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# Hot extrusion of SiC<sub>p</sub>/AZ91 Mg matrix composites

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**Abstract:** SiC particles reinforced AZ91 Mg matrix composites (SiC<sub>p</sub>/AZ91) with SiC volume fractions of 5%, 10% and 15% were fabricated by stir casting. After T4 treatment, these composites were extruded at 350 °C with an extrusion ratio of 12:1. In the as-cast composite, particles segregated at a microscopic scale within the intergranular regions. Hot extrusion almost eliminated this particle aggregation and improved the particle distribution of the composites. In addition, extrusion refined the grains of matrix. The results show that hot extrusion significantly improves the mechanical properties of the composites. In the as-extruded composite, with the increase of SiC<sub>p</sub> contents, the grain size of the extruded composites decreases, the strength and elastic modulus increase but the elongation decreases.

Key words: extrusion; Mg matrix composites; SiC<sub>p</sub>; AZ91 magnesium alloy; volume fraction

# **1** Introduction

As magnesium matrix composites have high specific stiffness and specific strength, they promise wide applications in aerospace, defense and automobile industries [1-3]. In order to extend the applications of magnesium matrix composites, secondary processing has to be well developed. Extrusion can yield a full integrity wrought material because of its combination of the hot compaction and mechanical working [4]. Therefore, extrusion is an increasingly attractive processing route for a class of materials which are difficult to successfully deform [5–10]. Most metal matrix composites belong to this class of materials. Many discontinuously reinforced aluminum matrix composites are thermomechanically processed by extrusion [7-10]. However, the effect of extrusion on the microstructure and mechanical properties of magnesium matrix composites has not been fully investigated [5,6].

It has been reported that extrusion can improve the particle distribution and refine the grains of matrix through recrystallization in magnesium matrix composites [11,12]. Particle cracking induced by extrusion has also been observed in magnesium matrix composites [12]. All these certainly influence the mechanical properties of the extruded composites.

Although there are some reports on the effect of extrusion on the microstructure evolution and properties of Mg matrix composites, the effects of particle sizes and volume fractions on the microstructure and mechanical properties of as-extruded magnesium matrix composites are limitedly studied. Therefore, this work is aimed to study the influence of particle volume fraction on the microstructure and mechanical properties of SiC particles reinforced AZ91D magnesium matrix composites (SiC<sub>p</sub>/AZ91).

## **2** Experimental

#### 2.1 Fabrication and extrusion of composites

SiC<sub>p</sub>/AZ91 composites with SiC volume fractions of 5%, 10% and 15% and particle size of 10  $\mu$ m were fabricated by stir casting in a protective atmosphere of CO<sub>2</sub> and SF<sub>6</sub>. The details of fabrication process were described in Ref. [13]. The billets of the composite and AZ91 alloy were solutionized at 415 °C for 24 h (T4 treatment). Then, the composites were extruded at 350 °C with constant extrusion ratio of 12:1 and constant ram speed of 15 mm/s. Extrusion was conducted in a 2000 kN press machine. The billets, pressure-pad and dish-shaped die were put into the extrusion container. The extrusion container was heated to the given temperature in a muffle furnace. The temperatures of

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the container were monitored by a K-type thermocouple which was inserted into the hole drilled in the container. Once the container reached the desired temperatures, a period of 1 h was allowed to elapse before the extrusion was carried out. This time was long enough to allow the billet to reach a steady-state temperature, as determined from previous tests. For the purpose of comparison, AZ91 alloys were also extruded under the same conditions as the composites.

In this work, the composite or alloy extruded at extrusion temperature of 250 °C and ratio of 12 was designated by 250R12 composite or alloy (by parity of reasoning, 300R12 and 350R12).

#### 2.2 Material characterization

Microstructural examinations were carried out by optical microscope (OM) and scanning electron microscope (SEM). The specimens for OM were ground, polished and etched in acetic picral (5 mL acetic acid + 6 g picric acid+10 mL H<sub>2</sub>O+100 mL ethanol (95%)). Specimens for SEM were ground and polished with great

care in order to avoid causing particle damage in this stage.

#### 2.3 Mechanical properties test

The tensile mechanical properties of the extruded  $SiC_p/AZ91$  composites and alloys were measured by an Instron1186 universal testing machine at a constant cross-head speed of 0.5 mm/min. The tensile property data (yield strength, ultimate tensile strength, elastic modulus and elongation) were based on the average of 3–5 tests.

### **3 Results and discussion**

#### 3.1 Particle distribution

The particle distribution is not very uniform in the as-cast composites, as shown in Fig. 1. SiC particles are segregated at a microscopic scale within the intergranular regions, i.e. there are particle-dense regions (PDRs) and particle-free regions in the composite and PDRs correspond to the intergranular regions [13]. This is



**Fig. 1** SEM images of as-cast and as-extruded composites with different volume fractions of SiC particles: (a) As cast, 5% SiC<sub>p</sub>; (b) As-extruded, 5% SiC<sub>p</sub>; (c) As cast, 10% SiC<sub>p</sub>; (d) As-extruded, 10% SiC<sub>p</sub>; (e) As cast, 15% SiC<sub>p</sub>; (f) As-extruded, 15% SiC<sub>p</sub> (parallel to extruded direction)

called necklace-type particle distribution, which is very typical in metal matrix composites fabricated by stir casting caused by the "push" effect of solidification front [14]. During the solidification of SiC<sub>p</sub>/AZ91 composite produced by stir casting, most particles are pushed ahead of the solidification front while primary magnesium grains grow, so these particles cluster in the intergranular regions during the impingement with other growing grains [14]. Particle segregation restricts the flow of liquid before the composite is completely solidified, so microcavities can be formed in these regions and the particle/matrix interface in PDRs is weak and defective [14,15]. Therefore, it is necessary to eliminate those cast defects using secondary deformation, such as hot extrusion.

Hot extrusion can improve the particle distribution of the composites, although SiC particles are aligned parallelly to the extrusion direction. Figure 1 shows the particle distribution parallel to the extrusion direction. The segregation of particles is largely eliminated by extrusion. As a result, the necklace-type particle distribution is not evident in the extruded composites. In addition, the particle alignment induced by extrusion causes that the longest axes of particles are parallel to the extrusion direction, resulting in the fact that the particle sizes in the extrusion direction are larger than those in the as-cast composites where the particle orientation is random. Uniform particle distribution is beneficial to the mechanical properties of composites. What is more, the particle alignment can improve their mechanical properties along the extrusion direction.

#### 3.2 Matrix microstructure

Hot extrusion can significantly refine the grains of the alloy and the composites, as shown in Fig. 2. Before



**Fig. 2** Optical micrographs of AZ91 alloy and composites under as-cast and as-extruded conditions: (a) As-cast AZ91 alloy; (b) As-extruded AZ91 alloys; (c) As-cast composites, 5%  $SiC_p$ ; (d) As-extruded composites 5%  $SiC_p$ , (e) As-cast composites, 10%  $SiC_p$ ; (f) As-extruded composite, 10%  $SiC_p$  (parallel to extruded direction)

hot extrusion, the average grain sizes of the composites with 5% and 10% SiC particles are about 95 and 65  $\mu$ m, respectively. After extrusion, the grain sizes are refined to 15 and 10  $\mu$ m, respectively. In addition, the grain size of AZ91 alloy is refined from 350 to 20  $\mu$ m by hot extrusion. The grain refinement is caused by dynamic recrystallization (DRX) which takes place during hot extrusion.

Particles affect the DRX of the matrix during hot deformation. The average grain sizes decrease as the volume fraction of SiC particles increases, as shown in Figs. 2(b), (d) and (f). Additionally, the grains in particlerich zones are much smaller than those in particle-poor zones, indicating local content of particles can also influence the DRX of the matrix. Particles affect the DRX of matrix by forming particle deformation zones (PDZs) near particles [16]. PDZ has been widely observed and studied in Al matrix composites [7,16], and recently it has been also investigated in Mg matrix composites [6]. During deformation, the mismatch between a non-deforming particle and ductile metal matrix during deformation leads to the enforced strain gradient in the matrix near a particle [16]. The strain gradient creates a region which contains high dislocation density and large orientation gradient. This region is called PDZ [16]. PDZ can extend to a distance of about a diameter of the particle from the surface of the particle and it may be misoriented by tens of degree from the adjacent matrix [16-18]. Therefore, PDZ is an ideal site for the development of a recrystallization nucleus, and particles can stimulate the nucleation of DRX [16–18]. Thus, the nucleation rates of composites are higher than those in the matrix alloy. Besides PDZ, particles can hinder DRX gain growth because particles can pin DRX grain boundaries and retard their movement. As shown in Figs. 2(d) and (f), many DRX grain boundaries contact with SiC particles, and those particles certainly retard the movement of those grain boundaries. It is expected and observed that the grains away from particles are larger than those near particles. In a word, the addition of particles refines the grains of matrix during hot deformation.

#### 3.3 Mechanical properties

Figures 3 and 4 show the mechanical properties of the as-cast and extruded composites, respectively. It can be seen that hot extrusion significantly improves the mechanical properties of the composites. As mentioned above, extrusion significantly improves the particle distribution and reduces particle segregation in the grain boundaries. It destroys the original grain boundaries and new grain boundaries are formed through DRX. These new grain boundaries are stronger than the original ones because the cavities, microcracks and segregation of particles or inclusions at the boundaries are largely eliminated. All these can improve the matrix plastic flow and decrease the possibility of crack in the matrix during deformation. So the elongation of the composite is improved significantly by extrusion. In addition, grain sizes are significantly refined due to DRX, as shown in Fig. 2. Extrusion can also increase the dislocation density in the matrix. Therefore, the strength of the matrix increases after extrusion and a larger effective load can be transferred to the reinforcement. SiC particles reinforce the matrix in the extruded composite more effectively. All these contribute to the enhancement of mechanical properties.



**Fig. 3** Effect of volume fraction of particles on mechanical properties of as-cast composites: (a) Yield strength and ultimate tensile strength; (b) Modulus and elongation

For both as-cast and extruded composites, with the increase of particle volume fraction, the yield strength and elastic modulus increase while the elongation decreases. According to Hall–Petch relation, the yield strength of a polycrystalline material increases with the decrease of grain size. The average grain size decreases as the volume fraction of particles increases, as shown in Fig. 2. Besides, ceramic particles can also induce multidirectional thermal stress at the particulate/matrix interface due to the large difference of coefficients of thermal expansion between matrix and reinforcement,



**Fig. 4** Effect of volume fraction of particles on mechanical properties of extruded composites: (a) Yield strength and ultimate tensile strength; (b) Modulus and elongation

and cause high dislocation density in the matrix. With the increase of particle contents, thermal stress and dislocation density are enhanced. Therefore, in terms of grain size and dislocation density, the strengths of composites increase with the increase of particle contents.

As the particle content increases, the ultimate tensile strengths change differently in as-cast and extruded composites. For as-cast composites, ultimate tensile strengths decrease with the increase of particle contents. This is caused by particle segregation, which restricts the flow of liquid before the composite is completely solidified. Therefore, micro-cavities are formed in these regions, leading to the weak and defective interface between particle and matrix at grain boundaries [13-15]. This micro-cavity has been observed at the interface in Mg matrix composites fabricated by stir casting [13]. In addition, large stress concentrations occur inside particle segregation while the composite is under load. Thus, the weak interface can not withstand the stress which is large enough to cause particle cracking and matrix rupture, resulting in the fact that particles can not effectively reinforce the matrix. As the particle contents increase, particle segregation at grain boundaries becomes more serious, as shown in Figs. 1 and 2. Hence, there are more micro-cavities in the composites with higher particle content, causing lower ultimate tensile strengths. For the extruded composites, on the contrary, the ultimate tensile strengths increase with the increase of particle contents. After hot extrusion, particle segregation and cast defects are almost eliminated. So, the addition of particles can effectively improve the mechanical properties of the extruded composites.

# **4** Conclusions

1) Hot extrusion of the as-cast and as-extruded  $SiC_p/AZ91$  composites improves the particle distribution, refines the grains of matrix, and significantly improves the mechanical properties.

2) The grain sizes of the extruded composites decrease with the increase of  $SiC_p$  contents.

3) For as-extruded  $SiC_p/AZ91Mg$  composites, the yield strength, ultimate tensile strength and elastic modulus increase with the contents of  $SiC_p$ .

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# SiC<sub>p</sub>/AZ91 镁基复合材料的热挤压

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摘 要:采用搅拌铸造法制备 SiC 体积分数为 5%、10%和 15%的颗粒增强 AZ91 镁基复合材料(SiC<sub>p</sub>/AZ91)。复 合材料经过 T4 处理后,于 350 °C 以固定挤压比 12:1 进行热挤压。在铸态复合材料中,颗粒在晶间微观区域发生 偏聚。热挤压基本上消除了这种偏聚并有效地改善颗粒分布。另外,热挤压有效地细化基体的晶粒。结果表明: 热挤压明显提高复合材料的力学性能。在挤压态复合材料中,随着 SiC 颗粒含量的升高,基体的晶粒尺寸减小, 强度和弹性模量升高,但是伸长率降低。

关键词:挤压;镁基复合材料;SiC<sub>p</sub>;AZ91 镁合金;体积分数

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