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Influence of Y_2O_3 nanoparticles on the twinning of single crystalline magnesium

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ABSTRACT

The influence of Y_2O_3 nanoparticles introduced by Friction Stir Processing on the deformation of magnesium has been studied with microcompression testing of single-crystal pillars oriented favorably for tensile twinning. It was found that the nanoparticles lower the twin nucleation stress and affect the twin morphology, as well as mitigate the size effects usually observed in pure magnesium single-crystals. While single twin nucleation is consistently observed in pure magnesium pillars, multiple twins of identical variants are observed in the pillars with nanoparticles, having nucleated at the Y_2O_3 particles. Consequently, the resultant twin–twin boundaries lead to an effective hardening.

With a specific weight as low as $1.74\,\mathrm{g \cdot cm^{-3}}$, magnesium is the lightest of all structural metals and possesses a huge potential to be used in applications that require weight reduction. However, its strength and toughness need to be increased in order to compete with other light metals such as aluminum and titanium. One solution is the reinforcement of magnesium and its alloys with oxide nanoparticles, where the addition of a low volume fraction of reinforcements allows the improvement of the mechanical properties [1–5] without compromising the density.

The use of magnesium nanocomposites as structural materials demands a fundamental study of their deformation mechanisms. Plasticity in magnesium, which has a hexagonal close packed (hcp) crystal structure, is characterized by a very strong plastic anisotropy as well as a complex twinning activity. Understanding these deformation mechanisms in the presence of nanoparticles is then crucial for the development of high performance nanocomposites for widespread industrial use.

Several studies have already been carried out on the deformation mechanisms of magnesium single crystals [6–9]. Nevertheless, to the authors' knowledge, the single crystalline deformation mechanisms of magnesium based nanocomposites reinforced with oxide particles has not been fully elucidated and is the subject of the present study. In order to focus on the role of the nanoparticles on the deformation behavior, microcompression testing of single crystalline volumes was carried out, thus eliminating the effects of grain boundaries and strain incompatibilities.

In the present study, attention has been made in order to fabricate microcolumns with an orientation such that the *c*-axis is oriented almost perpendicularly to the loading axis during the microcompression testing, ensuring a maximum Schmid factor for tensile twinning and minimum for basal slip. This orientation has been shown to undergo tensile twinning as its primary mode of deformation [8]. Tensile twinning ($\{10\bar{1}2\}$ twin) is activated when a tensile stress is applied along the *c*-axis. It leads to a reorientation of the *c*-axis in the twin of about 86° around the $[11\bar{2}0]$ axis. A few studies have already been published regarding the effect of particles on the deformation behavior of magnesium alloys. Garcés et al. [10] found that the introduction of a second phase can suppress twin nucleation, while Gharghouri et al. [11] investigated the interactions between second phase precipitates and twins. A more recent study from Stanford et al. [12] has shown that a larger number of twins is present in the alloys containing precipitates and the presence of these precipitates increases the hardening rate on the twinning system more than on the slip systems. The advantage of working with single crystalline specimens in the present case is that the compression stress within the microcolumn is not modified by the neighboring grains. This means that the shear stresses on the different slip or twin systems is

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just defined by the Schmid factors imposed by the orientation of the compression axis.

Magnesium nanocomposites reinforced with Y_2O_3 nanoparticles have been elaborated using Friction Stir Processing (FSP) [13–16]. Y_2O_3 powder is disposed in a groove between two plates of pure magnesium (99.95% purity). During FSP, a non-consumable rotating tool penetrates in the material and advances along the processing area. The heat generated by the friction of the tool during rotation renders the material malleable and creates a material flow that follows the tool movement [15]. It leads to a mechanical mixing between the particles and the base material and provides a homogeneous distribution of the particles with a volume fraction of 0.3% within the stir zone. Further information about the samples is presented in Ref. [17]. In order to circumvent potential influences of the FSP effect, pure magnesium samples have also been elaborated using the same friction stir processing conditions without the introduction of particles.

During FSP, a dynamic recrystallization takes place [16,18] leading to a final grain size close to 10 μm [17]. However, large grains that allow the machining of single crystalline microcolumns inside a single grain with a given orientation are needed. Thus, a subsequent annealing treatment at 500°C for 3 hours has been performed in order to induce abnormal grain growth. Electron Backscatter Diffraction (EBSD) has been performed in order to identify grain orientations of interest and to avoid preexisting twins in the subsequent sample preparation. The microcolumns have been fabricated in selected grains using focused ion beam (FIB) milling using a FEI Nova Nanolab 200 Dualbeam scanning electron and FIB microscope. Each microcolumn is fabricated with annular milling, which involves a series of successive circular cuts at varying diameters with an attempt to ensure an aspect ratio of 3:1 between column height and mid-height diameter. Two nominal column diameter were used: 5 μm and 10 μm, in order to consider the known size effects associated with microcompression testing of magnesium [8,9]. Annular milling has been used in preference to the lathe milling technique for magnesium columns in order to minimize the Ga-rich surface oxide layer, as shown in reference [19].

The microcompression tests were conducted in a Nanoindenter XP (Agilent) equipped with a flat-ended conical indenter with a 15 μm circular punch. The microcompression experiments were run in a load-control mode up to different maximum strain using a nominally constant strain rate within the range of 2.10^{-4} and 10^{-3} s^{-1}. The stress–strain response was computed using the true stress convention.

Fig. 1 presents the stress–strain curves for 5 μm and 10 μm columns favorably oriented for tensile twinning. For all the columns, the stress increases smoothly until a first strain burst occurs. A clear difference between the mechanical behavior of both materials can be noticed: a staircase stress-strain curve is observed for the nanocomposite (Fig. 1 (b)), while a single step is observed for pure magnesium (Fig. 1 (a)). This single strain burst observed for pure magnesium microcolumns is similar to the results obtained by Kim [8], when performing $[10\bar{1}0]$ microcompression tests in high purity (99.999%) magnesium single crystals. It confirms that the presence of even a small volume fraction of Y_2O_3 particles (0.3%) introduces a significant change to the mechanical behavior of magnesium, the influence of FSP is not the cause of the new behavior.

Studies performed by Kim [8,20] have shown that twinning nucleates at the early stage of the deformation and propagates stably and slowly. Once the strain burst occurs, rapid and unstable twin propagation takes place. In the case of nanocomposites columns, multiple small bursts followed by intermittent increasing flow stress is observed. This is in contradiction with a single twin propagation throughout the entire column. It is noticeable that the twins nucleate more easily in the nanocomposite since the first strain bursts are triggered at lower values of the applied stress in the nanocomposite. The average stress for unstable twin propagation in pure magnesium and the nanocomposite is, respectively, 140 ± 20 MPa and 80 ± 30 MPa (data set for 18 experiments). The lower critical stress found in the case of the nanocomposite can be attributed to the presence of stress concentration due to the Y_2O_3 particles during compression, facilitating twin nucleation inside the nanocomposite. While Garcés et al. [10] found that particles suppressed twinning due to tensile residual stresses induced by SiC whiskers in AZ31, such residual stresses are not relevant in the present case. Both the much smaller particle size and much smaller volume fraction of the Y_2O_3 nanoparticles negate any significant residual stress development. Instead, the nanoparticles serve as stress intensity sites during loading which lead to preferential twin nucleation at the particle/matrix interface.

In order to investigate the twinning activity, FIB cross sectioning followed by EBSD analysis have been performed on some of the deformed columns. Cross sectioning EBSD of a 10 μm column of the pure magnesium and the nanocomposite are presented in Fig. 2 (a) and (b), respectively. In microcompression of pure magnesium, a single twin is typically nucleated at the top of the column. Even in the rare case of the nucleation of two twins, as is shown in Fig. 2 (a), the twins nucleate at the top of the pillar due to the stress concentration imposed by the flat punch.

A similar twin morphology is observed in the upper part of the microcolumn in the nanocomposites (Fig. 2 (b)), but many additional twins of the same variant can be observed emitting from Y_2O_3 particles (indicated by the arrows) likely due to stress concentrations at the particle–Mg interface. This is in agreement with the observations of Robson et al. in Mg-5 wt%Zn polycrystalline alloys [21]. The presence of multiple twins can be correlated to the multiple strain bursts observed in the stress-strain curve (Fig. 1 (b)) for the nanocomposite. The twin propagates until it finds an obstacle such as a nanoparticle, another twin or the surface. Then, the stress increases, as shown in Fig. 1 (b), until another twin nucleates, propagates and again stops when it encounters an obstacle.

Yu et al. [22] studied twin–twin interactions in magnesium showing that a growing twin cannot transmit into another favorably growing twin; rather, a twin–twin boundary forms and new twins will nucleate in order to accommodate the applied strain. What is unique in our case, is that while the twin–twin boundaries envisaged by Yu et al. [22] were for different twin variants, all of the observed twins in the present case are of the same variant. Such twin–twin boundary structures are observed in Fig. 2 (b), where the overlay of an image quality map over the colored orientation map show dark regions (black arrows) throughout the twinned region. This is strong evidence of dislocation structures accommodating twin–twin boundaries between twins of the same variant. This would suggest that a dislocation structure is present at the growing twin front, a view shared by many [22–24].

Nonetheless the different mechanical behavior of the nanocomposite, an effective hardening is observed below 6% strain, after which a more significant slope change in the stress–strain curve occurs similar to that found in pure magnesium. The longitudinal strain due to twinning shear can be calculated by the following expression [8]:

$$\varepsilon = f_{v,twin} m \gamma \quad (1)$$

where $f_{v,twin}$ is the volume fraction of the twin, m is the Schmid factor (0.49 for the studied orientations) and γ is the twinning shear for tensile twinning in magnesium (0.129 according to Yoo [25]). If the twin had propagated through the entire column, it would lead to a strain of 6.321%. Thus, it is clear that the entire column has been twinned once the strain hardening takes place. This hardening after twinning is a consequence of the new crystal orientation after twinning. The deformation is very likely accommodated by multiple pyramidal slip systems, as shown elsewhere [8,26]. Thus, the