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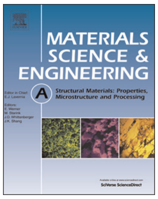

The potential link between high angle grain boundary morphology and grain boundary deformation in a nickel-based superalloy

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ABSTRACT

Focused ion beam (FIB) based serial sectioning was utilized to characterize the morphology of two high angle grain boundaries (HAGB) in a nickel based superalloy, one that experienced grain boundary sliding (GBS) and the other experienced strain accumulation, during elevated temperature constant stress loading conditions. A custom script was utilized to serial section and collect ion-induced secondary electron images from the FIB-SEM system. The MATLAB based MIPAR^{TM} software was utilized to align, segment and reconstruct 3D volumes from the sectioned images. Analysis of the 3D data indicates that the HAGB that exhibited GBS had microscale curvature that was planar in nature, and local serrations on the order of ± 150 nm. In contrast, the HAGB that exhibited strain accumulation was not planar and had local serrations an order of magnitude greater than the other grain boundary. It is hypothesized that the serrations and the local grain boundary network are key factors in determining which grain boundaries experience GBS during creep deformation.

1. Introduction

The creep deformation in polycrystalline nickel-based superalloys is a heterogeneous process, the primary mechanism that controls this deformation is the interaction of dislocations with the γ' precipitate distribution, while ultimate creep life is dependent on damage accumulation near grain boundaries [1]. Predicting the behavior of polycrystalline materials by simulation techniques [2,3] is dependent on producing relevant experimental data at appropriate time and length-scales to validate the predicted behavior. Recent experiments on nickel-based superalloys that quantify local deformation at grain boundaries in two-dimensions (2D) [4] and three-dimensions (3D) [5] have confirmed that experiments that capture only the 2D deformation fields can provide statistical insight but are insufficient to fully validate 3D deformation models. The deformation behavior of grain boundaries is a function of the grain boundary misorientation, the orientation of the grain boundary with respect to the loading direction, the morphology of the grain boundary, and the local stress state which can be very different from the far-field boundary conditions [5]. These last three parameters are difficult to characterize with scanning electron microscopy (SEM) or transmission electron microscopy (TEM) because these techniques only provide a single projection of the grain boundary structure. Therefore, complete characterization of grain boundaries requires collection of 3D data through a technique such as serial sectioning. Full field strain mapping and grain boundary sliding (GBS) measurements from discrete offsets in grid markers indicate that some grain boundaries in nickel-based superalloys experience strain accumulation while other grain boundaries exhibit GBS when the material is subjected to creep conditions [6]. No conclusive evidence could be found that either of these types of behaviors could be predicted based on grain boundary misorientation or the orientation of boundary projections in SEM images with respect to the tensile axis [7]. This led to the hypothesis that grain boundary structure and sub-surface grain neighborhood also play a pivotal role in dictating the local deformation response at grain boundaries. In this study, two different high angle grain boundaries (HAGB) were characterized by focused ion beam (FIB) serial sectioning: one exhibited grain boundary sliding, and the other exhibited strain accumulation during creep conditions.

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2. Experimental

Creep testing: Constant load, elevated temperature testing was conducted in an SEM under the following conditions: 700 °C and 1100 MPa. Specimens were polished and initial grain boundary locations were quantified prior to testing using electron backscatter diffraction (EBSD). A speckle pattern was subsequently applied to the samples to enable digital image correlation analysis of surface deformation fields. After testing, high resolution images were acquired and analyzed to quantify the contribution of grain boundary sliding (GBS) to plastic strain accommodation. Details of the sample preparation and experimental results are presented elsewhere [6,8]. From the many boundaries identified, two high angle grain boundaries were selected for 3D characterization by serial sectioning. One exhibited strain accumulation with no GBS and the other exhibited GBS but no strain accumulation beyond average macroscopic strain state, as shown in Fig. 1a and b respectively.

Sample preparation: The 3D structure of the grain boundaries shown in Fig. 1 was investigated through serial sectioning using an FEI Nova 600 SEM-FIB. Micrometer-scale volumes of material were extracted and each placed on an molybdenum OmniProbe grid using an OmniProbe in situ micromanipulation system in a manner similar to thin foil preparation [9], Fig. 2 shows one of the volumes prepared just prior to lift-out from the bulk sample. This extraction method was necessary in order to minimize the negative effects due to redeposition during serial sectioning. First a platinum cap was deposited over the area of interest, and then trenches were milled along three faces of the volume. Once trenches were cut, the sample was under-cut on two sides to create a cantilever beam of material. The volume was attached to the OmniProbe in situ micromanipulation needle using platinum deposition and then the sample connection to the bulk material was severed. Next, a molybdenum OmniProbe Grid was attached with silver painted to an SEM stub and inserted flat into the SEM-FIB chamber such that when imaged with the electron source, the grid looked like what is presented in Fig. 3. A molybdenum grid was used because it mills more slowly than conventional copper grids. The sample was then attached to the end of one of the grid fingers using platinum deposition.

A custom script utilizing FEI Runscript software was used to automate the serial sectioning process, and consisted of cross-section milling, collection of ion-induced secondary electron (ISE) images, and repositioning of the stage between the two conditions [10]. The FIB column was set to an accelerating voltage of 30 kV, and the current was switched between 2.8 nA for cross-section milling and 0.47 nA for ISE imaging. The sample stub was mounted on a 45° pre-tilted specimen holder in order to enable the script to perform both cross-section milling and normal-incidence imaging of the cross-section surface, which required both rotation and tilting of the microscope stage, as illustrated in Fig. 3. A pair of circular fiducial marks were used for automated alignment purposes. ISE images were used for this study because ISE images displayed significant channeling contrast that accentuated the crystallographic orientations of the grain structures, making it possible to construct automated segmentation processes that define the grain boundary morphology.

Two grain boundary volumes were extracted for serial section characterization, in both cases, the section thickness was approximately 50 nm. The sample containing the HAGB that exhibited GBS was reconstructed from 116 slices with each image having in-plane resolution of 223 pixels/ μm, resulting in a 406 μm^3 volume. This leads to highly anisotropic voxels $4.48 \times 4.48 \times 50$ nm in size. The sample containing the HAGB that exhibited strain accumulation was approximately 2080 μm^3 in size and was reconstructed from 110 slices with each image having in-plane resolution of 24 pixels/ μm. This leads to slightly anisotropic voxels $41.6 \times 41.6 \times 50$ nm in size. This larger volume contained several grain boundaries of interest for reconstruction. In this case, normal-incidence images did not provide adequate channeling

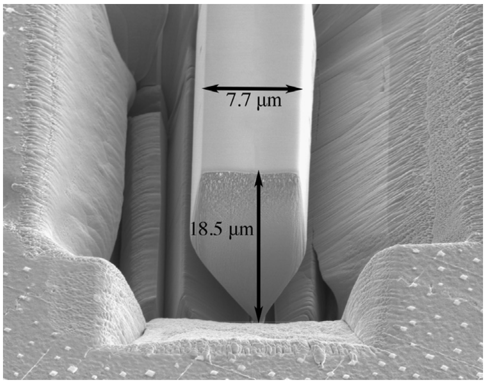

Fig. 2. Secondary electron micrograph showing the platinum capped serial sectioning sample ready for lift-off from the bulk material.

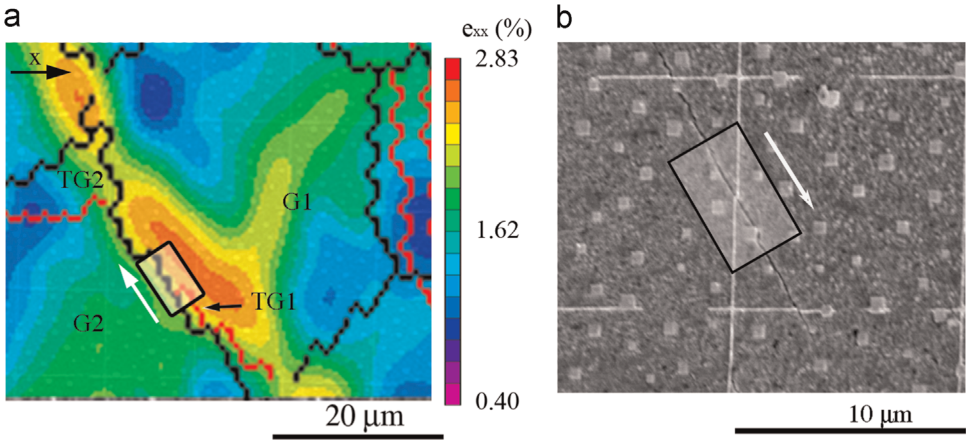

Fig. 1. (a) A DIC strain map with overlaid grain boundaries determined by EBSD prior to deformation (the black lines are HAGB and red lines are annealing twins) shows that the HAGB between grains 1 and grain 2 (G1–G2) experienced strain accumulation, also visible are annealing twins in G1 and G2 (TG1 and TG2 respectively). The (b) secondary electron SEM image highlights the HAGB that experienced GBS as measured via the offset in hafnium oxide grid lines (white) deposited prior to deformation. Rectangular boxes indicate volumes selected for serial sectioning, and white arrows indicate the sectioning direction. (For interpretation of the references to color in this figure caption, the reader is referred to the web version of this paper.)