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Microstructure evolution and mechanical properties of SiC nanoparticles reinforced magnesium matrix composite processed by cyclic closed-die forging



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ABSTRACT

Magnesium composites containing different amounts of SiC nanoparticles were processed by a two-step cyclic closed-die forging (CCDF), which was carried out at 400 °C for 3 passes (1st-CCDF) and further at 300 °C for 2 passes (2nd-CCDF). Microstructure evolution and mechanical properties of the composites were investigated. After processing, the average grain size is significantly refined to ~2.5 μ m, and the morphology exhibits a flow-lined feature. The β -Mg₁₇Al₁₂ phases in the as-cast alloy dissolve completely after 1st-CCDF, and then precipitate out with an average size of ~650 nm during 2nd-CCDF. After CCDF, the as-cast SiC clusters are uniformly dispersed as separate particles, and the yield strength and ultimate strength of the composites reach 258 MPa and 365 MPa, respectively. The ductility of the composites is enhanced after 1st-CCDF but decreased after 2nd-CCDF, which is in accordance with the observed fracture surfaces. From the perspective of strength and ductility, the optimal content of SiC is 0.5 wt%.

1. Introduction

Magnesium (Mg) matrix composites are widely studied to improve the mechanical properties of magnesium alloys for the potential of automobiles and aerospace applications due to their low density, high specific strength and stiffness [1,2]. Compared with other kinds of Mg matrix composites, particles reinforced composites exhibit the advantages of lower cost, easier fabrication and better machinability [3–5]. Recent studies indicate that decreasing the particle sizes improves the mechanical properties of metal matrix composites (MMCs) [6]. It is found that nano-sized reinforcements enhance the mechanical properties more effectively than the micron-sized ones [7]. A variety of techniques for fabricating nano-composites are developed, such as high energy milling [8], ball milling [9] and nano-sintering [10]. Ultrasonic cavitation based dispersion is a new developed casting method, which has excellent ability of cleaning surfaces of nanoparticles, enhancing the wettability between nanoparticles and matrix, and breaking nanoparticle clusters. Li et al. found that the SiC nanoparticles are well dispersed in Mg matrix composites after

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http://dx.doi.org/10.1016/j.msea.2015.06.079 0921-5093/© 2015 Elsevier B.V. All rights reserved. ultrasonic dispersion. However, there are still some small clusters exist, which have negative effects to the mechanical properties [11–13].

Severe plastic deformation (SPD) has been recognized as a promising processing technique to fabricate bulk ultrafine-grained (UFG) materials. Generally, UFG materials exhibit extraordinary mechanical properties with enhancement of both strength and ductility [14–16]. When SPD is applied to MMCs, the extremely high accumulated strains and the unique shear deformation mode are expected to reduce the grain sizes of matrix, and disperse the particle clusters simultaneously [17-19]. To date, particle reinforced MMCs have been processed successfully by several types of SPD techniques, such as equal channel angular pressing (ECAP) [20–22], high pressure torsion (HPT) [23,24], accumulative roll bonding (ARB) [25,26], cyclic extrusion compression (CEC) [27,28] and cyclic closed-die forging (CCDF) [29,30]. Among these methods, CCDF introduces intensive strain [31] and is able to process less ductile materials due to its compressive deformation characteristic [30]. In addition, CCDF is particularly suitable to fabricate bulk materials, and its multi-directional deformation leads to a more uniform microstructure.

In this situation, CCDF is supposed to be a promising method to fabricate bulk UFG composites with uniform distributed nanoparticles. However, utilizations of CCDF in the fabrication of magnesium matrix composites are limited [29,30,32], and there is

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still no research about CCDF processed Mg nanocomposites. To investigate the effect of CCDF on nanocomposites, current research was implemented. In this paper, Mg matrix composites with different content of SiC nanoparticles were processed by a two-step CCDF method. The microstructure evolutions of Mg matrix, second phases, and SiC nanoparticles are investigated, and the relationships between microstructures and mechanical properties in different processing status are bridged.

2. Experimental procedures

The magnesium matrix used in this study is the commercial AZ91 alloy with a nominal composition of Mg-8.66Al-0.56Zn-0.16Mn (in wt%), and the SiC nanoparticles are exhibiting an average size of 40 nm. The magnesium nanocomposites with SiC content of 0%, 0.5%, and 1.5% were fabricated by ultrasonic cavitation-based dispersion method [11–13].

The Mg MMC billets, designated as AZ91, AZ91-0.5% SiC and AZ91-1.5% SiC, were machined into $100 \times 100 \times 30 \text{ mm}^3$ cuboid samples for CCDF. Fig. 1 shows the schematic illustration of CCDF. The sample was erected at the center of the lower die chamber, which has a cross section of 100×100 mm². A punch with the same cross section pressed down to the chamber with a constant speed of 3 mm/s to forge the sample to a thickness of 30 mm. Then the sample was rotated and re-inserted into the chamber to continue the next pass as shown in Fig. 1. In current research, a twostep processing method was implemented as 3 passes of CCDF at 400 °C (defined as 1st-CCDF) followed by 2 more passes at 300 °C (defined as 2nd-CCDF). The 1st-CCDF was supposed to enhance the deformability of the materials, and then 2nd-CCDF was aimed to further refine and strengthen the materials. The sample and the die were lubricated by graphite and pre-heated at corresponding processing temperature for 30 min. After each pass, the sample was guenched into water immediately.

Specimens for microstructure analysis were taken from five typical positions in the samples after CCDF as shown in Fig. 2. To illustrate the 3D morphology of the samples, microstructures from different directions of each specimen were characterized. The etching solution was composed of 1 ml nitric acid, 20 ml acetic acid, 60 ml ethylene glycol, and 19 ml water. The grain size distribution of the specimens was measured from OM micrographs using Image-Pro Plus software. Morphologies of the second phases and distributions of SiC nanoparticles were studied by a FEI Quanta 250 field-emission scanning electron microscope (FE-



Fig. 1. The schematic illustration of CCDF process.



Fig. 2. Positions and dimensions of the specimens in a sample after CCDF.

SEM). Tensile test was performed to evaluate the mechanical properties of Mg MMCs. The flat tensile specimens with a cross section of $1.5 \times 3 \text{ mm}^2$ and a gauge length of 18 mm were cut at the position shown in Fig. 2. Tensile test was performed on a WDW-10S universal tensile tester (Jinan, Shandong, China) with a constant cross-head speed of 0.5 mm/min at room temperature, and the fracture surfaces were also studied by FE-SEM.

3. Results and discussion

3.1. Microstructure evolution during CCDF

Fig. 3 shows a typical optical micrograph of the as-cast composite (AZ91-0.5% SiC). The microstructure consists of coarse dendritic α -Mg, massive β (Mg₁₇Al₁₂) and lamellar β structures, and the average grain size of the matrix is about 150 µm [33]. Dendritic massive β phases, exhibiting length of 15–100 µm, are observed along the grain boundaries, connected by much finer lamellar β .

Fig. 4 illustrates the microstructure evolution of the composites after 1st-CCDF. The average grain sizes of the AZ91, AZ91-0.5% SiC and AZ91-1.5% SiC composites were reduced to 26.0 μ m, 22.7 μ m and 22.7 μ m, respectively. The addition of nano-scaled SiC slightly affects the microstructures of the Mg matrix during CCDF. The wide variation of grain sizes, from less than 10 μ m to 100 μ m, indicates the occurrence of partial dynamic recrystallization (DRX). Compared with the AZ91 sample, samples with SiC



Fig. 3. Microstructure of the as-cast AZ91-0.5% SiC composite.

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