



Improvement in creep resistance of G91 steel by conventional ausforming thermomechanical treatment

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OUTLINE

- Background and context

- New thermo-mechanical treatment

 - Austentization

 - Effect of ausforming temperature

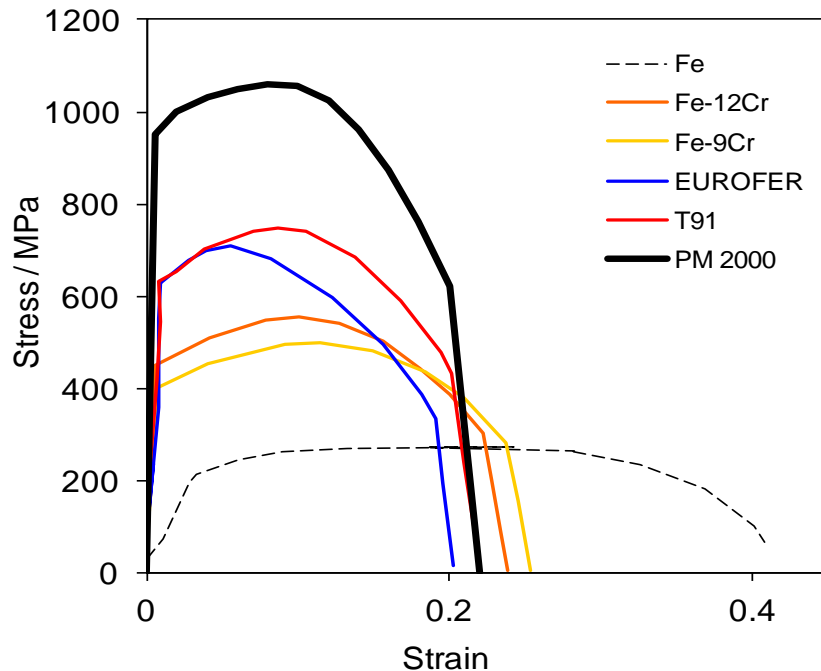
 - Effect of ausforming deformation

- Quick check for creep response

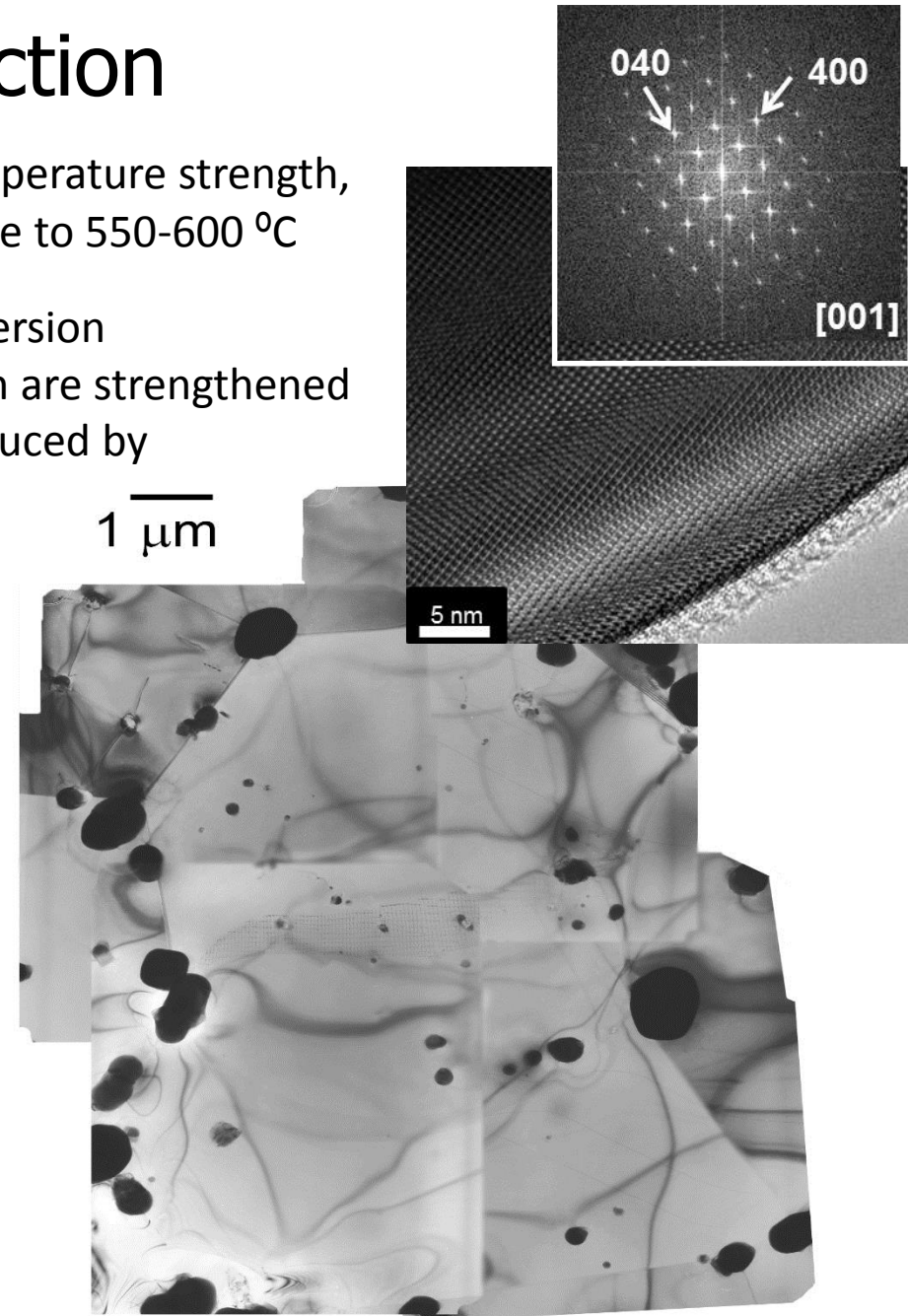
- Conclusions

Introduction

- ❑ A major short-coming of the steels is high temperature strength, which limits the maximum service temperature to 550-600 °C
- ❑ This has led to the development of oxide dispersion strengthened (ODS) steels. These steels, which are strengthened by nanometric in size oxide particles, are produced by complicated and expensive routes.

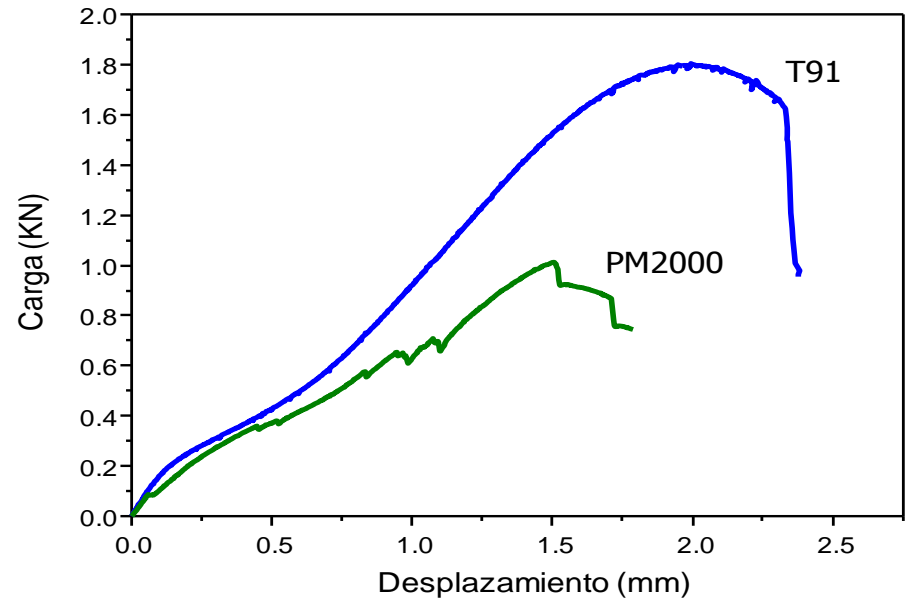
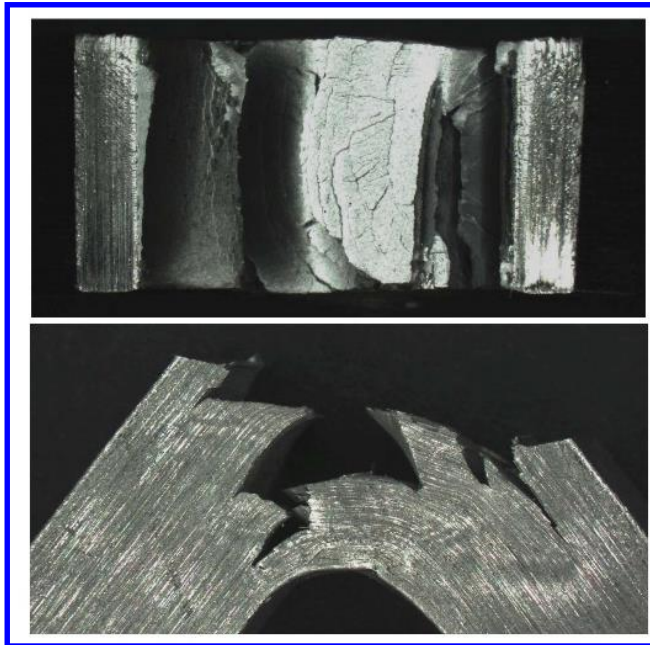


(S. Ukai, M. Fujiwara, J. Nucl. Mater. 307–311 (2002) 749)



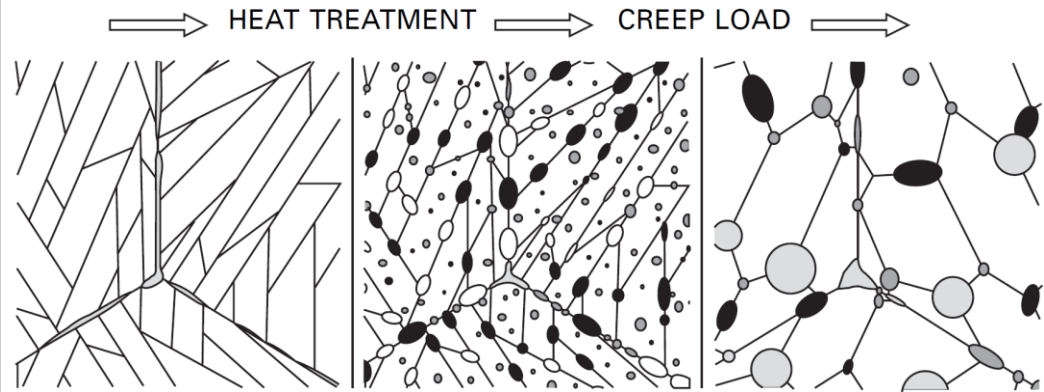
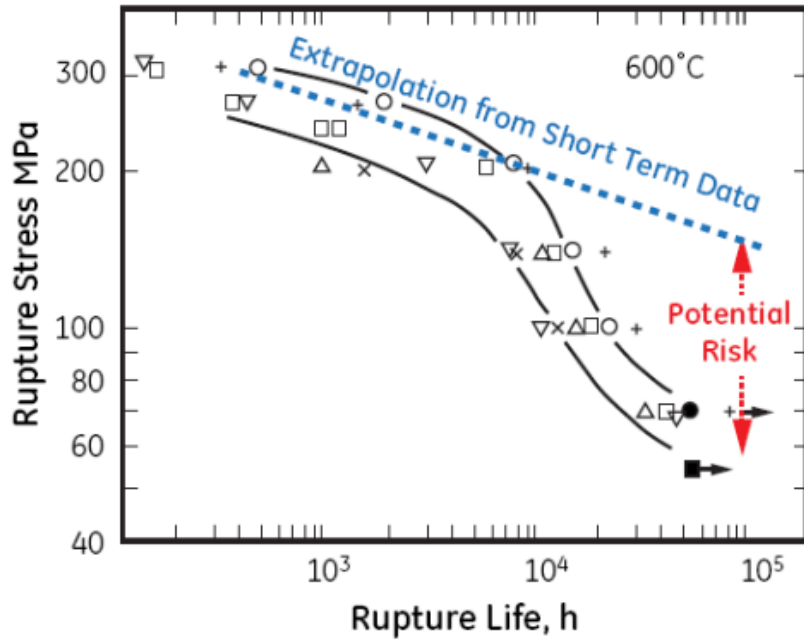
(C. Capdevila et al., JOM 2015, 67, 2208-2215)

- ❑ ODS ferritic or ferritic/martensitic steels are materials with high temperature strength but inferior impact properties with respect to conventional FM steels



(M. Serrano, results from FeCrADS Project, 2013)

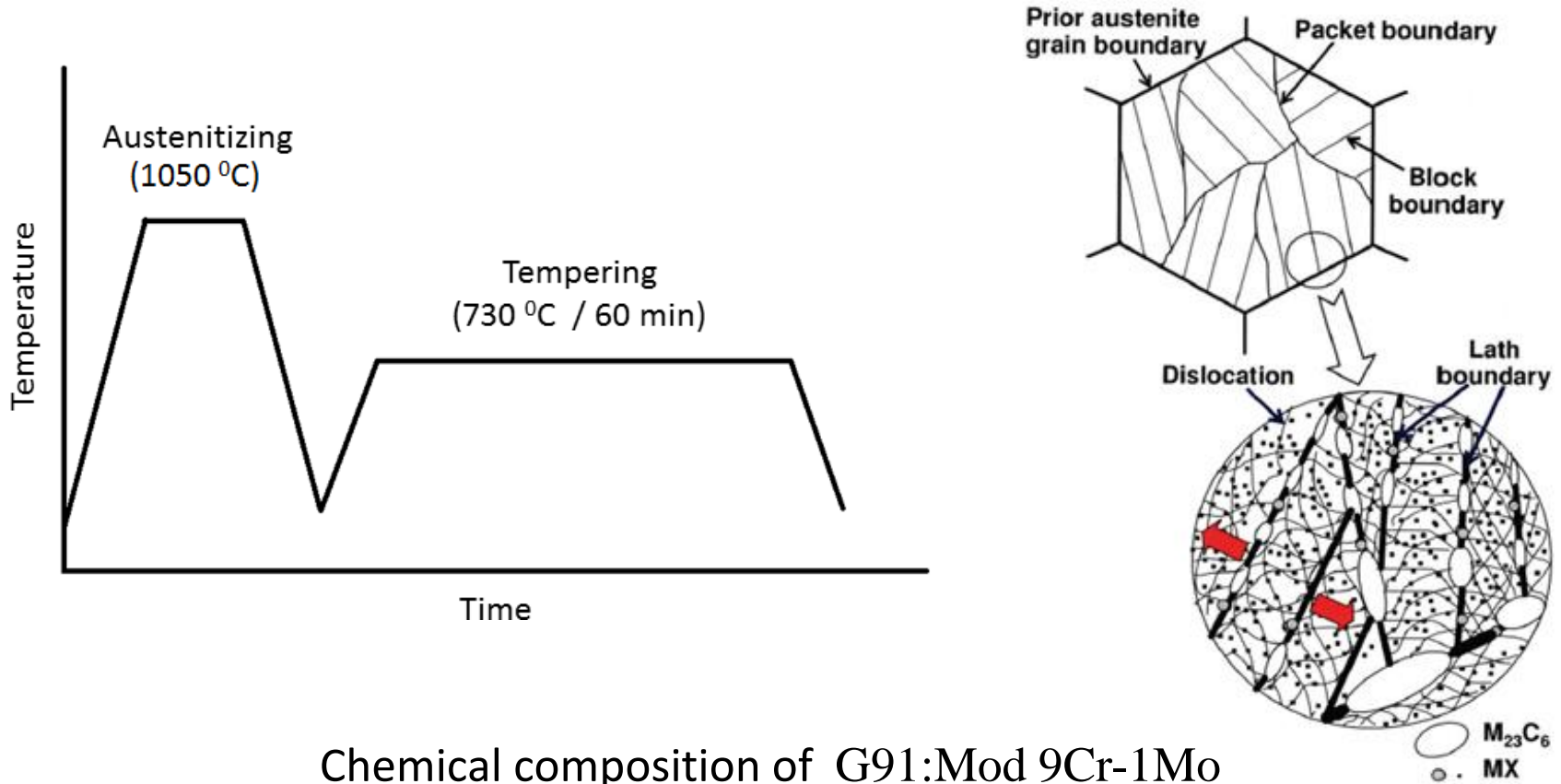
GOAL: Are we able to develop a new and different dispersion-strengthened Steel using conventional processing routes for application above 650 °C?



Many competing effects occurs in 9% Cr steels during creep. Past experience has shown that the instability of any of the Z-phase, Laves, MX or $M_{23}C_6$ can cause unexpected decrease in rupture stress as a function of time. The goal is to slow down the destabilization of the microstructure focusing in MX and $M_{23}C_6$

GOAL2: Does this steel present a more stable microstructure for application above 650 °C?

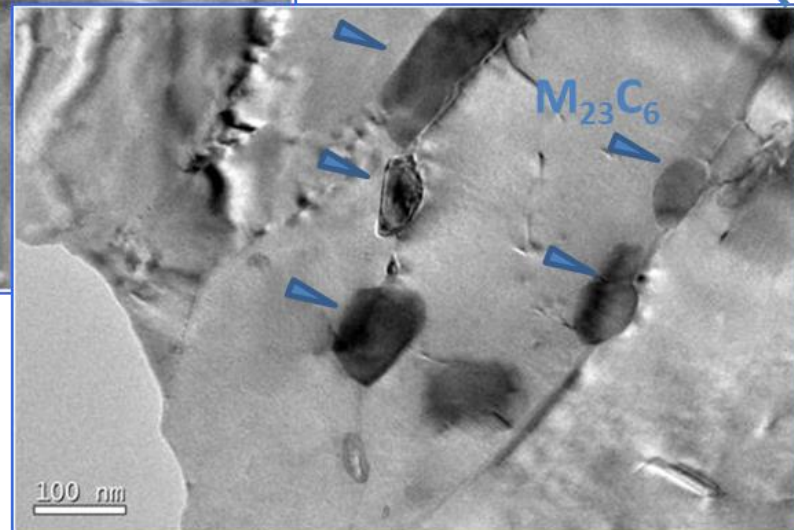
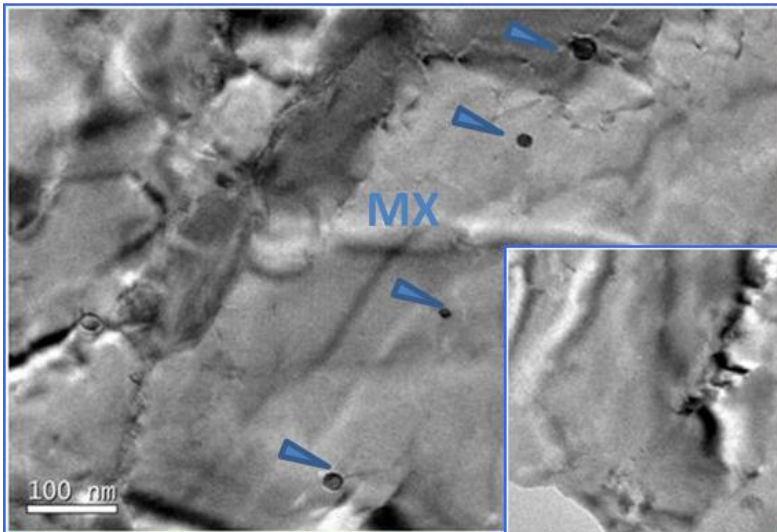
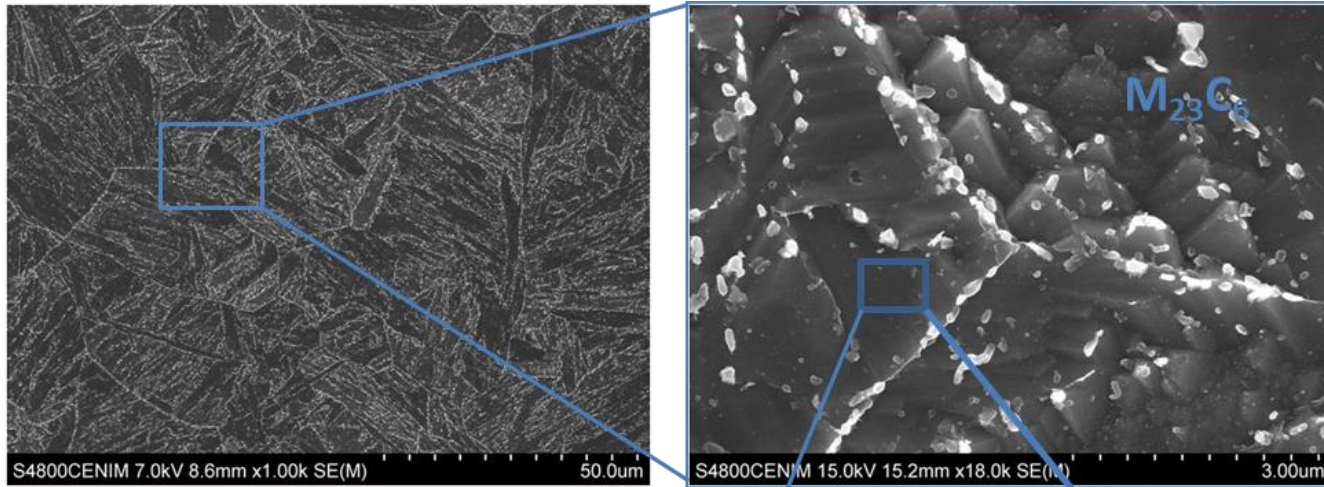
G91 – Reference state (AR condition)



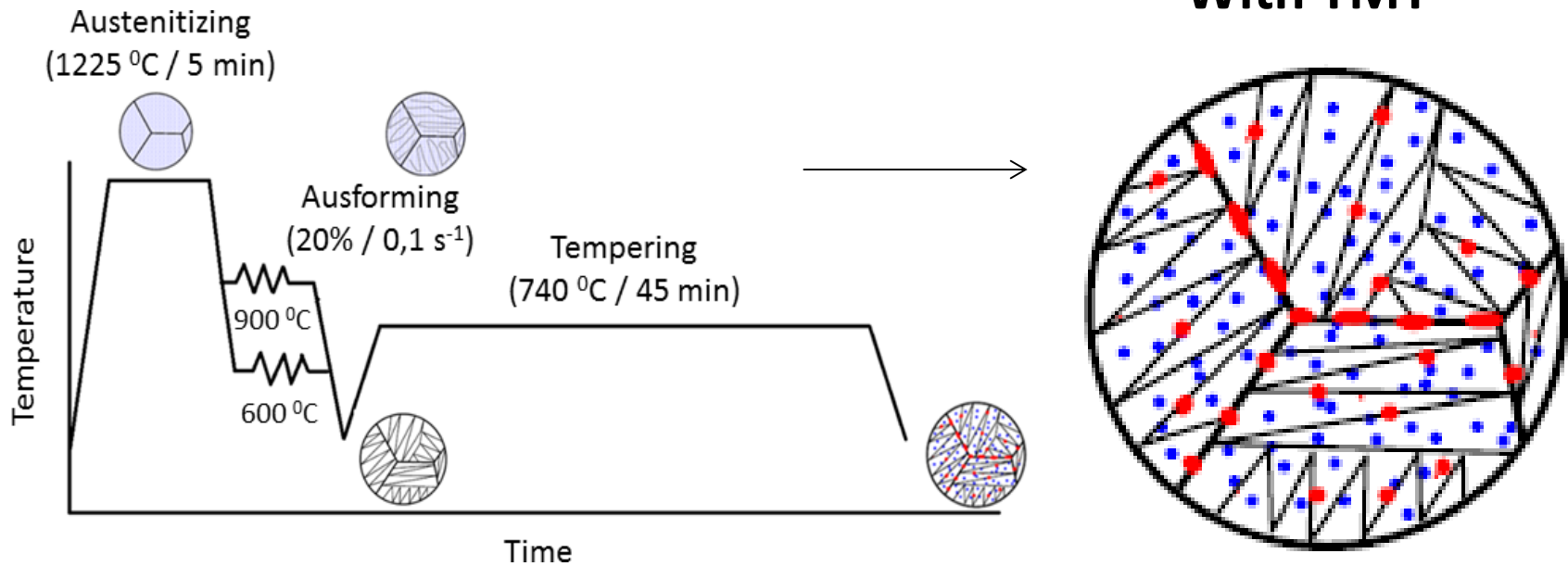
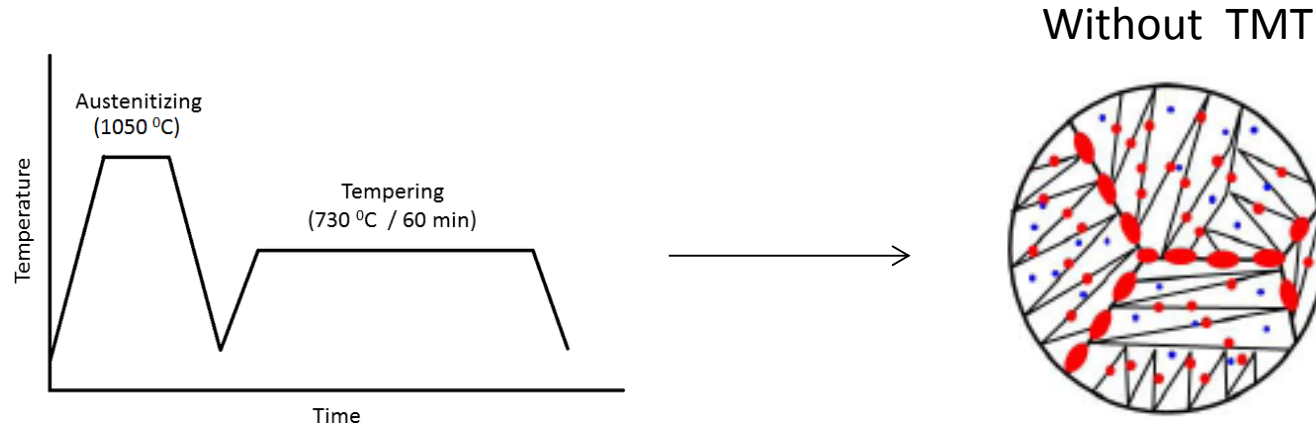
Chemical composition of G91:Mod 9Cr-1Mo

Elements	C	Si	Mn	Cr	Mo	V	Nb	N	Fe
% wt.	0.1	0.4	0.4	9	1	0.2	0.07	0.038	balanced

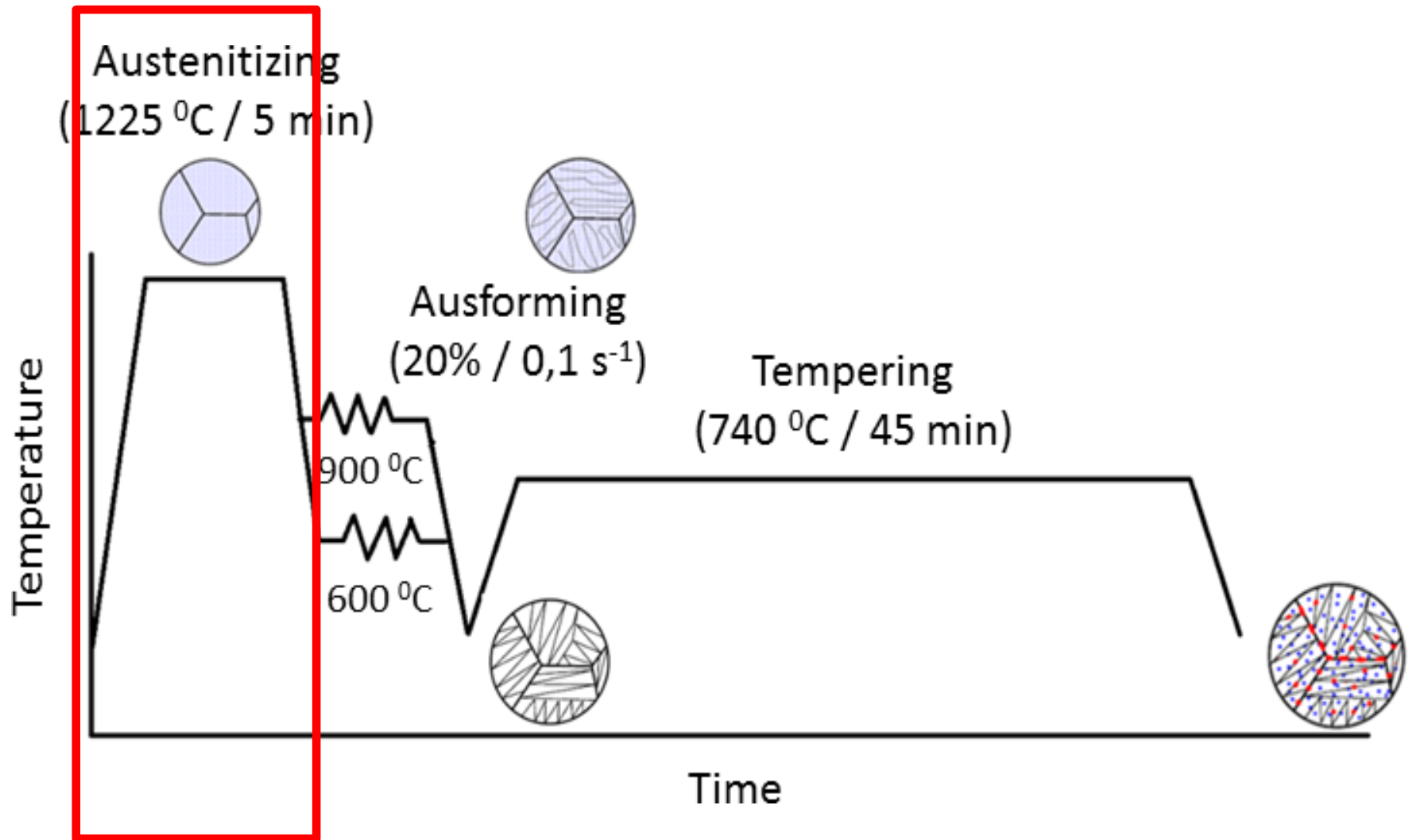
G91 – Reference state (AR condition)



G91 – Thermo-mechanical treatment

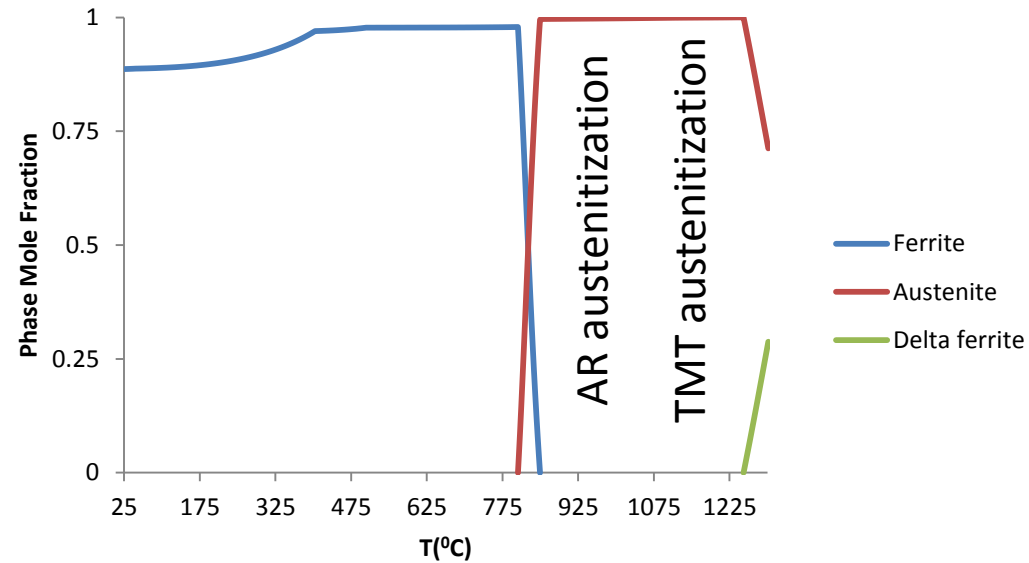
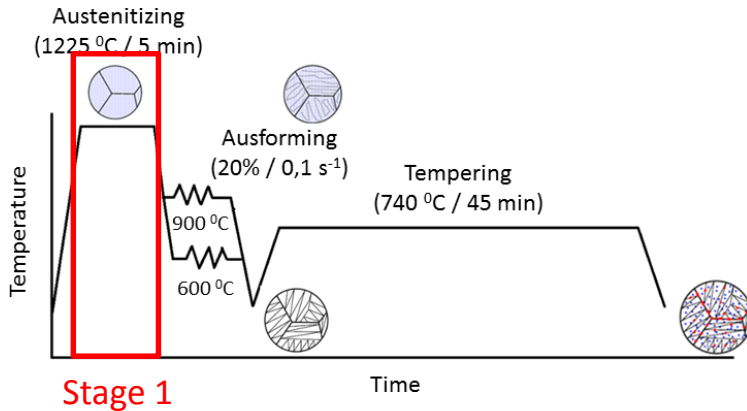


G91 – Thermo-mechanical treatment



G91 – Thermo-mechanical treatment

High Austenitization Temperature (HAT)



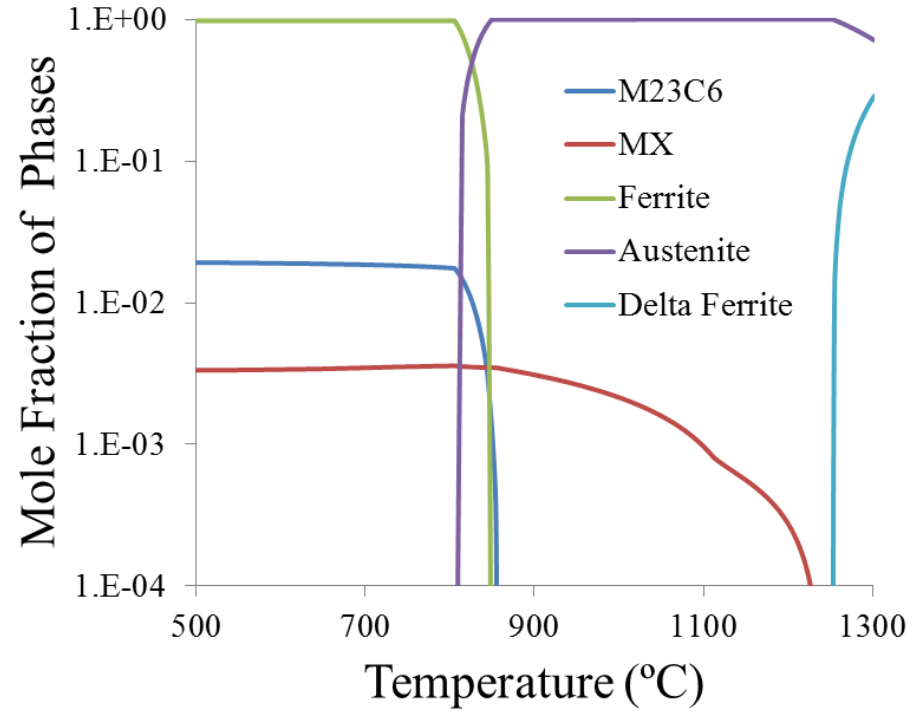
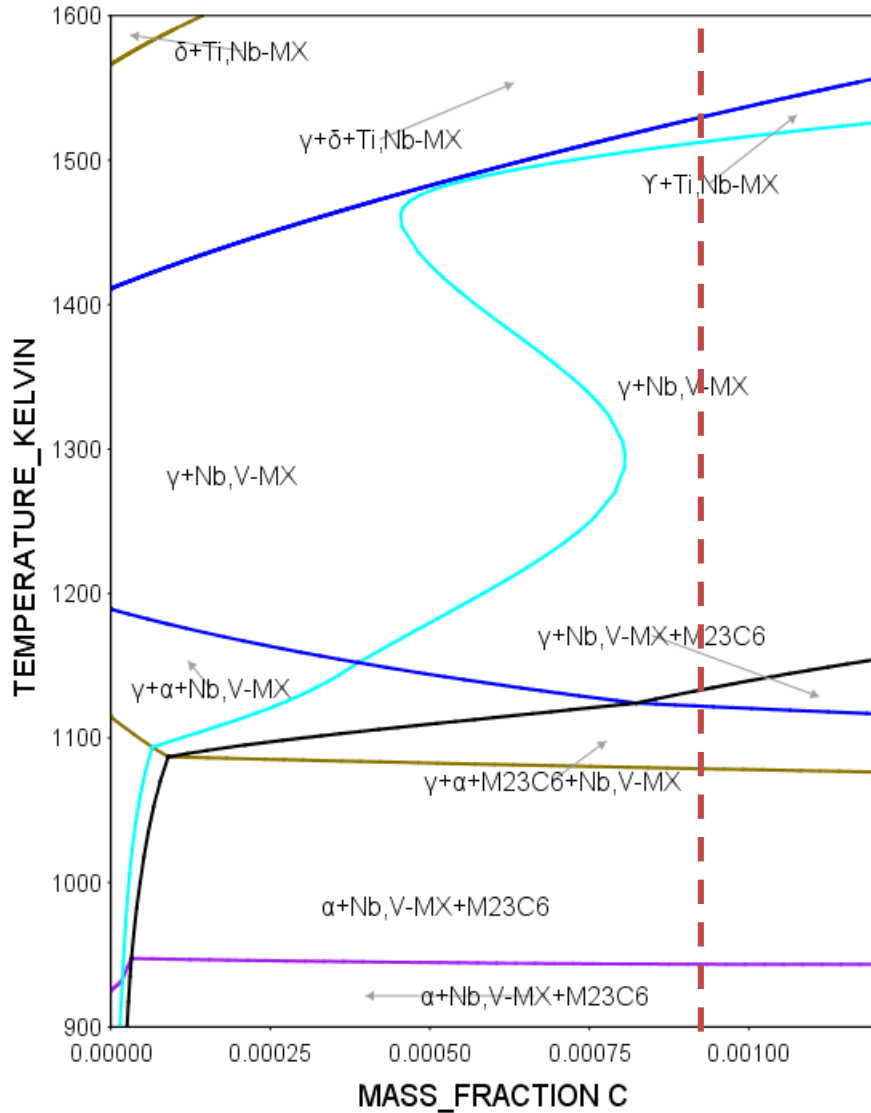
- ❑ Reach full solid-solution
- ❑ Control precipitation of MX using ausforming and tempering
- ❑ Avoid delta ferrite formation. Delta ferrite deteriorates creep properties
- ❑ Achieve higher grain/block size

G91 – Phase Diagram

2015.09.22.13.07.05

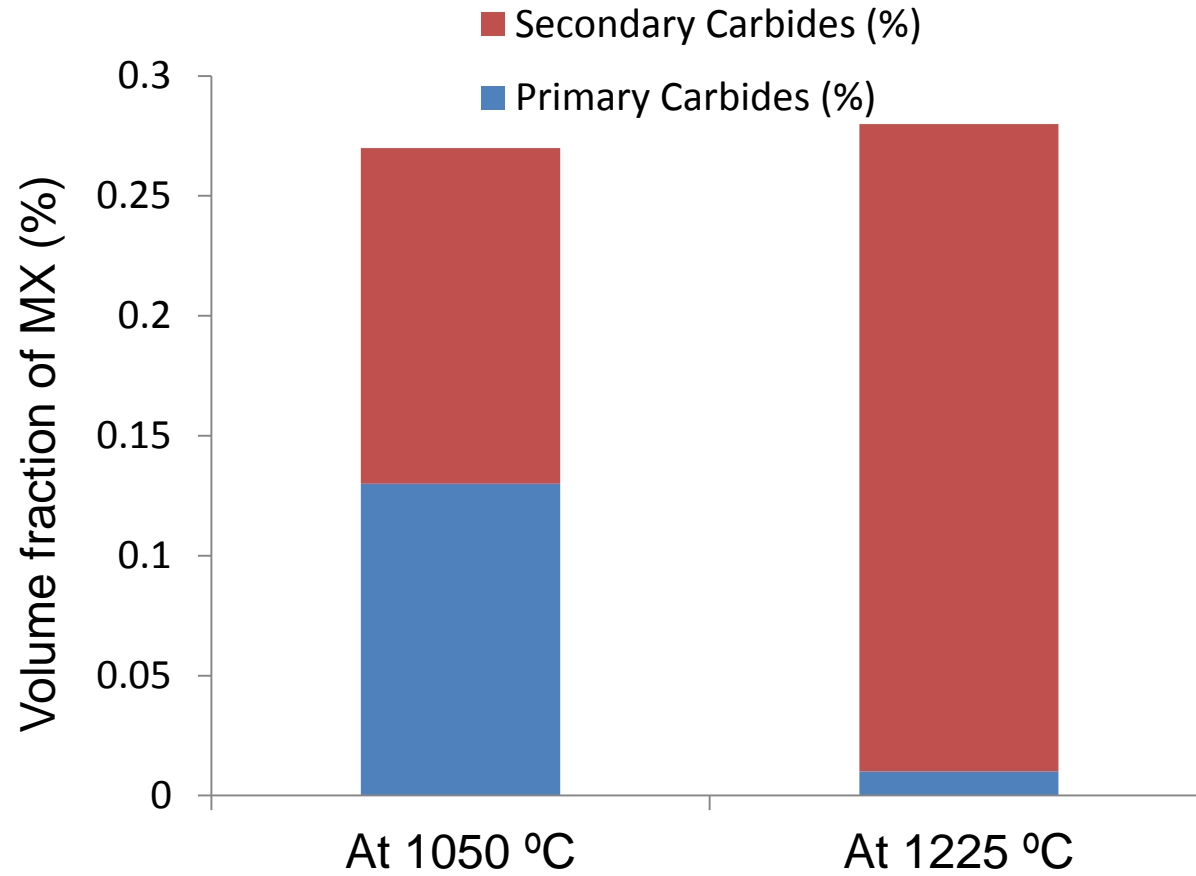
TCFE7: C, CR, FE, MN, MO, N, NB, NI, SI, TI, V

N=1, P=1E5, W(SI)=3.17E-3, W(MN)=5.97E-3, W(NI)=9.9E-4, W(CR)=8.76E-2, W(MO)=8.62E-3, W(V)=1.86E-3, W(NB)=7.3E-4, W(TI)=3E-5, W(N)=3.73E-4



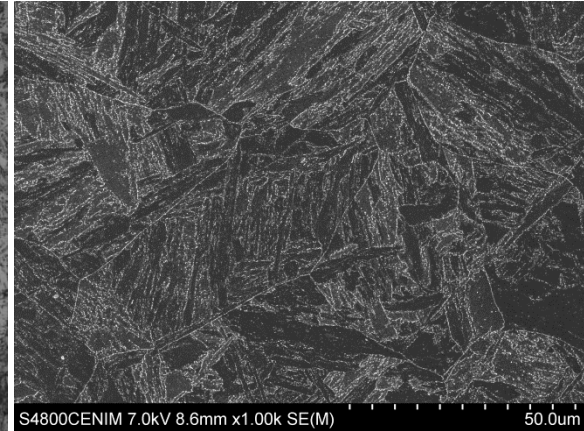
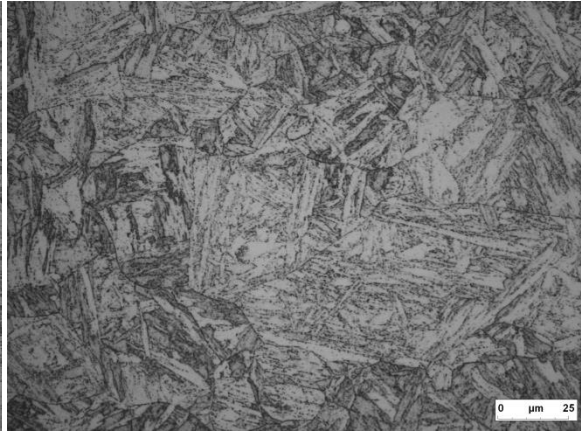
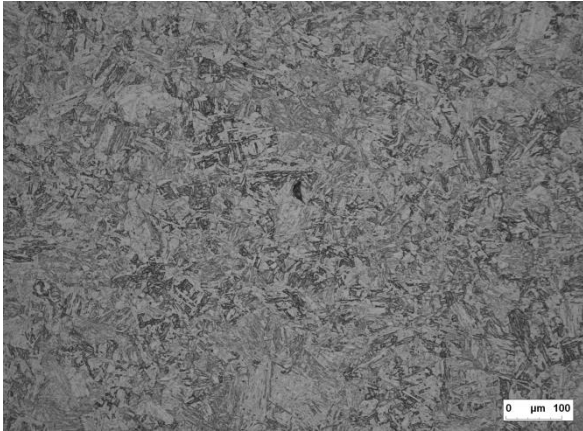
Thermo-Calc TCFE7 database

G91 – HAT

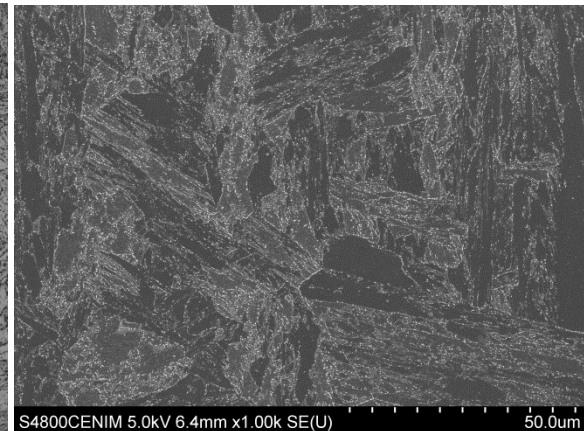
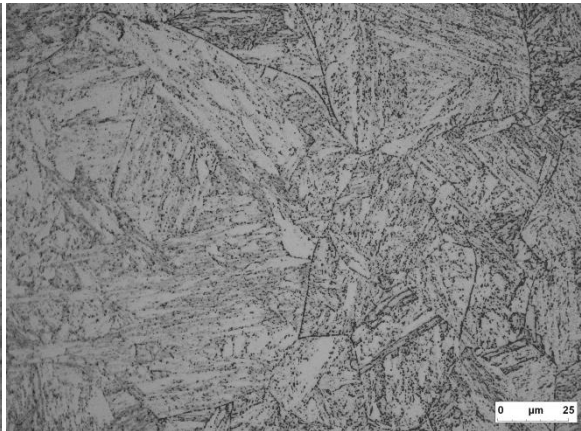
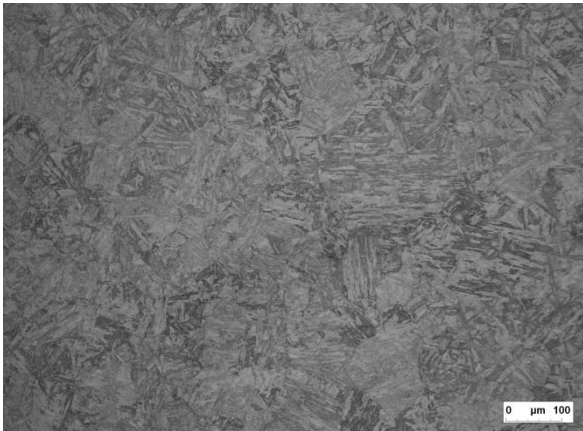


G91 – HAT

AR

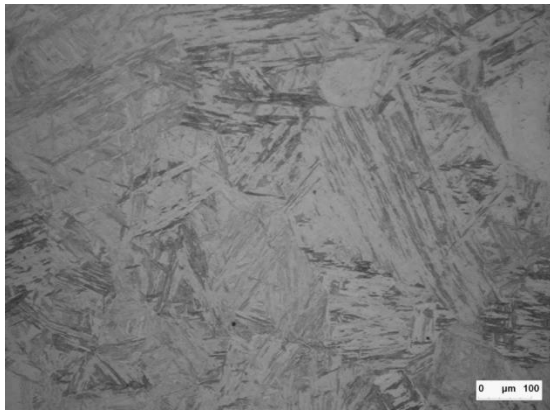
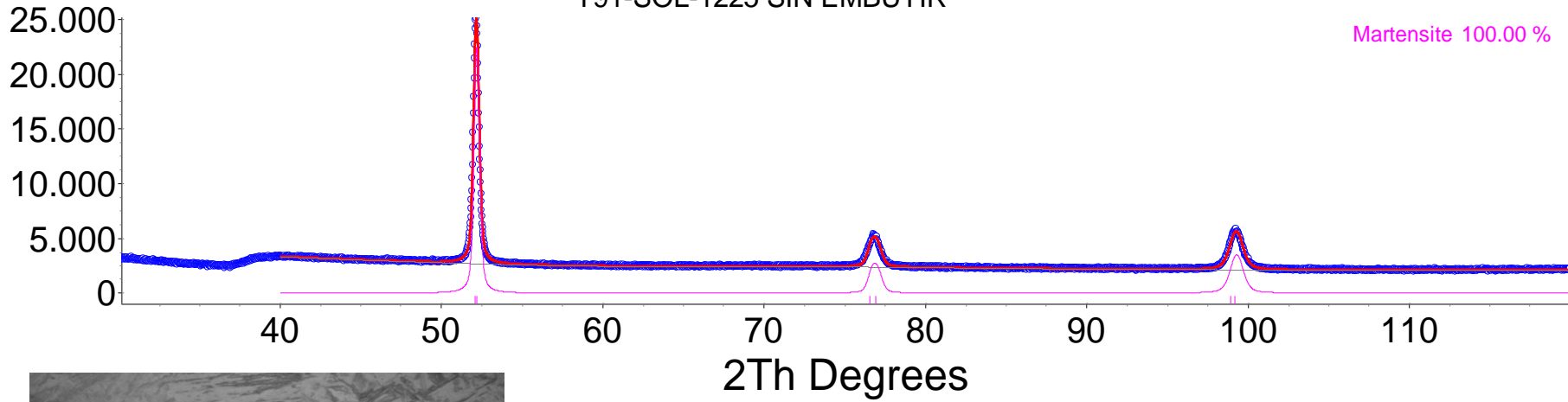


HAT

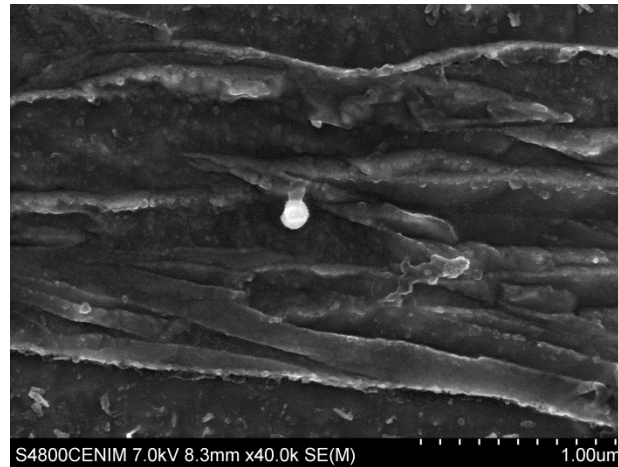
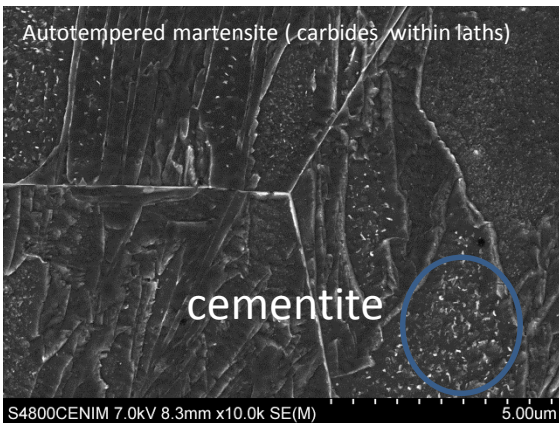


G91 – HAT

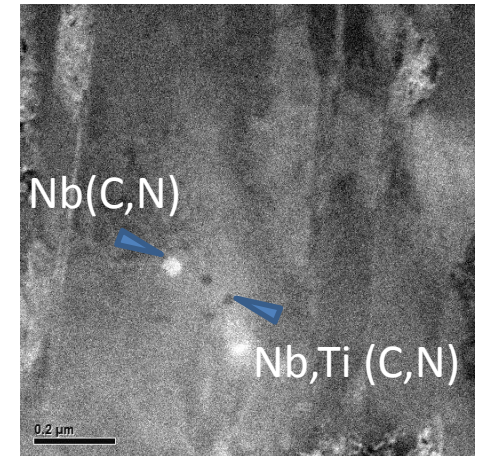
T91-SOL-1225 SIN EMBUTIR

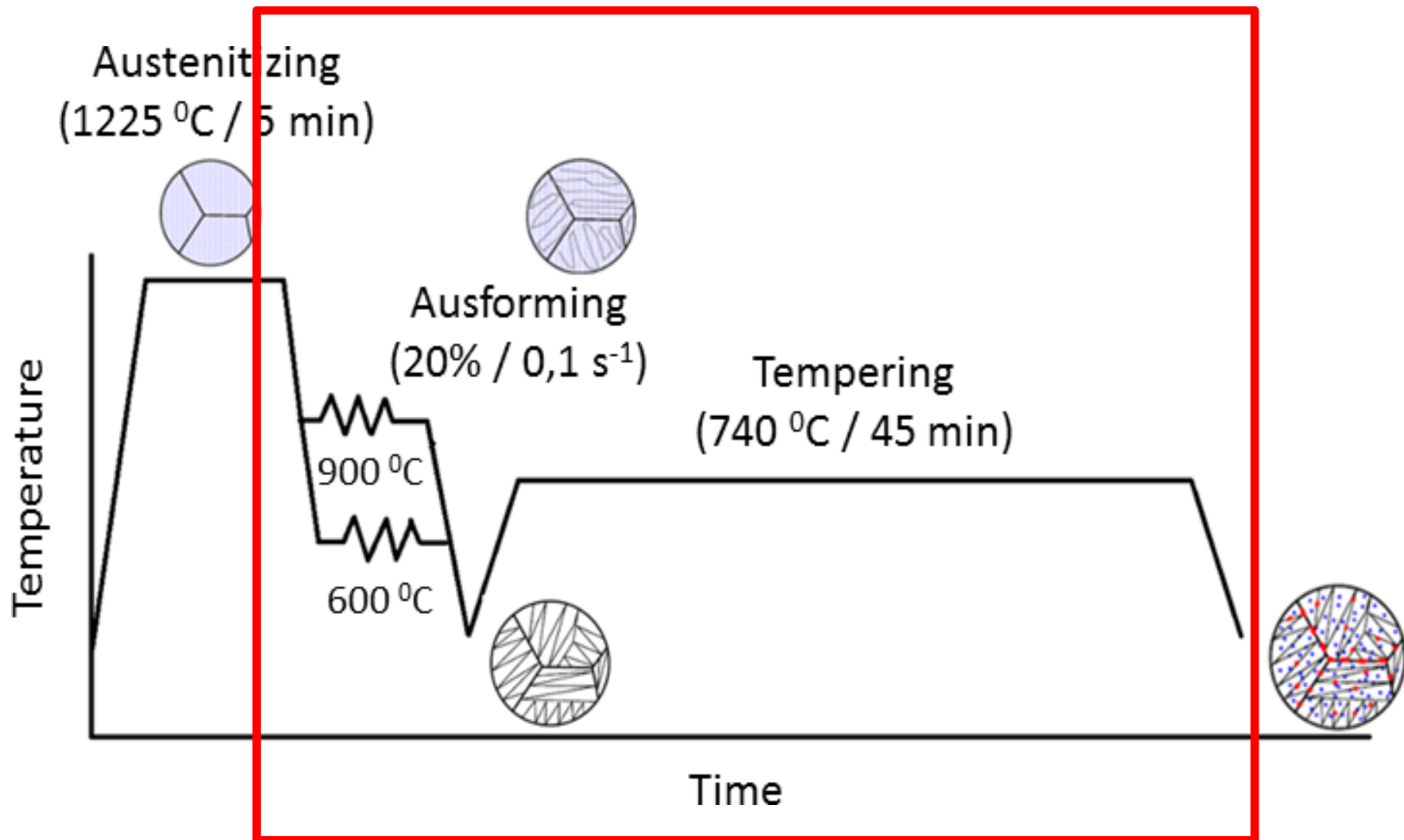


Autotempered martensite (carbides within laths)



Primary carbides (50 -80 nm)





G91 – Thermo-mechanical treatment

Effect of Ausforming Temperature (TMT_T)

Effect of Ausforming Strain (TMT_S)

Ausforming Temperatures

Reduction in thickness

900 °C	20%	40%	TMT_S
600 °C	20%		

TMT_T

AIM: To increase dislocation density in parent γ and of the resultant α' and refine microstructure. Dislocations act as nucleation sites for precipitates promoting finer precipitation

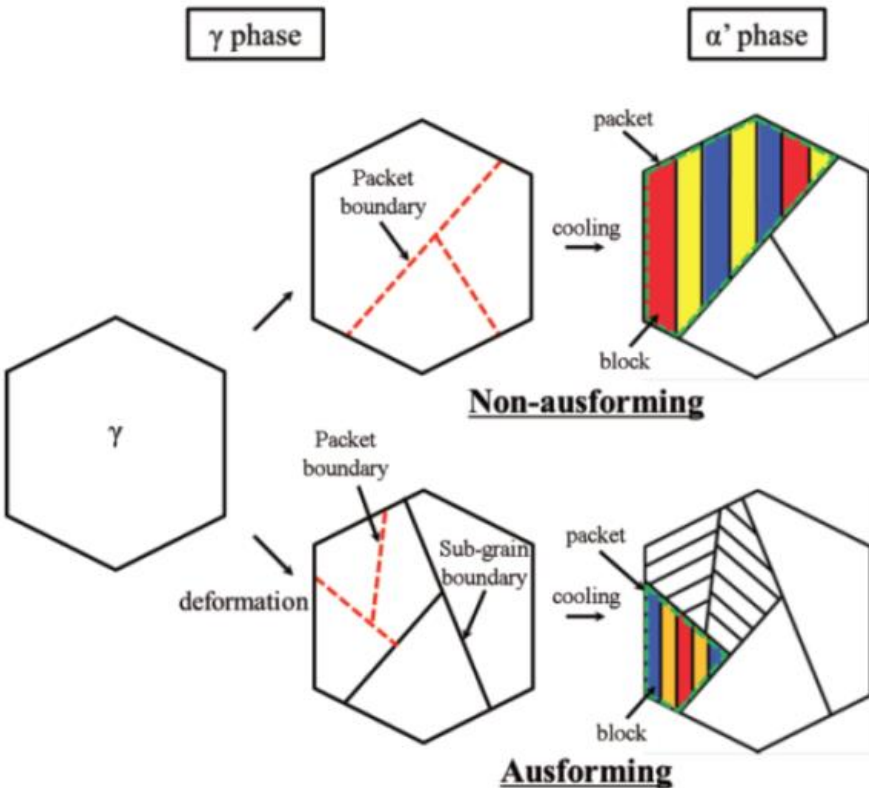
$$T_{NR} = 203 - 310C - 149\sqrt{V} + 657\sqrt{Nb} + 683 e^{-0.36\varepsilon}$$



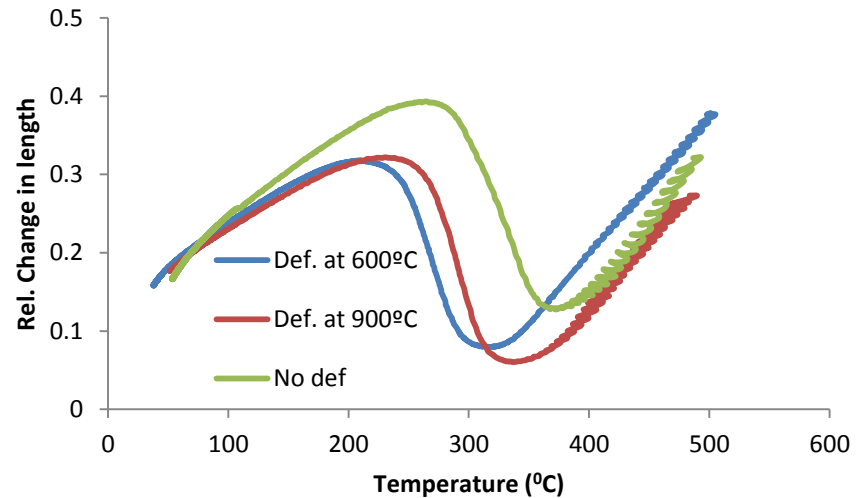
918 °C (20%)

875 °C (40%)

G91 – TMT



SAMPLE	Ms (°C)	Mf (°C)	ΔM (°C)
HAT	385	220	165
TMT _T 900 °C	374	195	179
TMT _T 600 °C	338	145	193

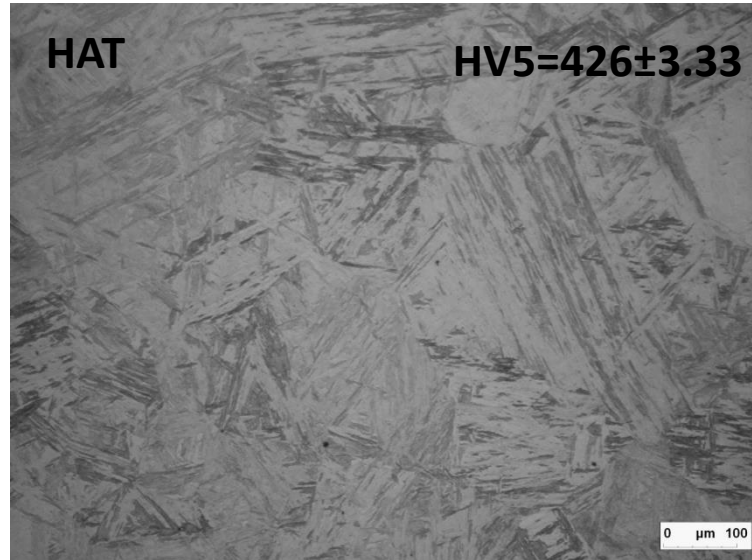


The strengthening of the γ matrix enhances self-accommodation of the transformation strain by formation of different variants (variants selection)

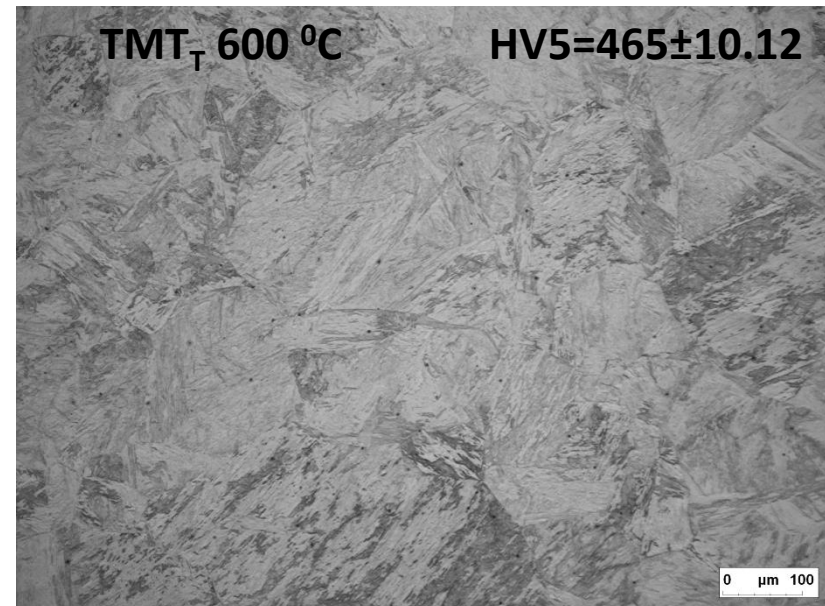
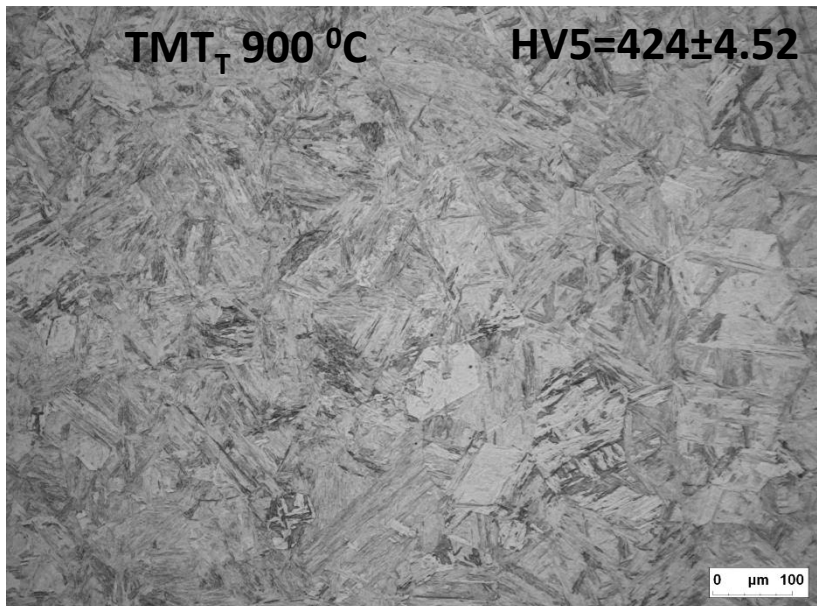
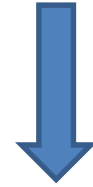
G91 – TMT_T

Matrix after Tempering at 740 °C

Refinement of microstructure because of certain degree of recrystallization



By ausforming, T91 suffers from variant selection, which is different from the unstrained scenario. Martensite with shorter laths is typical in ausformed samples



G91 – TMT

Matrix after Tempering at 740 °C

Prior Austenite Grain Size

Average (μm)

Error (μm)

AR

43.39

4.28

HAT

256.14

6.56

TMT_T 600 °C/20%

259.44

12.64

TMT_T 900 °C/20%

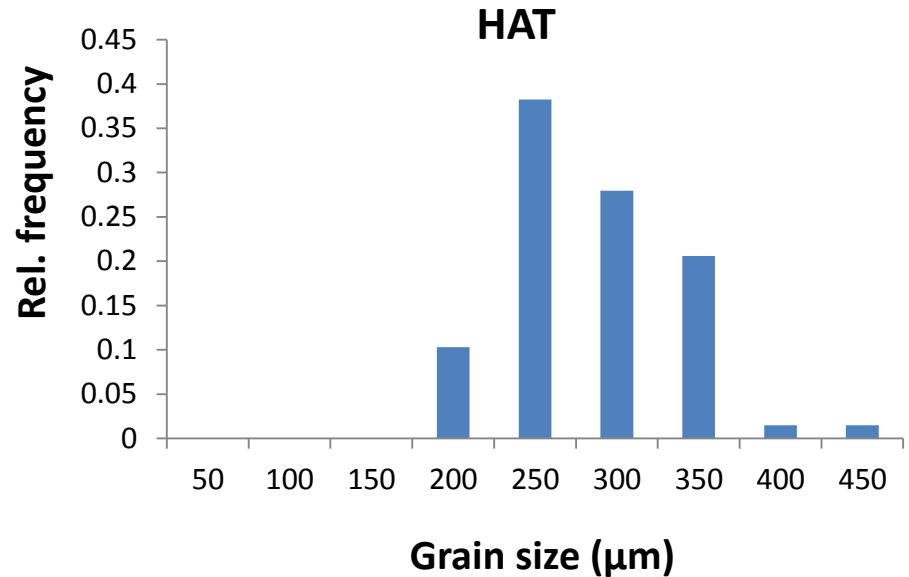
140.87

18.86

TMT_T 900 °C/40%

128.05

17.55



TMT 900 °C/20%

Recrystallization = 23%

TMT 900 °C/40%

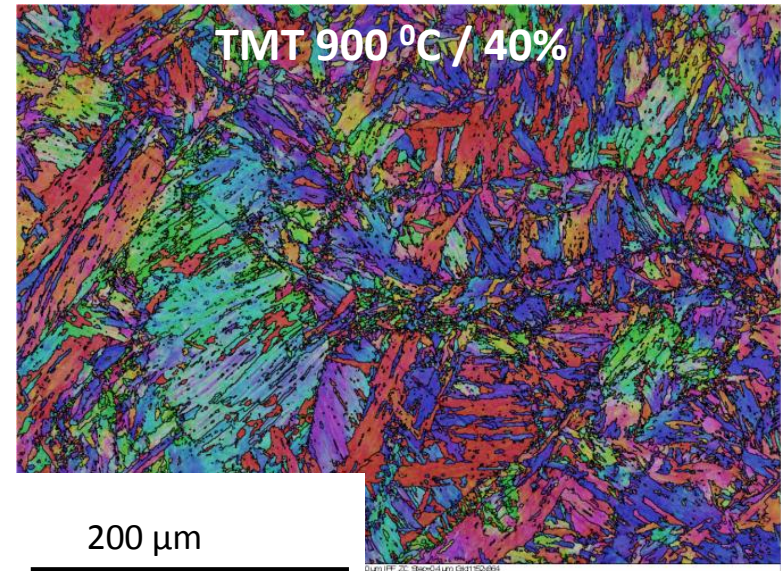
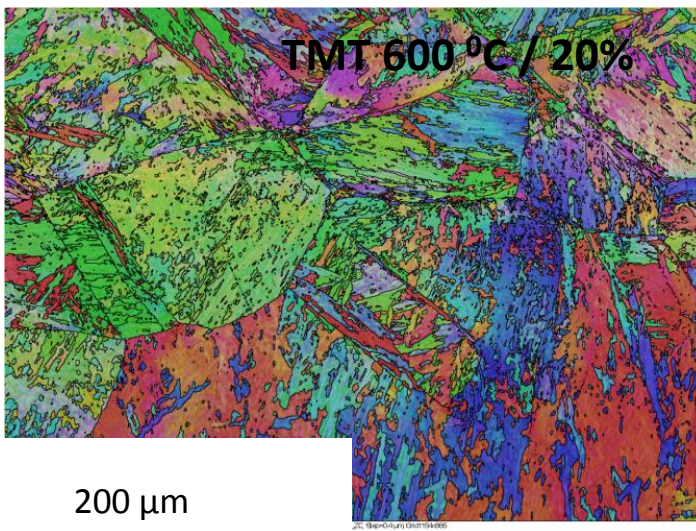
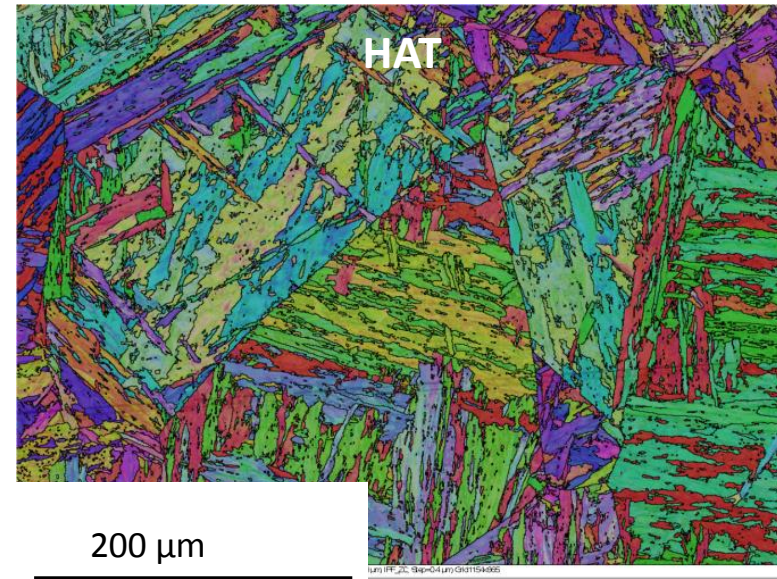
Recrystallization = 50%

G91 – TMT

Matrix after Tempering at 740 °C

	Block width (μm)	Error
AR	2.71	0.23
HAT	4.12	0.37
TMT 600°C 20%	3.91	0.36
TMT 900°C 20%	3.23	0.26
TMT 900°C 40%	3.21	0.27

Deformation of austenite introduce dislocations and these begin to tangle generating cell structure. These cell-structures act as boundaries in deformed austenite grains refining martensite microstructure

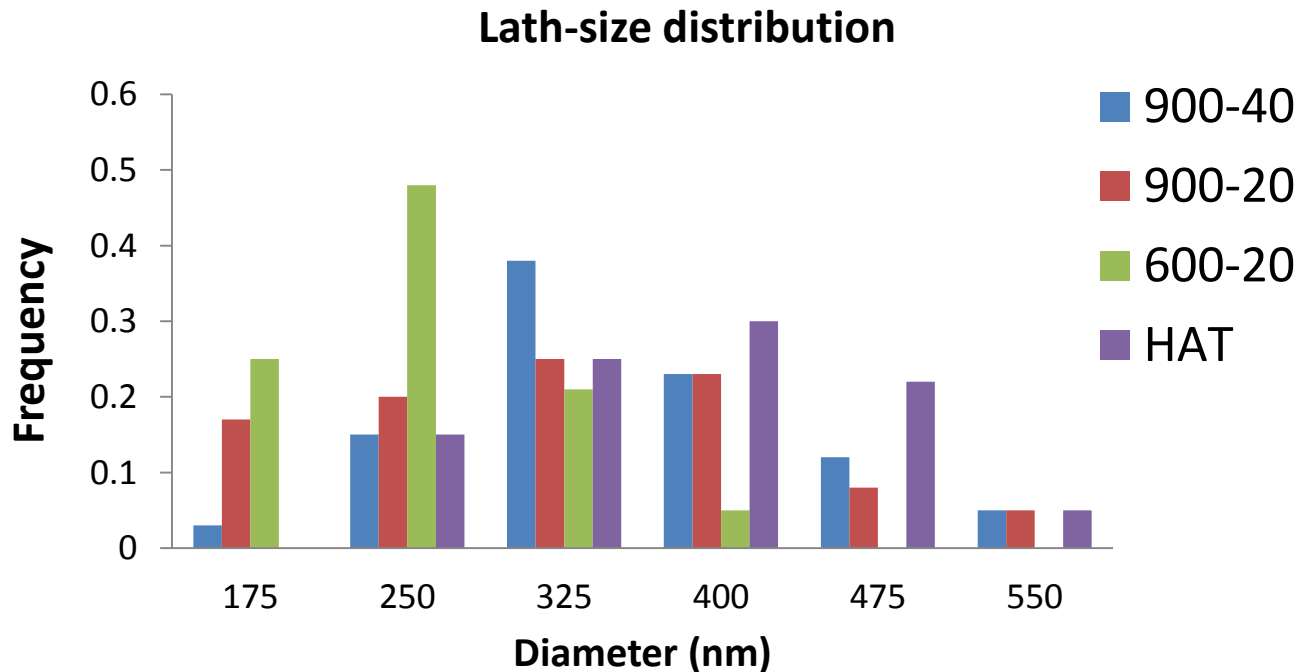


G91 – TMT

Matrix after Tempering at 740 °C

	Lath width (nm)	Error (nm)
AR	356	35
HAT	350	22
TMT 600°C 20%	212	58
TMT 900°C 20%	285	33
TMT 900°C 40%	318	32

Decreasing ausforming temperature produce an reduction in lath width. This fact can be explained because lath width depends on strenght of austenite

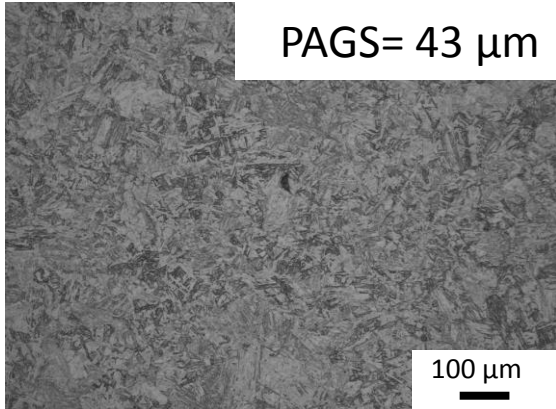


G91 – TMT

Precipitates after Tempering at 740 °C

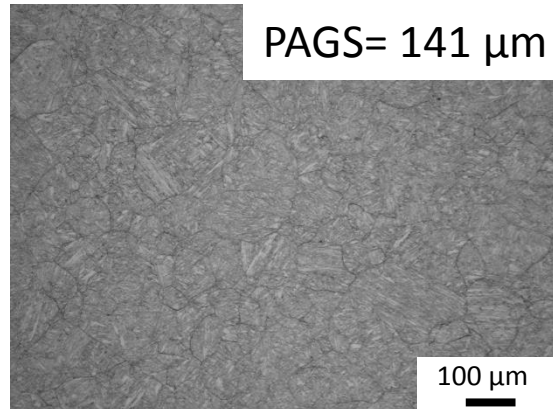
AR

PAGS= 43 μm



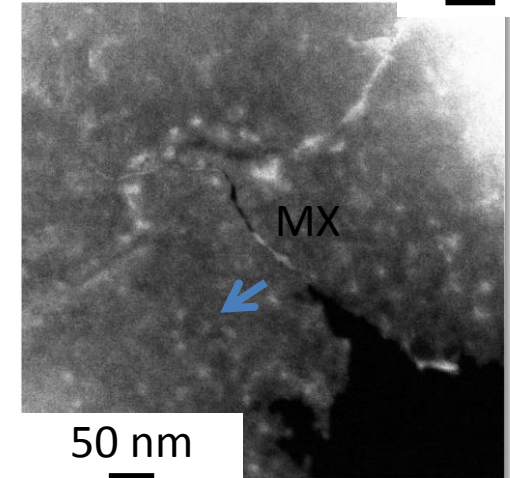
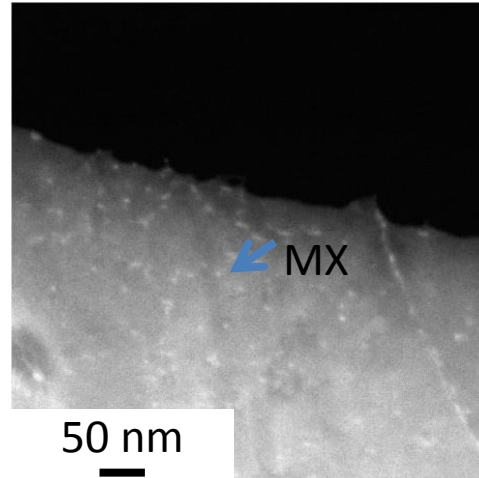
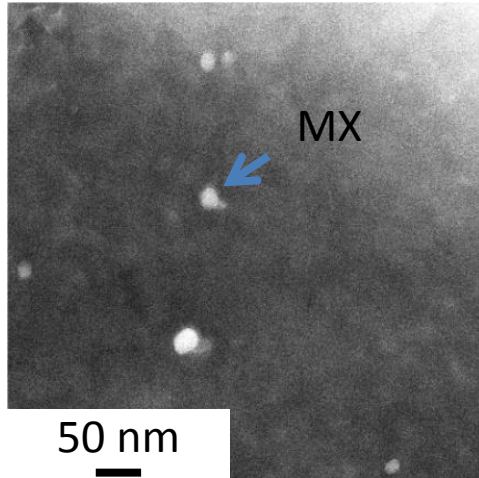
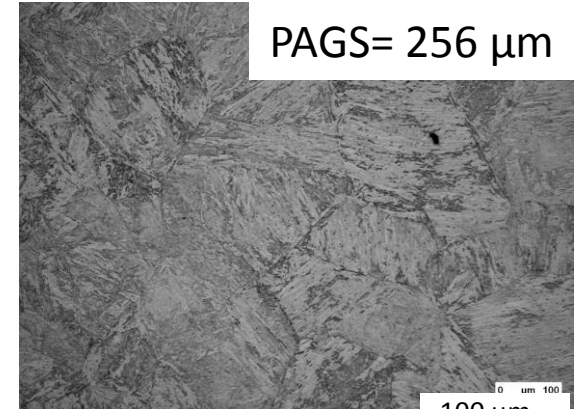
TMT_T 900 °C

PAGS= 141 μm



TMT_T 600 °C

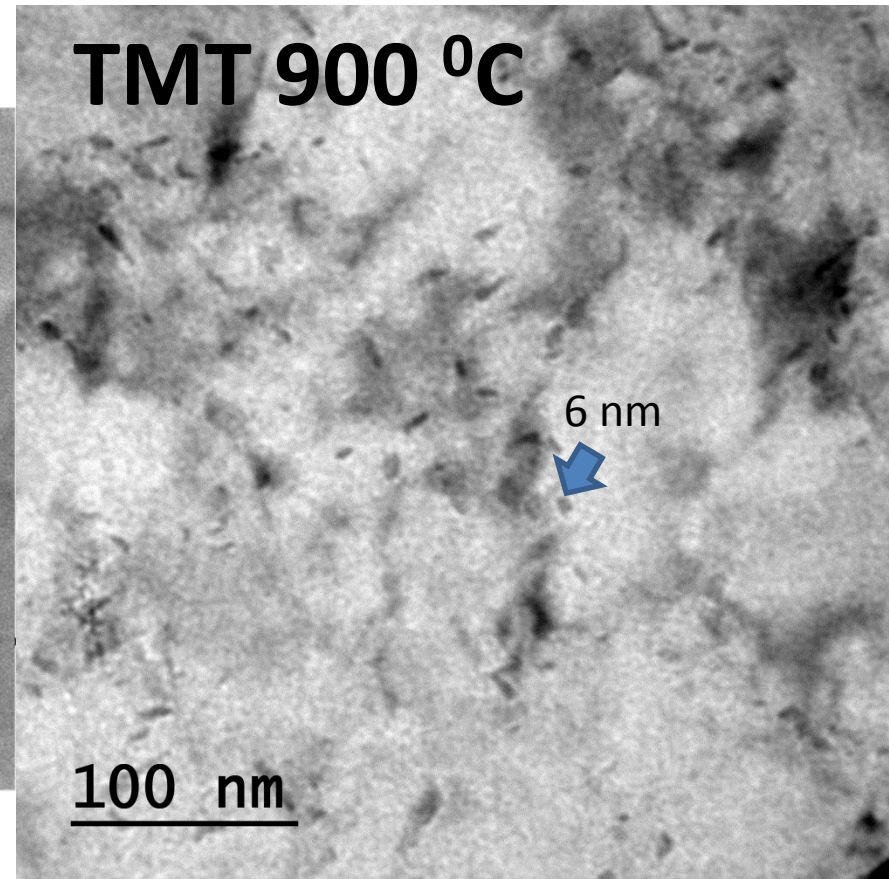
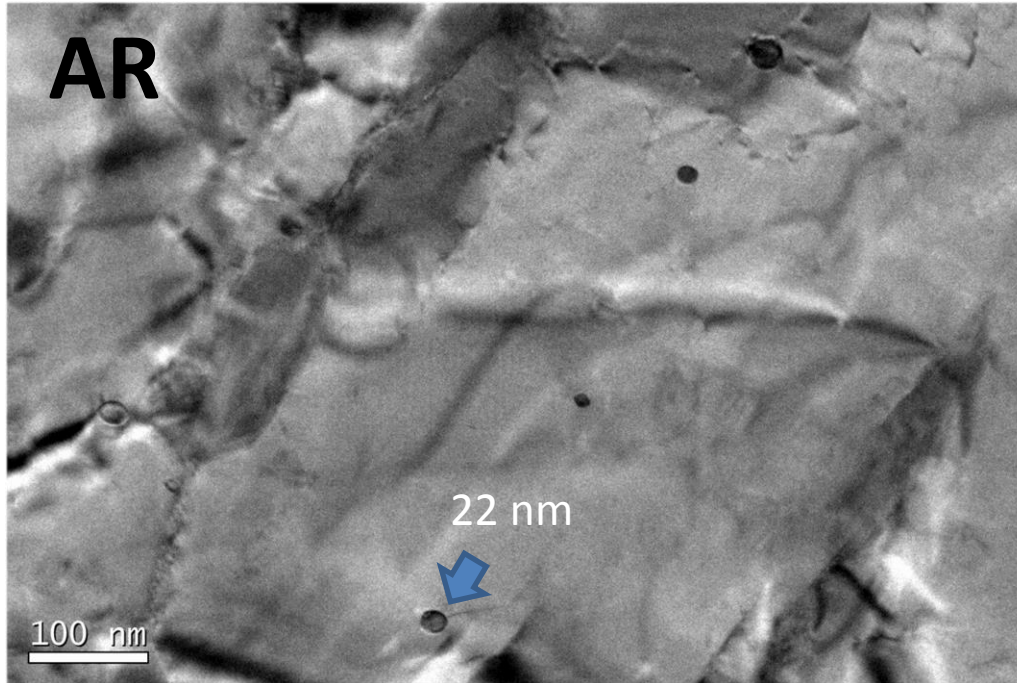
PAGS= 256 μm



Finer precipitation and better distribution of MX precipitates after TMT

G91 – TMT

Precipitates after Tempering at 740 °C

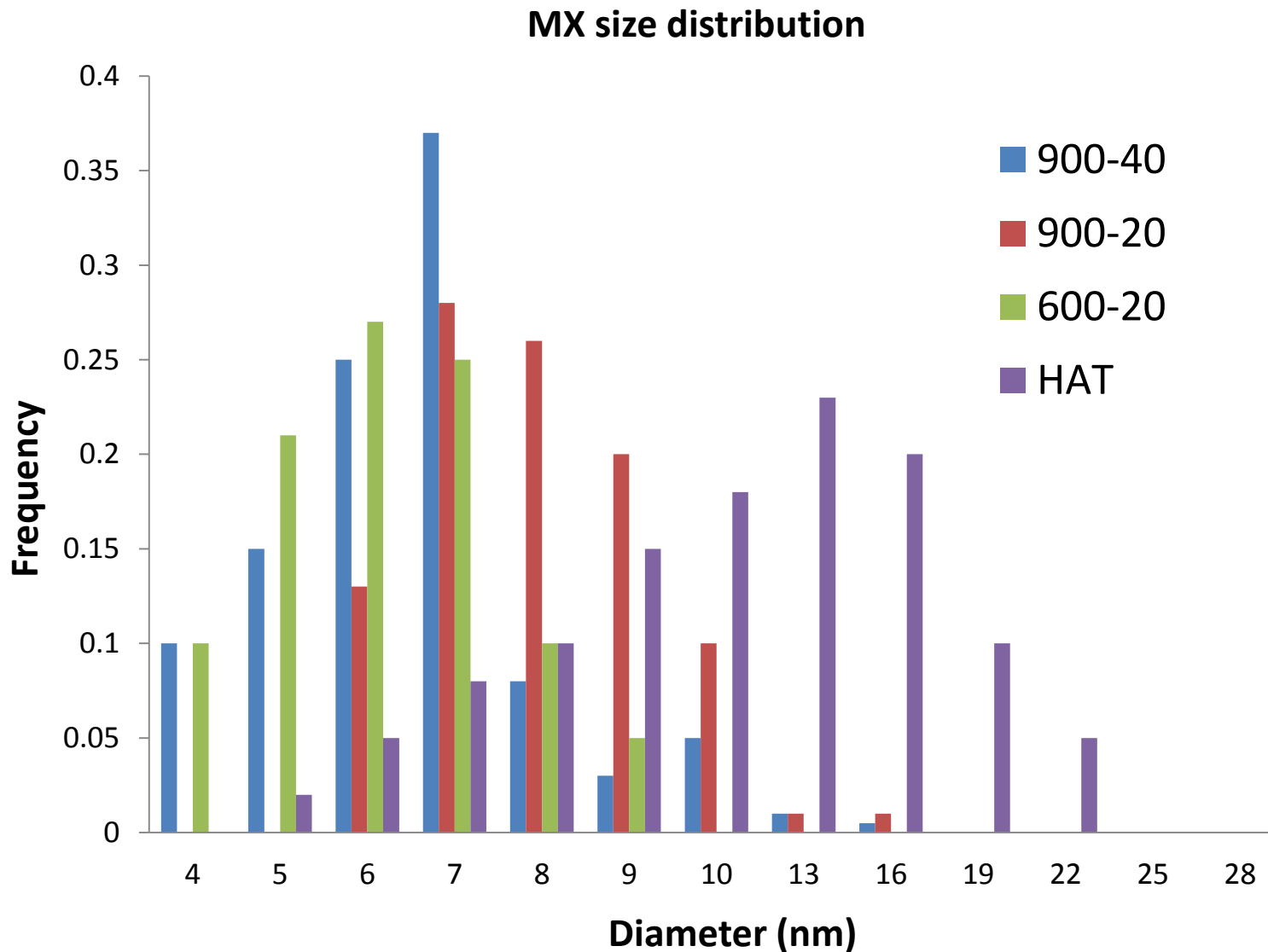


	V (%W)	Cr(%W)	Fe(%W)	Nb(%W)
V,Nb-MX	4.81	9.05	82.02	4.13
Matrix	0.64	12.18	86.24	0.93

V,Nb-MX secondary precipitates

G91 – TMT

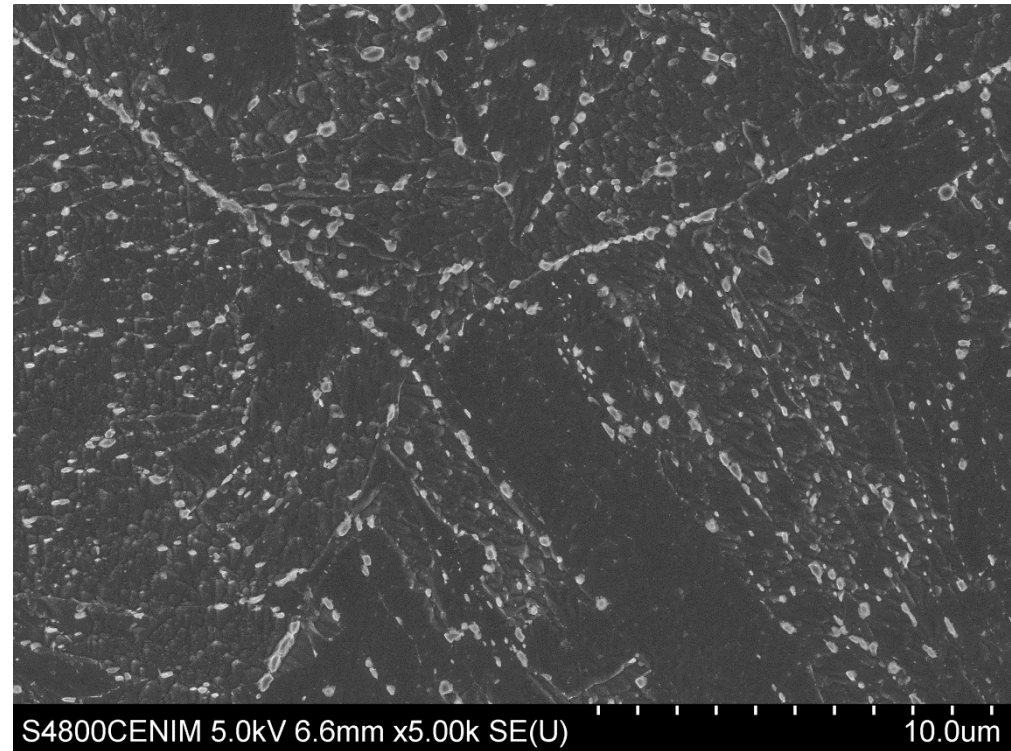
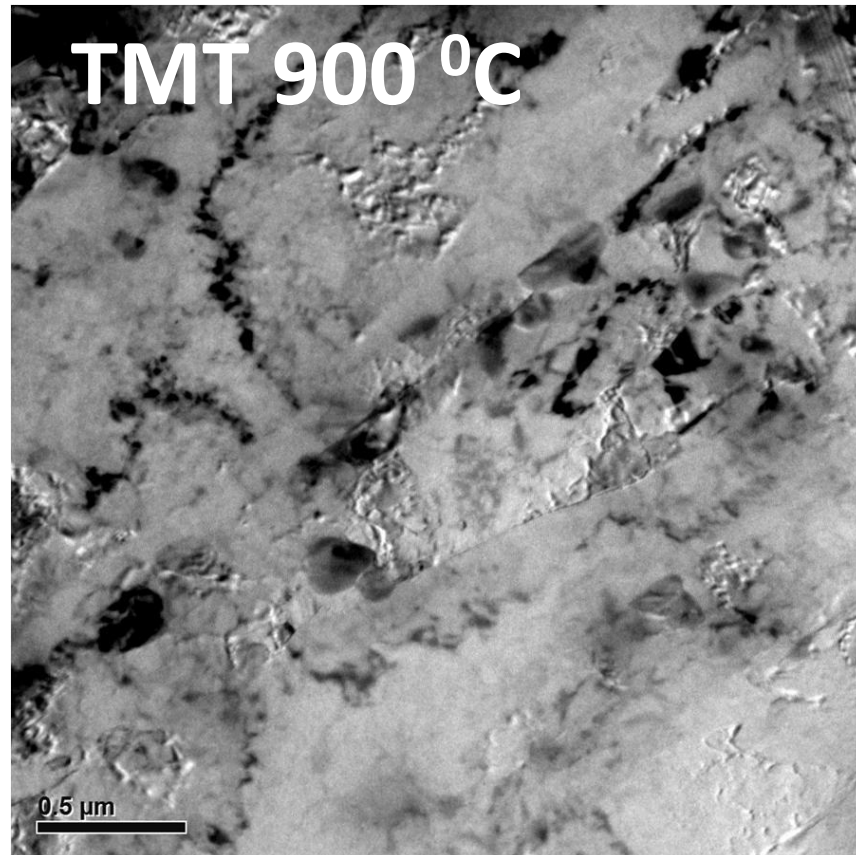
Precipitates after Tempering at 740 °C



G91 – TMT_T

Precipitates after Tempering at 740 °C

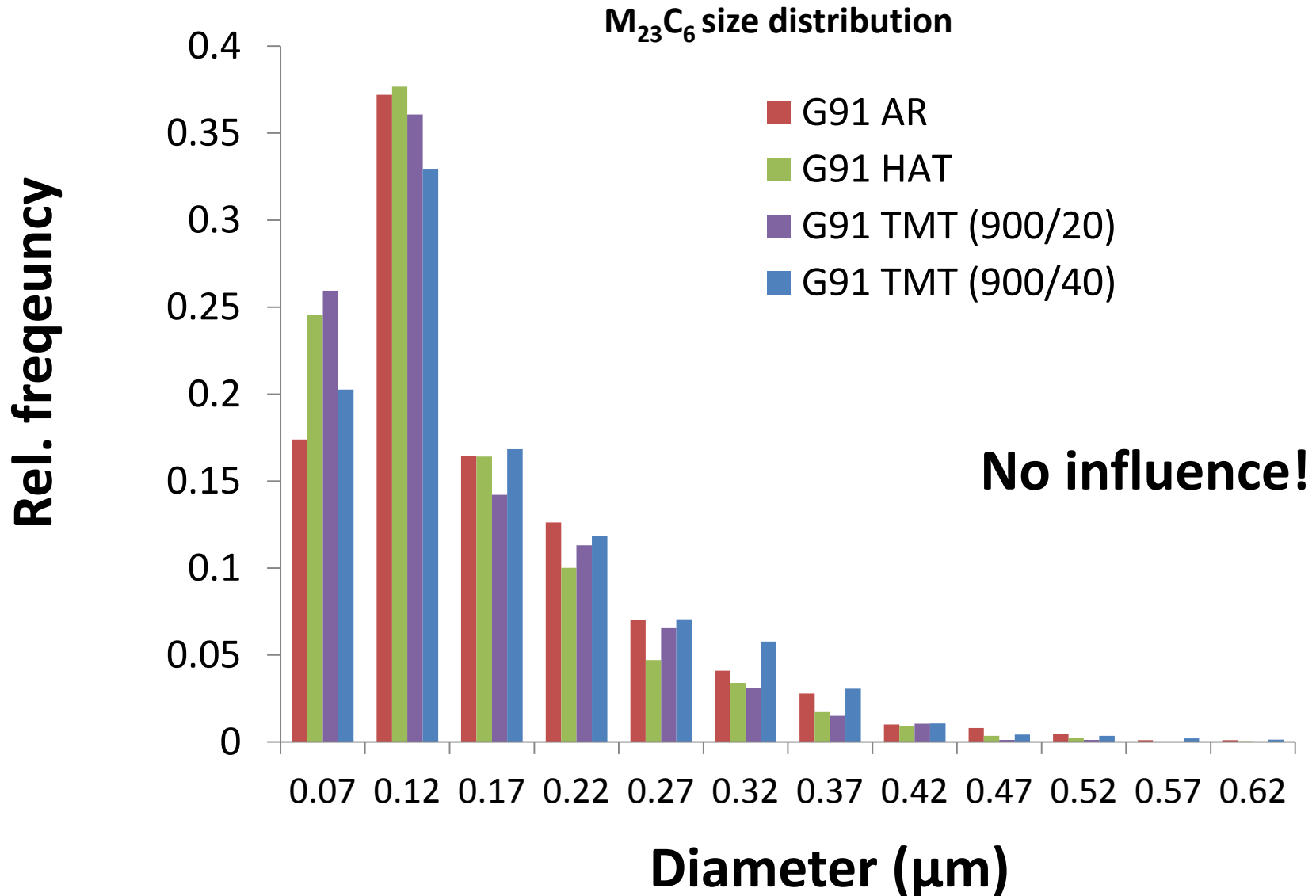
M₂₃C₆ on block and prior grain boundaries after TMT



	V (%W)	Cr(%W)	Fe(%W)	Nb(%W)
M ₂₃ C ₆	0	56.87	43.63	2.16
Matrix	0.64	12.18	86.24	0.93

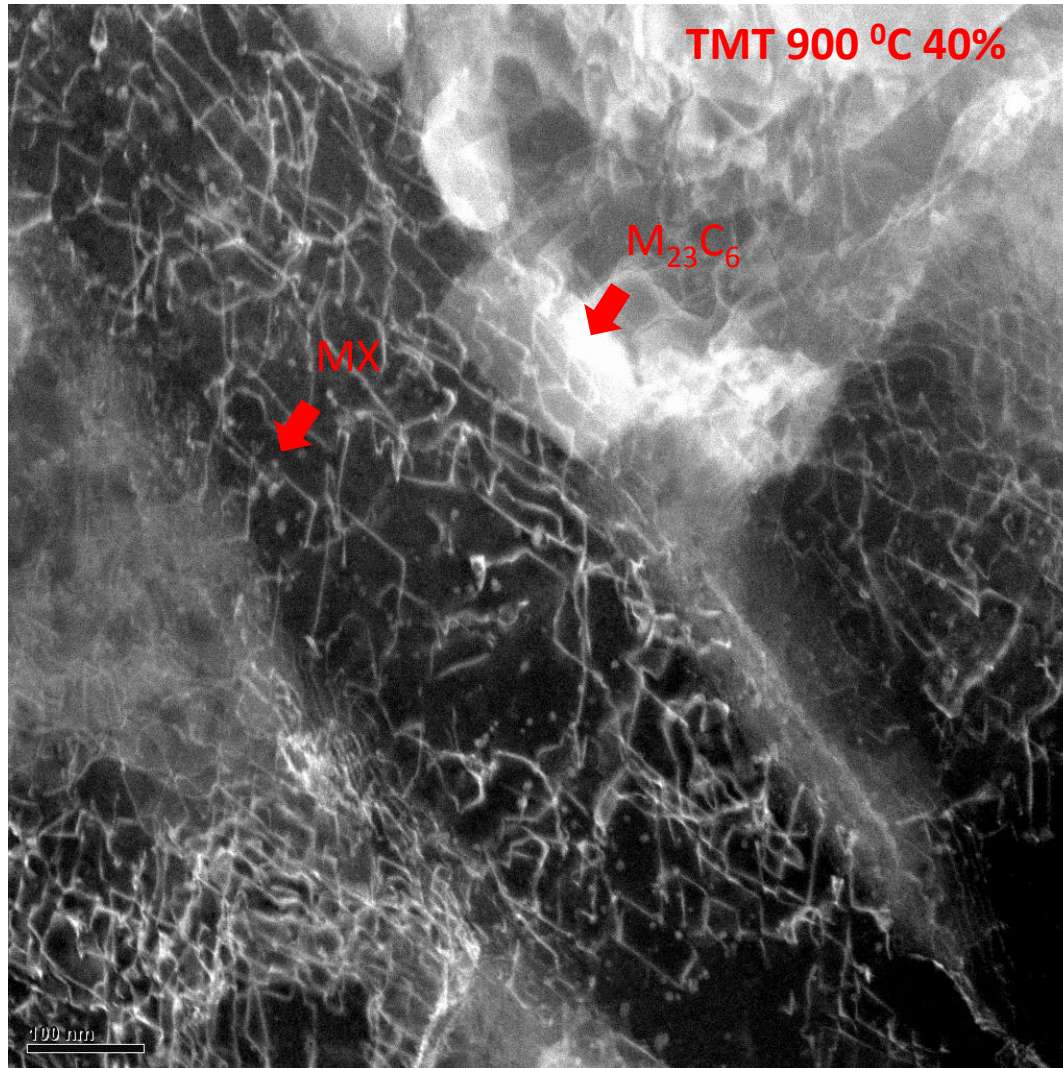
G91 – TMT_T

Precipitates after Tempering at 740 °C



G91 – TMT

Precipitates after Tempering at 740 °C

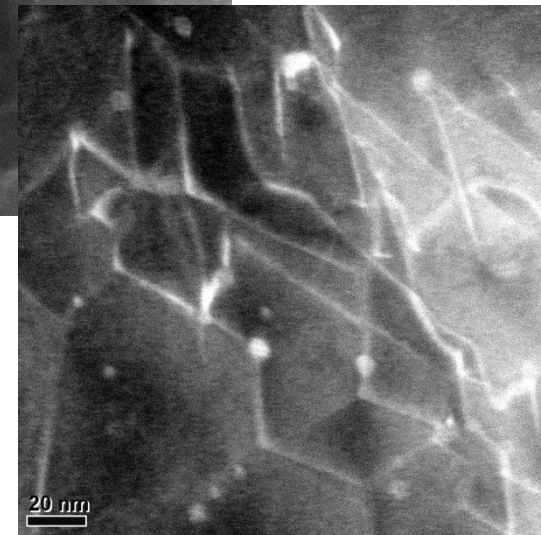
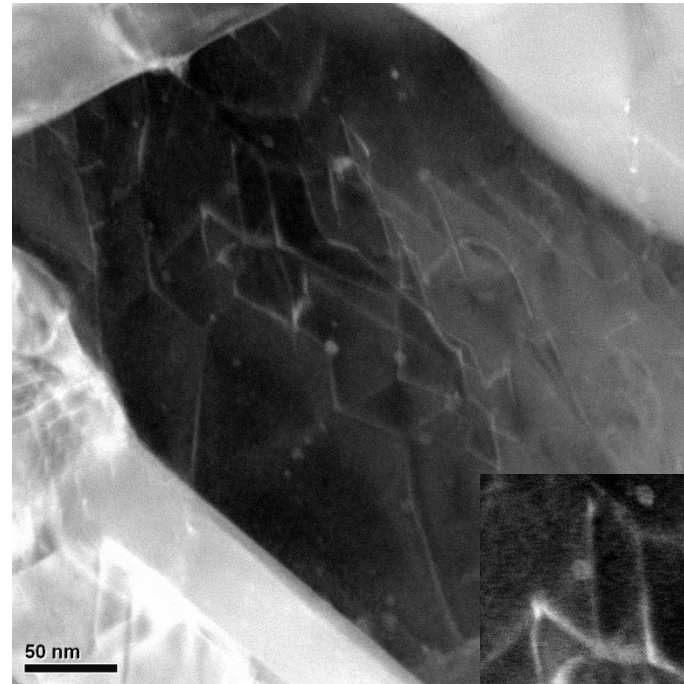
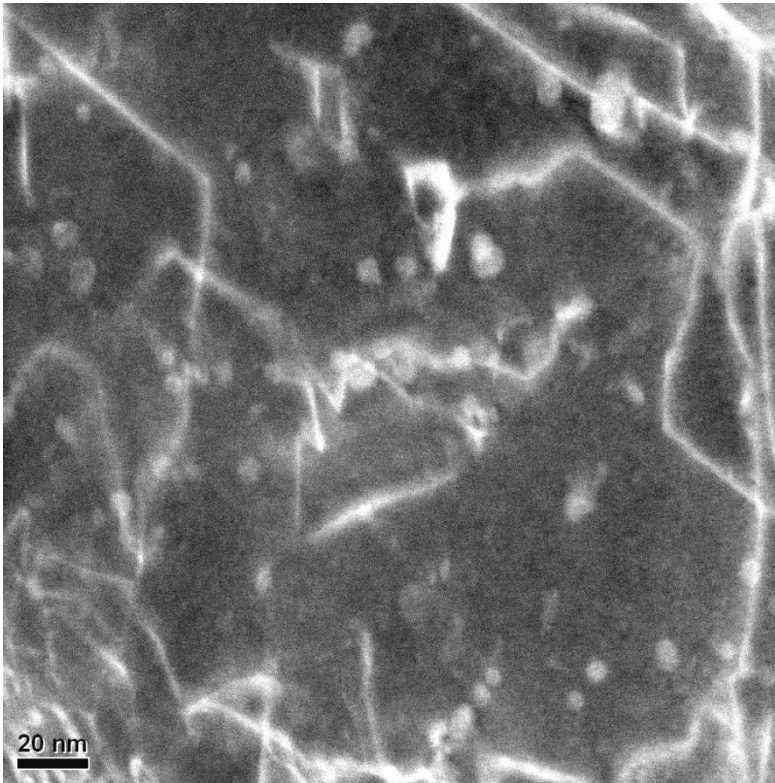


Sample	Precipitate	Diameter (nm)	Number density (m^{-3})
AR	$M_{23}C_6$	141	6.19×10^{19}
	MX	25	8.14×10^{19}
HAT	$M_{23}C_6$	124	8.24×10^{19}
	MX	11.5	7.20×10^{21}
TMT 600 °C	$M_{23}C_6$	136	3.78×10^{19}
	MX	5.59	9.39×10^{22}
TMT900 °C 40%	$M_{23}C_6$	143	4.11×10^{19}
	MX	8.74	1.69×10^{22}
TMT900 °C 20%	$M_{23}C_6$	125	8.50×10^{19}
	MX	7.4	6.7×10^{21}

MX measured within no recrystallised grains

G91 – TMT

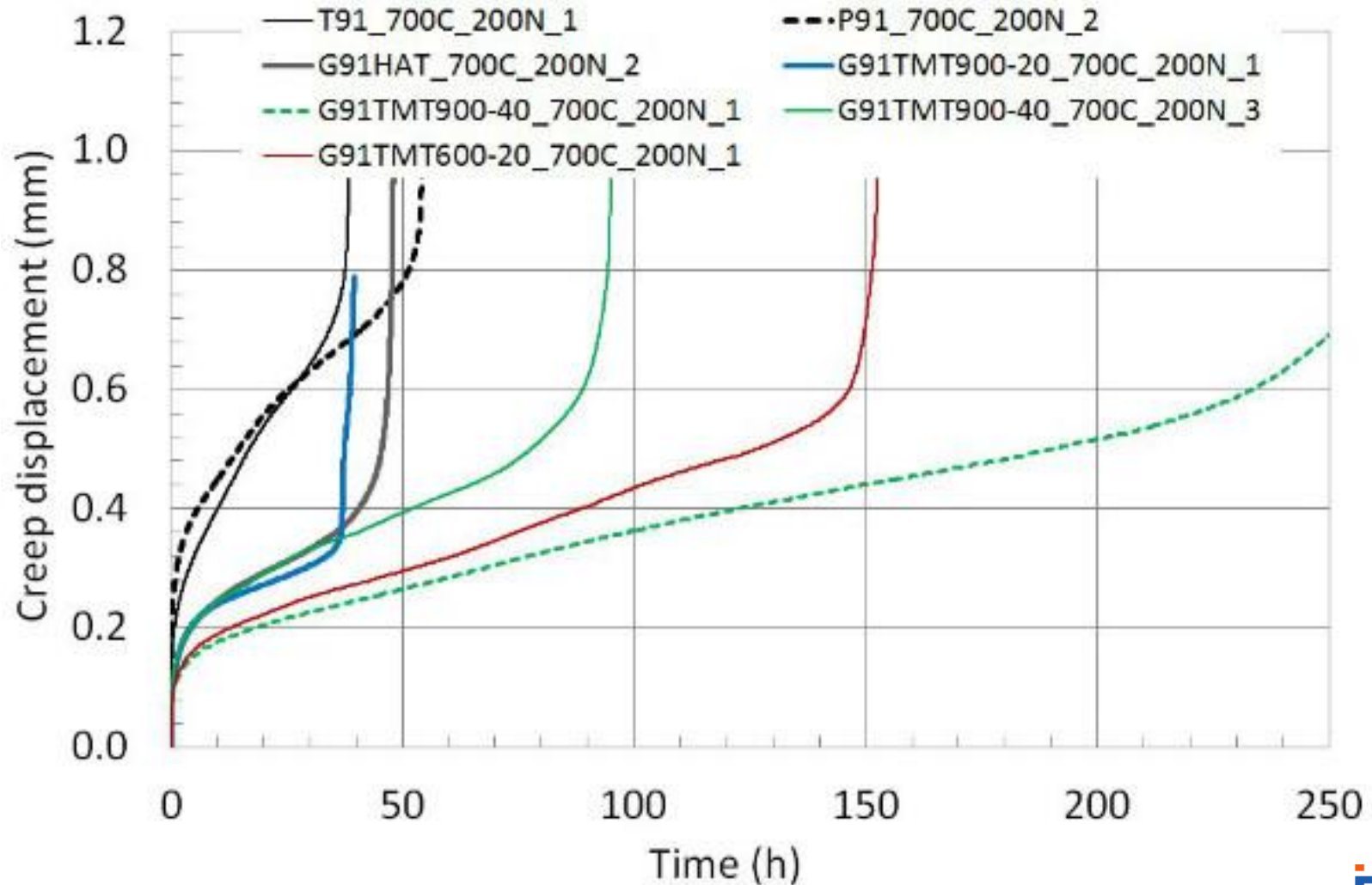
Precipitates after Tempering at 740 °C



- ❑ MX size is up to 7 times smaller than in AR conditions (2 times smaller than when deformation is 20 %
- ❑ No important changes in $M_{23}C_6$ size
- ❑ Number density of MX is up to 3 order of magnitude higher than AR conditions

G91 – TMT

Quick Screening of creep performance: SPC Test



Conclusions

1. Applying a thermomechanical treatment or a conventional treatment increasing austenitization temperature from 1040 to 1225 °C allow increasing number density of MX up to 3 order of magnitude which raise strengthening capability of MX at 700°C up to 6.5 times. These microstructures reduced considerably minimum disk deflection rate and showed greater time to rupture during SPCT carried out at 700°C with a load of 200N.
2. The steel after TMT showed the best creep strength. This result was attributable to its highest number density of MX precipitates. This higher number density of MX precipitates was obtained by the deformation applied in austenite previous tempering.
3. Recrystallization and recovery suffered during the ausforming at 900°C affect the microstructure of the matrix avoid exploiting the potential of this step triggering loss of dislocations which are responsible for nucleation site for MX precipitates.

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