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Growth twins in high stacking fault energy metals: Microstructure, texture and twinning

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ABSTRACT

This work explores the formation of twin boundaries in high stacking fault energy metals (SFE > 125 mJ/m^2) by synthesizing thick films (> 10 µm) of Al, Al-5.3 wt%Mg, and Ni using magnetron sputtering. We report the observation of twin boundaries that are inclined with respect to the film growth direction across the entire thickness of the films. The formation of these inclined twin boundaries results in localized changes in the texture of the columnar grains. Microstructural analysis revealed that the fraction of twinned grains in the Al film (46%) is four times higher compared to other similar studies in Al, while the Al-5.3 wt% Mg and Ni films can be considered highly twinned since the fraction of twinned grains is 70% and 90%, respectively. The experimental observations provide an explanation on the formation of twin boundaries during the synthesis of the films, and emphasize that, in addition to stacking fault energy restrictions, high grain boundary mobility is a limiting factor on the nucleation of twin boundaries.

1. Introduction

In crystalline solids, the propensity for twin boundary (TB) formation is highly correlated to the stacking fault energy (SFE) of the material; the lower the SFE, the higher the probability for TB formation [1]. In the case of low to intermediate SFE (< 45 mJ/m^2) metals such as Ag, Cu or Cu-alloys, films with a high density of TBs have been successfully synthesized by a variety of methods [1–8]. The inclusion of TBs in the film microstructure for low-intermediate SFE metals has shown a direct impact on the properties of the material, where the mechanical, thermal, and chemical properties are enhanced [1–8]. These improved properties have increased the working space of nanotwinned (nt) metals and contribute to the development of fundamental research for material's behavior at the nanoscale, particularly in low SFE metals with high TBs density [2,5,9–14].

In contrast, in metals such as Ni, and Al which have a high SFE (125 mJ/m^2 and 166 mJ/m^2, respectively [15,16]), TB formation is unlikely. During the last decade, research efforts have been made concerning the synthesis of TBs in Al and Al alloys at both the macroscale and at the nanoscale [17–21]. At the nanoscale, a study by Xue *et al.* has shown the occurrence of a few TBs in a low fraction of grains (< 9%) in thin (< 80 nm) Al films [22]. In Ni, the synthesis of TBs has been mainly achieved by annealing, and to a lesser extent by electrodeposition and sputtering [23–25] but only the electrodeposited Ni presented TBs at the nanoscale [25]. Despite the many efforts to introduce TBs in high SFE metals, the density of TBs in high SFE materials is limited [1]. Thus the possibility of enhancing mechanical, thermal or chemical properties by introducing TBs in high SFE metals requires further development.

In this work, thick films (~10 µm) of three different high SFE systems (Al, Al-5.3 wt% Mg, and Ni) were magnetron sputtered to elucidate the formation and density of TBs in high SFE metals (SFE > 125 mJ/m^2). A detailed description of the film microstructures was conducted, where it was found that the TB plane is usually inclined with respect to the film growth direction and it is not perpendicular to the [1 1 1] grain growth direction. These results are contrary to the observations in magnetron sputtered nt metals with low SFE, where the TBs are perpendicular to the film growth direction [4,26–30]. Further analysis of the microstructure revealed changes to the texture of the columnar grains due to the inclined TBs. A comparison between the nanostructural features of the three high SFE metals revealed that the three films have higher TB density and fraction of twinned grains than previous values reported in literature [22,24]. The physical evidence shows that in addition to SFE restrictions, the formation and stability of nucleated TBs can be limited by high grain boundary mobility during the synthesis of the films. Overall, the experimental observations allowed discussion on the formation of TBs in high SFE metals and provide an explanation regarding the nucleation of TBs during continuous grain growth in magnetron sputtering.

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2. Experimental details

Ni (99.995% purity), Al (99.99% purity), and Al-5.3 wt% Mg (99.99% purity) targets (7.62 cm in diameter) were magnetron sputtered onto 2.54 cm Si (100) substrates, the SFEs of these three materials is within the range of 125 mJ/m^2 – 166 mJ/m^2 [15,16]. Although, there is no specific value for the SFE of the Al-5.3 wt% Mg, it can be assumed that it is slightly less than the SFE of pure Al (166 mJ/m^2) based on computational studies by Muzyk *et al.* [31] and experiments by Kannan *et al.* in Al-1% Mg [32]. For simplicity, the Al-5.3 wt% Mg alloy film will be referred to as Al-Mg. The sputtering chamber was evacuated prior to deposition to a base pressure < 1.2×10^{-6} Torr (1 Torr=133.3 Pa). During sputtering, the Ar working pressure was 2 mTorr, the target-substrate distance 7.62 cm, and the deposition rate > 7 nm/sec. The thickness of the synthesized films was > 10 µm, as measured with an XP-2 profilometer (AMBios).

The microstructural characterization of the synthesized films was conducted by transmission electron microscopy (TEM) using a JEOL JEM-2100F operated at 200 kV. The specimens for TEM analysis were prepared by two methods, mounting a cross section of the film in silicon, dimple grinding, and ion milling using a Fischione Model 1050 TEM Mill and by performing focused ion beam (FIB) lift-out using a JIB-4500 FIB (JEOL). The texture of the films was then analyzed by taking X-ray diffraction (XRD) patterns using a Rigaku Ultima IV, and by transmission electron backscatter diffraction (T-EBSD). The FIB lift-out specimens used for TEM were also used for T-EBSD, which was carried out on the cross-section of the films by using a JSM-7001F-LV scanning electron microscope (JEOL) with a Hikari detector (EDAX). The FIB lift-out specimens were tilted 20° from the horizontal plane to give rise to a strong signal (more information in regards to the geometry of the experiment can be found elsewhere [33]); the accelerating voltage and working distance were 30 kV and 15 mm, respectively, while the step size was 5–10 nm. The T-EBSD collated data was analyzed with the TSL orientation image microscopy (OIM) software. Data with confidence index lower than 0.1 were removed and showed as black pixels in the orientation maps. Σ3 TBs were identified by applying boundary trace analysis as described elsewhere [34,35].

3. Results and discussion

3.1. Microstructural characterization

Cross-sectional TEM images were taken to study the microstructure of the films and identify TBs. In Fig. 1a, c, and e, low magnification bright field images of columnar grains with inclined TBs (marked by red dotted lines) are presented for Al, Al-Mg, and Ni, respectively. The insets depict the selected area electron diffraction (SAED) patterns obtained from the columnar grains. The typical double hexagon pattern of the (110) zone axis oriented twinned grain is highlighted by the dotted blue and magenta lines; each SAED pattern was also indexed with the corresponding plane orientation. Fig. 1b, d, and f show zoomed in high resolution TEM images of the square regions in Fig. 1a, c, and e. The TBs are marked by the red dotted lines and lie in a {1 1 1} family plane that is not perpendicular to the [1 1 1] direction (for instance the (1 1−1) plane), while the blue and magenta dotted lines show the (1 1 1) or (−1−1−1) planes at each of the two sides of the TBs. The yellow lines mark the (1 1−1) planes that are parallel to the (1 1−1) TB plane. The insets in Fig. 1b, d, and f show the fast Fourier transform of the high resolution TEM images confirming the presence of a TB. Since the planes at each side of the TB are (1 1−1) planes (yellow lines in the high resolution images), the TBs depicted in Fig. 1 are symmetrical, tilt, and twist boundaries, which is the definition used to classify Σ3 {1 1 1} coherent twin boundaries (CTBs) [36,37].

Representative cross-sectional low magnification TEM images of the Al, Al-Mg, and Ni films microstructures are displayed in Fig. 2(a-c) respectively. The three microstructures have columnar grains, and several TBs are highlighted by the dotted red lines. The TBs were identified following the characterization procedures shown in Fig. 1. In general, the TBs are not perpendicular to the film growth direction, which is in contrast from what is commonly observed in the synthesis of TBs in low SFE materials by magnetron sputtering [26–28,38]. In Fig. 2, α is the angle between the inclined TB and a plane that is perpendicular to the film growth direction. In Fig. 2a and b the TBs are preferentially inclined at an angle α ~70°, while in Fig. 2c the TBs are inclined at different angles and α varies from 0° to ~75°. Notice that in Fig. 1, the angle α between the TB plane and a plane perpendicular to the film growth direction is ~70.5° for both Al and Al-Mg (Fig. 1b and d), while in the case of Ni (Fig. 1f) α is ~ 50°. Overall, the microstructural characterization reveals that the inclined TBs are present across the thickness of the films (Fig. 2), and that the inclined TBs can induce changes in the columnar grains texture (Fig. 1), which is discussed in the following section.

3.1.1. Texture Analysis

The experimentally observed inclined CTBs can be associated with a change in the texture of the films. For example, in the case of Al and Al-Mg films at one side of the CTB, there are (1 1 1) planes that are perpendicular to the [1 1 1] grain growth direction as well as the film growth direction, while on the other side of the CTB, the (1 1 1) planes are inclined with respect to the film growth direction (Fig. 1b and d). In the case of the Ni film, the (1 1 1) planes are inclined with respect to the film growth direction at each of the two sides of the CTB (Fig. 1f). XRD and T-EBSD were used to investigate the overall film texture and to support the observations by TEM. Specifically, T-EBSD was used to identify the texture of the grains at each side of the Σ3 TBs. Fig. 3 shows the XRD patterns of the Al, Al-Mg, and Ni films. The Al and Al-Mg films XRD patterns both show a strong {111} texture, while the Ni film XRD pattern presents two strong textures {111} and {200}.

In EBSD, a boundary is considered a Σ3 TB if the misorientation angle between two grains is ~60° and if the misorientation axis between the same two grains is perpendicular to a {111} plane [35]. Fig. 4 shows T-EBSD scans of single columnar grains from the films cross-section, where the colors correspond to the orientation of the grains in the film growth direction. In Fig. 4(a-d), the inverse pole figures of a columnar grain with Σ3 TBs are identified by the change in color in each columnar grain. The legend of the inverse pole figure is included to identify the change in texture for each of the columnar grains after an inclined TB. Notice that in the case of Ni, two scans are included to show TBs that are nearly perpendicular (Fig. 4c) and inclined (Fig. 4d) to the film growth direction. Table 1 lists each of the columnar grains shown in Fig. 4, where the planes at each side of the TBs are labeled by the white numbered circles and are perpendicular to the paper plane in Fig. 4. The OIM software analysis was used to find the plane orientation for each of the white numbered circles with respect to the film growth direction, as well as the misorientation angle and misorientation axis between consecutive numbered white circles. For example, in Fig. 4a the columnar grain contains three numbered circles that are also shown on the right-hand side texture legend at their corresponding orientations, while in Table 1 the plane that corresponds to the number 1 and number 2 white circles are listed as (−1 1 1) and (−1 1 5) respectively. The misorientation angle and the plane perpendicular to the misorientation axis between the two planes is 60° and (1−1 1), respectively. In general, the Al and Al-Mg columnar grains showed mainly two textures before and after the Σ3 TBs {1 1 1} and {1 1 5}, while Ni columnar grains showed various textures, similar to the observations in the XRD patterns in Fig. 3.

Despite the strong {1 1 1} texture in Al and Al-Mg observed in Fig. 3, the TBs were not observed in planes perpendicular to the film growth direction, or to the [1 1 1] grain growth direction. In the case of the Ni film, some TBs are nearly perpendicular to the growth direction of the film, but due to the fact that the grains have different