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ARTICLE

Structural and Hardness Evolution of Pure Magnesium Subjected to High Pressure Torsion

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Abstract: Pure Mg was subjected to high-pressure torsion (HPT) at room temperature, and the structural and hardness evolutions were studied. In addition, the grain size evolution during the hardness steady state was investigated. The results indicate that the hardness HV initially increases with increasing the equivalent strain, reaches a maximum value of ~530 MPa, and then decreases to a steady-state level at large strains. However, although the hardness reaches the steady state, the average grain size does not reach a steady state. The evolution of the grain size is different from that the hardness during HPT processing. The continuously decreased grain size during harness steady state may be caused by the annihilation of the dislocations during dynamic recovery or dynamic recrystallization which is resulted from the temperature rising during the continuous HPT processing and the low melting temperature of Mg.

Key words: Mg; high pressure torsion; hardness; grain refinement; dislocation

High-pressure torsion (HPT) is a severe plastic deformation (SPD) technique where a thin disc or ring is placed between two massive anvils under a high pressure and intense shear strain is introduced by rotating the anvils with respect to each other[1,2]. Application of HPT has shown that the microstructure as well as the hardness and strength evolves into a steady state with continuous straining[3-6]. It was shown that the homologous temperature, T/T_m (T is the processing temperature and T_m is the melting temperature), is a dominant parameter to determine the steady-state grain size in pure metals processed by HPT and the grain size increases with increasing the homologous temperature[7-10]. Numerous reports are now available to describe the application of HPT to a range of pure metals. It is well accepted that three kinds of different hardness-strain behavior can be concluded for pure metals: (1) The hardness initially increases with increasing the strain but saturates to a steady state at large strains, such as Nb[11], Ni[9] and Cu[12-14]; (2) after reaching a maximum, the hardness decreases to a steady-state level, such as high purity Al [15,16] and Mg[17] (Mg was also reported for the behavior 1[18]); (3) the hardness slightly decreases and reaches a steady state at large strains, such as Zn[19], Pb[7], Sn[7] and In[7]. It is commonly considered that when the hardness reaches the steady state, the grain size also reaches a steady state[8], and few studies have focused on the grain size evolution during the hardness steady state. In addition, the average grain size at the steady state of the same metal was reported variously. For high purity Al, the average grain size at the steady state was reported to be 1.2 μm[20], 1.5 μm[1], 1.9 μm[21] and 2.1 μm[6]. The large steady-state grain size in Al is attributed to its high stacking fault energy (SFE) [2,22] or to its low melting temperature[23]. The melting temperature (T_m) and SFE of Mg are similar to Al, and the average grain size at the steady state of Mg was reported 1.0 μm[17]. Up to now, few investigations were focused on the grain size evolution of HPT treated pure Mg. Thus in this paper, the structural and hardness evolutions of pure Mg subjected to high pressure torsion were studied, and the grain size evolution during the hardness steady state was investigated and discussed.

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1 Experiment

This work was carried out on commercially pure Mg with a purity of 99.8%. Cylindrical rods of pure Mg, 20 mm in diameter, were cut from the as-received ingot. The rods were sliced into discs with 0.8 mm in thickness using a wire-cutting electric discharge machine. HPT was conducted using a quasi-constrained HPT facility[24]. Discs were subjected to HPT under a pressure of 4 GPa and subsequently shear strain was imposed through either N = 1, 3, 5, 10, or 20 revolutions with a rotation speed of 1 revolution per minute (r/min).

After HPT processing, the disks were mechanically polished to produce a mirror-like surface using diamond lapping films for hardness testing. Hardness measurements were carried out by an FEI-VM50 PC Vickers hardness testing machine at room temperature with a load of 50 g and dwell time of 15 s. The hardness was measured at points with radius values from 0.5 mm to 9.5 mm with an increment of 0.5 mm and the hardness value for each radius value was averaged from 8 datum points positioned by a rotational increment of 45^{o} around the disk centre. TEM specimens were prepared by a twin-jet polishing technique in a mixture of 5% perchloric acid and 95% ethyl alcohol at 233 K. The TEM investigations were carried out by a JEOL 2100F microscope operating at 200 kV. In order to facilitate discussion, the locations for TEM foil specimens were named regions Ⅰ, Ⅱ and Ⅲ, corresponding to the region at r=1.5 mm, r=4.5 mm and r=7.5 mm, respectively. The average grain sizes of the samples were examined using TEM. The values were obtained by measuring two orthogonal axes of the grains for more than 100 grains, and the low-angle grain boundaries were excluded in the measurements.

2 Results and Discussion

Fig.1a shows the variation in hardness with the distance from the center of the samples after 1 to 20 revolutions. The hardness variation is irregular and strongly depends on the revolution number. The hardness increases with respect to the distance from the center for 1 revolution. However, the hardness exhibits a decrease with an increase in the distance from the center to the edge for 3, 5, 10 and 20 revolutions. It should be noted from Fig.1a that the hardness of 10 and 20 revolutions are lower than that of samples after 3 and 5 revolutions. To demonstrate the hardness evolution with respect to equivalent strain, all hardness values in Fig.1a are plotted against the equivalent strain in Fig.1b as attempted in the earlier papers[2,17,22]. The minimum average grain size is ~343 nm. The equivalent strain was calculated as[3]:

$$\varepsilon = \frac{2\pi rN}{\sqrt{3}t} \quad (1)$$

where r is the distance from the disc center, N is the number of revolutions and t is the thickness of the disc. It is apparent that all data points now lie on a unique curve, reaching a maximum at an equivalent strain of ~30, thereafter leveling off at an equivalent strain of ~200. This is then followed by the onset of a steady state where the hardness remains unchanged with further straining. The hardness-strain evolution agrees well with the previous investigations on high purity Al[15,16] and Mg[17]. The mechanism for the unusual softening of pure Al at large strains was considered to be attributed to dynamic or fast static recovery due to easy cross-slip and/or dislocation climb arising from large SFE and low melting temperature[22,23]. Edalati et al.[17] indicated that although it is difficult for the cross-slip to occur in the hexagonal close-packed (hcp) metals, dislocation climb can still be activated to promote the softening in Mg because self-diffusivity of Mg is high and the homologous temperature of 0.32 is high enough to drive the recovery process. It should be noted that the equivalent strains of reaching a maximum and leveling off hardness are delayed compared with previous study[17], which may be due to the lower HPT pressure (4 GPa) and larger specimen diameter (20 mm).

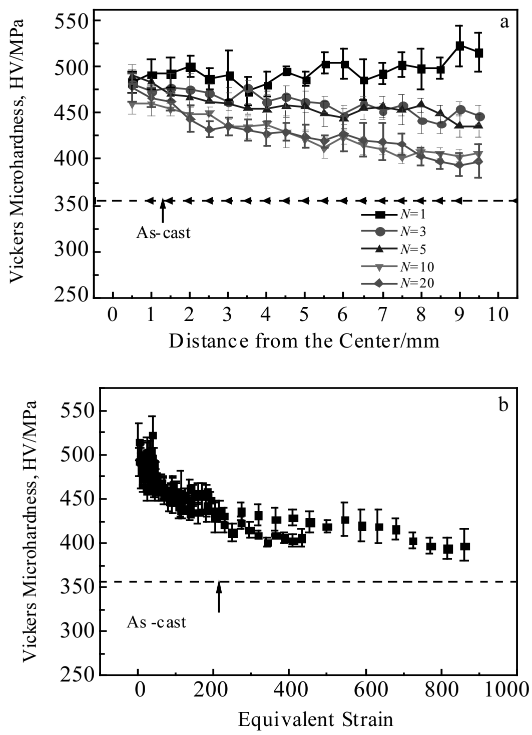

Fig.1 Vickers microhardness as a function of the distance from the disc center (a) and the equivalent strain (b) of the samples processed by HPT for various revolutions

Optical microscopy micrograph (not shown here) shows an average grain size ~1.5 mm for the undeformed sample. Fig.2 shows the bright field TEM images of the samples after HPT treatment. Figs.2a~2c are recorded from the regions Ⅰ, Ⅱ and Ⅲ of the sample after HPT for 1 revolution, respectively. Figs.2d~2f are recorded from the region Ⅲ of the samples after HPT for 3, 5 and 20 revolutions, respectively. After HPT processing, a large number of the dislocations are visible in the still large grains in Fig.2a (ε= 8). With increasing the equivalent strain to 20, the dislocations are tangled, subgrains are formed along the shear direction and many dislocations are visible in the grains, as shown in Fig.2b. With further increasing the equivalent strain to 34, the high angle grain boundaries are apparent and some dislocations can still be visible within the grains, as shown in Fig.2c. The increased dislocations density, dislocations tangling and grain refinement lead to the increase of the hardness. On the other hand, less dislocations are visible within the grains in Fig.2d when the equivalent strain reaches 102. Recrystallized grains can be observed in Figs.2e (ε=169) and 2f (ε= 678) since very few dislocations can be observed within the grains. The decreased dislocation density and dynamic recrystallization lead to the decrease of the hardness. It is worth mentioning that the grain size in Fig.2f are smaller than that in Figs. 2d and 2e. It is commonly accepted that after continuous processing by HPT a steady state should be reached, and the grain size should remain unchanged with further straining [8]. While in this study, it can be observed that the grain size continues to decrease after the hardness reaches the steady state.

To further understand the grain size evolution during the HPT processing, statistical analysis on the grain size of different samples are shown in Fig.3. The average grain sizes of the samples were examined using the TEM. The values were obtained by measuring the two orthogonal axes of the grains for more than 100 grains. The low-angle grain boundaries were excluded in the measurements. From the statistical analysis in Fig.3, it can be seen that the average grain size decreases with decreasing the distance from the center and increasing the revolution number. To demonstrate the grain size evolution with respect to the equivalent strain, all the average grain sizes in Fig.3 are plotted against the equivalent strain in Fig.4. It is apparent that the average grain size decreases initially with increasing the equivalent strain (stage 1), and then reaches a steady state (stage 2). With further increasing the equivalent strain the average grain size decreases again (stage 3). It is worth mentioning that although the hardness reaches a steady state, the grain size continues to decrease with further straining, and the minimum average grain size in the present study is ~343 nm. Traditionally, during the steady state by HPT processing, both the grain size and the hardness remain constant because of the balance between dislocation accumulation and grain refinement, and dislocation annihilation and destruction of the grain boundaries[8]. Different mechanisms were reported about the steady state in HPT processing, including dynamic

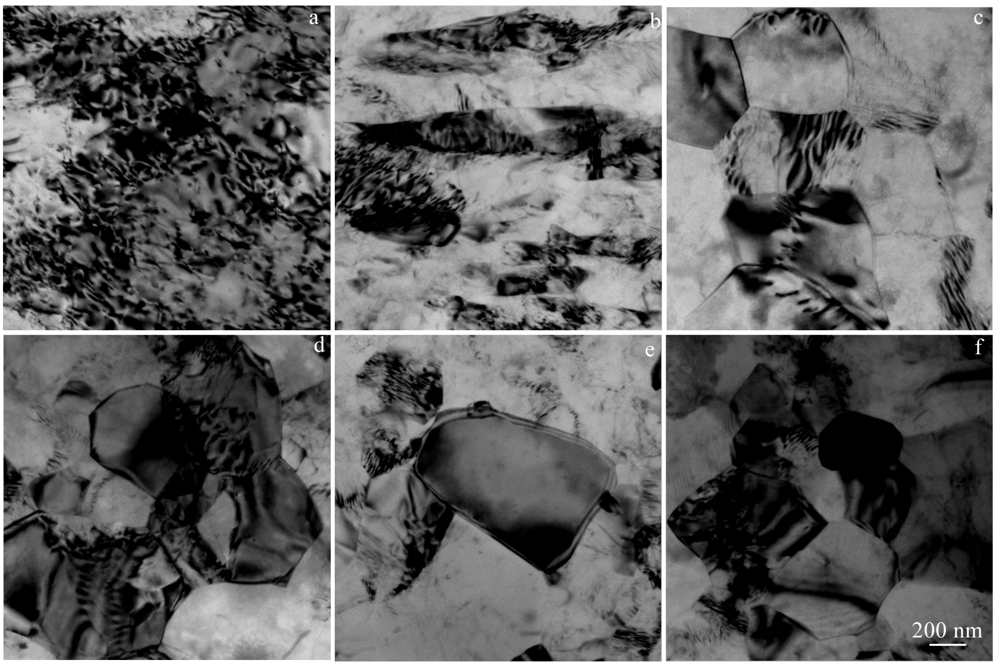

Fig.2 TEM micrographs in the different regions of the samples processed by HPT for various revolutions: (a) region Ⅰ, (b) region Ⅱ, and (c) region Ⅲ for 1 revolution; region Ⅲ for 3 (d), 5 (e) and 20 (f) revolutions

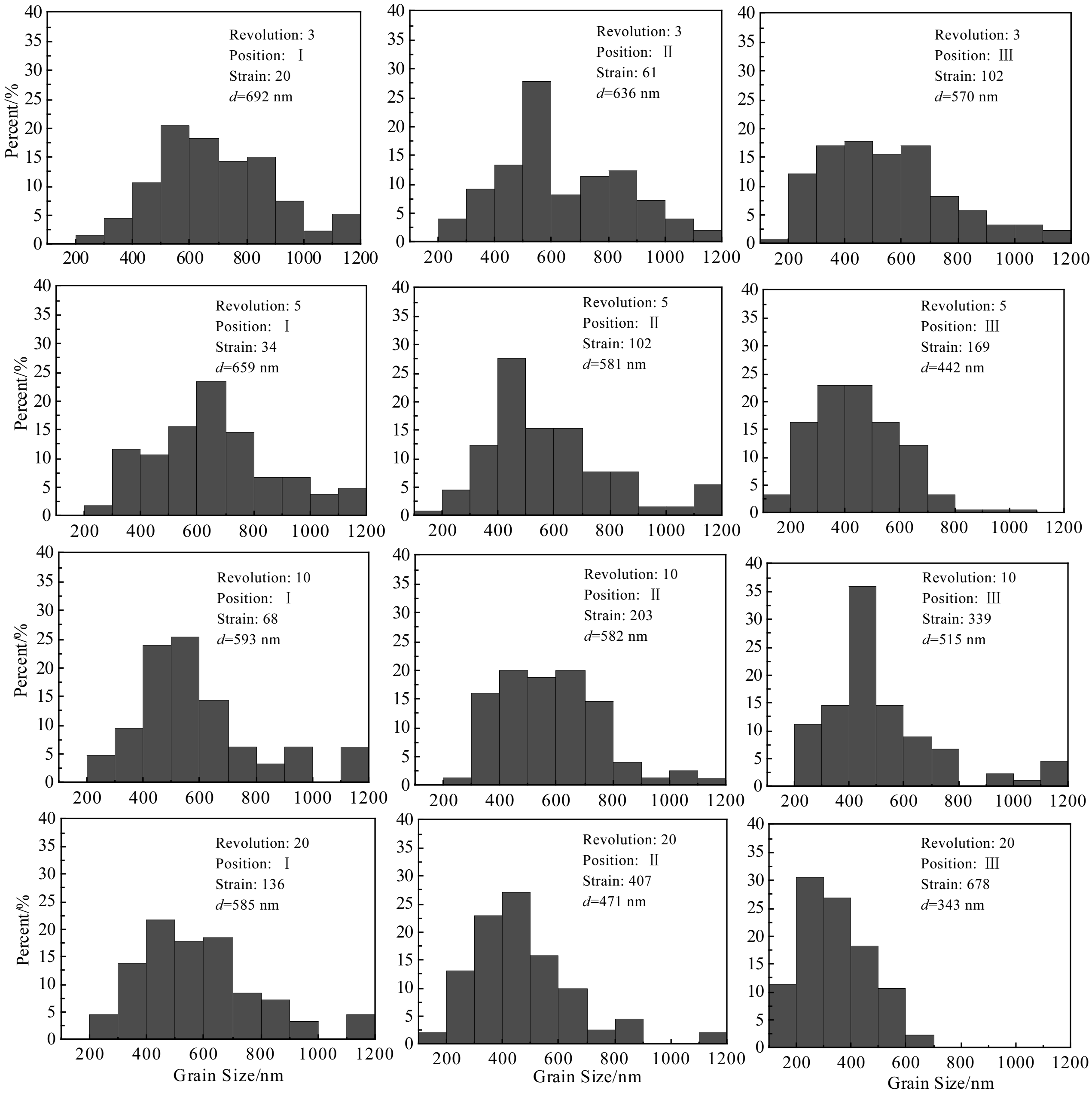

Fig.3 Statistical analysis of the grain size in the different regions of the samples processed by HPT for various revolutions

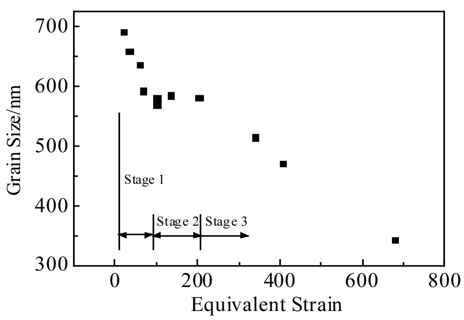

Fig.4 Plot of the average grain size against the equivalent strain

recovery[25], dynamic recrystallization[26,27], grain boundary migration[28] and grain rotation[29]. The average grain size decrease in the stage 3 with a steady hardness may be caused by the annihilation of the dislocations during dynamic recovery or dynamic recrystallization, resulted from the temperature rising during the continuous HPT processing and the low melting temperature of Mg. Our study showed that the grain size evolution of pure Mg during HPT is different from the hardness evolution, due to the different dominant mechanisms during the deformation processing.

3 Conclusions

1) The hardness HV initially increases with increasing the equivalent strain, reaches a maximum value of ~530 MPa, and then decreases to a steady-state level at large strains.

2) The grain size of pure Mg during HPT processing is independent on the hardness evolution, and the average grain size continues to decrease with further straining although the hardness reaches a steady state. The minimum average grain size in this study is ~343 nm.

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纯镁在高压扭转处理中的结构及硬度演变

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摘 要：研究了纯镁在室温高压扭转处理过程中的结构及硬度演变，系统探索了硬度达到稳态后时晶粒尺寸的演变。结果表明，在变形的初始阶段硬度 HV 随着应变的增加而增大，并达到一个最大值（~530 MPa），随后硬度 HV 随着应变的增加将降低到一个稳定值。然而，当硬度值处于稳态时，晶粒尺寸并不是处于稳态。在高压扭转的过程中，硬度值随应变的演变和晶粒尺寸随硬度值的演变是不一致的。当硬度值处于稳态时，晶粒尺寸进一步减小是由于动态回复和动态再结晶造成的位错湮灭。而动态回复和动态再结晶的发生来源于高压扭转过程中的温度上升以及纯镁较低的熔点。

关键词：Mg；高压扭转；硬度；晶粒细化拼；位错

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