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Diffusion processes during creep at intermediate temperatures in a Ni-based superalloy

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ABSTRACT

The local compositional changes associated with stacking fault and microtwin formation during creep at intermediate temperatures in a commercial Ni-base disk superalloy are explored. In order to investigate microtwin formation, an [001] single crystal of ME3 was tested in compression at 760 °C under a stress of 414 MPa — a stress-temperature regime found to promote microtwinning. Atomic resolution scanning transmission electron microscopy combined with state-of-the-art energy dispersive X-ray (EDX) spectroscopy analysis reveals the presence of Co and Cr rich Cottrell atmospheres around leading dislocations responsible for the creation of SISFs, SESFs, and microtwins. This analysis also highlights the role that tertiary γ particles inside γ' precipitates have on γ' shearing deformation mechanisms. Through the use of CALPHAD calculations, combined with new experimental observations, new insights into the rate-controlling processes during creep deformation are discussed.

1. Introduction

Ni-base superalloys are essential materials used primarily in the hot section of jet turbine engines. With the creation of each new generation of turbine engine the goal remains the same: increase the operating temperature of the engine, thereby reducing CO_2 emissions and fuel costs. This increase in temperature promotes changes in deformation modes in Ni-base superalloys that are still not fully understood. Multiple studies have found that the manner in which the γ' precipitates are defeated shifts from an athermal APB shearing at lower temperatures to a diffusion-mediated reordering at stacking faults during creep at temperatures above 700 °C [1–5]. One of these precipitate shearing modes is microtwinning, which has been found to adversely affect creep properties in Ni-based disk superalloys [6–9]. Thus, improved understanding of the rate-limiting mechanisms responsible for the formation of microtwins is needed to further improve the high temperature properties of these superalloys.

Mechanical microtwinning was first reported in Waspalloy by Guimier and Strudel [10]. Interestingly, it was found that a similar alloy tested using comparable parameters but without the presence of γ' precipitates did not exhibit twinning, demonstrating that γ' precipitates played an important role in the formation of microtwins [11]. In later work, microtwinning was determined to be orientation dependent, occurring in [110] and [111] oriented single crystals under tensile creep and [001] oriented crystals under compression creep [4,9,11–13]. Knowles and Chen [12], found that microtwinning occurred in orientations that also promoted superlattice extrinsic stacking fault (SESF) formation, implying the formation of both SESFs and microtwins were connected. This correlation was supported in future studies; however, early work incorrectly supposed that these faults were created by adjacent a/3<112> super-Shockley partials shearing the γ' precipitates on adjacent {111} planes [12,14]. Rather, later experimental evidence revealed that SESFs and microtwins were created by like-sign a/6<112> Shockley partials shearing adjacent {111} planes [2,13] [15]. In order for this shearing process to produce a low energy SESF, a reordering process, first proposed by Kolbe, must occur to the high energy complex extrinsic stacking fault (CESF) that would otherwise be created in the wake of the shearing Shockley partials [16]. Karthikeyan et al. [15] developed a quantitative model for microtwinning based on the hypothesis that the rate of partial shearing is controlled by the rate by which reordering can lower the energy of the CESF. Kovarik et al. expanded upon Kolbe's theory, using density functional theory analysis, and revealed an energetically favorable

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diffusion process that could remove wrong nearest-neighbor violations, thereby converting the high energy CESF into a low energy SESF [17]. Later, Smith et al. first reported the presence of a Co and Cr Cottrell atmosphere around shearing Shockley partials in a γ' precipitate, which they hypothesize is necessary to further reduce the energy of two layer CSF and promote precipitate shearing [18].

Kovarik et al. also provided qualitative evidence for the presence of segregation along SESFs and microtwin interfaces using atomic resolution high angle annular dark field (HAADF) imaging [17]. Indeed, elemental segregation has been recently confirmed along superlattice intrinsic (SISF) and SESFs in both Co and Ni based superalloys after intermediate temperature creep [18–20]. Furthermore, Smith et al. discovered that the type of elemental segregation along SESFs controlled whether the fault would thicken into a twin or not, thereby affecting the overall creep properties of the alloy. They predicted that the formation and extension of microtwins were reliant on the segregation of Co and Cr along the twin's interface, although no direct evidence for this was provided. Most recently, using atom probe tomography, Barba et al. [21], report evidence of Co and Cr along microtwins in a single crystal Ni-base superalloy, with higher concentrations along the twin interface.

The purpose of this study is to provide new insights into the formation of microtwins and the diffusional processes that enable it. Using high resolution Super-X EDX and CALPHAD calculations, a new microtwin formation model is presented highlighting the role of elemental segregation. Other observations include the presence and effect that newly discovered tertiary γ particles inside secondary γ' precipitates [18,22] have on creep performance. In addition, new evidence of Cottrell atmospheres around shearing Shockley partials inside γ' precipitates is provided, including for the first time around partials at a microtwin interface in association with the twin thickening process. Together, these new insights will help the creation of improved deformation models and ultimately provide insights that may enable improvement of the high temperature capabilities of disk superalloys.

2. Experimental methods

2.1. Creep sample and testing

A single crystal analog of the currently used commercial disk alloy ME3 was obtained from the GE research center. The composition of ME3 can be found below in Table 1.

After a heat treatment that resulted in a bimodal γ' precipitate distribution, an [001] oriented rectangular parallelepipeds with a 1:1:2.5 dimension ratio were extracted using electrical discharge machining (EDM). The sides of the sample were then polished to a 1200 fine grit using SiC polishing pads to remove the subsequent damage layer. A 414 MPa monotonic compression creep test was performed at 760 °C on an [001] oriented sample in order to promote re-order mediated precipitate shearing modes; specifically, microtwinning and isolated faulting. The test was performed using a MTS 810 Compression creep frame with two linear variable displacement transducers (LVDTs) to record plastic strain. Both tests were ended when about 0.5% plastic strain was reached and the specimens were quickly fan-cooled to room temperature in order to capture the deformation and microstructure present at the end of the test, as well as to minimize diffusional changes during cool-down.

2.2. Microscopy and chemical analysis

Post creep, the sample was again polished to a fine 1200 grit using SiC polishing paper and then followed by a 0.05 μm colloidal silica finish. To confirm that Microtwinning had occurred during the creep test, electron channeling contrast imaging (ECCI) was conducted using a FEI Sirion scanning electron microscope (SEM) while utilizing the backscatter detector at high accelerating voltages. ECCI allows for large areas to be imaged for improved statistical deformation analysis [23,24]. To validate the ECCI results, which found microtwinning to be the prominent deformation mode, transmission electron microscopy (TEM) samples were created using a FEI Helios Nanolab Dualbeam 600 focused ion beam (FIB). The FIB foils were further cleaned to remove ion damage using a Fischione Nanomill at 900eV. First, foils were extracted orthogonal to the compression axis to confirm the orientation of the sample and for STEM analysis. These foils were analyzed using low angle annular dark field (LAADF) detectors on an FEI Tecnai F20 STEM at 200 kV and again found evidence for microtwinning.

For atomic resolution HAADF imaging and high resolution EDX, [110] oriented FIB foils were extracted from the post compression creep samples to image the microtwins and superlattice stacking faults (SFFs) edge on. HAADF Imaging was conducted on a FEI probe-corrected Titan^3 80–300 kV STEM. While the EDX was performed on an image-corrected Titan^3 60–300 kV with Super-X detector technology utilizing the Bruker Esprit software. All quantified and summed EDX spectra were done using standard Cliff-Lorimer k factors and the Bruker Esprit 2™ software.

3. Results

3.1. Evidence of segregation along microtwins

To better understand if segregation occurs along microtwins, low magnification EDX maps were obtained from a γ' precipitate containing a microtwin that had sheared through its center. The results are shown below in Fig. 1. In the HAADF image and EDS maps, the microtwin is observed along a [10−1] direction such that the coherent (111) interfaces of the microtwin are observed "edge-on." In this case, the microtwin is about 15 nm in width.

Several noteworthy features can be seen in Fig. 1. The presence of tertiary γ particles can be seen in the Co and Cr elemental maps. Instead of these particles being present throughout the bulk of the precipitate there appears to be a denuded zone along the edge of the precipitate where they dissipated during the heat treatment and creep testing. The denuded zones have also been reported by Yardley et al. [22]. The elemental maps in Fig. 1 also indicate the presence of Co and Cr segregation along the microtwin; yet, the segregation does not appear to be uniform. In the region of the γ' precipitate where the tertiary γ particles are present, the twin has Co and Cr along the interface as well as inside the twin region, although the intensity of the Co and Cr signal inside the twin is not consistent.

In the region of the precipitate without the tertiary γ particles, chemical partitioning still occurs (although this is not obvious from Fig. 1, as the Co and Cr signatures are much less pronounced approaching the main γ'/γ interface). Confirmation of this feature is provided by the higher magnification EDX scan of Fig. 2 which was taken from the region indicated by the white box shown in the

Table 1
The elemental composition of ME3 in wt%.

Alloy	Ni	Co	Cr	Mo	W	Nb	Ta	Al	Ti	Hf	C	B	Zr
ME3	Bal.	20.6	13.0	3.8	2.1	0.9	2.4	3.5	3.4	0	0.05	0.03	0.03