ESRF	**Experiment title:** Determination of the internal strain of Ni based single crystal superalloy as a function of the thermo-mechanical history of the sample : relation between lattice parameters, precipitates morphology and internal stress state.	**Experiment number:** HS242
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Report :

Due to their mechanical properties Nickel based superalloys are important industrial materials. In particular in the field of gas turbine engines, they play an important role in the manufacture of turbine blades. Their high mechanical performances are due to a precipitation hardening : an ordered γ ' phase close to Ni3Al precipitates inside a matrix of disordered FCC γ phase and prevents the dislocation motions. Among the factors which influence the strength and the creep resistance of these alloys, the lattice parameter mismatch ($\delta = (a_{\gamma'} - a_{\gamma})/<a>$) is considered as very important. Its value evolves with temperature and during heat treatment. Due to its small value and to the intrinsic mosaicity of the material, measurments are delicate and tricky to performe. Our experiments have shown that the Triple Crystal Diffractometer installed on high energy beam line ID15A is well suited to perform this type of mismatch measurements on bulk sample, even when using a furnace for “in-situ” characterization (1-3). Measurements described here were performed at 150 keV.

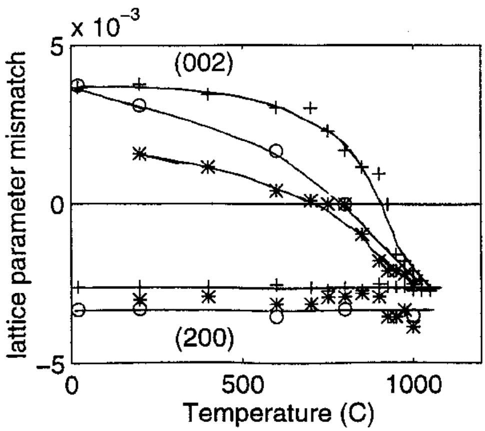

Figure 1: temperature dependence of the lattice parameter mismatch δ measured in the [002] and [200] directions for three AM1 crept samples
+ sample 1: T=1050°C σ=150MPa ε=1.033%
* sample 2: T=1000°C σ=160MPa ε=28.2%
o sample 3: T=1000°C σ=250MPa ε=35.4%

The fundamental reflections (with same indices of parity) are related to the diffraction of both phases while superstructure reflections (with different indices of parity) are related to the diffraction of the γ ' phase only. The analysis of both types of reflection allows to determine the lattice parameter distribution for each phase and then provides information about the strain distribution inside the material.

In a first step, we tried to measure the superstructure reflection (100). In accordance with the calculated structure factor values this reflection is very weak and then difficult to measure. However if the mosaicity of the sample is not too high, we have shown that this reflection can be measured with reasonable statistics but it seems difficult to measure superstructure reflection of higher indices with a good accuracy for crept deformed sample. This measurement becomes more difficult when the crystalline quality is damaged. Such a degradation occurs during creep deformation. Indeed, if the typical mosaicity of sample after standard heat treatment is of about 30' to I degree, it becomes higher, around 2 degrees and even more, for sample creep deformed up to rupture. However in most of the cases in this study, measurements remained possible even at high temperature.

In a next step, we studied three different AM1 superalloy specimens submitted to a tensile deformation applied along the <001> direction at high temperature. The γ ' precipitates have coarsened and present a raft shape perpendicular to the axis of deformation. The first specimen, referenced as sample 1, was creep deformed at 1050°C under 150 MPa during 118hours, the resultant deformation is ε=1.033%. The others were both deformed at 1000°C up to rupture. For the sample referenced as sample 2, the applied stress was 160 MPa and the resultant deformation after 708.5 hours of deformation was ε=28.2%. For the last sample, sample 3, the applied stress was 250 MPa and the resultant deformation after 46.3 hours of deformation was ε=35.4%.

We measured from room temperature up to the temperature of deformation the reflections (100) and (200) and also the reflections (001) and (002) providing information related to directions respectively parallel and perpendicular to the precipitate rafts. In order to obtain exact lattice parameter distribution for both phases, fundamental reflections have to be deconvoluted using superstructure ones. The lattice parameter mismatch behaviours were determined using such a deconvolution and are shown figure 1. This is only a preliminary estimation which already showed a large variation of the lattice mismatch along the <00h> direction and a dependence on the thermo-mechanical history of the material. A more accurate lattice parameter determination is in progress taking into account the evolution of the volume fraction of each phase as a function of temperature and the occurrence of a probable broadening of the superstructure reflections related to particle size. Nevertheless, these results show already the effect on the lattice parameter mismatch of the applied stress but also of the temperature at which the deformation is performed.

References :

(1) ESRF Highlights 1995/1996 pp.48-49

(2) A. Royer and P. Bastie, Scripta Mater., 1997, “in press”

(3) A. Royer, P. Bastie and M. Véron, Scripta Mater, “submitted’