High-temperature deformation mechanisms in a polycrystalline nickel-base superalloy studied by neutron diffraction and electron microscopy

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Abstract

To study the effect of γ′ precipitate size on the deformation behaviour of a polycrystalline nickel-based superalloy, model microstructures with a unimodal γ′ size distribution were developed and subjected to loading experiments at 750 °C. Neutron diffraction measurements were carried out during loading to record the elastic lattice strain response of the γ and γ′ phase. A two-site elasto-plastic self-consistent model (EPSC) assisted in the interpretation of the elastic lattice strain response. In addition, the microstructures of the deformed specimens were analysed by (scanning) transmission electron microscopy (STEM). Excellent agreement was found between the EPSC and STEM results regarding a joint deformation of the γ and γ′ phase in the fine γ′ microstructures and for low plastic strains in the medium γ′ microstructures. With increasing γ′ size and increasing degree of plastic deformation, both experimental methodologies revealed a tendency of the two phases to deform independently.

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1. Introduction

Nickel-base superalloys are a structural material for applications that demand high strength at elevated temperatures as well as hot corrosion resistance [1,2]. The fundamental basis for their high-temperature strength is that they contain a significant volume fraction of γ′ precipitates, whose ordered L12 structure provides precipitation strengthening, particularly at high temperature. One of the main drivers for developing advanced nickel-base superalloys has been the desire to increase the turbine entry temperature (TET), since the performance and efficiency of the engine is greatly improved if the TET can be raised [3]. For high-pressure compressor and turbine discs, where polycrystalline nickel-base superalloys are applied because of the required balance of high-temperature strength/creep resistance and fatigue properties, an important development has been the rising volume fraction of γ′. As

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a consequence, conventional γ′ strengthened polycrystalline nickel-base superalloys, such as Waspaloy, are now being replaced in the most demanding parts of an aero engine by more advanced alloys with ~50 vol.% γ′ [4]. The current understanding and theories of precipitation strengthening in polycrystalline alloys were developed for materials with low precipitate volume fractions, where negligible interaction between the precipitates is expected. In the case of large γ′ volume fractions, the precipitates will constrain the γ matrix, which needs to be considered. In addition, a great deal of work has been performed on understanding the deformation mechanisms in single crystal nickel-based superalloys, which generally contain very high γ′ volume fractions (e.g. Ref. [5]). In contrast, studies on deformation mechanisms in polycrystalline nickel-base superalloys with a balanced volume fraction of γ and γ′ are rare [6–8]. However, in order to optimize their microstructure for best performance and for providing guidance when developing new alloys, it is imperative to improve the micromechanical understanding of the interaction between γ and γ′ for different γ′ particle sizes during mechanical loading and at temperature.

Nickel-base superalloys for disc application in aero engines generally possess a complex γ′ size distribution within the face-centred cubic (fcc) γ matrix. Depending on whether the material was heat treated above or below the γ′ solvus (usually between 1100 and 1200 °C), either a bimodal or a trimodal γ′ size distribution is observed [9]. In the latter case, non-coherent primary γ′ pins the grain boundaries during the sub-solvus solution heat treatment and effectively reduces the level of intragranular γ′. In the case of intragranular γ′, a cube–cube crystallographic relationship exists with the γ matrix and, because of their similar lattice parameter, the interface is coherent. Typically, the diameter of primary γ′ precipitates is in the range of 1–3 μm, whereas it is between 50 and 500 nm for secondary γ′ (intragranular) and 5–30 nm for tertiary γ′ (intragranular) [10].

A number of potential deformation mechanisms in γ′ strengthened nickel-base superalloys have been identified, including weakly and strongly coupled dislocations cutting γ′ [11], cross-slip of superdislocations in γ′ forming a Kear–Wilsdorf lock and dissociation of dislocations into partials [12], as well as Orowan looping of dislocations [13], dislocation climb and microtwinning [14]. The activity of many possible deformation mechanisms in the material during deformation is often closely related to the size distribution of the γ′ precipitates. For instance, if a single dislocation were to move through an ordered γ′ precipitate, it would leave an anti-phase boundary (APB) behind, increasing the energy of the crystal. Consequently, dislocations that cut through γ′ tend to move in weakly or strongly coupled pairs with the second dislocation cancelling the APB [15,16]. The difference between weakly and strongly coupled dislocations is related to the distance between the two dislocations and whether that distance is larger or smaller than the width of a precipitate [11]. Furthermore, there are a number of ways in which the dislocations can dissociate into partials, which can be favourable because the dislocation elastic energy is proportional to \( b^2 \), where \( b \) is the Burgers vector.

The bulk of deformation studies have been obtained by post-mortem analysis using, for example, transmission electron microscopy (TEM). Such studies alone often make it difficult to pinpoint the onset of a certain deformation mechanism during plastic deformation and the relative importance of the different mechanisms, especially in polycrystalline materials. For this reason, in situ studies have become more common, using, for example, highly penetrating neutron or high-energy synchrotron X-ray diffraction to measure the evolution of intergranular strains during plastic deformation [17–19]. The elastic lattice strain evolution recorded for a particular material during mechanical loading can be understood as a fingerprint of the dominant deformation mechanisms. In order to use such fingerprints for identifying deformation modes, crystalline plasticity modelling is required. The most commonly used plasticity model for such an analysis is the elasto-plastic self-consistent model (EPSC), which uses the Eshelby–Hill formulation [20,21]. For the interpretation of two phase materials, such as γ strengthened nickel-base superalloys, a two-site EPSC model was developed by Daymond et al. [7]. This adaptation of the model uses two inclusions inside an infinite medium. The two inclusions have, for the case of nickel-base superalloys, a cube–cube orientation relationship, and the volume fraction is determined by the relative sizes of the inclusions. Daymond et al. used in situ neutron diffraction to study deformation mechanisms in Udimet 720LI, with a trimodal γ′ size distribution, at various temperatures between 20 °C and 750 °C. Using the two-site EPSC modelling approach, it was demonstrated that a change in deformation mechanism occurred with increasing test temperature. In order to obtain a good fit between predicted and measured intergranular strain evolution, as well as predicting the measured flow curve accurately, the addition of cube slip was needed above 400 °C [7].

The deformation mechanisms in precipitation strengthened materials are known to be strongly dependent on the precipitate size. However, the microstructures that Daymond et al. tested were trimodal, and therefore the responses of the γ′ diffraction peaks consisted of diffraction signal from three different sizes of precipitate, which are expected to behave differently. For the present work and to circumvent this issue, material with three model microstructures with unimodal γ′ size distributions was produced. These were deformed at 750 °C, using neutron diffraction to record, in situ, the elastic lattice strain evolution. These data were then used in conjunction with EPSC modelling to study the effect of γ′ size on the deformation mechanisms of this superalloy. Note that the same methodology was applied previously to study deformation mechanisms at room temperature [28]. The temperature region of 750 °C is of particular interest, because it is considered to
be near the maximum temperature that an advanced nickel-base superalloy for disc application can sustain for an extended period of time.

2. Experimental and modelling methods

2.1. Material

The material studied was RR1000, a nickel-base superalloy developed by Rolls-Royce plc and used in disc components in the high-pressure compressor and turbine of aero engines [22]; see Table 1 for the composition [10]. Compared with more conventional nickel-base superalloys such as Waspaloy and Inconel 718, RR1000 has a higher volume fraction of γ′ (at ~50 vol.%). As mentioned above, disc alloys exhibit a complex bimodal or trimodal γ′ size distribution. This is related to the continuous nucleation and growth of γ′ when the material is cooled from the solution heat treatment that is carried out either slightly below or above the γ′-solvus [9]. For this reason, developing an improved understanding of the γ′ particle size on deformation mechanisms in such alloys has been hampered. To overcome this problem, model microstructures with unimodal γ′ size distributions have been developed that exhibit γ′ precipitation sizes of 90, 130 and 230 nm (Fig. 1). This was achieved by first heat treating the material above the γ′-solvus for 1 h at 1180 °C to dissolve all γ′, followed by oil quenching, which produced a very fine bimodal γ′ distribution with sizes of ~60–70 and 10 nm. A second heat treatment of 800, 925 or 1050 °C was then applied to allow growth of the precipitates to 90, 130 or 230 nm, respectively. In each case, the material was cooled very slowly (1 °C min⁻¹ above 925 °C, and 0.1 °C min⁻¹ below 925 °C) in order to allow the precipitates present during the second heat treatment to grow without nucleating new γ′. In addition, such slow cooling rates should ensure that the chemistry of γ and γ′ stays comparable for the three microstructures. Previous work had demonstrated effects of cooling rates on lattice mismatch and γ′ chemistry because of differing diffusion rates of the various γ′ stabilizers through the γ matrix [23]. Here, X-ray diffraction on electrochemically extracted γ′ was used to confirm that, despite their different size, the chemistry of the γ′ particles was similar. These experiments revealed variations of less

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Cr</th>
<th>Co</th>
<th>Mo</th>
<th>Al</th>
<th>Ti</th>
<th>Ta</th>
<th>Hf</th>
<th>Zr</th>
<th>C</th>
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</tr>
</thead>
<tbody>
<tr>
<td>RR1000</td>
<td>14.35–15.15</td>
<td>14.0–19.0</td>
<td>4.25–5.25</td>
<td>2.85–3.15</td>
<td>3.45–4.15</td>
<td>1.35–2.15</td>
<td>0.0–1.0</td>
<td>0.05–0.07</td>
<td>0.012–0.033</td>
<td>0.01–0.025</td>
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Fig. 1. FEG-SEM images showing the (a) fine, (b) medium and (c) coarse γ′ microstructure. Note that, apart from (a), fairly unimodal γ′ size distributions were achieved.
than $7.5 \times 10^{-4}$ Å in the $\gamma'$ lattice parameter for the three different particle sizes.

2.2. In situ high-temperature loading experiment

The three different model microstructures were deformed in uniaxial tension while monitored in situ, using neutron diffraction on the ENGIN-X beam line at the UK neutron spallation source ISIS [24]. It is important to note that the intensity of the $\gamma'$ superlattice reflections is significantly stronger when using neutron compared with X-ray diffraction. In the diffraction spectrum of a fcc structure, the (100) and (110) reflections are extinct because of the existence of half planes, resulting in destructive interference. However, with an L1$_2$ structure, the atoms in the adjacent planes have different scattering lengths and therefore the (001) peak is not completely extinguished. The X-ray scattering lengths of nickel and titanium are relatively similar and determined by the atomic number. However, neutron scattering is a nuclear interaction, and neighbouring elements in the periodic table can have substantially different scattering characteristics [25,26]. Most importantly for the present case, titanium displays a negative neutron scattering length, while nickel and other alloying elements have a positive neutron scattering length [27]. Since titanium partitions to $\gamma'$, the tendency for extinction of the superlattice reflections is relatively weak when using neutron diffraction. Despite this, a relatively long counting time of $\sim$20 min is still required on ENGIN-X to obtain sufficient signal-to-noise ratios for the various superlattice reflections.

The tensile samples were of cylindrical shape with a gauge length of 50 mm and 6 mm diameter. The high-temperature loading experiments were carried out on an INSTRON 100KN tensile rig. Macroscopic strain was monitored on the samples using a dynamic high-temperature extensometer clip gauge, while the diffraction spectrum of the loading and transverse directions were recorded using the two-detector banks, as indicated in Fig. 2. The experimental setup is explained in more detail in Ref. [18]. The diffracting gauge volume was defined using slits that were 4 mm high and 8 mm wide on the incident side and 4 mm collimators on the diffracting side. The tensile samples were heated to 750 °C using an optical furnace, which uses a thermocouple spot welded to the sample to control the temperature. The two-detector bank setup at ENGIN-X enables the simultaneous measurement of the elastic lattice strains in the loading and transverse directions. Because of the comparatively long counting times to acquire good enough data to enable the deconvolution of the $\gamma$ and $\gamma'$ spectra, a continuous strain rate could not be applied during tensile loading. Therefore, the samples were loaded in steps and held at each load for 20 min, while the diffraction spectra were recorded. The experiment was carried out in load control to avoid any stress relaxation during data acquisition. Consequently, some creep strain was measured during the experiment, which increased with increasing load. The amount of strain accumulated during each holding period can be deduced by comparing the strain between each data point in the stress–strain curves in Fig. 3. The frequency of measurement points was increased by decreasing the load steps around the yield point, which is the area of most interest.

The behaviour of different grain families was investigated using single peak analysis. Owing to the similar lattice parameter of $\gamma$ and $\gamma'$, the only distinguishable difference in the diffraction spectra comes from the ordered nature of the L1$_2$ structure. Hence, the $\gamma'$ phase has
additional reflections compared with the γ phase, which does not have any peaks that can be fitted independently. The individual phase responses can only be “deconvoluted” from the (200) and (220) reflections using the information of the corresponding γ‘ superlattice peaks [28]. The exact knowledge of the (100) and (110) reflections enables one to fix the γ‘ (002) and (022) peak position and width when using a double peak fitting routine to isolate the (200) and (220) responses of the γ matrix.

2.3. Plasticity modelling

The two-site EPSC model allows one to model the deformation of two-phase materials. By fixing the crystallographic relationship between the two inclusions to a cube–cube orientation, the orientation relationship between γ and γ’ can be simulated. In the EPSC code, the possible slip systems for each orientation are defined, and shear will be allowed where the resolved shear stress exceeds a critical value, which is determined by the critical resolved shear stress and amount of hardening. An extended Voce hardening law, given by Eq. (1), is used to calculate the hardening on each group of slip systems and for each grain. Here, τ is the resolved shear stress, τ₀ is the initial resolved shear stress, τ₁ + τ₀ is the value of the shear stress where the secondary hardening rate has a shear strain of 0, θ₀ is the initial hardening rate, and θ₁ is the final hardening rate.

\[
τ = τ₀ + (τ₁ + θ₀γ)(1 - e^{θ₀γ/τ₀})
\]  
(1)

In recent years, this modelling approach has been used extensively on a range of quasi-single-phase engineering alloys to identify possible slip modes [17,18,29].

A central aspect of the elastic lattice strain data recorded by neutron diffraction is that they provide additional information that can be applied to constrain the choice of parameters used in the plasticity model. In other words, input parameters from Eq. (1) are not only chosen to fit a stress–strain curve, but also to the elastic lattice strain evolution measured in the longitudinal and transverse direction by neutron diffraction [20]. The fitting parameters used in this case are the stiffness values for the elastic region, the slip systems and the coefficients of the Voce hardening law for the plastic behaviour. It is important, particularly in the case of a dual phase material, that a large enough number of grains is modelled to gain suitable statistics for the lattice strain of individual grain families. In the present case, it was sufficient to use 1000 grains in the calculations along the loading direction, while to capture the (100) reflection in the transverse direction required ~5000 grains, and for the (110) reflection in the transverse direction, ~50,000 grains were required.

2.4. Scanning TEM

Scanning TEM (STEM) was employed to image the deformation structure after failure [30]. Foils were cut from the tensile specimens parallel to the loading direction and ground down before 3 mm discs were punched out of the material. The discs were thinned further to a thickness of 100–150 µm and then electropolished using 8% perchloric acid in acetic acid and a twin-jet Tenupol at 10 °C and 40 V. STEM imaging was performed on an FEI Tecnai F20 XT with a field emission gun (FEG) at 200 kV at Ohio State University, Columbus, OH, USA.

3. Results

3.1. Generation of the model microstructures

Fig. 1 presents FEG-SEM images of the model microstructures. For the medium and coarse γ‘ microstructures, a unimodal γ‘ size distribution was successfully generated. The average particle size, including standard deviation using the linear intercept method, and the γ volume fraction are given in Table 2. The 800 °C heat treatment did not result in a strictly unimodal γ‘ size distribution, as the very fine γ‘ formed during oil-quenching did not dissolve at this temperature. In this case, only the slightly coarser γ was considered in the quantitative analysis, while the very fine γ was estimated to be ~20 nm.

3.2. Yield stress and elastic lattice strain

When determining the best parameters for the EPSC model, agreement is sought with the bulk stress–strain curve and the elastic lattice strain curves. Fig. 3 shows
the measured bulk stress–strain curves for each microstructure tested at 750 °C compared with the EPSC predictions. The yield stresses (estimated from the deviation from the elastic region) for the fine, medium and coarse γ' microstructures were 550, 450 and 350 MPa, and the strain to failure was 2.5, 2.9 and 3.1%, respectively. Hence, as the γ' particle size is increased, the yield stress reduces, but the ductility increases slightly. The reason for the low ductility at 750 °C is currently not known. It is possible that the long holding periods at high stress levels, required for the accurate measurements of the diffraction spectrum, lead to creep cavitation, or that the unusual heat treatment procedure for generating the model microstructures resulted in either defects from the oil quench or the formation of the brittle sigma phase (although the latter could not be detected in the diffraction pattern). In addition, with reduced γ' particle size, both the initial and final hardening rates decrease. Fig. 3 also shows that the EPSC model is capable of closely fitting the measured stress–strain curves of the three microstructures by adjusting the parameters used in the Voce hardening law. Therefore, the stress–strain curves were fitted alongside the elastic lattice strains measured by neutron diffraction in the loading and transverse direction.

Figs. 4–6 show the measured and best fitted elastic lattice strain evolution of the (200) and (220) γ grain and γ' precipitate families for the three microstructures. In the case of the fine γ' microstructure (Fig. 4), the elastic lattice strain responses of the γ and the γ' phase are almost identical throughout the deformation process (with only a slight variation just before the sample failed) implying a joint deformation of γ and γ'. In contrast, the elastic lattice strain responses of γ and γ' deviate in the plastic regime for the medium (Fig. 5) and coarse (Fig. 6) γ' microstructures. In the case of the coarse γ' microstructure, the deviation occurs close to the yield point of the material, while in the case of the medium γ' microstructure, more plasticity is required before load partitioning between γ and γ' takes place. After the point of deviation, the γ' phase takes up more elastic lattice strain than γ in both the medium and coarse γ' microstructure. This behaviour was also observed for the coarse γ' microstructure at 20 °C and 500 °C [28]. As the γ' phase takes up more elastic lattice strain, the γ phase takes up more plastic strain, indicating a load transfer from γ to γ'.

Generally, reasonably good agreement between the recorded and predicted elastic lattice strains was obtained in the loading direction, whereas in the transverse direction this was less the case, as previously reported in other materials, and discussed in Ref. [31]. Most importantly, it was possible to predict the different levels of load transfer between γ and γ' for the different types of microstructures by adjusting the phase-specific critical shear stresses and hardening rates.

3.3. Dislocation studies by STEM

The STEM images of the deformed microstructures after failure (Fig. 7) show stacking faults in all three microstructures. In the fine γ' microstructure, these stacking faults tend to extend through both the γ and γ' phases equally, whereas in the medium and coarse γ', the stacking faults...
faults are exclusively limited to the $\gamma'$ precipitates, highlighting a difference in the respective deformation behaviour. Dislocations are found to pile up around the precipitates in the medium and even more so in the coarse $\gamma'$ microstructure, but not in the fine $\gamma'$. No rafting, coalescence or coarsening of particles was observed in the microstructures after failure.

4. Discussion

The in situ loading experiments at 750 °C show that the yield strength of the material increases as the $\gamma'$ particle size decreases in the range of precipitate sizes studied in this work. Using the additional information recorded during the in situ experiment and the information obtained from
the EPSC model, it is now possible to obtain a better micromechanical understanding of the early stage of deformation.

Fig. 8 plots the hardening curves used for the fine, medium and coarse $\gamma'$ microstructures, highlighting the more detailed information available from the EPSC model. These curves represent the hardening behaviour used by the model to achieve the fits shown in Figs. 4–6. It can be seen that, with increasing $\gamma'$ particle size, the $\gamma$ matrix starts to yield at lower stresses. It should be noted that this trend is not only a result from the EPSC model, but can also be clearly identified from the neutron diffraction data in terms of non-linear increases in the elastic lattice strain measured in the $\gamma$ matrix. This non-linear response of the $\gamma$ reflections, indicating plastic deformation of that phase, appears at increasing applied stress levels for decreasing $\gamma'$ particle size. An increase in the secondary hardening rate with increasing $\gamma'$ size can be deduced from the curves presented in Fig. 8. This trend is also seen in Fig. 3 when comparing the flow curves of the fine, medium and coarse $\gamma'$ microstructure and when plotting the evolution of full width at half maximum (FWHM) as a function of plastic strain (Fig. 9). In this case, the evolution of the normalized FWHM (normalized by the FWHM for the unloaded condition) of the $220^{\gamma'}$ and $110^{\gamma'}$ peaks are plotted against the total strain. The formula used for normalizing the FWHM is given in the following equation:

\[
FWHM_{\text{norm}} = \frac{FWHM}{FWHM_{\text{unloaded}}} - 1
\]

It is clear that, with increasing particle size, peak broadening is greater for a given strain. The FWHM results presented in Fig. 9 therefore suggest increased dislocation interaction and retained stored energy with increasing $\gamma'$ particle size during plastic deformation, which is again characteristic of a higher hardening rate. A higher
Dislocation density in the γ matrix is also observed qualitatively in the STEM images of the medium and coarse γ’ microstructure (Fig. 7).

The STEM images of the deformed fine γ’ microstructure show stacking faults extending through both phases (Fig. 7a), which means that the same slip system is active in γ and γ’. There is little evidence for accumulation of dislocation content in the matrix around the precipitates. Therefore, the γ and γ’ phases appear to be deforming jointly, a hypothesis that is supported by the neutron diffraction and ESPC modelling results. In the elastic lattice strain response of the fine γ’ material (Fig. 4), hardly any load transfer is observed throughout the course of plastic deformation, implying that γ and γ’ always take up a similar amount of plastic strain, as is seen in the phase-specific plastic strain results of the EPSC model (Fig. 10a). A further indication of the joint deformation in the fine microstructure is that the two phases display almost identical hardening behaviour according to the prediction of the EPSC model (Fig. 8a). The joint deformation of the two phases in the fine microstructure might be related to the presence of very fine γ’ precipitates in addition to the 90 nm size precipitates. These particles might impede the independent deformation of the matrix phase, which was observed in the other two microstructures.

The joint deformation of the two phases also appears to result in the most efficient hardening of the γ matrix by the precipitate phase, as the phase-specific stress of the γ phase as well as the yield strength are highest for this microstructure. Furthermore, recent phase field modelling results indicate that matrix dislocations will tend to dissociate and decorrelate in microstructures with finer γ’ precipitates and narrower channels, owing to the relative forces acting on the leading and trailing Shockley partials. The decorrelation of matrix dislocations forms intrinsic stacking faults in the matrix, and is furthermore considered to be a necessary precursor to shearing of the γ’ precipitates by superlattice stacking faults and microtwins.

In the medium and coarse γ’ microstructures, the stacking faults are confined to the precipitates and not seen in the matrix (Fig. 7b and c), which highlights a change in deformation behaviour from the fine γ’. Similar deformation behaviour is found after creep in single-crystal nickel-base superalloys, where <112> type dislocations cut through the γ’ precipitate. It is not surprising that the deformation structure observed here resembles creep deformation, considering the holding periods and stepwise loading pattern applied during the neutron diffraction experiment. Since the stacking faults are only found in the γ’ phase, a type of dislocation cuts through the precipitate, different from that travelling through the γ matrix. At the particle interface, the dislocations of the matrix have to combine in order to create a dislocation that can cut through the precipitate. The resultant barrier to dislocation movement is also evident from the accumulation and pile-up of dislocations in the γ channels, which is observed in the STEM images of the medium and especially the coarse γ’ microstructure (Fig. 7b and c).

A change in deformation mechanism from the fine to the medium and coarse γ’ can also be deduced from the elastic lattice strain data (Figs. 5 and 6). Unlike in the fine γ’, there is a difference in the elastic lattice strain response of γ and γ’ in the case of the medium and coarse microstructures.
Load transfer from $\gamma$ to $\gamma'$ is observed. With increasing $\gamma'$ size, this load transfer starts at lower plastic strains and becomes more pronounced. The difference in the elastic lattice strain responses shows that $\gamma$ and $\gamma'$ no longer deform together, which correlates well with the STEM results. As the dislocations have to wait at the precipitate interfaces for a suitable dislocation for reaction, a higher plastic strain is achieved in the $\gamma$ matrix than in the precipitate. The difference in plastic strain is also seen in the results of the EPSC model where, for the medium and coarse $\gamma'$ microstructures, the phase-specific plastic strain of the $\gamma$ phase is higher than for $\gamma'$ (Fig. 10b and c). An important observation of the phase-specific hardening curves is the apparent softening of the $\gamma$ matrix with increasing $\gamma'$ particle size. The greater interparticle spacing in the coarse $\gamma'$ microstructure allows the matrix to deform on its own, while in the fine $\gamma'$, the small interparticle spacing as well as the tertiary $\gamma'$ present only in this microstructure forces the matrix to deform jointly with the precipitates, leading to a higher yield strength.

The differences observed between the three microstructures might be linked to the occurrence of cross-slip in the material. The alloy studied here was developed to have a low stacking fault energy in order to promote planar slip, which is beneficial for low crack growth rates [33]. This desired behaviour seems to occur in the fine $\gamma'$ microstructure with stacking faults extending through both $\gamma$ and $\gamma'$, hence encouraging planar slip patterns in both phases. In the other two microstructures, where stacking faults are found only in the precipitates, but not in the matrix, cross slip might be activated in the matrix but inhibited in the precipitates because of these stacking faults. This might further contribute to the difference in the elastic lattice strain of the two phases observed in the medium and coarse microstructures. Without the activation of cross-slip in the precipitates, their deformation might be mainly elastic at this point during the plastic regime, explaining the larger elastic lattice strains compared with the $\gamma$ phase.

At this stage it is interesting to compare the present observations with previous studies of exactly the same alloy and microstructures, but loaded at 20 °C and 500 °C [28]. Remarkably, the behaviour seen in the elastic lattice strain response at these lower temperatures was essentially the same as observed here at 750 °C. It showed the same tendency for increased load to transfer from $\gamma$ to $\gamma'$ with increasing $\gamma'$ size, with no load transfer in the fine precipitates and observation of such transfer in the medium and coarse $\gamma'$ microstructures. It was also more pronounced and started “earlier” in the coarse $\gamma'$ microstructure compared with the medium $\gamma'$ microstructure, i.e. at a lower plastic strain. However, what was indeed different during the high-temperature experiments was that less plastic strain was required for the onset of load transfer, and that the degree of load transfer was much greater compared with the room temperature experiments. Although the general behaviour is the same, the amount of load transfer, when it occurs, is more pronounced at higher temperatures. This indicates that the strength of the $\gamma'$ phase in the material decreases more slowly than that of the $\gamma$ phase as a function of increasing temperature. Furthermore, the contribution of the $\gamma'$ phase to the overall strength of the material changes differently for different particle sizes as the temperature is increased. At room temperature, a large percentage of the plastic regime of the medium microstructure was spent in joint deformation of $\gamma$ and $\gamma'$, a behaviour that is closer to the behaviour of the fine precipitates, whereas at 750 °C this percentage is much lower. The behaviour at 750 °C is therefore more similar to that shown by the coarse particles. In other words, when the temperature is raised, the particles need to be smaller to ensure joint deformation.
In RR1000 tested at 20 °C and 500 °C, coupled dislocations were observed at least in the fine γ′ microstructure, and the occurrence of load transfer (after a certain amount of plastic deformation) was attributed to the onset of dislocation bowing in addition to particle shearing, as both processes were found in the microstructures showing load transfer [28]. In contrast, here single dislocations appear to operate in all three microstructures, as the abundance of stacking faults indicates, and load transfer seems to be associated with the presence of stacking faults that are limited to the precipitates, as demonstrated by the STEM analysis.

Another interesting comparison can be made with previous work carried out on UDIMET 720LI with a conventional trimodal γ′ distribution [7]. In that particular work, load partitioning between the phases was observed from the outset when the material was tested at 750 °C, but not at room temperature. So, while at room temperature the load partitioning observed for the UDIMET trimodal γ′ size distribution resembles the behaviour of the fine γ′ microstructure in RR1000 [28], in contrast at high temperatures, the trimodal UDIMET microstructure displays similar behaviour to the coarse γ′ microstructure of RR1000.

Whereas in the work of Daymond et al. [7] a change in deformation mode from octahedral to cube slip was required to model the diffraction elastic strains at high temperatures, the modelling results here and in Ref. [28] suggest that the slip modes in γ and γ′ at 750 °C and 20 °C are the same. In particular, there seems to be no need to invoke cube slip. The biggest discrepancies between the predicted and measured elastic lattice strains occur at higher plastic strains, where the particles behave harder than modelled, for all microstructures. This effect is stronger for the 100 reflection, and therefore it is suggested that this has to do with the onset of multiple slip. This discrepancy was also observed at 20 °C and 500 °C, where the deviations were even larger.

The findings presented here show that the fine γ′ microstructure displays the deformation structures desired for good fatigue strength. While it does have the highest yield strength, hence taking best advantage of the increase in strength of the γ′ phase, the fine microstructure is not very ductile at high temperatures. This might lead to early failure if, for example, the yield strength is locally exceeded as a result of stress concentration around carbides or other particles. In that event, the higher ductility of the material with coarse γ′ particles would be preferable.

In conclusion, a comparison of the elastic lattice strain data with the electron microscopy results shows excellent agreement between the two methods. The most striking difference in the elastic lattice strain results when the γ′ size increases is the increasing load transfer between the two phases. In the STEM results, this is manifested in the difference in dislocation density between γ and γ′. In the STEM images of the fine γ′ microstructure, it is hard to distinguish between the two phases, just as no difference is seen in the elastic lattice strain response. In the coarse γ′ microstructure, the precipitates can easily be distinguished from the matrix, as dislocations pile up around them, which correlates well with the difference in the elastic lattice strain response that is found. The medium γ′ microstructure displays a behaviour that is in-between that of the fine and the coarse microstructures regarding both the STEM images and the elastic lattice strain response.

5. Summary and conclusions

In situ loading experiments using neutron diffraction were carried out at 750 °C on model microstructures with a unimodal γ′ size distribution based on the polycrystalline nickel-base superalloy RR1000. The results were fitted using an EPSC model to identify possible deformation modes. The dislocation structure of the failed specimens was revealed by STEM analysis. The main findings can be summarized as follows.

1. The elastic lattice strain data gained from the neutron diffraction experiment as well as the EPSC model’s predictions indicate the γ and γ′ deform jointly in the fine γ′ microstructure, but not in the medium and coarse γ′.
2. A load transfer between γ and γ′ is observed in the elastic lattice strain data for the medium and coarse γ′ microstructures, indicating a different deformation mechanism from that in the fine microstructure.
3. This change in deformation behaviour is supported by the results of the STEM analysis, which shows that the same slip system involving single dislocations is active in both phases in the fine γ′ with continuous stacking faults extending through both phases. In contrast, in the medium and coarse γ′ microstructure, stacking faults are restricted to the γ′ phase, and the matrix dislocations do not penetrate the precipitates in a similar fashion as observed for the fine γ′ microstructure.
4. By undertaking comparisons with previous studies of the same material and microstructure, but tested at room temperature, only very subtle differences could be detected in terms of deformation mechanism, with the critical particle size for load transfer from γ to γ′ decreasing with increasing test temperature.
5. While evidence of cube slip in γ′ was anticipated when testing the material at 750 °C, neither the elastic lattice strain data in combination with plasticity modelling nor the STEM analysis provided any evidence of it.

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References