EXPERIMENTAL REPORT
RAPPORT D'EXPERIENCE

Programme Committee Proposal Number
N° Projet Comité de Programme

02-01-111

PROJECT TITLE : *TITRE DU PROJET :*

Compétition entre démixtion /mise en ordre dans un superalliage modèle Ni Cr Al
Competition between ordering and phase separation in a model superalloy Ni Cr Al

LIGNE : **D2AM**

INSTRUMENT : 7 circle diffractometer (in-house) et SAXS

NUMBER OF RUNS USED
NOMBRE DE SESSIONS EFFECTUEES : 11

STARTING DATE
DATE DE DEMARRAGE : 21 February 2002

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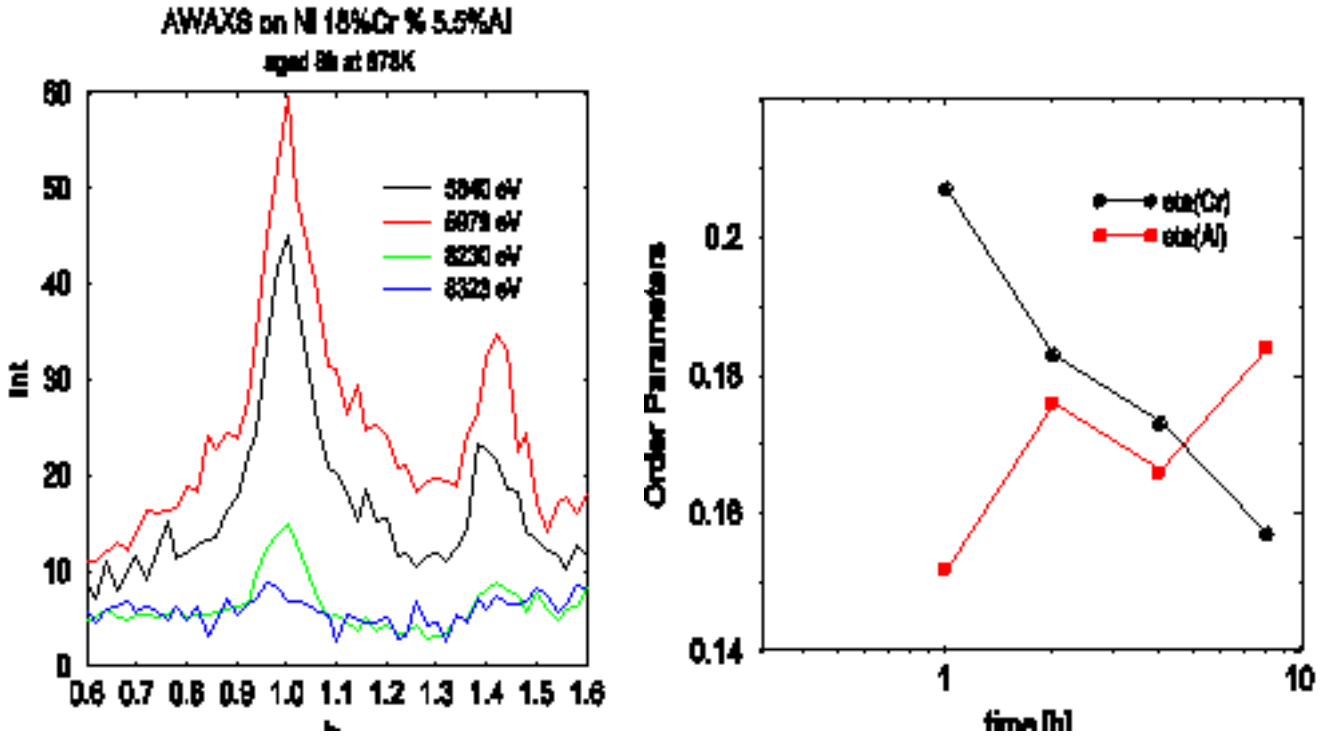
The mechanical properties of superalloys are related to a large volume fraction of an ordered phase ($L1_2$ structure as Ni_3Al, simple cubic), coherent with the disordered fcc matrix. Intermetallic ordered phase strengthens the alloys while matrix limits brittleness. The fairly regular pattern of ordered precipitates is obtained after a quench from the homogeneous state down to room temperature, followed by an ageing in the two phases domain of the phase diagram.
Kinetics is controlled by two processes, well known when they separately occur:
- *Ordering*, in the family of non-conservative transitions
- *Phase separation*, a conservative transition
Both of them can be continuous like spinodal decomposition or first order with nucleation-growth. The final stage is coarsening, where the driving force is the reduction of the antiphase/interphase area.

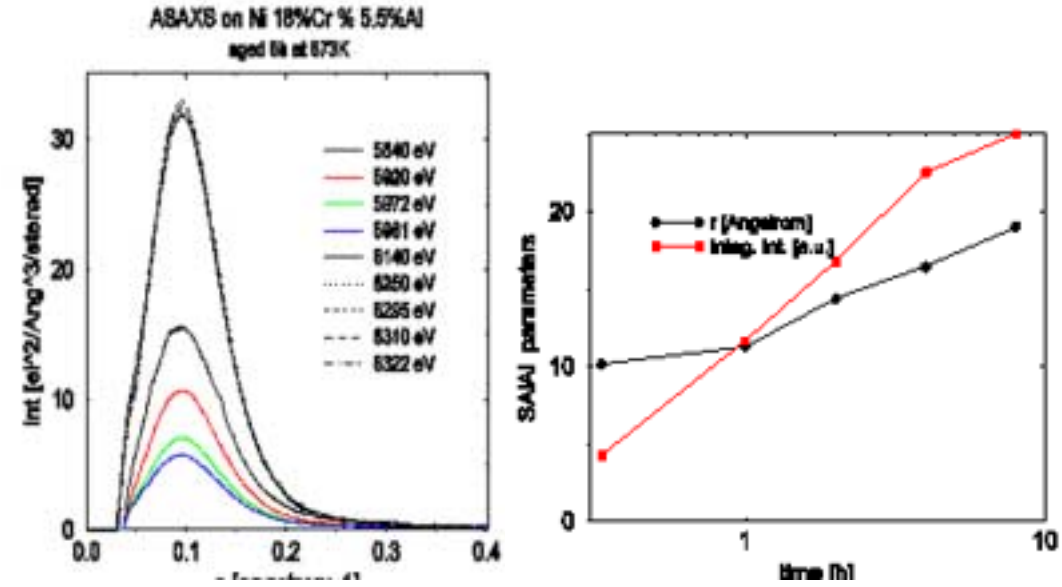
For the first stages of the phase separation in a system pronated to ordering, *the question is which fluctuations first occur, ordering fluctuations or composition fluctuations?*

The $Ni_{0.77}Cr_{0.18}Al_{0.05}$ alloy has been chosen because, at reasonable temperatures, its composition is close to the miscibility gap; its kinetics is not too fast so that very early stages can be observed, not too slow in order to reach coarsening.

- For AWAXS, the Roentec detector (borrowed to the detector pool, having an energy selection ~250eV, a high counting rate, >100000cts and no noise) allows to define several energy windows, such as $K_{\alpha\text{-}Cr}$, $K_{\beta\text{-}Cr}$, $K_{\alpha\text{-}Ni}$, $K_{\beta\text{-}Ni}$ + elastic. Moreover all other parameters have been kept fixed: beam-size on the optics and on the sample, needing only to tune the monochromator at the four energies 5.84, 5.979, 8.230 and 8.323keV (in-house time, 11-16th July 2001). Moreover, data were recorded so that three ways of calibration will be crosschecked: Al powder intensities, fundamentals and Cr fluorescence yield. Quantitative analysis (fig.1) gives a consistent result: A fairly constant Al long range order parameter , η_{Al}, and a decrease of the Cr order parameter.(fig2)



- For ASAXS [1], the experiments have been performed with 4 energies near the Cr edge and 5 near the Ni edge, with absolute calibration (fig3): there is a strong intensity decrease when approaching the Cr edge while there is no variation near the Ni edge. The partial structure factors [2] are consistent with a bi-phased system (the disordered γ matrix and ordered γ' precipitates. The inter diffusion occurs between Al and Cr only, the Ni concentration remaining constant on the whole lattice. There is an increase of the integrated intensity in a factor of two, which indicates that the observed stages are nucleation and growth (and probably simultaneous coarsening); but the volume fraction increases much less rapidly and the interpretation is a Al enrichment which takes place of the Cr atoms.



As a first tentative conclusion, it seems that ordering between Al, Cr and Ni takes place on the whole lattice and that the formation of the L_{12} precipitates tends towards $AlNi_3$ which ,and through out the Cr atoms with a decrease of their order parameter.

[1] J. P. Simon, S. Arnaud, F. Bley, J. F. Berar, B. Caillot, V. Comparat, E. Geissler, A. de Geyer, P. Jeantey, F. Livet and H. Okuda, J. Appl. Cryst., **30**, 1997, 900-904
[2] O. Lyon in «Methods in the Determination of Partial Structure Factors» edited by JB Suck, D Raoux, P Chieux, C Riekel, World Scientific (1993) p142-150.