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Elemental segregation to antiphase boundaries in a crept CoNi-based single crystal superalloy

Surendra Kumar Makineni [a,*], Malte Lenz [b], Steffen Neumeier [c], Erdmann Spiecker [b], Dierk Raabe [a,*], Baptiste Gault [a,*]

[a] Max-Planck-Institut für Eisenforschung GmbH, 40237 Düsseldorf, Germany
[b] Institute of Micro- and Nanostructure Research & Center for Nanoanalysis and Electron Microscopy (CENEM), Friedrich-Alexander-Universität Erlangen-Nürnberg, Cauerstraße 6, 91058 Erlangen, Germany
[c] Institute I: General Materials Properties, Friedrich-Alexander-Universität Erlangen-Nürnberg, Martensstr. 5, 91058 Erlangen, Germany

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ABSTRACT

We report on the full three-dimensional compositional partitioning among the features of a planar imperfection comprising a superlattice intrinsic stacking fault (SISF) with its leading and trailing partials, as well as the antiphase boundary (APB) in the wake of the trailing partial formed in the $L1_2$-ordered γ′ phase of a CoNi-based single crystal superalloy. The partial dislocations and the APB are found to be Cr/Co rich relative to the surrounding γ′ and richer in W/Ta/Ti compared to the γ matrix phase. Solute diffusion mechanisms are derived from the compositional gradients in the vicinity of the imperfection.

Antiphase boundaries (APBs) are among the most common planar crystalline defects in γ/γ′ based superalloys [1] and are related to an anomalous positive flow stress behavior at high temperatures [2,3]. The γ matrix is a face-centered-cubic (fcc) disordered solid solution in which the $L1_2$-ordered γ′ phase precipitates are coherently embedded. In Ni-based alloys, APBs are present between two strongly coupled a/2 ⟨110⟩ superpartial dislocations gliding on {111} planes through γ′ during creep deformation at temperatures above 850 °C [4–6]. Their energy determines both, the threshold stress for γ′ cutting and the spacing among the superpartials. Hence, the APB energy is a crucial quantity that governs the alloy's high-temperature mechanical behavior [1,7–10].

The γ/γ′ microstructure was also discovered recently in Co- and CoNi-based alloys [11–14]. The CoNi-based γ/γ′ alloys [15–17] were shown to deform by the formation of APBs, however, with an a/2 ⟨112⟩ shear mechanism [17]. Additionally, it was shown that the formation of SISFs and APBs involves local reordering and chemical segregation driven by the reduction of the SISF [18] and APB energies [17] according to Gibbs. The APBs were shown to be enriched in Co/Cr, both γ stabilizers, relative to the surrounding γ′ lattice [17]. Similarly, in Ni-based superalloys, a few experimental reports show that γ stabilizing solutes segregate to the APBs [19–22]. This phenomenon was regarded as "wetting" of APBs [23,24] with the disordered γ phase, which was first reported in a Fe—Al based alloy by Swan et al. [25] and Allen et al. [26]. In CoNi-based superalloys, by utilizing an in-situ heating approach [17], it was also shown that the APB composition approaches that of the corresponding disordered γ phase, resulting in fragmentation of the γ′ precipitate. The growth and coarsening of APBs involve solute diffusion and, therefore, their respective rates depend on the diffusivities of the various solutes. However, quantitative measurements of the local diffusion and associated mechanisms occurring during high-temperature deformation have not been reported yet. Unveiling and explaining these mechanisms will help the design of novel alloys.

Until now, local chemical compositions of such types of microstructures were analyzed mainly by means of energy dispersive X-ray spectroscopy (EDXS) in transmission electron microscopes (TEM) operated in STEM mode [18,27,28]. When using STEM imaging, projection effects cannot be avoided and the sensitivity and quantification, especially for minor alloying elements can be limited. Here, we quantify the compositional partitioning among all of the constituting features of a planar imperfection comprising a SISF, the two bounding partial dislocations and the APB in three-dimensions with the aid of correlative atom probe tomography (APT) and electron microscopy. The imperfection was formed upon creep in the ordered γ′ phase of a new CoNi-based single crystal superalloy. The compositional gradients derived

* Corresponding authors.
E-mail addresses: sk.makineni@mpie.de (S.K. Makineni), d.raabe@mpie.de (D. Raabe), b.gault@mpie.de (B. Gault).

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from our analysis allow us to predict the diffusive processes occurring during creep.

A CoNi-based single crystal alloy, named ERBOCo-1, with the composition Co-32Ni-8Al-5 W-6Cr-2.5Ti-1.5Ta-0.1Hf-0.4Si was prepared by the Bridgman process. It was subject to a multi-step heat treatment, viz. 1280 °C/8 h + 1050 °C/5 h + 900 °C/16 h to achieve a uniform γ/γ′ microstructure. The γ′ volume fraction in the alloy is measured to be ~ 61% which was evaluated by image analysis using ImageJ software [29]. Tensile specimens were cut and crept at 850 °C for 380 h at an applied stress of 400 MPa acting along the [001] direction up to 4.6% plastic strain. Cross-section samples with their plane normal close to the [110] direction were cut and mechanically polished for further microstructural characterization.

The creep deformed microstructure was observed by controlled electron channeling contrast imaging (cECCI) revealing the location of crystal defects [30]. A Zeiss Merlin scanning electron microscope (Carl Zeiss SMT, AG, Germany) equipped with a Gemini-type field emission gun electron column was used to perform electron backscattered diffraction (EBSD) mapping followed by cECCI in the same region-of-interest. The operating accelerating voltage was 30 kV with a probe current 4 nA and a working distance of 6 mm during imaging. cECCI exploits information from EBSD orientation determination, combined with simulation of electron channeling pattern (ECP) for different sample tilts, which we obtained from the software TOCA (Tools for Orientation Determination and Crystallographic Analysis) [31]. TOCA enables to determine the accurate sample stage settings to orient the specimen so as to obtain backscattered electron imaging in strong two-beam conditions [32].

Atom probe specimens for correlative investigation by TEM and APT were fabricated using a dual beam SEM/focused-ion-beam (FIB) instrument (FEI Helios Nanolab 600) following the protocol described in reference [33]. Regions containing the defects of interest were extracted from the bulk and subsequently sliced and welded using in-situ Pt-deposition on the end of the arms of an electropolished halved TEM Mo-grid inserted in a dedicated holder [34]. Slices were then sharpened by FIB milling at 30 kV followed by a final cleaning procedure at 2 kV and 16 pA to remove severely damaged regions. Transmission electron microscopy (TEM) on individual APT specimens was carried out in a Phillips CM-20 operated at 200 kV. A Cameca Instrument Inc. LEAP™ 5000XR, equipped with a reflectron for enhanced mass-resolving capability, was used for compositional analysis. APT data was acquired in laser pulsing mode at a repetition rate of 125 kHz and a pulse energy of 50 pJ. The specimen's base temperature was maintained at 40 K and the target detection rate was set to be 5 ions detected every 1000 pulses. The software package IVAS 3.8.0 was used for data reconstruction and analysis.

Fig. 1 shows an overview cECC image of the tensile crept CoNi-based superalloy along the [110] viewing direction. The micrograph was acquired in a two-beam condition with g = $(00\overline{2})$, where g is the diffraction vector. A clear atomic density contrast reveals rafted γ′ along the tensile loading direction embedded in darker γ channels. The bright contrast in the rafted γ′ corresponds to typical stacking faults (SFs) with bright lines formed due to the intersection of SF planes and the sample surface, as visible in the inset (a). Near the γ/γ′ interface region, a feature appears with a bright contrast extending away from the stacking fault shown in inset (b) and bows upwards towards the interface. This feature resembles the observation of APBs by TEM reported by Eggeler et al. [17], indicating the presence of such defects in the present crept alloy. In the subsequent analysis, we show that these features are indeed APBs. The curvature of the feature occurs due to the migration of the APB habit plane from {111} to {100}, which results in a reduction in the APB area and minimizes its specific energy [35,36].

An APT specimen was prepared from the location delimited by the dashed golden line in Fig. 1. Prior to APT probing, the presence of planar imperfections was confirmed by TEM. Figs. 2(a–b) respectively show a brightfield (BF) and a darkfield (DF) image taken from a superlattice $L1_2$ spot along the [110] zone axis of the APT specimen. The BF image shows clear contrast of straight dark lines resembling SFs. The absence of fringe contrast indicates that these faults are observed nearly edge-on. The DF image taken with a (010) superlattice reflection shows the γ′ phase in bright contrast. Regions where the $L1_2$ ordering is not maintained appear dark. This holds true for the γ phase, APBs and the damaged outer shell of the APT tip. Subsequently, a low energy cleaning of the specimen in the FIB (5 kV, lower current of 8 pA) was carried out to remove the e-beam contamination from the surface prior to APT analysis. Fig. 2(c) shows the distribution of Cr (pink dots) and Co

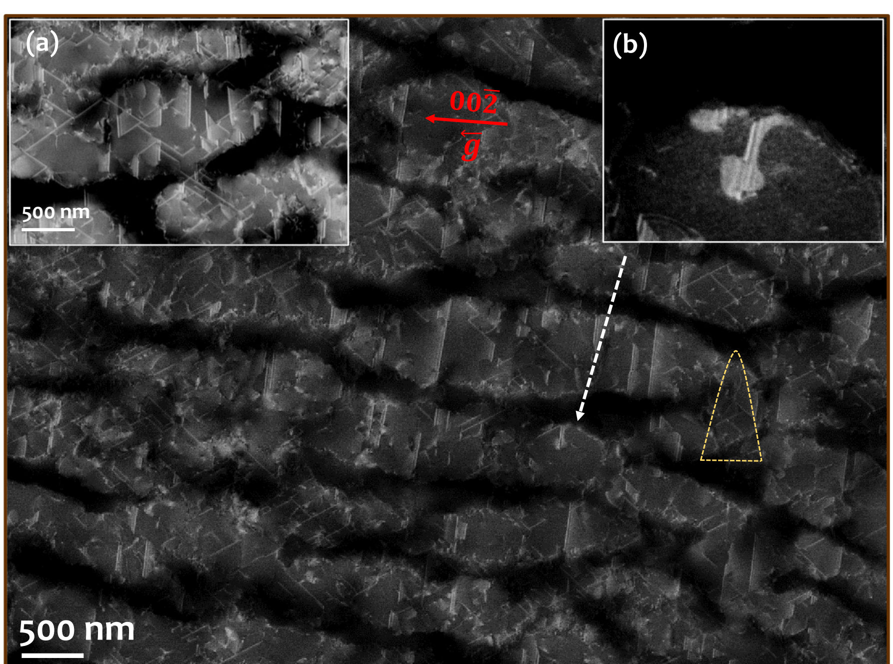

Fig. 1. An overview cECC image for the crept CoNi-based superalloy in two-beam diffraction condition (g = $(00\overline{2})$) showing a high number density of stacking faults (inset (a)) in the γ′ phase; inset (b) shows the bright contrast bowing upwards towards the γ/γ′ interface corresponding to an APB. (APB: Antiphase Boundary).