Microstructure and Texture Evolution of Magnesium alloy after Shear Assisted Processing and Extrusion (ShAPE™)

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Abstract. Shear Assisted Processing and Extrusion (ShAPE™) is a novel procedure used for fabricating thin-walled magnesium tubing. The technique involves pressing a billet through a rotating sleeve, thus causing severe deformation in the metal as the tube forms. This study analyzes the microstructure and crystallographic texture of the alloy in three different states by using electron backscatter diffraction. Notable differences in grain size and texture were observed depending on the location examined within the extrusion. Wall thicknesses of 1.5 and 3.0 mm (0.06 and 0.12 in) were characterized showing that both the grain size and basal plane orientation changed significantly as a function of position through the wall thickness, with abrupt changes near the outside wall. This type of microstructural information can be used to tune the parameters of the forming process to obtain the desired properties.

1. Introduction
With a view to fuel efficiency, the demand for materials with a high ratio of strength to weight increases significantly [1-3]. To this end, magnesium alloys are excellent candidates to be substituted for steel and other metal alloys due to their exclusive mechanical properties such as the lowest density as a metal, noticeable strength, suitable for casting and machining, great damping characteristics, high thermal stability, conductivity, and recyclability [3-4]. However, the lack of sufficient slip systems because of the HCP crystal structure causes restricted ductility and toughness at room temperature [5]. Thus, one of the main obstacles to achieve this goal is forming while optimizing the microstructure to obtain strength and ductility [6]. Recently, severe plastic deformation (SPD) has been used to alter grain size and texture to gain superior mechanical properties [7]. SPD can be applied by different techniques, for instance, shear force in high-pressure torsion [8], compression force in rolling [9], or equal channel angular pressing (ECAP) [10], among others. Size and cost of the product are two important challenges required to be addressed to turn these into appropriate metal forming processes in industry [11-12]. Indirect shear extrusion has been utilized to overcome tension-compression yield strength asymmetry in ZK60 and other magnesium alloys [13,3]. Recently, Shear Assisted Processing and Extrusion (ShAPE), a new direct shear extrusion procedure, has been applied on ZK60 for manufacturing large pipes [12]. It is important to investigate the microstructure along with its temporal and spatial variability in such processes. Electron backscatter diffraction (EBSD) is a robust technique that can be leveraged to quantify grain size, crystallographic texture, grain boundary structure, and deformation character in such materials [14].
In this paper, the gap between the die and the mandrel (wall thickness) and the number of spiral scrolls (starts) on the die shoulder were varied to determine their effect on the grain size, crystallite lattice orientation, and defect structure for specimens processed using the ShAPE procedure. Hence, three states were investigated, 1) 4 starts and 3 mm (120 mil) wall thickness, 2) 4 starts and 1.5 mm (60 mil) wall thickness, 3) 16 starts and 1.5 mm wall thickness. For each state, at least two samples were examined in different locations along the tube length to ensure sample uniformity.

2. Experimental procedure

2.1. ShAPE procedure
ShAPE is a direct extrusion procedure that applies shear loading for optimizing the grain size and texture. Figure 1 contains a schematic picture of the ShAPE system, in which the extrusion die is fixed and other parts rotate at 200 RPM. The actuator pushes the rotating billet and applies axial force and torque to the extrusion die shoulder while the applied torque, the rotational speed, and the chilled water flow rate through the mandrel control the generated heat. At the critical point, the area of billet touching the extrusion die softens and plastically flows through the spiral scrolls on the extrusion die. According to the combination of applied forces and thermal conditions, a strong gradient in crystallographic texture has been obtained. The texture depends on the thermal conditions, rotational speed, extrusion rate, and the number of spiral scrolls (starts) on the extrusion die. These quantities are expressed in Table 1[12]. In this work, eight specimens from different areas of the tube for three distinct states have been investigated. To study the effect of the wall thickness and number of starts as the two main parameters that control the microstructure in ShAPE, three states were defined. These are shown in Table 2.

| Processing Details          | Details           |
|-----------------------------|-------------------|
| Ram force                   | 40 kN (9000 lbs)  |
| Torque                      | 550 N-m (405 lb-ft) |
| Power consumption           | 11.5 kW           |
| Temperature 1 mm from die surface | 450°C          |
| k factor                    | 3.33 MPa          |
| Outside diameter of pipe    | 50.8 mm           |
| Feed rate                   | 3.81 mm/min       |

| State’s number | Number of starts | Thickness |
|----------------|------------------|-----------|
| 1              | 4                | 3 mm      |
| 2              | 4                | 1.5 mm    |
| 3              | 16               | 1.5 mm    |
2.2. Microstructural investigation
The specimens were cut and mounted in the longitudinal direction for the three states. They were mechanically ground and polished to a final step of 0.05 μm colloidal silica on a low nap cloth until the quality of Kikuchi patterns was acceptable. EBSD with an accelerating voltage of 20 KeV and a probe current of ~10 nA was used. For analyzing EBSD data, Orientation Imaging Microscopy (OIM) was leveraged to define grain size, misorientation angles, crystallite lattice orientation distributions, and recrystallization fraction [15]. Grain orientation spread (GOS) was used to obtain a measure of deformation in the grains and to estimate the fraction of recrystallization. GOS of less than 1.3 degrees was used to determine that the grain was recrystallized [16]. In this paper, the blue color represents the recrystallized grains in the GOS images.

3. Result and discussion
3.1. Microstructure analysis
As shown in Figure 2-a, the mass flow lines were created as a function of axial and shear force. These lines effectively show the material flow direction at each position in the structure. The change in grain shape orientations and crystallographic plane direction follows these flow lines. As shown Figure 2-a, the slope of these curves changes smoothly from the inside to the outside of the tube wall, while it abruptly comes back to the initial slope near to the outside edge. Although grains were refined to less than 10 μm, large grains were detected that created the bimodal structure (Figure 2-b). In the orientation map (Figure 2-a), the mass flow line can be observed by the orientation of the major axis of the elongated larger grains and by the path traced by the contiguous fine grains.
Figure 2 a) Optical micrograph of sample after final polishing step shows the mass flow line from the inside to the outside, b) Image quality map of EBSD near to the center.

In Figure 3, grain structures through the thickness of the three states are shown. As demonstrated, by increasing the number of starts and reducing the wall thickness, the number of large grains decreased. As a result, a more homogeneous structure was obtained except near the outside edge. The average grain size for the three states were 7.3 +/- 3.71 µm, 6.06 +/- 3.06 µm, and 5.94 +/- 3.0 µm, respectively. The error bars are determined by the standard deviation of the distribution. Large grains were detected in two different locations in all the states. The size of these grains was near 20 µm, approximately three times the average grain size, while there were very fine grains (1-3 µm) near these larger grains. Larger grains near the outside edge are created as a result of greater stored energy and higher temperatures in the structure due to frictional forces, allowing for more rapid grain growth. On the other hand, larger grains in the mid-thickness region occurred in conditions where lower stored energy was imposed, including tubes with thicker walls and for processing with lower expected mass flow (smaller number of spiral scroll starts). In tubes with the 1.5 mm wall thickness, the grain size distribution was generally more homogeneous.

Figure 3 Grain size histogram through the thickness a) 3 mm 4 starts b) 1.5 mm 4 starts c) 1.5 mm 16 starts
3.2. Texture Analysis

All calculations were done with respect to the radial direction. As shown in Figure 4-a, Basal poles were always perpendicular to the material flow line that gradually rotated due to the change of flow line slope. Thus, according to the majority of grain orientations, the thickness could be divided into five different regions each demonstrating a given texture (Figure 4-b). Although the first region demonstrated weak texture at the inner edge, the second region had a strong texture with basal poles lying along the radial direction. On the other hand, region 3 was a transition between regions 2 and 4 that had a c-axis fiber texture at 45 degrees while region 4 had a strong texture approximately along the extrusion direction. Finally, in region 5, a strong texture in a small area near the outside edge was observed where the crystallite lattices were rotated near 90 degrees with respect to those seen in region 4. The misorientation angle histogram for a randomly textured HCP metal is expected to follow that described by Mackenzie [17-18]. The ideal histogram for a perfect basal texture in this material has a constant frequency from 0-30 degrees and nothing outside of that range. The misorientation angle histograms in this structure were consistent with a random texture overlaid on a c-axis fiber texture in the region 1. In contrast, the disorientation distributions were similar and consistent with c-axis fiber textures in the rest of the structure. This pattern was revealed in all three states as observed in Figure 5-a except that in the 1.5 mm pipe, the grains in the extruded direction were eliminated and the region 2 grains grew by increasing the number of starts that led to the stronger texture seen in Figure 5-b.

![Figure 4](image_url)

**Figure 4** a) Orientation map through the thickness from inside to outside, b) five different texture conditions according to basal plane orientation, and c) misorientation histogram for defining the regions
Figure 5  a) Changing basal plane angle along the thickness for the three states, b) (0001) pole figure for 3 mm - 4 starts, 1.5 mm - 4 starts, and 1.5 mm - 16 starts, respectively

EBSD scans were also taken at two positions separated by 20 cm along the tube length. The results showed that as opposed to the radial direction, no significant change was observed, in crystallographic texture or grain size distribution, as shown in Figure 6.

Figure 6  Misorientation histogram, grain size distribution and pole figures ((0001) & (10-10)) at two different locations in the longitudinal direction

3.3. Recrystallization

Although more than 90% of grains were recrystallized as a result of dynamic recrystallization, the number of un-recrystallized grains increased near both edges. According to Figure 7-a all pipes thickness and number of starts did not have a considerable effect on recrystallization. Thus, no significant difference was observed in the three states (Figure 7-b). A closer look reveals that the un-recrystallized grains were larger grains that had been dynamically recrystallized and deformed again as the process progressed. This process was likely repeated in these regions through the entirety of the process. As mentioned, large grains were in two locations including those near the outside wall and those near the middle of the wall thickness. For the recrystallized larger grains near the outside wall, static grain growth likely occurred; therefore, they do not have in-grain orientation gradients.
Figure 7 a) Different recrystallization on large grains of two different locations, b) recrystallization through thickness in the three different states

4. Conclusion
Shear Assisted Processing and Extrusion (ShAPE) is a procedure that can easily be scaled up for cost reductions in such processing. ShAPE processing leads to grain refinement that gives a somewhat bimodal structure depending on processing parameters. The basal plane orientations change from 90 degrees to near zero with respect to the extrusion direction, through the wall thickness. However, raising the number of spiral scrolls and decreasing the wall thickness led to more uniform microstructures. Tuning the mentioned parameters successfully addressed the issue of the larger grains in the middle of the wall thickness. However, those near the outer wall thickness persisted, due to the increased strain energy and higher temperatures in these locations.

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