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Dependence of compressive deformation on pre-strain and loading direction in an extruded magnesium alloy: Texture, twinning and de-twinning

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ABSTRACT

This study was aimed at identifying compressive deformation behavior of an extruded AM30 magnesium alloy after different amounts of pre-strain along the extrusion direction (ED), with focus on the effect of re-loading direction, texture evolution, and deformation mechanisms. Compressive loading in both ED and transverse direction (TD) resulted in a similar sigmoidal true stress–true strain behavior due to the presence of two sets of basal textures with *c*-axes aligned nearly parallel to the plate normal direction which facilitated the occurrence of $\{10\bar{1}2\}$ extension twinning. In the two-step ED–ED compression, the compressive yield strength (YS) linearly increased, while the ultimate compressive strength (UCS) and hardening capacity linearly decreased with increasing pre-strain. The disappearance of twin boundaries or the coalescence of twins via twin growth was observed, but the twin boundaries were more visible and the intensity of cumulative textures with *c*-axes rotating towards the anti-compression direction was lower, in comparison with the one-step continuous compression at an equivalent strain level. In the two-step ED–TD compression, both YS and UCS increased, and hardening capacity decreased with increasing pre-strain. After a 4.0% pre-strain along the ED plus 4.0% re-compression along the TD, two seemingly opposite phenomena, i.e., the formation of new twins and de-twinning were observed to be co-existent due to the presence of multiple sets of textures after the first-step pre-straining. Both the reversal of textures and microstructural examinations corroborated the occurrence of de-twinning or twin narrowing/shortening as a result of the change of deformation path in the second-step compression.

1. Introduction

As an ultra-lightweight metallic material, magnesium alloy has recently rekindled a considerable interest in the automotive and aerospace sectors, and it is being intensively studied for the potential load-bearing structural applications to reduce vehicle weight and increase fuel efficiency [1–8]. Wrought magnesium alloys have in general better mechanical properties including tensile properties [9,10] and fatigue resistance [11,12] compared to cast magnesium alloys [13–15]. However, their applications are still limited because of poor room temperature formability and anisotropy related to the presence of crystallographic textures in wrought magnesium alloys, which are developed during forming or manufacturing processes (extrusion, rolling, forging, etc.). Since the extruded magnesium alloy contains a strong basal texture where *c*-axes of hexagonal close-packed (hcp) unit cells in most grains were perpendicular to the extrusion direction, a compressive or tensile load along the extrusion direction (i.e., perpendicular to the *c*-axis of a hcp unit cell) may cause the formation of extension or contraction twins [16–18].

Twinning in the hcp materials plays a key role in both texture evolution and hardening behavior [19]. The former effect is related to the crystallographic re-orientation between the twinned and untwined regions within grains, while the later is associated with the interaction between twin boundaries and dislocations. During the interactions, twin boundaries act as barriers to the dislocation motion, as do grain boundaries. Depending on the stress field, dislocations at twin boundaries can either dissociate into partial twinning dislocations (TDs) or aid twin growth when the dissociation products are glissile twinning dislocations and the stress state favors their motion in the twin direction [20–23]. The occurrence of $\{10\bar{1}2\}$ extension twinning can re-orient the crystallographic lattice by 86.3° [16,18,24,25]. Indeed, the activation of such $\{10\bar{1}2\}$ extension twinning in the extruded magnesium alloy causes *c*-axes in the twinned regions to be aligned towards the compression direction from the basal (0001) texture where the *c*-axes in most grains are initially positioned perpendicularly to the loading axis [25].

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It has also been reported that the twinned areas can be aligned to either twin growth or de-twinning if the strain path (or loading path) is changed appropriately [2,26–32], which can have a significant influence on the behavior of magnesium alloys during forming, e.g., rolling, leveling, coiling, bending, stretching, since it can result in an increase or decrease in the flow stress when such alloys are subjected to strain reversals [33,34]. It is therefore necessary to understand the effect of reversing the strain path on the stress–strain response, as well as the role of twinning in bringing this about. The effect of $\{10\bar{1}2\}$ extension twinning generated by pre-straining on the subsequent deformation behavior with a change of strain path during reloading has been studied by several authors [33,35–39]. Xin et al. [36] showed that after compressive pre-straining along the rolling direction (RD) in an AZ31 alloy, re-compression along the transverse direction (TD) led to the further occurrence of $\{10\bar{1}2\}$ extension twinning within the pre-existed $\{10\bar{1}2\}$ extension twins, which constituted a new type of 'double extension twinning'. On the other hand, Park et al. [33] observed the occurrence of de-twinning in the $\{10\bar{1}2\}$ twin bands when the rolled AZ31 alloy was subjected to consecutive in-plane compressions along two orthogonal directions (RD and TD). The results were explained by the fact that the twin variants generated between the compression along the RD and subsequent compression along the TD were similar, hence the double twinning in twin bands became equivalent to de-twinning.

Although twinning and de-twinning in hcp materials such as magnesium and zirconium have been reported in the literature [2,29,33,36,38,40–47], and they have a significant influence on the strain hardening, the mechanisms about twinning and de-twinning are still not fully understood. The microstructure evolution that influences hardening and texture behavior upon re-loading (both longitudinal and transverse) depends on pre-strain amounts [33]. However, it is unclear how twinning and de-twinning occur with a change of strain path, if the formation of new twins and de-twinning can be co-existent, and how they affect the texture change and hardening behavior. The objective of this study was, therefore, to identify the influence of twins introduced by pre-straining on the subsequent texture and hardening behavior of an extruded AM30 alloy upon re-loading with and without changing the strain path.

2. Experimental

AM30 extruded magnesium alloy was selected in the present study. Two types of sample dimensions were machined for the compression tests: cylindrical samples with a diameter of 5 mm and a height of 8 mm based on ASTM E9-09, where the cylinder (or compression) axis was parallel to the ED, and rectangular samples with dimensions of 5 mm × 6 mm × 4 mm (ED × TD × ND) used for the strain-path change tests. Samples were first pre-strained in compression along the ED at different strain levels of 1.5%, 2.3%, 4.0%, 5.8%, 7.5% and 8.2% (denoted as 1.5%ED, 2.3%ED, 4.0%ED, 5.8%ED, 7.5%ED and 8.2%ED, respectively) using a computerized Instron machine at a strain rate of $1.25 \times 10^{-4}\,s^{-1}$ and at room temperature. The pre-strained cylindrical and rectangular samples were subsequently subjected to re-compression along the ED or TD until failure. To observe the microstructural change and texture evolution, some pre-deformed samples were re-deformed to a strain amount of 4.0%. It should be noted that in the strain-related evaluation (i.e., strain amounts, stress–strain curves and strain-hardening rates), the machine deformation was eliminated using a calibration curve to arrive at the actual or net strain values of all test samples, as mentioned above. For the microstructural characterization, the deformed or re-deformed samples were cut along the compression axis using a slow diamond cutter, cold-mounted, ground up to a grit of #1200, polished with 6, 3, and 1 μm diamond paste, respectively, and etched using acetic picral solution containing 4.2-g picric acid, 10-ml acetic acid, 10-ml H_2O, and 70-ml ethanol to examine the evolution of deformation twins formed during compression. Texture was determined using a PANalytical X-ray diffractometer (XRD) by measuring a set of five incomplete pole figures ($\{0001\}$, $\{10\bar{1}0\}$, $\{10\bar{1}1\}$, $\{11\bar{2}0\}$, $\{10\bar{1}3\}$) between $\Psi = 0°$ to 75° in a back reflection mode using Cu K_α radiation at 45 kV and 40 mA. Then the pole figures were evaluated using MTEX software. In the texture analysis to determine the pole density, defocusing stemming from the rotation of XRD sample holder was corrected using experimentally determined data from the diffraction of magnesium powders.

3. Results and discussion

3.1. Initial microstructure

The microstructure of the as-extruded material is shown in Fig. 1(a), consisting of a mix of large and small grains which were twin-free. The initial texture of this material, shown in Fig. 1(b), exhibited a strong basal texture with a maximum intensity of 8.6 multiples of random distribution (MRD). While the basal (0001) poles had about 20° tilt towards ED, the $(10\bar{1}0)$ poles were observed towards both ED and TD, indicating that the prismatic $\{10\bar{1}0\}$ planes of hcp unit cells in most grains were parallel or perpendicular to the ED, respectively. As a result, two sets of initial basal textures, i.e., $\{0001\}<2\bar{1}\,\bar{1}0>$ and $\{0001\}<10\bar{1}0>$, could be identified in the extruded AM30 Mg alloy, with a detailed analysis presented in [25]. Based on these results, a schematic diagram could be plotted in Fig. 1(c), where the *c*-axes of hcp unit cells in most grains were nearly perpendicular to the ED (or pre-compression axis) in both cases of cylindrical and rectangular samples.

3.2. Effect of pre-strain and loading direction on compressive properties

The true stress–true strain curves of the initially as-extruded AM30 magnesium alloy compressed in the ED and TD directions are shown in Fig. 2(a). It is seen that both curves were similar and the average compressive yield stress was ~91 MPa in both ED and TD directions. Also, both curves showed a skewed/sigmoidal shape, indicative of the activation of deformation twinning [25,35,48]. An ultimate compressive strength of ~325 MPa and ~295 MPa was observed along the ED and TD, respectively. However, when compressed in the ED, the true stress increased more rapidly with increasing true strain, compared to the TD. Because of the presence of strong basal textures in the as-extruded material with the *c*-axes of most grains aligned almost parallel to the ND (Fig. 1(b) and (c)), both ED and TD compressed samples were subjected to a compressive loading perpendicular to the *c*-axes. Such a loading condition facilitated the occurrence of the $\{10\bar{1}2\}$ extension twinning. Therefore, the flow curves exhibited a similar sigmoidal shape (Fig. 2(a)), which is known to be a typical feature of the twinning-dominated deformation [25,33,36,37,48–51].

The true stress–true strain behavior of the samples that were subjected to different amounts of pre-strains along the ED is shown in Fig. 2(b), where the re-compression was also conducted along the ED, as symbolized by *x*%ED–ED, where *x* indicates the pre-strain amount after the exclusion of machine deformation. It is of interest to see that a marked change in the sigmoidal shape of the true stress–true strain curves occurred. With increasing pre-strain from 0%ED, 1.5%ED, 2.3%ED, 4.0%ED, 5.8%ED, 7.5%ED and 8.2%ED, the skewed curve shape gradually vanished. When the pre-strain reached about 7.5–8.2%, the true stress–true strain curve