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Evolution of Microstructure and Hardness in an AZ80 Magnesium Alloy Processed by High-Pressure Torsion

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Abstract

An AZ80 magnesium alloy with an initial grain size of ~25 μm and a hardness of Hv ≈ 63 was processed by high-pressure torsion (HPT) at room temperature for up to 10 turns under an imposed pressure of 6.0 GPa. After processing, the specimens were examined by optical microscopy and transmission electron microcopy and measurements were taken of the Vickers microhardness along diameters of the HPT discs. The results show that the grains are refined to ~200 nm after 5 and 10 turns of HPT and the hardness increases to Hv ≈ 120 at an equivalent strain of ~30. There is a saturation condition and no further hardening at additional equivalent strains up to >200.

Keywords: hardness; high-pressure torsion; magnesium AZ80 alloy; ultrafine grains

1. Introduction

Magnesium alloys are among the lightest structural materials in current use having high specific strength and low density compared to alternatives such as steel and aluminum. They are excellent candidate materials for achieving weight reductions in transportation applications [1]. In addition, they have dimensional stability, a high damping capacity, electromagnetic interference shielding, good machinability and they can be recycled at low cost making them attractive for a range of applications in electronic portable devices [2]. Nevertheless, the alloys are difficult to deform at room temperature due to their hexagonal close-packed (HCP) crystal structure and the presence of only a limited number of slip systems so that their overall utilization tends to be restricted.

From an economic perspective, the use of lightweight structural materials will improve fuel consumption and reduce cost, thereby having less impact on the environment. This has driven researchers to seek methods of improving the strength and other mechanical properties of magnesium alloys in order to compete with other metallic options.

It is now well established that the yield stress, σ_y, of a polycrystalline material varies with the grain size, d, through the Hall-Petch relationship which is given by [3,4]

$$\sigma_y = \sigma_o + k_y d^{-1/2} \tag{1}$$

where σ_o is the lattice friction stress and k_y is a constant of yielding. It follows directly from Eq. (1) that a reduction in grain size will increase the strength of the material, where grain refinement may be achieved by thermo-mechanical processing [5,6] or through the application of severe plastic deformation (SPD) [7-9]. In practice, the use of SPD processing is a convenient and effective method for producing ultrafine-grained (UFG) materials since the average grain size is generally refined to the submicrometer or nanometre scale and this is significantly smaller than the grain sizes attained by thermo-mechanical processing.

The two main techniques of SPD processing are equal-channel angular pressing (ECAP) [7] and high-pressure torsion (HPT) [8]. Although ECAP is attractive because samples can be scaled up to larger sizes [10, 11], it is difficult to use ECAP to process magnesium alloys at room temperature because the limited slip systems produce a lack of ductility and the initiation and development of cracks during deformation [12]. Accordingly, the ECAP processing of magnesium alloys is usually conducted at elevated temperatures to avoid cracking [13].

Processing by the HPT process is generally more convenient because, due to the imposed hydrostatic pressure, it can be conducted at low temperatures without any cracking [14,15]. In processing by HPT, the sample is in the form of a thin disc and it is placed between two large upper and lower anvils which apply a high compressive pressure and concurrent torsional straining. In straining through HPT, the equivalent von Mises strain, ε_{eq}, is given by a relationship of the form [16,17]

$$\varepsilon_{eq} = \frac{2\pi N r}{h\sqrt{3}} \tag{2}$$

where N is the number of HPT revolutions and r and h are the radius and height (or thickness) of the disc, respectively. It follows from Eq. (2) that the strain introduced in HPT is remarkably inhomogeneous since no strain is introduced in the center of the disc where $r = 0$. Nevertheless, extensive microstructural observations and hardness measurements show that a reasonably homogeneous microstructure and hardness generally develop if the discs are subjected to sufficiently high applied pressures and torsional straining is continued through a sufficient number of turns [18,19]. This development of homogeneity is also consistent with a theoretical approach based on strain gradient modeling [20].

Among wrought magnesium alloys, the AZ80 alloy has high tensile strength and relatively low cost so that it has become an attractive commercial alloy for a range of applications. The present research was therefore initiated to investigate the evolution of microstructure and microhardness in this alloy after processing by HPT. To date, only very limited information is available describing the processing of the AZ80 allloy by HPT and in this earlier work the HPT processing was conducted at room temperature using the relatively low applied pressure of 3.0 GPa [21]. In the present experiments, HPT processing was conducted at room temperature using an applied pressure of 6.0 GPa, the microstructural evolution was examined using optical microscopy (OM) and scanning transmission electron microscopy (STEM) and measurements were taken to determine the Vickers microhardness, Hv, across disc diameters.

2. Experimental material and procedures

The experiments were conducted using a commercial AZ80 magnesium alloy supplied by Magnesium Electron, Ltd (Swinton, Manchester, UK) and having a chemical composition of Al 8.7, Zn 0.51, Cu <0.001, Fe 0.005, Mn 0.18, Ni 0.0005, Si 0.02 and balance Mg (wt.%).

The material was supplied in the form of extruded rods having lengths of 1000 mm and diameters of 9.5 mm. In the as-received condition, the initial average grain size was ~25 μm and the initial Vickers microhardness was Hv ≈ 63. The extruded rods were sliced into discs with thicknesses of ~1.5 mm and then ground with abrasive papers to final thicknesses of ~0.85 mm. Inspection of the unprocessed discs verified homogeneity across the surface, as demonstrated by the distribution of microhardness values and grain sizes.

The AZ80 discs were processed by HPT under quasi-constrained conditions [22,23] where there is an outflow of some material around the periphery of the disc during the processing operation. The HPT was conducted at room temperature (296 K) through different numbers of turns from 1/4 to 10 turns using an applied pressure of 6.0 GPa and a rotation speed of 1 rpm.

After HPT processing, the discs were cold-mounted, mechanically ground using abrasive papers and polished to a final mirror-like surface. The surfaces were etched to reveal the microstructures and then were examined using OM. For this condition, the microstructures were recorded with images at the centre and edge of selected discs. A STEM Hitachi S-5500 was used to characterize the microstructure development at high magnifications. The TEM foils were prepared by ion milling. The centres of the TEM foils were 3 mm away from the centres of the HPT processed discs. The grain size was determined after processing through the maximum of 10 turns. The Vickers microhardness, Hv, was recorded on the polished mirror-like disc surfaces at selected positions across the diameters using an applied load of 100 gf and a dwell time for each measurement of 15 s. Each selected point was surrounded by four individual points each separated at 0.15 mm from the selected position and separated from each other by 0.3 mm. A similar procedure was described in detail in an earlier report [24]. Finally, the average of these four points was recorded as the hardness for that position.

3. Experimental results

3.1 Microhardness observations

All of the AZ80 magnesium alloy discs were successfully processed by HPT at 296 K without introducing any cracks into the material. Hardness measurements were then taken across selected diameters of each disc and the results are shown in Fig. 1 where the lower dashed line indicates the microhardness in the as-received condition of ~63 Hv.

Inspection shows that the values of Hv are consistently higher than in the as-received condition and this increased hardness is especially evident around the edges of the discs. In practice, there are significant differences between the edges and the centres of the discs at the lower numbers of turns. For example, after 1/4 turn the microhardness is ~110 Hv at the edge but only ~88 Hv in the center of the disc. There is an increase in the average microhardness value at the disc centre for the sample processed by one turn with Hv ≈ 92 whereas the changes at the periphery are essentially negligible. The microhardness values of samples processed by 5 and 10 turns showed reasonable homogeneity across the disc diameters with an average value of ≈ 120 Hv at all points. An earlier report described the processing of the AZ80 alloy by HPT up to 15 turns using an applied pressure of 3.0 GPa and there was no significant change in the hardness values at the larger numbers of turns [21]. Thus, it is concluded that the microstructure reaches a reasonable level of stability after 10 turns.

3.2 Microstructural observations

Processing by HPT for up to 10 turns showed that the microstructure was significantly refined by the HPT processing and there was a gradual evolution into highly deformed grains. Due to the heterogeneity introduced by the HPT processing in the early stages of processing as after 1/4 and 1 turn, it was found that the periphery contained highly refined grains whereas in the central region the grains were relatively coarse. Thus, the microstructure developed gradually from the edge towards the centre with increasing numbers of revolutions and this evolution was clearly apparent after 3 turns. This is shown by the OM images in Fig. 2 where the left column corresponds to the center position and the right column corresponds to the edge position: separate images are shown for (a,b) the as-received condition, (c,d) after 3 turns and (e,f) after 10 turns. After 3 and 10 turns the microstructures became reasonably uniform with similar highly refined grains across the diameter of each disc.

The evolution in microstructure was also further investigated using STEM as shown by the images and diffraction patterns in Fig. 3 for specimens processed through (a) 1/4 (b) 1, (c) 5 and (d) 10 turns. These TEM observations revealed a difference in microstructure between the early and later stages of HPT because initially the grains were elongated as in Fig. 3(a) and (b) whereas after 5 and 10 turns the grains were reasonably equiaxed and uniformly distributed with an average size of ~200 nm as shown in Fig. 3(c) and (d). This was confirmed by the selected area electron diffraction (SAED) patterns where there is clear evidence, after 5 and 10 turns, for the production of ultrafine grains separated by boundaries having high angles of misorientation. These microstructural changes are similar to those reported in early studies of pure Al [25,26] and Al alloys [27] processed by ECAP.

4. Discussion

Two factors generally influence the development of homogeneity in HPT processing: the applied pressure and the imposed strain as represented by the number of turns [28]. In this investigation, the imposed pressure was constant at 6.0 GPa for all samples and therefore the results demonstrate the importance of the imposed strain on the evolution of microstructure and microhardness towards a reasonable level of homogeneity.

The present results demonstrate that the AZ80 magnesium alloy may be processed successfully by HPT at room temperature without the appearance of any cracking when using an imposed pressure of 6.0 GPa. The measured values of the microhardness correlate well with the observed microstructure of the AZ80 alloy when processing up to 10 turns. Thus, although the results in this study revealed clear evidence of heterogeneous deformation across the diameters of the disc surfaces in the early stages of processing, continuing the HPT processing to 5 and 10 turns produced excellent grain refinement and a generally homogeneous structure.

It was shown in a very early study that the various measurements of hardness attained in HPT processing after different numbers of turns may be readily correlated by plotting the values of Hv against the calculated equivalent strain at each point of measurement using the relationship for strain given in Eq. (2) [29]. This approach is especially useful in confirming the development of a saturated or steady-state condition in which the hardness remains constant over a large range of strain. An example of this approach is shown in Fig. 4 where all of the individual datum points are now plotted together and delineate essentially a single curve. Thus, the as-received hardness of Hv $\approx$ 63 initially increases to >80 at the edge of the disc, there is a continuous increase in Hv up to ~120 at an equivalent strain of ~30 and thereafter, at even higher equivalent strains, the hardness remains reasonably constant up to equivalent strains of >200. The development of a steady-state or saturation condition is consistent with earlier reports for several metals processed by HPT [30] and also the same effect was achieved in an Al-7075 alloy processed by a combination of ECAP and HPT [31].

A recent review of hardness evolution in HPT processing described three different types of behaviour that may be observed experimentally [32]. First, there is hardening without recovery in which the values of Hv increase with increasing strain and then level off at a saturation condition. This is representative of a very wide range of different metals and it is consistent with the plot visible in Fig. 4. Second, there is softening with rapid recovery in which the hardness initially increases with strain, reaches a peak value and then decreases to a lower saturation condition. This behaviour occurs in metals such as pure aluminium where the very high stacking fault energy leads to easy recovery through cross-slip [33]. Third, there is weakening in which the hardness decreases from the onset of straining and ultimately levels off in a very low saturation condition. This behaviour has been reported in some two-phase alloys such as the Zn-22% Al eutectoid and the Pb-62%

Sn eutectic [34]. Nevertheless, the present results on the AZ80 magnesium alloy are consistent with the behaviour of a large number of metallic alloys.

There are not many reports of high-pressure torsion of magnesium alloys and even less on the AZ80 alloy. Table 1 summarizes the final grain size and the maximum Vickers microhardness of pure magnesium and magnesium alloys processed by HPT at room temperature for different numbers of rotations from published data and the present investigation. As shown in Table 1, with different initial grain sizes and different applied pressures, HPT can successfully achieve significant grain refinement and strength enhancement in AZ80 and other magnesium alloys.

5. Summary and conclusions

[1] An AZ80 magnesium alloy was processed by high-pressure torsion at room temperature for up to 10 turns under an imposed pressure of 6.0 GPa.

[2] Processing by HPT reduced the grain size from an initial value of ~25 μm to a value of ~200 nm after 5 and 10 turns.

[3] Measurements showed there was hardness heterogeneity in the initial stages of processing but a gradual evolution towards a reasonable level of hardness homogeneity after 5 and more turns.

[4] By plotting the hardness against the equivalent strain, it is shown that the hardness initially increases abruptly from an initial value of Hv ≈ 63 to a value of Hv ≈ 120 at an equivalent strain of ~30 and thereafter the hardness remains reasonably constant up to equivalent strains of >200.

Acknowledgements

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Figure captions

Fig. 1 Vickers microhardness Hv plotted as a function of position on discs processed through 1/4 to 10 turns: the lower dashed line at Hv ≈ 63 shows the as-received condition.
Fig. 2 Optical images showing the centre (left column) and edge (right column) for (a,b) the as-received material and after HPT processing through (c,d) 3 turns and (e,f) 10 turns.
Fig. 3 Images by STEM combined with the diffraction patterns for the AZ80 alloy after processing by HPT for (a) 1/4 turn, (b) 1 turn, (c) 5 turns and (d) 10 turns.
Fig. 4 Vickers microhardnmess Hv plotted against equivalent strain showing the development of a saturation condition at strains above ~30.

Table caption

Table 1 Final grain size and the maximum Vickers microhardness of pure magnesium and magnesium alloys processed by HPT at room temperature for different numbers of revolutions

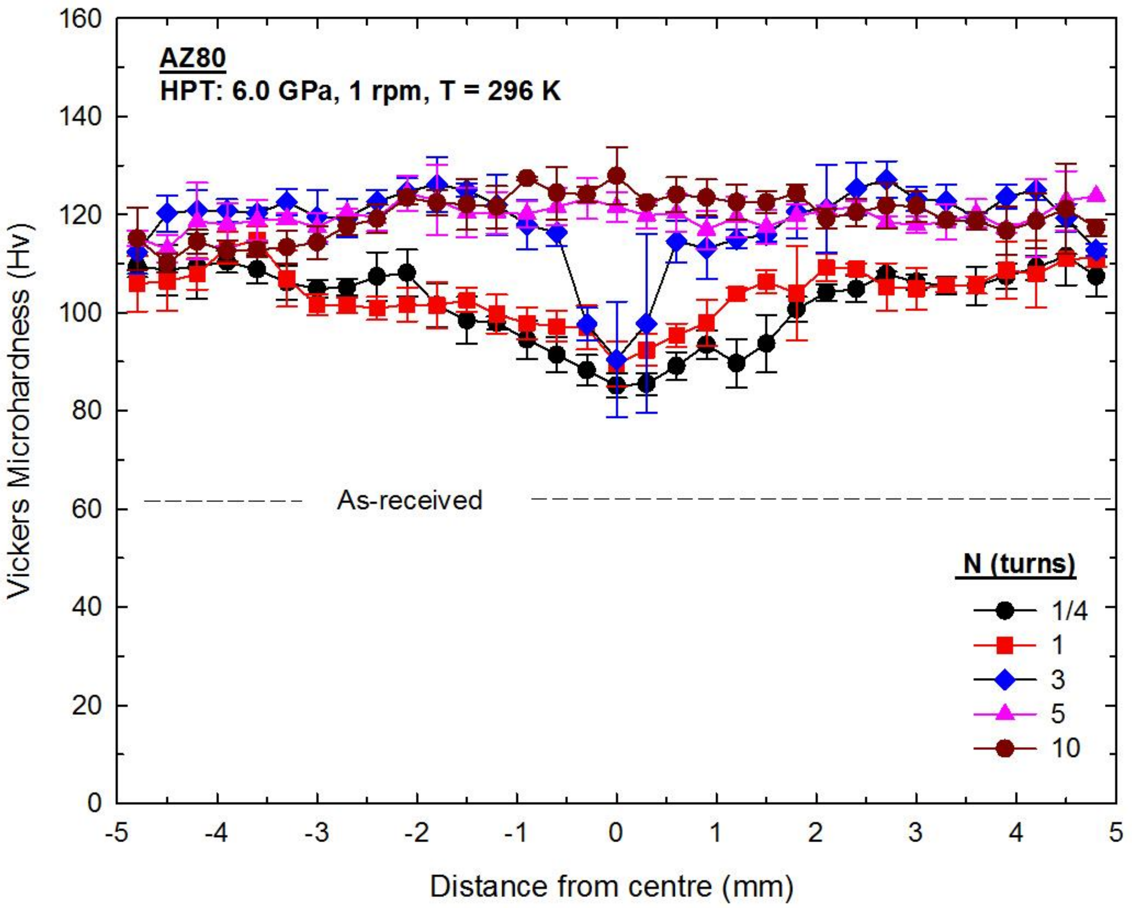
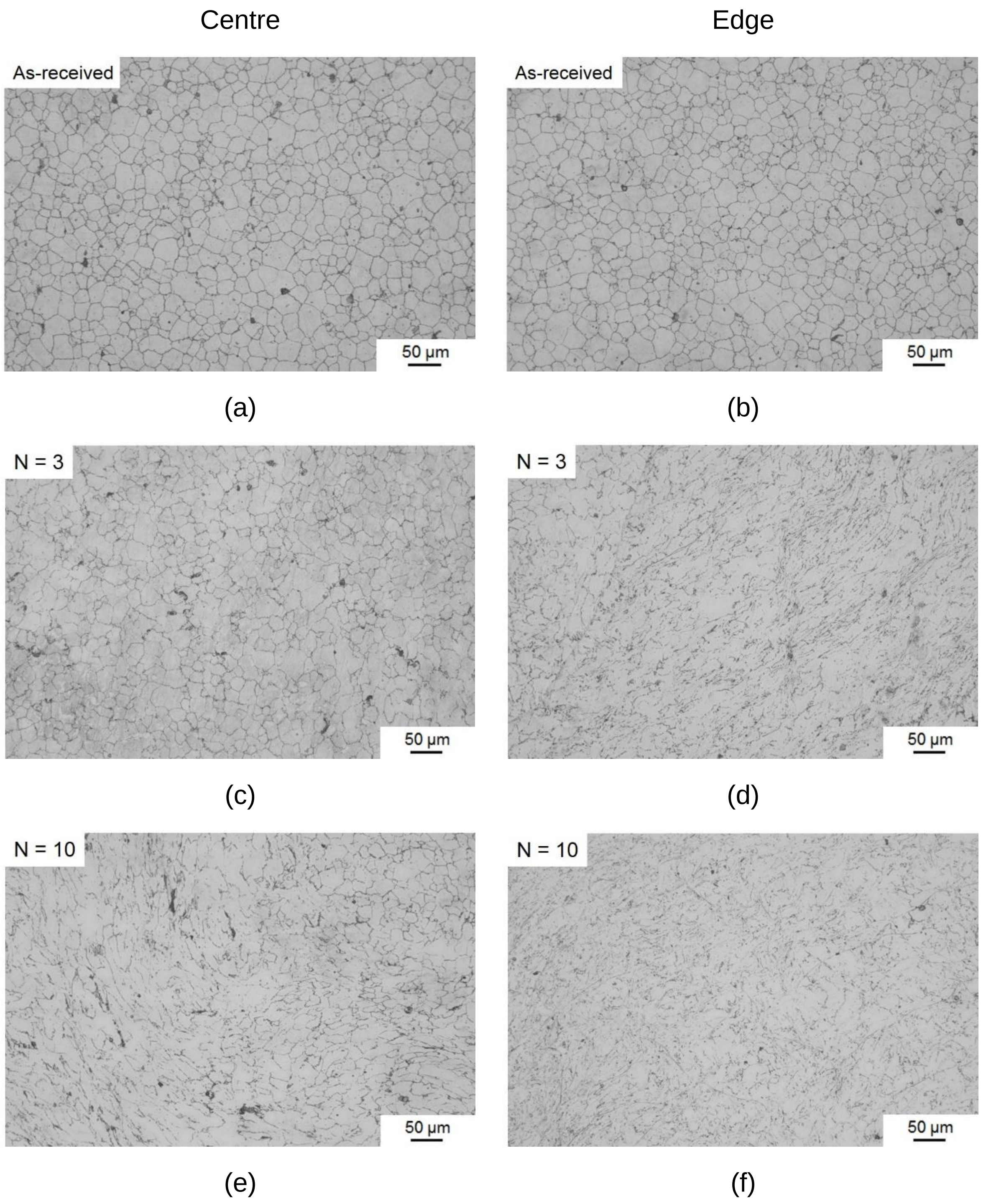


Fig. 1 Vickers microhardness Hv plotted as a function of position on discs processed through 1/4 to 10 turns: the lower dashed line at Hv ≈ 63 shows the as-received condition.



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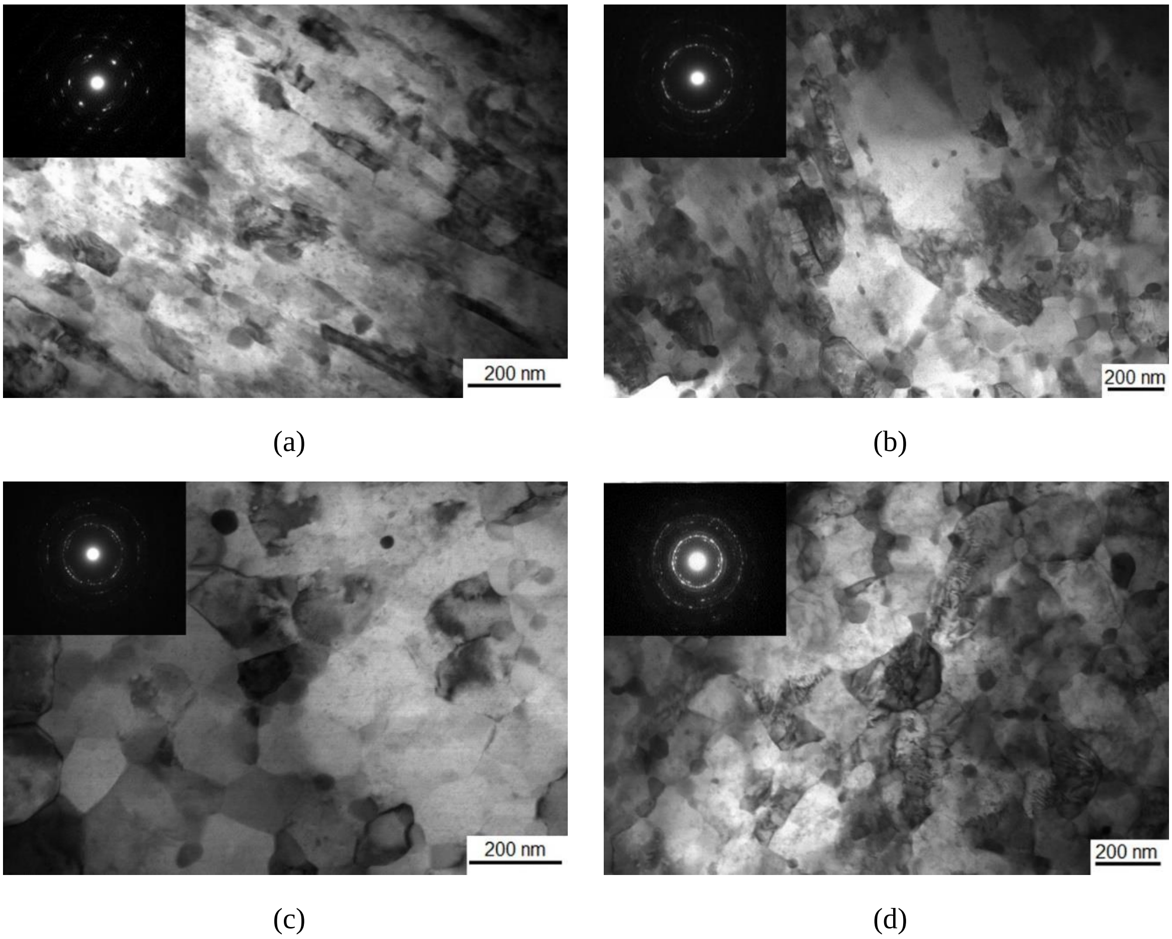

Fig. 3 Images by TEM combined with the diffraction patterns for the AZ80 alloy after processing by HPT for (a) 1/4 turn, (b) 1 turn, (c) 5 turns, and (d) 10 turns.

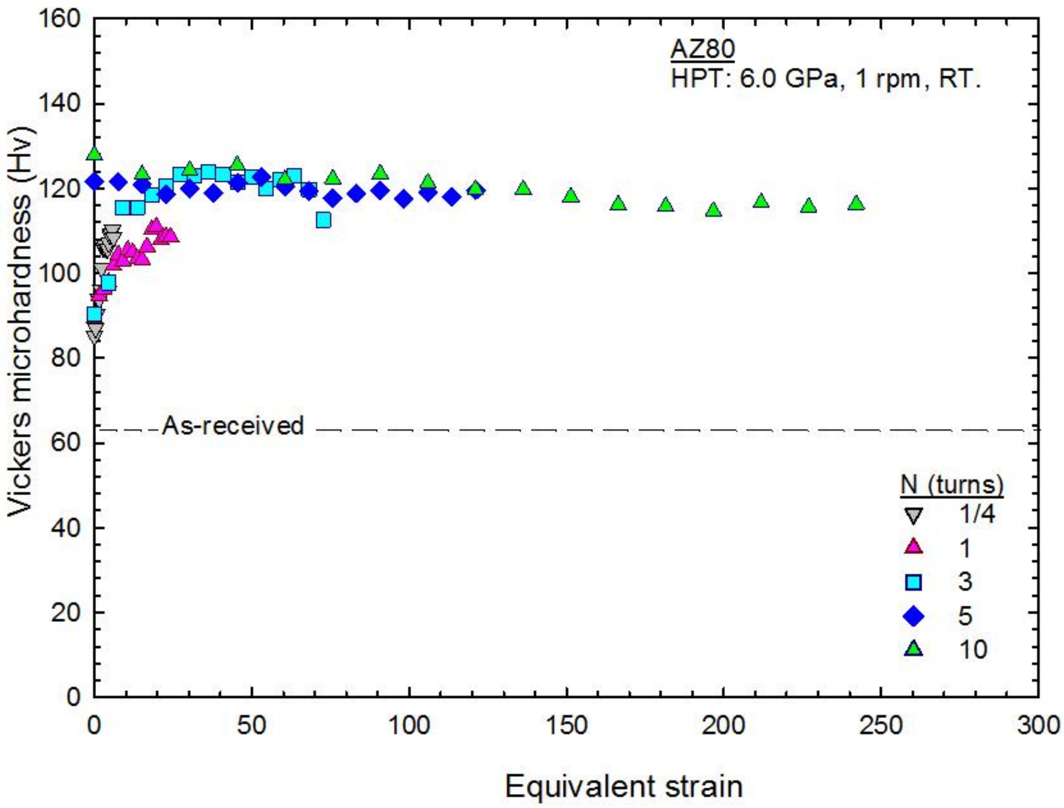

Fig. 4 Vickers microhardness Hv plotted against equivalent strain showing the development of saturation condition at strains above ~30.

Table 1 Final grain size and the maximum Vickers microhardness of pure magnesium and magnesium alloys processed by HPT at room temperature for different numbers of revolutions

Magnesium and magnesium alloy	**HPT processing**		**Grain size (μm)**		**Maximum Vickers microhardness, Hv**	**Reference**
	Pressure (GPa)	**Number of turns**	**Before HPT**	**After HPT**		
Pure Mg 99.8 %[a]	6.0	8	≈ 50	≈ 0.6	55	Qiao *et al.* [35]
AZ31[b]	2.5	15	≈ 150-200	0.15-0.2	113	Stráská *et al.* [36]
AZ31[c]	6.0	5	≈ 10	0.9-1.2	110	Huang *et al.* [14]
AZ61[d]	3.0	5	≈ 22	0.11	-	Harai *et al.* [37]
ZK60[e]	2.0	5	≈ 9.4	≈ 1	124	Torbati-Sarraf & Langdon [38]
ZK60A[f]	6.0	5	≈ 10	2-5	110	Lee *et al.* [39]
Mg–8%Li[g]	3.0	5	-	≈ 0.5	63	Matsunoshita *et al.* [40]
Mg-9 wt% Al[h]	3.0	5	≈ 12	≈ 0.15	120	Kai *et al.* [41]
AZ80[i]	3.0	15	≈ 6-31	≈ 0.1	125	Arpacay *et al.* [21]
AZ80[j]	6.0	10	≈ 25	≈ 0.2	120	This investigation

Material condition prior to HPT

[a] Extruded at temperature of 350 ^{o}C prior to HPT
[b] Homogenized at 390 ^{o}C for 12 h prior to HPT
[c d e f j] Extruded
[g] Extruded at temperature of 100 ^{o}C prior to HPT
[h] Extruded at temperature of 200 ^{o}C prior to HPT
[i] Extruded at temperature of 300 ^{o}C then solution heat treated at 400 ^{o}C for 11 h prior to HPT