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Microstructure and mechanical properties of AZ31−Mg_2Si in situ composite fabricated by repetitive upsetting

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Abstract: AZ31−4.6% Mg_2Si (mass fraction) composite was prepared by conventional casting method. Repetitive upsetting (RU) was applied to severely deforming the as-cast composite at 400 °C for 1, 3, and 5 passes. Finite element analysis of the material flow indicates that deformation concentrates in the bottom region of the sample after 1 pass, and much more uniform deformation is obtained after 5 passes. During multi-pass RU process, both dendritic and Chinese script type Mg_2Si phases are broken up into smaller particles owing to the shear stress forced by the matrix. With the increasing number of RU passes, finer grain size and more homogeneous distribution of Mg_2Si particles are obtained along with significant enhancement in both strength and ductility. AZ31−4.6% Mg_2Si composite exhibits tensile strength of 284 MPa and elongation of 9.8% after 5 RU passes at 400 °C compared with the initial 128 MPa and 5.4% of original AZ31−4.6%Mg_2Si composite.
Key words: AZ31−Mg_2Si composite; Mg_2Si particle; repetitive upsetting; microstructure; mechanical properties

1 Introduction

Mg matrix composites are superior candidates for lightweight structural applications in the industry due to their low density and high specific strength. Mg–Si alloys are in situ Mg matrix composites containing hard Mg_2Si particles [1]. It has been proved that adding Si to pure Mg or its alloys can produce substantial improvement in strength, hardness, creep resistance and wear property [2–4]. However, a mass of coarse dendritic Mg_2Si and Chinese script type Mg_2Si phases incline to form in the as-cast Mg–Si alloys when a high amount of Si is presented, and they are harmful to the mechanical properties [5]. For the sake of obtaining fine Mg_2Si particles with beneficial shapes, different kinds of methods have been employed, such as rapid solidification [6], modification [7,8], solution treatment [9,10], mechanical alloying [11], and hot extrusion [12]. However, refining of Mg_2Si is very limited, and it is difficult to achieve uniform distribution. Recently, researchers have paid much attention to the severe plastic deformation (SPD) techniques such as equal channel angular pressing (ECAP) [13,14], cyclic extrusion compression (CEC) [15,16], high pressure torsion (HPT) [17,18], cyclic closed-die forging (CCDF) [19], and repetitive upsetting (RU) [20]. SPD has been accepted as one of the most effective methods for processing bulk ultrafine-grained (UFG) materials. SERRE et al [21] studied the microstructure of AZ31 Mg alloy processed by HPT through 5 turns at 453 K. The results showed that HPT introduced significant grain refinement to an average grain size of ~0.5 μm. GAN et al [22] investigated the effect of ECAP on Mg–3.2Si alloy. They found that grains and eutectic Mg_2Si particles were refined. Furthermore, the yield strength and tensile strength were notably increased. WANG et al [23] studied microstructure and mechanical properties of AZ31–0.5Si alloy processed by ECAP. The results indicated that the Chinese script type Mg_2Si phase broken up and dispersed in the matrix, and the ductility was significantly improved. The study by ZHANG et al [24] revealed that most Mg_2Si particles in Mg−9Al−6Si alloy were less than 20 μm after 12 CEC passes. In

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addition, the investigation by METAYER et al [25] showed that wear resistance of Mg−1.5Si alloy improved with CCDF process passes.

Compared with other SPD techniques, RU is a novel processing method and it produces higher strains per pass. More homogeneous structure of the material may be obtained because it involves multi-directional deformation. The relevant investigation showed that AZ31 alloy exhibited mean grain size of 1.3 μm and yield strength of 294 MPa after 5 RU passes at 250 °C. Moreover, more homogeneous microstructure of the AZ31 alloy was obtained [26]. The microstructure and texture evolution of Mg−9.8Gd−2.7Y−0.4Zr alloy processed by RU were also investigated. Reasonably equiaxed uniform microstructure was achieved and randomized texture was obtained [20]. The study by LIU et al [27] showed that with the increase of RU passes, the grains of the Mg−3.03Nd−0.24Zn−0.49Zr alloy were significantly refined and the alloy exhibited elongation of 30.1% after 8 RU passes. It is expected that the RU process will be an excellent candidate for refining Mg_2Si particles and homogenizing their distribution in Mg matrix. However, few studies have been carried out on RU processing of Mg matrix composites.

In the present work, the deformation flow process of the sample during repetitive upsetting was simulated by using DEFORM-3D. Then, the improvements of microstructure and mechanical properties were examined in an as-cast AZ31−Mg_2Si composite subjected to RU at high temperature. Emphases were put on the refinement of grain and the homogeneous distribution of Mg_2Si particles induced by RU.

2 Experimental

Commercial AZ31 Mg alloy ingot (Mg−3Al−1Zn−0.4 Mn, mass fraction, %) and Si particles (99.9%, mass fraction) were used as starting materials to fabricate the AZ31−Mg_2Si composite. After the melt was poured into a steel mold and the ingot was obtained, the composition was measured and the actual Si mass fraction of the AZ31−Mg_2Si composite was 1.7% (i.e., AZ31−4.6% Mg_2Si).

Before RU, as-cast AZ31−4.6%Mg_2Si billet was machined into samples with dimensions of *d*100 mm× 20 mm. The sketch of the RU process is shown in Fig. 1. The device consisted of upper die, lower die, and a punch for external loading. The chamber of the lower die and the sample were of equal dimension. A sample lubricated with graphite was placed into the upper die before being heated to 400 °C followed by holding at this temperature for 30 min. Then it was pressed into the lower die by the punch with a constant speed (4 mm/s). After pressing, each sample was taken out and rotated 90° around *Y*-axis, and reinserted into the upper die for next pressing. Samples were exposed to 1, 3, and 5 passes of RU processing, respectively.

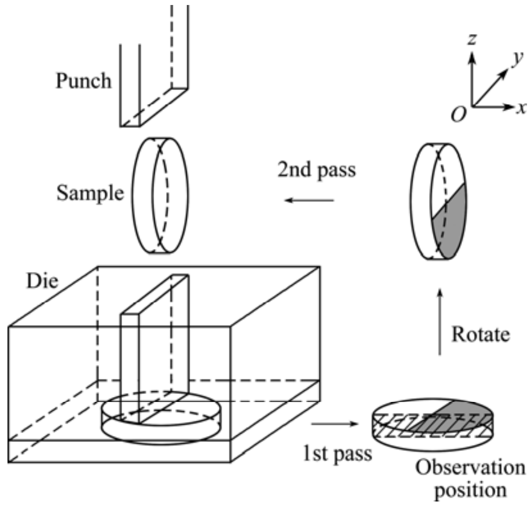

Fig. 1 Schematic representation of RU processing

The position of microstructure observation is marked in Fig. 1, which locates at the largest *Y*-plane of the sample. The burnished AZ31−4.6% Mg_2Si composite was eroded by a mixture of 1 g oxaldehyde, 1 mL nitric acid and 1 mL acetic acid in 150 mL water. Microstructure was observed by using optical microscopy. The flat tensile specimens (with a gage section of 10 mm×3.5 mm×2 mm) were machined from the central part of the RUed samples with the tensile axes parallel to *Y*-direction. Tensile tests were conducted at room temperature with an initial strain rate of 8.3×10^{-4} s^{-1}.

3 Finite element modeling

The RU process of AZ31−4.6%Mg_2Si composite at 400 °C was simulated by using the commercial finite element (FE) code DEFORM−3D. During RU processing, a flat-faced punch was applied to pushing the composite sample through the chamber of upper die for a total displacement of 80 mm, and the constant speed of the punch was 4 mm/s. The die geometry was taken with chamber angle Φ=90° and corner angle Ψ=0°. The cylindrical sample had a diameter of 100 mm and a height of 20 mm. The punch and die were modeled with rigid bodies. The sample was modeled as elastic-plastic material with Poisson ratio (ν) of 0.35 and elastic modulus (E) of 45 GPa, which were based on the data of AS41 alloy [28]. The stress−strain relationship of the plastic deformation of AZ31−4.6% Mg_2Si at different strain rates were established using the testing data generated by GLEEBLE 3500 thermal simulator, and the data loaded into the software included material behavior at temperatures from 300 °C to 450 °C with strain rates from 0.005 to 0.5 s^{-1} [29]. The sample was meshed with 6001 four node tetrahedron elements. All simulations used automatic remeshing to adapt the high strains and the germination of flow localization during RU. A friction coefficient between the die and the sample was adopted to be 0.3, which was based on the data of AZ31 alloy [30].

4 Results and discussion

4.1 FE simulation of RU process

Figure 2 shows the different stages of deformation flow net of the sample from 1 to 5 RU passes. Figures 2(a), (b) and (c) show the beginning, 80 % completing, and the finish of the first upsetting pass, respectively. The flow net clearly reveals that the deformation is concentrated in the bottom region of the sample. In order to understand the deformation behavior easily, four corners (1, 2, 3, 4) of the sample are indicated. During the first upsetting pass, the bottom corners (3, 4) are extensively elongated. However, the top corners (1, 2) stay in the same location even at the finish of deformation. This explicitly shows that the initial bottom region of 20 mm is elongated to the final bottom region of about 100 mm, and the top region doesn't experience any deformation. In addition, very high tensile deformation is attained in the bottom region. Figures 2(d) and (e) show flow net of the sample after 3 and 5 RU passes. After 3 passes, it can be seen that the flow net becomes very disorder. Furthermore, more homogeneous blend of the flow net is obtained after 5 passes. This suggests that more uniform deformation of the AZ31−4.6%Mg_2Si composite will be achieved with the increase of pass number.

The velocity vector field of the sample during upsetting process at 50% of deformation is shown in Fig. 3. It is the stage that the sample is forced to flow through the 90° die-chamber intersection after the upper die is filled. The material divides into two flows in the lower die and each stream is similar to a typical ECAP. On *X*-plane, the material points at the upper end have quicker flowing speed.

4.2 Microstructural evolution during RU process

Figure 4(a) shows the microstructure of as-cast AZ31−4.6%Mg_2Si composite, which is composed of α-Mg matrix (light gray color), Mg_2Si phase (gray color) and $Mg_{17}Al_{12}$ phase (white color). The Mg_2Si phases are presented as dendritic and Chinese script type morphologies. The dendritic primary Mg_2Si particles have a length along the first axial direction more than 80 μm. The microstructures of the composite subjected to 1, 3, and 5 RU passes at 400 °C are shown in Figs. 4(b), (c) and (d), respectively. After 1 pass, the average grain size is greatly refined to ~11.3 μm, which is attributed to the dynamic recrystallization (DRX) during upsetting. Grain refining by DRX in deformed Mg has been widely observed, peculiarly at high temperatures [31,32]. Since Mg has fairly lower stacking fault energy (60−78 kJ/mol), DRX generally dominates in deformed Mg at elevated temperatures (above 513 K) [33]. SU et al [34] studied the microstructure evolution of AZ31 alloy during ECAP at 200 °C, and the results showed that the grain refinement mechanism was a combination of mechanical shearing and subsequent continuous recovery, recrystallization, growth of grains and subgrain cells to produce refined and equiaxed grains [34]. It is known that large particles (>1 μm) activate nucleation for recrystallization while small particles prohibit recrystallization [35]. In the present work, since the Mg_2Si particles are much larger than 1 μm, the particles activate nucleation for recrystallization during upsetting. The grain distribution of the composite is still not homogeneous with blended microstructure of coarse

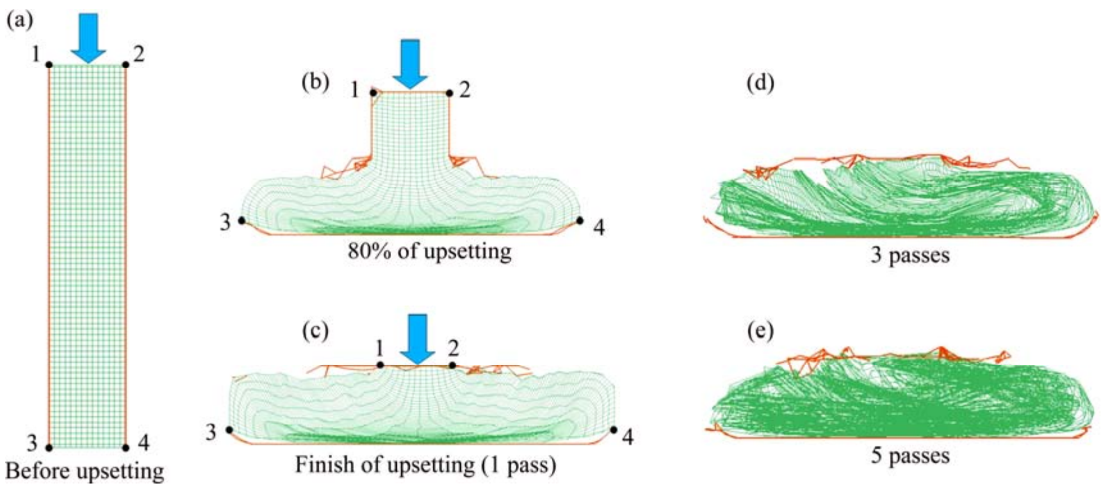

Fig. 2 Deformed sample drawn by flow net during different stages of RU: (a) Beginning; (b) 80% completing; (c) Finish of first upsetting pass; (d) After 3 passes; (e) After 5 passes

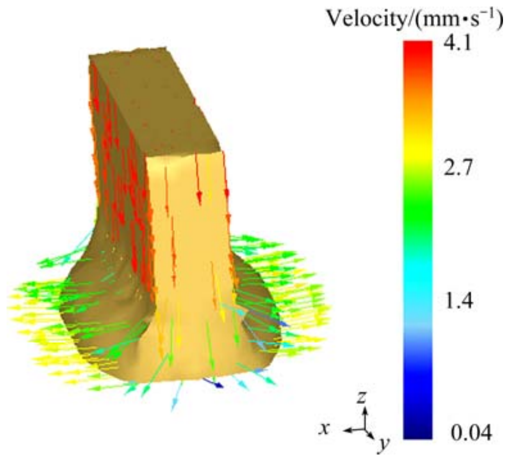

Fig. 3 Velocity vector field of sample during upsetting process at 50% of deformation

and fine grains. The grains continue to be refined upon further upsetting. After 3 passes, the average grain size is refined to ~7.2 μm. A uniform grain structure is obtained after 5 passes and the mean grain size decreases to 6.3 μm. Activation of recrystallization and limitation of grain growth by the presence of the particles lead to the grain refinement.

The effect of RU processing on the Mg_2Si morphology is also notable. After 1 pass, the orderly distribution of dendritic and Chinese script type Mg_2Si particles is broken up. These coarse Mg_2Si particles are divided into smaller ones and the microstructure is still very inhomogeneous. With more upsetting pass, higher strain is imposed to the composite and the proportion of broken particles increases. With continuous 90° revolve of the sample for following pass processing, a cross-shear exerts on the sample. Mg_2Si particles are further broken into finer ones and their distribution becomes more homogeneous. After 5 passes, most of the Mg_2Si particles are nearly equiaxed with a size less than 5 μm. In this work, upsetting introduces the mushrooming deformation and makes the Mg matrix as well as Mg_2Si particles flow transversely at the former pass, and then upsetting makes them move longitudinally at the following pass. During the multi-pass processing, Mg_2Si particles are broken into smaller fragments by the shearing stress imposed by the matrix. Thus, the Mg_2Si particles are refined gradually and their distribution becomes uniform. It should be noticed that there are still some large polygonal Mg_2Si particles of ~7 μm. This could be attributed to that the shearing stress imposed by the matrix flow is not sufficient to further break these Mg_2Si particles into smaller ones.

4.3 Mechanical properties improvement after RU

The tensile stress–strain curves at room temperature for AZ31−4.6% Mg_2Si composite after RU processing are shown in Fig. 5. The strength and ductility continuously improve with an increase of the RU pass number. Finally, the composite shows an improvement in the ultimate tensile strength (UTS) from 128 MPa for

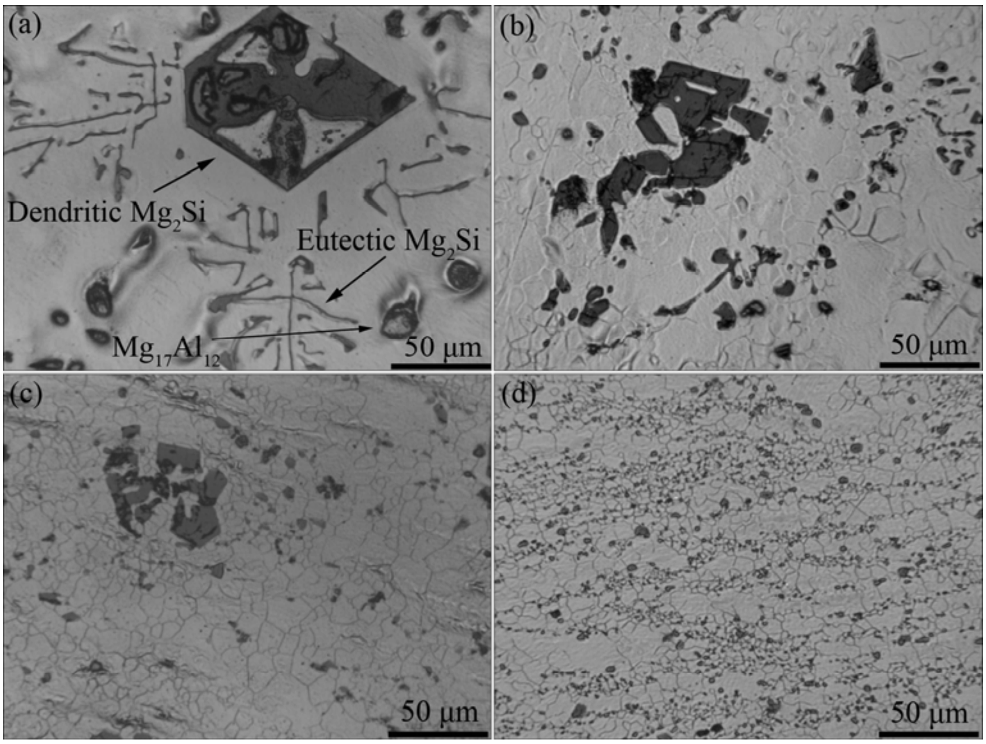

Fig. 4 Microstructures of AZ31−4.6% Mg_2Si composite after different passes of RU processing at 400 °C: (a) As-cast; (b) 1 pass; (c) 3 passes; (d) 5 passes

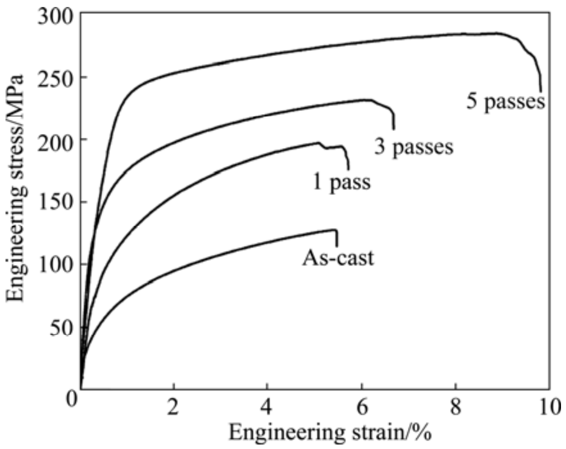

Fig. 5 Tensile stress−strain curves of AZ31−4.6% Mg_2Si composite processed by RU at 400 °C

0 pass (the as-cast sample) to about 284 MPa for 5 passes. The influence of RU on the yield strength (YS) is more effective, and the YS increases from 43 MPa for 0 pass to about 205 MPa for 5 passes. The composite after 5 passes exhibits higher elongation to fracture of about 9.8% in comparison to only 5.4% for the as-cast sample. The mechanical properties of Mg−Si alloys are widely known to strongly relate to the size, shape, and distribution of the Mg_2Si phases in the microstructure [36,37]. In the as-cast AZ31−Mg_2Si composite, coarse dendritic or Chinese script morphology Mg_2Si particles make the AZ31 matrix discontinuous. The matrix may strongly deform and it leads to fracture during tensile testing. Also, stress concentrations and high strain occur easily in the AZ31matrix near the sharp tips of the coarse dendritic or Chinese script type Mg_2Si particles. As such, the microcrack inclines to nucleate and propagate in the particles, and void is formed at the interface between the matrix and the particles. With the accumulation of strain, microcrack quickly grows and void coalesces. Thus, the tensile properties of as-cast composite are deteriorated. After 5 passes of RU, the refined Mg_2Si particles distribute homogeneously in the grains and along grain boundaries. Acting as strong obstructor, the particles can pin and retard the dislocation's movement and therefore strengthen the composite by dispersion strengthening mechanism. The finer Mg_2Si particles are not easy to fracture under external stress and lead to higher UTS. The transformation of coarse irregular Mg_2Si particles to polygonal ones reduces stress concentration on Mg_2Si and the matrix. Thus, the fracture possibility of them decreases. Also, the uniform distribution of Mg_2Si confines Mg matrix deformation path and results in complex deformation pattern for the enhancement of ductility. Refinement and uniformity of the matrix grain after RU also contribute to the improvement of strength and ductility. Mg alloy with large size grain is prone to form twins in a narrow region at higher strains leading to fracture under tension, whereas smaller size grain results in more homogeneous deformation.

5 Conclusions

1) The FE simulation by DEFORM-3D indicates that deformation is concentrated in the bottom region of the sample after 1 pass, and much more uniform deformation is obtained after 5 passes.

2) Mg_2Si and grain size gradually decrease with the increasing pass number. After 5 passes, the original large dendritic and Chinese script type Mg_2Si particles are broken up into much finer ones with homogeneous distribution in matrix with an average grain size of about 6.3 μm.

3) Both strength and ductility of the composite improve notably with the increasing RU pass number. AZ31−4.6% Mg_2Si composite shows a tensile strength of 284 MPa and elongation of 9.8% after 5 RU passes at 400 °C compared with the initial 128 MPa and 5.4% of as-cast alloy.

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反复镦压 AZ31−Mg_2Si 原位复合材料的组织和力学性能

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摘 要：采用常规铸造方法制备 AZ31−4.6% Mg_2Si 复合材料，在 400 °C 对铸态复合材料进行 1、3 和 5 道次的反复镦压(RU)剧烈变形。材料流动的有限元分析表明：1 道次后变形集中于试样底部区域，5 道次后可获得均匀的变形。多道次反复镦压过程中基体施加的剪切应力使树枝状和汉字状 Mg_2Si 相破碎成小颗粒，随着反复镦压道次的增加，晶粒尺寸逐渐减小，Mg_2Si 颗粒分布逐渐均匀，同时强度和塑性显著提高，在 400 °C 反复镦压 5 道次后，AZ31−4.6% Mg_2Si 复合材料抗拉强度和伸长率分别为 248 MPa 和 9.8%，而原始铸态复合材料的抗拉强度和伸长率分别只有 128 MPa 和 5.4%。

关键词：AZ31−Mg_2Si 复合材料；Mg_2Si 颗粒；反复镦压；组织；力学性能

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