ESRF	**Experiment title:** Transient variations of the dislocation densities within the Gamma Prime rafts of a Single Crystal superalloy during in situ HT mechanical tests with stress increments	**Experiment number**: MA 2891
Beamline: ID 11	**Date of experiment**: from: 25th April 2016 to: May 1st 2016	**Date of report**: Feb 24th, 2017
Shifts: 15	**Local contact(s)**: Vadim DIADKIN	*Received at ESRF:*

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Report:

Cylindrical specimens of the AM1 single crystal superalloy 2 mm in diameter (vertical tensile axis a few degrees from the nominal [001] direction) were oriented for (200) Bragg reflection with a 67.4 KeV (below the Ta K absorption edge) monochromatic beam (50 µm wide and 100 µm high, Si (111) monochromator, $2\theta B \approx 5.81°$). Far field images of the diffracted beam were recorded with a 4 Megapixels FReLoN camera (50 µm pixel size) at 8.4 m from the specimen.

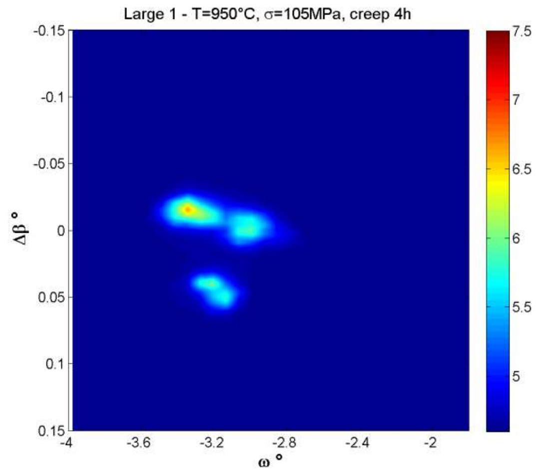
Two procedures were then followed:

- 3 D images of the (200) peak were obtained by scanning the ω angle (typical twenty 0.1°, one second steps for a total 2° width.
- ω integrated images were obtained by continuous ω rotation of the specimen during a 8 seconds camera recording.

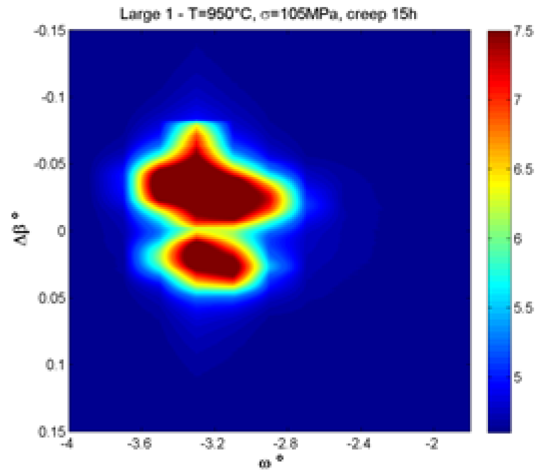
As can be seen in Figure 1 (top), integration of the 3D images on the $2\theta_B$ angle gives the distribution of orientations within the specimen, which contains subgrains (ω misorientation in the 0.1° range) which have slightly different average lattice parameters related to chemical segregation during solidification. The ω integrated images (bottom) show a main peak (γ' precipitates) and a tail on the large lattice parameters side (γ channels) for the initial cuboids microstructure (left) and a pair of peaks after microstructural change (rafting, right). Streaks parallel to the vertical [001] direction are visible for rafted specimens, and are due to dislocations at the γ/ γ' interfaces, which are perpendicular to the tensile axis after rafting.

Compared with the 1D diffraction profiles (integrated along ω and β) obtained by Three Crystals Diffractometry (TCD) during former experiments, 2D and 3D images bring the possibility to follow the behaviour of individual subgrains with slightly different chemical compositions and precipitate fraction, for which the kinetics of rafting and the mechanical behaviour are different.

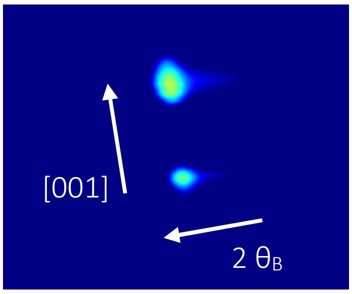
Three specimens prepared with different initial microstructures were tested under a variable tensile load at temperatures between 950°C and 1050°C in a home made mechanical testing device, while diffraction peaks were continuously recorded (more than 40000 images in five days !)



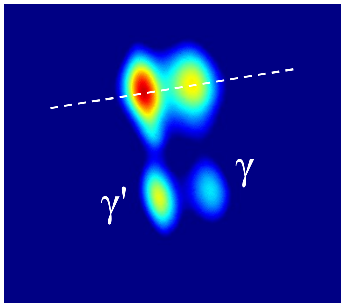
Peak intensity: (ω,φ) (2θ integration), Log scale
Before rafting



Peak intensity: (ω,φ) (ω integration) Log scale
After rafting



Peak intensity: (2θ ω,φ) (ω integration) Log scale
Before rafting



Peak intensity: (2θ ω,φ) (ω integration) Log scale
After rafting

Figure 1

The main result is the $\delta_{100} = 2*(a'_{100}-a_{100})/(a'_{100}+a_{100})$ mismatch in the rafts' plane, i.e. the distance between the γ and γ' peaks along the (200) direction. Because of the symmetry of the microstructure, once the applied load, the total strain of the material and the temperature dependent "natural" mismatch δ(T) are known, δ_{100} is the only parameter needed to calulate the strains and stresses in both phases.

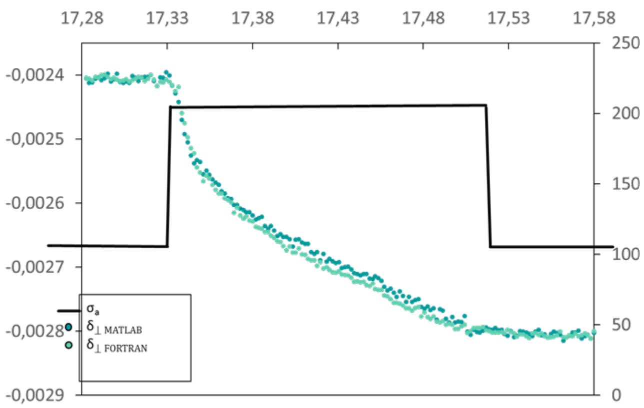


Figure 2

As seen in Figure 2, the precision on the value of δ_{200} (green dots, left scale, two independent analysis in light and darker greens), recorded during a 18 mn load step (right scale), is a few 10^{-5}, i.e. as good as obtained by Three Crystals Diffractometry at ID15 under stable conditions during former experiments. The possibility to record an image every 8 seconds (instead of every 300 seconds) also allows measurements during load changes without blurring: it is now possible to follow very short transients in the behaviour of the material.

Thus 3D far field images give much more details on the evolution of the diffraction peaks (and the microstructure evolution) during mechanical testing than one dimensional TCD peaks, and at a much higher rate. The experiment was thus quite successful, and we hope to use the same method to investigate superalloys under different sollicitations (temperature jumps) in a near future. The analysis of the results of this experiment (together with post mortem investigation of the specimens' microstructure) forms the bulk of PhD student R. Trehorel's Thesis, and peaks simulation will be part of the Thesis of PhD student K. Eloh.