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ARTICLE

Effects of Solution and Aging Treatment on Microstructure and Properties of Semi-solid Extruded SiC/AZ91D Alloy

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Abstract: In order to obtain the magnesium matrix composites with good mechanical properties, the semi-solid 15wt% SiC/AZ91D billet with 10 μm in length was prepared by the near-liquidus heat-holding method and later extruded to get the magnesium matrix composites. Solution treatment at 415 °C for 24 h (T4) was conducted, followed by further aging treatment at 220 °C for 8 h (T6). The results show that during the solution and aging treatments, $Mg_{17}Al_{12}$ phases at the grain boundary dissolve, and the secondary-precipitated phases appearing in grains become layered and spheroidized gradually. The magnesium matrix composites 15wt%SiC/AZ91D after T6 treatment show better integrated mechanical properties when compared with those of the composites before T6 treatment. The tensile strength, yield strength, elongation, and hardness reach 242 MPa, 204 MPa, 2.3% and 1322 MPa, respectively.

Key words: semi-solid extrusion; SiC; AZ91D; solution and aging; near-liquidus heat-holding method

In recent years, the particle-reinforced magnesium matrix composites are widely used in aerospace, automobile, and electronic industries due to their great specific strength, specific stiffness, and excellent wear resistance. As for the preparation method, the mechanical stirring and casting method attracts great attention because of the easy process, free size of ingot, and easy bulk production. However, particle-reinforced magnesium matrix composites prepared by mechanical stirring and casting have some weaknesses. For example, the particles may agglomerate easily in the base material and get air entrapped, thus resulting in numerous pores in the castings[1].

In the contrast, extrusion casting shows the benefits, such as improving the surface bonding strength between the reinforced particles and the base metal and reducing the porosity of metal matrix composites (MMCs), which makes this method an effective way of improving the properties of particle-reinforced MMCs. The near-liquidus casting method is easy and capable to prepare semi-solid slurry by simply adjusting the temperature to change the solidification process of liquid metal[2]. Due to the unique properties and benefits of non-ferrous metals, the magnesium matrix composites obtained by the near-liquidus casting method gain good mechanical properties after extrusion[3]. Wang et al[4] obtained the MMCs by combining the near-liquidus casting and extrusion casting. The mechanical properties of MMCs prepared by combination method are better than those of MMCs prepared by the common method of extrusion casting. Sun et al[5] studied the influence of reinforcement on the aging behavior of AZ91 magnesium alloy. The tensile strength, yield strength, and elongation of magnesium matrix composites in their research are 382 MPa, 316 MPa, and 1.6%, respectively. Nevertheless, in recent years, few researches of the heat treatment of semi-solid extruded SiC/AZ91D Mg matrix composites prepared by the near-liquidus casting are found.

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In this research, semi-solid SiC/AZ91D billet was prepared by mechanical stirring, casting, near-liquidus casting and solution and aging treatment to get the semi-solid extruded SiC/AZ91D Mg matrix composites. The microstructure, mechanical properties, and rupture characteristics were investigated, providing references for the application of composites.

1 Experiment

AZ91D alloy was heated to the molten state at 740 °C under the CO_2/SF_6 atmosphere and later cooled down to 670 °C. 15wt% SiC was preheated at 600 °C holding for 5 min and then added to the alloy. The composites were cooled down to 575 °C and stirred mechanically at 400 r/min for 30 min. And then the composites were heated to 720 °C and stirred at 400 r/min for another 5 min. Add 0.1wt% C_2Cl_6 to the composites and refine for 30 min. The composites were cooled down to 595 °C holding for 30 min to obtain semi-solid slurry. Pour the slurry into the mould pre-heated at 200 °C and obtain the semi-solid billet. Extrude the billet through the backward extruding mould, as shown in Fig.1, in a semi-solid way with the extrusion force of 2000 kN by a forging press of 200 t load, at the extrusion temperature of 575 °C holding for 30 min. The extrusion ratio was 1.6:1, and the extrusion speed was 1 mm/s. Finally, the extruded SiC/AZ91D matrix composites were solid solution treated at 415 °C for 24 h (T4) and aging treated at 220 °C for 8 h (T6).

Microstructure and composition of the sample were observed and analyzed by metallographic microscope Leica 6000M and the scanning electron microscope Tescan. The grain size, phase size, and phase volume fraction were measured by the software Image Pro Plus 5.0. The tensile test was conducted at tensile velocity of 0.5 mm/s by the stretcher. The hardness of the sample was measured at the load of 0.981 N with the Vickers hardness measurement instrument (Yantai Huayin).

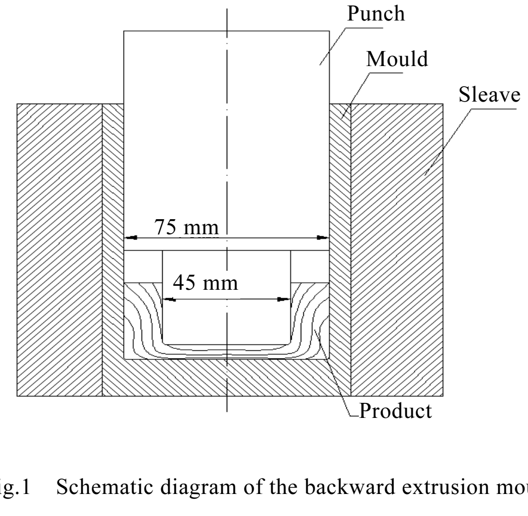

Fig.1 Schematic diagram of the backward extrusion mould

2 Results and Discussion

2.1 Microstructure

As shown in Fig.2, the $Mg_{17}Al_{12}$ phase and SiC exist in the magnesium matrix composite and no new phase appears. Fig.3 shows the microstructure of the semi-solid billet. A great deal of $Mg_{17}Al_{12}$ phases are distributed on grain boundary, and SiC particles are distributed along the grain boundary like a necklace. Such distribution of SiC is due to the "push" effect on the liquid-solid interface during the process of solidification[6].

Fig.4 shows OM and SEM images of microstructure of the semi-solid extruded 15wt%SiC/AZ91D composites under different treatments. As indicated in Fig.4a and 4d, the matrix grain is rose-like, and its grain boundary of the matrix is occupied by SiC particles distributed like a necklace, while the punctate $Mg_{17}Al_{12}$ (P-$Mg_{17}Al_{12}$) and the island $Mg_{17}Al_{12}$ (I-$Mg_{17}Al_{12}$) phases are distributed uniformly at the grain boundary. After the T4 treatment (Fig.4b and 4e), the grain shape of matrix begins to change but still looks like a certain rosette shape. The $Mg_{17}Al_{12}$ phases at the grain boundary dissolve in the matrix. Furthermore, a large number of the secondary precipitated phases of $Mg_{17}Al_{12}$ are precipitated in layered shape (2L-$Mg_{17}Al_{12}$) near the

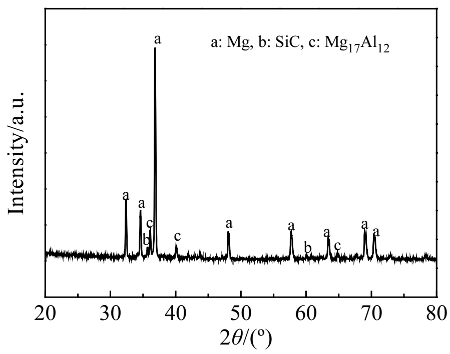

Fig.2 XRD pattern of 15wt%SiC/AZ9D semi-solid composite billet

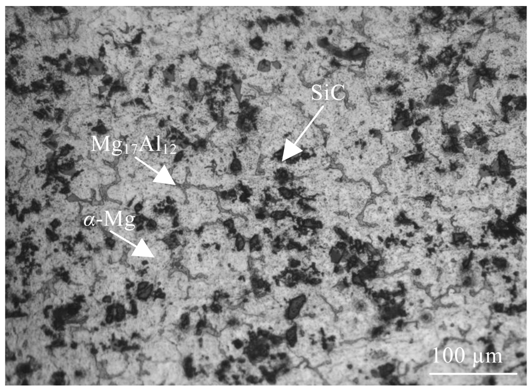

Fig.3 Microstructure of the semi-solid 15wt%SiC/AZ91D billet

grain boundary and finally diffuse into the grain. After T6 treatment (Fig.4c and 4f), the matrix grain becomes equiaxed spherical grain, and the 2L-$Mg_{17}Al_{12}$ phases are further precipitated and fully fill the whole grain. Moreover, punctate secondary precipitated phases (2P-$Mg_{17}Al_{12}$) can be observed in the microstructure. It can be confirmed that such phases come from the spheroidization of the 2L-$Mg_{17}Al_{12}$ phases according to the distribution orientation.

The grain size of the semi-solid extruded 15wt% SiC/AZ91D composites under different treatment was measured by Image Pro Plus 5.0 (Fig.5). It can be seen that the grain size reduces after the progress of heat treatment and reaches the minimum (15.78 μm) after T6 treatment.

The second phase size and volume fraction of the semi-solid extruded 15wt%SiC/AZ91D composites under different treatments were measured (Table 1). With the progress of heat treatment, the second phase size decreases, while the volume fraction increases. After T6 treatment, the second phase size of 15wt%SiC/AZ91D drops to the minimum (0.16 μm) while the volume fraction reaches the maximum (26.87%).

Fig.6 shows SEM image and EDS analysis of the semi-solid extruded 15wt%SiC/AZ91D Mg matrix composites. According to Fig.6, lots of $Mg_{17}Al_{12}$ phases are distributed at the grain boundary, while a few $Mg_{17}Al_{12}$ phase particles are adhered to SiC particles, which further proves the results of Fig.4.

In the process of extrusion, dynamic recrystallization can easily take place due to the low stacking fault energy, wide dislocation width, difficult glide and climb from dislocation, and increasing dislocation density and deformation energy

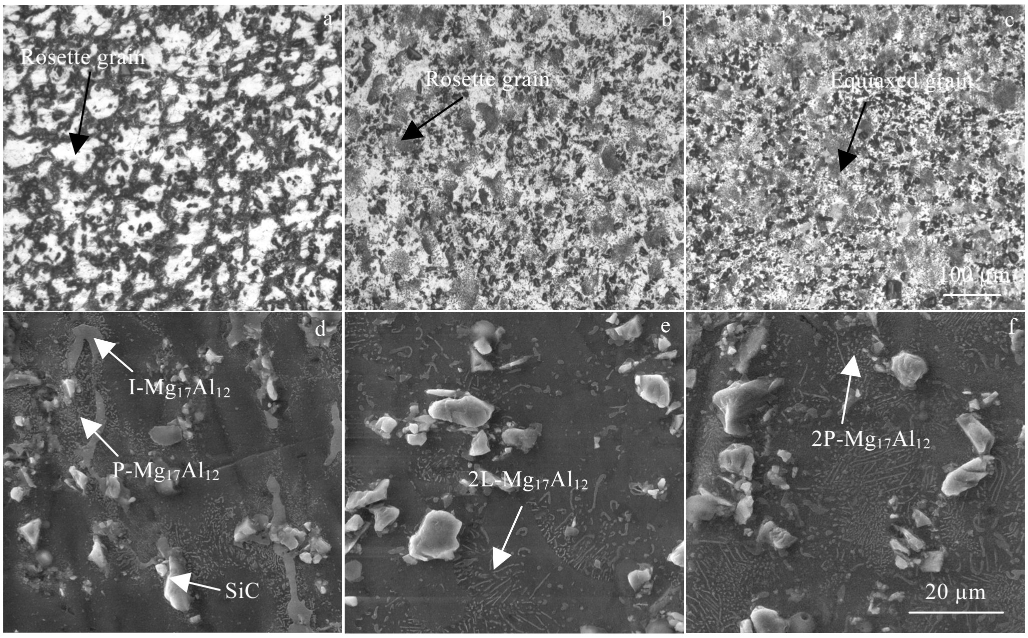

Fig.4 OM (a~c) and corresponding SEM (d~f) images of 15wt% SiC/AZ91D composites under different treatments: (a, d) as-extruded, (b, e) T4, and (c, f) T6

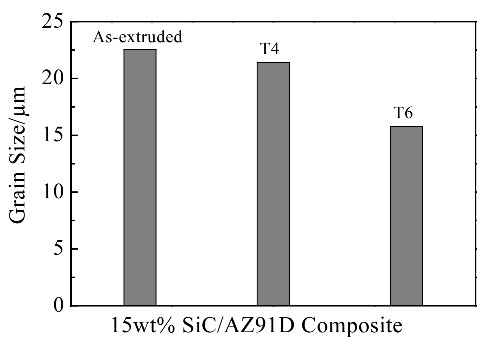

Fig.5 Grain size of 15wt% SiC/AZ91D composites under different treatments

of magnesium alloy[7]. Because the semi-solid 15wt% SiC/AZ91D Mg matrix composites prepared by near-liquidus pouring was heated to the semi-solid extrusion temperature and kept at such temperature for 30 min, the grains partially remelt, the I-$Mg_{17}Al_{12}$ phases at the grain boundary partially dissolve at the matrix, and the P-$Mg_{17}Al_{12}$ phases are precipitated from the remaining solid

Table 1 Phase size and volume fraction of $Mg_{17}Al_{12}$ in 15wt% SiC/AZ91D composites under different treatments

Treatment	As-extruded	T4	T6
Phase size/μm	0.42	0.22	0.16
Phase volume fraction/%	16.12	20.49	26.87

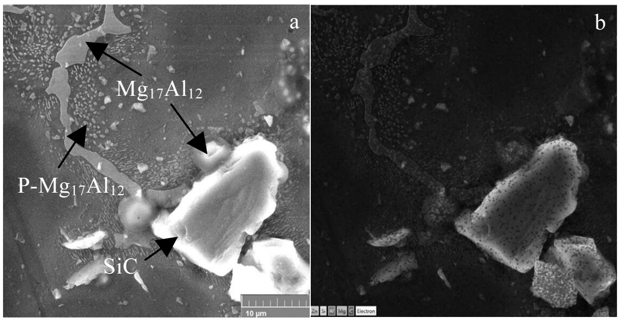

Fig.6 SEM image (a) and EDS analysis (b) of semi-solid extruded 15wt%SiC/AZ91D composites

phases at the grain boundary. Later, in the process semi-solid extrusion, the SiC particles distributed like the necklace are flattened and dispersed with the deformation of grains, solidifying the liquid phase and hardening the materials gradually. In this process, adding SiC results in grain refinement and the emergence of many grain boundaries, which is beneficial for the generation of $Mg_{17}Al_{12}$ phases. $Mg_{17}Al_{12}$ phases then hinder the grain boundary migration and pin the dislocation[8]. The dispersively distributed SiC particles and punctate $Mg_{17}Al_{12}$ phases are precipitated at the grain boundary providing nucleation particles for the dynamic recrystallization. Thus, the dynamic recrystallization occurs. In the process of extrusion, the dynamic recrystallized grains go through deformation all the time, and the central dislocation density of the grains increases constantly, leading to repeated dynamic recrystallization. Finally, because the softening from dynamic recrystallization can hardly stand the hardening from extrusion deformation, no complete dynamic recrystallization can be realized. In the process of solution treatment, the recrystallization continues and the grains are further refined. $Mg_{17}Al_{12}$ phases dissolve at the grain boundary, are precipitated in the grain for the second time, and finally produce 2L-$Mg_{17}Al_{12}$ phases. Nevertheless, the precipitation density of the secondary precipitated phases decreases constantly due to the interfacial energy between the matrix and the secondary precipitated phases[9]. Furthermore, the added SiC facilitates the generation of 2L-$Mg_{17}Al_{12}$ phases and hinders the spheroidization[5]. After the aging treatment, 2L-$Mg_{17}Al_{12}$ phases get further precipitated in the grains and spheroidized gradually, and the matrix grain size further decreases. Eventually, recrystallized grains get more and more spherical and finally become equiaxed grains without distortion.

2.2 Properties

Fig.7 shows the stress-strain curves of the semi-solid extruded 15wt%SiC/AZ91D Mg matrix composites under different treatments. Results show that after T6 treatment, both the strength and elongation of composites improve to some extent, with the maximum tensile strength and yield strength of 242 and 204 MPa, respectively, and the elongation of 2.3%.

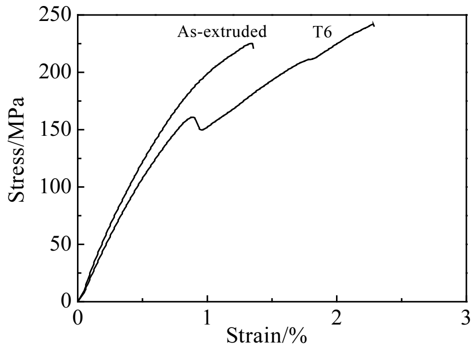

Fig.7 Stress-strain curves of semi-solid extruded 15wt%SiC/AZ91D Mg matrix composites under as- extruded and T6

In the process of extrusion, the thermal mismatch coefficient between the matrix AZ91D and SiC results in easy generation of dislocation near SiC when the composites cool down. And most $Mg_{17}Al_{12}$ phases are precipitated near the area of stress concentration at the time of stretching, thus the yield strength increases[8]. After T6 treatment, the 2L-$Mg_{17}Al_{12}$ phases can hardly trigger cracking and thus prevent the expansion of cracks[5]. Furthermore, the spheroidization of 2L-$Mg_{17}Al_{12}$ phases helps to improve the plasticity. Therefore, the massive high-density 2L-$Mg_{17}Al_{12}$ and 2P-$Mg_{17}Al_{12}$ phases prevent the slippage of grain boundaries and improve the tensile strength and elongation of composite.

Fig.8 shows the fracture surface of the semi-solid extruded 15wt%SiC/AZ91D Mg matrix composites before and after T6 treatment. Primary solid particles at the state of extrusion result in intergranular cracking and agglomeration (as marked by arrow 1). The bright and highly- concentrated $Mg_{17}Al_{12}$ phases (as marked by arrow 2) result in intergranular cracking and eutectic mixture (as marked by arrow 3). SiC particles distributed in a centralized way weaken the ductility of the composites, while the bigger primary grain agglomeration cannot compensate the reduced ductility. As a result, the composites after extrusion show poor ductility[10]. Nevertheless, after T6 treatment, matrix grains are further defined and the 2L-$Mg_{17}Al_{12}$ phases are spheroidized (2P-$Mg_{17}Al_{12}$), which improves the ductility of the composites, as well as the tensile strength to some extent[11].

Fig.9 shows the hardness of the semi-solid extruded 15wt%SiC/AZ91D Mg matrix composites under different treatments. The hardness of the composites decreases after the solution treatment, and increases after the aging treatment with the max hardness of 1322 MPa which is higher than that of the as-extruded sample. The reason is that after

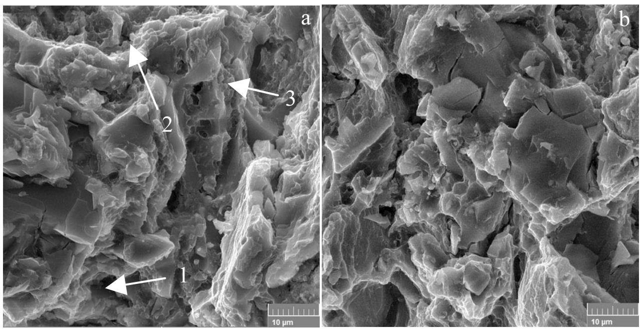

Fig.8 Fracture surface morphologies of semi-solid extruded 15wt%SiC/AZ91D Mg matrix composites before (a) and after (b) T6

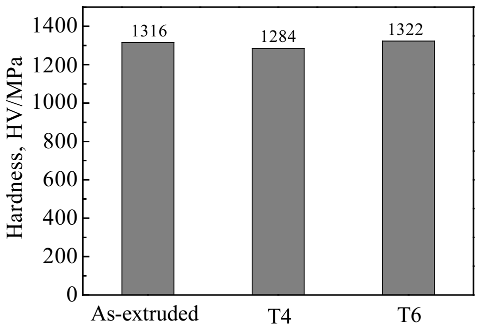

Fig.9 Hardness of semi-solid extruded 15wt%SiC/AZ91D Mg matrix composites under different treatments

T4 treatment, the 2L-$Mg_{17}Al_{12}$ phases with decreasing density diffuse into the crystal, leading to the decrease of hardness of the composites. In the contrast, after T6 treatment, the interior of the matrix grains is fully filled with 2L-$Mg_{17}Al_{12}$ phases, which improves the hardness of the composites.

3 Conclusions

1) With the progress of solution and aging treatment, the layered secondary precipitated $Mg_{17}Al_{12}$ phases are further precipitated and spheroidized gradually, but the spheroidization is restrained by the addition of SiC.

2) With the progress of solution and aging treatment, the matrix grain size decreases constantly, and the second phase grain size decreases all the time but the volume fraction increases. The 15wt%SiC/AZ91D composites have the matrix grain size of 15.19 μm, with the minimum second phase grain size of 0.16 μm and the maximum volume fraction of 26.87%.

3) With the progress of solution and aging treatment, the layered secondary precipitated phases and SiC improve the strength and hardness of 15wt%SiC/AZ91D composites jointly, gaining good integrated mechanical properties. The tensile strength, yield strength, elongation, and hardness are 242 MPa, 204 MPa, 2.3% and 1322 MPa, respectively.

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固溶时效对半固态挤压 SiC/AZ91D 组织与性能的影响

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摘 要：为了获得综合力学性能良好的镁基复合材料，采用近液相线保温法制备 10 μm 15%SiC/AZ91D(质量分数)半固态坯料进行半固态挤压后得到其复合材料，再经过 415 ℃固溶处理 24 h (T4)和进一步 220 ℃时效处理 8 h (T6)。结果表明：随着固溶时效的进行，晶界处的层片状和点状 $Mg_{17}Al_{12}$ 相溶解，然后在晶粒中二次析出，并逐渐球化。与 T6 处理前的 15%SiC/AZ91D 复合材料相比，经 T6 处理后其具有良好的综合力学性能，拉伸强度、屈服强度、伸长率和硬度(HV)分别达到 242 MPa、204 MPa、2.3%和 1322 MPa。

关键词：半固态挤压；SiC；AZ91D；固溶时效；近液相线保温法

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