

# Accurate very-high temperature creep-life prediction of Incoloy 800H addressing effects of creep mechanism transition and nitridation

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## Introduction

- Motivation
- Scientific context
- Scientific challenge

## Experimental campaign

- Microstructural evolution under operational conditions
- Macromechanical behaviour under creep

## Numerical campaign

- Numerical model
- Methodology
- Results & discussion

## Concluding remarks

- Conclusions
- Prospects

## Incoloy 800H: a Fe-Ni-Cr austenitic alloy

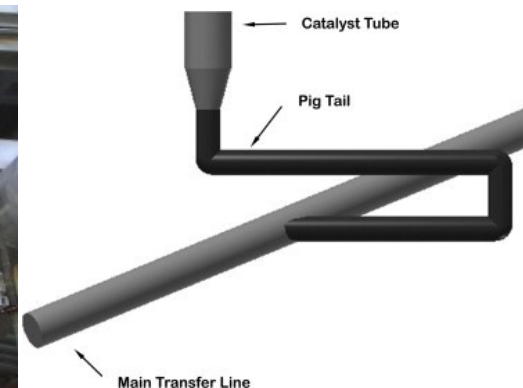
- Solution annealing: 1150°C + WQ
- Microstructure:



- Austenitic (FCC)
- Grain twinning
- Intra- & intergranular precipitates
  - $M_{23}C_6$
  - $M_6C$
  - $Ti_x(C,N)$
- Average Grain Size  
 $1 \leq \text{ASTM Gr.} \leq 5$

### Applications:

- Petrochemical
- Metallurgy
- Heat exchangers
- Power generation
- ...



after L.A. Spyrou et al., Eng. Fail. Anal., 2014

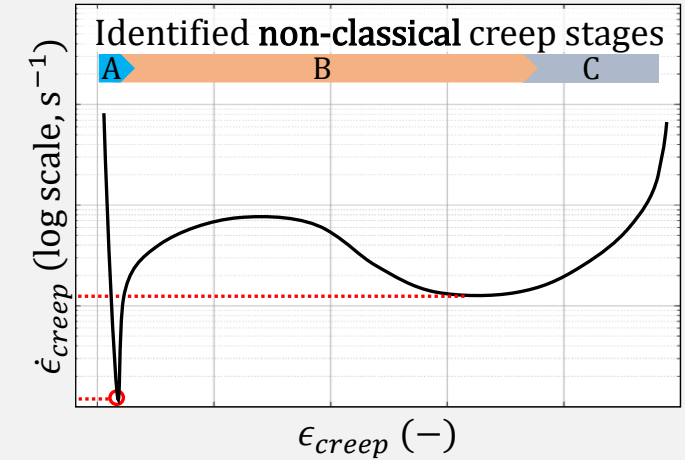
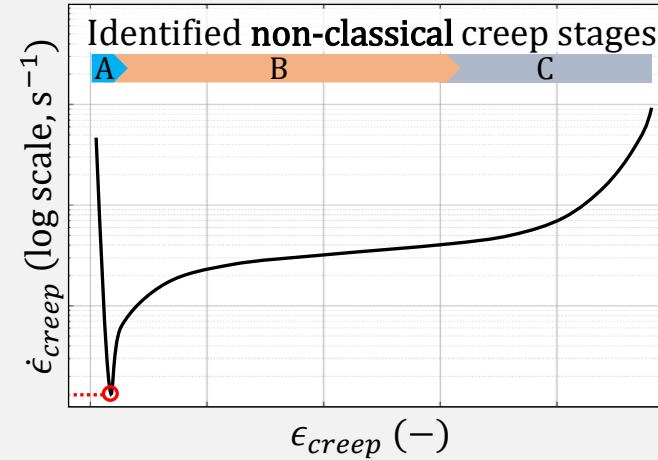
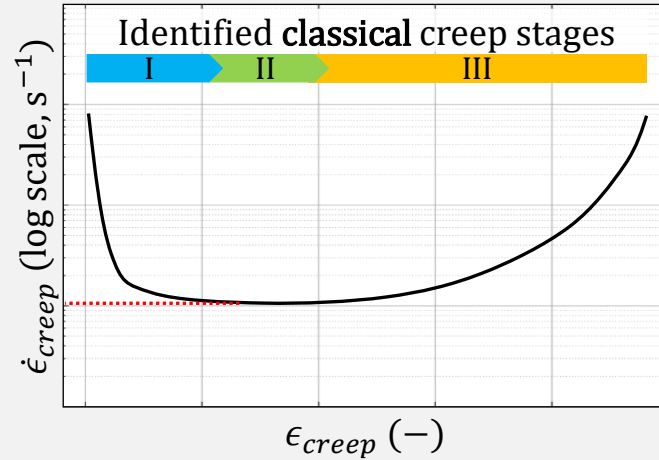
## Creep test

High-mid stress & Low-mid T  
 $\sigma \gtrsim 40$  [MPa] |  $T^\circ \lesssim 760^\circ\text{C}$

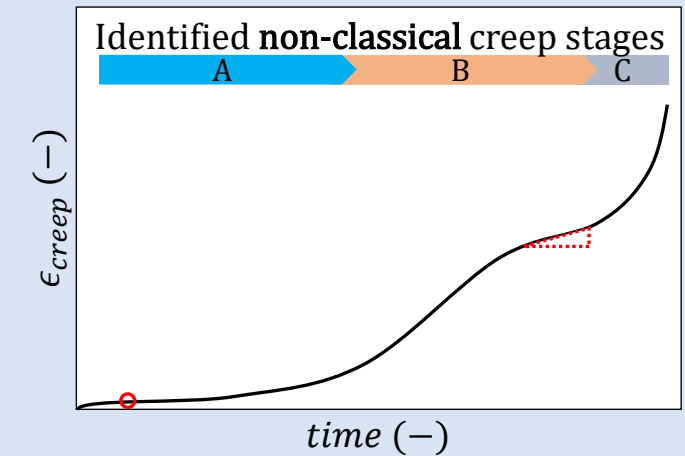
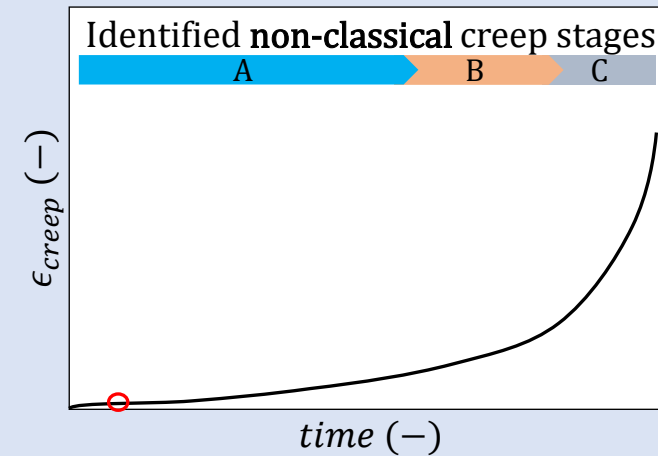
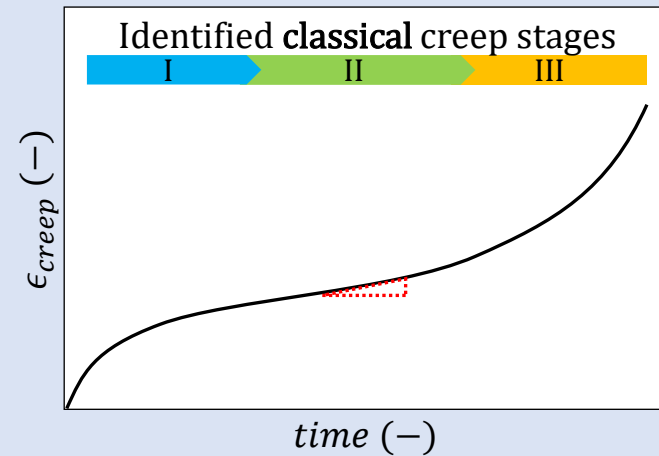
Low stress & high  $T^\circ$   
Short creep test | inert gas test

Low stress & high  $T^\circ$   
Long creep test | non-inert gas test

Creep strain rate  
v/s  
Creep strain



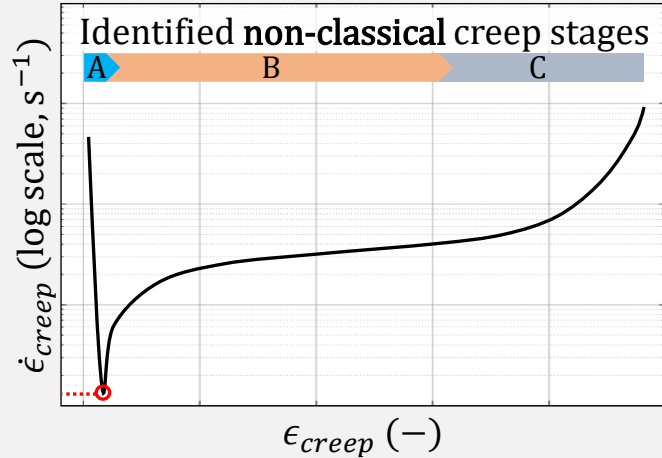
Creep strain  
v/s  
time



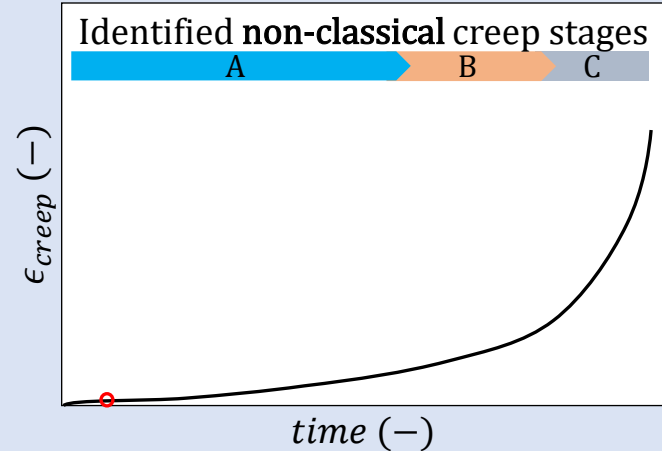
## Creep test

Low stress & high  $T^\circ$   
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Creep strain rate  
v/s  
Creep strain

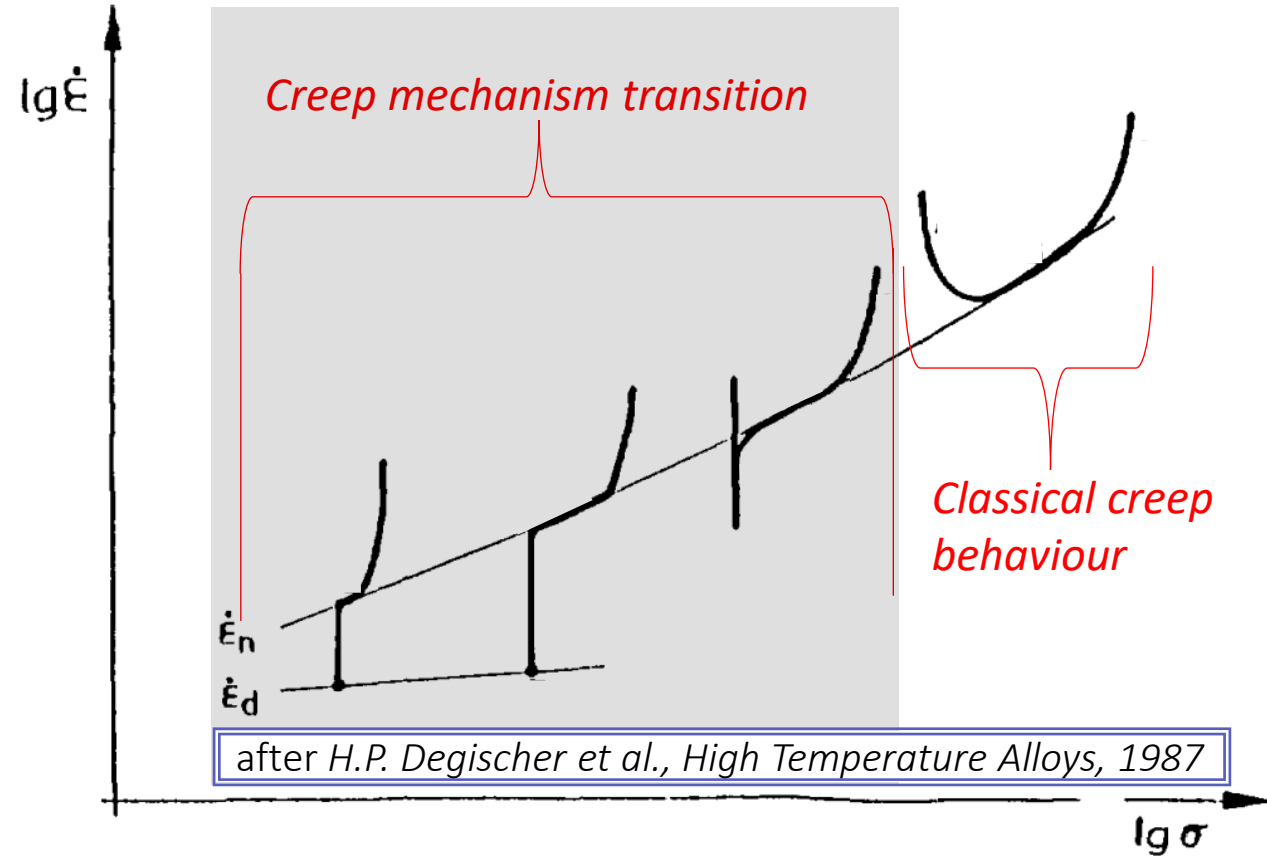


Creep strain  
v/s  
time



## Explanation: creep mechanism transition

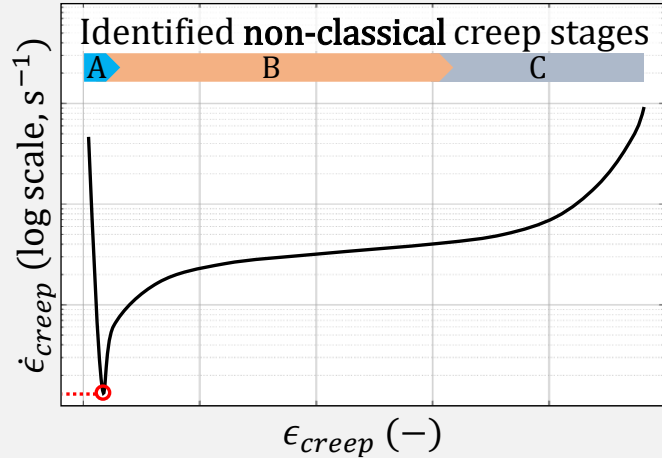
### Qualitative creep rate – stress relation at 800°C



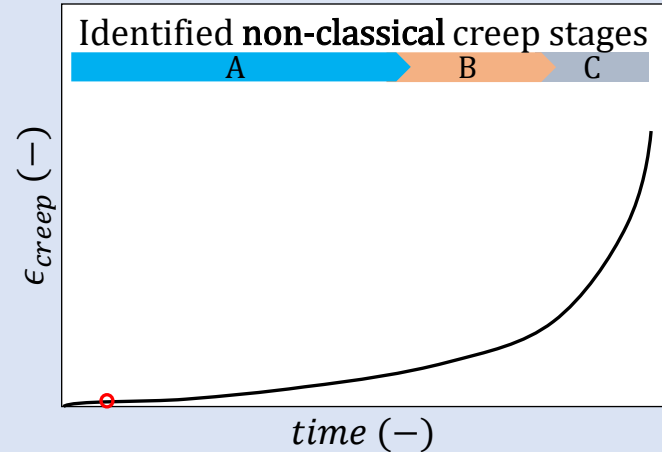
## Creep test

Low stress & high  $T^\circ$   
Short creep test | inert gas test

Creep strain rate  
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Creep strain

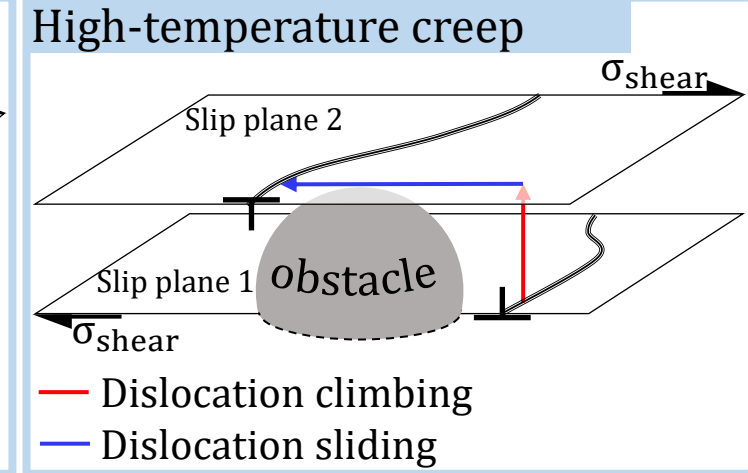
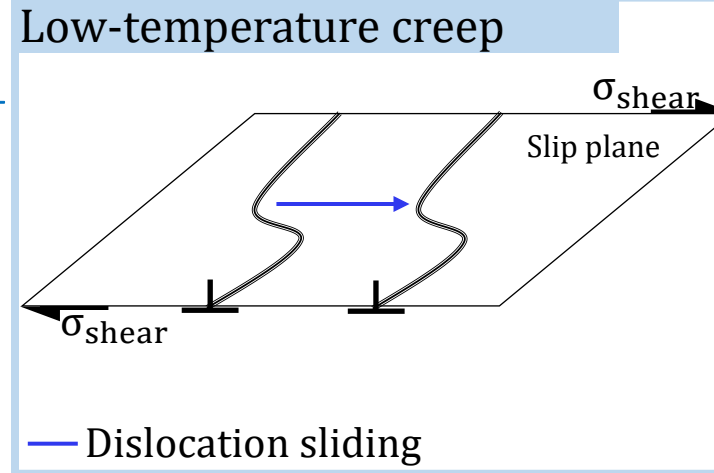


Creep strain  
v/s  
time

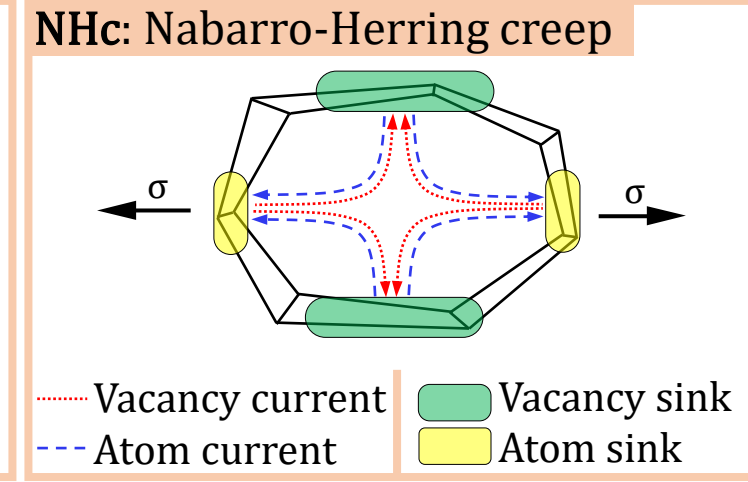
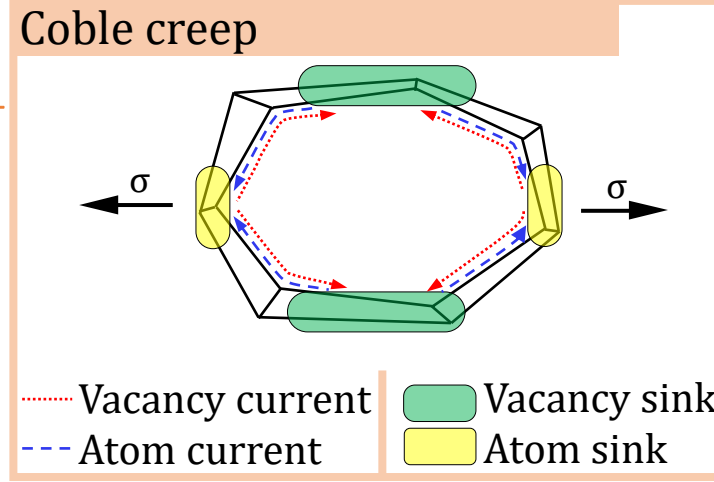


## Explanation: creep mechanism transition

A  
Dislocation-driven creep



B  
Diffusion-driven creep

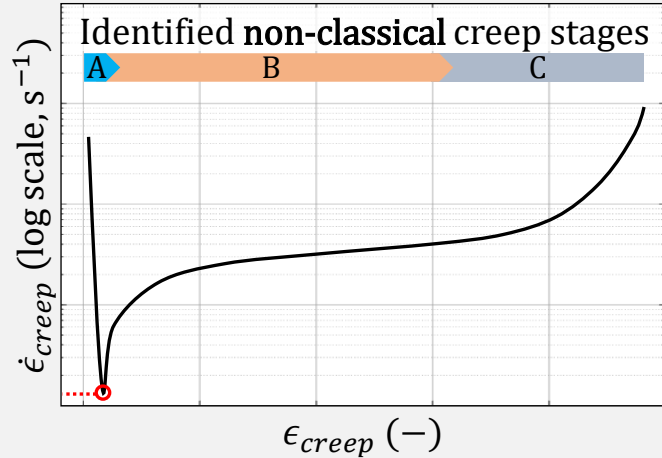


## Creep test

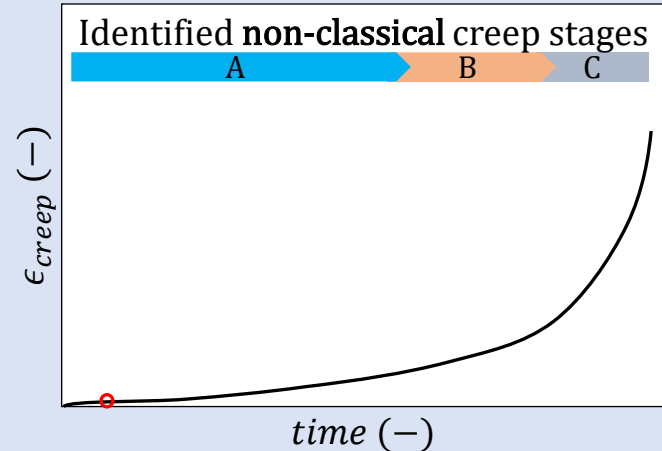
Low stress & high  $T^\circ$

Short creep test | inert gas test

Creep strain rate  
v/s  
Creep strain



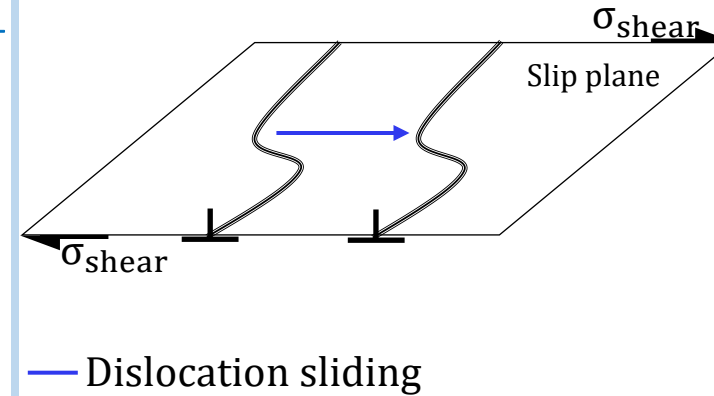
Creep strain  
v/s  
time



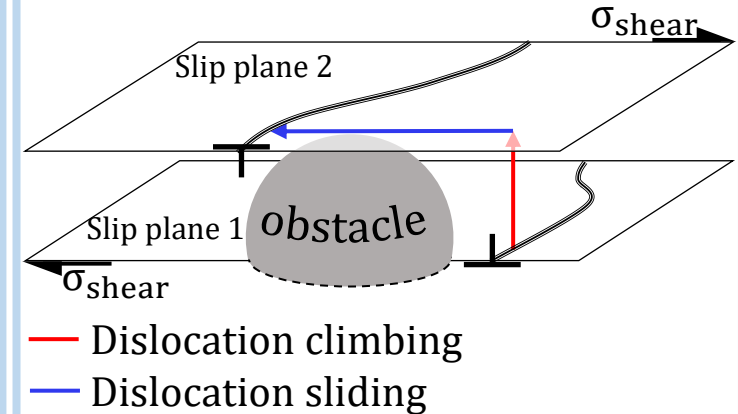
## Explanation: creep mechanism transition

### Low-temperature creep

A  
Dislocation-driven creep

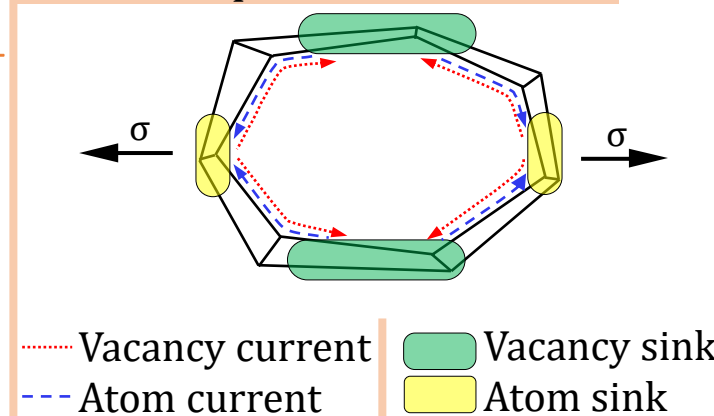


### High-temperature creep



### Coble creep

B  
Diffusion-driven creep



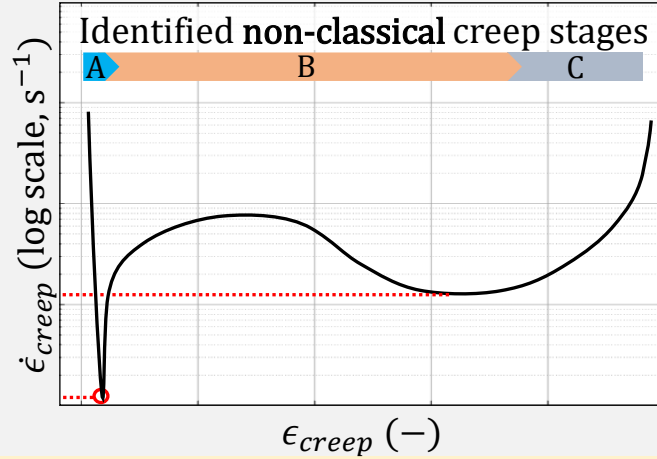
According to [\*], intergranular diffusion (i.e., Coble creep) is the preferential diffusion-driven creep mechanism for 800H

\*: after L.A. Beardsley et al., Metall. Mater. Trans. A, 2019

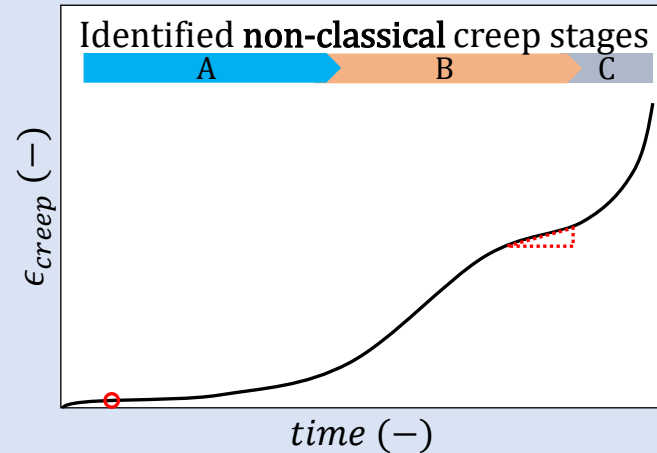
## Creep test

Low stress & high  $T^\circ$   
Long creep test | non-inert gas test

Creep strain rate  
v/s  
Creep strain

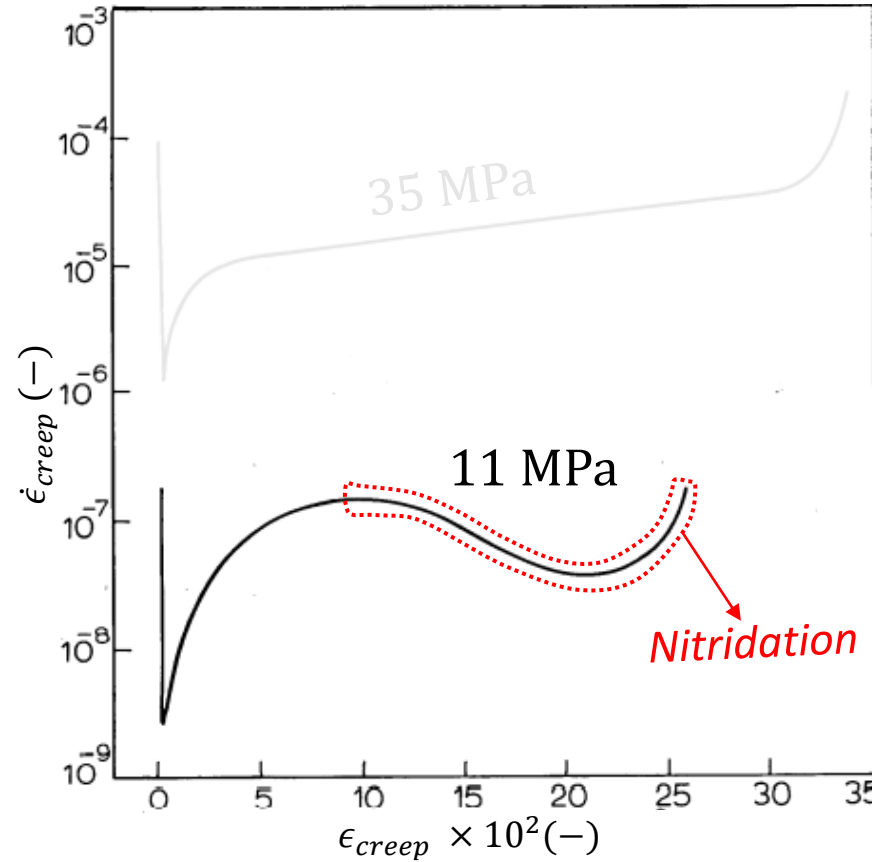


Creep strain  
v/s  
time



Explanation: Possible nitridation-induced creep hardening

## 1000°C creep-creep rate curves



Curves & micrograph after V. Guttman & R. Bürigel, Metal Science, 1983



## Finite Element numerical modelling of 800H alloy creep behaviour

### Lagamine: Our finite element software



Developed since 1980s

- Large & small deformations
- Applied to many processes & many material behaviours
- Elasticity, Thermal, **Viscosity**,...

Step 1: Use available Chaboche-type constitutive model [\*]

#### Yield function: von-Mises criterion

$$f_y = \Sigma_{VM}^{eq} - \sigma_y \leq 0$$

$$\left\{ \begin{array}{l} \tilde{\underline{\sigma}} = (1 - D)^{-1} \underline{\sigma} \quad \text{Effective stress (effect of damage)} \\ \underline{\dot{\mathbf{X}}} = \sum_{i=1}^n \underline{\dot{\mathbf{X}}}_{AF,i} + \underline{\dot{\mathbf{X}}}_{SR,i} \quad \text{Hardening \& Static Recovery} \\ \sigma_y = \sigma_0 + Q[1 - \exp(-b \cdot \bar{\epsilon}^p)] \quad \text{Voce isotropic hardening} \end{array} \right.$$

#### Damage functions

$$\dot{D} = \dot{D}_f + \dot{D}_c$$

$$\left\{ \begin{array}{l} \dot{D}_f = \left[ \frac{Y(\sigma)}{S_{f1}} \right]^{S_{f2}} \dot{\bar{\epsilon}}^p \\ \dot{D}_c = \left[ \frac{Y(\sigma^d)}{S_{c1}} \right]^{S_{c2}} \frac{1}{(1 - D)^k} \end{array} \right.$$

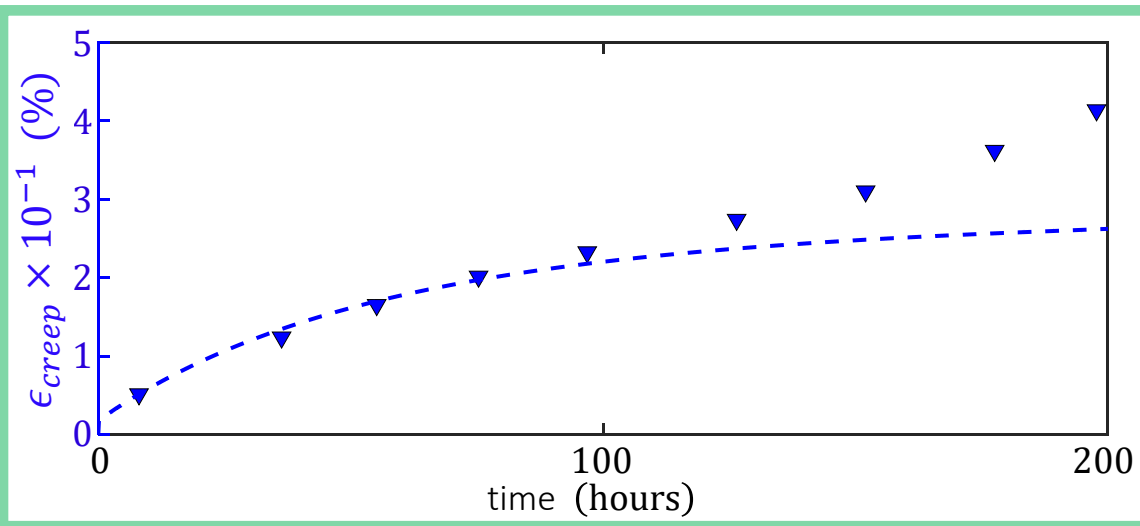
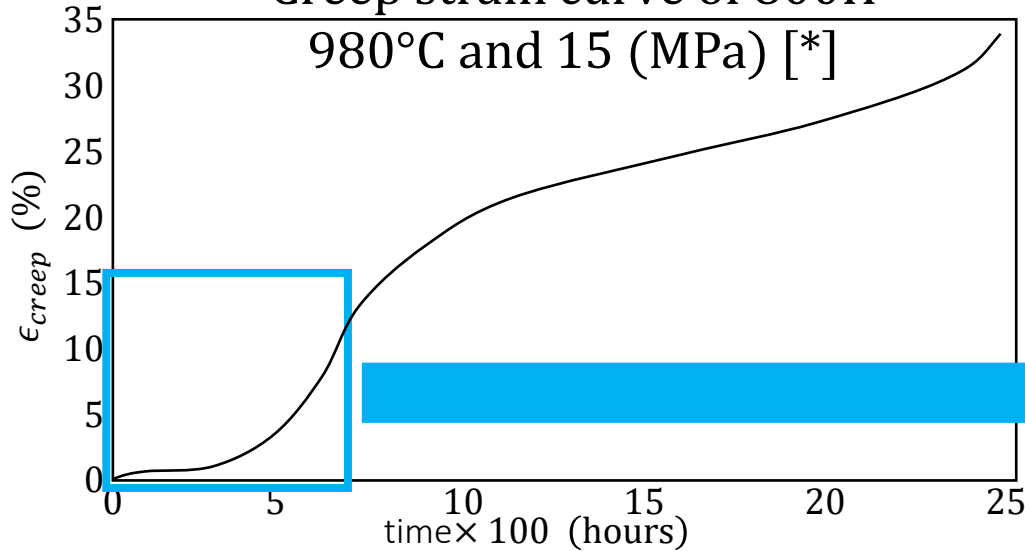
#### Viscoplastic function: Norton power law

$$\dot{\bar{\epsilon}}^p = \left\langle \frac{f_y}{K} \right\rangle^N \iff f_y = J_2(\tilde{\underline{\sigma}} - \underline{\mathbf{X}}) - \sigma_y - K(\bar{\epsilon}^p)^{1/N} \leq 0$$

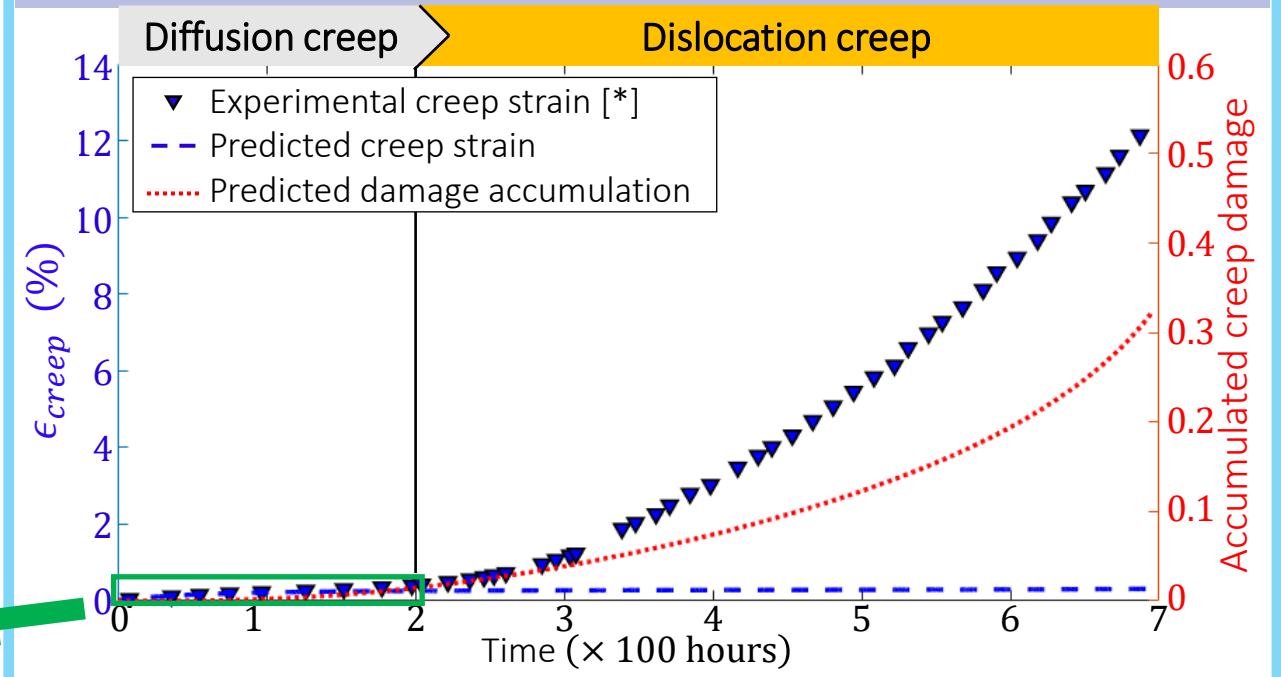
\*: Work after:

J.L. Chaboche, IJP, 2008; R. Ahmed et al., IJSS, 2016; R. Ahmed et al., IJSS, 2017;  
H. Morch et al., COMPLAS, 2017; H. Morch et al., EJM: A/Solids, 2021; H. Morch et al., FE in A&D, 2022)

Creep strain curve of 800H  
980°C and 15 (MPa) [\*]



## First modelling attempt, Chaboche-type law



\*: experimental curve after *B. Gardiner, PhD. thesis, U. Canterbury, 2014*

➤ We must implement a macromechanical model capable of predicting non-classical creep behaviour observed in 800H alloy

## Sample preparation

Smooth bar 800H samples

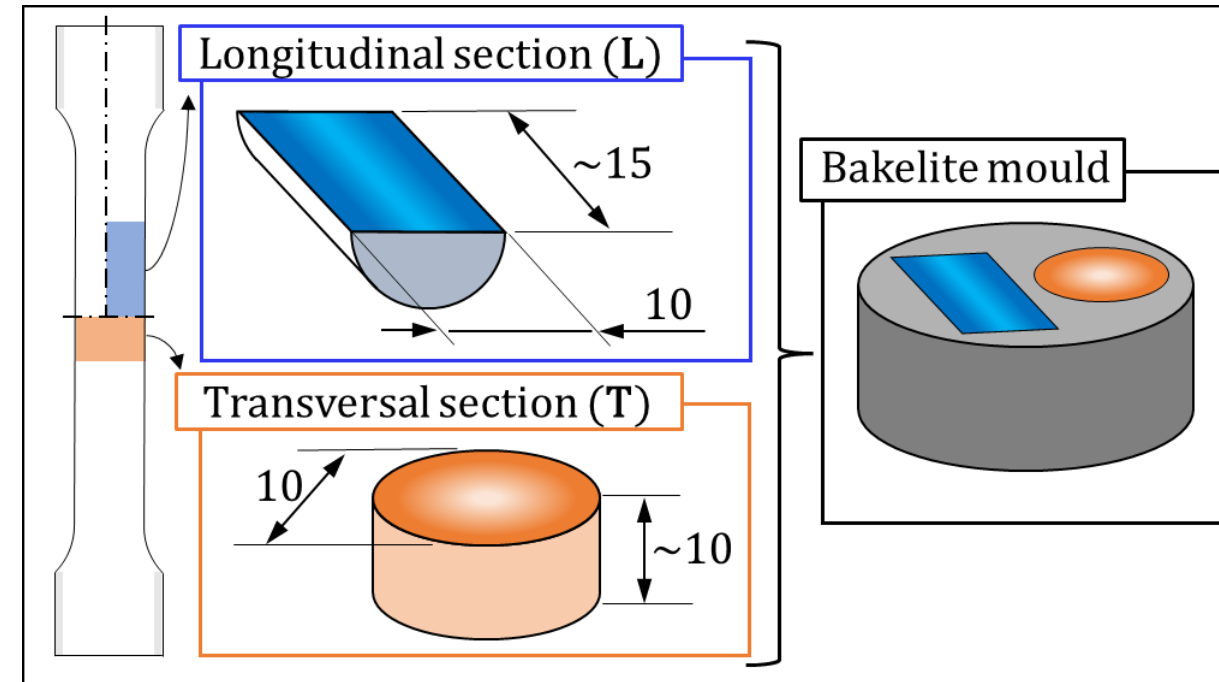
Geometries {  
D05 5 [mm] diameter  
D10 10 [mm] diameter

**Industrial furnace**

Exposed to realistic environmental and thermal loadings

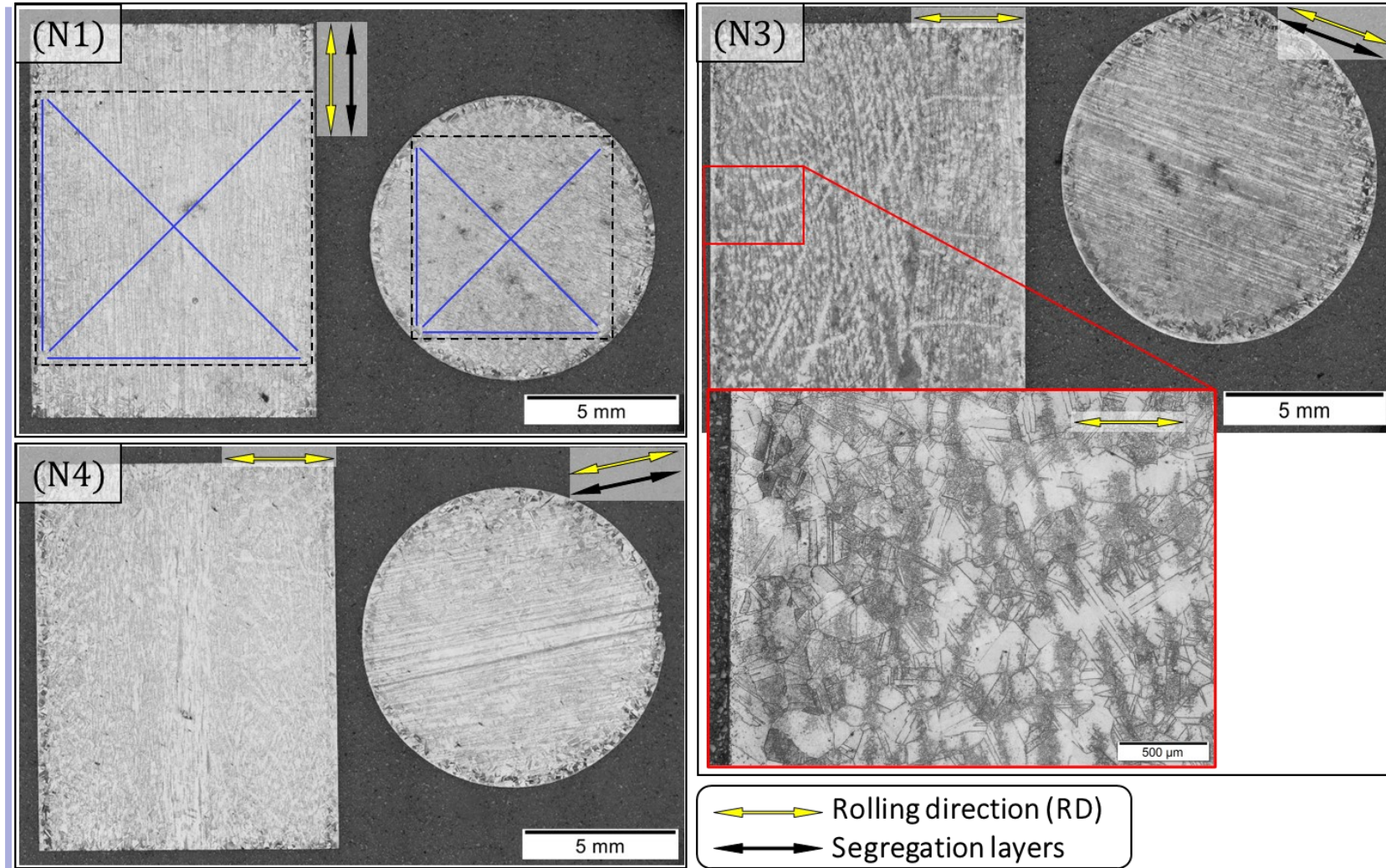
Tag	Description
N0	Sound material
N1	1 year in furnace
N2	2 years in furnace
N3	3 years in furnace
N4	4 years in furnace

1 D10 sample from each N-batch was selected to undergo microstructural characterization



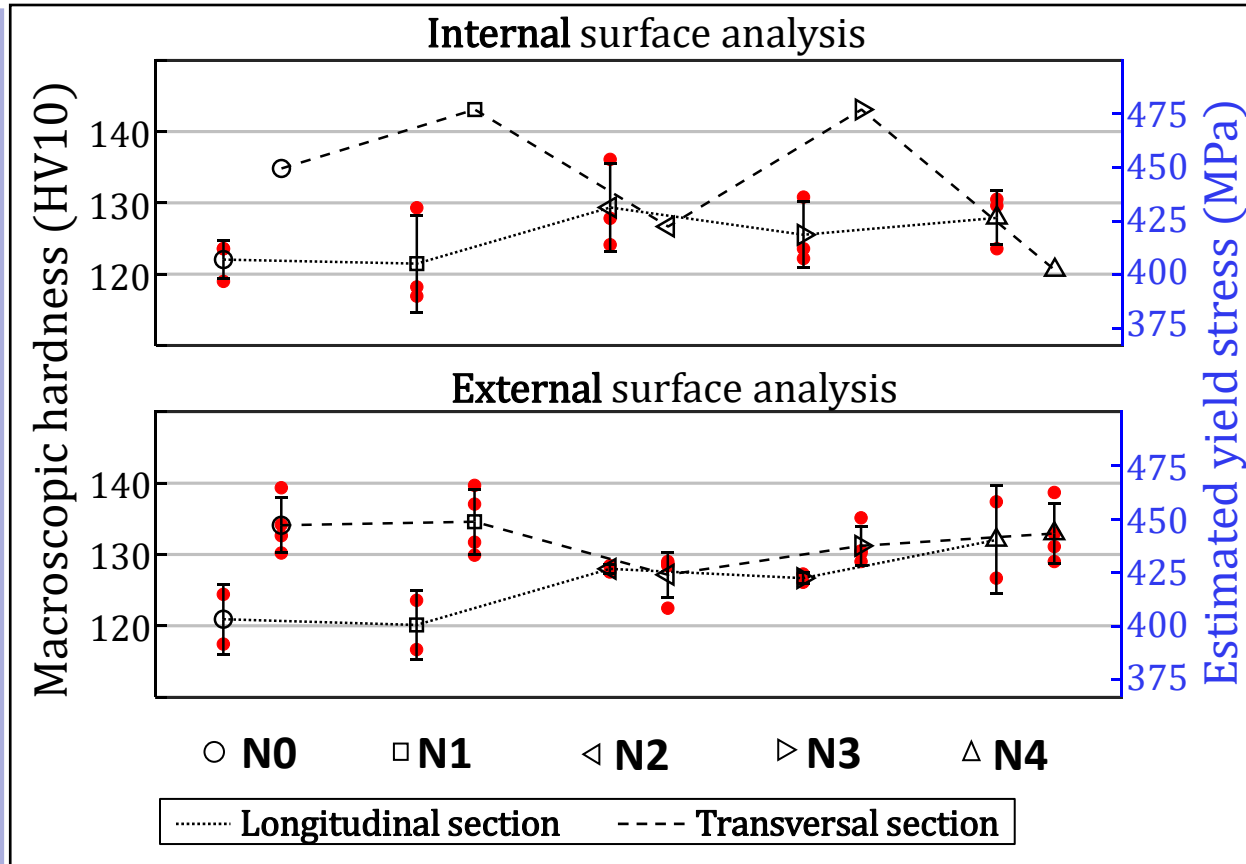
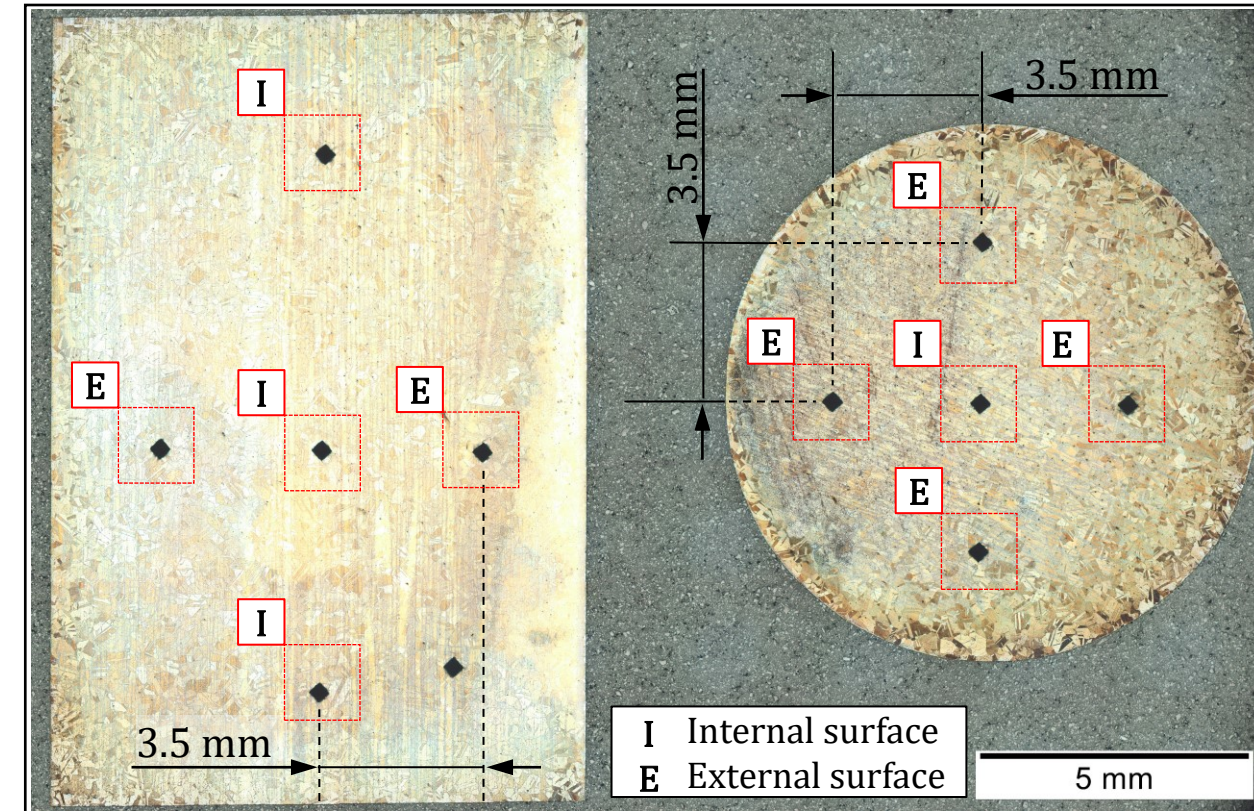
## Optical microscopy\* observations:

- AGS Gr.2 = 150 ( $\mu\text{m}$ )
- No visible external layer
- High precipitate segregation
  - Layers aligned to the RD
  - Dendritic-like segregation in N3



\*:Etching compound: Glyceregia

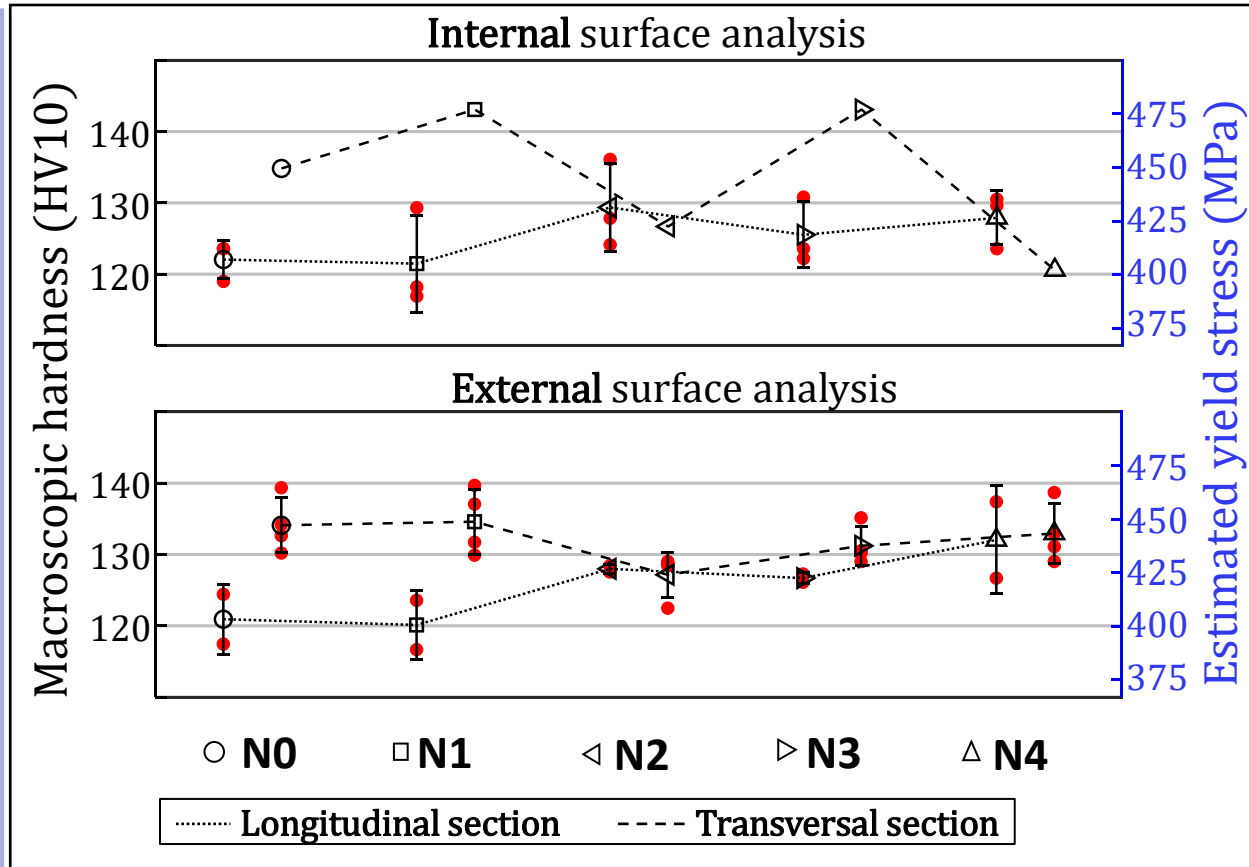
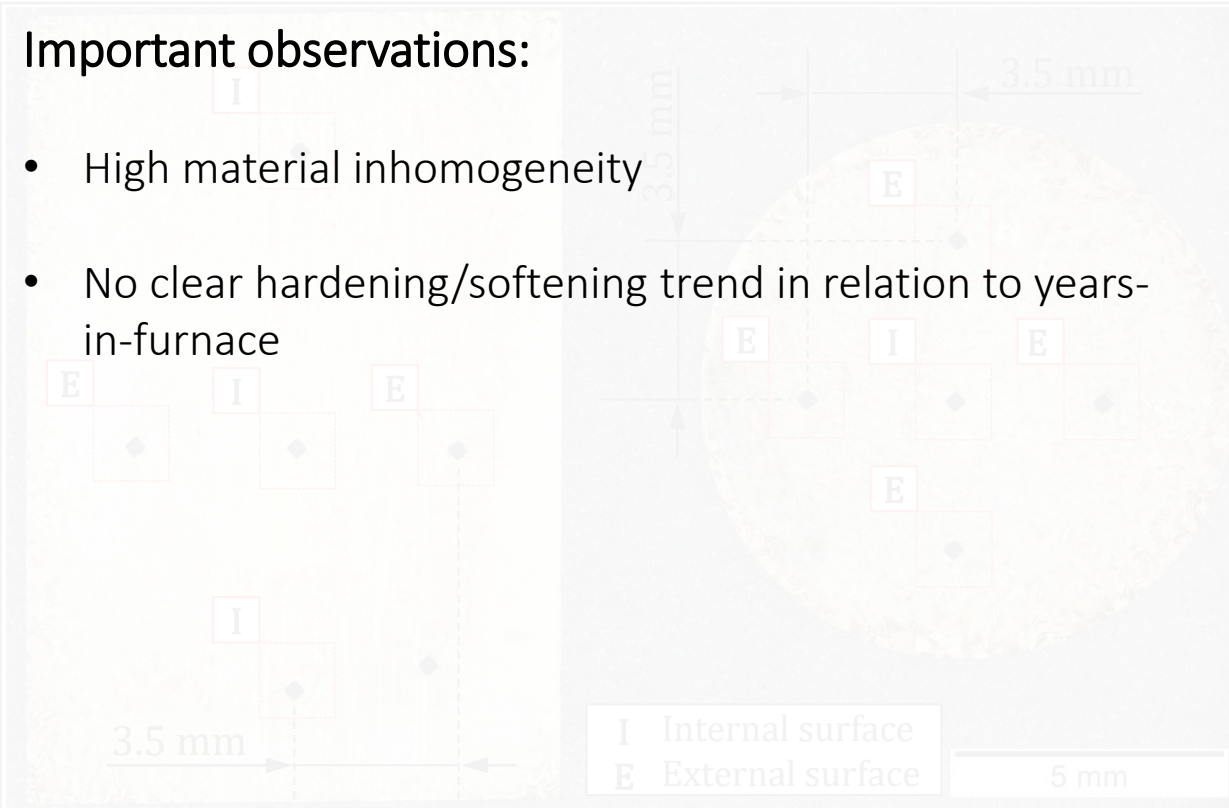
## Macro-indentation Vickers (HV10) observations:



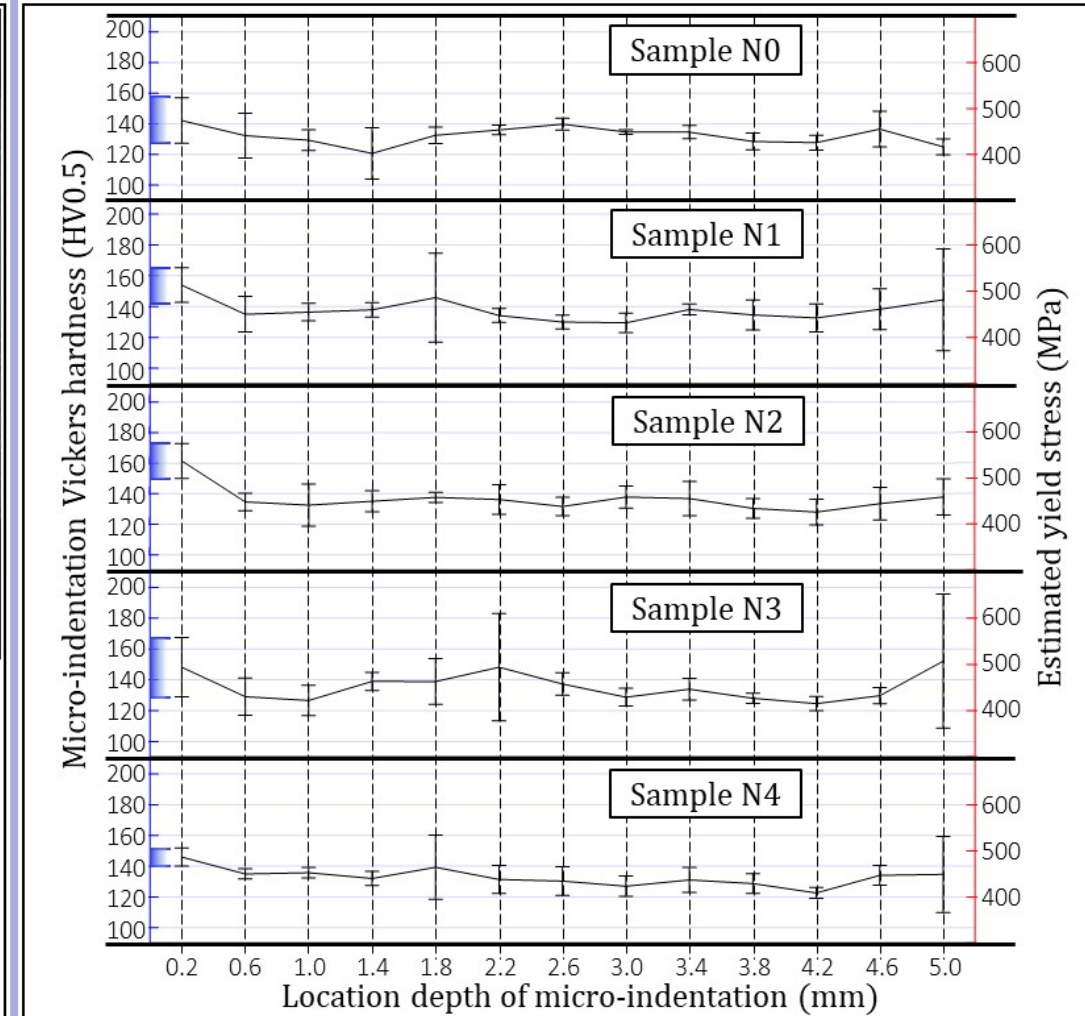
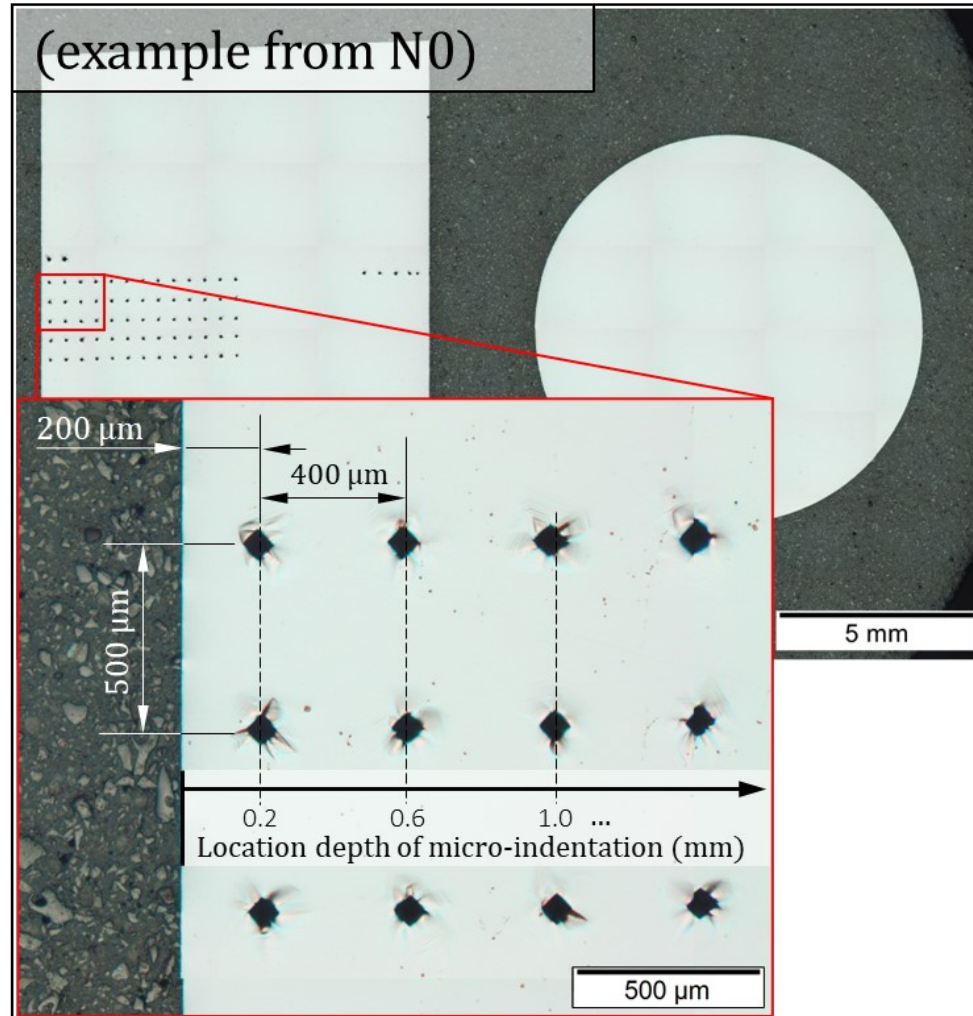
## Macro-indentation Vickers (HV10) observations:

### Important observations:

- High material inhomogeneity
- No clear hardening/softening trend in relation to years-in-furnace

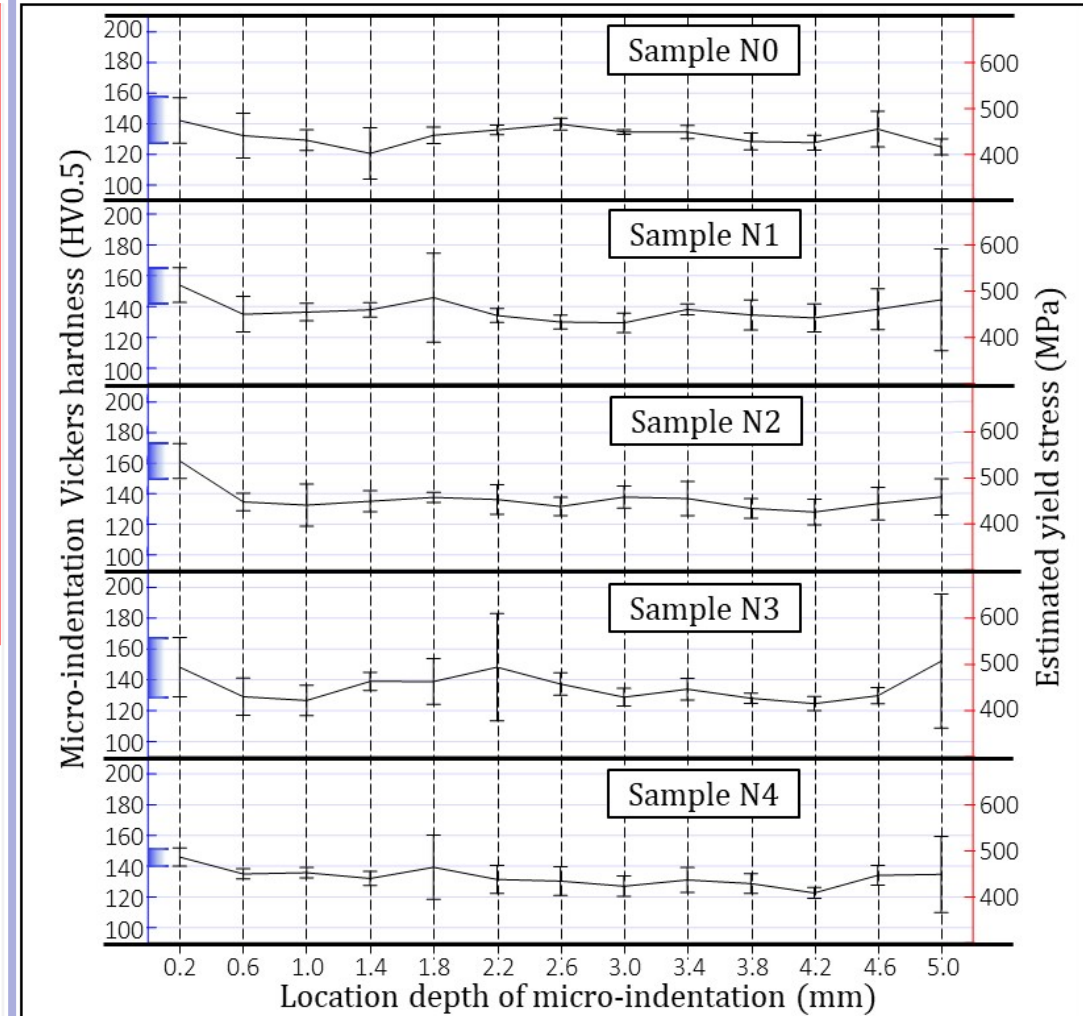
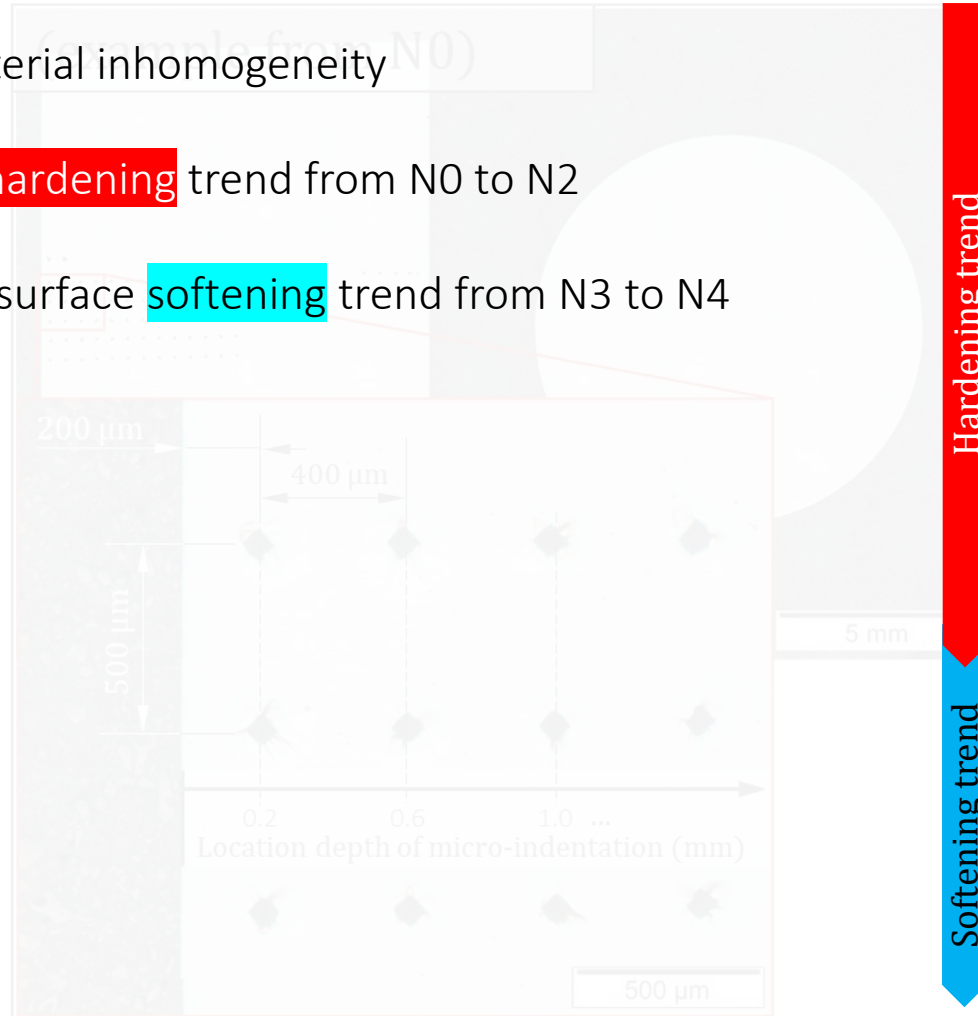


micro-indentation Vickers (HV0.5) observations:



## micro-indentation Vickers (HV0.5) observations:

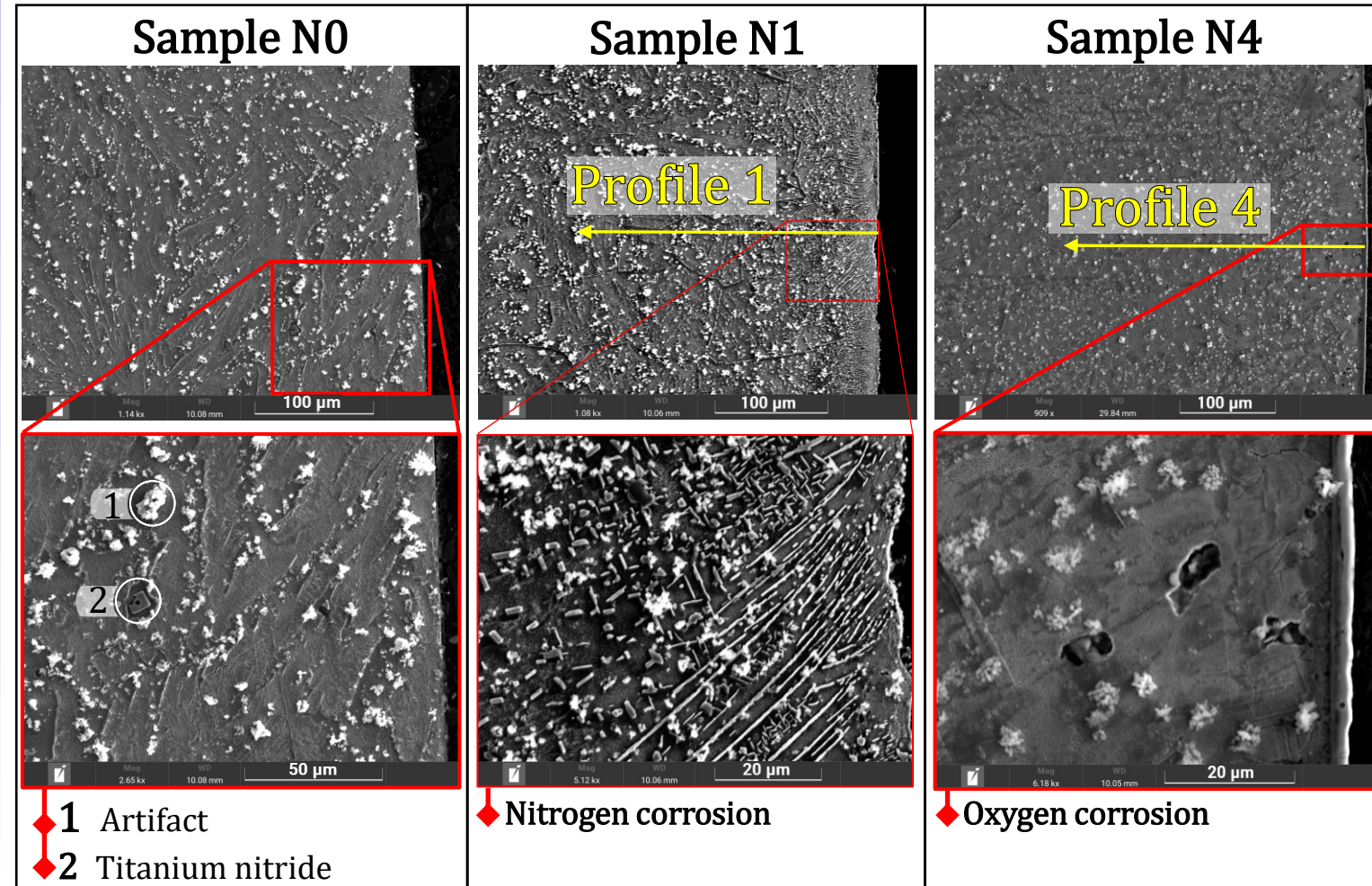
- High material inhomogeneity
- Surface **hardening** trend from N0 to N2
- External surface **softening** trend from N3 to N4





## SEM/EDX observations:

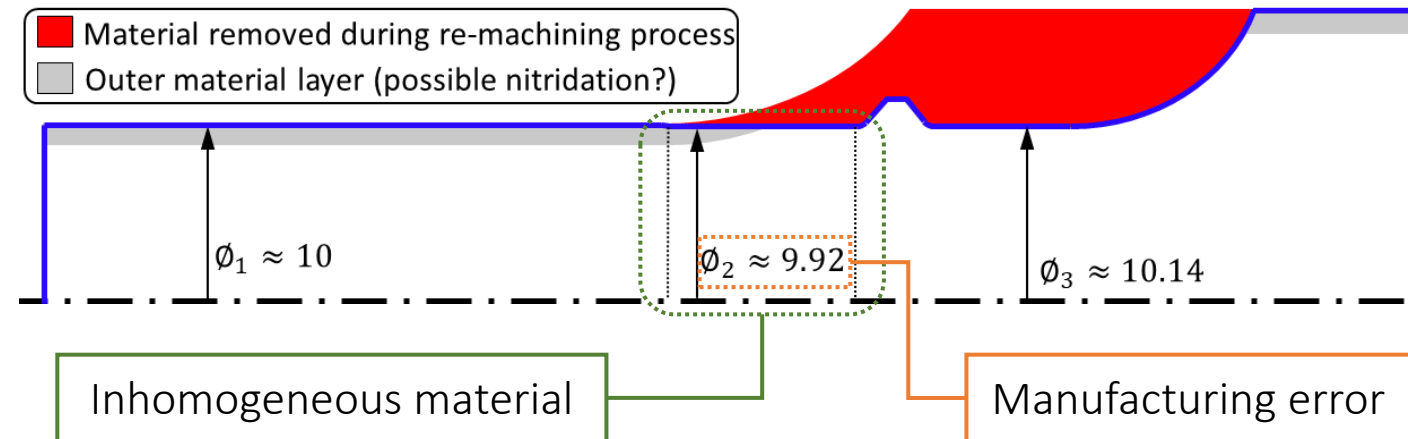
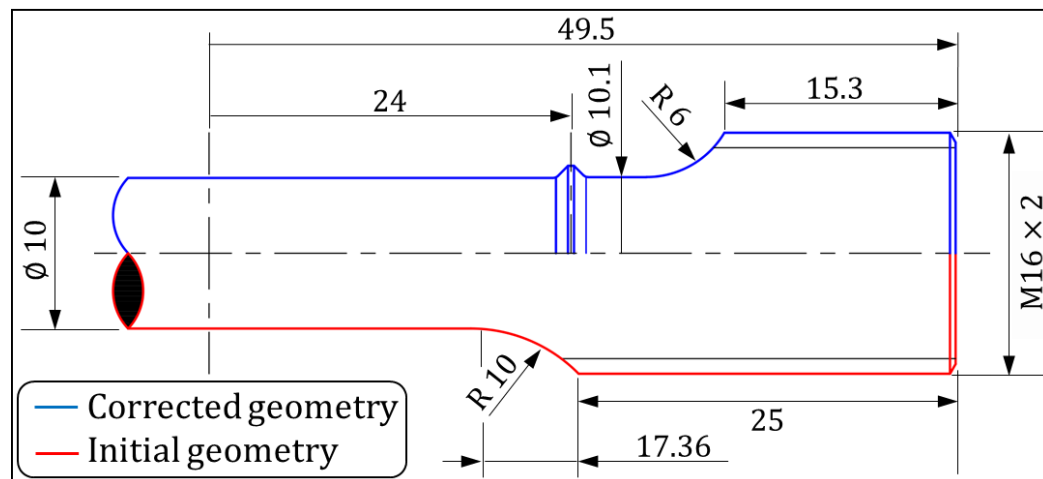
- Presence of artifacts
- Intergranular carbide precipitation
- Nitridation is dominant in sample N1
- Oxidation is dominant in sample N4



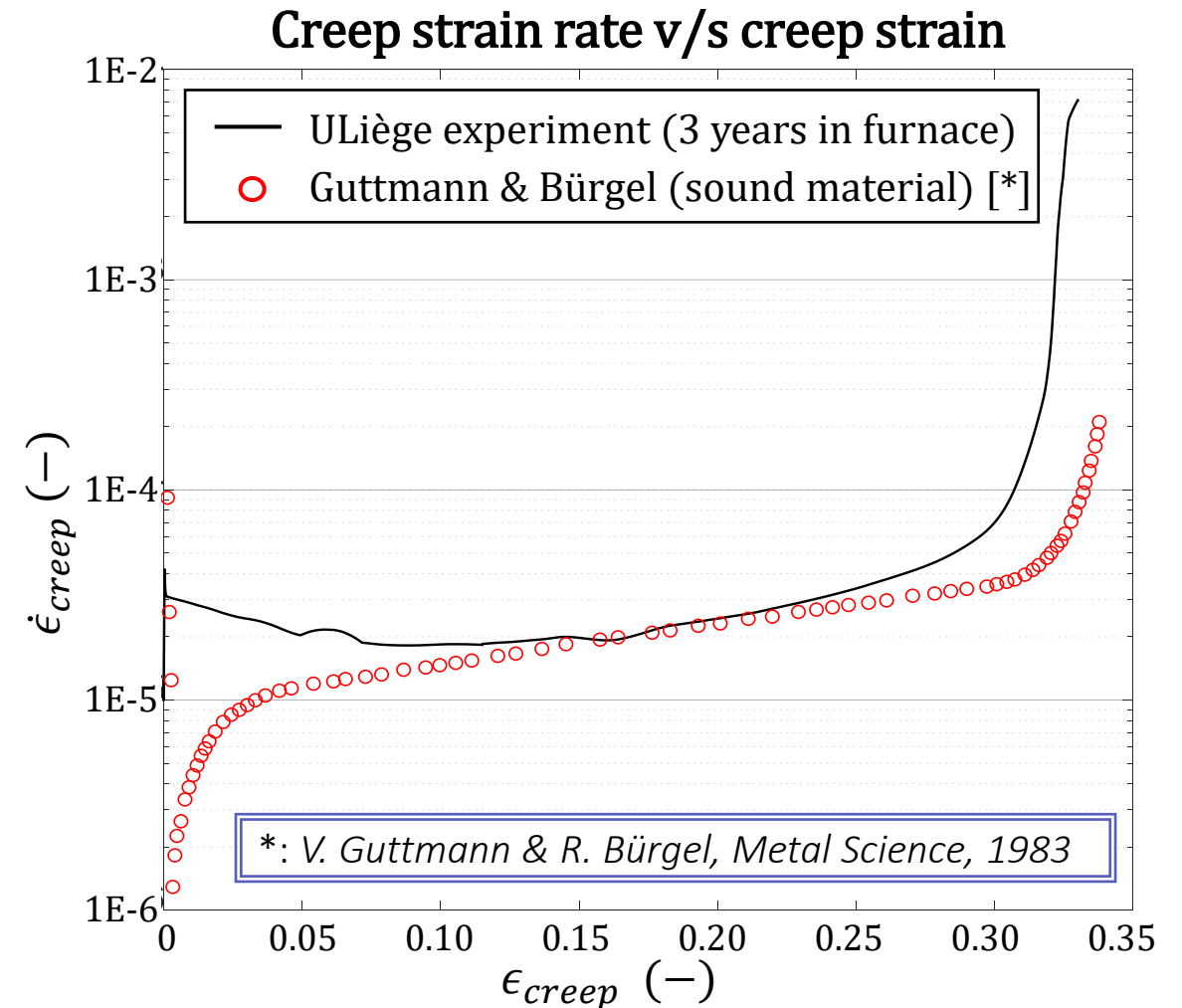
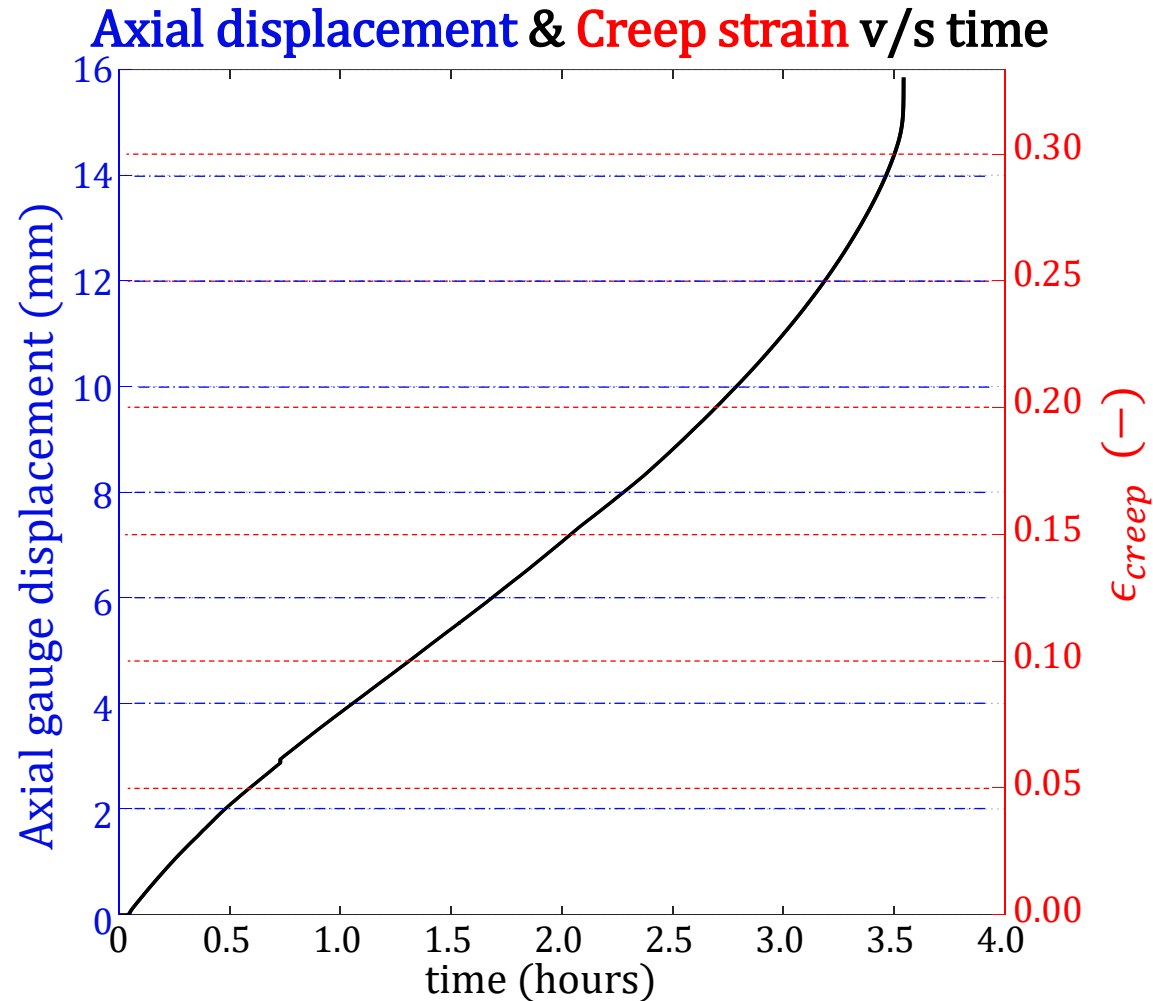
Samples had to be re-machined to include collars for the extensometer grip

... this is research...

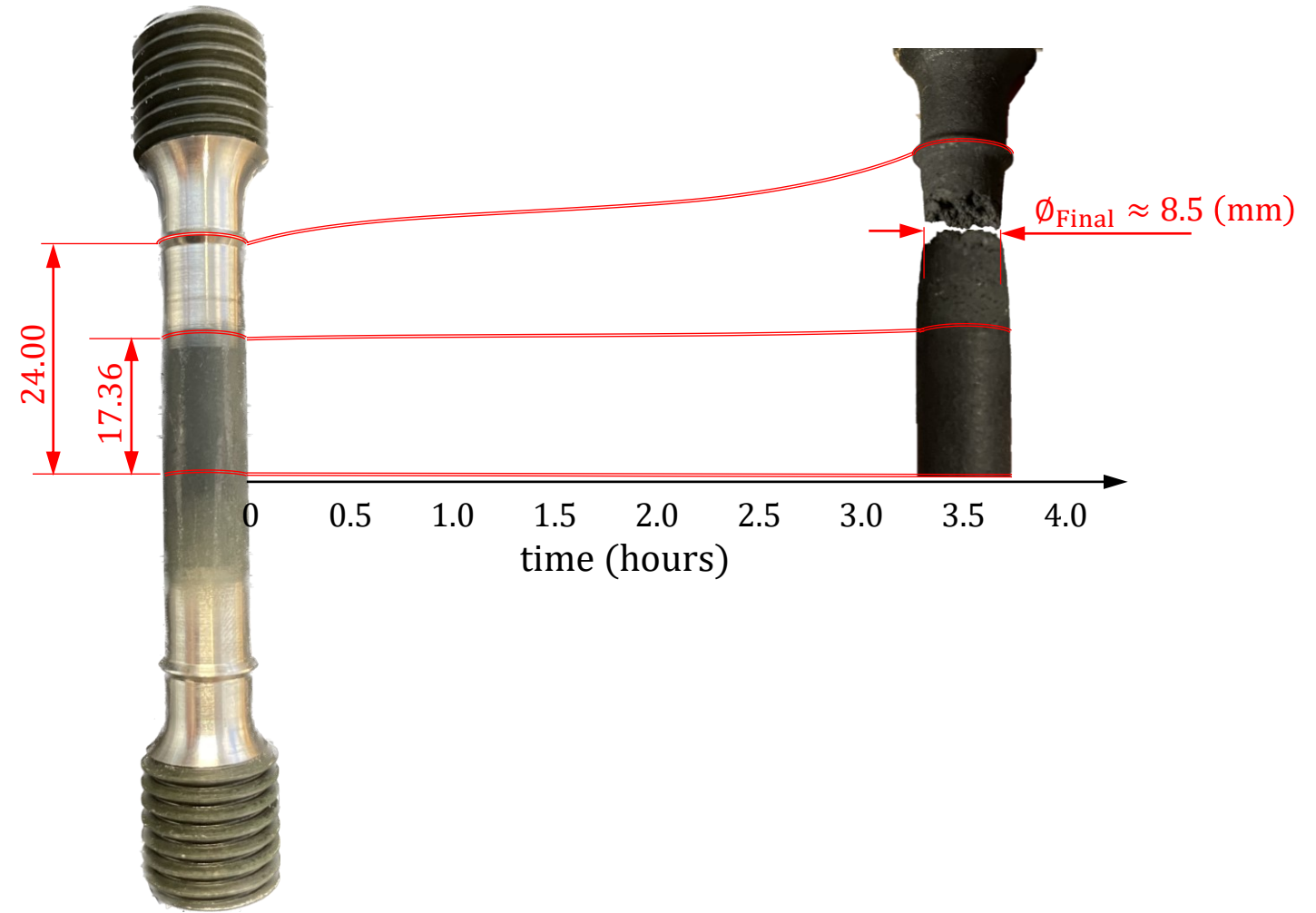
Original planimetry for geometry correction



Results for creep test at 1000°C and 35 [MPa] on N3 sample of 800H



Results for creep test at 1000°C and 35 [MPa] on N3 sample of 800H



## Yield function: von-Mises criterion

$$f_y = \Sigma_{VM}^{eq} - \sigma_y \leq 0$$

$$\left\{ \begin{array}{l} \underline{\tilde{\sigma}} = (1 - D)^{-1} \underline{\sigma} \quad \text{Effective stress (effect of damage)} \\ \underline{\dot{\mathbf{X}}} = \sum_{i=1}^n \underline{\dot{\mathbf{X}}}_{AF,i} + \underline{\dot{\mathbf{X}}}_{SR,i} \quad \text{Hardening \& Static Recovery} \\ \sigma_y = \sigma_0 + Q[1 - \exp(-b \cdot \bar{\epsilon}^p)] \quad \text{Voce isotropic hardening} \end{array} \right.$$

## Viscoplastic function: Norton power law

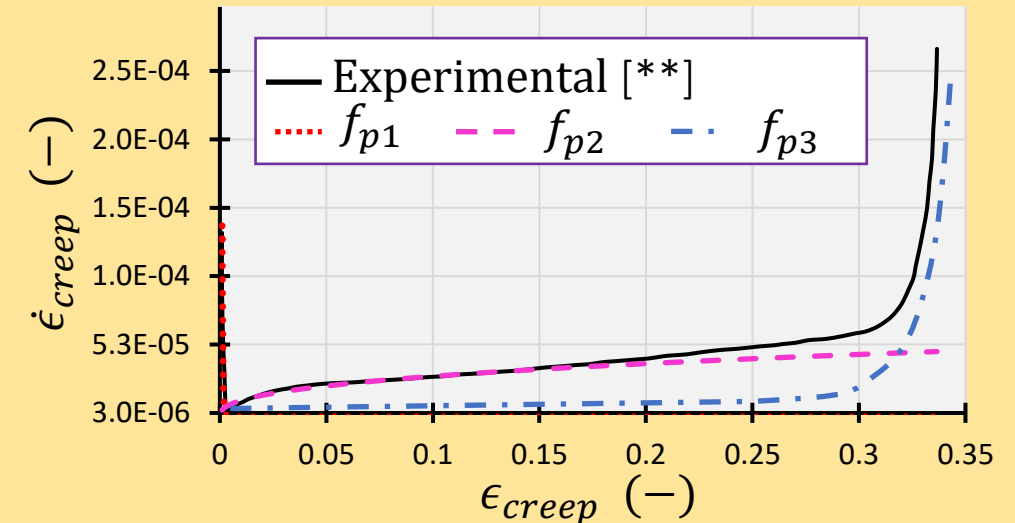
$$\dot{\bar{\epsilon}}^p = \left\langle \frac{f_y}{K} \right\rangle^N \iff f_y = J_2(\underline{\tilde{\sigma}} - \underline{\mathbf{X}}) - \sigma_y - K(\bar{\epsilon}^p)^{1/N} \leq 0$$

## Viscoplastic function: Graham-Walles approach [\*]

$$\dot{\bar{\epsilon}}^p = \sum_{j=1}^{vp_i} K_j e^{\bar{c}} [\Sigma_{VM}^{eq}]^{n_j} (\bar{\epsilon}^p)^{m_j} + K_T \sigma |\dot{T}| (\bar{\epsilon}^p)^{m_T}$$

$$\dot{\bar{\epsilon}}^p = f_{p1} + f_{p2} + f_{p3} + \dots + f_{pT}$$

1 Function  $\leftrightarrow$  1 phenomenon



\*\* : Curve after V. Guttman & R. Bürgel, Metal Science, 1983

## Damage function: Graham-Walles approach [\*]

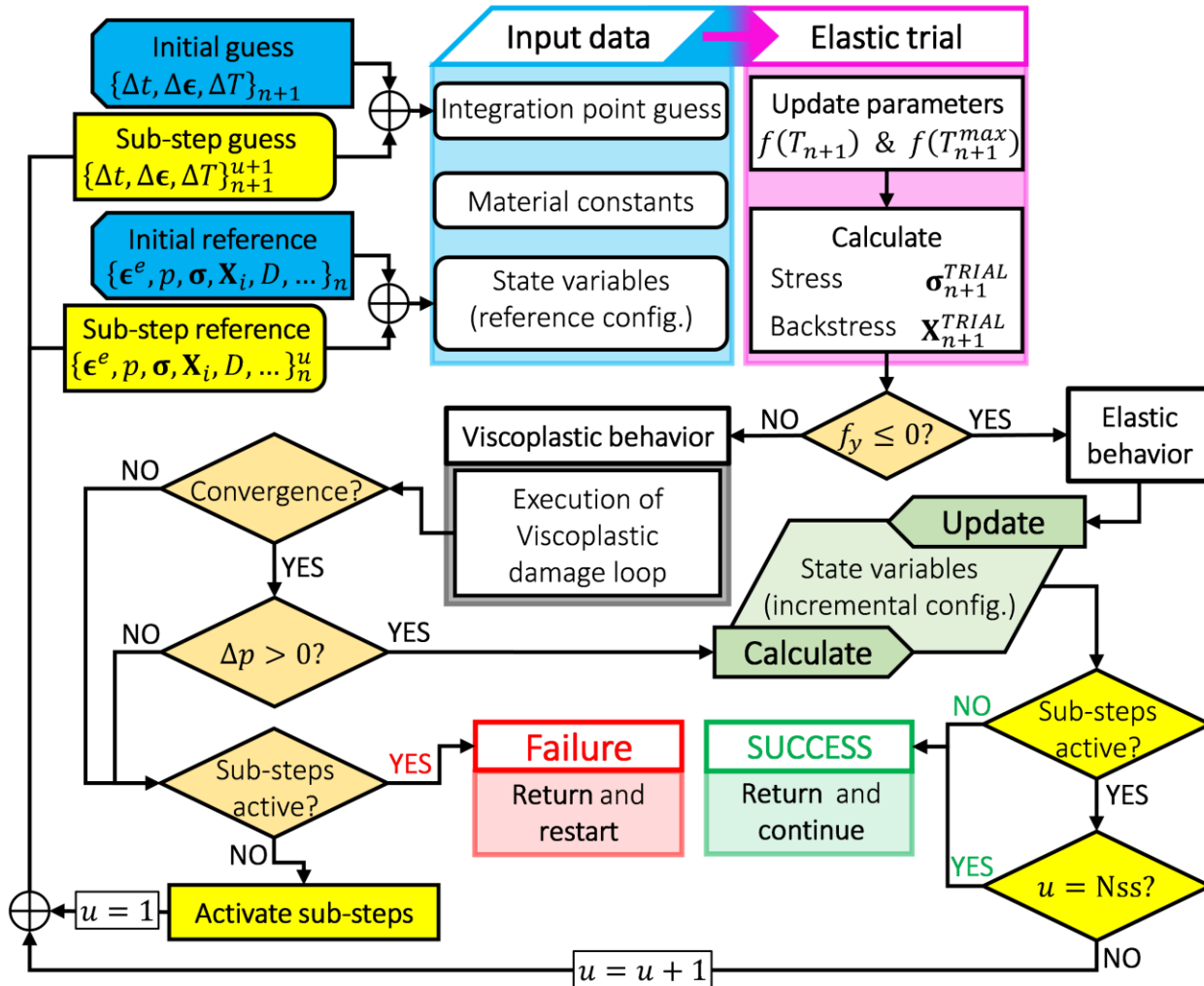
$$\dot{D} = \dot{D}_C + \dot{D}_f$$

$$\dot{D}_C = K_{TD} |\dot{T}| (\bar{\epsilon}^p)^{m_{TD}}$$

$$\dot{D}_f = K_D \Sigma_{VM}^{eq}$$

\* : Approach after N.K. Karthik, PhD. Thesis, RWTH Aachen & Darmstadt TU, 2016

## Constitutive law integration algorithm [\*]



## Viscoplastic behaviour: Fully implicit integration scheme

- Local residual matrix ( $\underline{\mathbb{R}}$ )

$$\underline{\mathbb{R}} = \begin{Bmatrix} \Delta \underline{\epsilon}^e \\ \Delta \bar{\epsilon}^p \\ \Delta \underline{\sigma} \\ \Delta \underline{X}_i \\ \Delta D \end{Bmatrix} \Rightarrow \begin{cases} \Delta \underline{\epsilon}^e = f(\Delta \bar{\epsilon}^p, \Delta \underline{\sigma}, \Delta \underline{X}_i, \Delta D) \\ \Delta \bar{\epsilon}^p = f(\Delta \bar{\epsilon}^p, \Delta \underline{\sigma}, \Delta \underline{X}_i, \Delta D) \\ \Delta \underline{\sigma} = f(\Delta \underline{\epsilon}^e, \Delta \underline{\sigma}, \Delta D) \\ \Delta \underline{X}_i = f(\Delta \bar{\epsilon}^p, \Delta \underline{\sigma}, \Delta \underline{X}_i, \Delta D) \\ \Delta D = f(\Delta \bar{\epsilon}^p, \Delta \underline{\sigma}, \Delta \underline{X}_i, \Delta D) \end{cases}$$

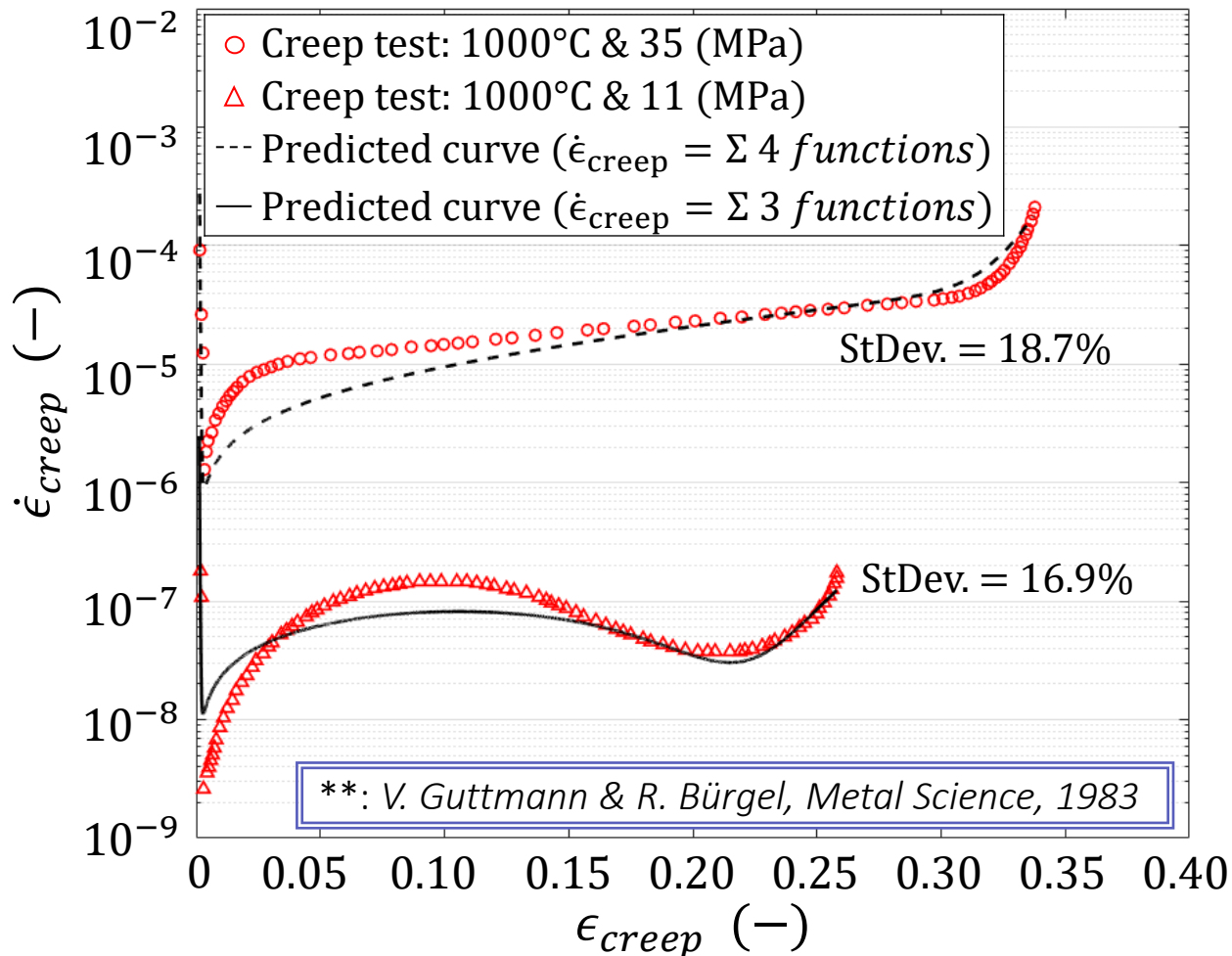
- Radial return mapping algorithm

$$\begin{Bmatrix} \Delta \underline{\epsilon}^e \\ \Delta \bar{\epsilon}^p \\ \Delta \underline{\sigma} \\ \Delta \underline{X}_i \\ \Delta D \end{Bmatrix}_{n+1}^{k+1} = \begin{Bmatrix} \Delta \underline{\epsilon}^e \\ \Delta \bar{\epsilon}^p \\ \Delta \underline{\sigma} \\ \Delta \underline{X}_i \\ \Delta D \end{Bmatrix}_{n+1}^k - [inv(J\{\underline{R}\}) \cdot \underline{R}]_{n+1}^k$$

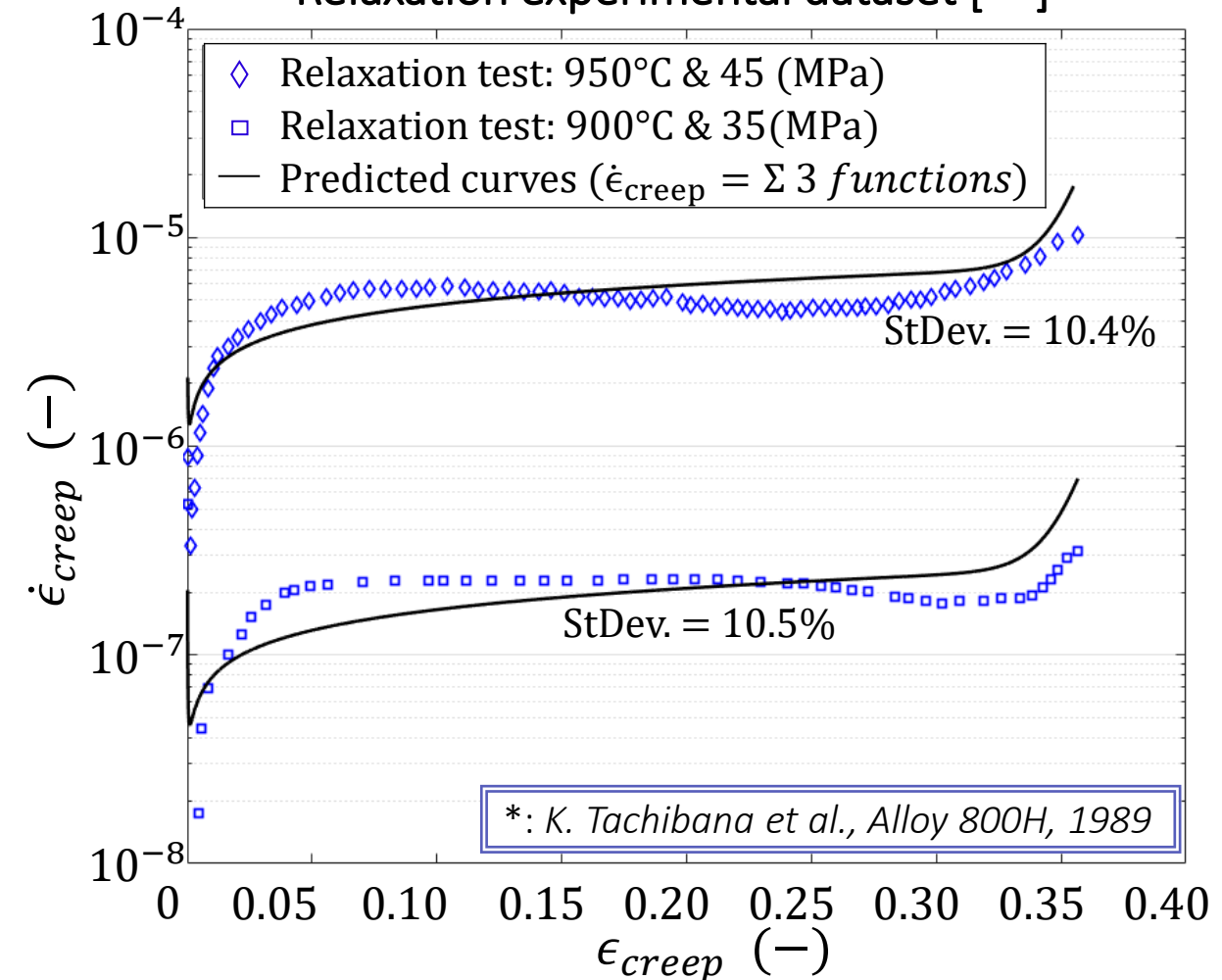
\*: Details in C. Rojas-Ulloa et al., Comput. Math. With Appl., 2023?

## Validation of the Chaboche-type + Graham-Walles viscoplastic function | 1 Finite Element

Creep experimental dataset [\*]

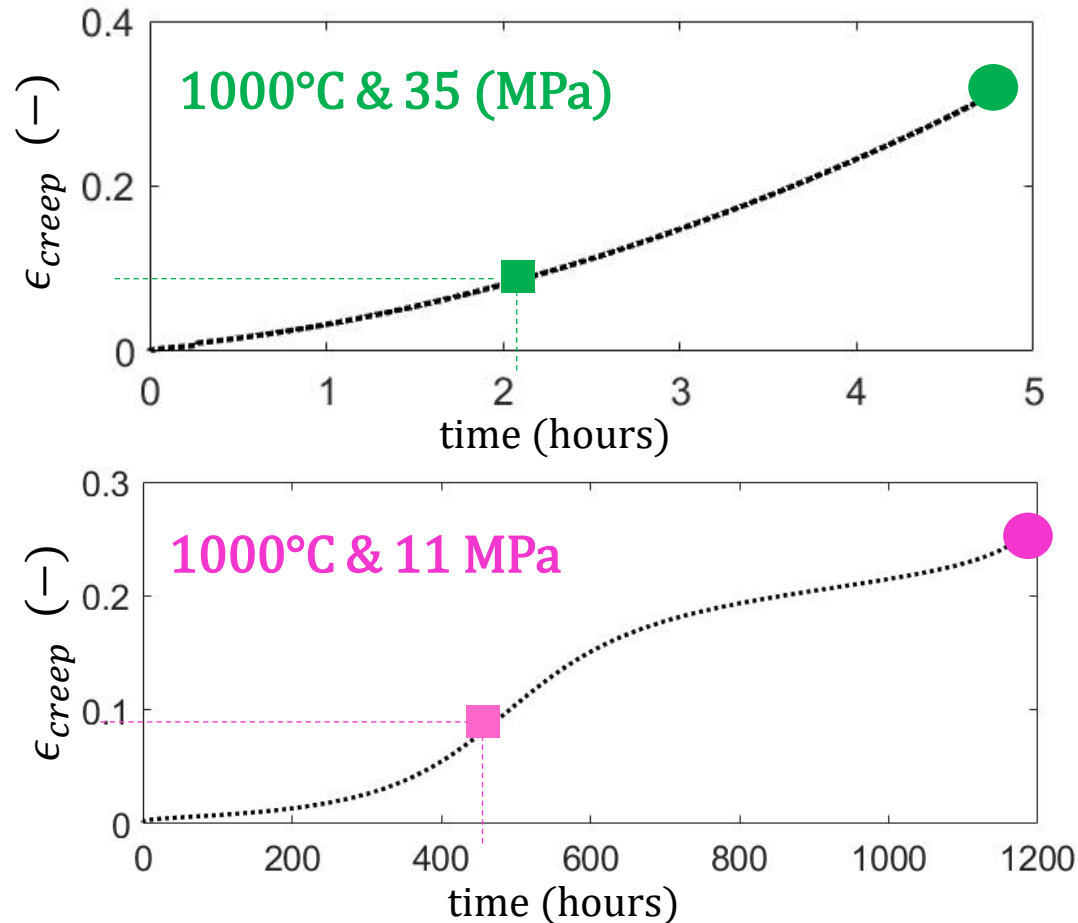


Relaxation experimental dataset [\*\*]



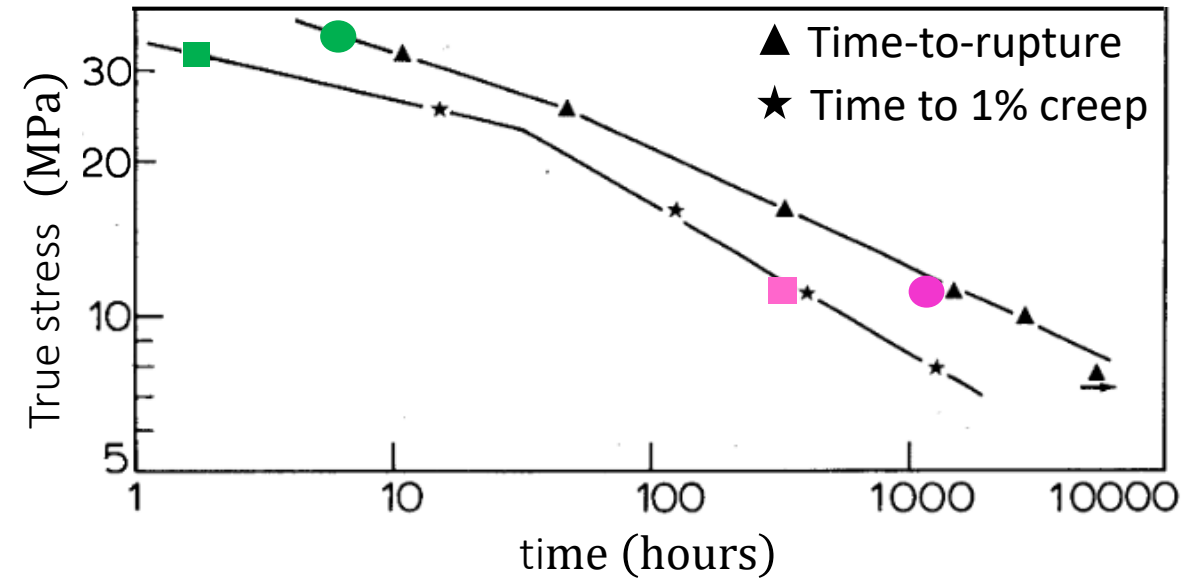
## Validation of the Chaboche-type + Graham-Walles viscoplastic function | 1 Finite Element

Numerical creep v/s time curves for 800H [\*]



Results are found within experimental limits

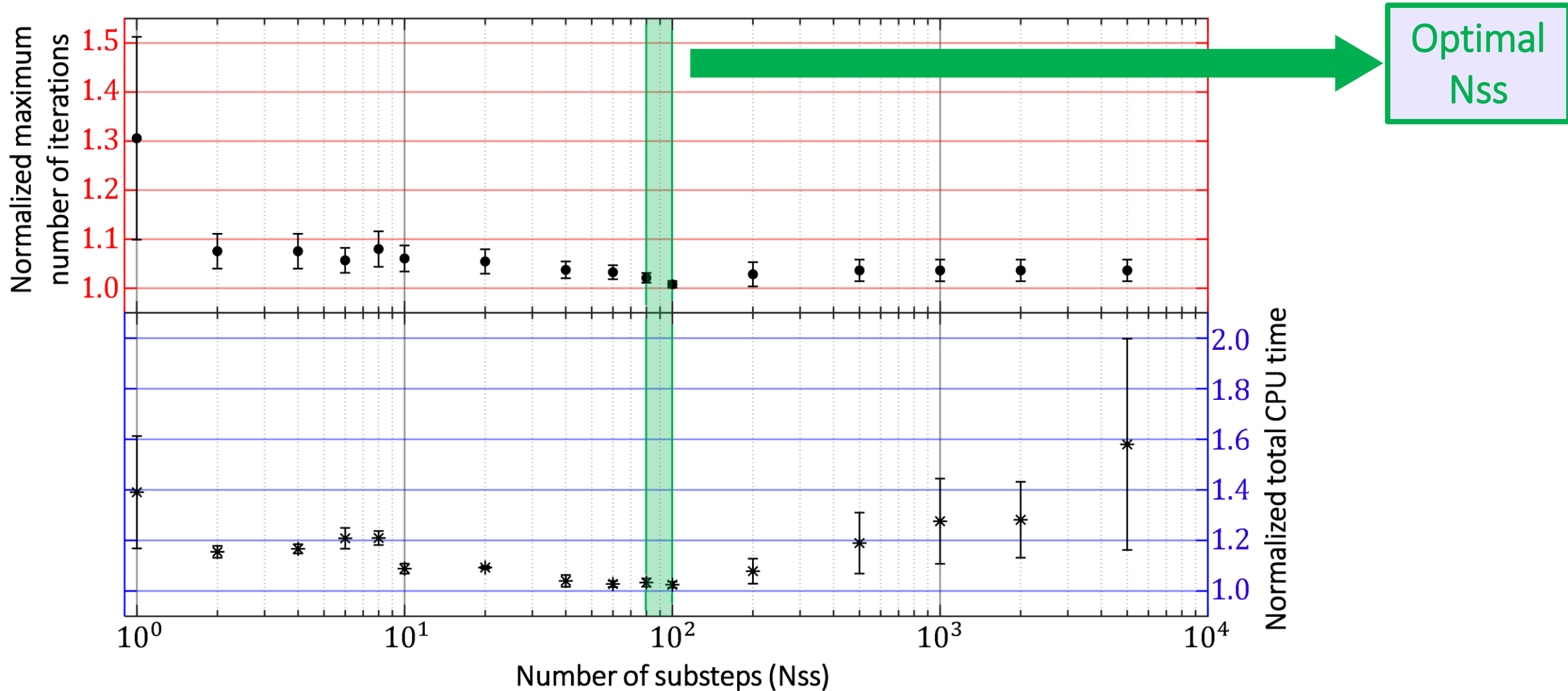
Creep rupture strength & time-to-1% creep [\*]



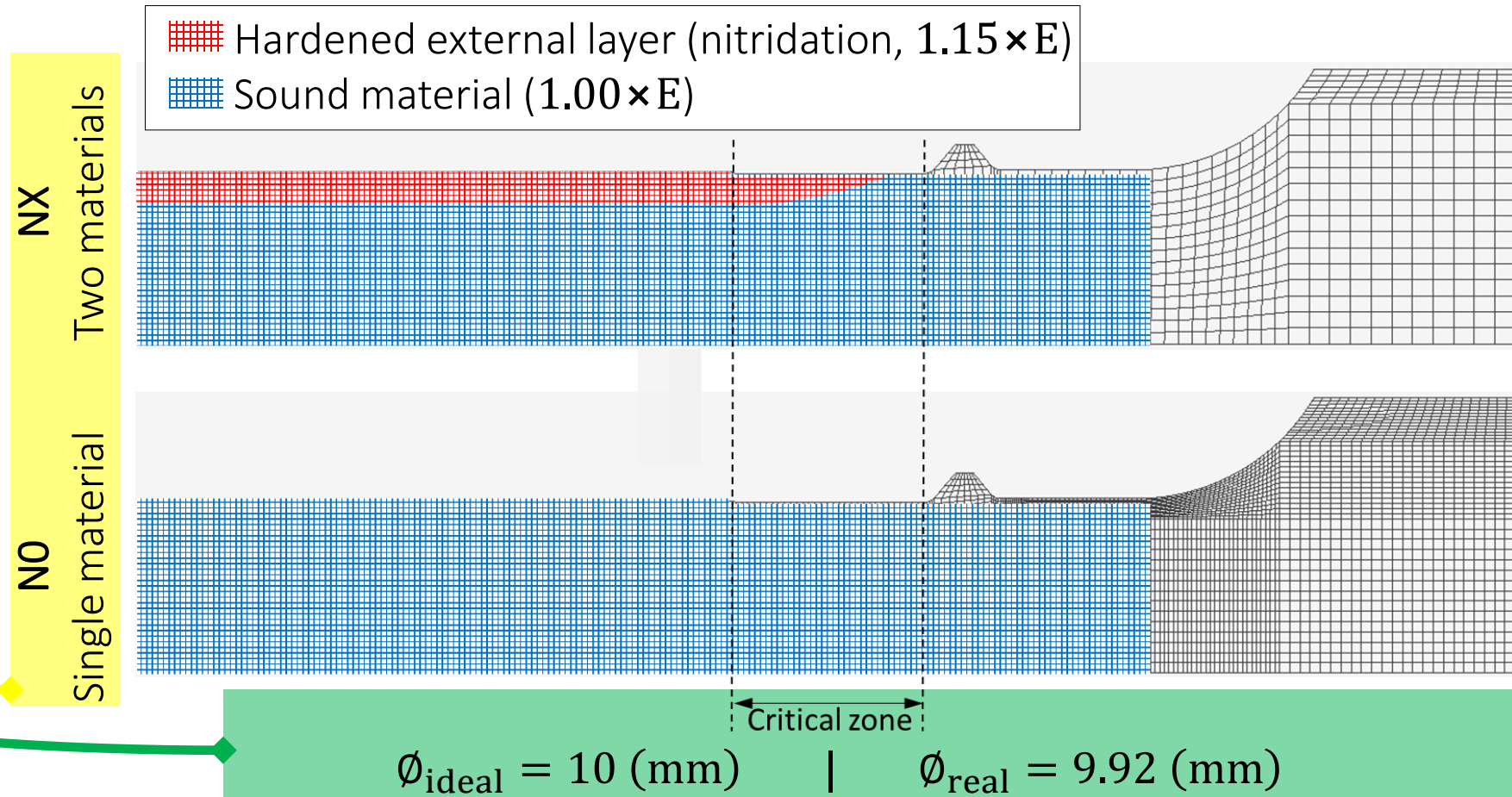
\*: V. Guttman & R. Bürgel, Metal Science, 1983



## Assessment of computational efficiency | 1 Finite Element



## ❖ Why did our samples failed this way?



Two theories were addressed:

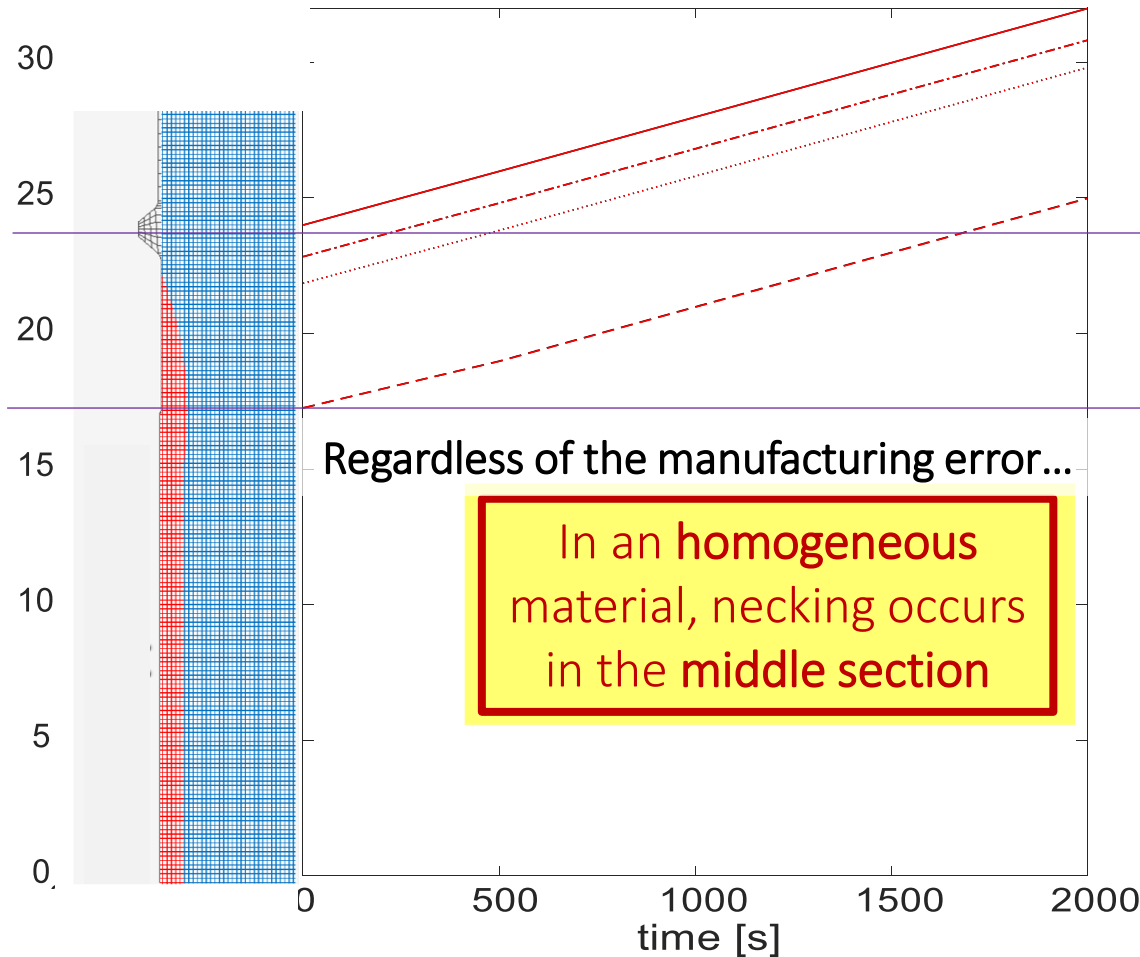
- Material inhomogeneity
- Manufacturing defect

Stage 1: 2D analysis | Hot quasi-static tensile test | simple elastoplastic law

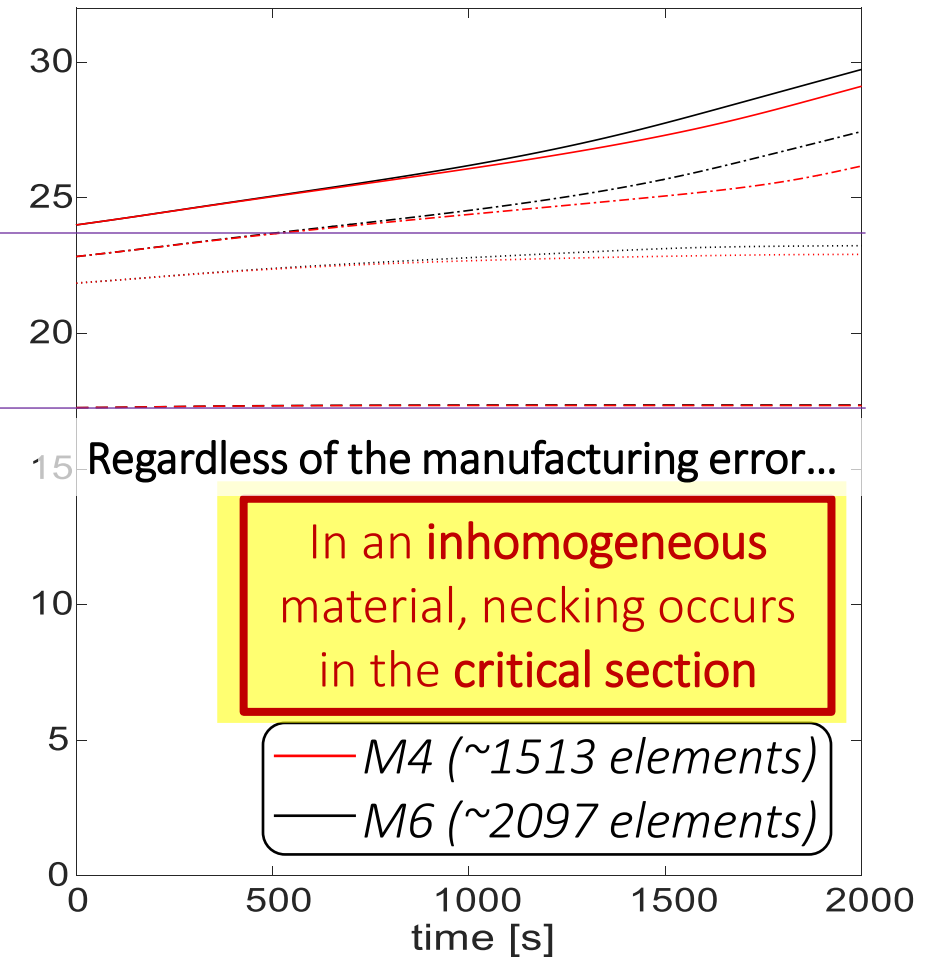
Model with manufacturing defect

Axial position of nodes of interest

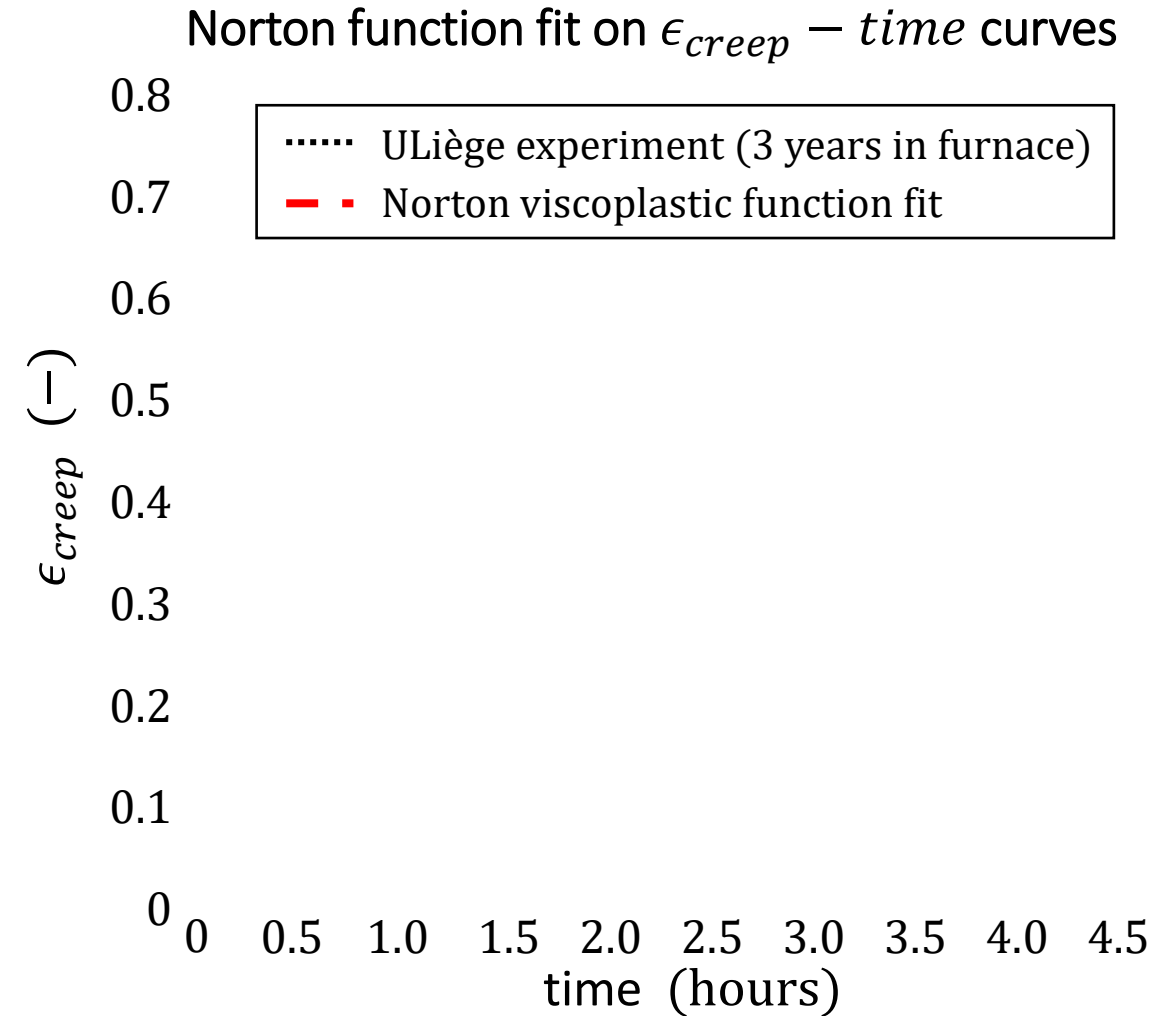
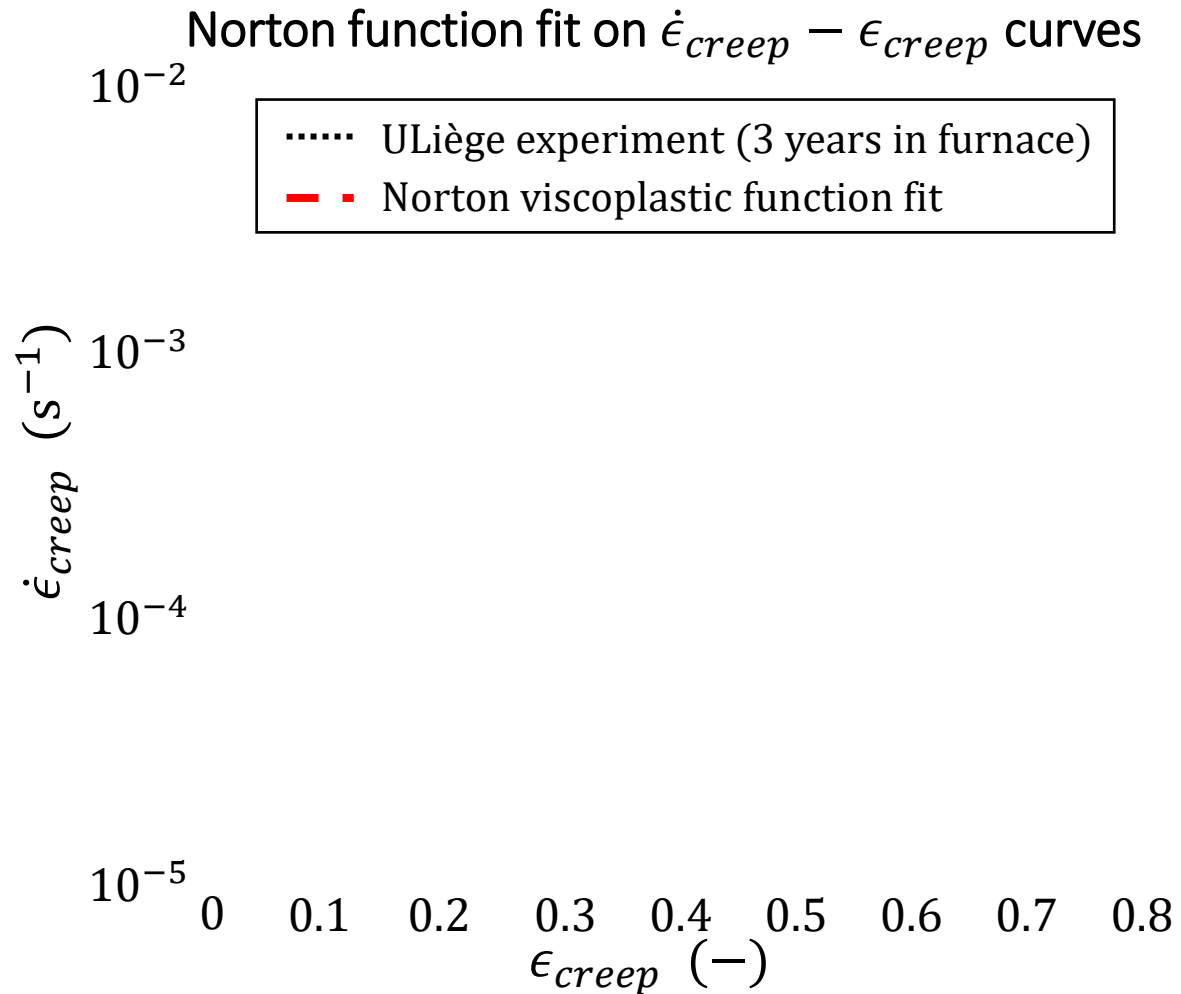
Displacements in N0 model



Displacements in NX model



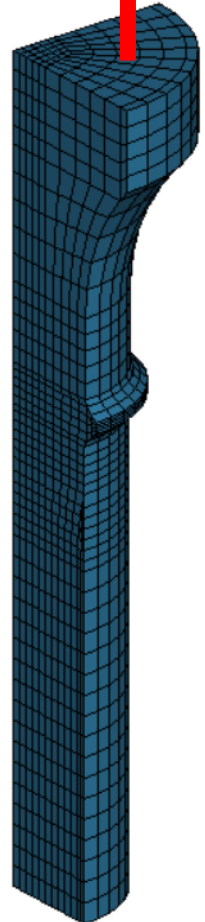
## Stage 2: 3D analysis | Chaboche-type colaw | Norton-Hoff viscoplastic function



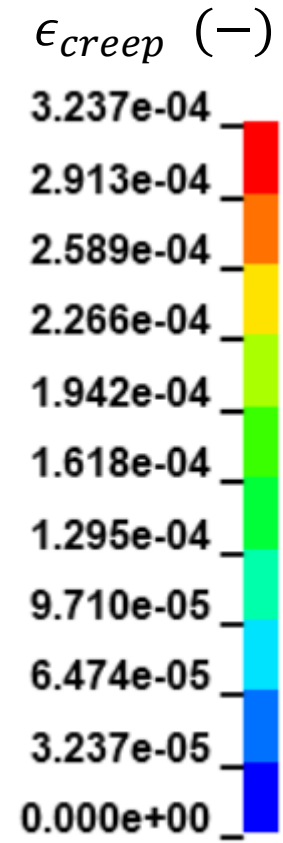
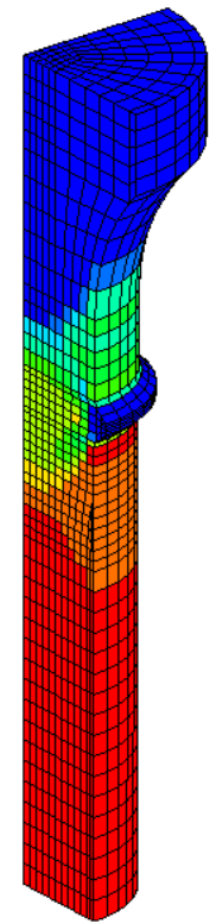
Stage 2: 3D analysis | Chaboche-type colaw | "identified" NH parameters

Model without manufacturing defect

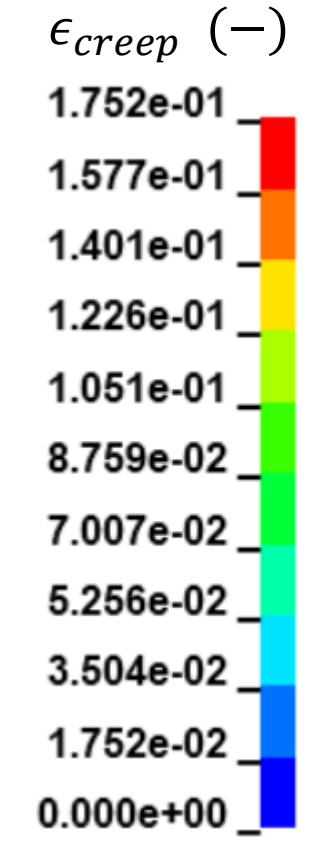
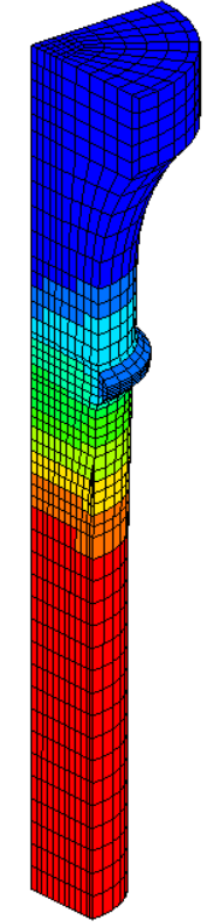
Constant force  
2.75 (kN)



Start of viscoplasticity (total load applied)



End of test -> time = 12k (s)  $\approx$  3.33 (hours)

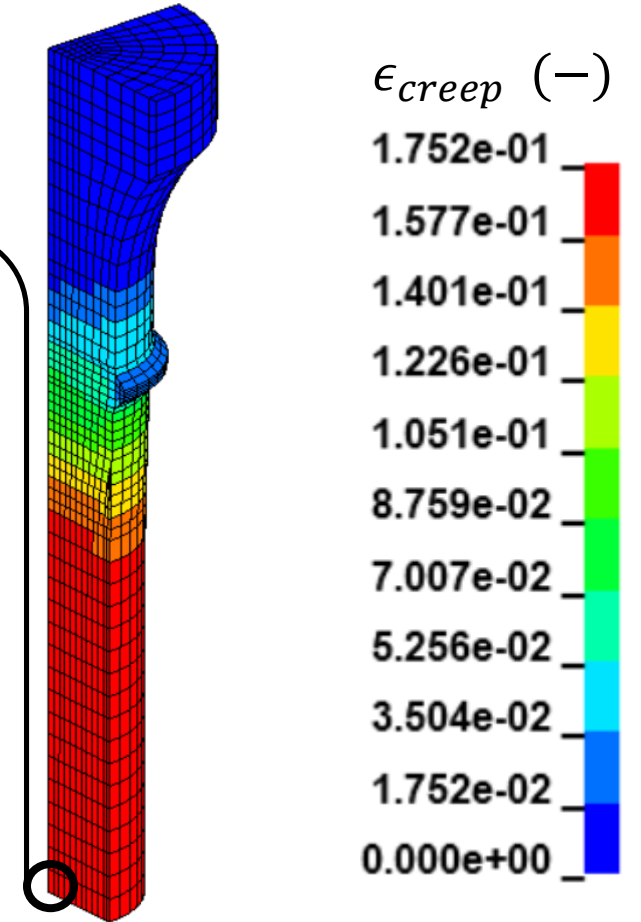
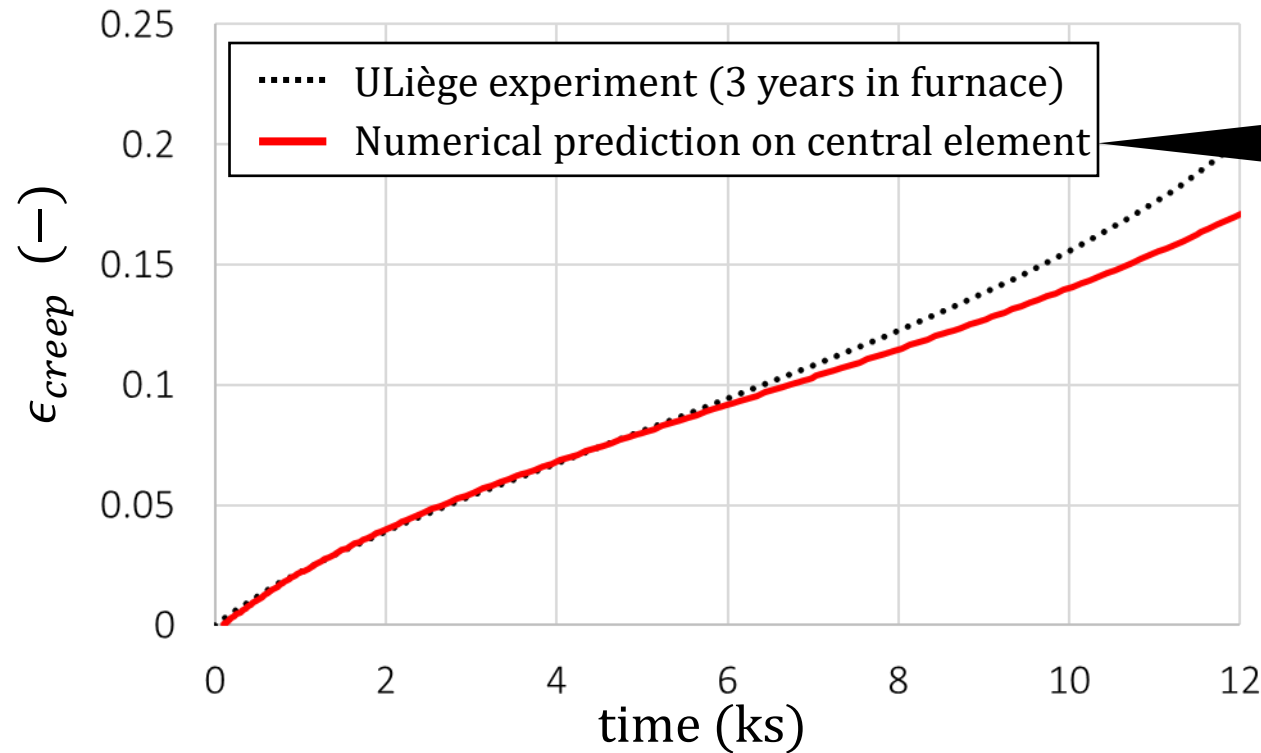


Stage 2: 3D analysis | Chaboche-type colaw | "identified" NH parameters

Model without manufacturing defect

End of test -> time = 12k (s) ≈ 3.33 (hours)

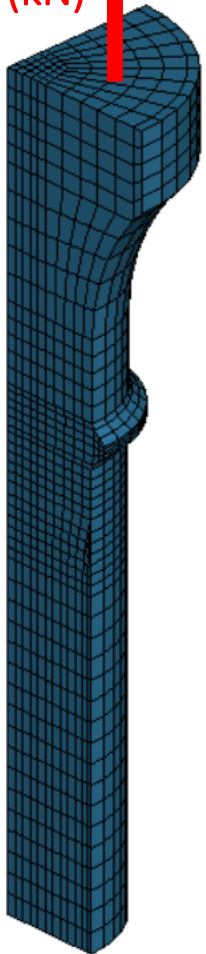
Experimental v/s numerical  $\epsilon_{creep}$  - time curve



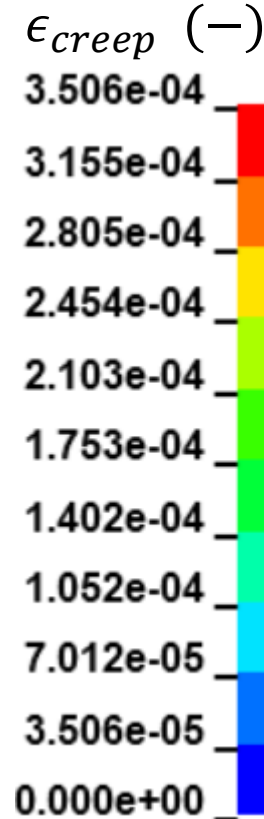
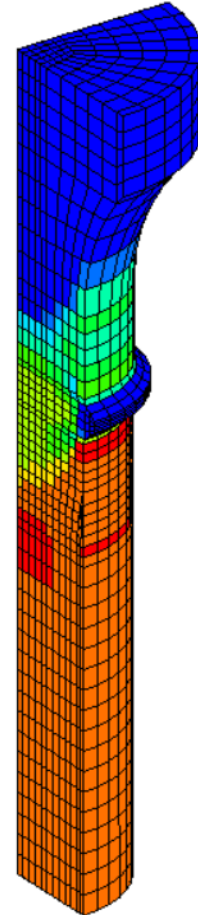
## Stage 2: 3D analysis | Chaboche-type colaw | "identified" NH parameters

Model with manufacturing defect

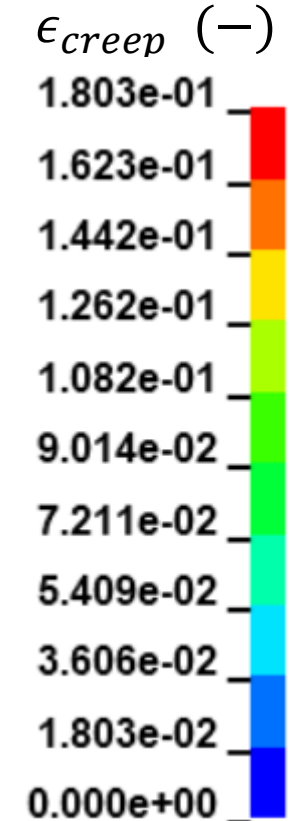
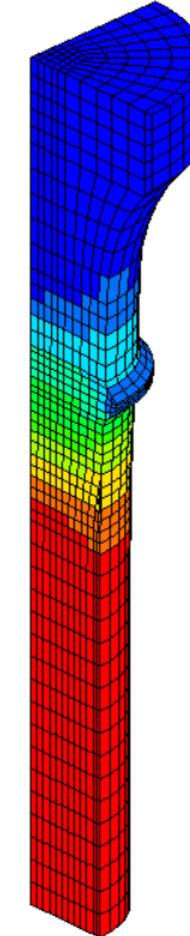
Constant force  
2.75 (kN)



Start of viscoplasticity (total load applied)



End of test -> time = 12k (s)  $\approx$  3.33 (hours)

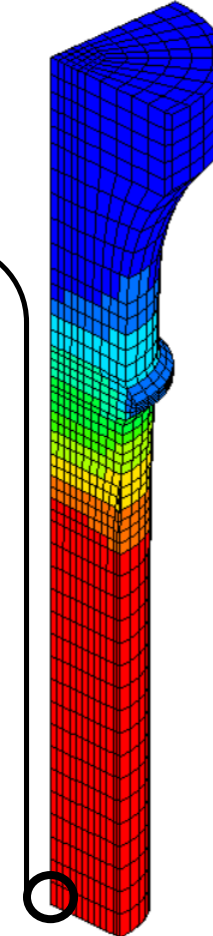
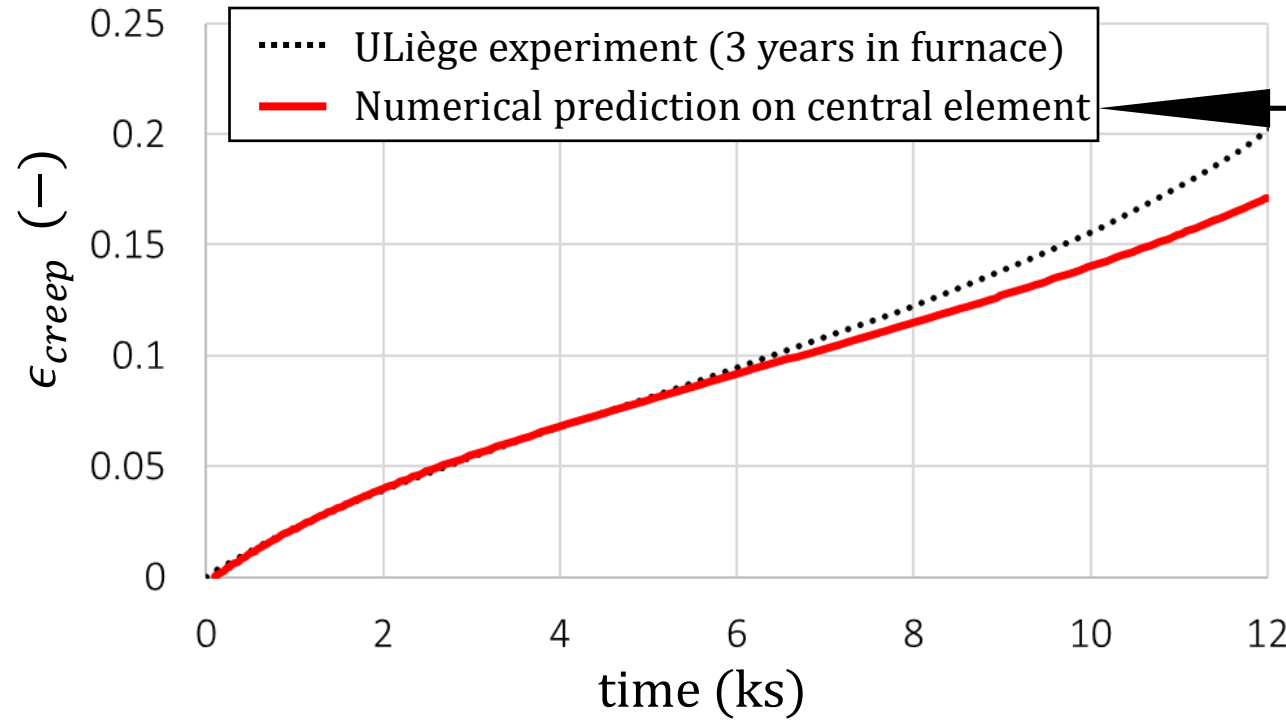


Stage 2: 3D analysis | Chaboche-type colaw | "identified" NH parameters

Model with manufacturing defect

End of test -> time = 12k (s)  $\approx$  3.33 (hours)

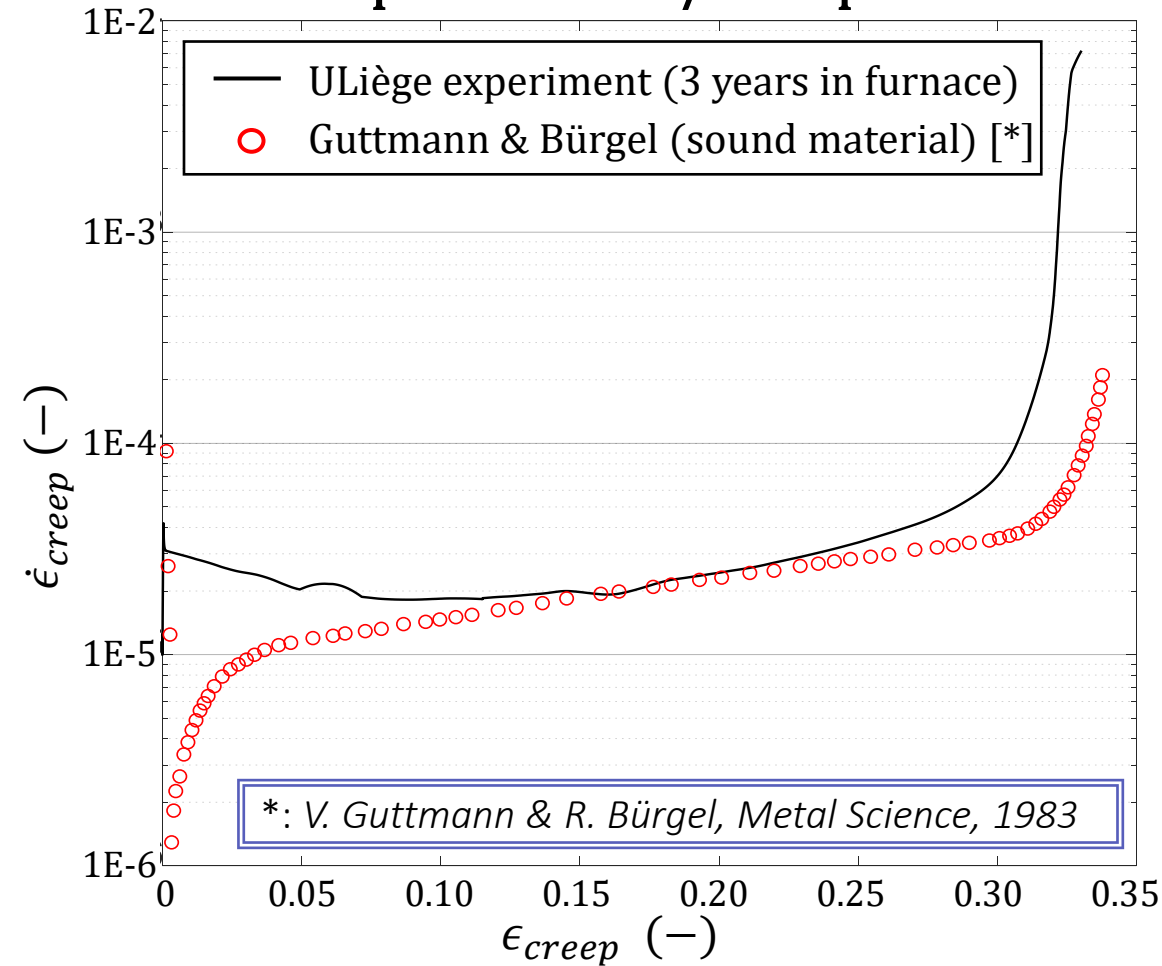
Experimental v/s numerical  $\epsilon_{creep}$  - time curve





## On our experimental campaign

Creep strain rate v/s creep strain



EP simulations suggest effect of material inhomogeneity

EVP simulations could not predict necking in critical zone

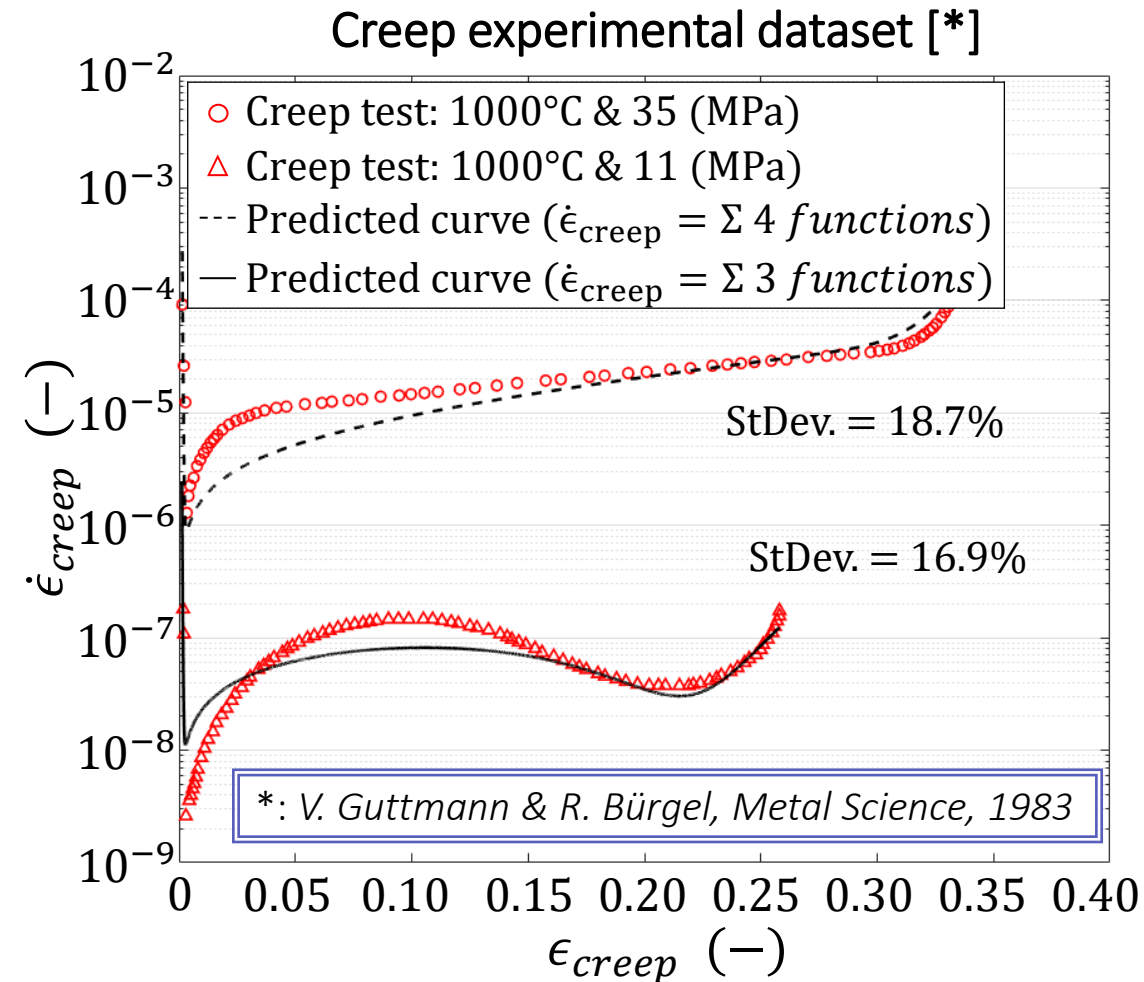
Microstructural evolution of 800H after 3 years in furnace consists in:

- Nitridation/oxidation competition
- Intergranular precipitation of  $M_{23}C_6$  carbides

To inquire on the effect of material inhomogeneity and microstructure evolution on the creep behaviour of 800H:

- Surface mapping via nanoindentation on more samples
- Pursue a thorough experimental campaign
- Perform simulations addressing material inhomogeneity

## On the numerical aspects of the project



The numerical model implemented exhibits **good adaptability for addressing classical & non-classical creep behaviour**

There is still a high uncertainty on the evolution of 800H microstructure and its effect on the creep behaviour

Microstructural evolution of 800H after 3 years in furnace consists in:

- Nitridation/oxidation competition
- Intergranular precipitation of  $M_{23}C_6$  carbides

To develop a creep micromechanics approach

- **Coupled** to our macromechanical model
- **Decrease uncertainty of the model**
- Provide **realistic and reliable** simulations

The final objective of my PhD. Project

# Accurate very-high temperature creep-life prediction of Incoloy 800H addressing effects of creep mechanism transition and nitridation

Carlos Rojas-Ulloa<sup>1</sup>, Hélène Morch<sup>1</sup>, Víctor Tuninetti<sup>2</sup>, Jérôme T. Tchuindjang<sup>3</sup>, Olivier Pensis<sup>4</sup>, Amedeo Di Giovanni<sup>4</sup>, Anne Mertens<sup>3</sup>, Laurent Duchêne<sup>1</sup> and Anne Marie Habraken<sup>1,5</sup>

<sup>1</sup>: ArGEnCo department, University of Liège, Belgium

<sup>2</sup>: Department of mechanical engineering, University of La Frontera, Chile

<sup>3</sup>: Department of Aerospace and Mechanical Engineering, University of Liège, Belgium

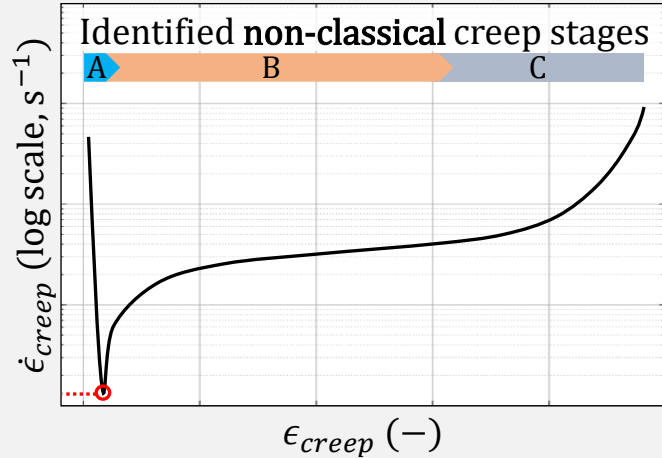
<sup>4</sup>: R&D department, Drever International, Liège, Belgium

<sup>5</sup>: Fonds de la Recherche Scientifique –F.R.S. –F.N.R.S., Belgium

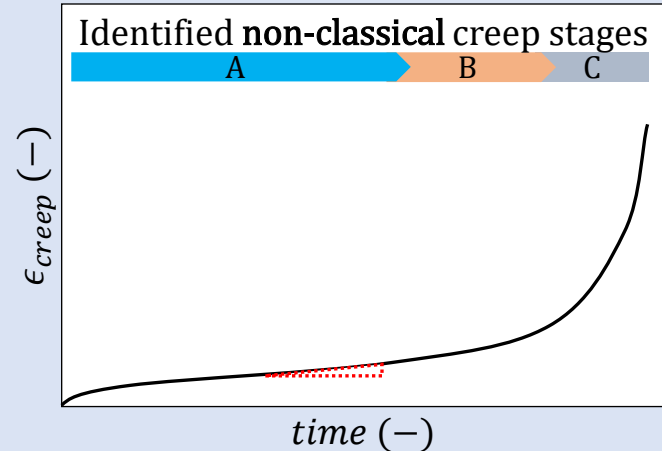
## Creep test

Low stress & high  $T^\circ$   
Short creep test | inert gas test

Creep strain rate  
v/s  
Creep strain

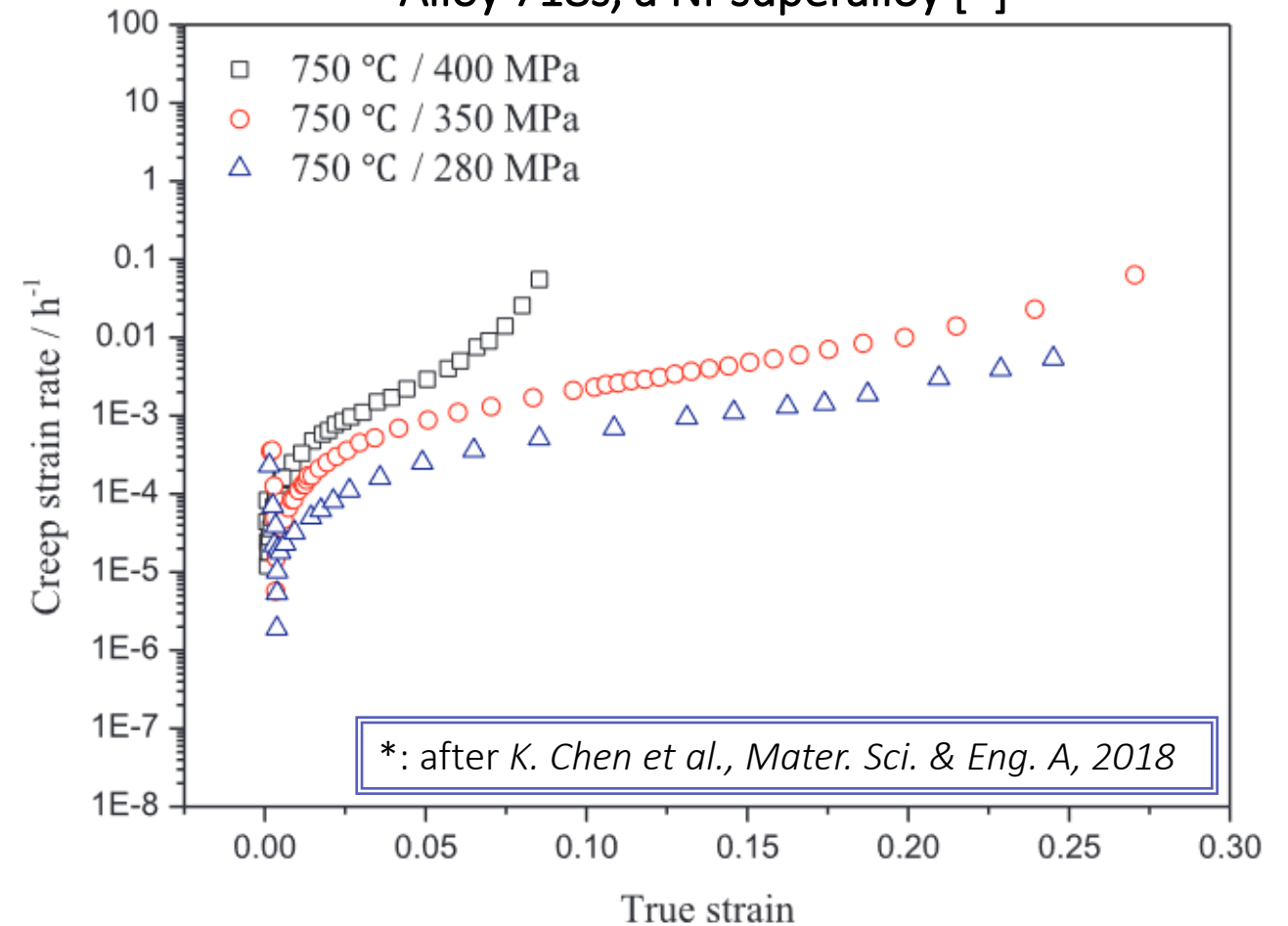


Creep strain  
v/s  
time



Similar behaviour has been reported for...

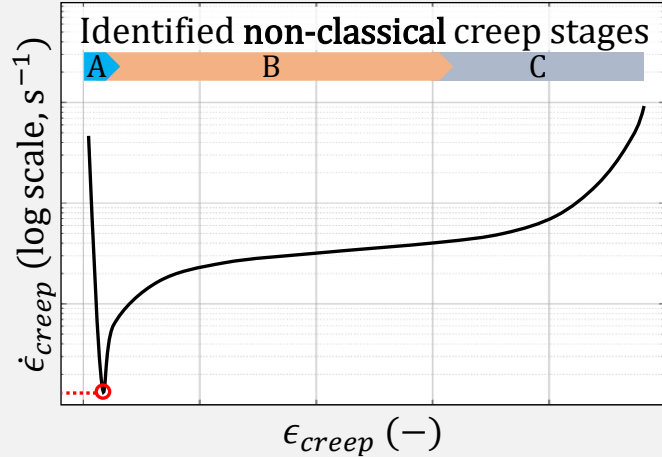
Alloy 718s, a Ni-superalloy [\*]



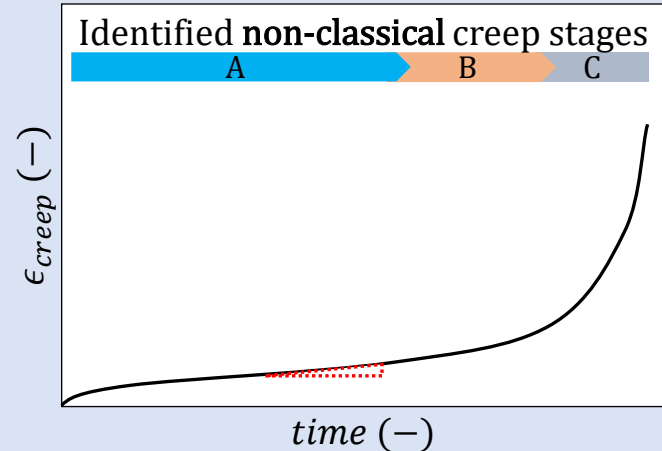
## Creep test

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Creep strain rate  
v/s  
Creep strain

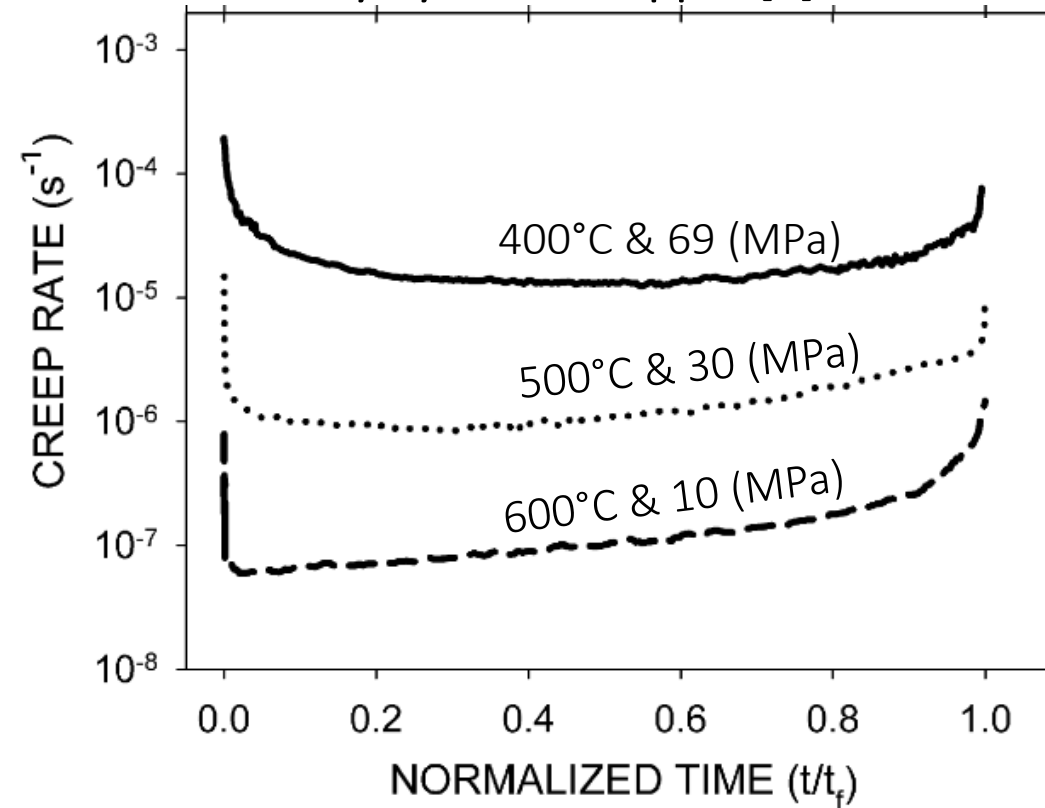


Creep strain  
v/s  
time



Similar behaviour has been reported for...

## Polycrystalline copper [\*]



\*: after B. Wilshire & A.J. Battenbough, Mater. Sci. & Eng. A, 2007