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LETTER

Fabrication of bulk UFG magnesium alloys by cyclic extrusion compression

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The fabrication of bulk ultrafine-grained (UFG) materials has attracted a great deal of attention in recent years because of the recognition that these materials appear to offer several engineering advantages, such as high strength and good ductility at ambient temperature [1–3]. Severe plastic deformation (SPD), such as equal channel angular extrusion (ECAE), high pressure torsion (HPT), cyclic extrusion compression (CEC) and accumulative roll bonding (ARB), has been successfully used to obtain fine grains for metals and alloys. In particular, the mean grain size in Cu and Ni subjected to HPT reaches 107 and 114 nm, respectively [4]. In Mg alloys subjected to ECAE it is 2.5–8.1 μm [5]. In Al alloys subjected to CEC it reaches 2 μm [6], and the mean grain size in IF steel subjected to ARB is 210–700 nm [7].

Magnesium alloys are being increasingly used in the electronics, automobile and aerospace industries due to their low density and high specific strength [8]. With their hexagonal close packed (hcp) structure and low stacking fault energy, they generally exhibit limited ductility and strength at ambient temperature. It is well known that both the mechanical properties and ductility can be improved by grain refinement. Thus, the application of SPD processing has the potential to produce UFG Mg alloys with high strength and good ductility, thereby broadening the use of magnesium alloys. Recently, studies have been performed to produce UFG magnesium alloys by ECAE [9]. Nevertheless, there are only a few reports on the development of the SPD method to address the problems of producing large billets with uniform microstructure. The CEC method originally proposed by J. Richert [10] has the advantage that it can be applied to metals and alloys with unlimited continuous deformation [10, 11]. However, the CEC method has some limitations. Specifically, the die usually consists of several parts. It is easy to make some materials flow into the gap which will lead to the increase of stress obviously. In addition, ductile materials are usually required for CEC processing [10]. The aim of this study is to investigate the possibility of fabricating an UFG AZ31 Mg alloy by the CEC method and to examine the grain refinement mechanism.

The CEC extrusion is a combination of alternating extrusion and compression cycles [12], as shown in Fig. 1. The extruder consists of two rams and one cylindrical die, all of which are made of H13 steel. The cylindrical die has two chamber of equal diameters d_0. The two chambers are connected by a smaller diameter d_m. The basic difference between our CEC extruder and those of M. Richert [12] and J.W. Yeh [11] is that the die is a solid die. The extrusion force F_A and F_B for processing is controlled and can be altered by the operator.

The processing operation involve several steps: first, the sample is placed into the upper chamber and is pushed by ram A to pass through the chamber with smaller diameter; meanwhile, ram B exerts a back extrusion force F_B on the extruded material to restore its initial shape. In this instance, F_B is smaller than F_A. Second, the ram B is reversed to push the sample to flow into upper chamber through the smaller chamber, and the ram A is compelled to move with the strained material because the F_B is greater than F_A. This deformation cycle is repeated so that unlimited true strain can be obtained. For the final extrusion, the opposite ram is removed, and the magnitude of the cumulated true strain is approximately

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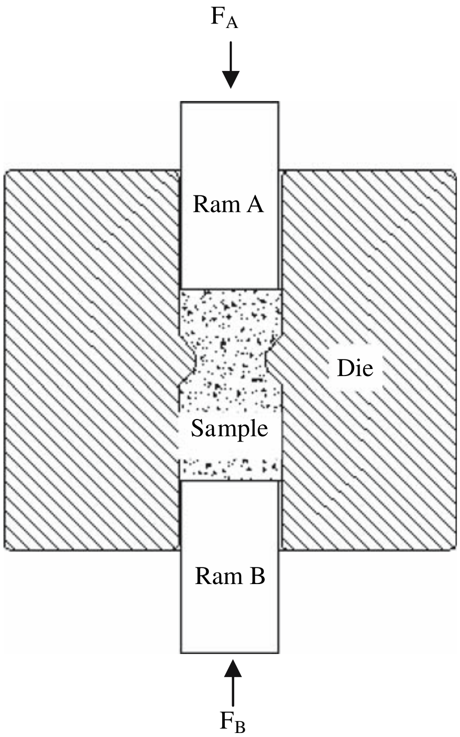


Fig. 1 A schematic drawing of CEC method

$$\varphi = 2n \cdot \ln\frac{d_0^2}{d_m^2} - \ln\frac{d_0^2}{d_m^2} = 2(2n-1)\cdot\ln\frac{d_0}{d_m} \quad (1)$$

where n is the number of passes through the smaller diameter d_m, d_0 the chamber diameter and d_m the smaller diameter. In the present paper, d_0 and d_m are 30 mm and 20 mm, respectively. This gives a strain of $\varepsilon = 0.81$ for first pass.

In this study, AZ31 (Mg–3.091%–1.023%Zn–0.421%Mn) ingots and the die were held for 10 min at 100 °C, and were coated with a lubricant of graphite powder. Next, they were heated to 300 °C and held for about 2 h. The sample was put into the upper chamber to start the CEC processing cycle. The extrusion rate was 7 mm/s. The die was air-cooled when the CEC cycle began. The extrusion passes for AZ31 Mg alloy were 1 (single extrusion) and 6, respectively. For the final pass, one of the rams was removed and the strained material was extruded as a rod 20 mm in diameter by another ram.

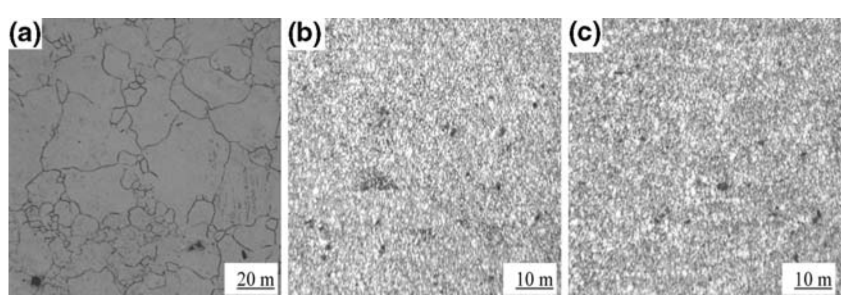
Longitudinal sections and cross-section specimens were prepared for optical microscopy. Thin foils for TEM examination were prepared from the longitudinal section.

Figure 2 shows the optical microstructures of AZ31 Mg alloy processed by the CEC method at 300 °C. It can be seen from Fig. 2a that fine grains have formed adjacent to the coarse grains after 1 pass (that is, one single extrusion) and appear at the original grain boundaries, particularly at triple junctions. The size of fine grains is about 6 μm, and the mean grain size is about 15 μm. After the sixth CEC pass, the grains have been significantly refined (as observed in Fig. 2b, c). In both the longitudinal section and cross section specimens, an UFG structure is observed; the mean grain size is less than 1 μm.

Transmission electron microscopy was performed to obtain detailed information about the microstructure of AZ31 Mg alloy processed by the CEC method after 6 passes. Figure 3 clearly shows that the UFG structure has formed after 6 passes with the CEC process. Dislocations are visible within some grains, especially close to the grain boundary, as shown in Fig. 4. It appears that the dislocations have the tendency to rearrange to network structures.

Based on the microstructural observations via OM and TEM, the grain refinement of the AZ31 magnesium alloy subjected to CEC is postulated as follows: First, the stresses are highly concentrated close to the grain boundaries where the motion of basal dislocations is impeded [13]. Therefore, the cross-slip and climb of basal dislocations occurs near grain boundaries [14]. Second, as the metal flows in the longitudinal direction during extrusion and in the reverse direction during compression, a high proportion of dislocations with different Burgers vector can be continuously produced by CEC. The increasing dislocation density can result in the generation of non-basal dislocations [13]. To further reduce the stress concentration, the dislocations may form network structures by cross-slip and climb when the dislocation density within the grains achieves some critical value (Fig. 4). Third, the network structures may develop into high angle grain boundaries (HAGBs) as a kind of continuous dynamic

Fig. 2 Optical microstructures of AZ31 Mg alloy processed by the CEC method at 300 °C. (**a**) 1 pass, longitudinal section; (**b**) 6 passes, transverse cross-section; and (**c**) 6 passes, longitudinal section



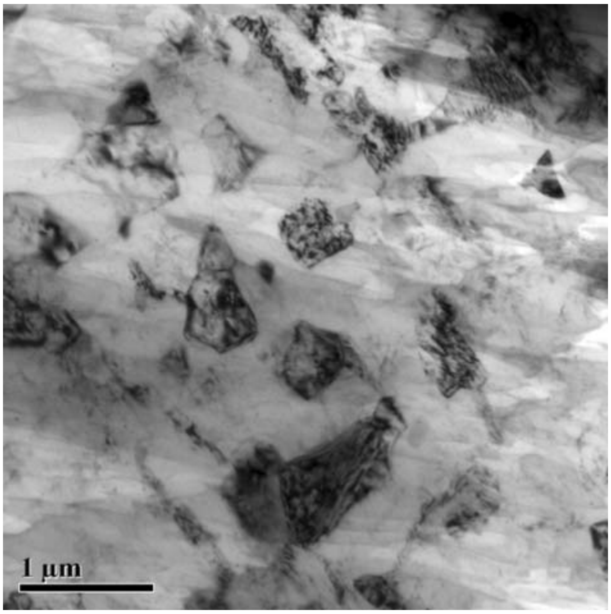


Fig. 3 TEM bright-field micrograph from the longitudinal section of AZ31 Mg alloy processed by the CEC method after 6 passes

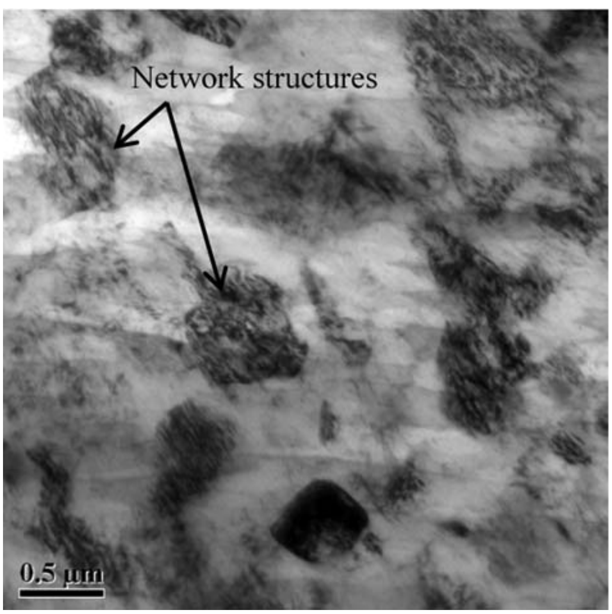


Fig. 4 TEM micrograph of the AZ31 Mg alloy processed by the CEC method after 6 passes showing the network structures

recovery and recrystallization (CDRR) [15, 16]. Lastly, with the accumulation of true strain, the dislocations are continuously produced in the new fine grains to refine the grains till a balance occurs between the generation and annihilation of dislocations.

In conclusion, a solid die for CEC was designed to overcome the problem of materials in gap. The CEC method was successfully applied to fabricate an UFG AZ31 Mg alloy. The microstructures of the AZ31 Mg alloy subjected to the CEC method with only 6 passes are reasonably equiaxed and homogeneous, and the mean grain size is less than 1 μm. Combined continuous dynamic recovery and recrystallization is the primary mechanism for grain refinement, and the balance of the generation and annihilation of dislocations appears to limit further grain refinement.

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