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Competitive twinning behavior in magnesium and its impact on recrystallization and texture formation

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ABSTRACT

Rolled pure Mg and Mg-1 wt% Gd alloy were subjected to room temperature in-plane compression along the rolling direction, followed by isochronal annealing treatments for 1 h. The results of deformation texture and microstructure showed substantial differences due to rare earth alloying. In spite of imposed c-axis extension during deformation, the Mg-1Gd alloy retained the initial texture with the majority of basal poles concentrated near the longitudinal direction of the used channel-die tool. Electron back scatter diffraction analysis of the deformation microstructure revealed a predominance of $\{10\bar{1}1\}$ compression and $\{10\bar{1}1\}$ - $\{10\bar{1}2\}$ double twins relative to coexisting $\{10\bar{1}2\}$ tension twins. This behavior was significantly contrasting in comparison with that of pure Mg, wherein first and second generation $\{10\bar{1}2\}$ tension twins were observed in profuse quantities. Continuous dynamic recrystallization took place inside compression and double twins by means of slip assisted subgrain rotation about the $[0001]$ axis giving rise to a sharp prismatic fiber of recrystallized orientations. This fiber was transformed into a randomized texture pattern during subsequent static recrystallization and grain growth due to a different discontinuous recrystallization mechanism. This resulted in a significant annealing texture weakening and an increase of the overall Schmid factor for basal slip.

1. Introduction

Deformation of hexagonal close packed (HCP) crystals takes place by activation of dislocation slip and mechanical twinning, and is characterized by a strong directional anisotropy. Deformation twinning is a very important feature of HCP plasticity due to its strong impact on texture evolution, anisotropy and mechanical response of the deformed material. Being an HCP metal, magnesium is unable to provide easily activated independent slip systems (particularly ones with a Burgers vector out of the basal plane) to ensure homogeneous deformation at room temperature [1,2]. In such case, the deformation behavior will strongly rely on twinning that can to some extent accommodate strain imposed along the c-axis. However, due to its characteristic polar nature, twinning impacts the deformation in primarily two ways: (1) the twinned volume reorients the crystal, such that mirror symmetry relative to the parent grain is created. Such crystallographic reorientation is usually associated with very high misorientation angles between parent grains and their twinned parts, which drastically modifies the crystallographic texture. Thus, understanding the role of twinning in the deformation texture evolution in magnesium and its alloys is indispensable. (2) The rapid reorientation of the twinned region leads to the formation of high angle twin boundaries acting as

strong barriers for further dislocation slip, and thereby promoting work hardening [3]. However, in the presence of anisotropic slip behavior, such twinning induced hardening can have a negative impact on the ductility of the material. Premature fracture frequently observed after profuse $\{10\bar{1}2\}$ extension twinning is normally a result of a strong concentration of orientations associated with very low Schmid factors (SF) for easy slip, when the forming load is applied along the crystallographic c-axis. Under such conditions, high stresses (exceeding the failure stress) are required to promote $\{10\bar{1}1\}$ contraction twinning and $c+a >$ pyramidal slip, which is why extrusion samples, for instance, tend to fail at low strains ($< 15\%$) during subsequent compressive loading (e.g. cold forging) along the extrusion direction [4].

In some cases, where contraction and double twins are activated, the related twinned areas can undergo much more plastic deformation relative to their parent grains owing to their favorable alignment for basal slip. Such a large difference in the degree of strain accommodation leads to incompatible plastic deformations between the 'soft' twin interior and the 'hard' adjoining matrix that trigger high local stress concentrations at the twin boundaries leading to subsequent shear failure [1]. A further consideration is that contraction twin boundaries have stable energetic configurations and are thus generally considered to be immobile, which is why contraction twins in magnesium are

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commonly resistant to growth, and thereby do not consume their surrounding matrix, unlike extension twins [1].

Several studies have already established that rare earth (RE) additions have the effect of randomizing the texture and in this way improve the ambient temperature formability of wrought magnesium sheets and extrusions [5–13]. With respect to twinning activation, previous studies on Mg-RE alloys indicate promoted contraction and double twinning during deformation as compared with conventional Mg-Al-Zn (e.g. AZ31) alloys [6,11,14,15]. In these studies, however, contraction twinning was still found to be less favorable than extension twinning when loaded accordingly. This particular aspect remains a subject of debate in the Mg literature and it is still yet to be determined whether contraction and double twin recrystallization contributes to the RE-texture modification phenomenon. Being regions of high dislocation density, these twins can be considered as preferred sites for nucleation of recrystallization.

The current study focuses on understanding the differences in twinning behavior with and without the presence of RE elements by investigating pure magnesium and a binary Mg-1Gd (wt%) alloy. Both materials were subjected to deformation in plane strain compression (PSC) at room temperature and subsequent annealing treatments at elevated temperatures between 350 °C and 450 °C to investigate twin formation and the response of formed twins to static recrystallization during annealing. The results readily indicate modified twinning behavior during deformation in the presence of Gd, characterized by competitive formation of both extension and contraction twin types. During annealing, static recrystallization promotes strong texture weakening, which is preserved during grain growth. The underlying mechanisms during deformation, recrystallization and grain growth are discussed within the purview of the addition of solute RE elements.

2. Experimental procedure

Commercial purity Mg (99.95%) and binary Mg-1Gd (wt%) alloy were cast as per Ref. [7]. Rolling blocks of dimensions 80 mm × 40 mm × 40 mm were machined from the starting cast materials and hot rolled at 450 °C (nominal furnace temperature) to a final thickness of 12 mm, imposing a total thickness reduction of 70% in seven passes. The rolling treatment was performed in order to achieve a suitable grain refinement and a well-defined sheet texture that was utilized later for obtaining specially oriented specimens for the plane strain deformation experiments. A subsequent recrystallization treatment was conducted at specific annealing parameters (Mg-1Gd was annealed at 350 °C for 5 min and pure Mg for 1 h, followed by rapid quenching in water), so that texture changes were insignificant, yet the microstructure was fully recrystallized and stress relieved.

Deformation experiments were carried out in plane strain compression (PSC) at ambient temperature and a constant strain rate of 10^{-2} s^{-1} . PSC was performed in a channel-die using a conventional screw-driven ZWICK testing machine. Channel-die specimens with dimensions of 12 mm (Longitudinal Direction; LD) × 10 mm (Transverse Direction; TD) × 4 mm (Compression Direction; CD) were machined from the hot rolled and annealed alloys and subjected to in-plane compression (IPC), wherein the PSC loading axis was aligned along the rolling direction. Hexagonal boron nitride powder was used as a lubricant to minimize friction between the die surfaces and the specimen. Deformation was conducted up to true strains of ~ 6% for pure Mg and ~ 7% for Mg-1Gd. The strains were chosen such that the deformed specimens exhibited strongly twinned microstructures. The stress-strain curves were obtained by means of automated measurement of the load and displacement with an average data acquisition frequency of 50 Hz. At least three tests were conducted for each strain and material to ensure reproducibility of the stress-strain data. The results presented here show the average flow curves. For the annealing study, the deformed specimens were annealed at 450 °C for 1 h followed by water quenching. Selected specimens were additionally annealed for 1 h at an

intermediate temperature of 350 °C in order to track the microstructural evolution during recrystallization.

Optical microscopy, X-ray diffraction (XRD) and electron backscatter diffraction (EBSD) measurements were utilized for texture and microstructural characterization. While texture evaluation was conducted on the compression plane, all microstructural characterization was performed on the transverse plane oriented normal to the TD. Specimens for optical microscopy were prepared by conventional grinding and diamond polishing (down to 0.25 µm), followed by electro-polishing in a 5:3 solution of ethanol and H_3PO_4 for 30 min at 1.93 V. Acetic picral and 5%-Nital were used as etchants to reveal the microstructure. XRD pole figure measurements were conducted using a Bruker D8 Advance diffractometer, equipped with a high resolution area detector, operating at 30 kV and 25 mA, using filtered iron radiation and polycapillary focusing optics. The measurements were performed on the specimen mid-plane to obtain the bulk texture and avoid any surface effects. The quantitative texture analysis toolbox MTEX [10] was employed to calculate the orientation distribution function (ODF) and full pole figures from the incomplete pole figure measurements. EBSD measurements were performed with a LEO-1530 scanning electron microscope (SEM) equipped with a field emission gun (operated at 20 kV) and an HKL-Nordlys II EBSD detector. Specimens for EBSD were subjected to electro-polishing in Struers AC-2 reagent using Lectro-Pol 5. For pure Mg, a voltage of 34 V was applied for 25 s and for Mg-1Gd, 41 V for 30 s. The final electro-polishing step ensured a mirror finish, removing any preparatory twins that may have been introduced during mechanical polishing. Specimen preparation was conducted under ambient temperature conditions. The acquired raw EBSD data was subsequently analyzed using commercially available EBSD analysis software and the MTEX toolbox.

3. Results

3.1. Thermomechanical treatment condition

Fig. 1a and b display the hot rolled and recrystallization annealed microstructures and sheet textures for pure Mg and Mg-1Gd prior to PSC deformation. Both materials revealed fully recrystallized microstructures. The measured average grain sizes with the lineal intercept method were 58.1 µm (S.E. = 1.1 µm) for pure Mg and 19.3 µm (S.E. = 0.65 µm) for Mg-1Gd. For pure Mg the thermomechanical treatment produced a typically observed sharp basal-type sheet texture in which the basal plane of the HCP lattice lies parallel to the sheet plane. The texture intensity exhibited a maximum of 16 multiples of random distribution (MRD). By comparison, the rolled and annealed Mg-1Gd sheet showed a much weaker texture (6 MRD) with a similar basal characteristic.

3.2. PSC flow behavior

Fig. 2a shows the obtained stress-strain curves for pure Mg and Mg-1Gd specimens during IPC deformation up to 6% logarithmic true strain. For the sake of clarity, the starting sheet textures shown in Fig. 1 were replotted in Fig. 2b and c with respect to the IPC deformation geometry. They indicate that the majority of grains were favorably oriented for deformation in c-axis extension. The initial ‘non-linearity’ observed in the flow curves is often seen in PSC stress-strain curves [8,13], and is possibly a result of compliance effects between the plunger of the channel-die and the sample. The ‘concave-up’ character seen in both flow curves arises from the operation of mechanical twinning and related orientation change.

In comparison to pure Mg, the Mg-1Gd alloy exhibits a much delayed onset of twinning (at ~ 3.5% vis-à-vis ~ 1.5% for pure Mg) occurring at higher flow stress values (~ 150 MPa for Mg-1Gd vis-à-vis ~ 75 MPa for pure Mg). Correspondingly, the change in strain hardening rate related to twinning also displays a delayed onset in the Gd

Fig. 1. Rolled and annealed optical microstructures and textures for a) pure Mg and b) Mg-1Gd from ref. [13].

containing sample relative to the pure Mg sample. The initial strain hardening rate marked by deformation strains below $\sim 1.5\%$, was considerably higher for pure Mg as compared to the Mg-1Gd alloy, which is most likely due to a more pronounced texture in the former.

3.3. Deformation and annealing textures and microstructures

Fig. 3a and b show the optical microstructures and XRD bulk textures for pure Mg and Mg-1Gd specimens after PSC deformation, and annealing at 350°C and 450°C for 60 min, respectively. In IPC deformation, loading was applied along the LD, almost perpendicular to the c -axis of the initial crystal to trigger $\{10\bar{1}2\}$ extension twinning. Hence, the (0002) recalculated pole figures of the deformed Mg specimens showed a drastic change in the starting texture, characterized by a rotation of basal poles from LD towards the center of the pole figure (CD). The resulting texture comprised two texture components, which led to a decrease in maximum texture intensity from an initial 16 MRD (multiples of random distribution) to 10 MRD (Fig. 3a). On the other hand, the Mg alloy containing Gd seemed to be less prone to texture changes during deformation since the PSC texture development showed a similar characteristic to that of the initial sheet texture accompanied by slight strengthening of the initial LD texture component (6 MRD to 9 MRD). Additionally, new orientations separated by $20\text{--}30^\circ$ from the predominant LD orientation were evident, yet with low intensities close to 3 MRD (Fig. 3b).

In accordance with the deformation texture, the microstructure of pure Mg upon 6% PSC remained coarse grained and showed a high proportion of deformation twins that were in part fully grown and already consumed their parent grains. For the Mg-1Gd alloy, deformed at a comparable strain, the deformation microstructure appeared to have a

higher level of inhomogeneity characterized by localization of plastic flow in banded morphology, and the presence of small fragmented grains. The area fraction of deformation twins was significantly lower (33.8% for pure Mg vis-à-vis 7.95% for Mg-1Gd) and the twins observed were comparatively thin and small (Fig. 3b).

Annealing treatments on pure Mg at 350°C and 450°C imparted considerable strengthening of the basal texture (18 MRD) accompanied by massive grain growth (Fig. 3a). By contrast, annealing of Mg-1Gd gave rise to a weaker texture (4 MRD) with a higher orientation spread around the same texture components seen in the deformation texture. At 350°C the microstructure indicated represented a partially recrystallized state comprising of recrystallized zones interspersed with twinned regions (Fig. 3b). Annealing at higher temperatures (450°C) was marked by complete recrystallization and subsequent grain growth. The microstructures indicate grain growth to occur discontinuously indicated by the presence of comparable proportions of fine ($\sim 8\ \mu\text{m}$) and coarse ($\sim 50\ \mu\text{m}$) grains (Fig. 3b).

3.4. EBSD characterization

For statistical orientation analysis of deformed and recrystallized grains and the characterization of twin types and fractions, EBSD measurements were performed on several deformed and annealed specimens. Fig. 4 shows EBSD data obtained for Mg-1Gd for the as-deformed condition. The original raw EBSD data was cleaned by careful noise reduction applying a minimum of 5 indexed nearest neighbors. The remaining zero solutions are shown here in black. The orientation map in Fig. 4a is presented in terms of inverse pole figure (IPF) coloring with respect to the CD. Grain boundary misorientation exceeding 5° are highlighted in black. The whole sampled area showed a very

Fig. 2. a) Flow curves for pure Mg and Mg-1Gd under plane strain compression (PSC); Starting textures for a) pure Mg and b) Mg-1Gd ([13]).

heterogeneous microstructure comprising of fragmented grains corresponding to the $\langle 11\bar{2}0 \rangle \parallel$ CD orientation shown in green and few large blue grains assuming $\langle 10\bar{1}0 \rangle \parallel$ CD orientation. Fig. 4b represents a unit cell mapping of the same region highlighting different twin types. The unit cells are shown in inverse pole figure coloring and oriented with respect to CD as the viewing direction. The characterization of twins was done on the basis of the characteristic misorientation angles and rotation axes (given in angle-axis pairs) of the most frequently observed twin types in Mg alloys, allowing an angular deviation of $\pm 6^\circ$. The identified twins in different colors revealed $\{10\bar{1}2\}$ extension twins (highlighted in red), $\{10\bar{1}1\}$ compression twins (blue) as well as $\{10\bar{1}1\} - \{10\bar{1}2\}$ double twins (green). Comparing the twinning behavior in the blue and the green regions from Fig. 4a and b, indicates that the latter gives rise to a high density of fine twins that seem to further fragment during deformation thus losing their characteristic twin morphology. In the blue parent, such fragmentation is only evident inside the compression and double twins. Fig. 4c presents the orientation spread of the matrix and twins corresponding to the blue and green colored regions in the microstructure. It is obvious that both areas exhibit a texture spread from LD to CD but only the blue colored regions of the map reveal a better coincidence of the single orientation data with the highlighted prismatic fiber that was correlated in a previous study with twin fragmentation [13,16]. The strain distribution for the microstructure in Fig. 4a is represented in terms of a grain reference orientation deviation (GROD) map. The distribution of GROD values is quite heterogeneous with the blue parent showing orientation spreads as high as $10\text{--}25^\circ$, indicating large plastic strain accumulation. On the other hand, majority of the green fragmented grains seem to be dynamically recrystallized, showing GROD values within 1° . Additionally few compression and double twins in the blue parent also evince GROD values less than 1° .

Considering that the imposed deformation should theoretically promote extension along the c-axis, analysis of $\{10\bar{1}2\}$ extension twin variant selection was performed on additional EBSD data acquired for Mg-1Gd in the as-deformed state. Fig. 5a and b shows representative areas corresponding to the blue colored $\langle 10\bar{1}0 \rangle \parallel$ CD and the green colored $\langle 11\bar{2}0 \rangle \parallel$ CD parent orientations, respectively. The orientations corresponding to the $\{10\bar{1}2\}$ twins observed in Fig. 5a and b, are shown in Fig. 5c. Interestingly, the blue parent grains give rise to two variants of extension twins (Fig. 5a) viz. ones shown in 'red' that assume a basal orientation (represented by red dots in Fig. 5c) and the 'light-green' colored variants having an off-basal orientation (shown as pale green squares in Fig. 5c). While the former is typical in commercial Mg alloys, the latter is an uncommon finding in the literature of polycrystalline Mg. Tension twins occurring inside the green-colored parent grains with $\langle 11\bar{2}0 \rangle \parallel$ CD orientation are colored in 'reddish orange' and correspond to the orange hexagons in Fig. 5c.

Fig. 6 shows the EBSD analysis for the Mg-1Gd alloy after annealing at 350°C . A region of interest, extracted from Fig. 6a, was investigated using EBSD. The mapping is presented in IPF coloring is shown in Fig. 6b. The recrystallized grains, exhibiting a vein-like morphology, are highlighted in the mapping. Fig. 6c shows a magnified view of the region highlighted in yellow (Fig. 6b). Misorientation analysis across the recrystallized grain boundaries exhibit a boundary misorientation within a range of $56^\circ \pm 6^\circ$ and a misorientation axis close to $\langle 11\bar{2}0 \rangle$, thus suggesting that these recrystallized bands might have originated from $\{10\bar{1}1\}$ compression twins. The corresponding unit cell mapping indicates that the recrystallized grains assume arbitrary orientations. Fig. 6d represents the single orientation scatter (0002) pole figure for the recrystallized subset. Unlike Fig. 4c, the recrystallized orientations are highly scattered and completely deviate from the prismatic fiber observed previously.

Fig. 7a and b show an IPF map for Mg-1Gd at 450°C , along with corresponding microtexture data measured by EBSD. The EBSD measurement indicates complete recrystallization succeeded by grain growth. The annealing texture reveals considerable weakening of the texture intensity accompanied with larger spread of orientations lying between LD and CD. The single orientation scatter, shown in Fig. 7b, is classified into regions of high ($SF > 0.3$) and low schmid factors ($SF < 0.3$) for basal slip. SF values were calculated for a compressive loading along CD and a constraint reactive stress, $\sigma_{TD} = 0.5\sigma_{CD}$. The average grain sizes corresponding to high and low SF orientations were $95.7\text{ }\mu\text{m}$ (Standard error (S.E) = $0.68\text{ }\mu\text{m}$) and $75\text{ }\mu\text{m}$ (S.E = $0.66\text{ }\mu\text{m}$), respectively. The scatter reveals relatively larger densities for grain sizes associated with the high SF orientations as compared to the low SF ones. The measured average grain size (c.f. Fig. 7c) for all grains was $89.17\text{ }\mu\text{m}$ (S.E = $0.65\text{ }\mu\text{m}$). The EBSD texture data shows good agreement with the XRD textures (c.f. Fig. 3b).

EBSD data for pure Mg in the as-deformed state is shown in Fig. 8. The original indexing fraction was 69%, which was subjected to careful cleaning with the noise reduction function explained earlier. Fig. 8a and b describes the respective grain orientations in IPF coloring and unit cell representation. Boundaries highlighted in red indicate the $\{10\bar{1}2\}$ tension twins. With respect to variant selection, a majority of primary tension twins originating from the $\langle 10\bar{1}0 \rangle \parallel$ CD (blue grains) or $\langle 11\bar{2}0 \rangle \parallel$ CD (green grains) parent orientations were associated with a single variant, depicted by 'red' and 'reddish-orange' orientations (c.f. Fig. 8a and b). Many grains displaying a basal/near-basal orientation (in red/orange-red), most likely arise out of extensive growth of twins to an extent where they entirely consume the original parent grain. The measured twin area fraction was $\sim 34\%$ (c.f. Fig. 8a). Few of the 'reddish-orange' tension twins additionally revealed secondary twinning. Fig. 8a depicts selected regions showing evidence of second generation $\{10\bar{1}2\} - \{10\bar{1}2\}$ twinning, giving rise to off-basal orientations encircled in Fig. 8c. The (0002) single orientation scatter and recalculated pole figures indicate a predominance of basal type orientations apart from the softer orientation of second generation twin

Fig. 3. (0002) recalculated pole figures from XRD and optical microstructures after PSC deformation, and static annealing at 350 °C and 450 °C for a) pure Mg and b) Mg-1Gd ([13]).

variants. The basal SF map shown in Fig. 8d displays low SF values for basal slip in most grains, indicating an overall ‘hard’ deformation texture in terms of further dislocation slip. On the other hand, SF values corresponding to the regions undergoing second generation extension twinning indicate a strong favorability of basal slip (c.f. Fig. 8a and d). Slip traces corresponding to basal slip are additionally indicated for each grain in Fig. 8d.

The annealed microstructures of pure Mg, as determined by EBSD are reproduced in Fig. 9 (350 °C) and Fig. 10 (450 °C). Due to considerably coarse final grain sizes, obtaining a sufficiently large number of grains from singular EBSD measurements was tedious. Hence, multiple mappings were analyzed in order to obtain statistically valid grain size distribution. The EBSD microstructure after 350 °C and 450 °C anneals revealed a fully recrystallized structure with obvious indications of strong grain growth. The IPF mapping shown in Fig. 9a comprises of primarily red/red-orange grains indicating a predominance of basal and near basal orientations. Few tension twins visible in the microstructure are most likely due to preparation induced surface deformation. Fig. 9b represents the single orientation scatter and (0002) pole figures, indicating that the majority of orientations have their c-axes aligned with the compression direction. The grain size distribution presented in Fig. 9c, gives a mean grain size of 104 µm (S.E. = 0.47 µm). The basal SF mapping in Fig. 9d shows majority of grains assuming low SF (< 0.3) values for basal < a > slip. Fig. 10a, b and c represent the EBSD microstructures in terms of image quality (IQ), IPF and basal SF coloring for 450 °C annealing treatment. Fig. 10d represents the single orientation scatter and (0002) basal pole figures

measured by EBSD. The orientations primarily assume a basal type texture as depicted by the recalculated pole figure, hence maintaining the deformation texture. The EBSD micro-texture coincidentally reveals reasonable agreement with the measured bulk texture at 450 °C (c.f. Fig. 3a). The average grain size (c.f. Fig. 10e) at 450 °C reported as 152 µm (S.E. = 0.66 µm) is significantly larger than its counterpart Mg-1Gd alloy. The pancake grain morphology observed in Fig. 10a-c, with preferred elongation towards LD is most likely as a result of specimen geometry constraints on the grain growth behavior, wherein the specimen length is 3 times the specimen height (along CD).

4. Discussion

The focus of the current work is a better understanding of the differences in twinning behavior with and without the presence of RE. The results indicate that both twinning and related recrystallization behavior in Mg-RE alloys show significant distinctions from that observed for pure Mg. While pure Mg displayed a conventional mechanical behavior, described by formation of basal texture that further intensifies during recrystallization and grain growth, the behavior of Mg-1Gd alloy was highlighted by its resistance to develop a basal-type texture. The forthcoming sections discuss in greater detail the deformation (Section 4.1) and annealing (Section 4.2) characteristics in Mg-1Gd with emphasis upon the observed deviations from the conventional response exhibited by pure magnesium.

Fig. 4. a) Inverse pole figure (IPF) + image quality (IQ) map for as deformed Mg-1Gd (viewing plane along the compression direction); b) Unit cell representation of microstructures, depicted in IPF coloring; c) Single orientation scatter datasets for matrix and twin orientations corresponding to blue colored $\langle 10\bar{1}0 \rangle \parallel \text{CD}$ and green colored $\langle 11\bar{2}0 \rangle \parallel \text{CD}$ parent grains; d) Grain reference orientation deviation (GROD) map for PSC deformed Mg-1Gd, with recrystallized grains within threshold value of 1° . (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

4.1. Deformation characteristics of slip and twinning

On the basis of the as-deformed textures shown in Fig. 3, it is evident that the two materials do not experience the same activation of twinning and slip, despite the same loading conditions and a similar starting texture. Microstructures obtained in the as-deformed condition reveal stark differences in the deformation response of magnesium with and without the addition of Gd. Considering a starting orientation conducive for twinning and an applied strain of 6%, the as deformed state would traditionally favor $\{10\bar{1}2\}$ extension twinning as seen in case of pure Mg (Fig. 8). However, PSC deformation of Mg-1Gd was quite the contrary, with relatively lower twinning fractions and simultaneous activation of $\{10\bar{1}1\}$ contraction and $\{10\bar{1}2\}$ - $\{10\bar{1}2\}$ double twinning. It is proposed that while plane strain compression in pure Mg was primarily $\{10\bar{1}2\}$ extension twinning dominated, deformation in the Gd containing alloy gave rise to activation of multiple slip and twinning systems.

The EBSD microstructures (Figs. 4a, 5a and b) represent two distinct regimes of deformation features corresponding to the twinned blue colored $\langle 10\bar{1}0 \rangle \parallel \text{CD}$ coarse parent grains and the severely fragmented green colored $\langle 11\bar{2}0 \rangle \parallel \text{CD}$ grains. Such an obvious orientation based distinction between the grain sizes i.e. blue grains vs. green grains can be attributed to the processing history of the alloy, prior to channel die compression. Since rolling induces a basal-type starting texture (c-axes parallel to sheet normal), subsequent annealing would promote preferential nucleation of green grains with their $\langle 11\bar{2}0 \rangle$ parallel to the rolling direction (or CD in case of channel die deformation) over the blue grains ($\langle 10\bar{1}0 \rangle$ parallel to the rolling direction). This is primarily owing to the predominance of the highly mobile $30^\circ \langle 0001 \rangle$ grain boundaries during recrystallization. On the other hand, solute drag effects due to the presence of Gd atoms at the grain boundaries [17,18], along with short annealing times (~ 300 s) can severely retard grain growth, wherein the green colored grains with higher nucleation densities remain relatively smaller than the blue grains. Contrarily, annealing of pure Mg sheet would result in rapid

recrystallization and grain growth, giving rising to uniform grain coarsening. The relative differences in the final grain sizes after thermomechanical treatment corroborates such a behavior.

During subsequent in plane compression of Mg-1Gd, the heterogeneous grain size distribution plays a key role in deciding the local strain accommodation mechanisms. Due to the larger size of the blue grains, twinning activity is relatively dominant as compared to the smaller green grains that favor dislocation slip, and thereby further grain fragmentation. Slip trace analysis (Fig. 11c) in parent grains revealed majority of slip lines coincident with $\{10\bar{1}1\}$ plane trace in the green parent $\langle 11\bar{2}0 \rangle \parallel \text{CD}$, whereas the blue grains showed activation of multiple slip systems comprising of basal and prismatic $\langle a \rangle$ dislocations (Fig. 11a and b). Prismatic orientations ('cyan' colored grains) lying in between $\langle 10\bar{1}0 \rangle$ and $\langle 11\bar{2}0 \rangle$ showed traces corresponding to either pyramidal $\{10\bar{1}1\}$ or prismatic $\{10\bar{1}0\}$ planes (Fig. 11b and c).

The observations indicate that the choice of slip mode strongly relies on the grain orientation and size. Such a behavior seems counter intuitive considering both blue and green grains should theoretically possess the same Schmid factor for prismatic slip, yet the clear favorability of prismatic slip in the blue grains vis-à-vis the green grains is evident by the slip trace analysis (Fig. 11a and b), as well as the in-grain orientation spreads (c.f. region 1 in Fig. 11d). However, considering that the local stress states can significantly vary due to additional stress components arising from grain shape compatibility, the resolved shear stresses on the highest stressed planes may not be identical. Under unbalanced shears the grains will experience rotations similar to the schematic shown in Fig. 12, wherein the prismatic slip induced rotation in blue grains results in an increase in the Schmid factor of the active slip system (shown in red in Fig. 12a), and subsequently promotes further slip. For grains oriented as $\langle 11\bar{2}0 \rangle \parallel \text{CD}$, the rotation due to the primary slip system (Fig. 12b) is counter balanced by the secondary slip system (shown in black) causing no change of orientation. In other words, the absence of prismatic slip activity in the green orientation is

Fig. 5. IPF and unit cell mappings for representative areas corresponding to a) blue ($\langle 10\bar{1}0 \rangle \parallel \text{CD}$) and b) green ($\langle 11\bar{2}0 \rangle \parallel \text{CD}$) parent grains; c) Twin variant analysis depicting twin orientations originating from blue ($\langle 10\bar{1}0 \rangle \parallel \text{CD}$) and green ($\langle 11\bar{2}0 \rangle \parallel \text{CD}$) parent grains. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

ascribed to its greater stability than the blue orientation.

In case of the green grains, traces corresponding to pyramidal planes were observed (Fig. 11c). Concurrently, the in-grain orientation gradients corresponding to the green grains indicated rotation of the c -axes (c.f. region 2 in Fig. 11d), alluding to activation of pyramidal $I \langle c+a \rangle$ slip. It was shown previously that Gd solutes tend to exist as clusters that facilitate the formation of point defects and stacking faults on pyramidal plane, leading to enhanced dislocation generation [19–21]. In light of restricted basal and prismatic slip modes inside the green parent grains, activation of pyramidal $I \langle c+a \rangle$ slip becomes feasible. Crystal plasticity simulations of plane strain compression behavior of Mg-1Gd also indicate the necessity of $\langle c+a \rangle$ slip activation for the observed deformation texture development [13]. Activation of non-basal slip is further aided by the relatively smaller grain sizes corresponding to the green grains, whereby deformation transitions from twinning dominated to a combination of twinning and slip.

Twinning characteristics in Mg-1Gd alloy were quite contrasting in comparison to that observed in pure Mg. Theoretically a grain can tensile twin on any of the six possible $\{10\bar{1}2\}$ planes, giving rise to 6 twin variants with 3 crystallographically equivalent $\langle 11\bar{2}0 \rangle$ rotation axes. However, in practice only those variants would form, for which the

resolved shear stress on the twin habit plane along the $\langle 10\bar{1}1 \rangle$ shear direction exceeds a critical value. In such conditions, it can be expected that the twin variant with the highest SF value will be preferentially chosen. Comparing the SF values for different variants (Fig. 5c), the red twin orientations of the blue parent assume an SF value of 0.499 vis-à-vis 0.12 for the low SF twin orientations. Similarly for the green parent, the high SF red-orange twins corresponding to a value of 0.437 are considerably higher than the other twin variants with SF equal to zero.

In case of pure Mg such variant selection is quite obvious from the IPF mapping and single orientation scatter data (c.f. Fig. 8a and c), wherein the majority of grains are colored as 'red' or 'red-orange'. Comparing with the calculated ideal twin orientations in the (0002) pole figure (Fig. 5c), it is clear that the 'red' and 'red-orange' twins correspond to the high SF variants originating from the 'blue' $\langle 10\bar{1}0 \rangle \parallel \text{CD}$ and 'green' $\langle 11\bar{2}0 \rangle \parallel \text{CD}$ parent grains, respectively. On the other hand, the low SF variants are completely absent. Contrary to pure Mg, Mg-1Gd reveals many large twinned grains comprising of more than one set of twins of different orientations (Fig. 5a). The appearance of multiple twin orientations ('red' and 'green' twins) inside certain matrix grains (corresponding to 'blue' $\langle 10\bar{1}0 \rangle \parallel \text{CD}$ in Fig. 5a) thus indicates that the resolved shear stresses for more than one twin variant

Fig. 6. a) Optical microstructure revealing still deformed parent and large twins along with bands of recrystallized grains for annealing Mg-1Gd at 350 °C for 60 mins; b) IPF mapping from selected area in Fig. 6a highlighting the bands of recrystallized grains; c) IPF and unit cell maps of a magnified recrystallized band showing the misorientation profile across the recrystallized – deformed interface; d) Single orientation scatter pole figure corresponding to the recrystallized subsets indicating appearance of orientations falling outside the prismatic fiber observed in Fig. 4c.

Fig. 7. a) IPF map and, b) (0002) recalculated pole figures and single orientation scatter for Mg-1Gd annealed at 450 °C for 60 mins. Circles in single orientation scatter indicate regions with (0001) $\langle 11\bar{2}0 \rangle$ basal schmid factor values greater (High S.F.) and smaller (Low S.F.) than 0.3; c) Grain size distribution for Mg-1Gd annealed at 450 °C for 60 mins.

Fig. 8. a) IPF and b) Unit cell representation for as deformed pure Mg; c) Single orientation scatter and (0002) recalculated pole figures highlighting $\{10\bar{1}2\}$ and secondary $\{10\bar{1}2\} - \{10\bar{1}2\}$ tension twins; d) Basal schmid factor map along with basal slip traces and corresponding stress tensor for schmid factor calculation. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

exceeded the critical twin nucleation stress. Furthermore, both red and green twins showed comparable volume fractions (Fig. 5a). The twins associated with the green parent, however, displayed activation of only the high SF variants described by the ‘red-orange’ orientations (Figs. 4a and 5b).

Typically twin variant selection behavior is dependent on the local stress-state of the twin nucleation and the accommodation strains involved during twinning associated atomic shuffling and shearing events [13,22–24]. With respect to the local stress state, the influence of lateral stresses arising from the channel die walls are important to consider. It must be understood that lateral stresses during channel die deformation strongly vary with the crystal orientation and can reach sufficiently high values, becoming comparable to the applied compression stress (σ_{CD}). For a completely isotropic case i.e. texture free material, the lateral stress can be assumed as $\sigma_{TD} = 0.5\sigma_{CD}$. With that, the calculated high and low SF values corresponding to $\langle 10\bar{1}0 \rangle \parallel CD$ become equal to 0.499 and 0.312, as opposed to the ideal values of 0.499 and 0.12. In a polycrystalline sample grains with diverse orientations will hence experience different lateral stresses. Considering the relatively weaker texture of Mg-1Gd, as compared to pure Mg, the tendency of variant selection would be abated in the former case.

Twin variant selection is also dependent upon the capability of local strain accommodation during twin nucleation and propagation. Strain accommodation in the twin vicinity is typically carried out by means of dislocation slip. Evidence of large amount of prismatic and pyramidal

slip traces in the matrix grains (Fig. 11) suggest the likelihood of significant dislocation activity and thereby easier strain accommodation. Assuming easier prismatic and pyramidal dislocation slip and a more homogeneous deformation in Mg-1Gd, the typical variant selection behavior would be modified, giving rise to appearance of both high and low SF twins. Contrarily in pure Mg, absence of homogeneous slip deformation in the twin vicinity leads to strong variant selection, whereby only the high SF twins that require lower accommodation work can form.

Despite the fact that most of the grains were initially oriented for extension twinning during IPC loading of Mg-1Gd, considerable amount of $\{10\bar{1}1\}$ compression and $\{10\bar{1}1\} - \{10\bar{1}2\}$ double twins were detected from the EBSD measurements, producing soft grain orientations lying between LD and CD in the (0002) pole figure. The feasibility of such twinning behavior can be macroscopically understood by the relatively weaker starting texture and the observed TD spread (Fig. 2c). In such case, grain orientations deviating from the ideal $\langle 0001 \rangle \parallel LD$ will experience a compressive stress component along their c-axes due to the die walls. However, even if such state existed for some grains, the significantly higher nucleation stresses for compression vs tensile twins, renders such an observation quite unusual. To discuss this in more detail, the effect of Gd-alloying on such deformation behavior needs to be taken into account. Solute Gd atoms present in the magnesium matrix can potentially modify the twinning response in the following ways:

Fig. 9. a) IPF representation for pure Mg annealed at 350 °C for 60 mins; b) Single orientation scatter and (0002) recalculated pole figures; c) Grain size distribution for pure Mg annealed at 350 °C for 60 mins; d) Basal schmid factor map along with basal slip traces and corresponding stress tensor for schmid factor calculation. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Fig. 10. a) IQ and b) IPF representation for pure Mg annealed at 450 °C for 60 mins; c) Basal schmid factor map; d) Single orientation scatter and (0002) recalculated pole figures; e) Grain size distribution for pure Mg annealed at 450 °C for 60 mins.

Fig. 11. Slip trace analysis on selected regions in as deformed Mg-1Gd corresponding a) blue ($\langle 10\bar{1}0 \rangle \parallel CD$) parent grains, b) 'cyan' colored parent grains lying in between $\langle 10\bar{1}0 \rangle - \langle 11\bar{2}0 \rangle$ and c) green ($\langle 11\bar{2}0 \rangle \parallel CD$) parent grains; d) Single orientation scatter displaying orientation spreads observed inside blue ($\langle 10\bar{1}0 \rangle \parallel CD$) grains (region 1 in Fig. 10a) and green ($\langle 11\bar{2}0 \rangle \parallel CD$) parent grains (region 3 in Fig. 10c). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Fig. 12. Schematic showing prismatic slip induced rotation in a) blue ($\langle 10\bar{1}0 \rangle \parallel CD$) parent grains and b) green ($\langle 11\bar{2}0 \rangle \parallel CD$) parent grains during plane strain compression of Mg-1Gd. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

a) Atomic size difference of Mg and Gd: Twinning typically involves simultaneous shearing, by means of dislocation glide of twinning partials or zonal dislocations, and atomic shuffling mechanisms. The favorability of activating a twinning mode over another is, however, strongly determined by the activation energy of atomic shuffling, i.e. the lower the shuffling energy, the easier it becomes to activate twinning [25]. While compression twinning is mostly driven by shear, the formation of extension twins is shuffle dominated [13,26]. This explains the frequent appearance of $\{10\bar{1}2\}$ tensile twins in pure magnesium, also observed in the present study. For the binary Mg-1Gd alloy, the solute Gd atoms can be expected to have an influence on the aforementioned twinning mechanisms. Considering the relatively larger atomic size of Gd (~ 0.18 nm) in comparison to Mg (~ 0.16 nm), shuffling of Gd atoms during twinning would require higher activation energies, making it the rate controlling process during twin formation and propagation. Higher activation energies would also mean higher critical stresses for extension twin formation in Mg-1Gd as compared to pure Mg. Hence, the nucleation stresses for compression and tensile twins become comparable. The delayed onset of twinning in Mg-1Gd in comparison with pure Mg further substantiates the higher activation energies for atomic shuffling, and thus the formation of tensile twins in case of Mg-1Gd.

b) Formation of Gd solute clusters: Numerous studies indicate the tendency of Gd solute atoms to undergo short range ordering, and thus to exist as solute clusters or pairs [17,27–31]. These features can act as effective twin hardening agents that retard nucleation and growth of $\{10\bar{1}2\}$ tension twins. Also, owing to their considerably larger sizes these clusters can generate significant atomic misfits, thereby inducing parastic strengthening. Furthermore, the considerably larger shear modulus of solute Gd (~ 21.8 GPa) as compared to that of Mg (~ 17 GPa) [32] would also give rise to additional dielastic and stacking fault strengthening effects.

In addition to the aforementioned mechanisms, the influence of solute RE on the intrinsic and extrinsic stacking fault energies, and the

interaction between solute RE atoms and dislocations are also important with respect to the formation of compression twins in this alloy [13,19]. It is also worth mentioning that apart from the first generation tensile twinning, secondary $\{10\bar{1}2\} - \{10\bar{1}2\}$ twinning (secondary extension twins within primary extension twins) was observed in the deformed pure Mg specimens. Appearance of such twinning was primarily observed in the green $\langle 11\bar{2}0 \rangle \parallel$ CD parent grains. These double extension twins are usually associated with soft non-basal orientations that potentially can witness multiple twinning generation, as was previously reported for monocrystalline magnesium samples deformed in plane strain compression perpendicular to the c-axis [16]. The observations indicate the feasibility of engineering microstructures in polycrystalline pure magnesium that display twinning related ductility.

4.2. Recovery and recrystallization behavior during deformation and annealing

The annealing response of Mg-1Gd was clearly distinct from the typically observed behavior for conventional Mg alloys, whereas 350 °C and 450 °C annealing of pure Mg led to full recrystallization and substantial grain growth accompanied by an expected texture strengthening of the basal component. This is due to the well-known favorable growth of grains with $30^\circ < 0001 >$ grain boundaries that does not impart notable changes to the basal character of the deformation texture. In other words, the maximum intensity of the basal poles remains in the center of the basal pole figure but the basal plane can reorient itself in one of three directions, namely $(0001)\langle 10\bar{1}0 \rangle$, $(0001)\langle 11\bar{2}0 \rangle$ or form a fiber spanning between both components.

Annealing of Mg-1Gd led to an increase in the orientation spread resulting from deformation, and hence to an unusual texture weakening. Nucleation of recrystallization initiated preferentially inside compression and double twins. Instances of strain-free grains inside twins were seen readily during deformation (Figs. 4a and 13a) and after annealing at 350 °C (Fig. 6d). Additionally, static annealing at 350 °C revealed bands of recrystallized grains (Fig. 6c and d) arising from compression and double twinned regions, interspersed with deformed matrix grains and tension twins. The formation of recrystallized grains

is proposed to take place by means of extensive dynamic recovery aided by dislocation slip, whereby dislocations of high density are rearranged into low angle grain boundaries that eventually become high angle boundaries.

Upon subsequent static annealing, nucleation of recrystallization can also occur through bulging mechanism at the boundaries of compression and double twins to reduce their dislocation density by forming strain-free grains, as shown in Fig. 6b. It is noted that this mechanism has distinct nucleation and growth stages, separating it from the continuous twinning recrystallization mechanism that occur during deformation. The latter is namely characterized by progressive plastic rotation of subgrains about their c-axes due to the activity of prismatic slip within their respective twin bands. When the subgrain rotation reaches a sufficiently large boundary misorientation ($\sim 15^\circ$), the subgrains evolve into recrystallized grains with high angle boundaries (Fig. 13a). The resulting 'prismatic' fiber of recrystallized grains populating the (0002) pole figure in the deformed state (c.f. Figs. 4c and 13b) can thus be interpreted by consecutive cycles of dynamic dislocation recovery and subgrain rotation. This type of mechanism has been recently suggested in discussing the role of grain boundaries in extrusion texture formation during dynamic recrystallization of AM30 magnesium alloy [33]. It was also proposed for twinning fragmentation and dynamic recrystallization observed in exceptionally ductile magnesium single crystals deformed in plane strain compression at room temperature [16]. With ongoing recrystallization during progressive deformation, the actual rotation axes can deviate from the ideal $[0001]$ Taylor axis for prismatic slip, which can in turn cause a broadening of the fiber.

Interestingly, during annealing and static recrystallization, the fiber of dynamically recrystallized twin orientations is not retained. This is most likely due to the effect of discontinuous twinning recrystallization that leads to development of arbitrary orientations residing outside of the prismatic fiber (c.f. Fig. 6d). Such tendency to transform a well-defined texture component into a randomized texture pattern explains the observed texture weakening during the annealing treatments (c.f. Fig. 3b). With regard to the influence of annealing on pre-existing tension twins in Mg-1Gd, it can be seen clearly from Fig. 6b that 'red'

Fig. 13. a) Slip trace analysis inside compression twins in as deformed Mg-1Gd indicating concurrent prismatic slip induced crystal rotation and twin fragmentation; b) Corresponding prismatic fiber observed in recalculated (0002) pole figure.

tension twins still retain their lenticular morphology, indicating their resistance to lateral twin growth. This is believed to arise out of Gd-solute segregation to twin boundaries during the annealing treatment [31] but still requires further future work to confirm. At higher annealing temperatures (450 °C), favorable growth of orientations nucleating in compression and double twins with high SF for basal slip takes place at the expense of low SF regions pertaining to unrecrystallized tension twins and recovered matrix grains.

5. Conclusions

- Micro-addition of 0.156 at% Gd (~ 1 wt%) to magnesium altered the undesirable {10 $\bar{1}2$ } twin dominated deformation that commonly causes high levels of mechanical anisotropy and asymmetry during in-plane tension/compression or through-thickness tension/compression of rolled sheets.
- Instead of profuse {10 $\bar{1}2$ }-twinning activity that fully reorients the initial texture towards the loading axis, the Mg-1Gd alloy witnessed competitive twinning behavior of both tension and compression/double twins that were critical for altering the recrystallization behavior and obtaining a significant texture weakening during subsequent annealing treatments.
- Upon deformation, instances of strain-free grains were readily observed inside compression and double twins forming a characteristic prismatic fiber in the basal pole figure. The formation of these new grains within their respective twin bands is suggested to take place by progressive plastic rotation of generated subgrains about their c-axes due to the activity of prismatic slip.
- Upon annealing, the well-defined fiber of dynamically recrystallized twin orientations is transformed into a randomized texture pattern. This is most likely due to the effect of discontinuous bulging mechanism at the twin boundaries that leads to development of arbitrary-oriented recrystallization nuclei residing outside of the prismatic fiber component.
- Onset of grain growth in Mg-1Gd during annealing was not observed until 450 °C, most likely owing to impending impurity drag effects. It promoted favorable growth of recrystallization orientations originating inside compression and double twins at the expense of their parent grains and unrecrystallized tension twins. On the other hand, pure Mg exhibited typical recovery-based annealing response, highlighted by extensive growth of extension twins.

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