



# Effects of microstructural parameters on creep flow and fracture of a 9Cr-1Mo-NNbV tempered martensitic steel

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# Effects of microstructural parameters on creep flow and fracture of a 9Cr-1Mo-NNbV (ASME Grade 91) tempered martensitic steel

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# BACKGROUND/ISSUE 9%Cr TEMPERED MARTENSITIC STEELS

## ■ Considered:

→ nuclear fusion reactors:

- tokamak blankets.

→ SFR (Generation IV) :

- steam generator and secondary piping.

**Need good mechanical properties :  
creep resistance at 550 - 650°C**

# BACKGROUND/ISSUE

## 9%Cr TEMPERED MARTENSITIC STEELS

### Considered:

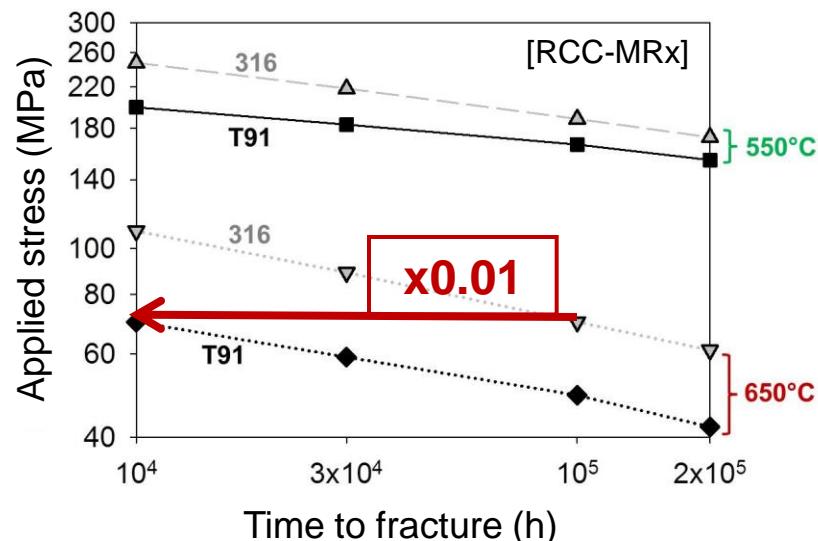
- nuclear fusion reactors:
- tokamak blankets.
- SFR (Generation IV) :
- steam generator and secondary piping.

**Need good mechanical properties:  
creep resistance at 550 - 650°C**

**Limited creep strength** compare to austenitic stainless steels

### Creep at 550°C and 650°C

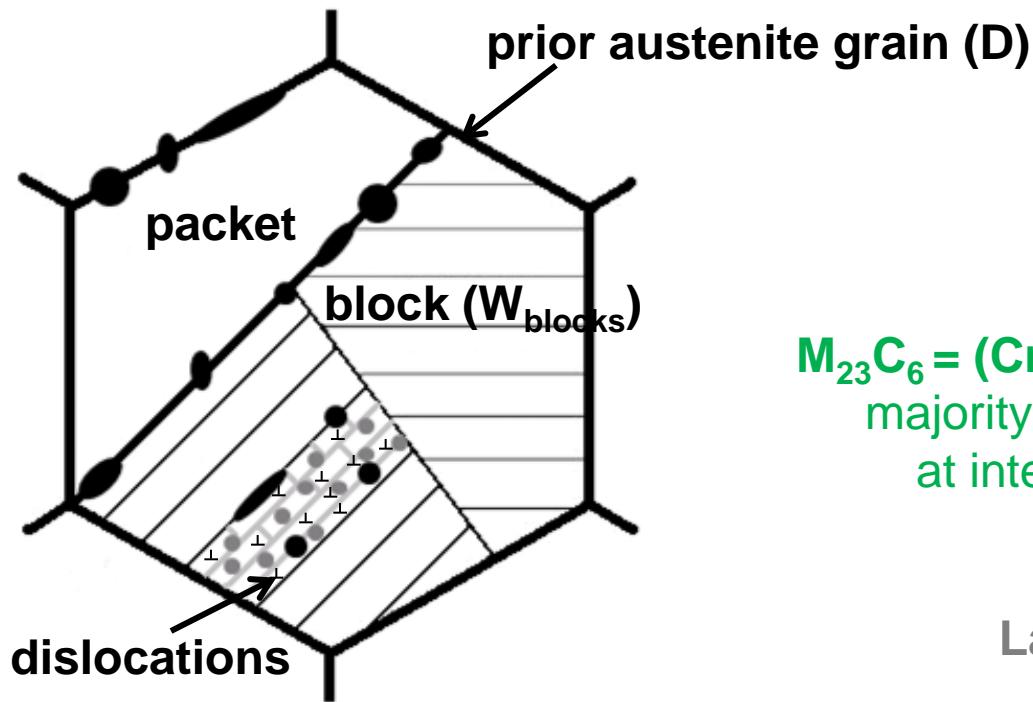
T91 =  
« standard »  
thermal  
treatment



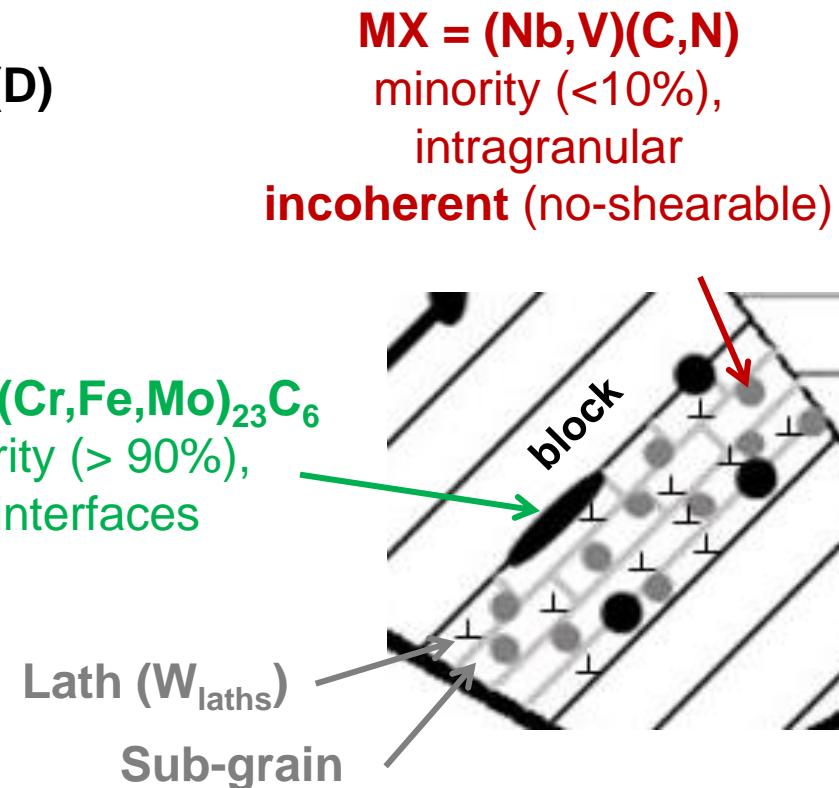
**Need of increasing  
strength**

# COMPLEX MICROSTRUCTURE OF GRADE 91 STEEL

- Simplified chemistry (wt %): Fe-9Cr-1Mo-0,1C-(Nb,V)
- Austenitized at 1050°C, 30 min, air cooled, tempered at 780°C, 1h, air cooled.
- Tempered martensite: multiscale microstructure



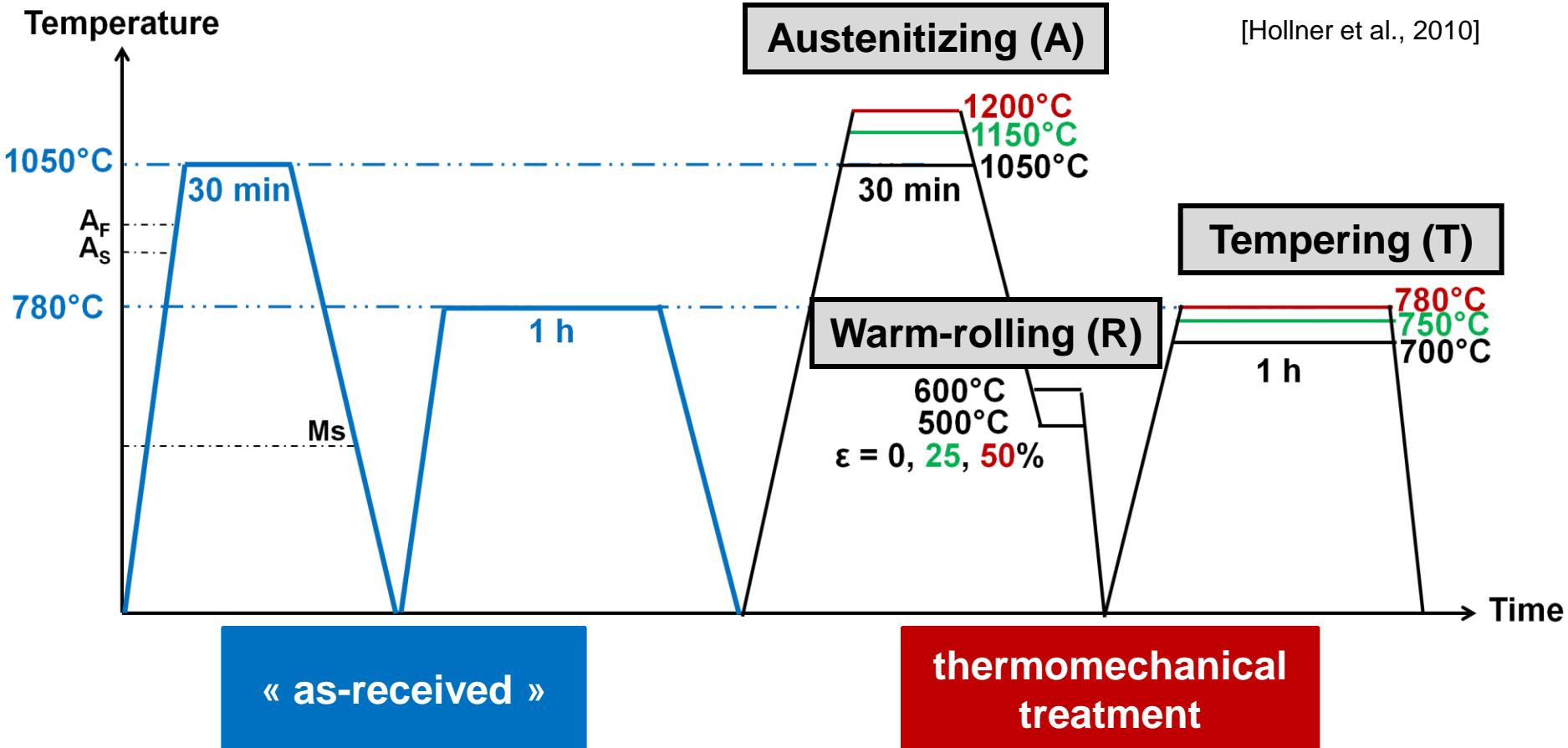
$M_{23}C_6 = (Cr,Fe,Mo)_{23}C_6$   
majority (> 90%),  
at interfaces



Microstructural parameters relevant to creep strength:  
 $D$ ,  $W_{blocks}$ ,  $W_{laths}$ , precipitates, dislocation density

# THERMOMECHANICAL TREATMENT

To vary microstructural parameters as independently as possible:  
thermomechanical treatment based on « ausforming »



# OUTLINE

More than 30 treated conditions



4 states

Part 1

$T_{\text{tempering}}$

$T_{\text{austenitizing}}$

Warm-rolling

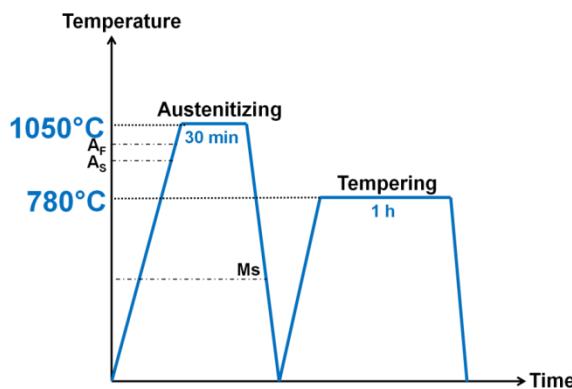
Time to fracture  
Minimal creep rate

Microstructure

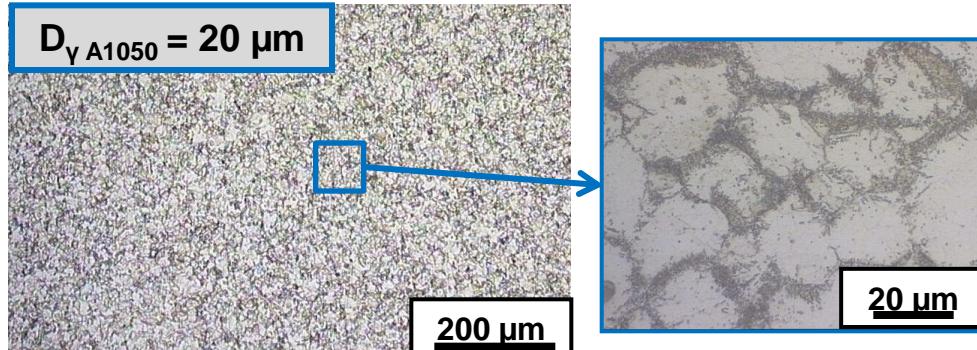
Part 2

Creep fracture mode

# “AS RECEIVED”: A1050-T780

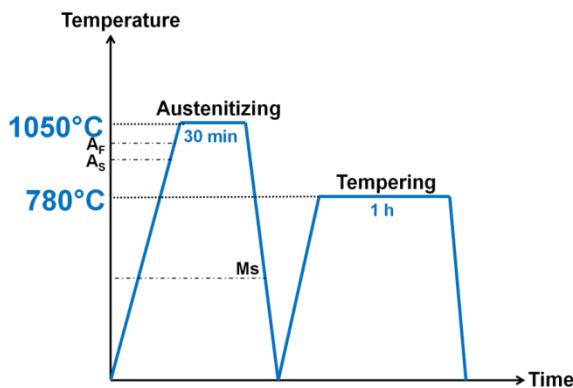


« as received »: A1050-T780

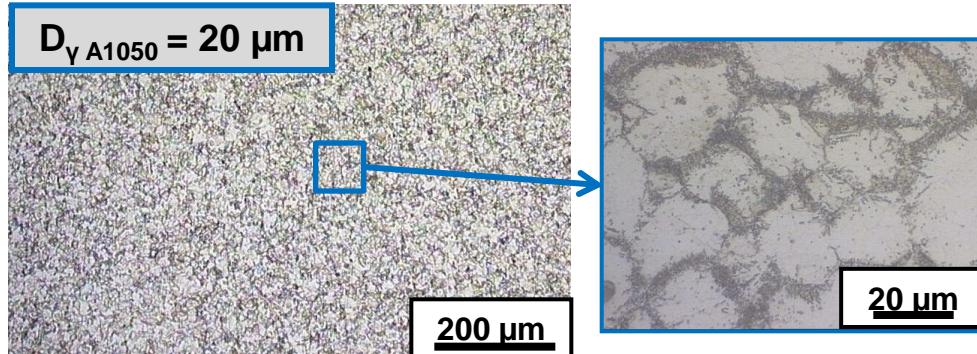


Fine prior austenite grain size

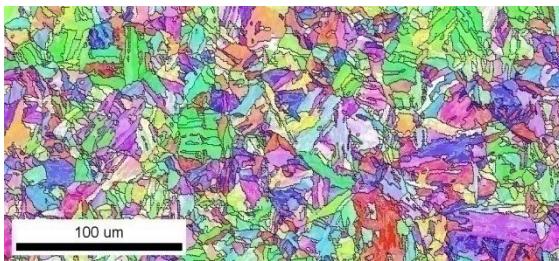
# “AS RECEIVED”: A1050-T780



« as received »: A1050-T780



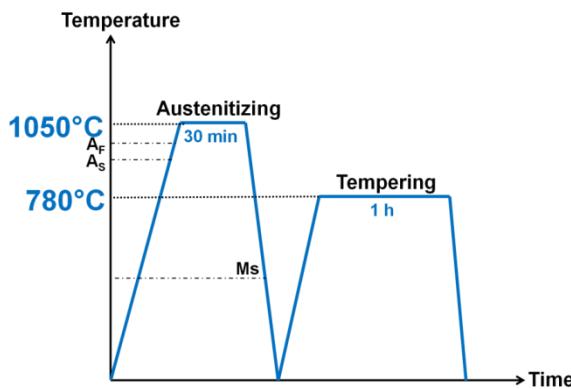
Fine prior austenite grain size



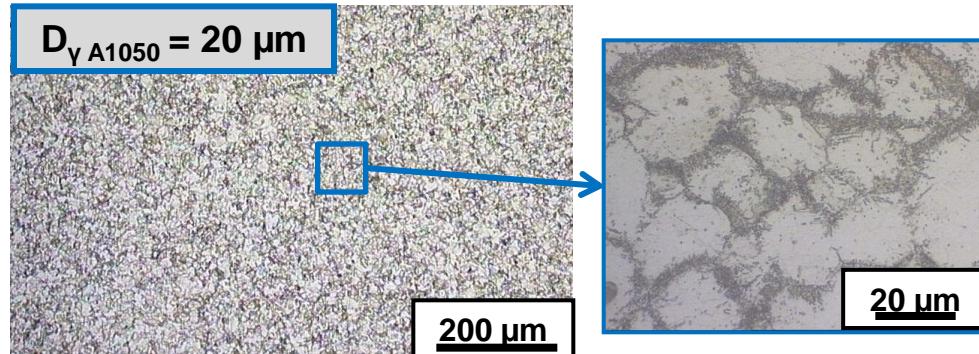
$W_{\text{blocks}} = 3.2 \mu\text{m } (*)$   
 $W_{\text{laths}} \sim 400 \text{ nm } (**)$

(\*) SEM-EBSD,  
(\*\*) TEM on thin foils.

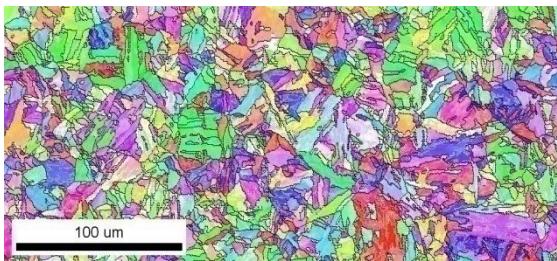
# “AS RECEIVED”: A1050-T780



« as received »: A1050-T780



Fine prior austenite grain size



$$W_{\text{blocks}} = 3.2 \mu\text{m} (*)$$

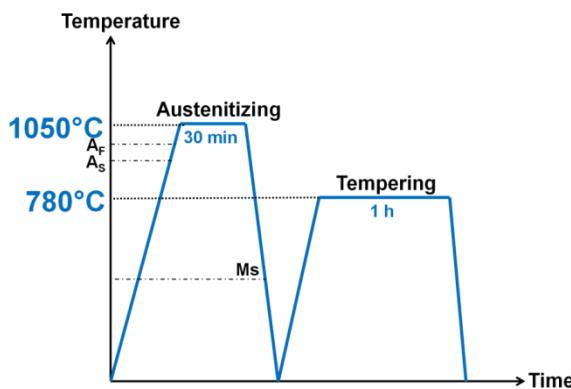
$$W_{\text{laths}} \sim 400 \text{ nm } (**)$$

(\*) SEM-EBSD,  
(\*\*) TEM on thin foils.

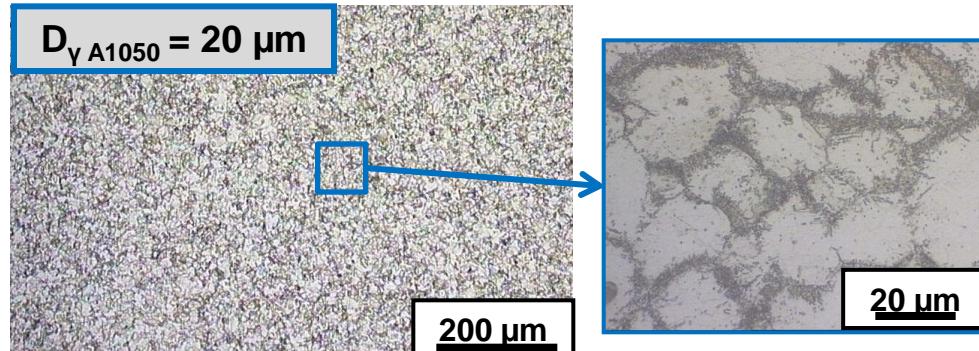
$$\rho = (0.62 \pm 0.05) \times 10^{14} \text{ m}^{-2} (***)$$

(\*\*\*) neutron diffraction (*FWHM martensite peaks*).

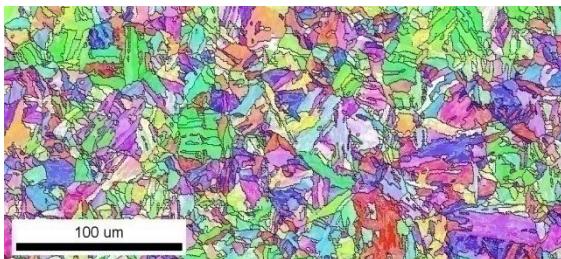
# “AS RECEIVED”: A1050-T780



« as received »: A1050-T780



Fine prior austenite grain size



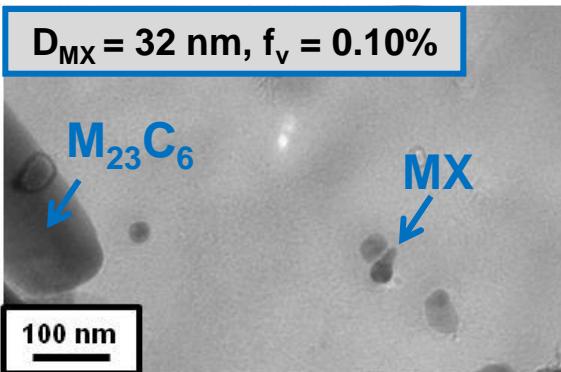
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(\*\*) TEM on thin foils.

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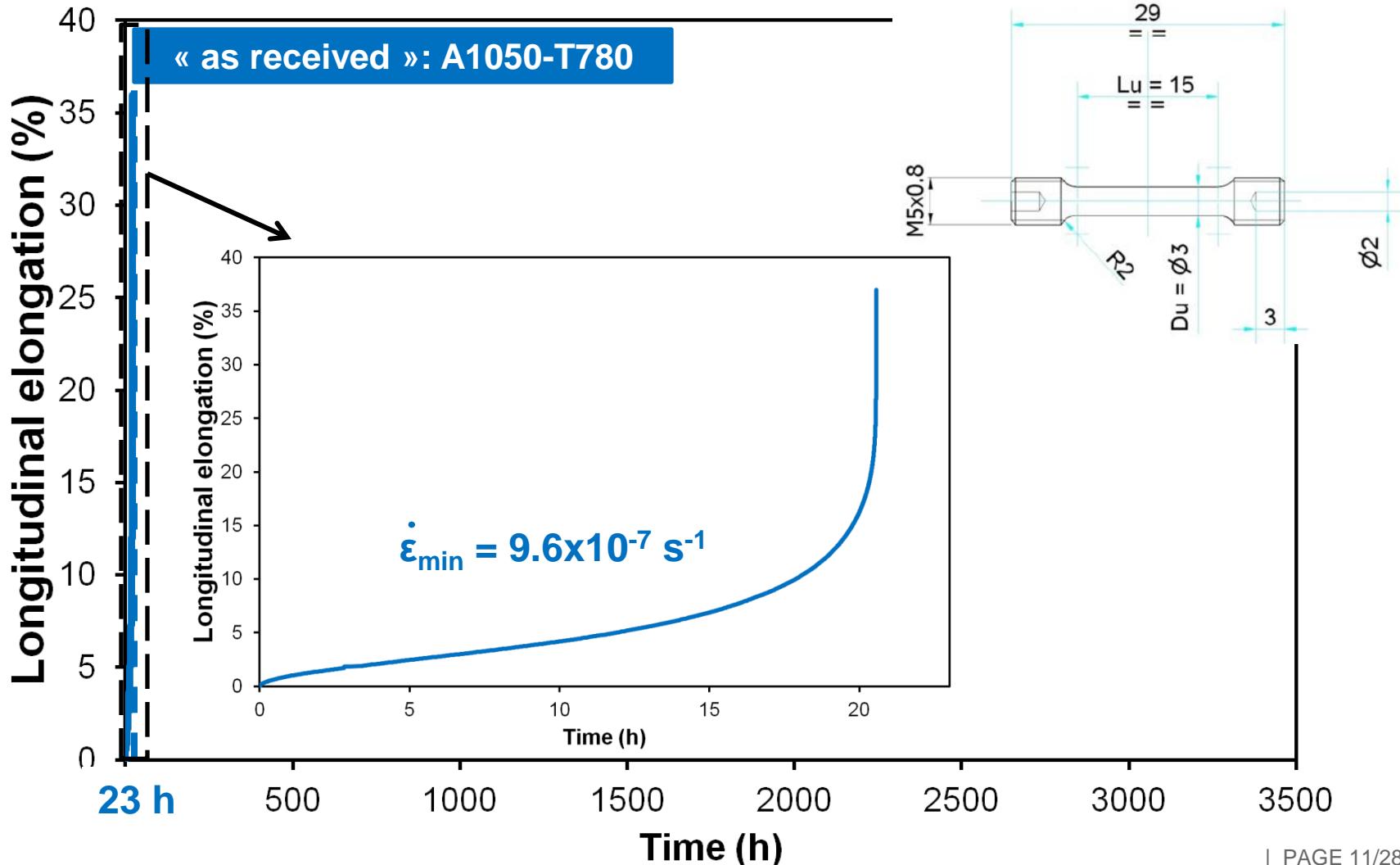
$D_{\text{MX}} = 32 \text{ nm}, f_v = 0.10\%$



Coarse  $\text{M}_{23}\text{C}_6$  precipitates at the different interfaces  
and few intragranular MX precipitates

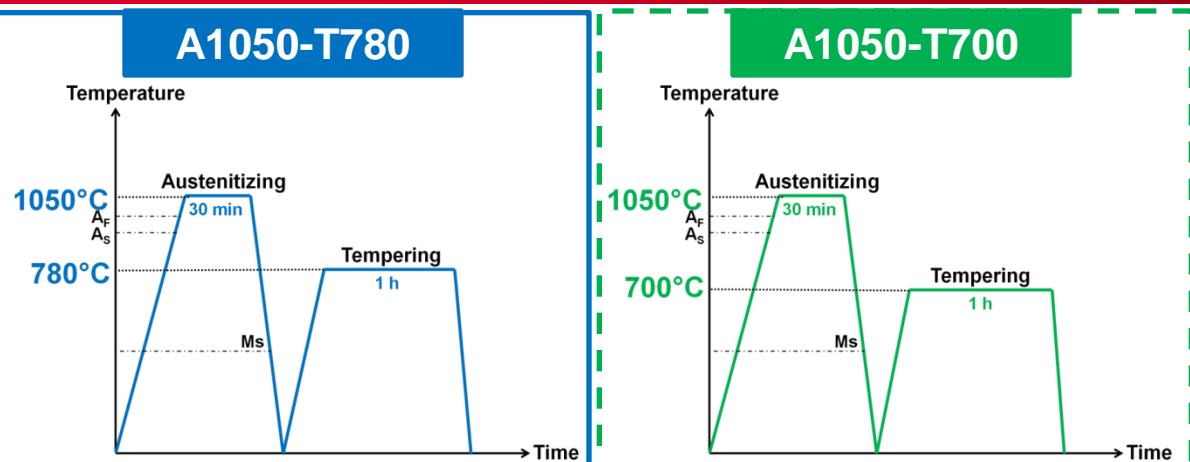
## “AS RECEIVED”: A1050-T780

- Creep conditions: 140 MPa at 650°C (*high stress and short lifetime*)



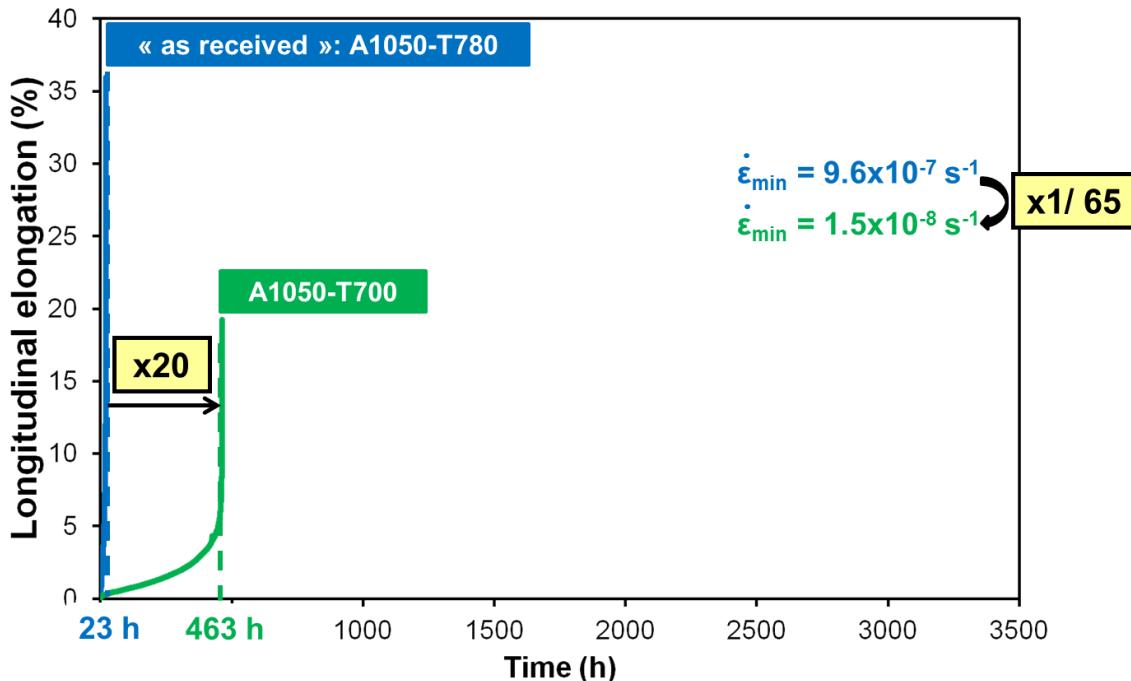
# COMPARISON “AR” VS A1050-T700

## ↓ TEMPERING TEMPERATURE FROM 780°C TO 700°C



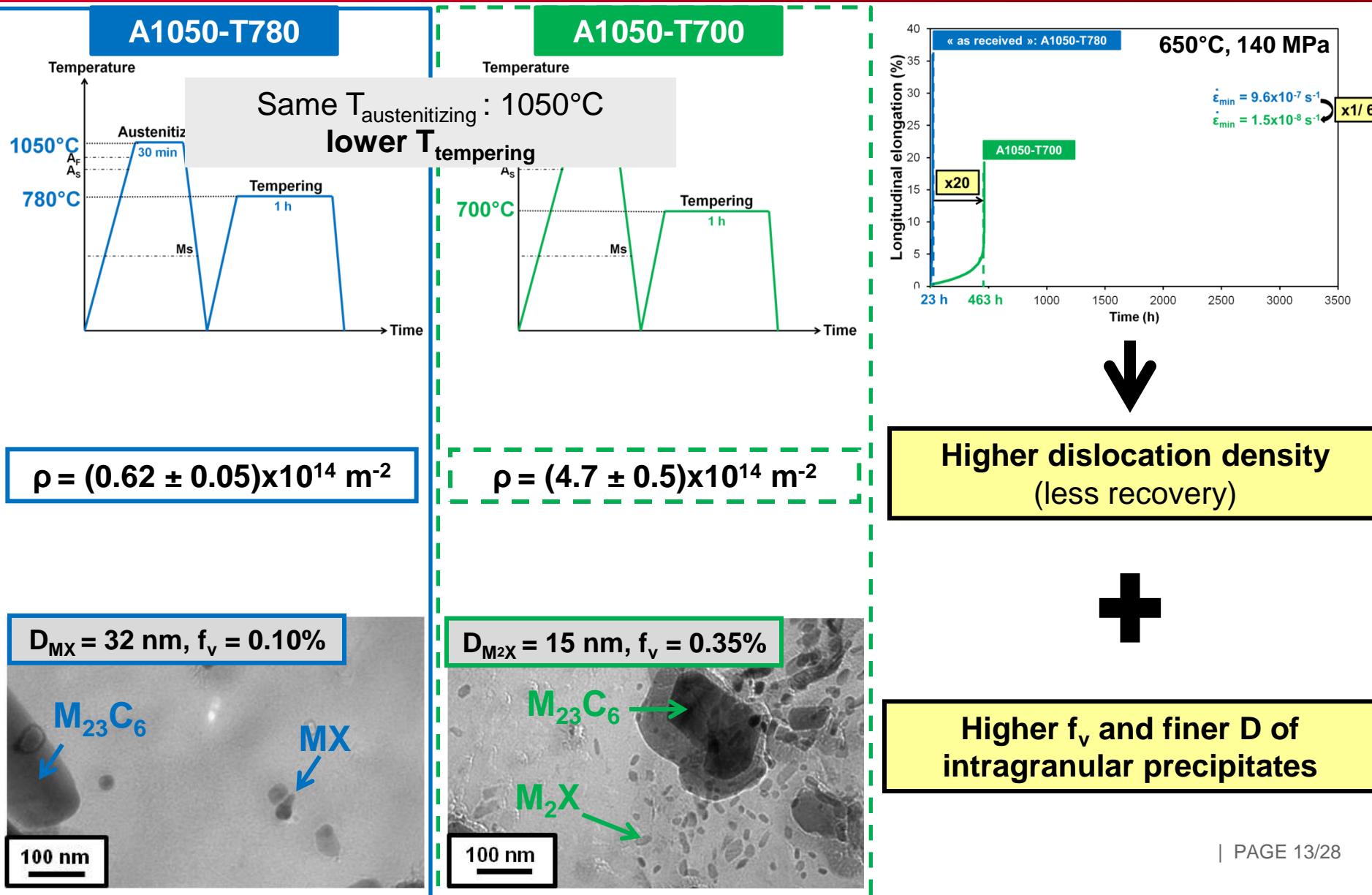
Same  $T_{\text{austenitizing}}$  : 1050°C  
lower  $T_{\text{tempering}}$

Creep conditions: 140 MPa at 650°C



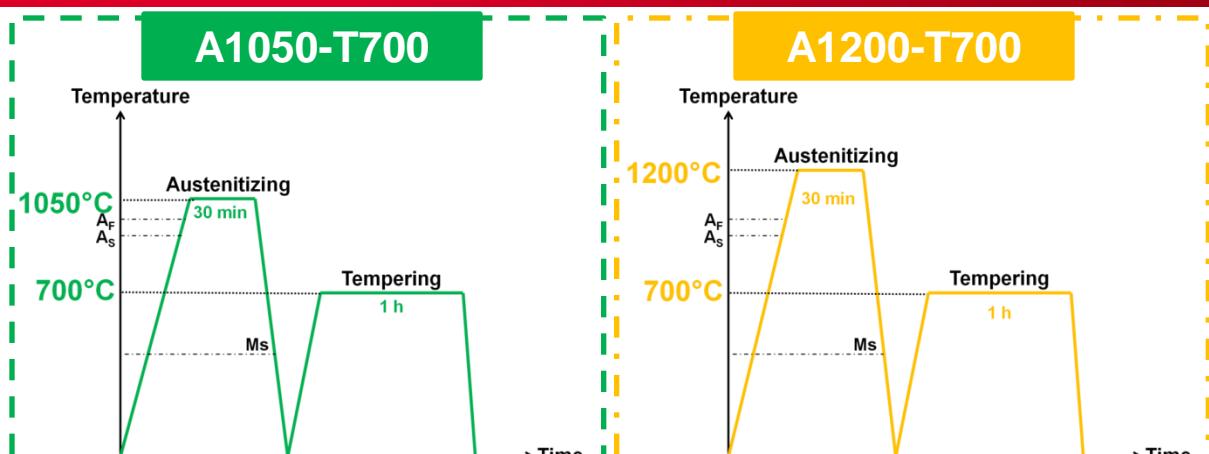
# COMPARISON “AR” VS A1050-T700

## ↓ TEMPERING TEMPERATURE FROM 780°C TO 700°C



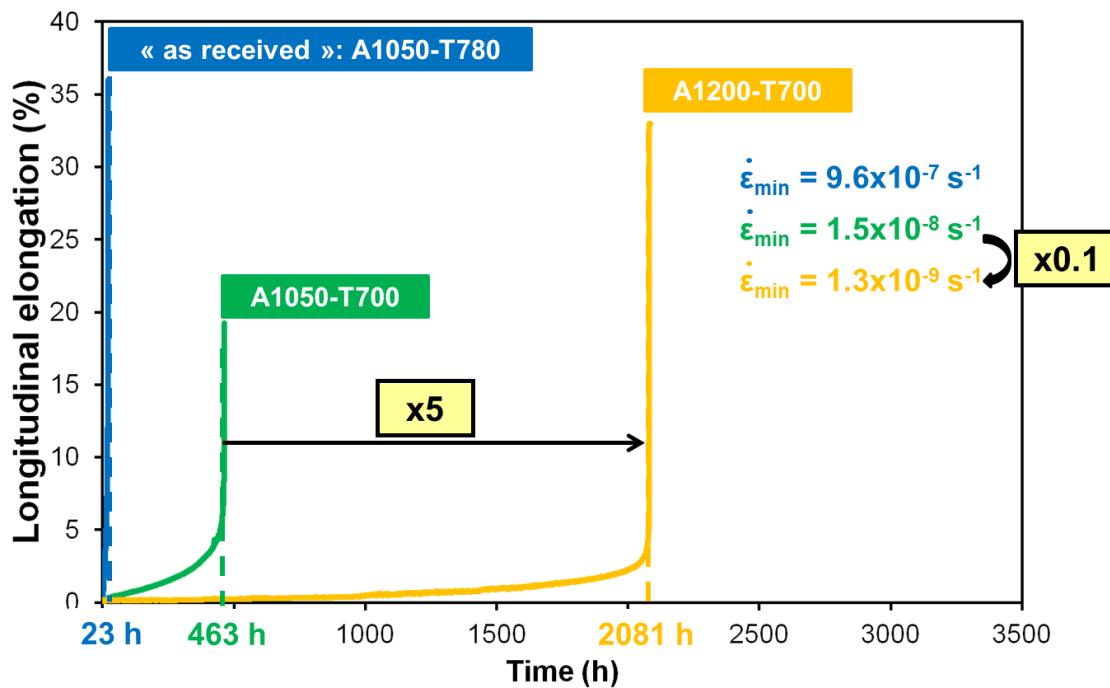
# COMPARISON A1050-T700 VS A1200-T700

↗ AUSTENITIZING TEMPERATURE FROM 1050°C TO 1200°C



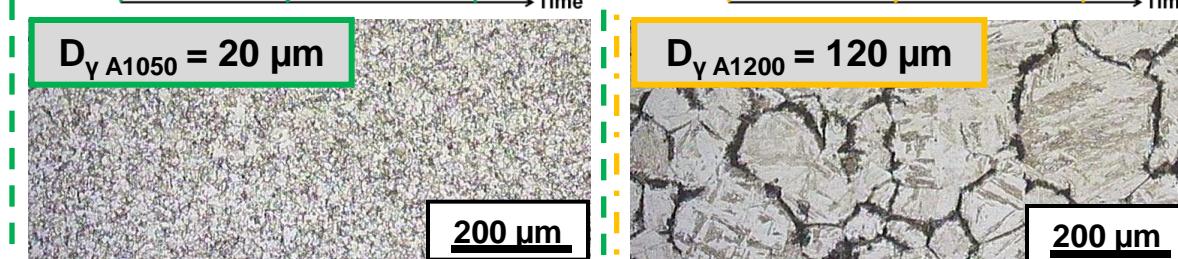
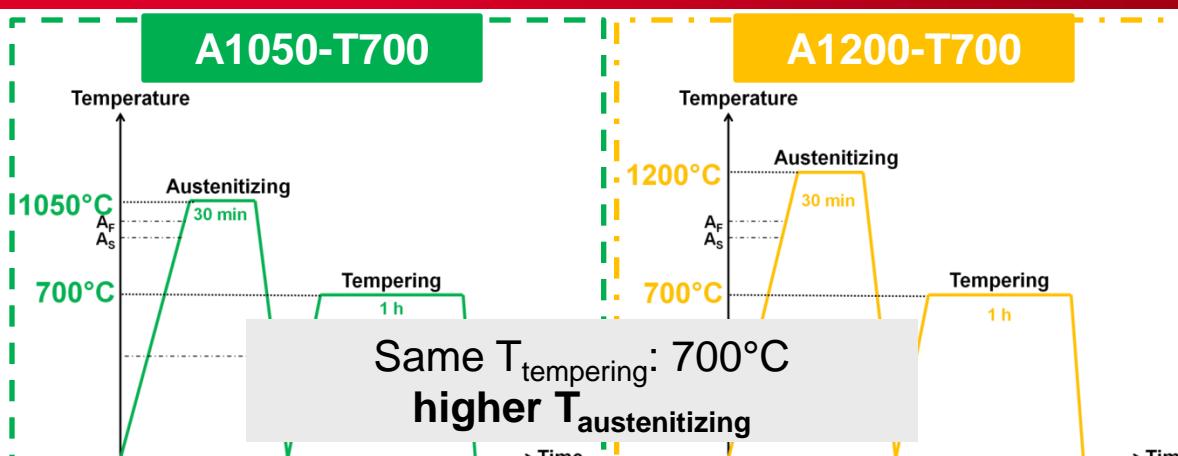
Same  $T_{\text{tempering}}$ : 700°C  
higher  $T_{\text{austenitizing}}$

Creep conditions: 140 MPa at 650°C



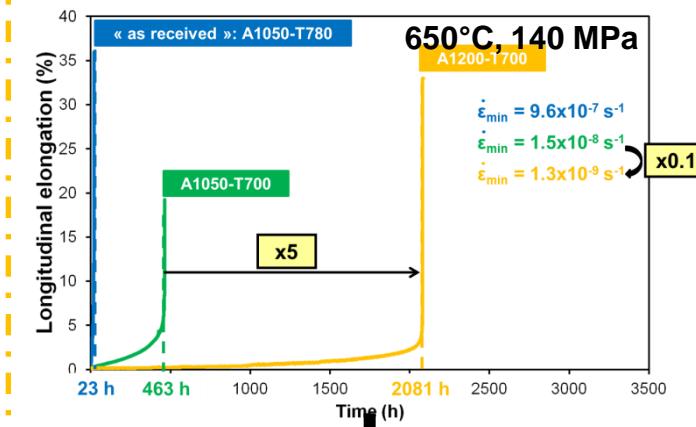
# COMPARISON A1050-T700 VS A1200-T700

↗ AUSTENITIZING TEMPERATURE FROM 1050°C TO 1200°C



$$W_{blocks} = 3.2 \mu\text{m}$$

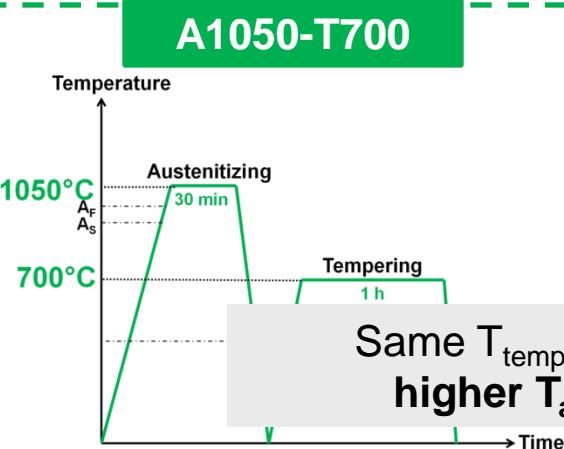
$$W_{blocks} = 4.3 \mu\text{m}$$



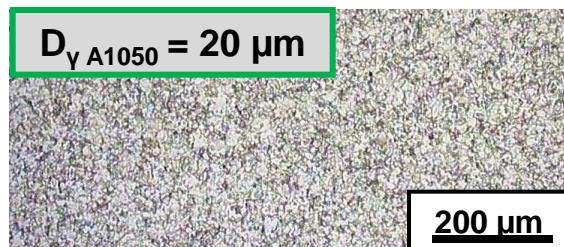
**Higher prior  $\gamma$  grain size  
(dissolution of small precipitates)**



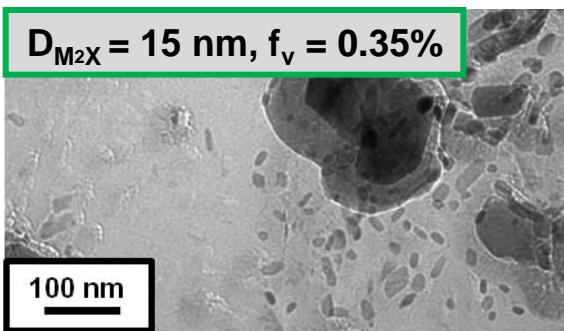
**Higher martensite block size**

**COMPARISON A1050-T700 VS A1200-T700****↗ AUSTENITIZING TEMPERATURE FROM 1050°C TO 1200°C****A1050-T700**

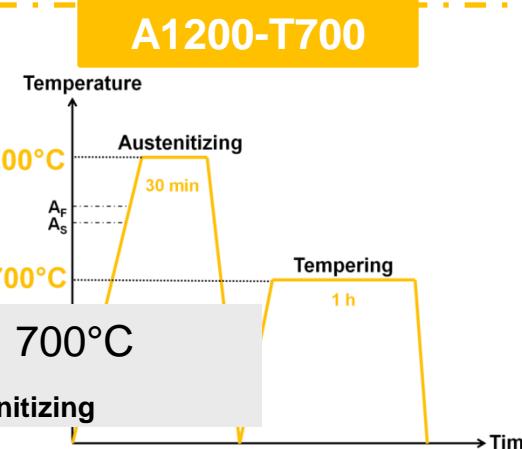
$$D_{\gamma, \text{A1050}} = 20 \mu\text{m}$$



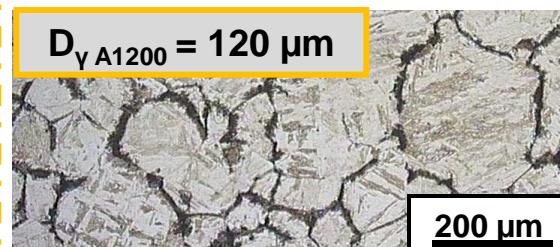
$$W_{\text{blocks}} = 3.2 \mu\text{m}$$



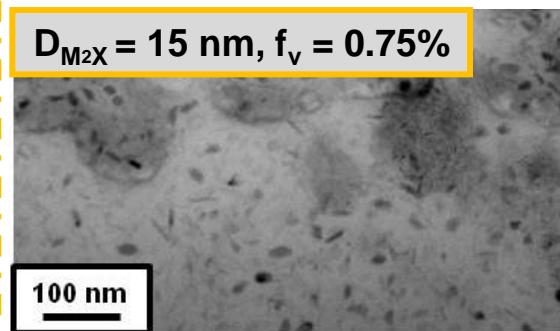
$$100 \text{ nm}$$

**A1200-T700**

$$D_{\gamma, \text{A1200}} = 120 \mu\text{m}$$

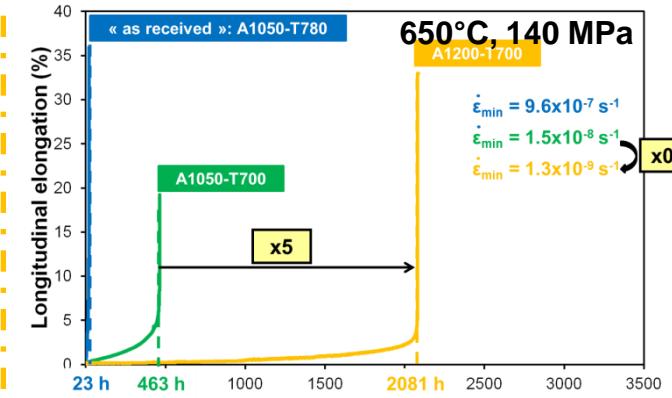


$$W_{\text{blocks}} = 4.3 \mu\text{m}$$



$$D_{M_2X} = 15 \text{ nm}, f_v = 0.35\%$$

$$D_{M_2X} = 15 \text{ nm}, f_v = 0.75\%$$



**Higher prior  $\gamma$  grain size**  
(dissolution of small precipitates)

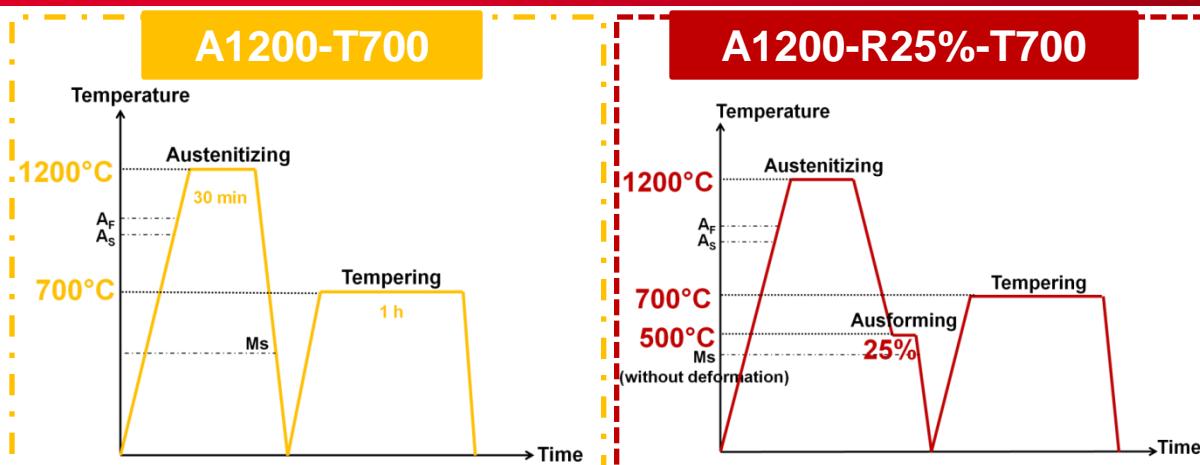


**Higher martensite block size**



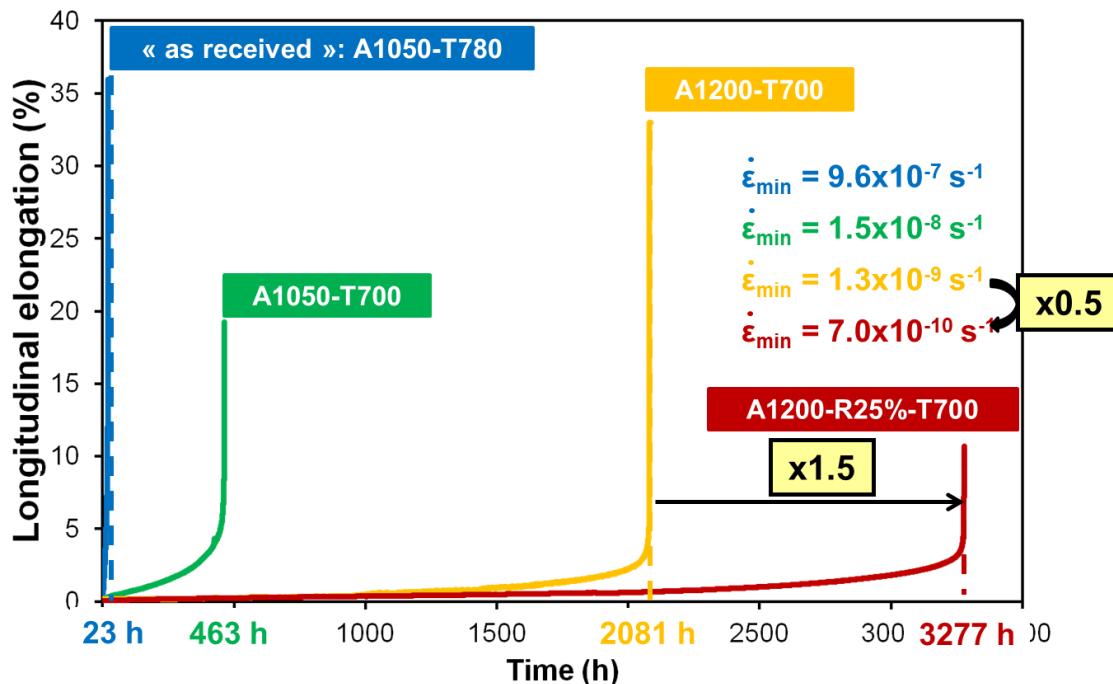
**Higher  $f_v$  of intragranular precipitates**  
(more microalloying elements available for precipitation)

# COMPARISON A1200-T700 VS A1200-R25%-T700 APPLYING WARM-ROLLING IN METASTABLE γ



Same  $T_{\text{austenitizing}}$ : 1200°C  
Same  $T_{\text{tempering}}$ : 700°C  
+ warm-rolling step

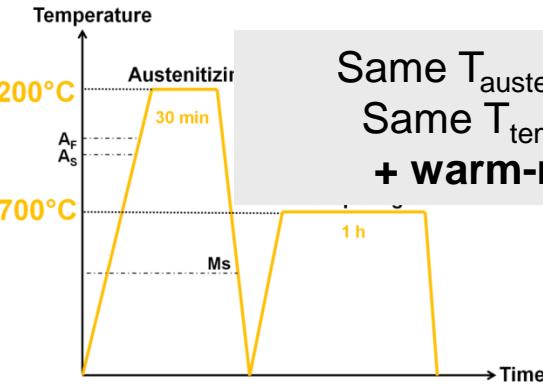
Creep conditions: 140 MPa at 650°C



# COMPARISON A1200-T700 VS A1200-R25%-T700 APPLYING WARM-ROLLING IN METASTABLE γ

## A1200-T700

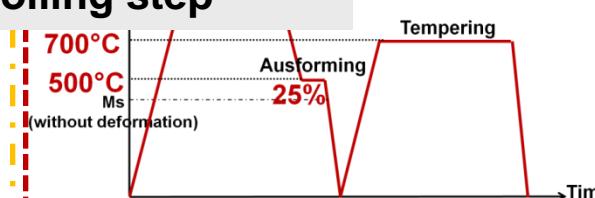
Same  $T_{\text{austenitizing}}$ : 1200°C  
Same  $T_{\text{tempering}}$ : 700°C  
+ warm-rolling step



$W_{\text{laths}} \sim 400 \text{ nm}$

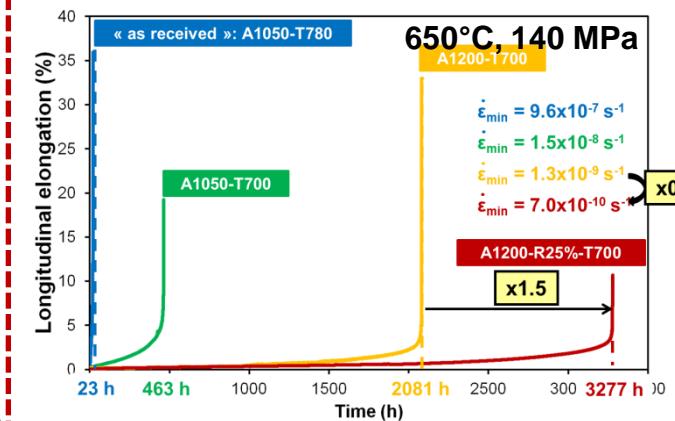
$$\rho = (4.7 \pm 0.5) \times 10^{14} \text{ m}^{-2}$$

## A1200-R25%-T700



$W_{\text{laths}} \sim 250 \text{ nm}$

$$\rho = (6.2 \pm 0.5) \times 10^{14} \text{ m}^{-2}$$



Finer lath width



Higher dislocation density  
(inheritance from warm-rolling)

# OUTLINE

More than 30 treated conditions



4 states

Part 1

$T_{\text{tempering}}$

$T_{\text{austenitizing}}$

Warm-rolling

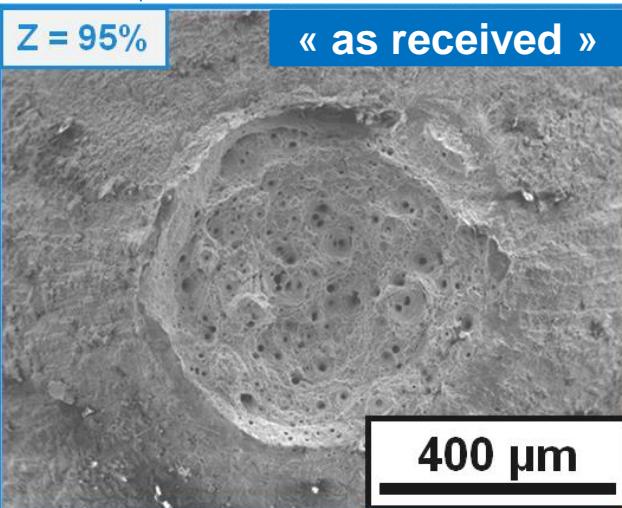
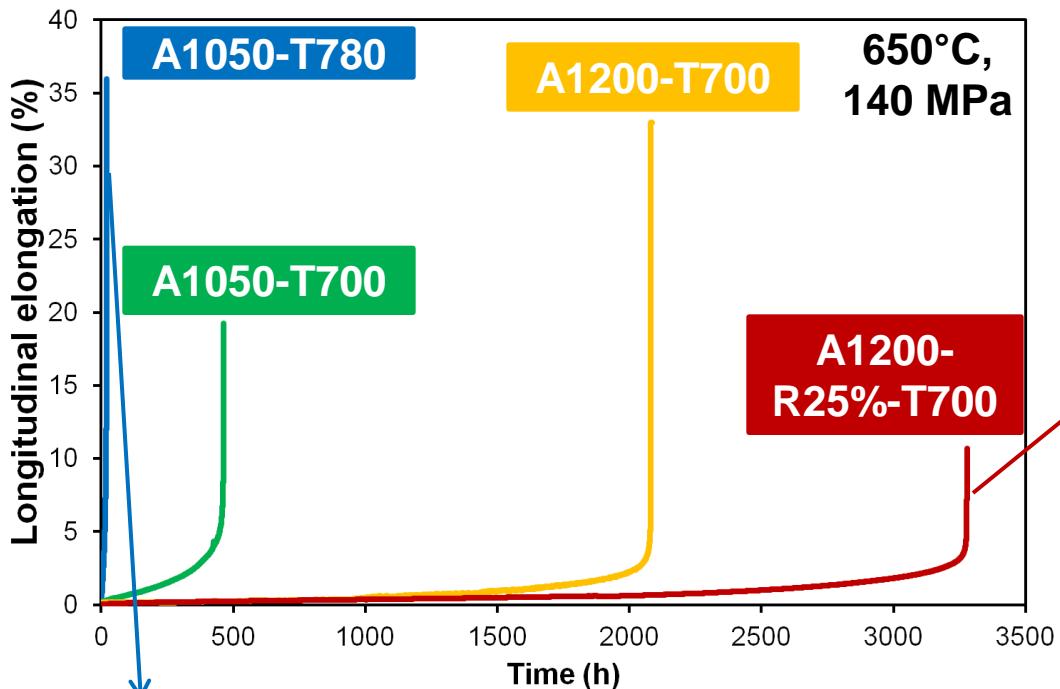
Time to fracture  
Minimal creep rate

Microstructure

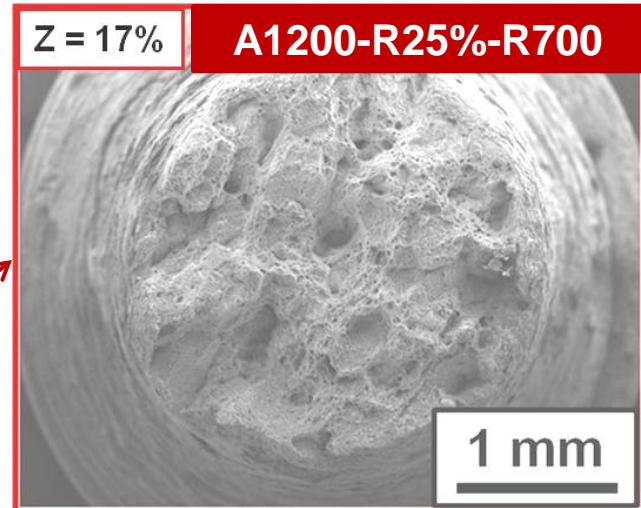
Part 2

Creep fracture mode

# CREEP FRACTURE MODE



Significant tertiary creep stage,  
high reduction of area at failure,  
**Ductile fracture.**



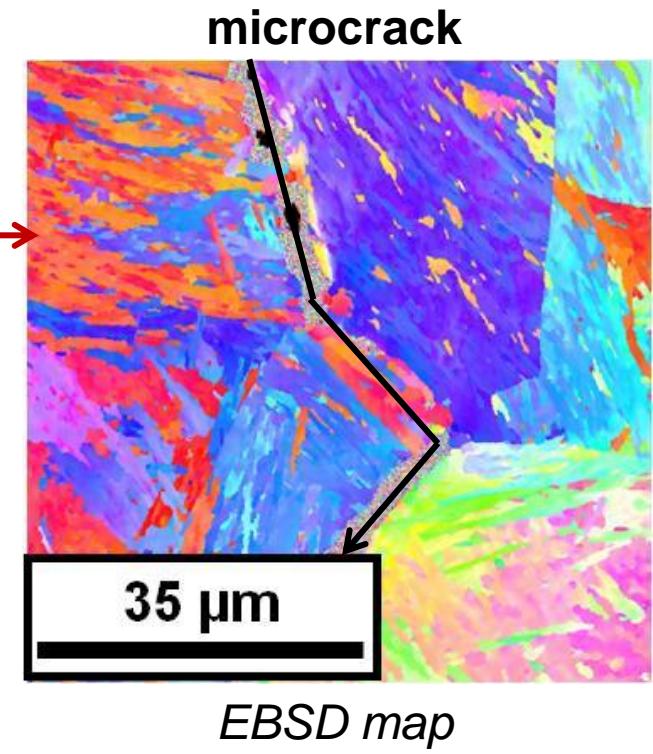
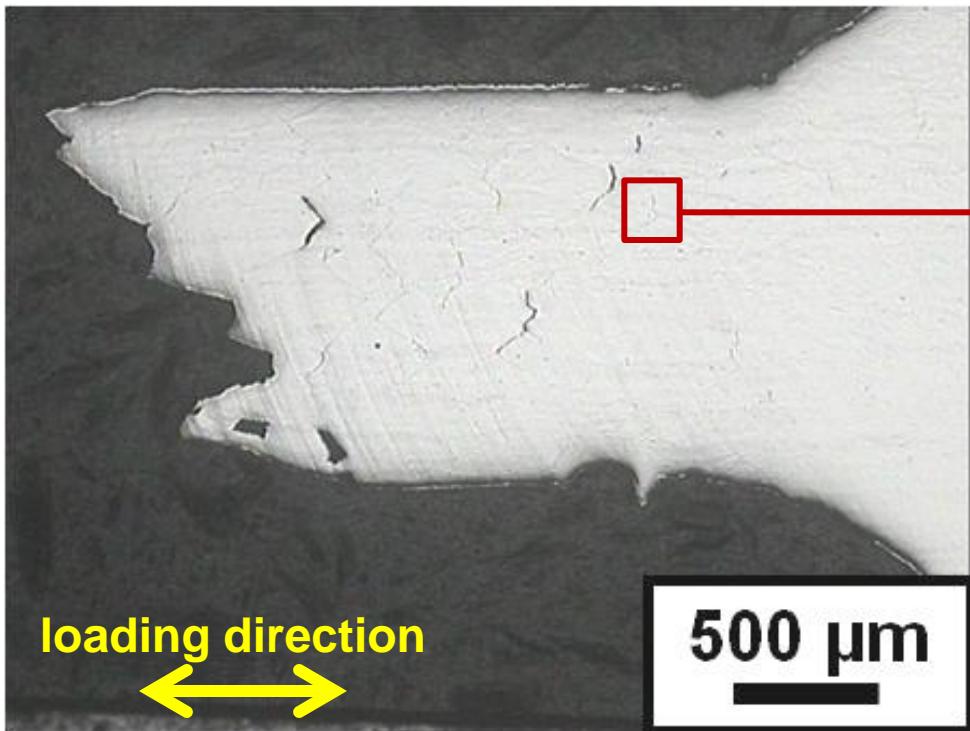
Very short tertiary creep stage,  
low reduction of area at failure,  
**Ductile + intergranular  
fracture.**

# CREEP FRACTURE MODE

Ductile + intergranular creep fracture mode observed for:  
**A1200-R(0-25%)-T(700-750°C) states (650°C, 140 MPa)**

- Confirmation by EBSD observations: damage at prior austenite grain boundaries,

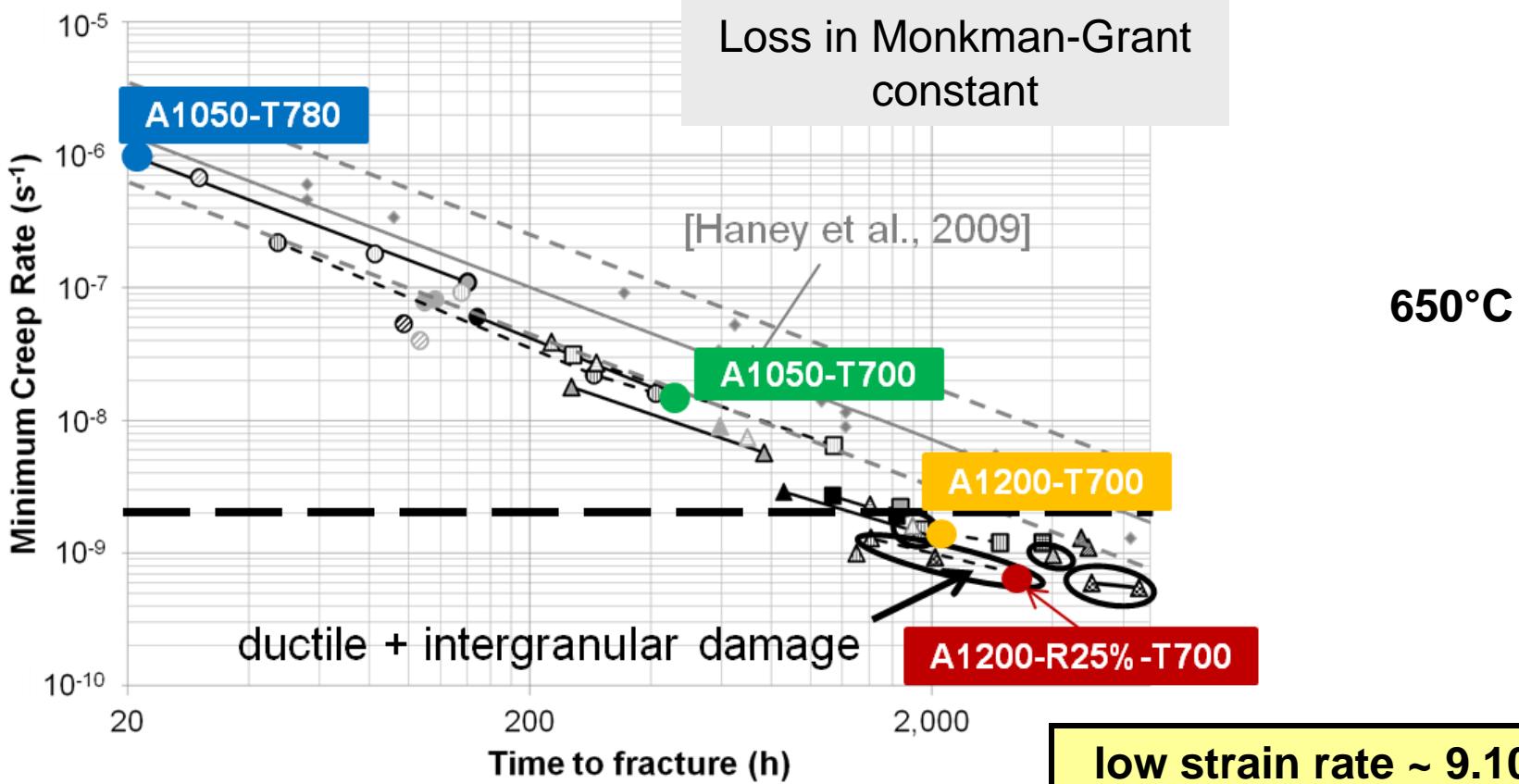
*cross section observation*



# CREEP FRACTURE MODE

Ductile + intergranular creep fracture mode observed for:  
**A1200-R(0-25%)-T(700-750°C) states (650°C, 140 MPa)**

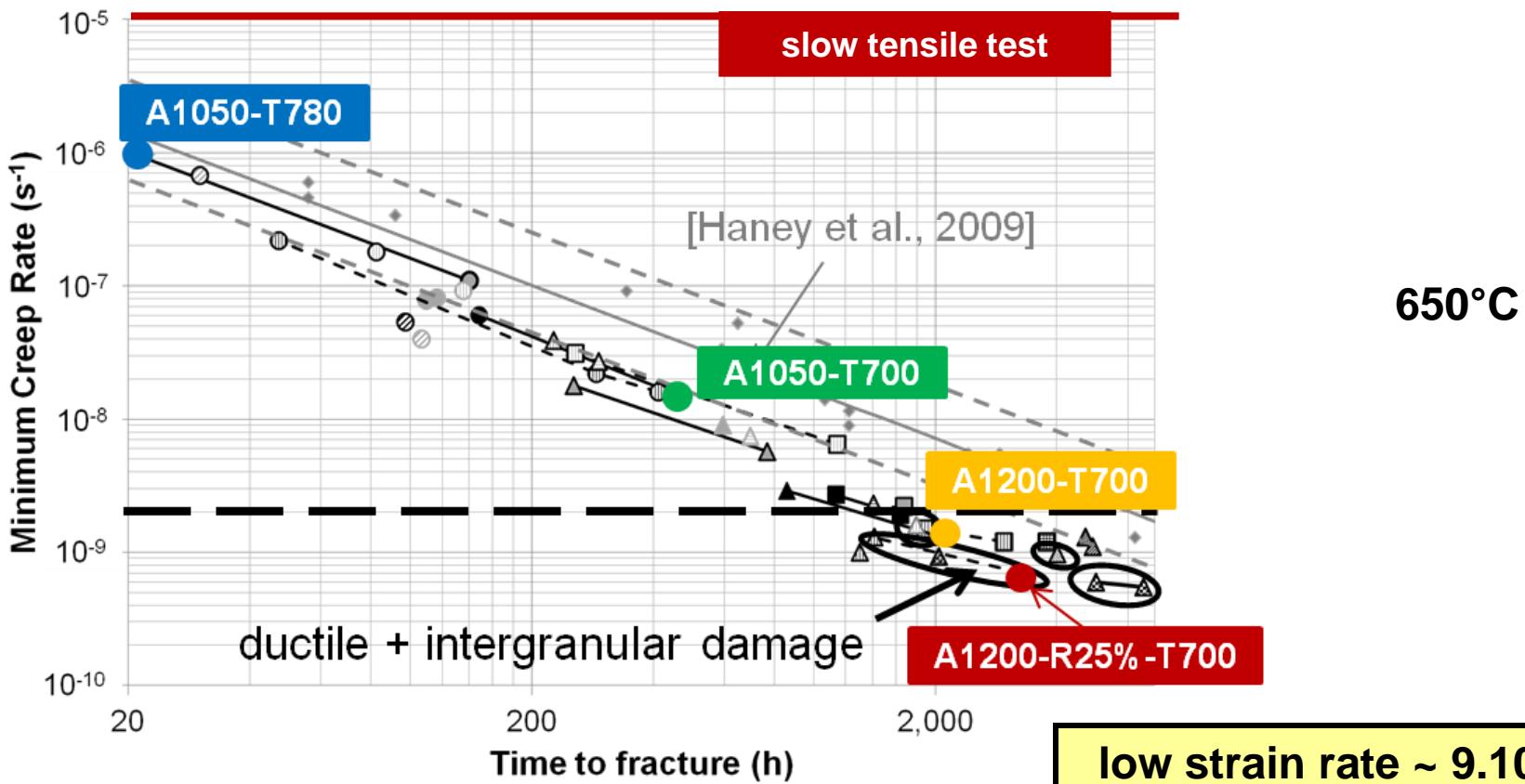
1. Related to a **slow deformation mechanism** typical of long-term creep of this steel (low strain rate and stress exponent, intergranular damage)?
2. Related to metallurgical ageing during the creep test?



# CREEP FRACTURE MODE

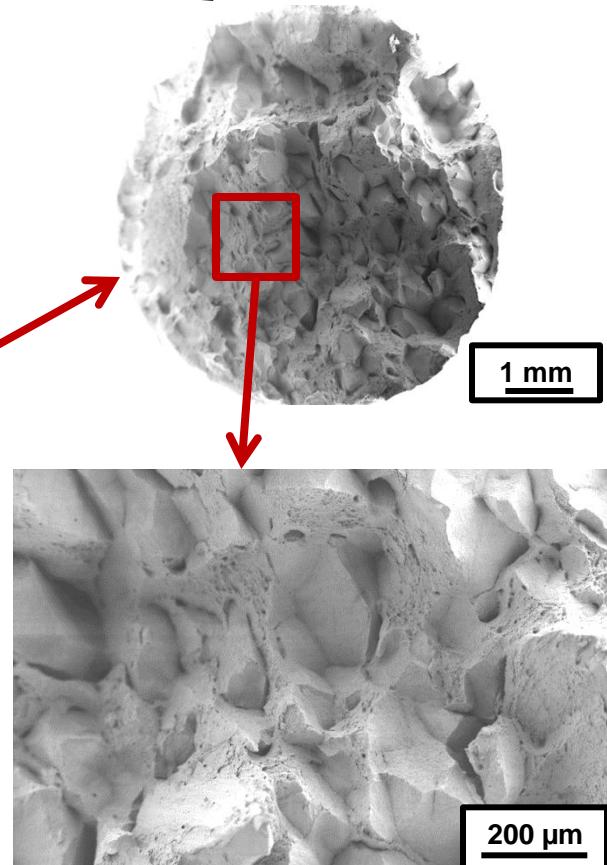
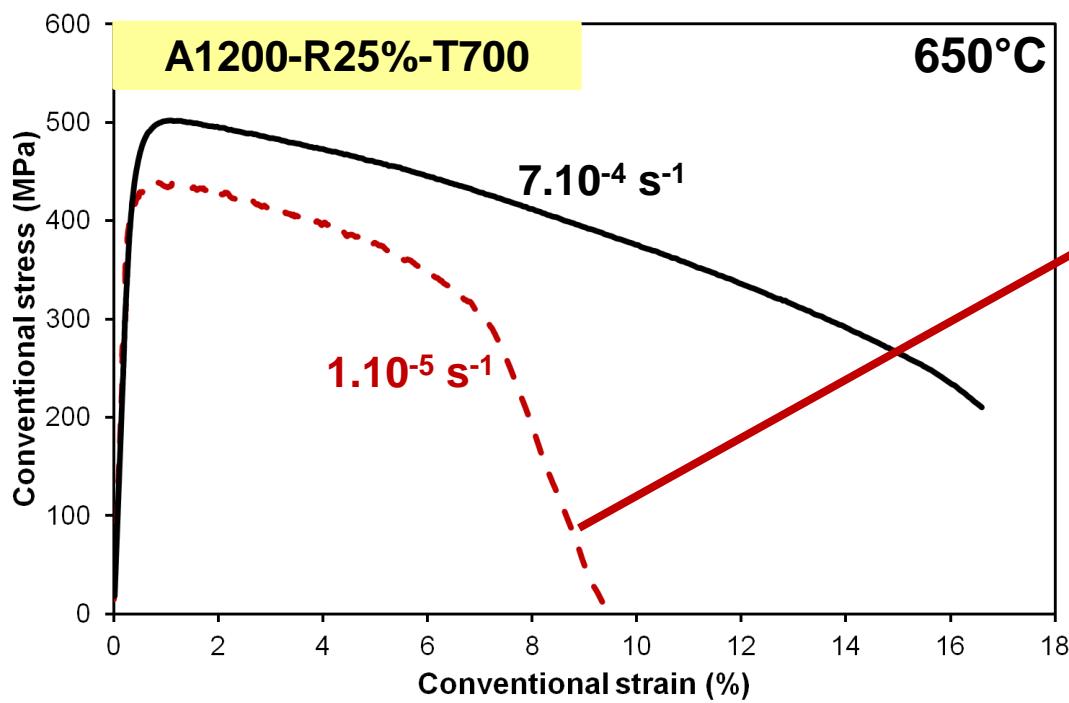
Ductile + intergranular creep fracture mode observed for:  
**A1200-R(0-25%)-T(700-750°C) states (650°C, 140 MPa)**

1. Related to a **slow deformation mechanism** typical of long-term creep of this steel (low strain rate and stress exponent, intergranular damage)?
2. Related to metallurgical ageing during the creep test?



# CREEP FRACTURE MODE

1. Related to slow deformation mechanism typical of the creep of this steel (low strain rate) → (what damage)?
2. Related to metallurgical ageing during the creep test?  
→ IG fracture mode also evidenced by a slow tensile test ( $10^{-5} \text{ s}^{-1}$ ).



**Due to some specific martensitic microstructures**  
(high  $D_Y$ , effects of impurities and/or high precipitates density at prior-γ grain boundaries, critical intragranular hardening...) ???

# SUMMARY AND PENDING ISSUES (1/2)

4 states (3 thermal + 1 thermomechanical treatments)

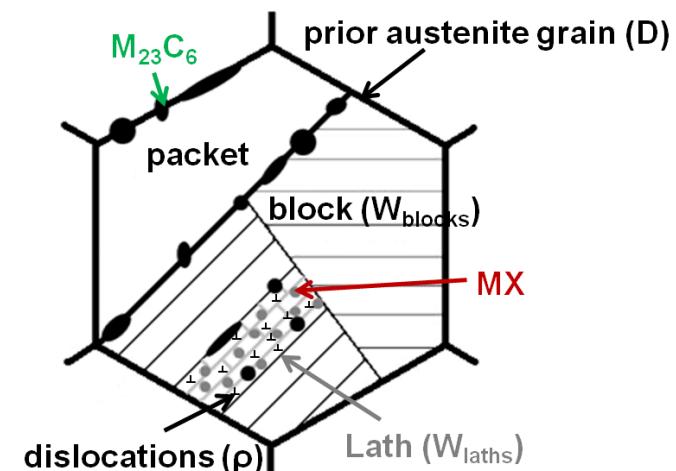
- Effects of  $T_{\text{austenitizing}}$ ,  $T_{\text{tempering}}$ , warm-rolling step on microstructural parameters and on creep flow:

Significant effects on  $t_r$  and  $\dot{\epsilon}$

	$\nearrow T_{\text{austenitizing}}$	$\searrow T_{\text{tempering}}$	+ warm-rolling
$D_\gamma$	$\nearrow$	=	=
$W_{\text{blocks}}$	$\nearrow$	=	=
$W_{\text{laths}}$	=	=	$\searrow$
$\rho$	=	$\nearrow$	$\nearrow$
$f_{\text{v intra}}$	$\nearrow$	$\nearrow$	=
$D_{\text{intra}}$	=	$\searrow$	=

1<sup>st</sup> order parameters

moderate effect



→ Further work: Modelling

## SUMMARY AND PENDING ISSUES (2/2)

### Effects of microstructural parameters on creep fracture:

**Ductile + intergranular** fracture mode : **A1200-R(0-25%)-T(700-750°C)**  
 (140 MPa, 170 MPa at 650°C)

#### Our assumptions :

1. Related to a **slow deformation mechanism** typical of long-term creep of this steel (low strain rate and stress exponent, intergranular damage)?
2. Related to **metallurgical ageing** during the creep test?



**Slow tensile test ( $10^{-5}$  s $^{-1}$ ) → FALSE !**



**Due to some specific martensitic microstructures ???**

#### → Further work:

Due to inclusions or coarse carbides ? Study “cleaner” or with low level C+N 9%Cr steels,

Due to intragranular hardening ? Reinforce prior γ austenite boundaries by « original TT ».



CENTRE DES MATERIAUX  
P. M. FOURT

The workers are grateful  
for financial support  
of EDF



# Thank you for your attention.

Afcen RCC-MRx code: Section III, vol. 1, Z, (2010) and, Section III, vol. 2: Materials, (2012).

Hollner et al., *Journal of Nuclear Materials*, **405**, pp 101-108, (2010).

H. Haney et al., *Materials Science and Engineering*, **510-511**, pp 99 – 103, (2009).

D.R. Palaparti et al., *Procedia Engineering*, **55**, pp 70-77, (2013).

**PhD thesis available on :**

<https://pastel.archives-ouvertes.fr/tel-01141826>

... but in French !

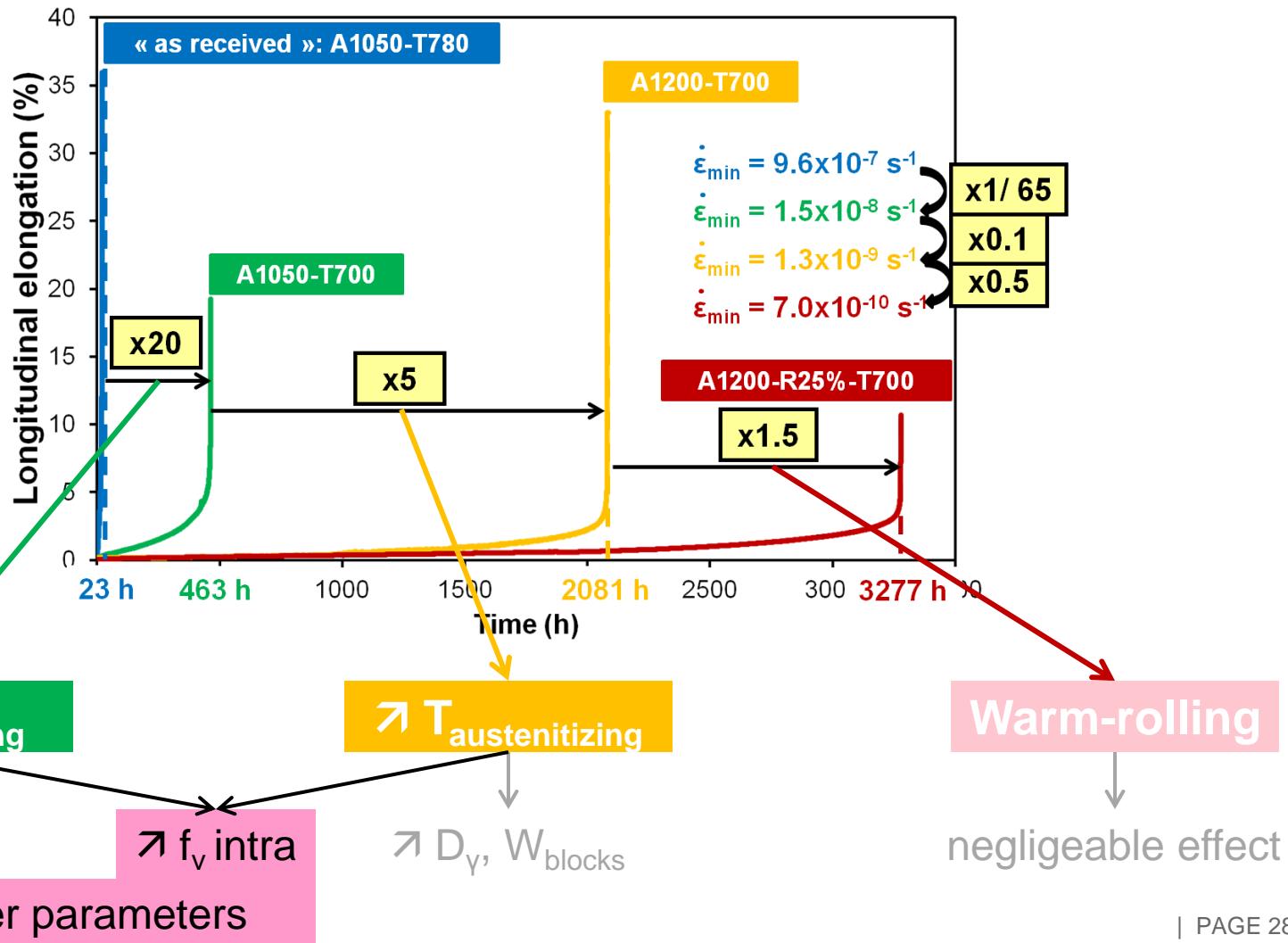
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Commissariat à l'énergie atomique et aux énergies alternatives  
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T. +33 (0)1 69 08 41 42 | F. +33 (0)1 69 08 71 30

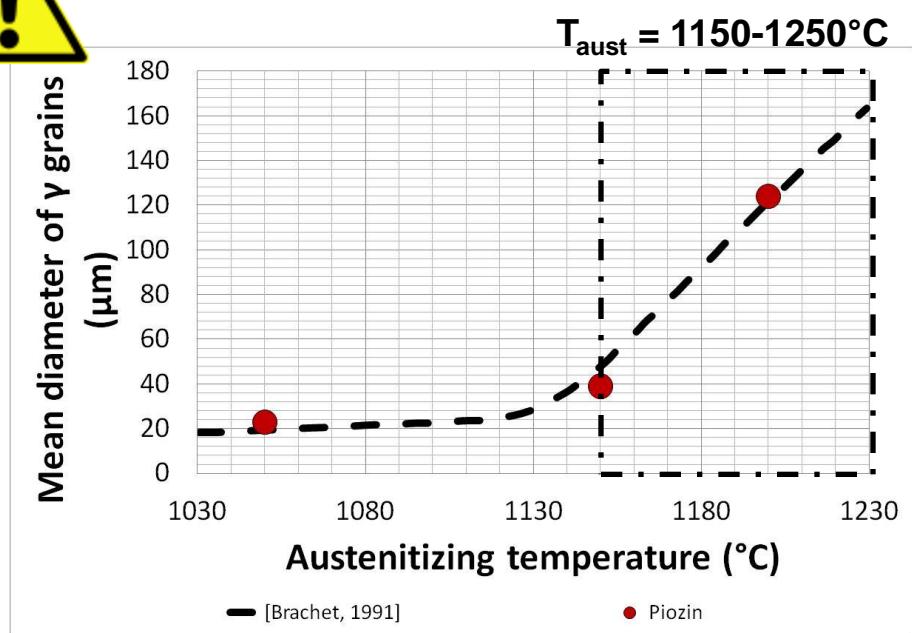
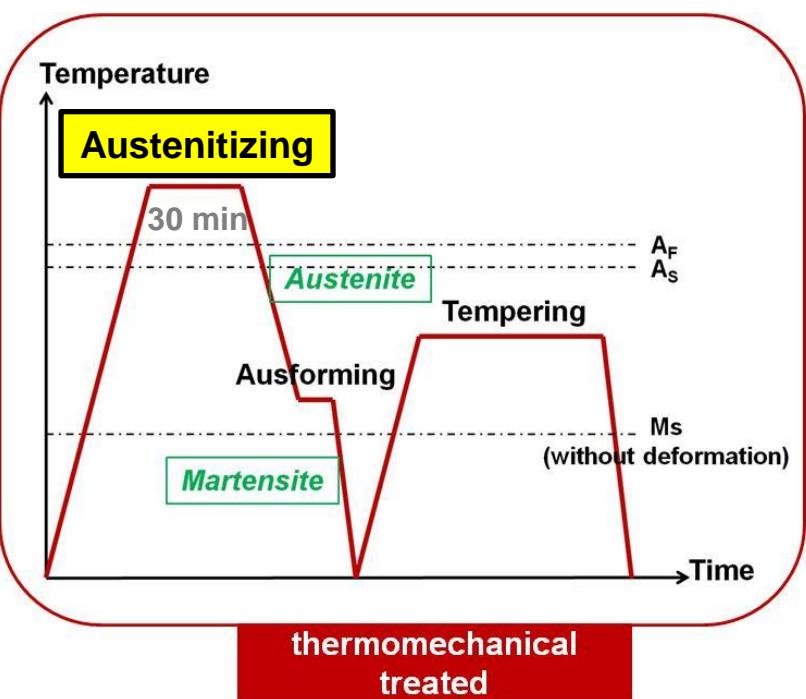
DEN  
DMN  
SRMA/LA2M

# SUMMURY – PART 1

Effects of  $T_{\text{austenitizing}}$ ,  $T_{\text{tempering}}$ , warm-rolling step on microstructural parameters and on creep flow:



# AUSTENITIZING STEP

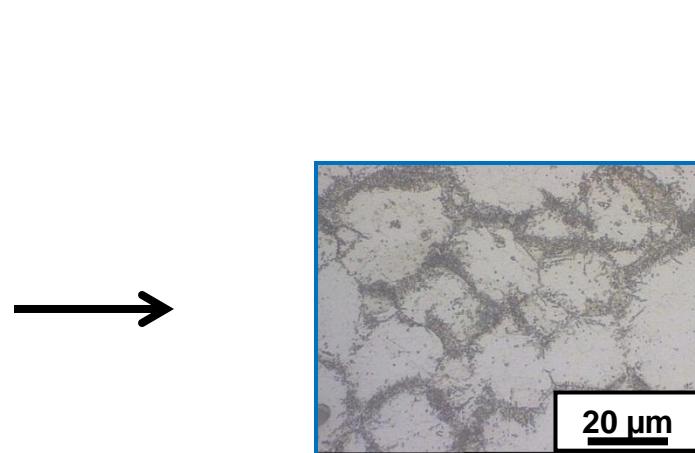
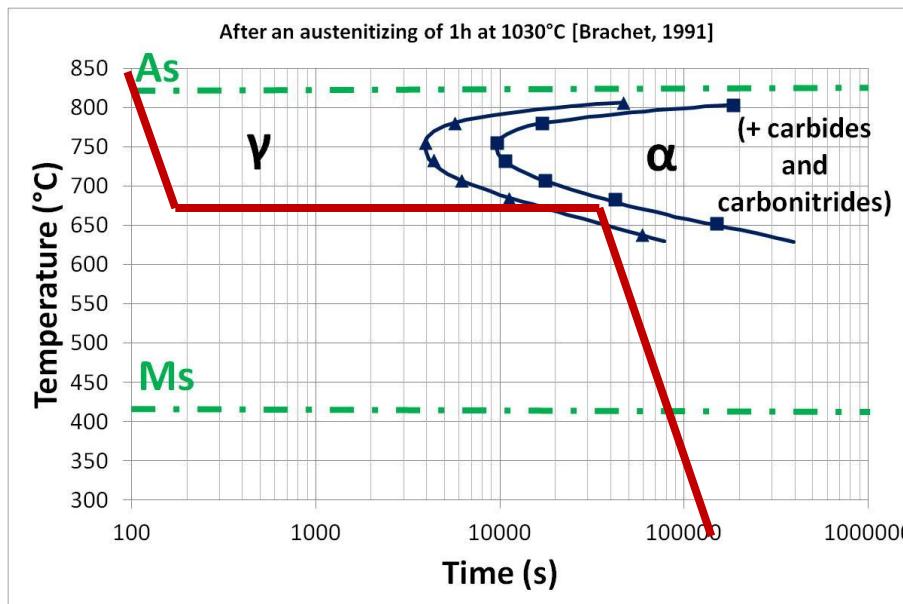
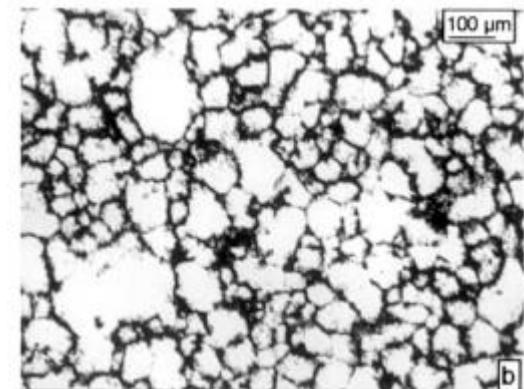
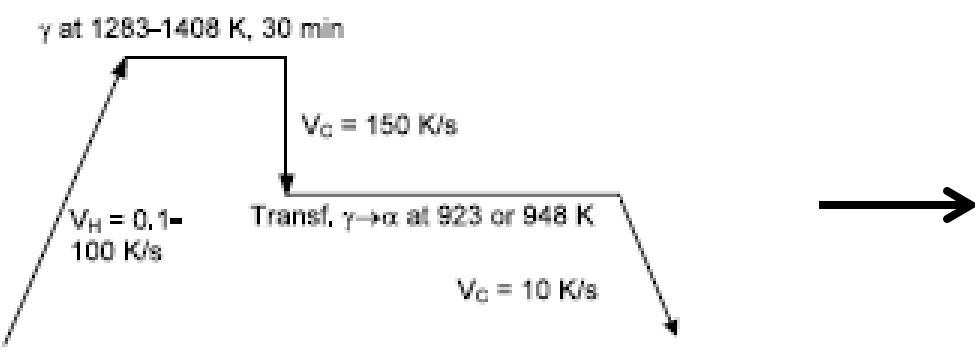


Austenitizing temperature ( $^{\circ}\text{C}$ )	
1050	"as received" condition
1150	to dissolve MX precipitates
1200	

# AUSTENITE GRAIN SIZE

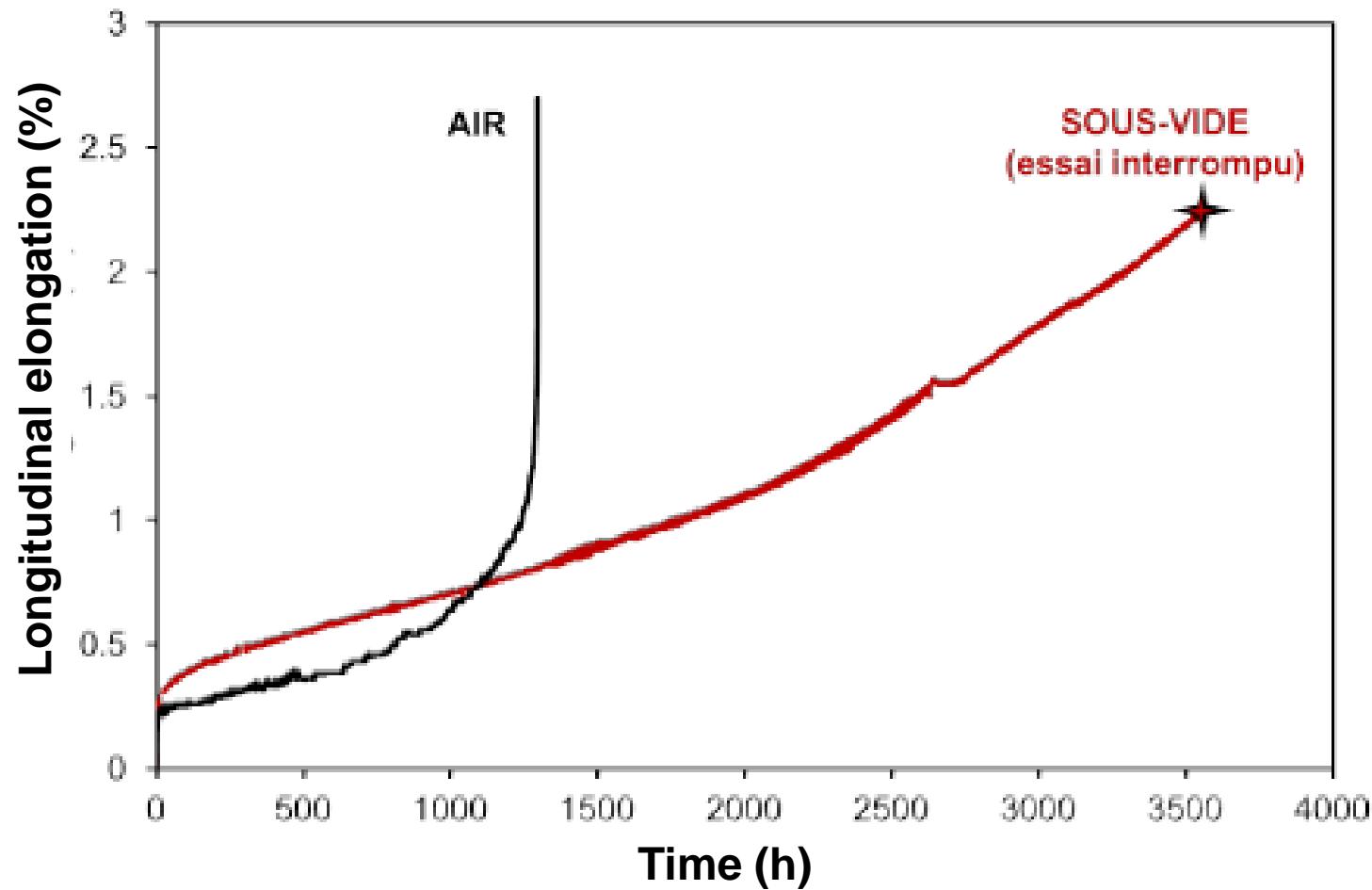
## Experimental procedure to measure prior austenite grain size :

From Danon et al., Materials Science and Engineering, vol. A 348, p. 122-132, 2003.



# INFLUENCE OF ENVIRONNEMENT

A1200-R500,25%-T700

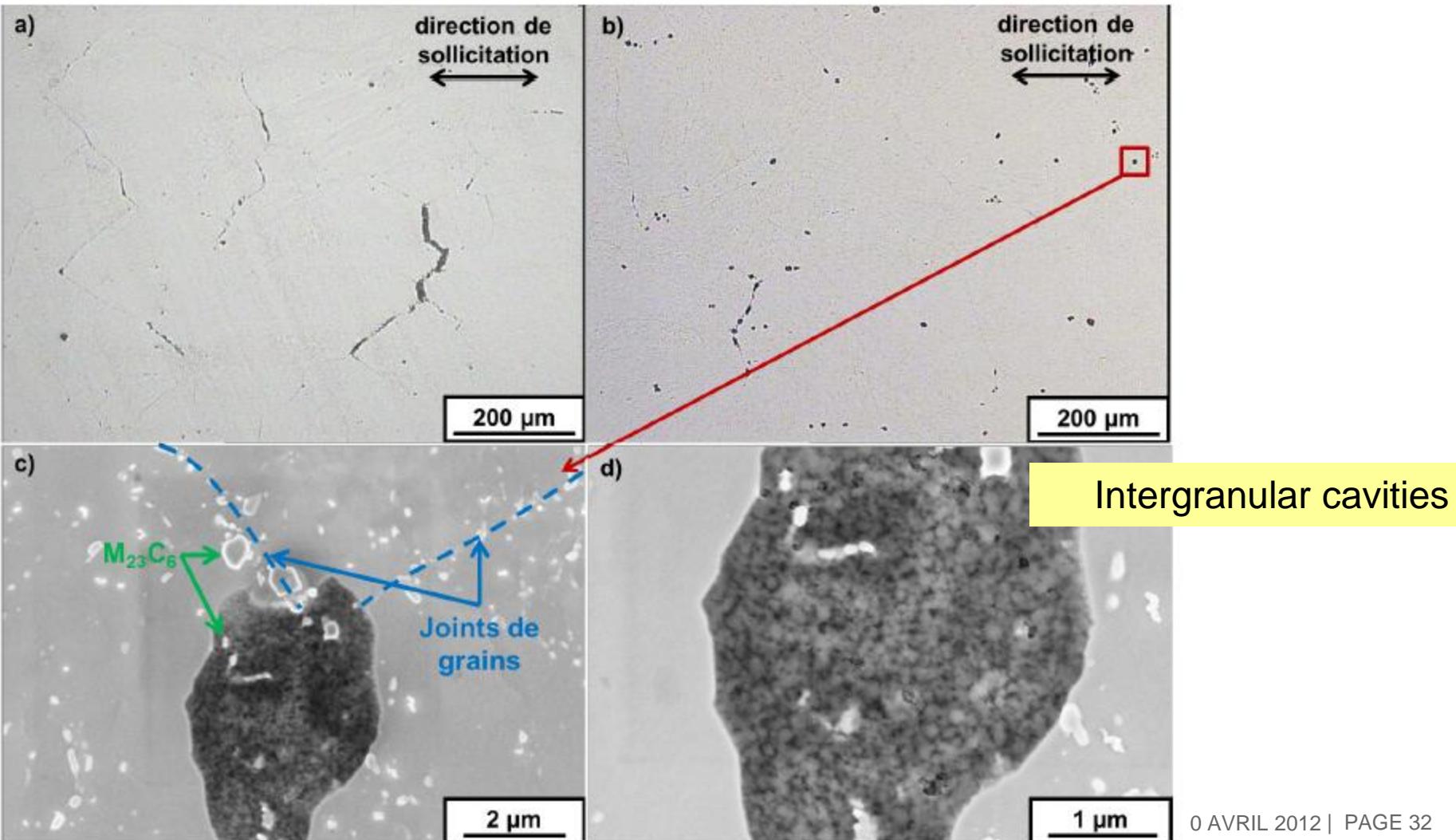


Same minimal creep rate:  
Air =  $1.10^{-9} \text{ s}^{-1}$ , Vacuum =  $9.10^{-10} \text{ s}^{-1}$ .  
But  $t_r$  higher for vacuum !

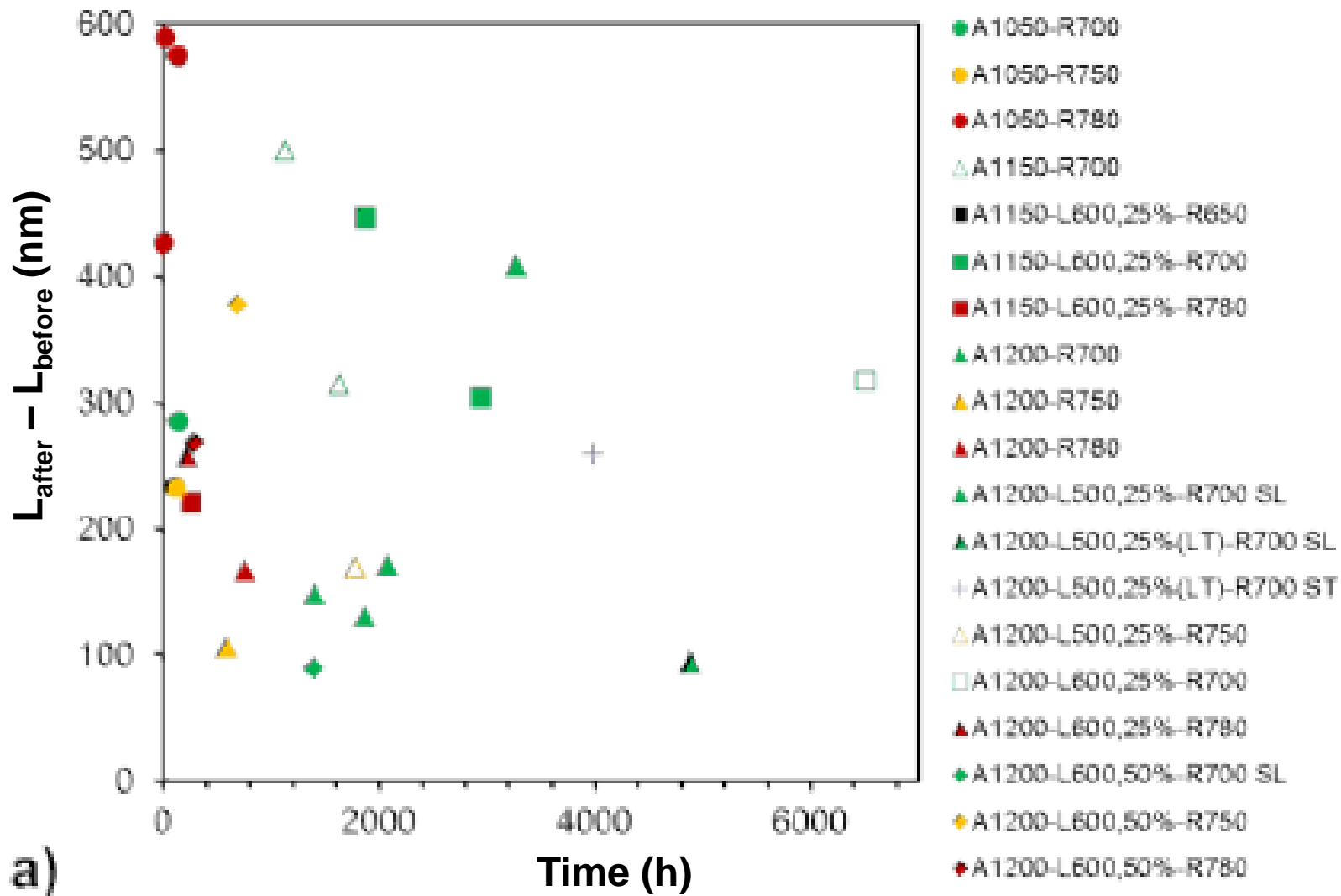
# INFLUENCE OF ENVIRONNEMENT

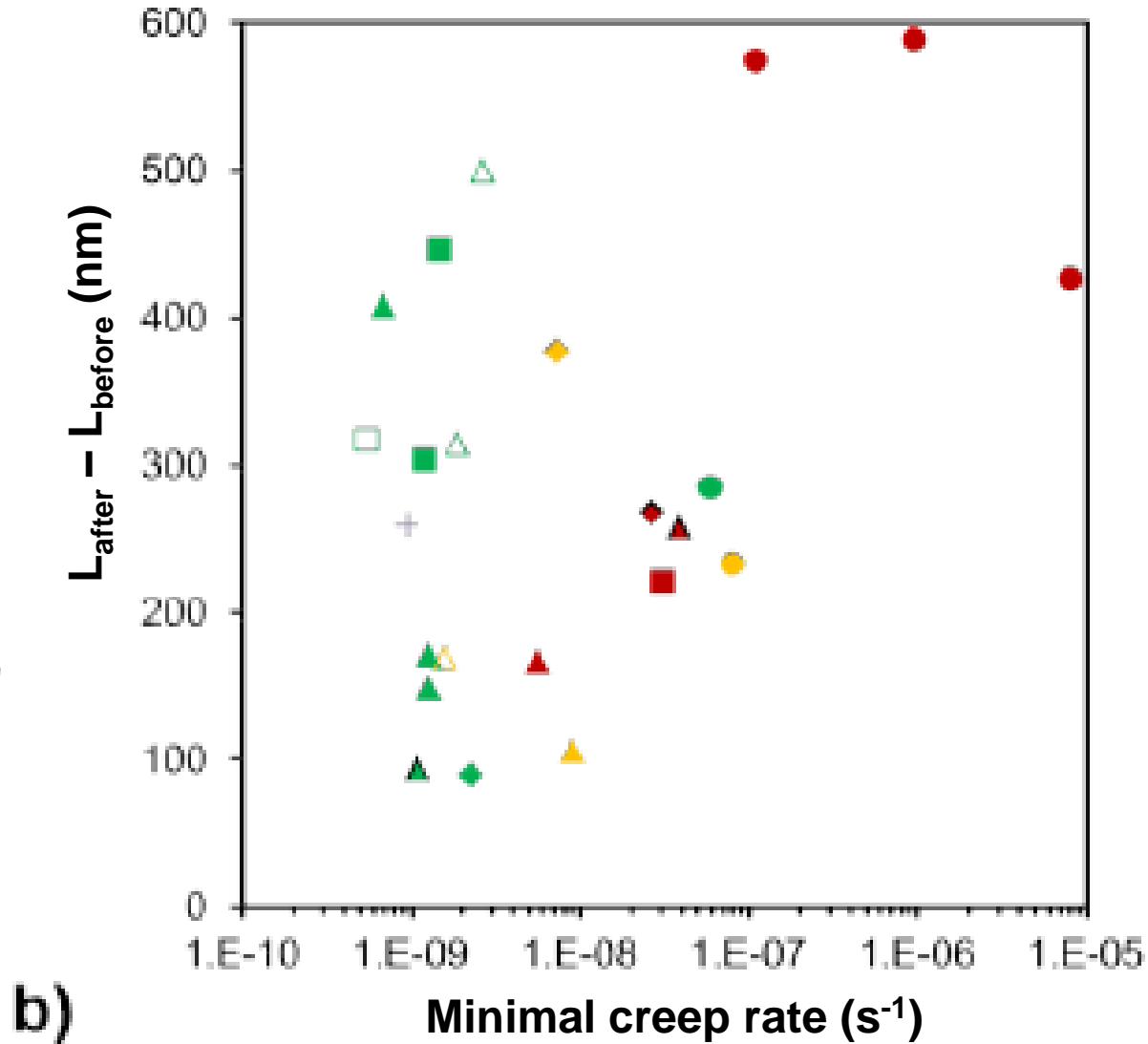
A1200-R500,25%-T700 (vacuum)

*cross section observations*

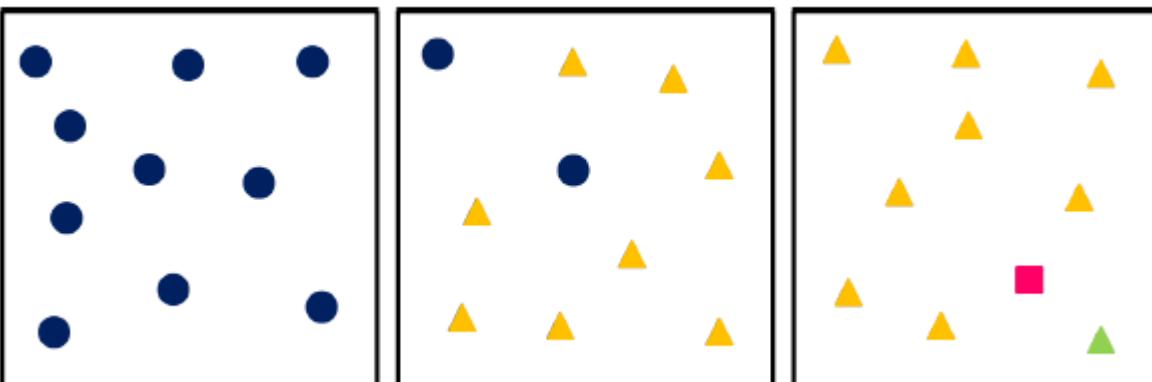


# MICROSTRUCTURE AFTER CREEP TESTS





# MICROSTRUCTURE AFTER CREEP TESTS



non sollicité

vieilli thermiquement à  
650°C pendant 6523  
heuresvieilli thermiquement à  
650°C pendant 6523  
heures + sollicité en  
fluage à 140 MPa

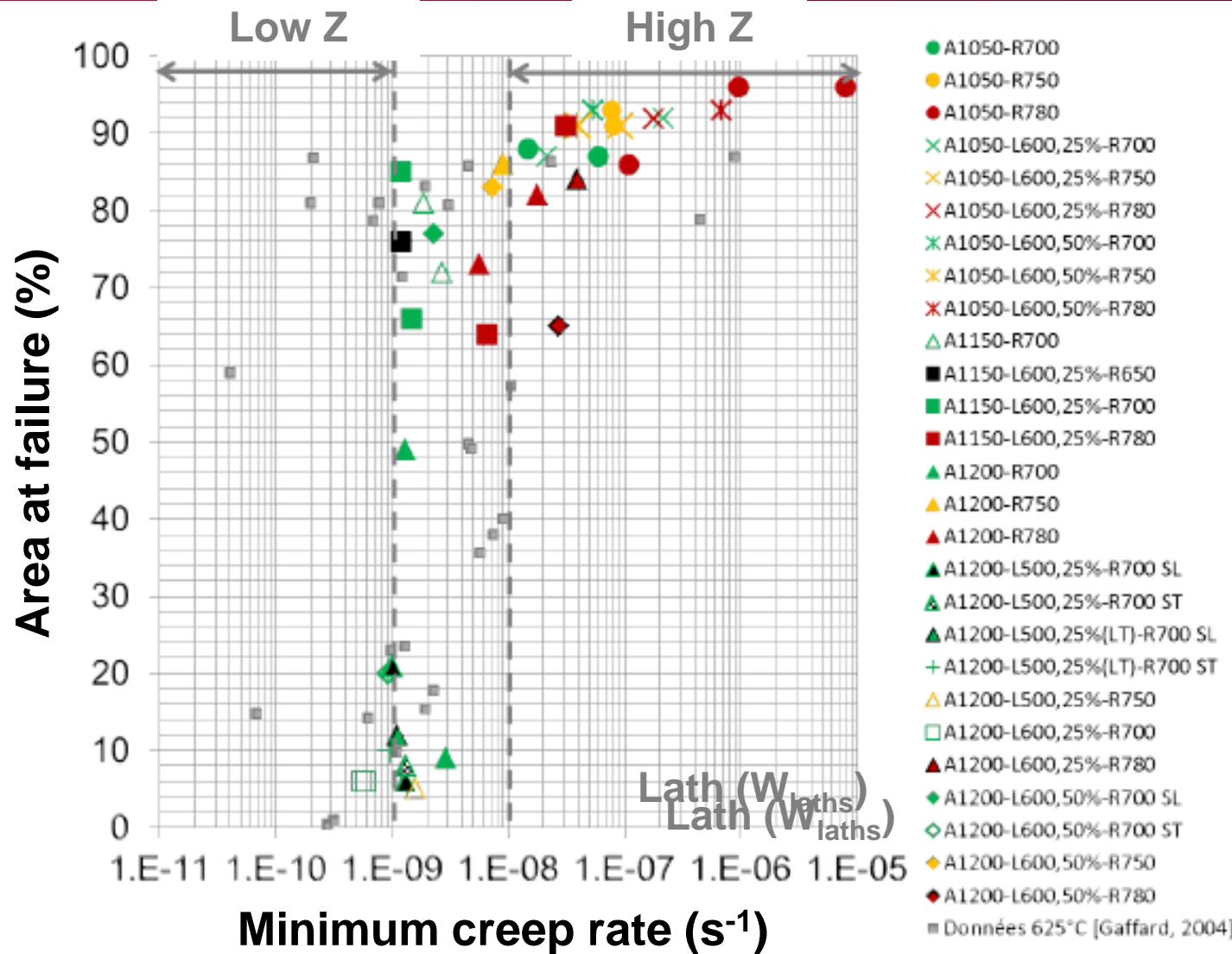
- $M_2X$  avec  $M=(80\%Cr, 15\%Fe, 10\%Mo, 5\%V)$  et  $X=(C,N)$
- ▲  $MX$  avec  $M=(50\%V, 30\%Nb, 20\%Cr)$
- ▲  $MX$  avec  $M=(80\%V, 20\%Cr)$
- $MX$  avec  $M=(80\%Nb, 15\%V, 5\%Cr)$

**A1200-R600,25%-T700**  
Creep test :  
140 MPa, 650°C  
 $t_r = 6523$  h

Change in chemical composition  
of MX precipitates

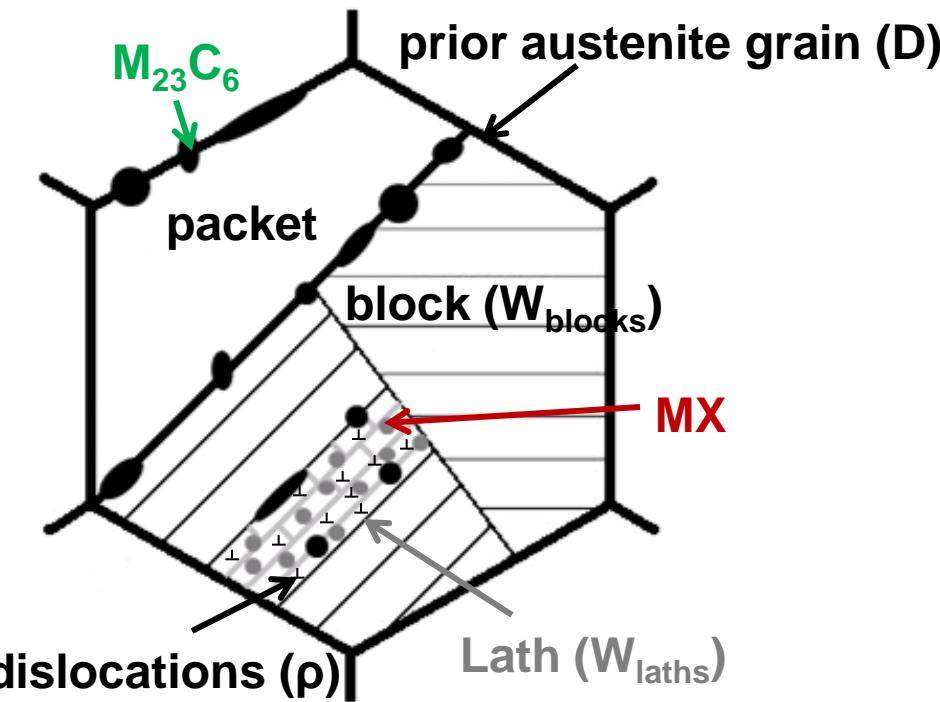
	No creep tested	Creep tested head	Creep tested gauge
$D_{M23C6}$ (nm)	$123 \pm 58$	$130 \pm 48$	$147 \pm 55$
$D_{MX}$ (nm)	$18 \pm 7$	$16 \pm 4$	$20 \pm 4$

# MINIMUM CREEP RATE VALUE LIMIT !

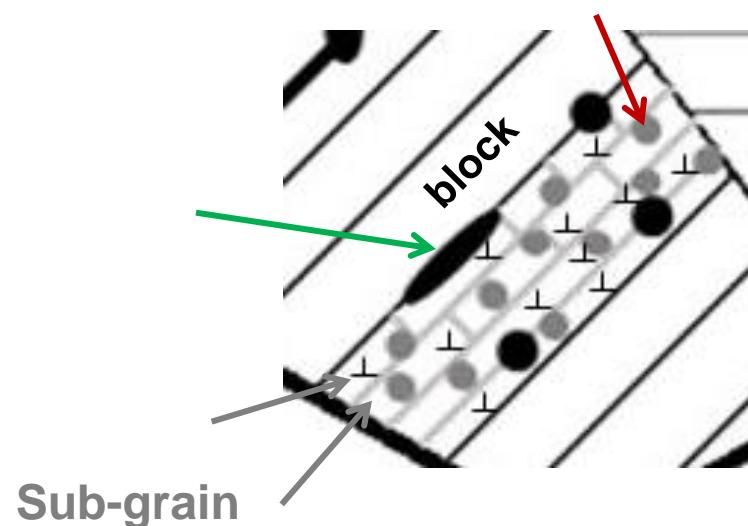


# COMPLEX MICROSTRUCTURE OF GRADE 91 STEEL

- Simplified chemistry (wt %): Fe-9Cr-1Mo-0,1C-(Nb,V)
- Austenitized at 1050°C, 30 min, air cooled, tempered at 780°C, 1h, air cooled.
- Tempered martensite: multiscale microstructure



**MX = (Nb,V)(C,N)**  
 minority (<10%),  
 intragranular  
**incoherent** (no-shearable)



Microstructural parameters relevant to creep strength:  
**D,  $W_{\text{blocks}}$ ,  $W_{\text{laths}}$ , precipitates, dislocation density**