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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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■ Considered:

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

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

■ Considered:

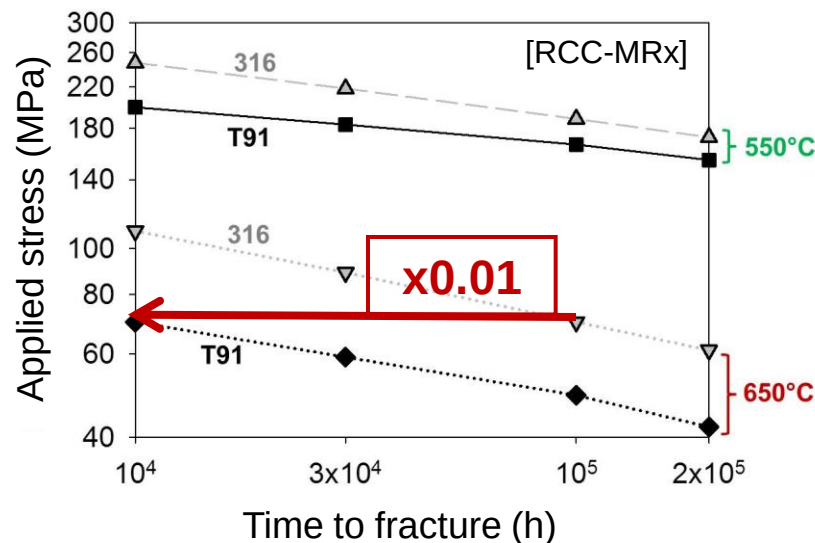
- nuclear fusion reactors:
- tokamak blankets.
- SFR (Generation IV) :
- steam generator and secondary piping.

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

Limited creep strength compare to austenitic stainless steels

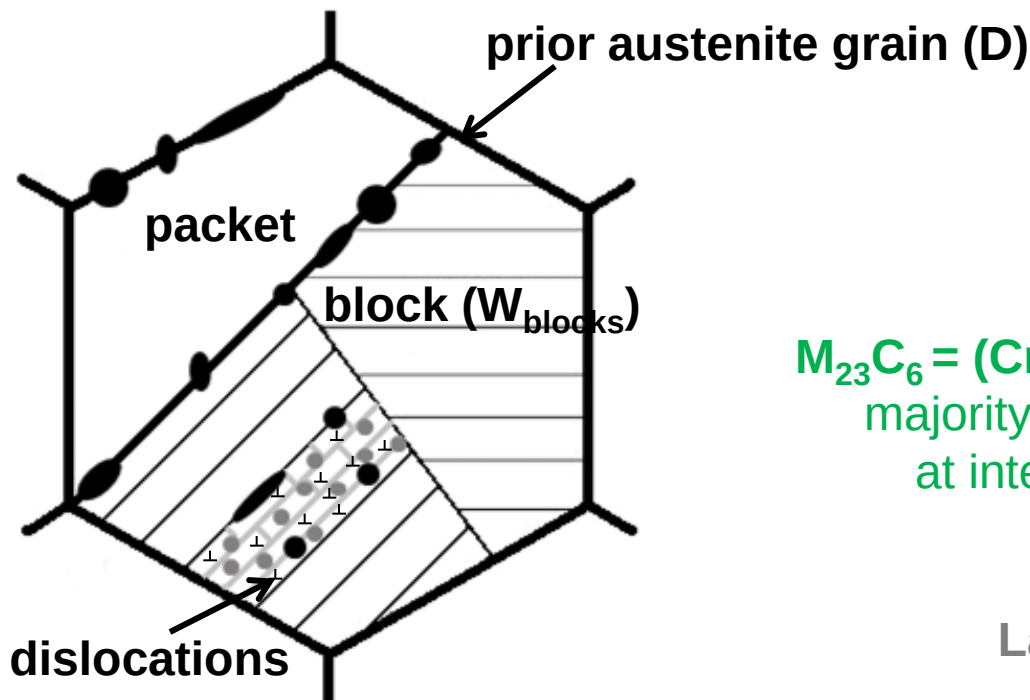
Creep at 550°C and 650°C

T91 =
« standard »
thermal
treatment



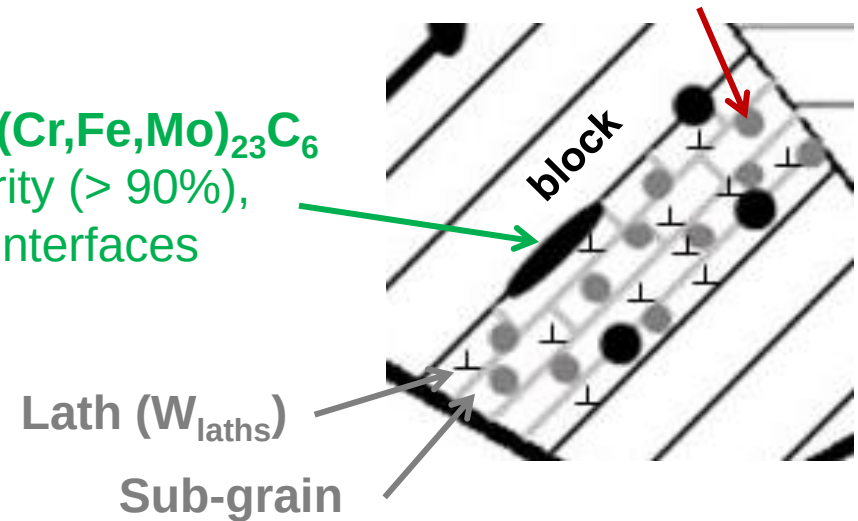
**Need of increasing
strength**

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



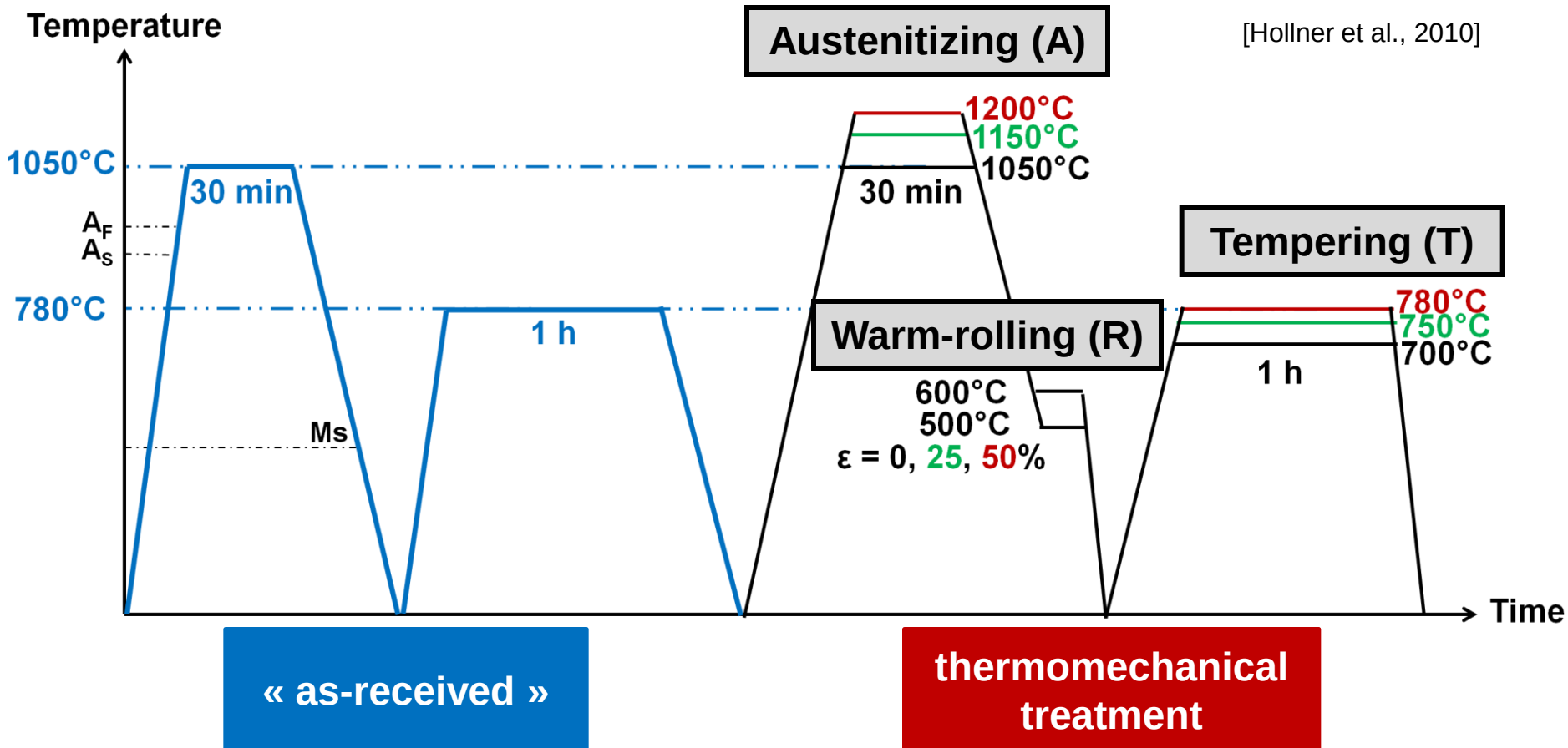
$MX = (Nb,V)(C,N)$
minority (<10%),
intragranular
incoherent (no-shearable)

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



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

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



More than 30 treated conditions

4 states

Part 1

$T_{\text{tempering}}$

$T_{\text{austenitizing}}$

Warm-rolling

Time to fracture
Minimal creep rate

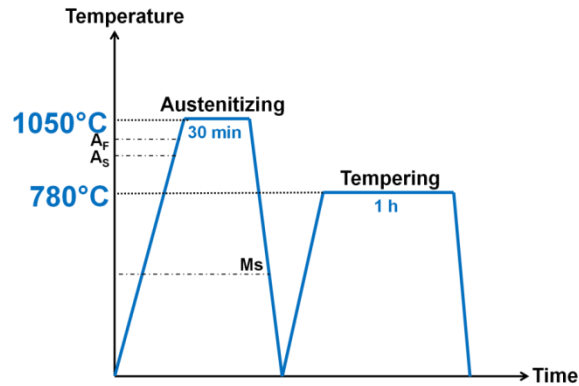
Microstructure

Part 2

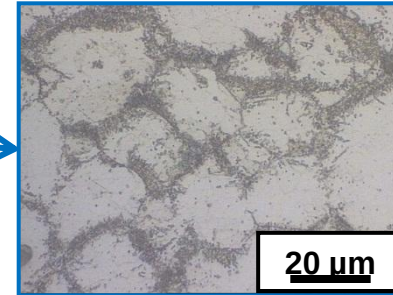
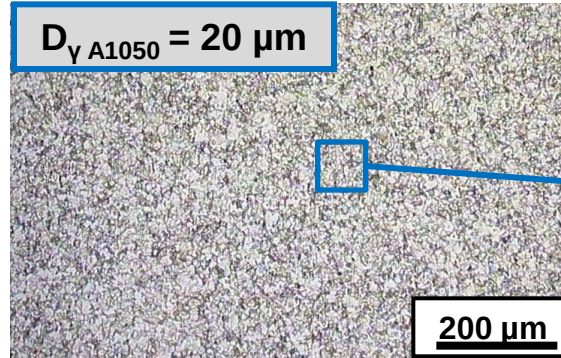
Creep fracture mode

“AS RECEIVED”: A1050-T780

« as received »: A1050-T780



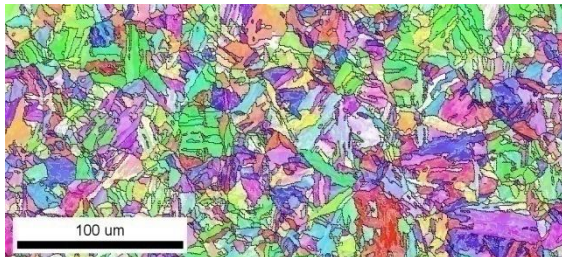
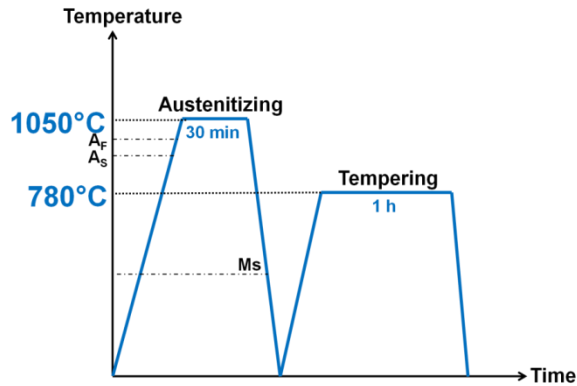
$D_{\gamma A1050} = 20 \mu m$



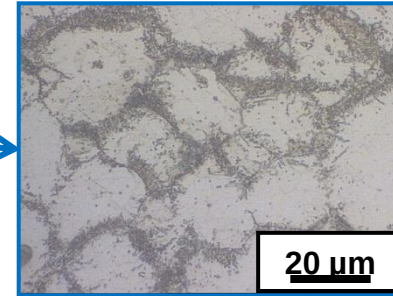
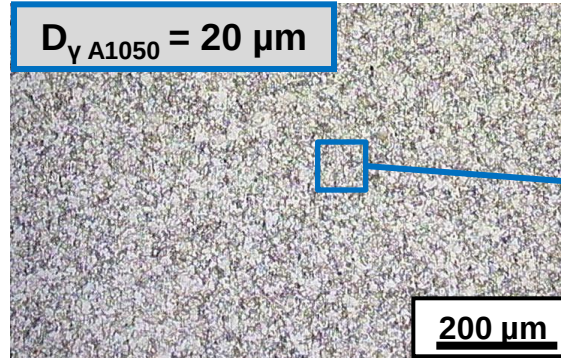
Fine prior austenite grain size

“AS RECEIVED”: A1050-T780

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



Fine prior austenite grain size

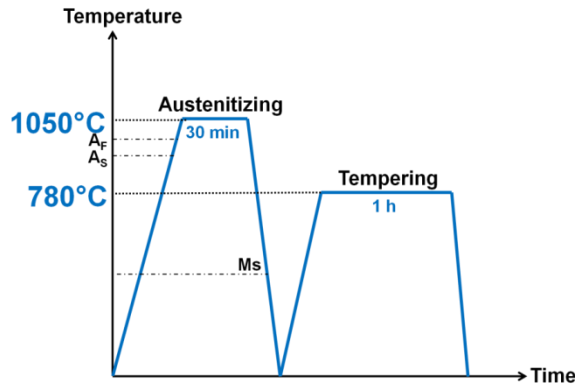
$W_{blocks} = 3.2 \mu m (*)$

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

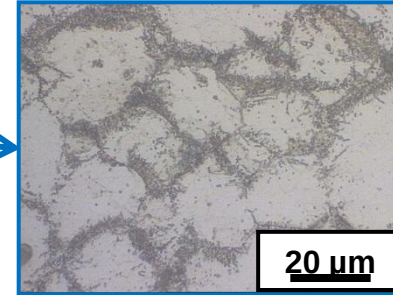
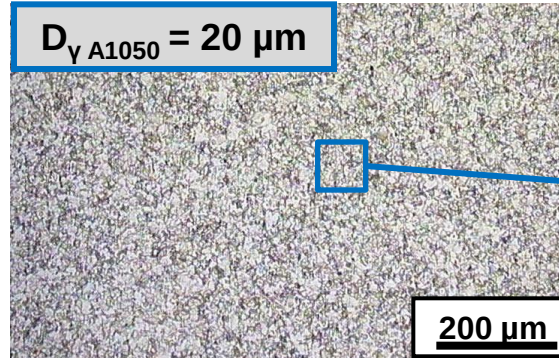
(*) SEM-EBSD,
(**) TEM on thin foils.

“AS RECEIVED”: A1050-T780

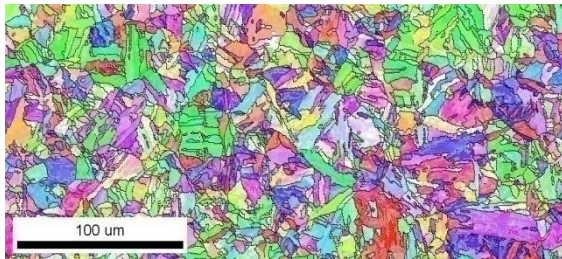
« as received »: A1050-T780



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



Fine prior austenite grain size



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

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

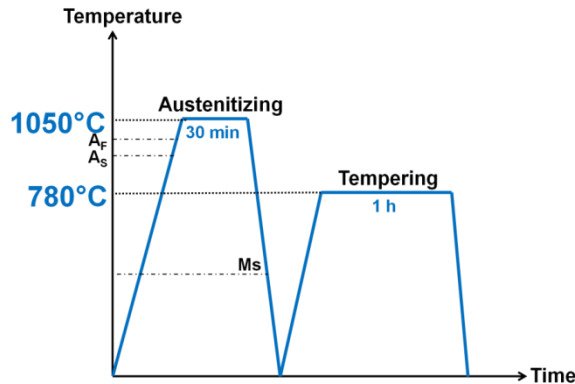
(*) SEM-EBSD,
(**) TEM on thin foils.

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

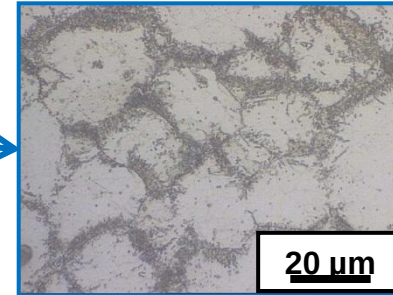
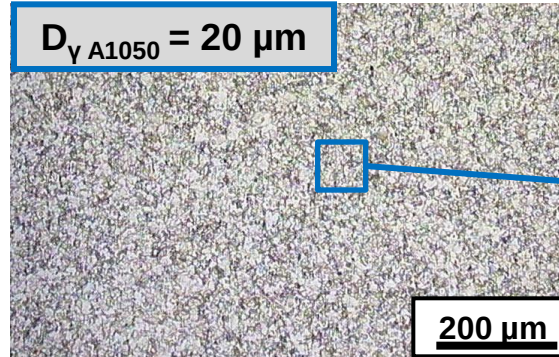
(***) neutron diffraction (*FWHM martensite peaks*).

“AS RECEIVED”: A1050-T780

« as received »: A1050-T780



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

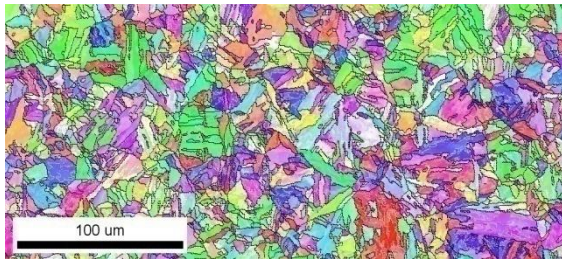


Fine prior austenite grain size

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

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

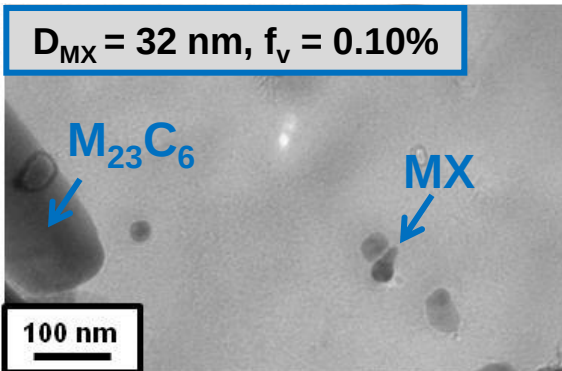
(*) SEM-EBSD,
(**) TEM on thin foils.



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

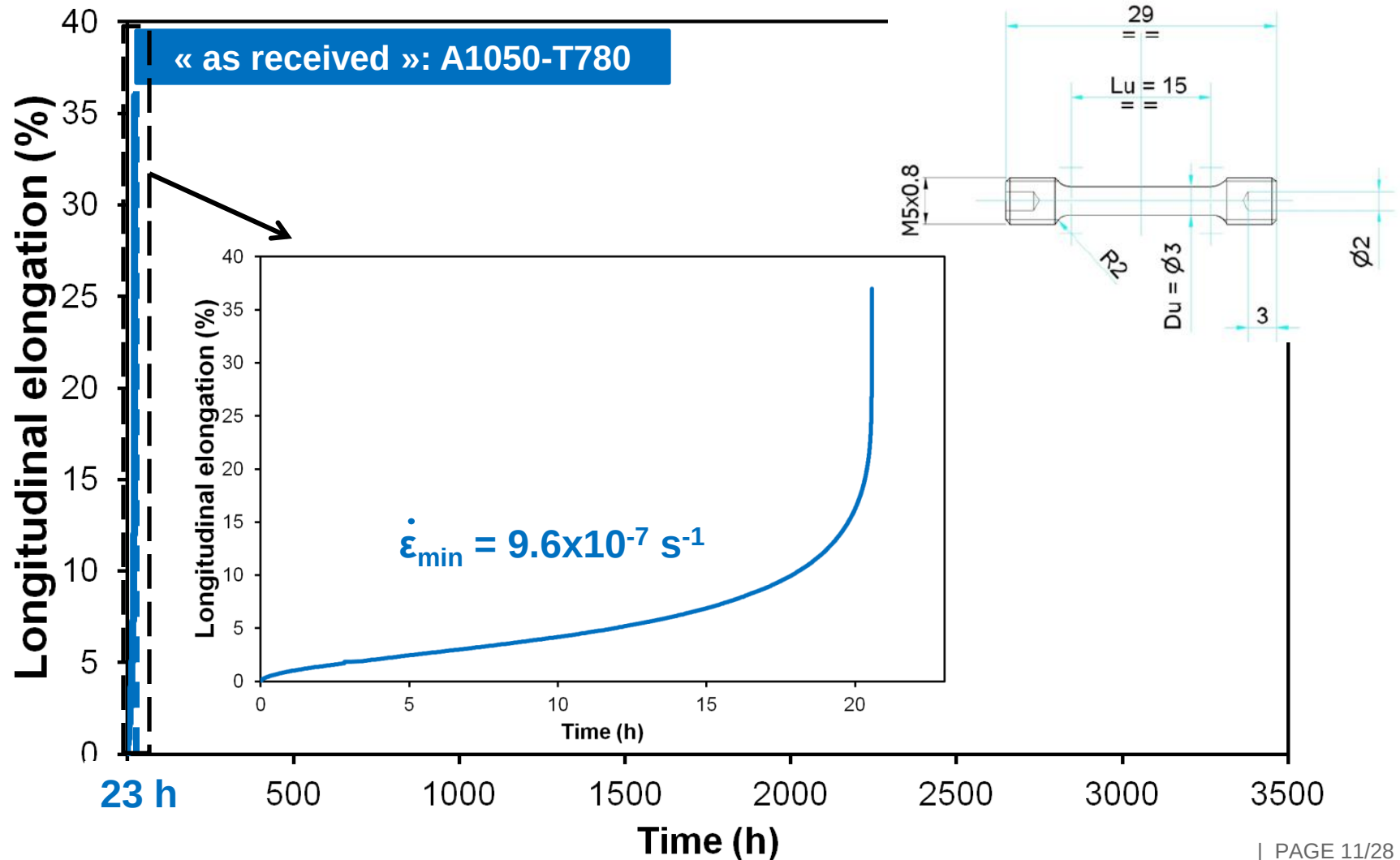
(***) neutron diffraction (*FWHM martensite peaks*).

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



Coarse $M_{23}C_6$ precipitates at the different interfaces
and few intragranular MX precipitates

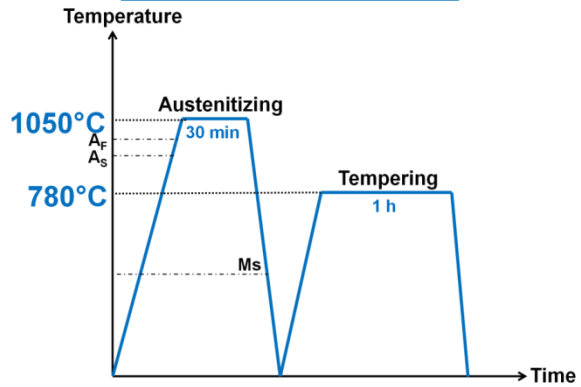
■ Creep conditions: 140 MPa at 650°C (*high stress and short lifetime*)



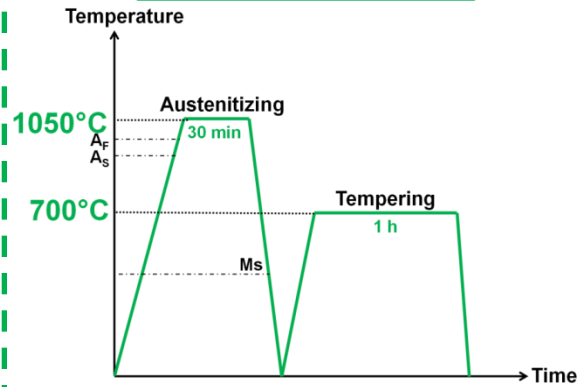
COMPARISON “AR” VS A1050-T700

↘ TEMPERING TEMPERATURE FROM 780°C TO 700°C

A1050-T780

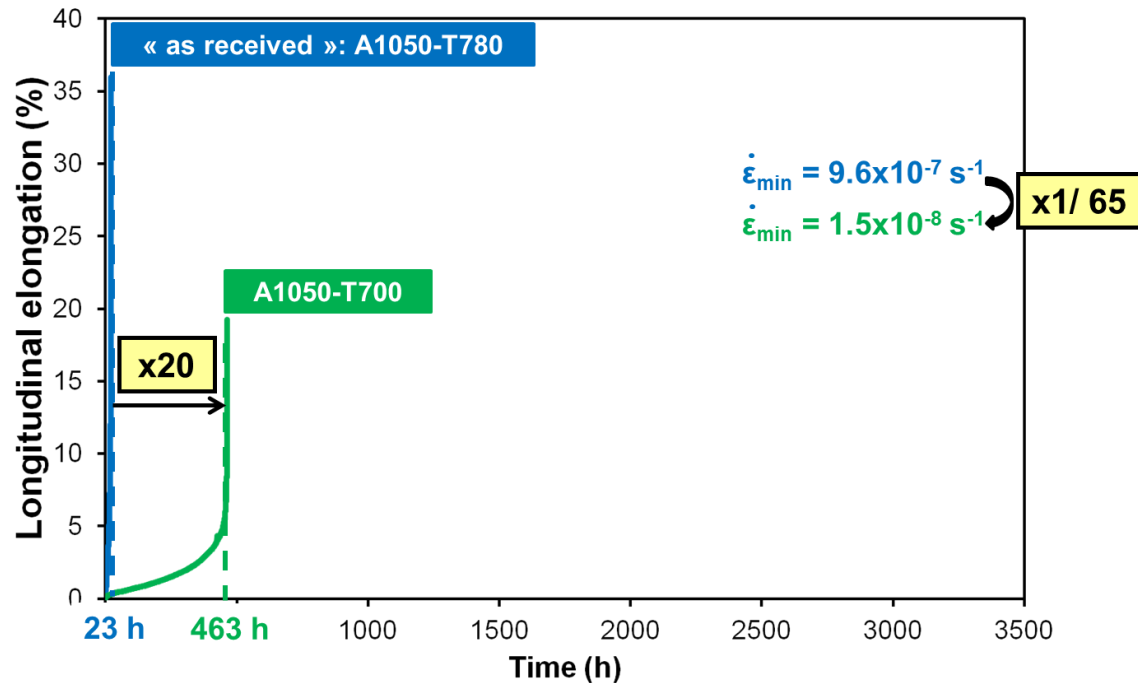


A1050-T700



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

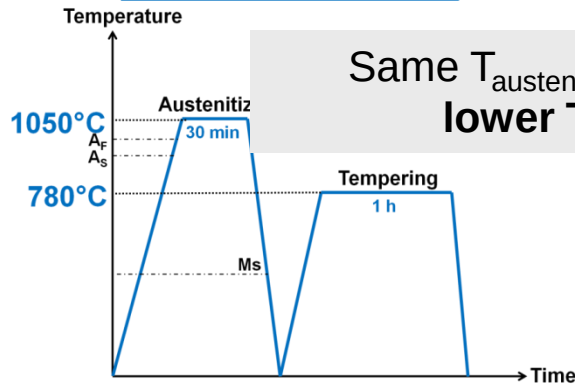
Creep conditions: 140 MPa at 650°C



COMPARISON “AR” VS A1050-T700

↘ TEMPERING TEMPERATURE FROM 780°C TO 700°C

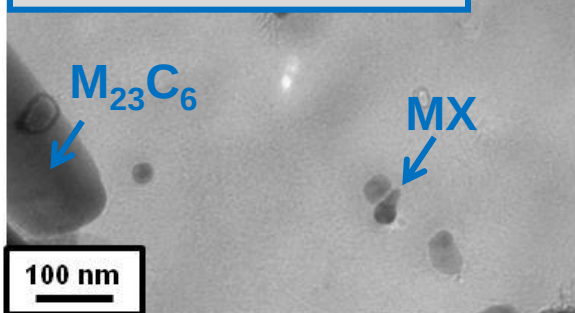
A1050-T780



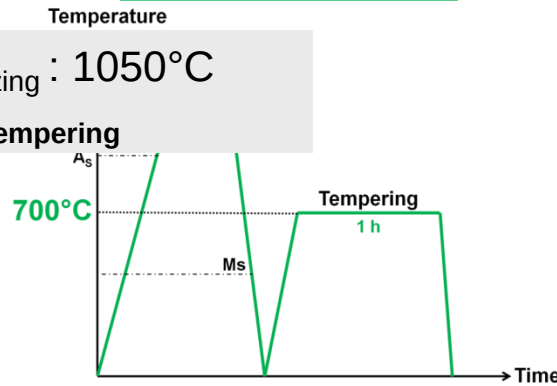
Same T_{austenitizing} : 1050°C
lower T_{tempering}

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

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

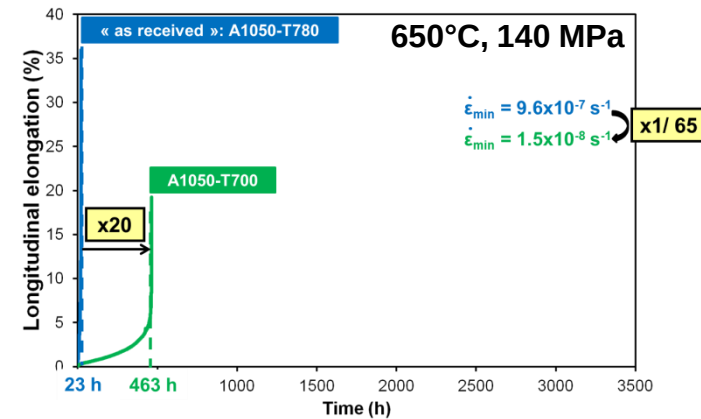
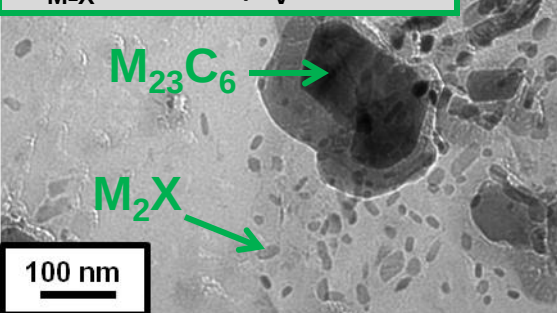


A1050-T700



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

$$D_{M2X} = 15 \text{ nm}, f_v = 0.35\%$$



Higher dislocation density
(less recovery)

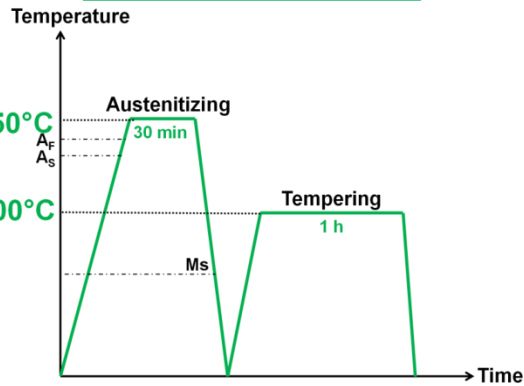


Higher f_v and finer D of
intragranular precipitates

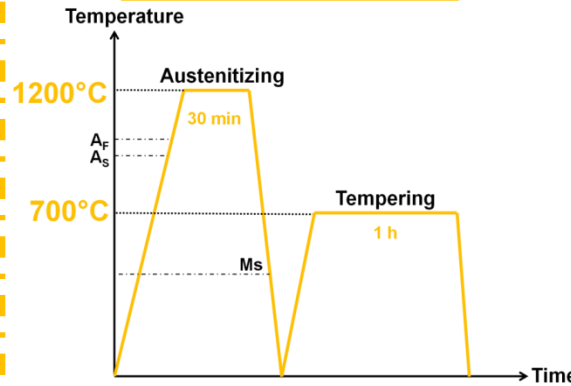
COMPARISON A1050-T700 VS A1200-T700

➔ AUSTENITIZING TEMPERATURE FROM 1050°C TO 1200°C

A1050-T700

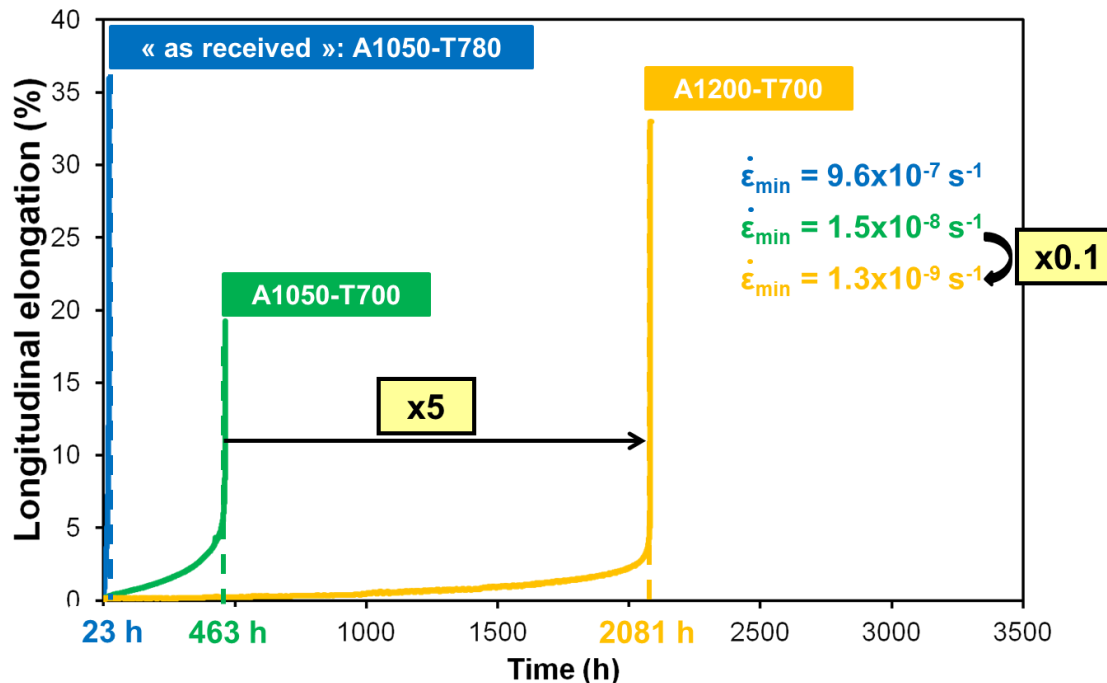


A1200-T700



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

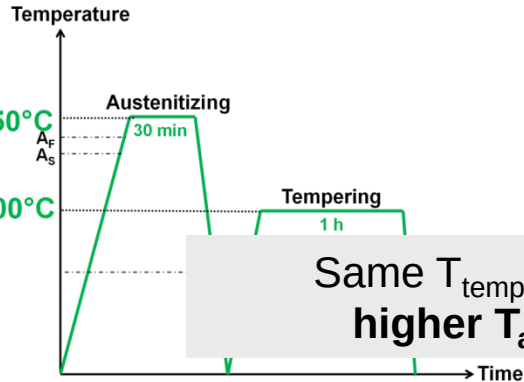
Creep conditions: 140 MPa at 650°C



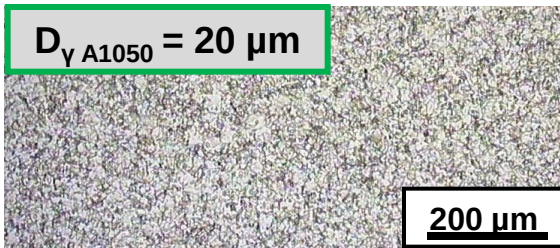
COMPARISON A1050-T700 VS A1200-T700

➔ AUSTENITIZING TEMPERATURE FROM 1050°C TO 1200°C

A1050-T700

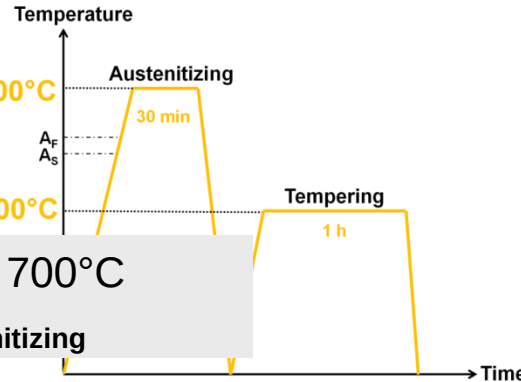


$D_{\gamma \text{ A1050}} = 20 \mu\text{m}$

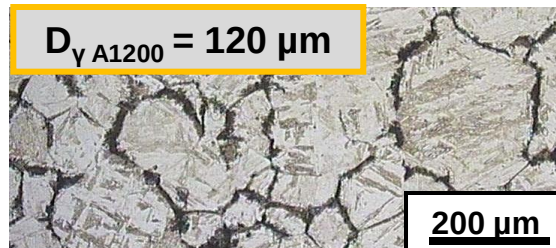


$W_{\text{blocks}} = 3.2 \mu\text{m}$

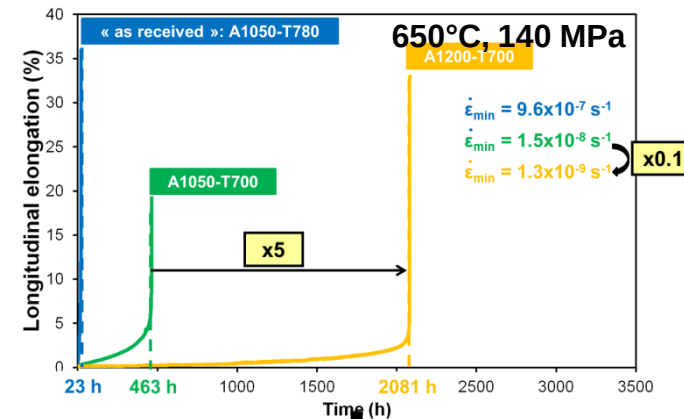
A1200-T700



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



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



Higher prior γ grain size
(dissolution of small precipitates)

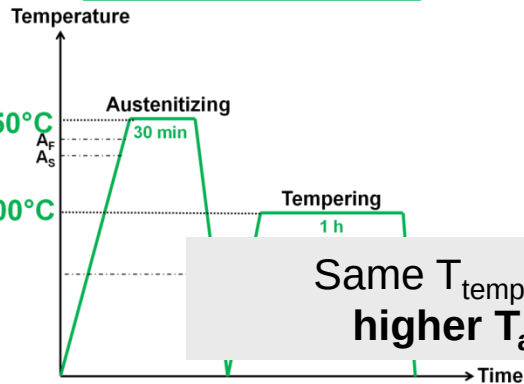


Higher martensite block size

COMPARISON A1050-T700 VS A1200-T700

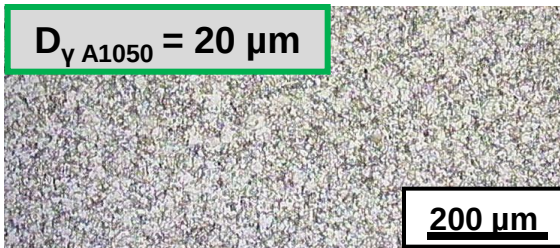
➔ AUSTENITIZING TEMPERATURE FROM 1050°C TO 1200°C

A1050-T700



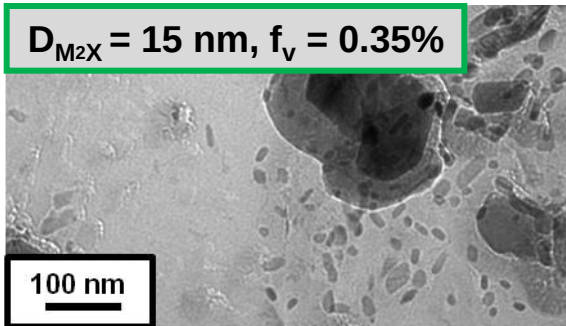
Same $T_{\text{tempering}}$: 700°C
higher $T_{\text{austenitizing}}$

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

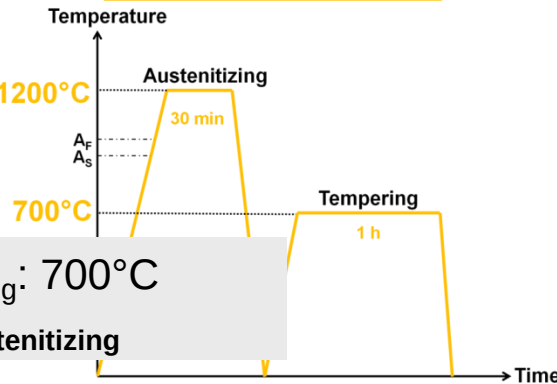


$W_{\text{blocks}} = 3.2 \mu\text{m}$

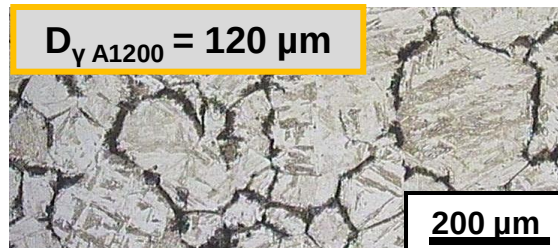
$D_{\text{M}_{2}\text{X}} = 15 \text{ nm}, f_v = 0.35\%$



A1200-T700

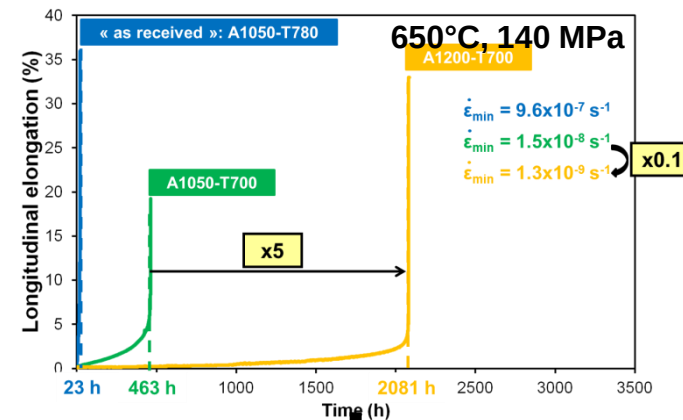
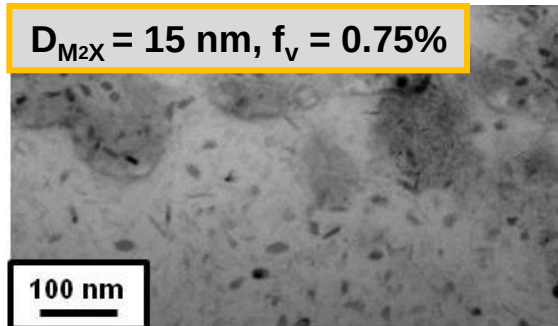


$D_{\gamma \text{ A1200}} = 120 \mu\text{m}$



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

$D_{\text{M}_{2}\text{X}} = 15 \text{ nm}, f_v = 0.75\%$



Higher prior γ grain size
(dissolution of small precipitates)



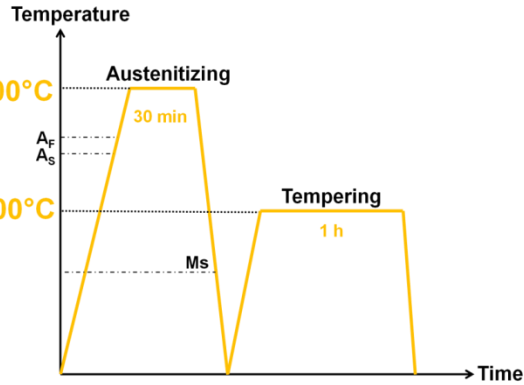
Higher martensite block size



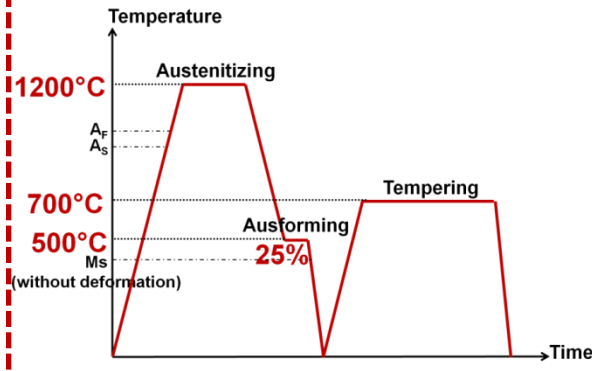
Higher f_v of intragranular
precipitates
(more microalloying elements
available for precipitation)

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

A1200-T700

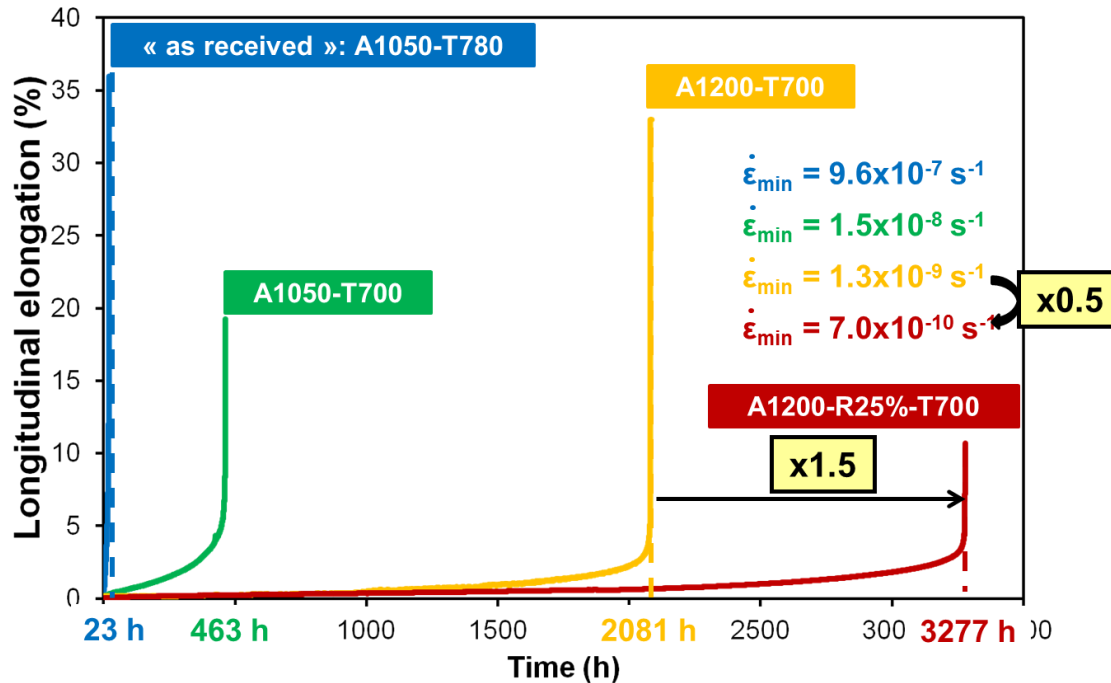


A1200-R25%-T700



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

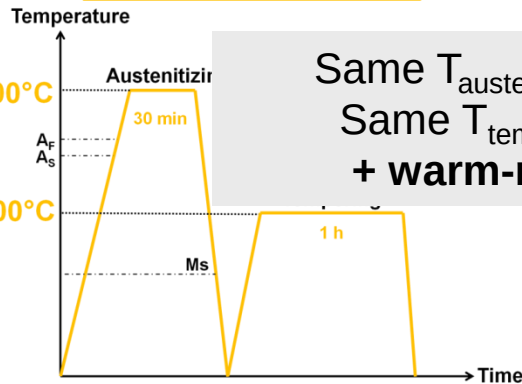
Creep conditions: 140 MPa at 650°C



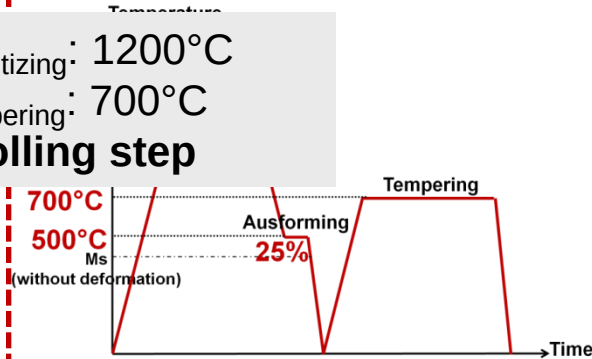
COMPARISON A1200-T700 VS A1200-R25%-T700 APPLYING WARM-ROLLING IN METASTABLE γ

A1200-T700

A1200-R25%-T700



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

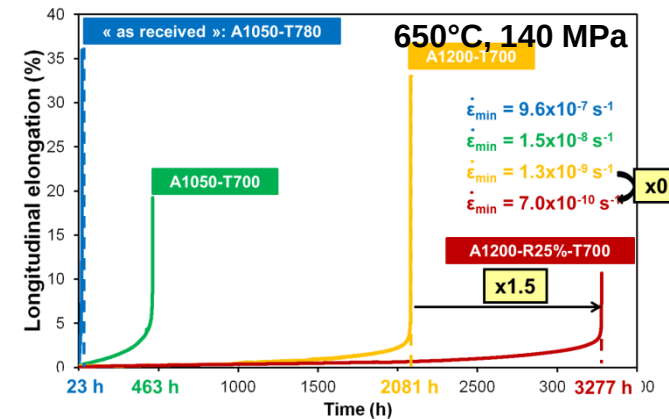


$W_{\text{laths}} \sim 400 \text{ nm}$

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

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

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



Finer lath width



Higher dislocation density
(inheritance from warm-rolling)

More than 30 treated conditions

4 states

Part 1

$T_{\text{tempering}}$

$T_{\text{austenitizing}}$

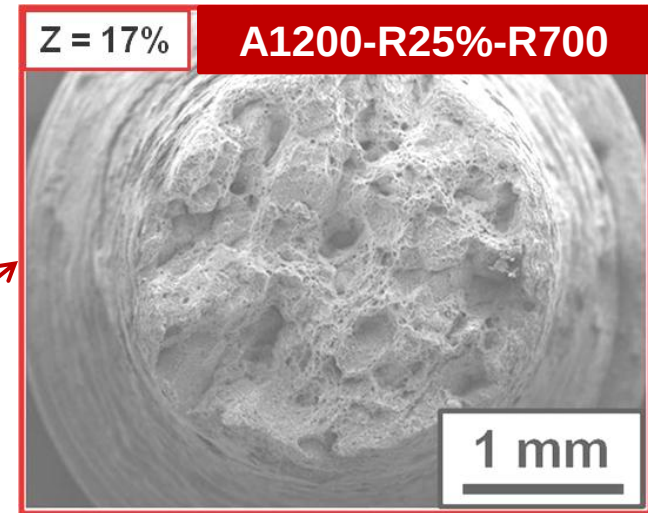
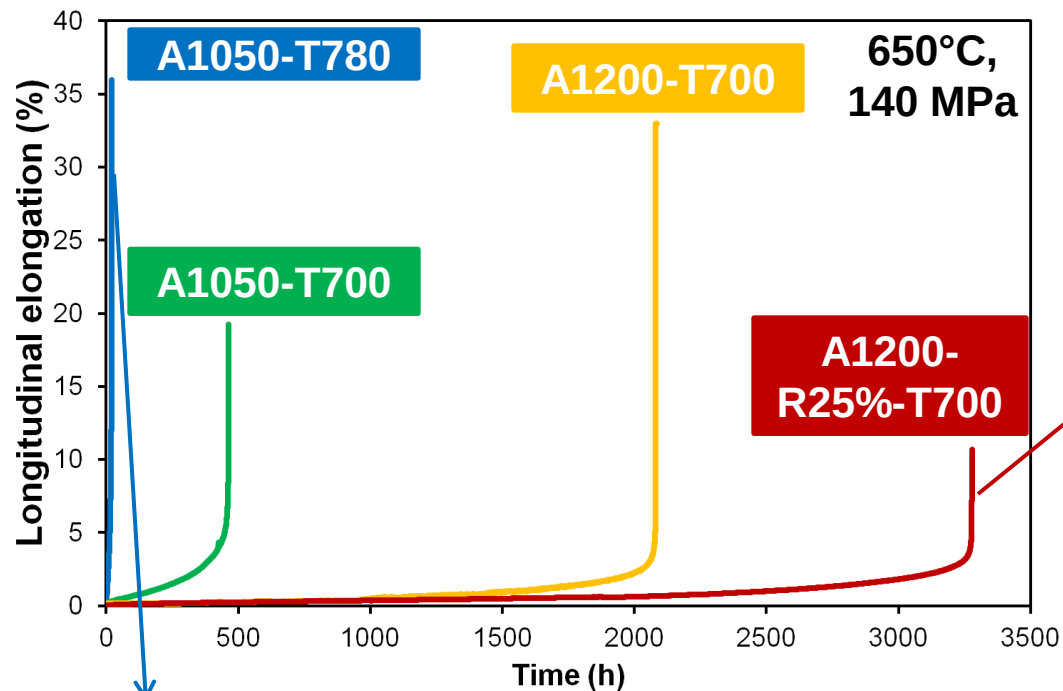
Warm-rolling

Time to fracture
Minimal creep rate

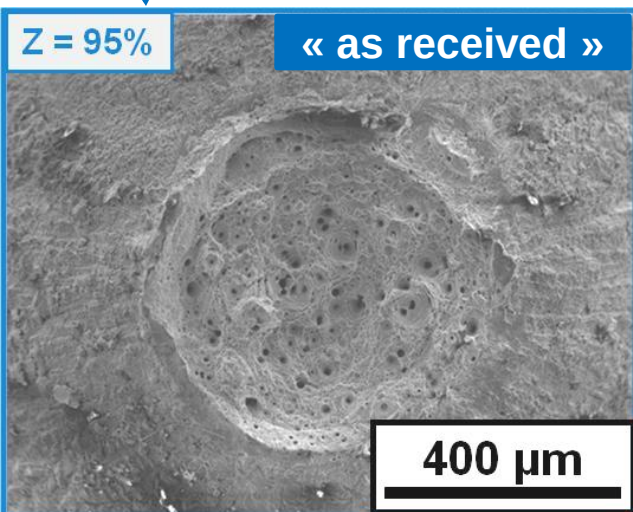
Microstructure

Part 2

Creep fracture mode



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

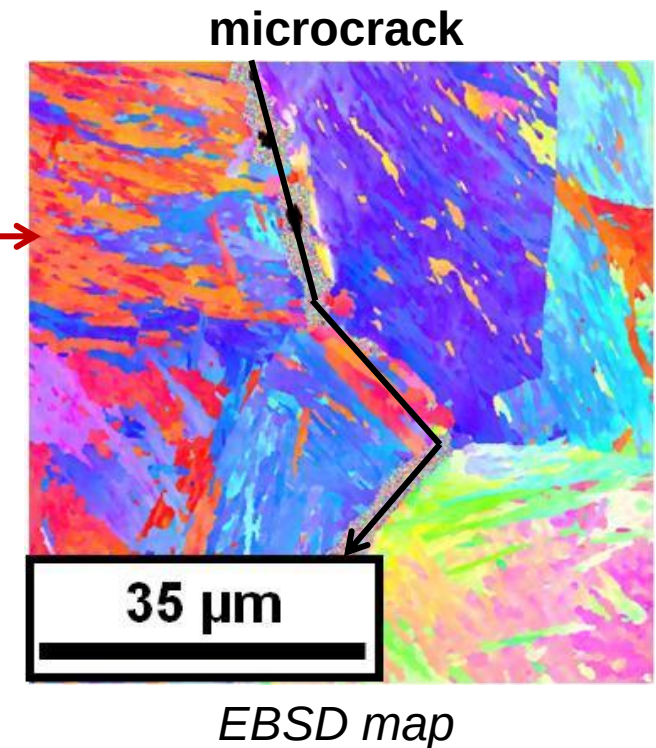
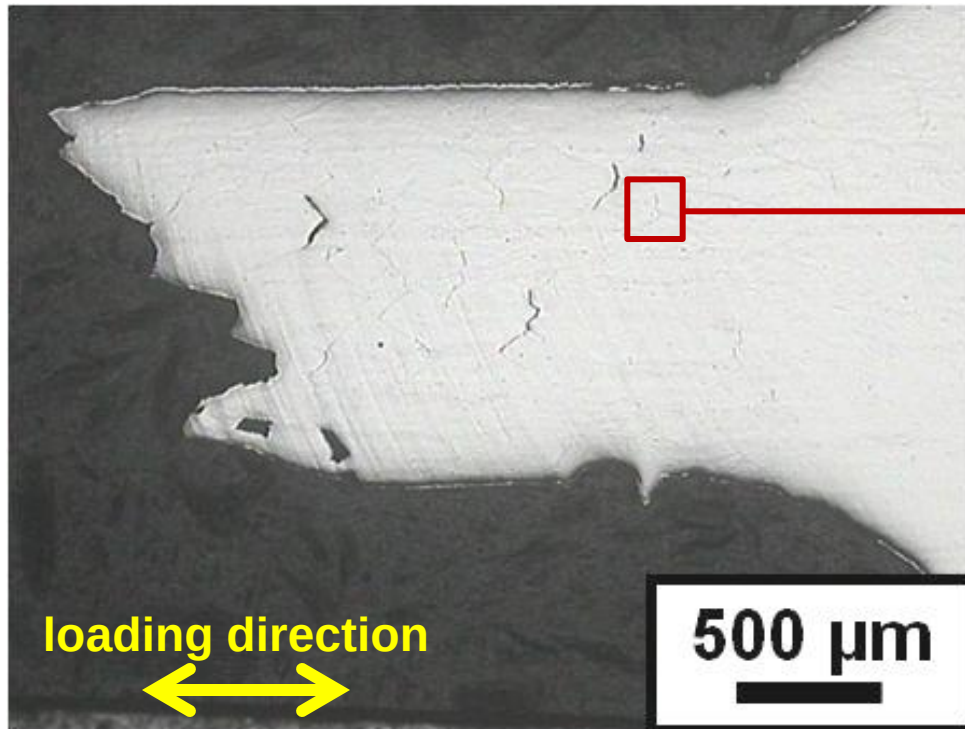


Significant tertiary creep stage,
high reduction of area at failure,
Ductile fracture.

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

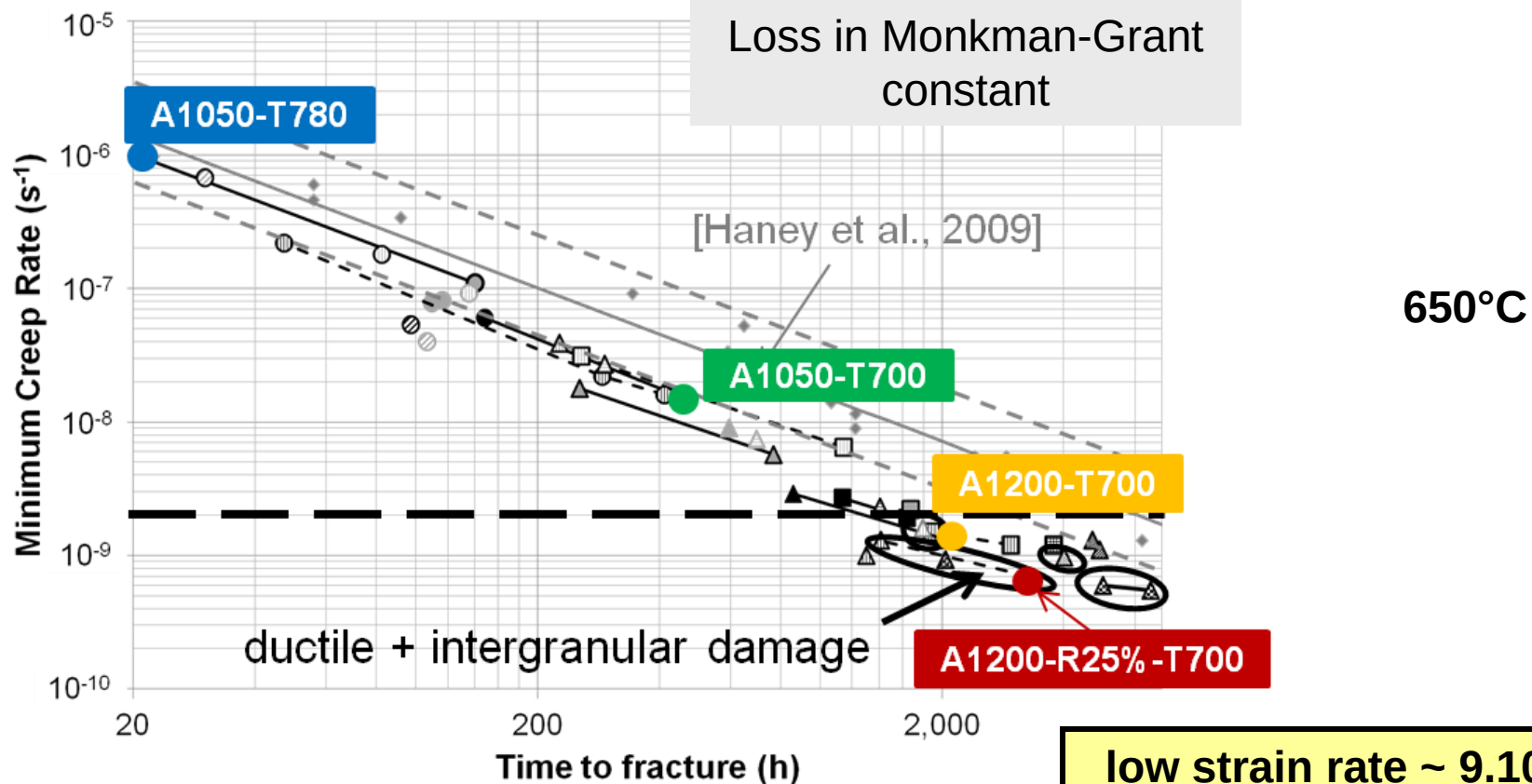
- Confirmation by EBSD observations: damage at prior austenite grain boundaries,

cross section observation



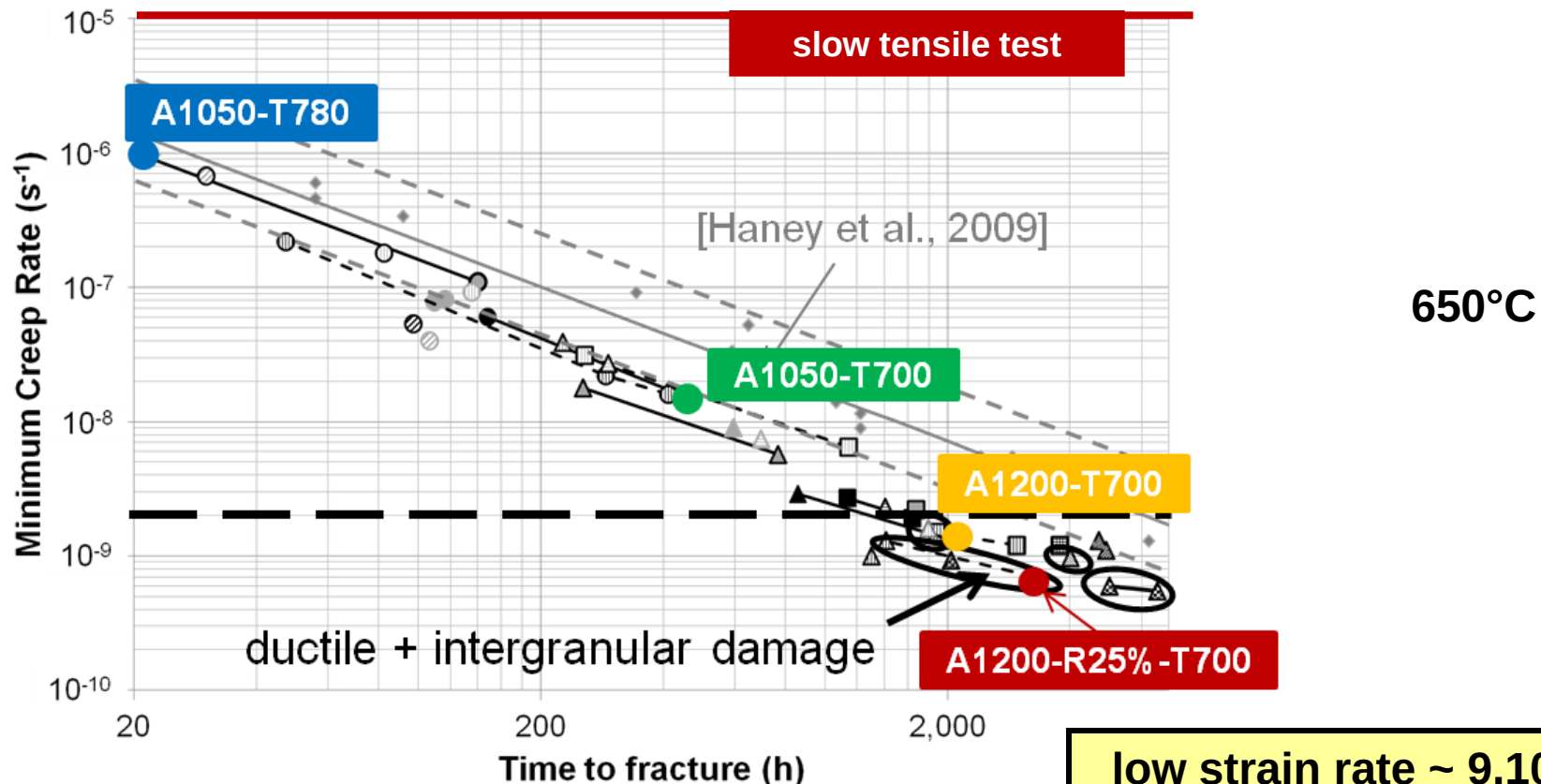
**Ductile + intergranular creep fracture mode observed for:
A1200-R(0-25%)-T(700-750°C) states (650°C, 140 MPa)**

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?

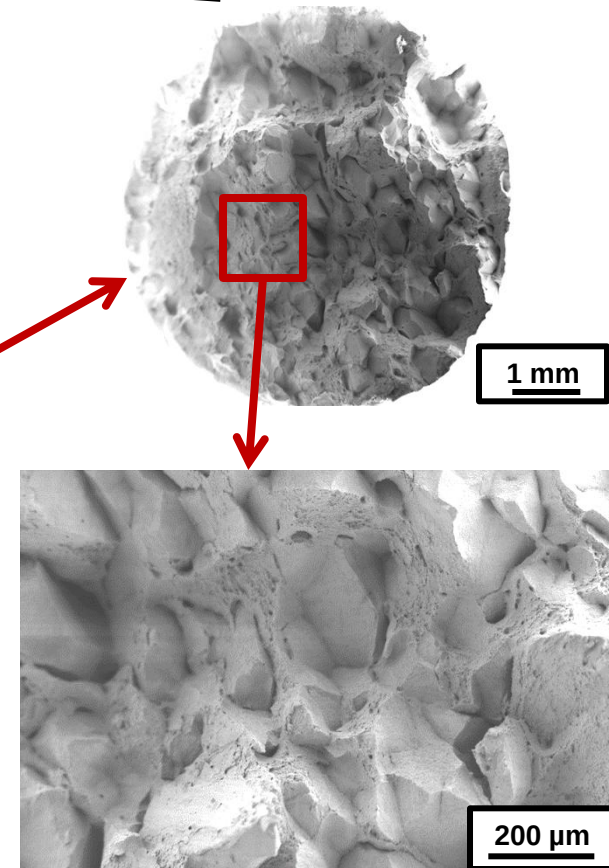
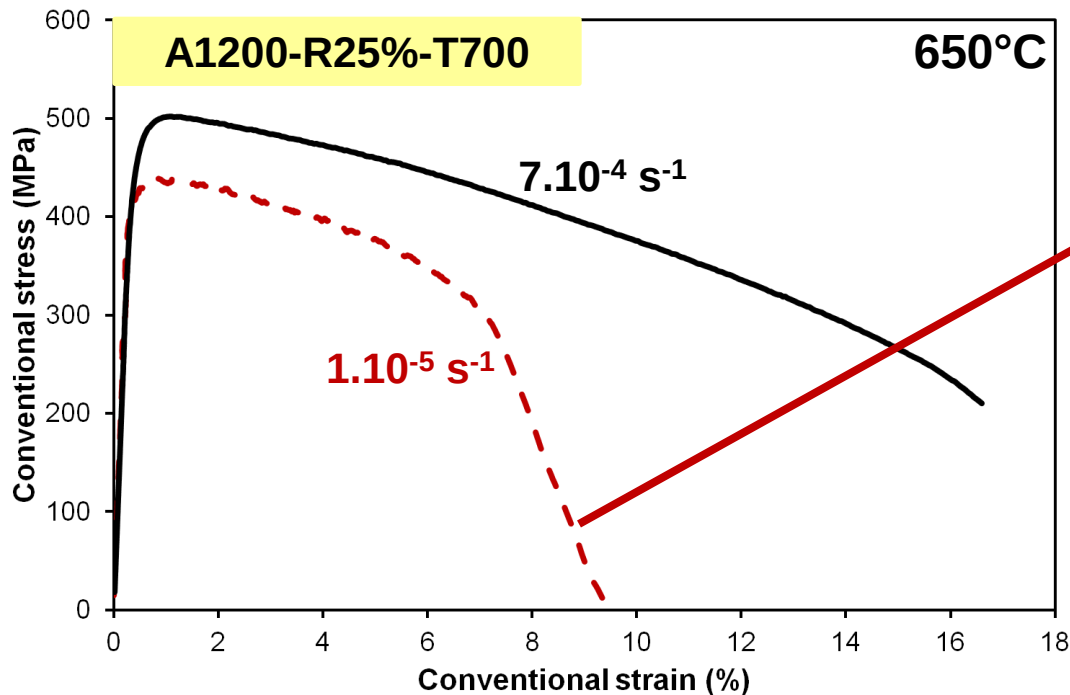


**Ductile + intergranular creep fracture mode observed for:
A1200-R(0-25%)-T(700-750°C) states (650°C, 140 MPa)**

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?



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

4 states (3 thermal + 1 thermomechanical treatments)

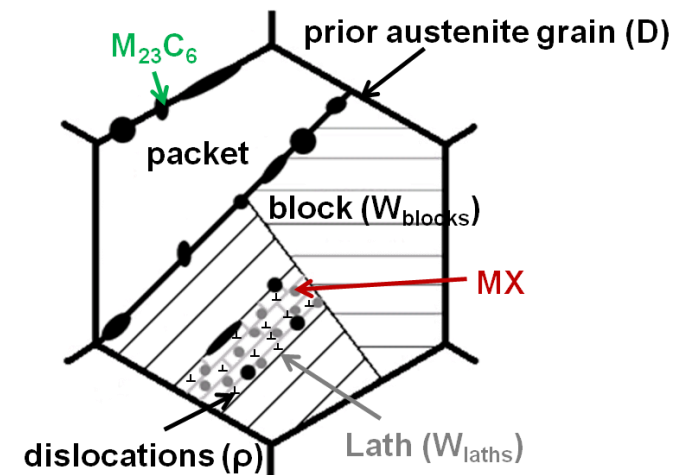
Effects of $T_{\text{austenitizing}}$, $T_{\text{tempering}}$, warm-rolling step on microstructural parameters and on creep flow:

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

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

1st order parameters

moderate effect



→ **Further work: Modelling**

■ 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 :

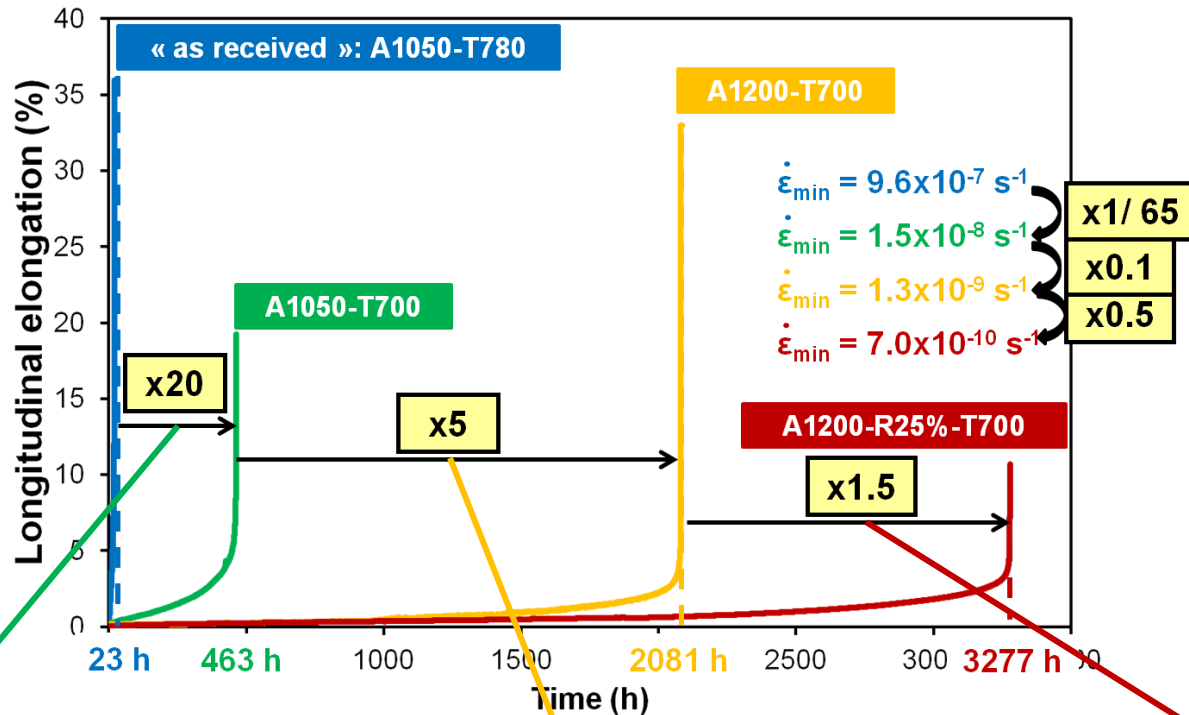
<https://pastel.archives-ouvertes.fr/tel-01141826>

... but in French !

Commissariat à l'énergie atomique et aux énergies alternatives
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DEN
DMN
SRMA/LA2M

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



↘ $T_{\text{tempering}}$

↗ $T_{\text{austenitizing}}$

Warm-rolling

↗ ρ

↗ $f_v \text{ intra}$

↗ D_v, W_{blocks}

1st order parameters

negligeable effect

Temperature

Austenitizing

30 min

Austenite

Tempering

Ausforming

Martensite

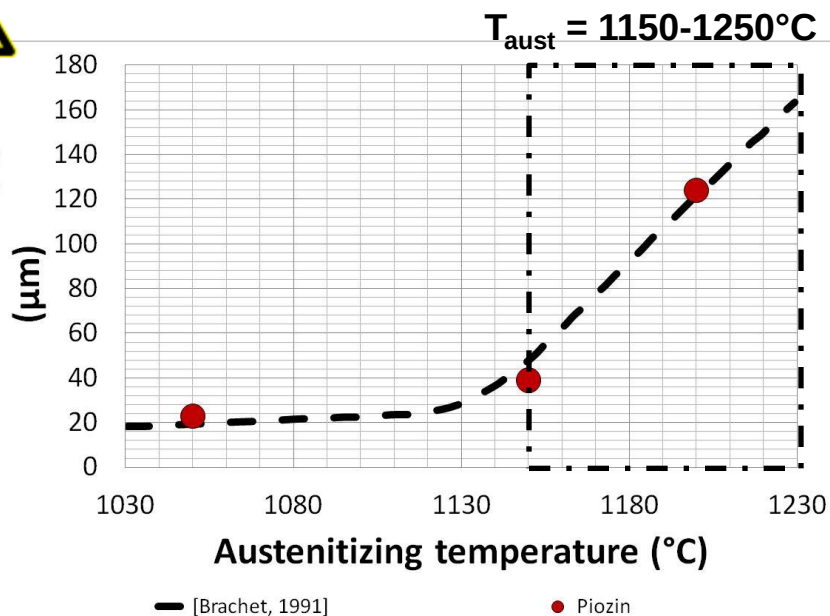
Ms
(without deformation)

Time

thermomechanical
treated



Mean diameter of γ grains
(μm)



Austenitizing temperature ($^{\circ}\text{C}$)

1050

“as received” condition

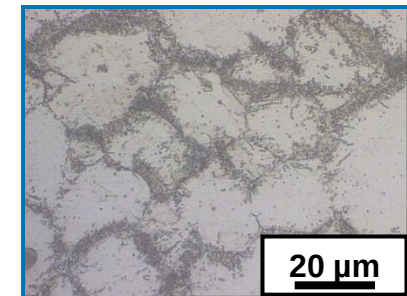
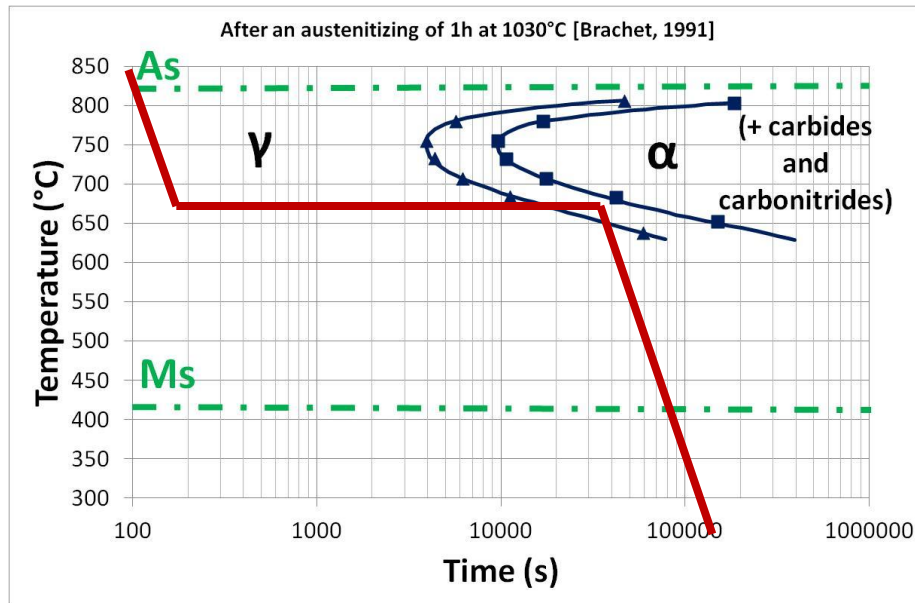
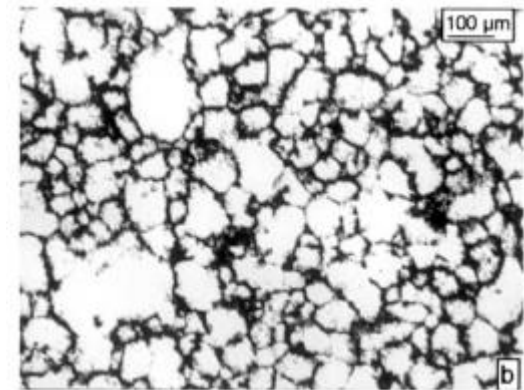
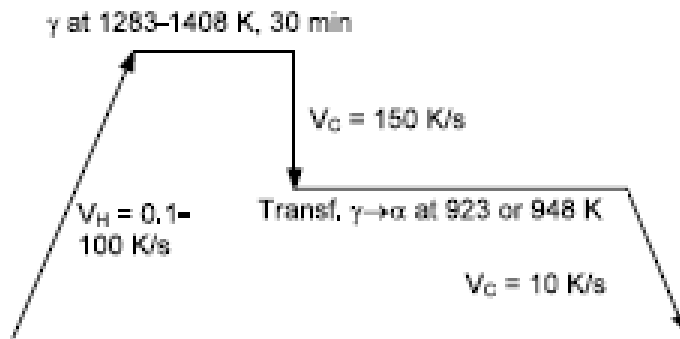
1150

1200

to dissolve MX precipitates

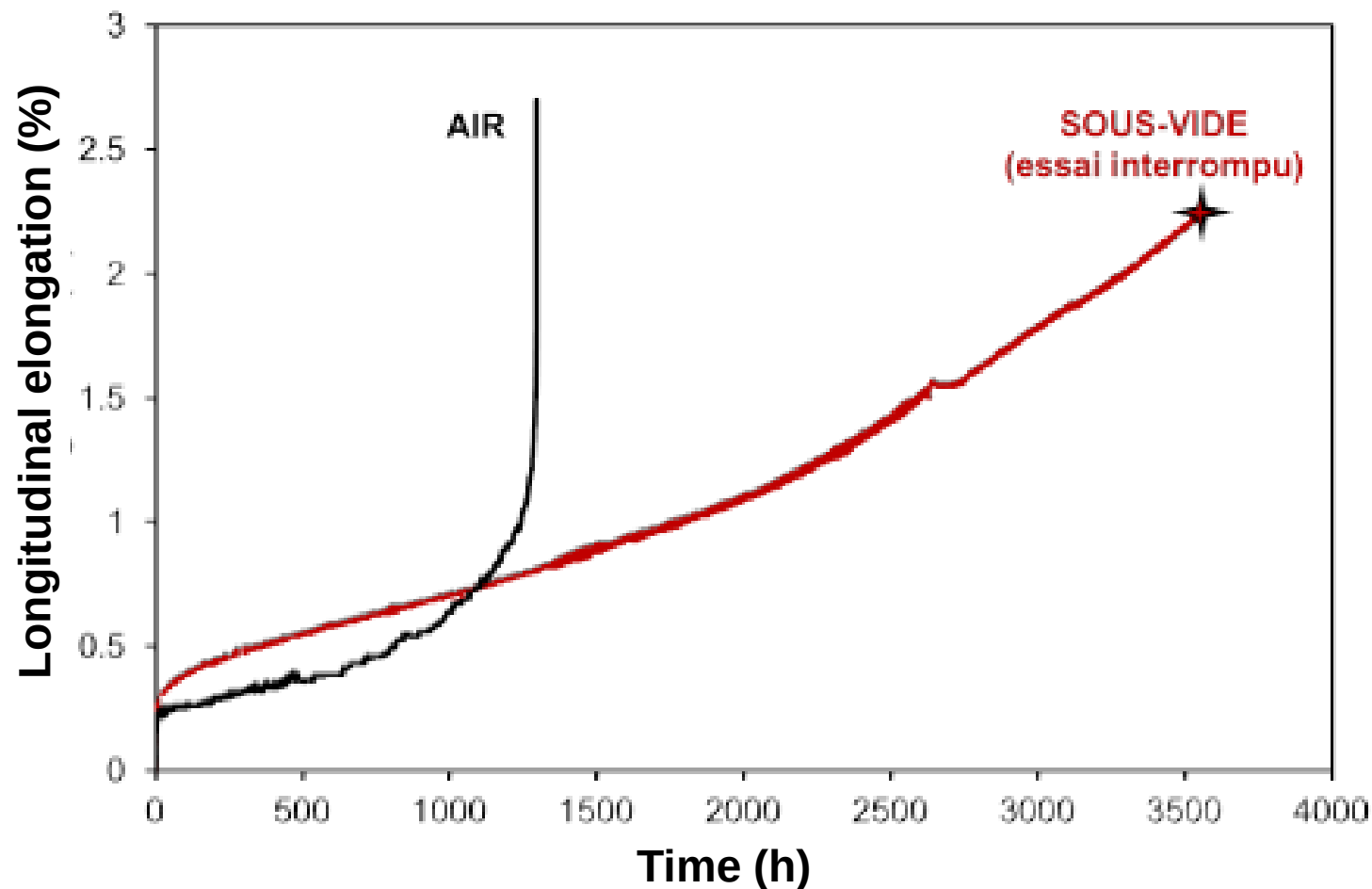
Experimental procedure to measure prior austenite grain size :

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



INFLUENCE OF ENVIRONNEMENT

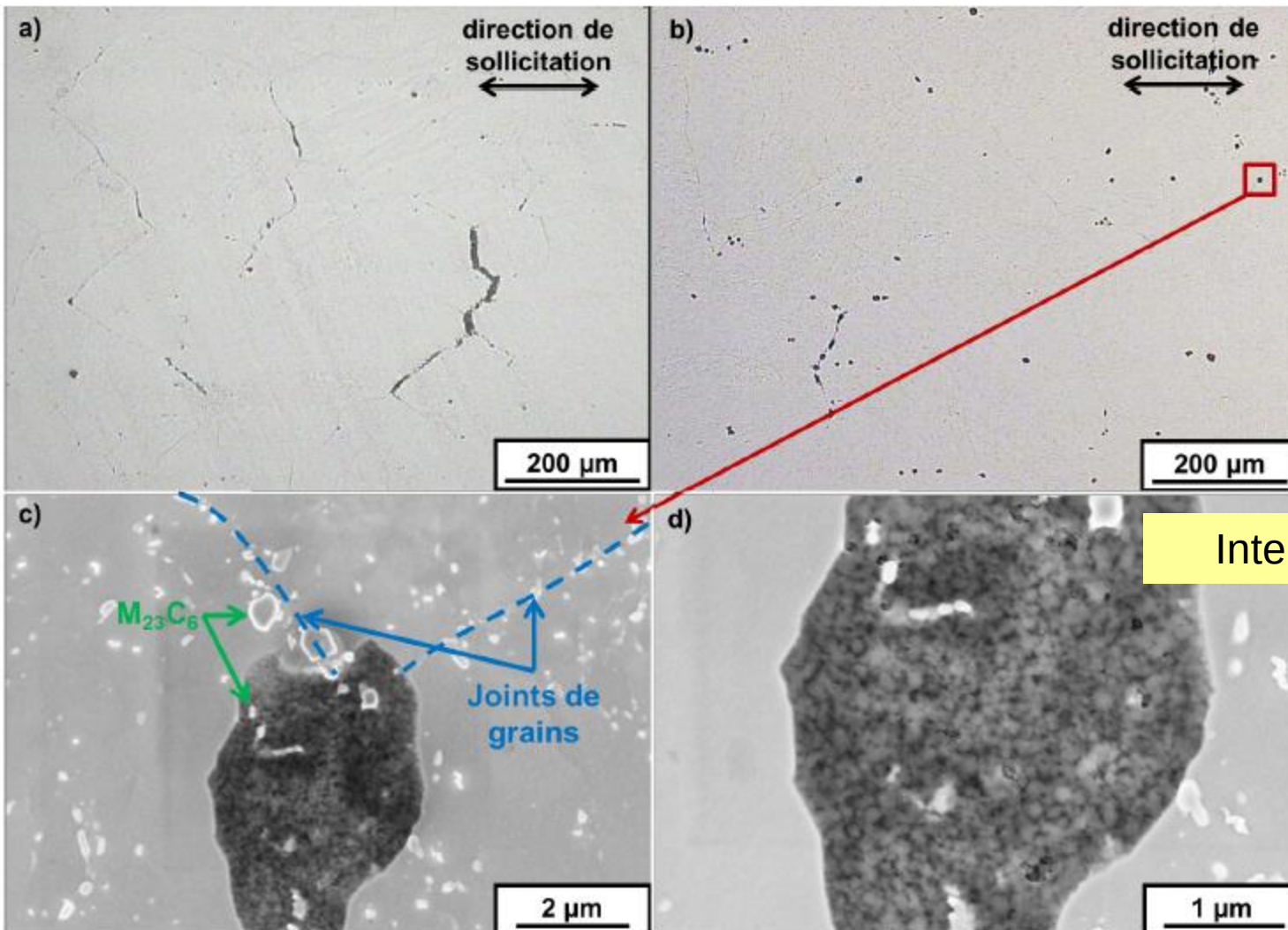
A1200-R500,25%-T700



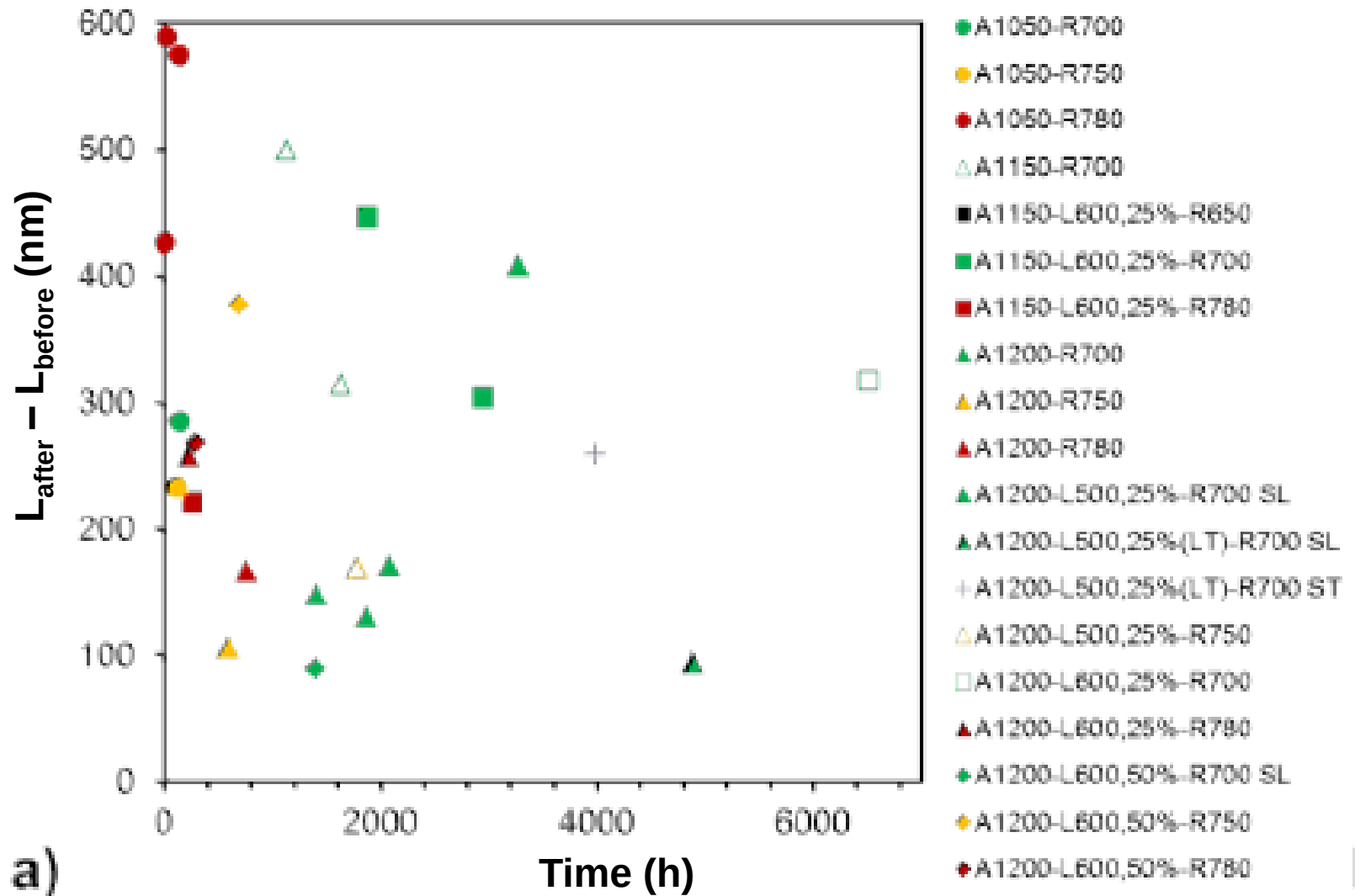
Same minimal creep rate:
 $\text{Air} = 1.10^{-9} \text{ s}^{-1}$, $\text{Vaccum} = 9.10^{-10} \text{ s}^{-1}$.
But t_r higher for vaccum !

INFLUENCE OF ENVIRONNEMENT

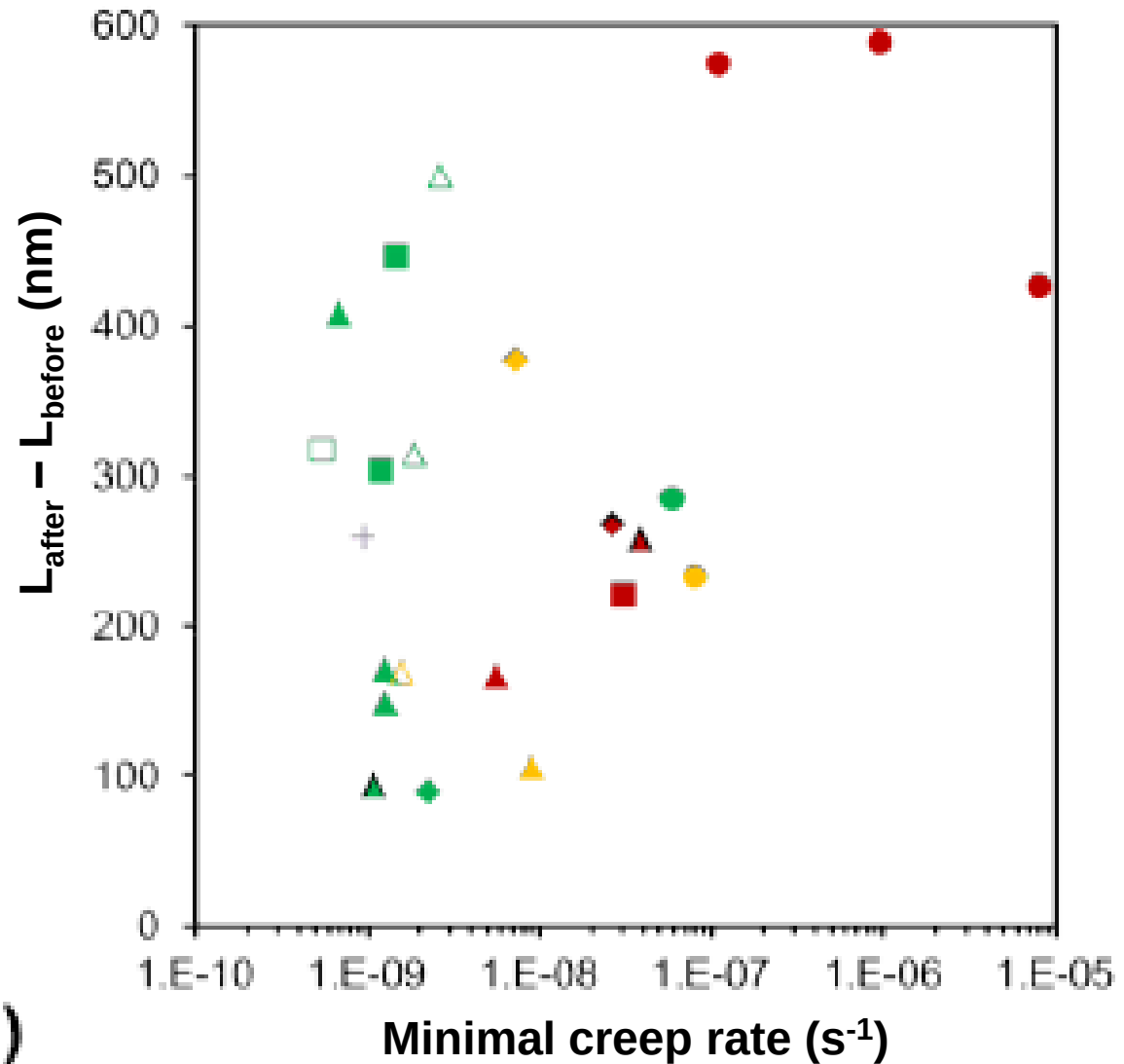
A1200-R500,25%-T700 (vacuum)

cross section observations

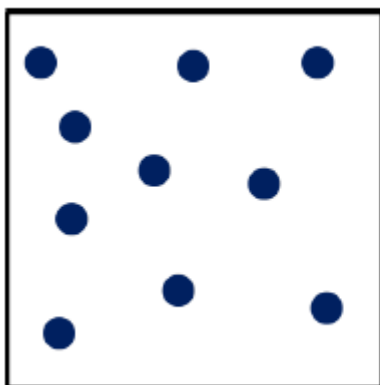
MICROSTRUCTURE AFTER CREEP TESTS



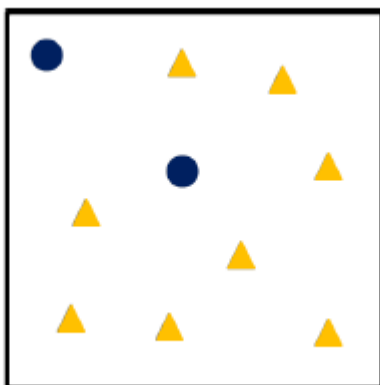
- A1050-R700
- A1050-R750
- A1050-R780
- △ A1150-R700
- A1150-L600,25%-R650
- A1150-L600,25%-R700
- A1150-L600,25%-R780
- ▲ A1200-R700
- ▲ A1200-R750
- ▲ A1200-R780
- ▲ A1200-L500,25%-R700 SL
- ▲ A1200-L500,25%(LT)-R700 SL
- + A1200-L500,25%(LT)-R700 ST
- ▲ A1200-L500,25%-R750
- A1200-L600,25%-R700
- ▲ A1200-L600,25%-R780
- A1200-L600,50%-R700 SL
- A1200-L600,50%-R750
- A1200-L600,50%-R780



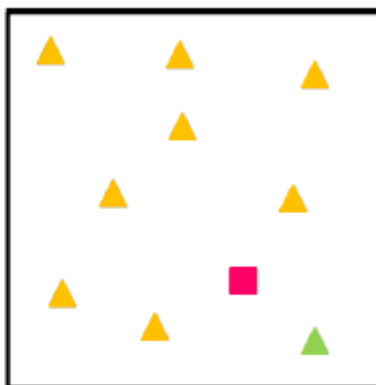
MICROSTRUCTURE AFTER CREEP TESTS



non sollicité



vieilli thermiquement à
650°C pendant 6523
heures



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

A1200-R600,25%-T700

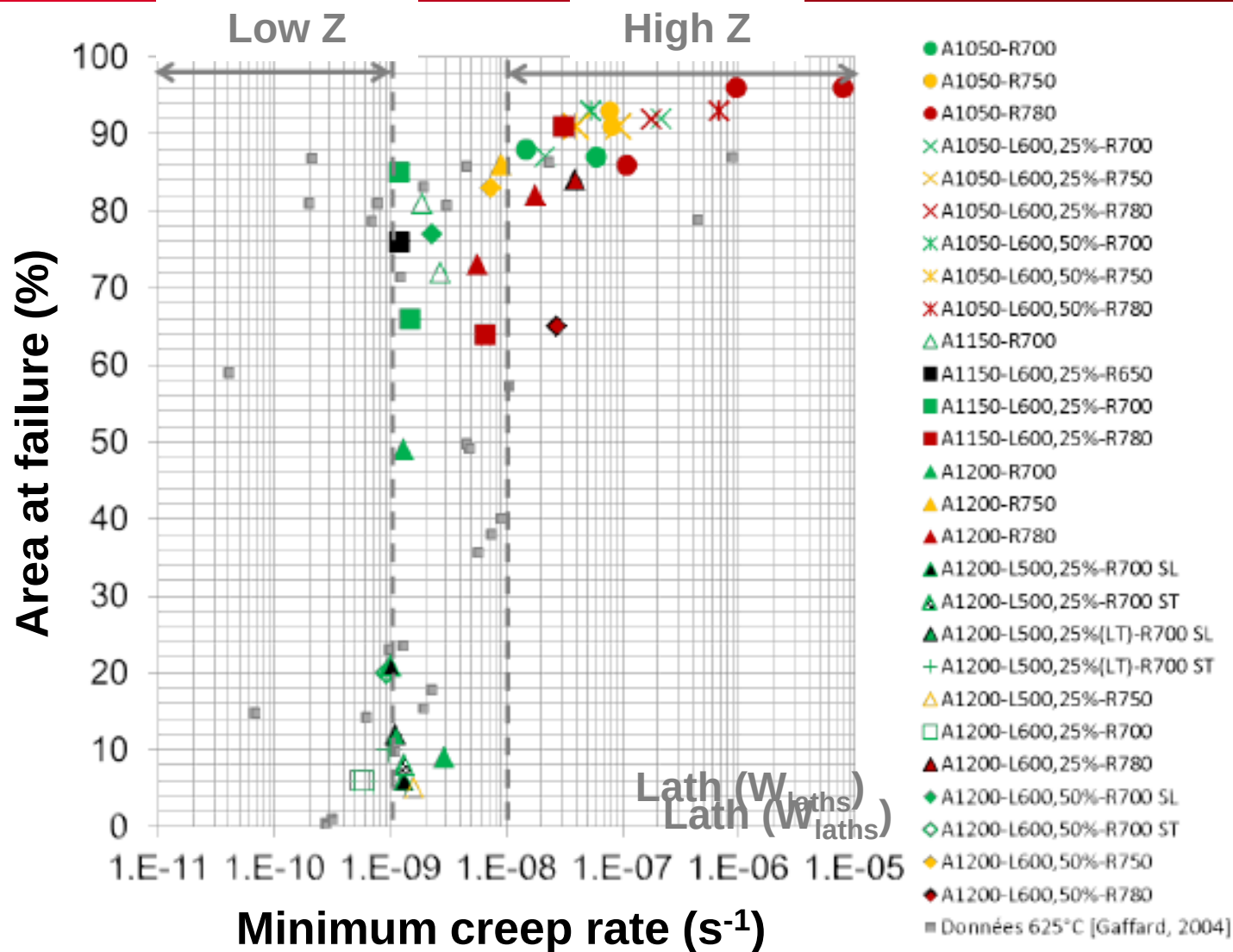
Creep test :
140 MPa, 650°C
 $t_r = 6523$ h

- 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 $M=(80\%Nb, 15\%V, 5\%Cr)$

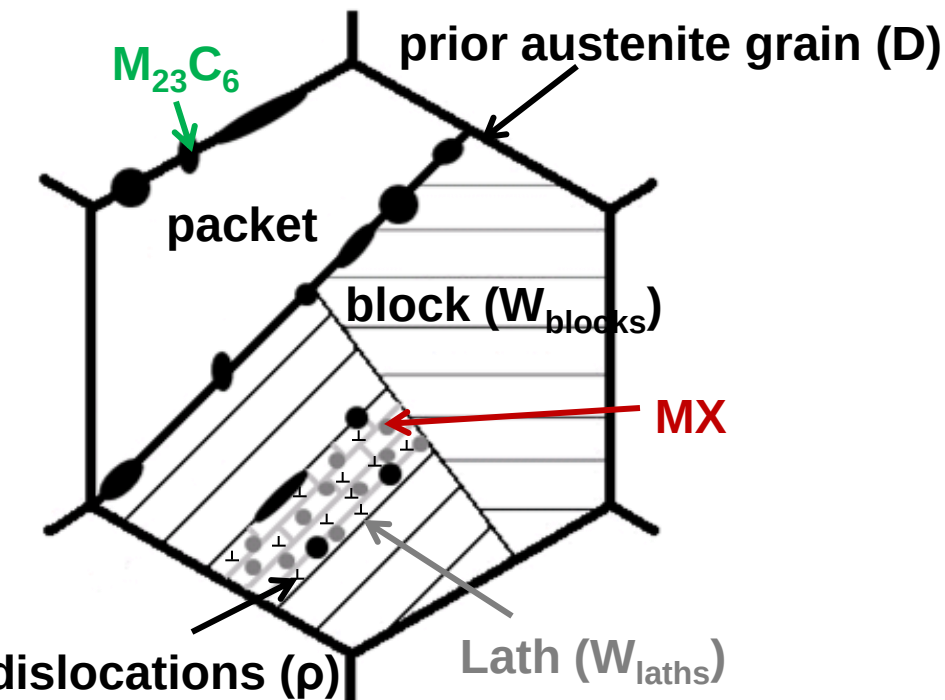
Change in chemical composition
of MX precipitates

	No creep tested	Creep tested head	Creep tested gauge
$D_{M_{23}C_6}$ (nm)	123 ± 58	130 ± 48	147 ± 55
D_{MX} (nm)	18 ± 7	16 ± 4	20 ± 4

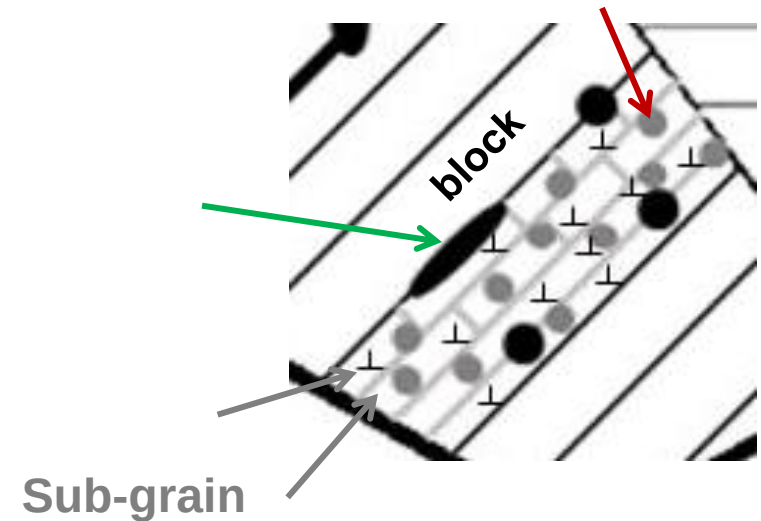
MINIMUM CREEP RATE VALUE LIMIT !



- Simplified chemistry (wt %): Fe-9Cr-1Mo-0,1C-(NNbV)
- 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_{blocks}, W_{laths}, precipitates, dislocation density