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Microstructural refinement and homogenization of Mg–SiC nanocomposites by cyclic extrusion compression

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ABSTRACT

An as-extruded Mg–1 wt%SiC nanocomposite was processed by cyclic extrusion compression (CEC) at 350 °C. The homogeneity of grain and SiC nanoparticle (~50 nm average diameter) distribution during the processing was investigated. With the increasing number of CEC passes, a finer grain size and more uniform particle distribution are obtained along with significant improvement in hardness. The matrix grain size is reduced remarkably from ~27.6 μm to ~6.5 μm after 8 passes of CEC. Nanoparticle declustering occurs through a mechanism of kneading caused by the intense turbulent flow of the Mg matrix during CEC, and the SiC nanoparticles are dispersed into the original particle-free regions. The property improvement is mainly attributed to Orowan strengthening and the Hall–Petch effect.

1. Introduction

Magnesium matrix composites are widely investigated because of their attractive high specific properties, especially in strength and stiffness. Among various Mg-based composites, particle reinforced Mg matrix composites have the advantages of lower cost, easier fabrication, and better machinability as compared to fiber reinforced Mg matrix composites. For most of the particulate Mg-based composites, the sizes of reinforcing particles are typically spanning from several microns to tens of microns. Addition of micron-sized particulates, such as Ti particles [1] or SiC particles [2–4], into Mg improves hardness, yield strength, wear resistance, as well as thermal stability. However, the improvement usually occurs at the cost of ductility or tensile strength reduction. Recent studies have indicated that the use of nano-sized reinforcements such as Al [5], Al_2O_3 [6], SiC [7,8], Y_2O_3 [9], and ZrO_2 [10], leads to a simultaneous increase in both strength and ductility of Mg composites. Nanoparticle reinforcements significantly enhance strength of the matrix by more effectively promoting particle hardening mechanisms than micron-sized particles. A fine and homogeneous dispersion of nanoparticles provides a better balance between the reinforcement and interparticle spacing effects to maximize the yield strength and creep resistance [11].

However, it is extremely difficult to distribute and disperse nanoscale particles uniformly in metal melts because of their high specific area and the poor wettability of nanoparticles. The occurrence of agglomeration and clusters [12] is detrimental to mechanical properties. In order to minimize these disadvantages, conventional secondary deformation processing methods such as hot extrusion have been employed to improve the homogeneity of the nanoparticle distribution [13,14]. However, these processing techniques are still difficult or impossible in the case of ultrafine particles, since very high strains would be required [15]. Severe plastic deformation (SPD) has been recognized as a promising method for fabricating bulk ultrafine-grained materials, having very small grain sizes typically less than 1 μm and exhibiting high yield strength and hardness with fairly good ductility [16–18]. Due to its unique shear deformation mode and extremely high accumulated strains, SPD of metal matrix composites is particularly attractive. After light metal matrix composites, such as Al–Si and Mg–Al–Mg_2Si, were processed by cyclic extrusion compression (CEC), the Si or Mg_2Si phases were refined and distributed uniformly in the matrix [19–21]. Therefore, it is anticipated that the SPD process may be a good candidate for producing nanocomposites with a homogeneous dispersion of nanoparticles. Nevertheless, few studies have been carried out on SPD of nanocomposites. Goussous et al. mixed carbon nanoparticles with pure Al particles and consolidated them using equal channel angular pressing (ECAP) to produce Al–C nanocomposites [22]. Asadi et al. added nanosized SiC and Al_2O_3 particles to an as-cast AZ91 magnesium alloy, and produced surface nanocomposite layers via friction stir processing (FSP) [23]. However, the distribution of SiC and Al_2O_3 particles was not uniform, and there were still clusters.

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Accordingly, this investigation aimed to study the effect of CEC on the homogeneity of nanoparticle distribution in a Mg–SiC composite. The influence of CEC on grain refinement and hardness was also investigated.

2. Experimental procedures

The material used in the present study was a Mg–1 wt%SiC (i.e. Mg–0.54 vol%SiC) nanocomposite fabricated by ultrasonic cavitation based solidification processing [24], and the average size of the SiC particles was ~50 nm. The ingots were extruded with an extrusion ratio of 25:1. Both the as-extruded sample and the CEC die coated with a lubricant of graphite powder were held for 10 min at 150 °C before heating to 350 °C for about 30 min. The CEC processing cycle was carried out by putting the sample into the upper chamber [25]. After 2, 5 and 8 passes of CEC processing, the corresponding equivalent strains were 2.4, 7.3 and 12.2, respectively [25]. After processing, longitudinal sections of the specimen were sectioned for microstructure analyses by optical microscopy (OM), field emission scanning electron microscopy (FESEM), and transmission electron microscopy (TEM). The hardness tests were performed using a Vickers indenter under a test load of 49 N and a dwell time of 15 s. The uniformity of the nanoparticle distribution in the composite was studied by the quadrat method [26]. The images to be studied were divided into a grid of square cells and the number of nanoparticles in each cell (N_q) was counted. Twenty images at a magnification of 100,000× were taken by the FESEM on each specimen. Each image was divided into 54 quadrats and the size of each quadrat was set to $a=300$ nm.

3. Results and discussion

Fig. 1 shows the evolution of the grain structure for the Mg–1 wt%SiC nanocomposite after CEC processing from the as-extruded condition. The initial average grain size for the as-extruded condition is ~27.6 μm as shown in Fig. 1a. After 2 passes it is apparent that the microstructure consists of coarse grains surrounded by much finer grains but with the coarse grains occupying a significantly larger area fraction. The mean grain size is reduced to ~14.2 μm. After 5 passes (Fig. 1c) there is again a mix of coarse and fine grains but the area fraction of finer grains less than 8 μm now becomes significant, and some coarse grains exhibit distorting with no refinement. Further deformation to 8 passes makes the grain structure more homogeneous with approximately all fine grains with an average size of ~6.5 μm. The decreasing grain size with increasing deformation is consistent with the results obtained on pure Mg [27], AZ31 alloy [28], and a Mg–Y_2O_3 nanocomposite [13]. Due to the relative high temperature of 350 °C ($T\sim0.7T_m$) in the CEC condition, dynamic recrystallization could occur, resulting in the observed grain refinement. On the other hand, the SiC nanoparticles possess the ability of nucleating Mg grains during recrystallization, and they also restrict the growth of the recrystallized Mg grains due to the pinning effect [29].

Fig. 2 illustrates the improvement of the nanoparticle distribution after 8 CEC passes. In the as-extruded condition, diffused clusters elongated in the extrusion direction are observed (Fig. 2a). The clusters have a size of up to 500 nm in the direction perpendicular to the extrusion direction. Large nanoparticle free zones around the clusters are also observed. It is clearly seen that the boundaries between the nanoparticle clusters and the particle-free matrix are sharp. After 8 passes of CEC, the SiC nanoparticles have debonded from the cluster and moved into the particle-free matrix. Thus, the large particle clusters dissociate and the particle distribution appears homogeneous (Fig. 2b). However, a few dense particle clusters remain visible in the longitudinal section, ascribing to the high surface energy associated with the nanoparticles. It should be noted that CEC does not degrade the microstructure of the investigated nanocomposite. No cavity formation is observed inside the specimen. The TEM study also reveals the uniform distribution of SiC nanoparticles (Fig. 2c) and good interfacial integrity between SiC nanoparticles and the Mg matrix (Fig. 2d) after 8 CEC passes.

The frequency histograms of the number of SiC nanoparticles per quadrat, *Nq*, for the as-extruded and 8 passes CEC-processed

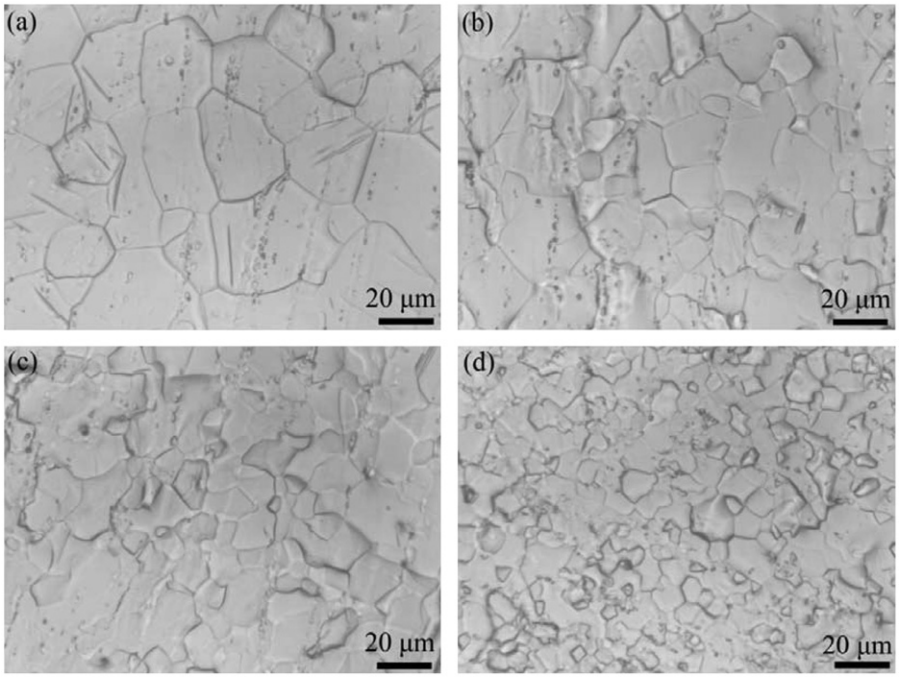

Fig. 1. Micrographs showing grain morphology of the Mg–1 wt%SiC nanocomposite: (a) as-extruded, and after (b) 2, (c) 5, and (d) 8 passes of CEC processing at 350 °C.