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Diffraction profile, strain distribution and dislocation densities during stage II creep of a superalloy

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Abstract. One of the major ingredients of modelling the mechanical behaviour of superalloys is the knowledge of dislocation densities and strain distribution. Both can be measured using post mortem BF TEM and CBED, but such methods do not allow following their variations during a test. The aim of the present work is to investigate the usefulness of in situ X-Ray Three Crystal Diffractometry (TCD) to measure the density and distribution of dislocations within a rafted superalloy, i.e. during stage II of high temperature creep. As the instrument contribution is very low, the two-peaked experimental profiles are representative of the lattice parameter distribution within the material. The profiles were measured within bulk specimens at the BW5 high energy beamline Hasylab (DESY), during high temperature (1050°C to 1180°C) tests under loads between 0 MPa and 300 MPa. The peak shapes were observed to change with varying experimental conditions. The peak width follows different patterns under low and high stress, i.e. with low and high strain rates.
The distribution of elastic strains was calculated by assuming two main contributions: dislocation segments trapped at the γ/γ’ interfaces in a more or less regular network, and dislocations moving within the γ’ rafts. A comparison between experimental and simulated peaks shows that several features of their behaviour can be explained: the absolute magnitude of the peak width, the observed decrease of the peak width under low loads with increasing interfacial dislocation densities. The larger increase in the width of the γ’ peak under high load (and strain rate) may be attributed to a dislocation density within the 10^{13} m^{-2} range within the rafts. The present results are presently being cross-checked by post mortem TEM observations.

Introduction

Single-crystal nickel-based superalloys are currently widely used in aircraft engines for their creep resistance under tension at high temperature. These alloys are two-phased materials, with a disordered fcc γ matrix containing a high relative concentration of $L1_2$ ordered γ’ precipitates. Under high temperature creep, the microstructure changes to a rafted one [1], ie the microstructure is a semi-coherent multilayer of the two phases γ and γ’ perpendicular to the [001] tensile axis.

It has been shown [2,3,4,5] that *in situ* diffraction methods using neutrons or high energy synchrotron radiation, especially Three Crystal Diffractometry (TCD) could give some insight on stresses and strain rates within the material.

The basic result from TCD (analyzer scans) using a (200) reflection is, indeed, the distribution of the lattice parameters within the material, along the [100] direction, i.e. perpendicular to the [001] tensile axis. In the case of a single crystal superalloy with a raft microstructure, it is a two-peak profile, corresponding to the γ' and γ phases. From the peak positions, the stresses and strains within both phases can be calculated [6]. From the peak shapes, it might also be possible to get some insight on the microstructure (dislocation densities).

Recent papers [7,8] suggested that the high temperature plastic strain of the γ' phase involved correlated climb of dislocations. The aim of the present paper is to investigate the effect of the experimental conditions on the shape of the TCD profiles, to link changes in these profiles and changes in the order of the interfacial dislocation array and in the dislocation densities within that phase, and the mechanical behavior of the rafts.

1. Experimental

1.1. In situ experiment with triple crystal diffractometer:

Experiments were performed on the high-energy BW5 beamline at HASYLAB (DESY). The measurements were carried out using TCD at 120 keV (λ= 0.01nm). The sample is a cylindrical AM1 single crystalline specimen ([001] tensile axis) with a 0.45 μm precipitate size grown by SNECMA, and machined by ONERA. It was pre-strained in tension (1080°C, 120 MPa), in a high temperature tensile device adapted to *in situ* X-Ray tests, in order to obtain a raft microstructure (stage II of creep), and stable lattice parameters. The specimen was loaded by successive steps up to 275 MPa, then unloaded to 120 MPa for 320 min, reloaded to 275, unloaded for 30 min, and reloaded for 15 min before cooling down under stress. The experiment lasted less than 24 hours, in order to limit the variations of raft and corridor thicknesses (Oswald ripening).

The data were analyzed following the procedure described in [6], as the sum of three peaks: γ' peak (rafts), γ peak (corridors), and a low intensity γ peak (distorted zones at raft ends and dislocation cores). The main parameters of the fit are their positions, heights, and shapes. The strain rate of the whole specimen was continuously measured during the test.

Recording a scan of the analyser takes 200-500s, depending on the number of points. It is thus possible to measure changes within the microstructure on a time scale > 5 min and to follow transient phenomena (such as response to loading or unloading steps)

1.2. TEM observation

At the end of the experiments, the specimens were cooled down, in order to freeze their microstructure (allowing for changes in the volume fraction of the γ' phase). Thin foils with a [301] normal were sliced, then polished mechanically down to 100 μm, and thinned by argon ion bombardment under a 3 kV acceleration voltage under a 5° angle. They were observed by conventional bright field methods on a CM 200 Philips microscope.

2. Results

2.1. Strain rate of the γ' phase

The Von Mises stresses and plastic strains in both phases were calculated using the procedure given in [6], taking into account the applied load and the elastic strain in the [001] direction, i.e. shifts of the TCD peaks position. In the present paper, only the behaviour of the γ' phase will be considered.

The data are summarized in Figure 1 as a function of time. Due to the high relative precision of the TCD measurements (a few 10^{-5}), the major sources of error are the specimen temperature and its elongation. While the uncertainty on the absolute values of the Von Mises stress in the γ' phase is ~ 10 MPa, the precision in its variations is within the MPa range. The same is true for the γ' strain rate. The scatter of the values of the strain rate in Fig.1 is a few $10^{-8}s^{-1}$. It should be noted for the high load steps, i.e. when the relative precision is better, that the γ' strain rate increases under constant stress. After unloading, it also remains high before slowly returning to its initial value: γ' strain rate depends on the stress, but also on a parameter which varies on longer timescales.

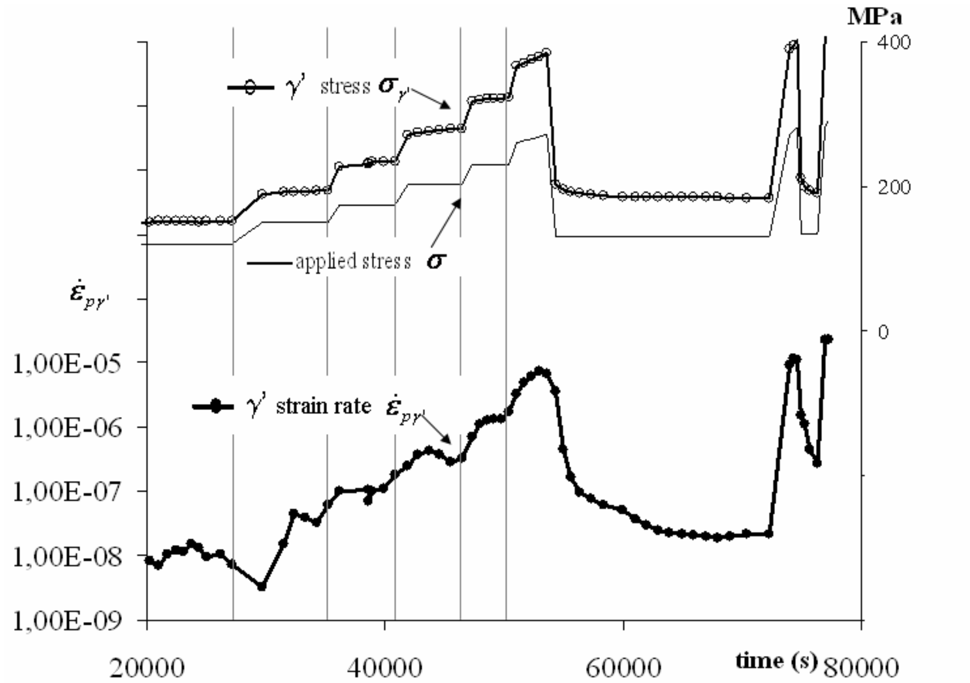

Figure 1. γ ' Von Mises stresses (top) and applied stress (middle) in the whole specimen (lines). Thick line with dot symbols gives the γ ' strain rate during the test.

2.2. Evolution of the TCD peak profiles

Figure 2 shows the definition of the width parameters w_γ and $w_{\gamma'}$ (inverse of the slope of the peaks on a logarithmic scale) of the experimental peaks and their evolution with the stress steps. The peaks become wider and their intensities lower (with a nearly constant area) when the stress increases. The center of both peak is shifted to higher angles as their lattice parameters (perpendicular to the tensile axis) decreases with increasing load. The area of the γ' peak is 57% of the γ + γ' areas.

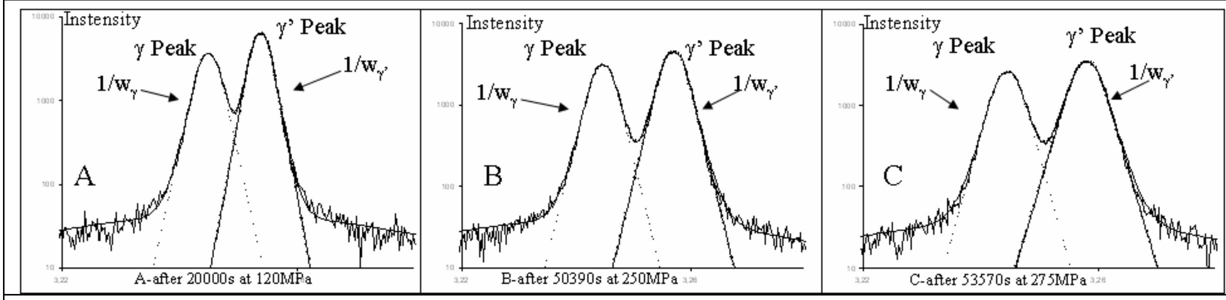

Figure 2. Fit of experimental TCD profiles recorded during the test and at different stress steps. The corresponding time is given in Fig. 3

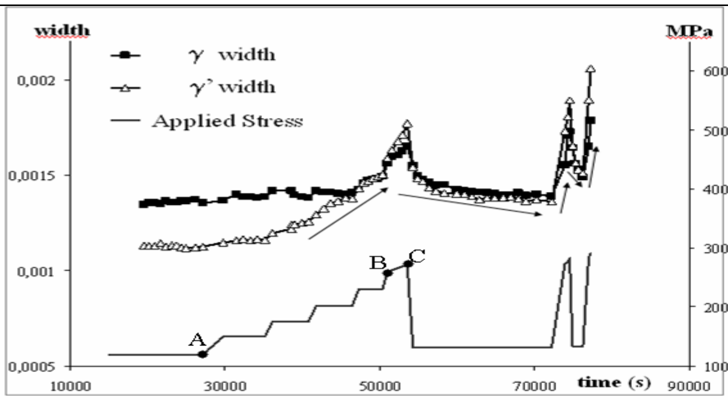

Figure 3. Evolution of w_γ and $w_{\gamma'}$ during the test

Figure 3 highlights the variation of w_γ and $w_{\gamma'}$ with time. Both parameters tend to increase with the applied stress, and decrease after unloading. This increase is stronger for $w_{\gamma'}$ especially during the first series of loading steps.

3.3. Microstructure after testing

The micrograph Fig. 4 shows the microstructure of the rest of a specimen cooled down under a 275 MPa load. The interface are seen end on with a high density of dislocations, and few dislocation are observed inside both γ’ rafts and γ channels. The density of γ‘ dislocations looks higher than γ one. The heterogeneous contrast inside γ suggest the presence of small γ’precipitates formed during cooling. Measurement of the fraction of γ‘ phase over the whole surface gives 58% which is coherent with X-Ray data. A 3.5 10^{12} m^{-2} density of dislocation can be deduced by dividing the numbers of dislocation on the γ‘ surface.

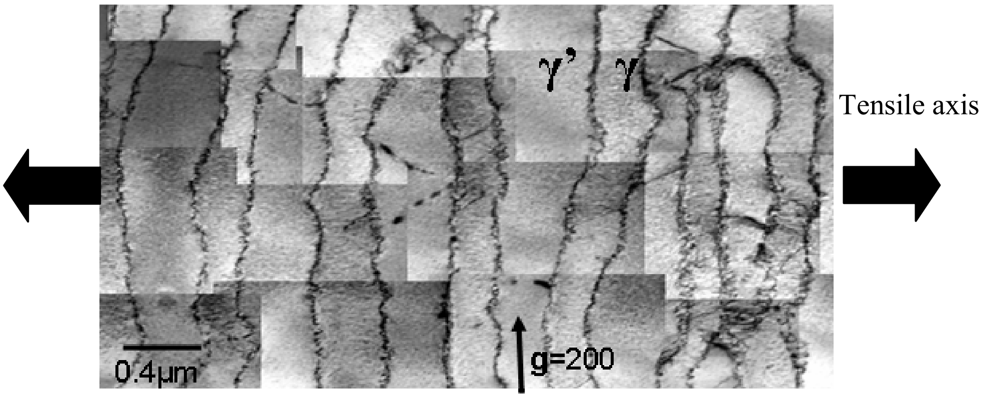

Figure 4. Transmission electron micrograph showing the microstructure after testing of nickel based superalloy AM1

3. Simulation and discussion

The short timescale of the variations of both the strain rate during unloading and the w parameters leaves changes in the dislocation microstructure (i.e. the dislocations trapped at the γ/γ’ interface, and possibly dislocations moving within the rafts), as their only possible explanation.

A periodic square array of edge interface dislocations having all their Burgers vector $b_\perp$ within the interface plane, with a distance: $d = b_\perp / \delta_\perp$, and their additional lattice plane on the γ’ side, will result in a two-peaks distribution of the lattice parameters within the material, as in Fig. 2, but with a very small width. However, some disorder (position fluctuations or presence of dislocations with a different Burgers vector) may result in long range fluctuations of the stress field, i.e. in wider peaks.

A first attempt to simulate the distribution of lattice parameters within the material was done, taking two arrays of dislocations with [110] and $[1\bar{1}0]$ line directions for each interface, $b_\perp = 2^{-1/2}$.a, and a = 3.45 10^{-10} m, with a distance d chosen so that $\delta_\perp$ = 2.7 10^{-3}. Five interfaces perpendicular to the [001] axis were considered, at positions z = - 500 nm (+), z = - 200 nm (-), z = 0 nm (+), z = 300 nm (-), z = 500 nm (+), where + (-) indicates a γ (γ’) to γ’ (γ) transition. Fluctuations of the raft/ channel thickness were not taken into account. The volume fraction of γ’ phase is 0.6. The strain due to both dislocation arrays was calculated at 200^3 points within a 2000^2 $nm^2 \times$ 250 nm (from z = -100 nm to z = 150 nm). The resulting strain distribution is given by plot A (Fig.5b). And the top of Figure 5a lets visualize the analysis for A. Using the same fit procedure as for experimental peaks, width parameters w_γ= 1.58 10^{-4} and $w_{\gamma'}$ = 1.16 10^{-4} are found (point A in Fig.5c). Due to the assumed periodicity of the γ/ γ’ stacking, these values are lower than that found for an experimental profile recorded at the beginning of the experiment.

Part of the initial dislocations were then replaced by a pair of dislocations belonging to one of the eight slip systems with a non-zero Schmid factor, with random Burgers vectors orientation, except the edge component in the interface plane, and put at a random position within the same interface as it is shown in the middle of figure 5a. These dislocations can be seen as dislocation segments left at an interface by a mobile dislocation gliding in the γ corridor. All the (w_γ, $w_{\gamma'}$) points (empty squares) are found on a straight line with a slope 0.68, with profile and point B standing for the replacement of 90% of the initial dislocations.

Adding dislocations with the same random Burgers vectors and line directions at random points within the γ' layers (Bottom of Fig.5a) moves the position in the diagram (Fig.5c) along a line with a slope ≈ 1.67 (dots and triangles). Going from 0 to 4 10^{13} dislocations m^{-2} changes profile B into C in Fig. 5b, and point B to point C in Fig.5c. The value of the slope is to be compared to the 1.61 slope measured for the experimental points (full diamonds). The changes in the peaks during the experiments might thus be linked to the introduction or annihilation of a few 10^{13} dislocations m^{-2} within the rafts.

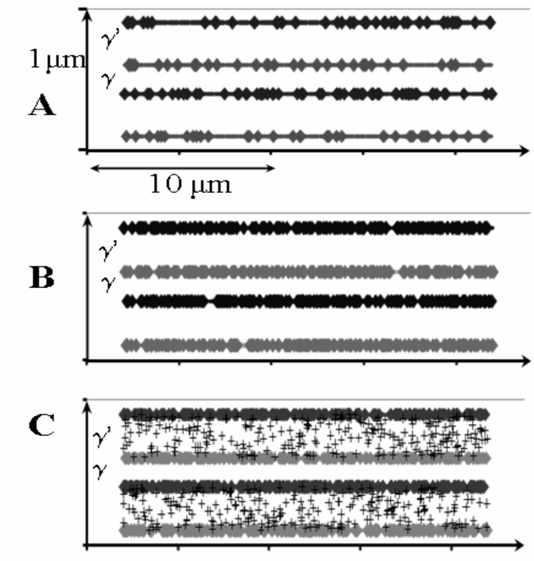

Figure 5a. Dislocation used for simulation: A: well-ordered interfacial dislocation array. B: highly disordered interface dislocation array. C: same plus dislocations within the γ' rafts.

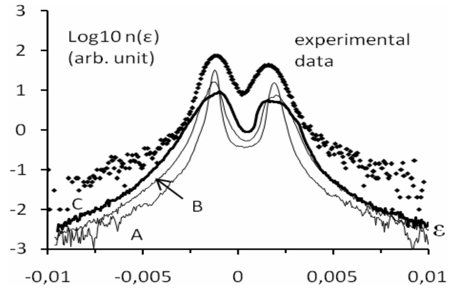

Figure 5b. Distribution of the strain within the material, due to disorder at the interface (A to B) and dislocations within the rafts (B to C).

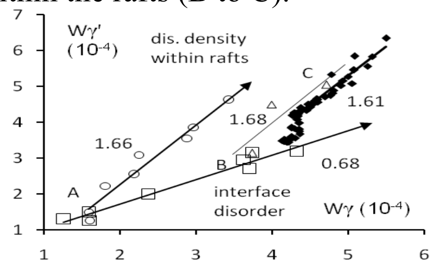

Figure 5c. Relative evolution of the w parameters with increasing interface disorder and/or increasing dislocation density within the rafts.

4. Conclusion

The strain rate of the γ' phase of a superalloy was measured during high temperature loading and unloading experiments. A correlation can be found between the long time response of the rafts and changes in the width parameters of the TCD profiles. These changes are themselves probably correlated with the introduction of a dislocation density of a few 10^{13} m^{-2} within the rafts.

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