Local analysis on dislocation structure and hardening during grain boundary pop-ins in tungsten

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ABSTRACT

In the present work, dislocation structures at the grain boundary (GB) pop-in events were studied around and below the Berkovich indentations using electron channeling contrast imaging (ECCI) in polycrystalline tungsten. The GB pop-in events were identified as displacement bursts in the load–displacement curve, and the indentation experiments were stopped just after the occurrence of the displacement burst. A significant hardness increase was observed before the GB pop-in event. The careful ECCI analysis on the sequential polished cross sections clearly shows the dislocation pile-up in the vicinity of the GB along with transmitted dislocations in the adjacent grain. The transmitted dislocations were also found to be influenced by the indenter orientation with respect to the GB.

Introduction

The mechanical behavior of polycrystalline materials is strongly influenced by grain boundaries (GBs) and their interactions with dislocations. For example, the classical Hall–Petch equation [1], which describes the relationship between grain size and yield strength of the material, has a prodigious impact on the modern structural materials. The resistance to slip transfer of GBs depends on the GB character [2–4]; however, often only an average hardening contribution is considered for a given material and microstructure [5]. Bayerschen et al. [2] have reported various possible interactions between dislocations and individual GB, starting from dislocations pile-up at GB

followed by different transmission mechanisms. Transmission of the dislocations requires thereby the activation of a dislocation source in the adjacent grain. Moreover, dislocations can be dissociated into the GB, which can result in zero dislocation emission in the adjacent grain. Furthermore, the re-emission of these absorbed dislocations in the adjacent grain is also possible with or without leaving the residual dislocations into the GB.

Nanoindentation has been frequently used to study the local-scale mechanical properties of materials [6–11] and GB-dislocations interactions [12–17]. The recent advances in nanoindentation technique allow performing the indentation experiments far away or close to the GB and capture different event during

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indentation from the load–displacement (LD) curve. For example, most crystalline materials show a displacement jump in the LD curve known as “pop-in,” depicting the elastic to a plastic transition of material [18–21]. If the indentations are performed near to a GB, along with this first pop-in, a second characteristic displacement excursion on the LD curve can also be seen at some higher loads, which is known as “GB pop-in” [12–15]. Interestingly, the GB pop-ins have been observed only in body-centered (BCC) materials and are generally believed to occur because of the dislocations transmission across the GB [5, 12–15]. The slip transfer in face-centered cubic (FCC) metals is considerably easier as compared to BCC metals due to the much lower Hall–Petch constants [12]. Therefore, the resistance to the slip transmission across the GB is expected to be more prominent in BCC metals as compared to FCC metals, which could be a possible reason why the GB pop-in phenomenon is not reported for FCC materials.

Wo and Ngan [22] conducted indentation experiments near the various GBs in Ni₃Al and found that the slip transfer across the GB depends on the local misorientation factor (m'). However, they did not observe any increase in hardness for the indentations conducted in the vicinity of the GB. Soer et al. [13] performed the indentations close to the high-angle GB in Fe-14%Si bicrystal and observed GB pop-in events in the LD curve. Though, they reported no appreciable long-range grain boundary hardening GB, which raises the question of whether the GBs contribute to the hardening effect in the material or not? Wang and Ngan [12] reported the absence of the increase in hardness possibly arising from the experimental errors. They also observed a strong dependency of the slip transfer across GBs on the misorientation factor and confirmed the occurrence of GB pop-in events in Niobium. They introduced a new protocol to predict whether the GB pop-in will occur or not. This protocol is based on the c/d ratio, where ‘ c ’ is the elastic–plastic boundary under the indenter and ‘ d ’ is the distance of the indent center from the GB [12]:

$$c = \sqrt{\frac{3P}{2\pi\sigma_{ys}}},$$

where P and σ_{ys} are the load at the GB pop-in event and the yield strength of the material, respectively. They found for different GBs that the c/d ratio can

vary between 1.5 and 5. However, for a specific GB, the c/d can found to be within a narrow range. Britton et al. [15] also validated that the occurrence of the GB pop-in event is strongly influenced by the m' and the c/d ratio falls in the range proposed by Wang and Ngan for Fe-0.01 wt% C polycrystals. Moreover, they reported a significant increase in hardness before the occurrence of GB pop-in event. Regardless of the dispute on the grain boundary hardening, all of the aforementioned studies unanimously confirm that the GB pop-ins are frequently observed in BCC materials and strongly influenced by the applied load, distance of the indent to the GB and misorientation between the adjacent grains.

One possible hypothesis of the GB pop-in considers that as the indenter penetrates into the material, dislocations are generated within the plastic zone and travel toward the GB, forming eventually a dislocation pile-up. After reaching a critical shear stress on the slip system of the adjacent grain, the dislocations are expected to transfer across the grain and leads to the GB pop-in event on the LD curve [5, 12–15]. The dislocation absorption at the GB and then their re-emission from the GB are proposed to be the mechanism behind the GB pop-in events [12, 13, 15, 23]. In the literature, this hypothesis is supported by in situ straining transmission electron microscopy experiments, which show dislocation absorption and re-emission at the GBs [24]. However, no direct experimental evidence relating the GB pop-in event with dislocation absorption and re-emission mechanism has been reported in the literature. Moreover, a detailed statistical analysis showing the repeatability of GB pop-in events for specific GB along with three dimensional (3D) dislocation structure analysis at GB pop-in event is still missing in the literature. Therefore, the aim of this work is to check the repeatability of GB pop-in events and study of dislocation structure around and below the indentations surface at the GB pop-in events for a single GB. Such analysis can provide better insight into the hardness of materials indented close to the GB and the underlying dislocations-GB interactions mechanisms at the GB pop-in event. Moreover, current strain gradient plasticity theories of grain boundary yield [25, 26] and three-dimensional crystal plasticity modeling [27, 28] can also benefit from the present comprehensive dislocation structure analysis at the GB pop-in events. This behaviour could also provide a further step for

understanding Hall Petch hardening effects on a local length scale.

For analyzing the grain boundary pop-in behavior, polycrystalline coarse grain tungsten (W) is chosen as a model BCC material in the present work, having moreover an isotropic modulus of elasticity. The nanoindentation experiments are performed near the GB and stopped just after the GB pop-in events. The electron channeling contrast imaging (ECCI) is used to study the dislocation structure around and below the indentations. Under optimum channeling condition (so-called two-beam diffraction conditions), the ECCI contrast is strongest and individual dislocation appeared as line, dark or white spot [29, 30]. Thus, they provide a direct evidence of the different GB-dislocation interactions like dislocations pile-up or transmission across the adjacent grains. A sequential polishing technique has been used to obtain the various cross sections underneath the indentations [21]. To obtain the better statistical analysis of the GB pop-in events, the plastic zone from the previous indentations is completely removed using sequential polishing and the new indentations are performed nearby the same GB. The dislocation structure around the indentations in the grain interior and in the vicinity of GB is also compared, which further sheds the light on how the GB restricts the dislocation movement and causes hardening effect in the presence of GBs.

Experimental

A high-purity (99.9%) coarse grain polycrystalline tungsten (W) supplied by Haines and Maassen Metallhandelsgesellschaft mbH (Bonn, Germany) was used in the present work. The specimen was ground with SiC abrasive papers through 4000 grit. The electro-polishing was conducted for 20–30 s (in 2% NaOH solution at 8 V) to reveal the grain boundaries. The specimen was further vibrational polished using a 40 nm silica suspension to perform the EBSD analysis in a TESCAN MIRA3 SEM equipped EDAX TSL DigiView EBSD system (EDAX, Mahwah, NJ, USA). A GB with a misorientation of 57° between the adjacent grains (A and B having the grain size of $\sim 150 \mu\text{m}$) is chosen as the reference GB (named as GB1) to study the GB pop-in events.

A nanoindenter G200 (from Keysight Technologies, USA) equipped with a diamond Berkovich indenter

tip was used to perform the indentations close to the reference GB in grain A (Fig. 1a, b). A continuous stiffness measurement (CSM) method and a strain rate of 0.05 s^{-1} were used for all the indentations, calculating the hardness via the Oliver–Pharr method [20]. A maximum indentation depth of $2 \mu\text{m}$ was used for the indent 1 (I_1). To study the dislocation structure at the GB pop-in event, indentations 2 (I_2), 3 (I_3) and 4 (I_4) were stopped just after the GB pop-in event (Fig. 1a). The occurrence of the GB pop-in event was directly identified from the nanoindentation load–displacement (LD) curve. The indentations I_1 to I_4 are named as “1st set of the indentations” performed close to GB1. The ECCI was used to study the dislocation–GB interactions on and below the surface. A four-quadrant Deben BSE detector (East Tilbury, UK) in TESCAN MIRA3 SEM was used to obtain ECCI images. All of the ECCI images were taken at a voltage of 20 kV and a working distance of 8 mm with a stage tilt between 0° and 15° . A sequential polishing technique was used to obtain the cross sections underneath the indentations at desired polishing depth [21, 31]. This was achieved by using a 40 nm colloidal silica suspension (OP-S, Struers A/S, Ballerup, Denmark) and a vibrational polishing machine (Jean-Wirtz GmbH, Düsseldorf, Germany). The required amount of material was removed from the surface of the specimen by measuring the indentation depth of the residual impression via confocal laser microscope (LEXT 4000, Olympus, Japan).

For the better statistical analysis of the GB pop-in events in the vicinity of GB1, the plastic zone from the previous indentations was removed via sequential polishing and new set of indentations were performed in grain A (close to the same grain boundary, GB1) named as “2nd set of the indentations” (Fig. 1b). The absence of the plastic zone from the previous indentations and misorientation between the adjacent grains was confirmed via ECCI and EBSD, respectively. The indentations 6 (I_6) and 7 (I_7) were stopped just after the GB pop-in event. In addition to these indentations, 1- μm -depth indentation was performed ($\sim 50 \mu\text{m}$ away from the GB1) in the grain A to compare the dislocation structure in the presence and absence of the GB.

Figure 1 SEM images of Berkovich indentations performed close to GB1, **a** 1st set of the indentations, **b** 2nd set of the indentations after removing plastic zone from the 1st set of the indentations along with representative LD curves **c**, **d**, respectively.

Results and discussion

Statistical analysis of GB pop-in events

Figure 1 shows the SEM images of the two sets of Berkovich indentations performed in the vicinity of GB1 along with their representative LD curves showing the GB pop-in events. As evident from the LD curves, all of the indentations show the GB pop-in event (Fig. 1c, d).

Figure 1a illustrates the 1st set of indentations, whereas Fig. 1b shows the 2nd set of indentations performed near the GB1 after removing the plastic zone from the 1st set of indentations. Before and after removing the plastic zone from the 1st set of indentations, the misorientation between the adjacent grains and the orientation of grain A and B was found to be the same (represented by unit cells in Fig. 1a, b). The indentations I₂, I₃, I₄, I₆ and I₇ were stopped just after the GB pop-in event (Fig. 1c, d);

therefore, these indentations have the smaller residual impression as compared to the I₁ and I₅ (Fig. 1a, b), which were not stopped after the GB pop-in events. It is also pertinent to mention that at the GB pop-in events, the indenter was not physically touching the GB1. This fact is evident from the indentations I₂, I₄, I₆ and I₇, which were stopped just after the GB pop-in event (Fig. 1a, b). One important difference between the indentations is also the orientation of the indenter to the GB. For I₄ and I₇, a face of the indenter is oriented to the GB1, whereas for the other indentations, the tip is facing toward the GB1.

All of the indentations also show a 1st pop-in event, which depicts the incipient plasticity of the material. The 1st pop-in load was found to ~ 1.2 mN as shown in the inset of Fig. 1c for 1st set of indentations. The GB pop-in load was strongly influenced by the distance of the indentation to the GB1. The indentations performed at the larger

distance to the GB1 show the GB pop-in event at much higher load (e.g., I_7) as compared to indentations performed close to the GB1 (e.g., I_6). The detail for each indentation is given in Table 1.

The c/d ratio is estimated to be in the range of 1.6–2.7 for all the indentations performed in the vicinity of GB1, which is in good agreement with Wang and Ngan's work [12]. Before the GB pop-in event, a significant increase in hardness was observed, which drops subsequently after the occurrence of the GB pop-in event as shown in Fig. 2 for the 1st set of indentations. As a reference, also the data of an indentation performed in the interior of grain A are shown, highlighting again the hardening contribution of the GB. After the GB pop-in event, the hardness values reach similar values as of the grain interior indentations (Fig. 2). The increase in hardness is also seen in the load–displacement curves, which show a change in slope before the second pop-in event (Fig. 1c, d). The difference in the hardness increases (ΔH) between the indentations performed in the vicinity of GB, and the grain interior is given in Table 1.

For both indents I_3 and I_4 , GB pop-in occurs at a load of ~ 130 mN. However, for the indent I_4 , a larger increase in hardness was found before the pop-in event as compared to the indent I_3 (Fig. 2). For all the indentations, a strain rate of 0.05 s^{-1} was used; therefore, the strain rate is not playing any role in the increase of hardness for the indent I_4 . Figure 1a clearly shows that due to the inclined GB1, the indenter face was facing the GB for indent I_4 , whereas for the indent I_2 , the indenter tip was facing the GB. This suggests that indenter orientation with respect to the GB influenced the degree of hardening in the

Figure 2 Hardness vs displacement into the surface plot showing hardness drop after GB pop-in events.

vicinity of GB. It is also pertinent to mention here that the c/d ratio and ΔH values for the indentations (I_4 and I_7) facing the side to GB were found to be higher as compared to the indentations facing the tip to the GB (see Table 1). Moreover, a trend of increase in ΔH was observed with increasing c/d ratio. As will be discussed in “Comparison of dislocation structure around the indentations: grain interior vs close to the GB” section, for grain A, a higher dislocation density was found at the indenter side, as compared to the indenter tip (Fig. 5). This leads to larger dislocation pile-ups near the GB when the indenter side is facing to the GB as compared to the indenter tip. Thereby results in higher hardness increase for the indent I_4 as compared to the indent I_3 .

Table 1 Data of GB pop-in events conducted in the vicinity of GB1

Indent no	Max. load at GB Pop-in (P) mN	GB Pop-in size nm	Distance of indent to the GB (d) μm	Elasto-plastic boundary (c) μm	c/d ratio	CSM hardness before GB pop-in GPa	ΔH GPa
I_1	162	109	6.1	11.9	1.9	6.04	1.24
I_2	152	103	7.1	11.5	1.6	5.86	1.06
I_3	130	100	4.2	10.6	2.5	6.39	1.48
I_4^a	129	106	3.9	10.6	2.7	7.16	2.23
I_5	92	117	5.1	8.9	1.8	6.59	1.54
I_6	74	88	3.6	8	2.2	6.49	1.35
I_7^a	386	142	8.2	18.3	2.2	6.38	1.76

^aIn Indents I_4 and I_7 , indenter side was facing the GB

3D dislocation structure analysis at the GB pop-in events

To study the dislocation structure at the GB pop-in events, indent I_2 (indenter tip facing toward GB) and indent I_4 (indenter side facing toward GB) are chosen as the reference indentations. Both of these indentations were stopped just after the GB pop-in event, and the residual impressions were not touching the GB1. Therefore, the GB pop-in is induced by an interaction of the dislocations within the plastic zone of the indentation and the GB. This clearly shows that a physical contact between indenter and the GB can be discarded as a possible mechanism for the GB pop-in event. Thus, the dislocations contrast/structure obtained from ECCI images truly depicts the dislocation–GB interactions at the GB pop-in events. Figure 3 shows the dislocation structure of the indent I_2 at 0 nm (without polishing), -300 nm and -1000 nm of polishing.

From Fig. 3a, it is obvious that the tip of the residual impression was $\sim 1.4 \mu\text{m}$ away from the GB1 at the GB pop-in event. The inset region in Fig. 3a' also shows some transmitted dislocations in grain B from grain A at the surface of the specimen. These transmitted dislocations in grain B can be easily identified by dark and white spots as indicated by exemplary white rectangular and dotted oval regions (Fig. 3a'). From Fig. 3b, it is also evident that below the surface the residual impression is away from the GB1 (indicated by dotted lines). In Grain A, a much higher dislocation contents were found close to the GB, compared to grain B (Fig. 3b,c green rectangular area). The dislocations pile-up at the GB in grain A is hindered to move into the adjacent grain B (exemplary region indicated by the dotted circle). After removing 1000 nm of the material from the surface (Fig. 3c), a higher dislocation density is found to pile up at the GB in the same dotted rectangular exemplary region as compared to the 300 nm polishing depth. This demonstrates that the dislocations

Figure 3 ECCI images of the indent I_2 (which was stopped after the GB pop-in) **a** at the surface along with inset region **a'** showing transmitted dislocation in grain B **b** at -300 nm **c** at -1000 nm.

pile-up in the vicinity of the GB varies underneath the surface. Moreover, some dislocations seem to be entrapped in the vicinity of the GB (exemplary regions are indicated by dotted circles in Fig. 3b, c). The content of the transmitted dislocation (from grain A to B) is also found to be increased below the indentation (Fig. 3b, c).

From Fig. 3b, it can also be seen that the transmitted dislocations appeared to be oriented in specific crystallographic directions. ECCI image at 1000 nm polishing depth also shows a similar trend (dislocation transmission in the specific crystallographic direction) along with the dense region of the dislocation density in the grain B (indicated by dotted arrow). For indent I_2 , the tip of the residual impression was facing the GB1 and the GB pop-in occurred at a load of 152 mN. On the other hand, for I_4 , the GB pop-in occurred at relatively lower load (as compared to I_2) of 129 mN. However, more transmitted dislocations were observed in grain B (on and below the surface) for I_4 at the GB pop-in event as compared to I_2 , as shown in Fig. 4.

At the surface, the GB1 is inclined to the indent I_4 as compared to the indent I_2 (Fig. 1a). Therefore, one

of the indenter sides is facing the GB1 for indent I_4 . As will be discussed in “Comparison of dislocation structure around the indentations: grain interior vs close to the GB” section, the dislocation density is considerably higher at the sides of the residual impression as compared to the tip. Therefore, when the indenter side was facing the GB (Indent I_4), clearly a higher transmitted dislocation content can be seen in grain B (Fig. 4c) as compared to Indent I_2 (Fig. 3c), where indenter tip was facing the GB. The observed higher content of the transmitted dislocation can also correlate to the measured hardness values of the indents I_2 and I_4 before the GB pop-in event. Indent I_4 shows higher transmitted dislocation content as well as the higher hardness value (7.16 GPa) before the GB pop-in event as compared to I_2 , which shows lower hardness value (5.86 GPa) and lower transmitted dislocation content in the adjacent grain. This suggests that due to the lower distance of the indent I_4 to the GB (3.9 μm), more dislocations reached and pile-up in the vicinity of the GB1 as compared to indent I_2 and results in the ~ 1.2 times higher hardness increase for indent I_2 .

Figure 4 ECCI images of the indent I_4 **a** at the surface along with inset region **a'** showing transmitted dislocation in grain B **b** at – 300 nm **c** at – 1000 nm.

For indent I_4 , along with transmitted dislocations in grain B, higher dislocation content in the vicinity of GB was also observed in grain A. At 1000 nm polishing depth, no residual impression was found; therefore, the contrast observed in Fig. 4c is truly the plastic zone, which consists of the dislocations. Javaid et al. [8], reported a high dislocation density (order of 10^{16} m^{-2}) below the indentation in tungsten, which decreases away from the indentation. Therefore, the dislocations far away from the indent center, pile-up at the GB in grain A, are clearly visible as a high density of dots (highlighted as the white rectangular area).

Moreover, similar to the indent I_2 , transmitted dislocations in grain B were found to be aligned in specific crystallographic orientation (Fig. 4a'–c) for I_4 . At 1000 nm polishing depth (Fig. 4c), interestingly, a small GB curvature can be clearly seen (depicted by dotted circle) in the dense region of dislocation density. The similar localized GB movement has already been reported by authors [17] in tungsten, and higher dislocation density in the vicinity of GB is believed to be the driving force for such a localized GB movement.

Figure 5 ECCI images of the 1 μm depth Berkovich indentations in grain A interior.

Comparison of dislocation structure around the indentations: grain interior vs close to the GB

Figure 5 shows an ECCI image of the 1 μm depth Berkovich indentation performed in the center of the grain A. It is evident from Fig. 5 that in the absence of GB, the dislocation contrast around the indentation in grain A extends at much larger areas. On the other hand, when the indentations were performed near the GB1 in the grain A, the dislocation contrast was found to be confined near the GB (Figs. 3 and 4), which clearly shows that the GB blocked the dislocations.

Due to higher dislocation density (order of 10^{16} m^{-2}) in the vicinity of the indentation [8], dislocations contrast appeared as dark regions (indicated by dotted the area in Fig. 5). However, away from the indentation, the dislocation contrast found to be aligned in the specific crystallographic directions (indicated by dotted lines). Moreover, due to the lower dislocation density away from the indentation [8], even at lower magnification, the individual dislocations are visible in the form of dots (exemplary dislocations are highlighted with arrows in Fig. 5).

It is also obvious from Fig. 5 that the dislocation contrast extends significantly from the sides of the residual impression as compared to the tip. This also suggests that when the indenter's side is facing the GB, more dislocations will be generated and pile up close to the GB (as compared to the residual impression facing tip to the GB), which results in the larger increase in hardness values before the GB pop-in event as observed for the indent I_4 as compared to I_2 .

Proposed mechanism of dislocation–GB interactions for GB pop-in

In the light of aforementioned experimental observations, it is plausible to suggest that during the indentation close enough to the GB, dislocations generated and travel toward the GB. The GB acts as an obstacle and causes the dislocation pile-up in the vicinity of GB (Figs. 3 and 4). Due to a continuous loading, the dislocation content in the vicinity of GB increases and results an increase in hardness before the GB pop-in event (Fig. 2). Depending upon the dislocation density in the vicinity of GB and back stresses from the dislocations pile-ups, increase in

hardness before the GB pop-in event can vary even for the same GB, as shown in Table 1. The indenter orientation with respect to the GB can lead to different dislocation content close to the GB, thereby influenced the hardening effect even for the same GB and under similar loading conditions as shown for the indentations I_3 and I_4 (Figs. 2, 3, 4). After reaching a critical stress level, the dislocations will be transmitted in the adjacent grain (Figs. 3,4) and appeared as a displacement bursts in the LD curve (Figs. 1c,d). The dislocation contrast in the vicinity of GB1 (Figs. 3c and 4c) also suggests that some of the dislocations might be trapped close to the GB and probably not all of the absorb dislocations emitted at the same time at the GB pop-in event. However, further computational approaches and in situ TEM experiments are necessary to understand the exact dislocation mechanisms.

Summary

Berkovich nanoindentation experiments were performed in the vicinity of a GB (having a misorientation of 57° between the adjacent grains) in coarse grained tungsten. A secondary pop-in event was observed in the load displacement curve, which is related to the transmission of dislocation through the GB. The GB pop-in load was strongly influenced by the distance of the indenter to the GB. Moreover, a significant hardness increase was observed before the GB pop-in events. The careful three-dimensional analysis confirms that at the GB pop-in events, the residual impressions were not touching the GB for all the indentations. Therefore, the GB pop-in is induced by an interaction of the dislocations within the plastic zone of the indentation and the GB. This clearly shows that a physical contact between indenter and the GB can be discarded as a possible mechanism for the GB pop-in event.

The dislocation structure at the GB pop-in event was studied using ECCI on sequentially polished cross sections below the surface at -300 nm and -1000 nm polishing depths. The ECCI images clearly show the dislocations pile-up and transmission in the vicinity of GB at the GB pop-in event. The indenter orientation with respect to the GB strongly influenced the content of the dislocation pile-up at GB and transmission in the adjacent grain. For the indentation where one side of the residual impression was

facing the GB shows more transmitted dislocation in the adjacent grain as compared to the indentation with the residual impression's tip facing the GB. The indentation conducted in the interior of the grain A revealed that the plastic zone extends to the larger extent from the side of the residual impression as compared to the tip. Therefore, when the indenter side is facing the GB, more dislocation will pile up at the GB and leads to larger hardening effect.

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Compliance with ethical standards

Conflict of interest The authors declare that there is no conflict of interests regarding the publication of this article.

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