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

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DE LA RECHERCHE À L'INDUSTRIE

CENTRE DES MATERIAUX
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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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(**MINES ParisTech – Centre des Matériaux**, Evry)

CREEP 2015 – TOULOUSE

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

Considered:

→ nuclear fusion reactors:

- tokamak blankets.

→ SFR (Generation IV) :

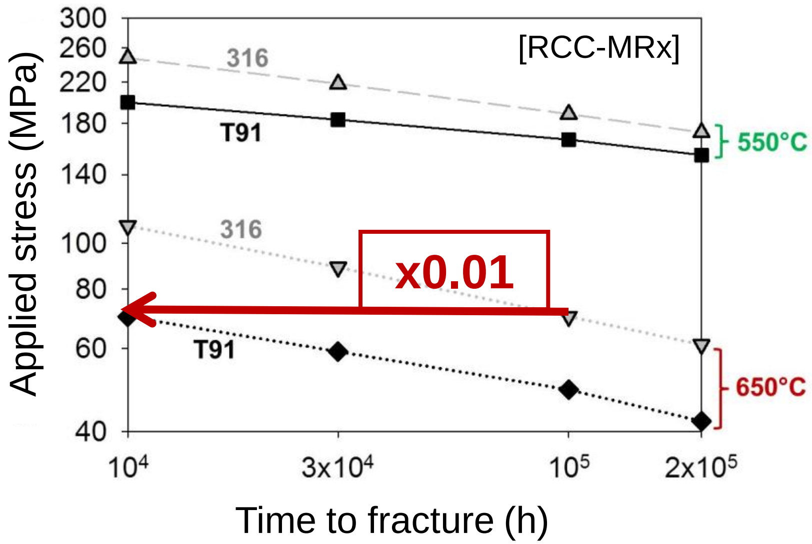
- steam generator and **secondary piping.**

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

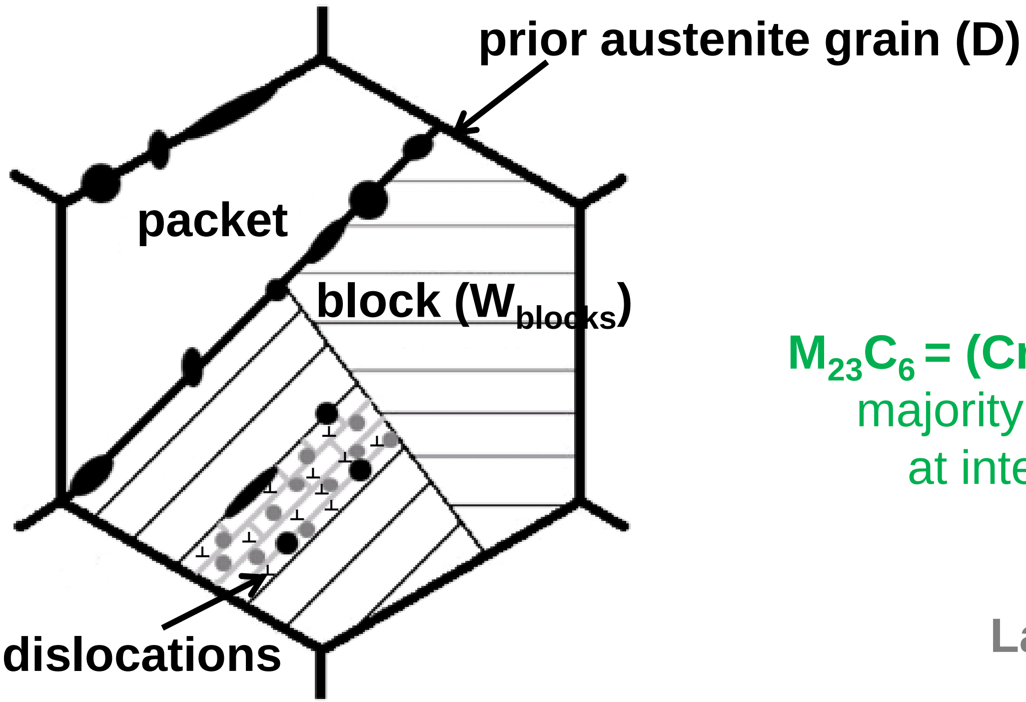
T91 = « standard » thermal treatment



Need of **increasing strength**

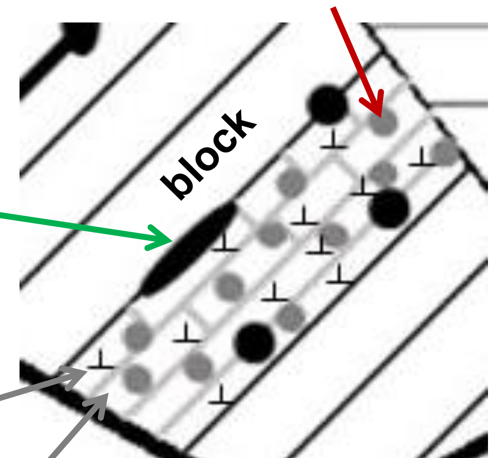
COMPLEX MICROSTRUCTURE OF GRADE 91 STEEL

- **Simplified chemistry** (wt %)**: Fe-9Cr-1Mo-0,1C-(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

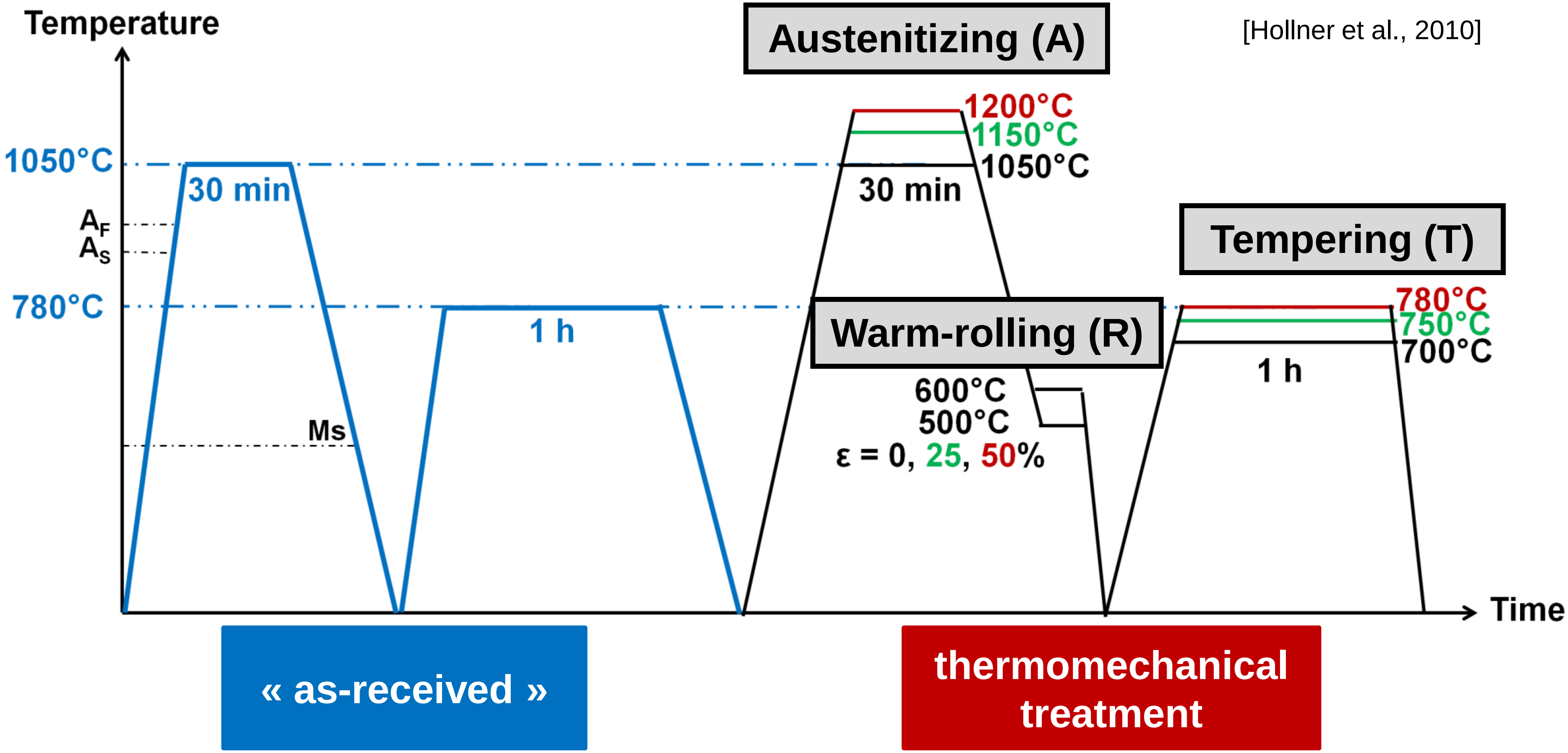


Lath (W_{laths})
Sub-grain

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

$T_{tempering}$ | $T_{austenitizing}$ | Warm-rolling

↓

Time to fracture
Minimal creep rate

↓

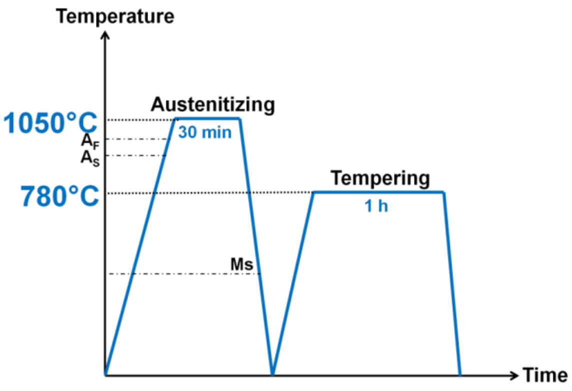
Microstructure

Part 2

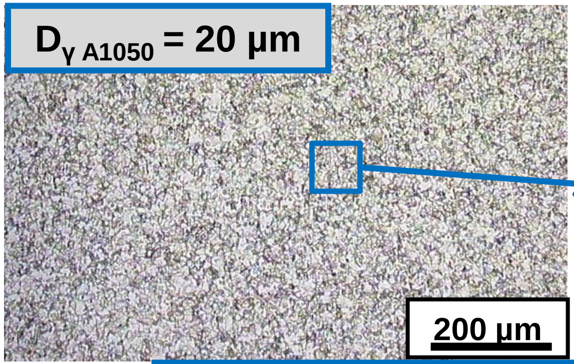
Creep fracture mode

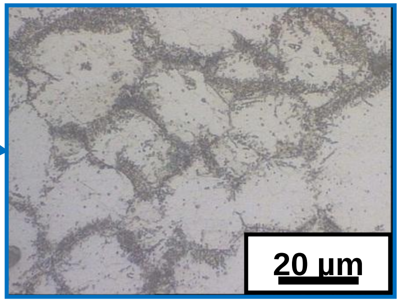
“AS RECEIVED”: A1050-T780

« as received »: A1050-T780



$D_{\gamma\ A1050}$ = 20 µm





Fine prior austenite grain size

"AS RECEIVED": A1050-T780

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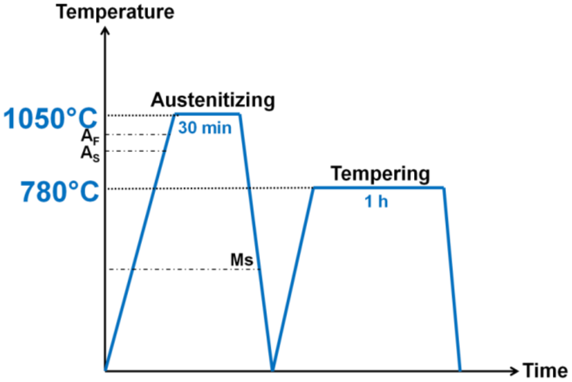

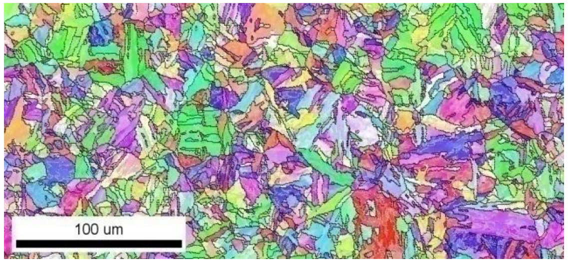

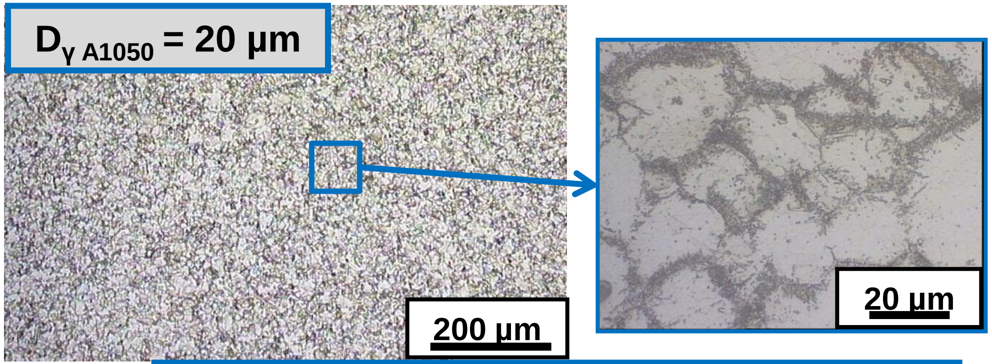

Fine prior austenite grain size

W_{blocks} = 3.2 µm (*)
W_{laths} ~ 400 nm (**)

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

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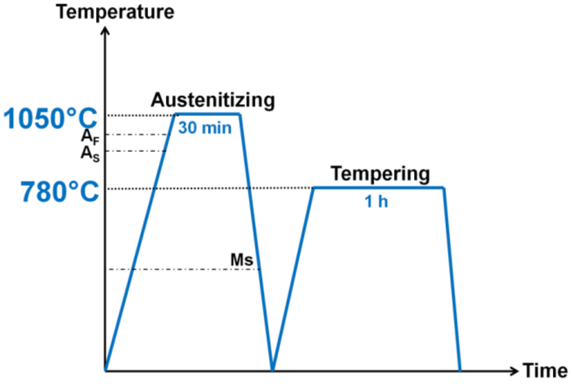
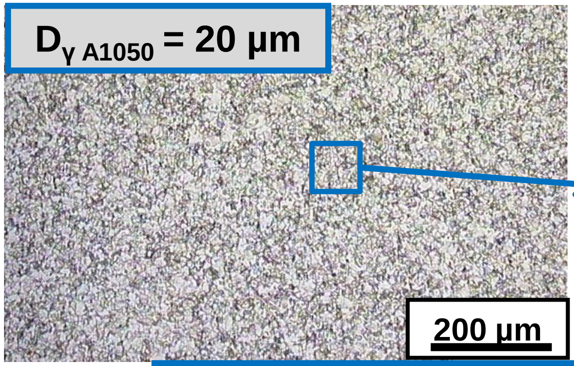
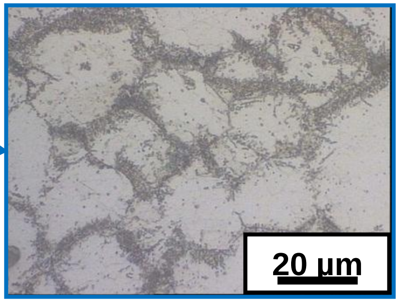
« as received »: A1050-T780



$D_{\gamma\ A1050}$ = 20 µm





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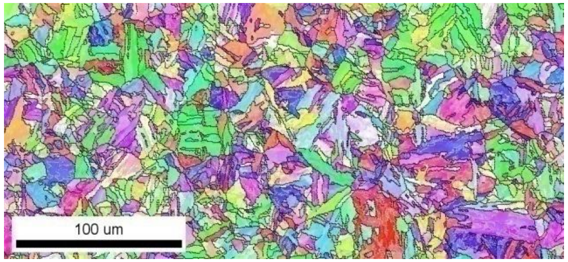

W_{blocks} = 3.2 µm (*)
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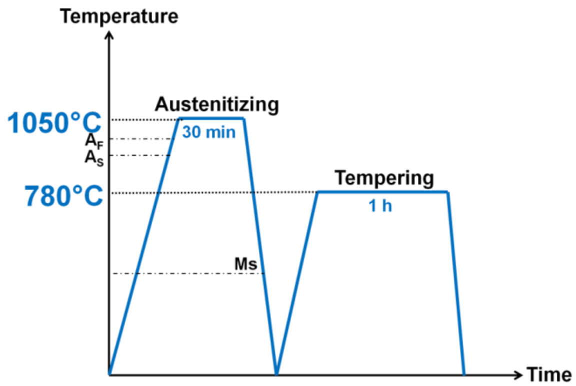
(*) SEM-EBSD,
(**) TEM on thin foils.

ρ = (0.62 ± 0.05)x10^{14} m^{-2} (***)

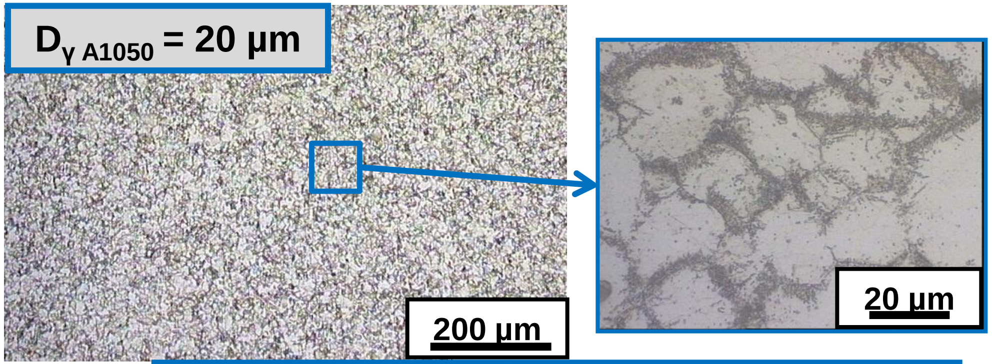
(***) neutron diffraction *(FWMH martensite peaks)*.

“AS RECEIVED”: A1050-T780

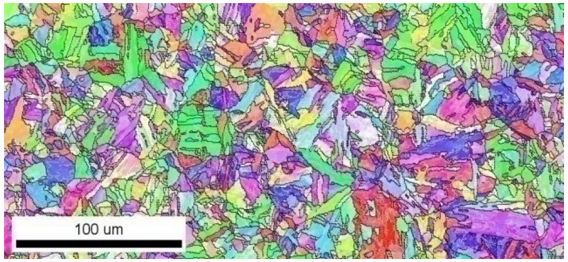
« as received »: A1050-T780



$D_{\gamma\ A1050}$ = 20 µm



Fine prior austenite grain size



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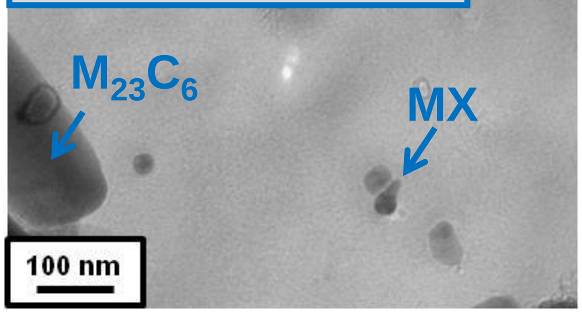
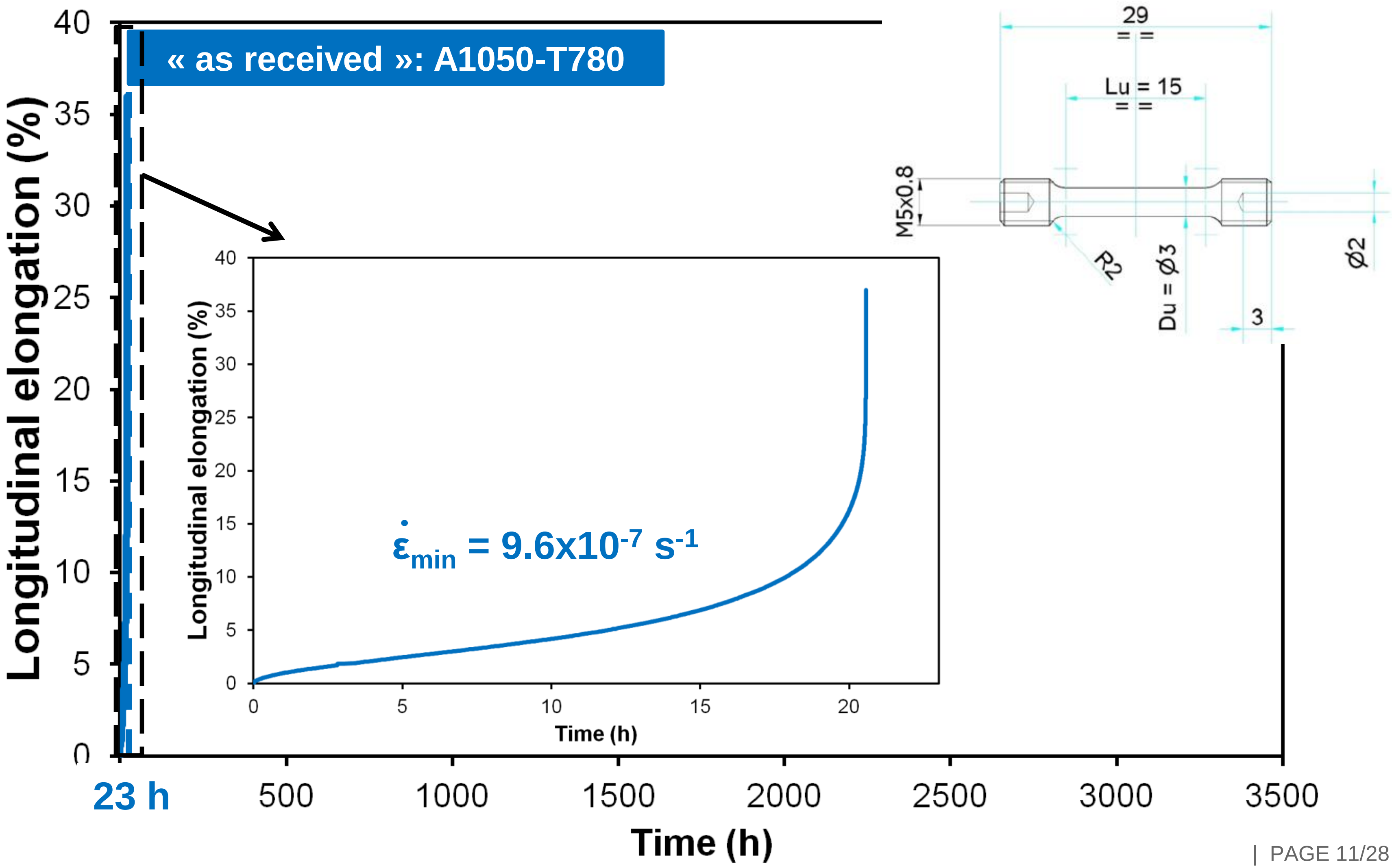
D_{MX} = 32 nm, f_v = 0.10%



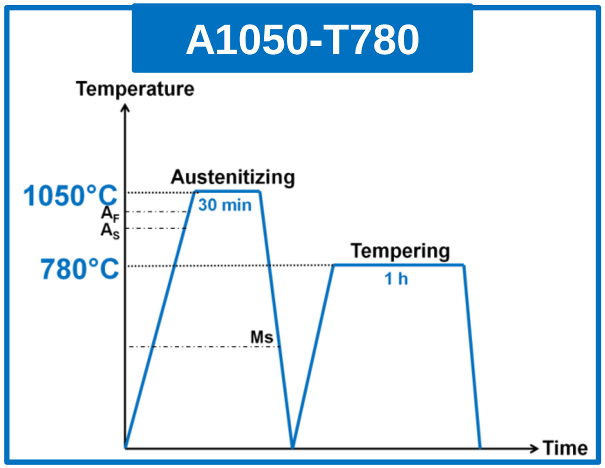
Coarse $M_{23}C_6$ precipitates at the different interfaces and few intragranular MX precipitates

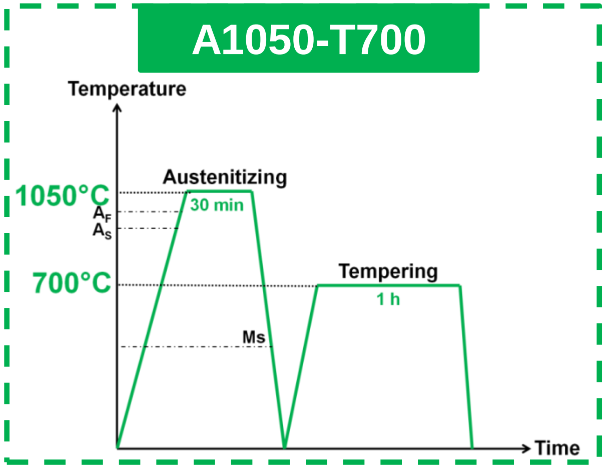
"AS RECEIVED": A1050-T780

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



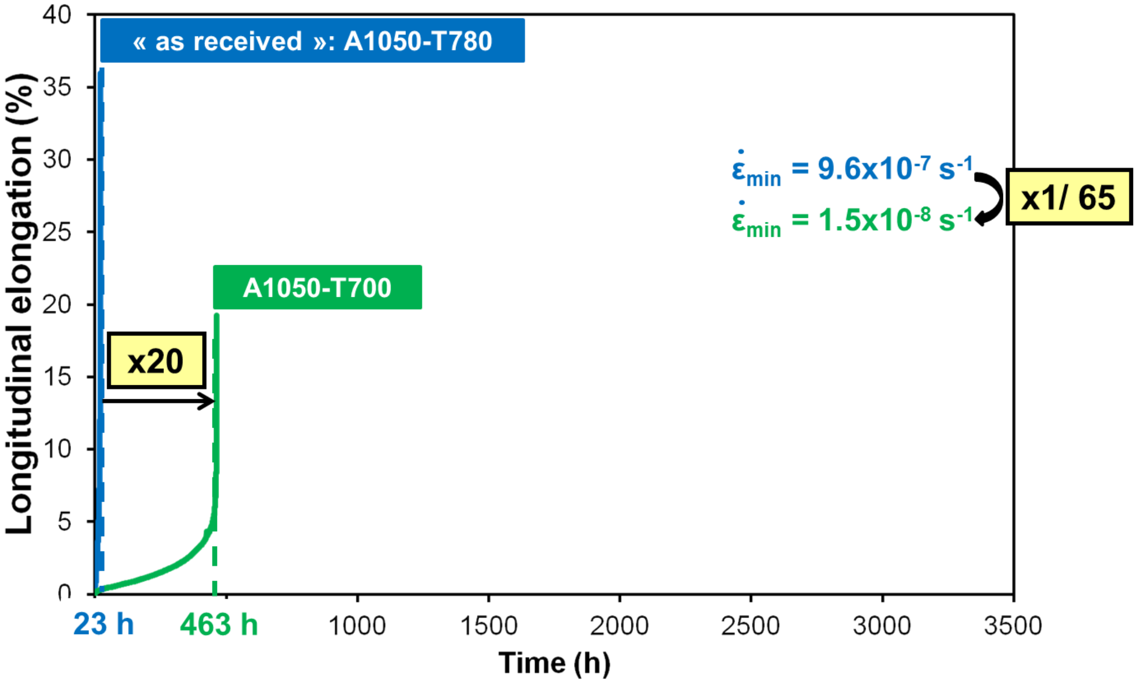
COMPARISON "AR" VS A1050-T700
↘ TEMPERING TEMPERATURE FROM 780°C TO 700°C





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

- **Creep conditions**: 140 MPa at 650°C



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

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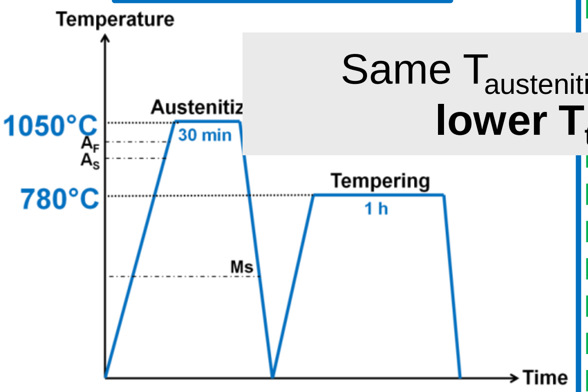
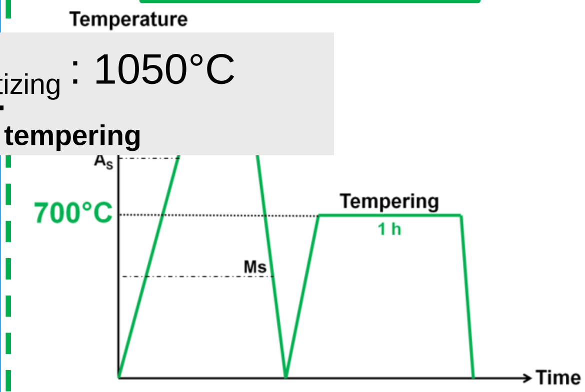
A1050-T780

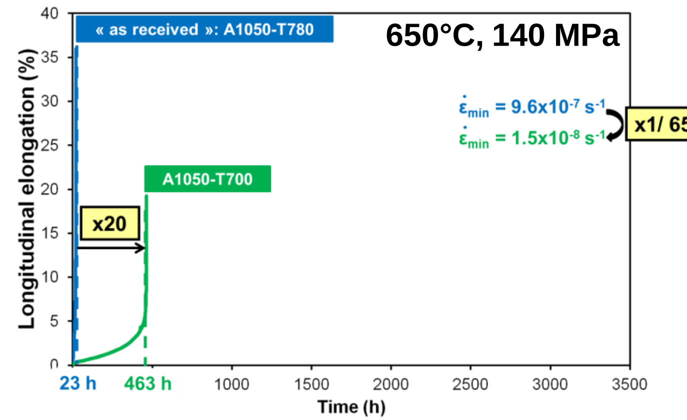


A1050-T700



Same $T_{austenitizing}$: 1050°C

lower $T_{tempering}$

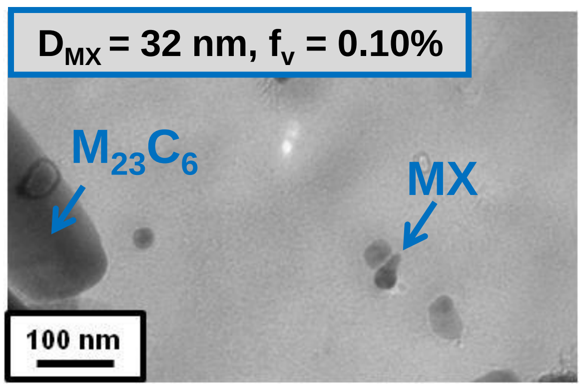


ρ = (0.62 ± 0.05)x10^{14} m^{-2}

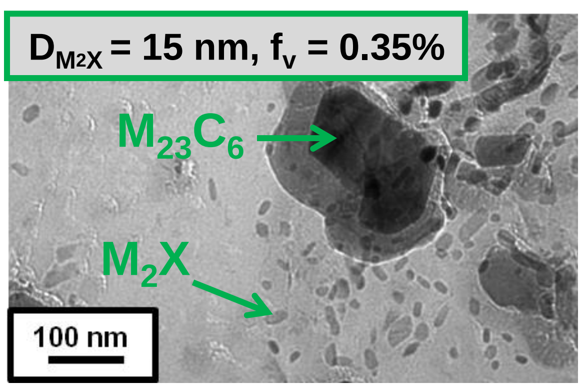
ρ = (4.7 ± 0.5)x10^{14} m^{-2}

Higher dislocation density
(less recovery)

D_{MX} = 32 nm, f_v = 0.10%

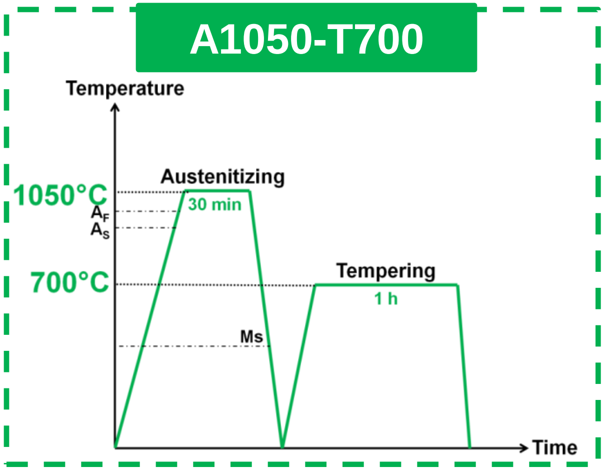


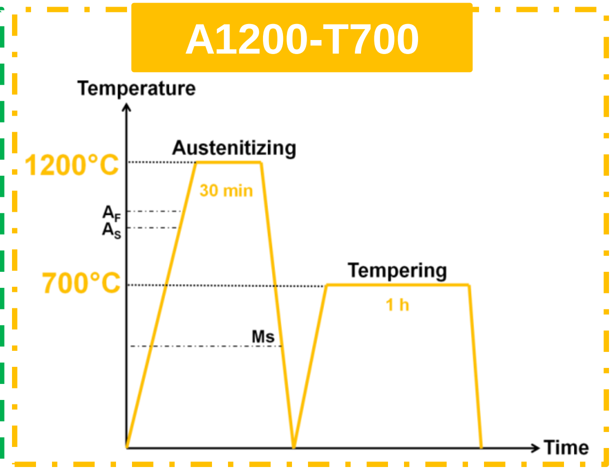
D_{M2X} = 15 nm, f_v = 0.35%



Higher f_v and finer D of intragranular precipitates

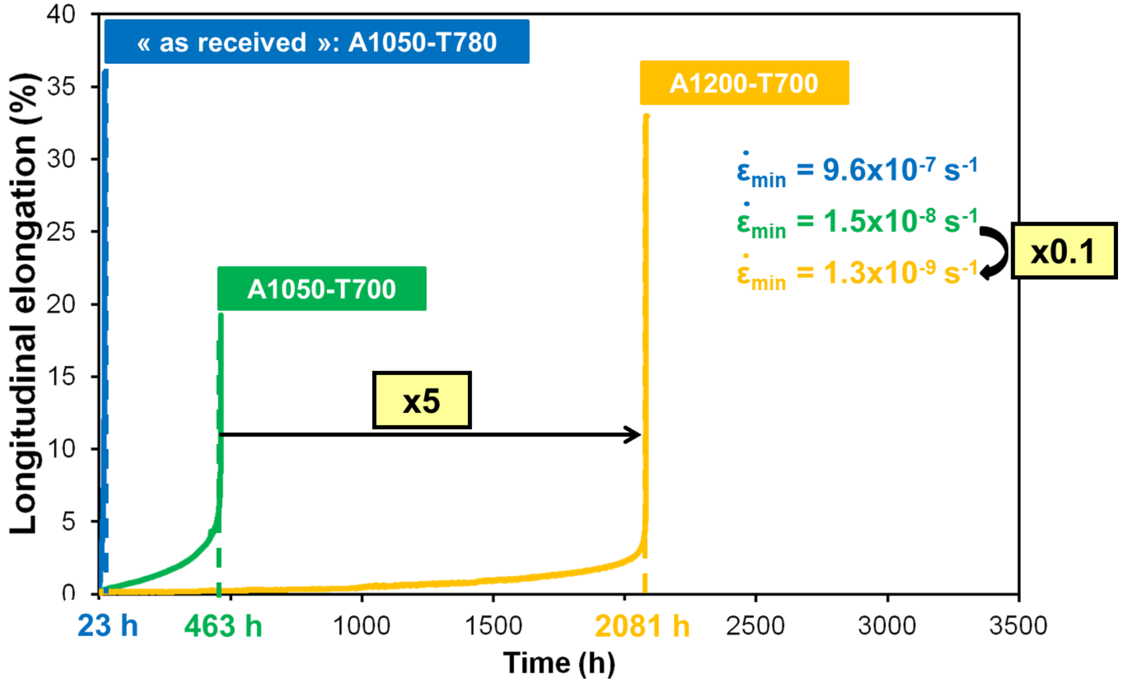
COMPARISON A1050-T700 VS A1200-T700
↗ AUSTENITIZING TEMPERATURE FROM 1050°C TO 1200°C





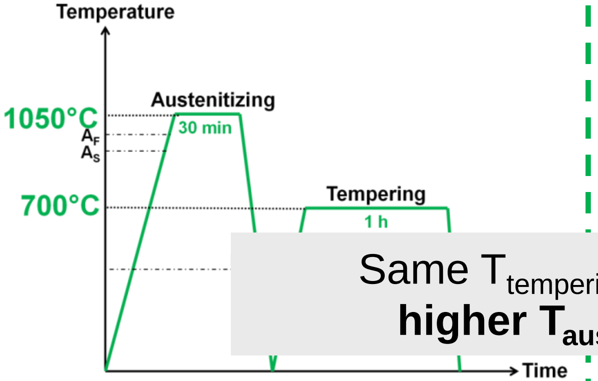
Same $T_{tempering}$: 700°C
higher $T_{austenitizing}$

- **Creep conditions**: 140 MPa at 650°C

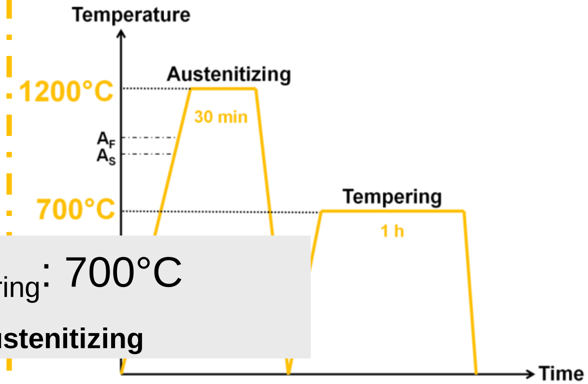


COMPARISON A1050-T700 VS A1200-T700
↗ AUSTENITIZING TEMPERATURE FROM 1050°C TO 1200°C

A1050-T700

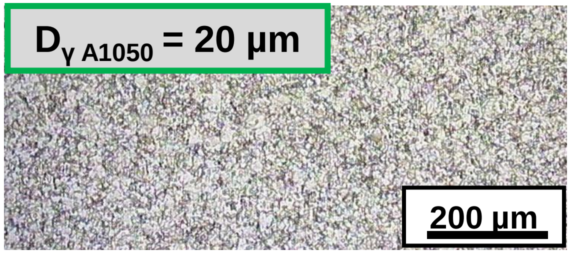


A1200-T700

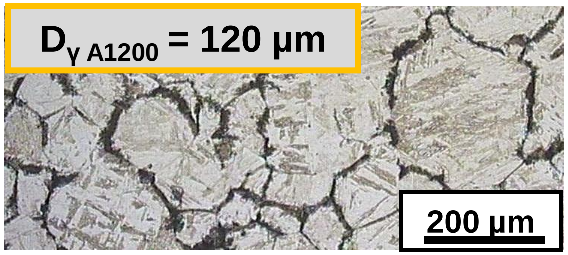


Same $T_{tempering}$: 700°C
higher $T_{austenitizing}$

$D_{\gamma\ A1050}$ = 20 µm

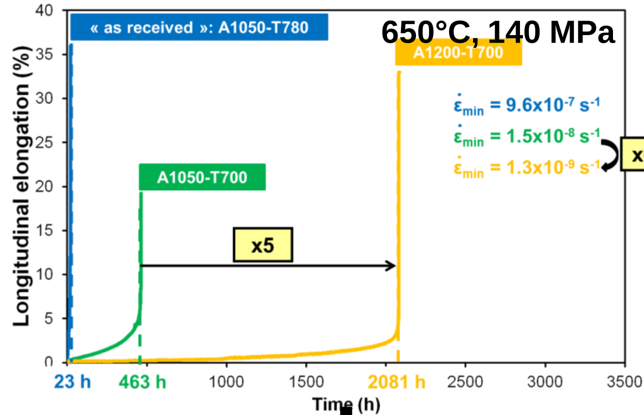


$D_{\gamma\ A1200}$ = 120 µm



W_{blocks} = 3.2 µm

W_{blocks} = 4.3 µm



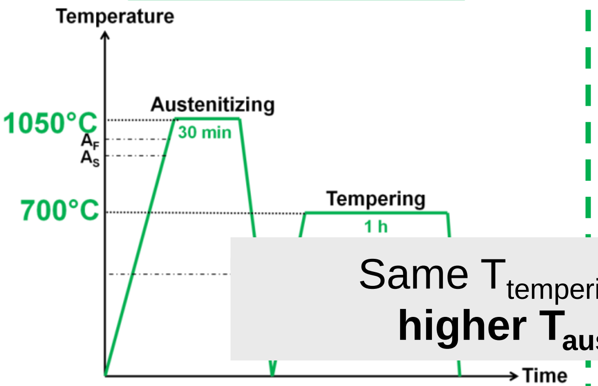
Higher prior γ grain size
(dissolution of small precipitates)

+

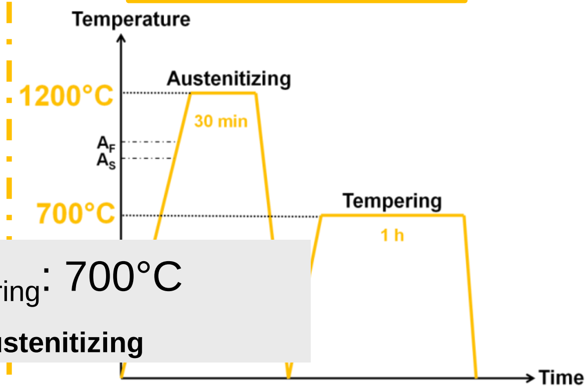
Higher martensite block size

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

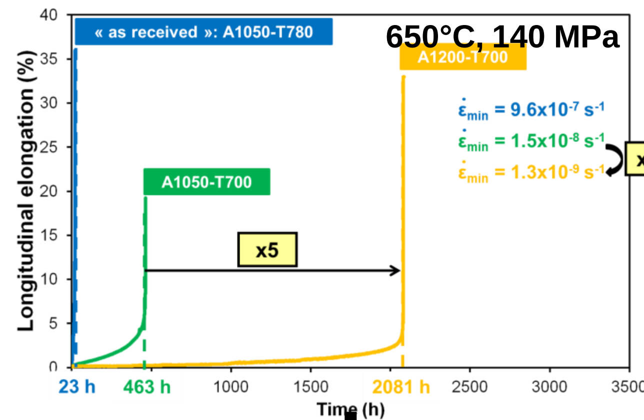
A1050-T700



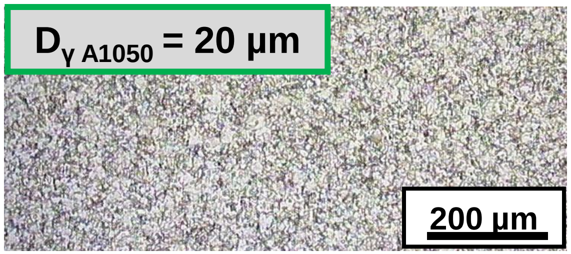
A1200-T700



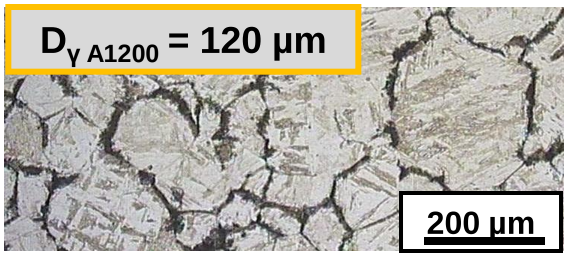
Same $T_{tempering}$: 700°C
higher $T_{austenitizing}$



$D_{\gamma\ A1050}$ = 20 µm



$D_{\gamma\ A1200}$ = 120 µm



Higher prior γ grain size
(dissolution of small precipitates)

W_{blocks} = 3.2 µm

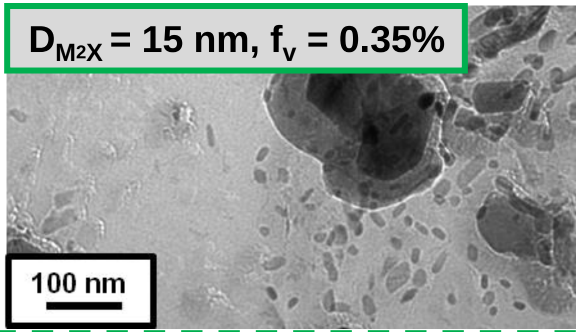
W_{blocks} = 4.3 µm

+

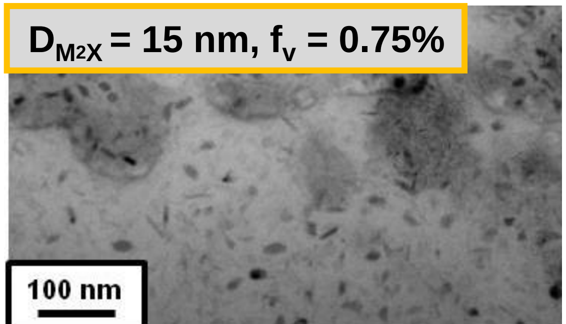
Higher martensite block size

+

D_{M2X} = 15 nm, f_v = 0.35%

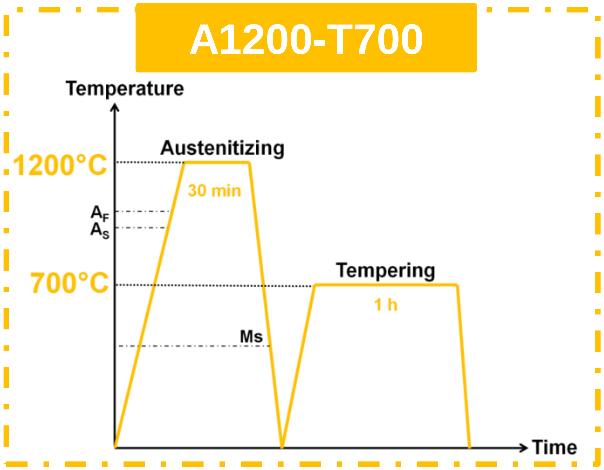


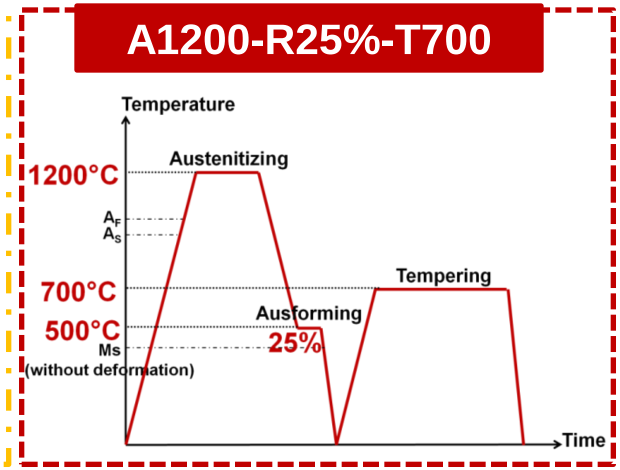
D_{M2X} = 15 nm, f_v = 0.75%



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

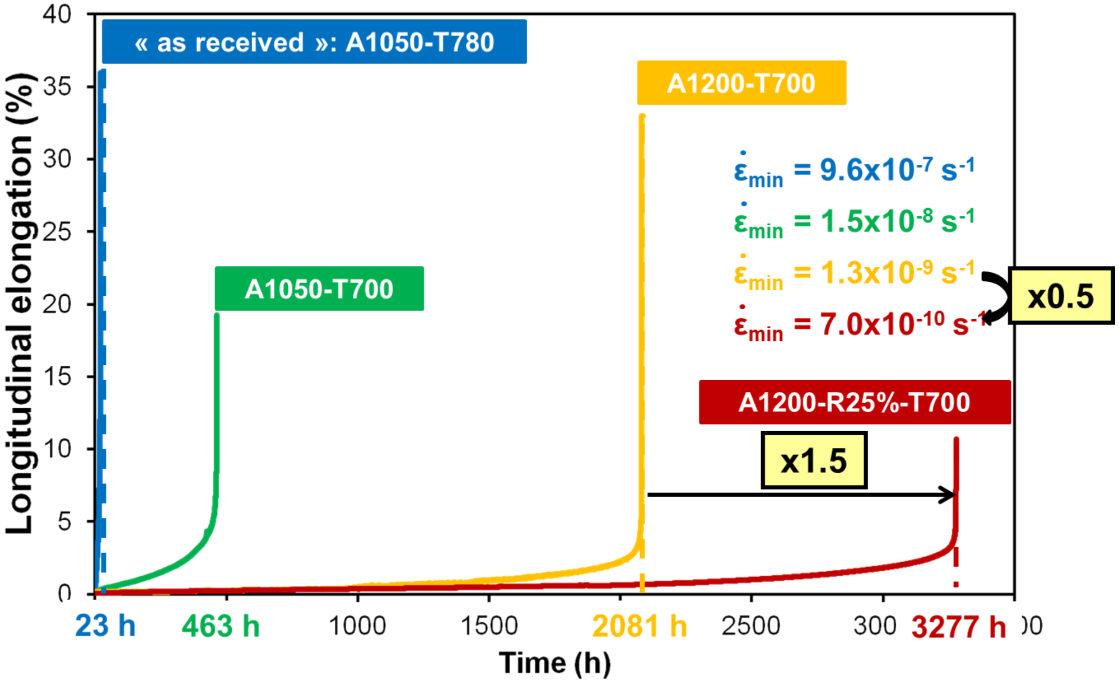
COMPARISON A1200-T700 VS A1200-R25%-T700 APPLYING WARM-ROLLING IN METASTABLE γ





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

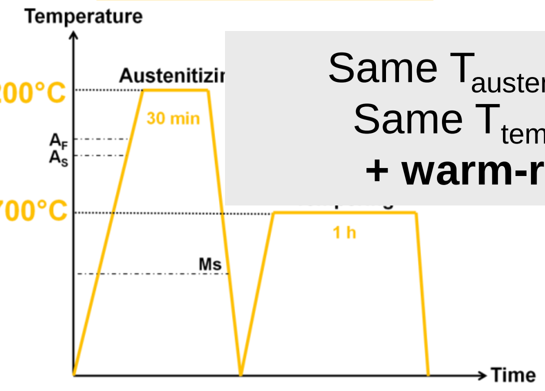
- **Creep conditions**: 140 MPa at 650°C



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

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

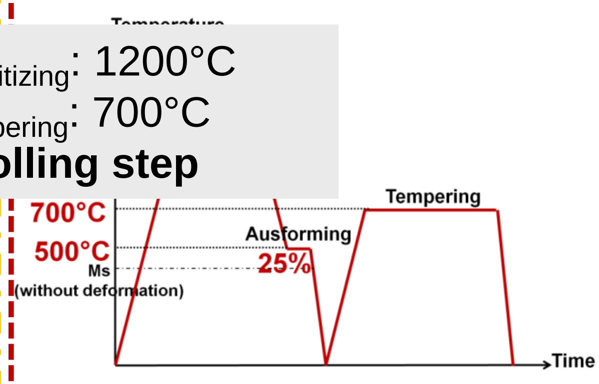
A1200-T700



W_{laths} ~ 400 nm

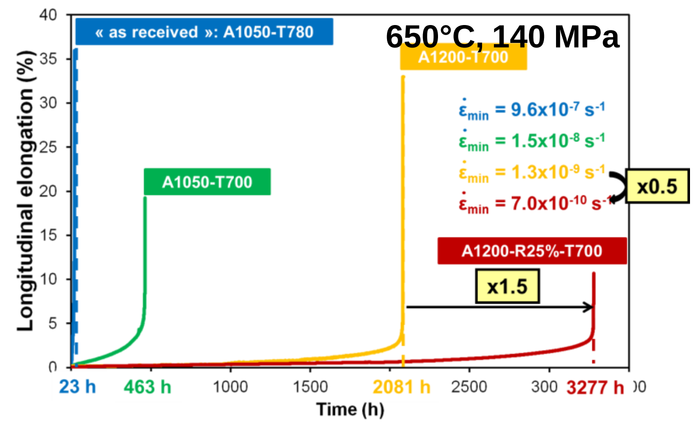
ρ = (4.7 ± 0.5)x10^{14} m^{-2}

A1200-R25%-T700



W_{laths} ~ 250 nm

ρ = (6.2 ± 0.5)x10^{14} m^{-2}

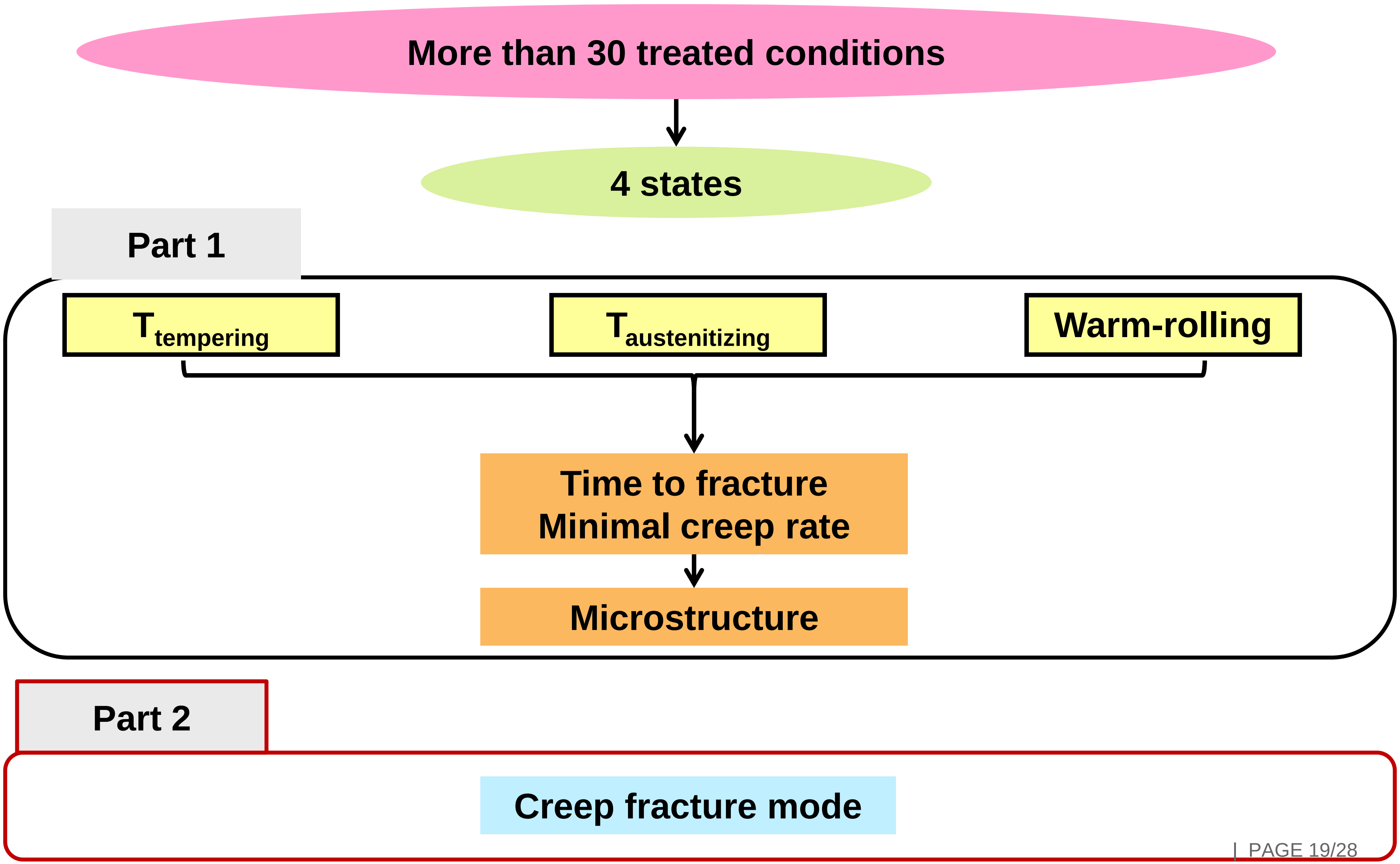


Finer lath width

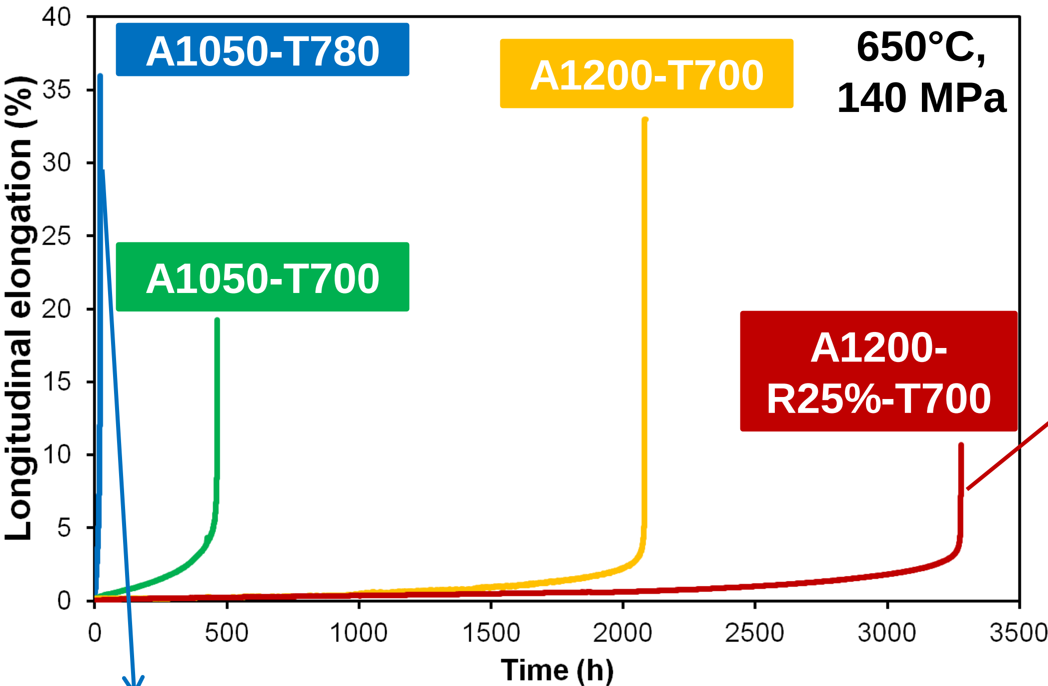
+

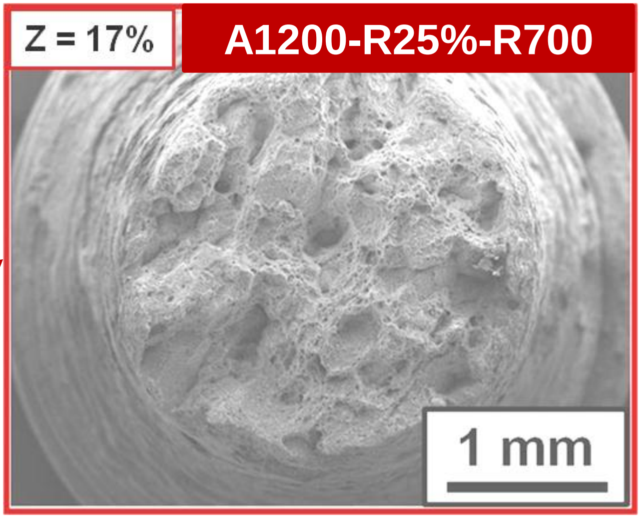
Higher dislocation density
(inheritance from warm-rolling)

OUTLINE

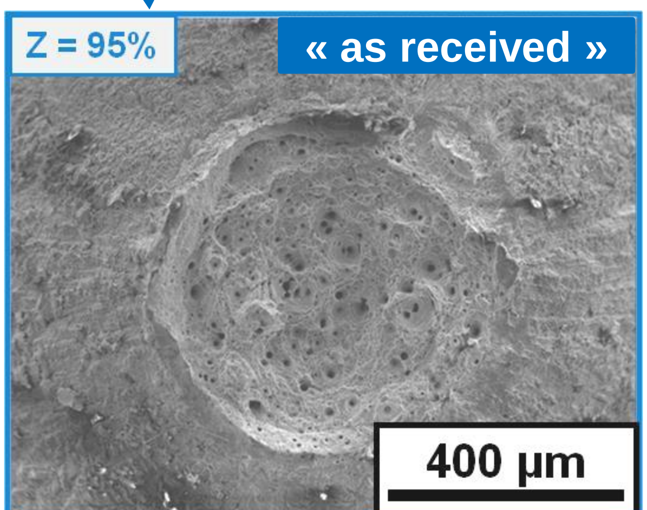

More than 30 treated conditions
4 states
Part 1
$T_{tempering}$
$T_{austenitizing}$
Warm-rolling
Time to fracture
Minimal creep rate
Microstructure
Part 2
Creep fracture mode

CREEP FRACTURE MODE





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



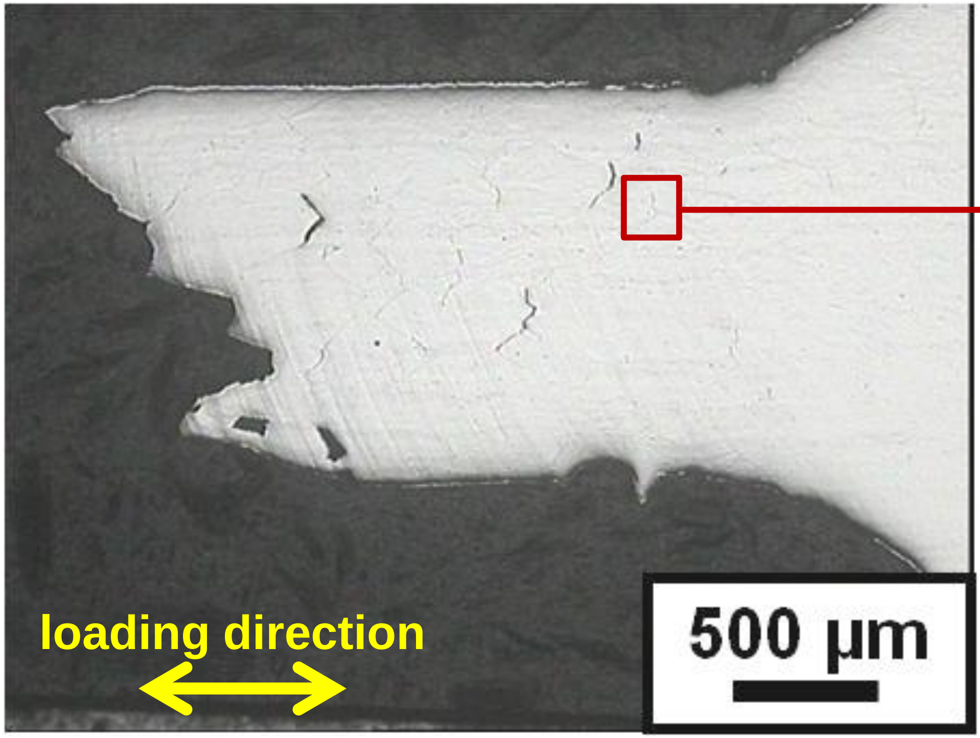
Significant tertiary creep stage, high reduction of area at failure, **Ductile fracture.**

CREEP FRACTURE MODE

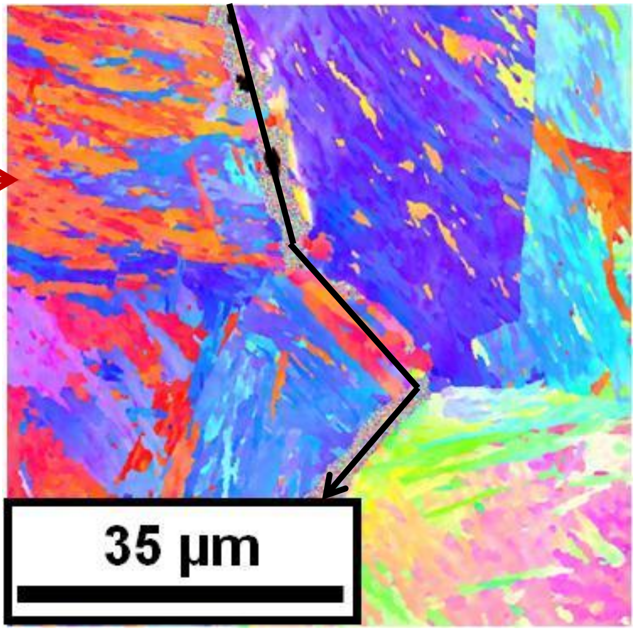
Ductile + intergranular creep fracture mode observed for:
A1200-R(0-25%)-T(700-750°C) states (650°C, 140 MPa)

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

cross section observation



microcrack

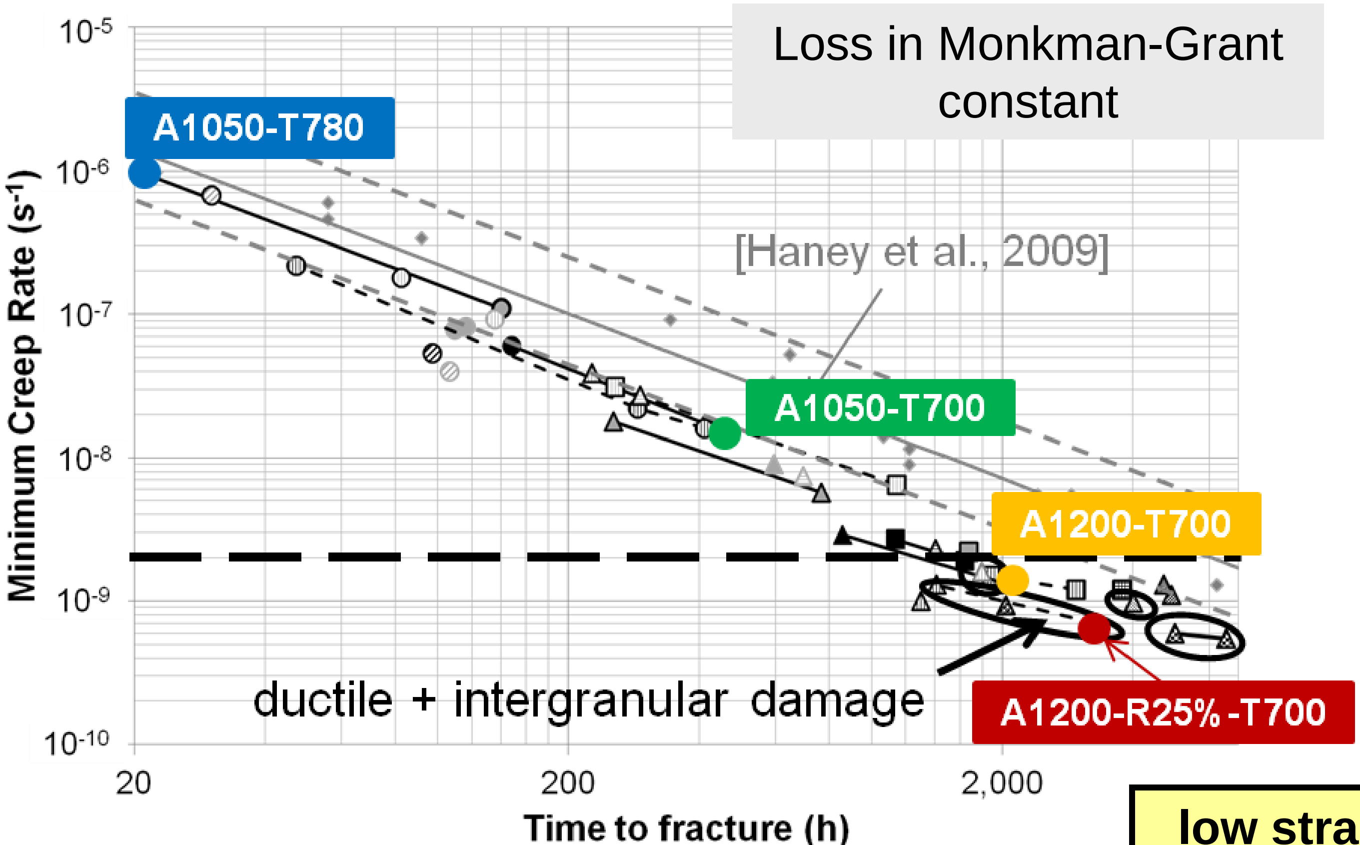


EBSD map

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?



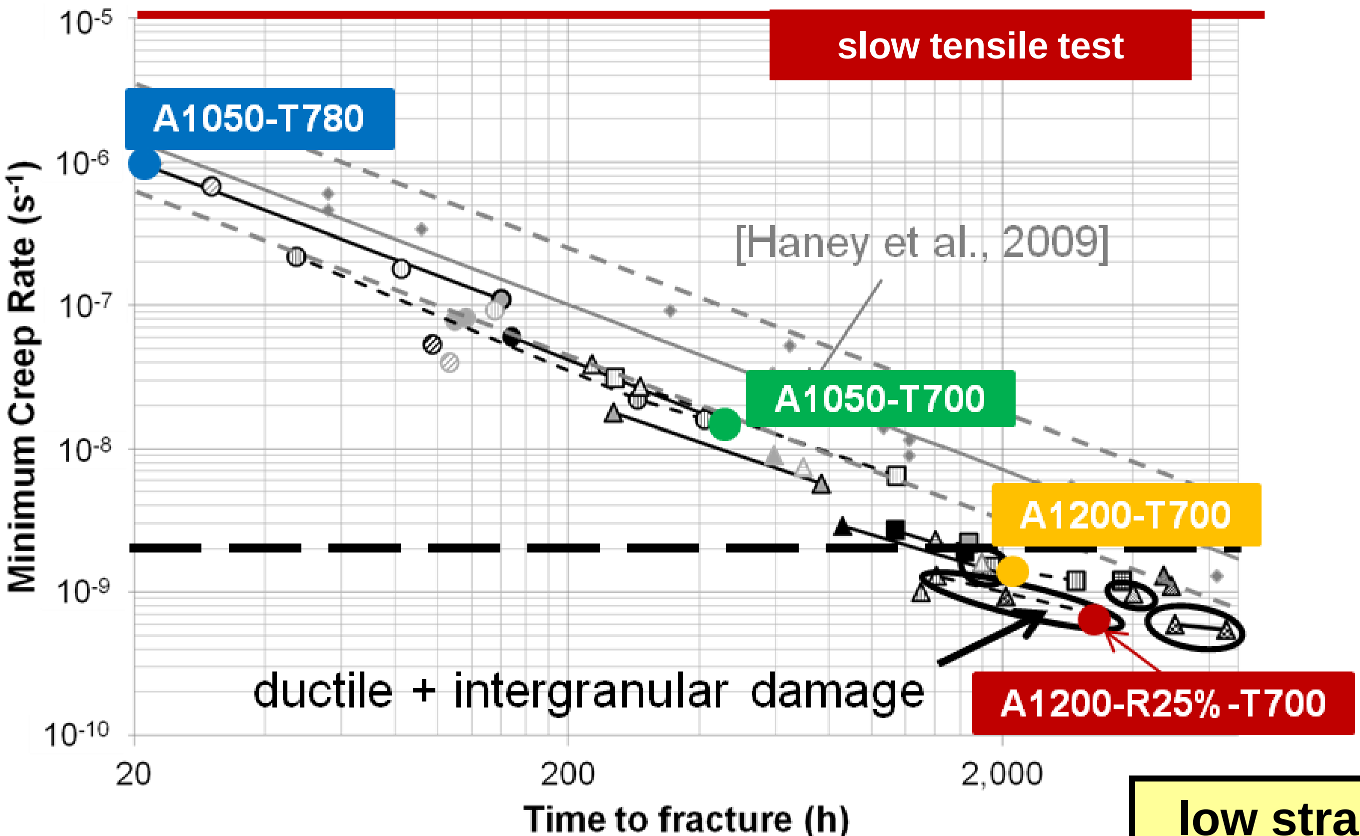
650°C

low strain rate ~ 9.10^{-8} s^{-1} ?

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?



650°C

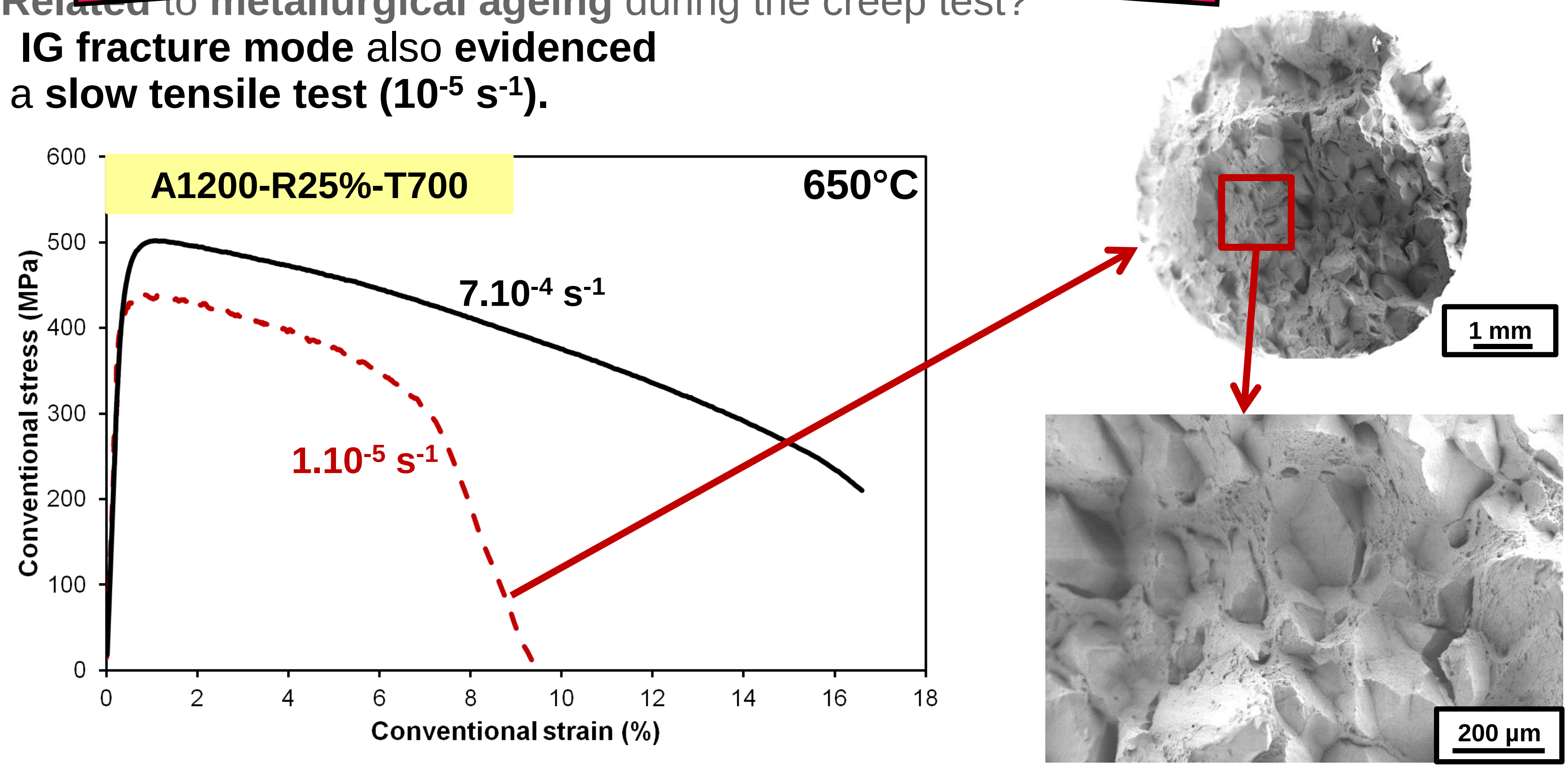
low strain rate ~ 9.10^{-8} s^{-1} ?

CREEP FRACTURE MODE

1. **Rel**... **low deformation mechanism** typical of l... creep of this steel (low strain rate ... damage)?

2. **Rel**... to **metallurgical ageing** during the creep test?

→ **IG fracture mode** also **evidenced** by a **slow tensile test (10^{-5} s^{-1}).**



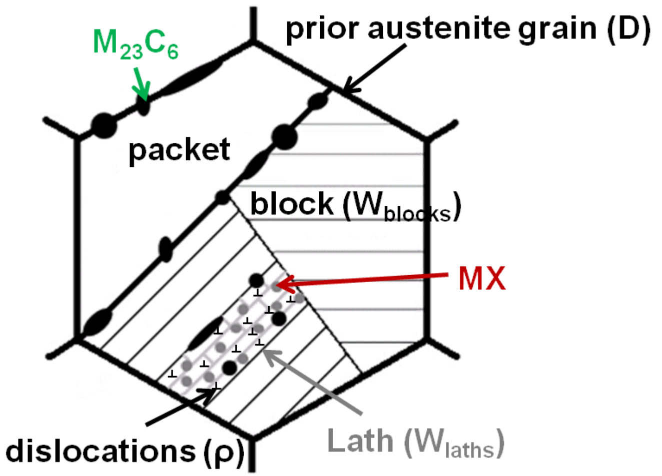
Due to some specific martensitic microstructures
(high D_γ, effects of impurities and/or high precipitates density at prior-γ grain boundaries, critical intragranular hardening…) **???**

4 states (3 thermal + 1 thermomechanical treatments)

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

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

	↗ $T_{austenitizing}$	↘ $T_{tempering}$	+ warm-rolling
D_γ	↗	=	=
W_{blocks}	↗	=	=
W_{laths}	=	=	↘
ρ	=	↗	↗
$f_{v\ intra}$	↗	↗	=
D_{intra}	=	↘	=



1^{st} 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 ».

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 !

The workers are grateful for financial support of EDF

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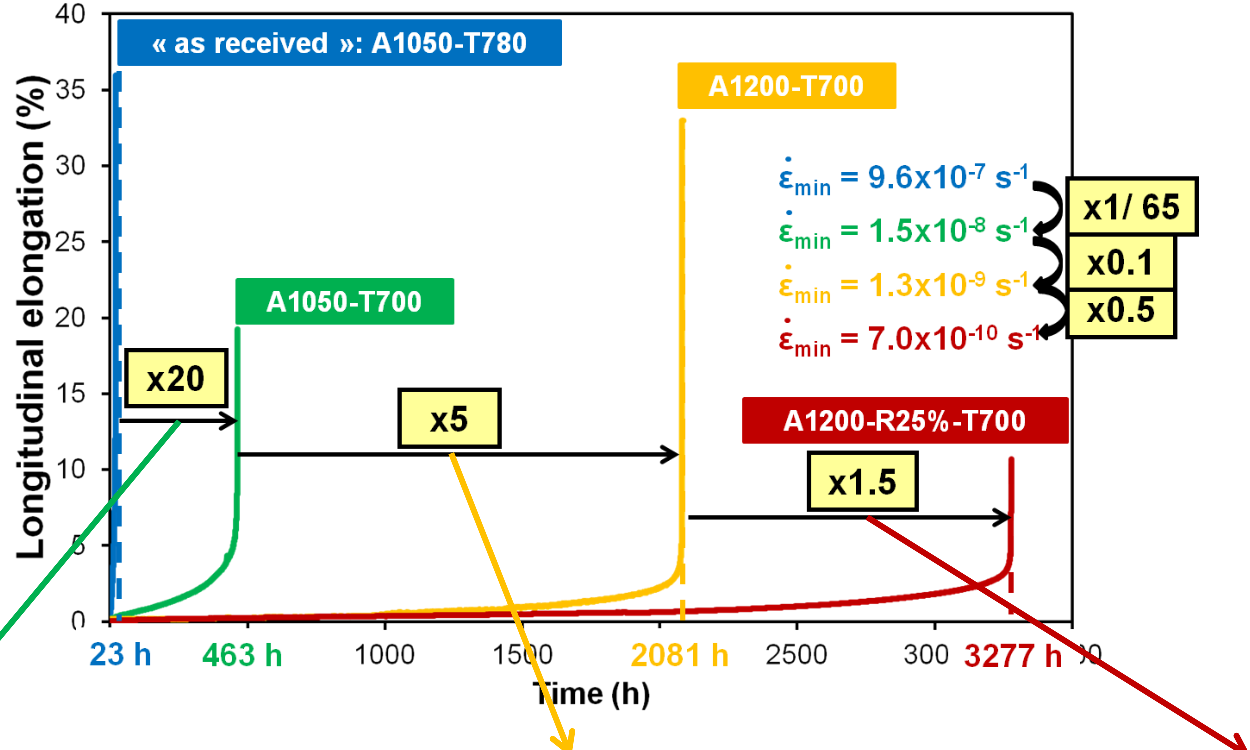
SUMMURY – PART 1

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

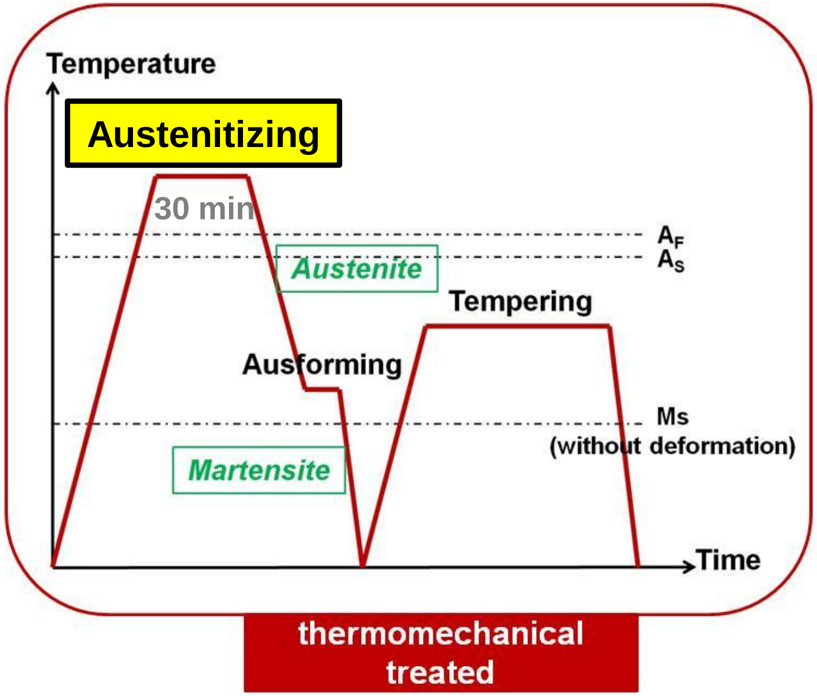


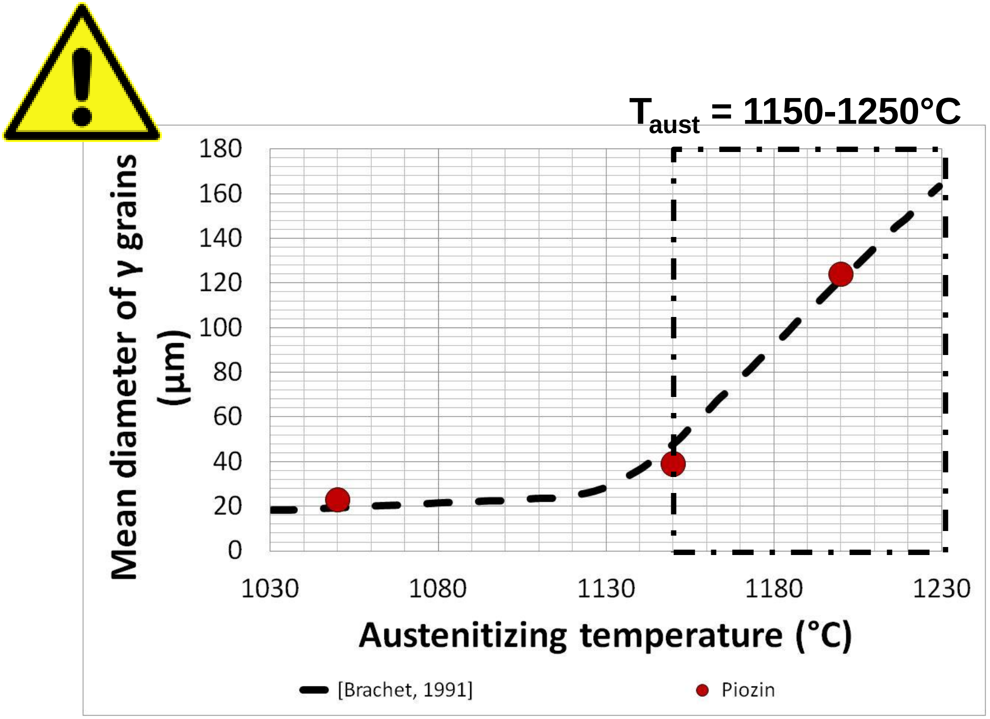
↘ $T_{tempering}$ | ↗ $T_{austenitizing}$ | Warm-rolling

↗ ρ | ↗ f_v intra | ↗ D_γ, W_{blocks} | negligeable effect

1st order parameters

AUSTENITIZING STEP

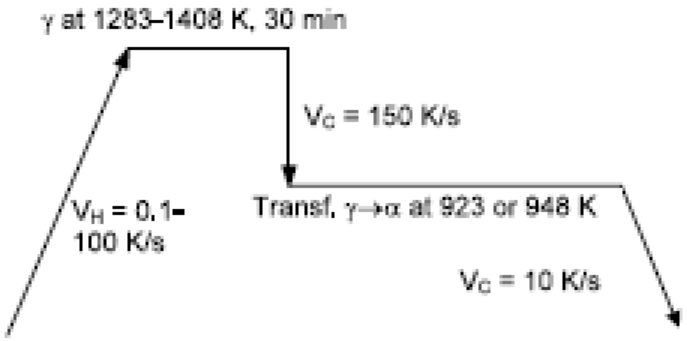


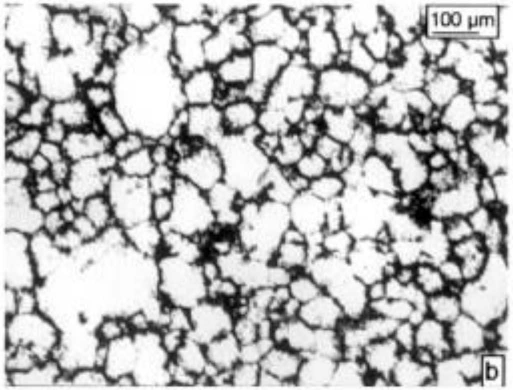


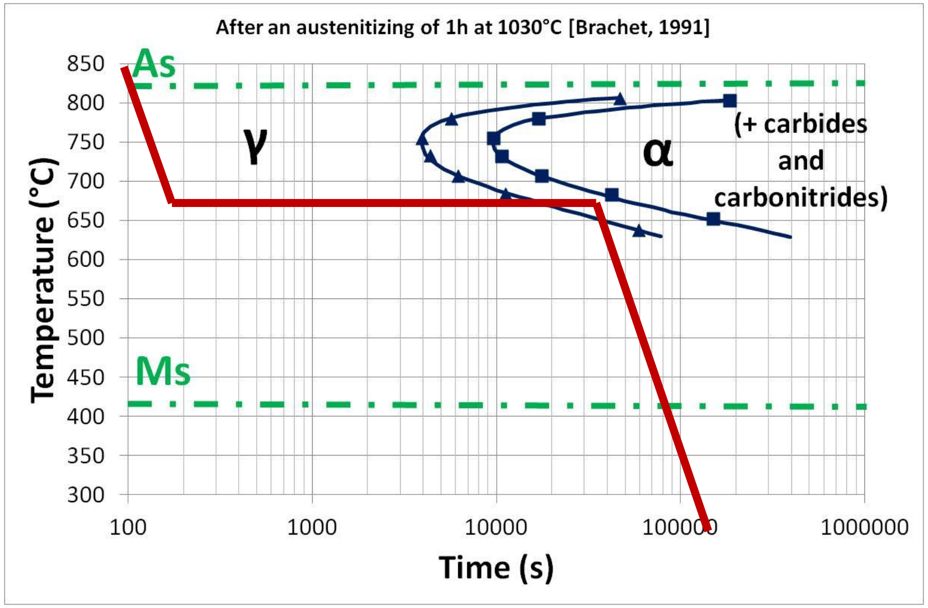
Austenitizing temperature (°C)	
1050	"as received" condition
1150	to dissolve MX precipitates
1200	

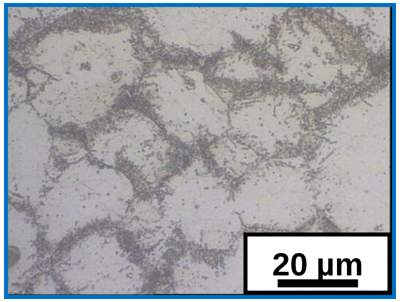
Experimental procedure to measure prior austenite grain size :

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



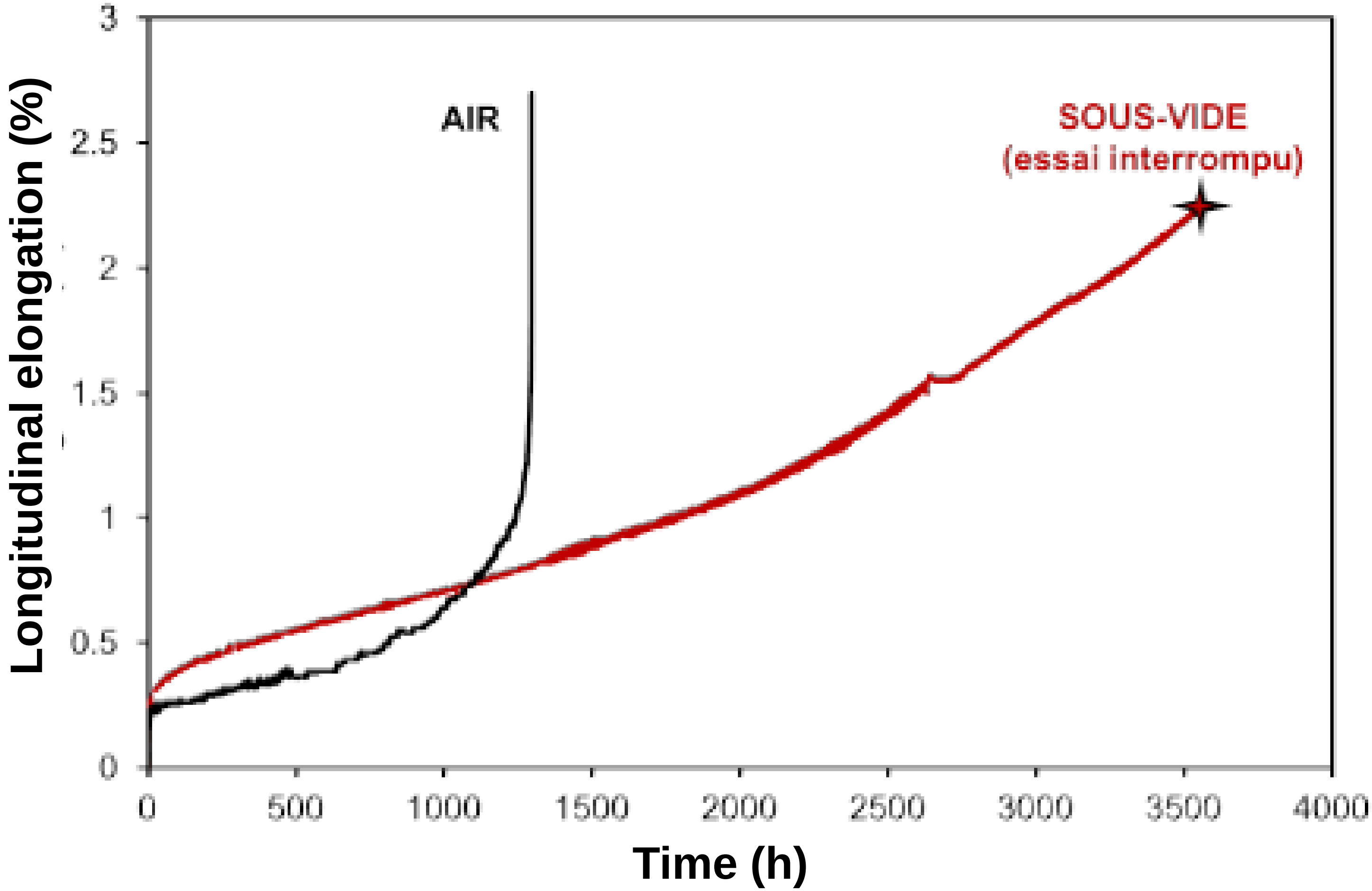






INFLUENCE OF ENVIRONNEMENT

A1200-R500,25%-T700

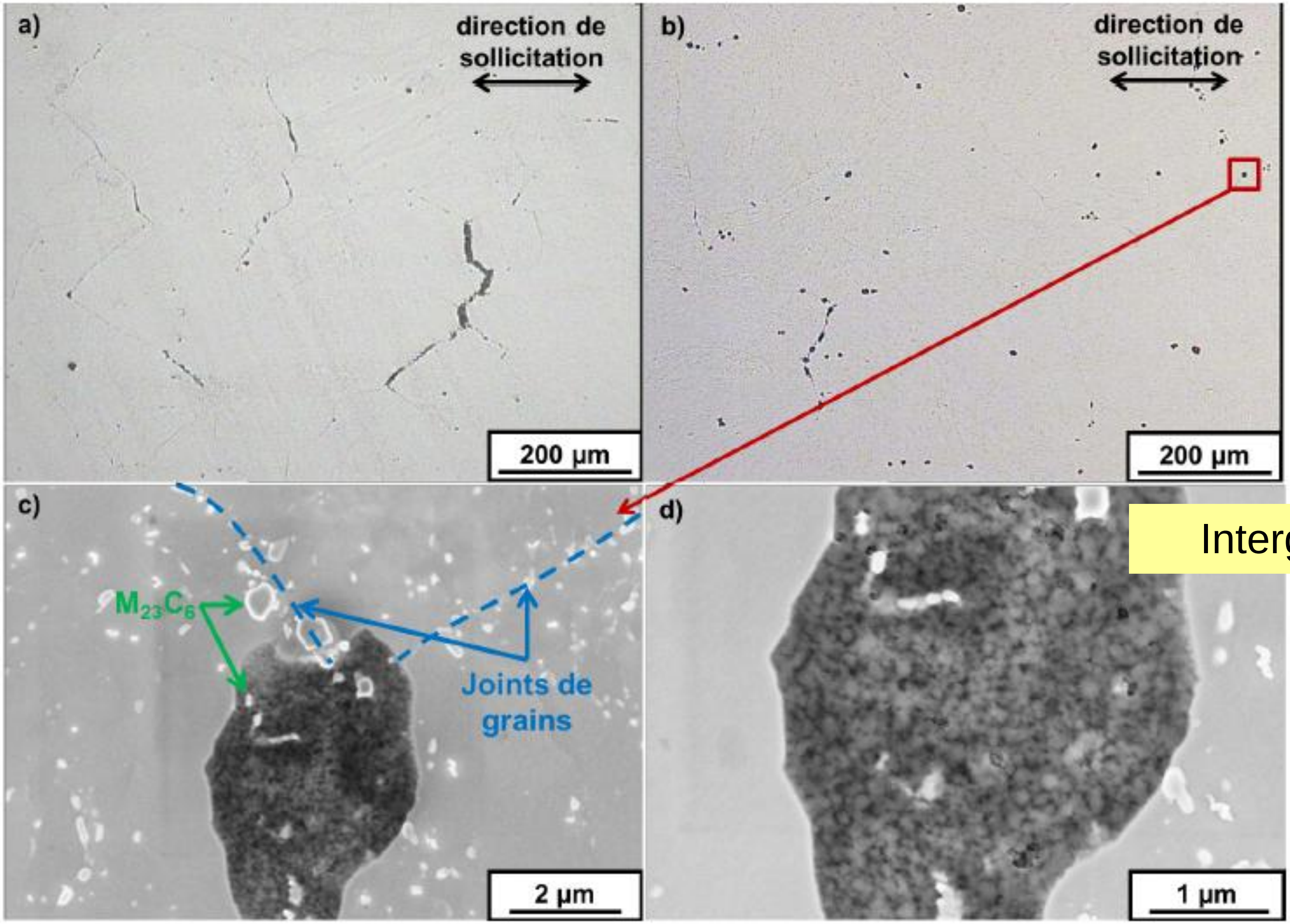


Same minimal creep rate:
Air = 1.10^{-9} s^{-1}, Vaccum = 9.10^{-10} s^{-1}.
But t_r higher for vaccum !

INFLUENCE OF ENVIRONNEMENT

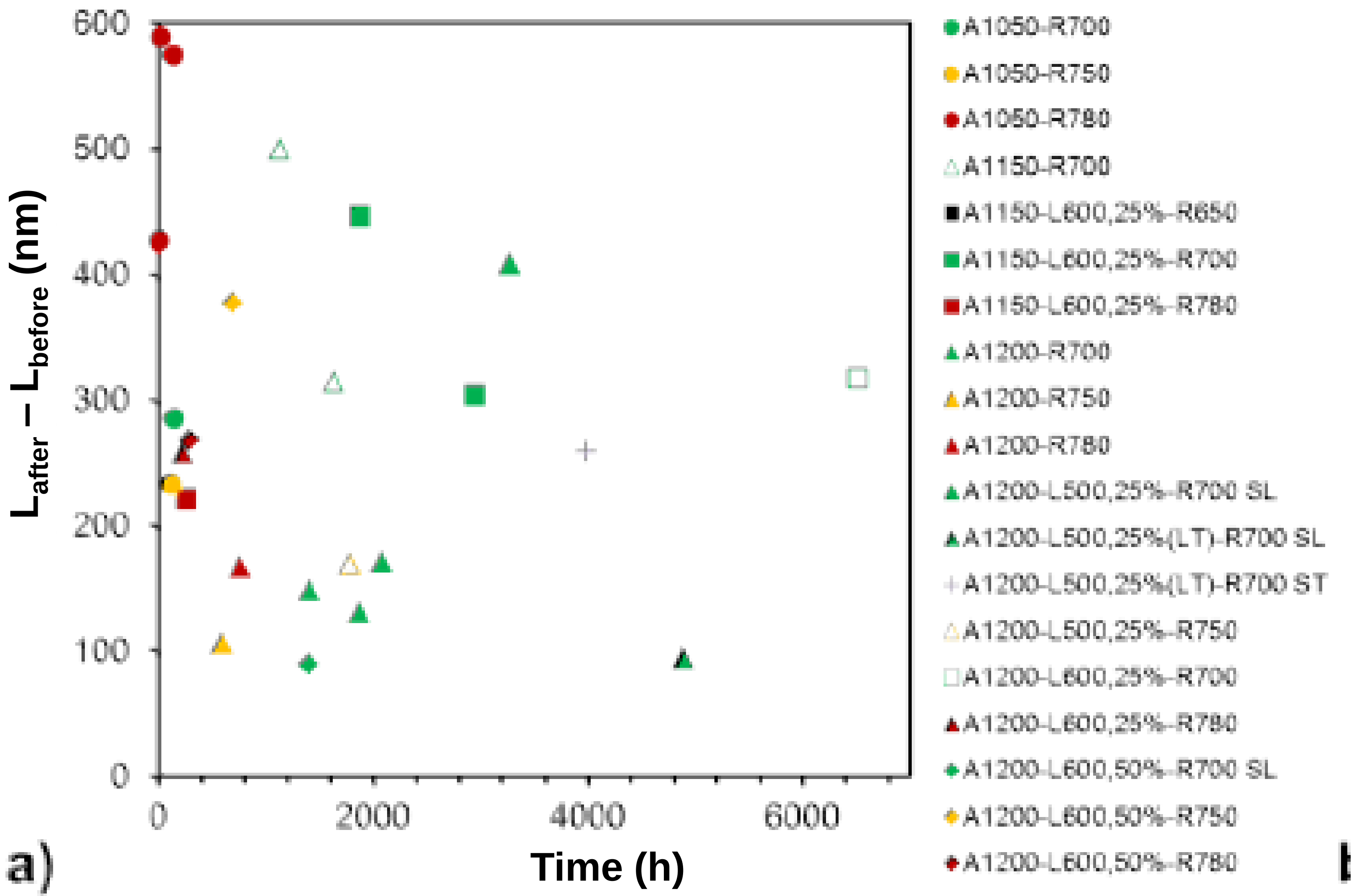
A1200-R500,25%-T700 (vaccum)

cross section observations

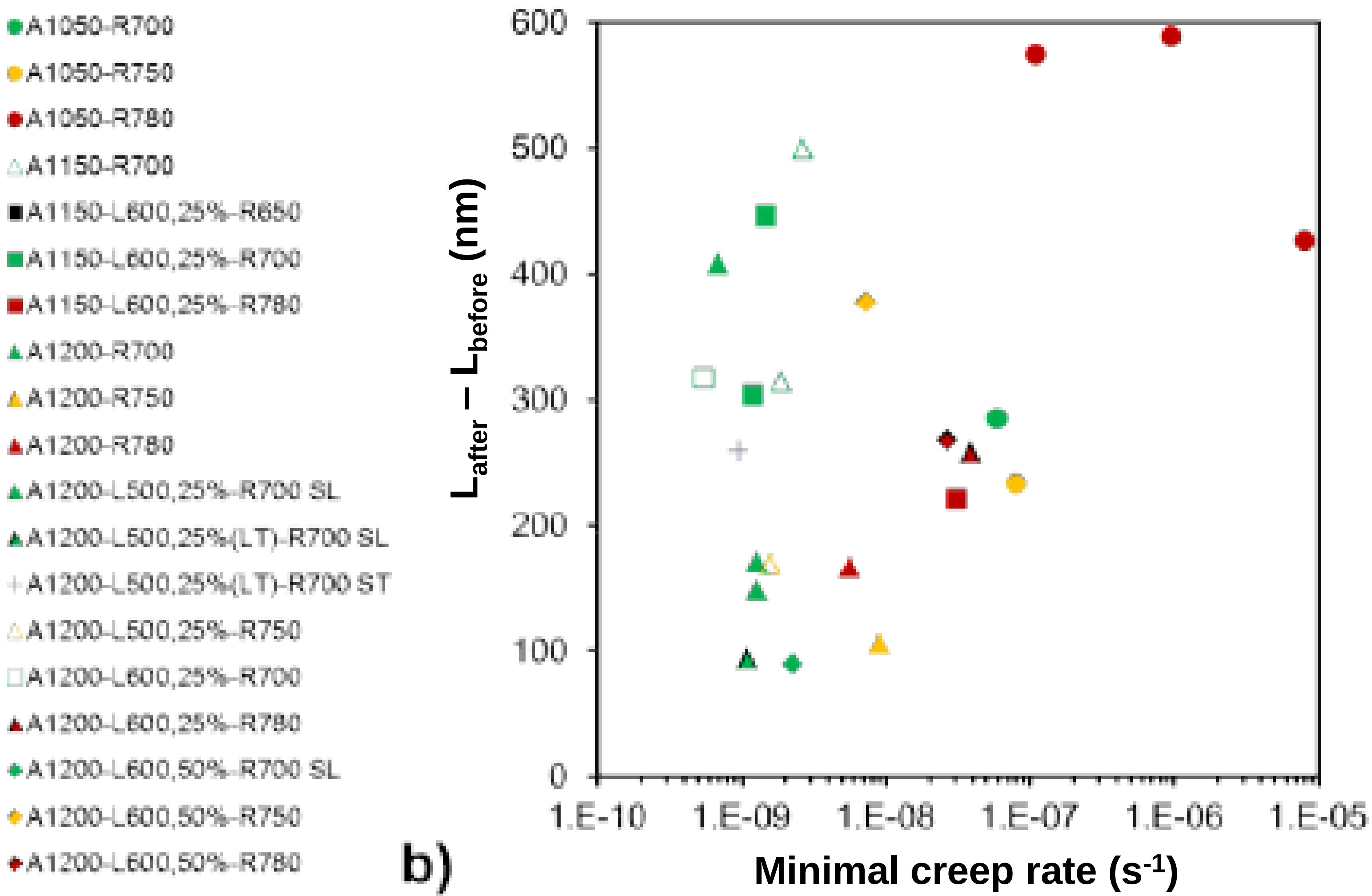


Intergranular cavities

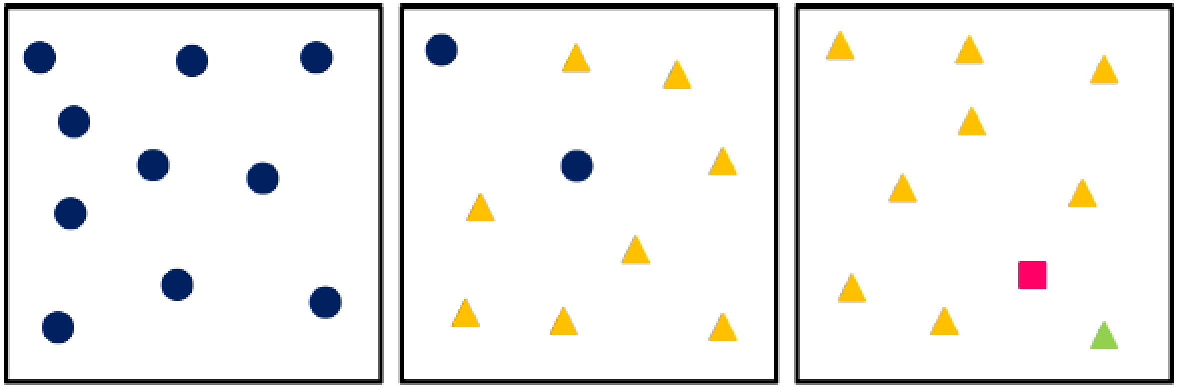
MICROSTRUCTURE AFTER CREEP TESTS



MICROSTRUCTURE AFTER CREEP TESTS

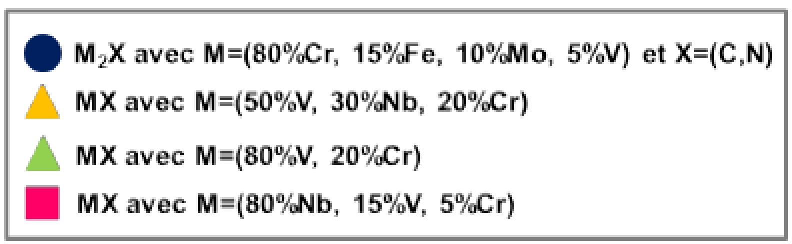


MICROSTRUCTURE AFTER CREEP TESTS



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

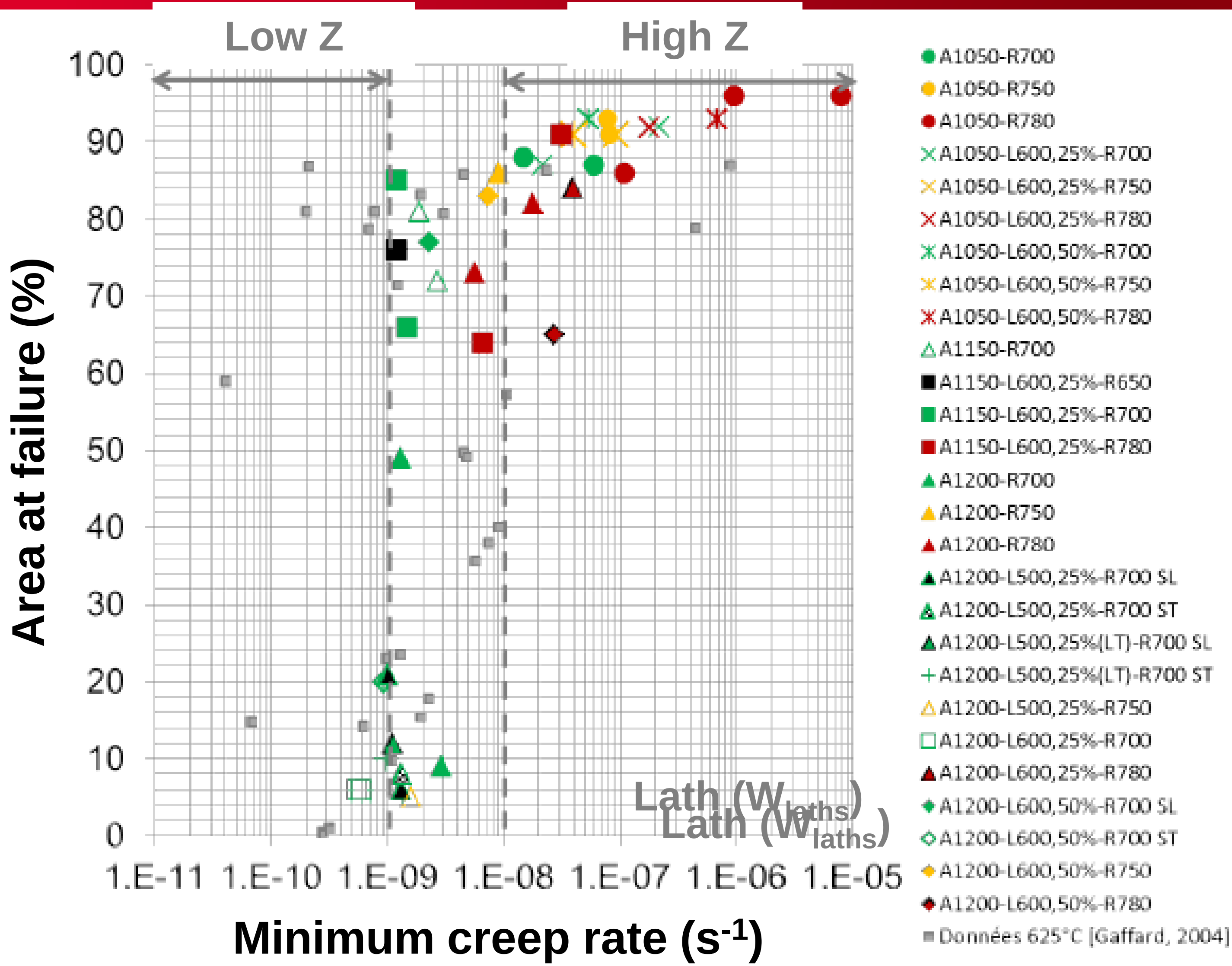
A1200-R600,25%-T700
Creep test :
140 MPa, 650°C
t_r = 6523 h



Change in chemical composition of MX precipitates

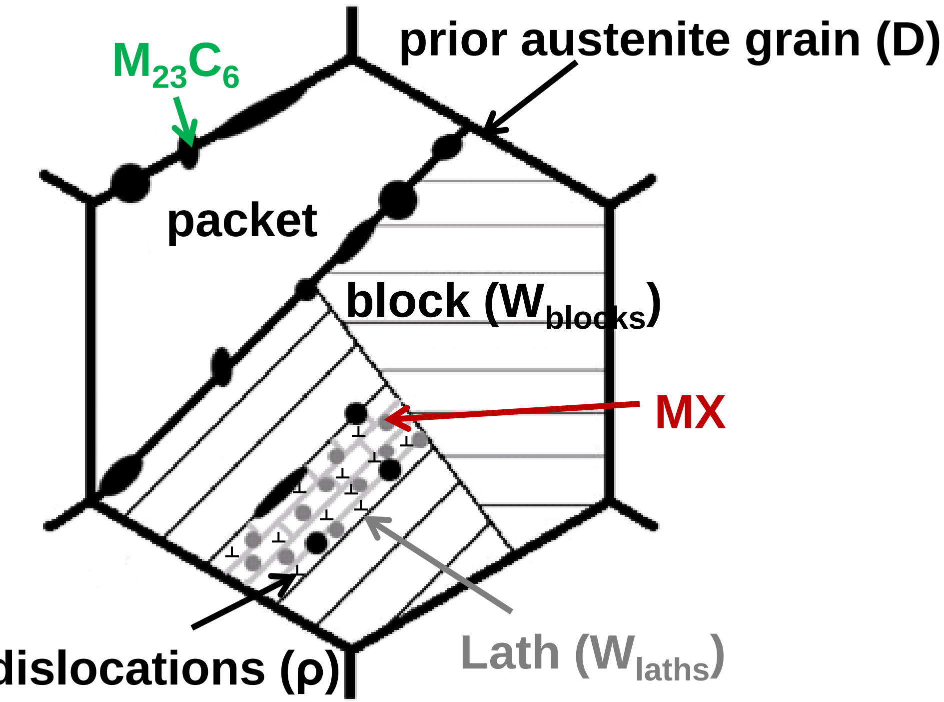
	No creep tested	Creep tested head	Creep tested gauge
D_{M23C6} (nm)	123 ± 58	130 ± 48	147 ± 55
D_{MX} (nm)	18 ± 7	16 ± 4	20 ± 4

MINIMUM CREEP RATE VALUE LIMIT !

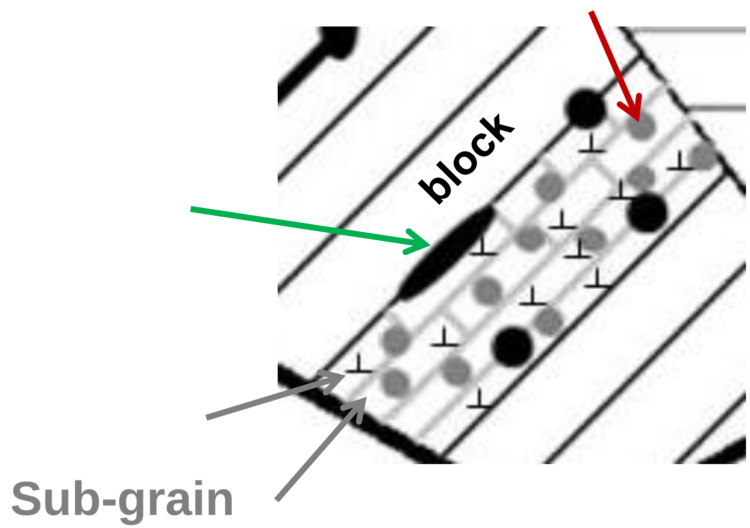


COMPLEX MICROSTRUCTURE OF GRADE 91 STEEL

- **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