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The β′-α′ interaction: A study of early stages of phase separation in a Fe-20%Cr-6%Al-0.5%Ti alloy

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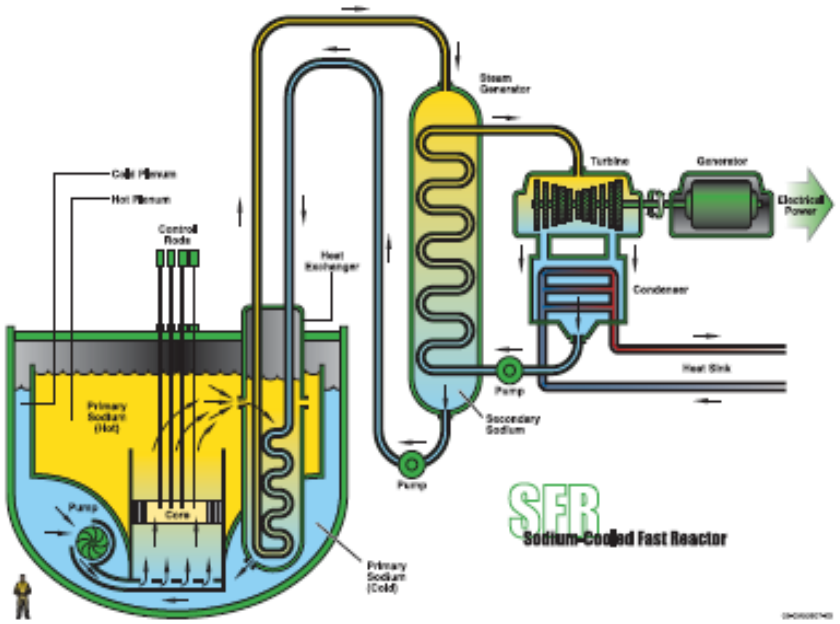
Introduction

- Due to their high creep rupture strength and excellent swelling resistance, many developments concerning oxide dispersion strengthened (ODS) ferritic steels are underway in several types of nuclear reactors such as sodium fast reactors (SFR), very-high temperature reactors (VHTR), super critical water reactors (SCWR), or other GEN IV nuclear energy systems.

- For such applications, these alloys face a severe embrittlement problem because their service temperature lies in the range of 300–500 ºC..

Sodium-cooled fast reactor



A Technology Roadmap for Generation IV Nuclear Energy Systems

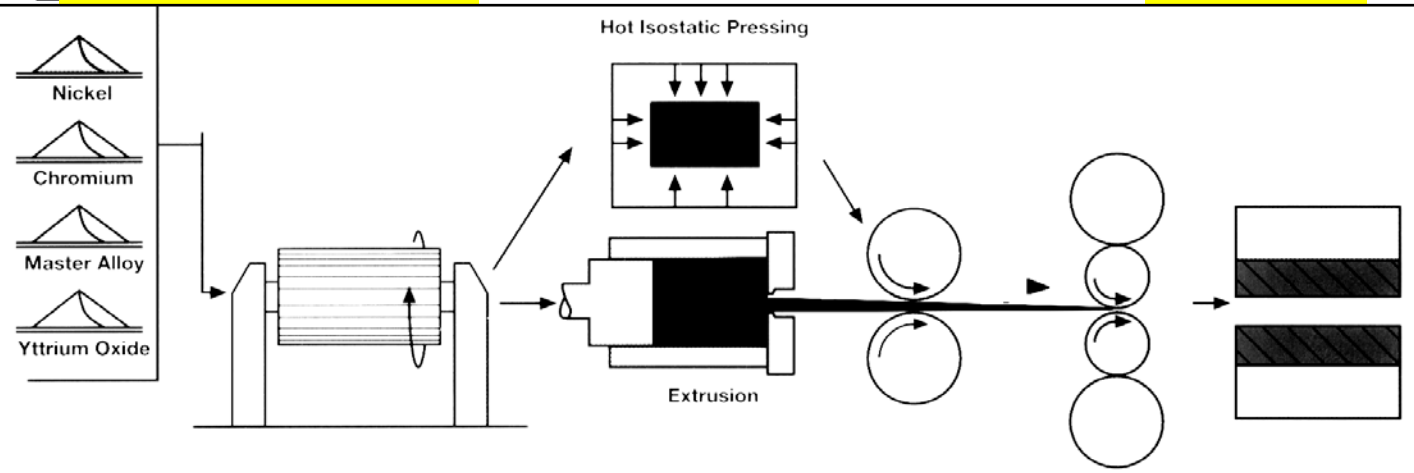
Spanish Goverment Funded Research Projects (I+D+i):
ENE2006-15170-C02-01
ENE2009-13766-C04-01

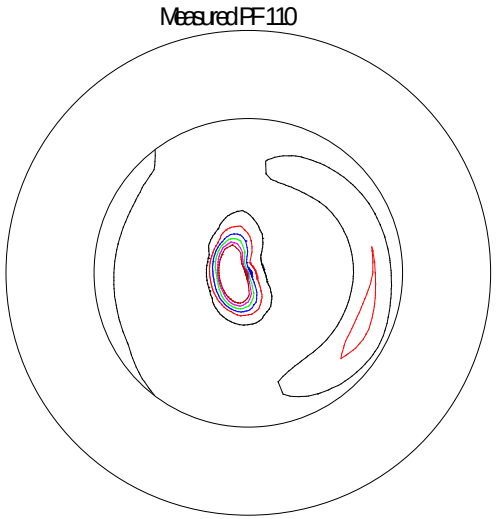
Material

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

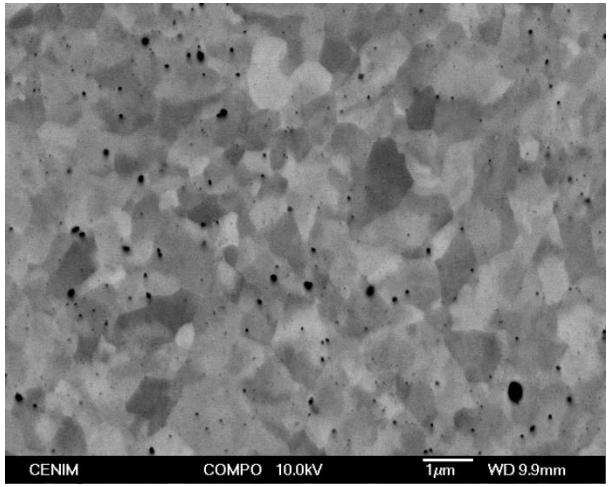
Chemical composition of PM 2000

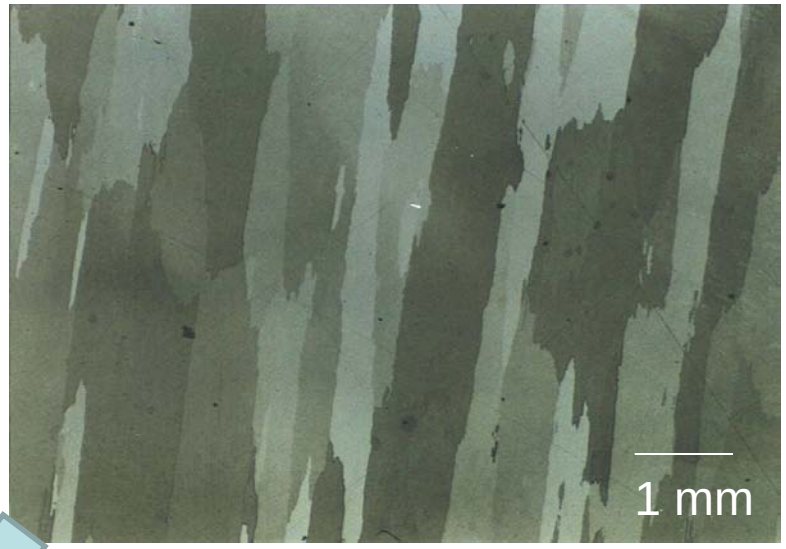
	Cr	Al	C	O	N	Ti	Y
wt. %	18.6	5.5	0.04	0.09	0.006	0.54	0.39
at. %	18.5	10.5	0.17	0.28	0.022	0.58	0.23



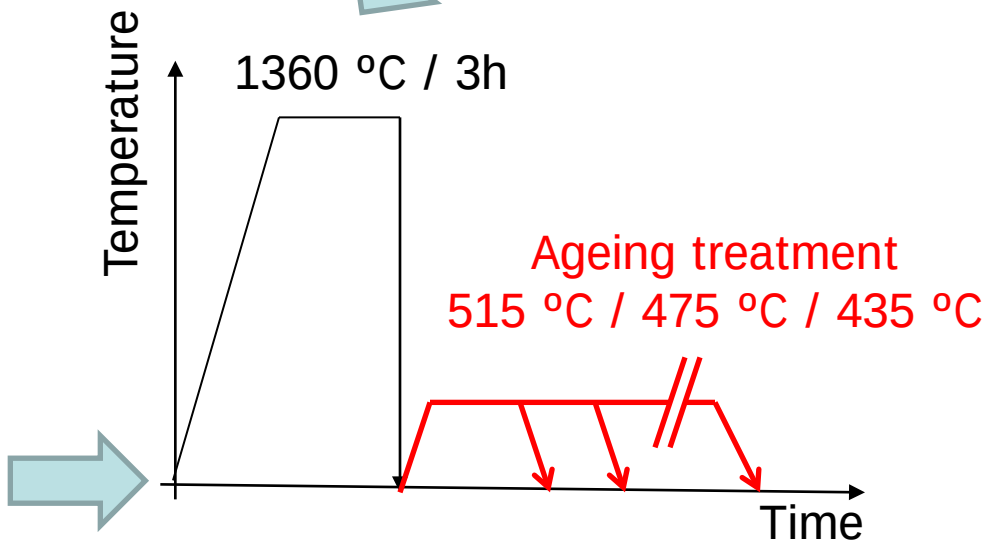


Transverse section



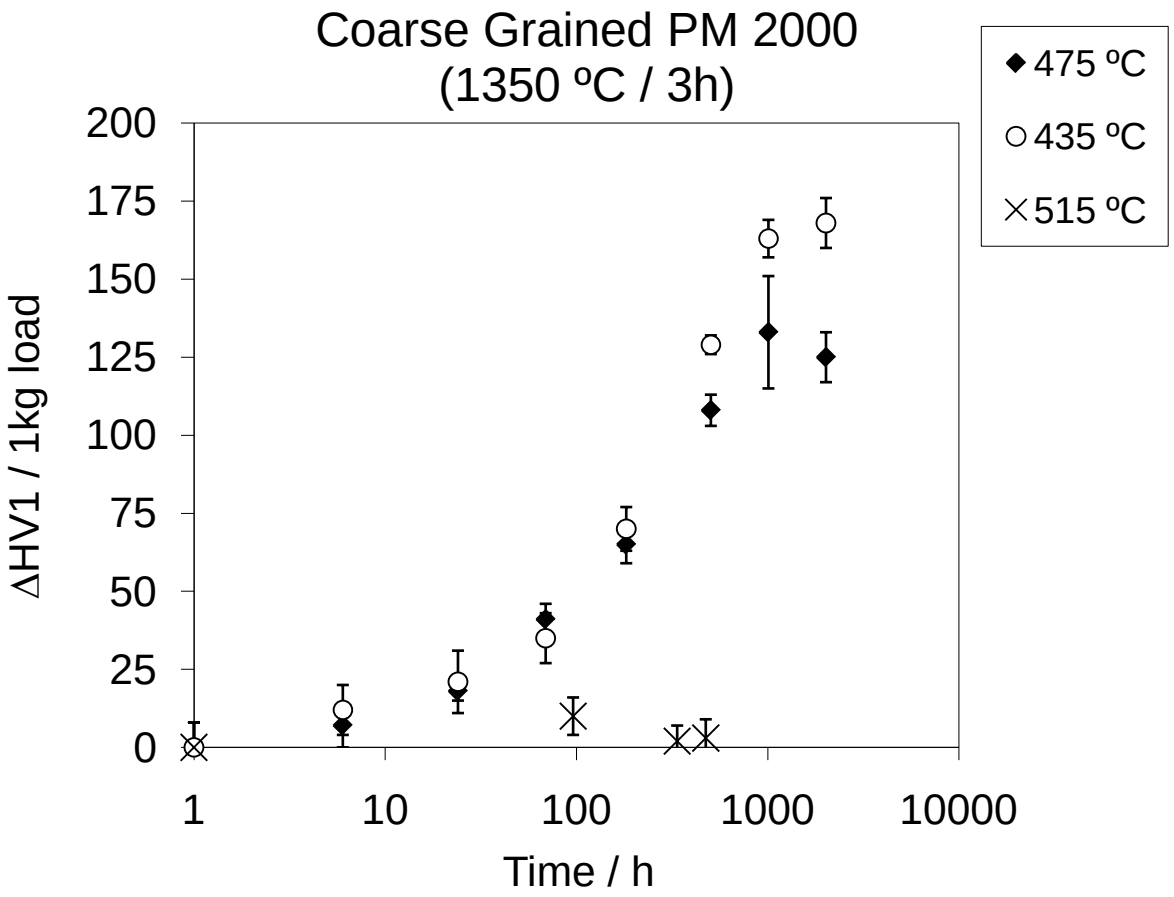


Thermal cycle

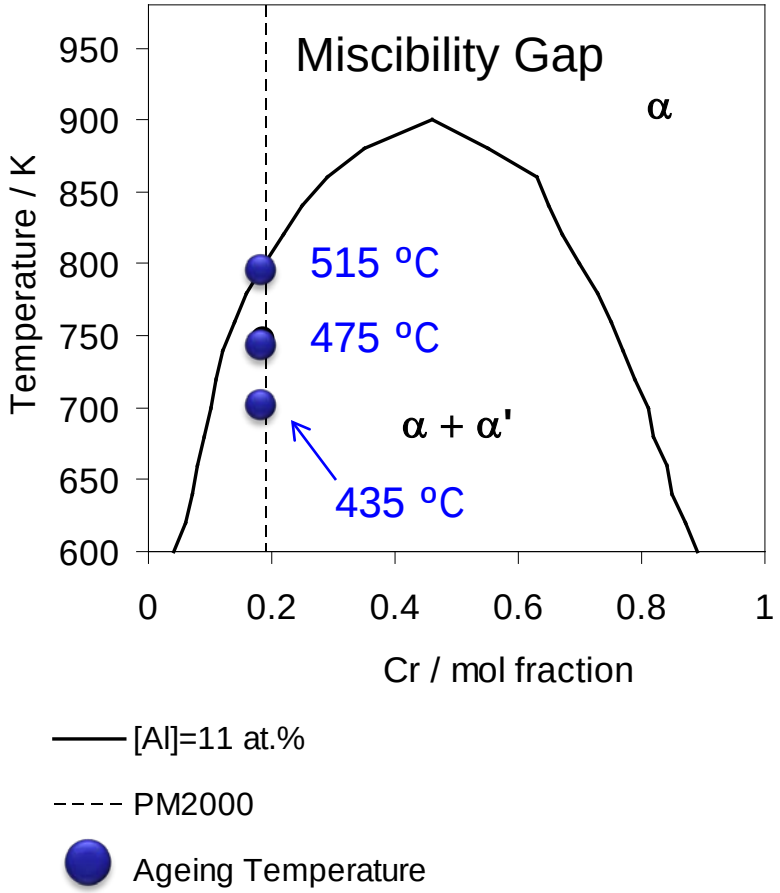


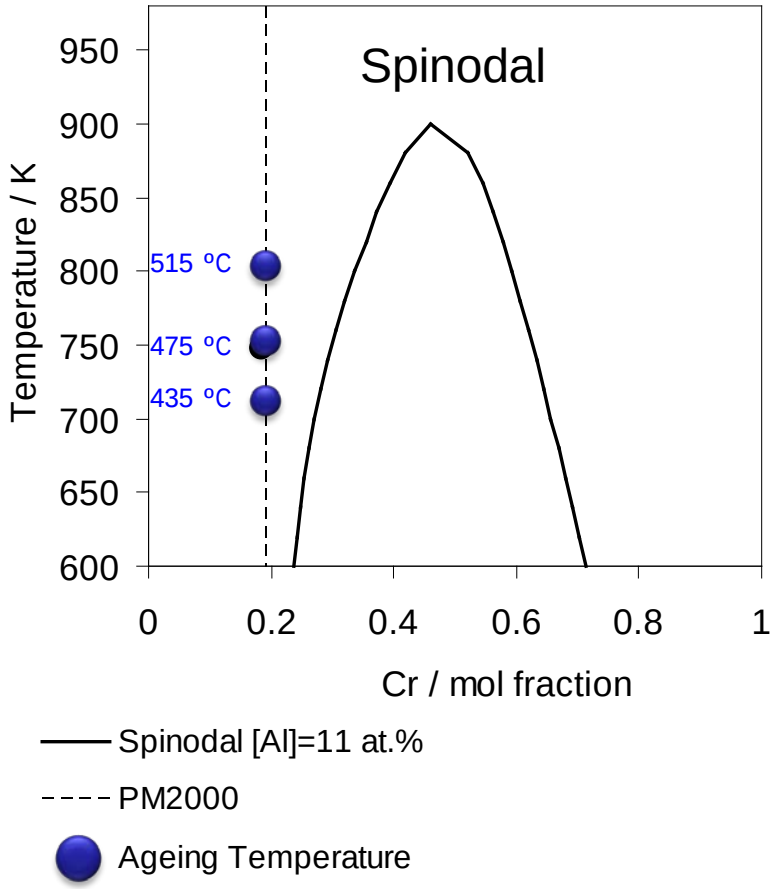
Hardness evolution

A continuous increase in hardness with respect to the initial state (before ageing), ΔHV1, was observed.



Thermodynamic analysis



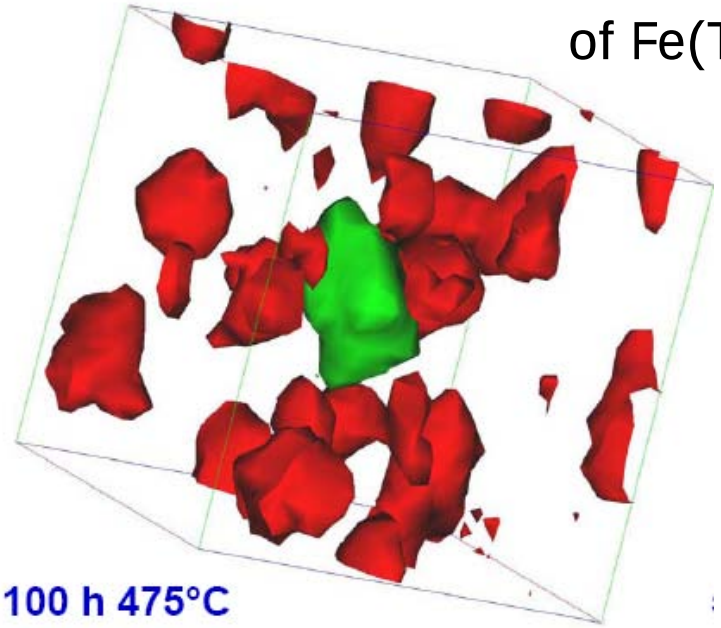


APT results

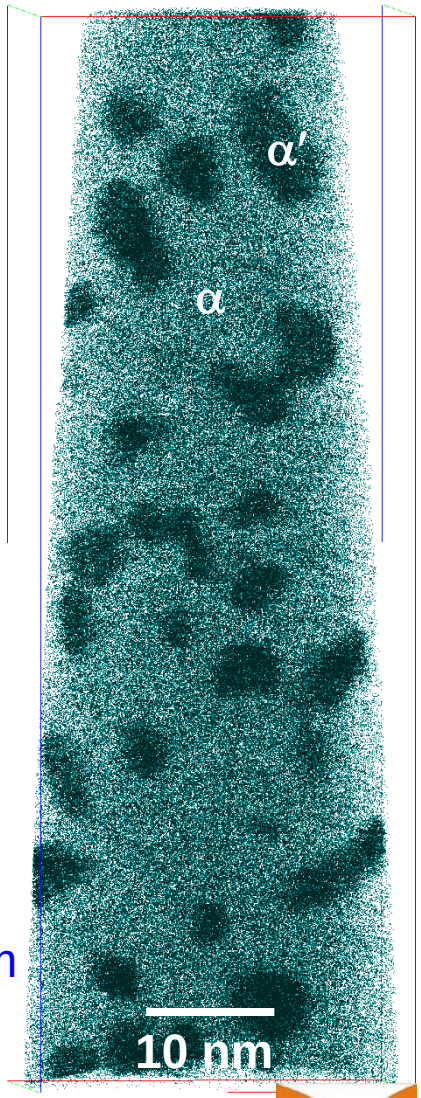
Red 30% Cr isoconcentration surface revealing the distribution and spherical morphology of Cr-rich α' phase.

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

Volume=20x20x20 nm



Cr atom map

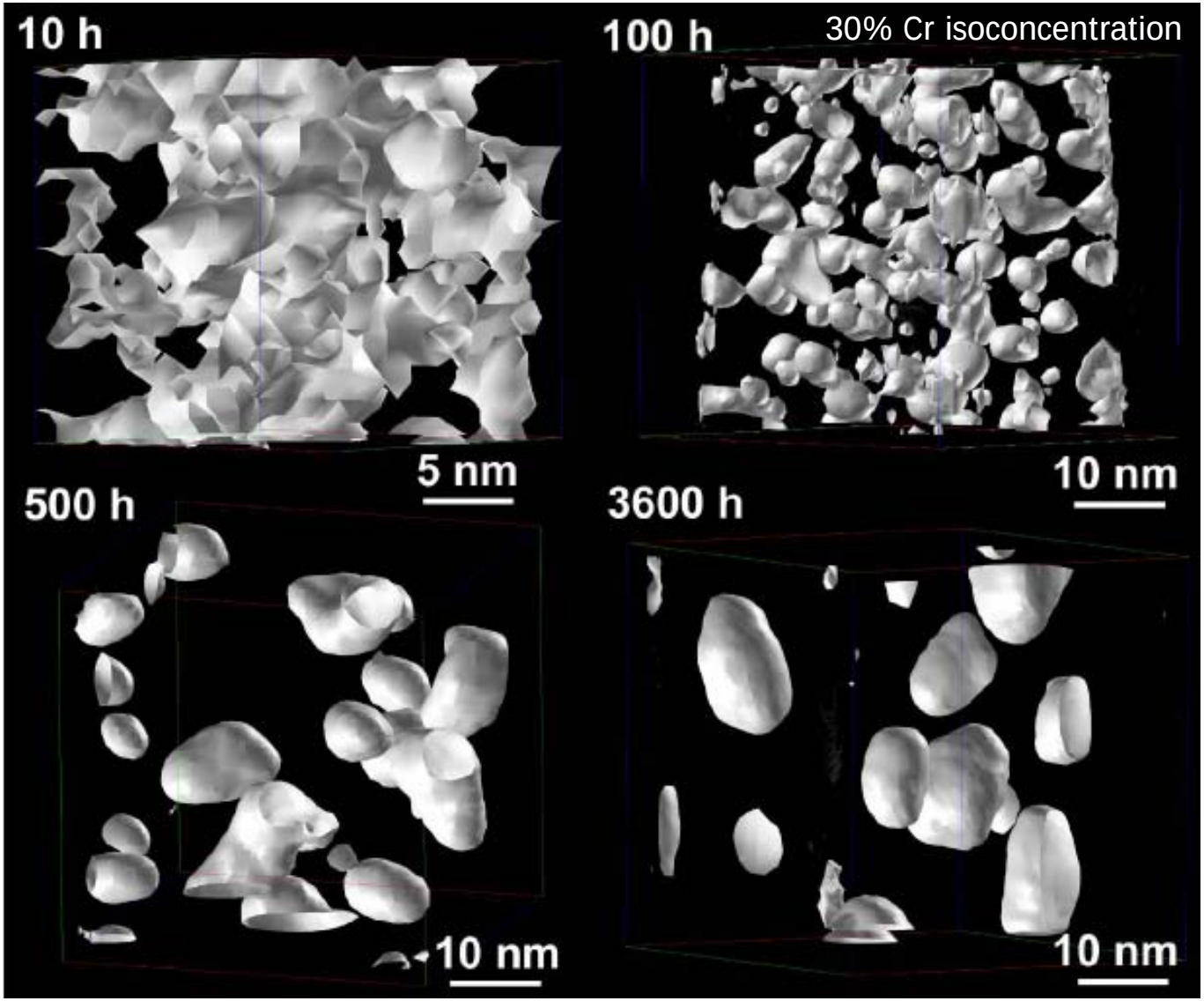


Volume=30x30x127 nm
2040 h 475 ºC

APT results

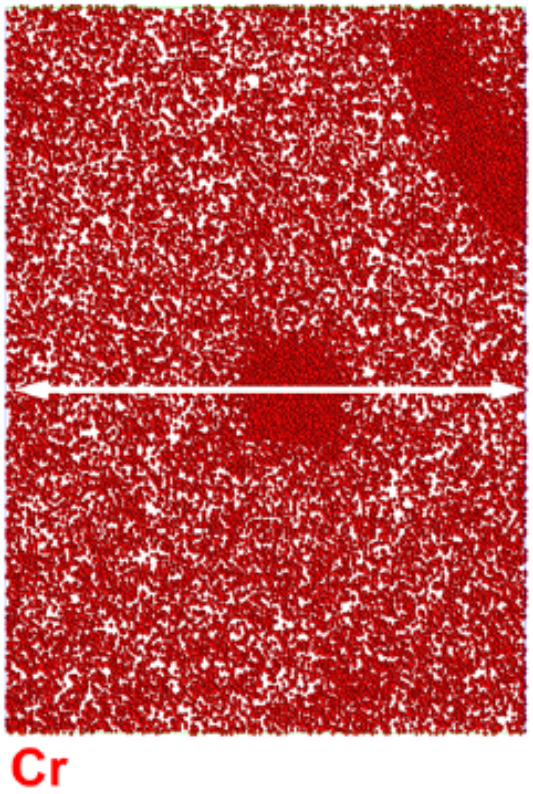
α' phase evolution with time at 475 ºC

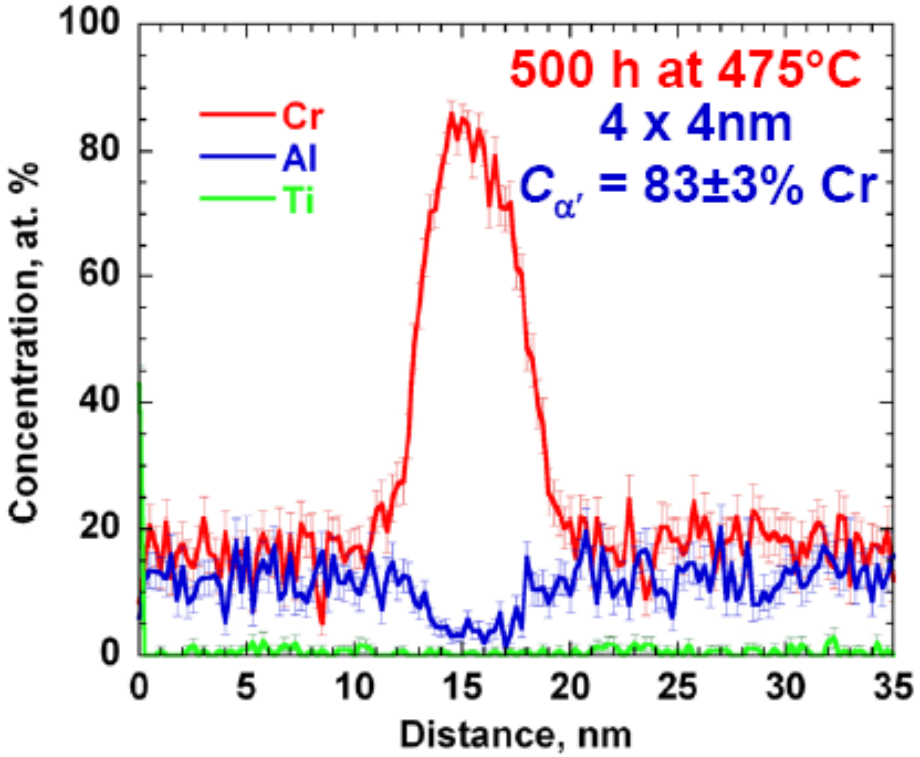
α´ phase exhibits some interconnectivity at early stages, but it becomes spheriodal and its size increases on further ageing.



APT results

α' phase composition

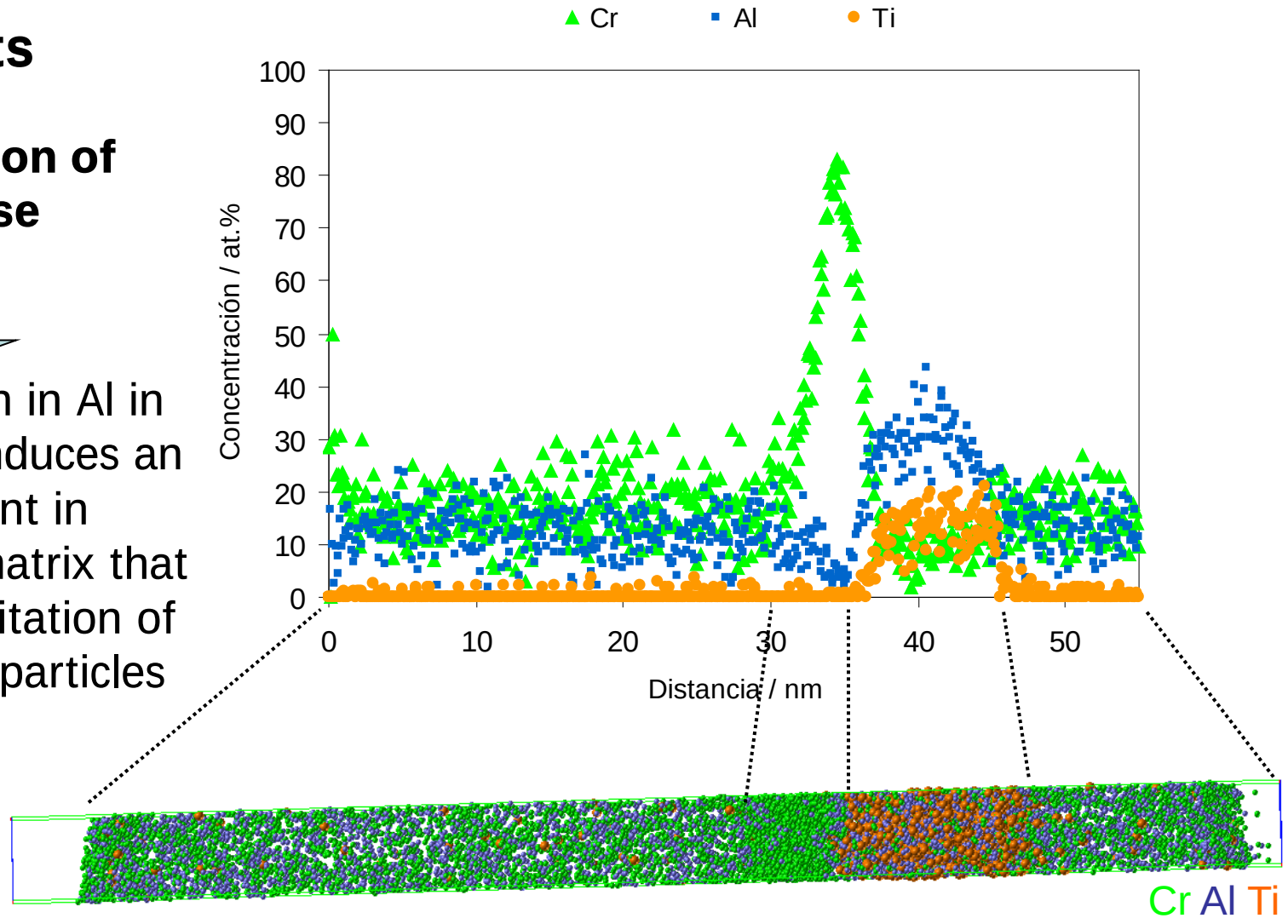




APT results

Precipitation of β' phase

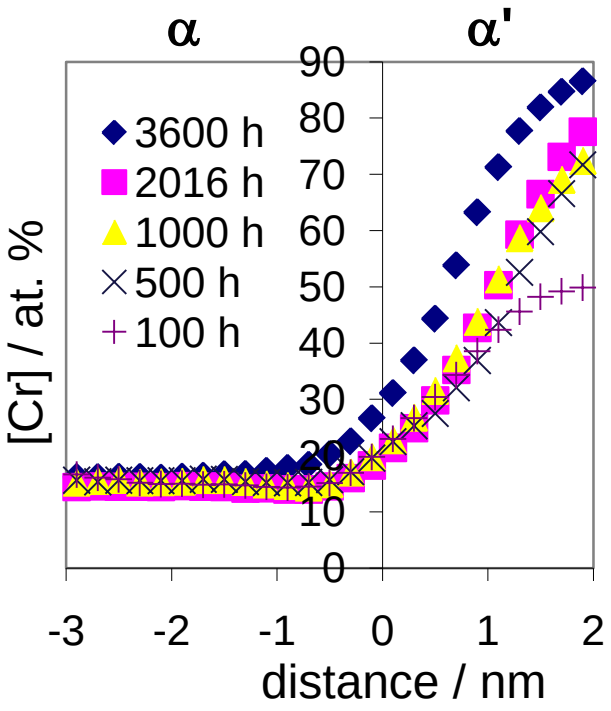
The depletion in Al in α´ particles induces an enrichemnt in surrounding matrix that causes precipitation of nanosized β' particles

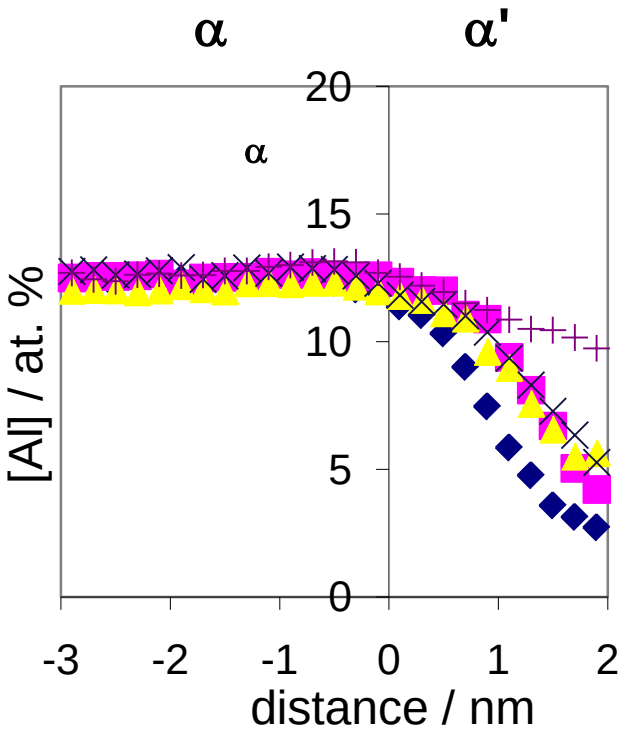


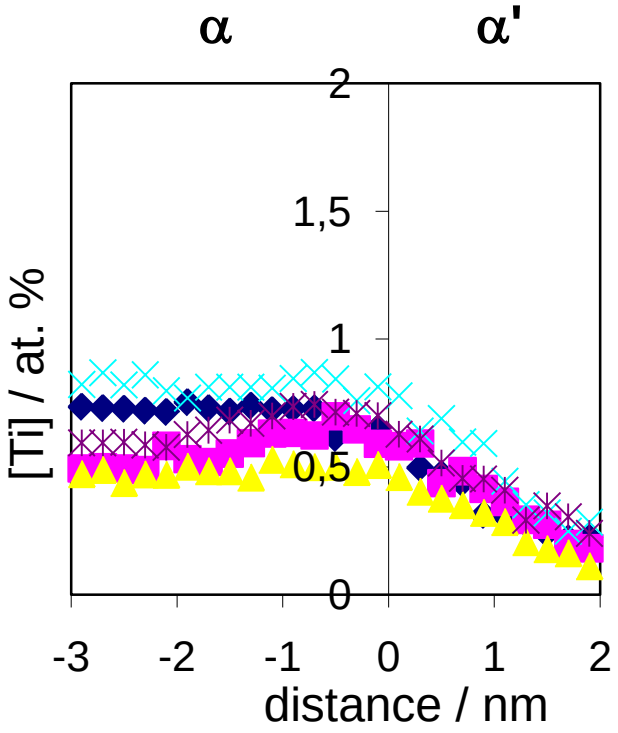
Volumen 4x4x57nm

APT results

Proximity histograms analysis





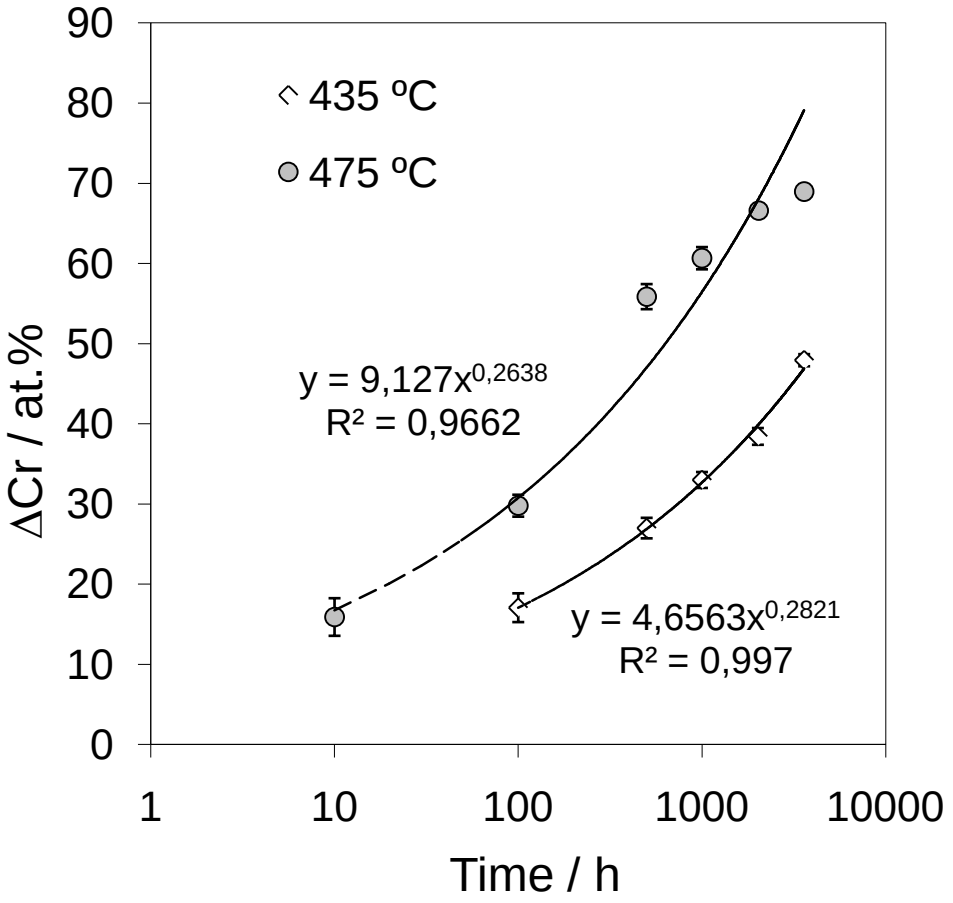


475 ºC

APT results

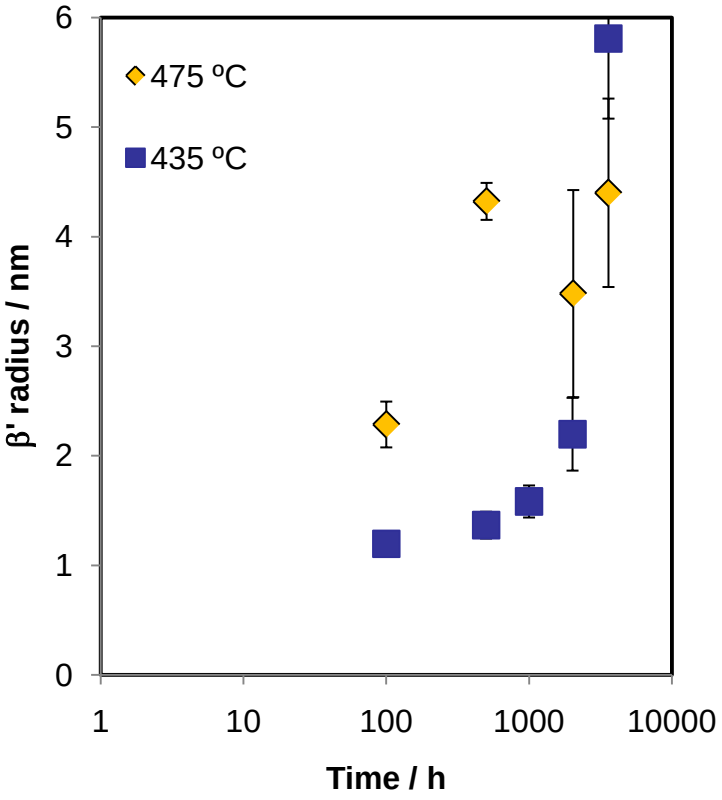
The kinetics of α - α´ phase separation process in PM 2000 were determined from the analysis of proximity histogram

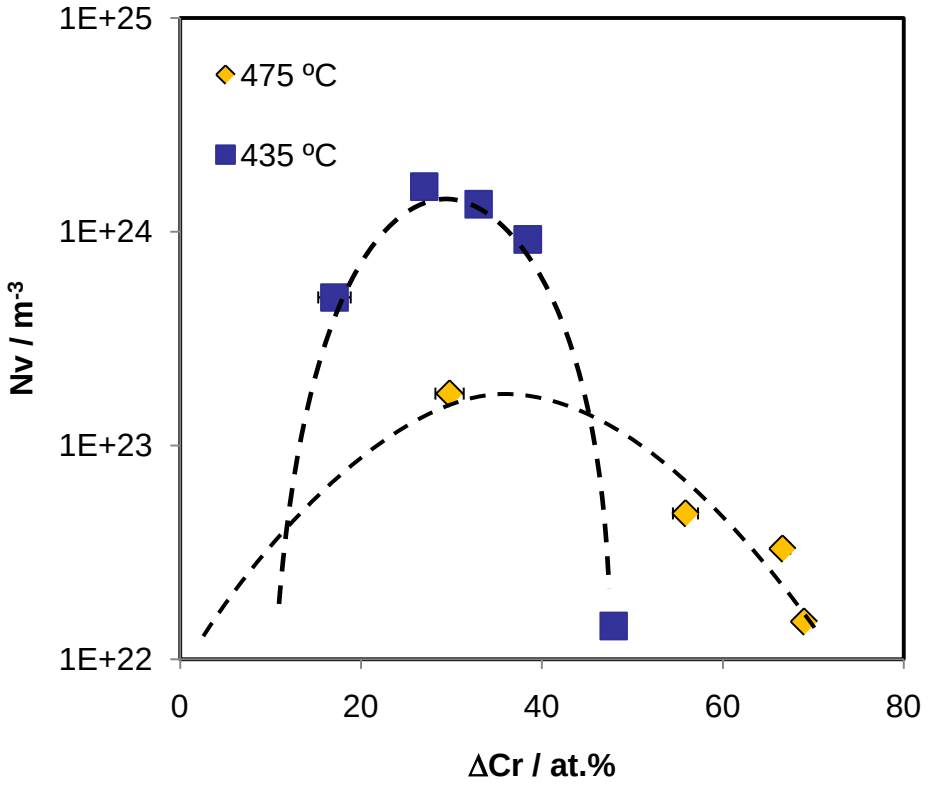
The size of the α´ increases with a time exponent of 0.32 which is consistent with the mean precipitate size R(t) varying as $\sim t^{1/3}$ predicted by the LSW theory.



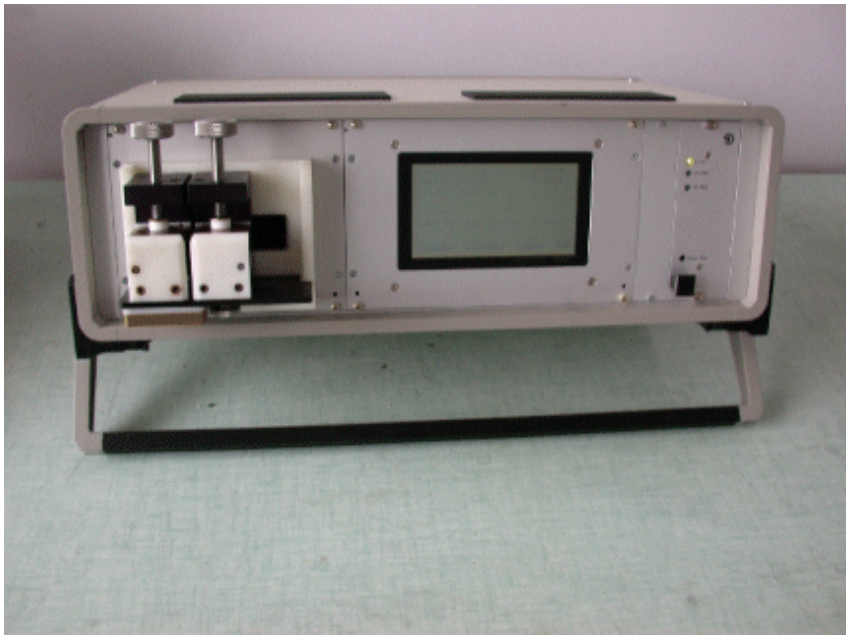
APT results

Maximum separation method

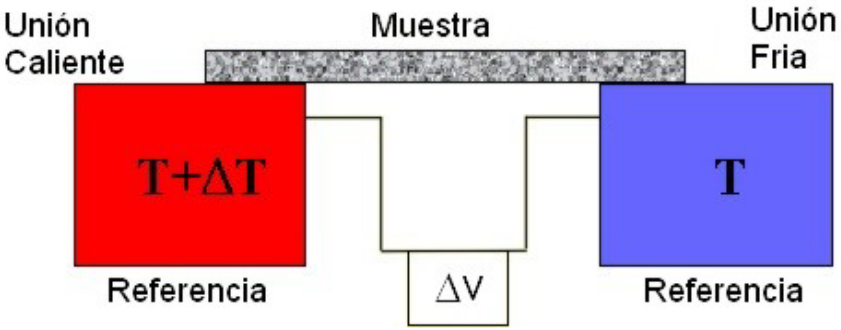




TEP results

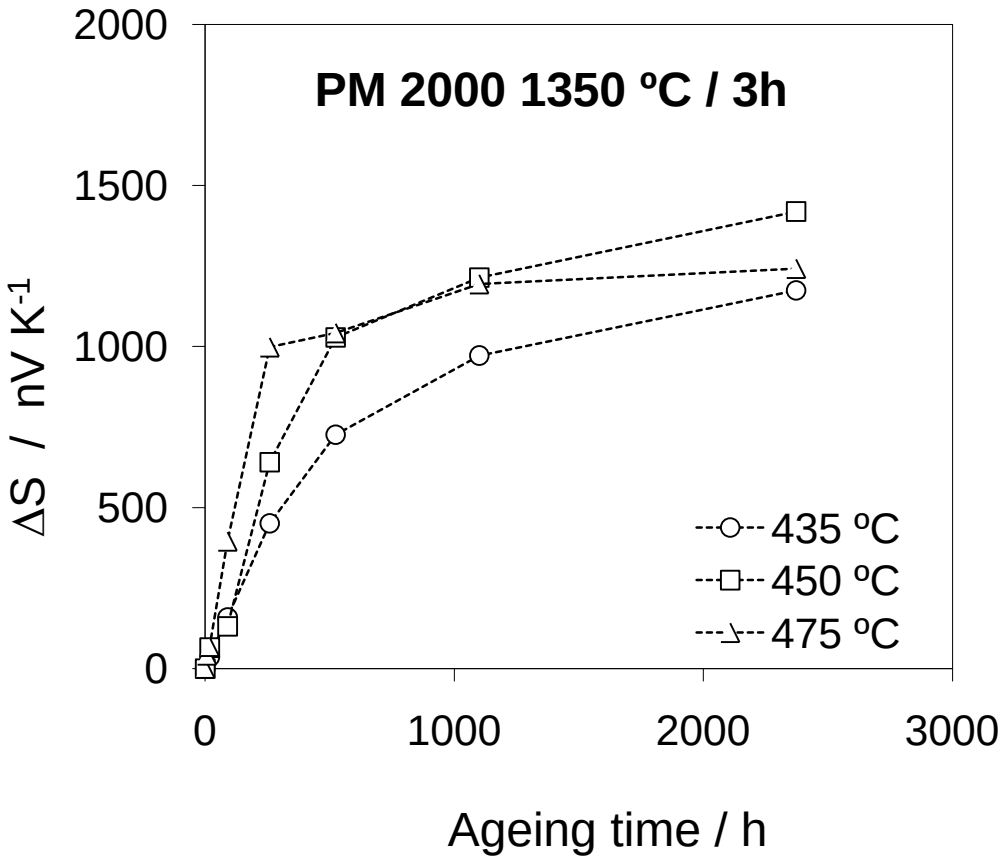


Thermoelectrical Power (TEP) measurements have been used to track microstructure evolution



$$\Delta S = \frac{\Delta V}{\Delta T} \text{ in nV/K}$$

TEP results


PM 2000 1350 ºC / 3h
ΔS / nV K-1
2000
1500
1000
500
0
0
1000
2000
3000
Ageing time / h
435 ºC
450 ºC
475 ºC

TEP results

Johnson-Mehl empirical equation

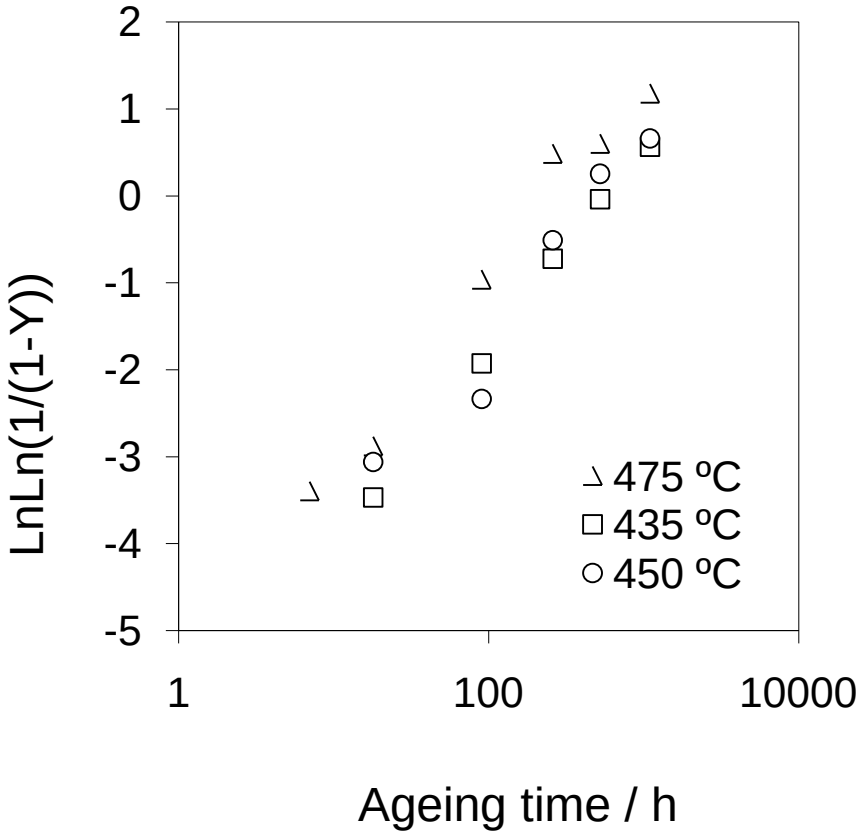
$$Y = 1 - \exp(-Kt^n)$$

where

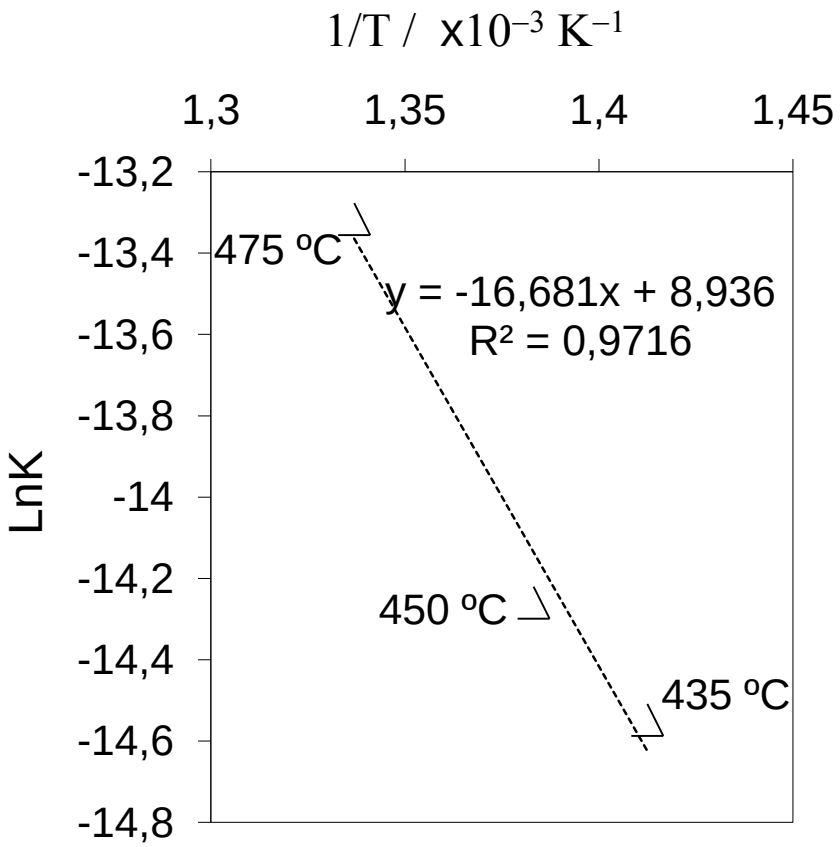
$$Y = \frac{(\Delta S - \Delta S_i)}{(\Delta S_f - \Delta S_i)}$$

and K follows an Arrhenius equation:

$$K = \ln K_0 - \frac{Q}{RT}$$



TEP results



Activation energy for α - α´ phase separation process in PM 2000

$Q = 138\ kJ\ mol^{-1}$

Conclusions

1. Atom probe tomography revealed that a nanometer-scaled phase separation betweeen α and α′ phases is the predominant mechanism responsible for strengthening a PM 2000 ODS alloy during isothermal aging at 435 and 475 ºC.

2. 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).

3. The maximum separation method estimated the size and number density of β′ particles. The β′ particles are more abundant and finer at 435 ºC than that at 475 ºC.

4. The TEP measurements is an easy and reliable technique to monitor the phase separation process in PM 2000. A value of the activation energy of 138 $kJmol^{-1}$ has been determined, which is consistent with that obtained by DSC.

Acknowledgemnts

Atom probe research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by Basic Energy Sciences, U.S. Department of Energy.

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