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Effect of Aging Treatment before Extrusion on Microstructure and Mechanical Properties of AZ80 Magnesium Alloy

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Abstract: This paper investigated the effect of aging treatment before extrusion on microstructure and mechanical properties of AZ80 magnesium alloy. Fractural microstructures of the test specimens were also analyzed by scanning electron microscopy. Results show that aging treatment before extrusion can remarkably promote grain refinement; the dispersed $Mg_{17}Al_{12}$ particles precipitated from the matrix during aging treatment before extrusion distribute in the grain boundaries; during the subsequent extrusion process these particles can pin grain boundaries migration and lead to fine microstructure; however, with prolonging the aging time, the grain refinement effect is weakened. After aging treatment and subsequent extrusion, the yield strength, tensile strength and elongation are increased. Based on fracture surface analysis, the premature cracks occur around big particles in grain boundaries lead to loss of elongation. The strengthening effect of aging before extrusion in AZ80 magnesium alloy proven in this paper provides a new method for the design of the relatively high-performance magnesium alloys.

Key words: magnesium alloy; recrystallization; microstructure; mechanical properties; Zener pinning effect

As the lightest structural materials, magnesium (Mg) alloys are widely used in the automotive, aeronautical and electronic industries for their light weight, high specific strength and specific stiffness, good castability and machinability, excellent damping properties and good electromagnetic shielding [1-3]. However, due to their poor mechanical properties, the applications of Mg alloys have been restricted. The poor mechanical properties of magnesium alloys result from the hexagonally close-packed structure (HCP), so it could not provide five independent slip systems uniform deformation requires at room temperature [4,5]. In order to improve the mechanical properties, many strengthening methods have been applied and studied to enhance the strength of magnesium alloys [6-9], such as heat treatment, pre-twinning, alloying and grain refinement. Although great efforts have been done, the mechanical properties of Mg alloys still cannot satisfy the needs of applications.

In order to enlarge the application field of magnesium alloys, the design of relatively high-performance wrought magnesium alloys has received much attention in recent years. The main reason is that wrought magnesium alloys often perform better mechanical properties than cast magnesium alloys because the deformation process can get rid of pores, homogenize composition and refine grains [10]. Mg-Al-Zn series alloys, one of the most commonly used wrought magnesium alloys including AZ31, AZ80 and AZ91, display good comprehensive mechanical properties and low cost. Among them, the AZ80 alloy has been the well-known commercial magnesium because it exhibits a good combination of medium strength, good corrosion resistance, excellent forging capability and lower price[6]. However, the AZ80 alloy still has some deficiencies that result in the limit

of applications, such as the insufficient strength and poor ductility[11].

It is widely believed that fine grain strengthening is the most important method to improve both strength and ductility at the same time [12,13]. Controlling the grain size during thermomechanical processing is the key to improving the properties of wrought magnesium alloys[14]. As to control the grain size during hot deformation, using the stable pinning particles to pin the migration of grain boundaries and prevent grain growth has been a new method in recent years[15]. This mechanism is called Zener pinning effect. A great many works based on this mechanism have been carried out so far. Zener pinning effect that results in grain refinement in magnesium alloys during dynamic recrystallization (DRX) has been applied to pure Mg, AZ31 alloy, AZ61 alloy, AZ91 alloy and ZK60 alloy [16-20]. However, up to now, there are few researches on the Zener pining effect on AZ80. What's more, most of the precursory studies focus on revealing the mechanism of Zener pinning effect during hot deformation and pay more attrition to the Zener–Holloman parameter. Hence, in the present study, using the particles precipitating from supersaturated solid solution during aging treatment before extrusion is the key point to refine grain using Zener pinning effect. Aging treatment was carried out before extrusion so that more particles precipitated from matrix during aging and the precipitates pinned grain boundaries migration and prevented grain growth due to Zener pinning effect. In consequence, the effect of aging treatment before extrusion on microstructure and mechanical properties of AZ80 magnesium alloy was investigated.

1 Experiment

The material used in the present study was the cast billets of commercial AZ80 magnesium alloy. The chemical composition was confirmed by X-ray fluorescence spectrometer (Shimadzu XRF1800) and the results are shown in Table 1. In order to remove macrosegregation and form supersaturated solid solution, the ingots were homogenized at 420 ^{o}C for 24 h and then quenched in hot water (~90 ^{o}C). After that the quenched alloys were aged at different status to form various precipitates before extrusion. Generally, the discontinuous precipitation would happen in the AZ80 alloy when the aging temperature is below 250 ^{o}C and continuous precipitation would happen when the aging time exceeds 310 ^{o}C. Hence, aging at 180 ^{o}C and 330 ^{o}C were conducted in the present study. According to the study of aging process for AZ80[8], the peak hardness would appear after aging for 10 h. However, with aging time increasing to 30 h, the aging hardness would decrease. It is demonstrated that more precipitates precipitate from the matrix when the aging time exceeds 10 h and overaging happens when the aging time exceeds 30 h. Therefore, the process selections of aging at 180 ^{o}C for 12 h, 180 ^{o}C for 48 h and 330 ^{o}C for 48 h are regarded as the aging

Table 1 Chemical composition of AZ80 alloys (wt%)

Elements	Mg	Al	Zn	Mn
Content	90.75	8.40	0.66	0.19

process of discontinuous and peak-aged, discontinuous and overaged, continuous and overaged, respectively. An AZ80 sample without aging treatment before extrusion was also used in these experiments for comparison. The detailed heat treatment schedule is given in Table 2. For the sake of refining the microstructure, the comparison alloy and aged samples were hot extruded to bars with the diameter of 16 mm at 330 ^{o}C with the extrusion ratio of 25:1 at a ram speed of 1 mm s^{-1}. Both the comparison and as-aged samples were pre-heated at 330 ^{o}C for 1 h before extrusion.

The microstructures of the samples before and after extrusion were observed by an optical microscope (OM, OLYMPUS OLS4000). The precipitates evolution of diverse heat treatment states was examined by a scanning electron microscope (TESCAN VEGA 3 LMH SEM) (SEM). After extrusion, uniaxial tensile tests were performed on a SANS CMT-5105 electronic testing machine at the strain rate of 2×10^{-3} s^{-1} at room temperature. The specimens for tensile tests were smooth dog bone-shaped with a gauge size of 5 mm in diameter and 35 mm in length and machined from the as-extruded bars under different heat treatment conditions. The tensile direction was parallel to the extrusion direction and the yield stress was determined as the 0.2% offset. Three tensile specimens were prepared for each condition and the average value was adopted. Afterwards, the fracture images of the tensile specimens were characterized by scanning electron microscopy (TESCAN VEGA 3 LMH SEM).

2 Results and Discussion

2.1 Microstructure before extrusion

Optical microstructure changes of AZ80 alloys under different conditions before extrusion are shown in Fig. 1. It should be noted that the grain size of the specimens subjected to aging treatment is a little bit larger than that of the comparison one. Except for that, there is no obvious difference among the specimens in various states. Fig.2 depicts the SEM morphologies of the AZ80 under different conditions before extrusion. As is shown in Fig.2a, few precipitates can be found in the specimen without aging treatment. After aging treatment, large quantities of second phases precipitate from supersaturated solid solution specimens. These precipitates

Table 2 Heat treatment process for the extruded AZ80 alloy

Designation	Heat treatment process
H1	ST^{a} at 420 ^{o}C/ 6 h, WQ^{b}
H2	ST at 420 ^{o}C/6 h, WQ, aging at 180 ^{o}C/12 h
H3	ST at 420 ^{o}C /6 h, WQ, aging at 180 ^{o}C/48 h
H4	ST at 420 ^{o}C /6 h, WQ, aging at 330 ^{o}C/48 h

^{a} ST, solution treated; ^{b} WQ, water quenched
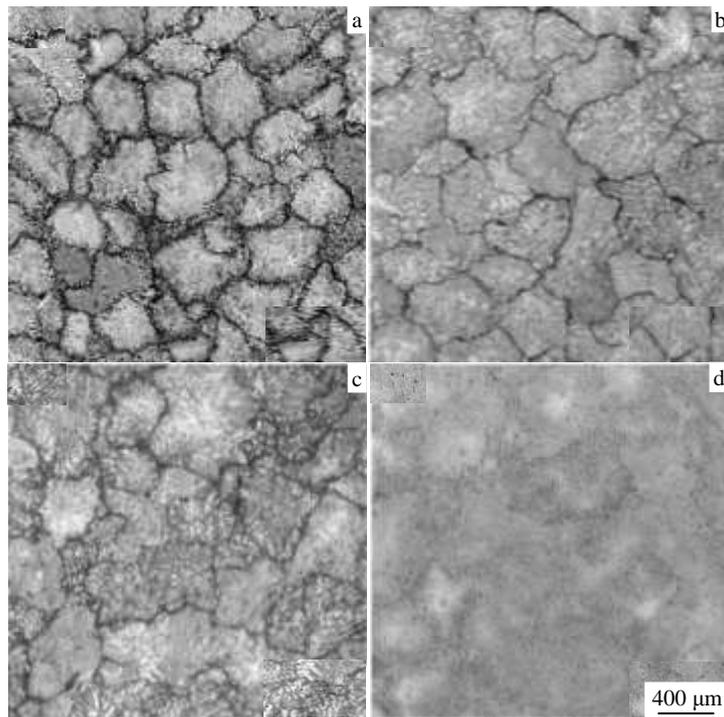
Fig.1   Optical microstructures of AZ80 alloy under different conditions before extrusion: (a) H1, (b) H2, (c) H3, and (d) H4
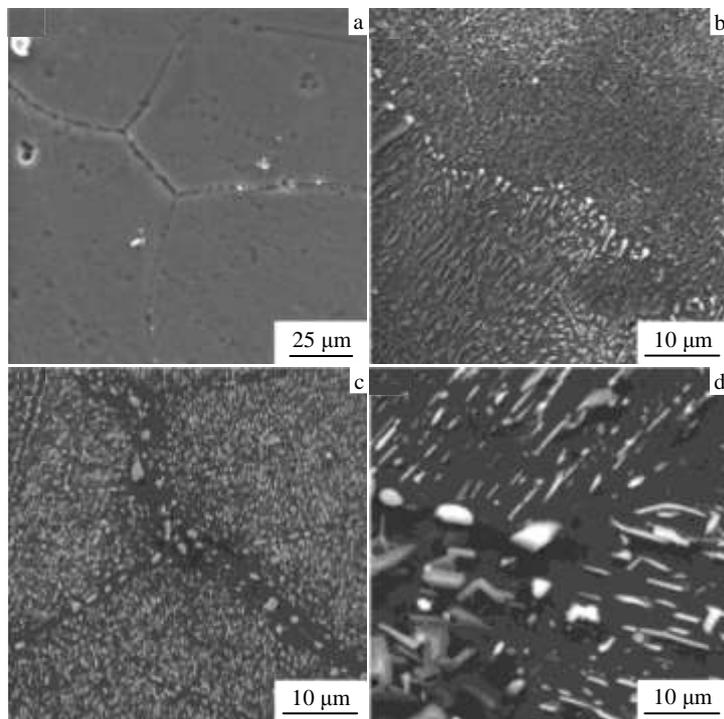
Fig.2   SEM of AZ80 alloy under different conditions before extrusion: (a) H1, (b) H2, (c) H3, and (d) H4

distribute in the grain interior and grain boundaries, and the amount of precipitates in the grain interior is more than that in the grain boundaries (Fig. 2b~2d)). It can be seen clearly that the precipitates get much coarser with elevating aging temperature or/and prolonging the aging time. Besides, it is noteworthy that the precipitates in the interior of the grains

retain a specific orientation relationship with matrix. Many researchers have reported this specific relationship in detail[21,22].

2.2 Microstructure after extrusion

Fig.3 presents the optical micrographs of transverse and longitudinal directions under different conditions after extrusion. Compared with the specimens before extrusion, the grains of all the specimens get refined remarkably due to the occurrence of DRX during hot extrusion. Fig.3a shows the microstructure of the specimen without aging treatment (H1) seen from transverse direction. It consists of approximately equiaxed grains with the size of the range from 20 μm to 40 μm. Fig.3c, 3e and 3g show the microstructures of specimens aged at 180 °C for 12 h (H2), 180 °C for 48 h (H3), 330 °C for 48 h (H4) seen from transverse direction, respectively. The grains of the specimens subjected to aging treatment are much finer than that without aging treatment (H1). Particularly in Fig.3c, it can be seen that the grain is fine and uniform. With prolonging the aging time or elevating the aging temperature, the inhomogeneous microstructures can be observed. Fig.3e and 3g show that a small number of large grains (~10 μm) are surrounded by regions of fine equiaxed grains (~3 μm). From the microstructures under different conditions seen from longitudinal direction (Fig. 3b, 3d, 3f, 3h), they are characterized by some large elongated grains (stripes) along the extrusion direction (ED), surrounded by regions of fine equiaxed grains. Meanwhile, the trend of microstructures' changes seen from longitudinal direction is corresponding to that seen from transverse direction.

SEM images of specimens under different conditions are shown in Fig.4. Apparently, the variation of grain size is consistet with that indicated in the optical microstructures (Fig. 3). Compared with Fig.2, the evolution of precipitates is of great significance. After extrusion, most precipitates are dispersed in the grain boundaries, and the amount of precipitates in the grain interior declines accordingly. Simultaneously, the specific orientation relationship between the precipitates and matrix disappears. In Fig.4a, a spot of precipitates distribute in the grain boundaries. Fig.4b shows almost all the precipitates of the specimen subjected to aging treatment at 180 °C for 12 h, which were initially segregated in the grain interior, distribute in the grain boundaries homogeneously. The occurrence of precipitates segregation in the specimens aged at 180 °C for 48 h and 330 °C for 48 h results from long time overaging (Fig. 4c and 4d). Fine particles' effect of pinning boundary migration is well known and widely observed in magnesium alloys[23]. Bai Jing et al.[24] systematically researched Mg-4Al-2Sr-0.3Mn and Mg-4Al-2Sr-1Ca-0.3Mn and found that the as-extruded Mg-4Al-1Ca-0.3Mn exhibited finer microstructure than as-extruded Mg-4Al-2Sr-0.3Mn. Further research indicated that a small amount of Ca addition had an effect on microstructural refinement for Mg-Al-Sr based alloys. To be specific, the tiny Al_2Ca particles exerted restraining forces upon grain
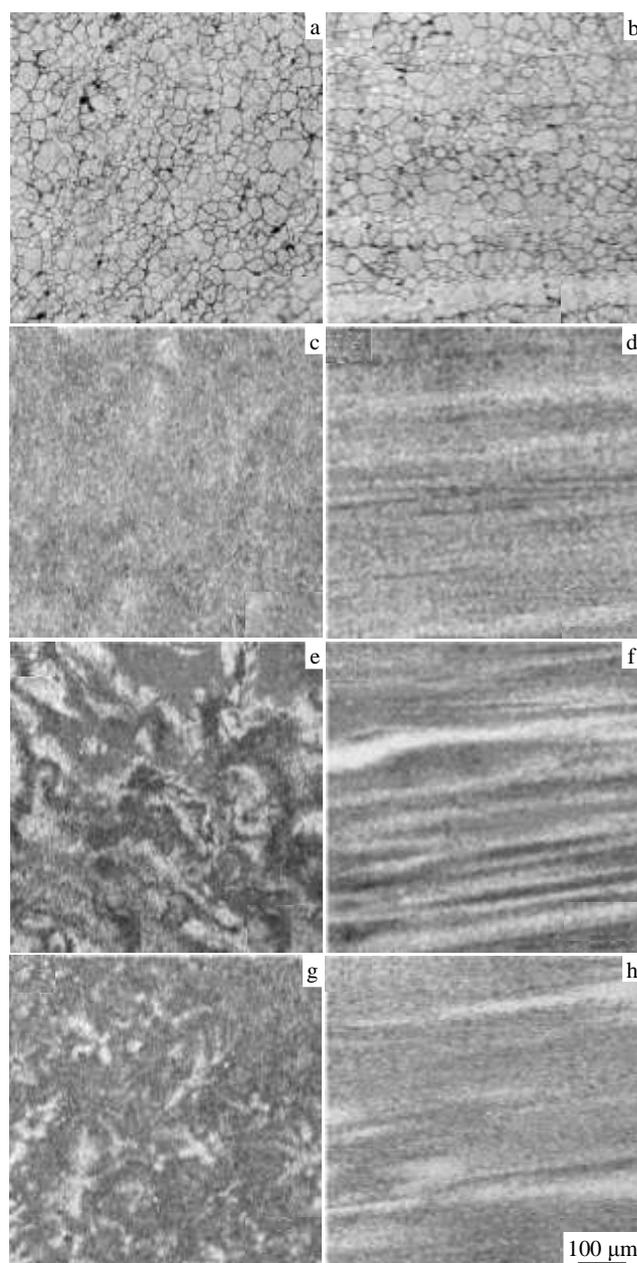
Fig.3 Optical micrographs of transverse (a, c, e, g) and longitudinal (b, d, f, h) direction for AZ80 alloy under different conditions after extrusion: (a, b) H1, (c, d) H2, (e, f) H3, and (g, h) H4

boundaries during recrystallization and were effective in decreasing the grain boundary mobility. The study of S. W. Xu et al.[19] demonstrated that the $Mg_{17}Al_{12}$ precipitates had pinning effect which was greatly influenced by compression temperature. Hence, a pronounced phenomenon in Fig.4c and 4d that the grain size gets finer with the increase of the precipitates in the grain boundaries due to the Zener pinning effect has been observed. The precipitates dispersed in the grain boundaries after extrusion have the effect of pining boundary migration and prevent grain growth availably
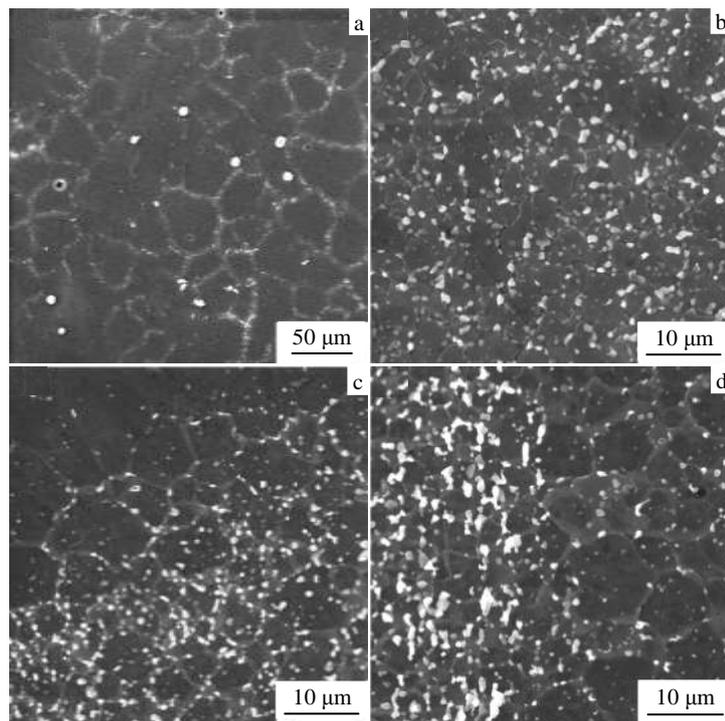
Fig.4   SEM of AZ80 alloy under different conditions after extrusion: (a) H1, (b) H2, (c) H3, and (d) H4

during the dynamic recrystallization. The effect is proportional to the amount of precipitates approximately. From Fig.4c and 4d, the grains with more precipitates in the grain boundaries are fine, on the contrary, the grains with less precipitates in the grain boundaries are coarse. This is the reason why the optical microstructures of the specimens aged at 180 ^{o}C for 48 h and 330 ^{o}C for 48 h (H3, H4) are inhomogeneous (Fig.3e, 3g).

2.3   Mechanical properties

The columnar sections of mechanical property indexes of as-extruded specimens under different conditions obtained through the uniaxial tensile tests are presented in Fig.5. Yield strength and tensile strength of the specimens aged at 180 ^{o}C for 12 h (H2), 180 ^{o}C for 48 h (H3) and 330 ^{o}C for 48 h (H4) are 244 and 360, 236 and 354, 230 and 343 MPa, respectively. In comparison with 213 and 322 MPa of the specimen without aging treatment, yield strength and tensile strength of the specimens with aging treatment increase sharply. The elongation of the specimens without aging treatment (H1), aged at 180 ^{o}C for 12 h (H2), 180 ^{o}C for 48 h (H3) and 330 ^{o}C for 48 h (H4) is 12.1%, 14.3%, 12.9% and 9.5%, respectively. The results accord with the microstructures of all the specimens under different conditions. It has been reported that fine grains help to prevent the formation of deformation twins and activate dislocations on non-basal planes which will lead to the enhancement of strength as well as ductility[25]. Thus, the yield strength, tensile strength and elongation of the specimen aged at 180 ^{o}C for 12 h (H2) are all improved because the microstructure gets finer and more uniform.
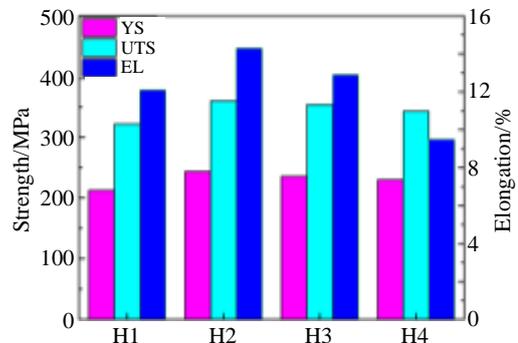
Fig.5   Columnar sections of mechanical property indexes of as-extruded AZ80 alloy under different conditions

Moreover, the Zener pinning effect of the precipitates distributing in the grain boundary during dynamic recrystallization is another important reason for the improvement of mechanical property indexes. Certainly, the enhancement of mechanical properties for AZ80 alloy improves the cost of machining to some extent. However, in some particular situations for the applications of magnesium alloys, compared with the increase of machining cost caused by the improvement of mechanical properties, the improvement of comprehensive mechanical properties results from the refinement of grains will expand the application range of AZ80 alloy and bring greater economic benefits.

2.4   Fracture morphology

Fig. 6 shows the fracture morphologies of the specimens under H1, H2, H3 and H4 conditions after tensile test. The fracture morphologies of all the specimens under different conditions exhibit hybrid fracture of cleavages and dimples. Compared with the specimen without aging treatment, dimples become more and cleavages become less by aging at 180 $^{\circ}$C for 12 h. Whereafter, with the aging time prolonged and/or the aging temperature elevated, dimples become less. Meanwhile, in comparison with the fracture morphologies of the specimens under other heat treatment conditions, some coarse and broken precipitates in the grain boundaries are presented in the fracture morphology of the specimen aged at 330 $^{\circ}$C for 48 h. It is acknowledged that coarse precipitates can lead to the stress concentration and crack initiation and microcracks tend to initiate in the $Mg/Mg_{17}Al_{12}$ interface and even in the $Mg_{17}Al_{12}$ particles[25,26]. Thus, based on the previous analysis, the coarsening of precipitates, segregation of precipitates and inhomogeneity of grains (Fig.3g, 3h and Fig.4d) are the reasons why the elongation of the as-extruded specimen under H4 conditions is lower than that of other conditions. It is well known that the precipitates distributing in the grain boundaries and interiors have the effect of pinning grain boundary migration and strengthening the α-Mg matrix[27]. Therefore, improving deformability of AZ80 by grain boundary migration and non-basal slip induced by grain rotation is strictly limited so as to reduce the ductility and elongation. However, the strength and elongation are both improved of the specimens under H2 and H3 conditions in this study. Based on Fig.3 and Fig.4, the precipitates that disperse in the grain boundaries dominantly after the process of aging

treatment and extrusion pin boundary migration and prevent grain growth during the dynamic recrystallization. Thus, the grains of the specimens subjected to aging treatment are finer than those of solid solution state. The strength and elongation are both improved accordingly.

3 Conclusions

1) The grains of AZ80 magnesium alloy can be refined obviously by aging before extrusion. But with prolonging aging time and/or elevating aging temperature, the grains grow up and become inhomogeneous.

2) The mechanical properties of AZ80 can be improved by aging before extrusion. In particular, both strength and elongation are improved simultaneously. Long aging time and/or high aging temperature lead to the decrease of elongation. To sum up, the most optimized process parameters is aging at 180 $^{\circ}$C for 12 h.

3) The mechanism of optimizing structure and improving mechanical properties by aging before extrusion is Zener pinning effect. The precipitates dispersed in the grain boundaries pin boundary migration and prevent grain growth effectively during the dynamic recrystallization.

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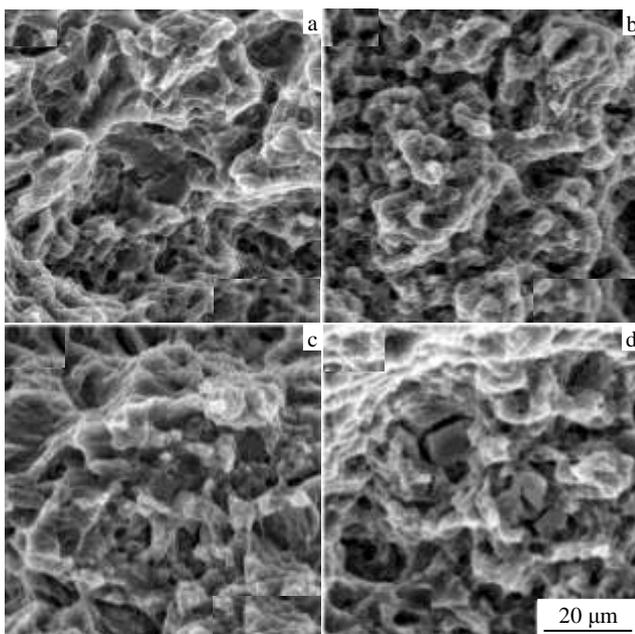
Fig.6 Fracture morphologies of AZ80 under different conditions: (a) H1, (b) H2, (c) H3, and (d) H4

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挤压前的时效处理对 AZ80 镁合金组织和力学性能的影响

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摘　要：主要研究了挤压前的时效处理工艺对 AZ80 镁合金显微组织和力学性能的影响，同时利用扫描电子显微镜对试样断口进行分析。结果表明：挤压前时效处理可以明显细化晶粒；时效过程中析出的 $Mg_{17}Al_{12}$ 粒子弥散分布在晶界处，在动态再结晶过程中起到阻碍晶界移动、阻止晶粒长大、细化组织的作用；随着时效时间的延长或者时效温度的升高，晶粒细化效果减弱；对时效后合金进行挤压，试样的屈服强度、抗拉强度和延伸率均提高。通过对断口形貌的分析发现，早期裂纹产生于晶界处粗大的第二相周围，导致了拉伸过程中延伸率的下降。挤压前时效处理对 AZ80 的强化效果为高性能镁合金的设计和开发提供了一种全新的思路。

关键词：镁合金；再结晶；显微组织；力学性能；齐纳钉扎效应

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