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Creep deformation-induced antiphase boundaries in $L1₂$ -containing single-crystal cobalt-base superalloys

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Abstract

Creep-induced antiphase boundaries (APBs) in new Co-base single-crystal superalloys with coherent embedded $L1_{2}$ - γ' precipitates have been observed. APBs formed during single-crystal tensile creep tests performed at 900 °C under vacuum at stresses between 275 and 310 MPa. The alloys investigated contained 30–39 at.% Ni, which was added to the Co–Al–W ternary system to expand the $\gamma-\gamma'$ phase field and increase the γ -solvus. Transmission electron microscopy (TEM) using two-beam conditions with fundamental and superlattice reflections was performed for defect characterization. The Burgers vector b of dislocations associated with the APBs was determined to be of type $\mathbf{b} = a_0/2[011]$ and $a_0/2[01\bar{1}]$. The displacement vectors, **R**, of the APBs matched the dislocation Burgers vectors, with $\mathbf{R} = \mathbf{b} = a_0/2[011]$. APBs were observed in nearly every precipitate beyond 0.5% creep strain for the compositions investigated. The implications for high-temperature properties are discussed.

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1. Introduction

The recent discovery of a ternary $L1_2$ phase in the Co–Al–W ternary system by Sato et al. [\[1\]](#page--1-0) has enabled new pathways for the development of high-strength, hightemperature materials for gas turbine applications. The L1₂ phase (γ') is in equilibrium with the solid-solution face-centered cubic (fcc) (γ) phase in the Co-rich corner of the Co–Al–W ternary phase diagram. Compositions lying in the two-phase field can possess a $\gamma + \gamma'$ microstructure, which is similar to Ni-base superalloys $[1,2]$. Ni-base superalloys are used in critical components in land-based and aircraft gas turbine applications as their high-temperature strength is largely unparalleled by other material systems [\[3\]](#page--1-0).

Creep is one of the primary deformation modes in these materials, as the turbine blades operate near 90% of their melting temperature $\lceil 3 \rceil$. The operating temperature of Ni-base superalloys is ultimately limited by the melting temperature, and there exists strong motivation to develop new alloy compositions which can further increase the operating temperature, and thus efficiency, of gas turbine engines. Pollock et al. [\[2\]](#page--1-0) have shown that Co-base alloys exhibit a higher melting temperature than Ni-base alloys and could therefore provide a new pathway to alloys possessing a higher operating temperature than Ni-base superalloys. Additionally it has been shown that Co-base superalloys exhibit superior single-crystal solidification characteristics compared to Ni-base alloys [\[4\],](#page--1-0) and compressive flow stresses comparable to Ni-base alloys above 900 °C [\[5\]](#page--1-0).

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The γ' solvus and solidus of various Co- and CoNi-base alloys have been investigated by numerous authors [\[1,2,4,6–8\]](#page--1-0). Ni additions have been reported to increase the γ' -solvus monotonically in Co-base alloys and thus serve as a way to tailor the γ' -solvus temperature for specific applications. The research reported here focuses on Ni-containing Co-base alloys.

Recently, single-crystal tensile creep tests have been conducted on Co- and CoNi-base alloys [\[6,9\]](#page--1-0). The Co- and CoNi-base alloys exhibit creep resistance comparable to first-generation Ni-base superalloys for alloys with a high volume fraction (\sim 70%) of cuboidal-shaped (\sim 0.5 µm in edge length) γ' precipitates. While for Ni-base superalloys, high-temperature deformation mechanisms have been extensively studied, investigations of high-temperature deformation mechanisms in Co-based superalloys are limited.

During mechanical testing at elevated temperatures, it has been reported [\[5,10,11\]](#page--1-0) that γ matrix dislocations of type $a_0/2\langle 110 \rangle$ glide through the γ channels during deformation. At greater strains, Suzuki et al. [\[5\]](#page--1-0) observed shearing of the γ' precipitates by glide of $a_0\langle 110 \rangle$ superdislocations on octahedral and cubic planes, and by motion of $a_0/3\langle 112 \rangle$ dislocations forming stacking faults across the precipitates. The authors also observed a high density of superlattice intrinsic stacking faults (SISFs) in the γ' precipitates in a Ta-containing Co-base alloy in compres-sive yield strength tests above 800 °C. Bauer et al. [\[10\]](#page--1-0) and Pyczak et al. [\[11\]](#page--1-0) also observed $a_0/2(101)$ dislocations in the γ channels after compressive deformation of polycrystalline Co-based alloys, as well as stacking faults.

Numerous creep deformation mechanisms in Ni-base superalloys have been identified by several authors at high temperatures (>850 °C) [\[3\]](#page--1-0). During primary and steadystate creep, dislocations have been observed to cross-slip and climb in the γ matrix channels [\[7,13,14\]](#page--1-0). Furthermore, as the stresses in the undeformed precipitates increase, dislocations have been observed to shear the γ' precipitates by $a_0/2\langle 110 \rangle$ matrix dislocations coupled by an antiphase boundary between them $[14–17]$. To the authors' knowledge, single $a_0/2\langle 110 \rangle$ dislocations shearing γ' precipitates have not been observed as a significant deformation mechanism in Ni- or Co-base superalloys containing a γ - γ' microstructure. In the present study, creep deformation mechanisms of CoNi alloys were investigated via transmission electron microscopy (TEM). Single $a_0/2\langle 110 \rangle$ dislocations were frequently observed to shear precipitates, leaving behind antiphase boundaries (APBs).

The present study focuses primarily on creep deformation-induced antiphase boundaries in CoNi-base alloys. A second paper will discuss the evolution of dislocation substructure present as a result of creep deformation in greater detail.

2. Experimental procedures

The compositions of the two alloys investigated are listed in [Table 1.](#page--1-0) Cobalt was partially substituted with Ni to expand the γ' phase field [\[18\]](#page--1-0), and Ta and Cr were added to increase oxidation resistance, creep resistance and γ' -solvus temperature [\[2\]](#page--1-0). The most significant change in composition between alloys was the substitution of Ni for Co, with one alloy containing equal levels of Ni and Co. Single-crystal bars of \sim 150 mm in length were cast by the liquid metal cooling process [\[19–21\],](#page--1-0) with a withdrawal rate of 142 mm s^{-1} , which is similar to the process used for Ni-base superalloys. The alloys investigated are from the same castings as in a previous study [\[9\]](#page--1-0). A homogenization heat treatment at 1215 \degree C for 12 h was utilized to assuage segregation during solidification, and aging was performed at 950 °C for 100 h to coarsen the γ' precipitates. Single-crystal tensile button-head creep specimens with a nominal gage diameter of 3.05 mm and a length of 25.4 mm were machined from the fully heat-treated bars with orientations near [001], as shown in [Fig. 1c](#page--1-0). The single-crystal bar axes were aligned $\sim 6.2^{\circ}$ and $\sim 23.0^{\circ}$ away from the [001] crystallographic axis for the CoNi-A and CoNi-C alloys, respectively. The crystal orientations were measured by electron backscatter diffraction and transmission electron microscopy (TEM). Single-crystal tensile creep tests were performed in a vacuum creep furnace at 900 °C under varying stresses in order to obtain a minimum creep rate near 10^{-8} s⁻¹, as shown in [Table 1](#page--1-0). The rupture lives of these alloys have recently been reported by Titus et al. [\[9\].](#page--1-0)

A FEI T20 transmission electron microscope with a dual-axis large-angle tilt stage was used to investigate creep deformation mechanisms. TEM specimens were extracted from the deformed gage length and from the undeformed region of the creep specimens—see dashed arrow in [Fig. 1](#page--1-0)c. Specimens were cut transversely and longitudinally to the tensile axis by wire electrical discharge machining. The discs with a diameter of \sim 3 mm were then mechanically ground and polished to a thickness of 0.12 mm before final thinning using a Fischione twin-jet electropolisher in a solution of 92.5% methanol and 7.5% perchloric acid by volume. The electrolyte was cooled with liquid nitrogen to a temperature of -50 to -40 °C, and polishing was performed at 16–18 V and 20–28 mA. Some samples were additionally polished using a Fischione Precision Ion Polisher at 1 keV with upper and lower beams set to 4° in order to remove oxidation and other contamination.

3. Results

3.1. As-aged microstructure

[Fig. 1a](#page--1-0) presents a centered dark-field (CDF) image of the as-aged microstructure of the CoNi-A alloy. The CoNi-A alloy exhibits a cuboidal precipitate morphology with a γ' area fraction of \sim 70–80%, a mean precipitate size of \sim 410 nm along the cube edge, and a mean channel width of 46 nm. The CoNi-C alloy contains mostly spherical precipitates due to a lower misfit between the γ' and γ phases [\[22\].](#page--1-0) The as-aged microstructure of CoNi-C is shown in

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