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Dislocation/precipitate interactions in IN100 at 650 °C

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ABSTRACT

The influence of γ' size on critical resolved shear stress in alloy IN100 at 650 °C has been examined by considering dislocation/precipitate interactions involving particle shearing and Orowan by-passing mechanisms. To achieve this, heat treatment procedures were carried out on smooth specimens to produce materials with variations in secondary and tertiary γ' size, while maintaining their respective volume fractions. These specimens were subjected to strain-controlled fully reversed cyclic loading at 650 °C. Thin foils extracted from these specimens, post-testing, were examined by transmission electron microscopy to identify the nature of the precipitate/dislocation interactions during plastic deformation. Results indicated the presence of shearing and Orowan by-passing mechanisms. These observations have been used as a basis to calculate the critical resolved shear stress as a sum of components contributed by solid solution and by γ' particles being sheared and looped. In this analysis, a critical particle size defining the shearing/looping transition has been determined and this has been used to calculate the relative volume fraction and size of particles contributing to the critical resolved shear stress. These analytical results have been compared with those experimentally obtained at 650 °C using smooth specimens with different precipitate sizes.

1. Introduction

Over the last few decades, many authors have examined the role of microstructure on deformation and damage mechanisms in precipitation strengthened Nickel based superalloys, with a focus on the role of dislocation/precipitate interactions [1–8]. Precipitate size and volume fraction have been shown to influence the hardening behavior and in particular, the yield strength. In this regard, the γ' precipitate/dislocation interactions are described in terms of shearing and/or Orowan by-passing mechanisms. The transition between these mechanisms is controlled by the precipitate size and volume fraction. The work of Shenoy et al. [9] on IN100 implies that all γ' particle/dislocation interactions are shearing. The initial critical resolved shear stress (CRSS) is described as being proportional to volume fraction and inversely proportional to size for secondary γ' (γ'_s) and primary γ' (γ'_p) particles. These precipitates are incorporated through shearing by strongly coupled dislocations, while the smaller particles, tertiary γ' (γ'_t), are incorporated through shearing by weakly coupled dislocations. For γ'_t, the CRSS is proportional to volume fraction and size. These patterns of γ' particle/dislocation interactions are consistent with how Milligan et al. [10] incorporates γ'_s and γ'_t into the yield stress for alloy IN100. However, their work shows that the γ'_p are of comparable strength as that of the matrix material and thus has no influence on the yield strength. These authors have shown that for IN100 there is no evidence of dislocation looping. Conversely, in the work by Heilmaier et al. [11] on IN100 at room temperature, CRSS is incorporated through shearing by weakly and strongly coupled dislocations, as well as, looping by edge and screw dislocations. Their work has shown that the transition between mechanisms is controlled by the particle size and suggests that both shearing and looping mechanisms exist simultaneously. Similar results are obtained by Reppich et al. [12] on Nimonic 105. For this alloy, large overaged particles of a diameter greater than 120 nm (volume fraction 22%) and 450 nm (volume fraction 51%), show differences in the experimental and theoretical data of yield stress, implying that both mechanisms operate. In addition, they suggest that the transition between shearing and looping occurs over a range of particle sizes, rather than a distinct critical size. Thus, for a continuum with a given mean particle size it is possible that both mechanisms operate. This is supported by transmission electron microscopy evidence of Nimonic 105 with particle sizes of 75 nm, 220 nm and 320 nm, showing the existence of shearing, shearing and looping, and looping respectively. Sinha's work [13] on IN-738LC, shows that during long creep tests, γ' precipitates ripen and coarsen, become irregularly shaped and develop rafted microstructures of plate or rod morphology. This has been addressed by using short-term

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strain relaxation (creep) and strain recovery tests. Sinha's work has shown that fitting of experimental data to a Norton power law form, has a strong dependence of the exponent on stress and microstructure. Sinha suggests that the change in value of this exponent is from a transition in which dislocations climb over gamma prime particles to that in which they cut the particles. Furthermore, the work of Del Valle et al. [14] on Inconel X-750 alloy have shown a critical γ' particle radius exists, below which particle shearing occurs and above which the Orowan looping mechanism operates.

The studies mentioned above, among others [see for example [15,16]], express different views on secondary γ' precipitate/dislocation interactions. The goal of this paper is to examine these mechanisms on the basis of the particle size and their relative influence on the hardening behavior in IN100. To achieve this, the first section of the paper describes heat treatment procedures aiming at producing microstructures with variations in their precipitate sizes while maintaining the respective volume fractions. In order to identify the nature of the precipitate/dislocation interactions during plastic deformation, strain-controlled fully reversed cyclic loading tests were carried out on specimens having as-received, as well as modified microstructures, at 650 °C. Thin foils were extracted from these specimens post-testing and were subjected to transmission electron microscope examination to identify the shearing and Orowan by-passing mechanisms in relation to the γ' particle size. These results were then used as a basis to calculate the components of critical resolved shear stress with contributions from secondary and tertiary γ' particles taking into consideration their relative dislocation/particle interactions.

2. Microstructure control

The role of secondary γ' particle size on the critical resolved shear strength is the focus of this work, thus, variation in this size is achieved by varying the heat treatment sequence of the as-received alloy. The model material in this work is the powder metallurgy Inconel 100 (IN100) with the chemical composition (in weight percent): 4.85 Al, 4.24 Ti, 18.23 Co, 12.13 Cr, 3.22 Mo, 0.71V, 0.071 Zr, 0.02 B, 0.072C and balance Ni [17]. The as-received condition, shown in Table 1, has seen the typical three stage heat treatment sequence; subsolvus solutioning, stabilization and aging. This treatment has resulted in the microstructure shown in Fig. 1a–c, which has an average grain size of 5 μm, and trimodal distribution of γ' particles, denoted primary, secondary and tertiary (γ'_p, γ'_s and γ'_t). The volume fraction and size of the γ'_p are 24.2% and 0.9 μm, γ'_s are 26.7% and 72.1 nm and γ'_t are 7.5% and 4.7 nm; respectively. The particle size is reported as mean equivalent particle radius. Two additional heat treatments were performed on the as-received material in order to vary their γ' statistics. These heat treatments, denoted overaged and water quenched, are listed in Table 1. Both heat treatments are performed at subsolvus conditions, thus maintaining the same grain size, as well as, the γ'_p volume fraction and size as that of the as-received material (see Fig. 1d and f). In addition, the volume fractions of the secondary and tertiary γ' show little variation, thus, an average value of 26.7% for γ'_s and 7.5% for γ'_t was considered for all microstructure conditions. The overaged heat treatment condition, shown in Fig. 1d and e, where an additional stabilization and aging sequence is applied, allows coarsening of both the γ'_s and γ'_t; whereas, the water quenched condition (see Fig. 1f and g) shows a decrease in the γ'_s particle size. In this case, the fast cooling rate from the solutioning temperature allows for precipitation of the γ'_s particles but does not provide sufficient kinetics for coarsening. The aging condition, which is similar to that of the as-received material, maintains the size of the γ'_t. The mean tertiary and secondary γ' sizes for the three microstructures are listed in Table 1. The mean particle radius of γ'_t for the three microstructure conditions were in the range of 2–16 nm, while γ'_s particles were in the range of 20–140 nm. Thus, a threshold particle size of 20 nm has been used here as the transition limit between γ'_t and γ'_s particle size.

3. Dislocation/precipitate interactions at 650 °C

In order to identify the nature of the precipitate/dislocation interactions during plastic deformation, a series of strain-controlled fully reversed cyclic loading tests were carried out, at 650 °C, on low cycle fatigue specimens that have been heat treated in the manner described in Table 1. The mechanical testing was carried out using a servo hydraulic test machine, equipped with a high temperature furnace and quartz rod extensometer. Fully reversed cyclic stress–strain tests ($R=-1$) are performed, at a strain rate of $1\times10^{-5}\ \mathrm{s}^{-1}$, with strain ranges varying from ± 0.6 to ± 0.9%. Cyclic stress–strain curves for the various microstructure conditions are shown in Fig. 2.

In order to analyze the deformation modes in term of dislocation mechanisms, transmission electron microscopy (TEM) investigations were carried out by means of Titan FEI TEM operating at 300 kV. TEM discs were prepared using a twin jet polisher, in an electrolyte of 10% perchloric acid in methanol, of the as-received and water quenched microstructure (as described in Table 1). These samples were cut from low cycle fatigue specimens, which have been cyclically loaded at a strain rate of $10^{-5}\ \mathrm{s}^{-1}$ and strain range of ± 0.08% at 650 °C, perpendicular to the loading axis. The deformation substructure of the as-received material is shown in Fig. 3 and the water quenched microstructure is shown in Fig. 4.

The as-received substructure shows dislocations which are curved and often blocked against secondary γ' precipitates which indicates high activation of the Orowan by-passing mechanism. While, there is some evidence of isolated shearing of particles, designated by **S** in Fig. 3, the Orowan by-passing mechanism is the prevailing one in the as-received material and is linked to the γ' particles size and their volume fraction which control the γ channel width. Similar deformation mechanisms were observed in failed specimens after dwell fatigue on U-720 and was related to the accumulated plastic deformation [18] and after creep tests in NR3 [19]. On the other hand, in the water quenched microstructure, the γ' precipitates seem to be sheared by supershockley dislocations a/3 (112) type (designated by $\mathbf{S_h}$ in Fig. 4a) leading to the stacking fault formation appearing as contrasted fringes between dislocations. This mechanism is described in References [20–22], as two super

Table 1
Heat treatment conditions of IN100 material and the corresponding γ'_t and γ'_s sizes (reported as mean equivalent particle radius).

HT condition	Solutioning	Stabilization	Aging	$\bar{r}_{\gamma' t}$ (nm)	$\bar{r}_{\gamma' s}$ (nm)
As-received	1149 °C/2 h/oil quench	982 °C/1 h/fan cool	732 °C/8 h/air cool	4.7	72.1
Overaged	/	815 °C/75 min/cool at 1 °C/min	732 °C/30 h/air cool	10.6	79.3
Water quenched	1135 °C/2 h/water quench	815 °C/2 h/ air cool	732 °C/8 h/air cool	4.7	37.9