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ARTICLE

Effect of Hot Extrusion on the Microstructure and Mechanical Properties of the SiCp/Mg-9Al-1Zn Nanocomposite Fabricated by Different Stir Time

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Abstract: 1wt% SiC nanoparticles reinforced Mg-9Al-1Zn magnesium matrix composite was fabricated by stir casting and hot extrusion. Effect of hot extrusion on microstructure and mechanical properties of SiCp/Mg-9Al-1Zn nanocomposite stirring for 10 min and 30 min was researched. The results show that the grains of the matrix in the as-cast SiCp/Mg-9Al-1Zn nanocomposite stirring for 30 min are mainly refined. But the mechanical properties of nanocomposite decline due to the increase of agglomerated SiC nanoparticles and $Mg_{17}Al_{12}$ phases with network morphology along the grain boundaries. After hot extrusion, a bimodal microstructure composed of alternate arrays of fine DRXed grains and relatively coarse DRXed grains forms. Particularly, for extruded nanocomposites stirring for 30 min, the fine DRXed region increases and the distribution of SiC nanoparticles is more homogenous, which exhibits superior mechanical properties compared with as-extruded SiCp/Mg-9Al-1Zn nanocomposite stirring for 10 min.

Key words: SiCp/Mg-9Al-1Zn nanocomposite; stir time; bimodal microstructure; mechanical properties

Magnesium alloys have gained more attention in scientific research and commercial application due to high specific strength and high specific modulus. However, relatively low strength, poor room temperature ductility and high coefficient of thermal expansion restrict their widespread application[1-4]. Magnesium matrix composites can provide positive mechanical properties which are superior to magnesium alloys. Particularly, particles reinforced magnesium matrix composite have been widely used because of low cost and simple fabrication technology[5-7]. In recent years, the use of nanoparticles as reinforcement phase has inspired considerable research interest since nanoparticles can lead to potential improvements in strength and ductility.

To add nanoscale size particles in matrix preferably, semisolid stirring casting is an simple and effective method compared with other ways such as powder metallurgy, squeeze cast, disintegrated melt deposition, infiltration and self-propagating high-temperature synthesis[8]. Semisolid stirring can be used to incorporate nanoparticles and disperse the nanoparticles[9-11]. It has been investigated that stir time has significant impact on microstructure and mechanical properties of magnesium matrix composite. When stir time is short, the grain size of the matrix in the composite is not decreased obviously. The as-cast composites show better mechanical properties due to fine $Mg_{17}Al_{12}$ phase along the grain boundaries. When stir time is long, the matrix grain is refined obviously while the amount of the refined $Mg_{17}Al_{12}$ phase along the grain boundaries decreases[12,13]. With the decrease of refined $Mg_{17}Al_{12}$ phases and the increase of SiC nanoparticles agglomeration, it would decrease the mechanical properties of as-cast composite[12]. If many uniform $Mg_{17}Al_{12}$ particles and homogeneously dispersed SiC nanoparticles exist in SiC nanocomposite reinforced Mg-Al composite, it would benefit mechanical properties of nanocomposite[14]. Exactly, hot ex-

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trusion could be applied to break $Mg_{17}Al_{12}$ phase into particles and improve SiC nanoparticles distribution. Based on the result, the mechanical properties of the nanocomposite containing plenty of coarse $Mg_{17}Al_{12}$ phases stirring for longer time would be likely to increase more significantly after extrusion while nanocomposite stirring for short time exhibits better as-cast mechanical properties.

To confirm the validity of this idea, 1wt% SiC nanoparticles reinforced Mg-9Al-1Zn magnesium matrix composite stirring for 30 min was subjected to hot extrusion, and the resulting microstructure and mechanical properties are herein discussed in comparison to those of an as-extruded SiCp/Mg-9Al-1Zn nanocomposite stirring for 10 min.

1 Experiment

Mg-Al-Zn alloy with the nominal composition of Mg-9Al-1Zn was used as the matrix alloy. SiC nanoparticles with an average diameter of 60 nm and weight fraction(wt%) of 1% were selected as the reinforcement. SiCp/Mg-9Al-1Zn magnesium matrix nanocomposites were fabricated by stir casting. The whole fabrication process was conducted in a protective atmosphere of CO_2 and SF_6 to avoid burning[15]. Mg-9Al-1Zn alloy was molten at 720 ℃, and then cooled to 590 ℃ which made the matrix alloy in the semi-solid condition. The SiC particles which were preheated to 550 ℃ were quickly added into the molten Mg-9Al-1Zn alloy. The mixture was stirred for 10 and 30 min. After adequately stirring the melt, the melt was rapidly reheated to 720 ℃ and held at this temperature for 30 min. Then the melt was cooled to 690 ℃ and poured into a preheated steel mould (300 ℃). The specimens for extrusion were machined to Φ30 mm×50 mm from as-cast ingots. Subsequently, the specimens were extruded at 360 ℃ with the ratio of 10:1.

Microstructure of the as-cast and as-extruded composites was determined by a Leica DM2500M optical microscopy(OM) and a Mira3 Tescan scanning electron microscopy(SEM) equipped with an energy dispersive X-ray spectrometer(EDS). The samples were ground, polished and etched in acetic picral [5 g picric acid+10 mL acetic acid+78 mL ethanol+7 mL H_2O]. The average grain size in the nanocomposite was analyzed from microstructure image using Image-Pro Plus software. Tensile tests were carried out at room temperature using a DNS 100 machine with a speed of 0.5 mm/min.

2 Results and Discussion

2.1 Microstructures of as-cast SiCp/Mg-9Al-1Zn nanocomposite fabricated by different stir time

Fig. 1 shows the typical OM and SEM images of as-cast SiCp/Mg-9Al-1Zn nanocomposite with different stir time. As shown in Fig.1a and 1b, the matrix grain of as-cast SiCp/Mg-9Al-1Zn nanocomposite stirring for 30 min (~60 μm) is refined compared with as-cast nanocomposite stirring for 10 min (~100 μm). It can be observed from Fig.1c that the microstructure of as-cast SiCp/Mg-9Al-1Zn nanocomposite stirring for 10 min is composed of matrix phase (Fig.1c-A) and secondary phase. Besides, the secondary phase with eutectic (Fig.1c-B) and lamellas (Fig.1c-C) morphologies can be found at the interior and boundary of grains. When the stir time is 30 min, the lamellar secondary phase (Fig.1d-D) will increase and form network morphology in the nanocomposite, as shown in Fig.1d. The EDS results of points "A", "B", "C" and "D" are demonstrated in Table 1. By EDS results, the matrix phase and the secondary phase can be recognized as α-Mg phase and $Mg_{17}Al_{12}$ phase, respectively.

As shown in Fig. 2, the composition of the SiC nanoparticle dense zones was investigated by EDS. Analyzing an area of SEM micrograph, EDS of Si K demonstrates that composition of the nanoparticles is SiC nanoparticles. For as-cast SiCp/Mg-9Al-1Zn nanocomposite stirring for 10 min, the distribution of SiC nanoparticle in the nanocomposite is uniform outside a few nanoparticle clusters located at the grain boundaries, as shown in Fig.2a. But for that stirring for 30 min, much SiC nanoparticle clusters locate at the grain boundaries, as shown in Fig.2b. It has been well documented that many SiC particles are pushed ahead by liquid-solid interface during solidification process with increasing the stirring time[16].

The microstructure of nanocomposite usually depends on the nucleation stage and subsequent growth condition. The "push" effect of the solidification front on the insoluble solid nanoparticles may cause the nuclei clusters along grain boundaries[12]. With increasing the stir time, much more SiC

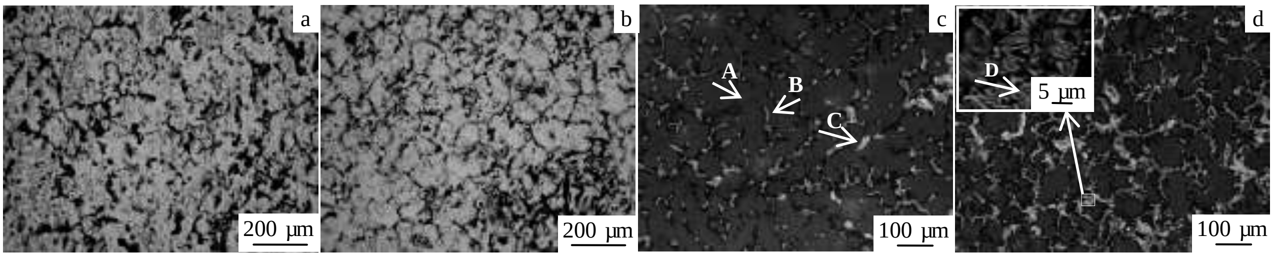

Fig.1 OM (a, b) and SEM (c, d) images of as-cast SiCp/Mg-9Al-1Zn nanocomposite with different stir time: (a, c) stirring for 10 min and (b, d) stirring for 30 min

Table 1 EDS results of point A, B, C, and D in Fig.1c and 1d (at%)

Point	Element			Possible compounds
	Mg	Al	Zn	
A	86.8	11.8	1.4	α-Mg
B	61.2	37.6	1.2	$Mg_{17}Al_{12}$
C	66.3	31.6	2.1	$Mg_{17}Al_{12}$
D	64.2	34.3	1.5	$Mg_{17}Al_{12}$

particles clusters along grain boundaries act as the nuclei, which increase the heterogeneous nucleation. In addition, SiC nanoparticles clusters along grain boundaries can restrict growth of grains during solidification. Thus, the grain refinement occurs in as-cast SiCp/Mg-9Al-1Zn nanocomposite stirring for 30 min. Furthermore, it is well known that a large amount of $Mg_{17}Al_{12}$ phases along the grain boundaries change to lamellae after addition of SiC nanoparticles[10]. For nanocomposite stirring for 30 min, grain boundaries increase with grain refinement, which can lead to the increase of precipitated lamellar $Mg_{17}Al_{12}$ phase along the grain boundary.

2.2 Effect of hot extrusion on microstructures of SiCp/Mg-9Al-1Zn nanocomposite fabricated by different stir time

Fig.3 shows the microstructural characteristics parallel to extrusion direction of as-extruded SiCp/Mg-9Al-1Zn nanocomposite. The optical micrographs in Fig.3a and 3b demonstrate a bimodal microstructure composed of fine DRXed grains and relatively coarse DRXed grains. For as-extruded SiCp/Mg-9Al-1Zn nanocomposite stirring for 30 min, it shows many relatively fine DRXed regions, as shown in Fig.3b. Consequently, the average grain size of as-extruded nanocomposite stirring for 30 min is refined from 19.28 μm to 13.72 μm compared with as-extruded nanocomposite stirring for 10 min. The SEM images in Fig.3c and 3d reveal the concentration of finer precipitated $Mg_{17}Al_{12}$ particles (~2 μm) and relatively lager cracked $Mg_{17}Al_{12}$ phases (~20 μm) that occurs in the fine DRXed region, while few such particles are seen in the coarse DRXed region, indicating that these $Mg_{17}Al_{12}$ particles play an important role in forming DRXed region. It is well know that during hot deformation an incompatibility between deformations occurs at the interface between the soft grains of the Mg matrix and hard second phases, and that this induces a strong stress concentration which can increase the formation of fine DRXed grains[17]. As mentioned above, there are more $Mg_{17}Al_{12}$ phases in as-cast SiCp/Mg-9Al-1Zn nanocomposite stirring for 30 min as compared with nanocomposite stirring for 10 min, which is the reason for the much more $Mg_{17}Al_{12}$ particles in as-extruded nanocomposite stirring for 30 min. A multitude of hard $Mg_{17}Al_{12}$ particles accelerate the formation of much more DRXed grains during hot extrusion by inducing a stress concentration around these particles. Similarly, fine $Mg_{17}Al_{12}$ particles play an important role in restricting DRXed grain growth by creating a pinning effect[18]. Thus, the greater number of fine particles dispersing along grain boundaries of the nanocomposite stirring for 30 min (Fig.3d) are considered to increase the fine DRXed grains through an enhanced grain boundary pinning effect.

Fig.4 shows the surface SEM-EDS for as-extruded SiCp/Mg-9Al-1Zn nanocomposite. The EDS of Si K is homogeneous which demonstrates that the distribution of SiC

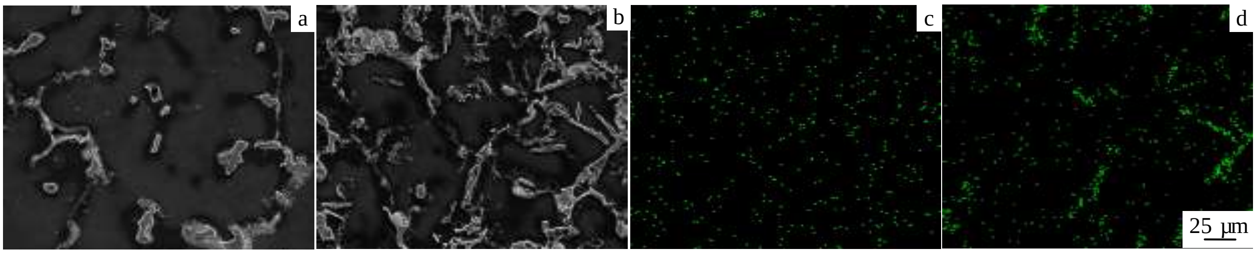

Fig.2 SEM images (a, b) and EDS maps (c, d) of as-cast SiCp/Mg-9Al-1Zn nanocomposite: (a) stirring for 10 min; (b) stirring for 30 min; (c) Si distribution corresponding Fig.2a; (d) Si distribution corresponding Fig.2b

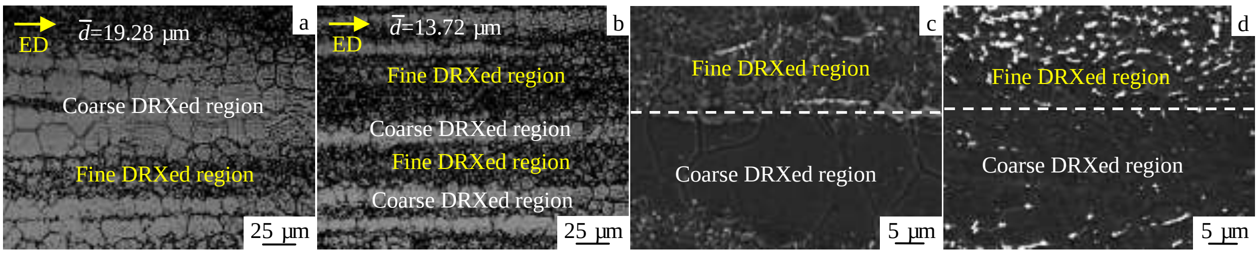

Fig.3 OM (a, b) and SEM (c, d) images of as-extruded SiCp/Mg-9Al-1Zn nanocomposite with different stir time: (a, c) stirring for 10 min; (b, d) stirring for 30 min

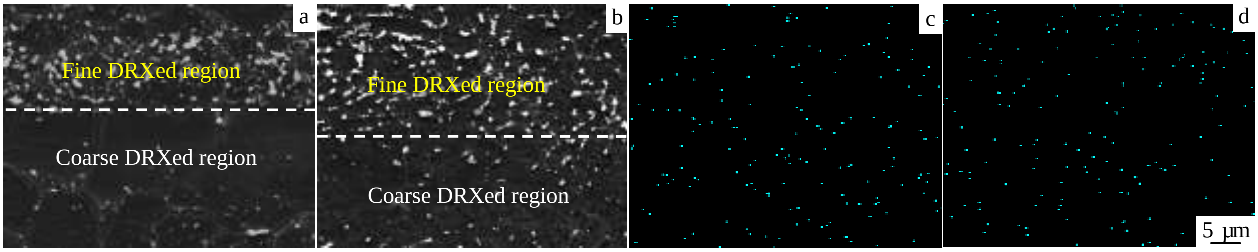

Fig.4 SEM images (a, b) and EDS maps (c, d) of as-extruded SiCp/Mg-9Al-1Zn nanocomposite: (a) stirring for 10 min; (b) stirring for 30 min; (c) Si distribution corresponding Fig.4a; (d) Si distribution corresponding Fig.4b

nanoparticle in as-extruded nanocomposite is uniform whether in fine DRXed region or in coarse DRXed region. Thus, fine $Mg_{17}Al_{12}$ particles play a major role in the formation of fine DRXed grains.

2.3 Tensile properties before and after extrusion

Fig.5 shows the yield strength (YS), ultimate tensile strength (UTS) and elongation of SiCp/Mg-9Al-1Zn nanocomposites in the as-cast and as-extrusion condition. For as-cast SiCp/Mg-9Al-1Zn nanocomposite, the YS, UTS and elongation of the nanocomposite after stirring for 30 min decline compared with the nanocomposite stirring for 10 min, which can contribute to the increase of the agglomeration of the SiC nanoparticles and the existence of network $Mg_{17}Al_{12}$ phase.

But for as-extruded SiCp/Mg-9Al-1Zn nanocomposite, the nanocomposite stirring for 30 min exhibits superior mechanical properties compared with nanocomposite stirring for 10 min. The YS, UTS and elongation of as-extruded nanocomposite stirring for 30 min are 255 MPa, 134 MPa and 11.5%, respectively, which are approximately 56%, 90% and 283% greater than that of the as-cast composite (163 MPa, 70 MPa and 3%). According to the classic Hall-Petch equation:

$$\sigma_y=\sigma_0+K_y d^{-1/2} \quad (1)$$

YS is proportional to $d^{-1/2}$, where d is the mean grain size[19]. The grains of extruded SiCp/Mg-9Al-1Zn nanocomposites stirring for 30 min are significantly refined due to increased fine DRXed grains as shown in Fig.3b. As a result the yield strength of nanocomposite is enhanced. Besides, SiC nanoparticles as well as fine $Mg_{17}Al_{12}$ particles in the as-extruded nanocomposite are much harder than that of the matrix alloy at room temperature. During tensile test, the deformation of as-extruded nanocomposite needs the dislocation to spread from grains to grains, which is hindered by the SiC nanoparticles as well as $Mg_{17}Al_{12}$ phases along the grain boundaries[20]. This Orowan strengthening effect can contribute to the improvement of ultimate tensile strength. In addition, uniform SiC particle and increased fine DRXed regions exist in the extruded SiCp/Mg-9Al-1Zn nanocomposites stirring for 30 min resulting in higher elongation.

Table 2 shows a comparison of the reported values of deformed SiC particles reinforced magnesium matrix composites. As compared with other researches of micron SiC particles

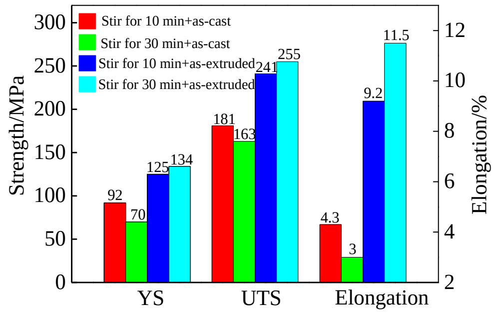

Fig.5 Tensile properties of SiCp/Mg-9Al-1Zn nanocomposite

Table 2 Comparison of reported values of deformed SiCp reinforced magnesium matrix composites

Composite	Process	UTS/MPa	Elongation/%
60 nm 1wt% SiCp/Mg-9Al-1Zn	Extrusion at 360 ℃ Ratio 10:1	255±9	11.5±1.0
0.5 μm 10vol% SiCp/AZ31B[21]	Extrusion at 350 ℃ Ratio 12:1	~350	~1.2
10 μm 10vol% SiCp/AZ91[22]	Extrusion at 350 ℃ Ratio 12:1	~325	~3.9
10 μm 9vol%+60 nm1% SiCp/AZ31B[23]	Extrusion at 350 ℃ Ratio 12:1	~308	~3.9

reinforced magnesium matrix composites, the nanocomposite of the present study shows better elongation, which is attributed to the existence of uniformly distributed nanoscale SiC particles. It is well known that uniformly distributed SiC nanoparticles can improve ductility of composite significantly. However, as-extruded 60 nm 1wt% SiCp/Mg-9Al-1Zn composites don't exhibit the best UTS in this study, which may be due to the higher extrusion temperature and the smaller extrusion ratio.

3 Conclusions

1) For as-cast SiCp/Mg-9Al-1Zn nanocomposite stirring for 30 min, the grain size declines contrast to that stirring for 10 min. But a multitude of SiC nanoparticle clusters locate at the grain boundaries and the $Mg_{17}Al_{12}$ phases exhibit network morphology with increasing the stir time.

2) The $Mg_{17}Al_{12}$ phases crack into fine particle and the seg-

regation of SiC particles is largely eliminated after hot extrusion compared with that of as-cast SiCp/Mg-9Al-1Zn nanocomposite. In addition, fine $Mg_{17}Al_{12}$ particles play a major role in the formation of fine DRXed grains.

3) For as-extruded SiCp/Mg-9Al-1Zn nanocomposite, the nanocomposite stirring for 30 min exhibit superior yield strength, ultimate tensile strength and elongation compared with nanocomposite stirring for 10 min, which is attributed to dispersed SiC particles and increased fine DRXed regions.

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热挤压对不同搅拌时间制备的纳米 SiCp/Mg-9Al-1Zn 复合材料组织和性能的影响

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摘 要：通过半固态搅拌铸造和热挤压变形复合工艺制备出 SiCp 质量分数为 1%的纳米 SiCp/Mg-9Al-1Zn 镁基复合材料。研究了搅拌时间分别为10和30 min时热挤压对纳米SiCp/Mg-9Al-1Zn镁基复合材料的显微组织和力学性能的影响。结果表明，对于铸态的纳米SiCp/Mg-9Al-1Zn 镁基复合材料来说，搅拌时间为 30 min 时，基体的晶粒细化，但在晶界处析出的 $Mg_{17}Al_{12}$ 相数量增多，网状化严重且 SiC 团聚增加，使得复合材料的力学性能下降。而通过热挤压后，复合材料形成了粗晶与细晶交替的组织结构。特别是对于搅拌时间为 30 min 的复合材料，细晶区增多且纳米 SiC 颗粒分布更加均匀，这就使得力学性能高于搅拌 10 min 的挤压态的 SiCp/Mg-9Al-1Zn 复合材料。

关键词：SiCp/Mg-9Al-1Zn纳米复合材料；搅拌时间；双峰结构；力学性能

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