



HAL
open science

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

E. Piozin, J.-C. Brachet, S. Vincent, A.-F. Gourgues

► To cite this version:

E. Piozin, J.-C. Brachet, S. Vincent, A.-F. Gourgues. Effects of microstructural parameters on creep flow and fracture of a 9Cr-1Mo-NNbV tempered martensitic steel. CREEP 2015 - 13th International Conference on Creep and Fracture of Engineering Materials and Structures, May 2015, Toulouse, France. cea-02491639

HAL Id: cea-02491639

<https://cea.hal.science/cea-02491639v1>

Submitted on 26 Feb 2020

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

DE LA RECHERCHE À L'INDUSTRIE



CENTRE DES MATERIAUX
P. M. FOURT

www.cea.fr

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

Emma PIOZIN,

Jean-Christophe BRACHET (CEA Saclay),

Sébastien VINCENT (CEA Saclay),

Anne-Françoise GOURGUES-LORENZON

(MINES ParisTech – Centre des Matériaux, Evry)

■ Considered:

→ nuclear fusion reactors:

- **tokamak blankets.**

→ SFR (Generation IV) :

- **steam generator** and **secondary piping.**

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

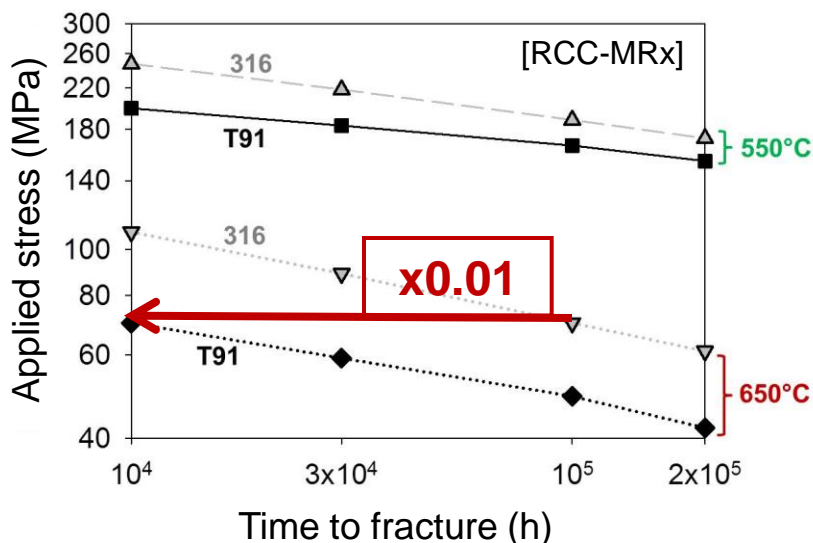
- **Considered:**
 - nuclear fusion reactors:
 - tokamak blankets.
 - SFR (Generation IV) :
 - steam generator and secondary piping.

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

Limited creep strength compare to austenitic stainless steels

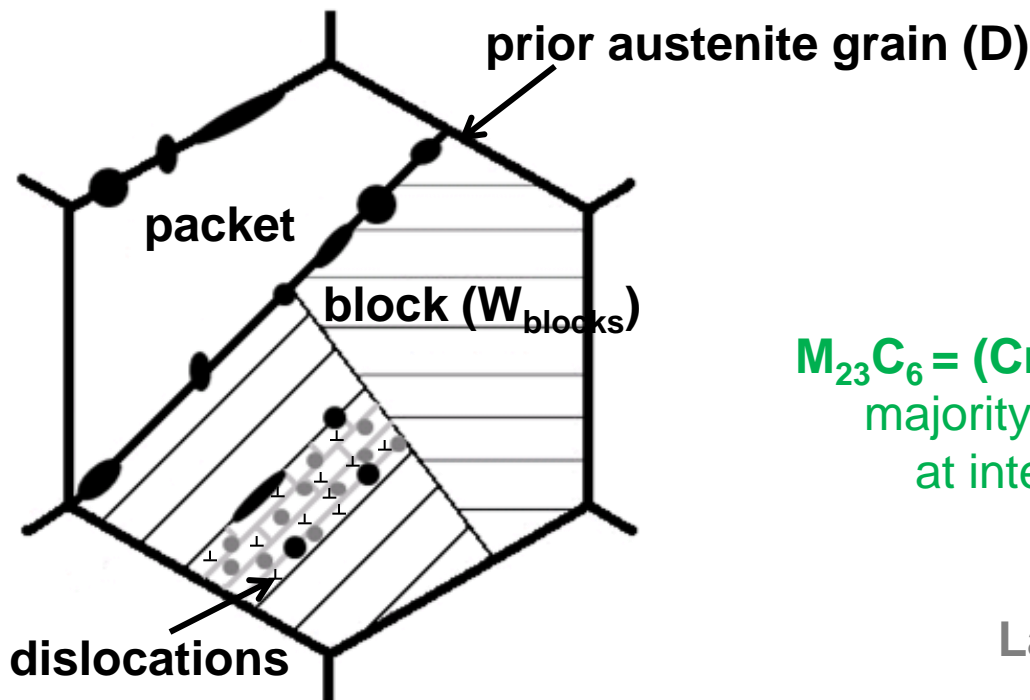
Creep at 550°C and 650°C

T91 =
« standard »
thermal
treatment



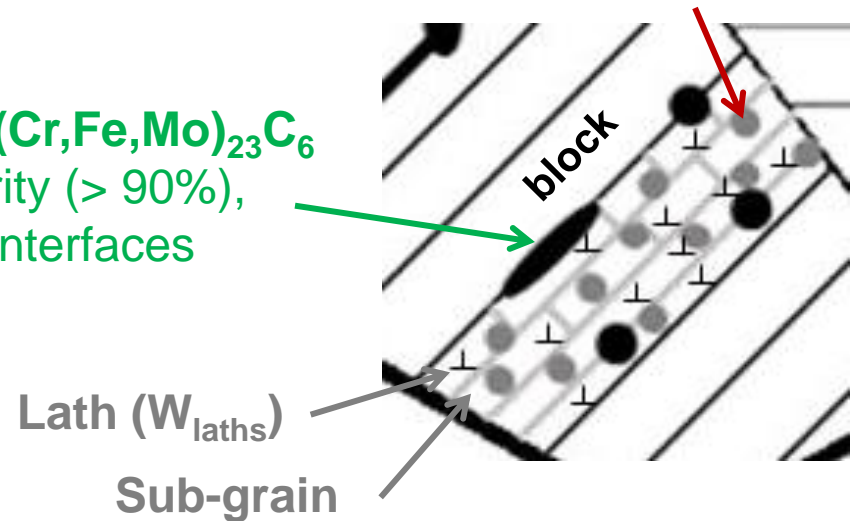
**Need of increasing
strength**

- 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**



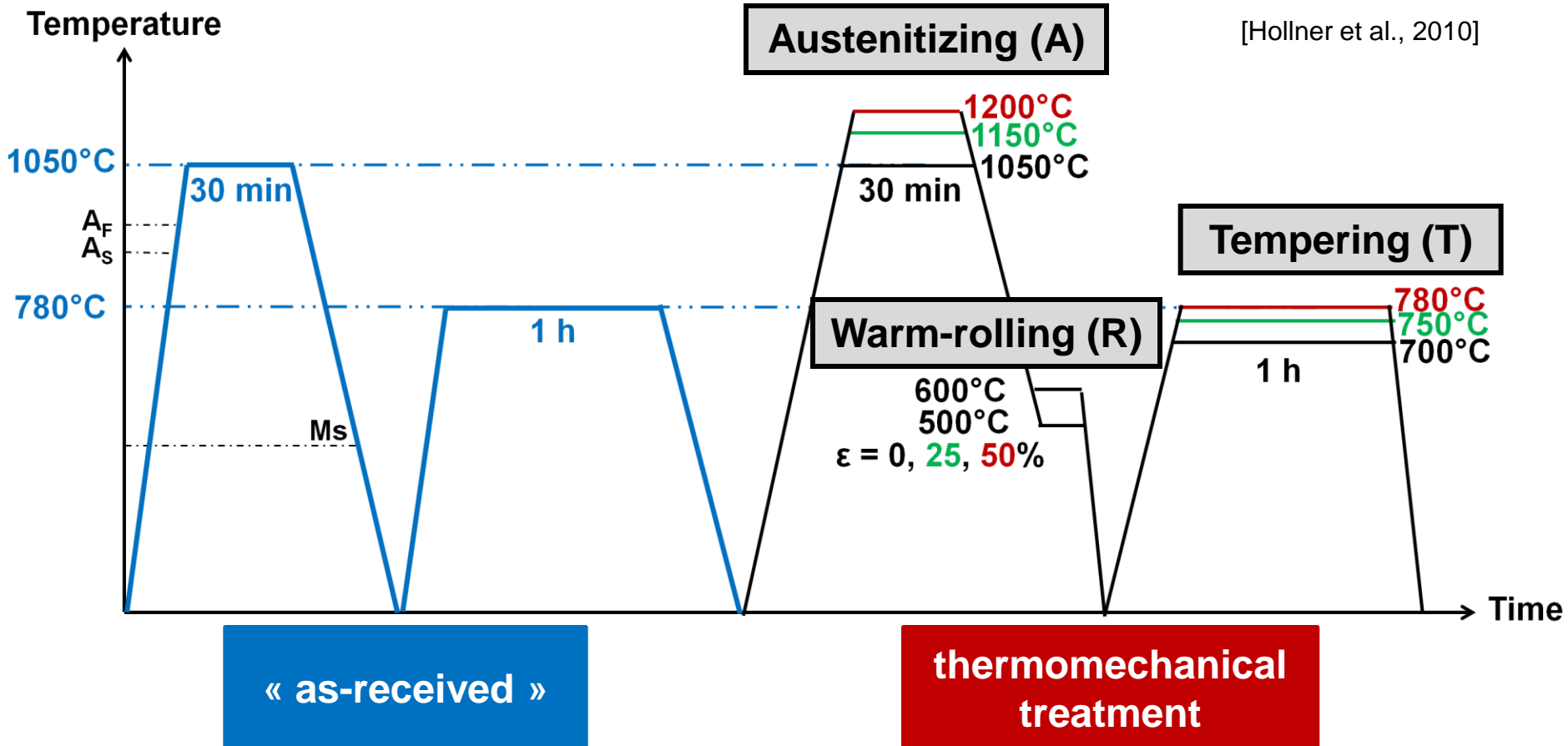
MX = (Nb,V)(C,N)
minority (<10%),
intragranular
incoherent (no-shearable)

$M_{23}C_6 = (Cr,Fe,Mo)_{23}C_6$
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

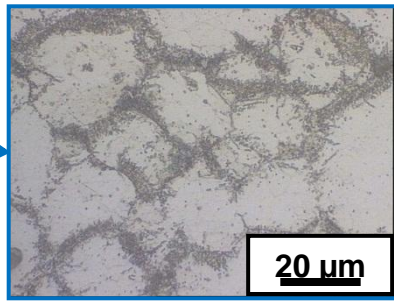
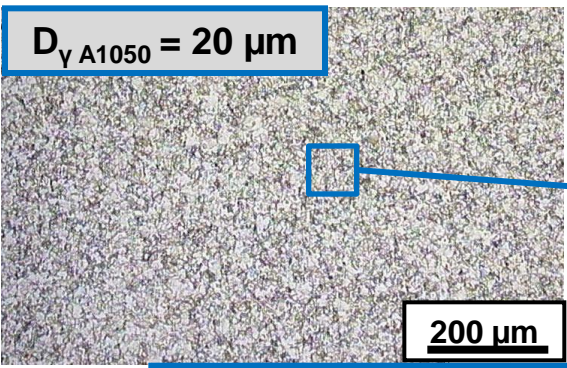
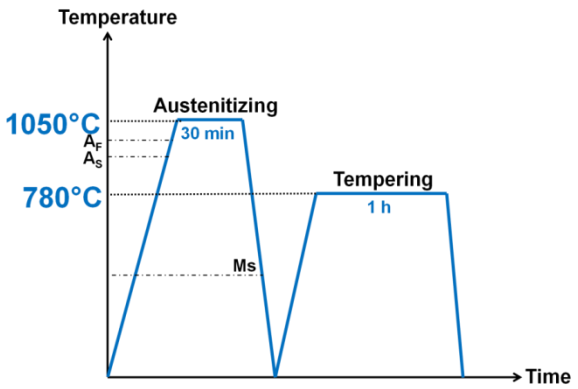
Microstructure

Part 2

Creep fracture mode

“AS RECEIVED”: A1050-T780

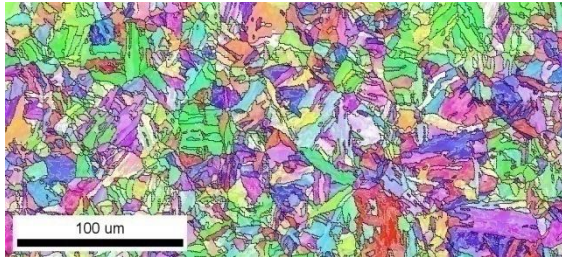
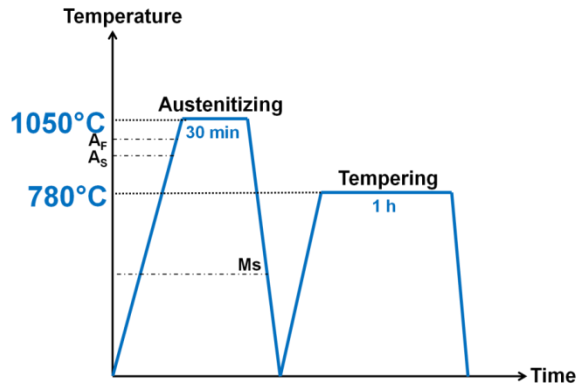
« as received »: A1050-T780



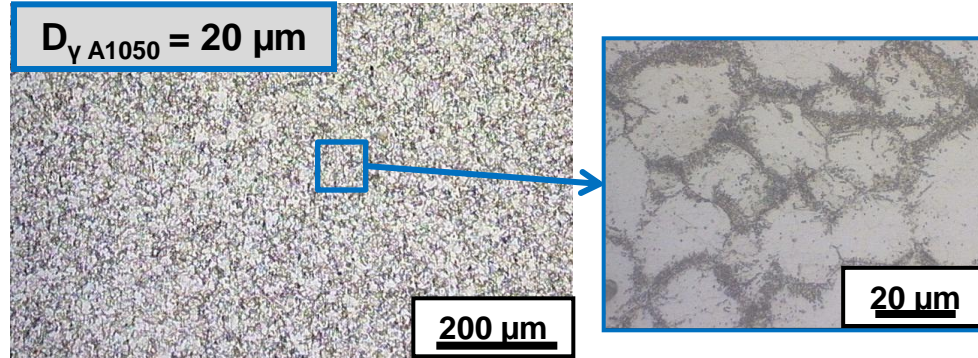
Fine prior austenite grain size

“AS RECEIVED”: A1050-T780

« as received »: A1050-T780



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



Fine prior austenite grain size

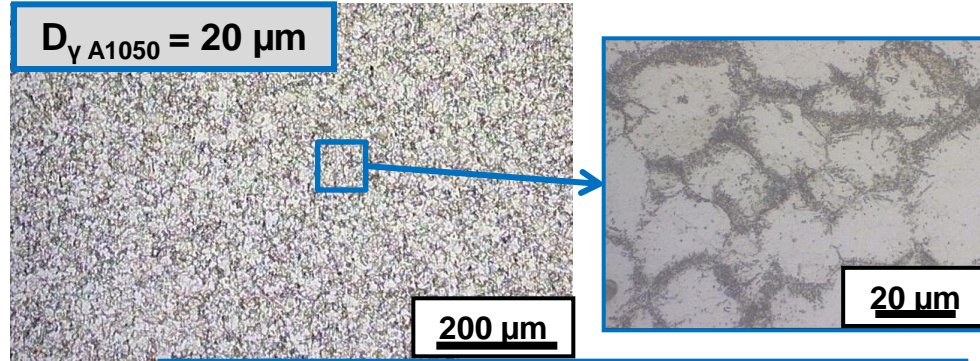
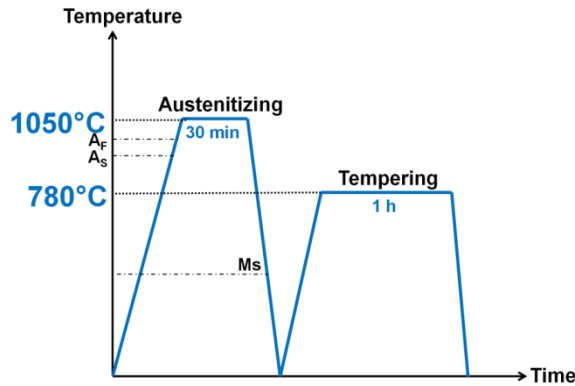
$$W_{\text{blocks}} = 3.2 \mu\text{m} (*)$$

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

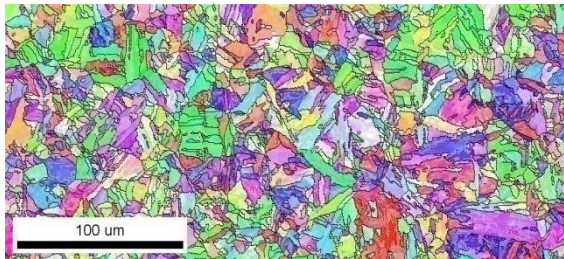
(*) SEM-EBSD,
(**) TEM on thin foils.

“AS RECEIVED”: A1050-T780

« as received »: A1050-T780



Fine prior austenite grain size



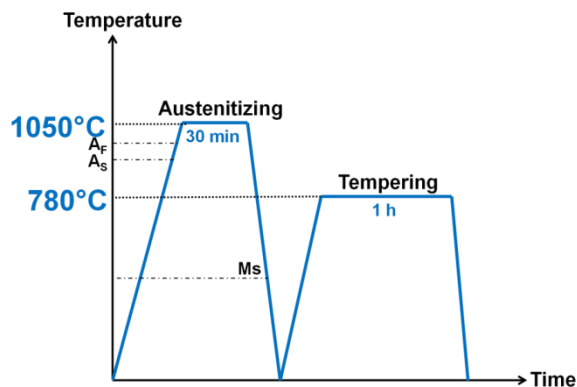
$W_{\text{blocks}} = 3.2 \mu\text{m}$ (*)
 $W_{\text{laths}} \sim 400 \text{ nm}$ (**)

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

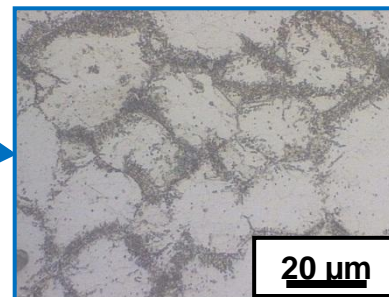
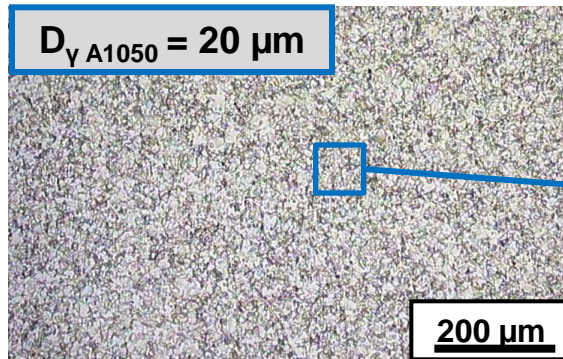
$\rho = (0.62 \pm 0.05) \times 10^{14} \text{ m}^{-2}$ (***)

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

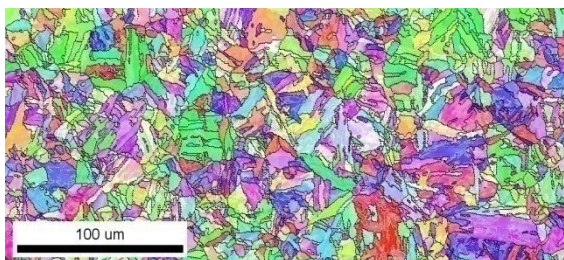
« as received »: A1050-T780



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



Fine prior austenite grain size



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

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

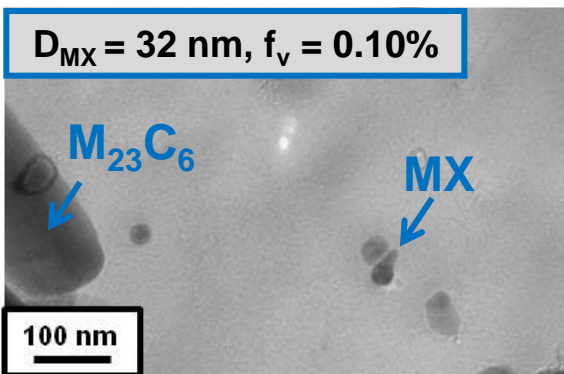
(*) SEM-EBSD,

(**) TEM on thin foils.

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

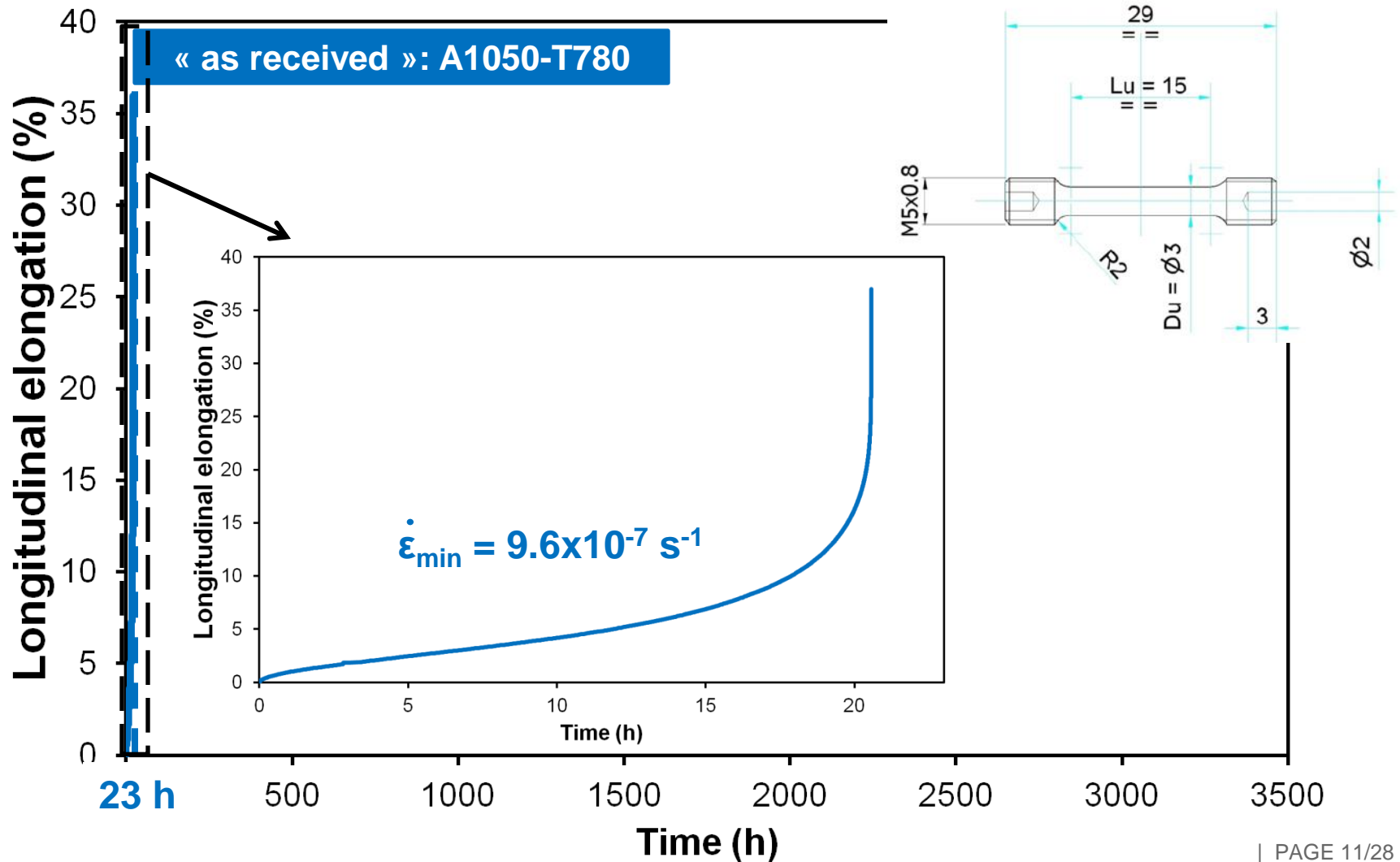
(***) neutron diffraction (*FWHM martensite peaks*).

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



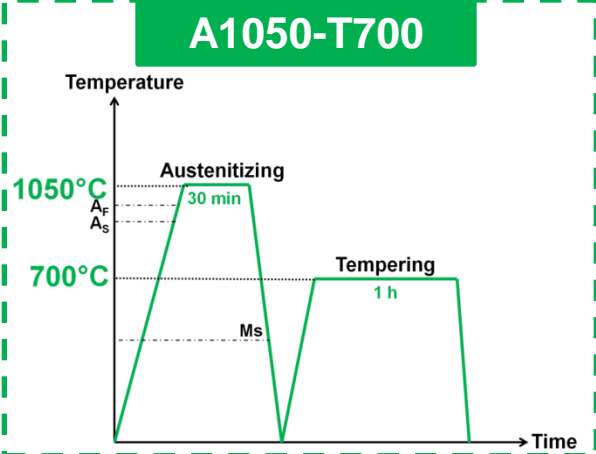
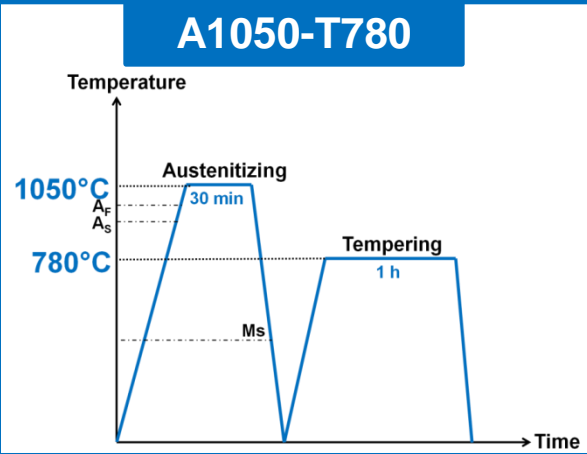
Coarse $M_{23}C_6$ precipitates at the different interfaces
and few intragranular MX precipitates

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



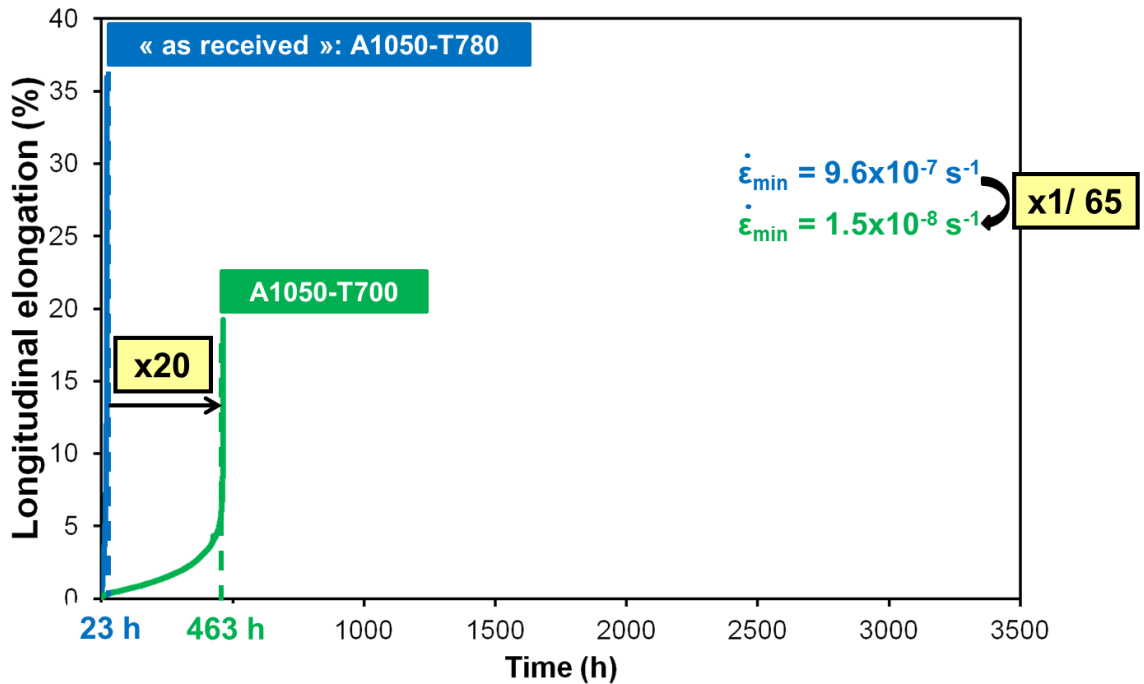
COMPARISON "AR" VS A1050-T700

↳ TEMPERING TEMPERATURE FROM 780°C TO 700°C



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

■ Creep conditions: 140 MPa at 650°C

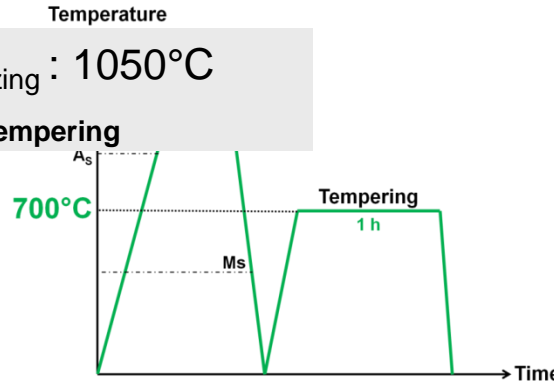
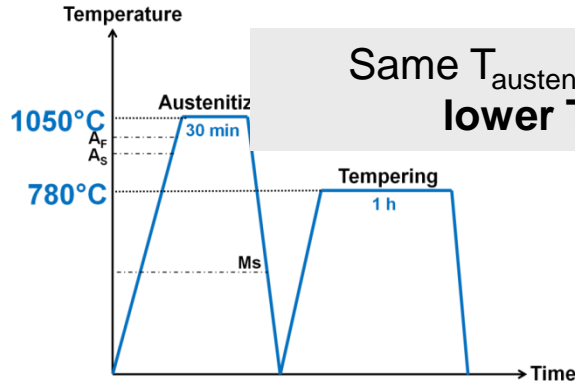


COMPARISON "AR" VS A1050-T700 ↳ TEMPERING TEMPERATURE FROM 780°C TO 700°C

A1050-T780

A1050-T700

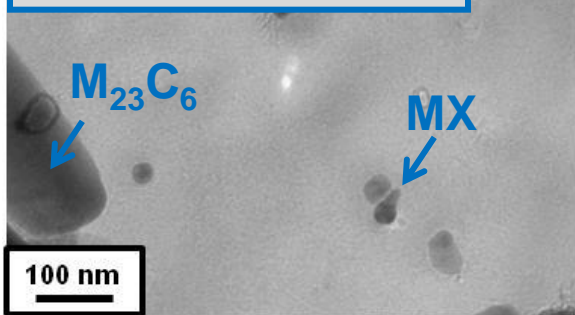
Same $T_{\text{austenitizing}} : 1050^{\circ}\text{C}$
lower $T_{\text{tempering}}$



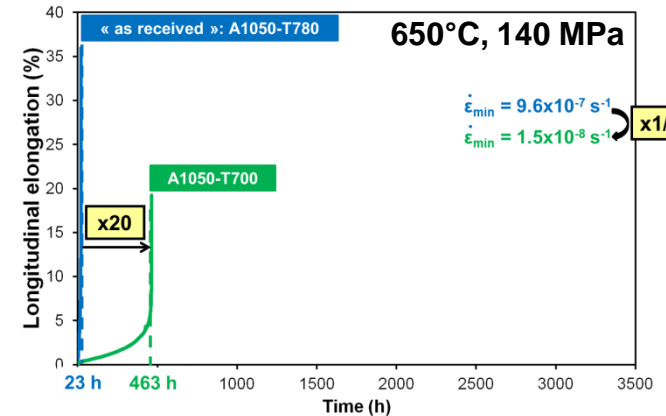
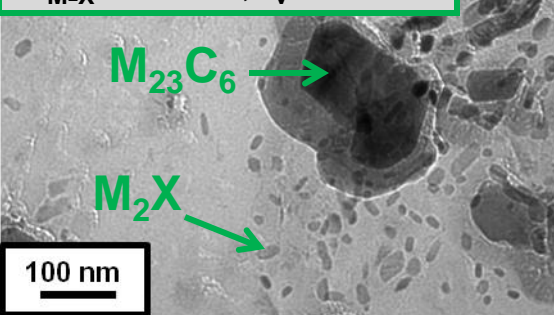
$$\rho = (0.62 \pm 0.05) \times 10^{14} \text{ m}^{-2}$$

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

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



$$D_{\text{M}_2\text{X}} = 15 \text{ nm}, f_v = 0.35\%$$



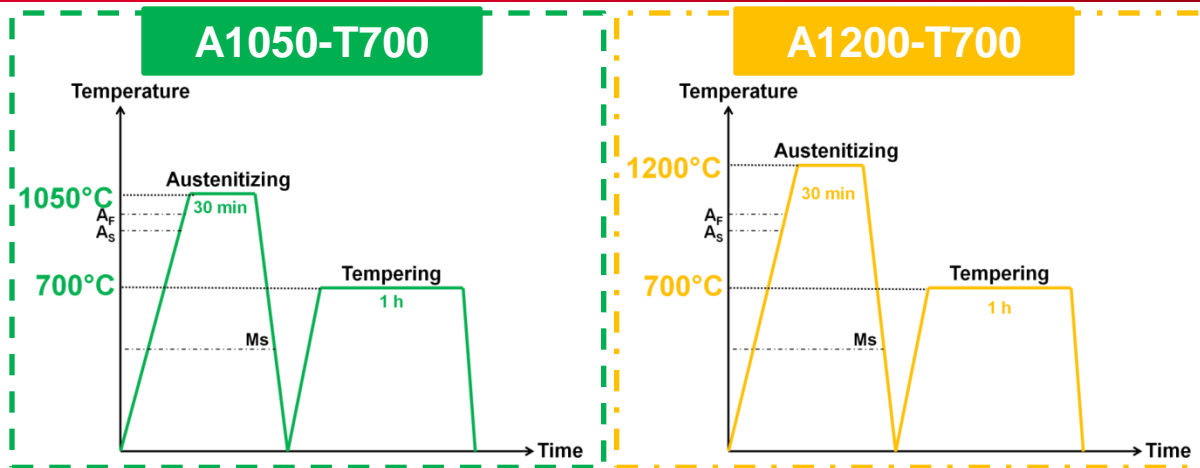
Higher dislocation density
(less recovery)



Higher f_v and finer D of intragranular precipitates

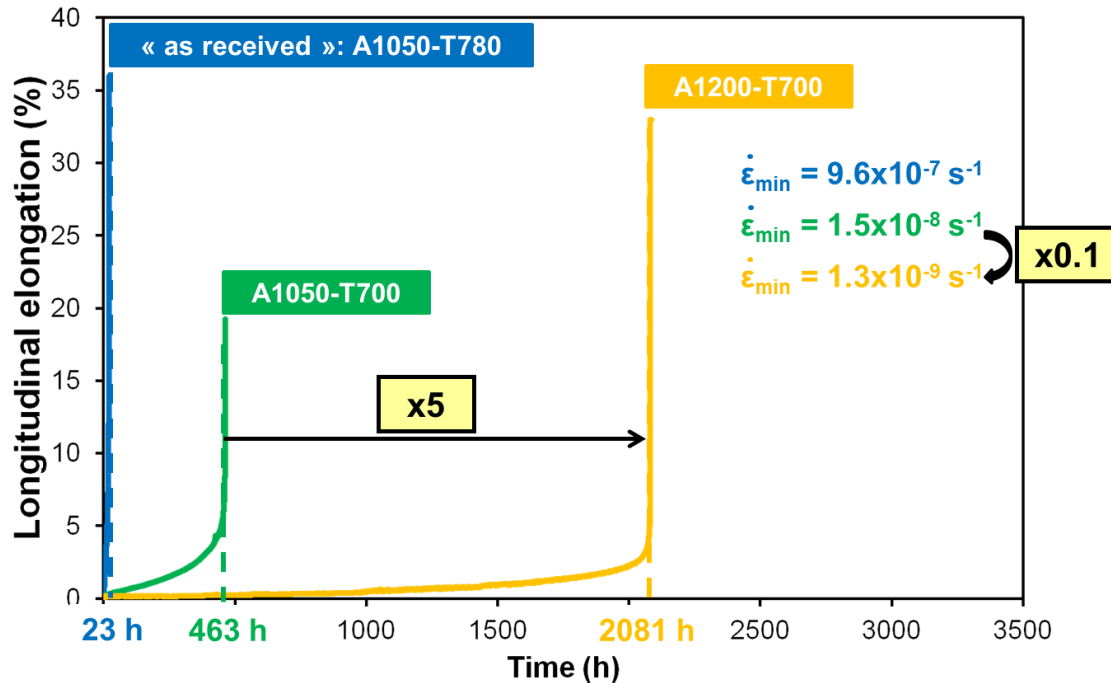
COMPARISON A1050-T700 VS A1200-T700

➔ AUSTENITIZING TEMPERATURE FROM 1050°C TO 1200°C



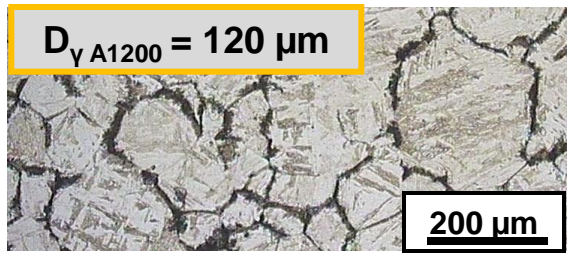
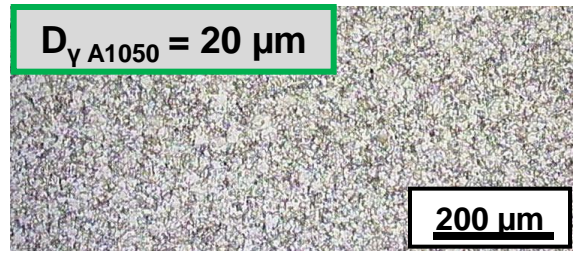
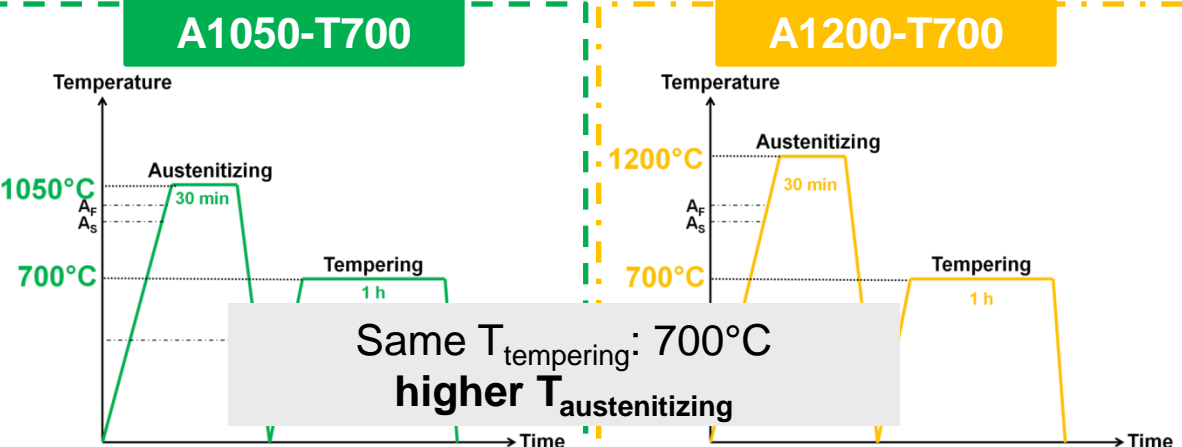
Same $T_{\text{tempering}}$: 700°C
higher $T_{\text{austenitizing}}$

■ Creep conditions: 140 MPa at 650°C



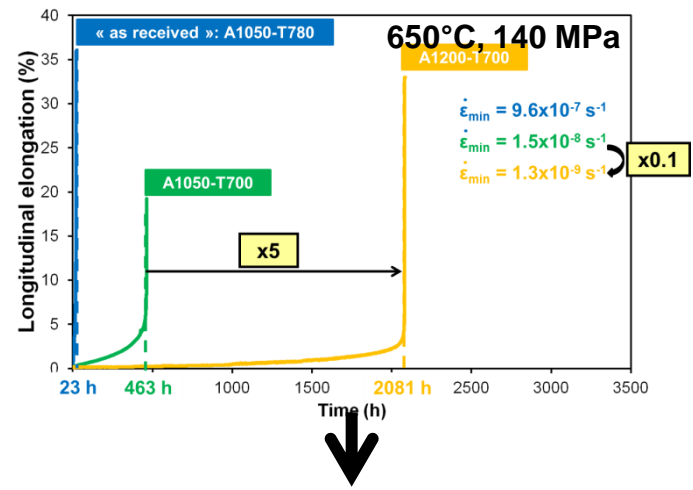
COMPARISON A1050-T700 VS A1200-T700

➔ AUSTENITIZING TEMPERATURE FROM 1050°C TO 1200°C



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

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



Higher prior γ grain size
(dissolution of small precipitates)

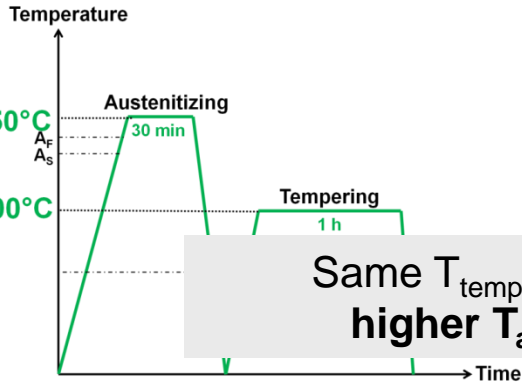


Higher martensite block size

COMPARISON A1050-T700 VS A1200-T700

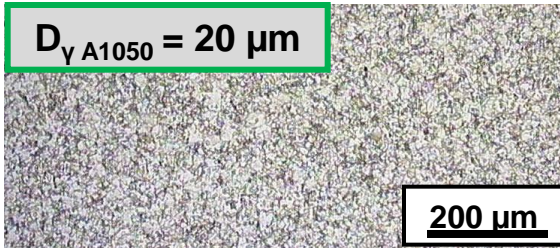
➔ AUSTENITIZING TEMPERATURE FROM 1050°C TO 1200°C

A1050-T700



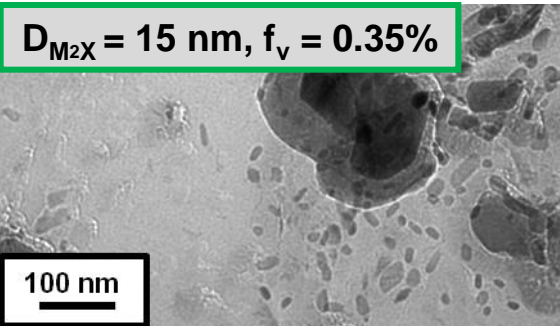
Same $T_{\text{tempering}}$: 700°C
higher $T_{\text{austenitizing}}$

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

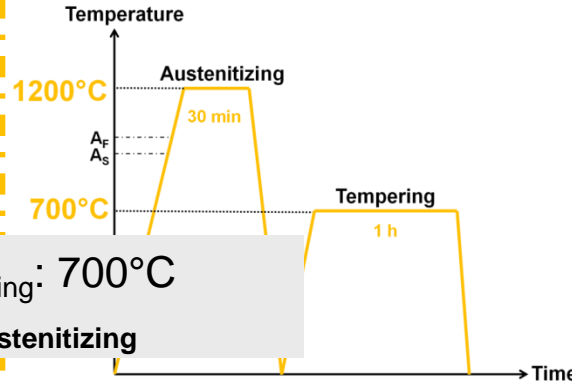


$W_{\text{blocks}} = 3.2 \mu\text{m}$

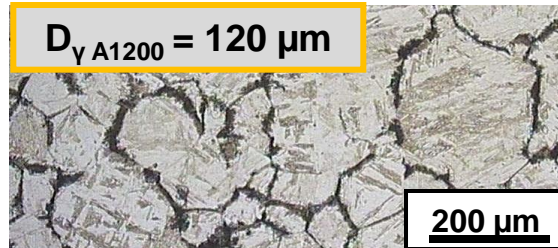
$D_{\text{M}_2\text{X}} = 15 \text{ nm}, f_v = 0.35\%$



A1200-T700

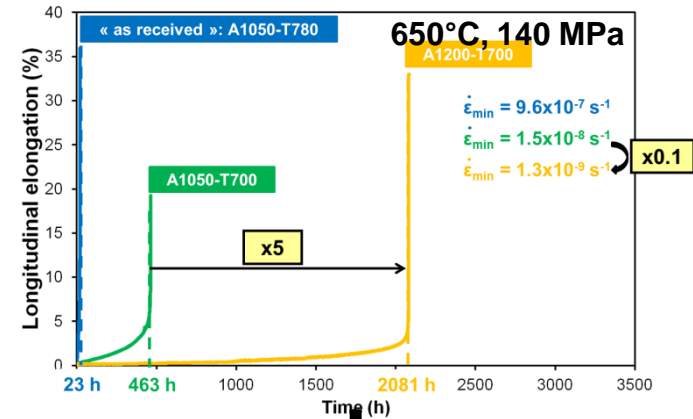
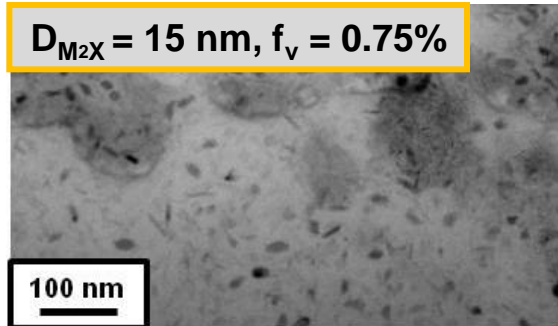


$D_{\gamma \text{ A1200}} = 120 \mu\text{m}$



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

$D_{\text{M}_2\text{X}} = 15 \text{ nm}, f_v = 0.75\%$



Higher prior γ grain size
(dissolution of small precipitates)



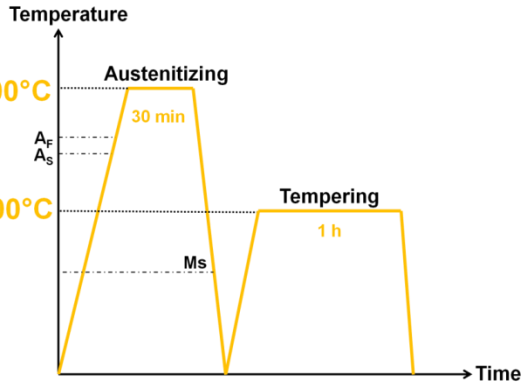
Higher martensite block size



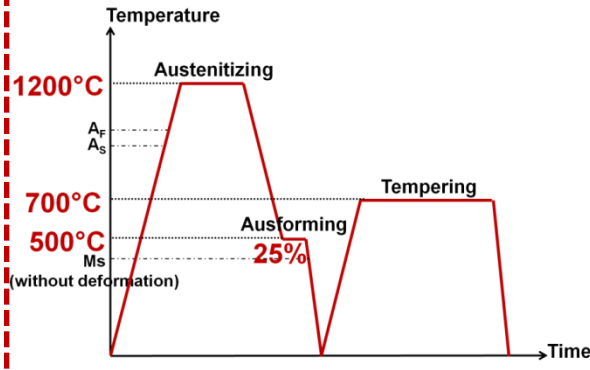
Higher f_v of intragranular precipitates
(more microalloying elements available for precipitation)

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

A1200-T700

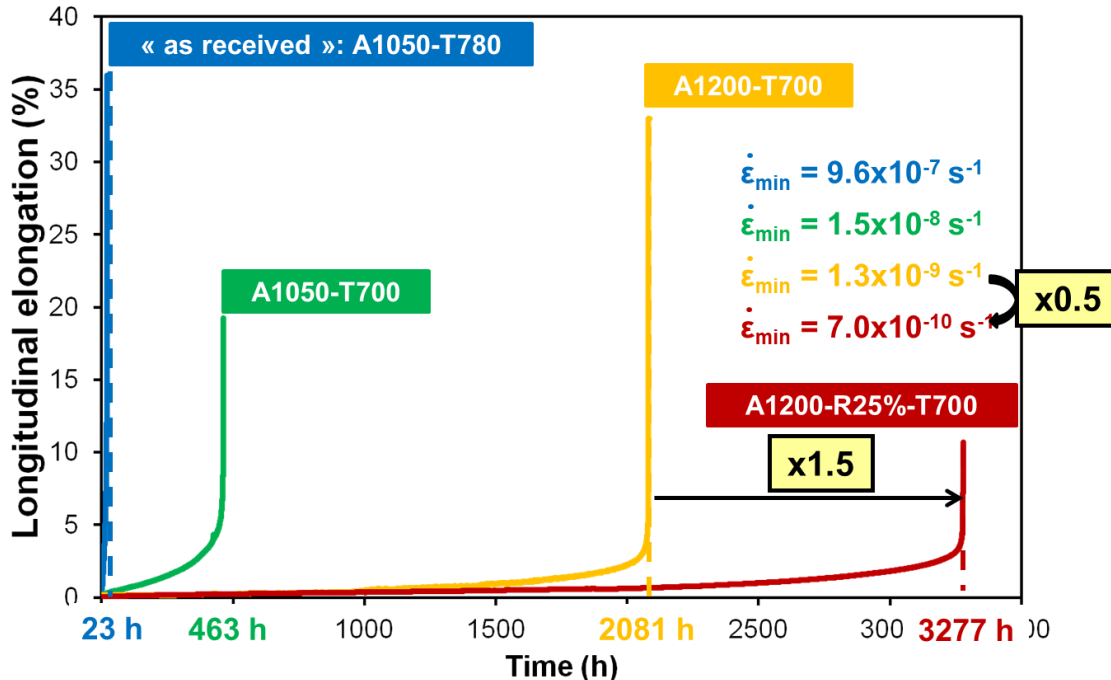


A1200-R25%-T700



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

Creep conditions: 140 MPa at 650°C

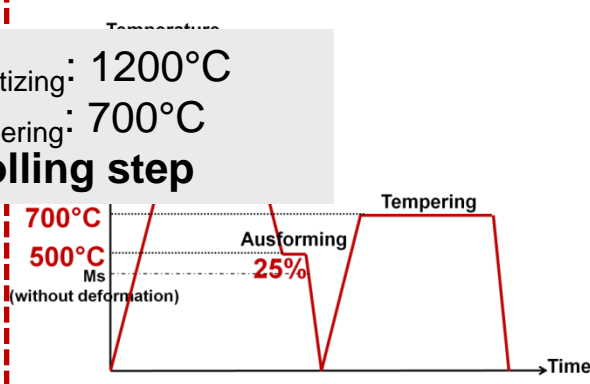
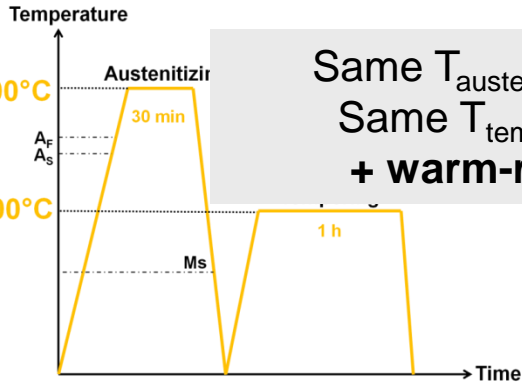


COMPARISON A1200-T700 VS A1200-R25%-T700 APPLYING WARM-ROLLING IN METASTABLE γ

A1200-T700

A1200-R25%-T700

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

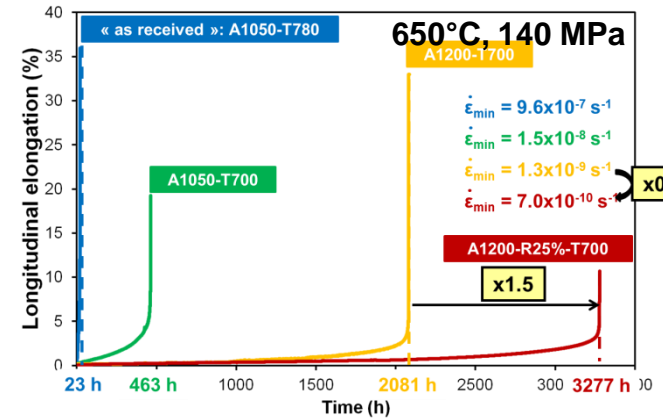


$W_{\text{laths}} \sim 400 \text{ nm}$

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

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

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



Finer lath width



Higher dislocation density
(inheritance from warm-rolling)

More than 30 treated conditions

4 states

Part 1

$T_{\text{tempering}}$

$T_{\text{austenitizing}}$

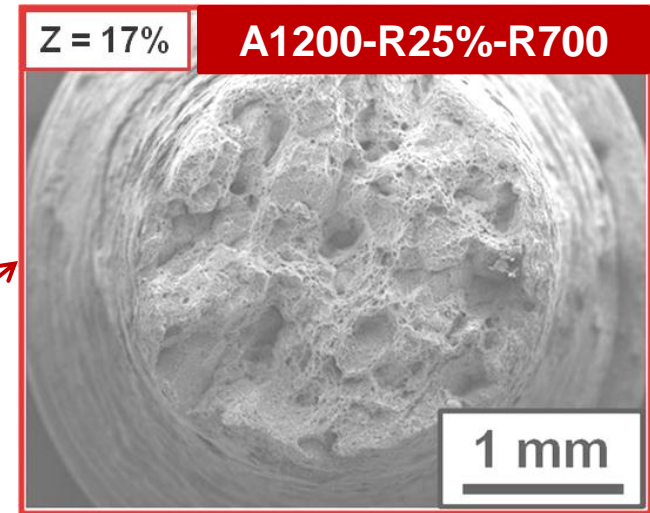
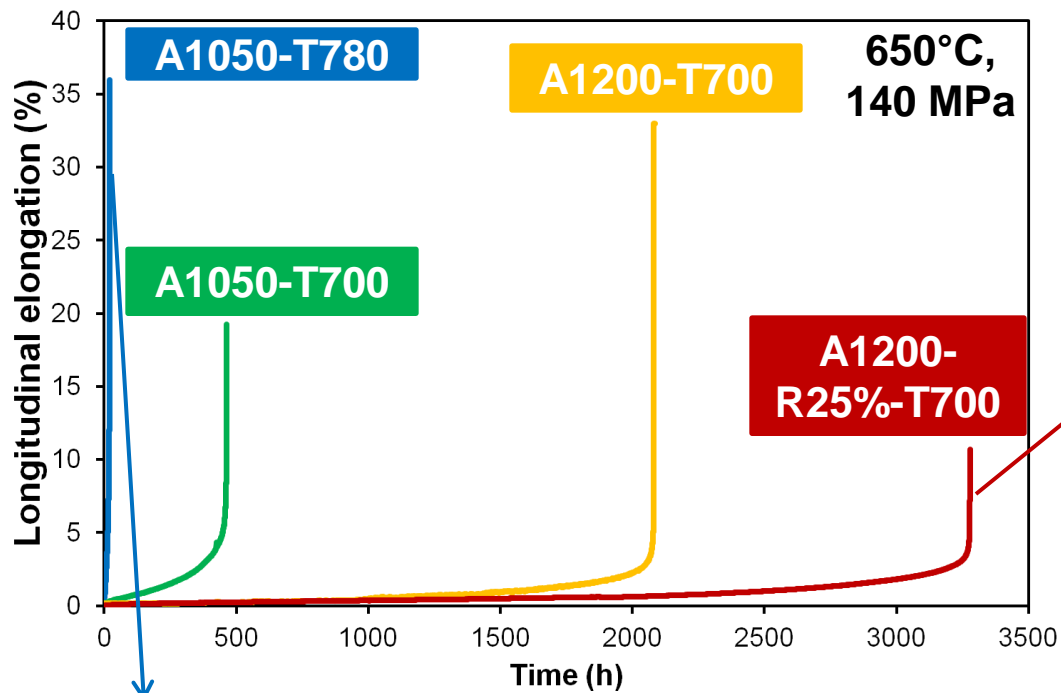
Warm-rolling

Time to fracture
Minimal creep rate

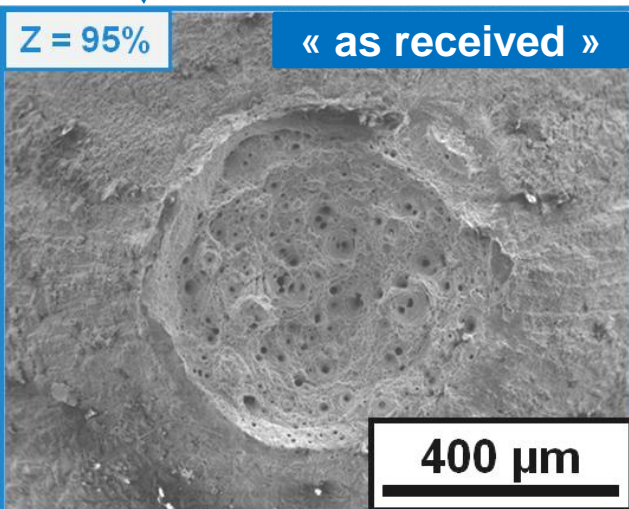
Microstructure

Part 2

Creep fracture mode



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

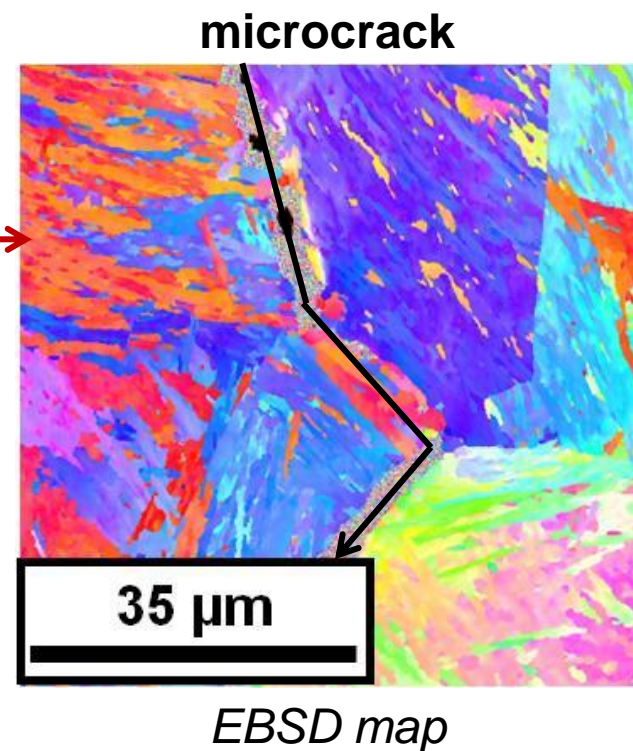
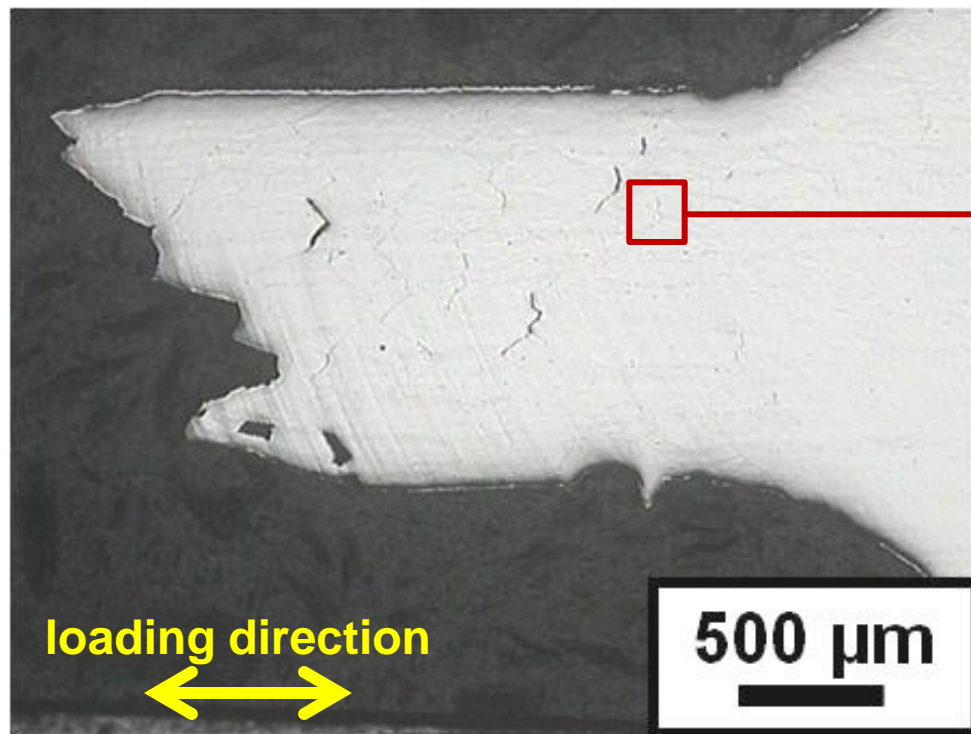


Significant tertiary creep stage,
high reduction of area at failure,
Ductile fracture.

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

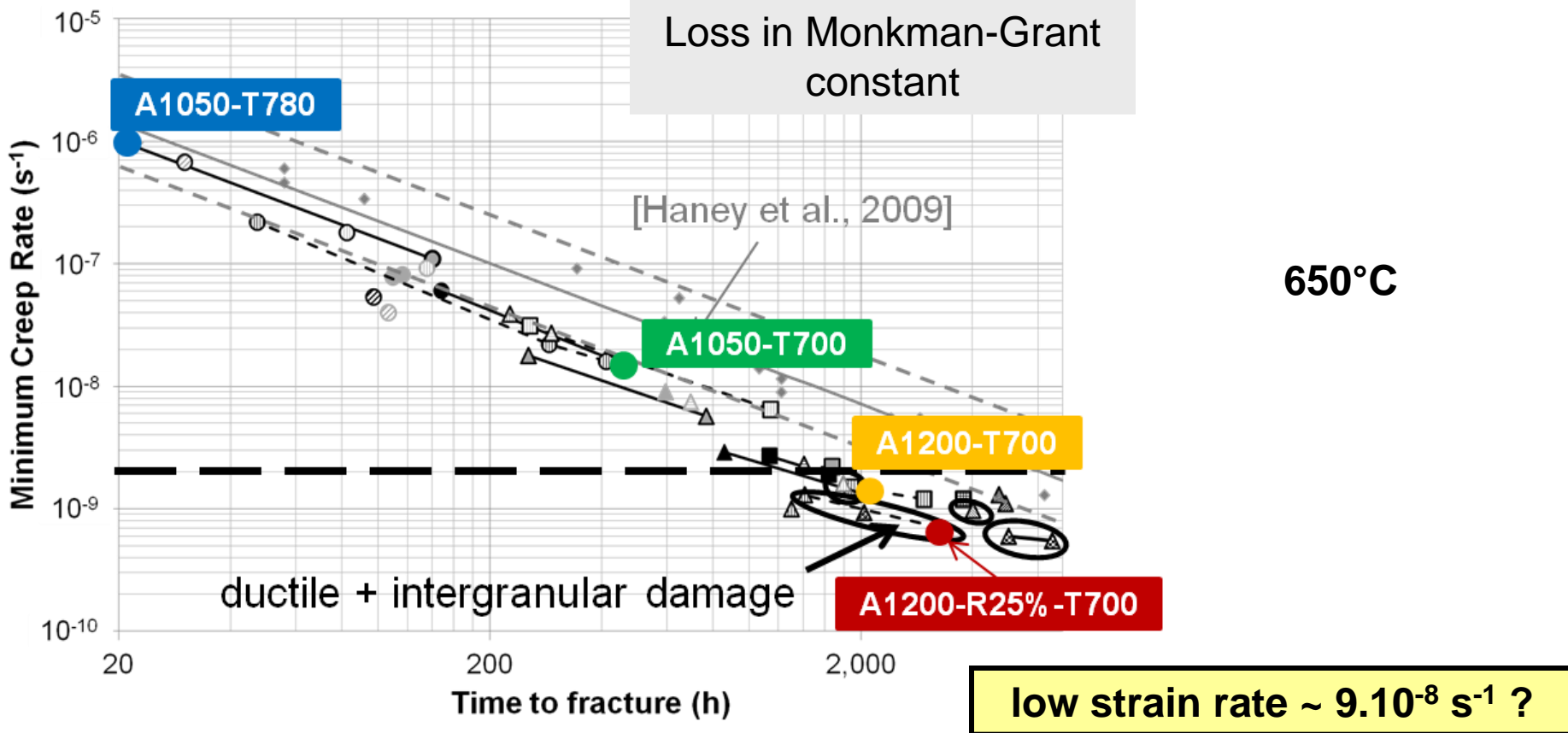
- Confirmation by EBSD observations: damage at prior austenite grain boundaries,

cross section observation



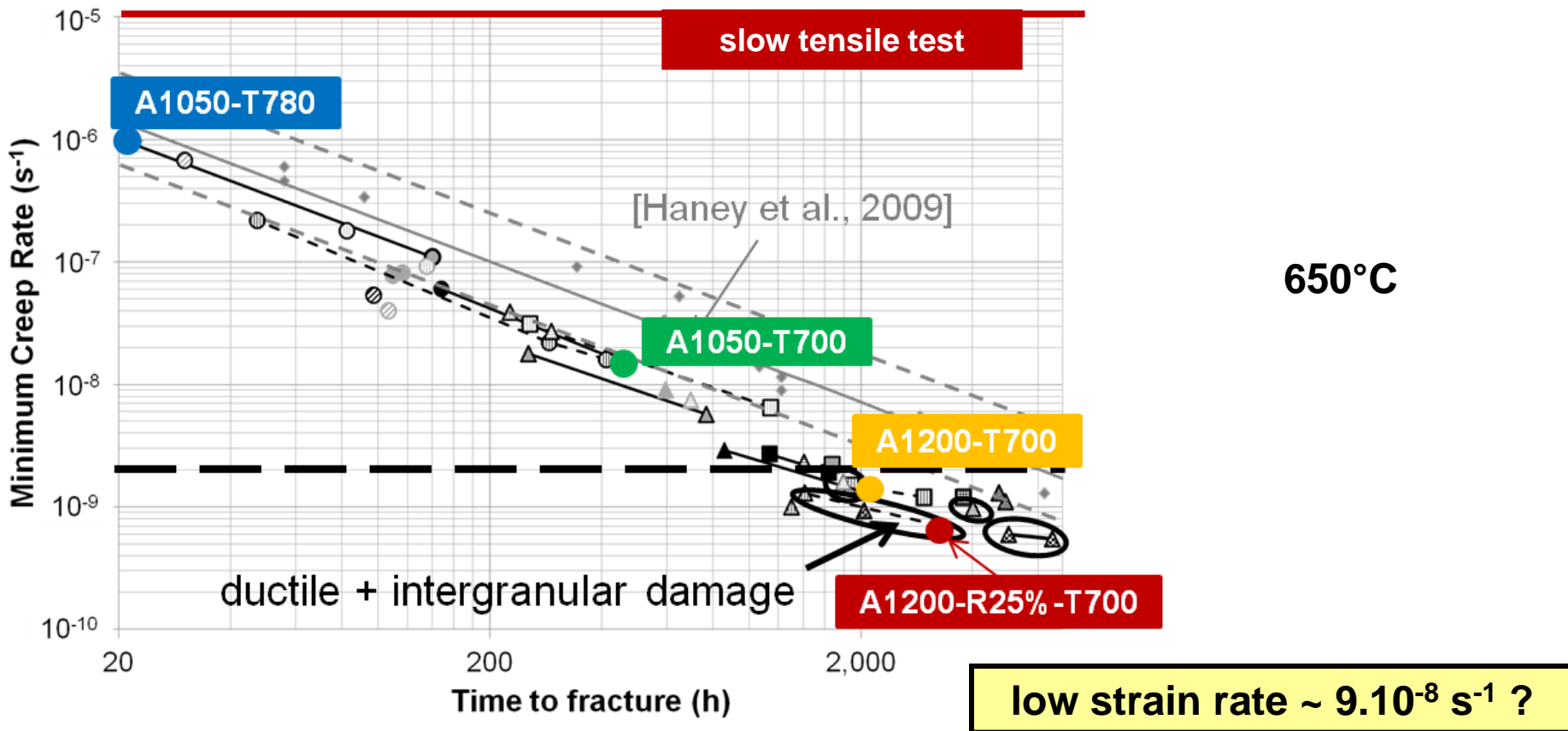
**Ductile + intergranular creep fracture mode observed for:
A1200-R(0-25%)-T(700-750°C) states (650°C, 140 MPa)**

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?

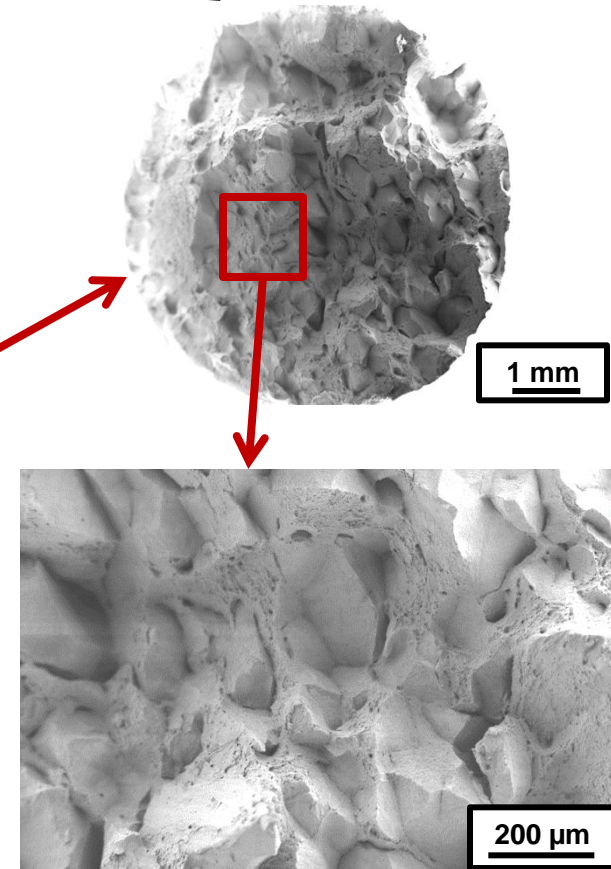
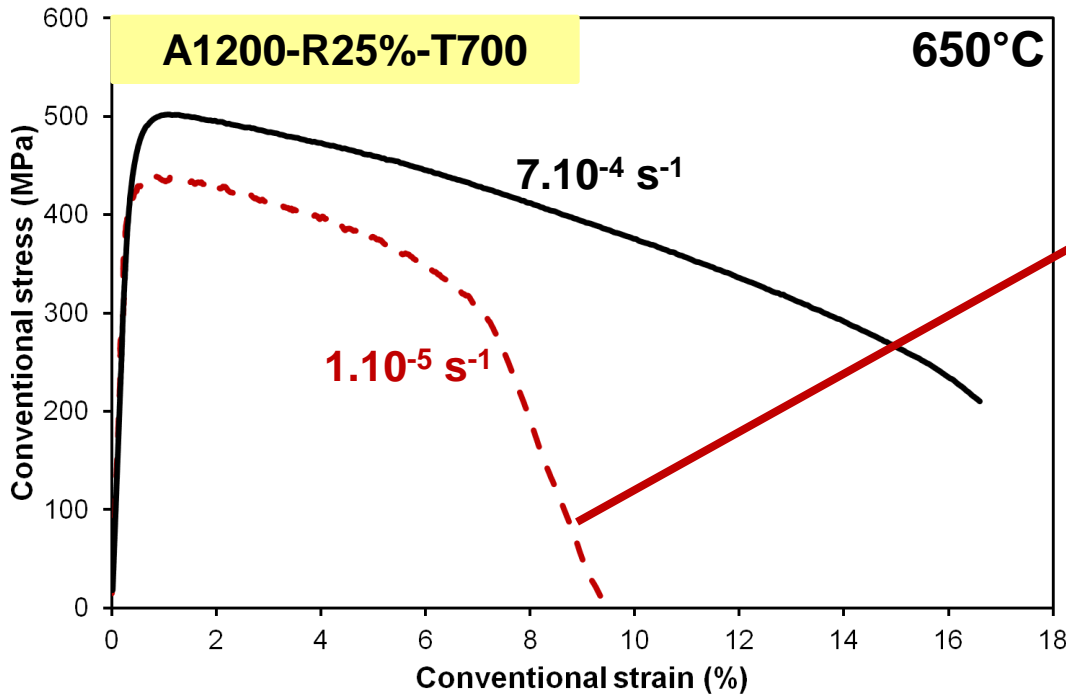


**Ductile + intergranular creep fracture mode observed for:
A1200-R(0-25%)-T(700-750°C) states (650°C, 140 MPa)**

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



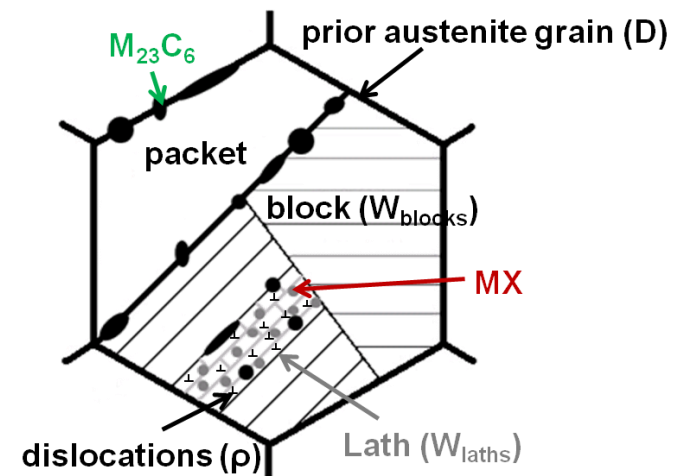
Due to some specific martensitic microstructures
(high D_Y , effects of impurities and/or high precipitates density at prior- γ grain boundaries, critical intragranular hardening...) ???

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 :

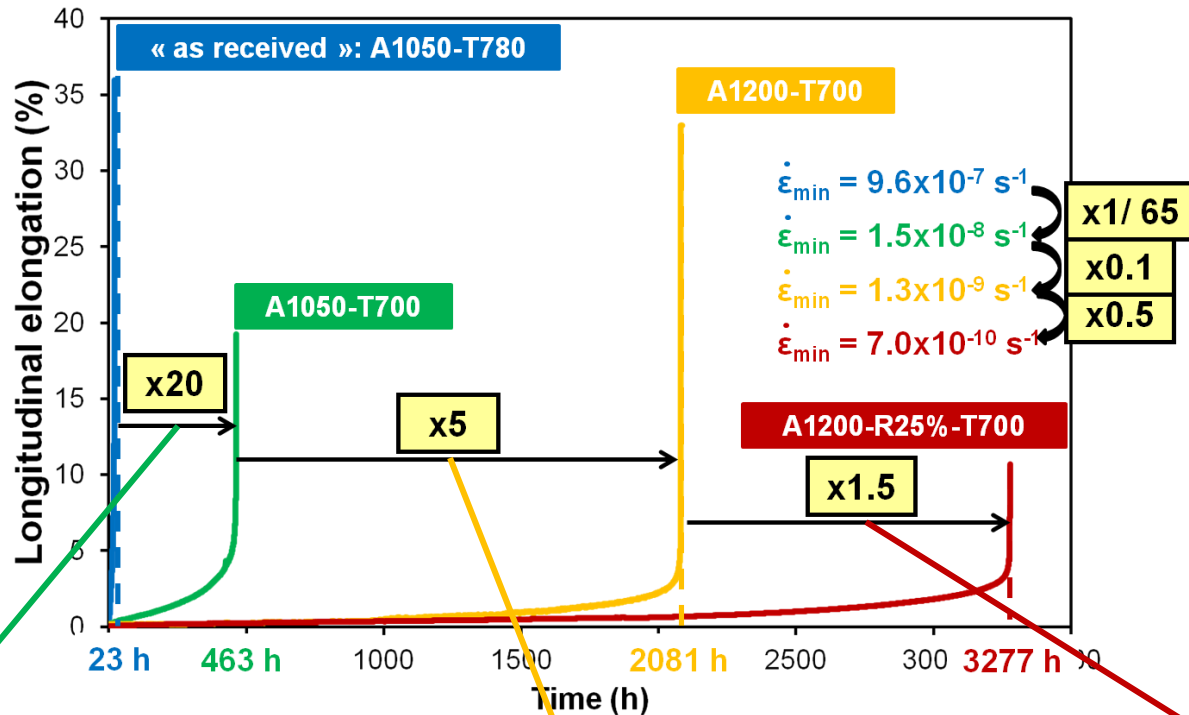
<https://pastel.archives-ouvertes.fr/tel-01141826>

... but in French !

Commissariat à l'énergie atomique et aux énergies alternatives
Centre de Saclay | 91191 Gif-sur-Yvette Cedex
T. +33 (0)1 69 08 41 42 | F. +33 (0)1 69 08 71 30

DEN
DMN
SRMA/LA2M

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



↘ $T_{\text{tempering}}$

↗ $T_{\text{austenitizing}}$

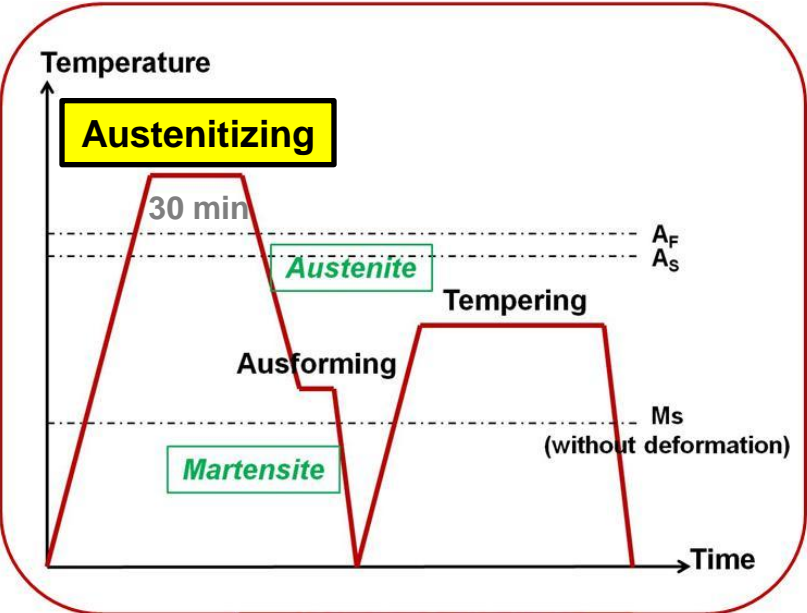
Warm-rolling

↗ ρ ↗ $f_v \text{ intra}$
1st order parameters

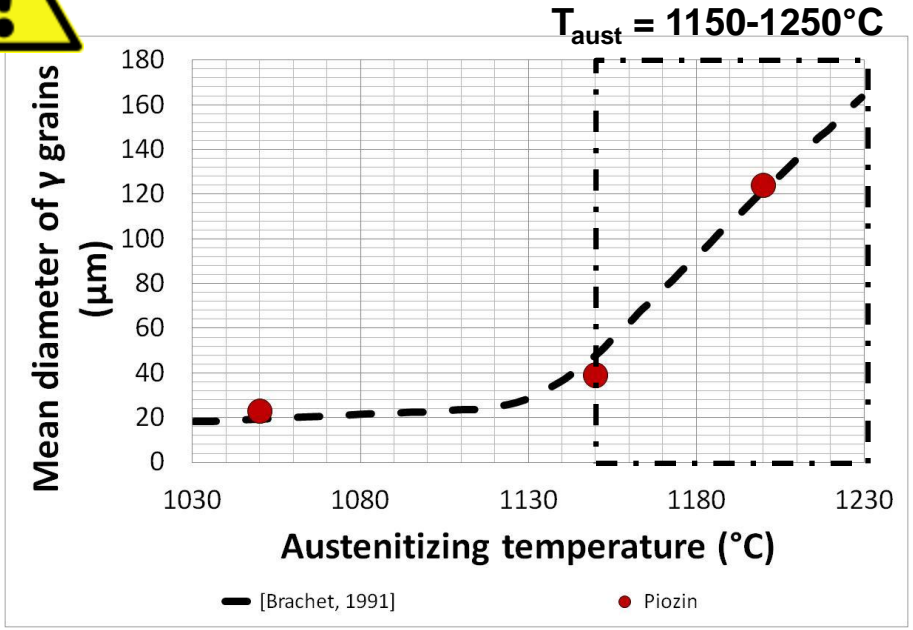
↗ D_V, W_{blocks}

negligeable effect

AUSTENITIZING STEP



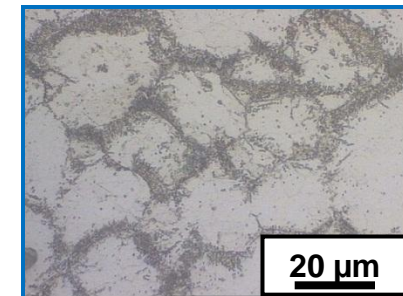
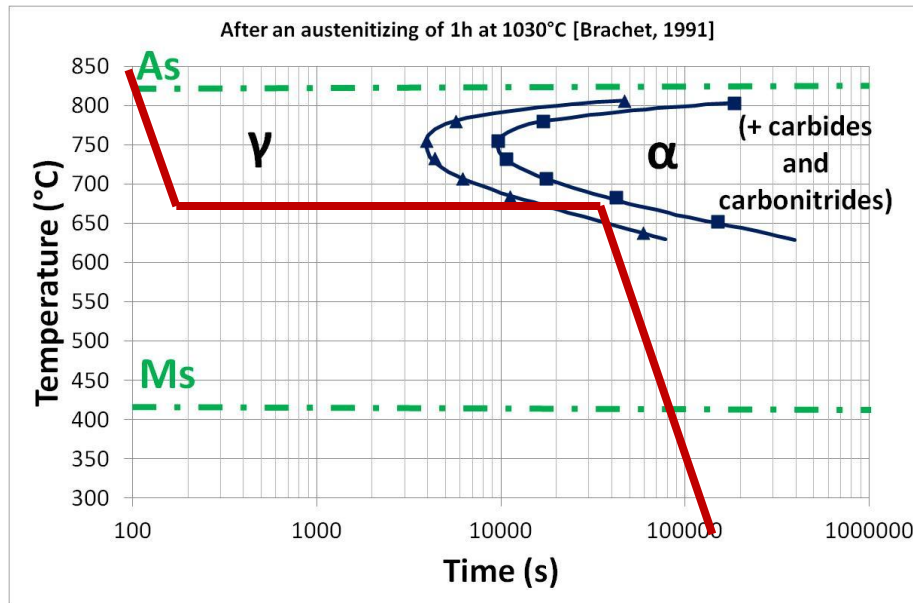
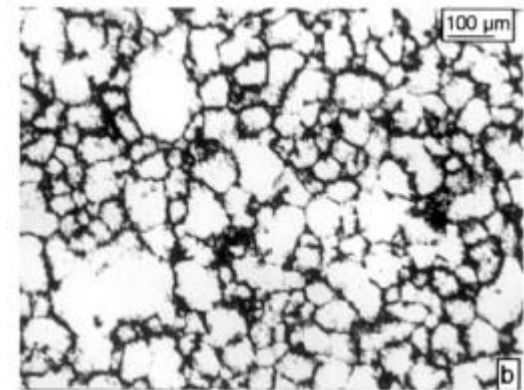
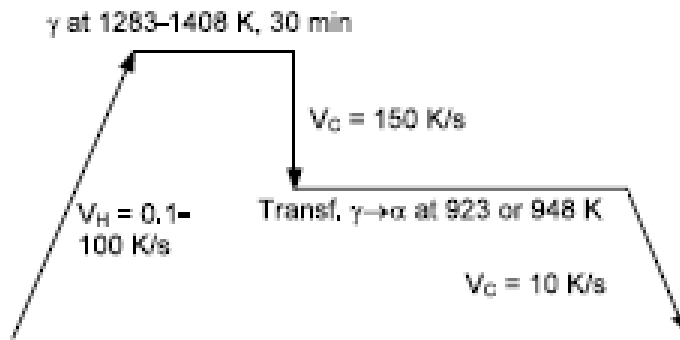
thermomechanical treated



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

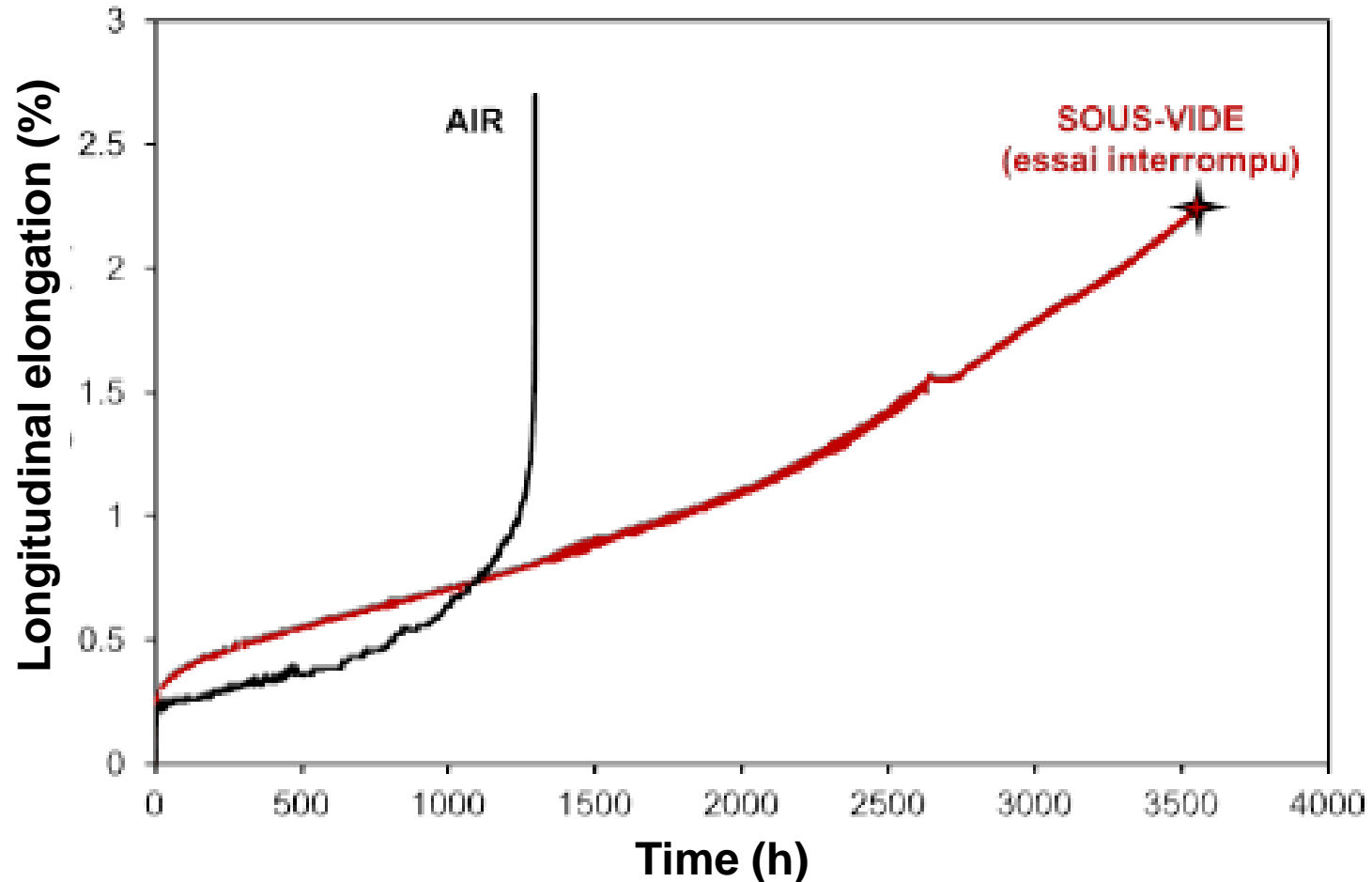
Experimental procedure to measure prior austenite grain size :

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



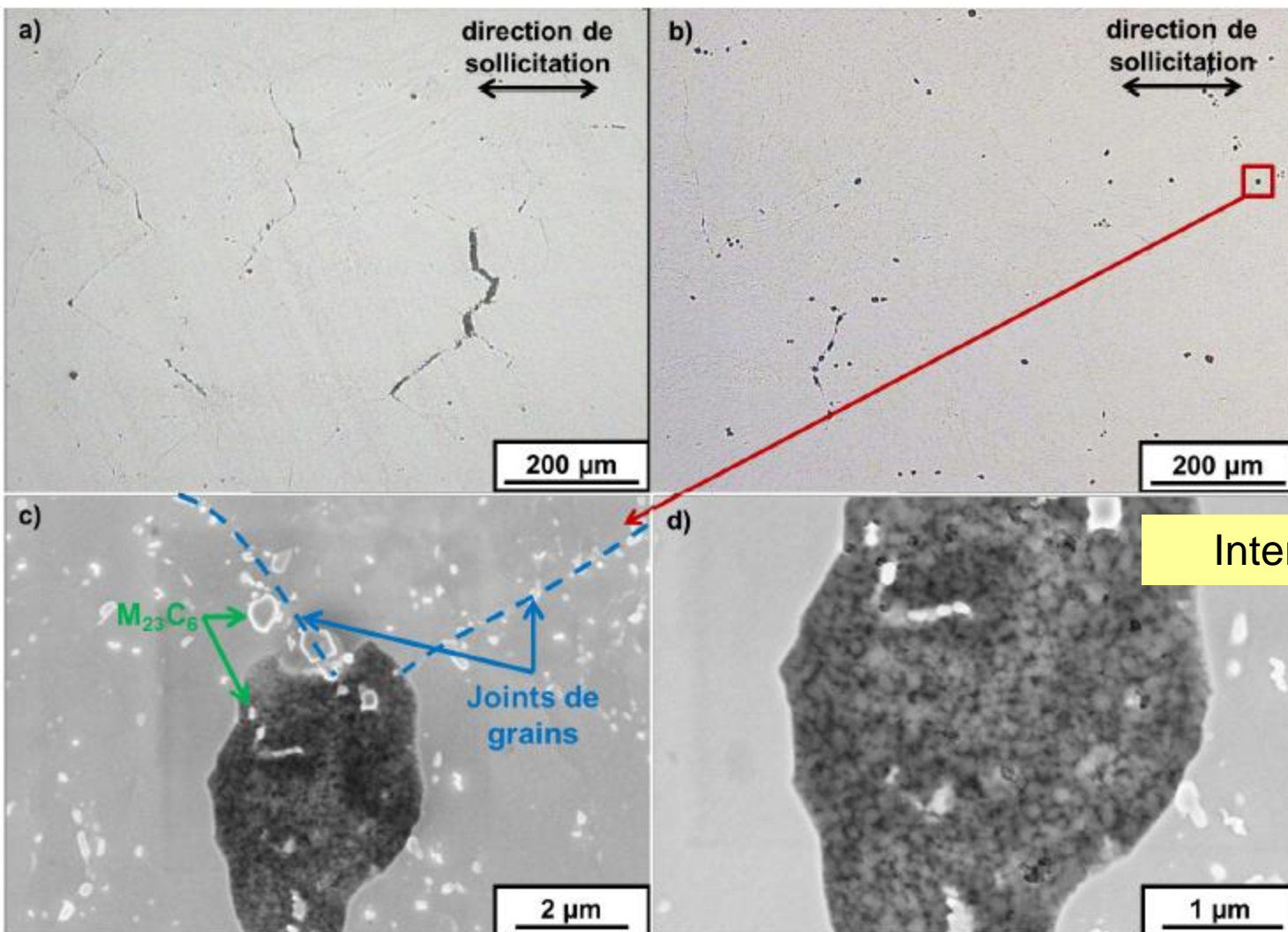
INFLUENCE OF ENVIRONNEMENT

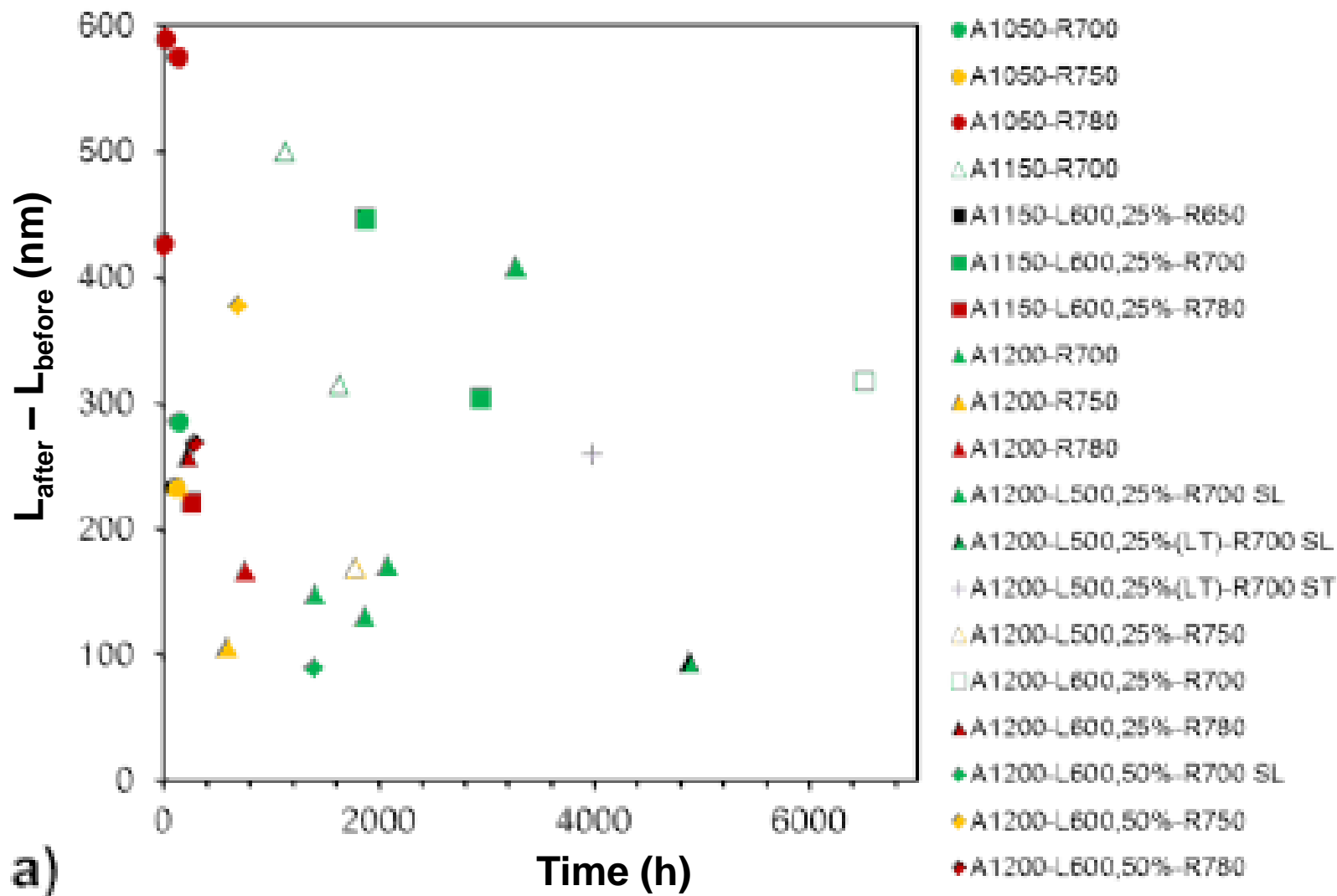
A1200-R500,25%-T700



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

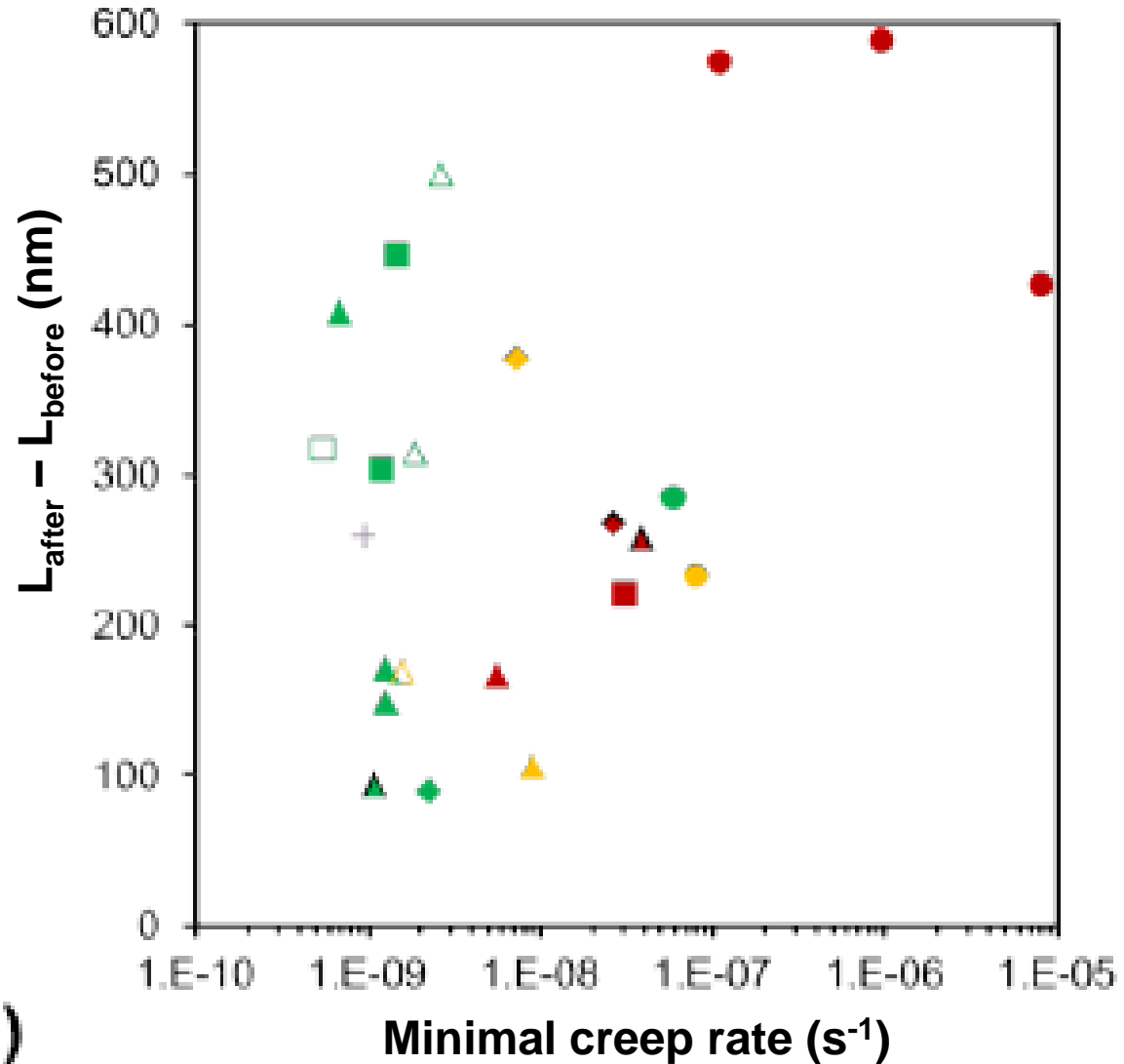
A1200-R500,25%-T700 (vaccum)

cross section observations



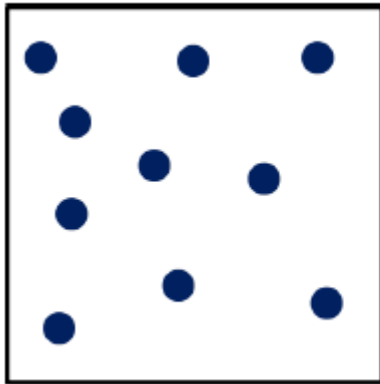
MICROSTRUCTURE AFTER CREEP TESTS

- 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

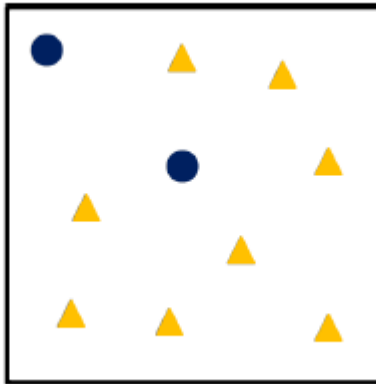


A1200-R600,25%-T700

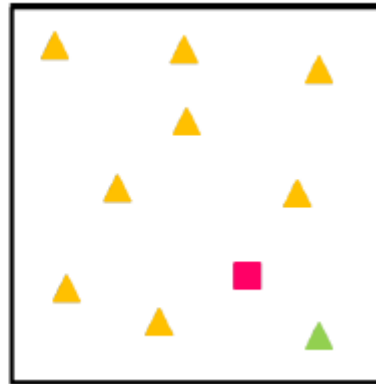
Creep test :
140 MPa, 650°C
 $t_r = 6523$ h



non sollicité



vieilli thermiquement à
650°C pendant 6523
heures



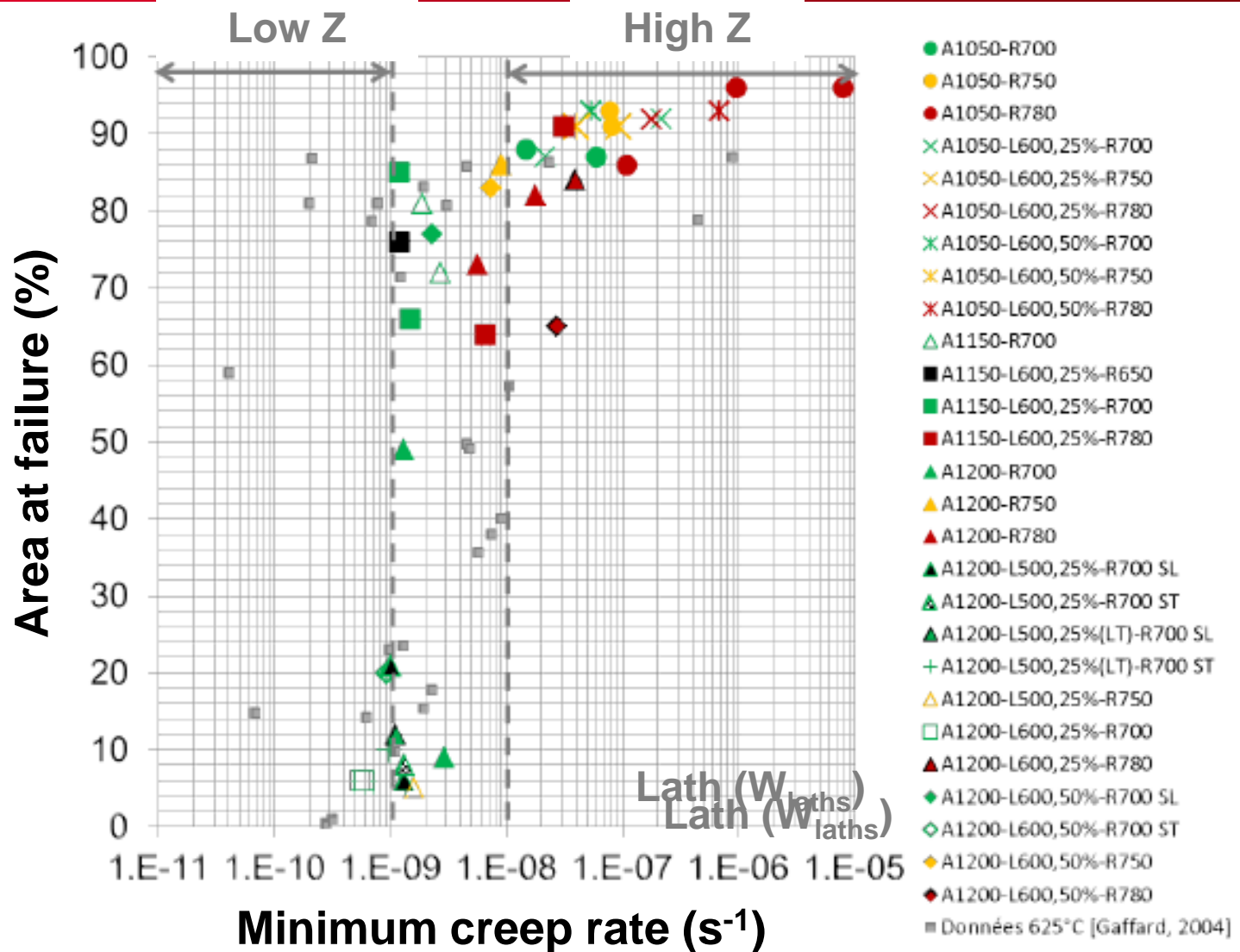
vieilli thermiquement à
650°C pendant 6523
heures + sollicité en
fluage à 140 MPa

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

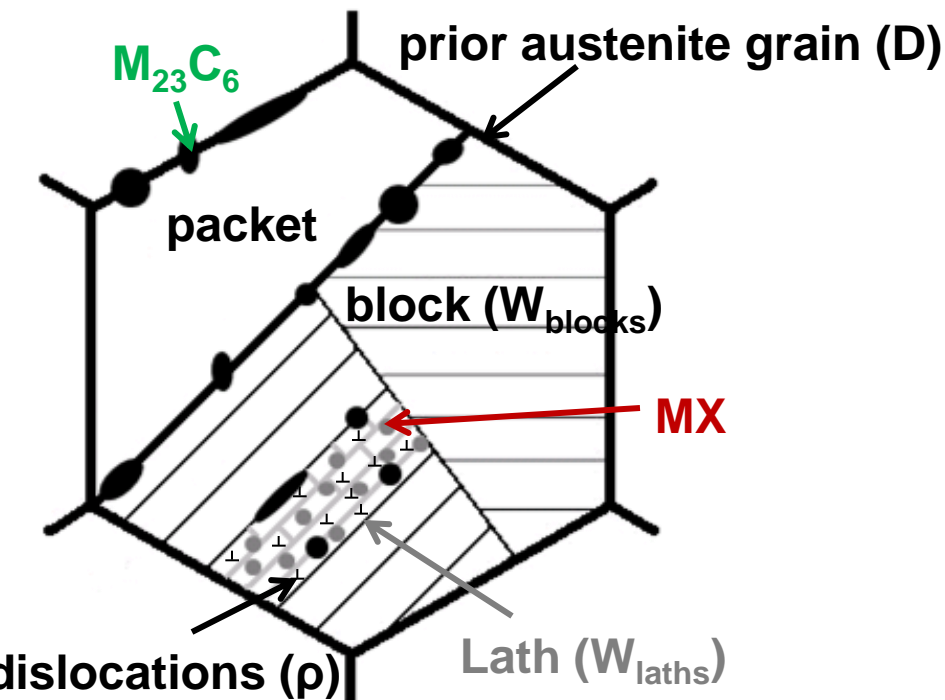
Change in chemical composition
of MX precipitates

	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

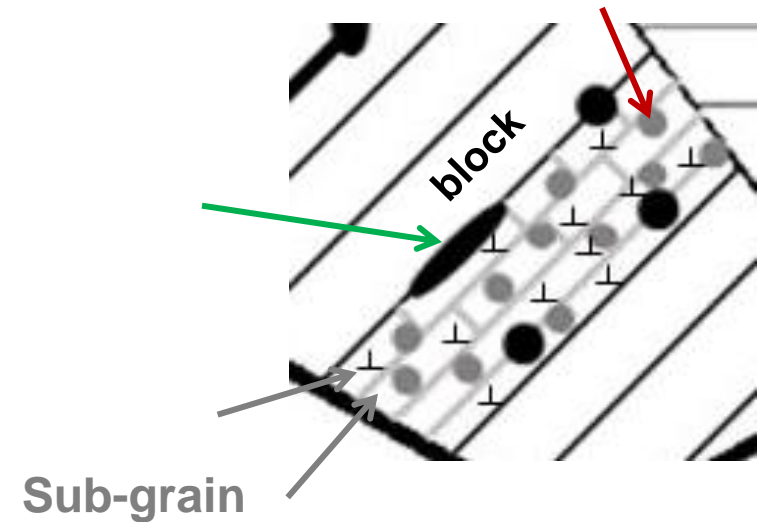
MINIMUM CREEP RATE VALUE LIMIT !



- 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**