field creep model adapted to predict the non-classical creep behavior of Incoloy 800H within high-temperature and mid-to-low stress loadings. Section 3.1 presents the peer-reviewed pre-print version of the scientific research paper **Mean-field prediction of transient solid solution hardening creep deformation response in Fe-Ni-Cr alloy** published in *Journal of Materials Science* (Springer), where a comprehensive summary of the mean-field model, its equations, development methodology and validation for Incoloy 800H as a case-study, are given. Details on parameter identification and validation for 800H alloy, as well as the analytical resolution of the partial derivatives of equation system of the model for a semi-implicit numerical implementation, are provided in Annex 3A. Its contents complement the information contained within the scientific publication of the model shared in section 3.1.1. Annex 3A is purposely not shared within the public version of this thesis due to confidentiality reasons.

3.1 Numerical model

Given the strong relationship between the high-temperature creep behavior of Incoloy 800H and its permanent and transient microstructural features (see section 1.2.4), creep modeling accuracy and predictability was deemed attainable only by means of a semi-physical creep model. In this context, the use of a mean-field approach (see section 1.1.3) was seen as an ideal choice. However, no creep-oriented mean-field model available in literature had yet combined all the main physical aspect entitled to the non-classical creep response of 800H alloy (see section 1.3). Thereby, the development of a new men-field set of equations addressing aspects such as precipitate-dislocation phenomena, SSH, and damage, was required. A graphical description of the creep micromechanics system envisioned for 800H alloy is provided in Figure 3.1.

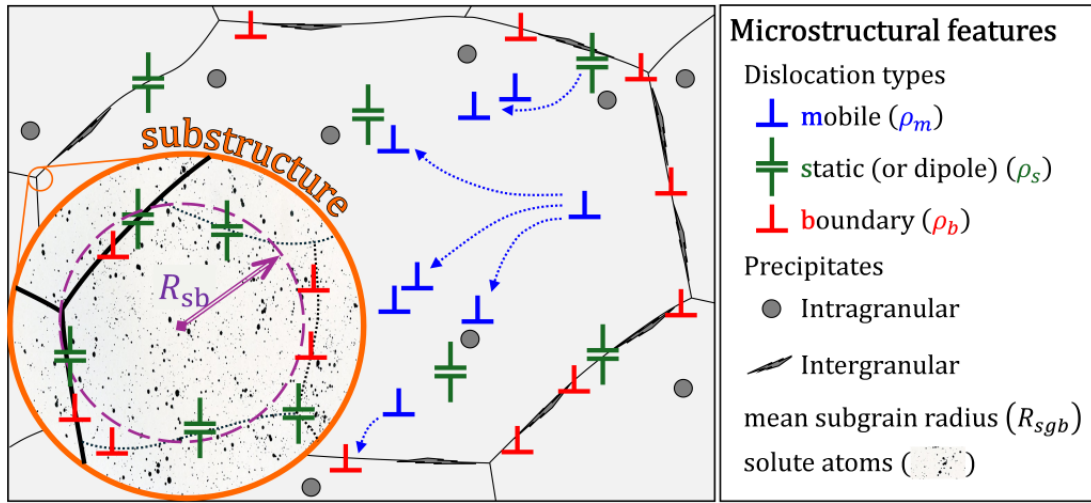


Figure 3.1: Sketch of Incoloy 800H microstructure and crystal defects interaction during creep. Adapted from [1].

The Mean Field Creep (MFC) model developed to recover the creep behavior of 800H alloy illustrated in Figure 3.1 is presented in the published article attached here after in section 3.1.1. It provides a comprehensive summary of the model. Following a comprehensive introduction, section 2 ("Mean-field creep model") addresses numerical aspects entitled to the MFC formulation. Then, an overview of the experimental characterization techniques applied on Incoloy 800H as a case study material to validate the MFC model is given in section 3 ("Material and methods"). It combines both creep and microstructural characterization techniques on AR and creep deformed material. Results entitled to the creep behavior characterization are given in normalized curves for confidentiality reasons. All physical and fitting parameters required for the model to recover the creep behavior of the alloy are described in Section 4 ("Input parameters"). In particular, physical parameters and initial state variables (namely dislocation densities) are given, whereas fitting parameters remain undisclosed. Numerical simulations of creep deformation and mobile dislocation density evolution are compared to their experimental values in Section 5 ("Results and Validation"). Finally, a brief discussion on the main novelties contained in the article is given in section 6 ("Discussion"), whereas section 7 deals with the main conclusions driven from this work.

3.1.1 Mean Field Creep article:

"Mean-field prediction of transient solid solution hardening creep deformation response in Fe-Ni-Cr alloy" Ulloa, C. E. R. *et al.* Mean-field prediction of transient solid

solution hardening creep deformation response in Fe-Ni-Cr alloy. en. *Journal of Materials Science*. ISSN: 0022-2461, 1573-4803 (June 2026)

Mean-field prediction of transient solid-solution hardening creep deformation response in Fe-Ni-Cr alloy

Carlos Rojas-Ulloa^{1*}, Fan Chen¹,
Jérôme Tchoufang Tchuindjang², Olivier Dedry²,
Tuan Nguyen-Minh³, Leo A.I. Kestens³, Anne Mertens²,
V́ctor Tuninetti⁴, Laurent Duchêne¹, Anne Marie Habraken^{1,5}

^{1*}Materials and Solid mechanics (MSM) team, Urban & Environmental Engineering (UEE) department, Université de Liège, 9 Allée de la Découverte, Liège, 4000, Wallonie, Belgium.

²Metallurgy and Materials Science (MMS) team, Aerospace & Mechanics (A&M) department, Université de Liège, 9 Allée de la Découverte, Liège, 4000, Wallonie, Belgium.

³Materials Science and Technology group, Department of Electromechanical Systems and Metal Engineering, Ghent University, Technologiepark 46 (Zwijnaarde), Gent, 9052, Flanders, Belgium.

⁴Department of Mechanical Engineering, Universidad de La Frontera, Av. Francisco Salazar 01145, Temuco, 4811230, La Araucanía, Chile.

⁵Fonds de la recherche scientifique - F.R.S., F.N.R.S, Rue d'Egmont 5, Brussels, 1000, Belgium.

*Corresponding author(s). E-mail(s): ce.rojasulloa@uliege.be;
Contributing authors: fchen@uliege.be; J.Tchuindjang@uliege.be;
Olivier.Dedry@uliege.be; t.nguyenminh@ugent.be; leo.kestens@ugent.be;
Anne.Mertens@uliege.be; victor.tuninetti@ufrontera.cl;
l.duchene@uliege.be; anne.habraken@uliege.be;

Abstract

High-temperature creep deformation is often accompanied by microstructure evolution. However, aspects affecting the creep properties of materials such as substructure formation and kinetics of primary and secondary precipitates are

not addressed by conventional models. In this work, a microstructure-dependent mean-field model is adapted to predict the non-classical creep deformation response of materials exhibiting solid-solution hardening regimes. Numerical novelties include an improved stress-sensitive apparent activation volume for the dislocation glide velocity, and a diffusion-based solid-solution stress formulation through the internal stresses of the material. An Incoloy 800H experimental campaign to identify and validate the model combining creep tests and microstructural characterization techniques was performed. Additionally, ex-situ thermokinetic simulations on Thermo-Calc[®] PRISMA module were conducted to recover the physical evolution of precipitates during creep. The numerical predictions are in good agreement with the experimental results for low-stress and high-temperature loadings. The loss of predictability observed at high-stress levels is attributed to a creep mechanism transition. Exact stress and temperature ranges cannot be openly disclosed for confidentiality reasons.

Keywords: High temperature creep, Solid-solution hardening, Particle strengthening, Cr₂₃C₆ thermokinetics, Incoloy 800H

1 Introduction

The continuous improvement in the efficiency and productivity of industrial processes in sectors such as power generation, metallurgy and aerospace [1] often relies on new technologies where higher operational temperatures are needed. In this context, resistance to the creeping viscous deformation observed in metallic components subjected to a combination of high-temperature and a constant mechanical load remains a major design aspect [2]. To satisfy these new requirements, researchers may explore the suitability of commercially available alloys [3, 4], develop new alloys [5, 6], or develop heat treatments aimed at enhancing the mechanical properties of commercially available alloys [7, 8]. In any case, accurate assessment of the creep performance during testing and service life is challenging. New creep testing methods such as indentation [9], miniaturized specimens [10, 11] or accelerated creep tests through the stepped iso-stress method [12] have been explored to reduce the costs of material characterization. Nevertheless, uniaxial tensile creep tests remain the most popular creep characterization technique. They consist of the application of a constant uniaxial load under high constant temperature (following either the ISO-204 [13] or ASTM E139 [14] standards). Conventional creep characterization campaigns covering wide stress and temperature ranges are, however, lengthy and costly. In this context, the development of reliable inter- and extrapolation methods that minimize experimental input to predict creep deformation and service life is a scientific challenge shared among industrial and academic researchers. Despite their popularity, conventional stress, temperature, and [time-to-rupture](#) (t_r) extrapolation methods such as the Larson-Miller parameter or the Manson-Haferd parameter assume a single creep mechanism and a constant microstructure per master curve. In general, simpler models fail to recover the creep behavior of materials that exhibit creep mechanism transition or microstructure evolution.

Under sufficiently high temperature and mid-to-low stress regimes, some austenitic steels exhibit complex creep behavior characterized by two consecutive local minima in the creep strain rate history. The second creep rate minimum corresponds to the conventional steady-state creep stage, whereas the first peak is often a consequence of complex microstructural transformations related to precipitate kinetics. For instance, [15] concluded that the non-classical regime observed in 2.25Cr-1Mo steel was the consequence of an Orowan-type particle-strengthening mechanism involving MX precipitates. More recently, [16] investigated the non-classical response of 18Cr-9Ni-3Cu-Nb-N at 873 K and 280 MPa by means of ex-situ microstructural characterization of material samples at different creep stages. The authors attributed the first local minimum to an intragranular NbX precipitate strengthening phenomenon, whereas the second minimum was ascribed to a grain boundary precipitate strengthening effect driven by the intergranular precipitation of Cr_{23}C_6 carbides associated with conventional aging.

Providing a balanced cost/weight ratio and good creep and corrosion resistance, alloys such as 800H, 800Rk and 800HT have experienced high popularity given their admissibility for the manufacturing of critical nuclear power plant components [17–19]. Investigations seeking to ascertain its applicability for temperatures roughly above 850°C were, however, limited by non-classical creep behavior. The first local creep strain rate minimum has been attributed to a [solid-solution hardening \(SSH\)](#) phenomenon linked to the incomplete precipitation of Cr_{23}C_6 carbides during [water quenching \(WQ\)](#) [20, 21], whereas the second minimum is consistent with a conventional steady-state creep rate; a delicate balance between dislocation production and annihilation. Incoloy 800H is hereafter chosen as the case study given its timeless industrial relevance and the modeling challenge it represents.

Accurate prediction of complex creep regimes involving microstructure evolution requires a numerical model in which the stress-strain dependency on dislocation and precipitate interactions is established. In this context, dislocation density-based [mean-field \(MF\)](#) models are a particularly prominent technique. They compute the microstructure dependent stress-strain mechanical response of materials by solving an equation system consisting of two main building blocks. The first is a mechanistic hardening model, such as those by E. Orowan [22] and the Bailey-Hirsh law [23], where the critical resolved shear strain and critical resolved shear stress dependency on the mean dislocation density (ρ) are established. The second block consists of the exploitation of ρ as a state variable. To express microstructure evolution within the equation system, variational ρ equation terms representing positive (e.g., dislocation production and multiplication) and negative (e.g., annihilation and immobilization) contributions are introduced. Altogether, this semi-physical framework provides ρ -based [MF](#) models with the ability to predict numerous microstructure-dependent inelastic phenomena. For instance, [24] achieved physical prediction of stress, strain and temperature assisted dynamic recrystallization by establishing a mean stored dislocation density threshold in addition to equations related to grain nucleation and growth. Such an innovative technique was used as a complement to cellular automaton simulations [25] or to predict the continuous inelastic deformation response and/or

flow stress of materials during hot forming processes [26, 27]. Multiscale approaches incorporating ρ formalisms into crystal plasticity models have also been explored [28].

In recent decades, creep-oriented ρ -based MF models have become increasingly popular among the scientific and industrial community because of the physically reliable creep deformation and service life predictions they provide. MF models developed during this period can be classified as static or continuum. Static creep models describe the stress and temperature dependency of a single feature (e.g., the **steady-state creep strain rate** ($\dot{\epsilon}_{ss}$)) for a steady microstructure [29], whereas continuum creep models describe the time-dependent creep deformation history for an evolving microstructure [30]. In view of the microstructural instabilities addressed in this work on the high-temperature creep of 800H alloy, the latter technique was chosen.

The creep model developed in this work follows the continuum MF modeling framework established in 1990 by N.M. Ghoniem et al. [31], where dependency to multiple mean dislocation density forests in combination with an evolving material substructure is introduced into a differential equation system consisting of 5 partial time-derivatives. Expressions describing ρ mobility and subgrain evolution are adopted from the analytical studies by H.P. Hirth and J. Lothe (lately revisited by P.M. Anderson in 2017 [32]), whereas terms representing dislocation production, annihilation and interaction with other microstructural features are largely inherited from the works by R. Sandström (a lifetime of creep modeling was recently summarized in [33]). The comprehensive MF model proposed by Ghoniem et al. [31] stands out as a versatile numerical framework capable of adapting to multiple materials, microstructures and problem types.

For instance, in 2012, M. Basirat et al. [34] achieved prediction of the full creep deformation response of 9Cr-1Mo steel by neglecting the subgrain evolution state variable, but introducing a climb-stress based SSH term, and a creep **continuum damage mechanics** (CDM) formulation addressing precipitate, cavitation, and lath depletion damage evolution. Later, S.D. Yadav et al. published on the application of creep MF models adapted to predict the creep behavior of a new 12% Cr [35] and a P92 [36] steel in 2016 and 2018 respectively. In addition to a CDM addressing precipitate coarsening and cavitation damage evolution, the authors compute the precipitate evolution through Mat-Calc [37] thermo-kinetic simulations. Noteworthy time-dependent ρ -based MF model applications to alloys such as new G115 steel [38], RAFM steel [39], austenitic stainless steel 304H Cu [30], IN718 superalloy [40], and steel 316LN [41] were published during the 2020s. In particular, the successful applications by F. Riedlsperger et al. to steel alloys P91 [42], P92 [43], and Ni-alloy 617 [44] have led the research team to consolidate a proper MF software called CreeSo [45]. A recent exploitation of the new CreeSo development environment by L. Witzmann et al. [4] showcases good numerical creep predictions for a 10% Cr steel in addition to remarkable mean dislocation density validation.

The present work deals with the development, implementation and validation of a creep-oriented ρ -based MF model belonging to the broad Ghoniem family introduced here above. It is adapted to account for the microstructure-dependent non-classical deformation response experimentally observed for 800H during low-stress high-temperature loadings [3, 46–48]. The creep MF model is presented in section 2. An experimental campaign aimed at studying the non-classical creep deformation

response and microstructure evolution of alloy 800H is described in section 3. A brief summary of the parameter identification procedure, as well as the physical parameters used for this alloy, is described in section 4. The numerical validation of the model and the numerical creep and damage prediction results are summarized in section 5. A brief discussion addressing the main contributions presented in this work is provided in section 6. Finally, section 7 contains the conclusions and prospects.

2 Mean-field creep model

The creep-oriented MF model implemented in this work establishes the creep strain rate ($\dot{\epsilon}$) dependency on 3 field state variables: the mean mobile (ρ_m), static (ρ_s), and boundary (ρ_b) dislocation density forests. The corresponding non-linear equation system is presented in Equation (1). The labels throughout Equation (1b) and Equation (1c) are assigned to facilitate the identification of dislocation interaction phenomena later on. In particular, "DWs" represents dislocation walls, whereas "SR" and "DR" represent static and dynamic recovery, respectively.

$$\dot{\epsilon} = \frac{b\rho_m v_{\text{eff}}}{m_T(1 - D_c)} \quad (1a)$$

$$\begin{aligned} \dot{\rho}_m = & \underbrace{v_g \rho_m (\rho_m^{1/2} + \rho_s^{1/2})}_{\text{Frank-Read source}} + \underbrace{v_g \frac{\beta \rho_s R_{\text{DW}}}{h_{\text{DW}}^2}}_{\text{I}} + \underbrace{v_g \frac{\rho_s}{2r_i}}_{\text{II}} - \underbrace{v_g \frac{\rho_m}{2R_{\text{DW}}}}_{\text{III}} - \underbrace{v_g \frac{\rho_m}{\lambda_{\text{eq}}}}_{\text{IV}} - \underbrace{v_g \frac{\rho_m}{h_{\text{DN}}}}_{\text{V}} \\ & - \underbrace{v_g \delta_{\text{anh}} (\rho_m^2 + \rho_m \rho_s)}_{\text{VI (DR)}} + \underbrace{v_c \frac{\rho_s}{\lambda_{\text{eq}}}}_{\text{VII}} + \underbrace{v_c \frac{\rho_s f_v b}{r_i}}_{\text{VIII}} - \underbrace{8v_c \rho_m^{3/2}}_{\text{IX (SR)}} \end{aligned} \quad (1b)$$

$$\begin{aligned} \dot{\rho}_s = & \underbrace{v_g \frac{\rho_m}{2R_{\text{DW}}}}_{\text{III}} + \underbrace{v_g \frac{\rho_m}{\lambda_{\text{eq}}}}_{\text{IV}} + \underbrace{v_g \frac{\rho_m}{h_{\text{DN}}}}_{\text{V}} - \underbrace{v_g \frac{\rho_s}{2r_i}}_{\text{II}} - \underbrace{v_g \delta_{\text{anh}} \rho_m \rho_s}_{\text{DR}} \\ & - \underbrace{v_c \frac{\rho_s}{\lambda_{\text{eq}}}}_{\text{VII}} - \underbrace{v_c \frac{\rho_s f_v b}{r_i}}_{\text{VIII}} - \underbrace{v_c \frac{\rho_s}{h_{\text{DW}}}}_{\text{SR at DWs}} \end{aligned} \quad (1c)$$

$$\dot{\rho}_b = \underbrace{v_c \left[8(1 - 2\xi) \frac{\rho_s}{h_{\text{DW}}} \right]}_{\star} \quad (1d)$$

Each dislocation density rate equation in Equation (1) incorporates mathematical terms representing physical effects of multiplication, annihilation and transformation of dislocation density forests embedded in an evolving microstructure. These are

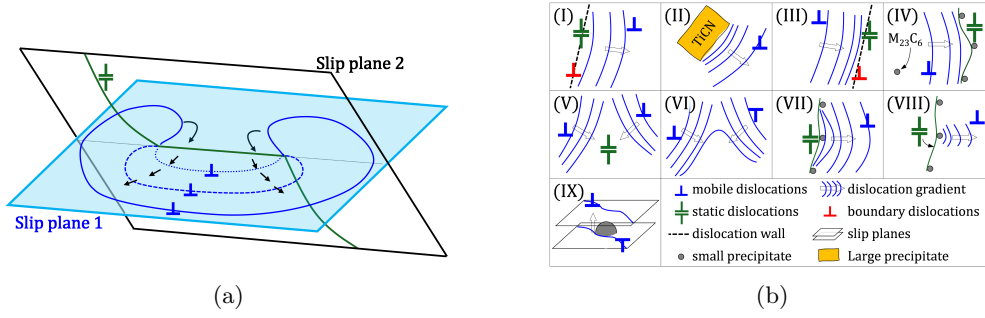


Fig. 1: Graphical representation of the multiple dislocation-precipitate interactions included in our MF model. (a) Mobile dislocation production following the Frank-Read source theory [49]. The mathematical expression in Equation (1b) incorporating dependency to ρ_m and ρ_s is recovered from [44]. (b) Illustrations of multiple creep-related microstructural phenomena according to the Roman numerical labeling system in Equation (1).

illustrated in Fig. 1b according to their roman numerical labels assigned throughout Equation (1b) and Equation (1c).

A brief description of the mathematical formula for each mean dislocation density type illustrated in Fig. 1b is offered below.

- I : emission of ρ_m at DWs. In the formula, R_{DW} is the mean radius of DWs [31]. The proportion of ρ_s dislocations participating in this depinning process is calculated as $\beta\rho_s$, where $\beta = 0.0375$ [31]. h_{DW} is the dislocation spacing ratio at the DW level and is calculated in Equation (2) [42].
- II : fraction of ρ_m produced at non-coherent precipitate-matrix boundaries that act as dislocation sources. r_i represents the mean radius of these precipitates. [44].
- III : fraction of ρ_m absorbed at DWs during creep deformation.[31].
- IV : fraction of ρ_m that become immobilized (ρ_s) upon finding a precipitate in their gliding path. λ_{eq} represents the mean particle spacing ratio of the precipitates participating in the process.[44].
- V : probability of ρ_m becoming immobilized upon encountering an equal-sign dislocation in their gliding path. The average mean dislocation spacing ratio (h_{DN}) is calculated by solving Equation (3).[44].
- VI : ρ_m annihilation probability following the encounter of two ρ_m of equal sign. δ_{anh} is the length parameter for spontaneous dislocation annihilation [31].
- VII : fraction of ρ_m produced from climbing [44].
- VIII : short ρ_m dislocation segments produced at small precipitates. In the formula, proposed by [44], f_v is the phase fraction of the precipitates participating in the process, and $\mathbf{b}$ is the Burgers' vector.
- IX : probability of dislocation annihilation during climbing [31].

★ : production of ρ_b from the static population. The subtracted quantity 2ξ ($\xi \ll 1$) represents the proportion of ρ_s annihilated in the process. After [31].

$$h_{\text{DW}} = [(\rho_s + \rho_b)R_{\text{DW}}]^{-1} \quad (2)$$

$$h_{\text{DN}} = 0.8 \cdot (\rho_m + \rho_s)^{-1/2} \quad (3)$$

Our MF creep model presented in Equation (1) is at its core a wisely chosen assembly of previous scientific works. Our contributions (namely the improvements enabling the prediction of the SSH regime, the enhanced stress sensitivity of 800H and the addition of a virtual inelastic work-based damage initiation criterion) are all introduced within Equation (1a) of our system: the Orowan equation. It describes the creep strain rate ($\dot{\epsilon}$) dependency on the field state variables, with m_T being the Taylor factor, D_c the equivalent creep damage, and v_{eff} the mean effective velocity of ρ_m . The latter is calculated as the geometrical summation of both glide (v_g) and climb (v_c) velocities [43], as shown in Equation (4).

$$v_{\text{eff}} = \left[\frac{1}{v_c} + \frac{\pi}{2} \sum_i (r_i^3 N_i) \frac{1}{v_g} \right]^{-1} \quad (4)$$

The equations describing v_g and v_c are shown in Equation (5) and Equation (6) respectively. They follow a thermodynamical interpretation that involves thermal and mechanical activation energy dependence [32]. In Equation (5), K_g is the pre-exponential factor [42], Q_g is the thermal activation energy for dislocation glide, k_B is the Boltzmann constant, T is the temperature in K, V_r is the apparent activation volume for dislocation glide, and σ_i and σ_a are the internal and applied (true) stresses, respectively. For Equation (6), Ω_{FCC} is the atomic size of the face centered cube (FCC) lattice per atom, and K_{cl} and K_{cp} are pre-exponential factors representing the lattice and pipe diffusion contributions respectively (see [42]).

$$v_g = 2K_g \exp\left(\frac{-Q_g}{k_B T}\right) \exp\left(\frac{-\sigma_i V_r}{k_B T}\right) \sinh\left(\frac{\sigma_a V_r}{k_B T}\right) \quad (5)$$

$$v_c = [K_{\text{cl}} + K_{\text{cp}}] \exp\left(\frac{-\sigma_i \Omega_{\text{FCC}}}{k_B T}\right) \sinh\left(\frac{\sigma_a \Omega_{\text{FCC}}}{k_B T}\right) \quad (6)$$

σ_a is calculated for a perfectly uniaxial deformation state and volume conservation as Equation (7a), where σ_0 is the initial (engineering) stress, ν is the temperature-dependent Poisson ratio and ϵ is the current plastic (creep) strain calculated in the model. σ_i refers to the internal mechanical stresses preventing free dislocation movement, a mechanical threshold that reduces the material ductility. In this work, Equation (7b) is calculated as the sum of the dislocation pile-up stress (σ_T) and SSH (σ_{ss}) contributions. σ_T is computed by solving the Bailey-Hirsch law [23] (more commonly referred to as the "Taylor stress") Equation (7c), where α is a material constant,

G is the shear modulus, and dependency on the square root of ρ_m is established for FCC alloys in [50].

$$\sigma_a = \sigma_0 (1 + \nu\epsilon)^{-2} \quad (7a)$$

$$\sigma_i = \sigma_T + \sigma_{ss} \quad (7b)$$

$$\sigma_T = \alpha m_T G b \sqrt{\rho_m} \quad (7c)$$

2.1 Solid-solution hardening

In this work, the SSH contribution to the inelastic deformation response is computed as a stress effect. σ_{ss} is calculated as the product of the strengthening function $f_{ss} = f_{ss0} \exp(\dots)$ and the solute atom concentration $\mathcal{X}_i^{2/3}$, where i represents the atom type found in solution, and the exponent 2/3 is optimized for FCC materials [51]. The Arrhenius expression governing f_{ss} in Equation (8) follows the interpretations of the kink-pair formation (KPF) model [52], inasmuch as it reduces the σ_{ss} calculation of the breakaway probability for a single dislocation segment. The form of f_{ss} in Equation (8) follows the proposal by [53, 54]. With only minor modifications, their model has seen successful applications for Inconel 718 in annealed [55] and "without-precipitate" configurations [27]. In Equation (8), C_1 is a fitting parameter, ΔE_b is the energy barrier for dislocation breakaway, σ_y is the temperature-dependent yield stress, and m is the hardening exponent. Our set of equations, guided by experimental observations, is described below.

$$\sigma_{ss} = f_{ss} \mathcal{X}_i^{\frac{2}{3}} = f_{ss0} \exp \left[\frac{-1}{C_1} \frac{k_B T}{\Delta E_b} \ln \left| \left(\frac{\sigma_a}{\sigma_y} \right)^m \right| \right] \mathcal{X}_i^{\frac{2}{3}} \quad (8)$$

The pre-exponential factor f_{ss0} is calculated in Equation (9), where ϵ_{eq} is the equivalent misfit parameter.

$$f_{ss0} = \frac{1}{\sqrt{2}} M_T G (\epsilon_{eq})^{\frac{4}{3}} \quad (9)$$

ϵ_{eq} is calculated in Equation (10) as the geometrical equivalent of the contributions of both the shear-modulus misfit η_i and the solute atom size misfit δ_i [56], where a is the solvent unit cell parameter; an experimental constant [57].

$$\epsilon_{eq} = \left[\left(\frac{\eta_i}{1 + 0.5|\eta_i|} \right)^2 + a^2 \delta_i^2 \right]^{1/2} \quad (10)$$

In Equation (8), ΔE_b represents the threshold energy barrier necessary for dislocations to surmount solute atoms. Equation (11a) is an extension of the one used in [55] and [27], where w/b is the mean atom travel distance ratio. The additional $\phi(T)$ function in Equation (11b) is introduced to recover the temperature dependency observed during model optimization for alloy 800H, where R is the gas constant and φ_1 and φ_2 are fitting parameters.

$$\Delta E_b(T) = \phi(T) M_T G b^3 \left(\frac{w}{b} \right)^{\frac{3}{2}} \sqrt{\frac{\sigma_a}{M_T G}} \quad (11a)$$

$$\phi(T) = \varphi_1 \exp \left(\frac{-\varphi_2}{RT} \right) \quad (11b)$$

The dissolution of submicron-sized Cr_{23}C_6 precipitates that nucleate at dislocations during **WQ** (see for instance [46, 58]) leads to the production of solid-solution Cr and C. A quick calculation involving the proportion and size differences between the two elements (see Supplementary material) suggests that Cr atoms are responsible for more than 98% of the total volume fraction of the particles produced from the dissolution of these carbides. Therefore, further σ_{ss} calculations involve exclusively Cr atoms. Inspired by the analytical works of [59], the form of the stress-assisted diffusion model of Cr atoms $\mathcal{X}_{\text{Cr}}$ is presented in Equation (12), where d_{AGS} is the average grain size (diameter), D_{eff} is the effective diffusion coefficient for Cr diffusion during **SSH**, t is the time, b_5 is a fitting parameter, and $\mathcal{X}_{\text{Cr},0}$ is the maximum Cr solute atom concentration participating in the **SSH** process. The latter is a temperature-dependent physical quantity recovered from ex-situ thermokinetic simulations (see section 4.3).

$$\mathcal{X}_{\text{Cr}} = \frac{\mathcal{X}_{\text{Cr},0}}{2} \left[1 + \operatorname{erf} \left(\frac{d_{\text{AGS}}}{2\sqrt{D_{\text{eff}} t}} + b_5 \right) \right] \quad (12)$$

The temperature and stress dependency of D_{eff} is established in Equation (13a) by numerical optimization. D_{ss}^0 is a pre-exponential factor, Q_{eff} is the activation energy, and b_4 is a material constant. The temperature dependency of Q_{eff} is described by the Arrhenius exponential function shown in Equation (13b), where b_1 , b_2 and b_3 are material parameters.

$$D_{\text{eff}}(T) = D_{\text{ss}}^0 \exp \left(\frac{-Q_{\text{eff},i}}{RT} \right) \exp \left(-b_4 \frac{\sigma_y}{\sigma_a} \right) \quad (13a)$$

$$Q_{\text{eff},i} = b_1 \left[1 + b_2 \exp(b_3/T) \right] \quad (13b)$$

2.2 Apparent representative volume

The stress-sensitivity of the model is largely determined by the apparent representative volume (V_r). Although similar creep-oriented **MF** models treat V_r as a fitting parameter varying within a reasonable physical range [44], a single V_r proved unsuitable for recovering the stress-sensitivity of 800H during creep. The independent magnitudes of V_r optimized to recover the $\dot{\epsilon}_{ss}$ across our experimental creep curves via inverse modeling depict an inversely proportional relationship to σ_a . The V_r dependency on σ_a was thereby recovered by the Arrhenius formula presented in Equation (14).

$$V_r(\sigma_a) = A_V \left[1 + B_V \exp(C_V \cdot \sigma_a) \right] \cdot b^3 \quad (14)$$

2.3 Creep damage

The addition of a **CDM** enables the prediction of the progressive loss of load carrying capacity and creep rupture time of the material [34, 35]. The creep damage denominator $(1 - D_c)$ in Equation (1a) is calculated by solving Equation (15), where D_i is the damage contribution of the i^{th} damage mechanism acting in our creep case study.

$$(1 - D_c) = \prod_i (1 - D_i) \quad (15)$$

The **MF** creep model presented in this work includes only two damage mechanisms: cavitation damage (D_{cav}) and precipitate coarsening damage (D_{ppt}). D_{cav} is defined by Equation (16). The temperature-dependent constant $A(T)$ in Equation (16a) is expressed as an Arrhenius-type function in Equation (16b), where A_1 , A_2 and A_3 are fitting parameters.

$$\dot{D}_{cav} = A(T) \dot{\epsilon} \quad (16a)$$

$$A(T) = A_1 \left[1 + A_2 \exp(T/A_3) \right] \quad (16b)$$

Precipitate damage (D_{ppt}) in 800H is associated with the massive precipitation and coarsening of $Cr_{23}C_6$ at non-coherent grain boundaries [58, 60, 61]. Equation (17a) recovers the D_{ppt} attributed to the j^{th} precipitate type following the particle-spacing ratio formulation by C.Ó. Murchú et al. [62], where $\lambda_{j,0}$ and λ_j are the initial and current average spacing ratios of the precipitate j respectively. The spacing ratio of a precipitate j is calculated by solving Equation (17b) and Equation (17c) as proposed by B. Sonderegger & E. Kozeschnik in [63]. In the case of multiple precipitates, the total D_{ppt} is calculated as in Equation (15).

$$D_{ppt,j} = 1 - \frac{\lambda_{j,0}}{\lambda_j} \quad (17a)$$

$$\lambda_j = \left[\frac{\ln(3)}{2\pi \sum_j (N_j \cdot r_j)} + (2r_A)^2 \right]^{1/2} - 2r_A \quad (17b)$$

$$r_A = \sqrt{\frac{2}{3}} \cdot \frac{\sum_j (N_j \cdot r_j^2)}{\sum_j (N_j \cdot r_j)} \quad (17c)$$

Additionally, our model incorporates a simplified (uniaxial) damage initiation criterion [64]. The accumulated plastic work formula is computed by Equation (18), where m_w is a fitting parameter. Damage is initiated once a user-defined critical threshold of virtual plastic work w_{crit} is reached.

$$w_s(\sigma, \epsilon) = \int_0^t \left(w_{iso}(\sigma, \epsilon) + w_{kin}(\sigma, \epsilon, \dot{\epsilon}) \right) dt \approx \sigma_a \epsilon^{1/m_w} \quad (18)$$

Table 1: Standard Fe-balanced stoichiometric composition of alloy 1.4959 (wt.%), after [65].

*: $0.46 \leq (\text{Al} + \text{Ti}) \leq 1.07$ wt.%

**: $30.09 \leq (\text{Ni} + \text{Co}) \leq 34.78$ wt.%

constituents	Ni **	Cr	C	Si	Mn	S	Ti *	Al *	Co **	Cu
min	29.4	19.0	0.05	0.32	0.50	0.001	0.21	0.19	0.028	0.006
max	34.45	21.5	0.10	0.74	1.34	0.017	0.59	0.58	0.76	0.070

3 Experimental campaign

Incoloy 800H material was acquired in as-received condition in the form of 20 mm thick sheets. A heat treatment **SA+WQ** consisting of **solution annealing (SA)** for 30 min at 1150°C followed by **WQ** preceded the hot rolling process. The standard chemical composition of the material is given in Table 1.

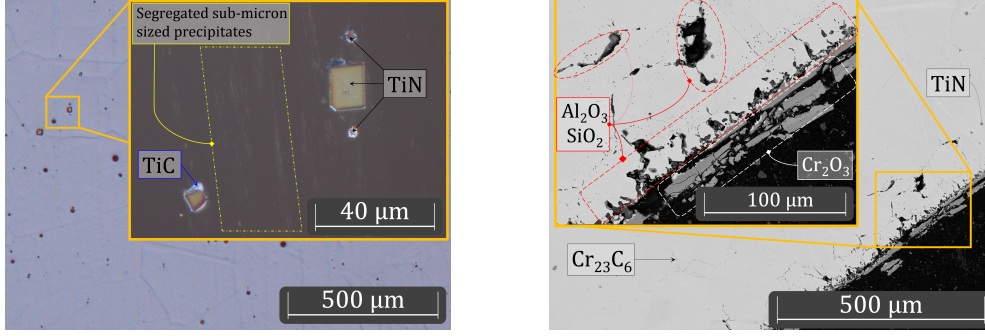
Material samples were extracted along the rolling direction of the sheets using a Charmilles Robofil **electric discharge machine (EDM)** model 310. These were destined to an experimental campaign combining mechanical creep tests and microstructural characterization techniques. A summary of the experimental results relevant for the application and validation of the **MF** creep model is given below.

3.1 Microstructure observations

The material microstructure is studied via an Olympus BX60 **Light Optical Microscopy (LOM)** coupled with an OLYMPUS UC30 CCD digital camera and equipped with a motorized stage together with the OLYMPUS STREAM Motion® software. The material samples were first ground and polished down to 1 µm prior to super finishing with **Oxide polishing suspension (OPS)** down to 0.3 µm on a Struers Tegramin 30 automatic device. Complementary **LOM** techniques include chemical etching with glyceresia (2 parts glycerol + 4 parts hydrochloric acid (HCl) + 1 part nitric acid (**HNO₃**)) to highlight carbide-decorated **grain boundaries (GBs)**, and light polarization to highlight precipitates. **Scanning electron microscopy (SEM)** analyses were carried out using a Tescan Clara SEM associated with **Back-Scattered electron (BSE)** for the images and a 129 eV BRUKER **energy dispersive x-ray spectrometer (EDS)** with a silicon drift detector. The system was connected to ESPRIT® software for pre- and post-treatment of the data. The accelerating voltage for the SEM analyses was set at 15 kV, with a minimum scanning step size of the electron beam of 1 µm.

As-Received (AR) material:

The **AR** microstructure shows coarse austenitic grains with abundant annealing twins and small micron and submicron-sized precipitates scattered throughout the lattice. Low-density titanium carbo-nitrides (**Ti_x(C, N)**) appearing dark in **BSE** images [48] are differentiated into **TiN** and **TiC** via in-situ polarized **LOM** analyses, as shown in Fig. 2a. Owing to the resolution limitations of our **SEM** machine, submicron-sized precipitates (**TiN** and **M₂₃C₆**, observed via **transmission electron microscopy (TEM)** by [58, 66]) could not be identified. Nevertheless, their presence can be correlated to



(a) Qualitative identification of precipitates in AR material via in-situ polarized LOM.

(b) BSE image showing oxide penetration in 800H after 2 months of high-temperature creep testing in air.

Fig. 2: Microstructural characterization (a) AR and (b) creep-affected microstructures of Incoloy 800H.

the bright reflection of polarized light at segregation lines (i.e., the agglomeration of submicron-sized precipitates formed during the hot-rolling process of the material) observed during LOM, as shown in Fig. 2a.

Finally, digital post-treatment of LOM images was performed using the open-source software ImageJ [67]. The grain aspect ratio was measured at 1.3 with no particular geometrical orientation. An austenite grain size of $135.7 \pm 30 \mu\text{m}$ was calculated by applying the linear intersection method according to the standard ASME E112-13 [68].

Creep-affected material:

Material samples from specimens affected by up to 2 months under high-temperature creep in air were subjected to similar microstructural analyses. A progressively greater number of Cr-rich M_{23}C_6 precipitates nucleating at GBs were observed in the samples from longer creep tests, which is consistent with aging. No grain coarsening was detected. Fig. 2b showcases the loss of the characteristic Cr_2O_3 protective layer [1, 69] as well as oxide penetration in the form of nucleation and coarsening of Si and Al oxides. These observations are consistent with those presented in [48]. The precipitation of oxides during creep is attributed to high-temperature exposure to air under sea-level atmospheric pressure conditions. In contrast with the observations presented in [48, 58], the samples analyzed herein showed no evidence of nitridation. The absence of nitrides is explained mainly by the short duration of the high-temperature creep tests in air.

3.1.1 Creep behavior

A total of 11 mechanical creep tests were conducted on a Zwick/Roell (Z/R) Kappa 100 DS machine equipped with a high-inertia resistance furnace MP00850. Creep strain was measured at the uniaxial gauge length of the specimen by means of a high-precision

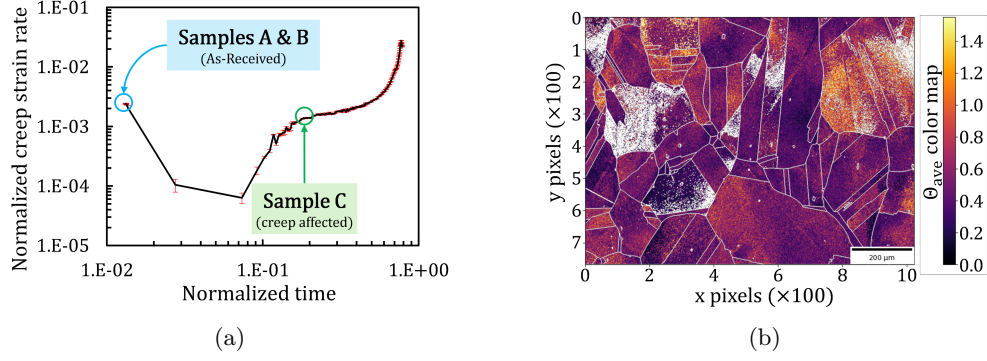


Fig. 3: (a) Normalized creep strain rate vs. time curve showing the source of samples A, B and C. Sample C is not extracted from a creep test interrupted after the $\dot{\epsilon}_{ss}$ was reached. (b) KAM map calculated from EBSD measurements for sample A (AR condition).

ceramic extensometer. To enable the SSH-driven minimum creep strain rate regime to take place, standard creep test (SCT)s experiments were primarily conducted under a combination of low-stress and high temperature loadings. In addition, the loading application methodology instructed in ISO-204 [13] was modified by decreasing the pre-load temperature holding time from 60 to 15 minutes [60]. Although specific stress-temperature regimes cannot be openly disclosed, they range between 10–80 MPa and 700–1050°C.

3.1.2 Dislocation density estimation

Experimental characterization of the geometrically necessary dislocation density (ρ_{GND}) was conducted on material samples under both AR (samples A & B) and creep-affected (sample C) conditions. In particular, sample C was obtained from a single creep test interrupted after reaching the $\dot{\epsilon}_{ss}$ (Fig. 3a). To calculate the ρ_{GND} , a kernel average misorientation (KAM) map was first constructed on the basis of the Nye-tensor approximation methodology for local misorientation of neighboring points detected by Electron backscattered diffraction (EBSD) (Fig. 3b). The ρ_{GND} is then estimated from the slope of the linear fit of the accumulated frequency of angular local misorientation (Θ_{ave}) as a function of the step size (length scale defined by the beam size definition and grid during EBSD) [70]. The ρ_{GND} values estimated for the three samples along with the corresponding misorientation gradient, background noise and R^2 error are given in Table 2. The grain map, KAM map and Θ_{ave} identification for specimens A, B and C are provided in the supplementary material file.

Table 2: Summary of results of the EBSD-based ρ_{GND} measurements (Fig. 3) for samples A, B (AR condition) and C (creep test interrupted at $\dot{\epsilon}_{ss}$).

	sample A	sample B	sample C
Estimated ρ_{GND} (m^{-2})	2.33×10^{11}	1.71×10^{11}	2.19×10^{12}
$\Theta_{\text{ave}} \times 10^{-6}$ ($\text{deg.} \cdot \text{m}^{-1}$)	0.0032	0.0025	0.0318
Background noise (deg.)	0.5651	0.5689	0.5802
R ² -error	0.9913	0.9918	0.9924

4 Input parameters

4.1 Initial state variables

The boundary conditions necessary to solve the non-linear equation system of our MF model are hereafter defined as the vector $W_0 = [\epsilon_0 \quad \rho_{m,0} \quad \rho_{s,0} \quad \rho_{b,0}]$, where ϵ_0 is the initial creep strain ($\epsilon_0 = 0.0$) and $\rho_{m,0}$, $\rho_{s,0}$ and $\rho_{b,0}$ are the initial mobile, static and boundary dislocation densities, respectively. $\rho_{s,0}$ is approximated as the 10% of $\rho_{m,0}$, as seen in [44], whereas $\rho_{b,0}$ is calculated in Equation (19) as a function of the R_{DW} and the mean DW misorientation angle [71].

$$\rho_{b,0} = \frac{3\Theta_{\text{DW}}}{2bR_{\text{DW}}} \quad (19)$$

Finally, a constant magnitude of $R_{\text{DW}}=250$ nm is used in this work. This value is estimated from the numerous TEM experimental observations of the AR hot-rolled microstructure of 800H alloy [72–74].

4.2 Physical and fitting parameters

4.2.1 Diffusion coefficients

Diffusion in solids is modeled as a thermally-activated process by means of the Arrhenius equation shown in Equation (20a), where D_i^0 and Q_i are the pre-exponential factor and activation energy for diffusion in a medium i , where $i = \text{l}$ (lattice), GB, or p (pipe). Following a similar approach to that proposed by [75], the parameters D_i^0 and Q_i to calculate D_{l} and D_{GB} are computed on the basis of the weighted average of those measured for Fe, Ni and Cr in an 800-type alloy. The weighted average function is shown in Equation (20b), where x is the property under evaluation and w_i are the relative molar mass of the alloy per element ($w_{\text{Fe}} = 0.335$, $w_{\text{Ni}} = 0.353$ and $w_{\text{Cr}} = 0.312$). A summary of the parameters used to calculate D_{l} and D_{GB} following this approach is given in Table 3.

$$D_i(T) = D_i^0 \exp\left(\frac{-Q_i}{RT}\right) \quad (20a)$$

$$x_{\text{eq},800\text{H}} = x_{\text{Fe}} \cdot w_{\text{Fe}} + x_{\text{Ni}} \cdot w_{\text{Ni}} + x_{\text{Cr}} \cdot w_{\text{Cr}} \quad (20b)$$

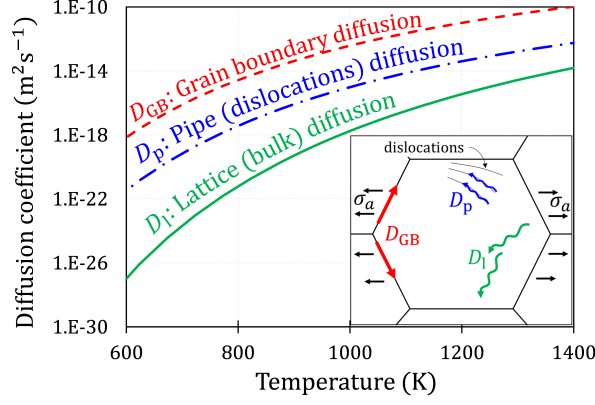


Fig. 4: Temperature dependency of D_{GB} , D_p and D_l used in this work, calculated after the methods of [75, 77–79]. The arrows inside the plot illustrate the vacancy diffusion under load (σ_a).

Table 3: Pre-exponential and activation energy parameters calculating D_l and D_{GB} for 800H alloy.

Parameter	Magnitude per γ lattice constituent			weighted average
	FCC-Fe (after [78])	FCC-Ni (after [77])	FCC-Cr (after [75])	
D_l^0 (m^2s^{-1})	3.260×10^{-5}	8.620×10^{-5}	3.240×10^{-4}	1.424×10^{-4}
Q_l (Jmol^{-1})	2.596×10^5	2.559×10^5	2.874×10^5	2.670×10^5
D_{GB}^0 (m^2s^{-1})	1.880×10^{-5}	3.820×10^{-5}	5.800×10^{-5}	3.788×10^{-5}
Q_{GB} (Jmol^{-1})	1.607×10^5	1.564×10^5	1.842×10^5	1.665×10^5

The pre-exponential and activation energy parameters to calculate D_p are extracted from [76]: $D_p^0 = 4.50 \times 10^{-5} \text{ m}^2\text{s}^{-1}$ and $Q_p = 1.85 \times 10^5 \text{ Jmol}^{-1}$). The temperature dependency of the three diffusion coefficients is depicted in Fig. 4.

4.2.2 Material elastoplastic constants

Elastic constants:

The shear modulus G and Poisson ratio ν of the material were introduced into the MF model as temperature-dependent polynomial functions $f(T)$ of the form shown in Equation (21). The equation and its fitting parameters (A, B, C and D; given in Table 4) are part of the quasi-static hot tensile material characterization conducted for Incoloy 800H by [80].

$$f(T) = A \cdot [1 + B \cdot (T - T_0) + C \cdot (T - T_0)^2 + D \cdot (T - T_0)^3] \quad (21)$$

Table 4: Fitting parameters for Equation (21) describing the temperature dependent shear modulus (G) and Poisson ratio (ν) for Incoloy 800H. Material characterization and parameter identification were performed by [80]. T is in $^{\circ}\text{C}$, and $T_0=20^{\circ}\text{C}$.

Property	A	B ($^{\circ}\text{C}^{-1}$)	C ($^{\circ}\text{C}^{-2}$)	D ($^{\circ}\text{C}^{-1}$)
G	76.74 ± 0.16 (GPa)	-2.50×10^{-4}	-4.32×10^{-7}	3.40×10^{-10}
ν	0.297 ± 0.0022 (-)	-6.05×10^{-4}	2.66×10^{-6}	-2.11×10^{-9}

Table 5: Optimized parameters for the polynomial function Equation (22) describing the temperature-dependency of the 0.2% offset σ_y for Incoloy 800H. Experimental characterization after [81, 82]

α_0 (MPa)	α_1 (MPa $^{\circ}\text{C}^{-1}$)	α_2 (MPa $^{\circ}\text{C}^{-2}$)	α_3 (MPa $^{\circ}\text{C}^{-3}$)	α_4 (MPa $^{\circ}\text{C}^{-4}$)
2.43×10^2	-3.14×10^{-1}	3.88×10^{-4}	5.69×10^{-12}	-2.60×10^{-10}

Yield stress:

Considering the temperature-dependent 0.2% offset yield stress characterization of Incoloy 800H at various temperatures conducted independently by [81, 82], σ_y is defined as a 4th order temperature-dependent polynomial function. It is presented in Equation (22), where T is the temperature in $^{\circ}\text{C}$, $T_0=20^{\circ}\text{C}$, and α_i are fitting parameters. The latter are identified via simple [root-mean squared percentile error \(RMSPE\)](#) minimization and are given in Table 5.

$$\sigma_y(T) = \sum_{i=0}^4 \alpha_i \cdot (T - T_0)^i \quad (22)$$

4.2.3 Physical and fitting parameters

The good balance between the accuracy and predictability of [MF](#) models is attained by combining both physical properties and reliable phenomenological formulations. To this end, correct identification of both physical and fitting parameters is paramount. The physical parameters reported in literature are given in Table 6, whereas the fitting parameters are listed in Table 7. To ensure physical consistency, the latter were identified via inverse modeling following a hierarchical optimization strategy (see section 4.4).

4.3 Complementary thermokinetic simulations

To consider the effect of precipitate evolution on the creep deformation response, this [MF](#) model requires the time and temperature-dependent precipitate history to be introduced as input data. That is, the mean radius r_i and number density N_i of precipitates and their corresponding nucleation sites. This information is outsourced

Table 6: List of physical parameters with literature sources.

Parameter	Description	Magnitude	Source
η_{Cr}	SSH: Cr-FCC shear modulus misfit	1.7541 –	[55]
α_{FCC}	SSH: FCC geometrical constant	16 –	[57]
δ_{Cr}	SSH: Cr-FCC size misfit	0.0298 –	[55]
w/b	SSH: mean atom travel distance ratio	1.70 –	[55]
d_{AGS}	Average grain size (diameter)	150 μm	[48]
a_g	Unit cell size (FCC)	0.356 nm	[83]
b	Burgers' vector (FCC Fe-Ni-Cr)	0.253 nm	[84]
α	Constant σ_T , optimized for 800H alloy	0.6 –	[50]
m_T	Taylor factor, optimized for FCC	3.06 –	[84]
β	Factor for dislocation production at GBs	0.0375 –	[31]
δ_{anh}	Dislocation annihilation coefficient	5 –	[31]
η_v	Coefficient for transfer of defects into jogs	1.00×10^{-6} –	[31]
Ω_{FCC}	Transfer parameter of dislocations into jogs	$1.21 \times 10^{-29} \text{ m}^3$	[31]
Θ_{DW}	Mean misorientation angle of DWs	5°	[74]

from ex-situ thermokinetic simulations conducted with [Thermo-Calc](#) PRISMA module, using the TCFE13 and MOBFE8 databases. These calculations, intended to recover the kinetics of precipitates in 800H during creep, correspond to isothermal simulations with no stress dependency (i.e., equivalent to aging). Such an approximation is admissible given the low stress levels addressed in this research.

4.3.1 Thermokinetic simulation methodology

The initial number density distribution of the precipitates in Incoloy 800H is largely determined by the mandatory [SA+WQ](#). In view of the influence of these precipitates (in particular, [Cr₂₃C₆](#) nucleating at dislocations [58]) on the [SSH](#) behavior of this material, accurate determination of the initial precipitate distribution is paramount. To this end, a thermokinetic simulation that replicates the [SA+WQ](#) process shown in Fig. 5a was first performed. The resulting [number density distribution \(NDD\)](#) of the primary precipitates obtained after this simulation are shown in Fig. 5b.

Given the high stability and rapid precipitation of [Ti_x\(C,N\)](#) and to ensure numerical convergence in TC-PRISMA, only the initial [NDD](#) of [Cr₂₃C₆](#) is considered for the subsequent creep/aging thermokinetic simulations conducted in TC-PRISMA. A total of 5 isothermal simulations were conducted at: 700°C, 800°C, 900°C, 1000°C, and 1050°C. The evolution of the mean radius and number density of [Cr₂₃C₆](#) precipitates for an isothermal thermokinetic simulation at 1000°C for a total of $1 \times 10^5 \text{ h}$ (≈ 10 years) is shown in Fig. 6. The predicted coarsening of intergranular [Cr₂₃C₆](#) precipitates (i.e., at grain boundaries, grain edges and grain corners) is consistent with aging, whereas the dissolution of those nucleating at dislocations is the source of the [SSH](#) phenomenon addressed in this work. The mean radius and number density of precipitates during [MF](#) creep simulations are continuously calculated internally by means of time and temperature dependent linear interpolations.

The tabulated *.csv files containing our thermokinetic simulation results for 800H alloy can be made available upon request.

Table 7: List of fitting parameters of the model and their calibration target in hierarchical order. While parameter magnitudes cannot be openly disclosed for confidentiality reasons, the physical definition and calibration target for each parameter are provided to facilitate model reproducibility.

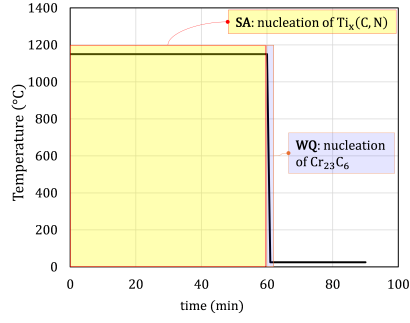
Calibration target	Parameter	Description	Unit
1. Steady-state creep rate ($\dot{\epsilon}_{ss}$)	a_1	Pre-exponential factor for v_g	ms^{-2}
	Q_g	Thermal activation energy for v_g	J
	A_V	Parameters for V_r in σ_{ss} :	—
	B_V	$V_r(\sigma_a) = A_V [1 + B_V \exp(C_V \cdot \sigma_a)] \cdot b^3$	—
	C_V		Pa
2. Solid-solution hardening (SSH)	C_1	f_{ss} – Equation (8)	—
	m		—
	φ_1	ΔE_b – Equation (11)	—
	φ_2		J
	b_1		J
	b_2	Parameters for diffusion of Cr Equation (12) & Equation (13)	—
	b_3		K
	b_4		—
	b_5		—
3. Tertiary creep stage (damage)	m_w	Stored plastic energy:	—
	w_{crit}	$w_s(\sigma_0) = \sigma_a \epsilon^{1/m_w}$	—
	A_1	Cavitation damage parameters:	—
	A_2	$\dot{D}_{cav} = A_1 (1 + A_2 \exp(T/A_3)) \cdot \dot{\epsilon}$	—
	A_3		—

4.4 Parameter uniqueness and identification methodology

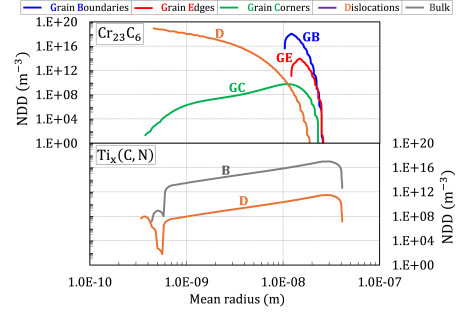
Given the dimensionality of the constitutive framework, an identification strategy was adopted to ensure physical consistency. First, a base model entitled to the case study alloy is defined by constraining the precipitate kinetics information inherited from ex-situ thermokinetic simulations (see section 4.3), physical parameters (see Table 6) and calibration of initial state variables (see section 4.1). Then, the inverse modeling of the creep deformation response addressing the individual contributions of $\dot{\epsilon}_{ss}$, SSH and damage is conducted. This is:

1. Identification of $\dot{\epsilon}_{ss}$ by optimization of parameters for v_g (a_1 , Q_g), and V_r (A_V , B_V , C_V).
2. Identification of SSH by optimization of parameters for f_{ss} (C_1 , m), ΔE_b (φ_1 , φ_2), and Cr-diffusion (b_i).
3. Identification of damage parameters for damage initiation (w_{crit} , m_w) and cavitation (A_i).

This hierarchical approach ensures that each parameter subset remains physically constrained to its respective deformation mechanism.

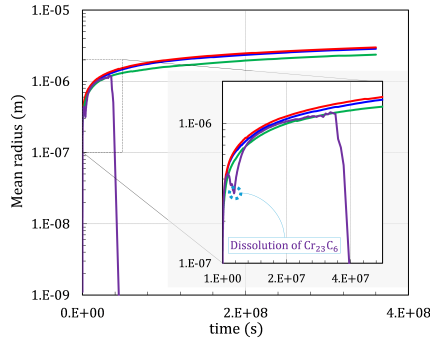


(a) Simulated SA (60 min at 1150°C) followed by WQ.

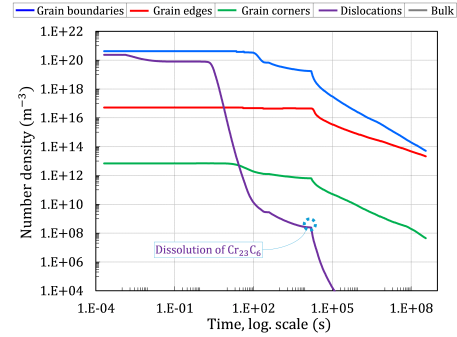


(b) NDD of Cr_{23}C_6 and $\text{Ti}_x(\text{C}, \text{N})$ nucleating during the SA+WQ, calculated with TC-PRISMA.

Fig. 5: Thermokinetic simulation of SA+WQ (Solution-Annealing followed by Water-Quenching) process conducted with Thermo-Calc PRISMA module for Incoloy 800H.



(a)



(b)

Fig. 6: Mean radius (a) and number density (b) history of M_{23}C_6 precipitates per nucleation site calculated after isothermal thermokinetic simulation conducted with Thermo-Calc PRISMA module at 1000°C for 1×10^5 h.

5 Results and validation

5.1 Creep deformation response:

A visual validation of the creep deformation predictions of the developed MF model is given in Fig. 7, where three experimental creep curves at equal temperatures and different stress levels are compared to their numerical counterparts. The exact scale information cannot be provided for confidentiality reasons.

The apparent prediction inaccuracies observed at the very beginning of the creep strain rate curves shown in their normalized form in Fig. 7b stem from the simplified stress-assisted diffusion model. The functions chosen to describe the time, stress and

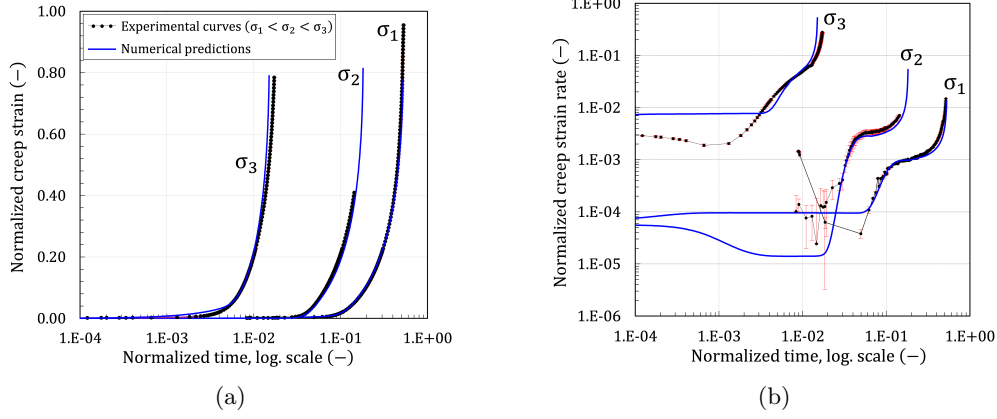


Fig. 7: Superposition of experimental and numerical creep strain (a) and creep strain rate (b) curves for Incoloy 800H for a single temperature and three different stress levels ($\sigma_1 < \sigma_2 < \sigma_3$). All the creep curves, with the exception of those at σ_2 , were conducted until rupture.

temperature dependent SSH (see section 2.1) are suited to recover the creep strain rate hardening effect within an order of magnitude of accuracy. Despite this approximation, the low level of creep deformation attained during the initial SSH creep strain rate regime leads to this error becoming negligible in the log term. Consequently, the SSH-induced creep hardening predicted in the creep strain vs. time curves in Fig. 7a is close to the experimental results.

Our MF model is identified and optimized for high-temperature and low-stress creep loadings. In the context of creep modeling, the applicability of the model is limited to a stress-temperature range whereupon a ubiquitous combination of creep mechanisms is involved. Venturing on stress levels falling outside this regime entails facing different creep mechanisms where new equations should be required. Let us illustrate this point by comparing the numerical predictions for two experimental creep curves at equal temperatures and different stress levels in Fig. 8. Both the creep strain and the creep strain rate benchmarks indicate that the model provides reasonable predictions for low(σ_4) stress levels. Conversely, the numerical curves obtained for the high stress case ($\sigma_5 \approx 2\sigma_4$) fail to reach the experimental response, dangerously underestimating the deformation response of the material.

During high stress creep deformation (see σ_5 in Fig. 8b), the $\dot{\epsilon}_{ss}$ is found to be an order of magnitude above than that predicted by the MF model. This finding indicates that creep is driven by a deformation mechanism that differs from that considered in Equation (1). In addition, no SSH effects are observed.

The creep deformation error calculated for those tests where the numerical predictions were consistent with the experimental results (e.g., σ_1 to σ_4 in Fig. 7 and Fig. 8) shows that for large creep strains $\epsilon \geq 5\%$, the model error is largely within a reasonable range: between 15% of underestimation and 5% of overestimation. Considering

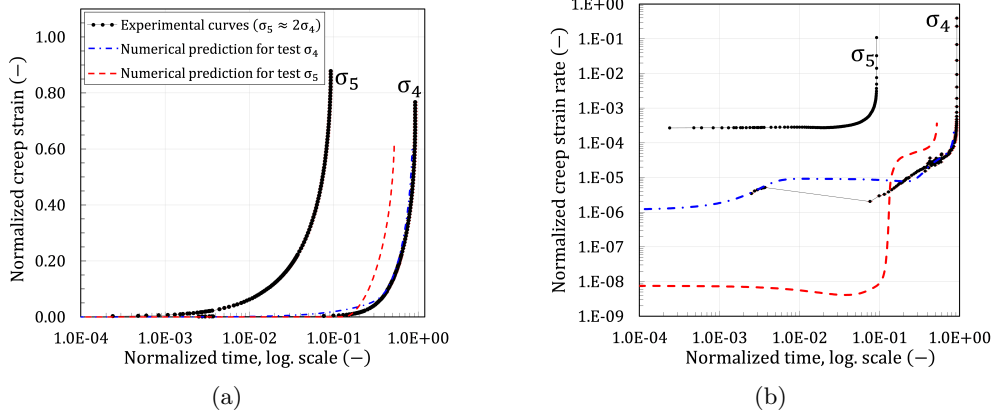


Fig. 8: Superposition of experimental and numerical creep strain (a) and creep strain rate (b) curves for Incoloy 800H for a single temperature and two different stress levels where $\sigma_5 \approx 2 \times \sigma_4$. All the creep tests presented here were conducted until rupture.

that experimental creep curve scattering is often found within a $\pm 10\%$ error, in addition to the fact that the model appears to exhibit a largely conservative error margin (i.e., underestimating the t_r), predictions for high-temperature low-stress loadings are validated. MF creep simulations can therefore be used for creep data extrapolation and creep rupture prediction.

5.2 Dislocation density evolution:

Given the inherent difficulties associated with accurately measuring the ρ_{GND} and the inability to effectively identify the respective contributions from ρ_m , ρ_s and ρ_b , validation between the numerical and experimental time-dependent evolution of these dislocation density types is possible only from a semi-quantitative standpoint [27, 44]. To this end, the ρ_{GND} values measured for 800H under AR condition and during creep (see Fig. 3) are compared to the dislocation density types predicted for the same creep testing conditions in Fig. 9. Let us compare the ρ_{GND} measurements to the predicted ρ_m , given the physical relationship between these two quantities [85]. The results show that the ρ_m history is within 1 order of magnitude of the experimental ρ_{GND} . In the context of MF modeling, such a deviation is considered to be in good agreement with the experimental results.

5.3 Damage evolution:

Creep damage (D_c) combines the effects of particle coarsening D_{ppt} and cavitation D_{cav} contributions. It is implemented following a semi-coupled approach, affecting the creep deformation response through the Orowan equation in Equation (1). In addition, an energy-based damage initiation Equation (17) is implemented. The fitting parameters (3 parameters for D_{cav} and the strain exponent m_w , see Table 7) are optimized for low-stress high-temperature creep tests following an inverse RMSPE error minimization

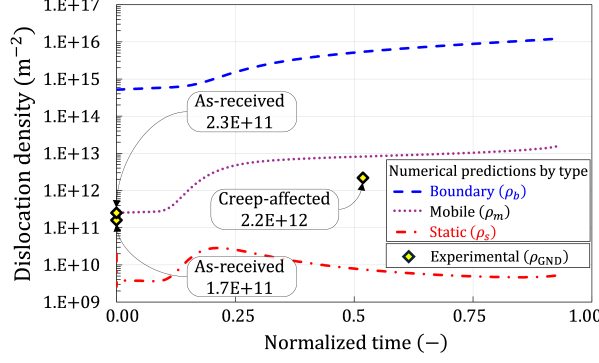
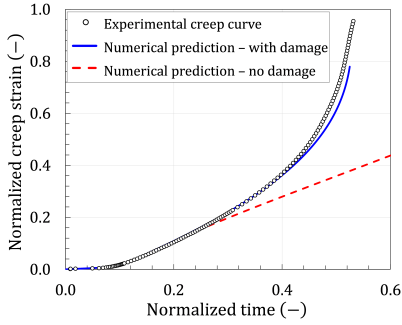
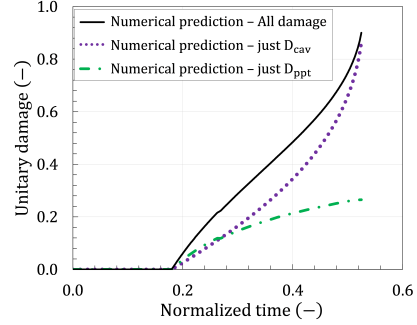


Fig. 9: Numerical prediction of dislocation density evolution by type (ρ_m , ρ_s and ρ_b) and experimental measurements (ρ_{GND}) for the same creep test (see Fig. 3).



(a) Creep strain benchmark for a single creep curve compared to numerical predictions with and without damage.



(b) Time-dependent D_c evolution for the test presented in (a) and contributions from D_{cav} and D_{ppt} .

Fig. 10: Effect of creep damage on the creep deformation response prediction and detailed assessment of the individual contributions from D_{cav} and D_{ppt} .

procedure. In addition, a critical damage level of $D_c^{crit} = 0.9$ is imposed to account for the rupture of the material. The simulation results presented in Fig. 7 and Fig. 8 show that the fracture prediction with these parameters is in good agreement with the experimental results within the stress-temperature applicability range of this model. The effects of individual damage contributions for curve σ_1 from Fig. 7 are illustrated in Fig. 10.

6 Discussion

6.1 About the stress-sensitivity of V_r

The $\dot{\epsilon}_{ss}$ dependency on stress and temperature is the core component of any viscoplastic model. The improved stress dependency of the apparent activation volume (V_r)

developed in this work was the result of several inverse modeling attempts. The optimal magnitude of V_r , identified independently on 4 experimental creep curves at equal temperatures and multiple stress levels, exhibited an inversely proportional Arrhenius-type dependency on σ_a . Similar V_r dependency on the stress has also been reported for 9Cr-1Mo martensitic steel [86], low-carbon steels [87], Ni₃Al polycrystals [88] and 316LN stainless steel [86], substantiating the interest in this generic formulation. Nevertheless, dependency on the strain rate [89], temperature [90], and grain size [91] has also been reported, indicting that the real mathematical form of V_r is strongly material dependent.

A similar Arrhenius-type V_r dependency to σ_a has been recently implemented for the MF modeling of the creep behavior of 30CrMoNiV5-11 martensitic steel by F. Chen et al.[92], further consolidating the applicability and generality of this approach.

Successful $\dot{\epsilon}_{ss}$ prediction entitled to the improved stress-sensitivity of V_r was only achieved for low stresses. The loss of $\dot{\epsilon}_{ss}$ predictability observed for high stresses (see σ_5 in Fig. 8) is attributed to a creep mechanism transition, either toward a diffusion-driven Coble creep or toward grain boundary sliding (GBS).

- The Coble creep theory is supported after the work of L.A. Beardsley et al.[18] in 2019. On the basis of the semi-physical BMD (Bird-Mukherjee-Dorn; see [93]) $\dot{\epsilon}_{ss}$ modeling formalism, the authors use a genetic algorithm to identify the combined diffusion and dislocation creep mechanisms involved in the high-temperature creep behavior of Incoloy 800H. The results provide phenomenological evidence of two dislocation-based (low and high-temperature) creep mechanisms and a single diffusion (Coble, where $\dot{\epsilon}_{ss} \propto \sigma^{-1}$) creep mechanism.

A complementary Coble creep term affecting the strain rate of the material in Equation (1a) could be implemented to improve our MF model predictability. Such an approach has already been implemented by F. Riedlsperger et al.[44] in 2023, and, more recently, by F. Chen et al.[92] in 2026, with outstanding results

- GBS is a plastic deformation mechanism in which grains move relative to each other. This shearing grain movement has been observed to promote cavitation damage during creep in materials with large grain structures (such as FCC in Cu- or Ni-alloys [33]). Its phenomenological interpretation follows the relationship $\dot{\epsilon}_{ss} \propto \sigma^{-2}$.

6.2 About the SSH approach

From a macromechanical perspective, SSH is a strengthening mechanism commonly associated with yield and tensile stress increments with negligible changes in ductility. This is consequence of the incomplete precipitation of alloying elements, which is purposely achieved by a combination of material composition and heat treatment optimization. For instance, the SSH effect attributed to variations in the matrix concentrations of elements such as Si, Cr, Mo, C, or N in stainless steels was determined by [94, 95] via regression.

For 800H alloy, while most of its alloying elements (such as Mg, Mn, Si or C) remain in solid solution state, a peak creep minimum attributed to a SSH mechanism

is observed at high temperatures. This creep strengthening is consequence of the incremental matrix concentration of Cr and C atoms following the rapid dissolution of the intragranular submicron Cr_{23}C_6 . Qualitative observation of the polarized light reflection response from these particles in AR material (see Fig. 2a) is corroborated via TEM by [58, 96]. Early attempts to link the first creep strain hardening peak observed in 800-type alloys to the precipitate strengthening effect of carbides achieved limited success. For instance, B. Reichert et al.[97] used a Kocks-Mecking-Estrin (KME)-based dislocation density model in which precipitate kinetics were included by means of an Ostwald-ripening approach with poor experimental validation. A similar but strongly phenomenological methodology was implemented by E. El-Magd et al.[98] for alloy 800HT with better results. Experimental evidence of a SSH process driving the plastic deformation of 800H alloy was obtained after quasi-static hot tensile test experiments conducted by Y. Cao et al.[21] in 2018, where a clear dynamic strain aging (DSA) regime characteristic of a SSH process is observed. In consideration of these latter findings, the solid-solution creep strain hardening peak exhibited by 800H alloy during low-stress high-temperature creep regimes is included in our MF creep model as a σ_i contribution. The combination of physical and phenomenological terms in the σ_{ss} formula (see section 2.1) allows for straightforward parameter optimization, whereas the Cr physical misfit parameters for Ni-Cr alloys given in [55] allow the finalization of the model set-up with no additional material characterization steps. However, accuracy limitations can be partially attributed to the following simplifications:

- The formula describing σ_{ss} (see Equation (8)) is a function of the Cr concentration in matrix ($\mathcal{X}_{\text{Cr}}$). The solid-solution contribution of the C atoms liberated during the Cr_{23}C_6 dissolution process is neglected because of their small size and number compared to those of Cr (see Supplementary material). Additional alloying elements that could contribute to the strengthening of 800H alloy in the form of solid-solution (e.g., P, Cu, Si, etc.) are disregarded in these calculations. Researchers aiming at more deterministic approaches could explore multiple SSH elements, following, for instance, the works of M.A. Moretti et al.[55] where multiple solid solution contributions are considered.
- Cr concentration function is inspired by diffusion in solids theory. The time-dependent Arrhenius function describing the decrease in $\mathcal{X}_{\text{Cr}}$ (see Equation (12)) is based on the theoretical works of P. Shewmon [59]. However the fitting parameters b_i ($i = 1, \dots, 5$) are purely phenomenological.

6.3 Final remarks

The results within low-stress and high-temperature creep loading regimes (i.e., σ_1 to σ_4 across Fig. 7 and Fig. 8) show that the strain rate hardening prediction is in good agreement with the experimental curves. The simplifications and concessions acknowledged here above are confined within a reasonably physical framework. The MF model developed in this work represents a balanced mesoscale approach where prediction accuracy, parameter uncertainty and numerical complexity are considered. This balance follows the modeling advice found in [99].

Exploitation of the physics-based results obtained from the current MF model can, for instance, complement experimental information gathered from conventional standard creep tests, decreasing the time and resources devoted to intensive creep experimental campaigns. Additionally, accurate predictions of creep scalars such as $\dot{\epsilon}_{ss}$, t_r or time-to-1% creep deformation can be used to feed surrogate models or construct physically-based stress-temperature material selection diagrams, exploiting machine learning approaches.

7 Conclusions

In this work, a dislocation forest based mean-field (MF) model adapted to predict the non-classical creep deformation response observed in 800H austenitic stainless steel was formulated and validated. The key conclusions are as follows:

- The integration of thermokinetic simulation results into the MF formulation establishes a direct, physically consistent link between the thermal history of the alloy and its evolving precipitate radius and number density. This eliminates the need for empirical precipitation parameters and enables a mechanistic description of solid solution hardening (SSH), precipitate strengthening, and coarsening-induced damage.
- The improved stress-sensitivity of V_r and the analytical diffusion-based SSH formulation capture the physical effects involved in the low-stress and high-temperature creep deformation response of 800H. Recent MF results reported in [5] showcased an Arrhenius-like V_r dependency on σ_a is a good indication of the generality of our approach. Nevertheless, more research is needed to corroborate the applicability of both the V_r and σ_{ss} formulations developed in this work.
- The creep prediction deviation observed for high stress is attributed to a transition towards an intergranular type of deformation. This finding is physically consistent with mechanisms such as Coble creep, where the strain rate is controlled by grain-boundary diffusion kinetics, or grain boundary sliding, where deformation is controlled by the shearing movement of grains relative to each other. The current model, focused on dislocation glide and climb mechanisms, systematically underpredicted strain for high stress. Identifying this transition provides a rigorous mechanistic basis for the observed discrepancy and clearly delineates the validity window of the dislocation-based approach. Additional high-stress creep tests and microstructural characterization instances are required to verify the deformation mechanism and extend the model to high stresses. The successful extension of a similar mean-field creep model has been recently reported for a 30CrMoNiV5–11 steel alloy by [92].
- The cavitation and precipitate coarsening damage formulations implemented in the MF model allow simulated creep curves to exhibit a strain softening consistent with the loss of load carrying capacity observed during experimental tests conducted until rupture. Additionally, the damage threshold enables accurate prediction of the final tertiary creep stage and fracture during standard creep tests.

Acknowledgements

All ULiège authors acknowledge the financial support of the European project AID4Greenest HORIZON-CL4-2022-RESILIENCE-01-19. Carlos Rojas-Ulloa acknowledges the FNRS FRIA grants B1 4000-8987 and B2 4002-1907. As Research Director F.R.S. - FNRS, Anne Marie Habraken acknowledges the F.R.S. - FNRS (Fonds de la Recherche Scientifique, Belgium, Walloon Region). Víctor Tuninetti and all ULiège authors acknowledge the international research cooperation agreement WBI/AGCID 2023-2025 RI-02 (DIE23-001).

Additionally, the authors acknowledge the helpful support of Dr. Bernhard Sonderegger, Dr. Florian Riedlsperger and PhD. candidate Laura Witzmann from the Institute for Engineering Materials at the Johannes Kepler University (Linz, Austria), and Dr. Surya Deo Yadav from the Department of Metallurgical Engineering at the Indian Institute of Technology (BHU) (Varanasi, India) for sharing their time and expertise on the capabilities of creep-oriented mean-field models. We also acknowledge Mg.Sc.Eng. Amedeo di Giovanni and Mg.Sc.Eng. Alexandre Venramini for their technical support during our experimental campaign.

Author contributions

- Carlos Rojas-Ulloa: Software, Formal Analysis, Funding acquisition, Investigation, Writing - Original draft preparation.
- Fan Chen: Software, Writing - review & editing.
- Jérôme Tchoufang Tchuindjang: Data curation, Investigation, Writing - review & editing.
- Olivier Dedry: Data curation.
- Tuan Nguyen-Minh: Formal analysis, Investigation, Writing - review & editing.
- Leo A.I. Kestens: Investigation.
- Anne Mertens: Supervision, Investigation.
- Víctor Tuninetti: Writing - review & editing
- Laurent Duchêne: Supervision, Writing - review & editing
- Anne Marie Habraken: Supervision, Investigation, Writing - review & editing, Funding acquisition

Declaration of interest statement

The authors declare no conflict of interest.

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Macromechanical creep model: VMVP

von-Mises viscoplastic constitutive model

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The non-classical creep response reported for 800H, described in literature [1–5] and revealed during our experimental campaign (see chapter 2), was successfully modeled for uniaxial tensile deformations within our OTR and OSR in chapter 3. However, industrial sector applications often require models to predict creep deformation and service life of complex geometries. This chapter deals with the implementation, identification and validation of a Chaboche-type FE constitutive model adapted to predict the non-classical creep behavior of 800H alloy.

The Chaboche-type unified viscoplastic constitutive model (UVCN) finite element (FE) law used herein is named VMVP (von-Mises viscoplastic). It is largely inspired in the work of Dr. Hélène Morch's PhD, consisting in the development and implementation of a Chaboche-type UVCN to predict the creep-fatigue behavior and service life of Hastelloy A230 components [6]. Cyclic hardening and softening, static and dynamic recovery, thermal effects and anisotropic damage evolution (Kachanov for creep [7], and Lemaitre for fatigue [8, 9]) are some of the main features of her formulation. In addition, the constitutive law allows to consider reversible and permanent changes in the mechanical properties of the material during simulation. Reversible changes were introduced by parametric dependency to temperature, whereas permanent changes are introduced throughout dependency to the maximum temperature in the thermal history. This constitutive model is hereafter referred to as "Chaboche-Morch" law.

FE simulations using Chaboche-Morch constitutive law (COLAW) were unable to recover the two-step creep strain rate minima. Its creep model, ruled by a Norton law and affected by temperature and damage, was deemed unsuitable for cases where complex creep regimes are observed. To solve this issue, a modification of the viscosity function by replacing it with the Graham-Walles (GW) equations was firstly explored. The results of this FE model were published in [10] (a peer-reviewed pre-print version of the article is provided in Annex 4A). Validated for literature results, the VMVP (von-Mises viscoplastic)-GW law showed limited capabilities once new ULiège experimental results arrived. A large number of parameters and numerical convergence difficulties were some of the detrimental aspects that motivated the development of a new constitutive model. With a greater understanding of the creep behavior of alloy 800H acquired during ULiège creep experimental campaign, a new approach consisting in the implementation of a modified Norton law was proposed: afN . This chapter describes the development, implementation and validation of this new VMVP- afN COLAW.

The mathematical formulation of the VMVP constitutive model is presented in Section 4.1. Special emphasis is placed in the modifications applied to the viscosity function, namely the GW and afN approaches. Note that the manuscript describing the development and implementation of the VMVP-GW viscous FE model is attached in annex 4A. Section 4.2 deals with the parameter identification, placing special attention to the identification of fitting parameters entitled to creep strain and damage computation. Numerical prediction of uniaxial creep deformation FE simulations are compared to ULiège experimental curves in Section 4.3. Section 4.4 discusses on the prediction capabilities of the identified model. The creep deformation and fracture prediction error is quantified and analyzed. Finally, the main conclusions derived from this analysis are summarized in Section 4.5.

4.1 Formulation

The formulation of Morch (and by extension our modified Morch-*af*N law, hereafter referred to as VMVP COLAW) follows that of a UVCN: a constitutive law where strain contributions falling into the inelastic category (e.g., plastic, creep, and thermal) are allocated into a single inelastic strain tensor (see [11]). It is defined by a single yield function Φ , where the yield stress σ_y is subtracted from the von-Mises equivalent stress as seen in Eq.(4.1a).

The Von-Mises equivalent stress is calculated by solving the J_2 -type function given in Eq.(4.1b), where the material yielding is linked to the second stress tensor invariant. $\tilde{\underline{\sigma}}$ is the effective component of the Cauchy stress tensor $\underline{\sigma}$ affected by the accumulated damage represented by the variable D as $\tilde{\underline{\sigma}} = \underline{\sigma} \cdot (1 - D)^{-1}$, with $(0 \leq D < 1)$.

The yield stress σ_y is defined in Eq.(4.1c) as the addition of the initial yield stress of the material σ_0 and the strain hardening component R . Note that in a UVCN, creep deformations cannot coexist with elasticity, so σ_y does not represent the actual yield stress of the material, but rather a fictional yield point from which inelastic (creep) deformations are allowed to occur. Finally, the material hardening is ruled by the Voce isotopic hardening law, described in its variational form in Eq.(4.1c) where B and Q are material constants, and $\dot{p}$ is the norm (scalar) of the inelastic strain rate tensor $\underline{\dot{\epsilon}}^p$ calculated in Eq.(4.1d).

$$\varphi = \Sigma_{VM}^{eq} - \sigma_y \leq 0 \quad (4.1a)$$

$$\Sigma_{VM}^{eq} = J_2(\tilde{\underline{\sigma}} - \underline{X}) = \left[\frac{3}{2} (\tilde{\underline{\sigma}} - \underline{X}) : (\tilde{\underline{\sigma}} - \underline{X}) \right]^{1/2} \quad (4.1b)$$

$$\sigma_y = \sigma_0 + R(p) \quad \text{where} \quad \dot{R} = -B(Q - R)\dot{p} \quad (4.1c)$$

$$\dot{p} = \left(\frac{3}{2} \underline{\dot{\epsilon}}^p : \underline{\dot{\epsilon}}^p \right)^{1/2} \quad (4.1d)$$

The J_2 -type function described in Eq.4.1b calculates the von-Mises equivalent stress based on two tensors: the deviatoric component of the effective Cauchy stress tensor $\tilde{\underline{\sigma}}$ and the backstress tensor $\underline{X}$. These are described hereafter in Eq.(4.2a) and Eq.(4.2b) respectively.

- 4.2b1: Swift-type hardening
- 4.2b2: Dynamic (strain-rate dependent) recovery.
 - γ_i : non-Masing behavior (see [12, 13])
 - $\dot{\underline{Y}}$: mean-stress evolution (see [14]).
- 4.2b3: Static recovery
- 4.2b4: Thermal inelastic contributions

The total backstress $\underline{X}$ is defined as a summation of Armstrong-Frederick type backstress functions $\underline{X}_i$, each representing a singular backstress contribution. The physical phenomena represented within each term are described hereafter according to the labels given in Eq.(4.2b).

$$\tilde{\underline{S}} = \tilde{\underline{\sigma}} - \frac{1}{3}\text{tr}(\tilde{\underline{\sigma}})\underline{I} \quad (4.2a)$$

$$\underline{X} = \sum_{i=1}^{n_{AF}} \left[\underbrace{\frac{2}{3}C_i\dot{\underline{\epsilon}}^p}_{4.2b1} - \underbrace{\gamma_i J_2(\dot{\underline{X}}_i - \dot{\underline{Y}}_i)\dot{p}}_{4.2b2} - \underbrace{b_i J_2(\underline{X}_i)^{(r_i-1)}\underline{X}_i}_{4.2b3} + \underbrace{\frac{1}{C_i} \frac{\partial C_i}{\partial T} \dot{T} \underline{X}_i}_{4.2b4} \right] \quad (4.2b)$$

A more detailed breakdown of each term contained in the backstress formulation from Eq.(4.2b) is offered in [10], or in the works of H. Morch [6], R. Ahmed & T. Hassan [15–18], or the outstanding review by J.L. Chaboche [11].

The damage formulation is inherited from the Chaboche-Morch COLAW [6]. The equivalent accumulated damage D is calculated as the linear summation of the creep (D_c) and fatigue (D_f) damage contributions as $D = D_c + D_f$. The creep damage is defined following the Kachanov [7] approach, whereas fatigue damage is introduced after the work of Lemaitre [8]. The Continuum Damage Mechanics (CDM) formulations are presented in their time differential form in Eq.(4.3a) and Eq.(4.3b) respectively, where S_c , S_{ce} , k_3 , k_4 & k are creep damage fitting parameters, and S_f , S_{fe} , k_1 & k_2 are fatigue fitting parameters. In addition, H. Morch et al.[19] incorporated creep and fatigue damage temperature-dependency through parameters S_c and S_f respectively.

$$\dot{D}_c = k_3 \left(\frac{k_4 \tilde{\underline{\sigma}}}{S_c} \right)^{S_{ce}} \frac{1}{(1-D)^k} \quad (4.3a)$$

$$\dot{D}_f = k_1 \left(\frac{k_2 \tilde{\underline{\sigma}}}{S_f} \right)^{S_{fe}} \dot{p} \quad (4.3b)$$

Important notes on the VMVP law used for Incoloy 800H

Backstress: It was implemented into VMVP to retain numerical consistency with the previous Chaboche-Morch UVCN. Since no cyclic loadings were addressed in this PhD. project, however, the backstress contributions are neglected.

Damage: Following the general Chaboche-type UVCN layout, both creep (D_c) and fatigue (D_f) CDM approaches were implemented in VMVP. However, since no cyclic loadings are considered in this PhD. project, only Kachanov creep damage initiation and evolution is considered.

4.1.1 Viscosity function

The final ingredient in the UVCN recipe is the viscosity function. The combined effects of the isotropic hardening function (R in Eq. (4.1c)), the backstress (in particular, the static recovery term 4.2b3 in Eq.(4.2b)), and the CDM approach affect the creep deformation response predicted by a UVCN, **the viscosity function is the core component defining the viscous deformation response in the UVCN**. It describes the uniaxial creep strain rate ($\dot{p}$) as a function of material constants and external loadings.

The original Chaboche-Morch COLAW uses a Norton viscosity function shown in Eq.(4.4a). Conversely to the conventional scalar Norton law (previously described in Eq.(1.3), see section 1.1.2), its formulation is adapted to fall within the context of a FE implementation. The

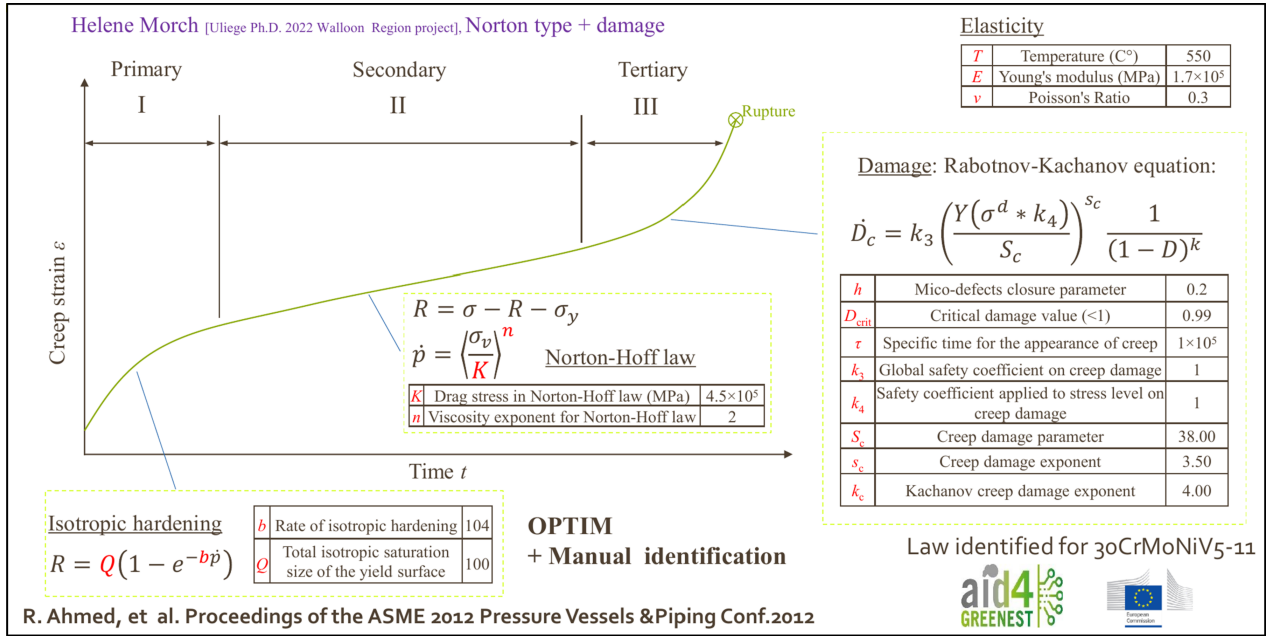


Figure 4.1: Standard creep strain vs. time curve showcasing the independent contributions from the isotropic hardening, viscosity function and damage law on stages I, II and III of creep, respectively. Figure extracted from slide #14 of MMELO (Metallic Microstructures European Lectures Online) presentation by Dr. Anne Marie Habraken in 2024 [20].

The parameters given were the result of a first optimization to recover the creep deformation response of a Martensitic steel alloy 30CrMoNiV5–11. To this day, new experimental results attained at ULiège in the context of AID4Greenest project have strongly evolved.

angle brackets represent a conditional function described in Eq.(4.4b), whereas σ_v is the over-stress. The latter is defined as the difference between the applied (true) equivalent stress (σ_a) and the fictional yield stress (σ_y), as seen in Eq.(4.4c). The effect of the isotropic hardening, Norton viscosity function, and Kachanov CDM optimized to describe the classical creep deformation response of another steel alloy 30CrMoNiV5–11 in the context of AID4greenest European research project is shown in Figure 4.1.

$$\dot{p} = \left\langle \frac{\sigma_v}{K} \right\rangle^n \quad (4.4a)$$

$$\langle x \rangle = \begin{cases} x & \text{if } x > 0 \\ 0 & \text{otherwise} \end{cases} \quad (4.4b)$$

$$\sigma_v = \sigma_a - \sigma_y \quad (4.4c)$$

The modeling methodology described in Figure 4.1 is applicable for classical creep responses [11] where a single well-defined steady-state creep strain rate ($\dot{\epsilon}_{ss}$) is observed. However, it cannot be used to recover non-classical creep curves such as those exhibited by Incoloy 800H. Case and point: an unsuccessful attempt to recover the non-classical creep response of 800H alloy is observed in Figure 4.2. Following a parameter optimization using OPTIM (automatic parameter optimization strategy based on the Levenberg-Marquardt algorithm [21]), the Chaboche-Morch FE model managed to only recover the first minimum creep strain rate (**mcr**) related to solid-solution hardening (SSH); the rest of the curve was neglected by the algorithm.

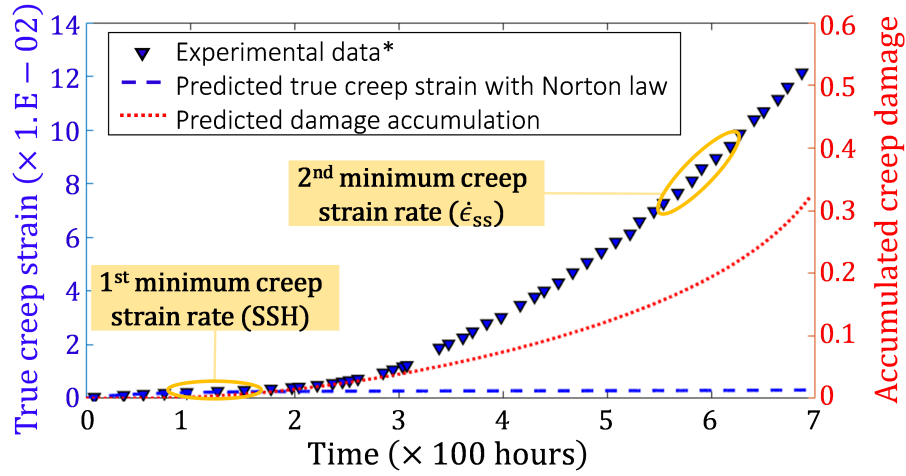


Figure 4.2: Numerical FE simulation results of a Chaboche-Morch [6] COLAW optimized to predict the experimental creep strain vs. time behavior of Incoloy 800H at 1000°C and 11 MPa (experimental curve * after [1]). The optimized model assumed an early damage activation and could not predict the second minimum creep strain rate of the material. Figure adapted from [10].

Non-classical creep deformation responses were naturally out of the scope of our conventional Norton-based Chaboche-Morch UVCN. Recovering the two-step creep strain rate minima exhibited by Incoloy 800H required a modification of the model, consisting of replacing the viscosity function.

First attempt: the Graham-Walles function.

The GW approach to creep (described in Eq.(1.6) in section 1.1.2) computes the equivalent creep response as the linear summation of empirical non-linear creep curves (see Figure 1.9). Its exploitation as a viscosity function within a FE code was first achieved in the context of N.K. Karthik's PhD. in RWTH Aachen University [22–24], where it was used to predict the creep-fatigue deformation of Ni-alloy components subjected to high-temperature low-stress thermal cycles.

Despite the lack of ULiège experimental curves at the time (2022), a GW viscosity function was implemented in our VMVP model. The creep curve summation strategy was thereby explored as a possible solution that would enable the modeling of non-classical creep curves in our FE code as seen in Figure 4.3. The resulting FE model was validated on 4 creep curves extracted from literature: V. Guttman & R. Bürgel [1] (1000°C at 11 & 35 MPa) and K. Tachibana [25] (950°C & 45 MPa and 900°C & 35 MPa). The results were published in a special issue of Materials at High Temperatures (Taylor & Francis) following the 6th edition of the ECCC conference (C. Rojas-Ulloa et al.[10], see peer-reviewed pre-print in Annex 4A of this thesis). The creep predictions were found in good agreement with the 4 experimental curves recovered from literature. The large number of parameters (3 per creep rate equation+1, 4 equations are required for a complex non-classical curve), the non-uniqueness of parameters, and convergence problems during steep creep rate slope changes, explain why the GW approach was impractical beyond this case study. The main contribution stemming from this first attempt lies in the computational aspects surrounding the GW equation implementation. It corresponds to the modification of a viscosity function within a fully-implicit UVCN integration scheme. Later, supported by a new ULiège experimental creep test campaign on 800H, an alternative viscoplastic law was developed.

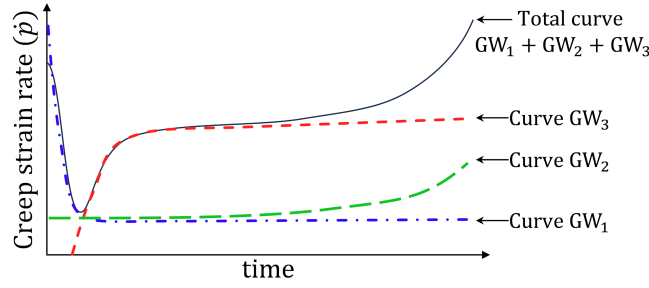


Figure 4.3: Computation of a non-classical creep strain rate curve following the Graham-Walles (GW) viscosity function approach involving 3 equations (GW_1 , GW_1 , and GW_3).

activation-function Norton (*afN*)

A Norton-based viscosity function for 800H alloy was envisioned after discovering that the 2nd **mcr** observed throughout our creep experimental campaign was found to be consistent with the $\dot{\epsilon}_{ss}$ of the material. The classical $\dot{\epsilon}_{ss}$ dependency on the stress (and temperature, throughout the drag stress K , see [19]) offered by the Norton law (see Eq.4.4) was the foundation of this new viscosity function adapted for materials with two **mcr**.

The 1st "SSH-attributed" **mcr** was incorporated into the Norton law as a stress (σ_a), temperature (T) and creep strain (p) dependent activation function. It delays the onset of the $\dot{\epsilon}_{ss}$ by applying a factor x ($0 < x \leq 1$) that evolves with the total creep strain rate ($\dot{p}$), thus recovering the gradual increment in $\dot{p}$ observed in experiments where two **mcr** are observed. Note that a magnitude $x = 1$ allows the creep strain rate $\dot{p}$ to equate to that computed by the Norton function. The intended effect of the proposed *afN* is compared to a conventional Norton viscosity function in Figure 4.4.

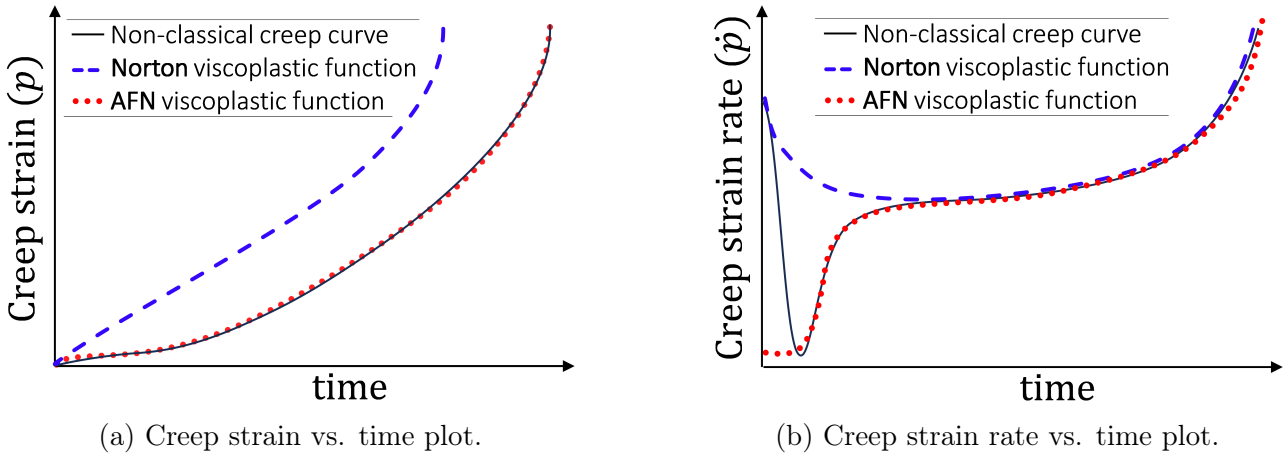


Figure 4.4: Creep deformation response prediction achieved with a proposed *afN* compared to a conventional Norton viscosity function in the context of a non-classical creep curve.

The form of the *afN* viscosity function is presented in Eq.(4.5), where a_N , b_N , and c_N are the activation function 'parameters' (temperature and stress dependency of these to be defined during the identification procedure), K is the drag stress and n is the Norton exponent. Note that σ in Eq.(4.5) comprises general stress dependency, e.g., engineering stress (σ_e), true applied stress (σ_a), yield stress (σ_y) or overstress (σ_v).

$$\dot{p}(\sigma, T, p) = \exp \left[-a_N (1.00 + p - 0.001 \cdot b_N)^{-c_N} \right] \cdot \left\langle \frac{\sigma_v}{K} \right\rangle^n \quad (4.5)$$

Following experimental observations of the variation of the $\dot{\epsilon}_{ss}$ of Incoloy 800H across multiple

stress and temperature levels (see chapter 2), temperature dependency is introduced into the Norton formulation throughout the drag stress (K). The corresponding Arrhenius-type formula, shown in Eq.(4.6), is inherited from the work of H. Morch [6, 19], where A_K , B_K , and C_K are fitting parameters.

$$K(T) = A_K \cdot \left[1 - B_K \cdot \exp\left(\frac{C_K}{T}\right) \right] \quad (4.6)$$

The dependency of *a*fN "parameters" a_N , b_N and c_N (see Eq.(4.5)) on stress and temperature was defined based on the best analytical curve fitting of all independent magnitudes identified for multiple ULiège experimental creep tests. This procedure is explained in detail in section 4.2. The analytical functions found to best fit these parameters are given in Eq.(4.7), where σ_e is the engineering (creep) stress and T is the temperature in °C. Ultimately, the activation function approach requires the following unique parameters to be identified: a_1 , a_2 , b_1 , b_2 , b_3 , and c_N .

$$a_N(\sigma_e) = \left[1 + \exp(\sigma_e - f_a) \right]^{-1} \quad (4.7a)$$

$$\text{with } f_a(T) = a_1 \cdot T + a_2 \quad (4.7b)$$

$$b_N(\sigma_e) = b_1 \cdot \exp(\sigma_e \cdot f_b) \quad (4.7c)$$

$$\text{with } f_b(T) = b_2 \cdot \exp(b_3 \cdot T) \quad (4.7d)$$

Finally, σ_e is computed during the FE code from the von-Mises applied (true) stress σ_a as in Eq.(4.8). This magnitude is used to facilitate the parameter identification procedure.

$$\sigma_e = (3/2)^{-1/2} \cdot \sigma_a \cdot \exp(-p) \quad (4.8)$$

Combined with the 4 fitting parameters (n , A_K , B_K and C_K) inherited from the Chaboche-Morch Norton viscosity function formulation, the total number of parameters needed for this modified *a*fN viscosity function reaches to 11.

4.2 Parameter identification

4.2.1 Material constants and base parameters

The material constants not directly involved in the creep deformation response of Incoloy 800H are listed hereafter:

- **Elastic & thermal constants:** the elastic modulus (E), shear modulus (G), Poisson ratio (ν) and thermal dilatation coefficient (α_T) are all defined as temperature-dependent functions as seen in section 1.2.3 of this thesis.
- **Fictional yield stress** (σ_y in Eq.(4.1c)): As explained at the beginning of section 4.1, the parameter σ_y is a yield stress, inherited from conventional plastic constitutive models. However, in the context of creep and UVCs, it represents a fictional stress threshold below which no inelastic deformations occur (see definition of σ_v for the Norton viscosity function in Eq.(4.4).

Setting all yield stress parameters to zero (i.e., σ_0 , B and Q) leads to a loss of numerical convergence as the concept of the yield surface is lost. To model creep, these parameters must be set to a very low value close to zero. In particular, B and Q are defined as a low positive number where $0 < B < Q < 1$.

- **Backstress ($\underline{X}$):** The original Chaboche-Morch UVCM is intended for the prediction of complex creep-fatigue interactions. The backstress formulation, presented in Eq.(4.2b), contains 4 terms entitled to different recovery mechanisms. Additionally, materials exhibiting multiple backstress formulations (useful, for instance, to incorporate combined recovery effects at multiple loading levels [6]). Given the creep focus of our Incoloy 800H research, the backstress recovery effects are neglected. However, in a similar fashion to that of the fictional yield stress, the backstress parameters cannot be set to zero, as this leads to numerical divergence. The following simulations are therefore conducted using a single backstress equation (i.e., $n_{AF} = 1$), and parameters C_1 , γ_1 , b_1 and r_1 are set to very low magnitudes. In particular, $C_1 \ll 1$, $b_1 \ll 1$, $\gamma_1 \ll 1$ and $r_1 \approx 2$.

4.2.2 Finite element boundary conditions

The FE model used across all the simulations presented in this chapter consists of a single hexahedral element of the type BLZ3T. The element, shown in Figure 4.5a, is defined with 8 nodes, a single integration point, 4 degrees of freedom per node (3 space coordinates + temperature) and no self weight. The dimensions of the element are set to $1 \times 1 \times 1$ (mm). Boundary conditions in Figure 4.5b allow a uniaxial deformation similar to that exhibited by our real life 800H specimens in our creep machine (see chapter 2).

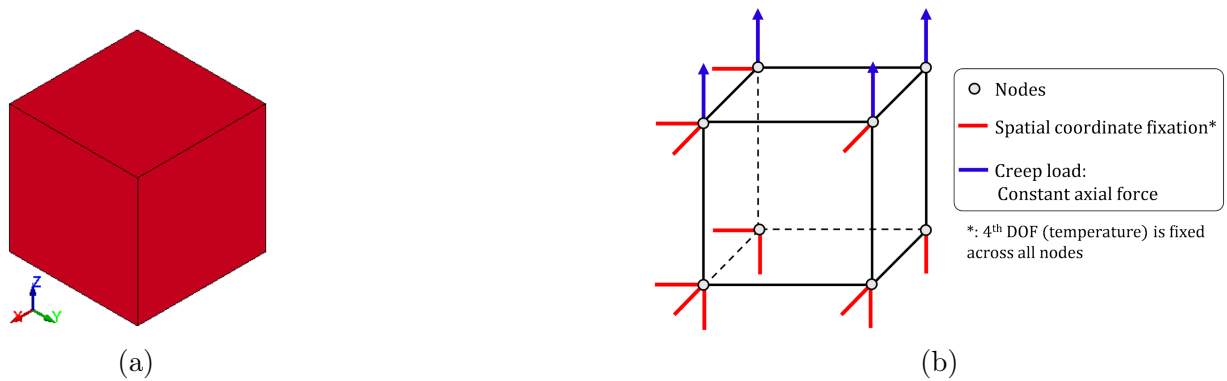


Figure 4.5: (a) BLZ3T element. (b) Boundary conditions (fixation and loadings) to achieve uniaxial deformation during creep. Figure (b) modified from [10].

4.2.3 Identification of fitting parameters

The methodology to identify the creep material parameters of VMVP for Incoloy 800H follows a 3-step hierarchical workflow. A flowchart describing this process, presented first in Annex 3A (not publicly available), is shown in Figure 4.6 .

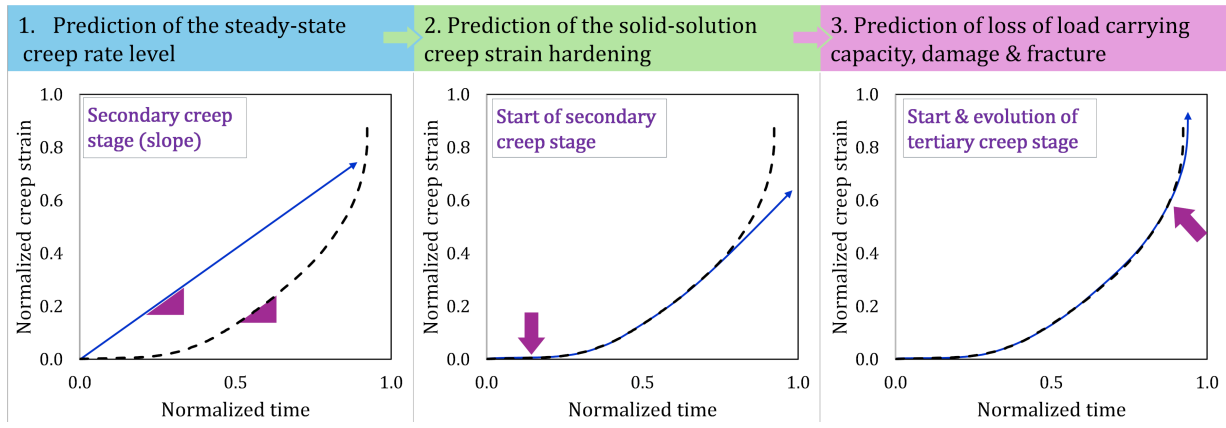


Figure 4.6: Flowchart describing the 3-step parameter identification of VMVP. Step 1 addresses the Norton component of the viscosity function, step 2 addresses the activation function parameters, and step 3 addresses the damage computation.

The parameter identification sequence (1→2→3) described in Figure 4.6 requires creep curves describing the creep deformation response of the alloy across multiple stress and temperature levels. The minimum number of experimental curves should be such that the non-linear $\dot{\epsilon}_{ss}$ dependency on stress and temperature can be captured. In other words, at least 3 temperature levels (e.g., T_1 , T_2 & T_3) and 3 stress levels for a single temperature (e.g., $\{T_1, \sigma_1\}$, $\{T_1, \sigma_2\}$, $\{T_1, \sigma_3\}$) are required.

In addition to a reliable set of experimental SCT curves, steps 1 and 2 of our hierarchical identification approach scheme require the 1st and 2nd minimum creep strain rates belonging to the non-classical creep response observed in Incoloy 800H to be correctly identified. The trends exhibited by these two different creep strain regimes are depicted for 800H in Figure 4.7, each addressing a single temperature and multiple stress levels. In particular, Figure 4.7a showcases a set of non-classical 800H creep curves obtained from MFC simulations, whereas creep curves in Figure 4.7b correspond to ULiège experimental results where both classical (σ_F) and non-classical creep responses are observed.

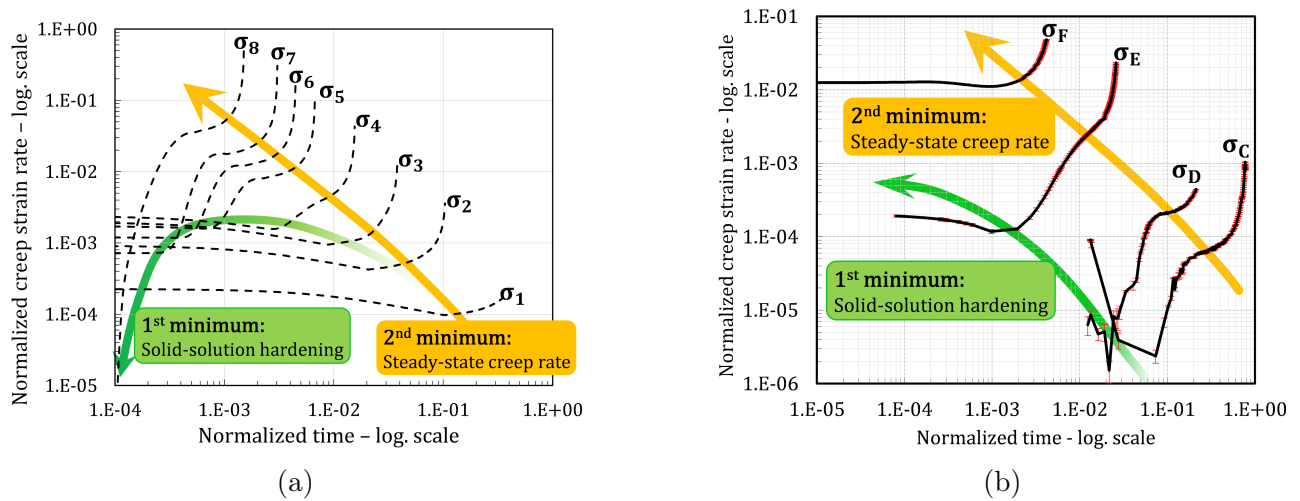


Figure 4.7: (a) Numerical and (b) experimental normalized creep strain rate vs. time curves showcasing the 1st (SSH) and 2nd ($\dot{\epsilon}_{ss}$) minimum creep strain rate trends for a single temperature and multiple stress levels. Curves (a) are the results from MFC simulations (see chapter 3), with $\sigma_1 < \sigma_2 < \dots < \sigma_8$; adapted from [26]. Curves (b) correspond to ULiège experimental results, with $\sigma_C < \sigma_D < \sigma_E < \sigma_F$.

Step 1 : Norton parameters

The fitting parameters of the Norton part of our *afN* viscosity function were found following a simple analytical procedure (i.e., no simulation required). To this end, the activation function component is disregarded (i.e., $a_N = 0$) and the Norton equation describing the stress-temperature- $\dot{p}$ relationship is evaluated for $\dot{p} = \dot{\epsilon}_{ss}$ as seen in Eq.(4.9a), where σ_v^{ss} is the overstress at $\dot{\epsilon}_{ss}$, p_{ss} is the creep strain at $\dot{\epsilon}_{ss}$ ($p_{ss} \approx 2.5 \times 10^{-2}$), and R is the isotropic hardening function evaluated at p^{ss} (see Eq.(4.1c)).

$$\dot{\epsilon}_{ss} = \left\langle \frac{\sigma_v^{ss}}{K} \right\rangle^n \quad (4.9a)$$

$$\sigma_v^{ss} = \sigma_a^{ss} - R(p^{ss}) \quad \text{with} \quad \sigma_a^{ss} \approx (3/2)^{-0.5} \cdot \sigma_e \cdot \exp(-p^{ss}) \quad (4.9b)$$

The Norton formulation used in this work (see Eq.(4.4a)) requires a single magnitude of n per creep mechanism (i.e., no stress or temperature dependency), whereas the drag stress K is considered temperature dependent following the Arrhenius-type function of Eq.(4.6). All experimental creep curves addressed hereafter in the context of the Norton parameter identification fall within a single creep mechanism, thereby requiring a single unique n . The temperature dependency of K implies a single K magnitude per temperature. The phenomenological relationship captured by the Norton component of this *afN* viscosity function is shown in Figure 4.8 for pedagogical purposes.

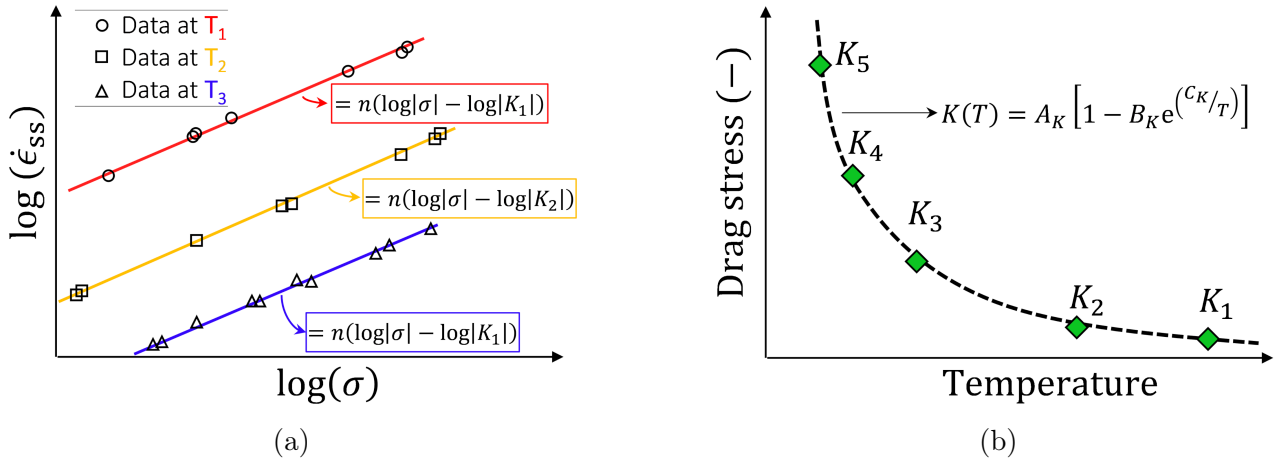


Figure 4.8: (a) Identification of Norton law parameters based on the inherent linear phenomenological $\dot{\epsilon}_{ss}$ - σ log. relationship. (b) Arrhenius curve fitting to recover the temperature dependency of the drag stress parameters (K_i) identified for each temperature T_i .

- **sub-step 1.a** (Figure 4.8a): identification of individual (K , n) points.
Here, a single set of (K_i , n) parameters was identified for each temperature level T_i by simple linear regression. The temperature dependency of the identified data points (K_i , T_i) is illustrated in Figure 4.8b.
- **sub-step 1.b** (Figure 4.8b): Fitting of the Arrhenius function parameters (A_K , B_K & C_K).
The series of K_i data points obtained for each temperature T_i in the previous step (1.1) exhibit an Arrhenius-type temperature dependency. Step 1.2 exploits this phenomenological relationship by fitting a temperature-dependent Arrhenius function of the form shown in Eq.(4.5). To this end, a new parameter optimization procedure consisting in an RMSPE minimization was conducted. The final set of parameters identified for Incoloy

800H (A_K , B_K , C_K & n) was found suitable for all creep stresses and temperatures addressed in this campaign. The presence or absence of a first SSH creep strain rate peak has no effect on the second creep strain rate peak ($\dot{\epsilon}_{ss}$).

step 2 : afN parameters

The optimal magnitude of the afN parameters (a_1 , a_2 , b_1 , b_2 , b_3 & c_N , see Eq.(4.6)) suited to recover the non-classical creep behavior of 800H alloy were found via an inverse modeling technique using OPTIM, an optimization software developed in-house based on the Levenberg-Marquardt algorithm [21] capable of handling multiple reference curves. In practice, the optimization methodology consisted of 3 sequential steps:

• **sub-step 2.a:** Individual OPTIM identification.

Individual magnitudes for afN parameters $a_{N,i}$ and $b_{N,i}$ (see Eq.(4.5)) were first identified for each creep test ($\sigma_{e,i}$, T_i) following an automatic parameter optimization strategy based on the OPTIM software. It must be noted that an educated guess of the magnitude of parameters $a_{N,i}$ and $b_{N,i}$ and a precision of $PREC = 0.005$ was required for each OPTIM simulation. Due to its high sensitivity, the magnitude of c_N was fixed and considered a constant (i.e., the steepness of the activation function change relied in its entirety on the magnitude of a_N and b_N). The outcome of step 2.1 is a set of ($a_{N,i}$, $b_{N,i}$) parameters for creep tests ($\sigma_{e,i}$, T_i).

• **sub-step 2.b:** Ascertaining stress and temperature dependency.

The stress dependency is established on the engineering stress (σ_e). To this end, a suitable analytical equation capable of recovering the stress-dependency of the data points ($a_{N,i}$, $b_{N,i}$) identified in step 2.a above must be chosen and fitted as $f(\sigma_e)$. The stress dependency exhibited by a_N and b_N was found to follow the phenomenological function defined in Eq.(4.7a) and Eq.(4.7c) respectively.

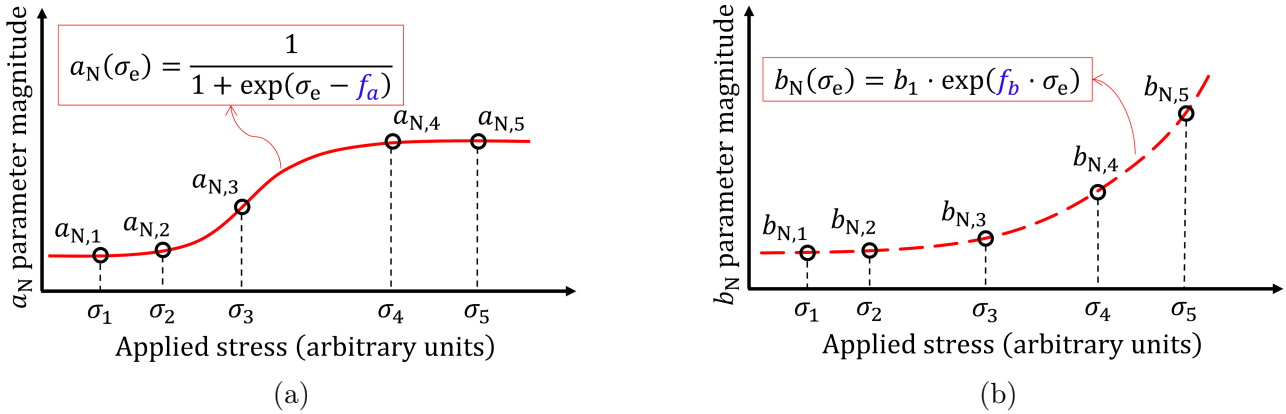


Figure 4.9: Stress-dependency exhibited by afN parameters (a) $a_{N,i}$ and (b) $b_{N,i}$ identified for a series of creep tests at different stress levels (σ_i). The analytical curves chosen to recover the engineering stress (σ_e) dependency of each parameter are displayed within.

Both of the stress-dependent functions identified here above in Figure 4.10a and Figure 4.10b contain a temperature-dependent function (f_a and f_b , respectively). The temperature dependency of these parameters is recovered in a similar fashion: finding a suitable analytical function that fits the magnitudes for the given dataset. The form of the analytical functions, are given for a_N and b_N in Eq.(4.7b) and Eq.(4.7d) respectively. Additionally, these are illustrated in Figure 4.10a and Figure 4.10b.

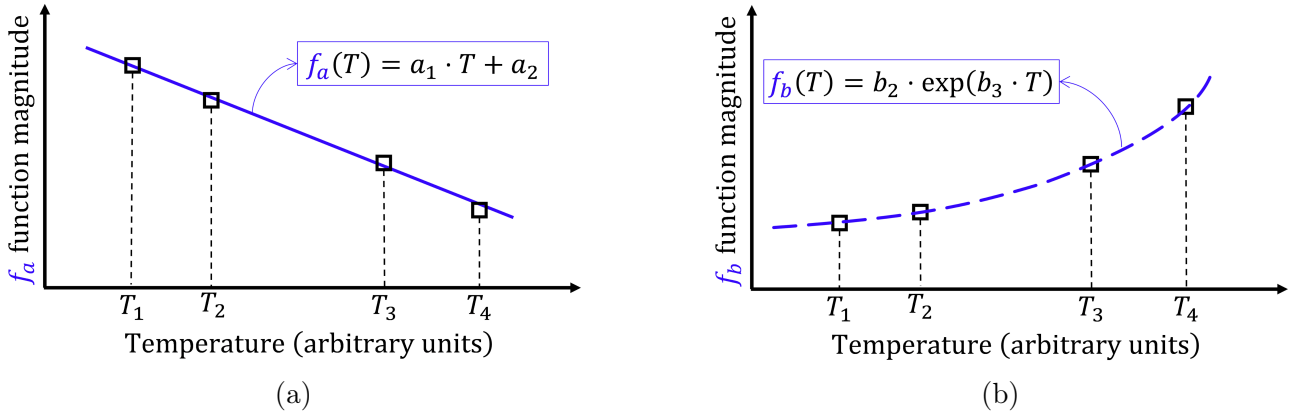


Figure 4.10: Temperature-dependency exhibited by afN functions (a) $f_a(T)$ and (b) $f_b(T)$ identified for a series of creep tests at different temperatures (T_i).

step 3 : Damage parameters

The objective of this creep damage identification procedure was to recover the creep strain softening attributed to the loss of load carrying capacity of the material until the fracture of the specimen was reached. Given the creep focus of our research, fatigue damage features inherited from the Chaboche-Morch COLAW, are hereafter disregarded.

The identification of creep damage parameters was conducted in three sub-steps:

- **sub-step 3.1:** Damage initiation criterion.

The creep damage initiation criterion chosen for Incoloy 800H consists of a temperature dependent creep strain threshold. It follows an Arrhenius-type function shown in Eq.(4.10), where p_1^c , p_2^c and p_3^c are fitting constants identified by simple linear regression. Damage is assumed to initiate when the current creep strain p reaches the single critical creep strain p^{critical} (i.e., when $p \geq p^{\text{critical}}$).

$$p^{\text{critical}}(T) = p_1^c \cdot [1 - p_2^c \cdot \exp(T/p_3^c)] \quad (4.10)$$

- **sub-step 3.b:** Creep damage accumulation.

This work uses the Kachanov creep damage formulation, presented in its semi-implicit incremental form for a time step t_{n+1} in Eq.(4.11a). Following the developments by H. Morch et al.[19], temperature dependency is introduced through parameter S_c as seen in Eq.(4.11b), while k_3 , k , S_{ce} and k are independent of temperature. In addition to the Arrhenius parameters s_1 , s_2 & s_3 in Eq.(4.11b), the total number of damage parameters is 7.

$$D_c^{n+1} = D_c^n + \Delta D_c^{n+1} \quad \text{with} \quad \Delta D_c^{n+1} = k_3 \left(\frac{k_4 \tilde{\sigma}^{n+1}}{S_c} \right)^{S_{ce}} \frac{1}{(1 - D^n)^k} \quad (4.11a)$$

$$S_c(T) = s_1 \cdot [1 - s_2 \cdot \exp(T/s_3)] \quad (4.11b)$$

To ease the parameter identification procedure, k_4 was fixed to $k_4 = 1$. The 6 other creep damage accumulation parameters (k_3 , k , S_{ce} , and Arrhenius parameters s_1 , s_2 & s_3) were identified following a combined analytical and inverse modeling methodology. A first analytical approach was calculated analytically, using 5 out of the 10 experimental curves available in ULiège. This analytical parameter set was considered as a first guess,

and used to initiate a final automatic parameter optimization strategy in OPTIM. The latter was arranged with all experimental tests available in ULiège, and was aimed at identifying parameters k_4 , k and S_{ce} (parameters k_4 , s_1 , s_2 & s_3 were kept fixed and constant). This decision is justified by the low sensitivity of these parameters.

• **sub-step 3.c:** Fracture prediction.

Once all damage initiation and accumulation parameters (steps 3.1 and 3.2 above) were completed, the CDM approach computes damage accumulation until convergence is lost. However, the latter occurs for $D \approx 1$, when unrealistic deformation levels are reached. Step 3.3 consists of introducing a critical damage threshold D^{critical} for which the material at the integration point is considered fractured. Empirical numerical-experimental comparison of results leads to the establishment of a single unique damage threshold for 800H of $D^{\text{critical}} = 0.20$.

4.3 Results

The creep deformation response of 800H alloy predicted using the recently identified *afN*-modified FE model is hereafter compared to that observed experimentally during the SCT campaign performed in ULiège. Experimental and numerical creep strain and creep strain rate curves are displayed for each creep test in their normalized form throughout Figure 4.11 to Figure 4.16; each figure addresses a single temperature level T_1 to T_6 respectively ($T_1 < T_2 < \dots < T_6$). Numerical predictions are shown in dashed blue lines, whereas experimental curves are shown in black. The standard deviation calculated for each experimental data points composing the experimental curves are shown in red.

T_1

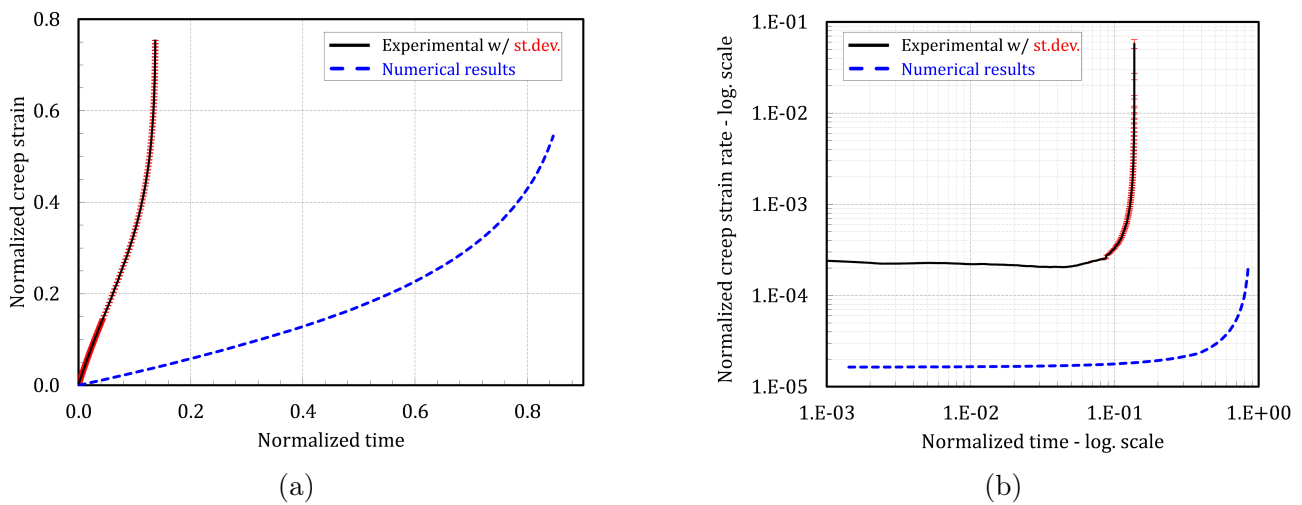


Figure 4.11: Comparison of experimental and numerical (a) creep strain vs. time and (b) creep strain rate vs. time curves for Incoloy 800H at temperature T_1 . Numerical curves are obtained from uniaxial FE simulations using a *afN* function fitted purely using ULiège creep experimental results.

No virtual mean-field creep tests are addressed herein.

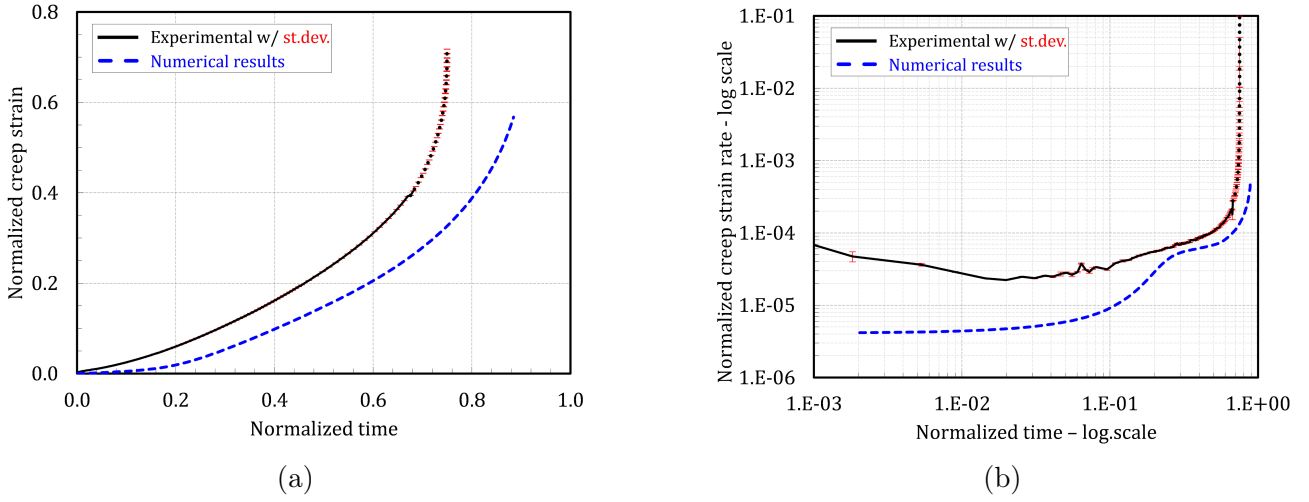
T_2 

Figure 4.12: Comparison of experimental and numerical (a) creep strain vs. time and (b) creep strain rate vs. time curves for Incoloy 800H at temperature T_2 . Numerical curves are obtained from uniaxial FE simulations using a afN function fitted purely using ULiège creep experimental results.

No virtual mean-field creep tests are addressed herein.

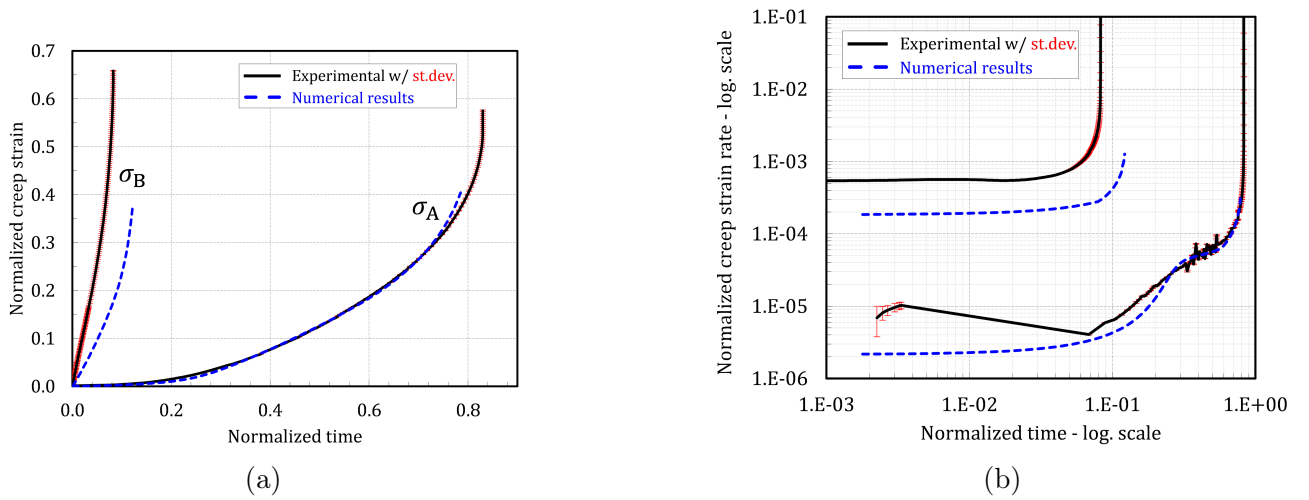
 T_3 

Figure 4.13: Comparison of experimental and numerical (a) creep strain vs. time and (b) creep strain rate vs. time curves for Incoloy 800H at temperature T_3 and multiple stresses ($\sigma_A < \sigma_B$). Numerical curves are obtained from uniaxial FE simulations using a afN function fitted purely using ULiège creep experimental results.

No virtual mean-field creep tests are addressed herein.

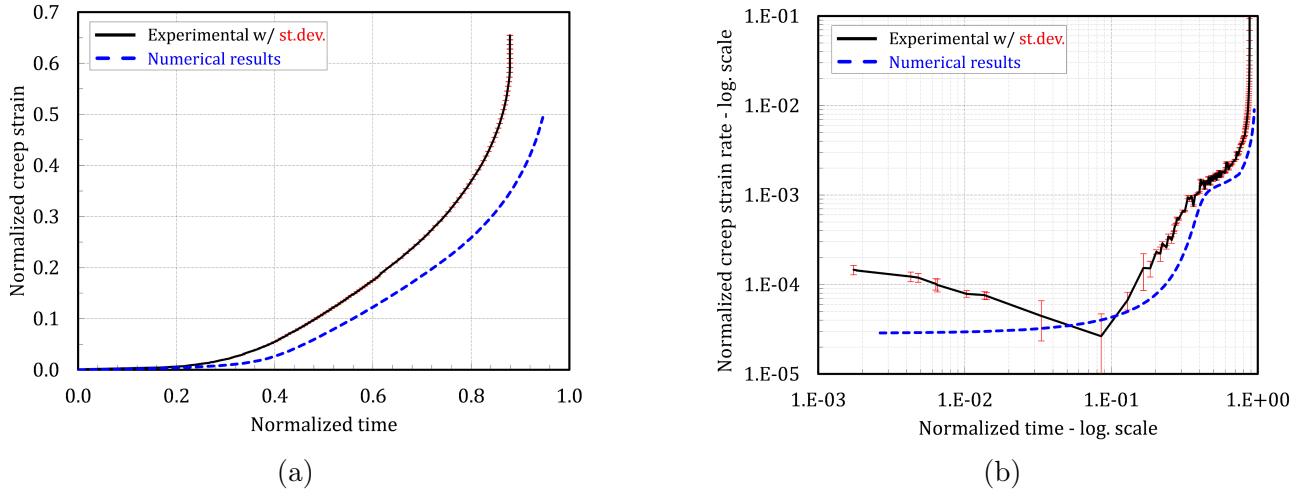
T_4 

Figure 4.14: Comparison of experimental and numerical (a) creep strain vs. time and (b) creep strain rate vs. time curves for Incoloy 800H at temperature T_4 . Numerical curves are obtained from uniaxial FE simulations using a afN function fitted purely using ULiège creep experimental results.

No virtual mean-field creep tests addressed herein.

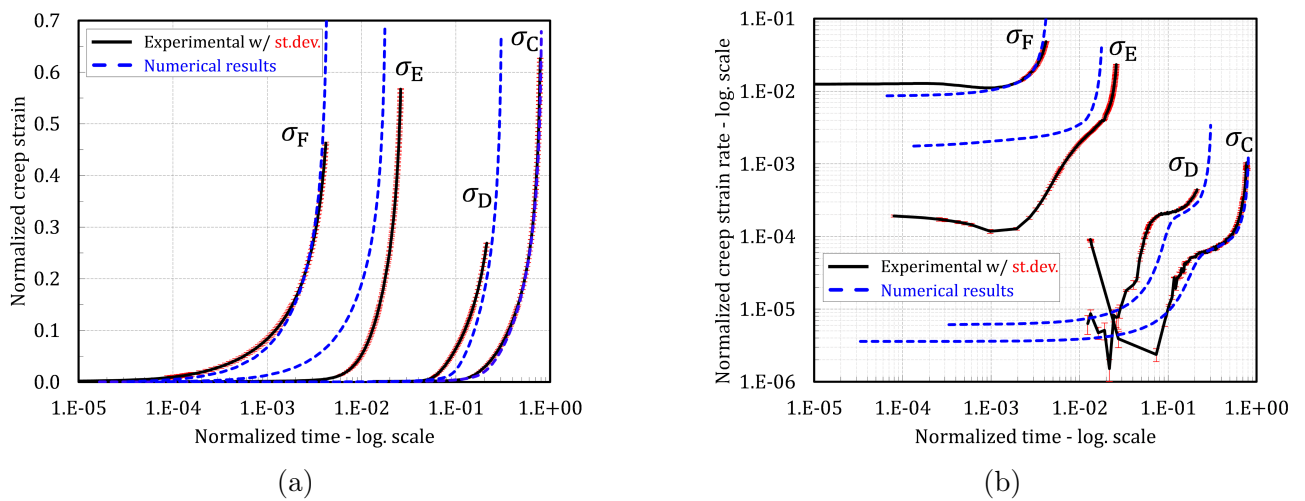
 T_5 

Figure 4.15: Comparison of experimental and numerical (a) creep strain vs. time and (b) creep strain rate vs. time curves for Incoloy 800H at temperature T_5 and multiple stresses ($\sigma_C < \sigma_D < \sigma_E < \sigma_F$). Numerical curves are obtained from uniaxial FE simulations using a afN function fitted purely using ULiège creep experimental results.

No virtual mean-field creep tests were addressed herein.

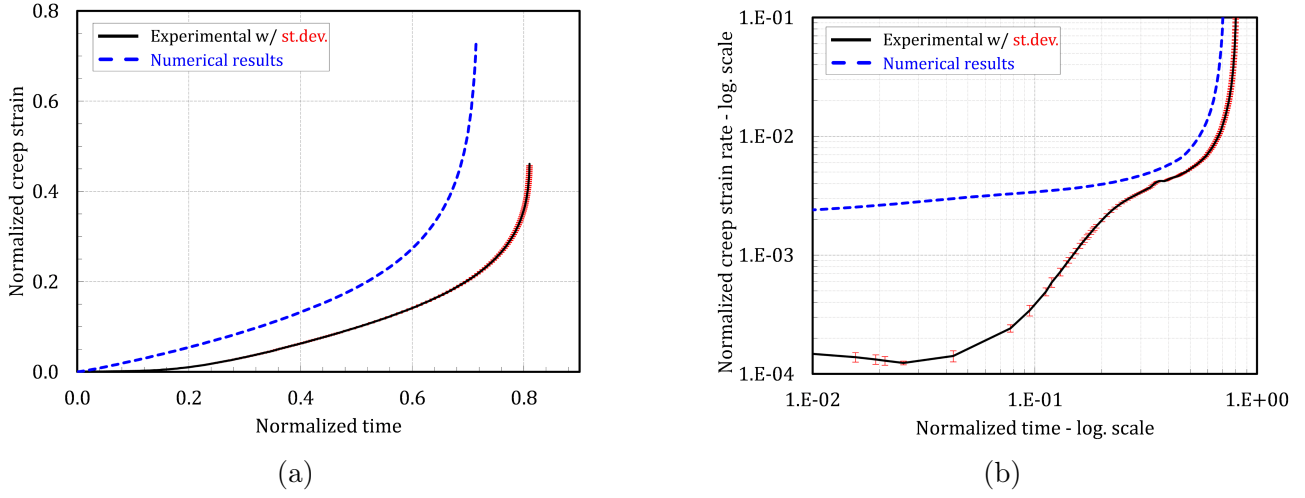
T_6 

Figure 4.16: Comparison of experimental and numerical (a) creep strain vs. time and (b) creep strain rate vs. time curves for Incoloy 800H at temperature T_6 . Numerical curves are obtained from uniaxial FE simulations using a afN function fitted purely using ULiège creep experimental results.

No virtual mean-field creep tests addressed herein.

4.4 Discussion

To better assess the creep predictability of the afN -modified FE model presented here above, the three key aspects of every creep curve presented from Figure 4.11 to Figure 4.16 are hereafter analyzed, namely, the 1st **mcr** peak (attributed to SSH), the 2nd **mcr** peak ($\dot{\epsilon}_{ss}$), and the fracture prediction. To this end, the relative (experimental-numerical) error at the 1st and 2nd minimum creep strain rate peaks, the time-to-1% creep strain, and the time-to-rupture (t_r), are summarized for all creep tests in Table 4.1. The relative error for the 1st and 2nd peak is calculated using the log. scale formula of Eq.(4.12a), whereas the time-to-1% creep deformation and t_r errors are calculated with the conventional formula of Eq.(4.12b). The nomenclature of each creep test analyzed in Table 4.1 follows the same temperature-stress creep loading designation used throughout Figure 4.11 to Figure 4.16.

$$\text{err}_{\text{rel}}^{\log} = \frac{\log_{10} |x_{\text{exp}}| - \log_{10} |x_{\text{num}}|}{\log_{10} |x_{\text{exp}}|} \quad (4.12a)$$

$$\text{err}_{\text{rel}} = \frac{x_{\text{exp}} - x_{\text{num}}}{x_{\text{exp}}} \quad (4.12b)$$

Table 4.1: Relative error at the prediction of the 1st (SSH) and 2nd minimum creep strain rate peaks, time-to-1% creep deformation, and time-to-rupture (t_r), calculated for FE simulations of ULiège SCTs conducted using the newly implemented and identified *afN*-modified model. A star (★) indicates that the test was used to identify the *afN* and Norton equation. Exceptionally good predictions (low error, error < 5%) are highlighted in **blue**, whereas particularly inaccurate predictions (error > 50%) are highlighted in **red**.

N.A.: not applicable; phenomenon not observed during the experiment.

N.P.: Not predicted; Numerical model wrongfully predicting no 1st creep hardening peak.

Creep loading			1 st min. (SSH)	2 nd min. ($\dot{\epsilon}_{ss}$)	time-to-1%	t_r
T ₁	—		N.A.	-16.7%	-1000%	-518.4%
T ₂	—		-9.8%	-0.02%	-159%	-18.0%
T ₃	σ_A	★	-4.0%	+0.07%	-15.4%	+5.4%
	σ_B		N.A.	-6.6%	-8.9%	+42.7%
T ₄	—	★	+1.2%	-0.8%	-28.3%	-0.6%
T ₅	σ_C	★	+1.1%	-0.1%	-11.1%	-2.61%
	σ_D	★	+4.3%	+0.4%	-42.9%	N.A.
	σ_E		N.P.	+1.4%	-23.1%	+30.8%
	σ_F	★	N.A.	-0.1%	-42.8%	-4.0%
T ₆	—		N.P.	-1.4%	+78.6%	+11.7%

A general overview of the relative errors calculated throughout Table 4.1 shows that the creep deformation prediction of the *afN*-modified FE model cannot be fully validated. Good agreement with experiments is only attained at very low stress levels (including those exploited during the parameter identification procedure, highlighted with a star ★ in Table 4.1). However, creep predictability at mid- to high stress regimes (e.g., test T₁, (T₃, σ_B), (T₅, σ_E & σ_F) is not achieved. In particular, numerical predictions for test T₁, conducted at a very high stress, are completely out of bounds.

A detailed analysis of each of the creep deformation quantities assessed within Table 4.1 is provided hereafter.

- **1st min. creep strain rate:** Prediction of this early creep stage was the primary target justifying the activation function modified viscosity formula. The relative error of our SSH minimum creep strain rate predictions, quantified for all creep tests in Table 4.1, shows good agreement with experiments conducted at very low stress levels. However, accuracy is lost within mid-to-high stress levels. For instance, the non-classical SSH regimes observed in experiments (T₅, σ_E) and T₆ were simply not predicted. This lack of consistency is likely the consequence of an inadequate parameter identification procedure involving a reduced number of tests.
- **2nd min. creep strain rate:** With exception of tests conducted at particularly high stresses (i.e., test T₁ and (T₃, σ_B)), all $\dot{\epsilon}_{ss}$ predictions using our FE model and parameters are found in very good agreement with those measured experimentally during the ULiège SCT campaign. The absolute average $\dot{\epsilon}_{ss}$ prediction error calculated excluding the two tests mentioned here above is 0.5%.

- **time-to-1% creep deformation:** Practical applications of computational creep models are often used to verify if creep deformations exhibited by metallic components under high thermal loadings do not exceed a maximum admissible threshold. In this context, the accuracy of the predicted time-to-1% creep deformation establishes a pragmatic method to evaluate the predictability of a numerical model. The relative error entitled to the time-to-1% creep deformation prediction attained with our *afN* model showcases a non-negligible margin of error compared to experimental observations. Excluding very high stress tests T_1 and (T_3, σ_B) , the absolute average relative error is $\pm 50\%$. With this numerical discrepancy, the model cannot be fully validated in this state.
- **time-to-rupture (t_r):** The relevance of the t_r as a quantity to evaluate the reliability of a numerical creep model lies in the number of aspects required to achieve a good rupture prediction. In other words, a consistently accurate t_r prediction is the result of an equally correct creep deformation and damage accumulation prediction. Altogether, the t_r prediction error achieved with the combined *afN* and Kachanov CDM formulation is found to be in relatively good agreement with experiments. Discarding the very high stress tests $(T_1$ and $(T_2, \sigma_B))$ and test (T_5, σ_D) for not being conducted to rupture, the absolute average error is 13%, which is quite accurate in the context of creep. Experimental creep work usually accepts large curve scattering; creep tests conducted by a single laboratory can exhibit curve scattering within 20-30%, as announced in Górnośląski Instytut Technologiczny (GIT) laboratory (Poland).

The creep deformation results presented here above indicate that the *afN* viscosity function implemented in the Chaboche-Morch UVCN allows the FE model to successfully predict the two-step creep strain rate hardening trend exhibited by Incoloy 800H. However, the relative error analysis of key creep deformation quantities shows predictability issues. In general, creep deformation quantities observed at large deformation levels such as the t_r and $\dot{\epsilon}_{ss}$, and the 1st SSH minimum creep rate, are found to be in good agreement for low-stress experiments. Conversely, creep deformation predictability within low-deformation levels (i.e., the time-to-1% creep deformation) appears to be inconsistent with experimental results. Allowable ($\leq 30\%$) scattering for time-to-1% creep deformation predictions is only achieved in 6 out of 10 tests (namely T_2 , (T_3, σ_A) , (T_3, σ_B) , (T_4, σ_C) & (T_4, σ_E)).

Good experimental agreement in regard to the 1st SSH minimum creep rate prediction combined with inaccurate prediction of the time-to-1% creep deformation seems contradictory at first glance since both quantities belong to the first stages of creep deformation. However, this observation is the key to comprehend the inherent complexity of modeling the non-classical creep behavior of 800H: accuracy at the minimum creep rate plateau is easily achievable, however **accuracy at the prediction of the transition between the 1st and 2nd minimum creep strain rate regimes is considerably more challenging**. The results can likely be improved by increasing the number of tests where two creep strain rate minima are observed, thus acquiring a better understanding of the stress and temperature sensitivity of the phenomenon.

4.5 Conclusions

In this chapter, prediction of the non-classical creep deformation response of Incoloy 800H observed during the creep test campaign conducted at ULiège was achieved via finite element (FE) simulations performed in the in-house developed FE software Lagamine. To this end, an activation-function Norton (*afN*) viscosity function was developed within a new VMVP (von-Mises viscoplastic) law, an extension of the Chaboche-Morch unified viscoplastic constitutive model (UVCN). The implementation followed a semi-implicit approach, similar to that

presented in C. Rojas-Ulloa et al.[10]. The fitting parameters entitled to the *afN* viscosity function and the Kachanov Continuum Damage Mechanics (CDM) approach were found following a combined analytical and inverse modeling approach. The numerical results yielded the following observations:

- The consistently accurate prediction of the $\dot{\epsilon}_{ss}$ across the 10 creep tests addressed during the experimental benchmark presented in section 4.2 validates the Norton law formulation (inherited from H. Morch, see [19]) used in this work. Please note that the reliability of this law is for this material entitled to the correct identification of the $\dot{\epsilon}_{ss}$ regime across the creep tests chosen to partake in the parameter identification procedure. In other words, differentiating both the 1st and 2nd minimum creep strain rate regimes is paramount.
- The reliability of the activation function component of the *afN* viscosity function at predicting the non-classical creep deformation response of Incoloy 800H across the 10 creep tests addressed throughout this chapter is limited. The prediction of the 1st minimum creep strain rate at low and mid-stress levels is found in good agreement with experiments; however, the prediction of the transient behavior towards the 2nd $\dot{\epsilon}_{ss}$ minimum creep strain rate is not consistently achieved. This yields a weak time-deformation relationship predictability, especially at low deformation levels.

Altogether, both conclusions presented here above show that the *afN* solution to the non-classical creep deformation behavior is a promising approach. Full validation using the experimental curves gathered during the ULiège experimental campaign was however not achieved. The prediction inconsistencies observed in the SSH creep hardening regime indicate that the activation function parameter identification must be improved following a more comprehensive approach, involving for instance a greater number of creep tests. The latter justifies the development of the micromechanical mean-field creep model (see chapter 3), and ultimately the multi-scale creep modeling approach presented hereafter in chapter 5.

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Multi-scale creep modeling approach: MSCreep

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The predictability issues observed for VMVP chapter 4 were mainly attributed to the limited experimental information used to parametrize and identify the COLAW. This chapter deals with this situation by developing a multi-scale creep modeling methodology where the experimental creep curves used to identify VMVP are complemented with virtual creep curves obtained from MFC (see chapter 3) simulations. The motivation, framework and methodology of the multi-scale modeling approach are provided in section 5.1. Section 5.2 presents the detailed sequence of activities associated with the application and validation of the multi-scale creep approach in the context of Incoloy 800H. The improved low-stress and transitional (SSH-to- $\dot{\epsilon}_{ss}$) parametrization in the final VMVP-multi-scale creep (MSC) identification was adopted from the combined virtual + experimental creep tests. These attained to a comprehensive and physically-consistent creep behavioral mapping of the material. A final creep experiment consisting of 6 cantilever beams loaded under high temperature were used to validate the inherent stress gradient sensitivity of the FE viscoplastic model, and to validate the VMVP-MSCreep parametrization. Finally, section 5.3 presents the the conclusions.

5.1 Multi-scale creep: a modeling methodology

The MSCreep modeling approach is not a single model but a methodology where the micromechanical Mean Field Creep (MFC) model (presented in chapter 3) is used to improve the predictability of VMVP (von-Mises viscoplastic) macromechanical constitutive model (see chapter 4).

5.1.1 Motivation

The end goal of this PhD. was the reliable and accurate prediction of the creep deformation response of Incoloy 800H using a UVCN. To this end:

- a thorough SCT experimental campaign was planned. The stress and temperature of each test were defined according to the range of interest, aiming to capture the non-classical creep deformation response exhibited by the alloy.
- a special viscosity function, consisting of an activation function-modified Norton law (*afN*), was implemented within the VMVP COLAW in LAGAMINE finite element (FE) code. The function, explained throughout chapter 4 and presented here again in Eq.(5.1a), allows for the successful prediction of the anomalous creep response exhibited by 800H during low-stress high-temperature creep loadings with remarkable numerical stability.

$$\dot{p} = \exp \left[\overbrace{-a_N (1.00 + p - 0.001 \cdot b_N)^{-c_N}}^{\text{activation function}} \right] \cdot \overbrace{\left\langle \frac{\sigma_v}{K} \right\rangle^n}^{\text{Norton}} \quad (5.1a)$$

$$\langle x \rangle = \begin{cases} x & \text{if } x > 0 \\ 0 & \text{otherwise} \end{cases} \quad (5.1b)$$

$$\sigma_v = \sigma_a - \sigma_y \quad (5.1c)$$

$$(5.1d)$$

The parameter identification procedure described in chapter 4 consisted of a consecutive three-step approach: **step 1** deals with the Norton parameters (K, n), **step 2** deals with the activation function parameters (a_N, b_N, c_N), and **step 3** deals with the creep damage computation. Both **step 1** & **2** have a sub-step hierarchical progression. Defined hereafter alphabetically as a & b (so that 1.a $\rightarrow$ 1.b $\rightarrow$ 2.a $\rightarrow$ 2.b), these entail:

- $\rightarrow$ **sub-step a:** identification of individual parameters for each creep curve. In other words, step 1.a aims to identify $\{K_i, n\}$, whereas step 2.a addresses $\{a_{N,i}, b_{N,i} \& c_{N,i}\}$ for each test i .
- $\rightarrow$ **sub-step b:** Parametrization. Finding suitable phenomenological curves that capture the stress and/or temperature dependency of the parameters previously identified for each creep test i . Note that the phenomenological function is not explicitly established in this generalized definition. This is intentional, given the uncertain stress and temperature dependency that materials exhibiting similar non-classical creep responses may exhibit.

The step and sub-step parameter identification sequence (1.a $\rightarrow$ 1.b $\rightarrow$ 2.a $\rightarrow$ 2.b) was conducted in chapter 4 for Incoloy 800H using exclusively experimental results obtained in ULiège. The particular sub-equations fitted to recover the stress and temperature dependency of parameters a_N and b_N and the temperature dependency of the drag stress K are presented in Eq.(4.7). Additionally, parameters c_N and n were found to be constant, independent of stress and temperature.

The model predictability was finally evaluated for all creep tests conducted in ULiège in section 4.4. The analysis of results in section 4.4 shows that the creep deformation predicted within ranges of interest for industrial applications (i.e., time-to-1% creep) was not consistent with experiments. This loss of accuracy takes place between the first (SSH) and second ($\dot{\epsilon}_{ss}$) minimum creep strain rate peaks. Chapter 4 concludes by attributing this issue to a wrongful parametrization of functions $a_N(\sigma, T)$ and $b_N(\sigma, T)$, given the limited number of creep tests available to describe the stress and temperature dependency of these parameters. This situation is better illustrated here in Figure 5.1. The lack of information within the zones highlighted in yellow substantially affects the inter- and extrapolation accuracy of the model. To improve the accuracy of our finite element model, more experimental curves were required in order to fill the gaps represented by yellow areas in Figure 5.1. **This is the role of our micromechanical MFC model.**

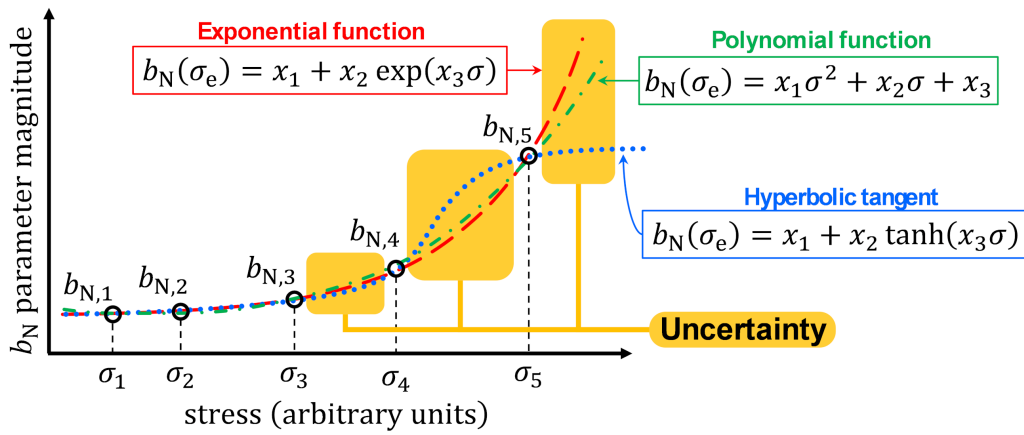


Figure 5.1: Engineering stress (σ_e) dependency of a_fN parameter b_N based on the multiple individual magnitudes $b_{N,i}$ for a number of creep tests at a single temperature and multiple stresses (σ_i). The illustration showcases the multiple phenomenological curves suitable to fit the behavior $b_N(\sigma_e)$. Note that the uniqueness of the exponential curve chosen in chapter 4 cannot be ascertained.

5.1.2 Methodology

The MSCreep modeling methodology is defined as a two-level procedure:

Level 1: Production of virtual tests

It consists of the exploitation of the MFC model to produce creep curves in complement to those obtained during the experimental campaign. The creep curves produced using the MFC model are hereafter referred to as virtual tests (VTs).

Level 2: Identification of parameters in VMVP

The new creep curve dataset, consisting of experimental and VTs, is used to identify the a_fN VMVP parameters. To this end, the same step and sub-step procedure (1.a $\rightarrow$ 1.b $\rightarrow$ 2.a $\rightarrow$ 2.b) applies. This combined experimental-virtual dataset is expected to provide a

much clearer description of the stress and temperature dependency of parameters, filling all information gaps represented by yellow zones in Figure 5.1. The researcher is free to fit new phenomenological curves during sub-step b, or to assign different weight factors to experimental and virtual curves.

5.2 Application to Incoloy 800H

Hereafter, the two-level MSCreep methodology described above in section 5.1.2 is applied to our Incoloy 800H alloy to improve the FE simulation results compared to those presented in chapter 4 where no VTs were employed. The FE COLAW identified in chapter 4 on ULiège experimental creep tests is hereafter named "VMVP - chapter 4", whereas the new VMVP model adapted to the MSCreep methodology is referred to as "VMVP - MSCreep". Additionally, to ensure consistency throughout this thesis, creep curves obtained with the old (chapter 4) model will be plotted in **blue**, whereas curves resulting from the VMVP-MSCreep approach will appear in **orange**.

Section 5.2.1 addresses **Level 1** of the MSCreep modeling methodology; a VT battery is organized in consideration of the experimental tests available in ULiège. Section 5.2.2 focuses on the second level of the MSCreep approach, dealing with the parametrization and identification of the afN viscosity function. The FE simulation results obtained using the newly identified afN are presented in section 5.2.3. Finally, a brief discussion on the predictability and perspectives of the model is provided in section 5.2.4.

5.2.1 Virtual tests battery

The stress and temperature loadings addressed within the battery of VTs were defined with two goals in mind: to complement the experimental curves already obtained in ULiège, and to acquire physically-accurate information within creep loadings where conventional SCTs would take years to reach rupture (i.e., very low stresses). The stresses and temperatures addressed in this context are hereafter referred to as σ_i and T_j , with $\sigma_1 < \sigma_2 < \dots < \sigma_{14}$ and $T_1 < T_2 < \dots < T_8$. The different loading combinations (σ_i, T_j) and the origin of the creep curve for such loading (i.e., virtual or experimental) are described in Table 5.1.

All Experimental and Virtual creep tests, represented as "**E**" or "**V**" in Table 5.1, are hereafter reference tests. A total of 87 reference creep curves were assessed in the context of the implementation of the MSCreep approach: 8 experimental curves complemented with 79 VTs. Note that 2 of our 10 creep experimental creep curves were dropped from this set as they deal with large stresses that are out of the scope for industrial relevance.

5.2.2 afN parameter identification

Level 2 of this MSCreep methodology addresses the identification of VMVP parameters. The following sections describe the parametrization and parameter identification procedures conducted on the afN -modified VMVP law. Following the hierarchical 3-step process previously introduced in section 5.1.1, steps 1 (Norton), 2 (activation function) and 3 (damage) are hereafter described in sections 5.2.2, 5.2.2 and 5.2.2, respectively.

Step 1: Norton law

Sub-step 1.a: independent identification

The first aspect differentiating this sub-step identification procedure from that described in chapter 4 is the much larger dataset used. To discover the new relationships stemming from our

Table 5.1: Stress-temperature distribution of Virtual and Experimental creep tests defining the dataset. Note that italic temperature (T) is used hereafter in chapter 5 to differentiate it from that (T) used throughout chapter 4.

V: virtual test ("V" used instead of VT due to space restrictions)

E: Experimental creep test

⊗: (σ , T) range not considered in our VTs battery, either because of an excessively long t_r (e.g., low- σ & low- T , with $t_r > 250$ h), or because the (σ , T) combination was found outside the range of applicability of MFC.

	σ_1	σ_2	σ_3	σ_4	σ_5	σ_6	σ_7	σ_8	σ_9	σ_{10}	σ_{11}	σ_{12}	σ_{13}	σ_{14}
T_1	⊗	⊗	⊗	⊗	⊗	⊗	⊗	V	V	V	V	V	V	V
T_2	⊗	⊗	⊗	⊗	⊗	V	V	V	V	V	V	V	V	V
T_3	⊗	⊗	⊗	V	V	V	V	V	V	V	V	V	V	V
T_4	⊗	V	V	V	V	V	V	V	V	V	V	V	V	E
T_5	V	V	V	V	V	V	V	V	V	V	V	V	E	V
T_6	V	V	V	V	V	V	V	V	V	V	E	V	V	⊗
T_7	V	V	V	V	V	E	V	E	V	V	E	⊗	E	⊗
T_8	V	V	V	V	V	V	V	V	V	E	⊗	⊗	⊗	⊗

reference curves, the steady-state creep strain rate ($\dot{\epsilon}_{ss}$) was identified for each curve. Each $\dot{\epsilon}_{ss}$ was then tabulated to as a stress-temperature layout similar to Table 5.1. The phenomenological stress-temperature- $\dot{\epsilon}_{ss}$ relationship defined by the Norton viscosity function (see Eq.(5.2)) was subsequently verified by plotting the logarithmic stress- $\dot{\epsilon}_{ss}$ relationship, as shown in Figure 5.2. The Norton power-law breakdown toward low stresses indicates a possible creep mechanism change.

$$\dot{\epsilon}_{ss} = \left\langle \frac{\sigma_{ov}}{K} \right\rangle^n \quad \Leftrightarrow \quad \log(\dot{\epsilon}_{ss}) = n[\log|\sigma_{ov}| - \log(K)] \quad (5.2)$$

The existence of a different creep mechanism acting at very low stresses is illustrated for T_7 in Figure 5.2, where the conventional linear Norton relationship diverges from the reference data points at stresses lower than σ_5 . To introduce this apparent creep mechanism change behavior into the conventional Norton power law, a stress-dependency on the exponent n is required. Contrary to conventional creep mechanism changes, however, the behavior predicted by the low-stress VTs corresponds to a soft saturation behavior (i.e., a smooth transition).

This observation was implemented in our analytical Norton parameter identification procedure (**sub-step 1.a**) by introducing a new set of instructions. Our analytical optimization algorithm was targeted at minimizing the RMSPE between the reference and calculated $\dot{\epsilon}_{ss}$ for each reference test (σ_i , T_i) by modifying both Norton parameters (K and n) instead of only K . In particular, the exponent n_i was allowed to change with the stress (contrary to the process described in section 4.2.3, where it was held constant), whereas the drag stress K_i was allowed to change with temperature.

Sub-step 1.b: parametrization & parameter identification

The engineering stress and temperature dependency identified during the parametrization of all the individual stress-dependent n_i and temperature-dependent K_i magnitudes was achieved by analytical interpolation of phenomenological curves via linear regression. The target of this first "raw" identification of parameters was the RMSPE minimization between the analytically calculated value (e.g., $K(T_i)$) and their corresponding magnitude (e.g., K_i). In particular,

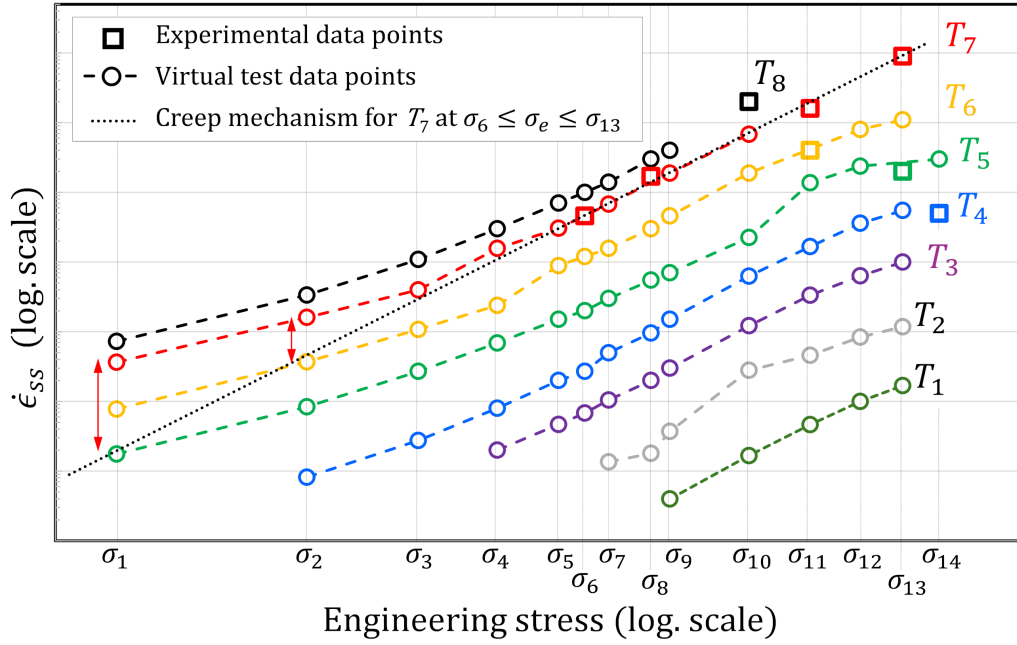


Figure 5.2: Normalized logarithmic stress- $\dot{\epsilon}_{ss}$ relationship identified from Incoloy 800H reference creep curves, addressing both virtual (MFC) and experimental tests.

the stress dependency of the Norton exponent $n(\sigma_e)$ was recovered with a hyperbolic tangent function, whereas the drag stress dependency on temperature $K(T)$ was found to follow an Arrhenius shape. The curves are shown in Eq.(5.3a) and Eq.(5.3b) respectively.

$$n(\sigma_e) = (n_1 - n_2) + n_2 \cdot \tanh(\sigma_e \cdot n_3) \quad (5.3a)$$

$$K(T) = A_K \cdot \left[1 - B_K \cdot \exp(T/C_K) \right] \quad (5.3b)$$

A second and final optimization of the Norton parameters stemming from the curve selection (i.e., n_1 , n_2 , n_3 , A_K , B_K & C_K) was subsequently performed. The target of this final optimization was the minimization of the RMSPE between the reference $\dot{\epsilon}_{ss}$ and that calculated by solving the Norton equation from Eq.(5.2) using the calculated functions $K(T)$ & $n(\sigma_e)$ for each reference curve (σ_i, T_i) via linear regression. The final results of this parameter optimization procedure are shown for the drag stress and Norton exponent in Figure 5.3a and Figure 5.3b respectively.

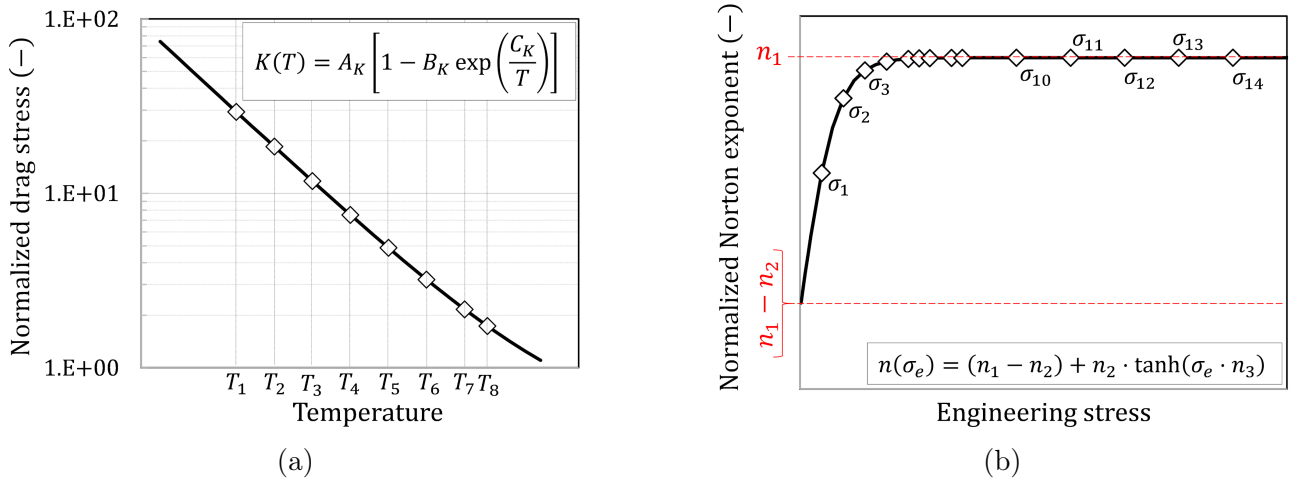


Figure 5.3: Analytical curve fitting recovering (a) the temperature-dependency of the drag stress $K(T)$ and (b) the stress-dependency of the Norton exponent $n(\sigma_e)$.

Step 2: activation function

Sub-step 2.a: independent identification

The identification of individual activation function parameters (a_N , b_N and c_N , see Eq.(5.1a)) was conducted by means of an inverse modeling approach involving our in-house optimization software OPTIM [1]. This optimization software based on the Levenberg-Marquardt algorithm was instructed to minimize the RMSPE between the reference and the FE-predicted creep curve for a given reference creep curve loading (σ_i , T_i) with a precision of 5×10^{-3} . To improve the computational efficiency and the overall accuracy of the process, only creep strain data points pertaining to the SSH regime were considered. The optimized "creep strain vs. time" and "creep strain rate vs. time" curves for the reference loading case (σ_{11} , T_6) are shown in Figure 5.4a and Figure 5.4b respectively. Note that the predicted LAGAMINE curve in Figure 5.4 does not consider damage accumulation, thus missing the final creep softening behavior. Full curves corresponding to the equivalent MFC VT simulation and experimental (ULiège) curve are included to emphasize the consistency of our model.

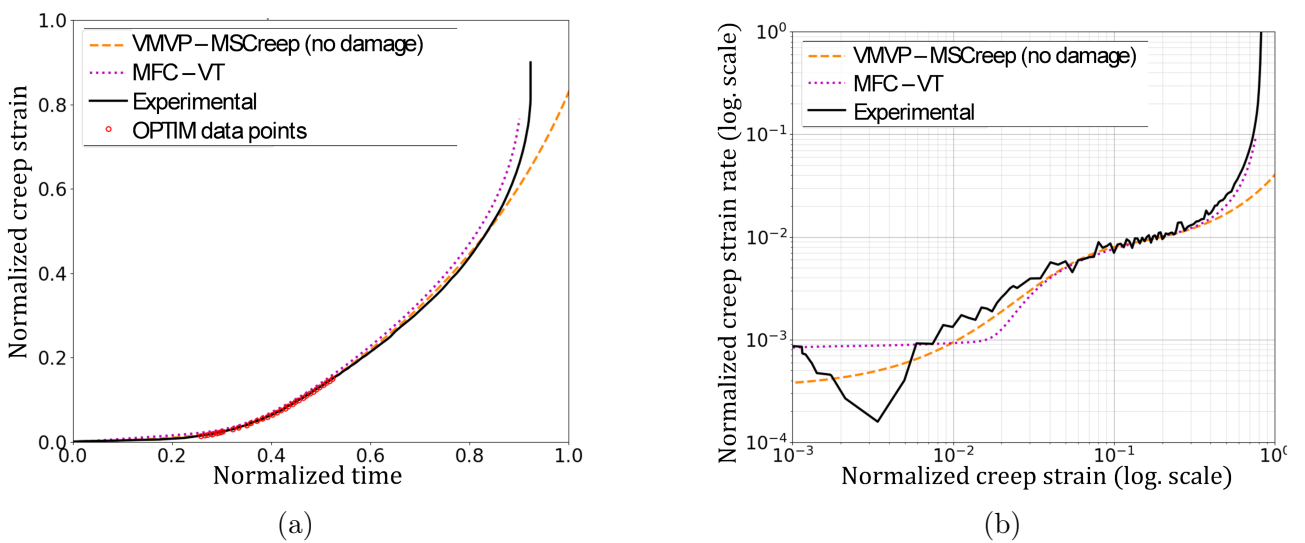


Figure 5.4: (a) Creep strain and (b) creep strain rate curves computed by the FE code (LAGAMINE) for the reference loading combination σ_{11}, T_6 with a set of afN parameters ($a_{N,i}$, $b_{N,i}$ & $c_{N,i}$) optimized by OPTIM. The target points loaded into OPTIM as the objective for LAGAMINE curve fitting are shown in red in (a).

Sub-step 2.b: activation function parametrization

The results acquired during **sub-step 2.a** indicate that parameters a_N and c_N are constant (i.e., independent from either stress or temperature), whereas parameter b_N exhibited strong stress and temperature dependency. **sub-step 2.b** consisted of the analytical parametrization of b_N as $f(\sigma_e, T)$. To this end, the RMSPE of points $b_{N,i}$ was minimized for all σ_i, T_i via linear regression. The stress dependency of b_N was found to follow the hyperbolic tangent function shown in Eq.(5.4a), where b_1 and b_2 are fitting parameters, and b_3 is a temperature function. The form of the function $b_3(T)$ is defined in Eq.(5.4b), where b_{31}, b_{32} and b_{33} are fitting parameters.

$$b_N(\sigma_e, T) = b_1 \cdot \tanh \left\{ b_2 \cdot [\sigma_e - b_3(T)] \right\} \quad (5.4a)$$

$$b_3(T) = \left| b_{31} \cdot \left[1 - b_{32} \cdot \exp(T/b_{33}) \right] \right| \quad (5.4b)$$

The function $b_N(\sigma_e, T)$ fitted on the original $b_{N,i}$ data points identified in **sub-step 2.b** is shown in Figure 5.5.

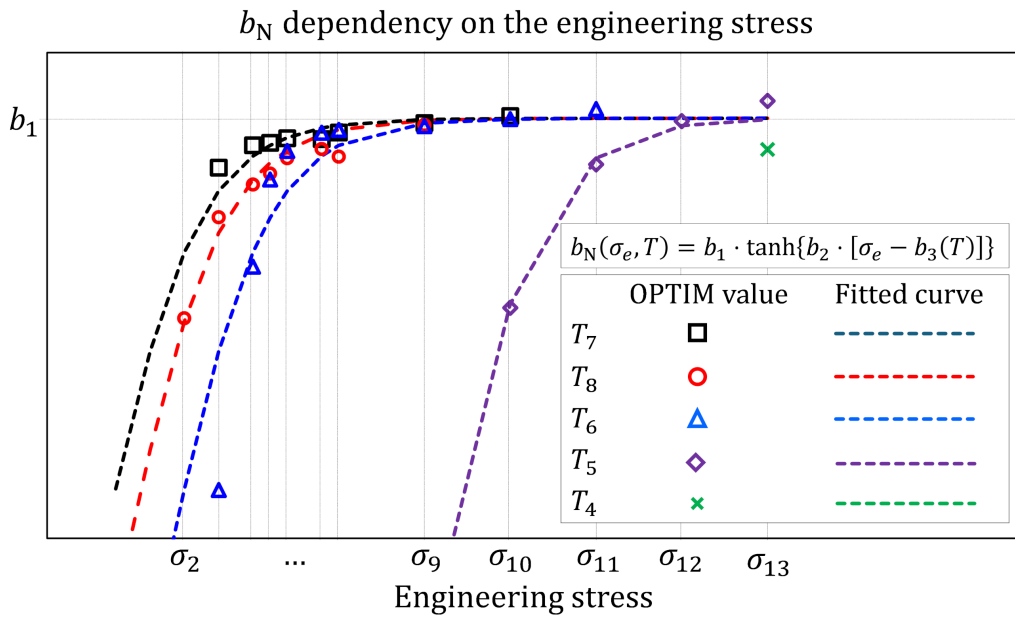


Figure 5.5: Stress dependency of b_N recovered by a hyperbolic tangent function $b_N(\sigma_e, T)$. Note that the sensitivity of the creep deformation response of the material to the parameter b_N is only attained for b_N values found above the vertical axis= 0 values in this plot.

The temperature-dependency of the function $b_3(T)$ described in Eq.(5.4b) as the absolute value of an Arrhenius expression is shown in Figure 5.6. Note that the absolute value prevents the expression from reaching negative values (i.e., $b_3(T) \geq 0$). Additionally, since $b_3(T)$ is subtracted from σ_e in Eq.(5.4a) (i.e., it is not a multiplying factor), the very low positive values reached within the inflection point at $T_6 < T < T_7$ do not affect the numerical convergence of the model.

Step 3: creep damage

The VMVP creep damage approach introduced previously in chapter 4 was not modified in the context of this MSCreep methodology. The damage formulations (namely the damage

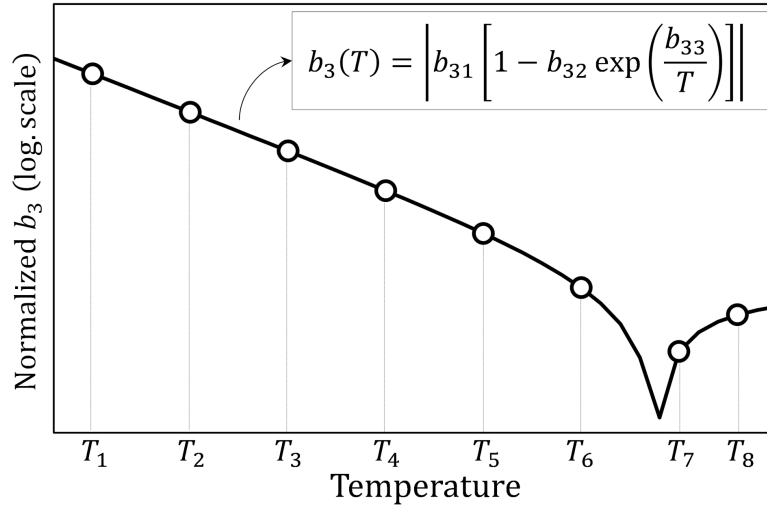


Figure 5.6: Temperature dependency of function $b_3(T)$.

initiation criterion, the Kachanov CDM approach, and the creep rupture criterion) and their parameters were directly inherited from section 4.2.3. The consistency of the creep damage accumulation and rupture predictions compared to ULiège experimental results ascertained in section 4.4 was confirmed to extend into this new MSCreep approach, as seen in the creep strain and creep strain rate plots of Figure 5.7a and Figure 5.7b respectively. Note that the newly identified afN , in addition to damage (initiation, accumulation & fracture), allows to recover the experimental results with remarkable accuracy.

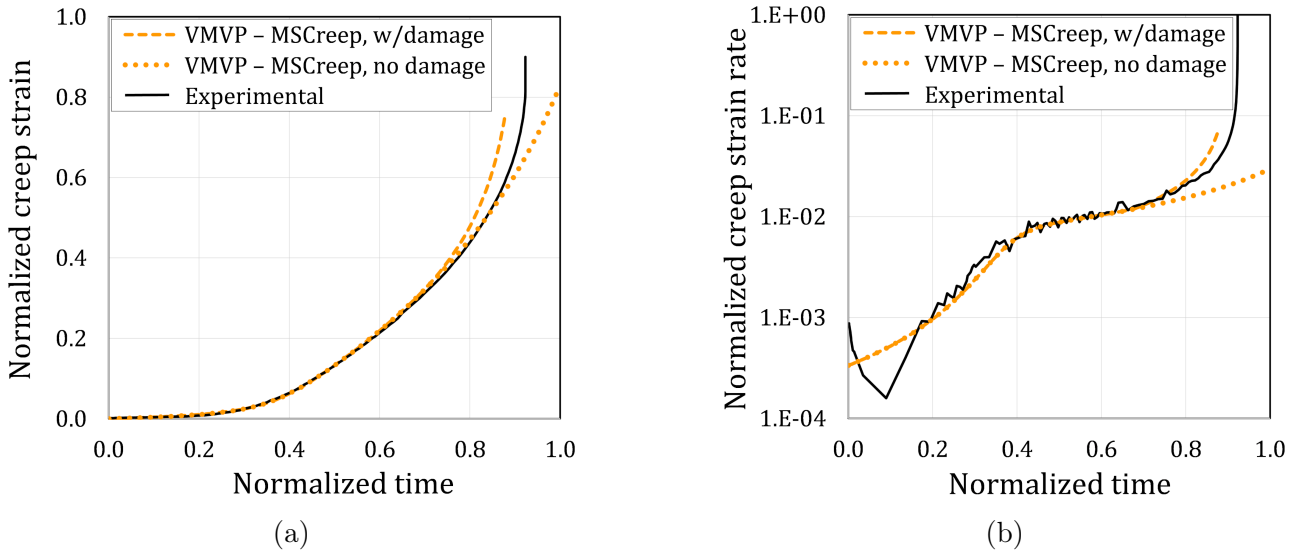


Figure 5.7: (a) Creep strain vs. time and (b) creep strain rate vs. time curves computed in LAGAMINE using the new VMVP-MSCreep modeling approach with and without damage.

Summary of creep fitting parameters

The VMVP formulation of chapter 4 (identified exclusively on ULiège experimental results) and the MSCreep approach VMVP are similar: both share the afN viscosity function and Kachanov creep damage formulations. The differences stem from the new set of stress and temperature dependent equations governing the evolution of the creep strain rate in afN . To further clarify the new VMVP functions found throughout this section, a list of all fitting parameters is provided in Table 5.2.

Table 5.2: Summary of parameters of the VMVP-MSCreep constitutive model identified by a combined experimental and virtual creep test campaign.

Parametric functions	Parameters	SI unit
Activation function		
$\exp \left\{ -a_N [1.05 + p - 0.001 \underbrace{b_N(\sigma_e, T)}]^{c_N} \right\}$	a_N	—
	c_N	—
$b_N(\sigma_e, T) = b_1 \tanh \left\{ b_2 [\sigma_e - \underbrace{b_3(T)}] \right\}$	b_1	—
	b_2	—
	b_{31}	MPa
$b_3(T) = \left b_{31} [1 - b_{32} \exp(b_{33}/T)] \right $	b_{32}	—
	b_{33}	°C
Norton law: $\underbrace{\left\langle \frac{\sigma_{ov}}{K(T)} \right\rangle^{n(\sigma_e)}}$		
	A_K	MPa
$K(T) = A_K \left[1 - B_K \exp \left(\frac{C_K}{T} \right) \right]$	B_K	—
	C_K	°C
	n_1	—
$n(\sigma_e) = (n_2 - n_1) + n_2 \tanh (\sigma_3 \cdot n_3)$	n_2	—
	n_3	MPa
Critical creep strain for damage initiation:	p_1^c	—
$p^{\text{critical}}(T) = p_1^c \left[1 - p_2^c \exp \left(\frac{T}{p_3^c} \right) \right]$	p_2^c	—
	p_3^c	°C
Kachanov creep damage accumulation:	k_3	—
$\Delta D_c^{n+1} = k_3 \underbrace{\left(\frac{k_4 \tilde{\sigma}^{n+1}}{S_c(T)} \right)^{S_{ce}}}_{\frac{1}{(1-D^n)^k}}$	k_4	—
	S_{ce}	—
	k	—
	s_1	—
$S_c(T) = s_1 \cdot [1 - s_2 \cdot \exp(T/s_3)]$	s_2	—
	s_3	°C

In summary, the *afN* viscosity function carries 13 fitting parameters: 7 of them are entitled to the activation function parametrization (a_N , b_1 , b_2 , b_{31} , b_{32} , b_{33} & c_N), and 6 belong to the Norton law formulation (A_K , B_K , C_K , n_1 , n_2 & n_3). In addition to the 3 parameters entitled to the damage initiation formulation (p_1^c , p_2^c , & p_3^c) and the 7 Kachanov creep damage parameters (k_3 , k_4 , S_{ce} , k , s_1 , s_2 & s_3), the number of creep fitting parameters for this new MSCreep VMVP approach ascends to **23**.

5.2.3 Results & validation

The creep curves provided in this subsection compare the numerical results obtained with our new VMVP-MSCreep approach to the corresponding reference (i.e., E or VT) creep curves, and to our previous VMVP-experimental iteration (where VMVP parametrization was performed exclusively on experimental tests, see chapter 4).

Experimental benchmark

Creep curves computed by LAGAMINE FE simulations of uniaxial creep tests conducted in ULiège (see "E" in Table 5.1) are hereafter presented for 5 temperatures: $T_4 < T_5 < \dots < T_8$. Note that these are equivalent to the temperatures ($T_2 < T_3 < \dots < T_6$) addressed in the experimental campaign in Chapter 2 section 2.2.2, the MFC experimental benchmark in Chapter 3, and the experimental parametrization of VMVP in Chapter 4 section 4.3. The creep strain and creep strain rate vs. time curves are provided in a normalized manner. VMVP-MSCreep FE simulations of uniaxial creep tests were obtained using a *afN* function parametrized and fitted on a combined experimental+virtual (MFC) tests (see Table 5.1). The *afN* function from the VMVP-chapter 4 curves was parametrized and fitted exclusively on ULiège creep experimental curves. Note that T_4 is equivalent to T_2 in section 4.3.

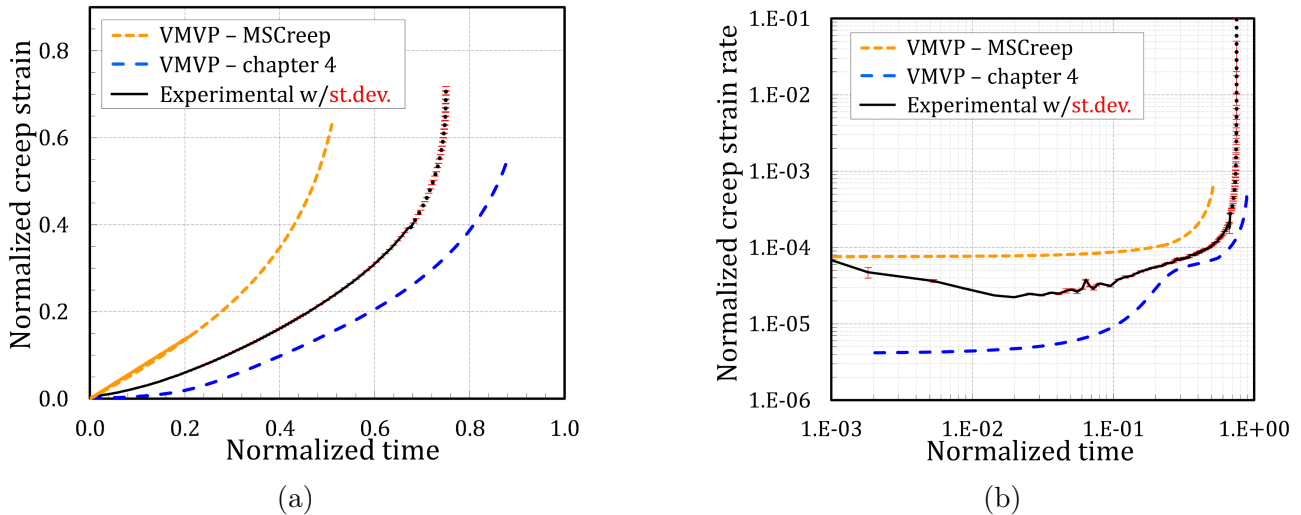


Figure 5.8: Experimental and FE numerical (a) creep strain vs. time and (b) creep strain rate vs. time curves for Incoloy 800H at temperature T_4 and stress σ_{14} .

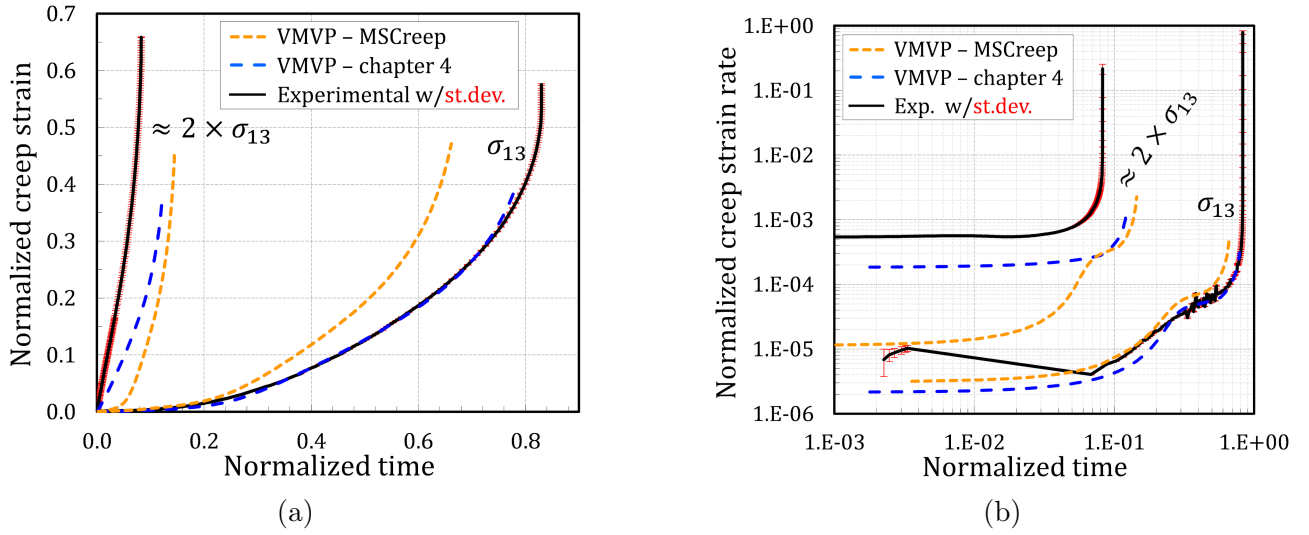


Figure 5.9: Experimental and FE numerical (a) creep strain vs. time and (b) creep strain rate vs. time curves for Incoloy 800H at temperature T_5 and stress σ_{13} & $\approx 2 \times \sigma_{13}$.

T_5

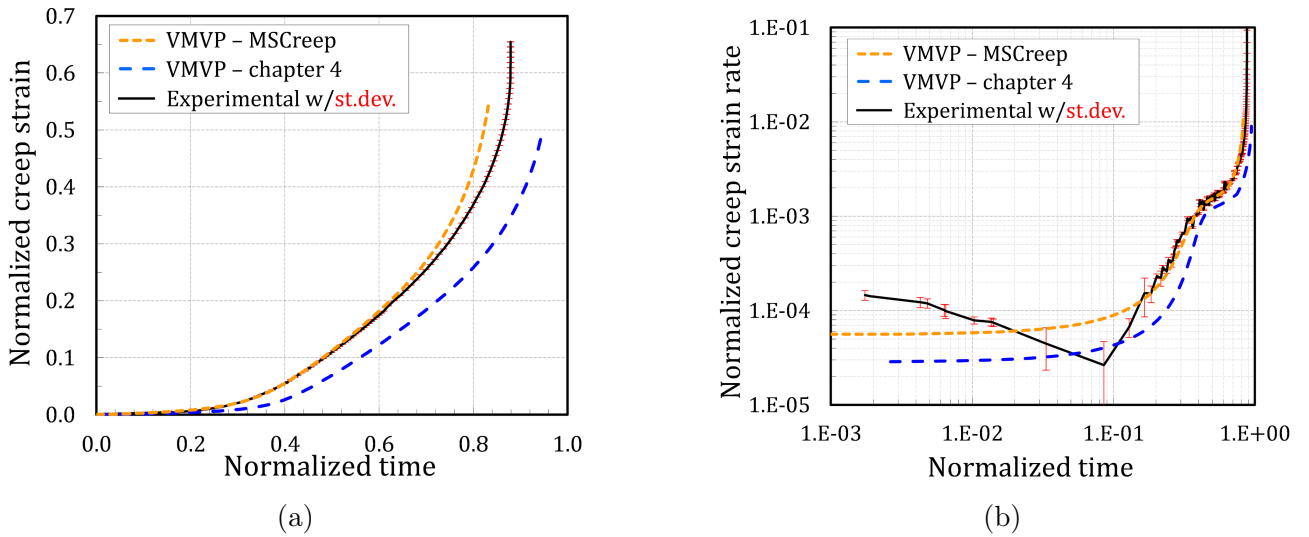


Figure 5.10: Experimental and FE numerical (a) creep strain vs. time and (b) creep strain rate vs. time curves for Incoloy 800H at temperature T_6 and stress σ_{11} .

T_6

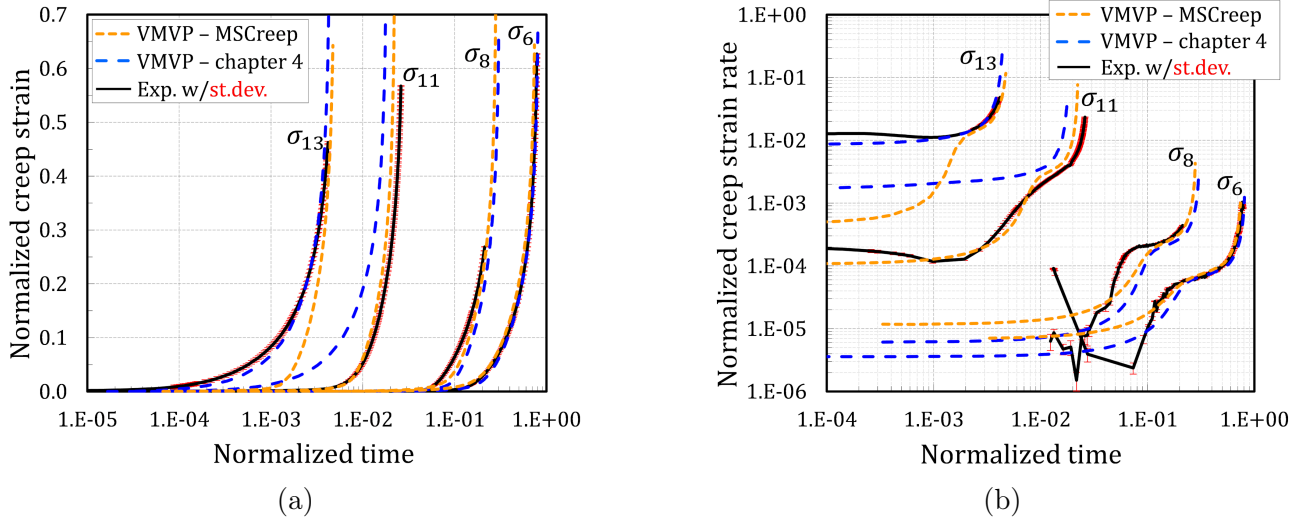


Figure 5.11: Experimental and FE numerical (a) creep strain vs. time and (b) creep strain rate vs. time curves for Incoloy 800H at temperature T_7 and stresses σ_6 , σ_8 , σ_{11} & σ_{13} .

T_7

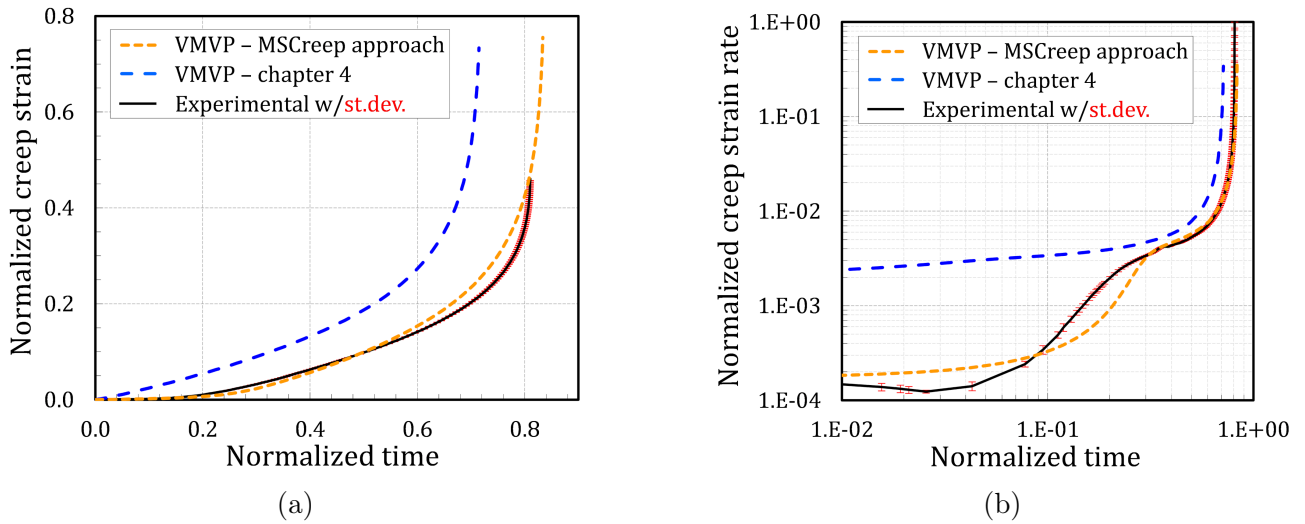


Figure 5.12: Experimental and FE numerical (a) creep strain vs. time and (b) creep strain rate vs. time curves for Incoloy 800H at temperature T_8 and stress σ_{10} .

T_8 The experimental benchmark shown from Figure 5.8 to Figure 5.12 shows that our VMVP-MSCreep model exhibits a more consistently accurate prediction of the creep strain and creep strain rate deformation response of 800H compared to VMVP-chapter 4, despite the latter being identified on experimental curves. Nevertheless, a high-stress discrepancy to the experimental results is also observed: for (σ_{14}, T_4) in Figure 5.8, for $\{(\sigma_{13}, T_5) \text{ \& } (\approx 2 \times \sigma_{13}, T_5)\}$ in Figure 5.9, and for (σ_{13}, T_7) in Figure 5.11. Altogether, these results indicate that the applicability range of our VMVP model identified following the new MSCreep approach is limited to high-temperature and low-mid stress ranges.

VMVP-MSCreep model applicability range:

The stress-temperature loadings of the experimental and VT curves chosen to take part in the MSCreep approach (see Table 5.1) were initially limited to σ_{14} in consideration of the applicability range defined for our MFC model (see chapter 3). Nevertheless, the results within

stresses close to σ_{14} show a non-negligible divergence from the experiments, resulting in a more restrictive applicability range compared to that of MFC. The final applicability range of our VMVP-MSCreep, defined within the same stress-temperature table layout of Table 5.1, is shown in Figure 5.13.

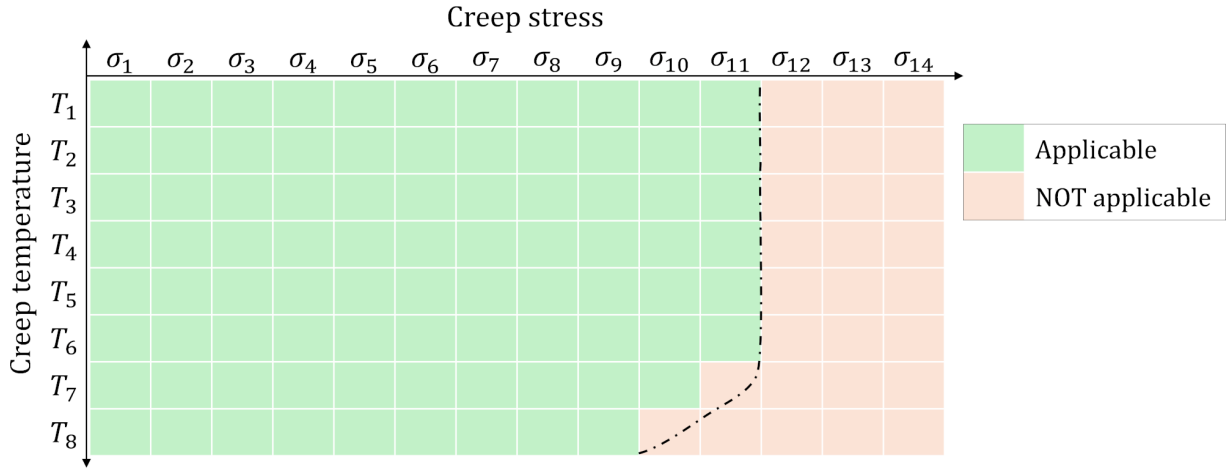


Figure 5.13: Graphical description of the stress-temperature creep loadings applicability range for the VMVP model identified under the MSCreep approach.

The numerical analyses of the VMVP-MSCreep model presented hereafter are conducted exclusively on (σ, T) creep loadings found within the **green** applicability zone shown in Figure 5.13

Low stress benchmark outside feasible experiments

The previous experimental benchmark provides a good depiction of the interpolation capabilities of our MSCreep modeling approach. Numerical improvements compared to the previous VMVP iteration in chapter 4 are mainly attributed to the exploitation of MFC VTs to fill the creep loading gaps in-between experimental tests (see Figure 5.1).

However, the greatest advantage of the MSCreep consists of the exploitation of MFC VTs to obtain physically-accurate creep curves within loading (σ_i, T_i) where conventional creep tests would take years to complete. This "low stress benchmark" presents the creep modeling improvements attained for low stresses (σ_1, σ_3 & σ_5) across 3 high temperatures: T_5, T_7 and T_8 . The VMVP-MSCreep FE results are compared to VMVP-chapter 4 results and to their respective MFC VTs curves. VMVP-MSCreep FE simulations of uniaxial creep tests were obtained using a *afN* function parametrized and fitted on a combined experimental+virtual (MFC) tests (see Table 5.1). However, the *afN* function from the VMVP-chapter 4 curves was parametrized and fitted exclusively on ULiège creep experimental curves.

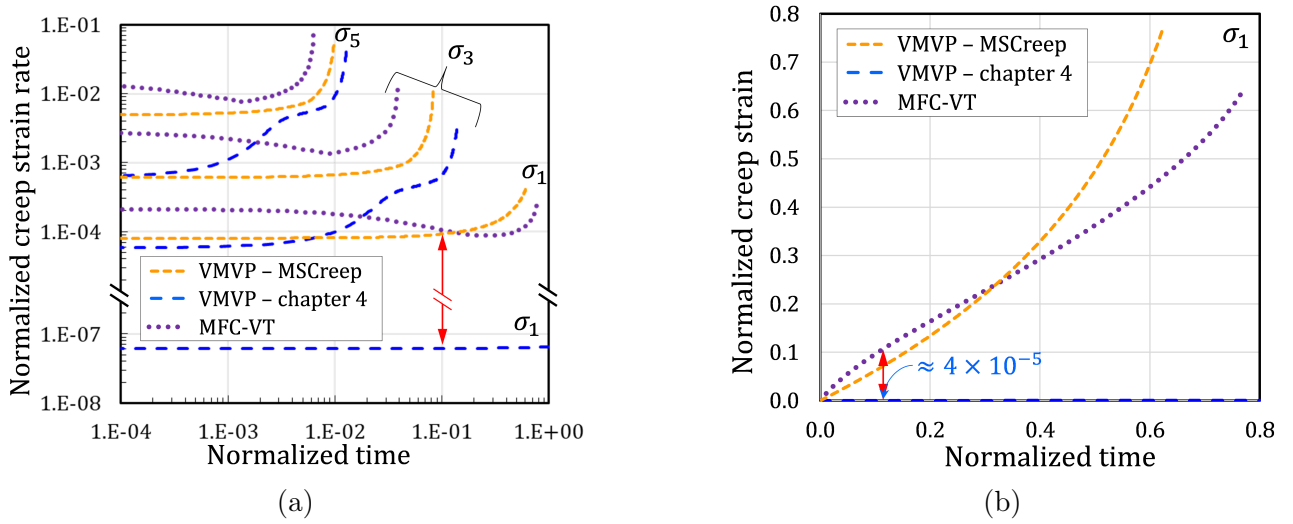


Figure 5.14: Virtual (MFC) and numerical (FE) (a) creep strain vs. time and (b) creep strain rate vs. time curves for Incoloy 800H at temperature T_5 and very low stresses $\sigma_1 < \sigma_3 < \sigma_5$. T_5 is equivalent to T_3 in section 4.3.

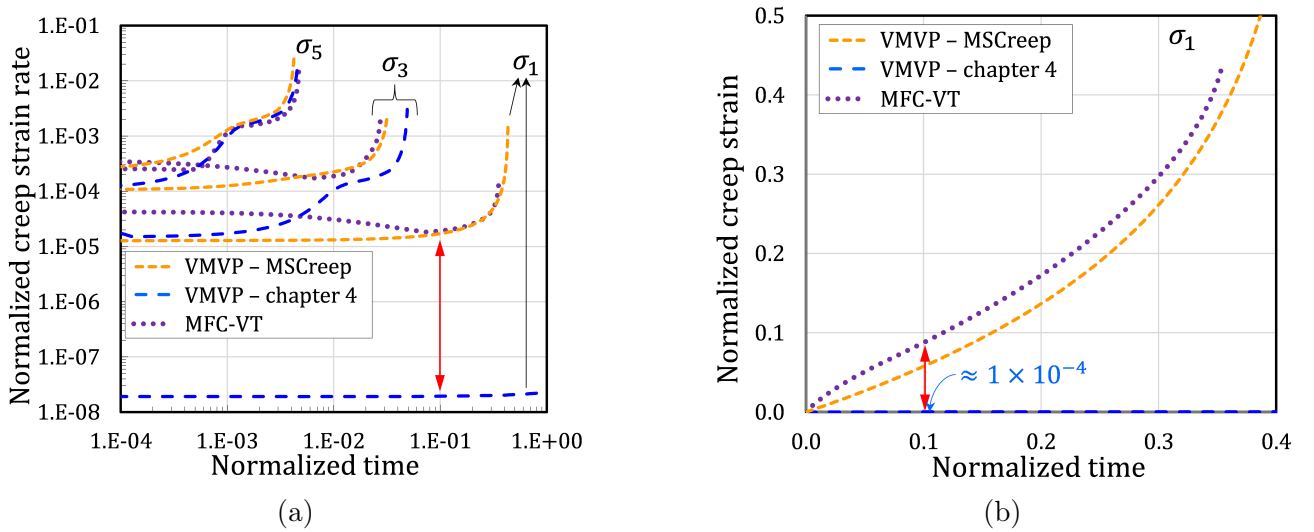


Figure 5.15: Virtual (MFC) and numerical (FE) (a) creep strain vs. time and (b) creep strain rate vs. time curves for Incoloy 800H at temperature T_7 and very low stresses $\sigma_1 < \sigma_3 < \sigma_5$. T_7 is equivalent to T_5 in section 4.3.

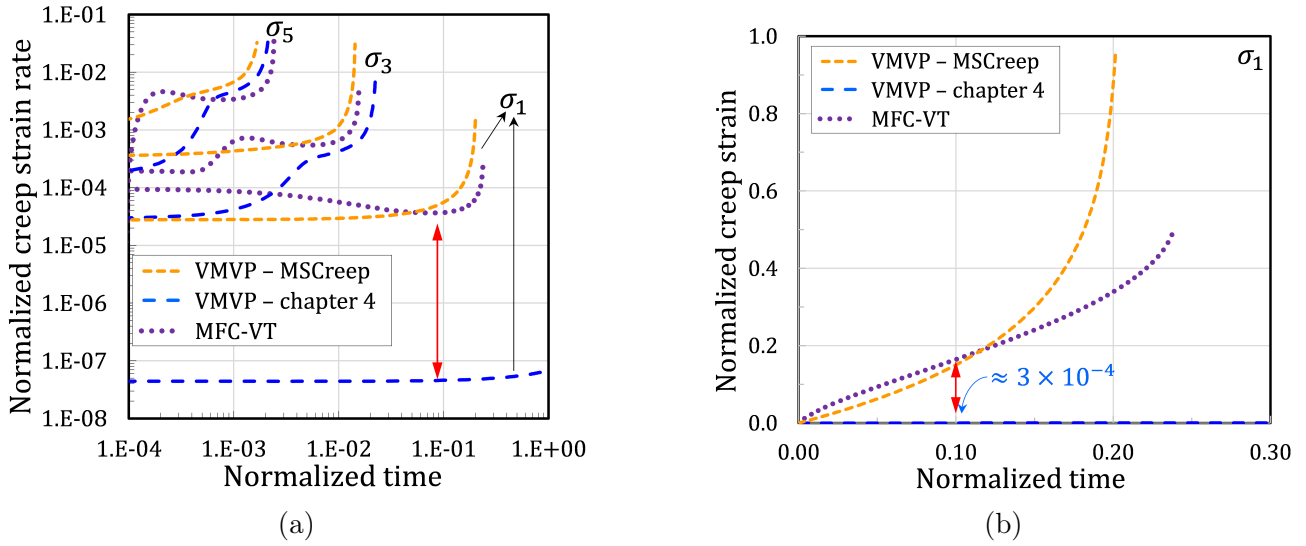


Figure 5.16: Virtual (MFC) and numerical (FE) (a) creep strain vs. time and (b) creep strain rate vs. time curves for Incoloy 800H at temperature T_8 and very low stresses $\sigma_1 < \sigma_3 < \sigma_5$. T_8 is equivalent to T_6 in section 4.3.

Assessment of creep deformation predictability by ECCC methods

The creep deformation response of a material can often be described by multiple creep models, each providing a fairly accurate prediction of the creep deformation response within a given stress-temperature range. Choosing the best model, or approving a particular model, requires the objective assessment of their numerical consistency and predictability. The first aims to verify numerical convergence and the absence of indeterminations during extrapolation, whereas the latter deals with analyzing the creep deformation response in relation to the ground truth material data. The procedure described here above has been condensed into a single standard methodology known as Post-Assessment Tests (PATs). Developed more than 3 decades ago by the ECCC (see ECCC Recommendation Volume 5 [2]), these consist of a series of numerical analyses intended to validate a creep model for a given material. Let us analyze the $\dot{\epsilon}_{ss}$ and time-to-1% creep deformation ($t_{1\%}$) prediction capabilities of our VMVP-MSCreep according to the PAT guidelines.

The PATs intended for creep predictability validation consists in a direct comparison of the reference (ground truth) vs. predicted creep deformation quantities (e.g., $\dot{\epsilon}_{ss}$, t_r , etc.) for a given material. The graphical analysis relies on the distribution and deviation of such data points from the 1:1 relationship (meaning the divergence from a perfect prediction). Additionally, a $\pm 20\%$ error (equivalent in a log-log. scale to a $\pm \log(2)$) is considered admissible in the context of creep modeling. Figure 5.17a presents the reference (experimental+VT) vs. predicted $\dot{\epsilon}_{ss}$ analysis of FE results obtained with VMVP from chapter 4 (identified exclusively on experimental results). Figure 5.17b shows a similar reference vs. predicted $\dot{\epsilon}_{ss}$ analysis addressing the FE results obtained from the new VMVP-MSCreep approach parametrization.

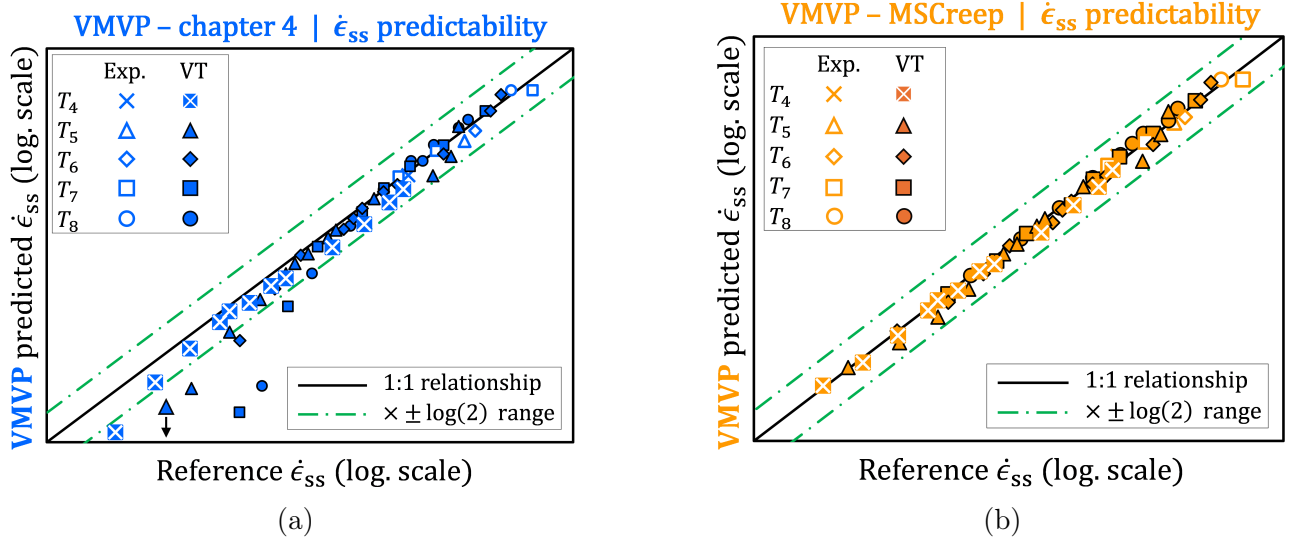


Figure 5.17: Reference vs. predicted steady-state creep strain rate ($\dot{\epsilon}_{ss}$) for (a) VMVP-chapter 4 and for (b) VMVP-MSCreep modeling approaches.

Another relevant creep deformation quantity to evaluate in this context is the $t_{1\%}$. A similar analysis assessing the log-log. error between the reference and the VMVP predicted $t_{1\%}$ is shown in Figure 5.18. In particular, Figure 5.18a deals with VMVP-chapter 4 results, whereas Figure 5.18b addresses the new VMVP-MSCreep modeling approach results.

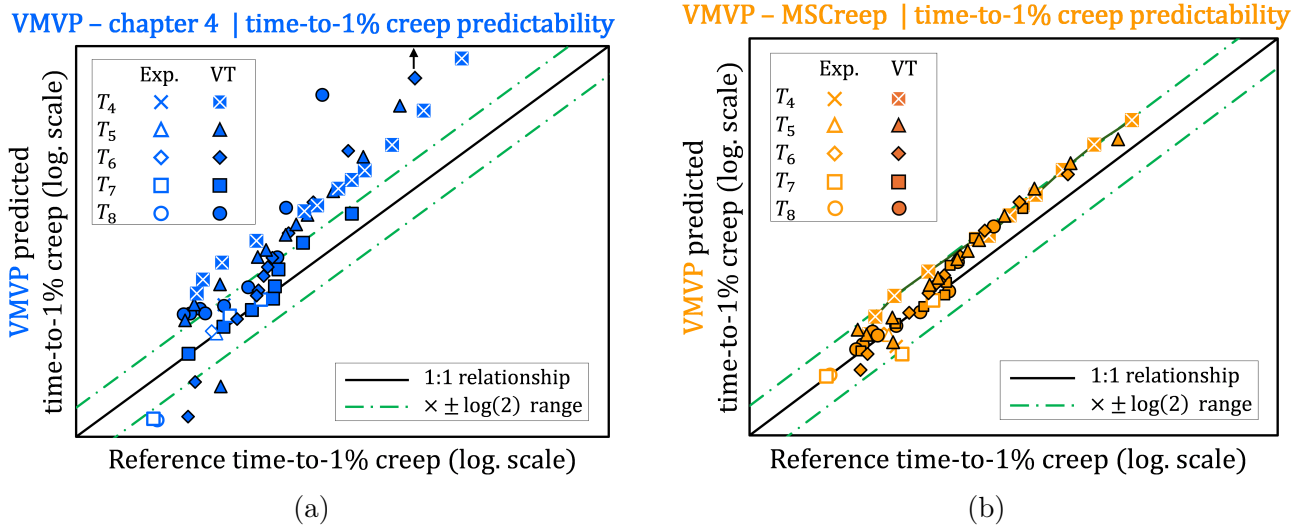


Figure 5.18: Reference vs. predicted time-to-1% creep deformation ($t_{1\%}$) strain for (a) VMVP-chapter 4 and for (b) VMVP-MSCreep modeling approaches.

The data points deviation observed in the log-log. plots shown in Figure 5.17 and Figure 5.18 above indicates that the VMVP-MSCreep does show a significant improvement over the previous VMVP-chapter 4 iteration. A final step in this brief PAT analysis of creep predictability is the quantification of the average (log-log.) error and standard deviation in each case. These quantities, calculated for each predictability log-log. plot in Figure 5.17 and Figure 5.18, are given in Table 5.3.

Table 5.3: Average absolute $\log . - \log .$ error and standard deviation (St.Dev.) calculated from the PAT analysis comparing the reference and FE predicted $\dot{\epsilon}_{ss}$ and $t_{1\%}$ using two versions of VMVP: chapter 4 (old version, identified only on experimental tests) and MSCreep (identified on both experimental and VTs). Note that an error and St.Dev. of $\pm \log(2)$ is considered admissible according to ECCC Reference Volume 5 standards [2].

	VMVP–chapter 4		VMVP–MSCreep approach	
	$ \log . - \log . $ error	St.Dev.	$ \log . - \log . $ error	St.Dev.
$\dot{\epsilon}_{ss}$	$\pm \log(2.21)$	$\pm \log(3.74)$	$\pm \log(1.23)$	$\pm \log(1.18)$
$t_{1\%}$	$\pm \log(7.83)$	$\pm \log(4.58)$	$\pm \log(1.93)$	$\pm \log(1.52)$

Industrial validation case study: bending

The improved very-low stress ($\lesssim \sigma_4$) creep deformation predictability achieved using the MSCreep identification approach on VMVP was demonstrated consistently throughout the low stress benchmark and the PAT analyses offered here above. However, such very-low stress results were achieved and validated after VTs conducted in MFC. The experimental validation of such low stress creep behavior using conventional uniaxial SCTs was deemed unfeasible given the limited resources of this research project.

An additional challenge falling within the FE validation context was the mesh sensitivity of VMVP as a viscoplastic damage COLAW. We validate the null mesh dependence of elastic FE results. However, viscoplastic FE models, with or without damage, exhibit strong mesh sensitivity. A gradient (or non-local) implementation of the numerical COLAW is a well-studied solution to this issue (see, for instance, [3] or the PhD. thesis by Dr. Marcus Lehmann [4]). However, the cost is often a loss of convergence and robustness of the implicit FE code. A more pragmatic approach is to establish an optimal mesh size defined as a function of the stress gradients distributed across the FE geometry in evaluation. Given the time constraints of this thesis, the latter was chosen.

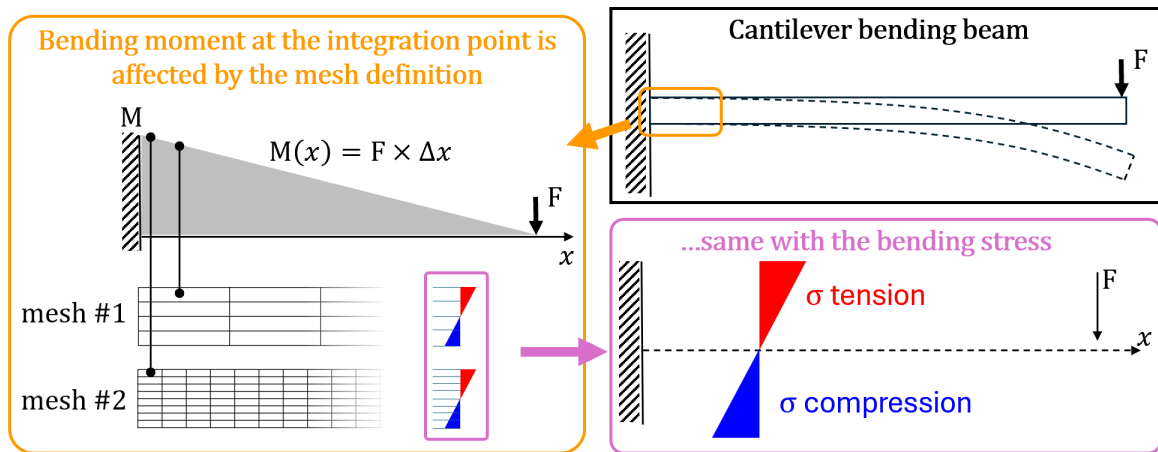


Figure 5.19: Mesh definition of a cantilever beam affecting the mechanical stresses calculated in a FE model.

The bending momentum varying with the distance from the tip makes the bending stress computation largely dependent on the mesh definition. In other words, a finer mesh (e.g., mesh#2 in Figure 5.19) "detects" a higher bending momentum, thus computing a higher bending stress. Additionally, one must consider the existence of stress gradients across both longitudinal and

transversal axes, inherent to the bending situation. Altogether, the well-studied but non-trivial stress distributions observed in a cantilever bending beam provide a numerical challenge for plastic, damage and viscoplastic models alike if no gradient formulation is used.

After careful consideration of the limited space available in the furnace used for the experiments, this bending creep experiment was envisioned to deal with the two remaining FE validation challenges: the validation of the low-stress creep prediction and the establishment of mesh size dependency on a given stress gradient. The experiment consisted of 6 cantilever round profile beams of diameter D and length $34D$ manufactured from Incoloy 800H material. Three of the beams were loaded with a weight w_1 positioned at a distance $34D$ from the origin, resulting in a maximum static bending stress (self weight + w_1) close to σ_5 . In a similar fashion, the other three beams were loaded with a weight w_2 , resulting in a maximum static bending stress of approximately σ_7 . A sketch of the experimental layout is given in Figure 5.20a. The equivalent tridimensional FE model describing the bending beam is described in Figure 5.20b.

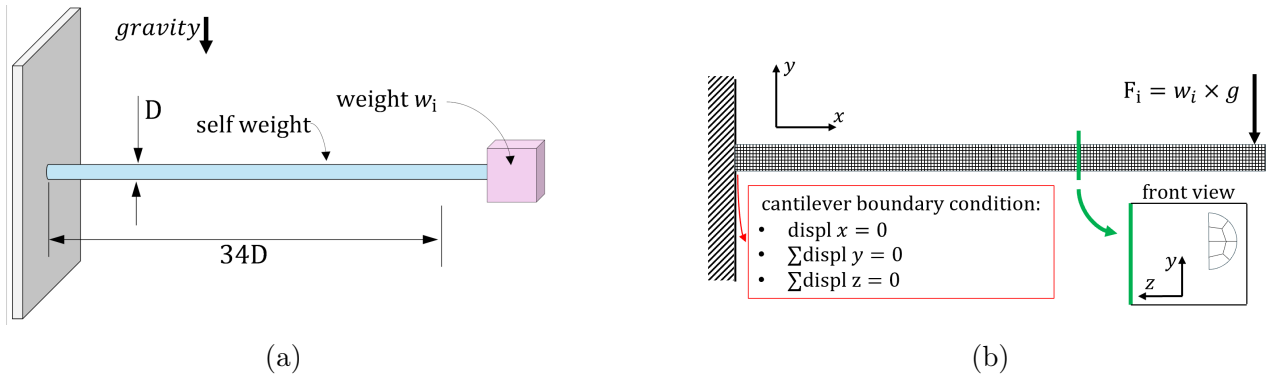


Figure 5.20: (a) 3D illustration and (b) FE model describing the bending beam experiment conducted to validate the low-stress predictability of VMVP-MSCreep and to ascertain the mesh sensitivity of the viscoplastic-damage model.

All cantilever beams were placed in an industrial furnace (an environment similar to that described in [5]) and left for a total time of 15 months at a temperature of approximately T_5 . Following their in-furnace time completion, all bars were manually unmounted for analysis. In particular, the final bending of the bars was measured via simple image correlation based on carefully taken photographs. The measured curves for both σ_5 – A, B, C and σ_7 – A, B, C beams are provided without scale for confidentiality reasons in Figure 5.21a and Figure 5.21b respectively. The large scattering of the curves is partially attributed to human intervention. In particular, curvatures inconsistent with natural bending (i.e., unable to fit a 2nd-degree polynomial) were detected in beams σ_5 – C, σ_7 – B and σ_7 – C, indicating that the beams were deformed by external forces, possibly during the dismounting procedure. In view of this situation, the following validations are conducted on a weighted average deflection curve (assigning the lowest weight to those showing signs of external intervention).

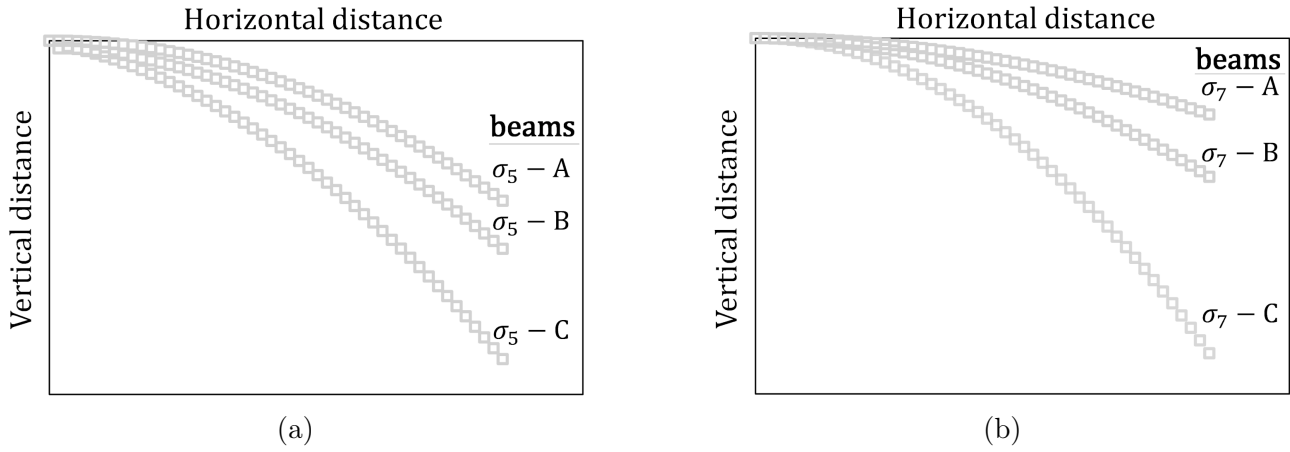


Figure 5.21: Permanent creep bending deflection of cantilever circular profile beams measured at RT after spending 15 months in an industrial furnace at $\approx T_5$ and an initial maximum static bending stress of (a) w_1 (σ_5) and (b) w_2 (σ_7).

Mesh sensitivity analysis

The cantilever beam problem described above was modeled and meshed using the 3D modeling software GMSH [6]. A total of 9 mesh definitions were assessed to study the mesh effect. Taking advantage of the symmetry of the cylindrical beam across the x, y plane (see Figure 5.20b), only one half of the beam was modeled. The different mesh discretizations were defined by combining multiple transversal discretizations (2, 4 or 6 elements across the radius) and longitudinal-to-transversal mesh definitions (ratio 1:1, 1:2 & 1:4), as illustrated in Figure 5.22.

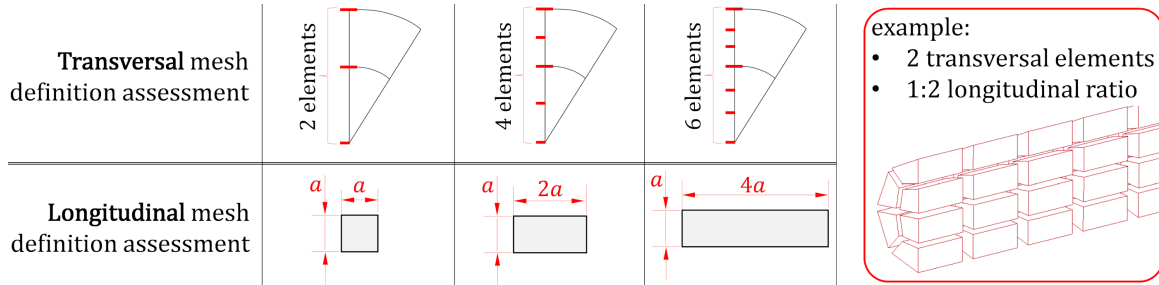


Figure 5.22: Description of the mesh sensitivity analysis methodology, showcasing the different transversal and longitudinal mesh definitions.

The meshed models were all transferred into LAGAMINE FE code. The beam geometry was meshed using "brick" (BLZ3T) elements with 8 nodes, 4 degrees of freedom per node (3 spatial dimensions + temperature) and a single integration point, whereas the special $\sum disp$ boundary conditions were introduced by means of BINDS type of elements (check [7] for more information). Our mesh sensitivity analysis study revealed that the longitudinal mesh definition (i.e., longitudinal-to-transversal ratio 1:1, 1:2 or 1:4, see Figure 5.22) had a negligible effect on the FE results. Conversely, the transversal mesh definition was observed to have a strong impact on the predicted bending creep deformation. A careful consideration addressing the maximum deflection curve under bending creep in all 9 mesh scenarios evidenced that a transversal mesh definition of 4 elements across the radius and a longitudinal-to-transversal ratio of 1:4 provided the optimal compromise creep deformation prediction accuracy and computational efficiency. This observation allowed us to provide an approximate formulation describing the optimal number of elements (N_{elem}) as a function of a stress σ varying across a section of distance

c. This relationship was established through the formula given in Eq.(5.5). Details on the *constant* found to recover the relationship will not be disclosed here.

$$\left. \begin{array}{l} N_{elem} \propto \sigma \\ \sigma \propto c \end{array} \right\} \frac{N_{elem}}{\sigma \times c} = constant \quad (5.5)$$

Simulation results

The creep deflection curves obtained from the LAGAMINE FE simulations for both σ_5 and σ_7 cantilever beam cases are compared to the weighted average experimental curves in Figure 5.23a and Figure 5.23b respectively. The results show that the FE simulation results obtained using the VMVP-MSCreep identification are in much better agreement compared to those using the -chapter 4 parametrization. The latter is attributed to the consistently

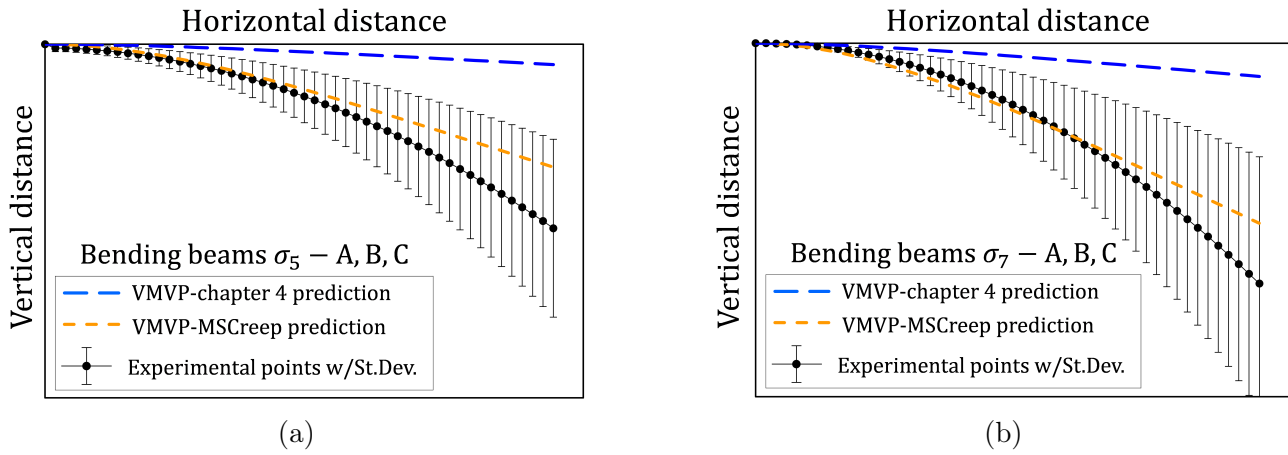


Figure 5.23: Weighted average of maximum creep bending deflection of cantilever circular profile beams under creep at temperature $\approx T_5$ and tip weight w_1 (σ_5) & (b) w_2 (σ_7) for 15 months, predicted in LAGAMINE using both VMVP-chapter 4 and -MSCreep parameter identification procedures.

5.2.4 Discussion

Advantages of the MSCreep approach

The lack of experimental information describing the transition from the SSH creep regime to the $\dot{\epsilon}_{ss}$ regime during chapter 4 was deemed insufficient to fully describe the stress and temperature dependency of the afN parameters. The non-uniqueness of the afN , illustrated in Figure 5.1, was identified as the main factor affecting the accuracy of the VMVP-chapter 4 FE simulations. This was established in section 5.1.1 as part of the motivations guiding the development of the MSCreep approach.

The accurate creep numerical predictions achieved with VMVP-MSCreep are directly linked to the MSCreep approach application and the realistic description of the creep behavior of 800H alloy produced by the MFC VTs. MFC was exploited as an inter- and extrapolation tool with two main targets:

1. **to reach extremely low stress levels:** The results of this target are first shown within the low-stress benchmark from Figure 5.14 to Figure 5.17 and later validated for a creep bending case study. Altogether, the FE simulation results obtained with the VMVP-MSCreep model are in very good agreement with the experimental results. Finally, the PATs benchmark shows a remarkable prediction improvement over very low $t_{1\%}$ and $\dot{\epsilon}_{ss}$

(occurring at very low stresses) compared to those obtained with the previous (chapter 4) iteration of VMVP.

2. **to fill the gaps in-between ULiège SCT experiments:** the effects of this second target are evidenced throughout the experimental benchmark. For instance, the creep curves at T_6 in Figure 5.10 or at T_7 in Figure 5.11 show that the VMVP-MSCreep approach provides more consistently accurate FE creep predictions for $\sigma \leq \sigma_{11}$. A more detailed inspection of the creep strain rate curves for these two cases show that both VMVP model identifications (-chapter 4 and -MSCreep) reach a correct $\dot{\epsilon}_{ss}$, indicating that the Norton parameters (i.e., K , n) are correctly identified in both cases. The source of discrepancy between both VMVP identifications (and ultimately loss of accuracy of -chapter 4 iteration) is found at the wrongful parametrization and identification of the afN function. This observation is represented for the case (σ_{11}, T_6) in Figure 5.24.

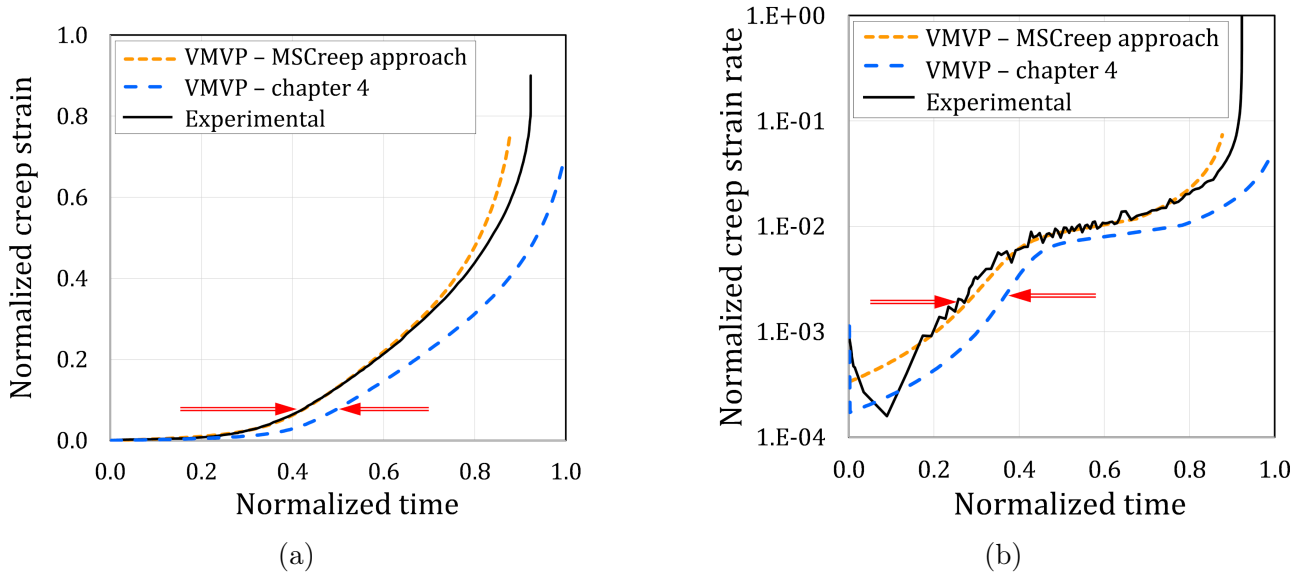


Figure 5.24: (a) Creep strain vs. time and (b) creep strain rate vs. time curves showcasing the LAGAMINE-VMVP prediction achieved with both VMVP-chapter 4 and VMVP-MSCreep approaches.

Note that, while both models predict the correct $\dot{\epsilon}_{ss}$, the incorrect afN parametrization of VMVP-chapter 4 leads to the loss of creep prediction accuracy during the SSH-to- $\dot{\epsilon}_{ss}$ creep regime transition. Conversely, the new MSCreep modeling approach allows to capture the experimental results with remarkable accuracy.

On the CDM approach

CDM approaches are a computationally inexpensive and reliable interpretation of the loss of load bearing capacity of the material. They have been incorporated in our numerical models to capture the progressive softening of tertiary creep and the rupture of the material. The validation of the models, namely MFC in chapter 3 and VMVP-MSCreep here above, must be ascertained independently from each other because the numerical aspects driving the creep deformation differ from one model to another. The core difference in each model stems from the stress configuration they were designed for. MFC deals exclusively with uniaxial deformations, whereas the FE COLAW VMVP deals with complex loading configurations. To this end, the true stress in MFC is calculated from the reduction of the cross section area as function of the uniaxial strain (ϵ) and the Poisson ratio (ν) of the material, whereas VMVP computes it as the

von-Mises equivalent stress from the stress tensor ($\underline{\sigma}$). The different true stress computation approaches are shown in Eq.(5.6).

$$\text{true stress: } \sigma = \begin{cases} \sigma_0 \cdot (1 - \nu\epsilon)^{-2} & \text{in MFC} \\ \sigma_{\text{vM}} = \sqrt{\frac{3}{2} \underline{\sigma} : \underline{\sigma}} & \text{in VMVP} \end{cases} \quad (5.6)$$

The two different true stress approaches shown in Eq.(5.6) predict a similar deformation response for their corresponding models. This entails that the hardening and softening information and phenomena adopted for each model cannot be exchanged. In other words, if a model working with a higher true stress requires a stronger softening (e.g., damage) to reach the desired deformation level. Let us illustrate this event in Figure 5.25a, addressing the case (σ_{13} , T_6). According to Figure 5.25a, both creep curves predict similar deformation levels consistent with the experiment. However, as seen in Figure 5.25b, the damage initiation and accumulation rate are in both cases different.

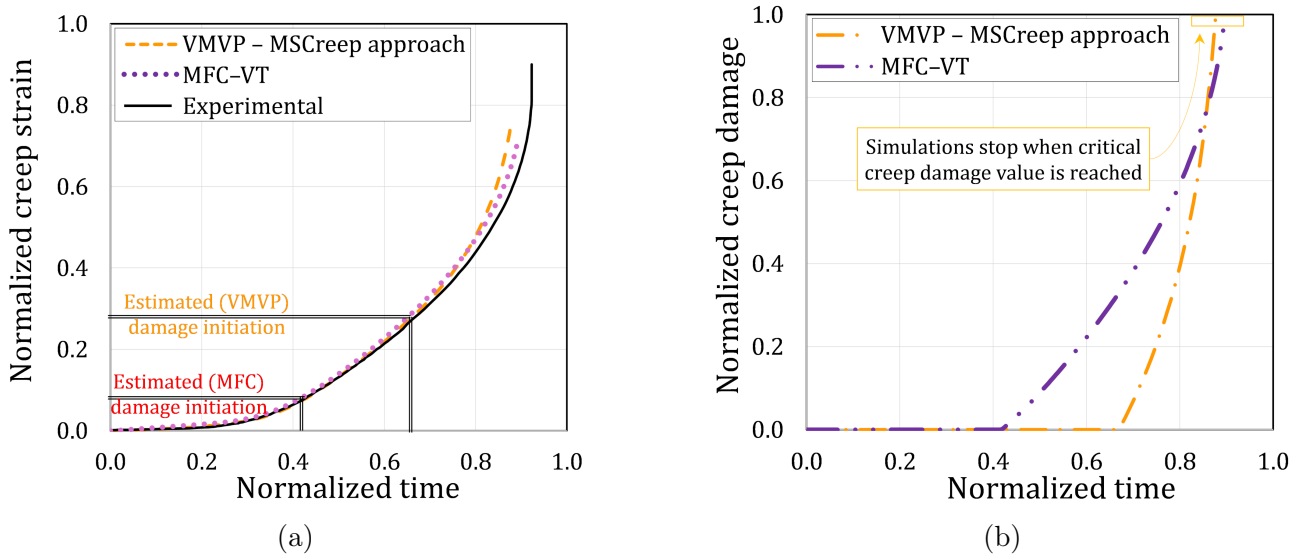


Figure 5.25: (a)

This evidences that creep damage remains a phenomenological concept aimed at recovering the loss of load-carrying capacity of the material. The damage laws and parameters identified in each model are validated because they capture the material softening observed in the experiments. They are, in other words, physically-founded phenomenological functions that predict the effect of damage accumulation rather than damage itself (see for instance the book by J. Lemaitre et al.[8]).

5.3 Conclusions

This chapter presents a multi-scale creep methodology consisting of the exploitation of a microstructure-based Mean Field Creep (MFC) model as to produce realistic virtual creep curves to complement experimental data. The multi-scale creep (MSC) modeling approach was applied to our case study alloy 800H, where the high-temperature low-stress uniaxial creep deformation response of the material was mapped by combining 8 real standard creep test (SCT) with a total of 79 virtual tests (VTs). The 87 creep curves were destined for the parametrization and parameter identification of the macroscopic FE viscoplastic constitutive model VMVP in Lagamine. The improved phenomenological parametrization of the stress and temperature

dependency of the multiple activation-function Norton (*afN*) terms was a direct result of the VTs involved in the process. In particular:

- VTs concerning stress-temperature creep loadings found in-between our experimental curves improved our understanding of the transitional creep behavior of Incoloy 800H observed between the first solid-solution hardening (SSH) minimum creep strain rate and the subsequent onset of the steady-state creep strain rate ($\dot{\epsilon}_{ss}$). The improved stress-temperature parametrization of the activation function components of the *afN* viscous law allowed the VMVP-MSCreep model to predict with higher accuracy and consistency those creep deformation responses found within low strains (e.g., time-to-1% creep deformation ($t_{1\%}$)).
- VTs conducted at very low stresses evidenced a creep softening of the material consistent with a low-stress Norton law breakdown. This creep response was incorporated into VMVP during this MSCreep procedure by introducing a stress-dependency into the Norton exponent n of our *afN* viscosity function. Low-stress creep deformation predictions ($t_{1\%}$ and $\dot{\epsilon}_{ss}$) obtained from FE simulations conducted using the MSCreep-identified VMVP law were found to be in good agreement with VTs. Interestingly, the results of our previous VMVP-chapter 4 iteration (identified exclusively on experimental results, not addressing VTs) failed to capture the low-stress Norton law breakdown behavior. Instead, they predict a creep strain rate almost 2 orders of magnitude higher than that predicted by our MFC VTs; a dangerous overestimation of the low-stress creep performance of our alloy 800H.

An experimental creep validation campaign consisting of 6 round-profile cantilever beams, 3 of which were loaded with a tip weight w_1 and 3 with a tip weight w_2 , was organized to validate both our VMVP parameters and our local FE model implementation. The permanent deflection of the beams after spending 15 months subjected to high-temperatures and a constant bending load was measured and used as a means to study the mesh sensitivity of our viscoplastic law and provide experimental validation for the low-stress predictability improvement following the MSCreep identification. The mesh sensitivity analysis established an optimal element size rule based on the stress gradient distribution of a given FE geometry. The low-stress predictability of the VMVP model was later studied for both cantilever bending cases (w_1 and w_2). The creep deflection curves predicted by VMVP identified with the MSCreep approach were in good agreement with the experimental results, whereas those predicted using the previous -chapter 4 iteration of VMVP predicted a deformation almost 10 times lower than that measured during the experiments.

The MSCreep modeling methodology represents a computationally efficient coupling of the two numerical models developed during this thesis: the micro-mechanical MFC and the macro-mechanical model VMVP. Results show that the physically accurate creep description provided by the MFC VTs allowed the MSCreep identification methodology to improve the FE predictability of VMVP.

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Conclusions

In this doctoral thesis, the accurate finite element (FE) prediction of the creep deformation response of Incoloy 800H was achieved by means of a multi-scale creep (MSC) modeling approach where the parametrization and parameter identification of the macromechanical VMVP (von-Mises viscoplastic) finite element (FE) constitutive law used in Lagamine code^[1] was conducted using a creep curve database consisting of experimental and virtual tests.

Experimental campaign

The tensile creep tests conducted in the Materials and Structures (M&S) laboratory in ULiège were complemented with a thorough microstructural characterization campaign in cooperation with Metallurgic and Materials Science (MMS) researchers and UGent colleagues, involving Light Optical Microscopy (LOM), indentation, and Scanning Electron Microscopy (SEM) analyses. These experiments revealed the following:

- Standard creep tests (SCTs) addressing low and medium stresses and covering medium to high temperatures revealed a non-classical creep behavior consistent with the two-step creep strain rate minima reported for 800H in scientific literature. The creep curves obtained during these experiments were used to feed the database used for the parameter identification and validation of our micromechanical (MFC) and macromechanical (VMVP) creep models.
- The creep deformation performance of Incoloy 800H recorded during our SCT campaign was consistently lower than that informed in available industrial datasheets. In particular, the time-to-rupture (t_r) calculated with the Manson-Haferd Parameter (MHP) extrapolation model given within the European Creep Collaborative Committee (ECCC) datasheet^[2] was higher than that observed during our experiments. This discrepancy is explained by the differences in each experimental procedure, namely the hot rolled sheet source material used to manufacture our specimens and the modified experimental methodology employed during our SCTs campaign to better capture the first (SSH) creep strain hardening peak.
- The non-classical creep regime of 800H alloy is the result of a complex microstructural evolution process. At high temperatures (below the Dynamic Recrystallization (DRX) point), Cr_{23}C_6 precipitates initially nucleating at dislocations during the SA+WQ heat treatment dissolve in favor of the progressive nucleation and coarsening of Cr_{23}C_6 at grain boundaries (aging). The first creep strain hardening peak observed in this material during high-temperature and low-stress viscoplastic deformations was thereby attributed to the solid-solution hardening (SSH) regime caused by the atoms liberated during the rapid

^[1]: Lagamine, finite element code developed at the Materials & Solid Mechanics team in University of Liège.
url: www.lagamine.uliege.be

^[2]: ECCC data sheet, ECCC Working group WG3.3, approved by Michael Spindler, pag.95-97, 1998

dissolution of these carbides. The second creep strain hardening peak was identified as the steady-state creep strain rate ($\dot{\epsilon}_{ss}$) regime of the material.

- All uniaxial creep test attempts on 800H specimens aged for years in an industrial furnace resulted in the rupture of multiple ceramic components belonging to the high-temperature extensometer of the Zwick/Roell (Z/R) Kappa 100DS creep machine in M&S (ULiège). The fracture of these brittle ceramic inserts was likely caused by the onset of non-uniaxial stress gradients within the vicinity of the specimen section in connection to the extensometer arms. This was attributed to the combined effects of the microstructure inhomogeneity (non-homogeneous corrosion penetration) and the irregular cylindrical geometry of these specimens caused by a re-machining process where the tolerances instructed by the creep machine manufacturer were not strictly considered. Ascertaining microstructural homogeneity and geometrical tolerances of the specimen destined for standard creep tests (SCTs) in machines with delicate ceramic components must thereby be considered paramount.
- The microstructure characterization analyses of industrially-aged and of creep-tested 800H specimens provided an exceptionally detailed perspective of the progressive aging and corrosion behavior of the alloy under a realistic atmosphere containing N_2 , O_2 and water vapor. The core material microstructure exposed only to high temperatures (i.e., no environmental effects) exhibits a progressive nucleation and coarsening of intergranular $Cr_{23}C_6$ precipitates (aging). Conversely, the high temperature combined with an O_2 and N_2 rich atmosphere leads to a very peculiar corrosion behavior involving oxidation and nitridation:
 - $t \approx 0 - 10$ hours: the low-diffusivity chromia layer ($Cr_{(2-x)}Fe_xO_3$), formed naturally upon contact with O at high temperatures, provides the alloy with protection against reactive elements such as O and N.
 - $t \approx 10 - 100$ hours: formation of cracks within the chromia layer. The precipitation of new chromia from the 800H material exposed to the atmosphere is limited by the coarsening of detrimental spinel oxide phases (mainly Al, Si and Mn oxides, associated with loss of load carrying capacity) and the progressive material sensitization (Cr depletion) in the zones underneath the chromia layer. In the absence of a protective oxide layer, nitrogen starts to penetrate into the matrix material located underneath the surface.
 - $t \approx 1$ year: the incremental concentration of N in the matrix induces the nucleation of Al and Cr nitrides. The maximum penetration depth reached by Cr nitrides was measured at $\lesssim 150\mu m$ after 1 year in the furnace.
 - $t \approx 2$ years and more: the material sensitization and oxygen penetration induces the dissolution of Cr-nitrides in favor of the nucleation of Cr-oxides. Hereafter, the nitridation front is survived only by AlN.

The increased surface hardness of the 800H microstructure decorated with nitrides, quantified via $\mu V^{[3]}$, indicated that the creep strengthening effect associated with nitridation in 800H can be attributed exclusively to the CrN and Cr_2N precipitates. Hardness analyses of the nitrided surface of specimens spending more than 2 years in-furnace (i.e., a microstructure where only AlN remain) showed negligible differences compared to the core material. Given the low maximum penetration depth and the subsequent dissolution of Cr nitrides, the creep strengthening contribution associated to nitridation in 800H alloy can safely be considered negligible from a creep modeling standpoint.

Mean Field Creep (MFC)

A creep-oriented mean-field model was chosen as a balanced semi-physical alternative to generate virtual creep tests within a physics-based framework. In this PhD., the accurate creep deformation prediction of Incoloy 800H under uniaxial loadings was achieved with MFC. It extends the classical dislocation density equations describing the creep deformation related to a fixed initial geometry. The new equation system combined with the ex-situ precipitate thermokinetic information provided by Thermo-Calc[®] PRISMA allowed to accurately represent the physical effects of the microstructural phenomena involved in the non-classical two-peak creep response. In particular, the first (SSH) peak was addressed as an internal stress contribution slowing down dislocation glide, whereas the second ($\dot{\epsilon}_{ss}$) peak was captured by an improved stress sensitivity formulation of the mechanical energy threshold in the mean dislocation glide velocity equation. Altogether, these features make our MFC an ideal modeling tool to produce virtual creep curves, combining modeling accuracy, physicality, reliability and computational efficiency.

Accurate prediction of creep curves using mean-field models (such as MFC) is not trivial. A safe number of experimental creep curves and extensive qualitative and quantitative knowledge of the material microstructure are required. This useful information is not only for conventional parameter identification but also in cases where model modifications are deemed necessary. The mean-field set of equations describing the relationship between the creep deformation response and the microstructural features involved in the mechanical response vary from one material to another. A similar methodology to that employed to develop MFC, can be used by other researchers to adapt a mean-field model to a different material, incorporating new terms representing, for instance, a new creep, hardening or softening mechanism. Finally, mean-field is a promising modeling technique that has yet to offer a unified model. Developers are required to have knowledge on microstructural and physical aspects of creep.

VMVP (von-Mises viscoplastic)

The FE prediction of the two-step creep strain rate minima observed in 800H alloy was achieved by modifying the viscosity function in unified viscoplastic constitutive model (UVCN) constitutive law. A Norton power law was chosen to capture the stress-temperature relationship observed during the second ($\dot{\epsilon}_{ss}$) creep strain peak, whereas the first (SSH) peak was captured with an activation function. The activation-function Norton (*a.fN*) viscosity law was implemented into VMVP: an extended Chaboche-Morch^[4] type UVCN in Lagamine. Numerical simulations conducted with the new VMVP constitutive law identified exclusively on ULiège experiments showed promising capabilities and computational stability. However, parameter uniqueness could not be ascertained given the small size of the creep dataset.

Multi-Scale Creep (MSC)

The MSCreep modeling approach consisted of the production of a comprehensive 800H creep behavior knowledge bank combining virtual and experimental creep curves. To this end, MFC

[3]: Carlos Rojas-Ulloa, Hélène Morch, Víctor Tuninetti, Jérôme Tchoufang Tchoundjang, Olivier Pensis, Amedeo di Giovanni, Anne Mertens, Laurent Duchêne and Anne Marie Habraken. "Microstructure evolution of Incoloy 800H in industrial environment and correlation with creep mechanisms from literature". *Materials at High Temperatures*, Vol.41(2), pg.311-321. ISSN: 0960-3409 (Feb. 2024). doi: 10.1080/09603409.2024.2342602.

[4]: Morch, H. *Thermomechanical modelling of the creep-fatigue behaviour and damage of Nickel-alloy receiver tubes used in Concentrated Solar Power plants* English. PhD. (Université de Liège, 2022)

simulations were conducted at loadings different from those addressed during the experimental campaign in ULiège, reaching stresses where conventional SCT would take years to complete. Finally, the parameter characterization of VMVP following the MSCreep modeling approach was comprised of 87 creep curves (79 virtual + 8 experimental).

The virtual tests conducted at creep loadings close to those addressed in the ULiège experimental campaign provided a physically accurate depiction of the SSH-to- $\dot{\epsilon}_{ss}$ two-peak creep deformation response of 800H alloy, whereas those conducted at very-low stresses revealed a creep softening trend consistent with a Norton power-law breakdown. The FE results show that the VMVP-MSCreep parametrization stemming from the comprehensive MSCreep behavior mapping resulted in reduced parameter uncertainty and improved numerical accuracy. In particular:

- Experimental validation addressing the SCTs conducted in ULiège was ascertained for creep loadings found within the stress-temperature applicability range defined for VMVP-MSCreep.
- Compared to industrial 800H datasheets, the creep deformation response predictions achieved with our VMVP-MSCreep modeling approach are consistently conservative. In other words, the Incoloy 800H creep performance informed by the datasheets corresponds to a material with a higher creep strength and a longer time-to-rupture (t_r) than those predicted by our VMVP-MSCreep by a factor of approximately 30%. This difference stems from the experimental results obtained during our SCT campaign. The conservative predictability error and the inherent ($\pm 20\%$) scattering of creep data, in addition to the creep testing uncertainty of the industrial datasheets, makes this an admissible error from a creep modeling safety standpoint.
- The post-assessment tests (PATs) conducted to study the numerical predictability of VMVP-MSCreep showed that the predicted $\dot{\epsilon}_{ss}$ and time-to-1% creep deformation ($t_{1\%}$) creep deformation were consistently found within the $\pm \ln(2)$ threshold required for validation according to the European Creep Collaborative Committee (ECCC) standards.
- Validation for non-uniaxial stress regimes was ascertained by finite element (FE) modeling of the creep deflection observed for cantilever beams spending 15 months in a furnace reproducing a realistic industrial environment. The VMVP-MSCreep FE simulation results were found to be in reasonable agreement with the experiments. Additionally, the FE sensitivity analysis allowed us to establish an optimal mesh size determined according to the stress gradient ratio. The application of the mesh size relationship rule ensures that the numerical results obtained with this FE model remain independent from the FE mesh, despite the inherent non-linearity of the UVCN and the lack of a non-local FE formulation used in VMVP.

Altogether, the MSCreep modeling approach developed in this thesis has demonstrated huge potential to reduce the cost and length required for a conventional SCT experimental campaign to characterize the creep behavior of a material. Using only conventional SCTs to achieve the improvements reported from the implementation of the MSCreep approach would have required time and resources far beyond those available during this project.

Perspectives

1. The robustness of the MFC-based MSCreep methodology developed in this PhD. should be ascertained by applying it to other materials. A first step in this direction has been

the use and development of MFC in the sister project AID4Greenets. This EU project has allowed us to:

- Adapt our MFC model to describe the microstructure-informed creep behavior of the steel alloy 30CrMoNiV5–11 applied for instance in large shaft manufacturing (Reinosa industry). The modified MFC, adapted to capture a single minimum creep strain rate peak ($\dot{\epsilon}_{ss}$), incorporates additional phenomenological parametrization to capture a stress-dependent creep mechanism transition. The model has been recently validated and published^[5].
 - Apply MFC to predict the creep behavior of materials during a stepped iso-stress method (SSM) test. SSM experiments consist of capturing the $\dot{\epsilon}_{ss}$ at different stress levels by applying incremental stress steps at a constant temperature, thus shortening the length of an otherwise time-consuming SCT campaign. More details on the modifications of the model to capture the creep strain during stepped stress loadings will be provided in an article still in preparation.
2. The VMVP FE constitutive law developed in this PhD. is expected to provide accurate creep life assessments for complex industrial applications. The robustness of the MSCreep modeling and parametrization approaches and the mesh size dependency on the stress gradient have been validated for standard and bending creep experiments addressing simple geometries. However, numerical studies addressing steep stress gradients or large metallic components may require a number of elements far beyond the computational limits of the Lagamine FE code. In this context, our extended VMVP model could be integrated into METAFOR (MEtal FORming FE software by ULiège): a more efficient and commercially oriented FE software that integrates parallel computation and a graphical user interface. A precedent for a Lagamine-METAFOR constitutive law migration is shown in the PhD. of H. Morch^[4]. This “Morch” constitutive law is currently used by GDTECH company to model creep-fatigue deformation and damage in industrial components.

Other interesting VMVP developments could be as follows:

- A non-local numerical implementation, allowing the computation of the stress to become effectively independent from the mesh element size.
- Extension of the model to predict inelastic deformations occurring during creep-fatigue loadings. The backstress equations required for creep-fatigue computations have been incorporated into VMVP; however, these have been purposely deactivated in the context of this creep-oriented PhD. thesis. Characterization and identification of fatigue-related parameters in VMVP is foreseen for industrial applications where cyclic loadings are reported.
- A “jump” version, where the lengthy cycle-to-cycle computation of inelastic deformations and damage accumulation is shortened by a simple linear extrapolation of the behavior after n –cycles. An approach of this kind was developed for the UVCm by H. Morch et al.^[6], showing good numerical predictability while optimizing the use of computational resources.

^[5]: Fan Chen, **Carlos Rojas-Ulloa**, Jérôme Tchoufang Tchuindjang, Olivier Dedry, Rishabh Bharadwaj, Mahesh Somani, Anne Mertens and Anne Marie Habraken. “Microstructure-informed creep model for 30CrMoNiV11-5 steel”. *Materials & Design*. ISSN: 02641275 (June 2026). doi: 10.1016/j.matdes.2026.116152

^[6]: Hélène Morch, Laurent Duchêne, Ridha Harzallah, Víctor Tuninetti and Anne Marie Habraken. “Efficient temperature dependence of parameters for thermo-mechanical finite element modeling of alloy 230”. *European Journal of Mechanics - A/Solids*, Vol.85. ISSN: 0997-7538 (Jan. 2021). doi:10.1016/j.euromechsol.2020.104116.

3. The development of a numerical approach capable of providing a reliable approximation of the creep deformation response of new alloys on the basis of the stoichiometric and phase composition of the material and reliant on CALPHAD software such as Thermo-Calc® is not an impossible dream. The instantaneous material composition and heat-treatment optimization offered by this approach would provide a huge advantage in the field of alloy design.

A model such as this could be a perfect tool to aid in the rapidly evolving field of alloy development by additive manufacturing. An experimental campaign combining laser powder bed fusion (LPBF) manufacturing, heat treatment assessment and SSM creep testing would enable rapid creep performance characterization and validation of the model predictions. Later exploitation of the validated model could potentially reduce experimental costs, guide the development of new alloys, and possibly be exploited as a deterministic tool to feed deep learning models.

4. The numerical models developed over the course of this thesis can be exploited to develop new deep learning modeling solutions that can provide fast and reliable creep strength, creep rupture and $\dot{\epsilon}_{ss}$ calculations to assist in the creep design of metallic components. Currently, a prototype application that requires a single SSM test to provide creep deformation quantities (currently $\dot{\epsilon}_{ss}$ and time-to-rupture (t_r)) has already been developed by our colleague L. Morand at Fraunhofer Society in the context of AID4Greenest project. The model is currently trained on a virtual database generated by simple uniaxial FE simulations in Lagamine[2] using an extended Chaboche-Morch UVCm for virtual materials. Further development of this tool involving curve feeding and validation using our deterministic creep models (VMVP and MFC) for multiple other real materials is undoubtedly a promising.
5. The scientific contributions and numerical tools developed during this thesis have also been integrated into the framework of a new research **Scale bridging** modeling project titled **RaMPANT** (**R**eliability and **M**ulti-scale **P**rediction **A**cross **N**anostructural **T**o microstructural behavior), led by PhD. candidate MScEng. Rayane Eloudrhiri under the supervision of Prof.Dr. Laurent Duchêne. This new project aims to improve the physicality of microstructure-informed creep predictions through an integrated numerical platform. The automated multi-scale modeling algorithm proposed by MScEng. Rayane Eloudrhiri addresses 2 length scales: At the macro-level, the creep deformation of complex geometries is computed via FE simulations in Lagamine or METAFOR (both FE software programs developed in ULiège). During the simulation, certain temperature or stress thresholds known to induce microstructural changes trigger a series of automated SSM-MFC simulations aimed at updating the FE viscosity function parameters, thus capturing the creep behavior effects attributed to local microstructural changes. To improve the physicality and computational efficiency of microstructure-informed MFC predictions, the thermokinetic information will be retrieved from phase-field (PF) simulations conducted in a PF software developed in ULiège by both Materials and Solid mechanics (MSM) and Metallurgic and Materials Science (MMS) research teams. Compared with commercial CALPHAD softwares (e.g., Thermo-Calc®PRISMA), our PF software is advantageous because it allows skilled PF ULiège researchers to have full control over the microstructure state variables. This integrated platform offers a computationally efficient methodology and a user-friendly environment in which the microstructure dependency of the creep deformation response of materials is incorporated into a fully automated framework. Such a numerical tool could potentially improve the prediction of the creep behavior of materials exhibiting high stress gradients and localized microstructural differences, such as in the case of the shaft of the European Project AID4Greeneest.

Carlos Eduardo ROJAS ULLOA

Civil-Mechanical Engineer, Msc.Eng.

 [linkedin.com/in/crojas-ulloa](https://www.linkedin.com/in/crojas-ulloa)
 orcid.org/0000-0002-6997-9128
 (ULiège) - ce.rojasulloa@uliege.be
 (Personal) - ce.rojasulloa@gmail.com
 Temuco, Chile (29)



Areas of expertise

- High-temperature materials
- Multiscale numerical modeling
- Plasticity & viscoplasticity
- Metallurgy & Material sciences

Personal interests

- Hiking
- Exercising
- Reading
- Cooking

Personal values

- Discipline
- Commitment
- Responsibility
- Communication

EDUCATION

2021-today	PhD. of Engineering Sciences, Université de Liège, (Liège, Belgium)
2018-2020	Master of Engineering Sciences, Universidad de La Frontera (Temuco, Chile)
2015-2018	Bachelor of Engineering Sciences, Universidad de La Frontera (Temuco, Chile)

TECHNICAL COMPETENCES

Programming languages	Matlab (8+ years), Python(3+ years), Fortran (6+ years)
Numerical modeling	Finite Element : ANSYS (user), Lagamine (developer) Metallurgy : Thermo-Calc; CAD : autoCAD, SolidWorks
Utility softwares	Microsoft Office, LaTeX, Visual Studio code & community
Languages	Spanish (native), English (advanced), Français (B1+)

PROFESSIONAL EXPERIENCE

today October 2025	Research Engineer PhD. Student, AID4GREENEST, ULiège ➤ Creep life assessment of steel alloy 30CrMoNiV5-11 : mean-field creep modeling European Project Horizon-CL4-2022-RESILIENCE-01-19
September 2025 January 2021	PhD. Student, MSCREEP, ULiège ➤ Multi-scale high-temperature low-stress creep life assessment of Fe-Ni-Cr alloys ➤ Development of microstructure-dependent model : Mean-Field Creep ➤ Development of finite element macro-mechanical constitutive law (Lagamine) ➤ Assessment of the effects of corrosion in creep life of austenitic steels FRIA B1 4000-8987 FRIA B2 40002-1907
December 2021 May 2021	Research Engineer, DOMMACO, ULiège ➤ Finite element modeling of quasi-static ductile fracture of Ti-6Al-4V (Lagamine) Wallonie-Bruxelles International

RESEARCH INTERNSHIPS

August 2018 June 2018	Non-linear optical systems, UNIVERSIDADE FEDERAL OF PERNAMBUCO (UFPE), Brasil ➤ Non-linear optical properties of materials : mounting of optical systems & data postprocessing. Laboratory of Photonics and Biophotonics
December 2017 February 2018	Linear optical systems & electronics, UNIVERSITY OF CONCEPCIÓN (UDEP), Chile ➤ Design & prototyping of a STM32-type microcontroller-based monochromator Center for Optics and Photonics (CEFOP)

