Deformation Heterogeneity on the Cross-Sectional Planes of a Magnesium Alloy Processed by High-Pressure Torsion

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Disks of an extruded AZ31 magnesium alloy were processed by high-pressure torsion (HPT) at a pressure of 6.0 GPa through 1/4, 1, and 5 turns either at room temperature (296 K (23 °C)) or at an elevated temperature of 463 K (190 °C). The cross-sectional planes of the disks were examined after processing, and it is shown that there is a high level of heterogeneity throughout all of the samples. This heterogeneity is revealed through the nature of the flow patterns, through the distributions of grain sizes, and by comprehensive microhardness measurements. The results demonstrate that it is possible to double the strength of the alloy in some areas of the disk after processing through 5 turns at room temperature.

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I. INTRODUCTION

SEVERE plastic deformation (SPD) processes have been studied extensively as convenient methods for the production of ultrafine-grained and nanostructured metals and their alloys.[1] Processing by SPD is generally imposed using one of three different basic techniques: accumulative roll bonding (ARB),[2] equal-channel angular pressing (ECAP),[3] and high-pressure torsion (HPT).[4]

Processing by ARB involves the cold rolling of sheets with a thickness reduction of about 50 pct per pass, cutting the samples transversally at their midlength between passes, and then placing these two halves on top of each other and rolling again. In practice, the success of ARB depends on an adequate bonding of the two samples during the cold rolling operation and, if successful, the process permits the production of materials having a sheet geometry. The most widely studied and fundamental SPD process is ECAP, where the sample is generally in the form of a rod or bar and a plane strain shearing is achieved inside a closed channel within a die. When processing by ECAP, high strains are attained by pressing through a series of successive passes. An important advantage of ECAP is that the process can be easily scaled-up for use with relatively large bulk materials.[5] Much recent attention has been devoted to HPT, because it generally produces greater grain refinement than ECAP[6] and the processed grains are often within the nanometer range, which is defined formally as grain sizes less than 100 nm.[7] In HPT, the sample is usually in the form of a thin disk that is subjected to a high imposed pressure and concurrent torsional straining. Some experimental results are now available where HPT was conducted using larger cylindrical samples.[8,9]

Processing by SPD was applied to a very wide range of materials. However, there is a special interest in using SPD processing for magnesium alloys, because these materials have an excellent strength/density ratio and there is a potential for achieving additional strengthening using SPD processing. In practice, however, it is now recognized that there are often significant difficulties in the processing of magnesium-based alloys through ECAP at low temperatures due to the availability of only limited slip systems, the nature of the grain refinement process, and the development of unstable flow and segmentation.[10–14] By contrast, it is relatively easy to use HPT to process magnesium[15,16] and its alloys[15] at room temperature without the introduction of any cracking into the samples.

HPT involves the deformation of thin metallic disks in torsion between massive anvils that also apply an axial pressure on the material. The applied pressure prevents the occurrence of cracking and, thereby, enables the processing of these difficult-to-work alloys. The similarity with conventional torsion tests permits an estimate of the deformation imposed to the sample in HPT using the following relationship:[17–19]

$$\varepsilon = \frac{2\pi N r}{h\sqrt{3}} \quad [1]$$

where ε is the imposed strain, N is the number of turns in HPT, r is the radius of the disk, and h is the disk thickness. According to this relationship, very high levels of strain may be imposed to thin samples by increasing the numbers of turns. The relationship implies also that deformation is homogeneous

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throughout the thickness of the sample and varies only in the radial direction.

From the point of view of the use of materials produced by HPT, the homogeneity of the final product is of critical importance. Nevertheless, recent reports have described unusual features in materials processed by HPT, including the development of agglomerates of Zn-rich and Al-rich grains near the edges of disks of the Zn-22 pct Al alloy[20,21] and the presence of unexpected flow patterns on the surfaces of disks of a duplex stainless steel[22–24] and a Cu-Ag alloy[25] processed by HPT including the formation of double swirls and shear vortices. These observations suggest there may be deviations from the ideal analysis used to estimate deformation in HPT through Eq. [1].

Most of the studies conducted to date to analyze the flow behavior in HPT concentrated on the distributions of hardness values on the disk surfaces, and usually there is no parallel investigation of the degree of homogeneity along the thickness direction. In practice, there are only a few recent reports where observations were conducted on cross-sectional planes at different distances between the top and bottom surfaces of the HPT disks,[8,26–28] and an examination of the published results reveals conflicting trends. For example, an analysis of the microstructures and the distributions of hardness at different positions along the thickness of disks of pure aluminum revealed a high degree of homogeneity in the thickness direction,[27] whereas microhardness distributions on sections along the axial directions of disks of pure iron revealed regions of higher hardness developing around the midplanes of the disks and with a degree of hardness heterogeneity that was dependent upon the ratio between the thickness and the diameter of the sample.[26] An analysis of the structure and hardness distribution of a bulk cylindrical sample of pure aluminum having a high thickness-to-diameter ratio revealed a pronounced heterogeneity along the axial direction.[8] Finally, a recent analysis of the cross sections of disks of the AZ31 magnesium alloy processed by HPT at a high temperature also revealed significant differences in the structure and hardness distributions along the axial direction.[28]

The present investigation, therefore, was initiated in order to analyze the heterogeneity of deformation on cross sections of HPT disks along the axial direction (hereafter, simply termed the cross section of the sample) of an AZ31 magnesium alloy processed through 1/4, 1, and 5 turns with the processing conducted both at room temperature (296 K (23 °C)) and at an elevated temperature of 463 K (190 °C). As will be demonstrated, the flow patterns, grain size distributions, and microhardness distributions may be used to reach conclusions on the degree of heterogeneity along the axial directions of the samples.

II. EXPERIMENTAL MATERIAL AND PROCEDURES

Experiments were conducted using a commercial AZ31 (Mg-3 pct Al-1 pct Zn) alloy supplied by Timminco Co. (Aurora, CO) in the form of extruded rods with a diameter of 10 mm and an average grain size of ~9.4 μm. These rods were sliced into disks with thicknesses of ~1.5 mm and then ground with abrasive papers to final thicknesses of ~0.8 mm. Each disk was processed by HPT under quasi-constrained conditions[4] either at room temperature (296 K (23 °C)) or at an elevated temperature of 463 K (190 °C). Heating of the samples to 463 K (190 °C) was achieved using heating elements embedded in the upper and lower anvils, and the temperature was controlled using a thermocouple placed within the upper stationary anvil. The time for heating to 463 K (190 °C) was ~15 minutes, and thereafter, the temperature was held constant during HPT processing to a temperature within the range of 463 ± 5 K. The imposed pressure was maintained constant during processing at 6.0 GPa, and separate disks were processed through totals of 1/4, 1, and 5 turns. Following processing, care was taken to identify the top and bottom surfaces of each disk.

After processing, the disks were sectioned along their axes and mounted in resin. These cross-sectional areas were ground with abrasive paper and polished with diamond paste, and a final polishing was conducted using a colloidal silica solution. The polished surfaces were etched to reveal the flow patterns and grain boundaries, and these surfaces were examined using optical microscopy.

Following the observations of the structure, the surfaces were repolished and microhardness measurements were taken at predetermined locations using a Vickers indenter. A rectangular grid of indentations was recorded using five indentations parallel to the top and bottom surfaces and five indentations perpendicular to these surfaces. The minimum distance between separate indentations was set at 100 μm in order to avoid interference between the measurements. For all measurements, the applied load was 100 gf and the dwell time was 10 seconds. The values of the Vickers microhardness, Hv, were determined as the average of five separate indentations and the average values were plotted as a function of the distance from the bottom surface of the disk.

III. EXPERIMENTAL RESULTS

A. *HPT at Room Temperature*

Figure 1 shows the appearance of the half cross section of the disks processed through 1/4, 1, and 5 turns of HPT at room temperature. Inspection shows that some areas within these samples exhibit features perpendicular to the top and bottom surfaces of the disks, whereas other regions display features parallel or inclined with respect to the faces of the disks. The areas near the centers of the disk, corresponding to the left-hand side in Figure 1, tend to exhibit perpendicular or inclined features, while the midplanes in areas away from the disk centers exhibit parallel features. It is also apparent that the flow patterns exhibit distinct appearances in areas at similar radial distances from the centers (corresponding to a horizontal displacement)

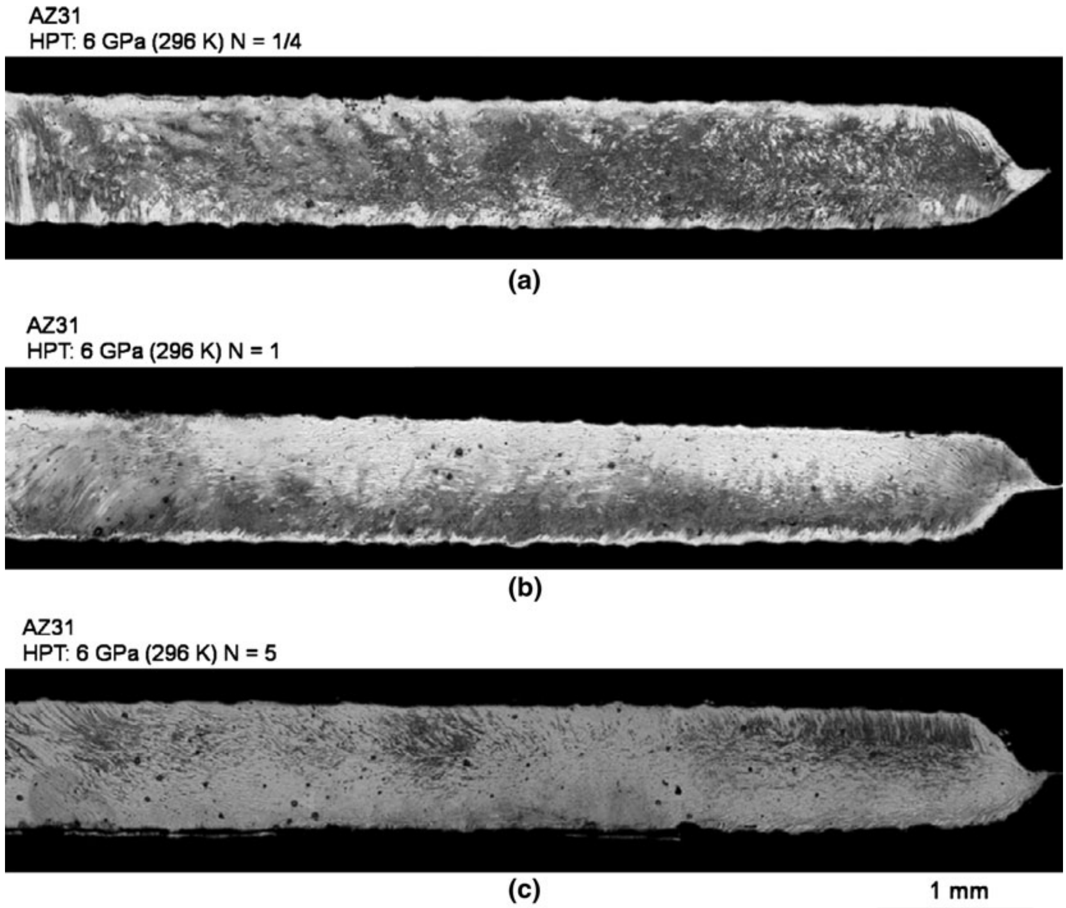

Fig. 1—Flow patterns in the cross-sectional planes of HPT samples after (*a*) 1/4, (*b*) 1, and (*c*) 5 turns at 296 K (23 °C).

but at different heights (corresponding to a vertical displacement), which suggests the occurrence of different deformation levels at various distances from the top and bottom surfaces of the HPT disks.

To extend these observations, Figure 2 shows higher magnification images at selected locations within the disk cross sections, where the flow patterns are clearly revealed; thus, the two upper images are for the disk processed through 1/4 turn at the center (on left) and at the edge (on right), and the two lower images are at the edges for the disks processed through 1 turn (on left) and 5 turns (on right), respectively. It is observed that the flow pattern is perpendicular to the disk surface at the center of the disk processed through 1/4 turn, and this is a characteristic feature of the as-received extruded material, thereby suggesting there was little or no straining in the center of the disk. By contrast, some areas near the edges of the disks, both after 1/4 turn and after 1 and 5 turns, exhibit structural features parallel to the disk surfaces, which are due to the torsional deformation imposed by HPT. It is also apparent that these areas are preferentially located near the midplanes of the disks for each processing condition. By contrast, some regions near the edges of the disks in the vicinity of the top and bottom surfaces exhibit flow patterns that are perpendicular or inclined to the disk surfaces, where this is again more characteristic of the as-received material. It is worth noting that such a situation is observed, for example, near the top surface at the edge of the disk processed through 5 turns despite the very severe torsional deformation imposed on the sample.

Figure 3 shows the distributions of hardness as a function of distance from the bottom surfaces at different radial locations within the disk cross sections for samples processed by HPT at room temperature through (a) 1/4, (b) 1, and (c) 5 turns. There are two conclusions from these results. First, the overall level of hardness increases with increasing numbers of turns in HPT so that the highest values are achieved after $N = 5$ turns. Second, these individual hardness measurements were recorded at the center of each disk and at radial positions of 1.3, 2.5, and 5.0 mm from the centers, where the latter position corresponds to the outer periphery of each disk.

Comparing the values obtained for the different radial positions, it is apparent that the hardness tends to increase with increasing distance from the disk center. However, the individual hardness values vary significantly with distance from the bottom surfaces. Thus, for the disk processed to 1/4 turn, the microhardness at the edge of the disk exhibits lower values near the bottom and top surfaces and higher values between these

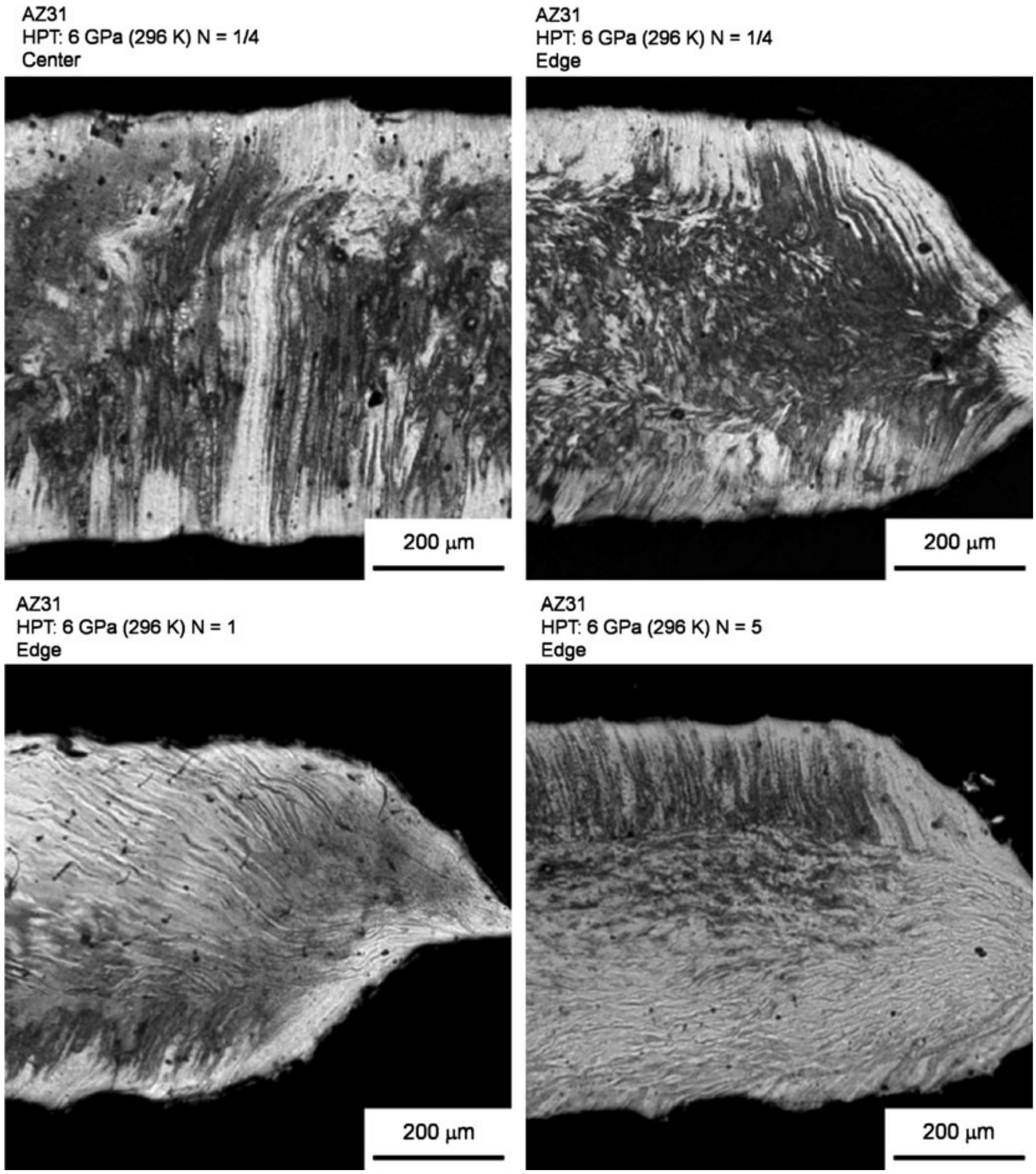

Fig. 2—Details of the flow pattern in the cross-sectional planes of HPT samples after 1/4, 1, and 5 turns at 296 K (23 °C).

surfaces; for the disk processed to 1 turn, there is a consistent higher hardness near the bottom surface than near the top surface; and for the disk processed to 5 turns, there is a sharp decrease in the hardness near the top surface. The conclusion from these data is that strain hardening is not constant at similar distances from the disk centers.

B. *HPT at an Elevated Temperature*

Figure 4 shows the appearance of the half cross sections of disks of the AZ31 alloy processed by HPT to 1/4, 1, and 5 turns at the higher temperature of 463 K (190 °C). The etching clearly reveals the grain boundaries on these cross sections, and areas having smaller grains appear darker. It is readily apparent that the structure in the through thickness (vertical direction) of the disks is not homogeneous. The sample processed by 1/4 turn exhibits a thin layer of finer grains which are darker and lie between the midplane and the bottom surface of the cross section. The sample processed by 1 turn exhibits an area of finer grains around the midplane of the cross section and a very distinct separation between areas of different grain size distributions, as demonstrated by the formation of an extensive shear band crossing the entire half cross section. The sample processed by 5 turns exhibits a finer grain structure near the bottom surface, alternate thin layers of finer and coarser grains through the middle volume, and a relatively coarser grain structure near the top surface.

The heterogeneous nature of these grain size distributions is evident in a higher magnification image taken near the bottom surface of the disk processed by 1/4 turn at a distance of 2.0 mm from the center, as shown

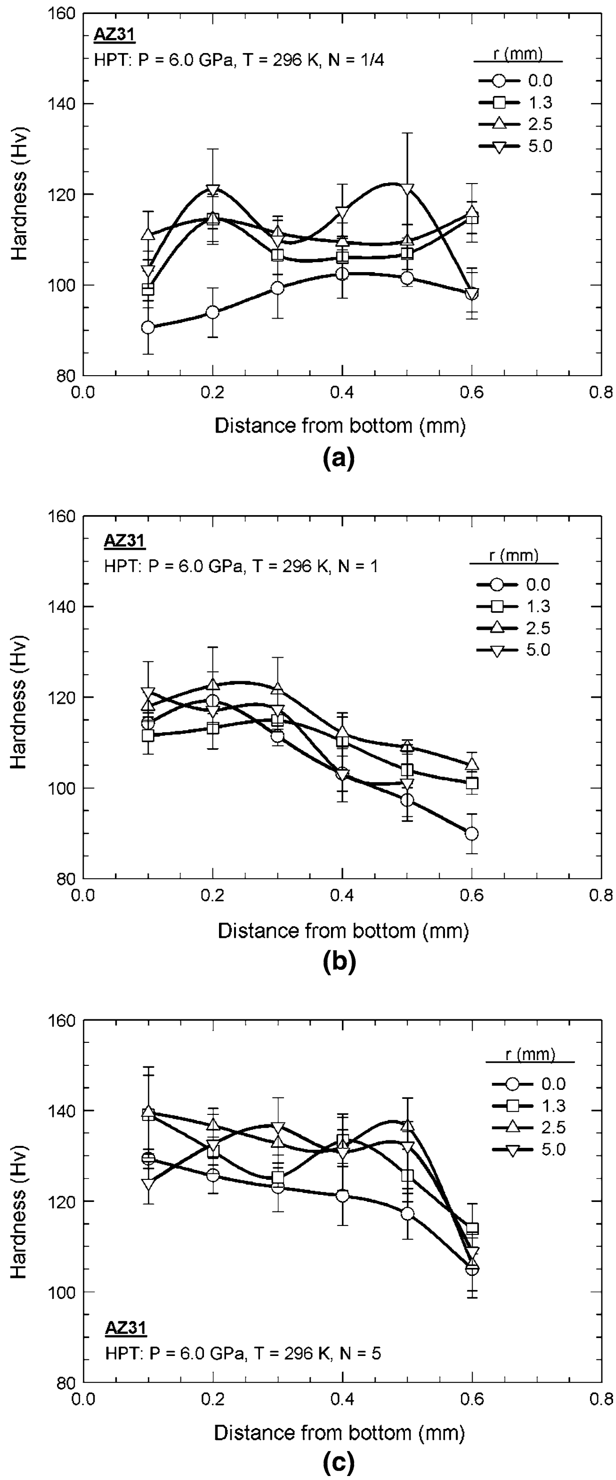

Fig. 3—Microhardness values at various radii as a function of the distance from the bottom of the disks for HPT samples after (*a*) 1/4, (*b*) 1, and (*c*) 5 turns at 296 K (23 °C).

in Figure 5. It is readily apparent from this image that there is a coarse grain structure with grains as large as ~10 μm near the bottom surface, where this is comparable with the initial as-received grain size of ~9.4 μm, whereas ultrafine grains having sizes of ~1 to 2 μm are observed over a large area at a distance of ~150 μm from the bottom surface (corresponding to the top area in Figure 5).

Figure 6 shows the distributions of microhardness values in vertical traverses on the cross-sectional planes after processing by HPT at 463 K (190 °C) for (a) 1/4, (b) 1, and (c) 5 turns. It is apparent that the hardness distributions lie in the range ~70 to 90 Hv after 1/4 turn, there is an increase to ~80 to 100 Hv after 1 turn, and then there is a decrease to ~70 to 95 Hv after 5 turns. The disk processed through 1 turn of HPT exhibits the expected increase in hardness with increasing distance to the center, but the disks processed to 1/4 and 5 turns exhibit similar levels of hardness at different distances from the center and also different hardness levels at different distances from the bottom surface. These results demonstrate that the heterogeneity of deformation is more pronounced along the bottom-top or axial direction than along the radial direction. The hardness distributions are also consistent with the grain structure such that higher hardness values are recorded in the areas where there is a finer grain size. From Figure 4, it follows that these areas of smaller grain size are between the bottom and midplane in the disk processed by 1/4 turn, in the vicinity of the midplane after 1 turn, and near the bottom surface of the disk after 5 turns.

IV. DISCUSSION

The present results provide very clear evidence for the occurrence of heterogeneous flow along the thickness of the samples of the AZ31 alloy processed by HPT at both room temperature and 463 K (190 °C). The results contrast with the assumption, which is inherent in Eq. [1], that the level of deformation is dependent only upon the number of turns and the radial distance from the disk center as in conventional torsion testing. Three separate observations support the heterogeneous nature of the deformation along the cross-sectional planes of the discs, and these observations are discussed in Sections IV–A through IV–C.

A. *Flow Patterns during Processing at Room Temperature*

Etching of the cross sections of the disks processed by HPT at room temperature reveals the flow patterns associated both with previous deformation and with HPT processing, as shown in Figures 1 and 2. The material used in this investigation was provided in the form of extruded bars and these rods were cut perpendicular to the extrusion direction before HPT. Therefore, the bottom-top or axial direction of the HPT disks lies parallel to the original extrusion direction, and flow lines are readily visible along the axial direction. A clear example is shown in Figure 2, where there are flow lines parallel to the axial direction of the disk at the center of the sample processed to 1/4 turn. Since this region experiences the lowest amount of deformation, the original flow lines of the extruded material are retained.

The torsional straining in HPT occurs in a plane perpendicular to the axial direction of the disks.

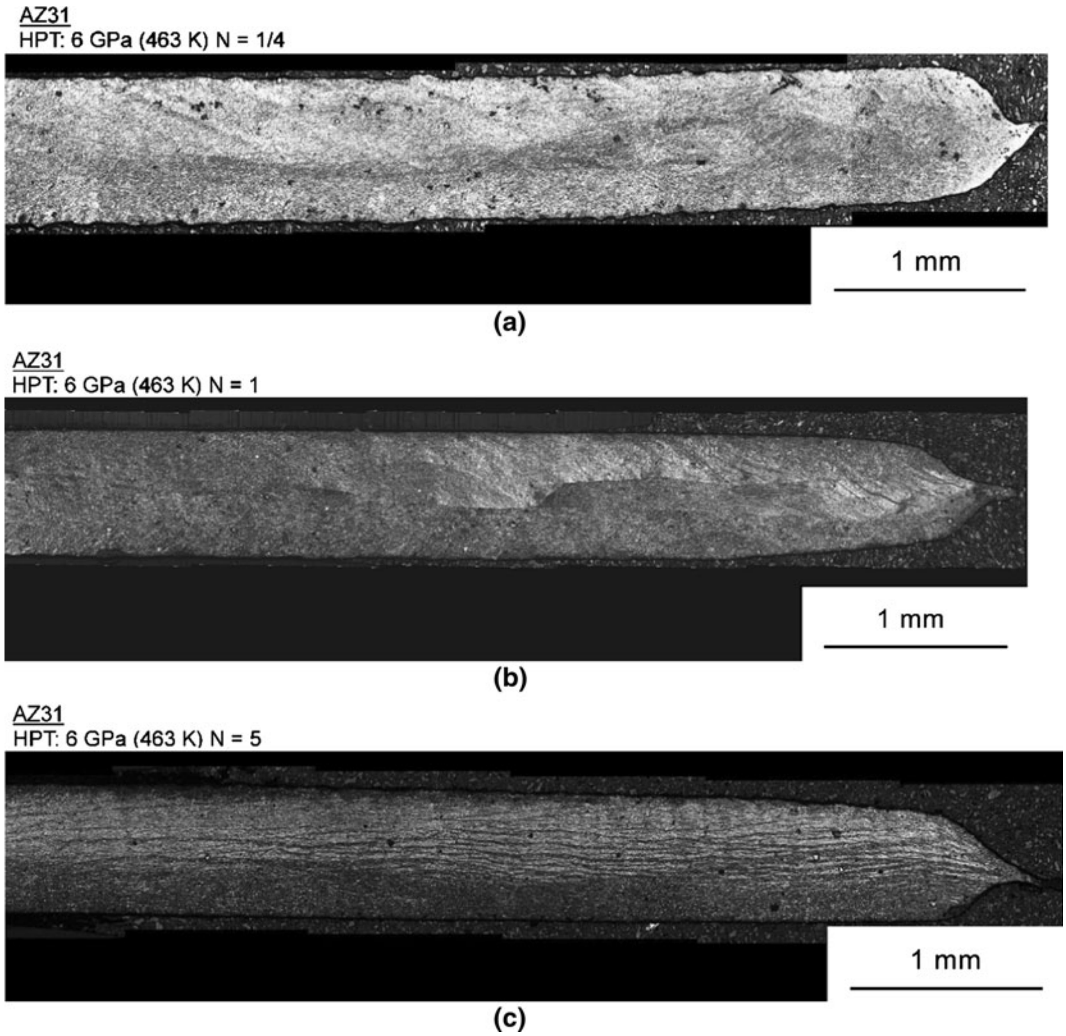

Fig. 4—Flow patterns in the cross-sectional planes of HPT samples after (*a*) 1/4, (*b*) 1, and (*c*) 5 turns at 463 K (190 °C).

Therefore, it is expected that the flow lines, initially parallel to the axial direction, will align with the plane perpendicular to the axial direction with increasing deformation. As a consequence, the orientation of the flow lines is indicative of the distribution of deformation during HPT.

An analysis of the cross sections of the disks in Figure 2 shows distinct features at different distances from the centers of the disks and also in directions perpendicular to the radial direction. The appearance of these flow lines, as in Figure 2, suggests that deformation is concentrated in the midplanes at the edges of the disks. This area is located near the gap between the two anvils in the quasi-constrained facility used for HPT processing. This suggests that at the edges of the disks, a ''dead zone'' may form in the areas constrained by the lateral walls of the upper and lower anvils, thereby reducing the amount of deformation imposed to the samples. This can be attributed, at least in part, to the friction between the samples and the inner anvil walls that prevent torsional deformation. It should be noted that the image of the cross section of the edge of the disk processed to 5 turns, as in Figure 2, shows that this area of low deformation extends to several hundreds of microns away from the anvil wall. A recent report also presented evidence of lower deformation at the top and bottom surfaces at the edges of disks processed by HPT, and it was shown that the degree of heterogeneity was increased with an increase in the ratio of the thickness to diameter of the sample.[26] It was also proposed that it may be possible to modify the anvil geometry in order to avoid the formation of these dead zones.

B. *Distribution of Grain Sizes during Processing at an Elevated Temperature*

The HPT processing was conducted at a temperature of 463 K (190 °C), and therefore it is appropriate to consider recent reports on the mechanism of grain refinement in magnesium alloys processed by ECAP at temperatures in the range of ~420 K to 600 K (~147 °C to 327 °C).[29–31] Refinement at these temperatures occurs by dynamic recrystallization, which is initiated along grain boundaries and twin boundaries to give an initial necklacelike appearance of fine grains and is then followed by a gradual evolution in which these fine

Fig. 5—Grain size heterogeneity on the cross-sectional plane of an HPT sample after 1/4 turn at 463 K (190 °C).

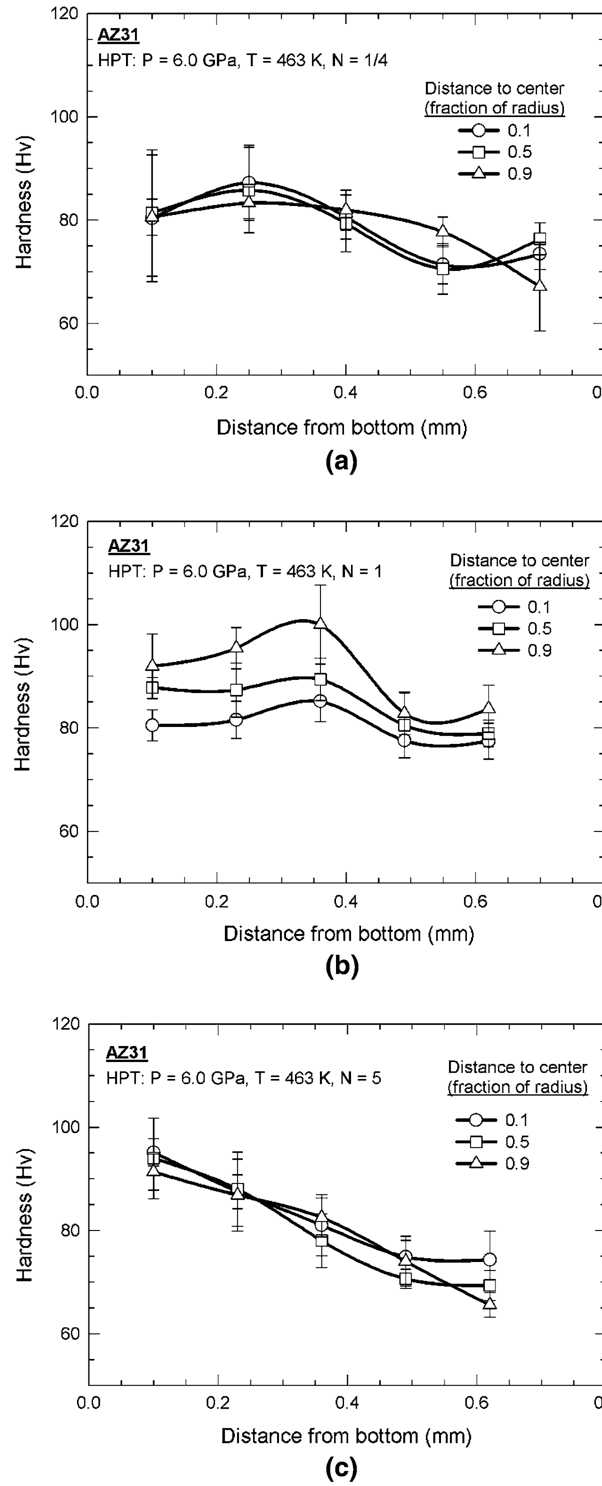

Fig. 6—Microhardness values at various radii as a function of the distance from the bottom of the disks for HPT samples after (*a*) 1/4, (*b*) 1, and (*c*) 5 turns at 463 K (190 °C).

grains spread into the inner core areas of the original grains. In practice, the size of these new grains is dependent upon the temperature and strain rate. Therefore, it follows that the distribution of grain sizes may be used to provide a quantitative estimate of the distribution of deformation associated with any processing conducted in this temperature range. Accordingly, areas experiencing higher deformation will exhibit a higher fraction of small grains, whereas areas experiencing low deformation will contain the coarse cores of the unrefined grains. Moreover, the occurrence of flow localization is associated with higher deformation rates, and this will lead to smaller grains by comparison with the neighboring areas.

The images of the grain structures along the cross sections of disks processed by HPT at 463 K (190 °C), as shown in Figure 4, reveal areas with distinct distributions of grain sizes. Areas with fully refined grain structures are present between the midplane and the bottom surface of the disk processed by 1/4 turn, around the midplane of the disk processed by 1 turn, and near the bottom surface of the disk processed by 5 turns. This suggests these areas were subjected to severe deformation. By contrast, coarse unrefined grains are present in the remaining areas, suggesting the occurrence of lower levels of deformation. This distinct distribution of grain sizes is evident in Figure 5 for the disk processed by 1/4 turn, where coarse unrefined grains are present near the bottom of the disk at a radial distance 2 mm from the center whereas fully refined grains are present nearer the central plane of the disk. The image in Figure 5 shows that areas with low levels of deformation are observed far from the lower surface, and therefore any friction between the disk and the anvil wall is insufficient to represent the only source of heterogeneous deformation.

C. *Distribution of Microhardness Values*

Microhardness tests were used in these experiments to provide information on the local distribution of strength. The as-received extruded material exhibited practically a homogeneous distribution of hardness throughout the sample, with a hardness value of ~68 Hv, but strain hardening and grain refinement increase the strength of metallic materials and are both dependent on the level of deformation imposed by SPD processing. Therefore, hardness tests can be used also to indirectly evaluate the level of deformation imposed on the sample during HPT.

The results for the testing of samples processed at room temperature show a pronounced increase in strength of the alloy. Microhardness up to ~135 Hv was observed in different areas of the disk processed through 5 turns, showing that HPT can practically double the strength of the magnesium alloy. However, the distribution of hardness displays significant heterogeneity at different positions along the axial direction of the sample. Variations larger than 20 Hv were observed at various radial positions and in the samples processed to a different number of turns. These results confirm that the deformation imposed to the sample varies significantly at different positions between the bottom and top surfaces.

It is important to note that the hardness level at a fixed distance between the bottom and top surfaces may stabilize or even decrease with increasing distance to the center of the disk without reaching the maximum hardness. Therefore, the use of microhardness tests along a plane perpendicular to the disk axial direction, as in conventional hardness testing of HPT samples, can suggest a saturation of hardness at levels below the maximum hardness.

The distribution of hardness in the samples processed at an elevated temperature shows that the increase in strength produced by HPT is not as effective as when the processing is conducted at room temperature. The maximum hardness values in these samples are in the range of ~90 to 100 Hv, whereas processing at room temperature gives hardness values up to ~135 Hv. This shows that an increase in processing temperature compromises the increase in strength produced by HPT, and this is reasonable because of the increase in the recovery rate and the occurrence of grain growth at high temperatures. Despite the lower hardness level, the samples processed at a high temperature also display significant differences in hardness distribution perpendicular to the disk surface. Thus, differences of hardness in the range of 10 Hv are observed in the samples processed to 1/4 turn and 1 turn and up to ~20 Hv in the sample processed to 5 turns. These measurements confirm that the deformation level imposed by HPT varies through the disk thickness.

All of these results demonstrate that there is a high degree of heterogeneity in disks of the AZ31 magnesium alloy processed by HPT, and this is in very sharp contrast with the high level of homogeneity recorded earlier in high-purity aluminum after HPT processing.[27]

V. SUMMARY AND CONCLUSIONS

1. Disks were machined from extruded bars of an AZ31 magnesium alloy and processed by HPT at a pressure of 6.0 GPa through 1/4, 1, and 5 turns either at room temperature (296 K (23 °C)) or at an elevated temperature of 463 K (190 °C). After processing, the disks were sectioned to reveal the cross-sectional planes.
2. It is shown that the flow patterns vary not only along the radii of the disks but also through the thickness. There is a high degree of heterogeneity in these disks after HPT processing and this is revealed by the flow patterns, by the distributions of grain sizes, and by the microhardness measurements.
3. Processing by HPT can double the strength of the magnesium alloy in some areas of the disk after processing through 5 turns at room temperature. However, the increased hardness is not constant either in the radial direction or along the axial direction within the cross-sectional plane.

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