ESRF	**Experiment title:** **Very high temperature mechanical behaviour of a single crystal superalloy: *in situ* experiments by high energy Three Crystal Diffractometry**	**Experiment number**: MA 978
Beamline: ID 15a	**Date of experiment**: from: 07/07/2010 to: 12/07/2010	**Date of report**: 08/2012
Shifts: 15	**Local contact(s)**: T. Buslaps	*Received at ESRF:*

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Aims of the experiment:

The aim of the experiments was to determine the mechanical behaviour of rafted single crystals superalloys at very high temperatures (>1060°C). Additionnal slower experiments at temperatures between 950°C and 1050°C were carried out at the BW5 beamline of Hasylab.
The rafted microstructure of single crystals superalloys is observed during high temperature tensile creep tests of alloys having a negative lattice mismatch δ. Besides its industrial importance, it is among the most simple two-phased microstructure, and is a very good model material to investigate the high temperature behaviour of polyphased materials.

Experiments

Four specimens of single crystals of the AM1 superalloy were initially pre-strained at IJL by high temperature tensile creep in order to reach the second stage of their creep curve and prepare well formed raft microstructures. They were then tested at high temperature at the ID15 High energy beamline. We performed mechanical tests under constant temperature and variable load while recording continuously Three Crystal Diffractometry scans of the (200) double diffraction peaks perpendicular to the tensile axis. The ID15 TCD, with a +-+ setup and operating with 150 keV radiation allows transmission experiments through 3 mm thick Ni3Al specimens, and makes possible measurement on bulk materials. The high beam intensity provided at ID15 allows recording full scans with a high precision in less than three minutes. This makes possible the investigation of fast transient phenomena. Knowing the variations of the lattice parameter of each phase in the [001] direction, it was possible to determine simultaneously the full stress tensor within each phase as well as the plastic strain of each phase. After the tests, the specimens microstructure was carefully investigated by SEM and TEM to determine their microstructure (rafts lengths, corridor thicknesses, dislocation densities, Burgers vectors…)

The length of data processing and specimen analysis explain the present late report.

Results

From the analysis of our data, we could measure:

- The temperature dependence of the Young modulus of both phases.
- The plastic strain rate of the γ corridors as a function of the local Von Mises stress (Figure a). We showed that plastic strain begins once the Von Mises stress is larger than a threshold stress. The temperature and microstructure dependence of this threshold was measured, and it was checked that it is equal to the Orowan stress within the rafts at temperatures higher than 1000°C, and about 20 MPa higher for lower temperatures. The necessary parameters, such as the Young modulus and corridor thickness were obtained both from in situ experiments and SEM investigation.
- The strain rate of the the γ' rafts as a function of the applied load and of the internal stress σ'_{xx} (Figure b). This latter parameter is apparently the most important one. It exhibits a threshold (about 80 MPa) which depends weakly on the temperature.
- The strain rate measured during unloading is lower than during loading at equivalent σ'_{xx} stress. This might be due to exhaustion of mobile dislocations.
- The shape of the TCD peaks was analyzed, as well as its dependence on experimental conditions. It is currently being compared to 3D simulations of the dislocation distribution within the rafted alloy, in order to separate the contribution of the microstructure and dislocation densities to the peaks profiles.

We would like to stress that such results are a world firsts, and that they could not have been obtained without the combination of *in situ* high temperature tests and TCD experiments at the ESRF.

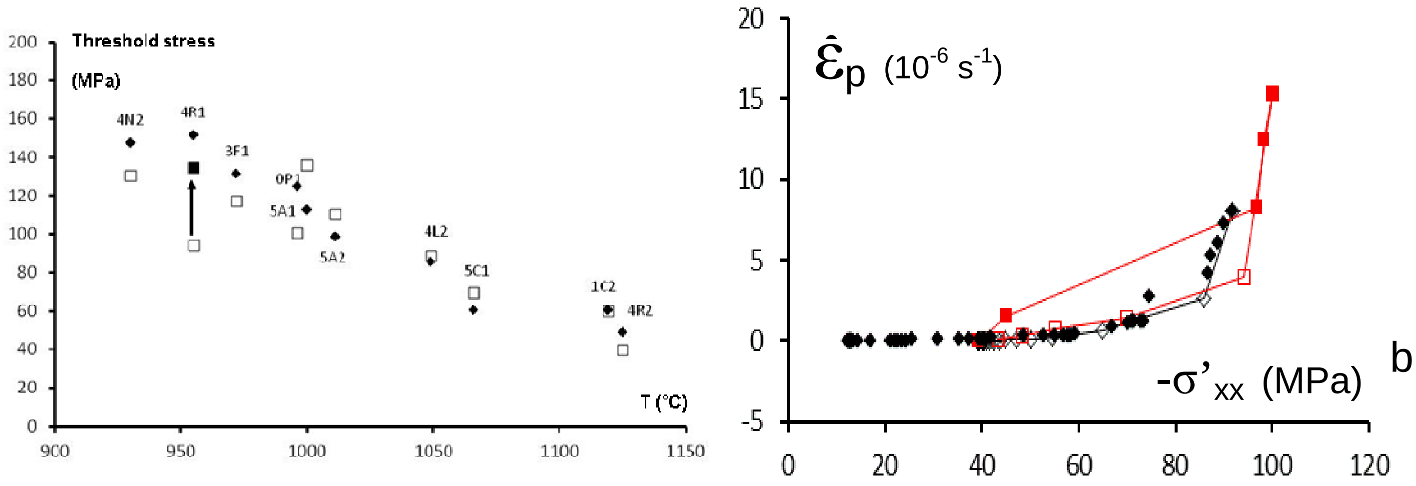

Figure a: threshold Von Mises stress for plastic strain of the γ corridors (full diamonds) and calculated Orowan stress using our data for Young Modulus and microstructural parameters (squares). b: plot of the strain rate of the γ' rafts during loading vs the σ_{xx} component of the strain tensor.

A first report of the results is given in:
Phase specific high temperature creep behaviour of a pre-rafted Ni-based superalloy studied by X-ray synchrotron diffraction L. Dirand et al. submitted to Phil. Mag.

Future work

This work on single crystal superalloys will be carried on in two directions:

- Determination of the physical mechanisms controlling the plastic strain of the the γ' rafts: experiments under the same conditions (temperature and stress tensor components) but different microstructural parameters (raft length and thickness, dislocation densities….)
- Measurement of the mechanical behaviour under transient temperature excursions.