Influence of plastic deformation on the rafting of the precipitates in single crystal nickel-based superalloys

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Rafting in single crystal (SX) Ni-based superalloys describes the directional coarsening of the gamma prime precipitates when the alloy is subjected to high temperature creep deformation. Different studies showed that rafting takes place during the annealing at high temperature of samples that have been previously plastically pre-deformed\cite{1}. This phenomenon is thus called plastic strain-induced rafting. It was also shown that the lattice parameter misfit between the matrix and the precipitates present in SX Ni-based superalloys plays a major role in the rafting of the precipitates and acts as a driving force for rafting to occur.

Rafting was observed at very localised areas in a fully heat-treated turbine blade. Neutron diffraction performed on a similar blade but cooled from the solution heat treatment showed the presence of residual strains in the areas where rafting was observed in the fully heat-treated blade. One explanation for the microstructural change observed in the fully heat-treated blade is the relaxation of the residual strains measured in the solution heat-treated blade during subsequent heat treatments.

The two objectives of this experiment were:

\begin{itemize}
  \item Characterise the influence of plastic strain on the lattice parameter misfit evolution during high temperature annealing. Samples (2x2mm\textsuperscript{2} cross section and 7mm length) from the two different alloys (Re-free and Re-containing superalloys) with different levels of plastic deformation (0.2, 0.4, 0.6, 0.8 and 1\%) have been prepared. The lattice parameter misfit evolution was measured in-situ during annealing at 1050 °C.
  \item Characterise the lattice parameter misfit in thin section (1mm thick) of a turbine blade similar to the one measured with neutron diffraction in order to characterise the influence of the residual strain measured on the lattice parameter misfit. This could then be related to the rafting observed in the fully heat-treated blade by measuring the misfit in a cut of a blade in the fully heat-treated condition.
\end{itemize}

The BW5 beamline is particularly suitable for the characterisation of the lattice parameter misfit in SX Ni-based superalloys since a triple axis set up can be mounted, which gives a d-spacing resolution better than 10\textsuperscript{-4} whereas the misfit between both phases is for the most of the case close to 10\textsuperscript{-3}. Furthermore, the ILL furnace available is used to anneal the samples at 1050 °C for long periods of time.

The first part of the experiment on the solution heat-treated cut of a turbine blade revealed a significant mosaicity of the single crystal. As it is illustrated in figure 1,
several high intensity peaks can be observed at different rotation angles, indicating a large degree of mosaicity in the sample. Hence calculation of misfit becomes complicated. Nevertheless, the mosaicity seems to be larger in the areas where residual strains were characterised using neutron diffraction and where rafting was observed in the fully heat-treated blade. Residual strain seems thus to increase the mosaicity of the sample in the solution heat-treated condition. The same areas were then measured on the cut in the fully heat-treated condition and they showed a lower mosaicity than in the solution heat-treated blade, which could be explained by a relaxation of the residual strains during subsequent heat treatments. However, the mosaicity remains significant and did not allow us to calculate the lattice parameter misfit from the collected data.

During the second part of the experiment and due to time restriction, only the 0.2% pre-deformed samples were annealed for approximately 10 hours. The beam energy was set to 120 keV to insure X-ray beam penetration through the thickness of the samples. During the annealing period, the (200) fundamental reflection was recorded continuously (see figure 2), with an acquisition time of 2 minutes. During the rafting of the precipitates, the (200) fundamental reflection usually splits. Hence the calculation of the lattice parameter misfit becomes easier since both peaks can be fitted separately. Unfortunately, the (200) reflection did not split during the annealing period, indicating that no rafting occurred. This was confirmed by microscopy analyses performed on the samples measured during the beamtime. One explanation could be that the annealing period of 10 hours was not sufficient to induce the rafting of the precipitates at a such low level of plastic deformation. A new experiment is scheduled this year (16th to 20th May) during which only 0.6 and 1% pre-deformed samples will be annealed in order to induce the rafting of the precipitates and calculate the lattice parameter misfit in both alloys.

![Figure 1. Omega scan of the SHT cut at a position where residual strains have been observed with neutron diffraction. The different peaks of similar intensity within 3 degrees shows the large mosaicity of the γ']

![Figure 2. (200) fundamental reflection of the Re-free material in the undeformed condition in 2θ scale. The main peak corresponds to the precipitates phase γ']