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# 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 $\gamma'$ precipitate size on the deformation behaviour of a polycrystalline nickel-based superalloy, model microstructures with a unimodal $\gamma'$ 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 $\gamma$ and $\gamma'$ 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 $\gamma$ and $\gamma'$ phase in the fine $\gamma'$ microstructures and for low plastic strains in the medium $\gamma'$ microstructures. With increasing $\gamma'$ size and increasing degree of plastic deformation, both experimental methodologies revealed a tendency of the two phases to deform independently.


*Keywords:* Nickel-base superalloy; High-temperature deformation; Elasto-plastic self-consistent (EPSC) model; Neutron diffraction; Transmission electron microscopy

## 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 $\gamma'$ precipitates, whose ordered $L1_2$ 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 $\gamma'$. As

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a consequence, conventional $\gamma'$ 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.% $\gamma'$ [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 $\gamma'$ volume fractions, the precipitates will constrain the $\gamma$ 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 $\gamma'$ volume fractions (e.g. Ref. [5]). In contrast, studies on deformation mechanisms in polycrystalline nickel-base superalloys with a balanced volume fraction of $\gamma$ and $\gamma'$ 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 $\gamma$ and $\gamma'$ for different $\gamma'$ particle sizes during mechanical loading and at temperature.

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

A number of potential deformation mechanisms in $\gamma'$ strengthened nickel-base superalloys have been identified, including weakly and strongly coupled dislocations cutting $\gamma'$ [11], cross-slip of superdislocations in $\gamma'$ 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 the many possible deformation mechanisms in the material during deformation is often closely related to the size distribution of the $\gamma'$ precipitates. For instance, if a single dislocation were to move through an ordered $\gamma'$ precipitate, it would leave an anti-phase boundary (APB) behind, increasing the energy of the crystal. Consequently, dislocations that cut through $\gamma'$ 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 $\mathbf{b}^2$, where $\mathbf{b}$ 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, crystal 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 $\gamma'$ 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 $\gamma'$ 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 $\gamma'$ 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 $\gamma'$ 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 $\gamma'$ 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