Original Article

Microstructure and texture characterization of Mg–Al and Mg–Gd binary alloys processed by simple shear extrusion

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\textbf{A B S T R A C T}

Microstructural and textural evolutions of pure Mg, Mg–2 wt% Al, and Mg–2 wt% Gd were investigated after extrusion and simple shear extrusion (SSE). Microstructural studies revealed that the grain size of all extruded samples decreased after 4 passes of SSE at 553 K (280 °C). In the fine-grained Mg–2Gd alloy, however, a duplex structure consisting of fine recrystallized grains and coarse unrecrystallized patches was formed. Although the 2.5 μm size of the recrystallized grains did not experience a significant change, the volume fraction of the coarse 15 μm-wide unrecrystallized patches decreased, and the overall microstructural homogeneity was improved after 4 passes of SSE. In Mg–2Gd, fragmentation of large grains and unrecrystallized patches encouraged the formation of HAGBs, while Mg–2Al did not experience significant changes in the fraction of HAGBs. Contrary to pure Mg and Mg–2Al alloy, Mg–2Gd alloy developed a new “rare earth texture component” with the (1 1 2 1) direction parallel to the extrusion direction in the extruded condition. However, all three materials developed the conventional “extrusion texture” after SSE.

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1. Introduction

Severe plastic deformation (SPD) techniques are known as effective tools for refining the grain structure and improving mechanical properties of metallic materials [1]. The limited deformation achieved by conventional thermo-mechanical processes of hot extrusion or rolling may lead to the non-homogenous refinement of the initial coarse structure of the material. As a result, the deformed structures usually have multi-modal grain size distributions. In the SPD processes, in contrast to the conventional forming operations, plastic deformation takes place without any changes in the cross-section of the samples. Therefore, processing can be repeated indefinitely resulting in large imposed deformations and exceptional structural refinement [2–4]. There has been a great deal of interest in using SPD methods to process Mg and its alloys. There are many reports on achieving fine- and ultrafine-grained microstructures in Mg alloys after deformation by these methods [5–7]. Grain refinement and textural modifications have been known as the main causes for improving the ductility of Mg alloys after deformation by SPD processes [8–11]. It has been demonstrated that by lowering the imposed strain rate and the strain per pass, it is possible to carry out SPD at lower temperatures [12,13]. Furthermore, application of back pressure can lead to more refined microstructures having a higher degree of homogeneity at lower temperatures and fewer passes [14–16].

Simple shear extrusion (SSE), as a novel SPD technique, was proposed and developed by Pardis and Ebrahimi (2009). Similar to some other SPD processes, SSE is currently considered as a bench-scale test for examining microstructural evolution and mechanical properties of severely deformed materials. The details of the testing arrangement and die design are explained elsewhere [17] and are only briefly described here. SSE is based on pressing the specimen through a direct channel with a specific shape. During the SSE process, the specimen is deformed gradually, while its cross-sectional area remains unchanged. The extent of plastic deformation per pass in SSE depends on the maximum distortion angle (α) in the middle of the channel and the level of applied back-pressure. The theoretical strain is equal to 1.15 in each pass of the SSE process with α = 45° [17]. Furthermore, the specific design of this process leads to the development of a back pressure to the samples passing through the SSE deformation channel, enhancing grain refinement effects of the deformation process [17–19]. The shear direction in the SSE method is reversed after passing the middle plane of the deformation channel. Recent research on the deformation of pure Cu using SSE method has revealed that this strain reversal influences the formation of high angle grain boundaries (HAGBs) and texture of the deformed samples [20,21]. It has been shown that the formation of high angle grain boundaries encourages hot ductility and superplasticity, while textural changes can influence the strength of Mg alloys [6,8,10].

In contrast to other SPD processes, many aspects of the material behavior such as microstructural and textural evolutions during SSE are still unknown. Accordingly, this study deals with the investigation of such aspects in pure Mg and two binary Mg alloys; one with 2 wt% Gd and the other one with 2 wt% Al after different deformation passes by the SSE method. Pure Mg was selected as a simple model material with hexagonal close packed structure. The binary Mg–2Gd alloy was selected, based on the results of previous studies on the high potential of Gd for grain refinement of Mg after plastic deformation [22–25]. Furthermore, the results of Mg–2Gd were compared with those of the Mg alloy containing 2 wt% Al, as the most common alloying element in magnesium alloys.

2. Experimental materials and procedures

This investigation involved pure Mg and two binary Mg alloys; Mg–2 wt% Al and Mg–2 wt% Gd. The actual chemical compositions of the alloys are given in Table 1. Pure Mg and two master alloys of Mg–30%Gd and Mg–50%Al were employed to prepare the Mg–2Gd and Mg–2Al alloys, respectively. The alloys were melted in a graphite crucible placed in an electrical furnace, under a covering flux to protect magnesium from oxidation. The melt was held at 1023 K (750 °C) for 20 min, mechanically stirred for 2 min, and then poured into a steel mold preheated to 473 K (200 °C). The cast billets of 42 mm diameter and 120 mm length were homogenized at 733 K (460 °C) for 6 h and extruded to 11 mm × 11 mm bars at 653 K (380 °C). The extrusion ratio was about 11.5 and the ram velocity was 5 mm min⁻¹.

The SSE billets having nominal dimensions of 10 mm × 10 mm × 30 mm were machined from the extruded bars. All samples were pressed at 553 K (280 °C) using an SSE die having a maximum distortion angle of α = 45°. A schematic representation of the SSE process is given in Fig. 1. Pardis and Ebrahimi have suggested four different routes for the SSE method [18]. In this study, repetitive pressing of the same sample was carried out with route C, in which the specimen is rotated 90° about the extrusion axis between consecutive passes. This route was applied because it has been found to be the most effective route in achieving homogenous refined microstructures [18]. Samples were wrapped in poly tetra fluoro ethylene (Teflon) tape to reduce friction, before being pressed at a speed of 0.6 mm min⁻¹ for 1, 2 and 4 passes. To extract the deformed samples from the die cavity and to prevent them from being held at high temperature for long time, three sacrificial samples of AA1050 aluminum alloy were used after each magnesium sample. The sacrificial Al samples drive the deformed Mg samples out of the hot deformation channel, while they remain there waiting for the next Mg sample to enter the channel. The presence of these Al samples also creates a back pressure on the deforming Mg samples, resulting in a more pronounced grain refinement. Hot compression tests were carried out on the samples machined along the extrusion axis at 553 K (280 °C) and initial

<table>
<thead>
<tr>
<th>Material</th>
<th>Al (wt%)</th>
<th>Gd (wt%)</th>
<th>Mg (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure Mg</td>
<td>–</td>
<td>–</td>
<td>99.9</td>
</tr>
<tr>
<td>Mg–2Al</td>
<td>1.93</td>
<td>–</td>
<td>Bal.</td>
</tr>
<tr>
<td>Mg–2Gd</td>
<td>–</td>
<td>2.05</td>
<td>Bal.</td>
</tr>
</tbody>
</table>
strain rate of 0.002 s⁻¹, using a screw-driven universal testing system equipped with a three-zone split furnace.

Optical microscopy (OM) and scanning electron microscopy (SEM) were used for microstructural examinations of the cross-sections parallel to the pressing direction of the extruded and SSEed billets. For optical observations, the specimens were polished with 0.3 μm alumina paste, followed by polishing on an abrasive-free microcloth. Etching was carried out using Nital 5% solution at room temperature. The macro texture evolution of the deformed samples was analyzed by X-ray diffraction. The measurements were performed using Cu Kα radiation at 50 kV with the sample tilt angle ranging from 0° to 90°. Electron backscattered diffraction (EBSD) was used to study the misorientation angles of grain boundaries and also textural evolution on the cross-sections perpendicular to the pressing direction. The specimen preparation procedure for EBSD involved grinding with 1200 grit SiC paper, polishing with 6, 3, and 1 μm oil-based diamond suspension, followed by a colloidal silica slurry and electro-polishing in a solution of 20% nitric acid and ethanol at room temperature, which provided a high quality surface finish for EBSD. Clemex image analyzer was used to obtain the grain size and volume fraction of the unrecrystallized grains.

3. Results and discussion

The optical microstructures of the investigated materials in the extruded condition are exhibited in Fig. 2. It is apparent that the extruded pure Mg has an equiaxed grain structure with an average grain size of about 35 μm. The microstructure of the Mg–2Al alloy is almost similar to that of pure Mg with the grain size of 16 μm in the extruded condition. This alloy has a single phase solid solution structure, as expected from the Mg–Al phase diagram [26]. The microstructure of Mg–2Gd, however, is in sharp contrast to those of pure Mg and Mg–2Al.
The Mg–2Gd alloy has a two-phase structure, consisting of Mg (α) solid solution and intermetallic particles. It has previously been shown by the authors that these particles have a typical composition of MgGd [24]. Another distinct feature of the Gd-containing alloy is the significant grain refinement, which has occurred after extrusion. Contrary to common alloying elements such as Al, used in Mg alloys, rare earth (RE) elements can segregate to the grain boundaries. This is due to the large size mismatch between RE and Mg atoms [27], resulting in very fine grain sizes in the extruded Mg–2Gd alloy, as compared to the Mg–2Al alloy.

It can further be inferred from Fig. 2 that the Mg–2Gd microstructure consists of two different regions; equiaxed recrystallized grains with an average size of about 2.5 µm, and highly deformed elongated patches of unrecrystallized grains in the form of 15 µm-wide bands. It seems that the extrusion strain of 2.44 and the deformation temperature of 653 K were not high enough to attain a fully recrystallized microstructure in the Mg–2Gd alloy; therefore, a pronounced bimodal grain structure has been achieved in this alloy after extrusion. It has been reported that the addition of Gd to Mg retards the nucleation of recrystallization [28,29]. It has also been reported that during thermo-mechanical processing, Mg-Gd alloys exhibit minimal dynamic recrystallization. In some cases, this could result in a non-homogenous microstructure after hot deformation of these alloys [24,30,31]. Therefore, it is expected that higher deformation temperatures and more severe deformations, achievable in SPD processes, may help attaining a more uniform recrystallized grain structure.

The optical microstructures of the materials after processing by SSE are exhibited in Fig. 3. The grain structure of pure Mg after processing by 1, 2, and 4 passes of SSE is shown in Fig. 3(a)–(c), respectively. The average grain size of the samples decreased with increasing the number of passes in such a way that it reached about 9 µm after 4 passes. The microstructural evolution of the Mg–2Al alloy is almost similar to that of pure Mg, as exhibited in Figs. 3(d) to 2(f). The grain size of Mg–2Al decreased from 16 µm in extruded condition to 10 µm after 1 pass of SSE. Further straining of the material during the subsequent passes reduced the grain size to 8.5 and 7 µm after 2 and 4 passes, respectively. The observed moderate grain refinement in both pure Mg and Mg–2Al alloy is attributed to their tendency for grain growth at the deformation temperature of 553 K (280 °C), which counteracts the grain refinement caused by severe plastic deformation. This kind of microstructural evolution has also been reported in other studies on Mg–Al alloys [10,32]. The microstructural evolution of Mg–2Gd after 1, 2, and 4 passes of SSE, depicted in Fig. 3(g)–(i), exhibits a different trend. It is evident that, while the recrystallized grain structure of the alloy remains almost unchanged, the unrecrystallized patches are depleted with increasing the number of SSE passes. The quantitative analysis of the volume fraction of unrecrystallized patches shows that it decreases from 18% in the extruded condition to 6% in the 4-pass SSE condition,
as shown in Fig. 4. A summary of the mean grain sizes and the corresponding standard deviations of the deformed alloys are tabulated in Table 2. As already mentioned in the previous section, the grain size of the tested samples was measured using the Clemex professional image analysis program according to the ASTM E112 standard.

As a support for the observed microstructural evolutions, hot deformation behavior of the extruded materials in the SSE channel was simulated by hot compression tests performed at the same temperature of 553 K (280 °C) and initial strain rate of 0.002 s⁻¹, prevailing in the SSE process. The results of compression test have previously been used to predict the deformation behavior of aluminum billets in the SSE process [17] processes. The compressive stress–strain curves are shown in Fig. 5. As can be seen, the flow curves of extruded pure Mg and Mg–2Al are similar to those typically observed in the hot deformation tests, in which recrystallization has been completed [33]. For pure Mg, the flow curve exhibits softening after a critical strain, typical of pure metals in which recrystallization and the consequent grain growth are likely to occur. In the Mg–2Al alloy, a steady-state behavior is observed. This phenomenon is due to the fact that dynamic softening mechanisms such as recovery and recrystallization are in equilibrium with work hardening mechanisms after reaching a maximum stress. The hot deformation behavior of the Mg–2Gd alloy, however, is indicative of a typical work hardening behavior that might have happened during the initial stages of recrystallization, in which some unrecrystallized grains are present. These findings are in agreement with the results of the microstructural observations.

For a more detailed evaluation of microstructural evolution during SSE, the Mg–2Al and Mg–2Gd samples were analyzed using EBSD, and the corresponding images are presented in Fig. 6. It should be noted that all EBSD maps were taken from the plane normal to the extrusion direction. The EBSD maps of Mg–2Al contain a mixture of fine and coarse grains with an average grain size of 8.5 μm after 2 passes of SSE. Further straining to 4 passes of SSE resulted in a finer and more recrystallized equiaxed structure with a more uniform grain size of about 7 μm. The respective quoted standard deviations of 6 and 4.5 mm for these conditions in Table 2 agree with these observations. The EBSD images of the Mg–2Gd alloy after 2 and 4 passes of SSE show similar recrystallized microstructures with mostly equiaxed grains having an average grain size of about 2 μm. The presence of some unrecrystallized patches is evident in the EBSD maps of the Mg–2Gd alloy even after 4 passes of SSE, indicative of partial dynamic recrystallization of the Gd-containing alloy.

<table>
<thead>
<tr>
<th>Condition</th>
<th>Pure Mg</th>
<th>Mg–2Al</th>
<th>Mg–2Gd</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grain size (μm)</td>
<td>SD (μm)</td>
<td>Grain size (μm)</td>
<td>SD (μm)</td>
</tr>
<tr>
<td>Extruded</td>
<td>35.0</td>
<td>17.0</td>
<td>16.0</td>
</tr>
<tr>
<td>1 pass of SSE</td>
<td>15.0</td>
<td>13.0</td>
<td>10.0</td>
</tr>
<tr>
<td>2 passes of SSE</td>
<td>12.0</td>
<td>10.0</td>
<td>8.5</td>
</tr>
<tr>
<td>4 passes of SSE</td>
<td>9.0</td>
<td>6.5</td>
<td>7.0</td>
</tr>
</tbody>
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during SSE. These unrecrystallized grains have already been detected in the optical images, Figs. 3(g) to 2(i), taken along the pressing direction in the form of elongated patches.

In many fine-grained materials, in addition to the grain size, the nature of grain boundaries is of great concern, as it affects the deformation characteristics at high temperatures. The misorientation angle of grain boundaries can be extracted from the EBSD results. The accumulated strain of the material during consecutive passes of SPD processes can alter the type and distribution of grain boundaries. The evolution of high angle grain boundaries (HAGBs) and low angle grain boundaries (LAGBs) with deformation by SSE is exhibited in Fig. 7. The curves were created by a fitting process, using the data extracted from the EBSD results. The HAGBs and LAGBs are considered as boundaries with a misorientation angle $>15^\circ$ and $<15^\circ$, respectively [34]. The fully recrystallized microstructure of the extruded Mg–2Al alloy showed a higher fraction of HAGBs (78%), as compared to that of the extruded Mg–2Gd alloys (62%). This can be attributed to the presence of the unrecrystallized grains, which are deemed to be of low angle
nature. In the Mg–2Al alloy, the volume fraction of the HAGBs sharply decrease from 78% in the extruded condition to 45% after 1 pass of SSE, and then remains almost unchanged with further straining in the subsequent passes. Obviously, the volume fraction of the LAGBs shows an opposite trend, where it increases from 22% to 55% after 4 passes of SSE. In both materials, after the first pass of SSE, the fraction of LAGBs rapidly increases due to the formation of new grains. During the subsequent SSE passes, however, the grain boundary evolution of the Mg–2Al and Mg–2Gd alloys are significantly different. In Mg–2Gd, fragmentation of large grains and unrecrystallized patches encourages the formation of HAGBs, so that the volume fraction of HAGBs increases from 33% in the 1 pass deformed sample to 54% after 4 SSE passes. Similar results have been reported in other Mg alloys after processing by equal channel angular pressing (ECAP) [35,36]. The formation of HAGBs can be a result of continuous dynamic recrystallization (CDRX) during SSE processing. It has been recently proposed that the formation of micro shear bands (MSBs) is the possible mechanism for the CDRX in SSE [21]. This mechanism has also been reported in other SPD processes [37,38]. Fig. 8 shows the appearance of such MSBs within the grains after first pass of SSE. The presence of MSBs in all tested materials after the first pass of SSE means that the transformation of MSBs to HAGBs results in the grain refinement of the SSEed samples.

In addition to the refinement of the grain structure, deformation by SSE can alter the size and distribution of the second phase particles. Fig. 9 shows the distribution of the second phase particles in the Mg–2Gd alloy in the as-cast and extruded conditions, as well as after 4 SSE passes in the sections along the extrusion direction. According to the results of our previous work [24], these particles are Mg3Gd intermetallics, which are aligned in the extrusion direction after deformation. It is clear that SSE has significantly fragmented and refined the particles. It is well accepted that the second phase particles can act as obstacles or barriers to dislocation motion [39]. Therefore, these particles can enhance
Mg alloys processed by different SPD methods such as ECAP, high pressure torsion (HPT), accumulative roll bonding (ARB), etc. [40-42]. However, for the SSE, as a novel SPD process, the influence of multi-pass deformation on the texture of Mg alloys is unknown. Accordingly, the textural evolution of pure Mg, Mg–2Al, and Mg–2Gd alloys were studied in the extruded condition and after 2 and 4 passes of SSE. Fig. 10 shows the \{0002\} pole figures of the extruded and SSEed samples. The conventional fiber texture of basal planes is observed in the pole figures of all extruded samples. As can be observed, the maximum intensity of \{0002\} planes decreases with the addition of both alloying elements in the extruded condition. This behavior can be related to the smaller grain size and different recrystallization mechanisms in the presence of alloying elements [28,30]. Stanford and Barnett [28], have reported that the unrecrystallized patches in the extruded Mg–2Gd alloy develop the conventional basal texture, whereas the recrystallized grains develop a weaker texture with a new texture component. Therefore, developing a sharper texture in the extruded Mg–2Gd alloy in comparison with Mg–2Al alloy could be ascribed to the retention of the unrecrystallized patches in the microstructure of the extruded Mg–2Gd alloy. The \{0002\} pole figures of the SSEed samples show that the fiber texture of basal planes was destroyed specially after 4 passes of SSE, while the angles of \{0002\} planes remain unchanged. The annihilation of fiber texture in the SSEed samples could be attributed to the direction of applied shear stress and the nature of shear reversal in the SSE method.

The inverse pole figures obtained from the EBSD data, depicted in Fig. 11, can be used for a more detailed study of the texture components. These pole figures demonstrate statistical distribution of crystallographic planes parallel to the sample surface. It is evident that \{0002\} basal planes are mostly oriented parallel to the extrusion direction in the extruded pure Mg and Mg–2Al alloy. In this state, the prismatic 1010 planes are inclined at an angle of 90° to the extrusion direction. Achieving this kind of fiber texture is common in the extruded magnesium alloys [24,28]. The extruded Mg–2Gd alloy is an exception in the investigated materials. This alloy shows an additional peak between \{0002\} and 1210 in the inverse pole figure. This peak, which shows the \{1121\} direction, is aligned parallel to the extrusion direction, being known as the “RE texture component” in the literature [28]. It has been reported that solute partitioning of Gd to dislocations and grain boundaries, and shear band nucleation are responsible for developing the observed modified texture in the Mg–Gd alloys [28,30,43].

Examination of the inverse pole figures of SSEed samples revealed that all of the investigated materials developed the conventional “extrusion texture”, usually observed in Mg alloys. This is evident in all samples, even in the Mg–2Gd alloy that had already shown a different texture component in the extruded condition. In the Mg–2Gd samples the basal planes are also aligned along the extrusion direction after SSE, in spite of having an initial texture different from pure Mg and Mg–2Al samples. It could be deduced from these results that, unlike some of the other SPD methods such as ECAP, SSE does not change the conventional texture of Mg alloys. Development of conventional texture in Mg alloys leads to mechanical anisotropy, which is not desirable in some
applications. Moreover, it is believed that weakening of the conventional texture enhances the mechanical properties of Mg alloys [43]. It has been shown that basal planes are rotated to approximately 45° from the extrusion axis during ECAP. This is believed to be caused by shearing parallel to the basal planes [44]. It has also been reported that there is a clear distinction between the shear directions in the SSE and ECAP processes [18]. The shearing of a hypothetical cubic element in SSE and ECAP is schematically presented in Fig. 12. It can, thus, be proposed that the difference between the direction of applied shear stress in SSE and ECAP leads to dissimilar textual evolutions in Mg alloys after deformation by these two SPD methods.

The presence of a strong texture peak at 12 10 is noticeable in the inverse pole figures of pure Mg after 2 and 4 passes of SSE. The activation of different slip systems determines the preferential orientation of the grains during hot deformation processes. The formation of (12 10) texture could have been caused by the activation of pyramidal slip system during the deformation of pure Mg by SSE. This argument is in accordance with the previous results, in which the same texture component has been formed by the activation of virtual {1120} <10 10> slip system rather than the prismatic slip system [5,45]. On the other hand, the inverse pole figures of Mg–2Al and Mg–2Gd alloys after SSE show the strong texture peak at (10 10), which confirms the activation of the prismatic slip system in the above-mentioned alloys, especially after 4 passes of SSE. The activity of non-basal slip systems depends on the test temperature and the amount of solute atoms. Addition of alloying elements can alter the value of c/a ratio in Mg alloys. It is believed that non-basal slip can hardly occur at large c/a ratios [46,47]. It has also been

Fig. 10 – {0002} pole figures of extruded and SSEed samples.
reported that addition of Al and Gd increases the c/a ratio in Mg alloys [45,48]. This is believed to be the main reason for the shift in the position of the strongest peak in the inverse pole figures of the tested Mg alloys, compared to that of pure Mg.

4. Conclusions

Microstructural and textural evolutions in the Mg–2Al and Mg–2Gd alloys were investigated and compared with that of pure Mg after conventional extrusion and simple shear extrusion (SSE). The following conclusions are drawn from this study:

- The as-extruded microstructure of pure Mg and Mg–2Al contained equiaxed grains with the respective grain sizes of 35 and 18 μm, while that of Mg–2Gd consisted of very fine equiaxed-recrystallized grains with an average size of about 2.5 μm, and some unrecrystallized grains in the form of 15 μm wide bands.

- Processing by SSE significantly refined the microstructure of pure Mg and Mg–2Al, to, respectively, 9 and 7 μm after 4 passes, whereas it had minute effects on the size of the recrystallized grains in Mg–2Gd. On the other hand, SSE reduced the volume fraction of the unrecrystallized grains, resulting in a more uniform microstructure.

- The extruded pure Mg and Mg–2Al alloy developed the conventional extrusion texture, implying that the basal planes are preferentially oriented parallel to the extrusion direction. In the Mg–2Gd alloy, however, a new texture component with the ⟨1121⟩ direction parallel to the extrusion direction was developed.

- All of the investigated materials developed conventional extrusion texture after 2 and 4 passes of SSE. However, the position of the strongest peak in the texture of pure Mg was different from those of Mg–2Al and Mg–2Gd.

- Adding small amounts of Gd to Mg enhances strength due to grain refinement and particle hardening. The observed textural changes can reduce anisotropic behavior. These parameters results in improved properties, making Mg-Gd
alloys more interesting materials, as compared to Mg-Al alloys.

**Conflicts of interest**

The authors declare no conflicts of interest.

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