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Strains and dislocation densities within a rafted superalloy: *in situ* investigation during mechanical testing at 1080°C

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Abstract. During *in situ* investigation of the distribution of lattice parameters within a superalloy with a rafted microstructure by Three Crystal Diffractometry, both the γ and γ' peak positions and shapes are shown to change. While the peak positions can be used to measure the average stresses and strains within both phases, the changes in shape are related to these in the dislocation distribution within the material. It is shown that the density and order of the dislocation array at the γ/γ' interface and within the γ' phase give different contributions to the peak width, and that the widening of both peaks under high stresses is due to an increase of the dislocation density within the rafts.

1. Introduction

During the major part of their high temperature creep life (i.e. stage II of the creep curve), single crystal superalloys have a so-called rafted microstructure [1]: semi-coherent platelets, or rafts, of the $L1_2$ γ' phase perpendicular to the [001] tensile axis, are embedded in a disordered fcc γ matrix. Such a microstructure can be seen as the simplest two-phase material, i.e. a stack of alternating phases where dislocations move in both phases by glide or climb, and are trapped or transmitted at the γ/γ' interfaces. It might thus be possible to build physically-based constitutive laws.

In order to build such laws with some confidence, we need to know several parameters, such as the stresses within both phases, the strain rate of each phase, and if possible to have some insight about dislocation densities and their variations. Such data are however difficult to obtain from usual post mortem TEM investigations, as the volume fraction of each phase and the lattice parameters change, and the dislocation microstructure might relax during cooling,

It has been shown [2-5] 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 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].

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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.

2. Experimental

A cylindrical AM1 single crystalline specimen ([001] tensile axis) with a 0.45 µm precipitate size was grown by SNECMA, and machined by ONERA. It was pre-strained in tension (1080°C, 120 MPa), until the onset of stage II of creep, in a high temperature tensile device adapted to in situ X-Ray tests, in order to obtain a raft microstructure, 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.

The distribution of the lattice parameters within the bulk of the specimens was measured *in situ* at ~ 300 s intervals by analyzing the diffraction profile of a (200) type reflection (i.e. perpendicular to the tensile axis), using scans of the analyzer crystal of the Triple Axis Diffractometer of the High Energy (BW5) beamline of Hasylab at 120 keV. 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 slopes (in logarithmic scale). The strain rate of the whole specimen was continuously measured during the test.

3. Results

3.1. 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.

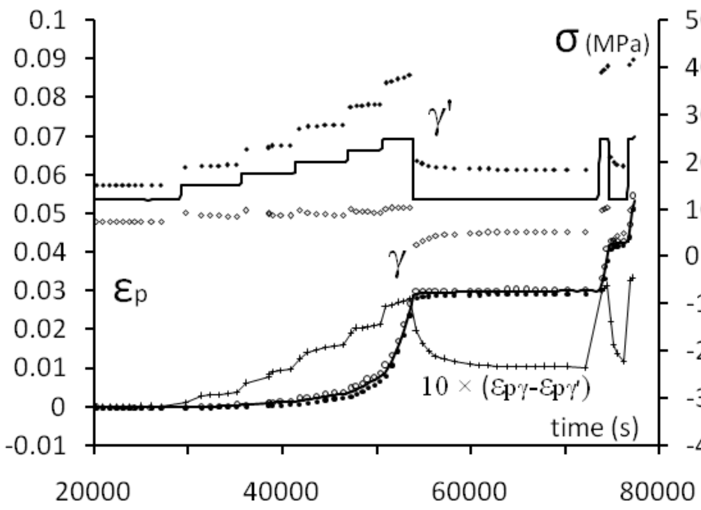

Figure 1. Von Mises stresses (top) and plastic strains (bottom) in the whole specimen (lines), in the γ ' (full symbols) and γ (empty symbols) phases. "Plus" symbols indicate the difference between the plastic strain of both phases, deduced from variations of $\delta_\perp$.

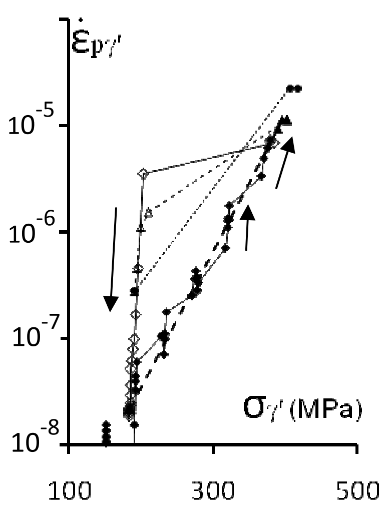

Figure 2. Stress dependence of $\dot{\varepsilon}_{p\gamma'}$. Diamonds, triangles, and dots (resp. full and empty) show the behavior during successive loadings (resp. unloadings) .

All internal stresses can be related to:

$$\delta_\perp = 2\left(a_{\gamma'100} - a_{\gamma100}\right)/\left(a_{\gamma'100} + a_{\gamma'100}\right) \quad (1)$$

where $a_{\gamma100}$ and $a_{\gamma'100}$ are the lattice parameters measured along [100]. The data are summarized in Figure 1 as a function of time, and the plastic strain rate of the γ' phase $\dot{\varepsilon}_{p\gamma'}$ is plotted vs. the Von Mises stress in Figure 2. 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 difference between the Von Mises stresses in the γ and γ' phases is ~ 10 MPa, the precision in their variations is within the MPa range. The same is true for the plastic strain: the precision in: $\varepsilon_{p\gamma}$ -$\varepsilon_{p\gamma'}$ is better than that in the plastic strain of the whole specimen. It should be noted (Fig. 2) that $\dot{\varepsilon}_{p\gamma'}$ increases under constant stress (arrows) and, after unloading, remains high before slowly returning to its initial value: $\dot{\varepsilon}_{p\gamma'}$ depends on the stress, but also on a parameter which varies on longer timescales.

3.2. Evolution of the TCD peak profiles

Figure 3 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 Figure 4 their variation with time. Both parameters tend to increase with the applied stress, and decrease after unloading.

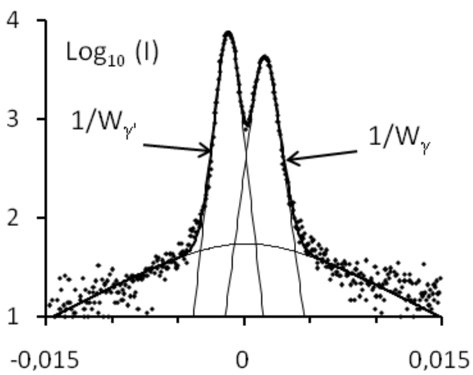

Figure 3. Definition of $w_{\gamma'}$ and w_γ.

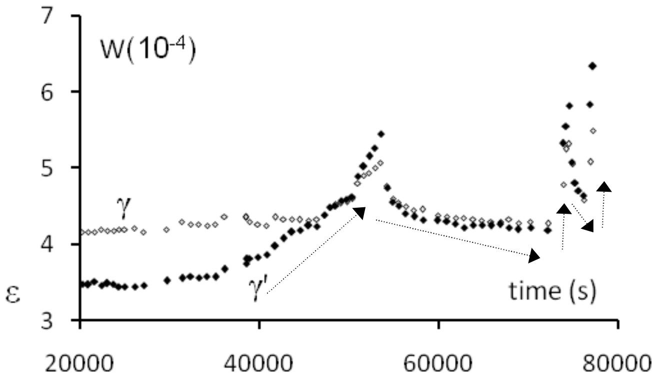

Figure 4. Evolution of $w_{\gamma'}$ and $w_{\gamma'}$ during the test.

4. 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. 3, 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. 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. 5). 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.6). 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. 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 moves the position in the diagram (Fig. 6) along a line with a slope ≈ 1.67 (dots and triangles). Going from 0 to 4 10^{13} dislocations m^{-2} and changes profile B into C in Fig. 5, and point B to point C in Fig.6. This value 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.

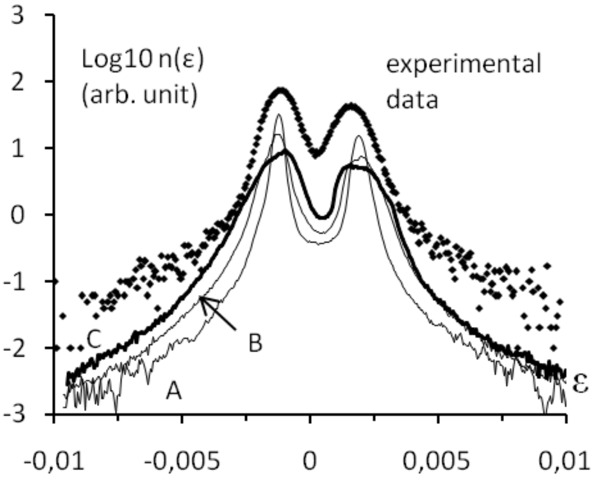

Figure 5. Distribution of the strain within the material, due to disorder at the interface (A to B) and dislocations within the rafts (B to C).

Figure 6. Relative evolution of the w parameters with increasing interface disorder and/or increasing dislocation density within the rafts.

5. 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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References

[1] Veron M, Brechet Y and Louchet F 1996 *Acta Mater.* **44** 3633.
[2] Kuhn H A, Biermann H, Ungar T, Mughrabi H, 1991 *Acta Metall.* **39** 2783
[3] Royer A, Bastie P, Bellet D, Strudel J L, 1995 *Phil. Mag .A* **72** 669
[4] Biermann H, Strehler M, and Mughrabi H 1996 *Metall. Mater. Trans. A* **27**, 1003
[5] Jacques A and Bastie 2003 *Phil. Mag. A* **83**, 26, 3005
[6] Jacques A, Diologent F, Caron P and Bastie P 2008 *Mater. Sci. Eng. A* **483-484** 568
[7] Sarosi P M, Srinivasan, R, Eggeler G F, Nathal M V and Mills M J 2007 *Acta Mater*. **55** 2509
[8] Mompiou F, and Caillard D 2008 *Mater. Sci. Eng. A* **483-484** 143