ESRF	Experiment title:  Non equilibrium microstructural evolutiion and plastic behaviour of a Superalloy during a creep test with very fast high temperature excursions.	Experiment number: HC 3231
Beamline: ID11	<b>Date of experiment</b> : from: 24/01/2018 to: 29/01/2018	<b>Date of report</b> : 01/03/2018
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## **Preliminary report:**

Ni base single crystal superalloys are used to manufacture aircraft engine turbine blades since they exhibit very good mechanical properties at high temperature. During the major part of their high temperature creep life, the microstructure of such single crystal superalloys is lamellar, with semi coherent alternating layers, perpendicular to the [001] tensile axis, of a disordered fcc  $\gamma$  matrix ( $\gamma$  channels) and of a hard ordered L12  $\gamma$ ' phase ( $\gamma$ ' rafts).

The aim of the proposed experiment was to measure the microstructure evolution and the transient mechanical behaviour of a single crystal superalloy during high temperature tensile creep tests (950°C-1200°C) with very short ( $\sim$  40 seconds) temperature excursions. During such excursions, the hard  $\gamma'$  phase partly dissolves, allowing a burst of plastic strain within the  $\gamma$  channels first, then within the  $\gamma'$  rafts.

However, as the only available alloy was a new one (CMS-X4+), we first needed to measure the volume fraction of the  $\gamma$ ' phase and the lattice mismatch between both phases as a function of temperature. This was done by modifying the projected setup, in order to measure the (002) (parallel to the tensile axis and (200) diffraction peaks, by use of two far field FReLoN cameras in the vertical (002) and horizontal (200) diffraction planes and an ILL furnace with a better thermal stability than our own device. The (200) (resp. (002)) double peaks (one for each phase) were recorded during omega (resp. rot-y) scans in order to measure the slight misorientations between different parts of the specimen within the beam's trajectory. (mainly areas with a different chemical composition resulting from the dendritic solidification of the alloy.) (See figure below.)

Because this more complex configuration and difficulties in the simultaneous computer control of both cameras and rotations, the time necessary for the setup was much longer than expected (seven shifts instead of three), and the first part of the experiment took ten shifts.

The remaining five shifts were too short to fulfil the expected program, and we decided to use these to follow the microstructural and mechanical evolution of the alloy during a full creep test using the (200) diffraction peak. Using omega scans, we plan to use the misorientations between subgrains to distinguish the dendritic and interdendritic areas of the specimens, which have a different behaviour in this alloy.

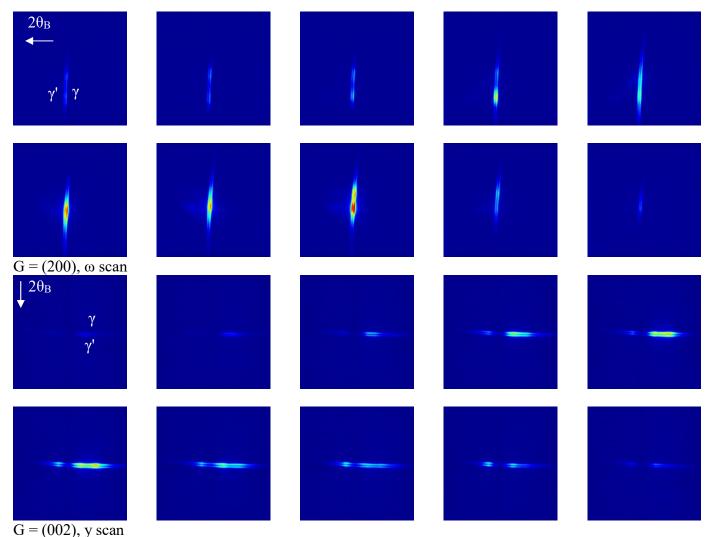


Figure: successive images (2000\*2000 pixels) of the diffraction (002) and (200) double peaks recorded during omega and rot-y scans of a specimen with a rafted microstructure at 1250°C. Different spots appear during the 3 degrees rotations. Quasi single crystalline areas (dendrites) give a sharp spot, while the more disordered interdendritic areas give a more diffuse one. The streaks perpendicular to the G vector appear during plastic strain and are related to the distorted areas near dislocations.

To conclude, we could not carry out the planned experiments, but we used the beamtime to obtain interesting and useful data on a new Superalloy for which chemical segregation between dendritic and interdendritic zones plays an important role in the high temperature mechanical behaviour.

A preliminary analysis of the results (40000 images) will take about six months, and a full analysis between one and two years. We thus plan to apply for additional beamtime for this project after the ESRF shutdown.