Materialia 000 (2018) 1–5

Contents lists available at ScienceDirect

Materialia

journal homepage: www.elsevier.com/locate/mtla

Full Length Article

Stress-driven grain boundary movement during nanoindentation in tungsten at room temperature

Farhan Javaid, Karsten Durst*

Materials Science Department, Physical Metallurgy Division, Technische Universität Darmstadt, Darmstadt, Germany

ARTICLE INFO

Keywords:
Tungsten
Grain boundary movement
Dislocations
Nanoindentation
EBSD
ECCI

ABSTRACT

Nanoindentations were performed in the vicinity of grain boundaries (GBs) in polycrystalline tungsten at room temperature, observing in some cases a secondary pop-in (known as GB pop-in) in the load–displacement curve. The dislocation microstructure in the plastic zone of the residual impression was analysed using sequential polishing, electron channelling contrast imaging (ECCI) and electron backscatter diffraction (EBSD) analysis on and below the surface. For some indentations, the interaction of the dislocations within the plastic zone and the GB leads to a localized GB movement on or below the surface. The occurrence and magnitude of GB movement are found to be strongly influenced by misorientation between the adjacent grains, the orientation of the indenter, as well as the applied load and the distance to the GB. The results show that the localized GB movement under an inhomogeneous stress field at room temperature is a possible deformation mechanism for tungsten.

1. Introduction

Plastic deformation of metal is typically carried out by dislocation motion. At low temperatures, grain boundaries (GBs) are traditionally considered to act as stationary obstacles to dislocation motion; the classical Hall–Petch strengthening mechanism is directly based on these effects [1]. However, during plastic deformation of nanocrystalline face-centred cubic (FCC) metals, GB motion has been frequently observed even at the room temperature [2–5], and this GB motion is associated with a softening of the deformed material instead of Hall–Petch strengthening [6,7]. Interactions between GBs and dislocations are important for understanding the stress-driven grain boundary migration at low temperature that characterizes such softening [8,9]. GB–dislocation interactions may include dislocation pile-up at the GBs, the absorption of dislocations in GBs, the emission of dislocations from GBs, or the absorption and direct re-emission of dislocations from one grain to another (with or without creating a residual dislocation at GB) [10–16]. These GB–dislocation interactions are relatively complex and still not understood in great details [17].

Nanoindentation is frequently used in conjunction with advanced microscopy techniques like transmission electron microscopy (TEM) and electron backscatter diffraction (EBSD) for probing small-scale mechanical properties [18–21] and local GB–dislocation interactions [22–24]. Minor et al. [2] reported GB mobility at room temperature during in situ nanoindentation experiments in ultrafine-grain aluminium (Al) thin films. Soer et al. [3] also confirmed extensive GB movement for ultrafine-grain Al thin films at room temperature.

In contrast to FCC metals like Al, body-centred cubic (BCC) metals exhibit larger resistance to intergranular slip transmission (a way for dislocations to travel despite GB non-movement) and nanoindentation experiments conducted close to the GBs of BCC materials reveal a distinct effect referred to as “GB pop-in”. GB pop-in appears as a strain burst on the nanoindentation load–displacement curve, and is believed to occur due to the dislocation transmission across the GB [22–24]. However, there is no direct evidence of the dislocation microstructure in the vicinity of GB before or after the GB pop-in event. Moreover, no localized GB movement has been reported for BCC metals at room temperature until now. Here it is shown that the interaction between dislocations generated in the plastic zone of an indentation can indeed lead to local GB movement below the surface in a BCC material at room temperature.

2. Methods

High-purity (99.9%) polycrystalline coarse-grain tungsten (W) was used as a model BCC material. After grinding and polishing the specimens (via SiC abrasive papers till 4000 grit), electropolishing was conducted for 20–30 s (in 2% NaOH solution at 8 V) to reveal the GBs and to remove any deformation layer from previous polishing steps. Berkovich nanoindentation experiments were performed in the vicinity of various straight grain boundaries with a G200 nanoindenter (Keysight Technologies, USA) using a continuous stiffness measurement method.

To study the GB–dislocation interactions, several cross-sections were obtained through the residual impressions with the surrounding plastic zones using a sequential polishing technique [25–26] and focused ion beam (FIB) milling. The sequential polishing was performed with a

* Corresponding author.
E-mail address: k.durst@phm.tu-darmstadt.de (K. Durst).

https://doi.org/10.1016/j.mtla.2018.04.002
Received 23 March 2018; Received in revised form 12 April 2018; Accepted 15 April 2018
Available online xxx

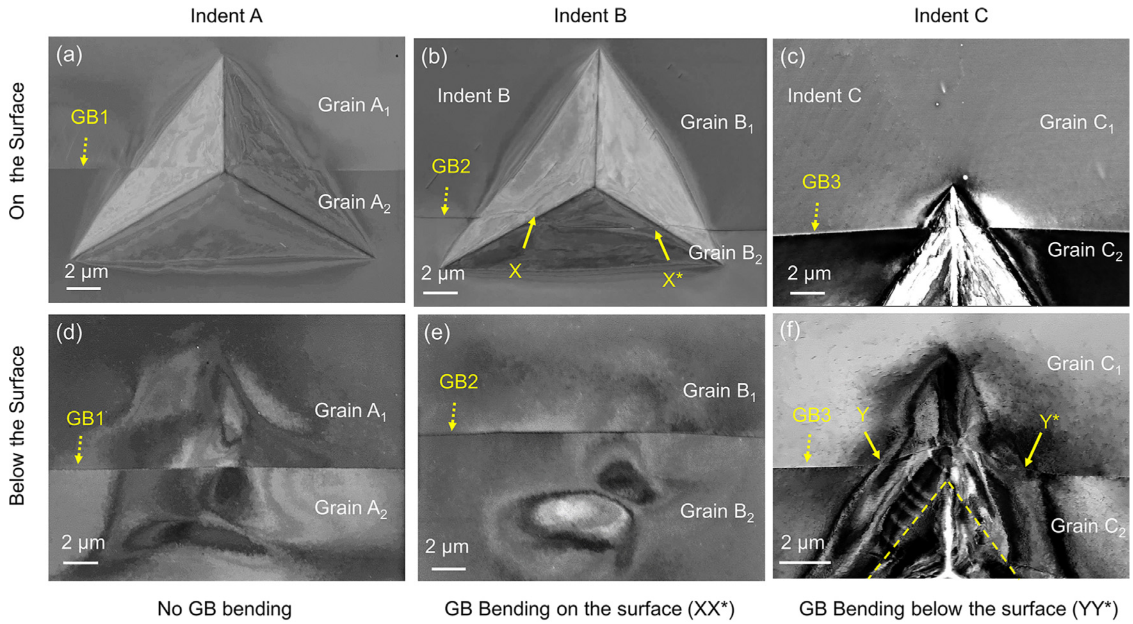

Fig. 1. SEM and ECCI images of representative 2 μm deep Berkovich indentations, (a–c) on the surface, (d–f) below the surface obtained from sequential polishing.

vibration polishing machine (Jean-Wirtz GmbH, Düsseldorf, Germany) and 0.04 µm colloidal silica suspension (OP-S, Struers A/S, Ballerup, Denmark). The material removal rate was determined by measuring the residual depths of reference indentations with a laser scanning microscopy (LEXT 4000, Olympus, Tokyo, Japan). The plastic zone below the polished indentations was revealed using ECCI in a TESCAN MIRA3 SEM equipped with four quadrant Deben BSE detector (East Tilbury, UK).

Cross-sections of the indentations were performed by FIB milling, using a dual beam electron microscope (Helios NanoLab 600i, FEI). For coarse and fine milling, ionic currents of approximately 20 nA and 10 pA at a voltage of 30 kV were used, respectively. Before FIB milling, the indent was covered with a protective Pt-layer to avoid Ga^+ damage during sectioning. EBSD measurements on the FIB cross-sections were performed with a Nordlys EBSD detector (Oxford Instruments HKL, UK). A step size of 100 nm was used for all measurements. The misorientation angle maps were analysed using the HKL Channel-5 software from Oxford Instruments HKL, UK.

For better statistics on the localized GB movement, 30 Berkovich indentation tests were performed in the vicinity of different GBs (having a misorientation of 7°–60° between the adjacent grains). Out of these 30 indentations, only 14 led to localized GB movement, with a misorientation of 20°–60° between the adjacent grains.

3. Results and discussion

Fig. 1 shows characteristic images of three different types of GB–dislocation interactions on and below the Berkovich indentations for three different GBs (GB1, GB2 and GB3 indicated by dotted arrowheads). Instead of following the expected straight line, a significant local GB curvature was observed on and below the surface (indicated by the solid arrowheads in Fig. 1b and f).This effect is most pronounced in Fig. 1f, in which a clear GB movement below the surface becomes visible.

From a careful analysis of the SEM and ECCI images in Fig. 1, we make the following three remarks:

I. For GB1, the GB line is not influenced by the indentation (indent A). It follows the expected straight line on and below the indentations (Fig. 1a and d).

II. For GB2, a significant local GB curvature (towards grain B_2) occurs inside the residual impression of indent B (indicated by XX* in Fig. 1b). However, below the indentation, the GB becomes straight again (Fig. 1e).

III. For GB3, no localized GB movement is visible inside the indent C (Fig. 1c). However, sequential polishing revealed a clear GB curvature (towards grain C_1) below the indentation in GB3 (indicated by YY* in Fig. 1f).

For indent A (Fig. 1a), the indent extends well beyond the GB, but no localized GB movement is observed in GB1 either on or below the surface. Indent C also extends well beyond the GB, but even though the residual impression of indent C (as indicated by the dotted lines in Fig. 1f) is not touching the GB, a strong GB movement is found below the surface. This evidence strongly suggests that in the YY* region (Fig. 1f), the interaction between the dislocations is generated in the plastic zone of the indent with the GB, leading to the formation of a curved GB, which is moved from grain C_2 towards C_1.

However, for indent B (Fig. 1b), the indenter is touching the grain boundary and the displaced volume of the indenter coupled with the dislocation activity may cause the localized GB movement. Fig. 1(a)–(c) also suggests that the Berkovich indenter orientation (with respect to GB) can also influence the local GB movement. Local GB movement inside the residual impression was only observed when one side of the Berkovich indenter was facing towards the GB. On the other hand, if the Berkovich tip was facing towards the GB, no GB movement inside the residual impression was found on the surface, but significant GB movement occurred below the surface. This evidence also suggests that both GB character and indenter orientation play a vital role in determining whether the GB movement will occur or not.

To check the reproducibility of the local GB movement under similar conditions, GB3 is chosen as a reference GB. Along with the 2 µm deep indentation (Fig. 1c), 1 µm depth indentations at various distance from the GB3 are performed in grain C_2. As expected, no GB movement was found on the surface (not shown). However, below the indentations (Fig. 2), local GB movement is found again. This local GB movement depends on the applied load as well as on the distance of the indent to the GB.

Fig. 2 shows ECCI images (obtained after removing ~300 nm of material from the surface of the specimen) of the exemplary 2 µm and 1 µm