

Segregation and η phase formation along stacking faults during creep at intermediate temperatures in a Ni-based superalloy



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ABSTRACT

In this paper, the local compositional and structural changes occurring in association with stacking faults in a Ni-base superalloy are characterized and related to the possible rate-controlling processes during creep deformation at intermediate temperatures. These rate-controlling processes are not presently understood. In order to promote stacking fault shearing, compression creep tests on specially prepared single crystals of an exploratory Ni-base superalloy were conducted at 760 °C in the [001] orientation. Scanning transmission electron microscopy (STEM) imaging was coupled with state-of-the-art energy dispersive X-ray (EDX) spectroscopy to reveal for the first time an ordered compositional variation along the extrinsic faults inside the γ' precipitates, and a distinct solute atmosphere surrounding the leading partial dislocations. The local structure and chemistry at the extrinsic fault is consistent with the η phase, a DO_{24} hexagonal structure. Density Functional Theory (DFT) and high angle annular dark field (HAADF)-STEM image simulations are consistent with local η phase formation and indicate that a displacive–diffusive transformation occurs dynamically during deformation.

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1. Introduction

Polycrystalline nickel-based superalloys are essential materials for disks in the hot section of jet turbine engines due to their high strength and microstructural stability at elevated temperatures. Until recently, the fatigue performance of polycrystalline turbine disks has been a property of primary importance. As the operating temperature and engine hold times increase, developing a complete understanding of creep deformation becomes imperative. Recent studies have shown that the prominent deformation modes are strongly temperature-dependent. Operation of $1/2\langle 110 \rangle$ dislocations in the γ matrix and cutting of the γ' precipitates by pairs of $1/2\langle 110 \rangle$ dislocations (linked by an antiphase boundary or APB) is observed predominantly following lower temperature (400–600 °C) testing [1–3], for which the temperature dependence

of the alloy strength is small. Stacking fault shearing and microtwinning become active at higher temperatures (600–800 °C), particularly under low strain rate or creep conditions [4–6]. Understanding the rate-limiting process for these higher temperature deformation modes is important for creating more accurate models of creep behavior and for improving the temperature capability of future alloys.

The mechanistic reason for the onset of time- and temperature-dependence at intermediate temperatures is not obvious since both stacking faults and microtwins are created by the shear movement of partial dislocations on close-packed {111} planes. For example, Chen and Knowles [7,8] proposed a mechanism for creation of superlattice extrinsic stacking faults (SESFs) based on the passage of two different $1/3\langle 112 \rangle$ superpartials on adjacent {111} planes. The $1/3\langle 112 \rangle$ superpartials are created by the interaction of two like $1/2\langle 110 \rangle$ dislocations at the γ/γ' interface:

$$1/2[110] + 1/2[110] = 1/3[12\bar{1}] + 1/3[211] \quad (1)$$

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There are several concerns with this mechanism: (a) the strong repulsive forces between the parent dislocations that would resist their interaction, (b) the two dissimilar $1/3\langle 112 \rangle$ dislocations cannot simultaneously experience a large Schmid factor driving the shearing event, and (c) since this is a pure shear process, it should not be strongly temperature dependent.

In early work by Kear et al. [9] and more recently by Decamps et al. [10,11], a mechanism for the creation of intrinsic and extrinsic faults in $L1_2$ precipitates was proposed that involves individual $1/2\langle 110 \rangle$ dislocations initiating shearing of the γ' precipitate, thereby forming an antiphase boundary (APB). This high energy fault is presumed to create a favorable situation for nucleation of a $1/6\langle 112 \rangle$ Shockley partial on the plane of (or adjacent to) the APB, and is equivalent to the net shear of a $1/3\langle 112 \rangle$ dislocation, which is a partial dislocation in the $L1_2$ superlattice. Kear [9] proposed that the nucleation of the partial loop could yield a temperature dependence. However, analysis by Zhou et al. [12] has criticized the Decamps model since the nucleation of the Shockley partial loop αB on the pre-existing APB would require overcoming a very large activation barrier.

The earliest proposed mechanism for forming superlattice extrinsic stacking faults (SESFs) was put forward by Kear et al. [13] who postulated that a the combination glide of three differently signed Shockley partials could create the DO_{24} crystal structure of the two layer SESF in an $L1_2$ precipitate. Soon after, they introduced the possibility that like-sign Shockley partial pairs could create a SESF; however, this scenario required the interaction of a dipole displacement near the dislocation core [14]. The introduction of the dipole displacement was their attempt to account for the wrong nearest neighbors that would be created in the ordered $L1_2$ structure due to the movement of Shockley partials. However, this concept of a dipole occurring at or near the dislocation core was not developed further and is not supported by more recent high resolution TEM analysis of partial dislocation configurations [15–17].

More recently [18], the concept of “reordering” has been suggested as a rate-limiting process under the assumption that stacking faults are created by the movement of combinations of $1/6\langle 112 \rangle$ Shockley partials rather than $1/3\langle 112 \rangle$ superpartials. For instance, Decamps et al. [19] proposed that the dissociation of two like $1/2\langle 110 \rangle$ type dislocations at the interface leads to shearing of the precipitates by $1/6\langle 112 \rangle$ Shockley partials. These partials form a high energy, complex stacking fault (CSF) during the shearing process. Therefore, if two partials shear a precipitate on adjacent planes, forming a two-layer CSF, the reordering process described by Kolbe et al. [18], and later in more detail by Kovarik et al. [20] could occur, converting the high energy, two-layer CSF into a low energy SESF. Since reordering is a local, diffusion-mediated process, it could potentially be responsible for the onset of time- and temperature-dependent behavior at intermediate temperatures. Indeed, Kovarik et al. used DFT calculations to explore possible diffusion pathways for reordering, and the associated activation barriers [3]. A simple model of deformation based on reordering as the rate-limiting process was developed by Karthikeyan et al. [21].

These previous models have been challenged recently by several studies that have revealed the presence of compositional fluctuations at stacking faults in both Ni-base [22] and Co-base [23] superalloys—studies enabled by the advent of advanced, thin-foil, energy dispersive spectroscopy (EDS) capabilities. These results have lent important support to the earlier observation of increased intensity of atomic column positions at stacking faults in alloy ME3 [6] and CMSX-4 [24]. For the Ni-base alloys, the indication from initial EDS analysis is that the composition at superlattice intrinsic faults (SISFs) within the γ' tend toward that of the FCC γ matrix. In the case of the Co-base alloys, the proposed explanation by Titus

et al. [20] is that the local DO_{19} structure of the SISF is tending toward that of the thermodynamically favored Co_3Ti phase. Thus, Titus et al. [20] argue that the shearing process is the result of a displacive–diffusive transformation at the fault.

The present paper focuses on an exploratory Ni-base superalloy, and provides the first structural and chemical analysis of superlattice extrinsic stacking faults (SESFs), which are the most prominent defect for this alloy under creep conditions at intermediate temperature. An alternative mechanism is proposed for forming the partials that enable γ' shearing involving unlike $1/2\langle 110 \rangle$ matrix dislocations. Definitive evidence for another type of local displacive–diffusive phase transformation, from faulted γ' to the η phase, is also provided for the first time. In addition, evidence for the formation of a distinctive solute atmosphere surrounding the leading partials of the SESF is also obtained for the first time. In order to explore the origin of this mechanism and confirm that it is sensible, we perform Density Functional Theory (DFT) [25] calculations and find that the observed segregation behavior of the solutes can be explained by a changed bonding environment around the SESF. The atomic structure used in the DFT calculations is validated by simulated HAADF images. Together, these results provide further evidence for the importance of long-range diffusional processes in association with shearing of γ' particles at intermediate temperatures.

2. Materials and experimental methods

2.1. Sample preparation

A single crystal analog of a disk alloy with minor compositional variations relative to the commercial alloy ME3 (with increases in Ta, Hf and Nb and decreases in Co and Cr as compared to ME3 which typically has 50.1% Ni, 20.6% Co, 13.0% Cr, 3.8% Mo, 2.1% W, 0.9% Nb, 2.4% Ta, 3.5% Al, 3.7% Ti, 0.04% C, 0.03% B, and 0.05% Zr) was obtained from GE Global Research Center in the form of a single, large casting after a heat treatment that formed a bimodal γ' precipitate microstructure. Before testing, microstructure analysis on this Alloy X was conducted to obtain volume fraction and average size for the secondary and tertiary precipitates. Samples were polished progressively down to a 1200 fine grit using SiC polishing pads and then further polished with a 0.05 μm colloidal silica finish. The alloy was then etched with a solution of 2 mL hydrofluoric acid, 30 mL nitric acid, and 50 mL lactic acid that preferentially etched the γ' precipitates. Using an FEI Sirion scanning electron microscope (SEM), backscattered electron micrographs of the alloy's microstructure were obtained and then analyzed using ImageJ [26]. For statistical significance, multiple images were obtained and over a thousand particles were included in the analysis. Fig. 1 shows an example of the microstructure present in Alloy

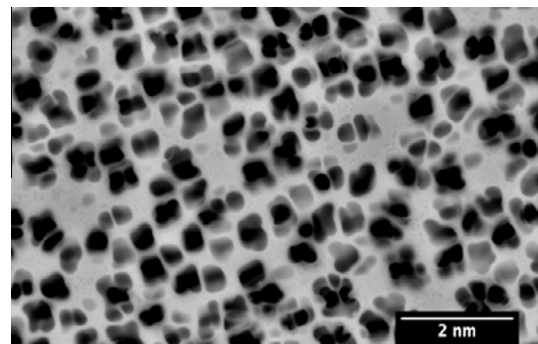


Fig. 1. Back-scatter SEM image of the microstructure of Alloy X where the γ' precipitates have been etched away (black) from the γ channels (grey).

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