

# Creep mechanisms of Incoloy 800H at high temperature

Carlos Rojas-Ulloa<sup>1</sup>, H el ene Morch<sup>1</sup>, J er ome T. Tchuindjang<sup>3</sup>, Olivier Pensis<sup>4</sup>, Amedeo Di Giovanni<sup>4</sup>, Anne Mertens<sup>3</sup>, Laurent Duch ene<sup>1</sup>, V ictor Tuninetti<sup>2</sup> and Anne Marie Habraken<sup>1,5</sup>

<sup>1</sup>: ArGEnCo department, University of Li ege, Belgium

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

<sup>3</sup>: Department of Aerospace and Mechanical Engineering, University of Li ege, Belgium

<sup>4</sup>: R&D department, Drever International, Li ege, Belgium

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

July 3<sup>rd</sup>, 2023

## Research context

Industry & Science on 800H alloy

## Creep macromechanics

- 800H creep macromechanics
- Finite element modelling

## Creep micromechanics

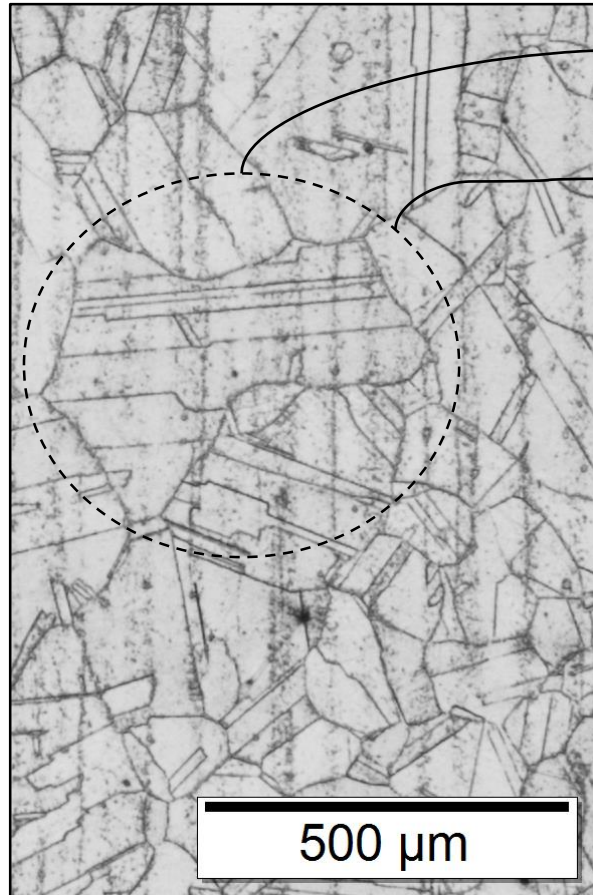
- 800H creep micromechanics
- Experimental findings

## Concluding remarks

- Multiscale correlation
- Research prospects

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

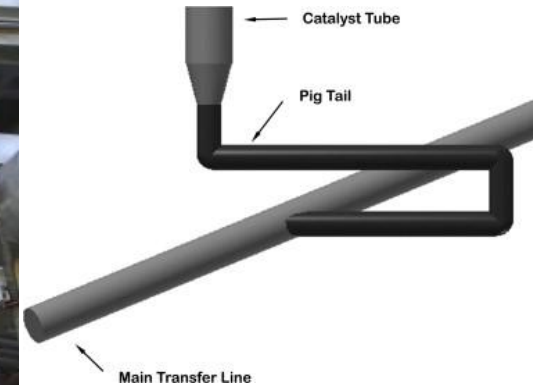
- Solution annealed: 1150°C + WQ
- Microstructure:



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

- Used in many applications:

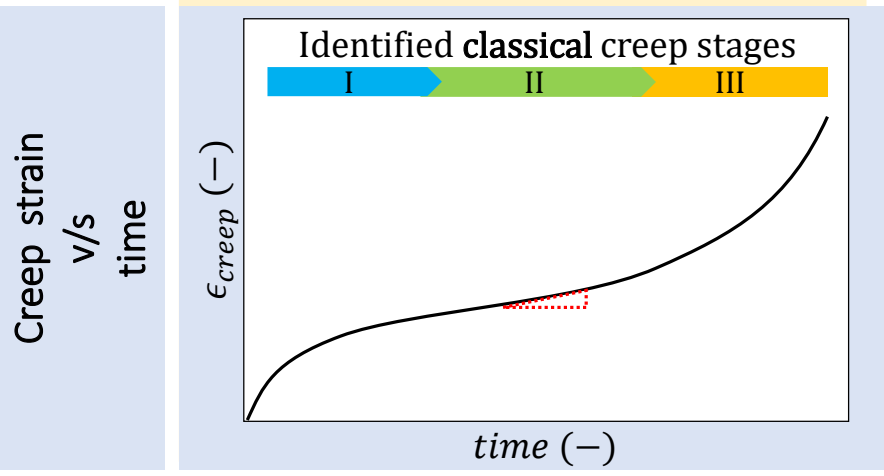
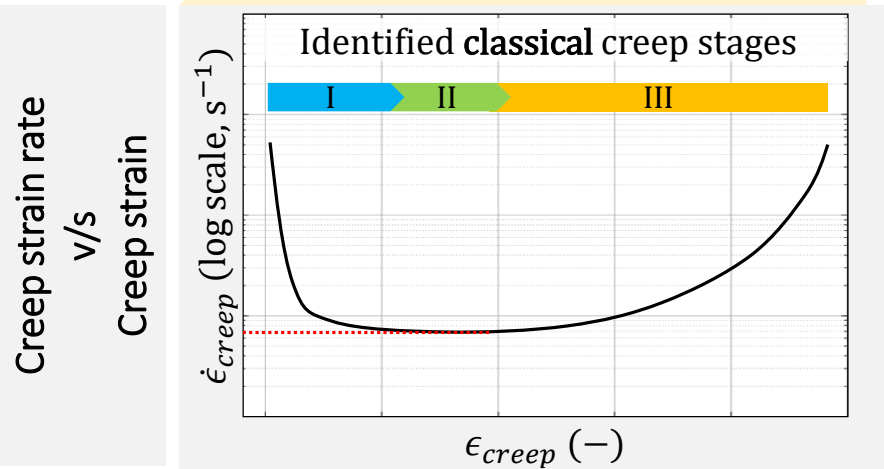
- Petrochemical
- Metallurgy
- Heat exchangers
- Power generation
- ...



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

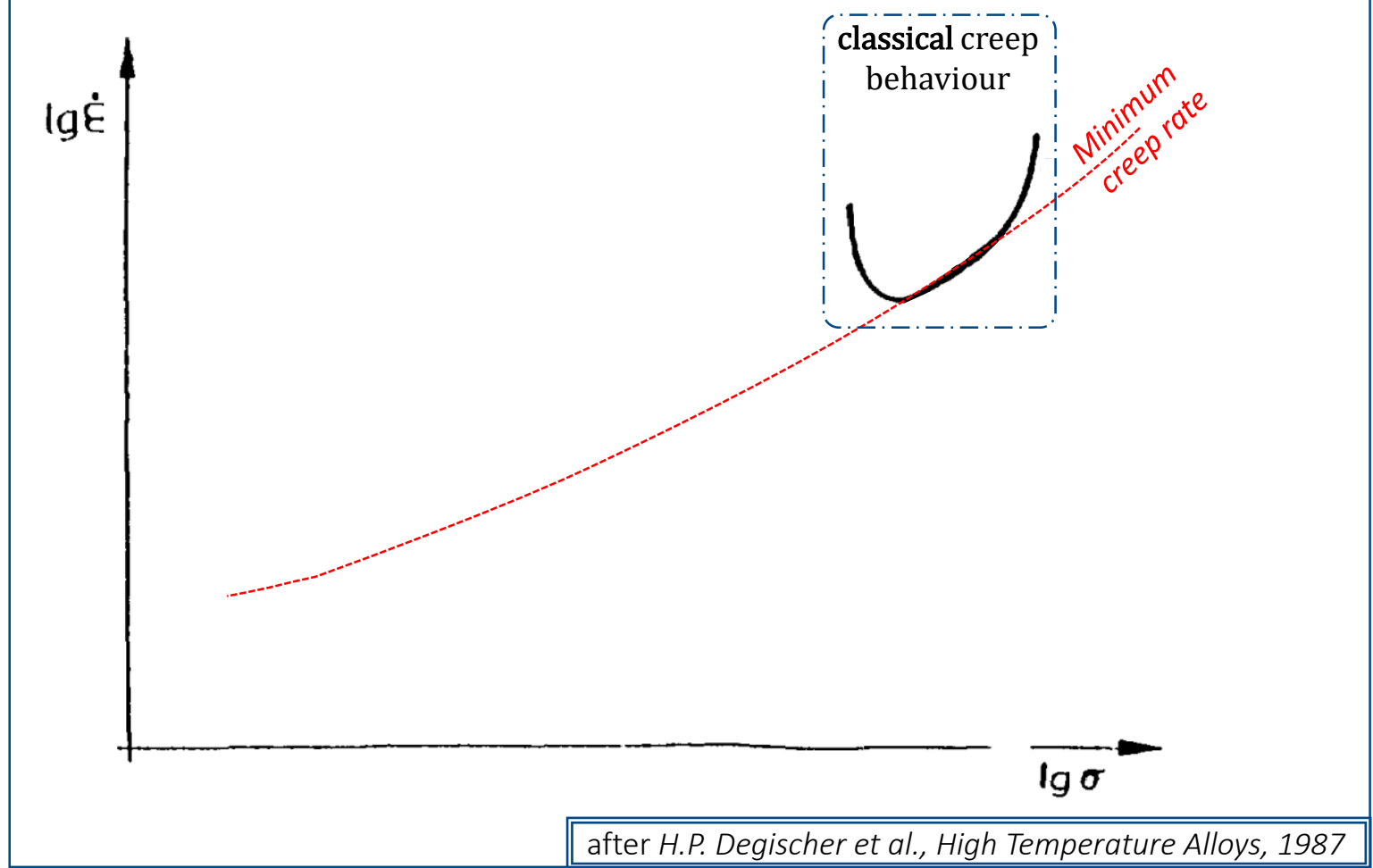
## Creep test

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



## Classical creep behaviour

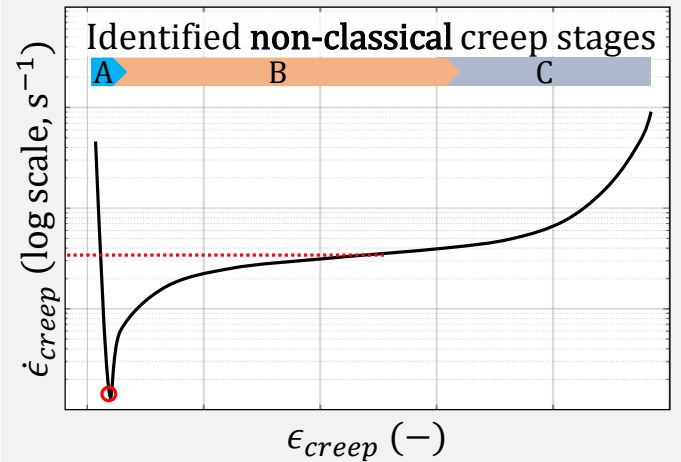
### Qualitative creep rate – stress relation at 800°C



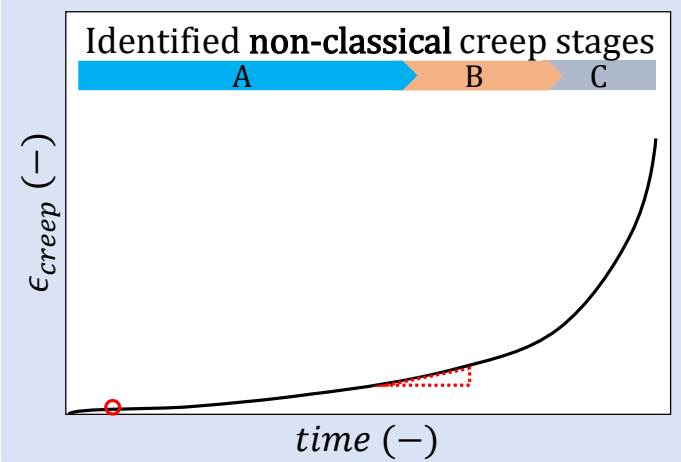
## Creep test

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

Creep strain rate  
v/s  
Creep strain

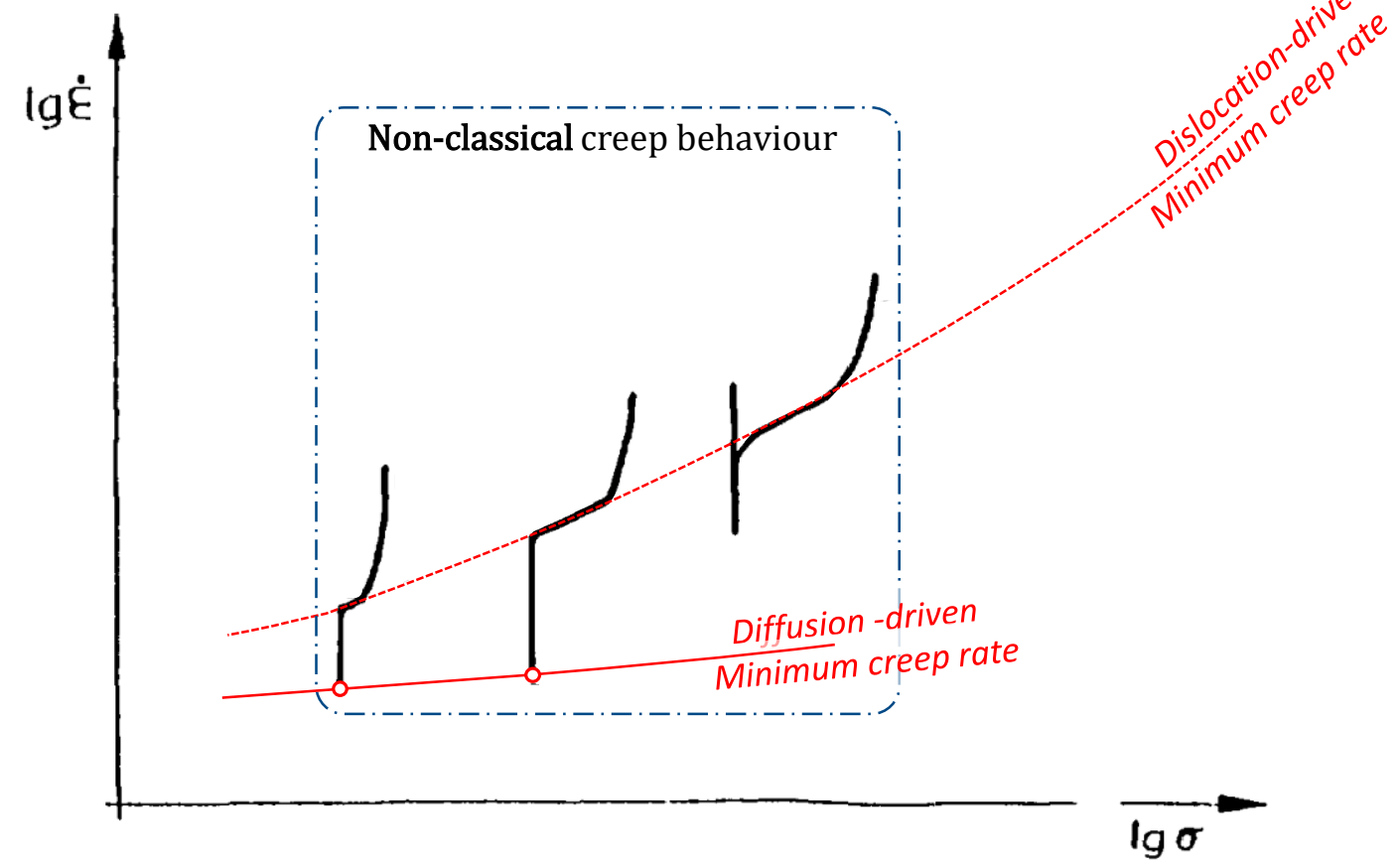


Creep strain  
v/s  
time

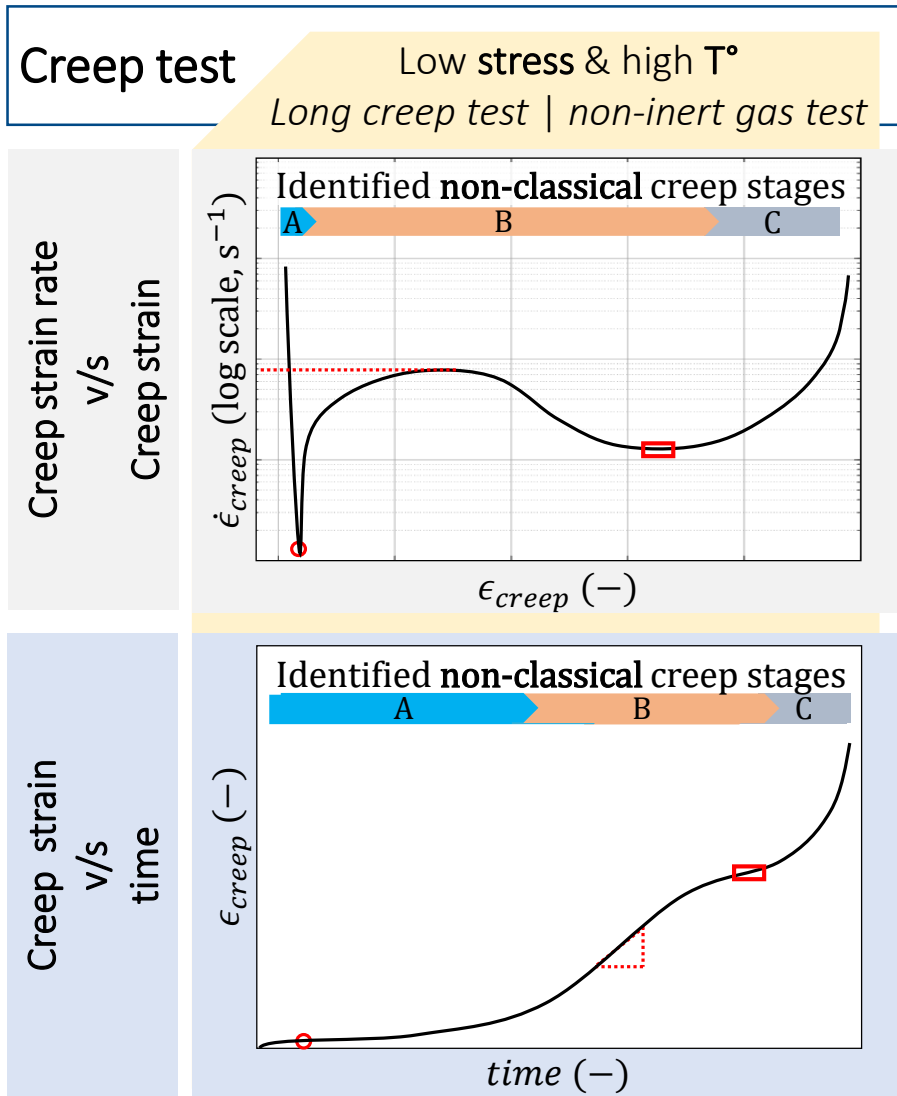


## Non-classical creep behaviour: creep mechanism transition

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

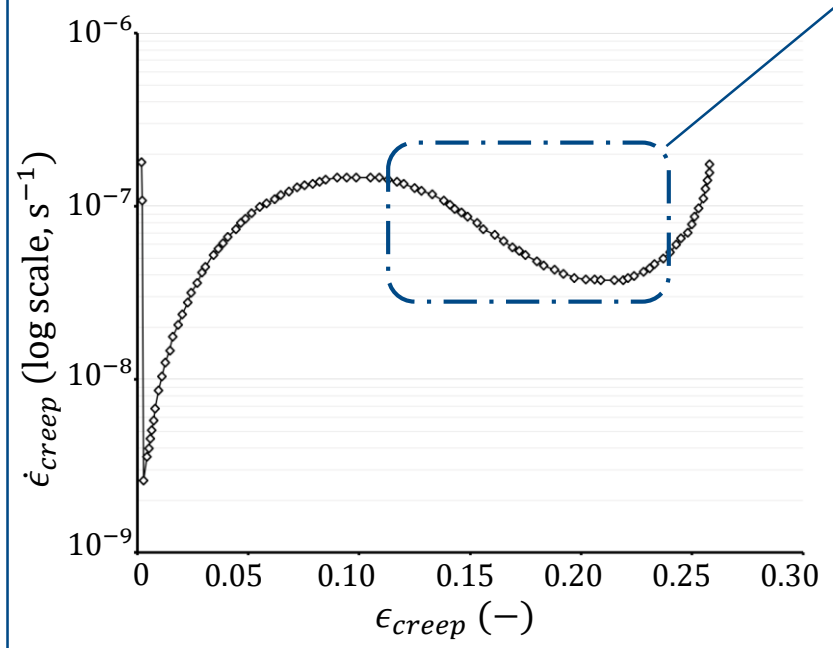


after H.P. Degischer et al., High Temperature Alloys, 1987



## Non-classical creep behaviour: Two-step minima

Creep on 800H at 11 [MPa] & 1000°C



after V. Guttman & R. Bürgel, Metal Science, 1983

- Why?
  - Nitridation-induced creep hardening

Observed in 800H  
 V. Guttman & R. Bürgel, 1983;  
 H.P. Degischer's team (GmbH, Austria)

- Intergranular  $M_{23}C_6$  carbides precipitation

Observed in similar Ni-austenitic alloys  
 T. Hatakeyama et al., 2021; 2022



## Constitutive law: Chaboche-type law + Graham-Walles viscoplastic & damage function approach

Yield function: **von-Mises** criterion

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

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

Viscoplastic function 1: **Norton power law**

Damage function 1: **Lemaitre + Kachanov**

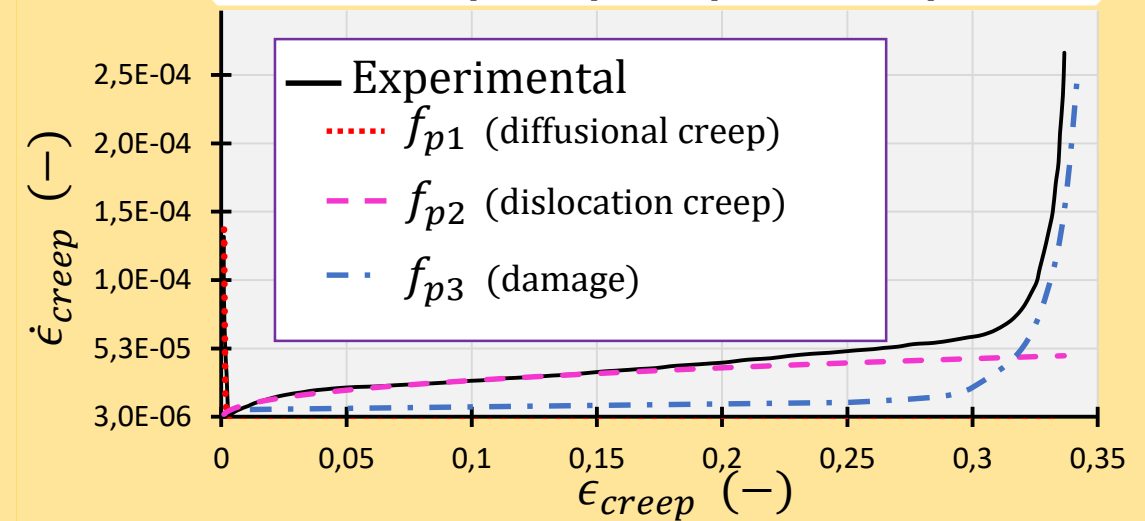
Viscoplastic function 2: **Graham-Walles approach** [\*]

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

\*: Model developed in IfW Darmstadt. See *Narayana K. Karthik, PhD Thesis, Aachen University, 2020*

1 Function  $\leftrightarrow$  1 phenomenon

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



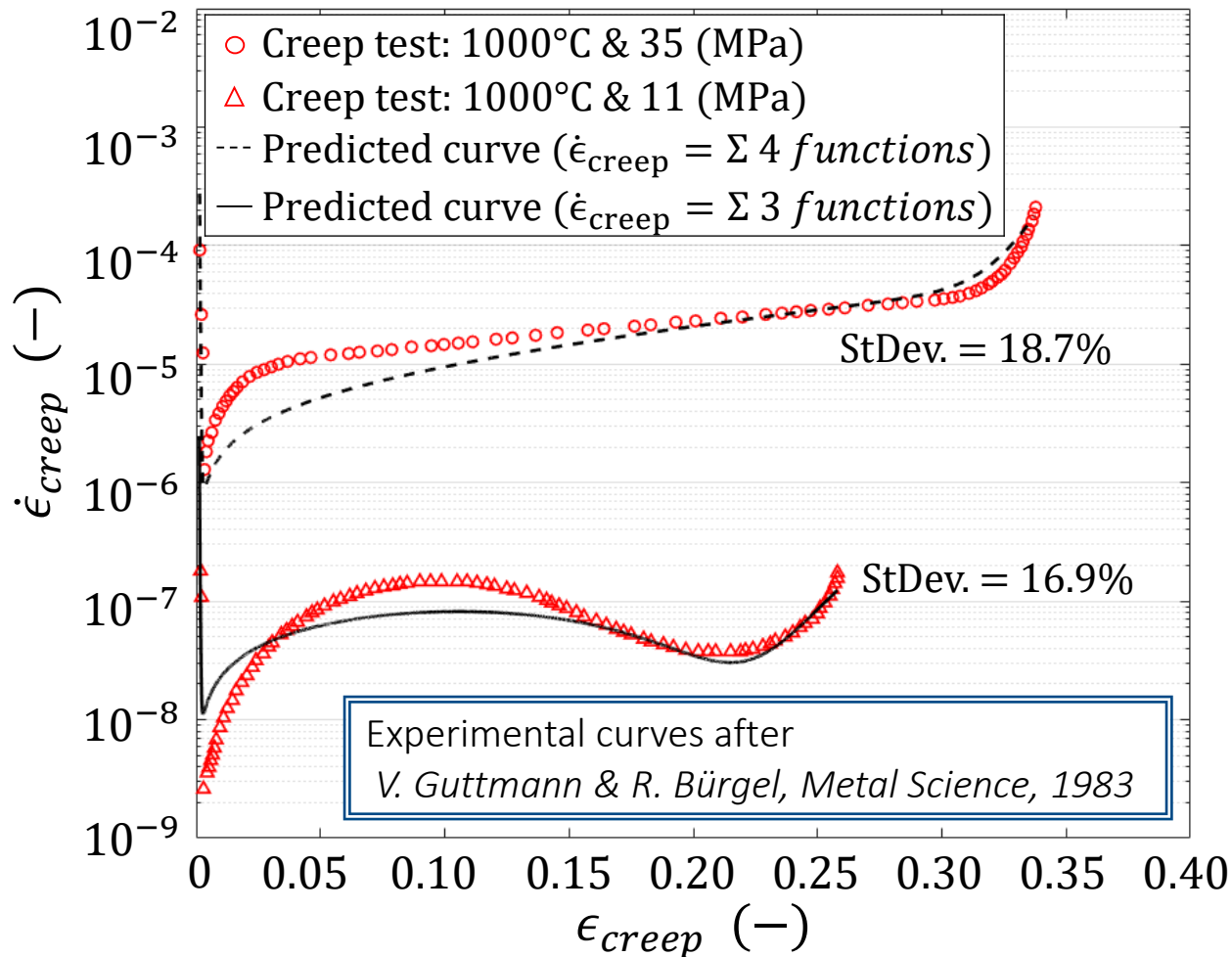
Curve after *V. Guttman & R. Bürgel, Metal Science, 1983*

Damage function 2: **Graham-Walles approach** [\*]

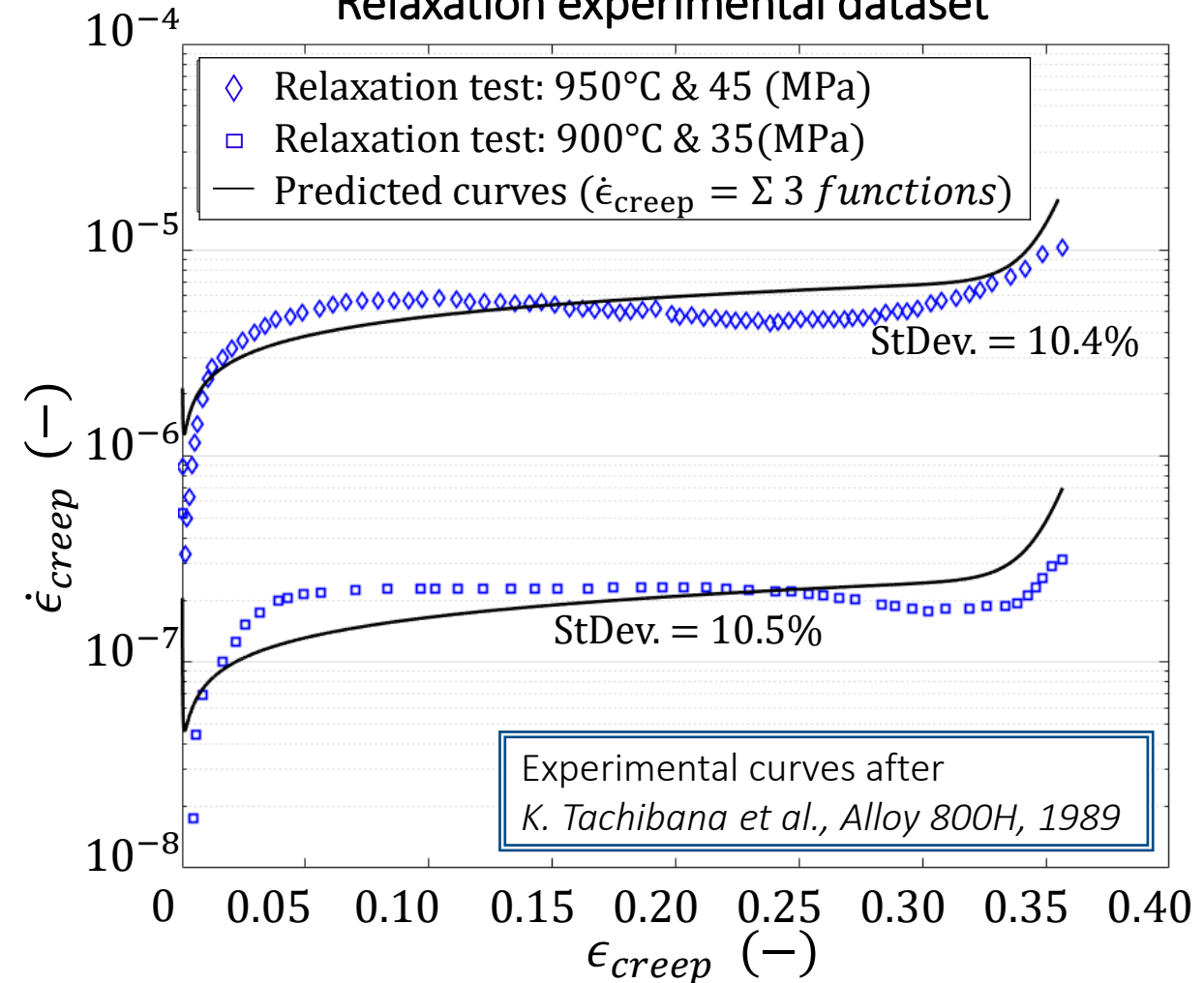
$$\begin{aligned} \dot{D} &= \dot{D}_C + \dot{D}_f \\ &= K_{TD} |\dot{T}| (\bar{\epsilon}^p)^{m_{TD}} \\ &= K_D \Sigma_{VM}^{eq} \end{aligned}$$

## Constitutive law: Chaboche-type law + Graham-Walles viscoplastic & damage function approach

### Creep experimental dataset



### Relaxation experimental dataset





## Creep in 800H: a multiscale phenomenon

Smooth bar samples were placed in a furnace, where thermal and environmental conditions were simulated

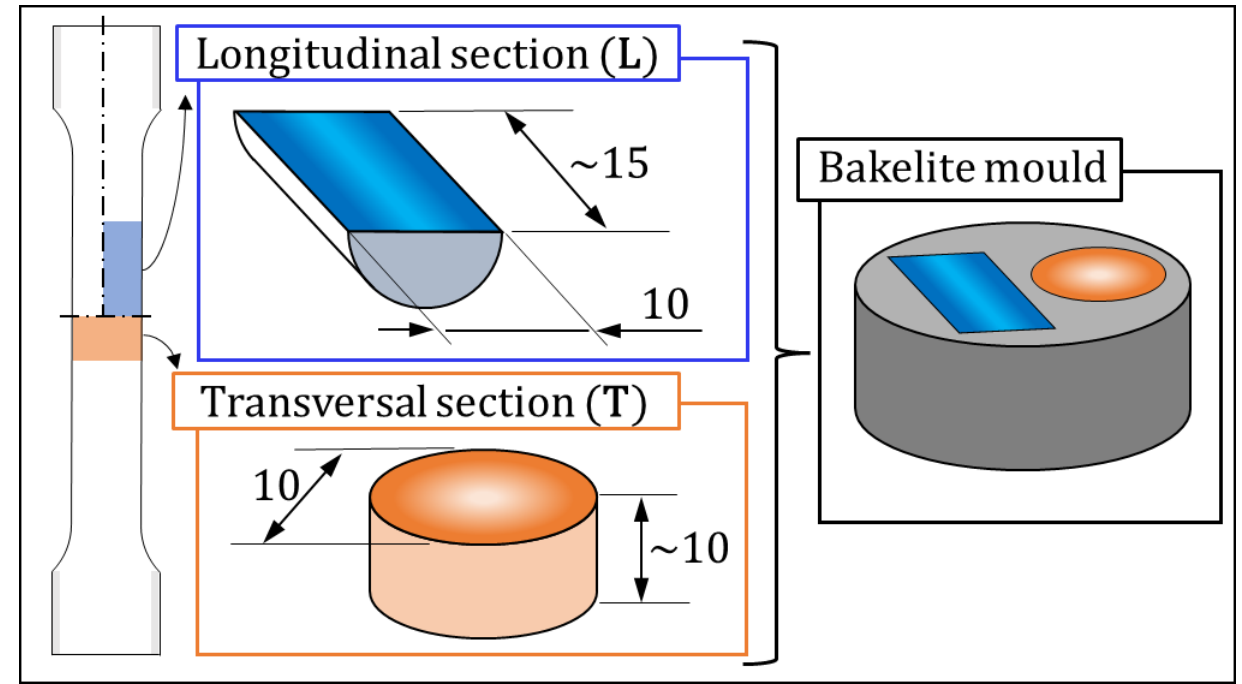
Smooth bar 800H samples

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

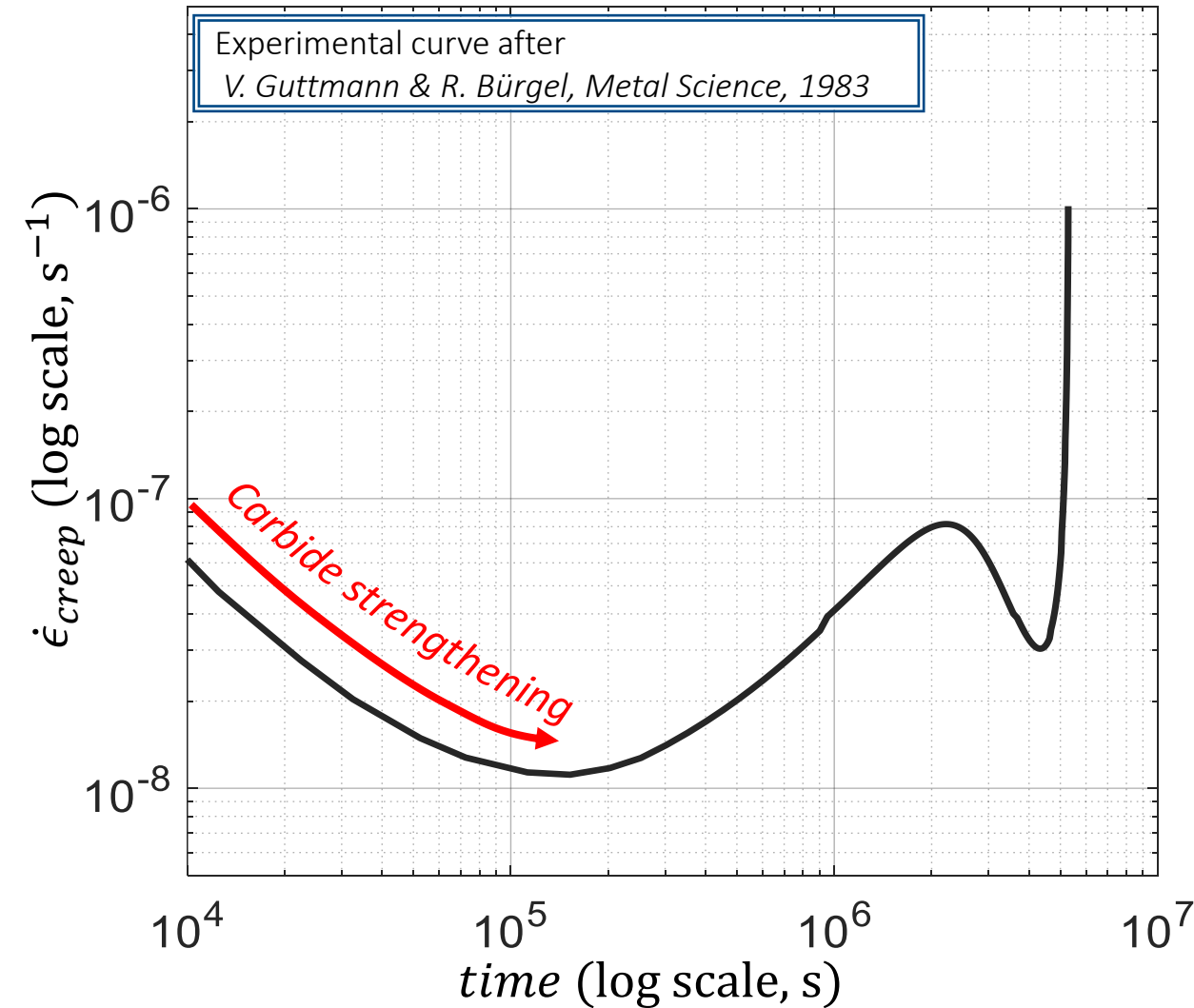
1 D10 sample from each N-batch



**Microstructural experiments performed at ULiège 2023**

Optical microscopy  
 macro-indentation (HV10)  
 micro-indentation (HV 0.5)  
 SEM/EDX

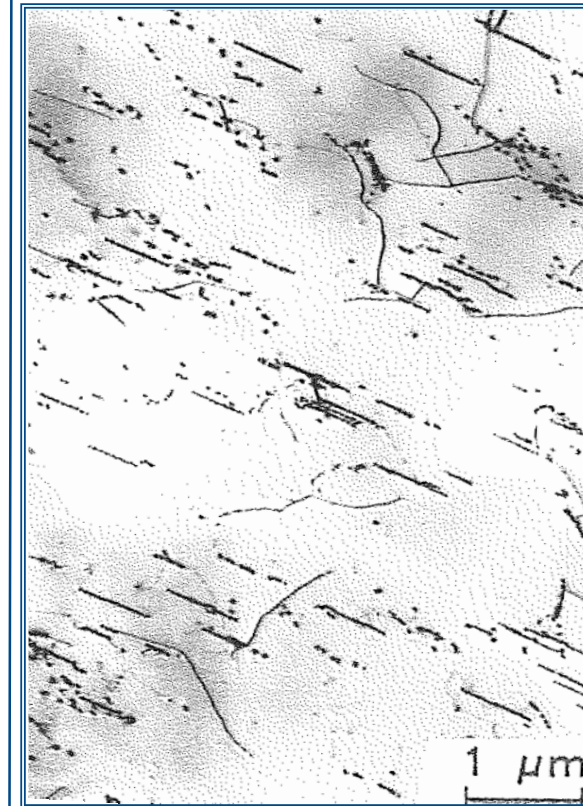
Predicted creep v/s time curve



## Carbide strengthening

- Presence of Nb, Cr & Ti carbides
- Dislocations are pinned

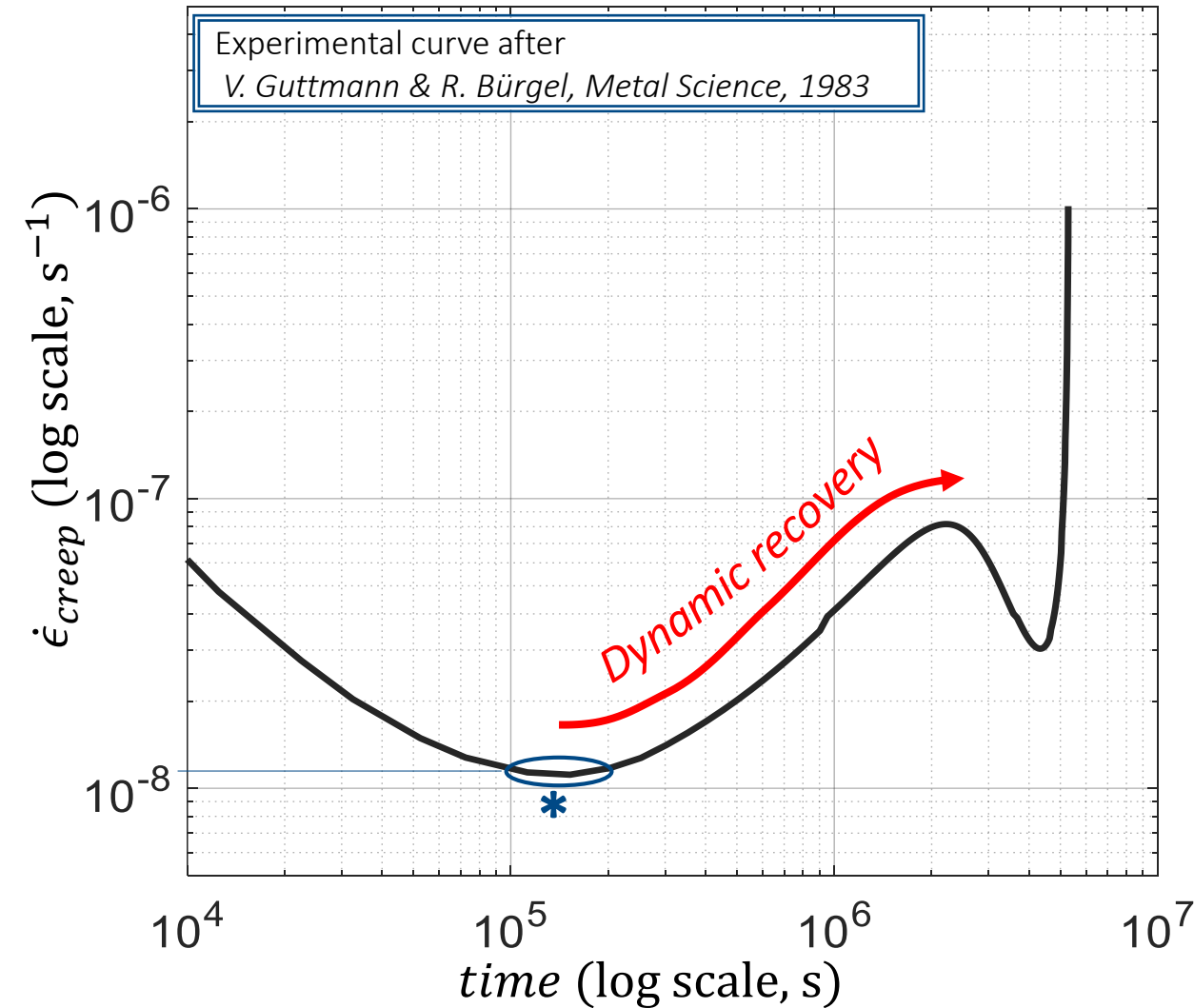
Diffusional-driven creep occurs



*MC carbides precipitating into dislocations after 30 min at 800°C*

TEM image after:  
A. Cyrska-Filemonowicz et al., ICEM 13, 1994

Predicted creep v/s time curve



## Dynamic recovery

- MC Carbides diffuse towards GBs
  - Dislocations are able to move freely
- Dislocation-driven creep occurs

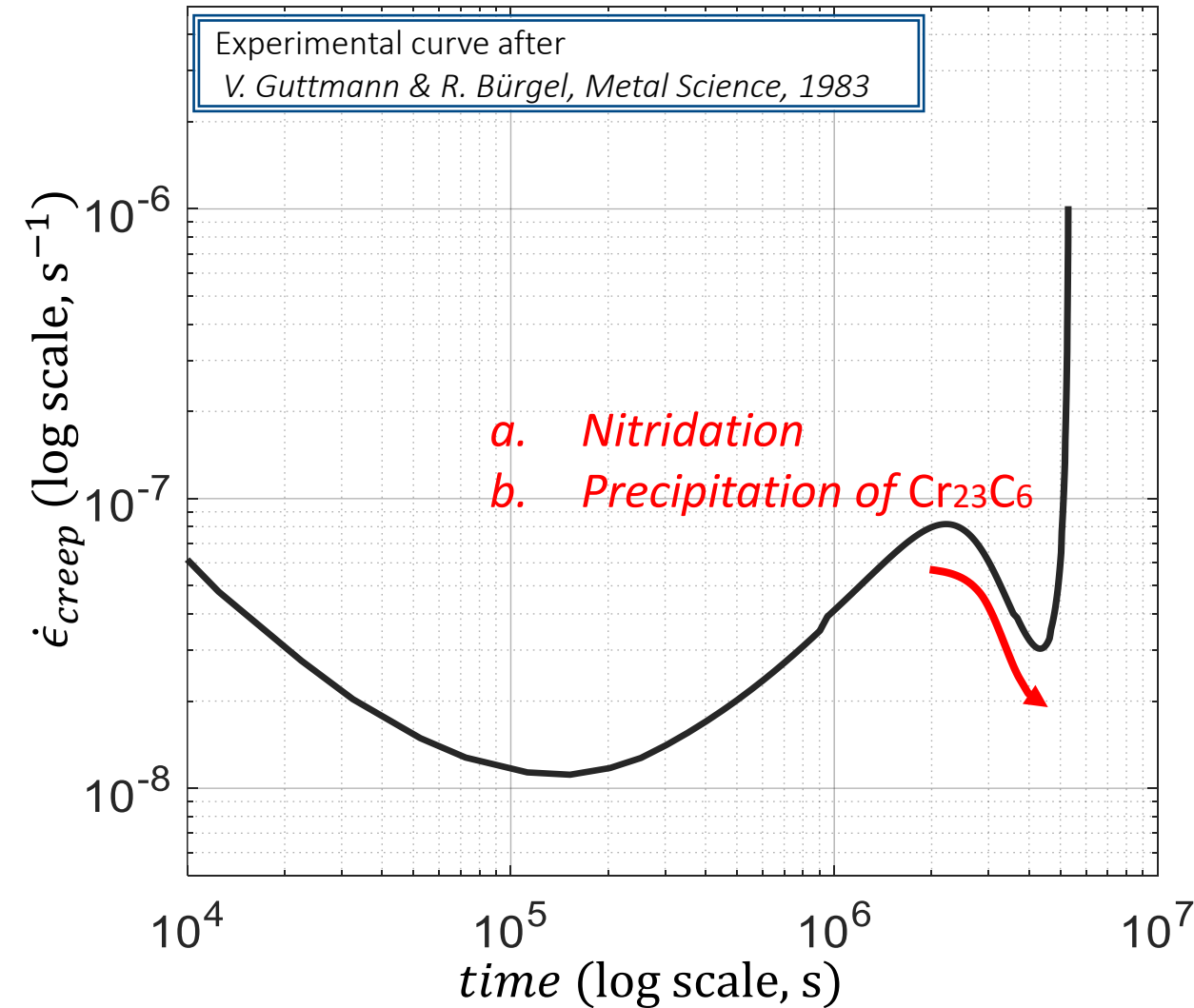


Dislocation pile-up in GBs of 800H alloy after **minimum creep strain rate\*** is reached

TEM image after  
*V. Guttman & R. Bürigel, Metal Science, 1983*



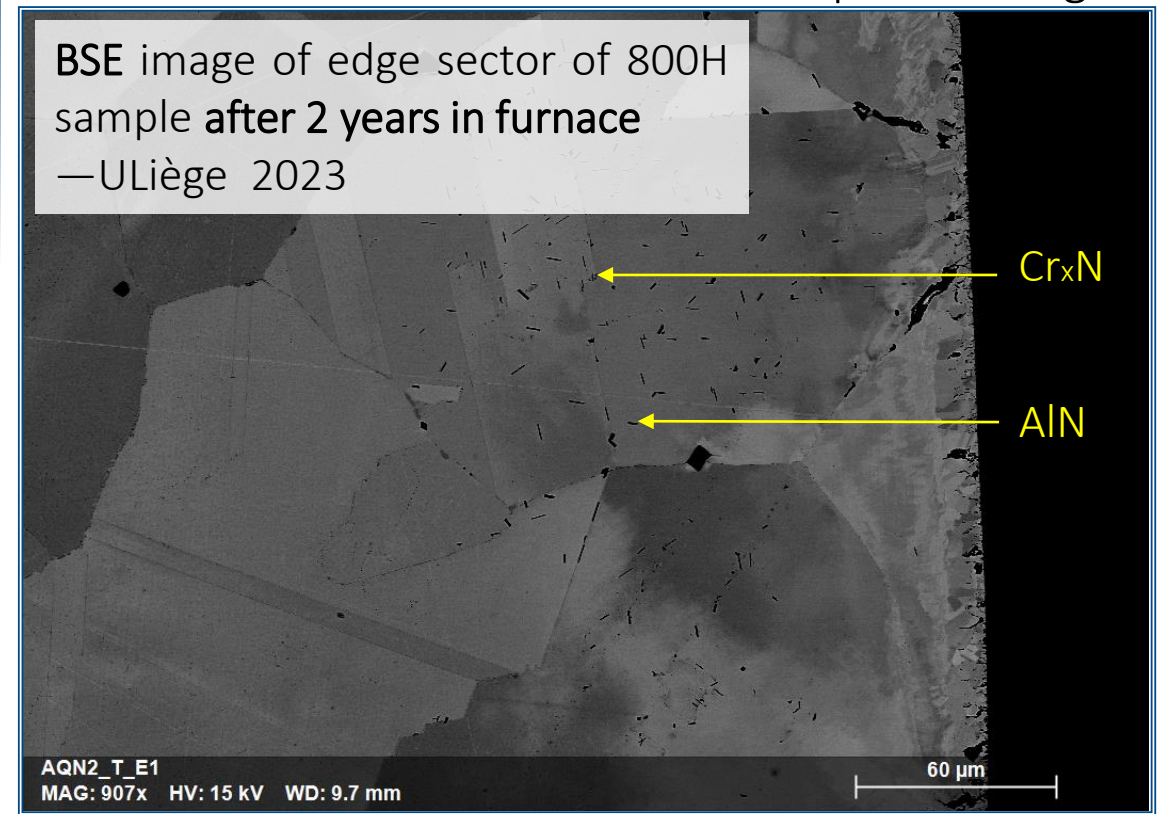
Predicted creep v/s time curve



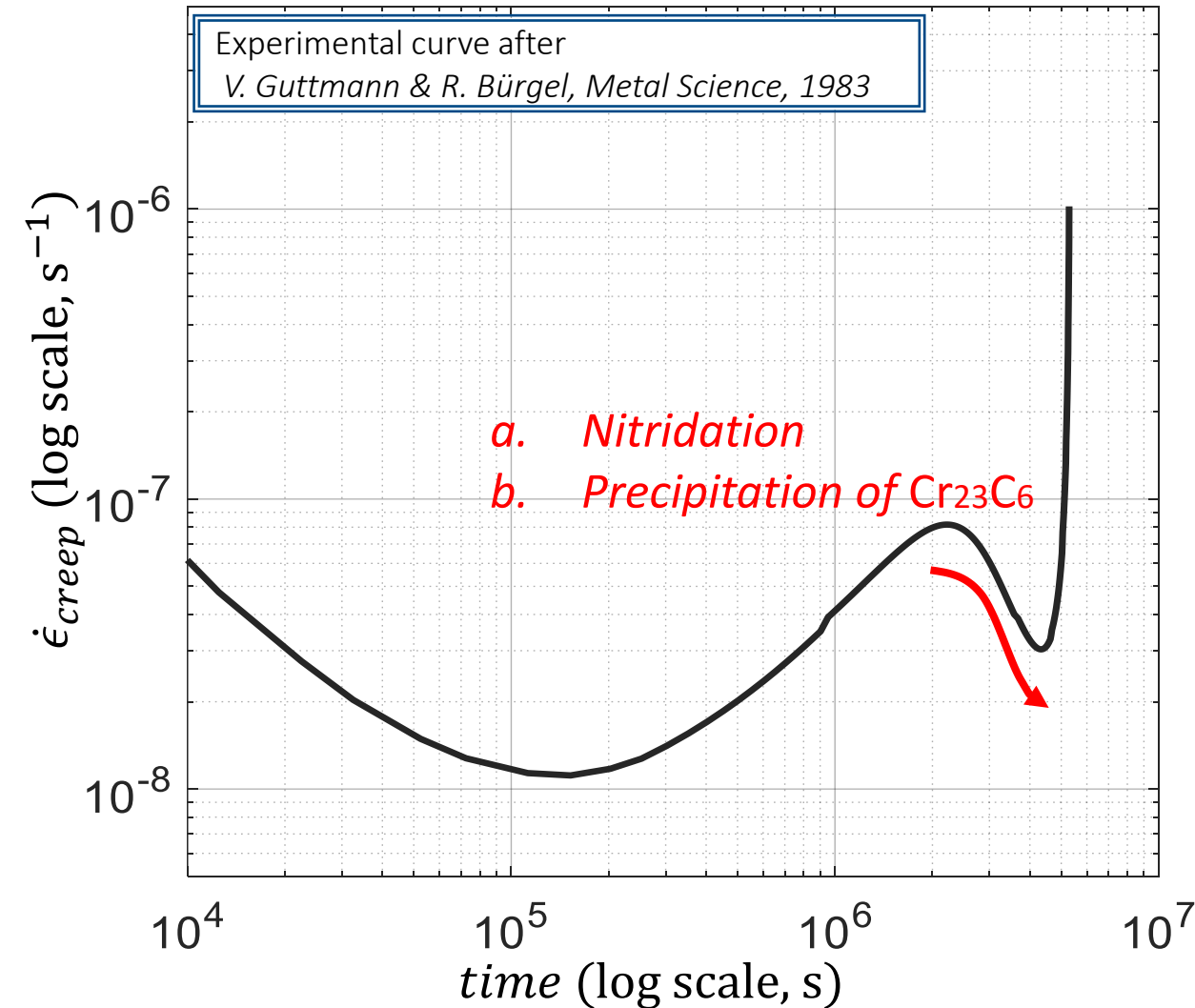
## a. Nitridation

- Intragranular precipitation of Cr- & Al- nitrides
- Material surface hardening

→ Nitridation-induced creep hardening



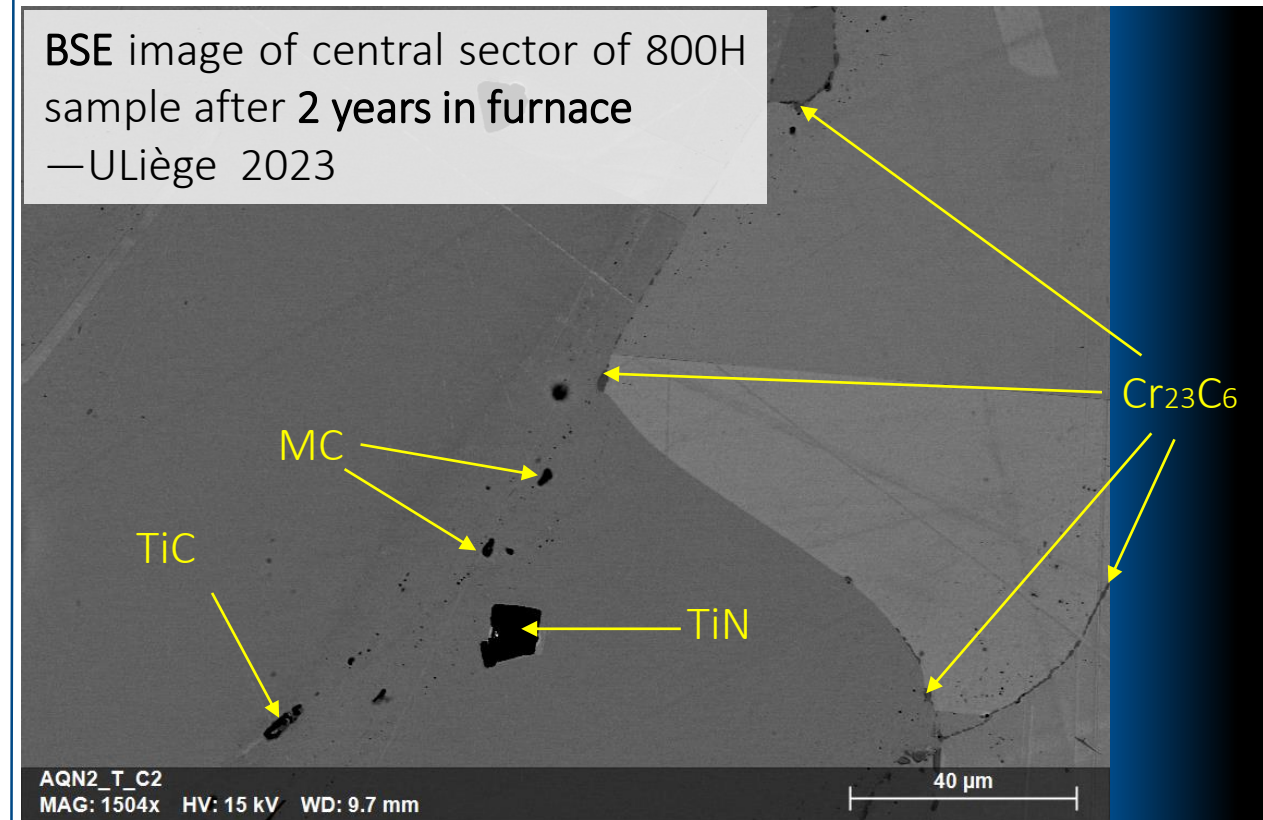
## Predicted creep v/s time curve



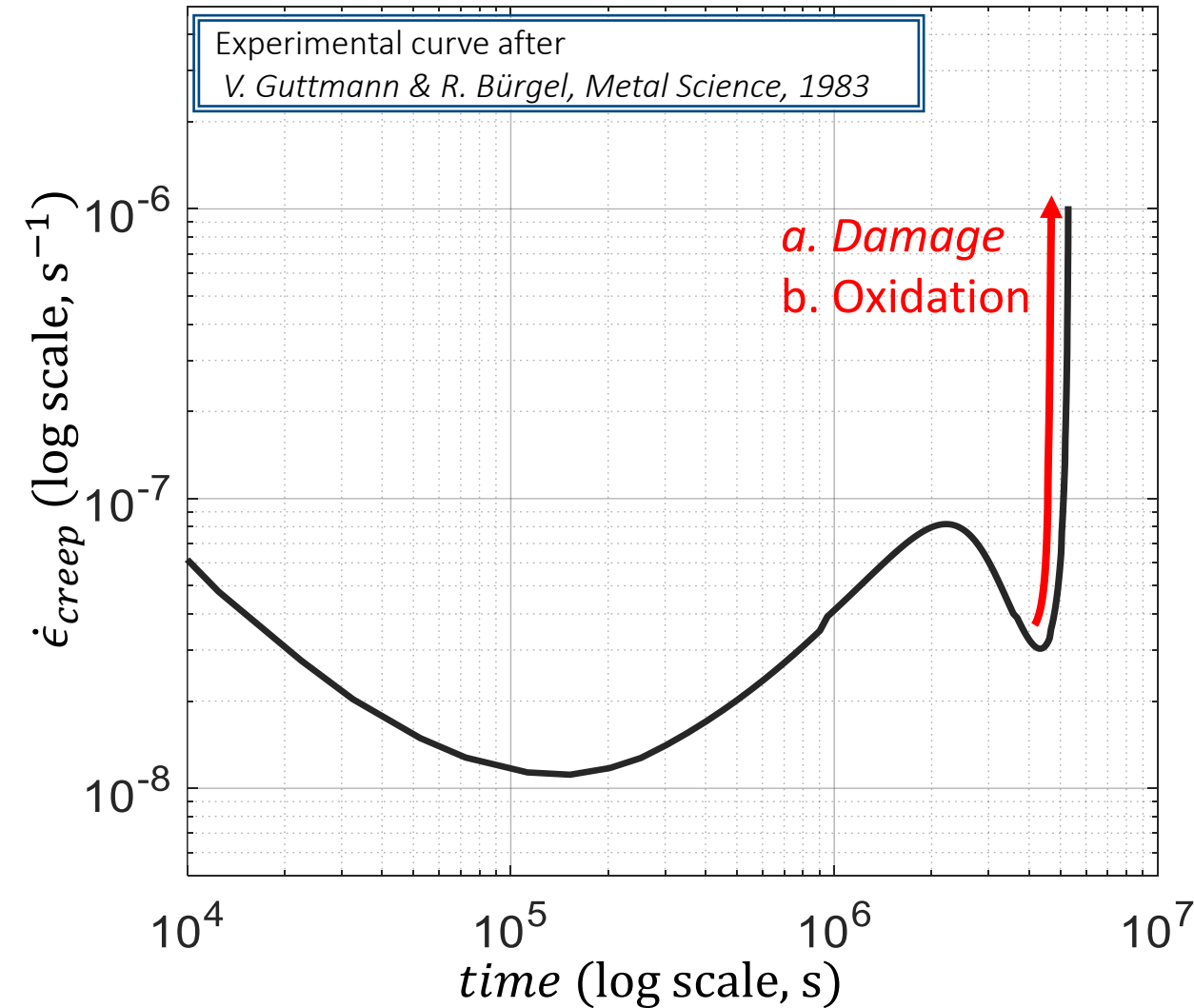
## b. Precipitation of $\text{Cr}_{23}\text{C}_6$ carbides

- Intergranular precipitation of  $\text{Cr}_{23}\text{C}_6$  carbides
- Grain boundary particle strengthening

BSE image of central sector of 800H sample after 2 years in furnace  
—ULiège 2023

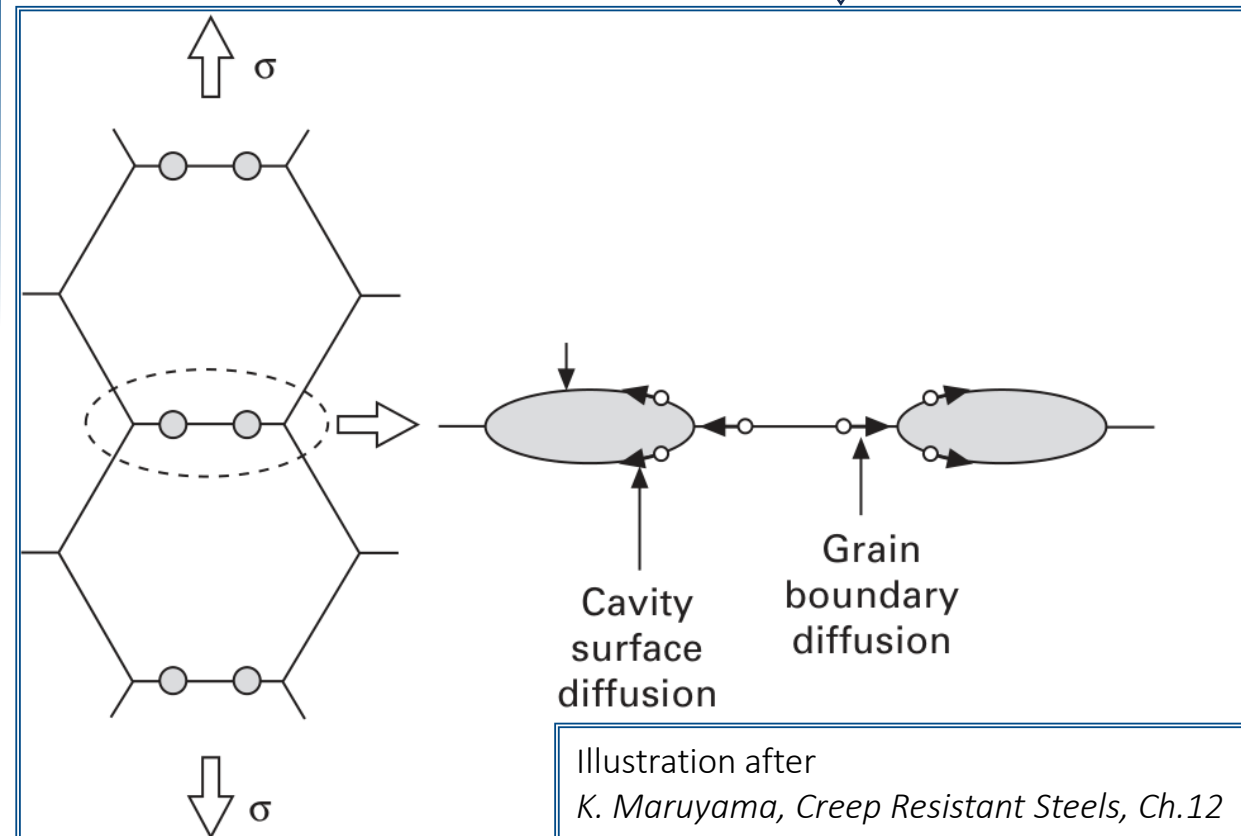


Predicted creep v/s time curve



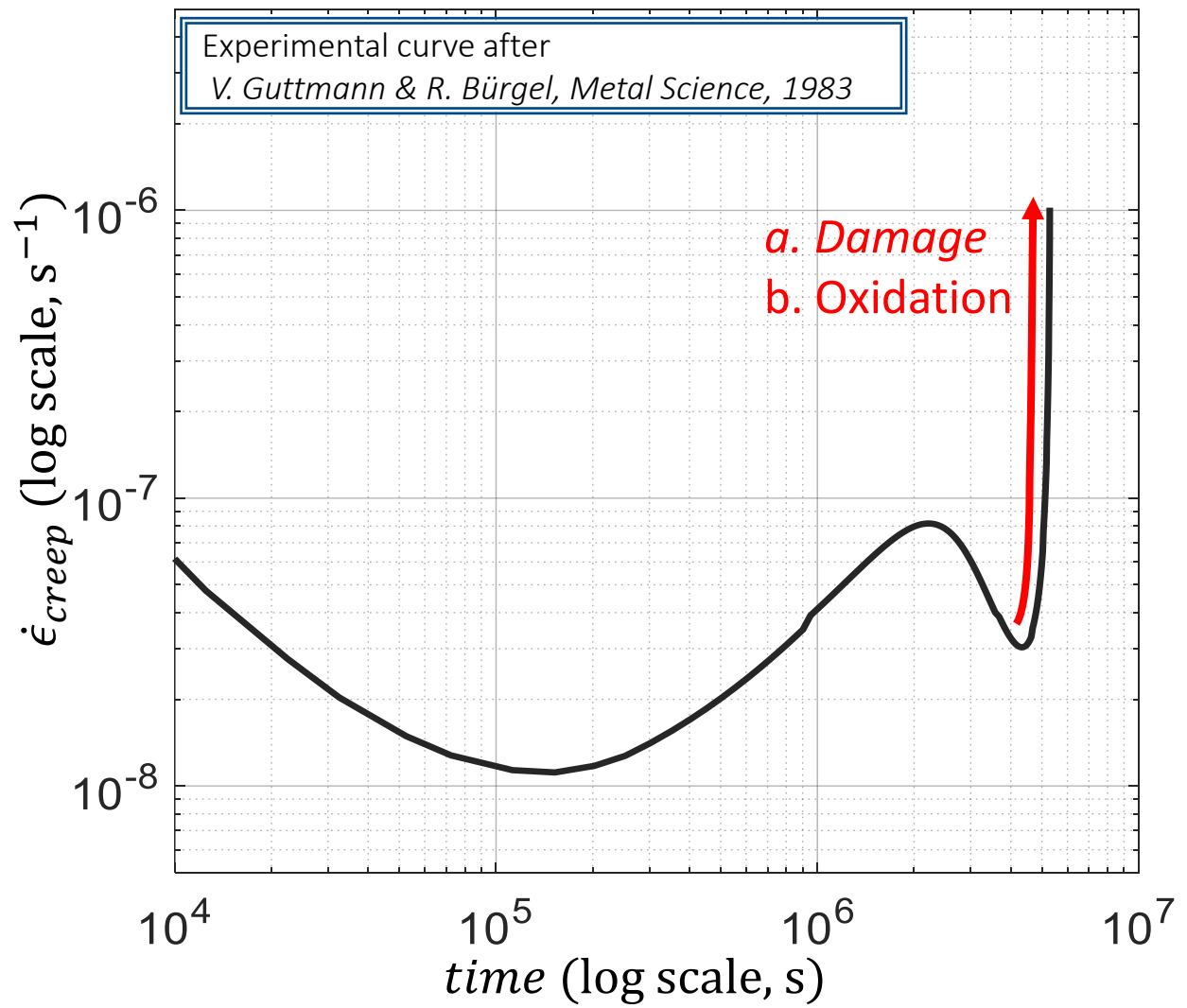
a. **Damage: grain decohesion**

- Intragranular void formation within triple joints
- Void growth at grain boundary





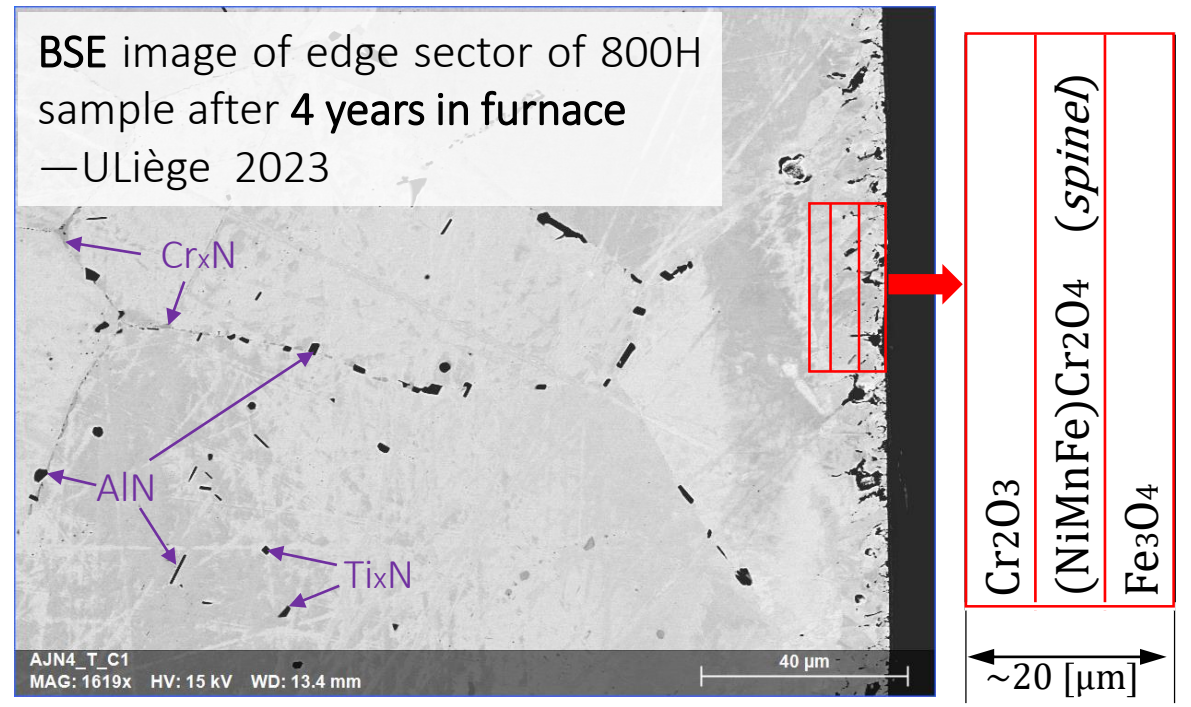
## Predicted creep v/s time curve



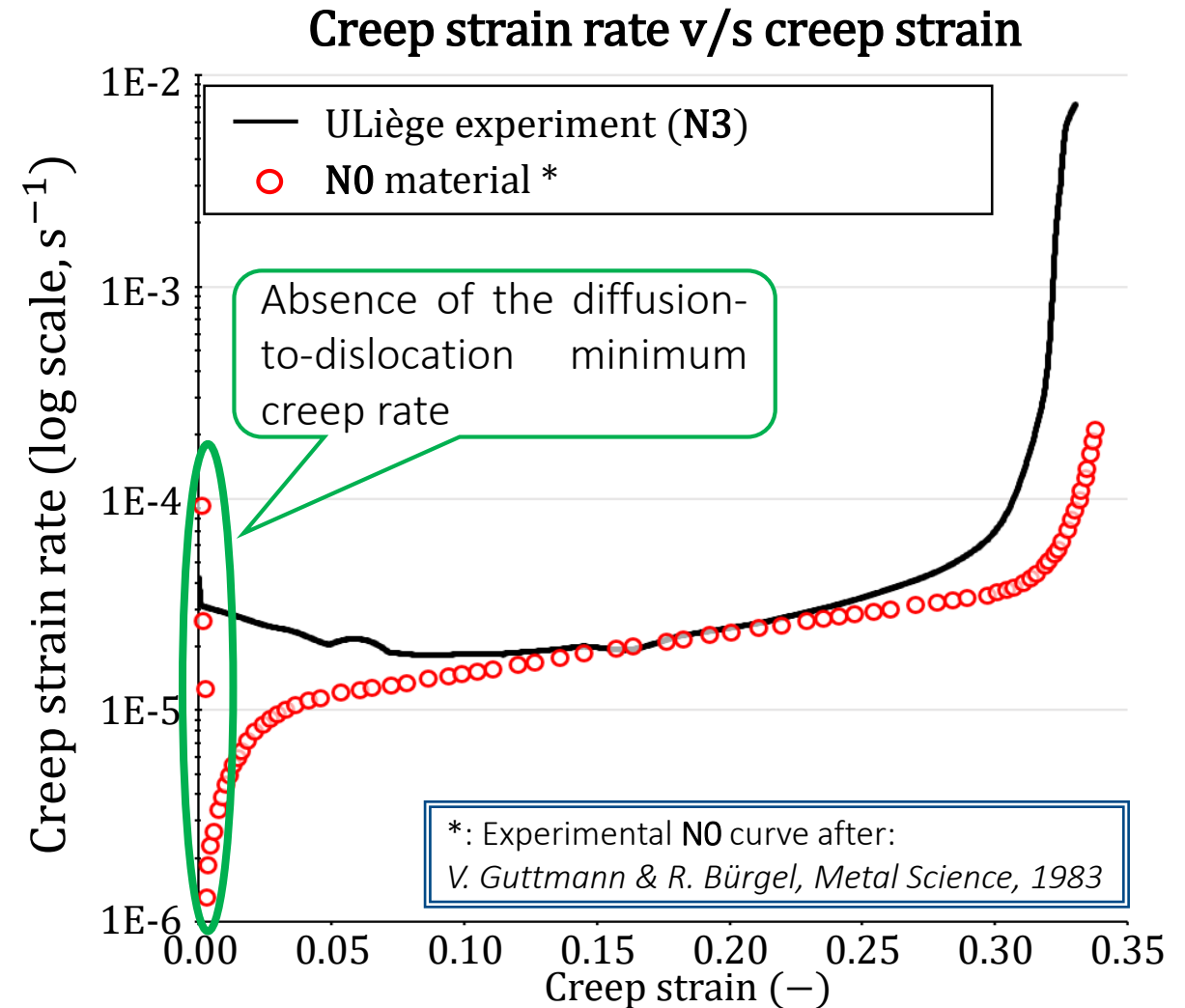
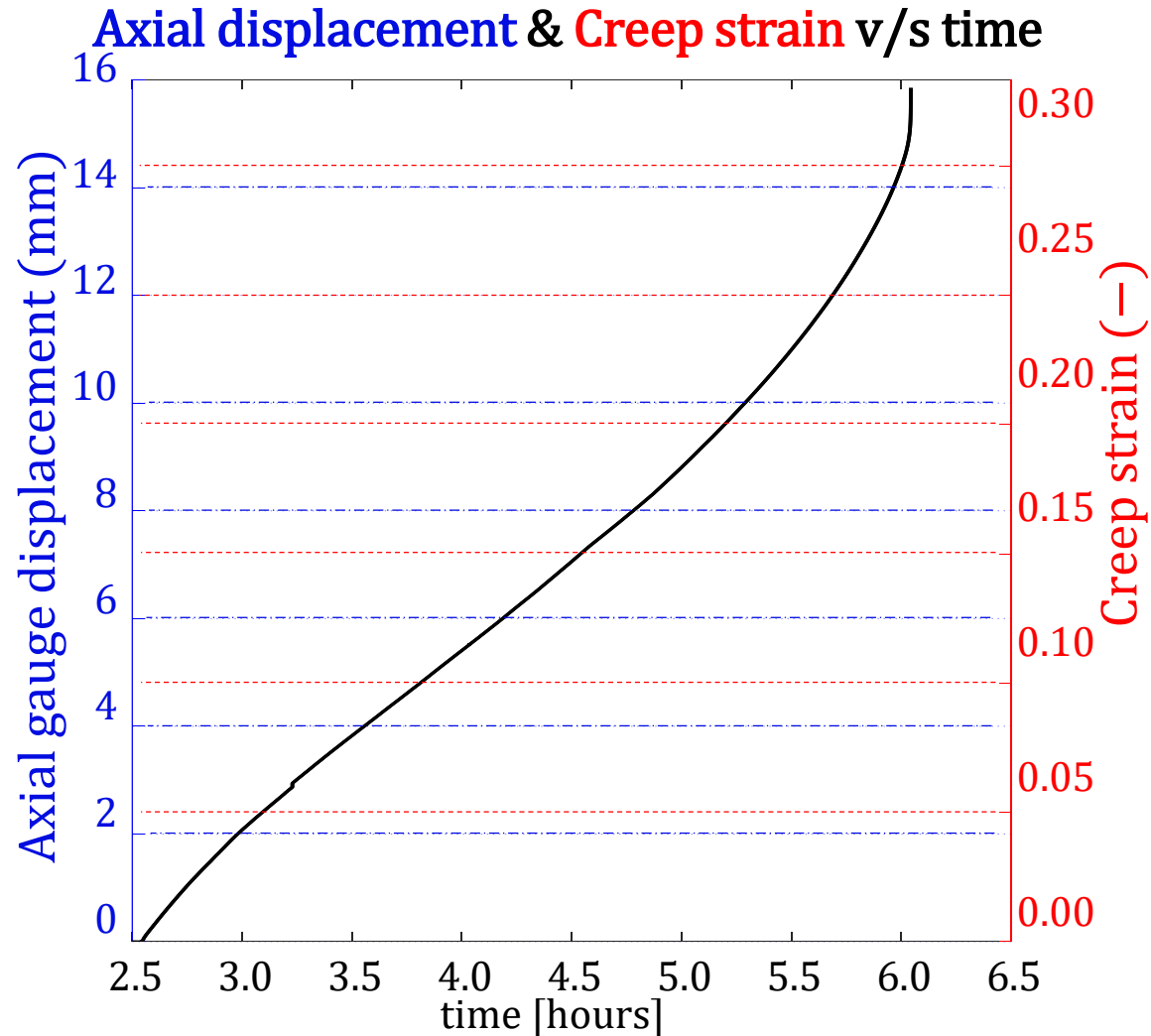
## b. Oxidation

Depleted Cr-zones due to nitridation enable deeper penetration of Fe, Mn & Cr oxides

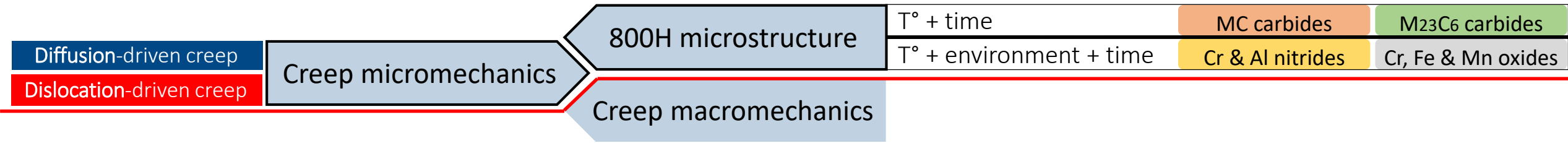
→ Favors intragranular fracture propagation



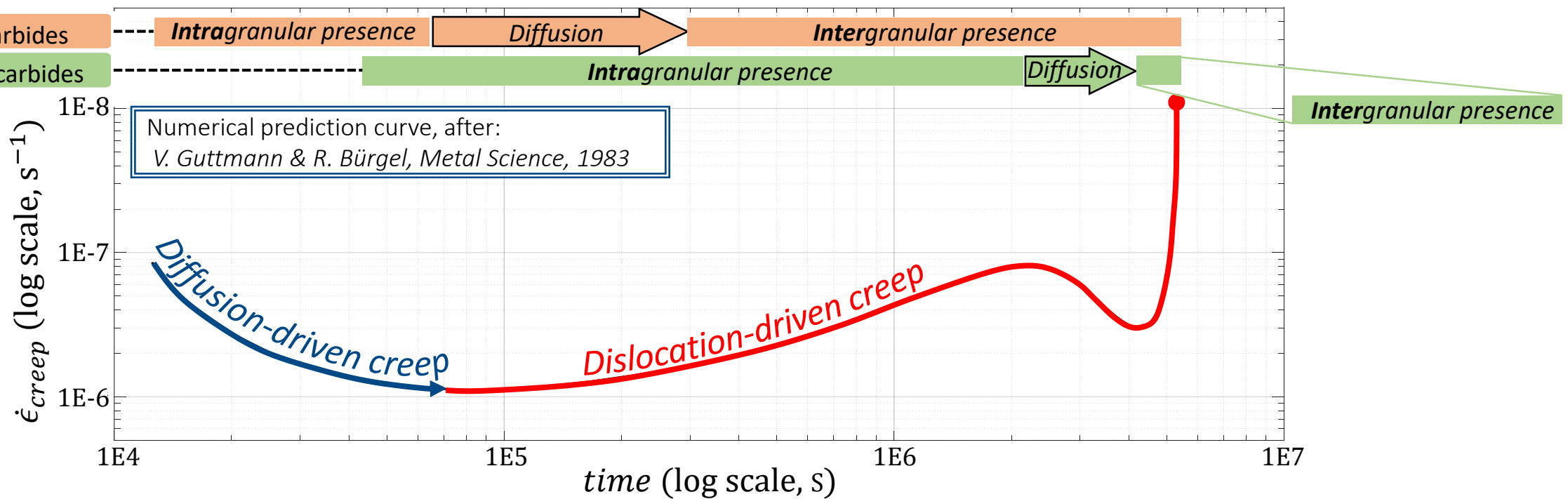
## 800H creep test on sample N3 (3 years in-furnace): 1000°C & 35 [MPa]



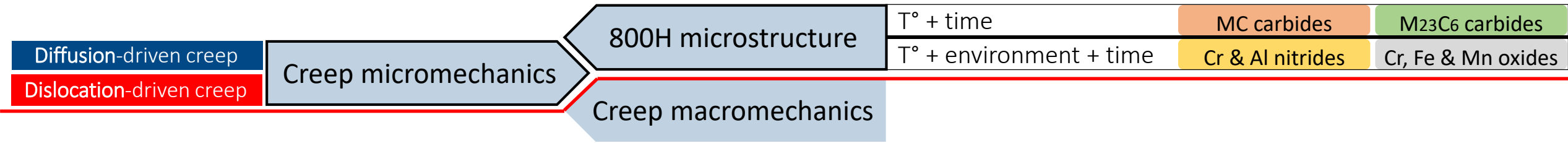
## Creep in 800H: a multiscale phenomenon



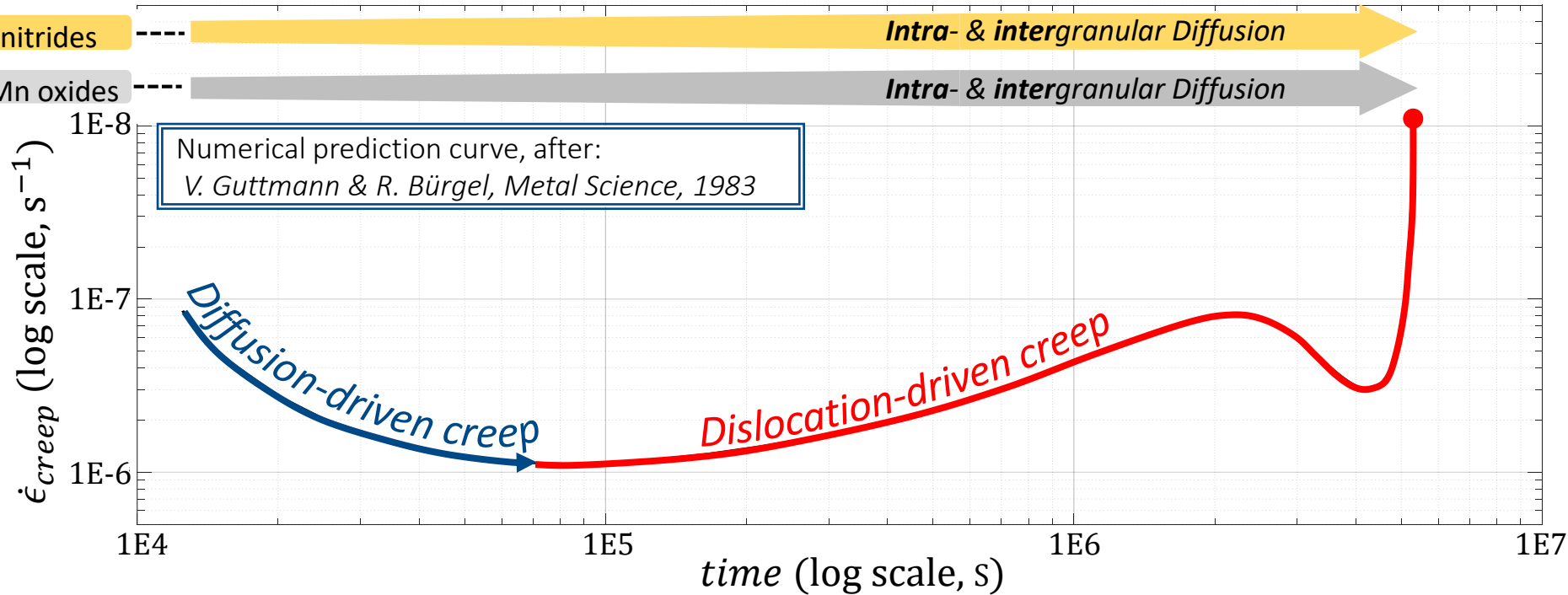
Creep rate v/s time (log. Scale) curve for 800H exhibiting non-classical behaviour



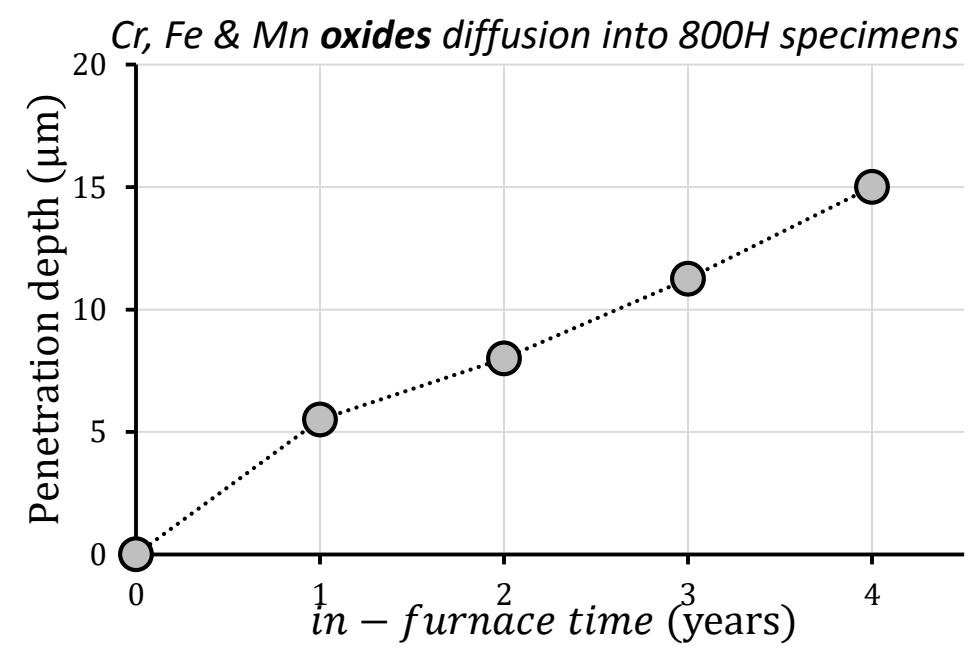
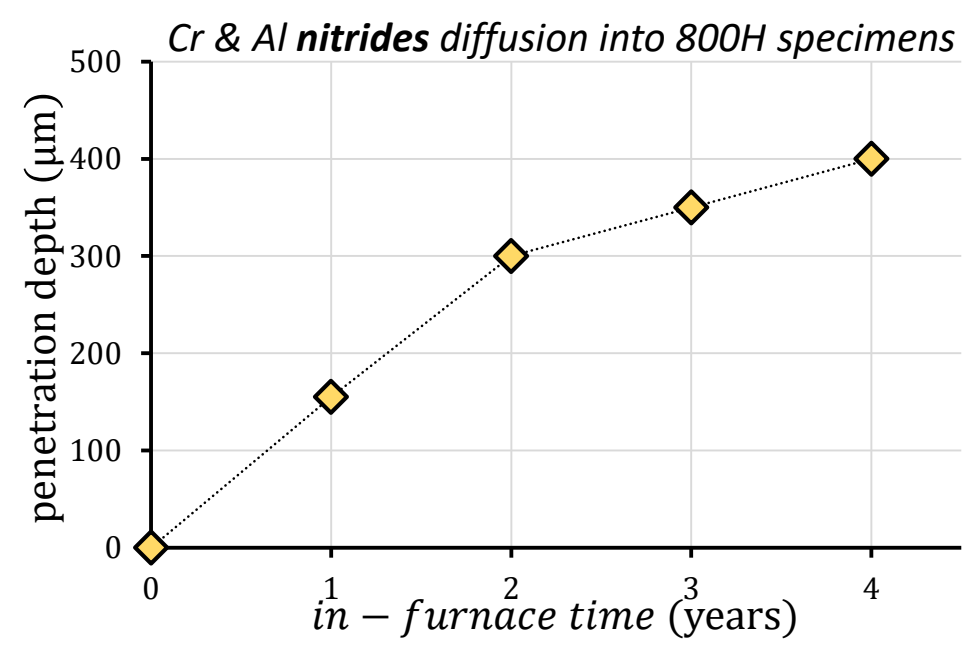
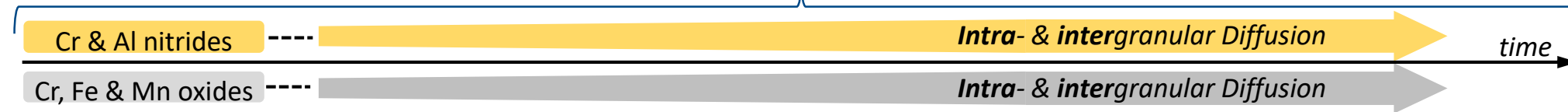
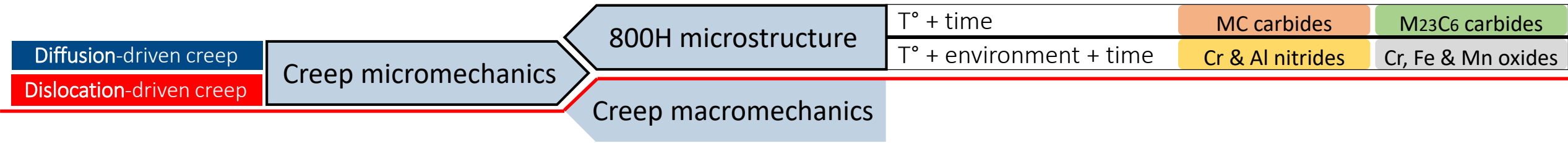
## Creep in 800H: a multiscale phenomenon



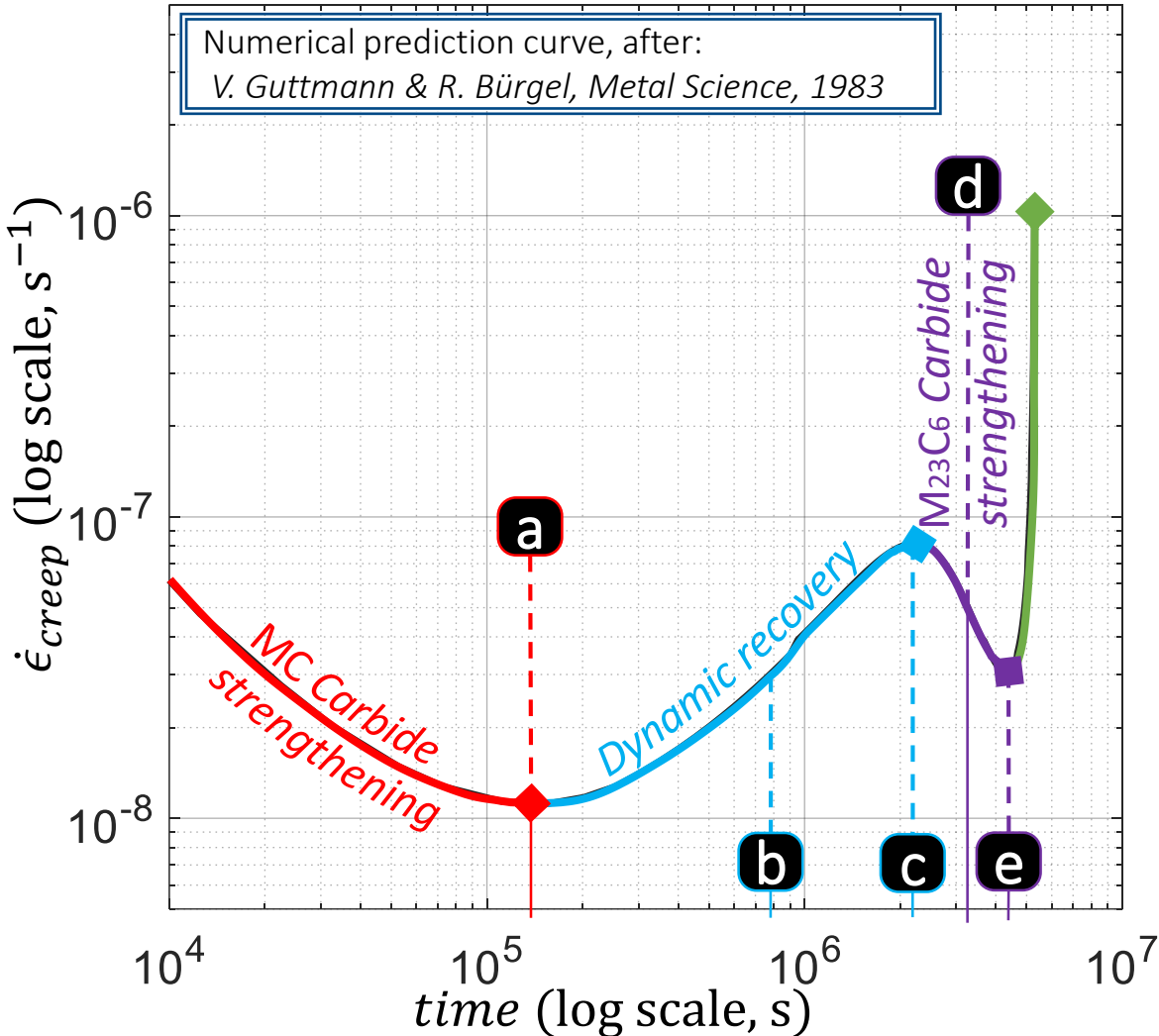
Creep rate v/s time (log. Scale) curve for 800H exhibiting non-classical behaviour



## Creep in 800H: a multiscale phenomenon



Predicted creep v/s time curve

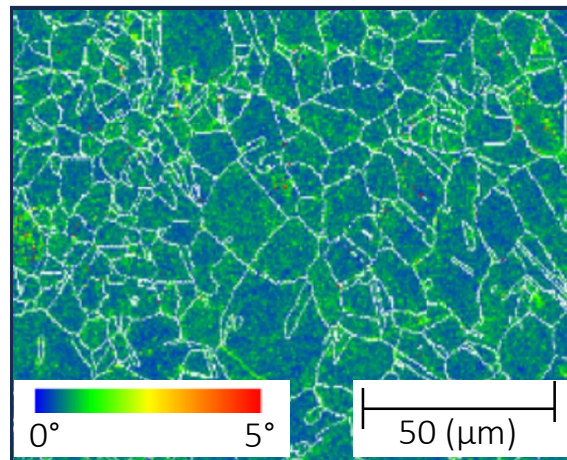


## Experimental campaign prospects

Micro characterization	Ascertain kinetics of <ul style="list-style-type: none"> <li>MC carbides</li> <li>M<sub>23</sub>C<sub>6</sub> carbides</li> <li>Cr<sub>x</sub>- and Al- nitrides</li> <li>Cr-, Mn- &amp; Fe- oxides</li> </ul> Study particle-dislocation interactions Study nitridation-oxidation interaction
Methodology	Perform <ul style="list-style-type: none"> <li>FESEM</li> <li>EDX</li> <li>TEM</li> <li>EBSD</li> </ul> and creep tests on 800H samples N0, N1, N2, N3, N4 <div style="float: right; text-align: center;"> <math>\left. \begin{matrix} a \\ b \\ c \\ d \\ e \end{matrix} \right\}</math> </div>

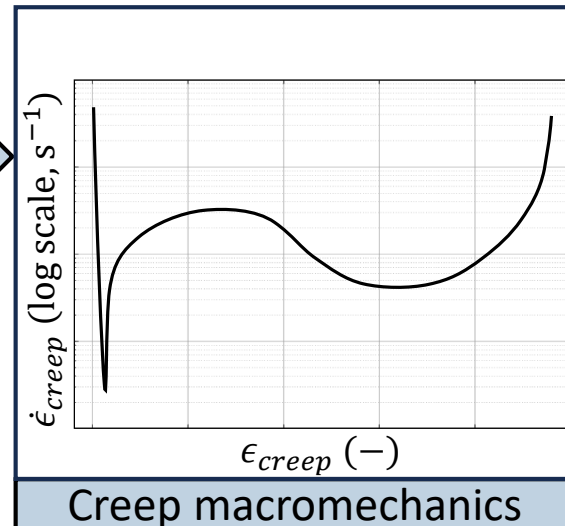
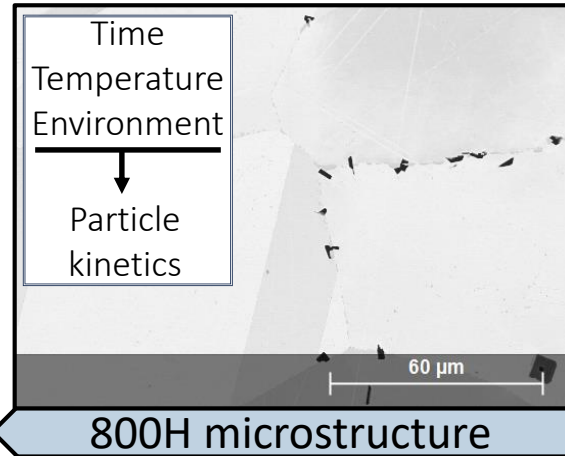


## Multiscale modelling: meso-to-macro scale



Creep micromechanics

KAM map of austenitic Ni-alloy after:  
T. Hatakeyama, *Mater. Sci. Eng. A*, 2022



## Numerical modelling

Development

Mesoscale modelling

- Constant microstructure
- Or evolving microstructure
- Precipitate kinetics
- Diffusion & dislocation creep

Validation

Simulation of real tests and results analysis

Prospects

Generation of reliable virtual tests  
for better identification, better modelling

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[anne.habraken@uliege.be](mailto:anne.habraken@uliege.be)

[ce.rojasulloa@uliege.be](mailto:ce.rojasulloa@uliege.be)

July 3<sup>rd</sup>, 2023



LIÈGE université  
Sciences Appliquées



*Materials  
and Solid  
Mechanics*



# Post Doc position - Horizon Europe project about Creep Experiments and Modelling

As partner of the European project entitled



**AI powered characterization and modelling for green steel technology ,**

the MSM Materials and Solid Mechanics team led by prof. L. Duchêne and AM Habraken is recruiting a Post-Doc researcher (a salary, not a grant).

Duration: 3 years, starting on 1 September 2023,

[anne.habraken@uliege.be](mailto:anne.habraken@uliege.be)