

TMS 2011

Effect of Elastic Stress on Phase Separation in Fe-20%Cr-6%Al-0.5%Ti ODS alloy

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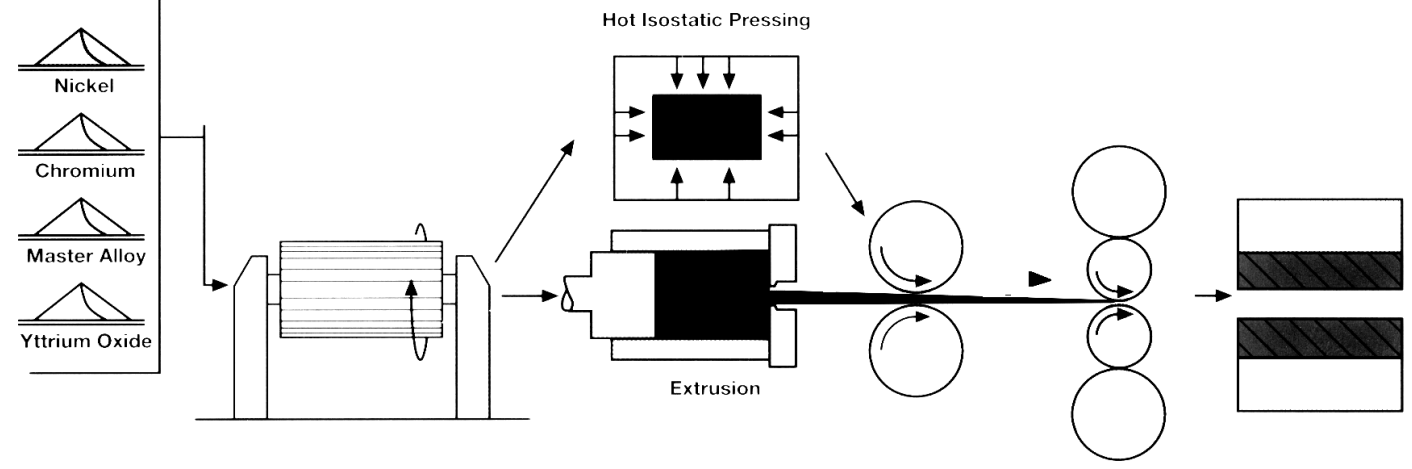
Marta Serrano, Mercedes Hernandez from CIEMAT; Javier Aldazabal from CEIT; and Monica Campos from Carlos III University (UC3) for their help with ODS for nuclear applications

Material

PM 2000tm is a commercial Fe-base ODS alloy manufactured by PLANSEE in Lechbruck, Germany

Chemical composition of PM 2000

	Cr	Al	C	O	N	Ti	Y
wt. %	18.6	5.5	0.04	0.09	0.006	0.54	0.39
at. %	18.5	10.5	0.17	0.28	0.022	0.58	0.23



Powder Raw Materials

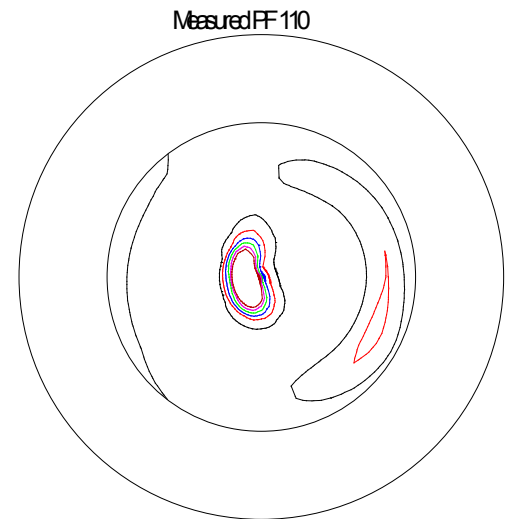
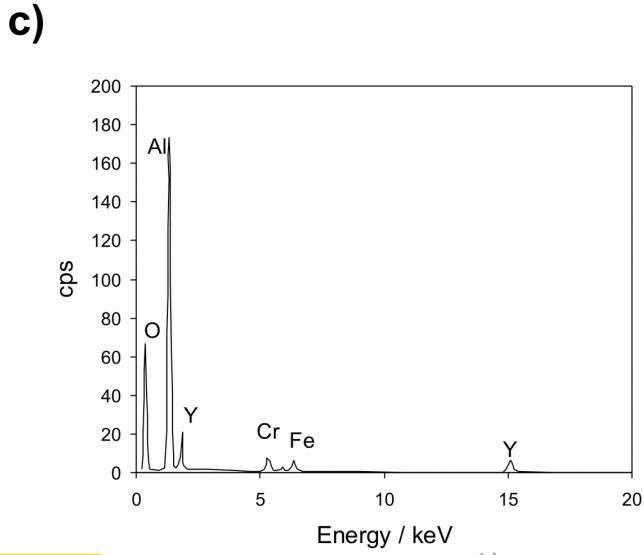
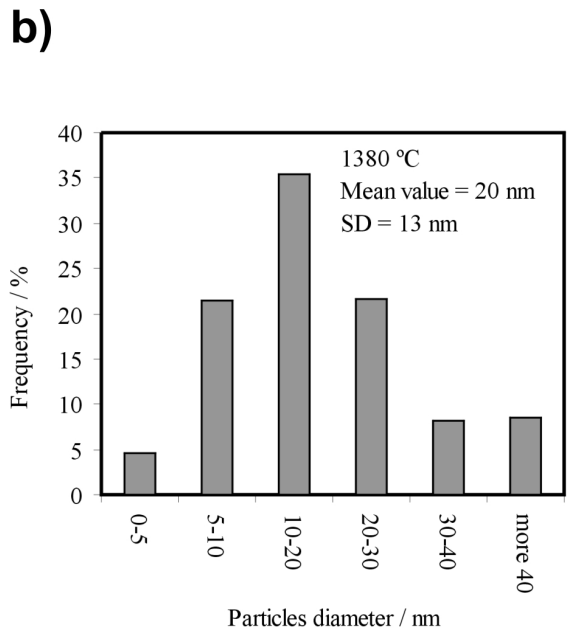
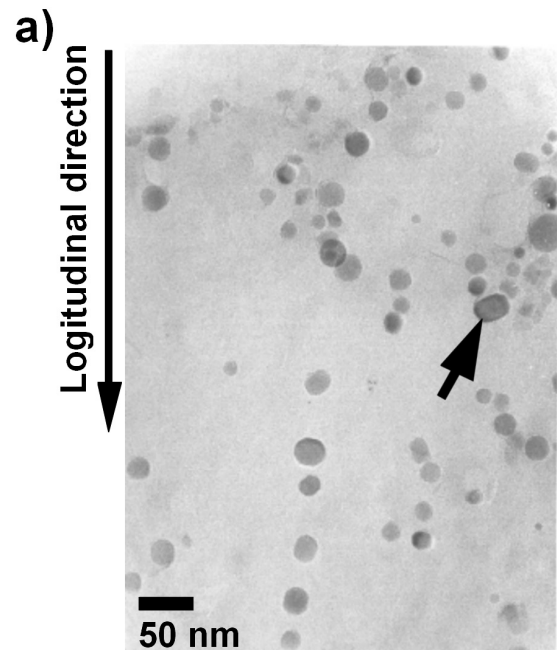
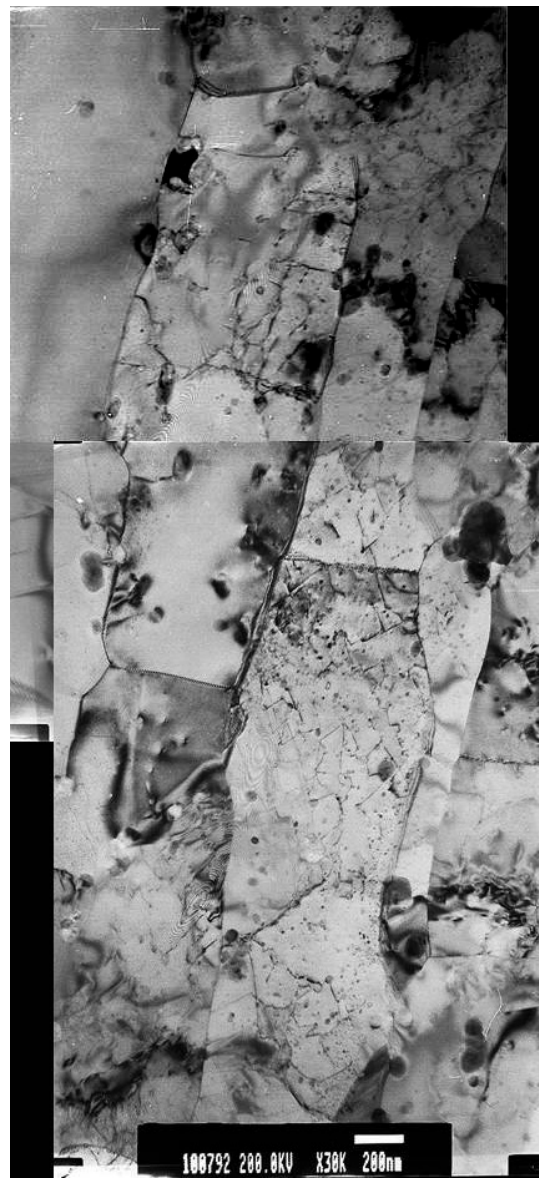
Mechanical Alloying

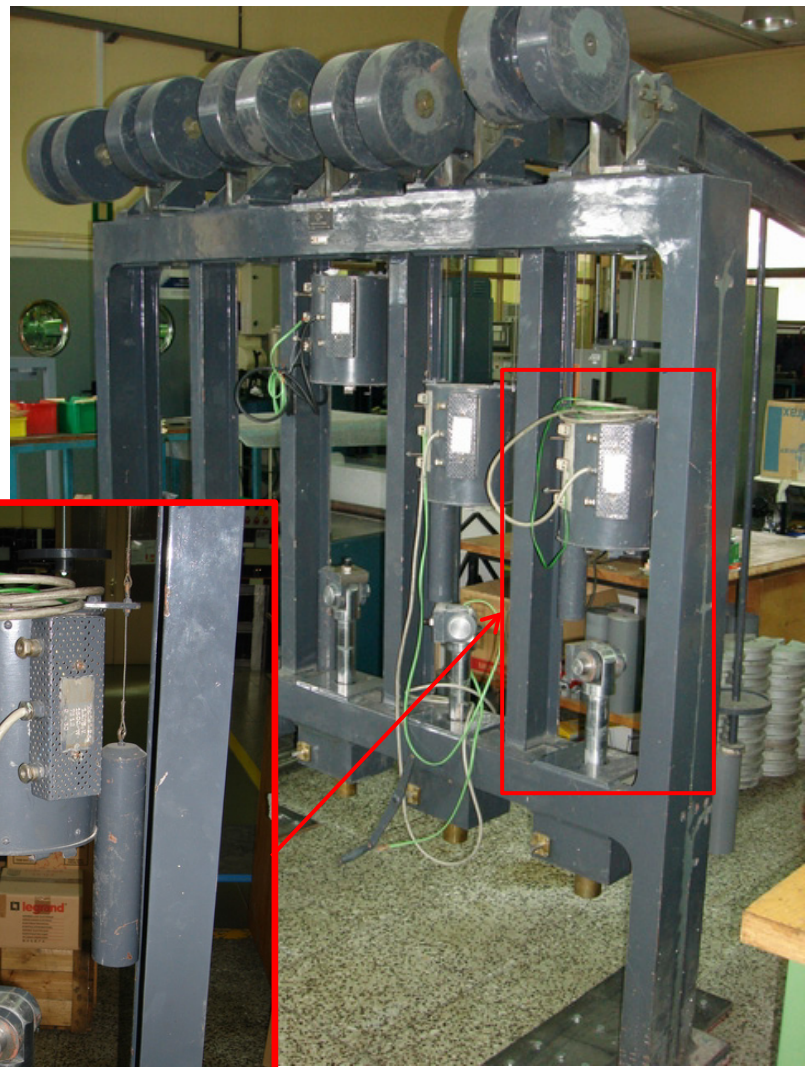
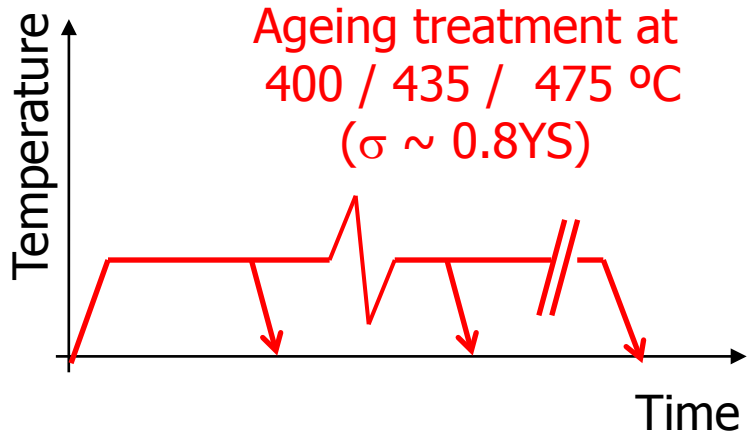
Hot Compaction

Hot Rolling

Cold Rolling

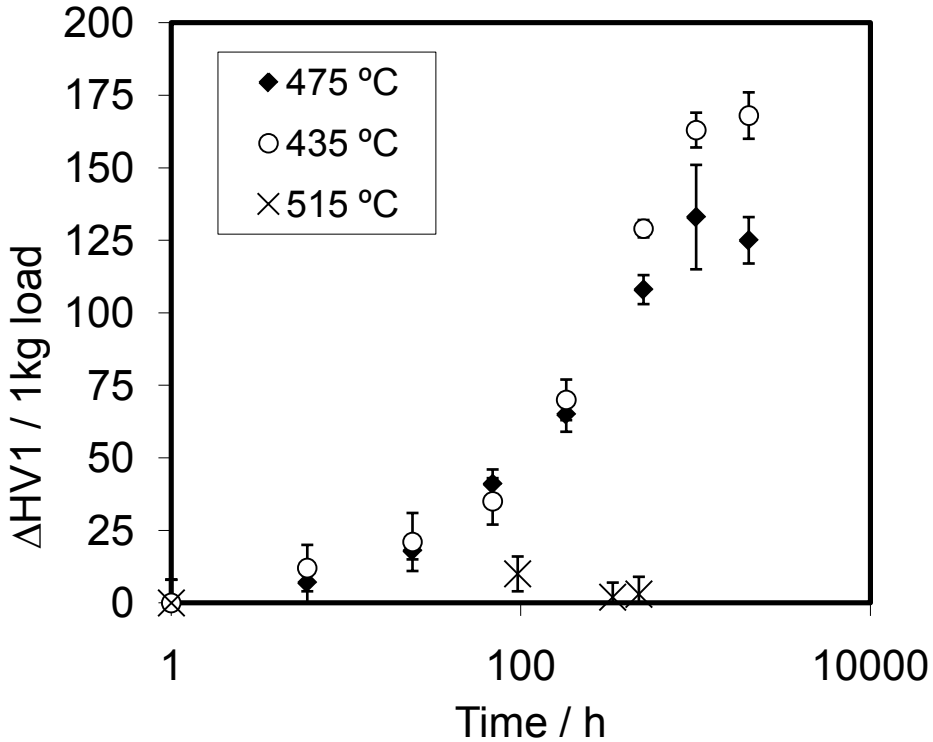
Heat Treatment



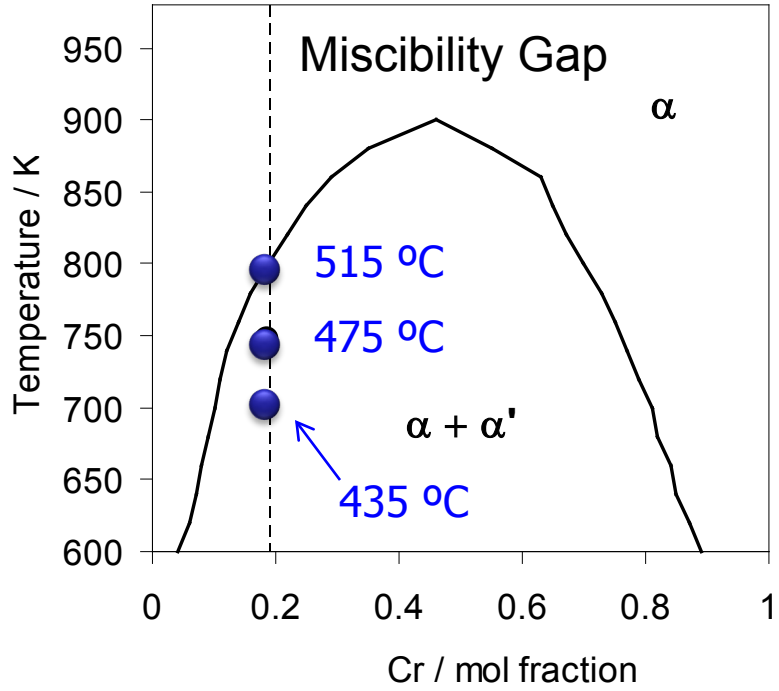


Hardness evolution

A continuous increase in hardness with respect to the initial state (before ageing), $\Delta HV1$, was observed.



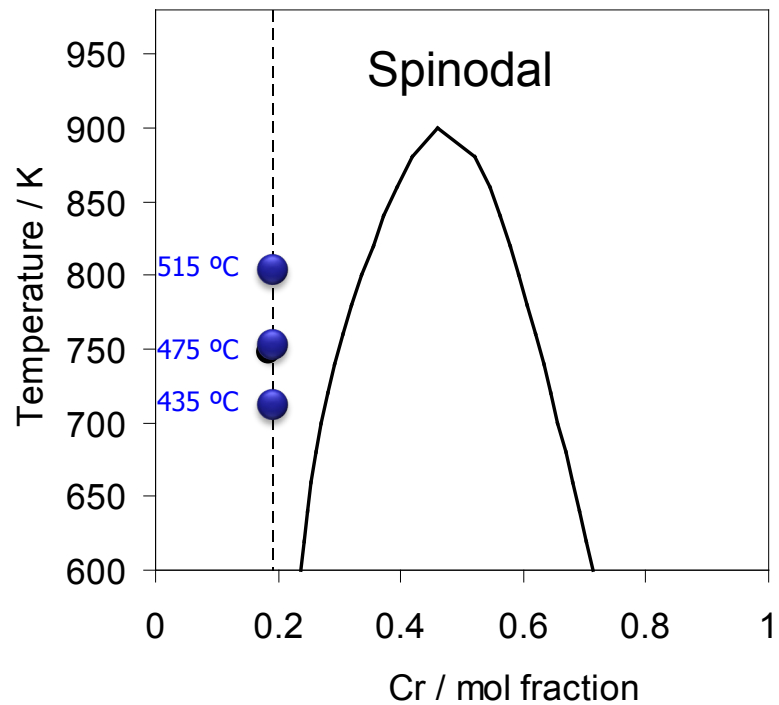
Thermodynamic analysis



— [Al]=11 at.%

---- PM2000

● Ageing Temperature



— Spinodal [Al]=11 at.%

---- PM2000

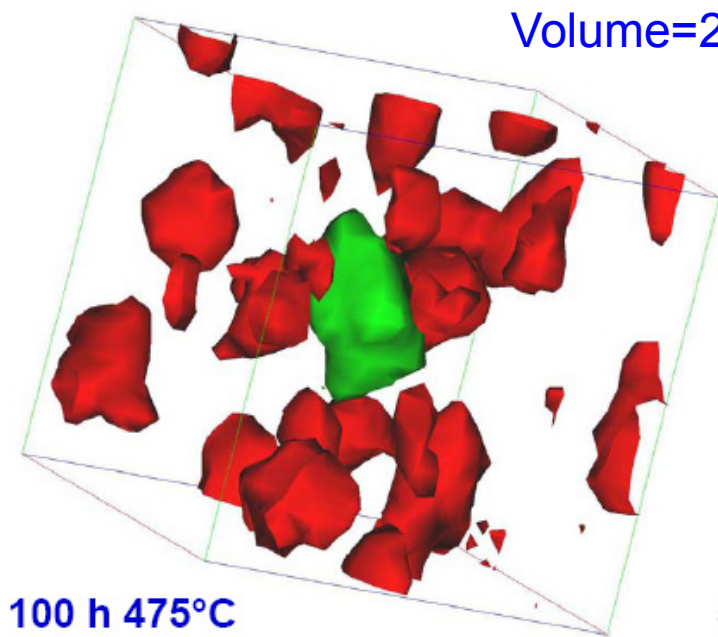
● Ageing Temperature

APT Results

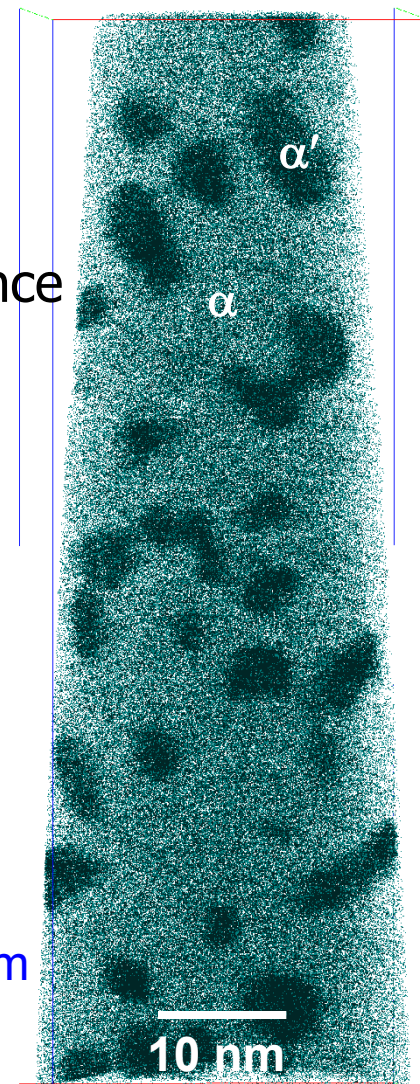
Red 30% Cr isoconcentration surface revealing the distribution and spherical morphology of Cr-rich α' phase.

Green 5% Ti isoconcentration surface revealing the existence of 3.2 nm in diameter nanoclusters of Fe(Ti,Al) (β' phase)

Cr atom map



Volume=30x30x127 nm
2040 h 475 °C

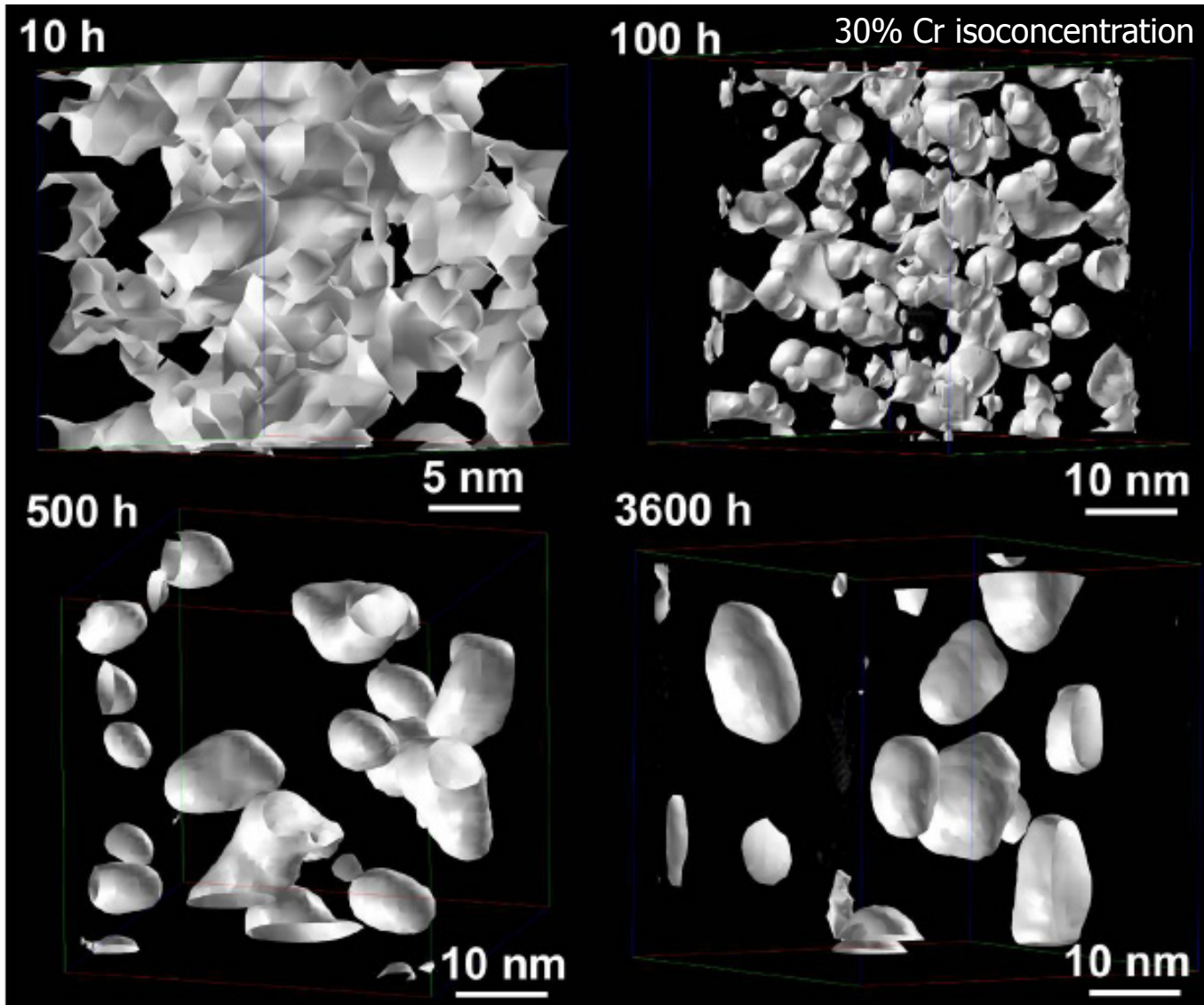


APT Results

α' phase evolution with time at 475 °C

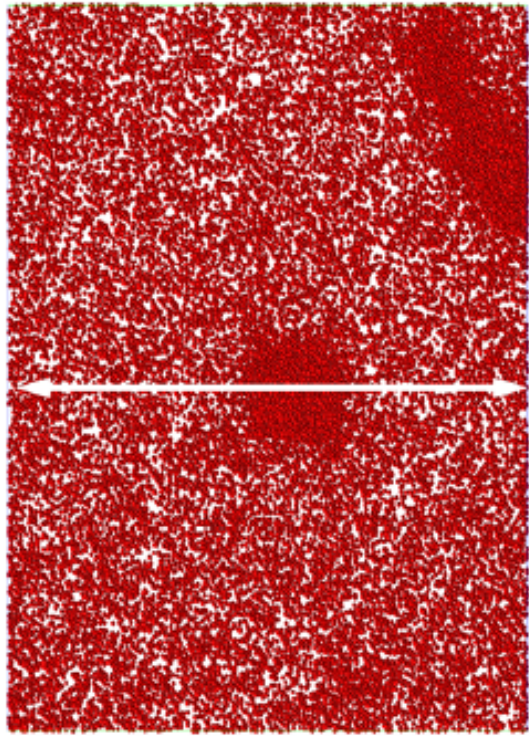


α' phase exhibits some interconnectivity at early stages, but it becomes spheroidal and its size increases on further ageing.

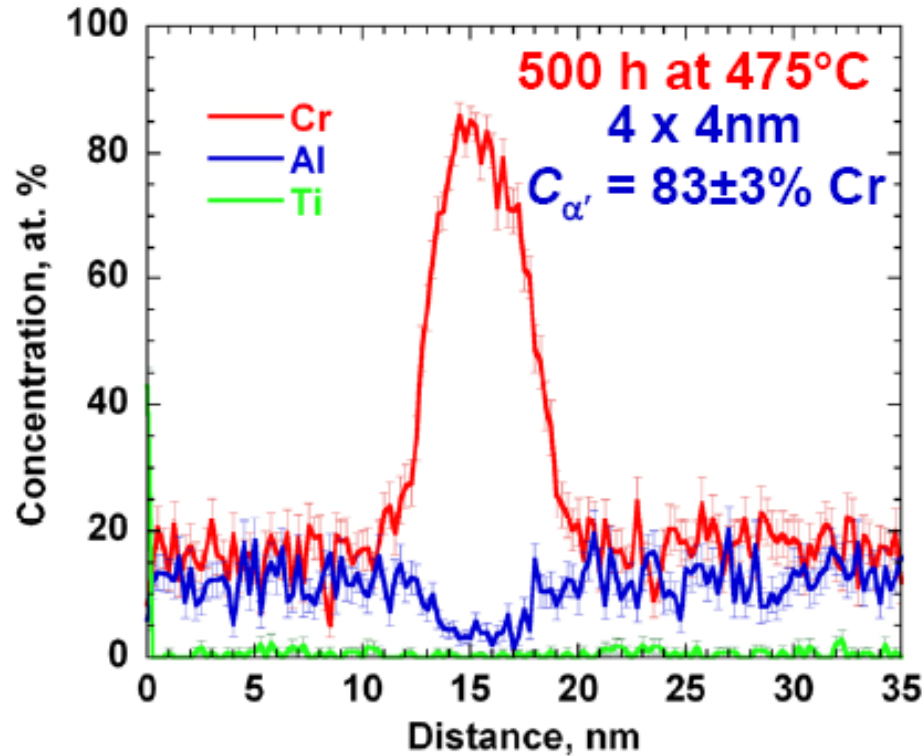


APT Results

α' phase composition

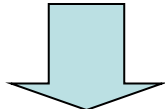


Cr

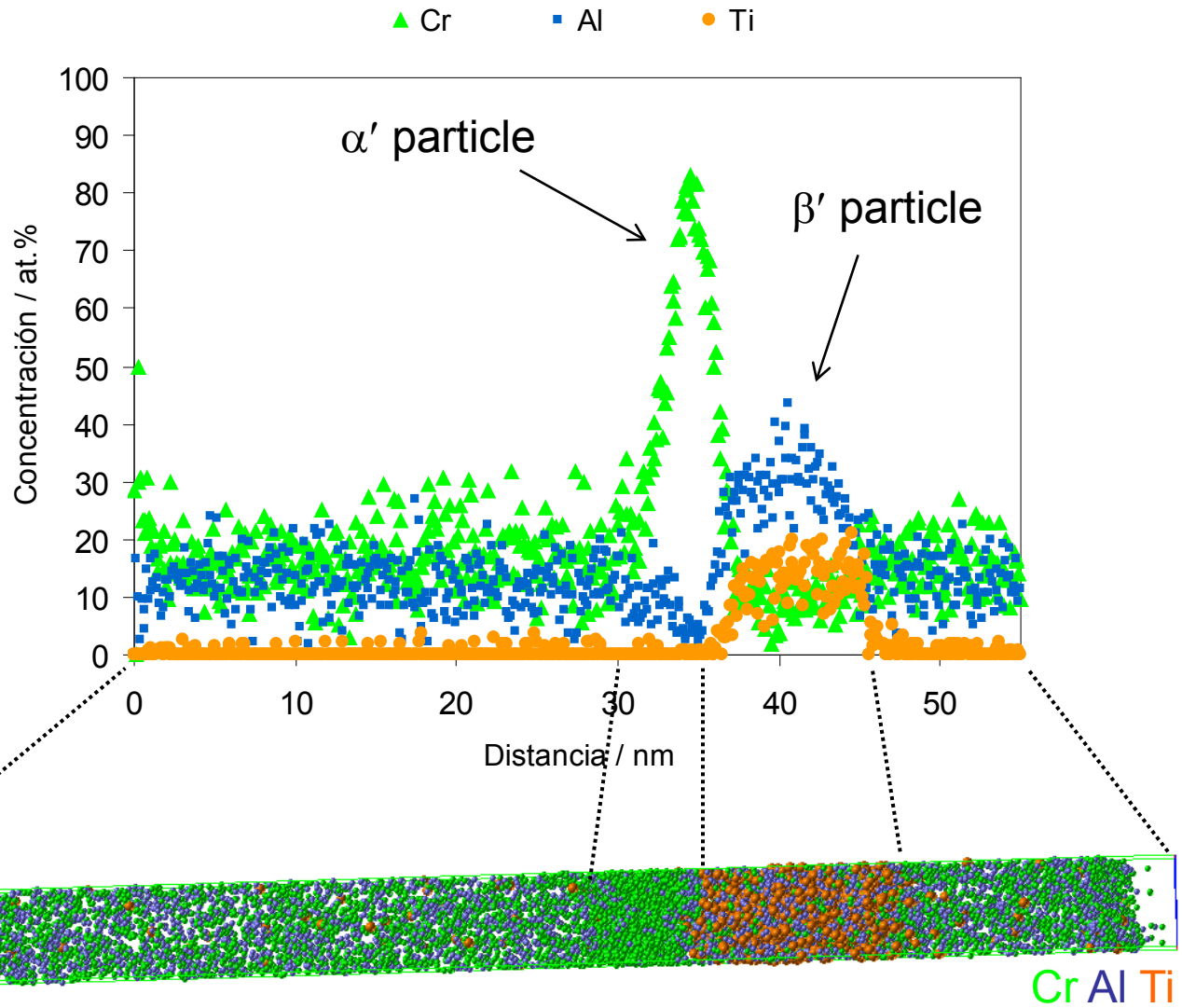


APT Results

Precipitation of β' phase



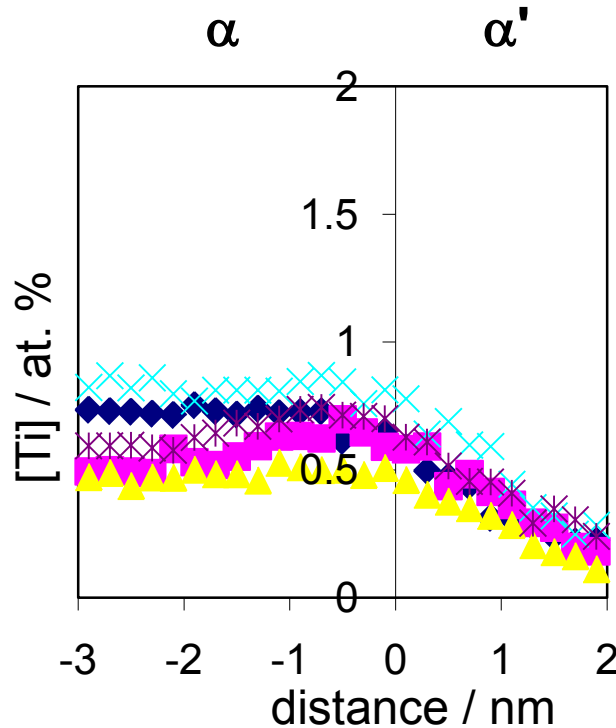
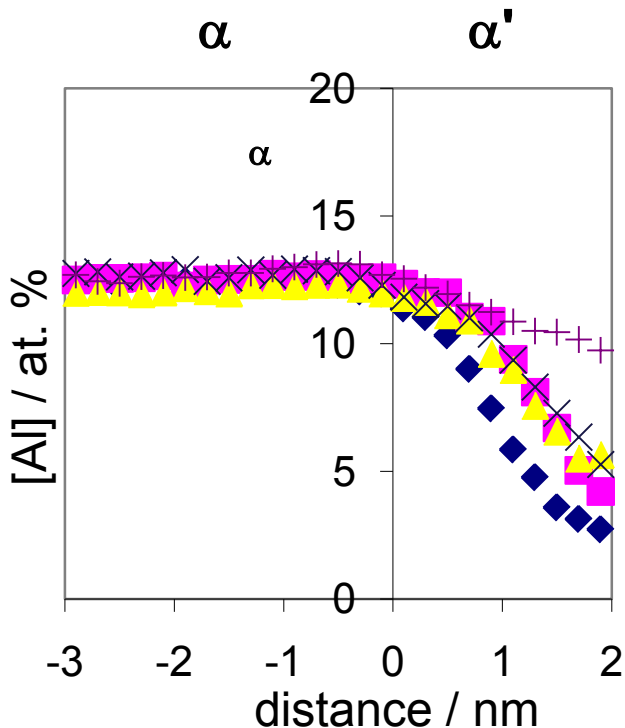
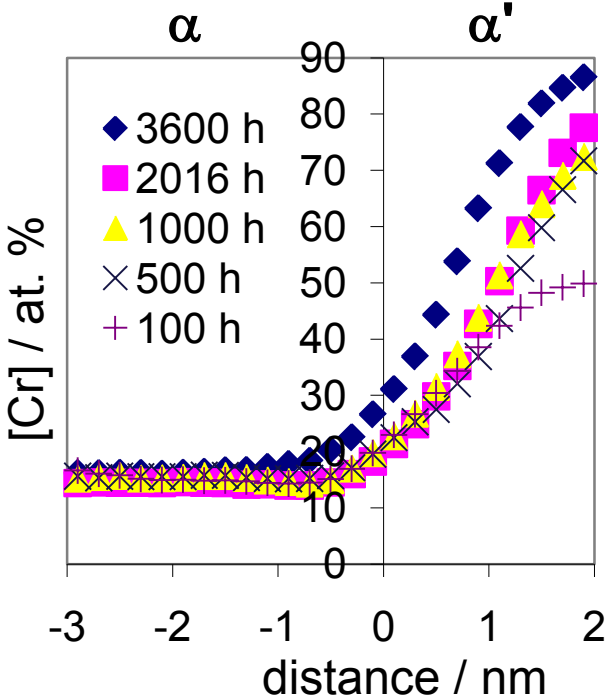
The depletion in Al in α' particles induces an enrichment in surrounding matrix that causes precipitation of nanosized β' particles



Volumen 4x4x57nm

α - α' phase separation

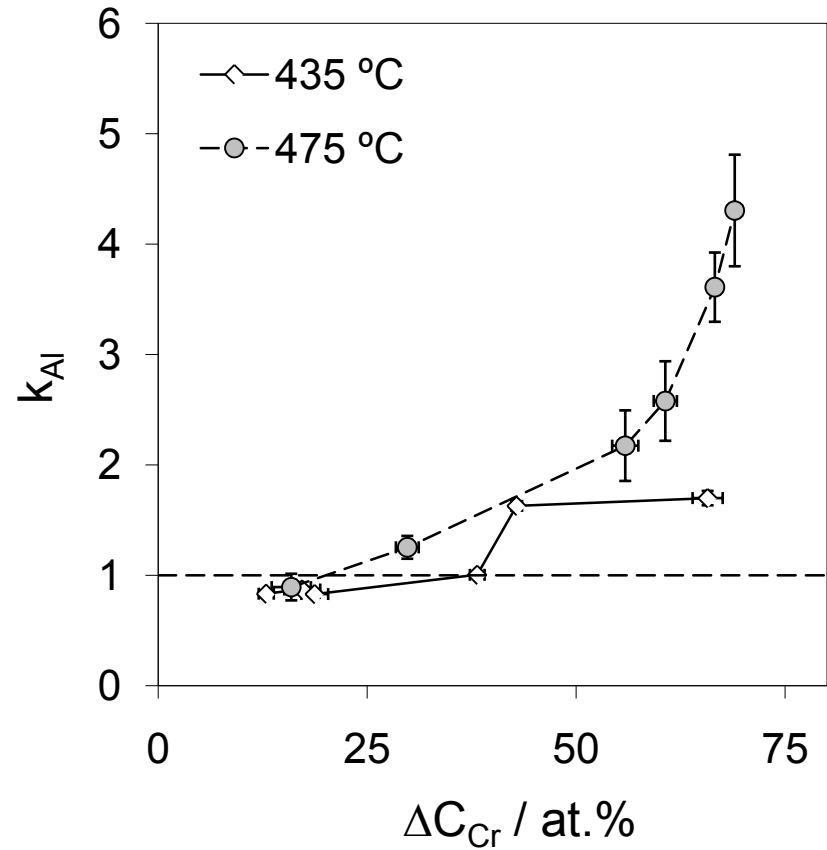
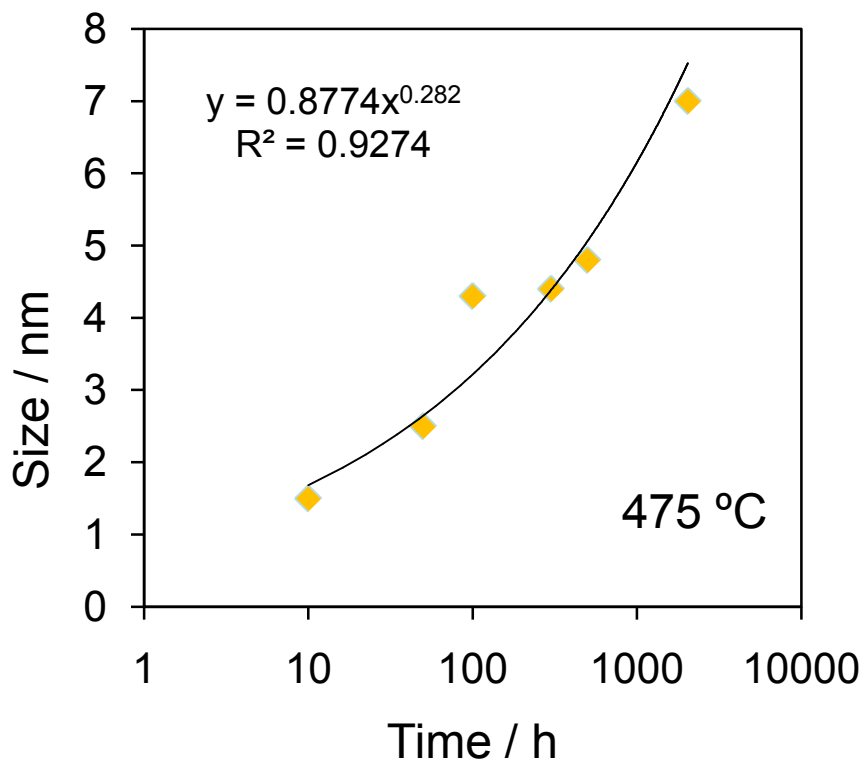
Proximity histograms analysis



475 °C

α' - size

$$k_{Al} = [Al]_{\alpha} / [Al]_{\alpha'}$$

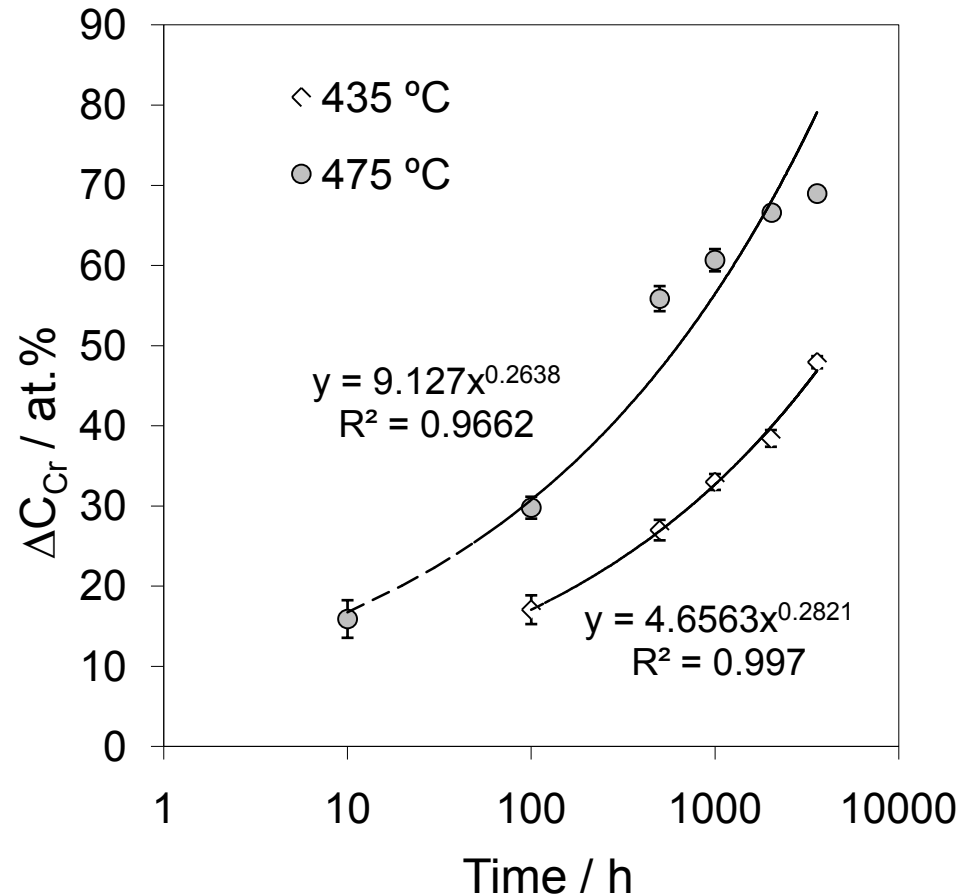


α - α' phase separation kinetics

The kinetics of α - α' phase separation process in PM 2000 were determined from the analysis of proximity histogram

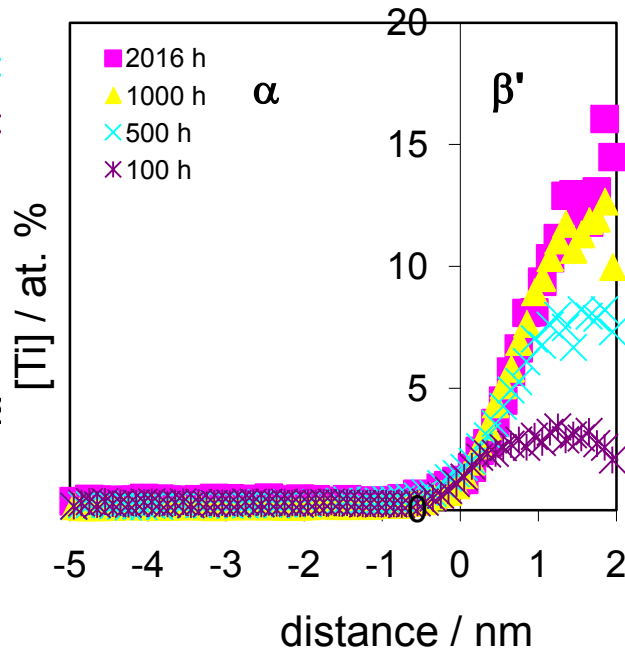
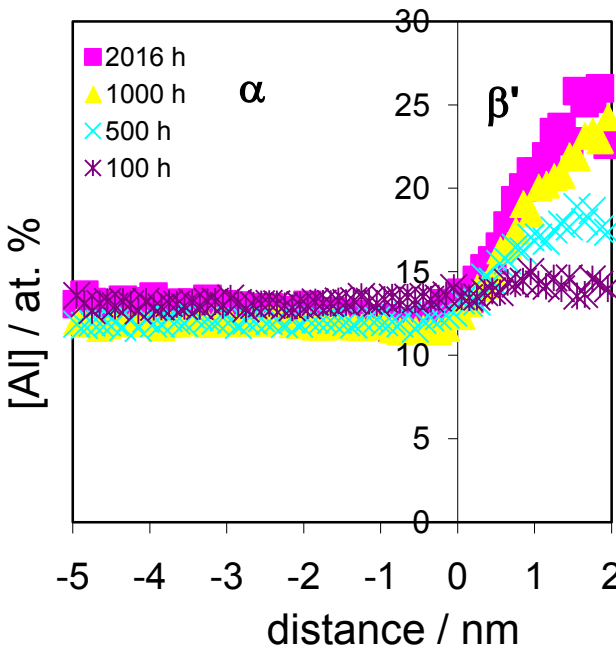
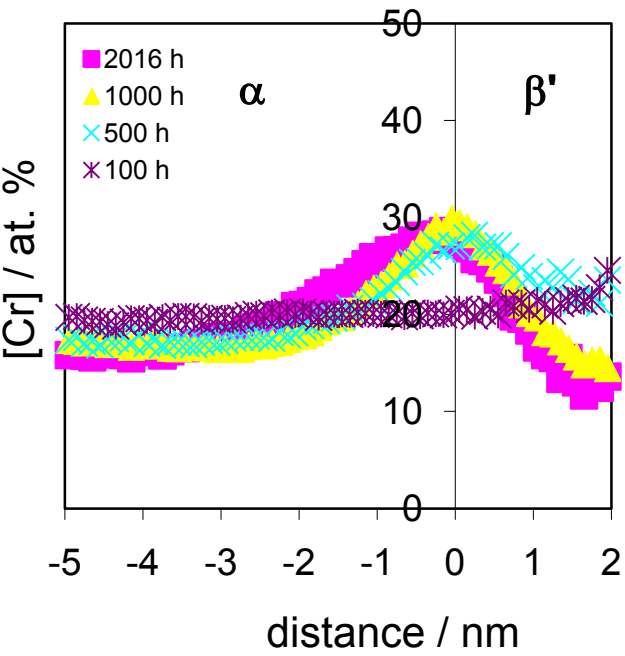


The size of the α' increases with a time exponent of 0.32 which is consistent with the mean precipitate size $R(t)$ varying as $\sim t^{1/3}$ predicted by the LSW theory.



β' precipitation

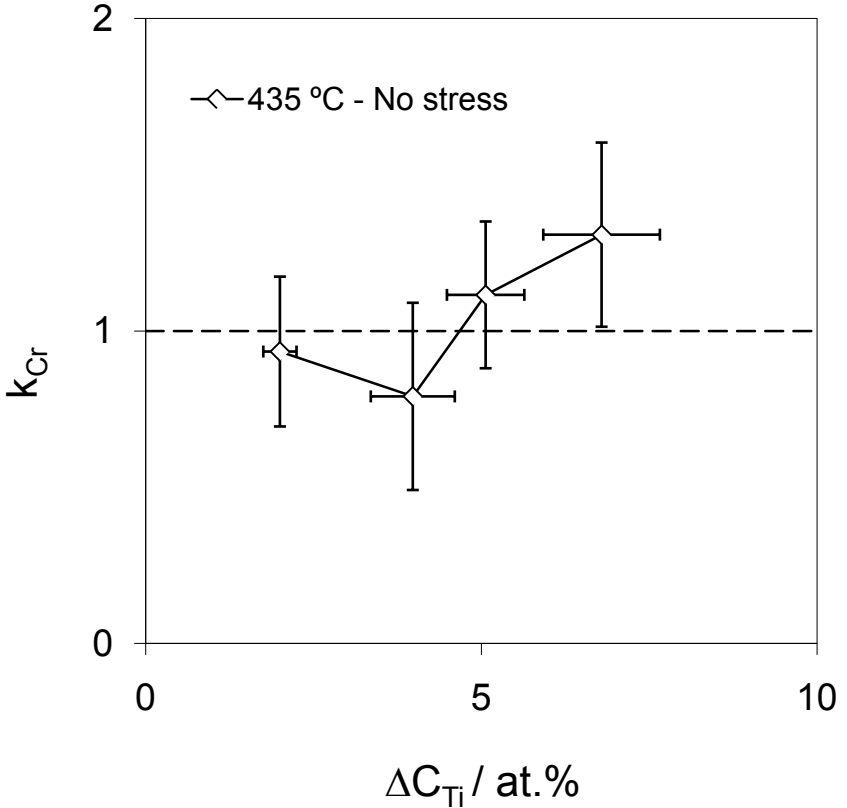
Proximity histograms analysis



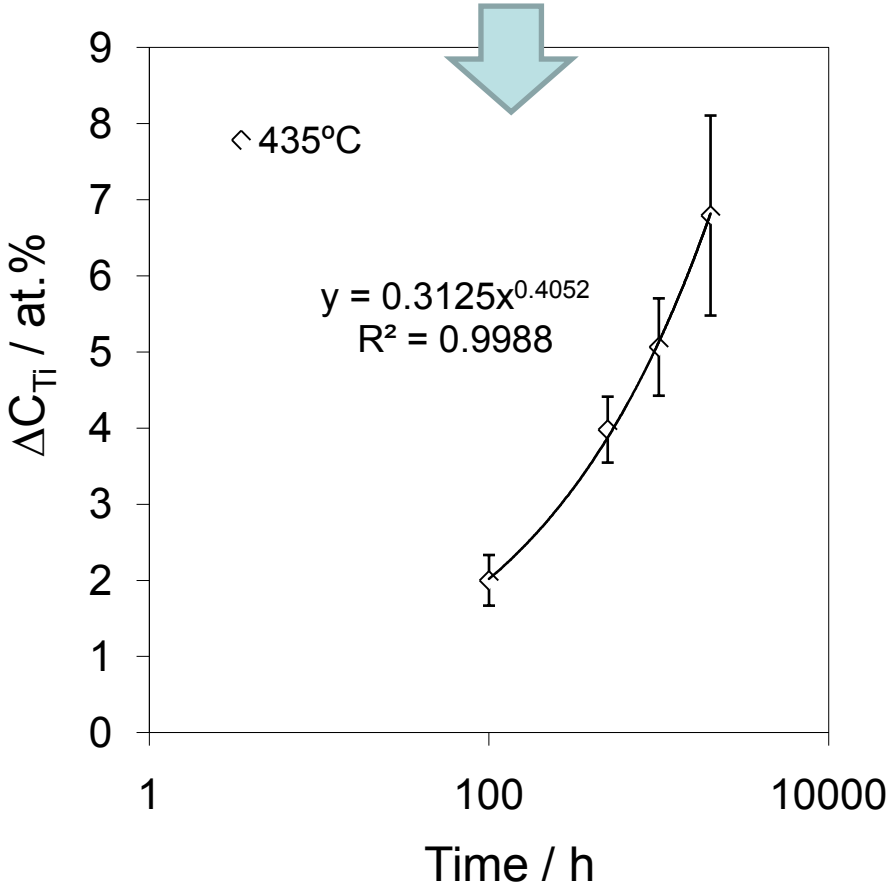
435 °C

β' precipitation kinetics

$$k_{Cr} = [Cr]_{\alpha} / [Cr]_{\beta'}$$

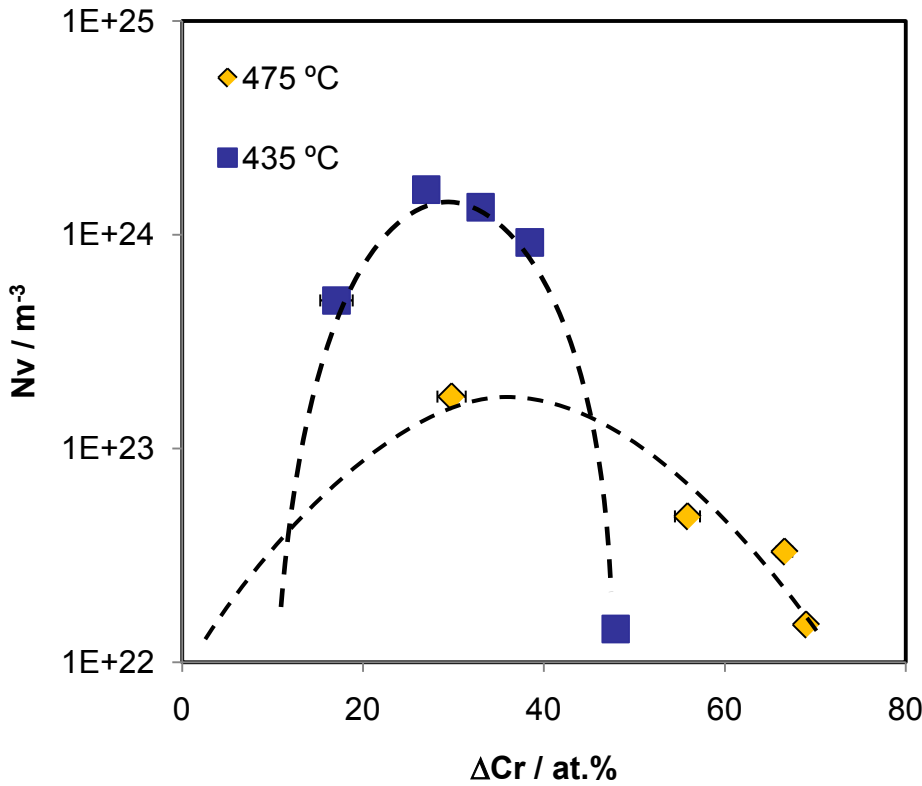
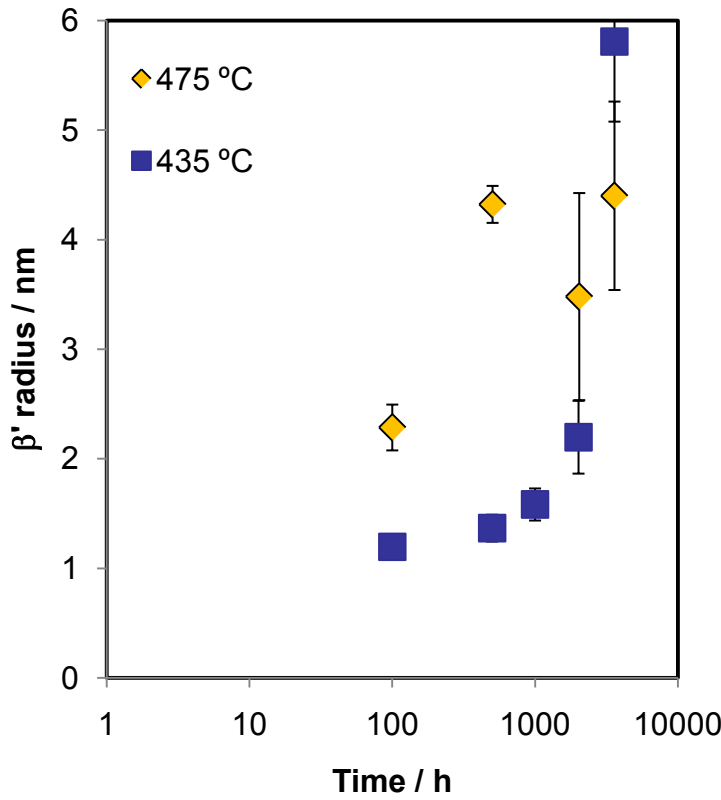


The kinetics of β' precipitation were determined from the analysis of 1at.%Ti- proximity histogram

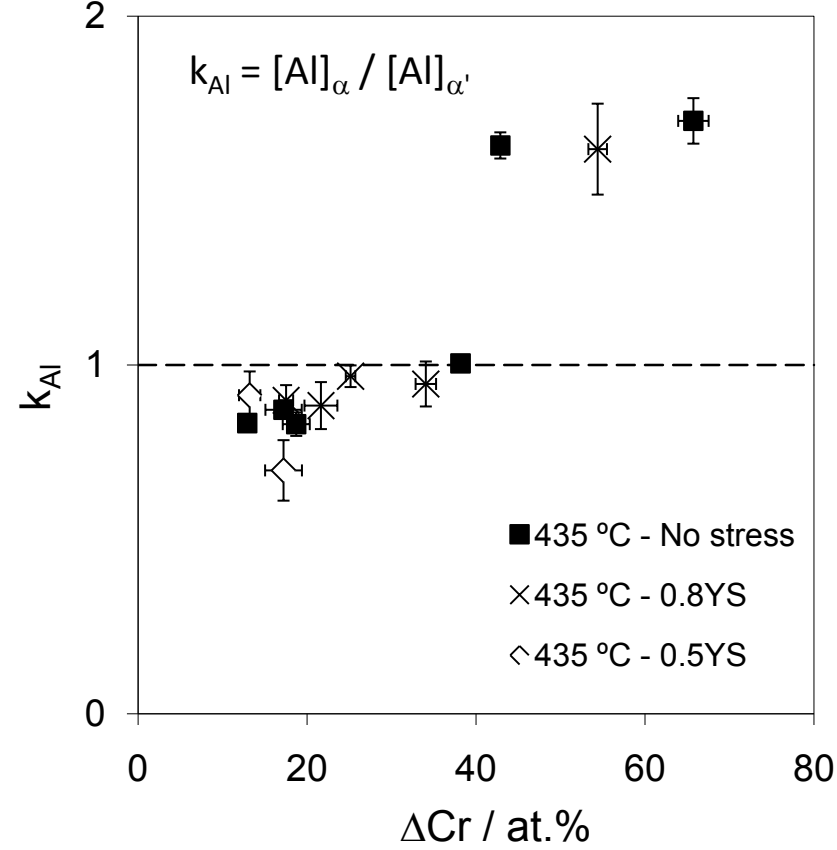
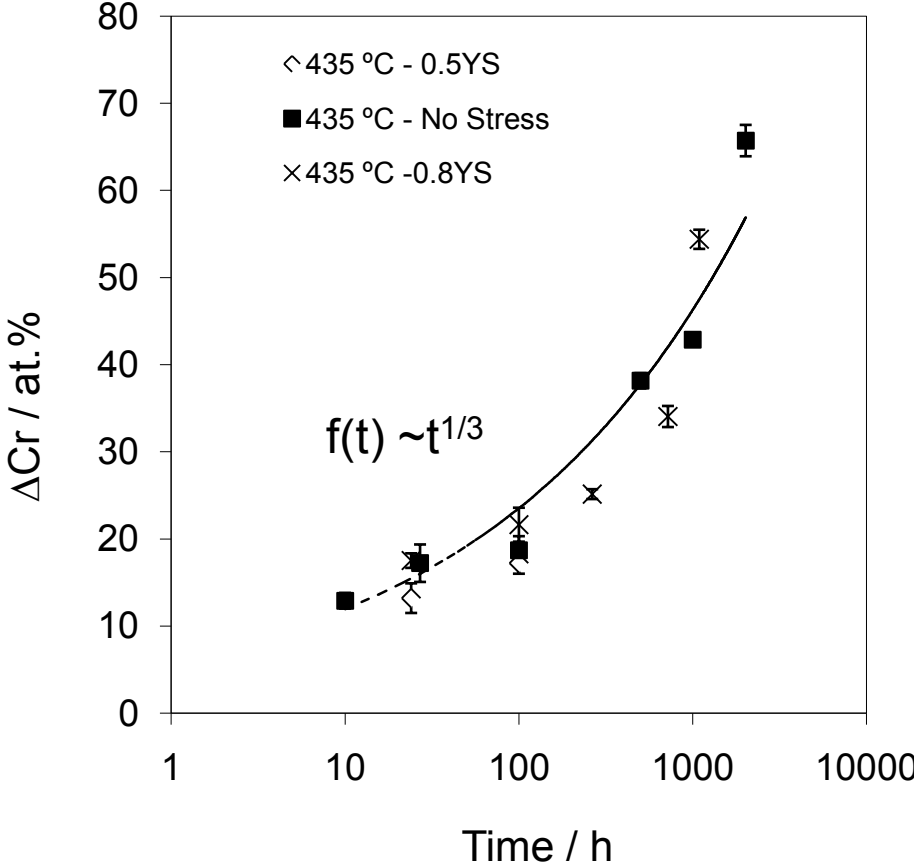


β' - size

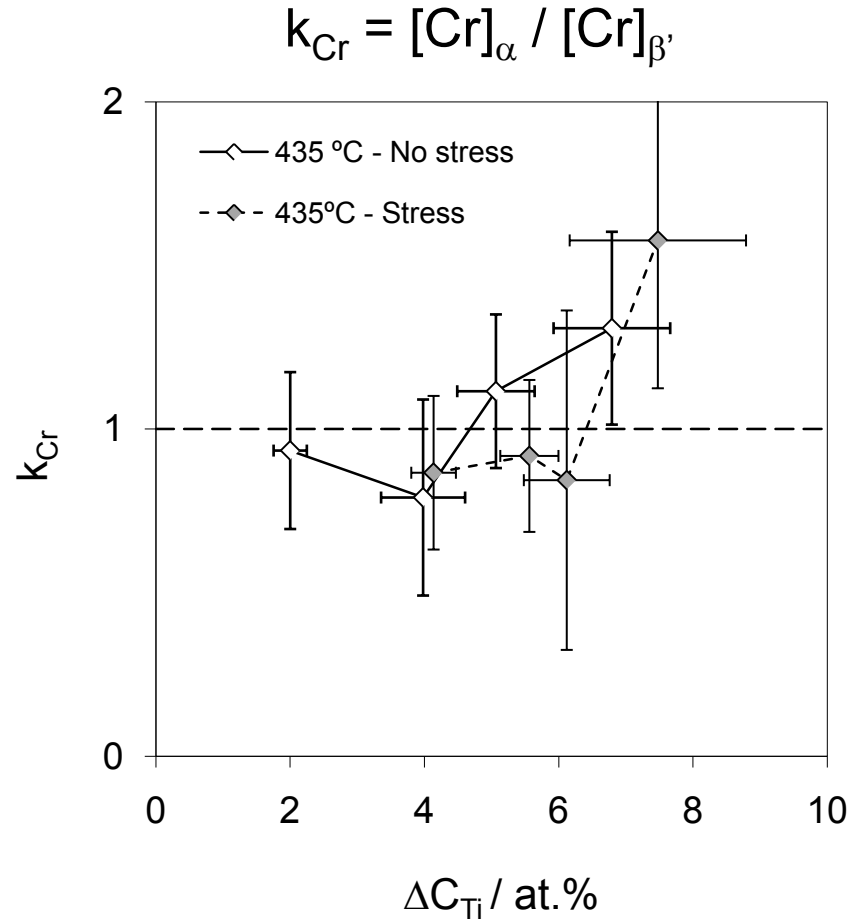
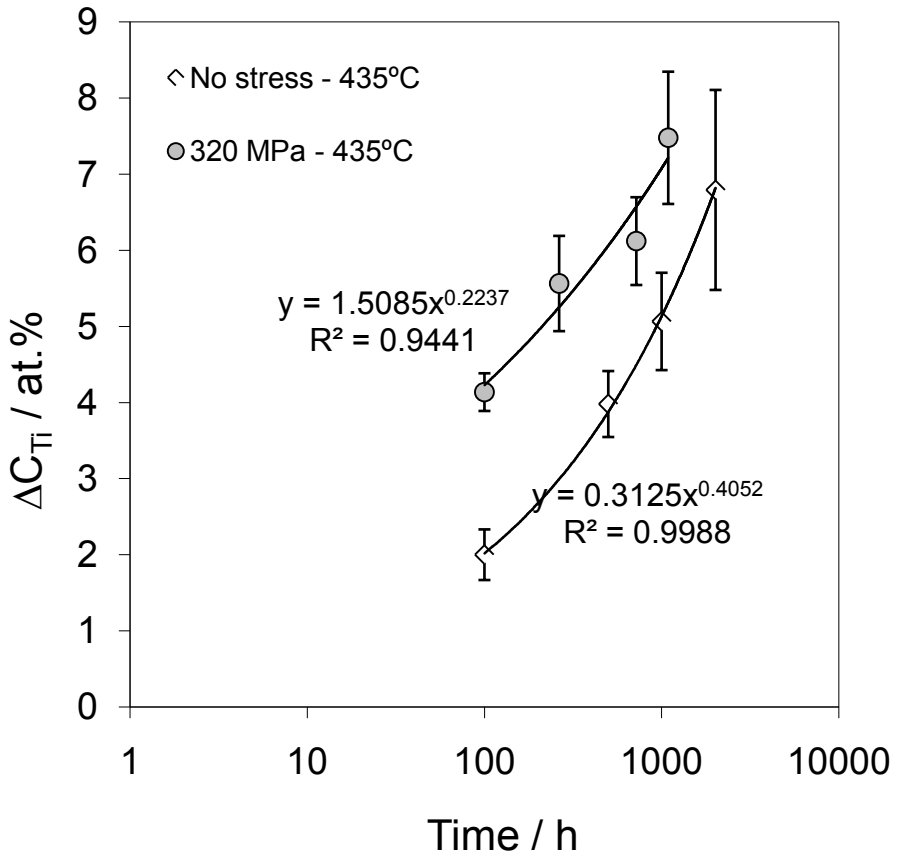
Maximum separation method



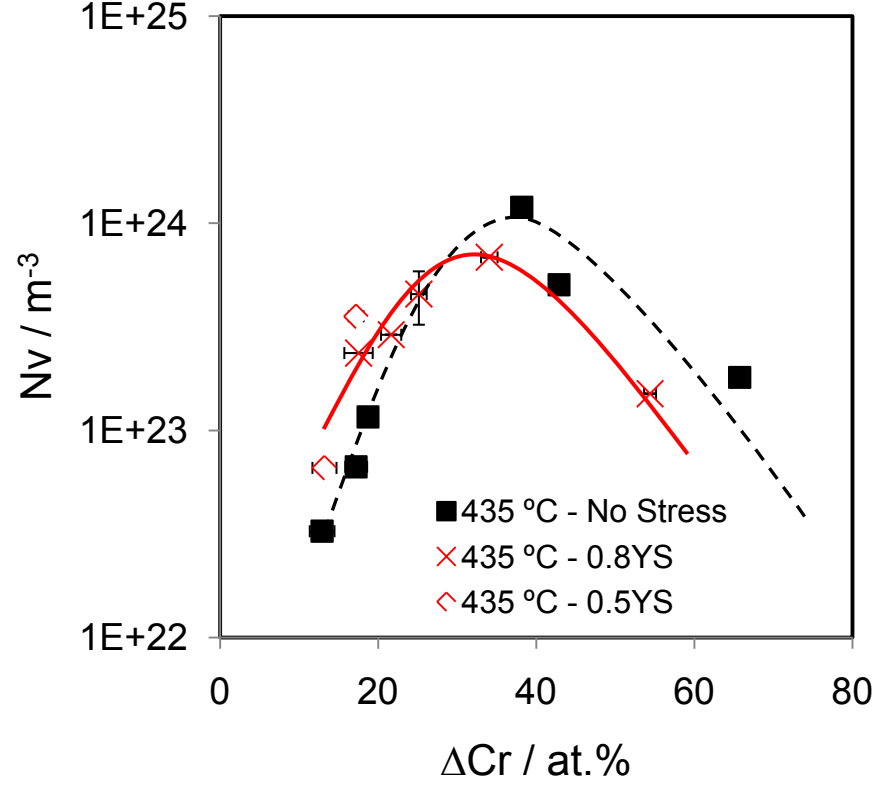
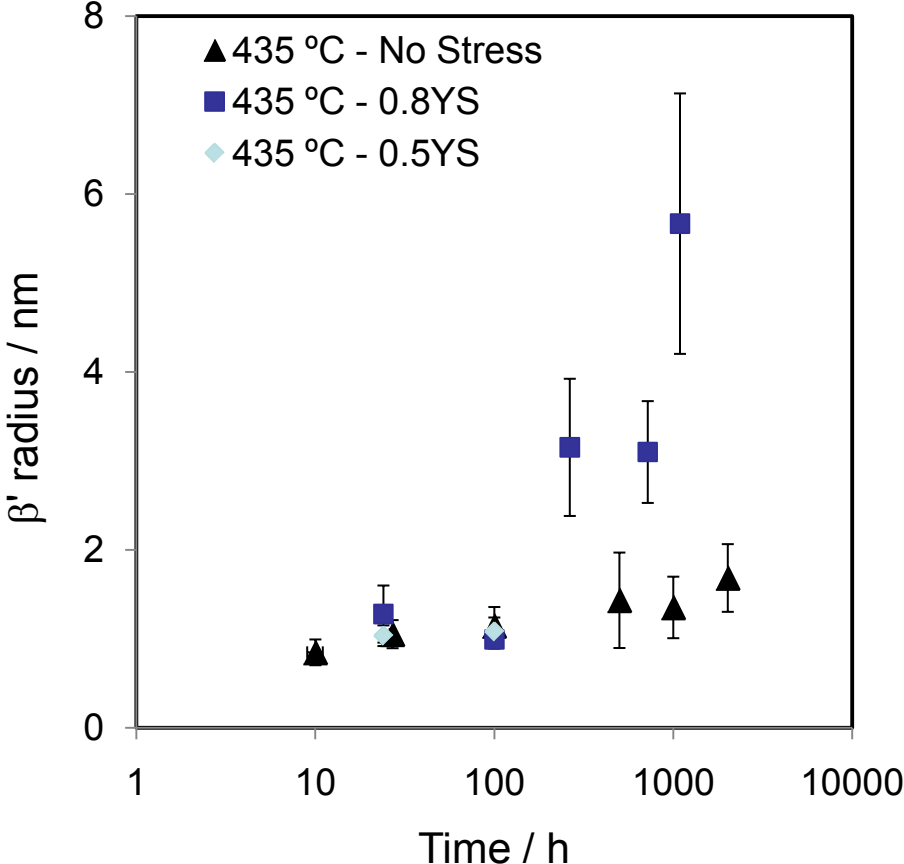
Effect of Stress: α - α' phase separation



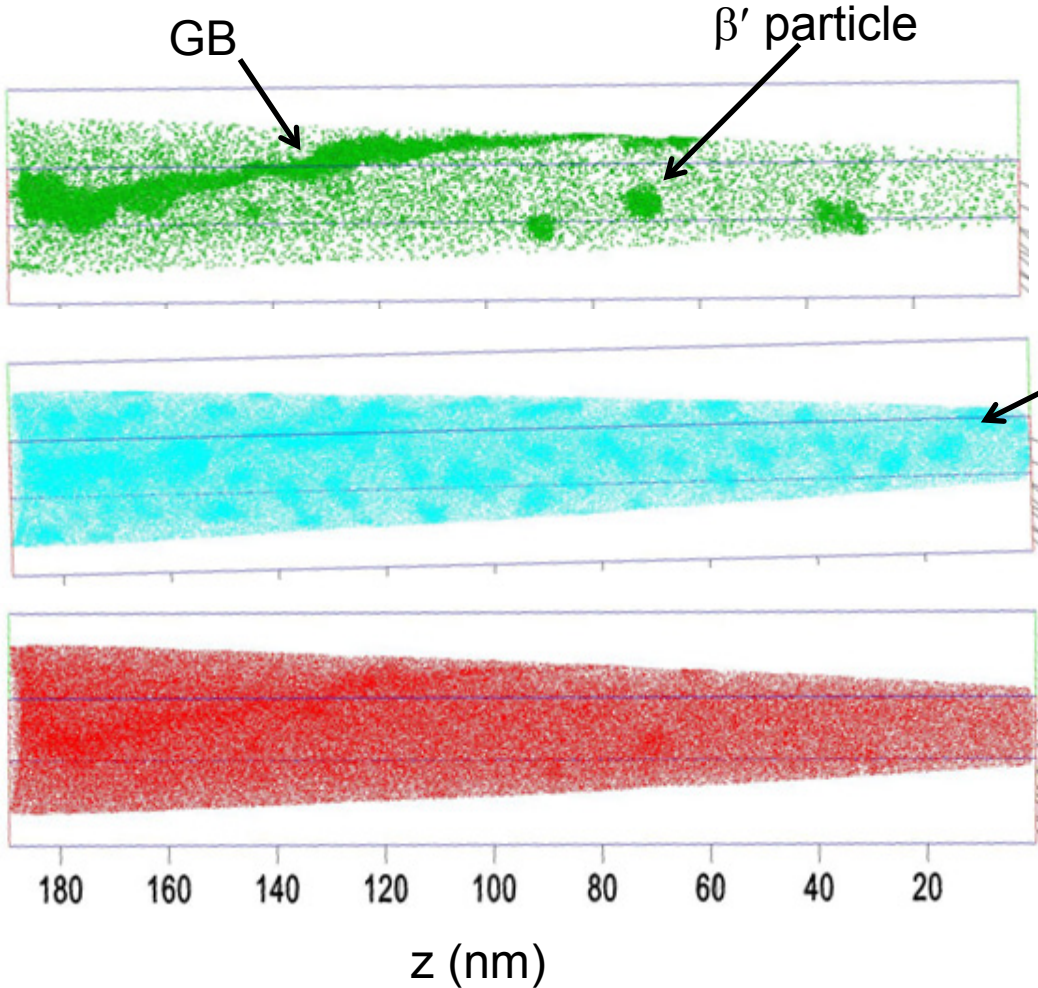
Effect of Stress: β' precipitation



Effect of Stress: β' precipitation



Effect of Stress: grain boundary segregation



$\sigma = 0.8Y_S$

Ti atom map

Cr atom map

Al atom map

Conclusions

1. Atom probe tomography revealed the nanometer-scaled phase separation between α and α' phases during isothermal aging at 435 and 475 °C.
2. Proximity histograms analysis revealed that the faster phase separation kinetics without stress applied is 475 °C.
3. Al is rejected from the α' to the matrix during the phase separation. This leads to the precipitation of Fe-Ti-Al intermetallics (β' phase).
4. The maximum separation method estimated the size and number density of β' particles. The β' particles are more abundant and finer at 435 °C than that at 475 °C.
5. Elastic stress does not affect $\alpha - \alpha'$ phase separation kinetics, but significant coarsening in β' phase precipitation is observed.
6. It was observed Ti and Al segregation at the grain boundary during elastic stressed ageing treatments.