Steel composites for energy generation systems

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ShaRE
Shared Research Equipment User Facility
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Background

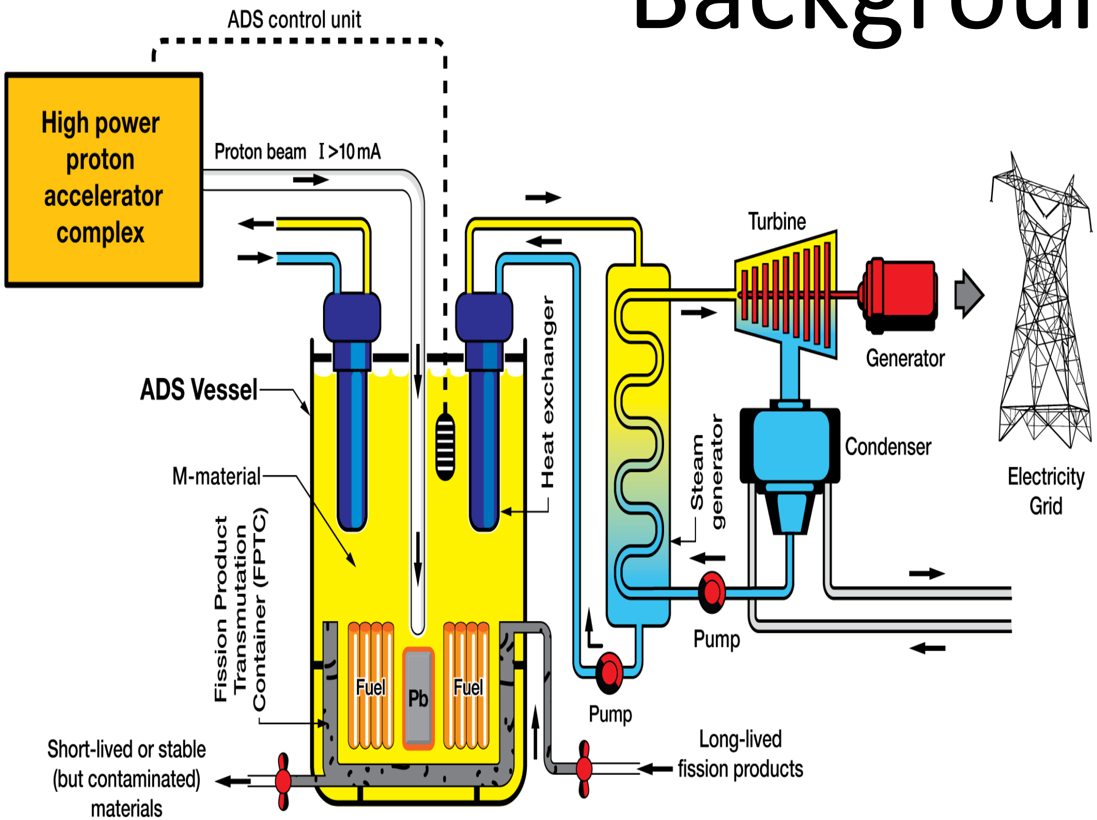
- The need to reduce CO_2 emissions coupled with the need to increase the quantity of electricity supplied are driving to the development of new power generation systems.

- Significant gains in efficiency for power generation systems can be made by increasing the steam temperatures and pressures. This lead to an improvement of the high-temperature properties of current heat resistant alloys.

- The low creep resistance at high temperatures of Fe-base alloys could be mainly improved by different methods:

  - One method consists on a combination of composition adjustments, guided by computational thermodynamics, and thermo-mechanical control process (TMCP) optimization.
  - Second method is to strength the steel by oxide dispersion, and this line led to work on ferritic oxide dispersion-strengthened (ODS) alloys. The advantages of ODS alloys at high temperatures are clear: high strength and high creep resistance.
  - Third method consists on compositional tunning to induce the formation of nanoclusters and nanophases. An example is illustrated here.

Background



(S.R. Hashemi-Nezhad, University of Sydney, Australia)

- Fe-Cr-Al-Ti ODS alloys have promising properties where corrosion and creep resistance is paramount, i.e. beam windows in subcritical accelerator driven systems (ADS) nuclear reactors.
- Phase separation of α (Fe-rich) - α' (Cr-rich) during service affect mechanical properties.

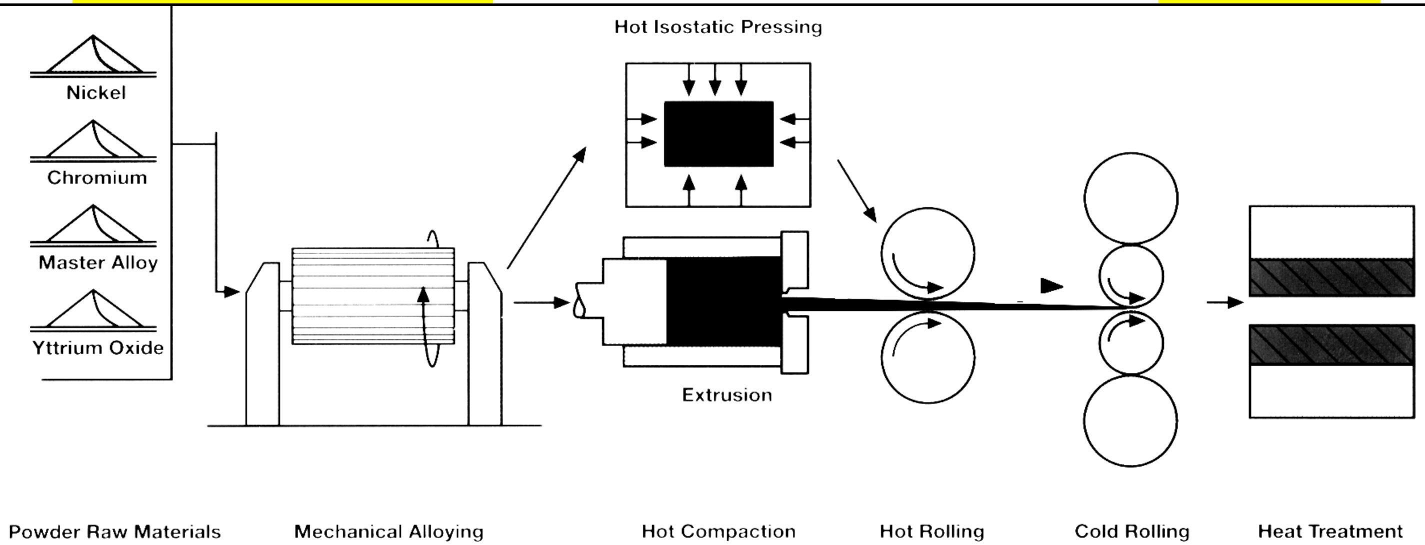
- Topics included in this presentation and its effect on phase separation:
  - Activation energy of α - α' phase separation
  - Novel nanophase formation of β' (Fe-Ti-Al precipitate)
  - Effect of elastic stress on α - α' + β' precipitation

Material

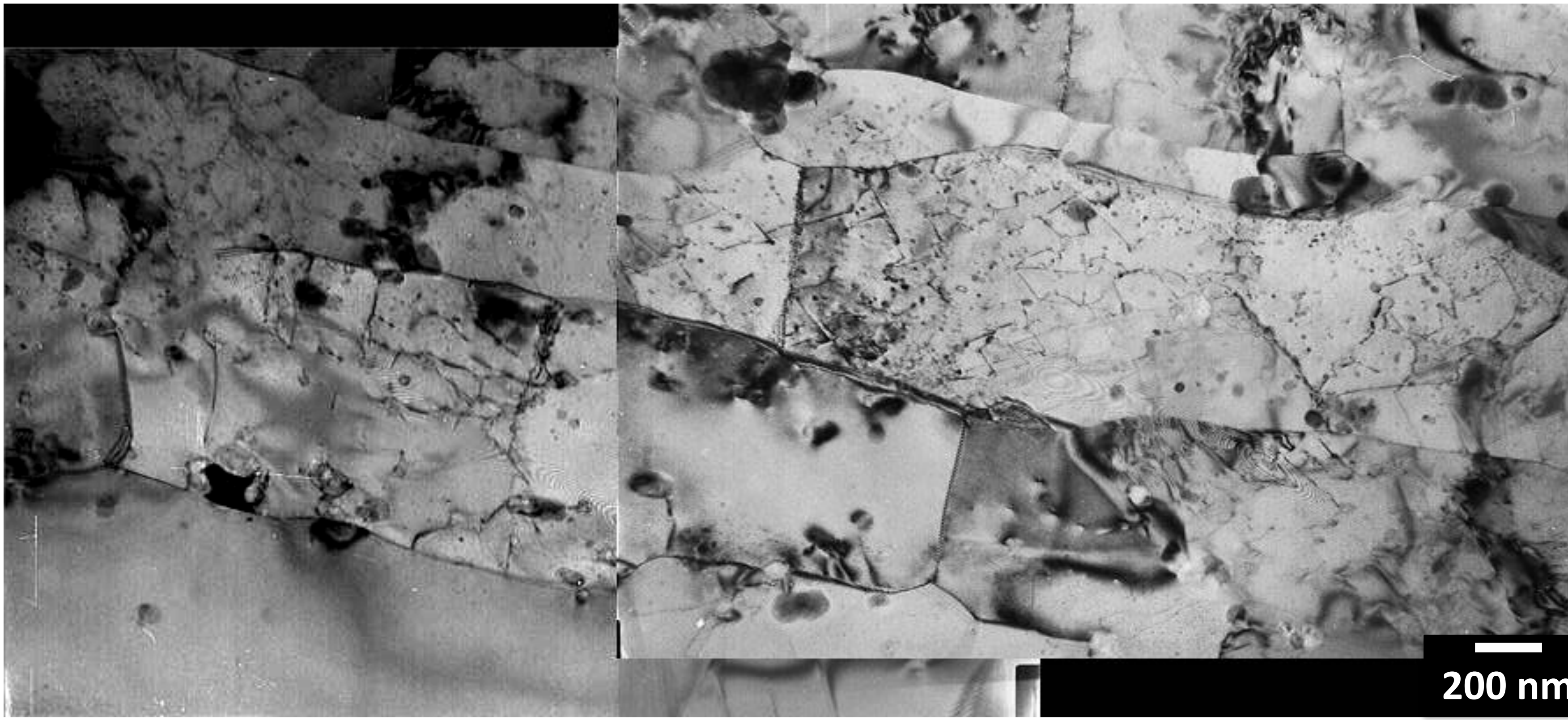
PM 2000tm is a commercial Fe-base ODS alloy manufactured by PLANSEE in Lechbruck, Germany

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



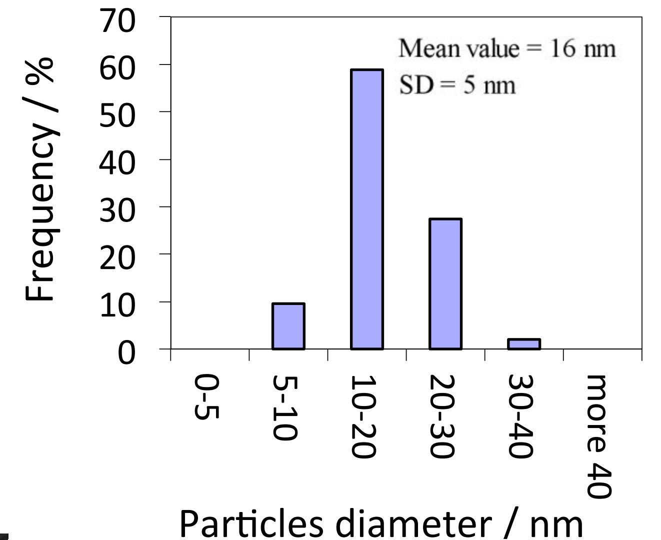
Microstructure

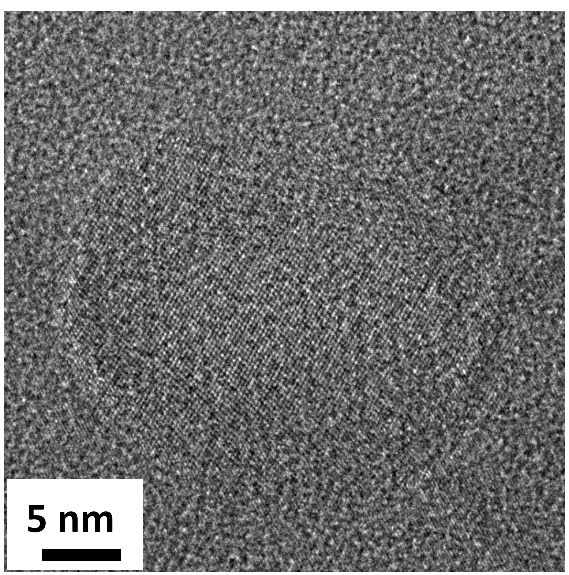


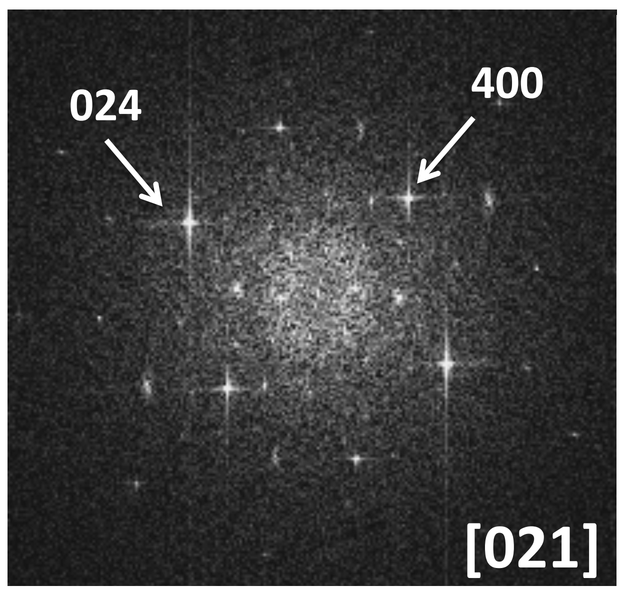
Anisotropic microstructure: Strongly textured (<110>||RD) elongated grains with high-dislocation density and homogenous distribution of particles

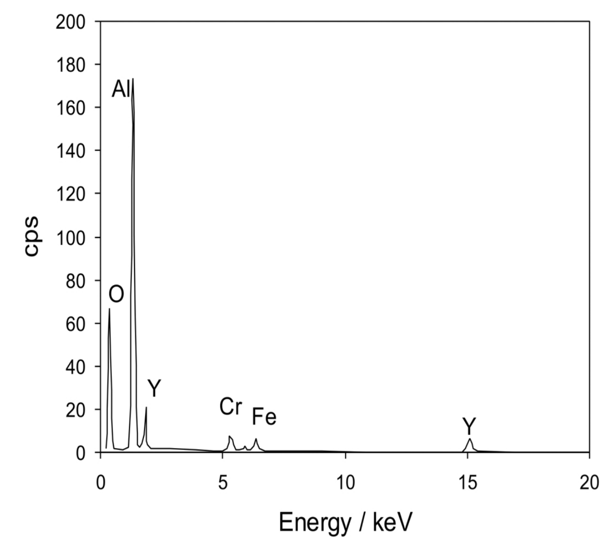
Microstructure

- The structure and composition of initial yttria particles is modified during mechanical alloying
- Particles in as-received condition:
  - Composition $Y_3Al_5O_{12}$ and garnet structure
  - Sizes: 3-40 nm

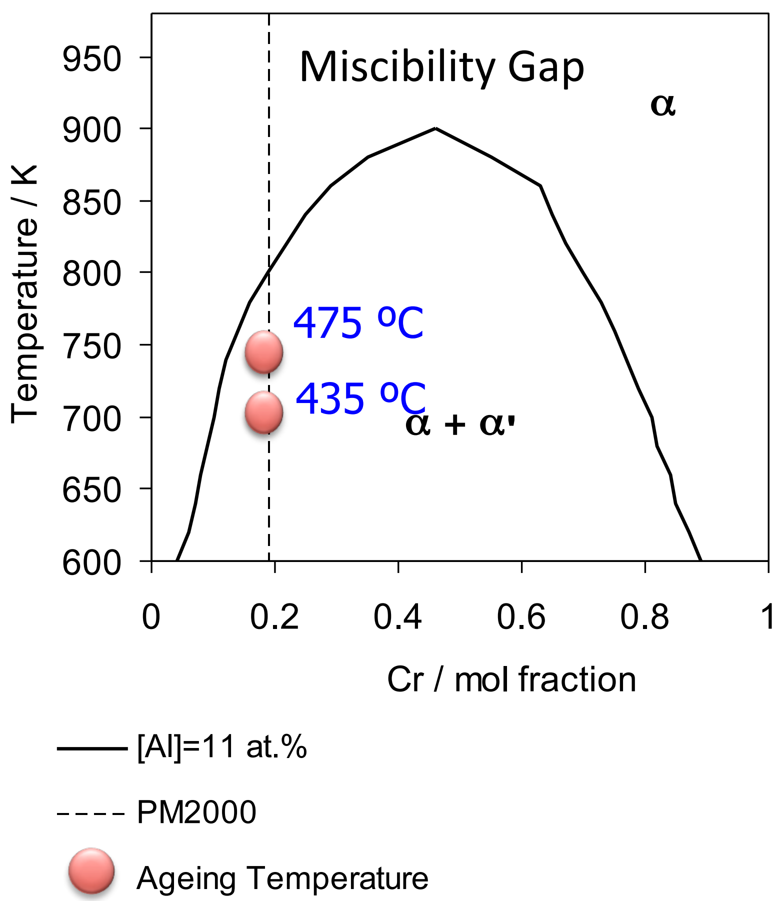


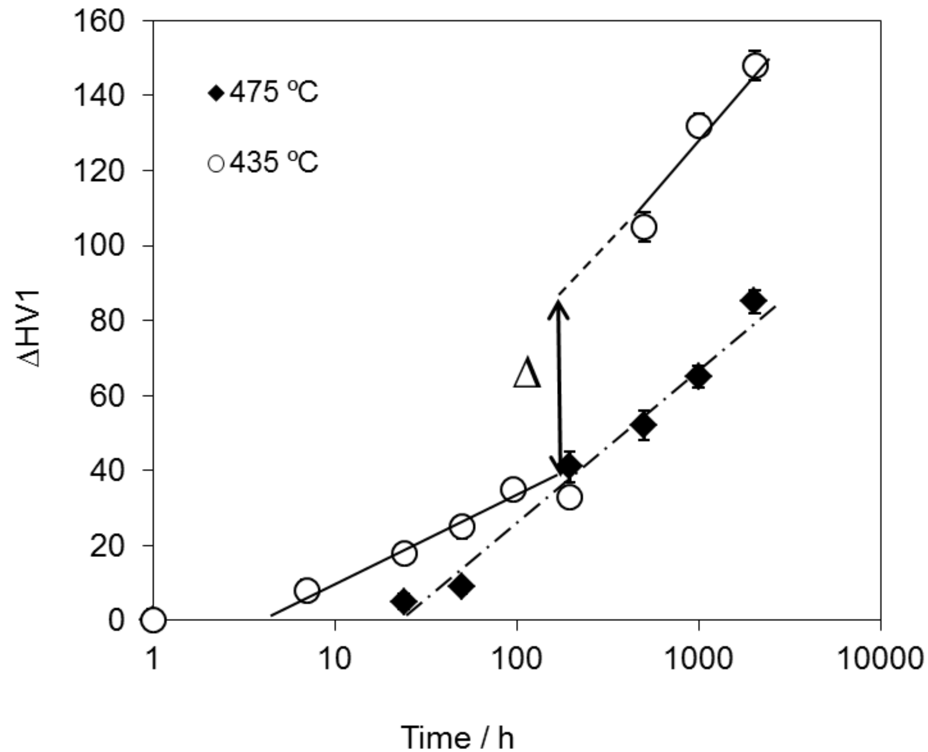






Hardening



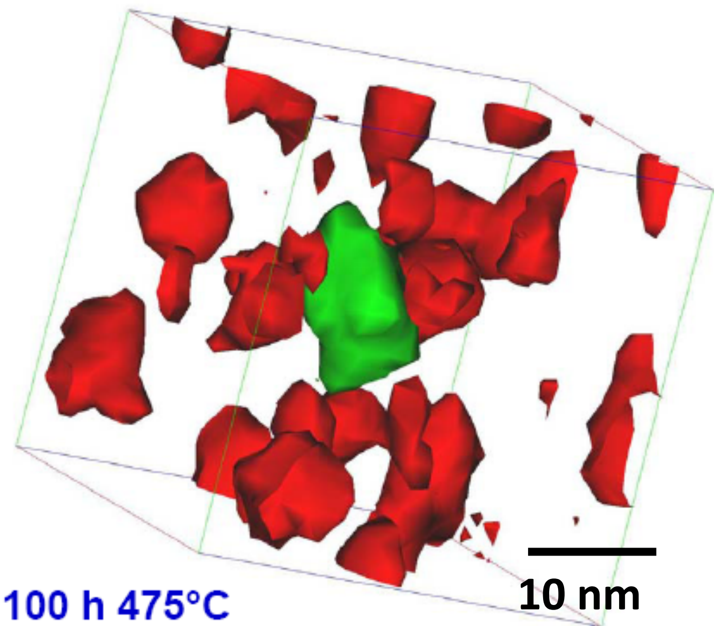


Annealing at temperatures below 500 ºC induce hardening. Surprising increase at 435 ºC is observed.

APT Results

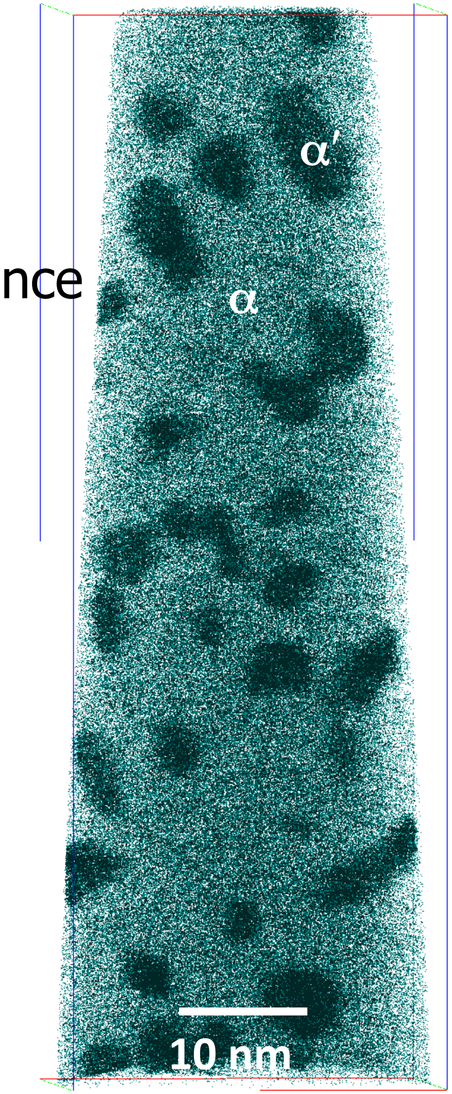
Red 30% Cr isoconcentration surface revealing the distribution and spherical morphology of Cr-rich α' phase.

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



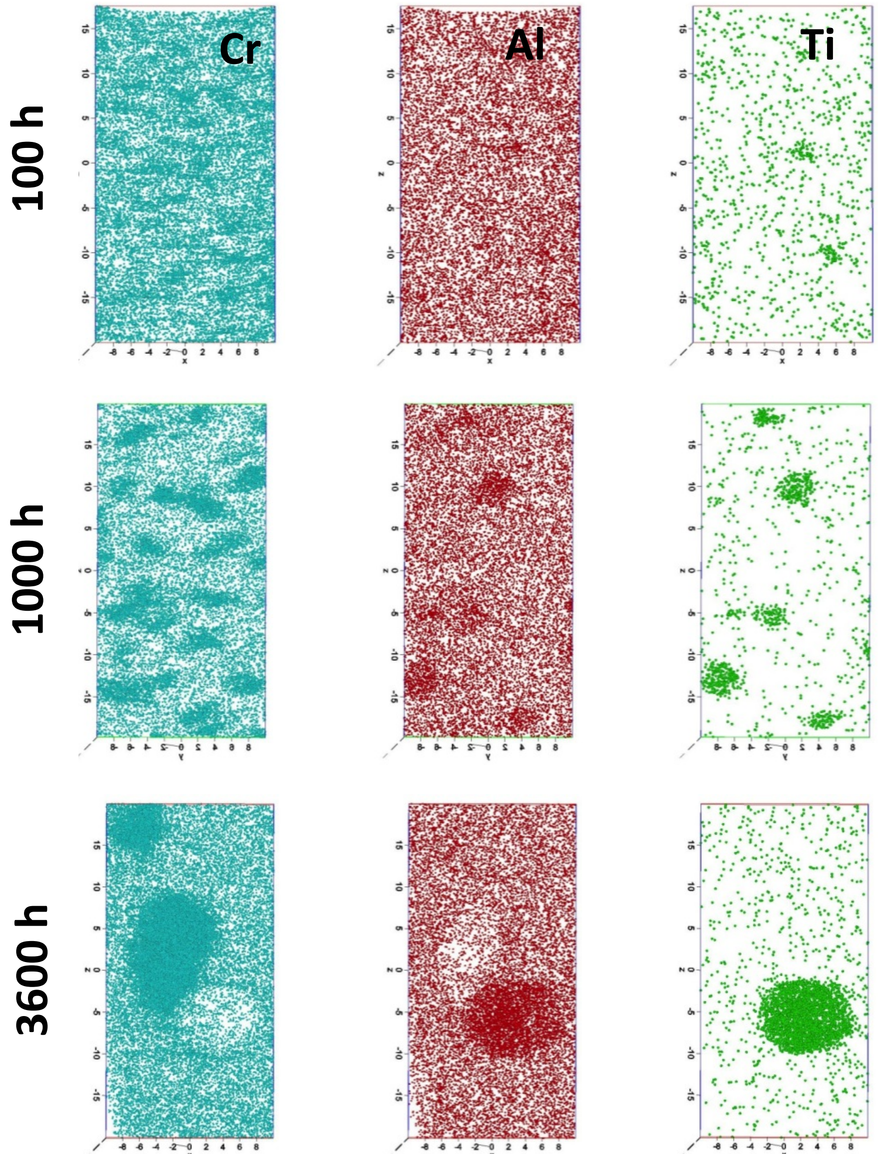
Cr atom map

2040 h 475 ^{o}C



C. Capdevila, M.K. Miller, K.F. Russel, J. Chao, J.L. González-Carrasco, Mater. Sci. Eng. A (2008) 490 277–288.

Nanoparticles at 435 ºC

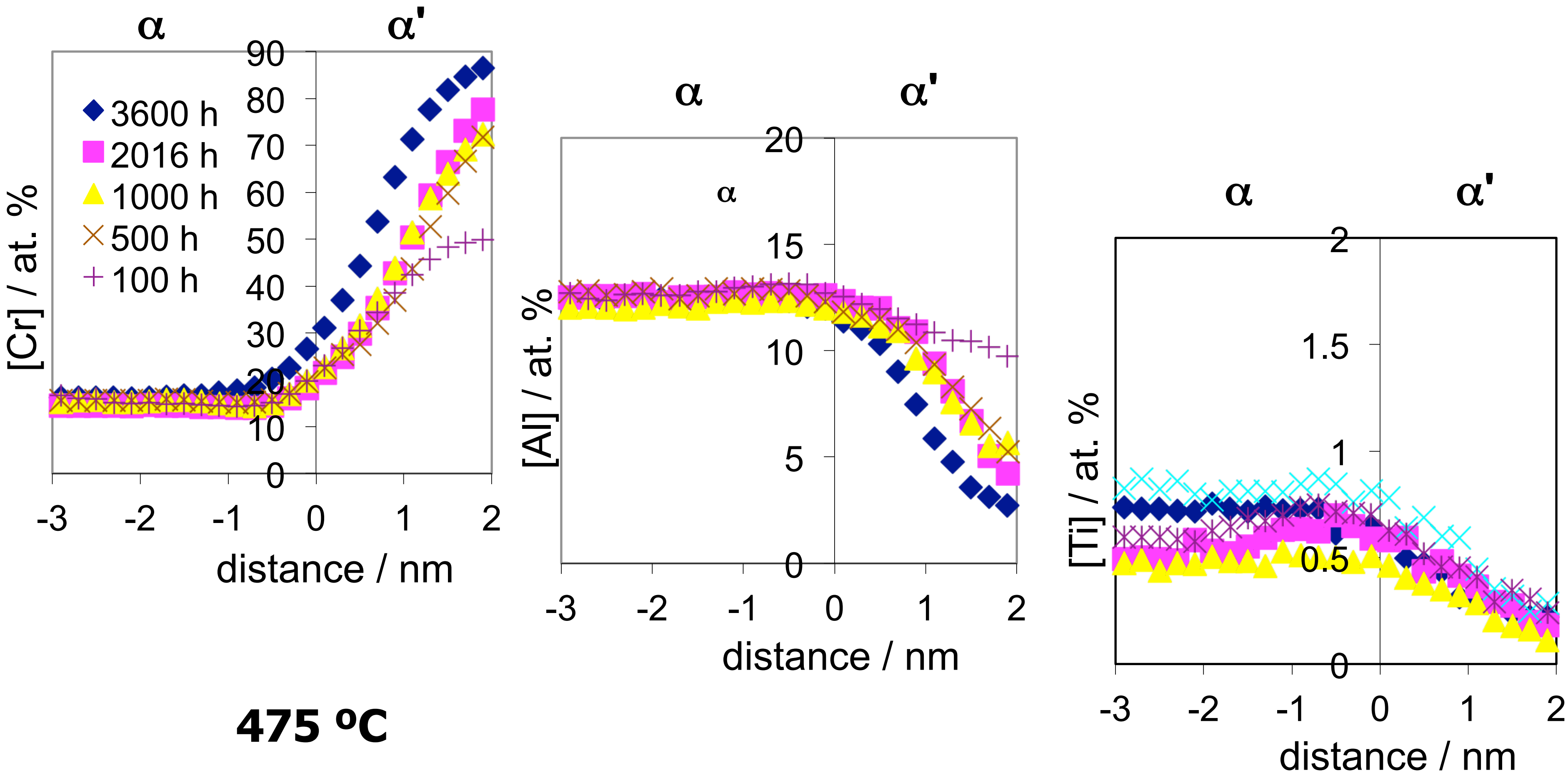


- 4-nm-thick atom maps for selected times (volume of 4×20×40 nm^3)
- The Cr-enriched α' regions were found to be depleted in Al
- The phase separation is of a finer scale and is less well developed at lower ageing temperatures
- The atom maps also reveal the presence of a Ti- and Al-enriched phase (β'). This phase is present at a significantly lower number density compared to the Cr-enriched α' phase, but its number density increases at lower ageing temperatures

C. Capdevila, M.K. Miller, I. Toda, J. Chao, Mater. Sci. Eng. A (2010) 527 7931-7938.

α′-phase composition

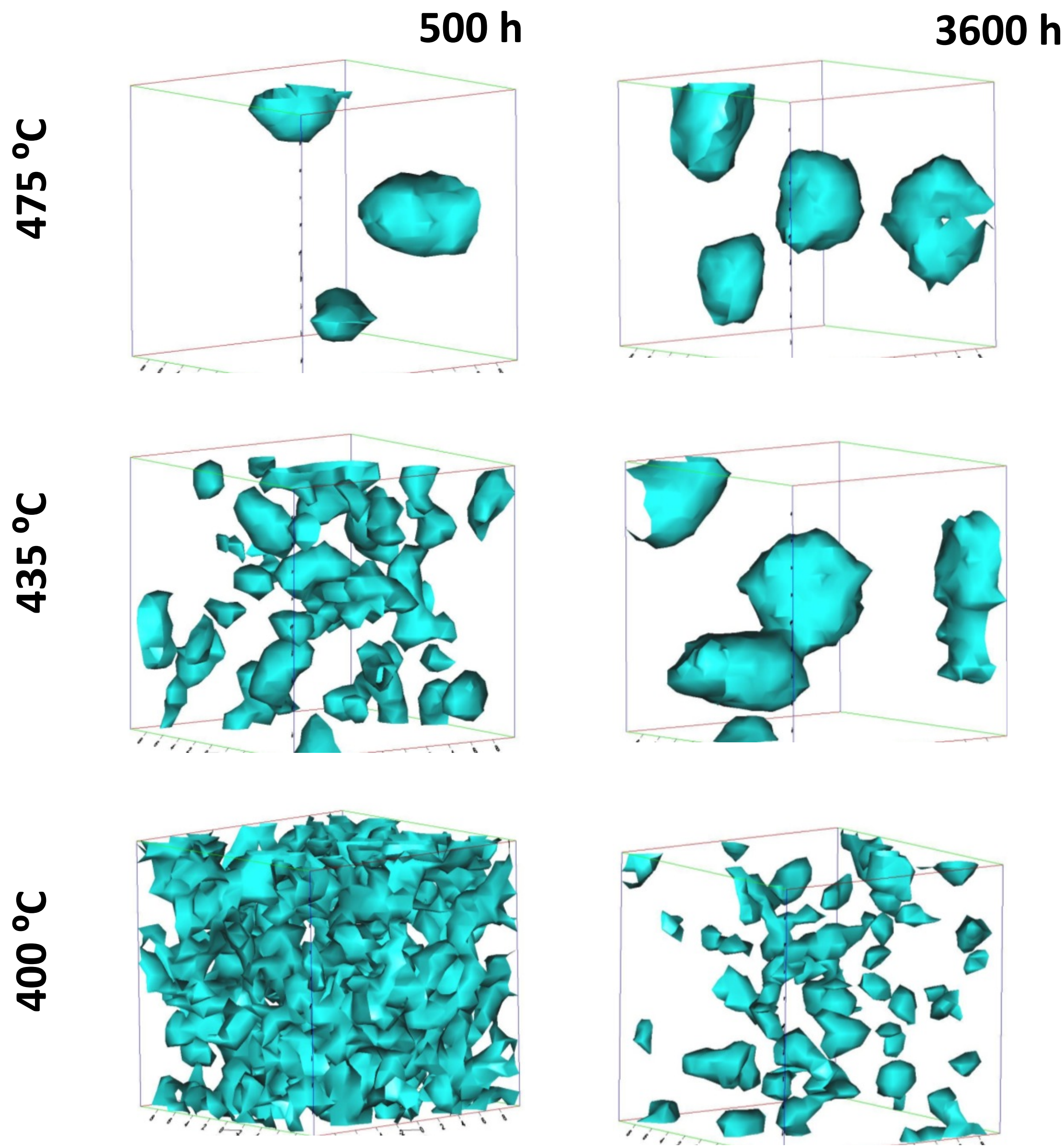
Proximity histograms analysis



475 ºC

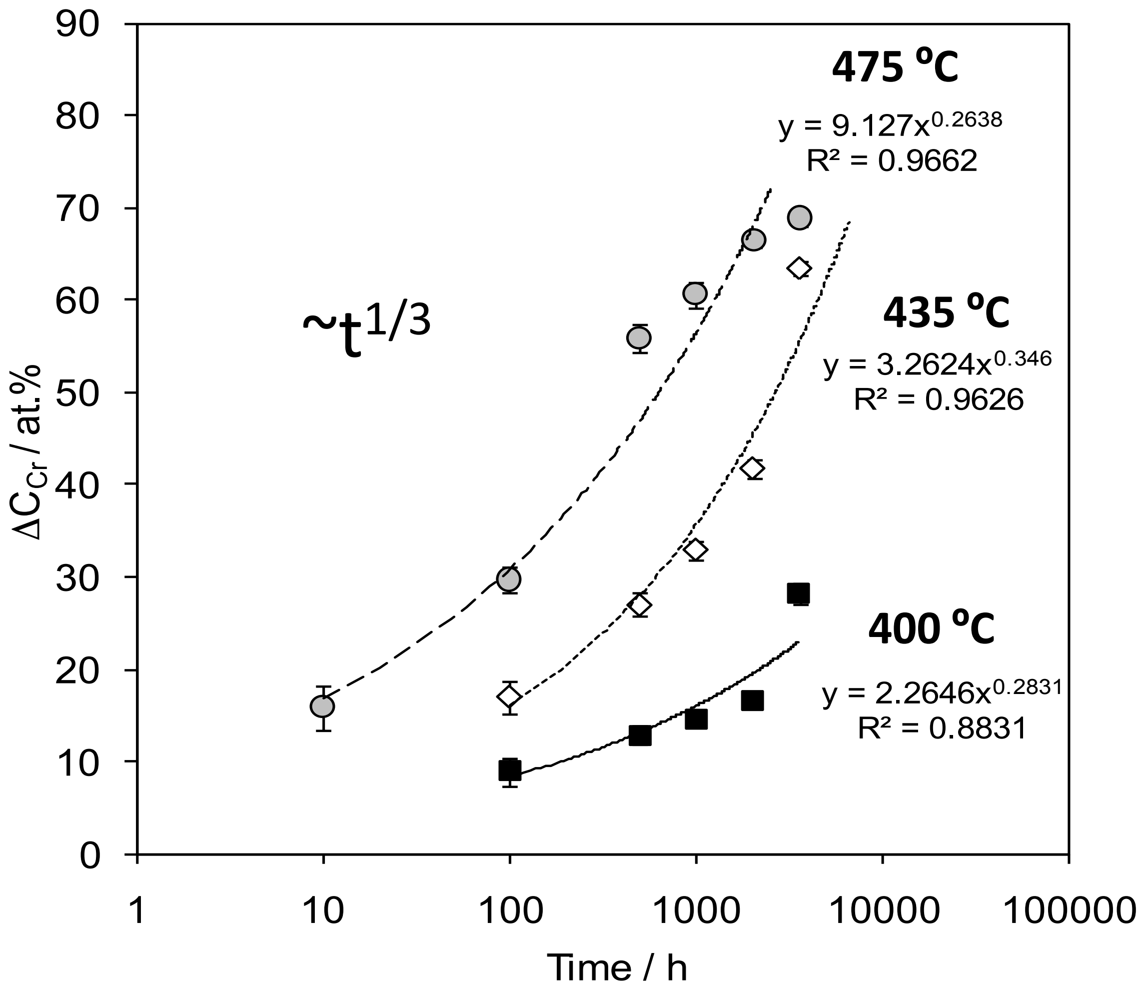
C. Capdevila, M.K. Miller, K.F. Russell, J. Mater. Sci. (2008) 43 3889-3893.

α'-phase morphology



- The spheroidal α' particles observed at 475°C are clearly isolated and do not form a percolated microstructure
- At the lower ageing temperatures the morphology of the Cr-enriched α' phase after 3600 h is of a finer scale and forms an interconnected network

α'-phase kinetics

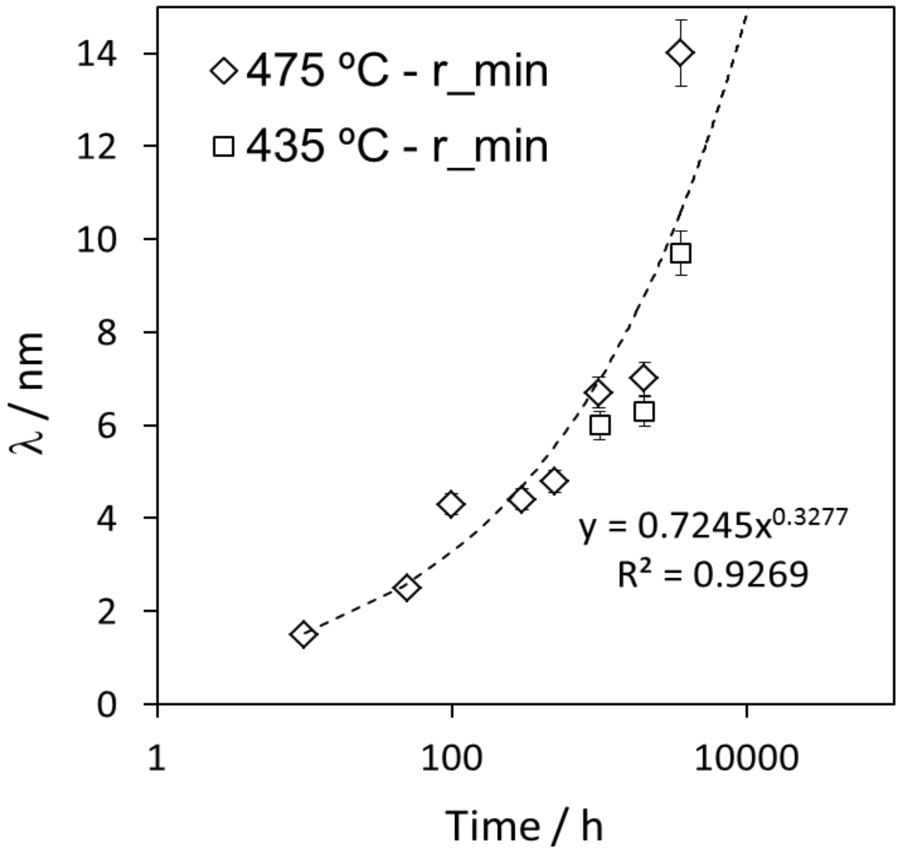


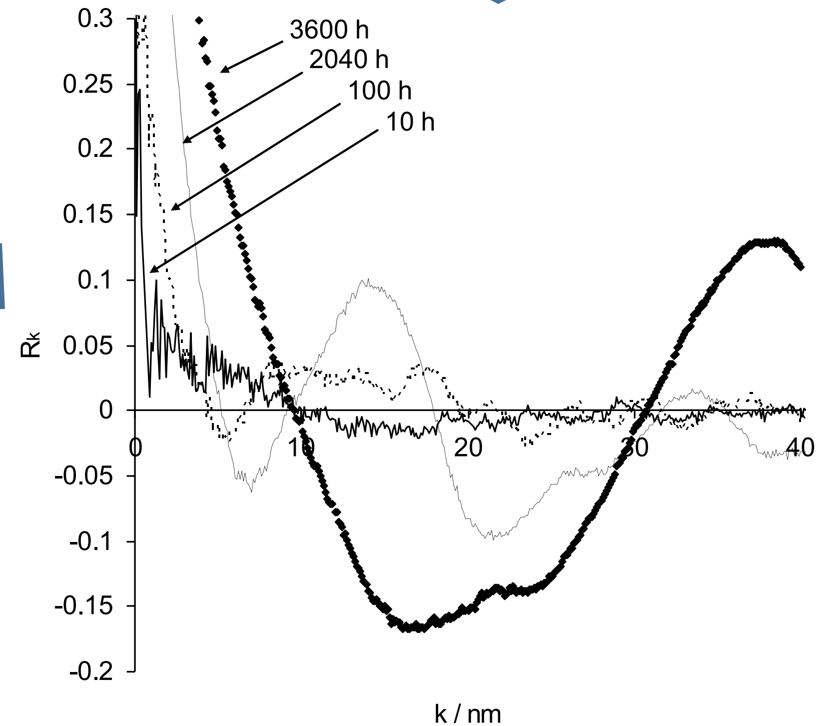
The kinetics of α - α' phase separation process were determined from the analysis of proximity histogram

α′-phase kinetics

α′- size

The size of the α′ particles (λ) is determine by the first minima (r_min) of the autocorrelation function

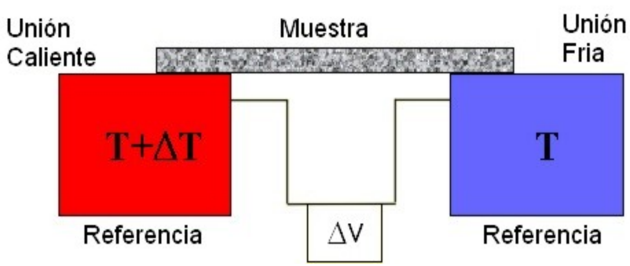




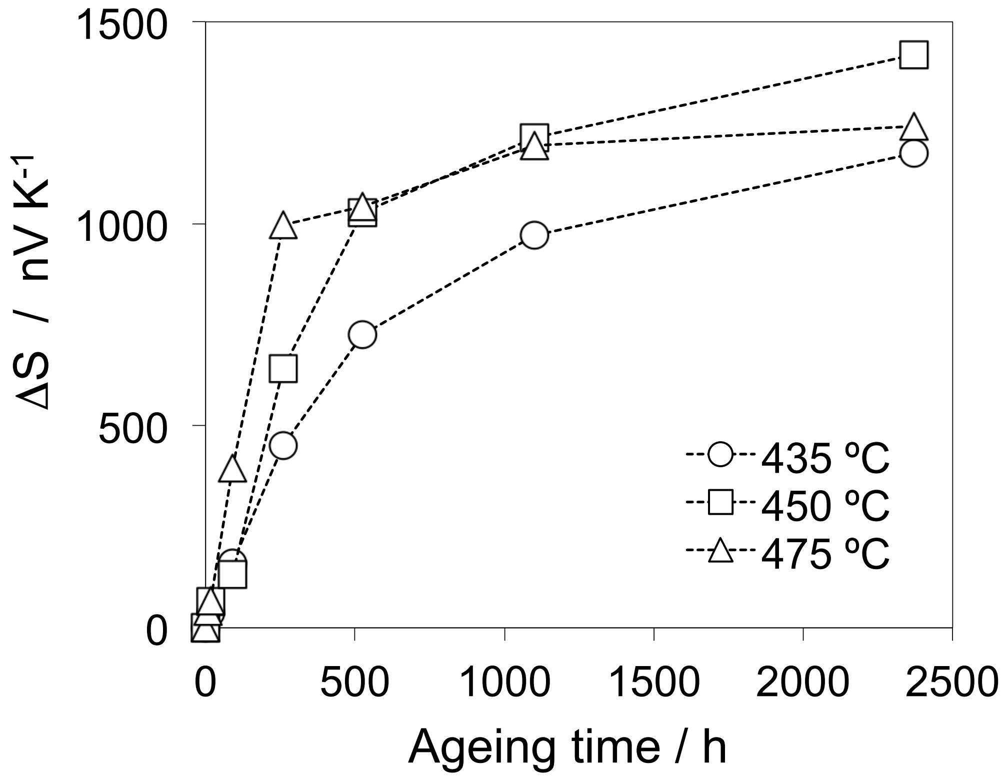
The size of the α′ increases with a time exponent of ~0.3 which is consistent with the mean precipitate size R(t) varying as ~$t^{1/3}$ predicted by the LSW theory.

Activation energy for α'-phase

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



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



Since volume fraction of β' is significantly lower than α', the influence of β' on TEP is negligible.

C. Capdevila, M. K. Miller, G. Pimentel , J. Chao, Scr. Mater. (2012) 66 254-257

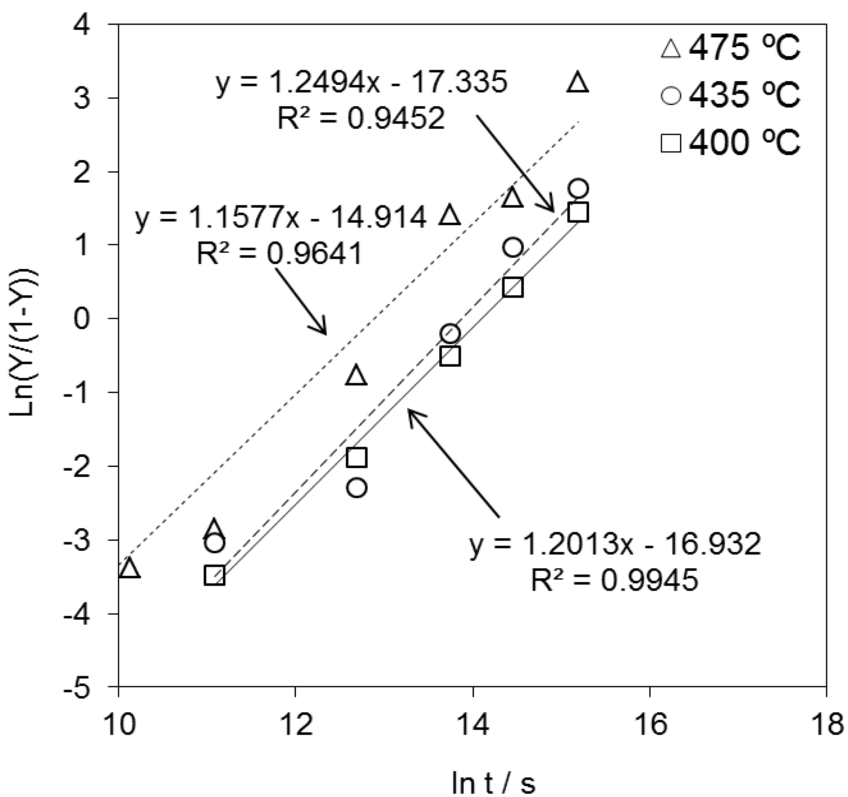
Activation energy for α'-phase

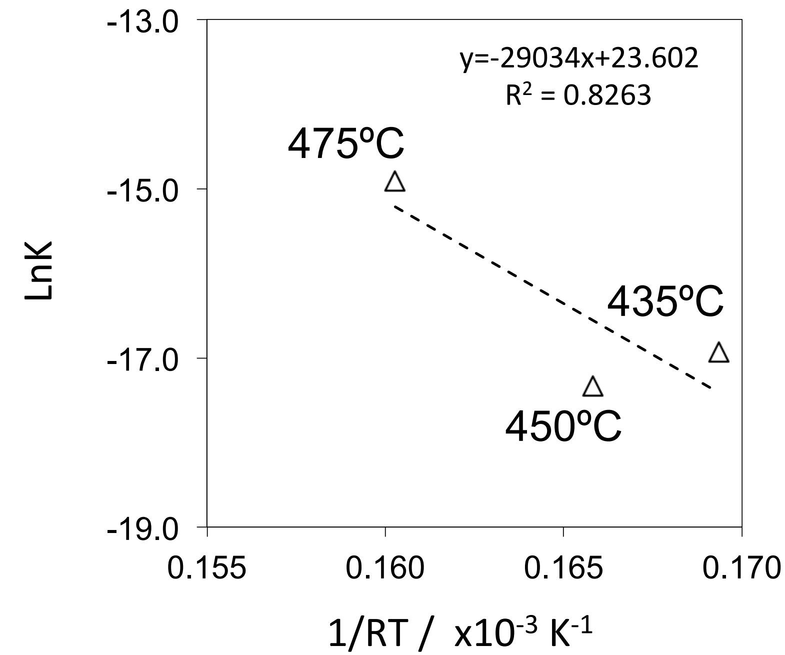
Fitting TEP (ΔS) to Austin-Rickett equation:

$$\frac{f(t)}{1-f(t)} = K(T)t^n$$

$$f(t) = \frac{(\Delta S - \Delta S_i)}{(\Delta S_f - \Delta S_i)} \quad ,, f(t) \equiv Y$$

$$K(T) = k_o \exp\left\{-\frac{Q}{RT}\right\}$$





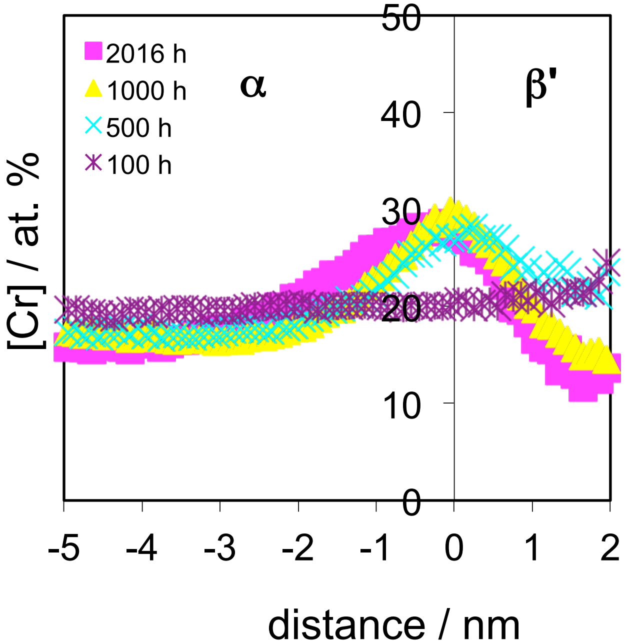
Q = 242 kJ mol^{-1} ,, n=1.2

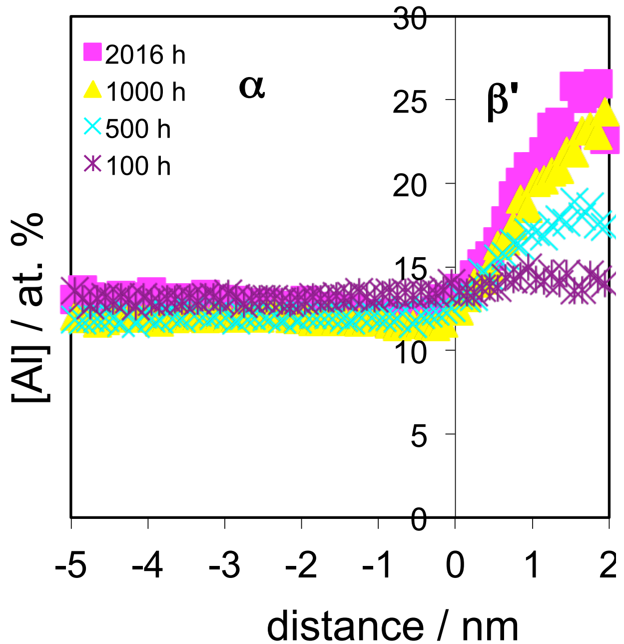
Activation energy for Cr self-diffusion in Fe → 248 kJ mol^{-1}

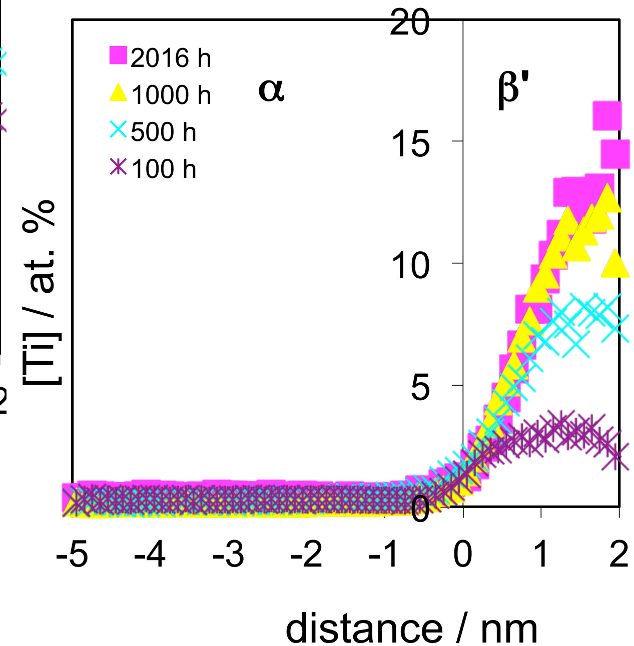
C. Capdevila, M.K. Miller, J. Chao, Acta Mater. (2012) 60 4673–4684

Novel β′-phase: Composition

Proximity histograms analysis







435 ºC

Nature of β'-phase

Composition of β' phase:

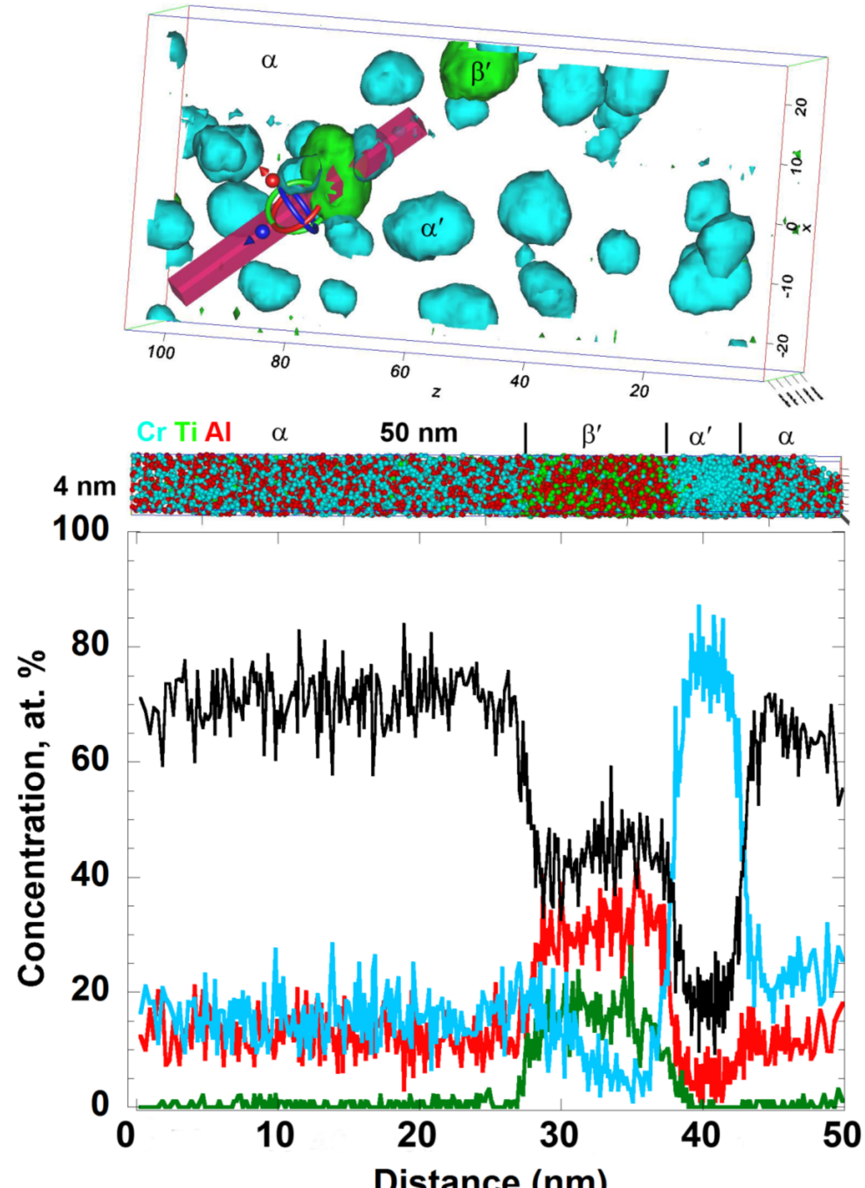
Fe = 49 at.%

Al = 25 at.%

Ti = 15 at.%

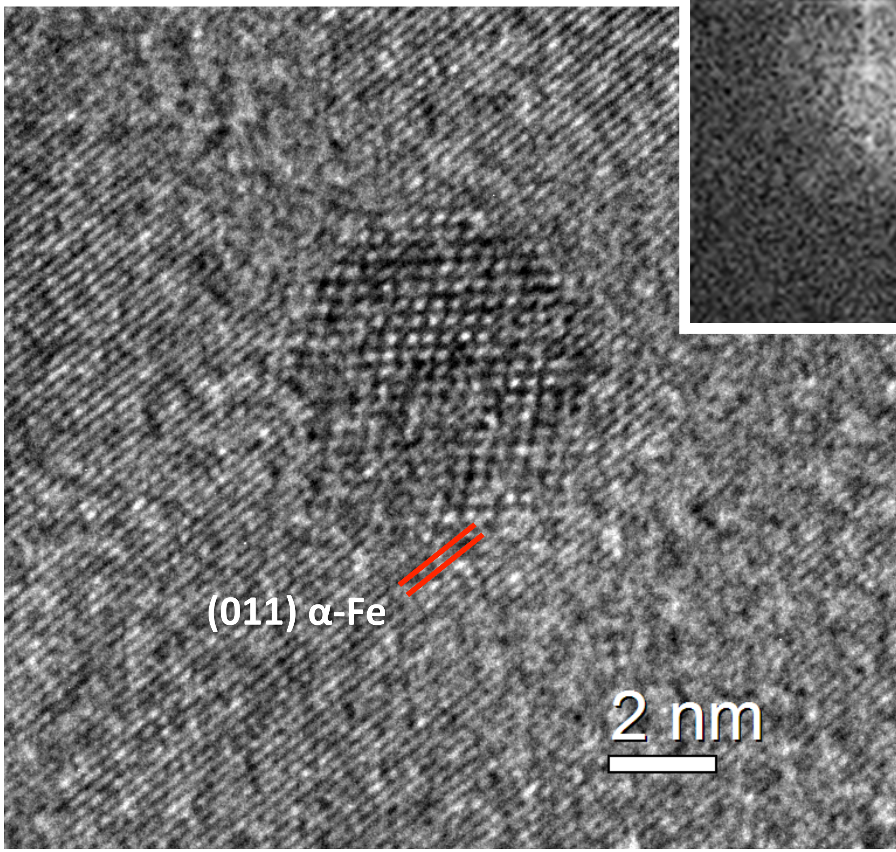
Cr = 11 at.%

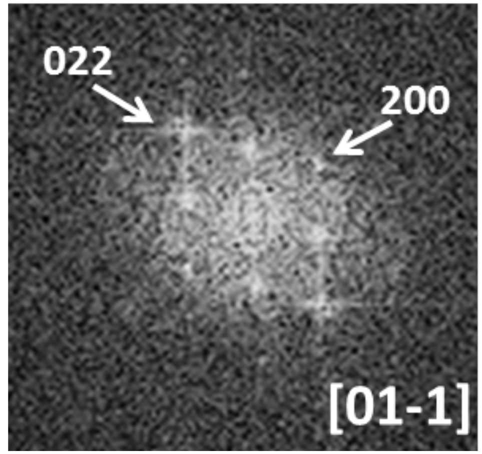
$Fe_2 (Al\ Ti_{0.6}\ Cr_{0.4})$



Nature of β′-phase

HRTEM Results





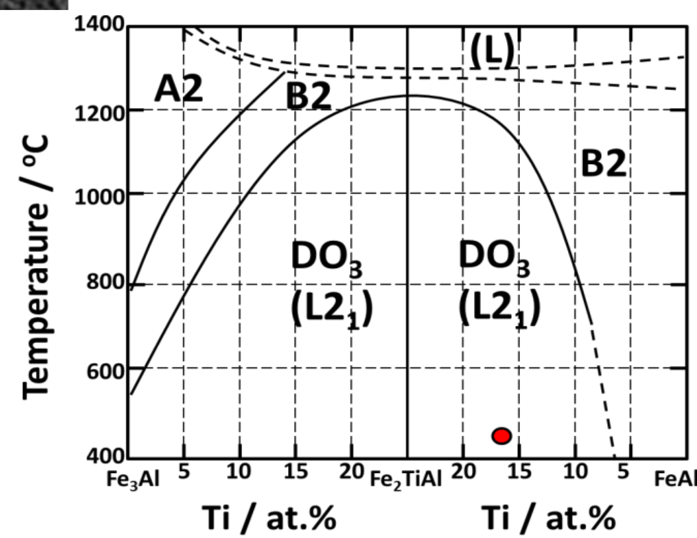
(FFT)

Solid-solution type:

$Fe_{(2-x)}\ Al_{(1+0.5x)}Ti_{(1+0.5x)}$

Cubic space group Fm3m

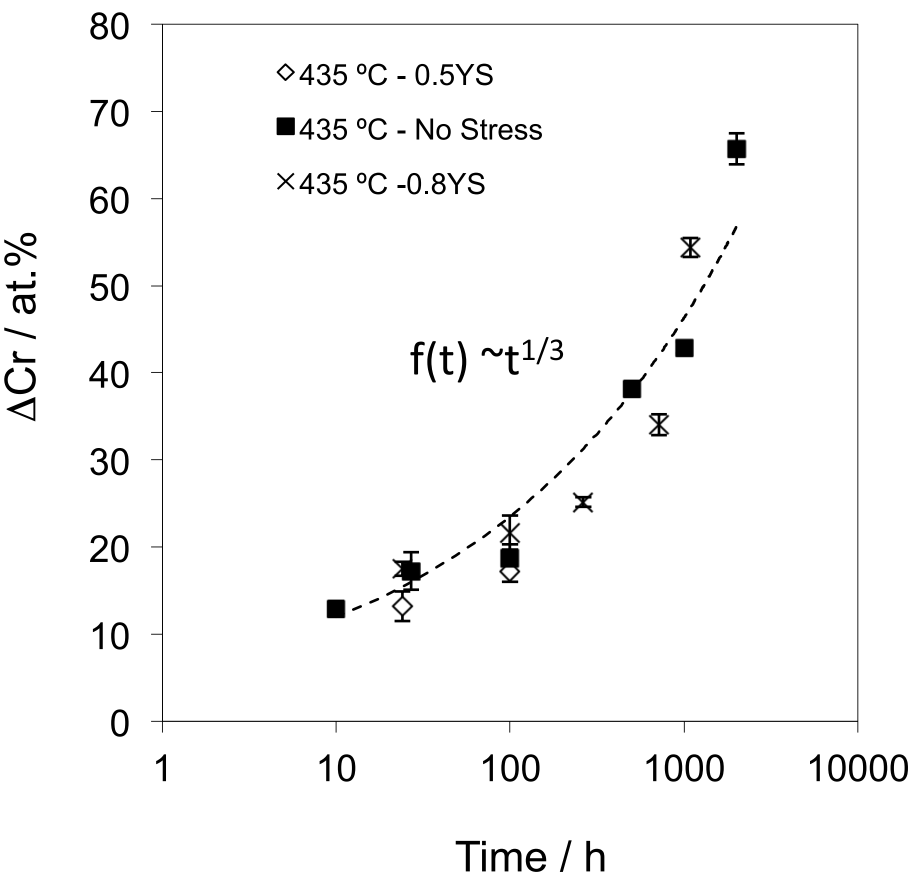
a=0.5879 nm



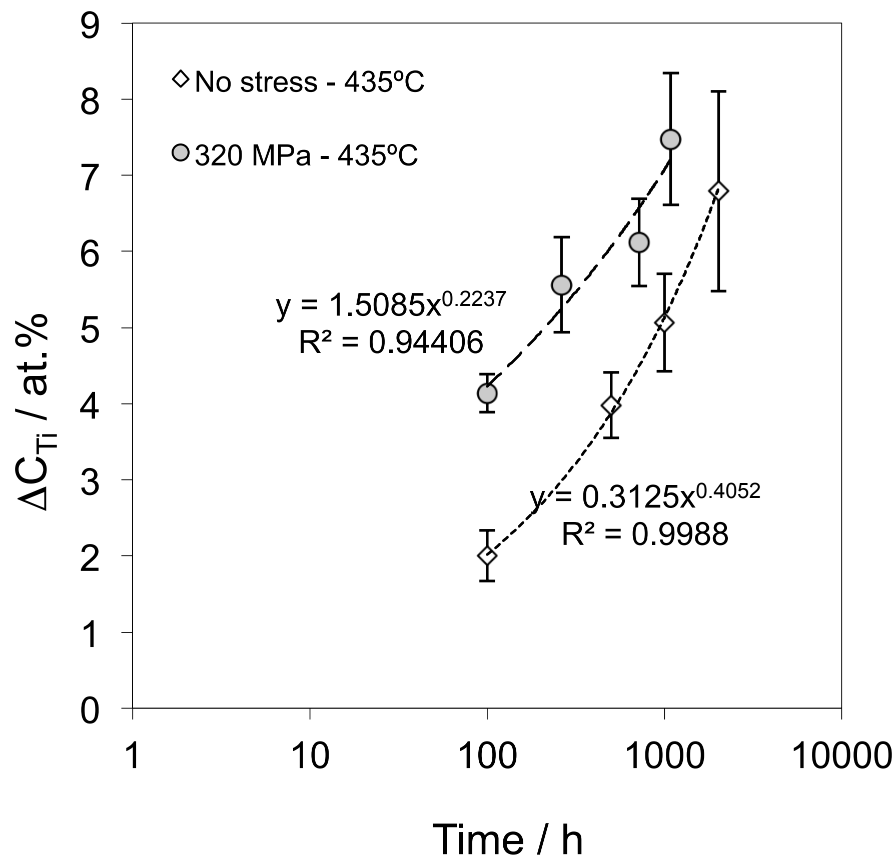
<u>Coherency with matrix</u>: (011) of Fe (0.203 nm) coincide with the planes (022) of the particle (0.208 nm)

Effect of elastic stress

α-α' phase separation



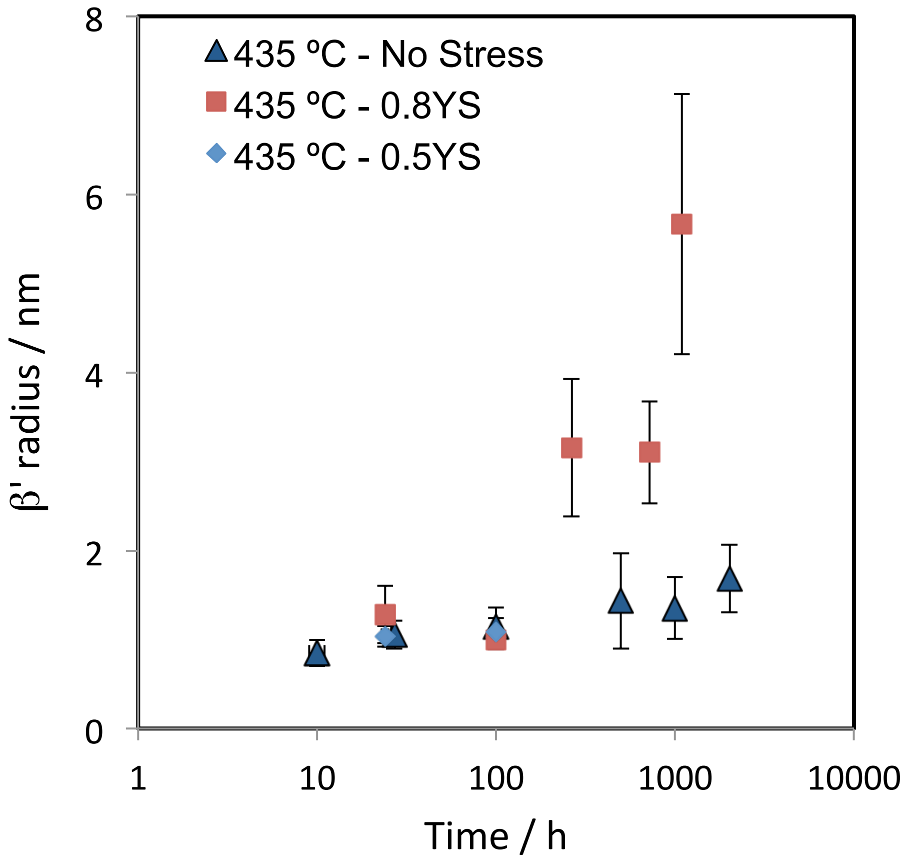
β' precipitation



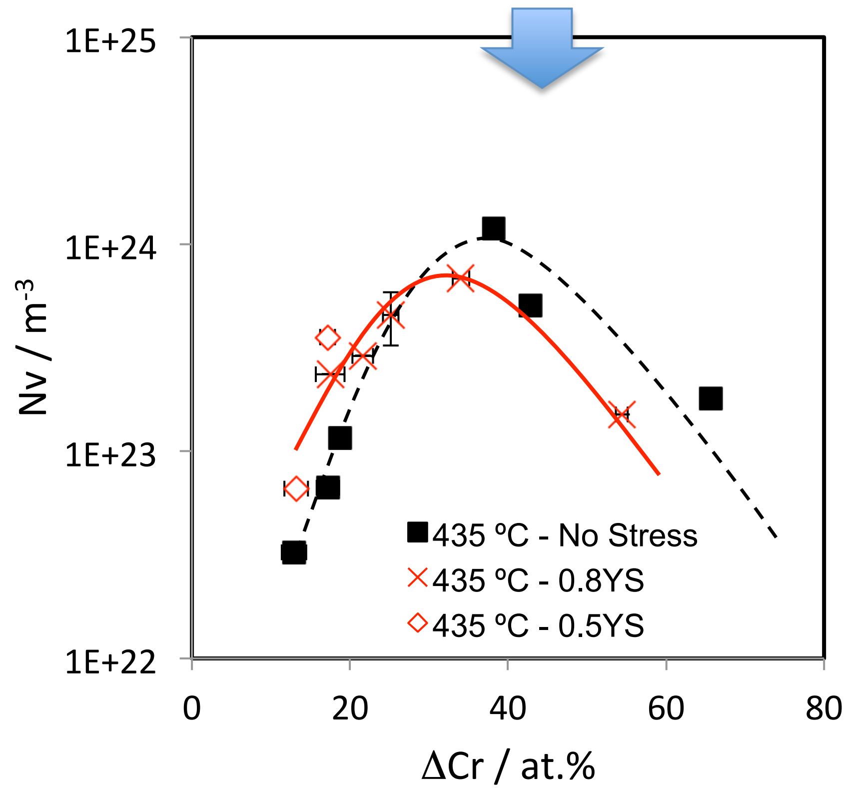
α'-phase precipitation is insensitive and β'-phase is very sensitive to elastic stresses

Effect of elastic stress

β′ precipitation



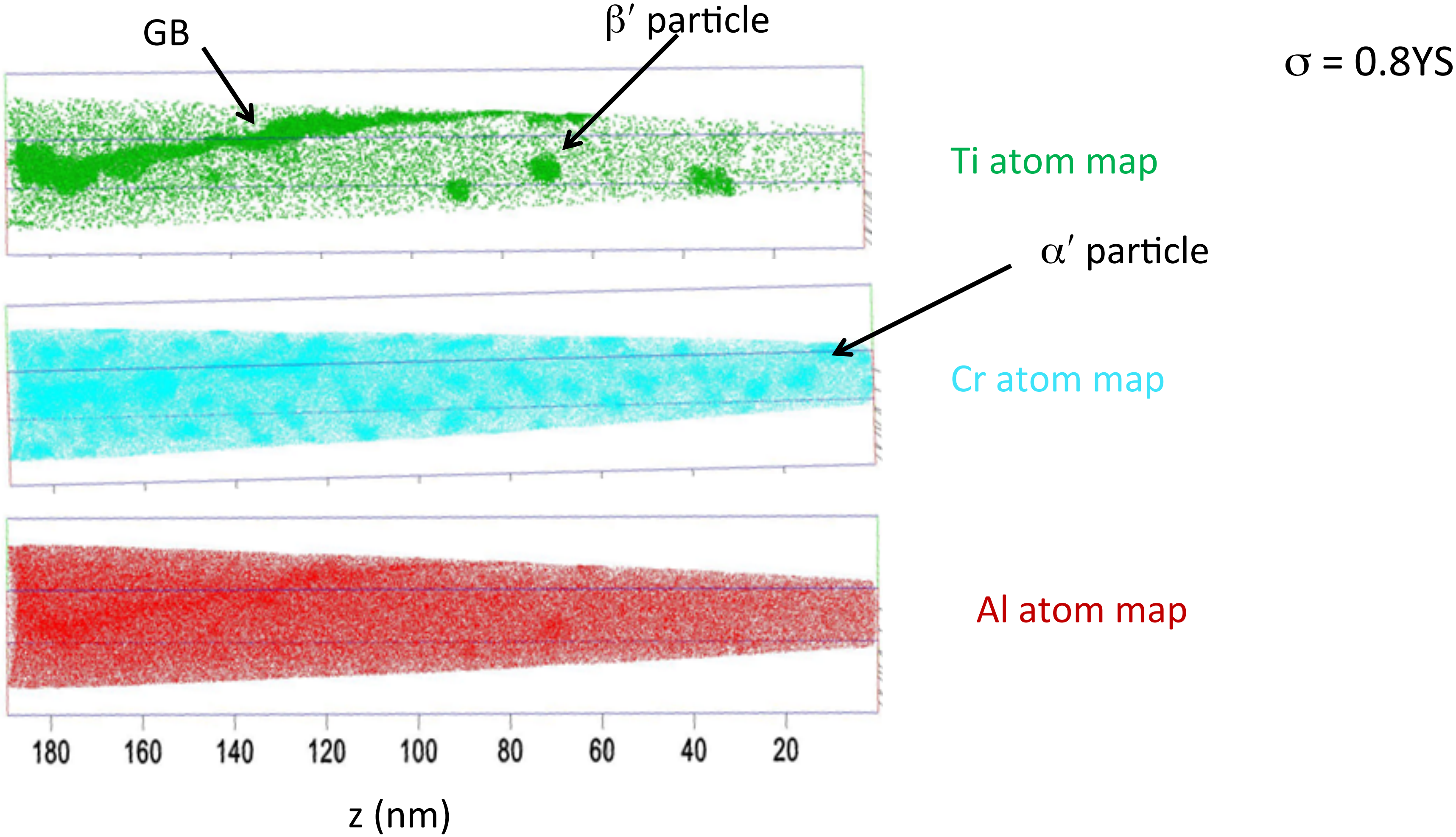
Maximum Separation Method (MSM) to APT data



Elastic stresses induce coarsen β' particles but no significant change in number density

Effect of elastic stress

Grain boundary segregation

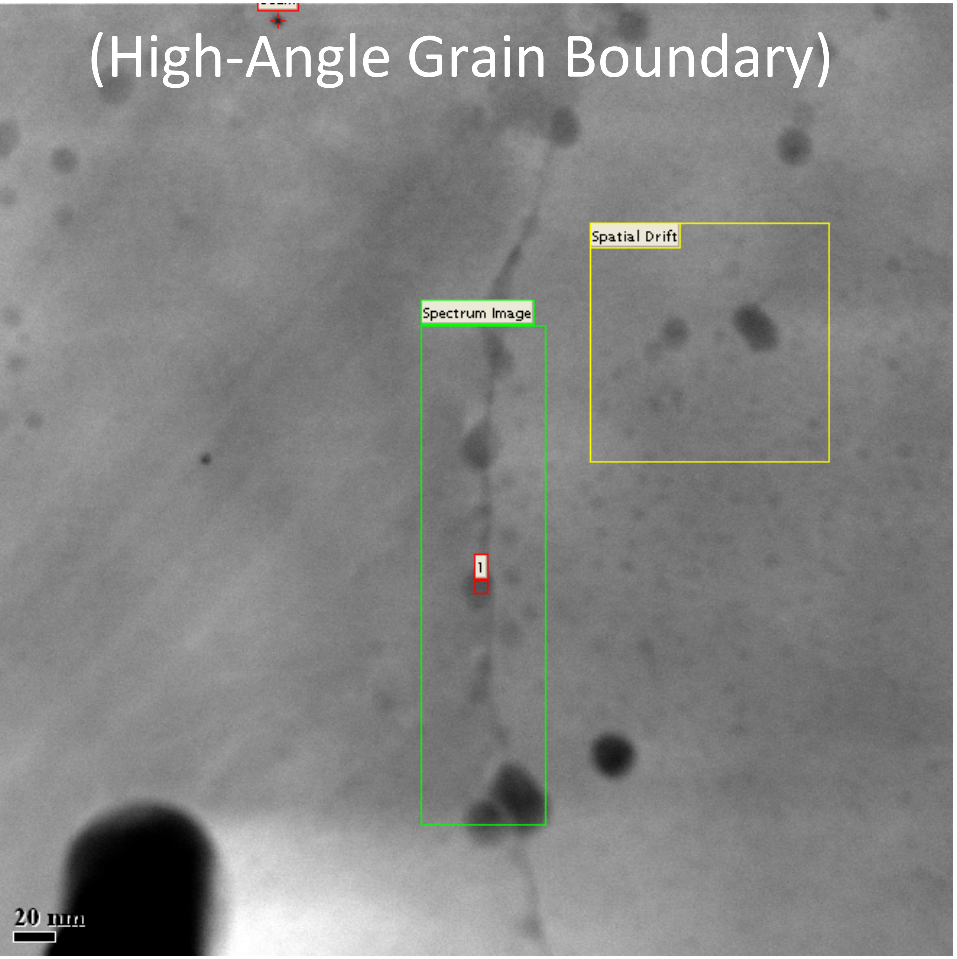


Elastic stresses induce GB segregation of Cr, Al and mainly Ti

Effect of elastic stress

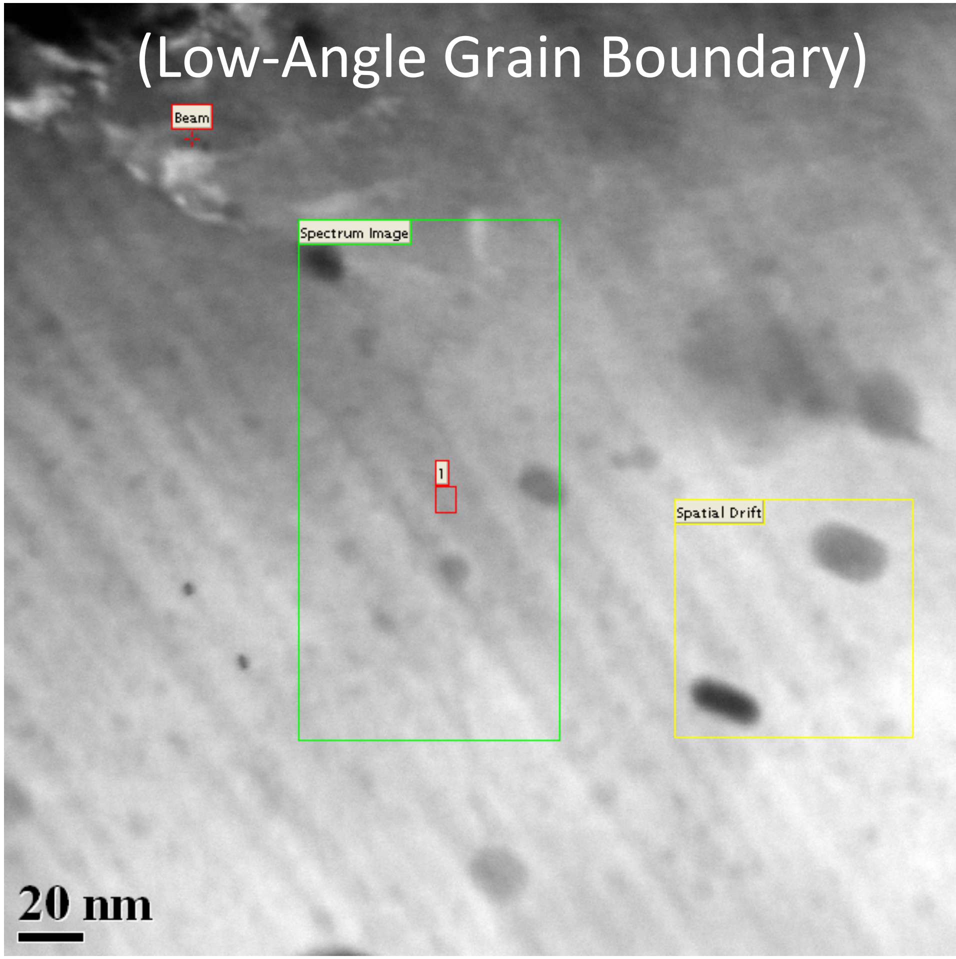
HAGB

(High-Angle Grain Boundary)



LAGB

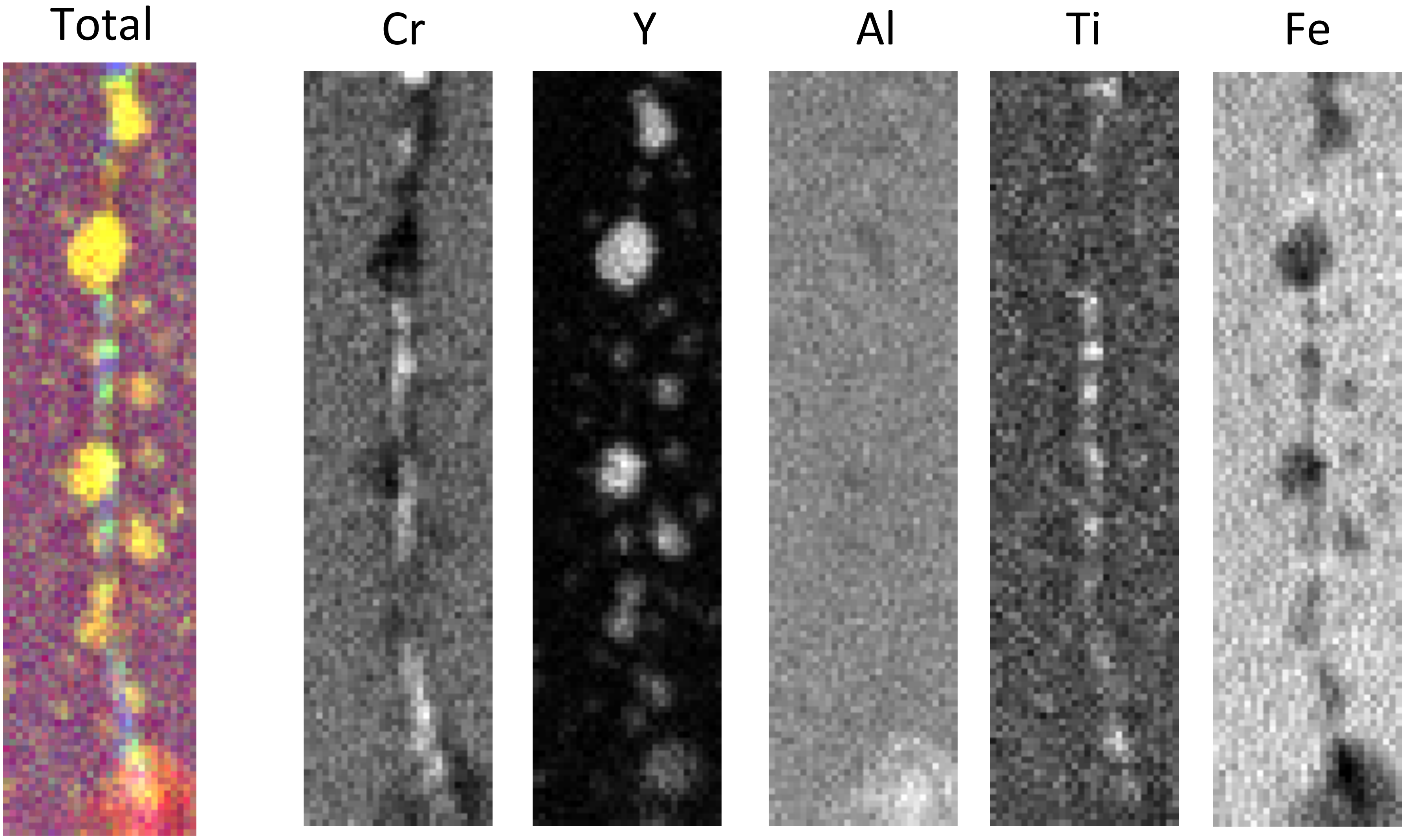
(Low-Angle Grain Boundary)



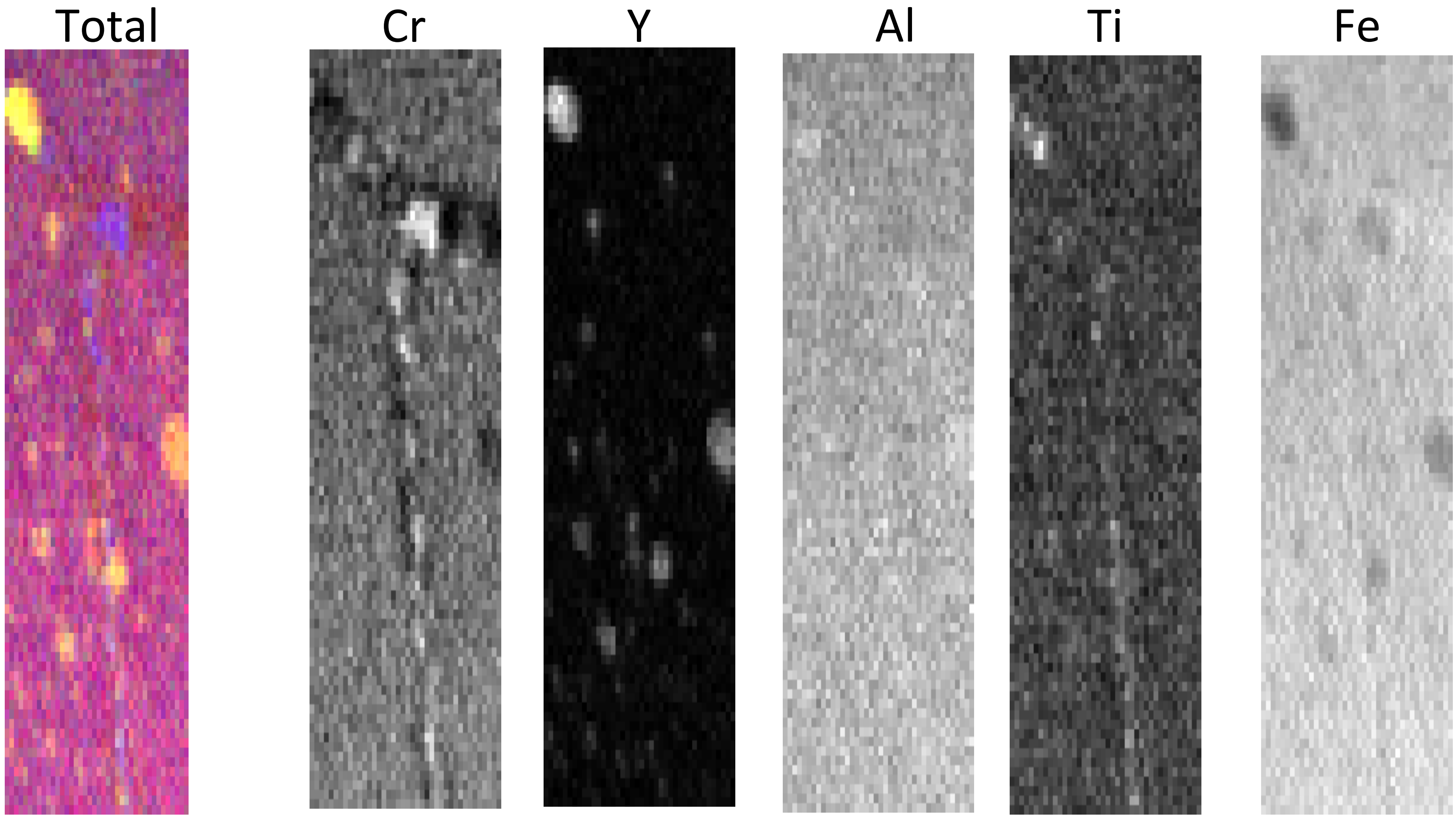
STEM energy-dispersive X-ray spectroscopy (EDS) mapping

435 ºC / 2000 h / 320 MPa

Effect of elastic stress



Effect of elastic stress



Conclusions

Hardening of FeCrAlTi alloy by both dispersion of nanometer in size oxides and precipitation of nanophases is studied. The following conclusions arise:

1. Proximity histograms analysis revealed that the faster phase separation kinetics without stress applied is 475 ^{o}C. The activation energy for α - α' phase separation is 242 kJ mol^{-1}, which is similar than Cr-self diffusion in Fe.
2. Al is rejected from the α' to the matrix during the phase separation. A simultaneous precipitation of Fe-Ti-Al intermetallics: β′ phase $Fe_2(AlTi_{0.6}Cr_{0.4})$ is found.
3. The maximum separation method estimated the size and number density of β′ particles. The β′ particles are more abundant and finer at 435 ^{o}C than that at 475 ^{o}C, which lead to an extra hardening.
4. Elastic stress does not affect α - α' phase separation kinetics, but significant coarsening in β′ phase is observed.
5. It was observed Ti and Al segregation at the grain boundary (HAGB and LAGB) during elastic stressed ageing treatments.