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Fabrication of SiC particles-reinforced magnesium matrix composite by ultrasonic vibration

K. B. Nie · X. J. Wang · K. Wu · L. Xu · M. Y. Zheng · X. S. Hu

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Abstract Magnesium matrix composites reinforced with two volume fractions (1 and 3%) of SiC particles (1 μm) were successfully fabricated by ultrasonic vibration. Compared with as-cast AZ91 alloy, with the addition of the SiC particles grain size of matrix decreased, while most of the phase $Mg_{17}Al_{12}$ varied from coarse plates to lamellar precipitates in the SiCp/AZ91 composites. With increasing volume fraction of the SiC particles, grains of matrix in the SiCp/AZ91 composites were gradually refined. The SiC particles were located mainly at grain boundaries in both 1 vol% SiCp/AZ91 composite and 3 vol% SiCp/AZ91 composite. SiC particles inside the particle clusters may be still separated by magnesium. The study of the interface between the SiC particle and the alloy matrix suggested that SiC particles bonded well with the alloy matrix without interfacial reaction. The ultimate tensile strength, yield strength, and elongation to fracture of the SiCp/AZ91 composites were simultaneously improved compared with that of the as-cast AZ91 alloy.

Introduction

Magnesium matrix composites (MMCs) have been extensively studied as an attractive choice for aerospace and automotive applications due to their low density and superior specific properties, including strength, stiffness, and creep resistance [1–5]. Conventional fabrication methods, such as stir casting [6–9], powder metallurgy [10], and squeeze casting [11] have been successfully applied to produce discontinuously microparticles-reinforced MMCs. However, the stir casting process is very easier to entrap of gases and unwanted inclusions to the Mg melting, which significantly reduced the mechanical properties of the composites. The powder metallurgy method requires Mg powders which are very expansive, and involves complicated processes during the material fabrication. Squeeze casting method is very complex, and difficult to control the fractions of reinforcements. In general, tensile strength and elastic modulus of the MMCs fabricated by conventional fabrication methods are improved, but their ductility is reduced. Their poor ductility significantly limits the widespread use of the MMCs.

The need for high-performance and lightweight materials for some demanding applications has led to extensive efforts in the development of novel and cost-effective fabrication technologies for magnesium matrix composites. A new solidification processing technique, ultrasonic vibration process, has been developed at UW-Madison [12–15]. It is efficient in dispersing nanoparticles in metal melts. Ultrasonic cavitation can produce transient (on the order of nanoseconds) "micro-hot-spots" that can have temperatures of about 5000 °C, pressures above 1000 atm, and heating and cooling rates above 10^{10} K/s [16]. The strong impact coupled with local high temperatures can break nanoparticle clusters and clean the particle surface. Compared with conventional metal matrix composites reinforced by micro particles or fibers, the good ductility of alloy matrix was retained or even improved in nanoparticles-reinforced metal matrix composites fabricated by ultrasonic vibration. However, the major disadvantage of nanoparticles-reinforced metal matrix composites

K. B. Nie · X. J. Wang (✉) · K. Wu · L. Xu · M. Y. Zheng · X. S. Hu
School of Materials Science and Engineering, Harbin Institute of Technology, No. 92, West Da-Zhi Street, Harbin 150001, People's Republic of China
e-mail: xjwang@hit.edu.cn

K. B. Nie
e-mail: kaibo.nie@gmail.com

fabricated by ultrasonic vibration lies in the relative high cost of the reinforcement materials and low elastic modulus. It is known that a volume fraction of at least 15–20% reinforcement is preferred for an effective increase of strength and elastic modulus in particle or short fiber-reinforced magnesium matrix composite. To realize a combination of excellent strength and ductility of these MMCs, it is necessary to select the type, size, and volume fraction of the reinforcements besides the fabrication methods.

Several studies have been reported in relation to the effect of ultrasonic vibration on microstructure and mechanical properties of metal matrix composites, but they are mainly focused on metal (including magnesium) matrix composites reinforced with nanoparticles rather than microparticles. Thus, the primary aim of the present study was to synthesize magnesium matrix composites reinforced with micro SiC particles by introducing ultrasonic vibration and to investigate the effect of ultrasonic vibration on the microstructure and tensile properties of the composites.

Experimental conditions

A commercial ingot of AZ91 alloy with a nominal composition of Mg–9.07Al–0.68Zn–0.21Mn was used as the matrix alloy. SiC particles with an average diameter of 1 μm were selected as reinforcements. The experimental setup for this study is shown in Fig. 1. The ultrasonic vibration device consists of a transducer with a maximum power of 2 kW and frequency of about 20 kHz. Figure 2 gives a flow to show the whole fabrication process against temperature. In each experiment, about 1 kg of the AZ91 alloy was first melt in the mild steel crucible under an atmosphere containing a gas mixture of CO_2/SF_6. Once the melt temperature of 700 °C was attained the aluminum foil containing SiC particles was submerged beneath the surface of the melt. Then, the ultrasonic probe was dipped into

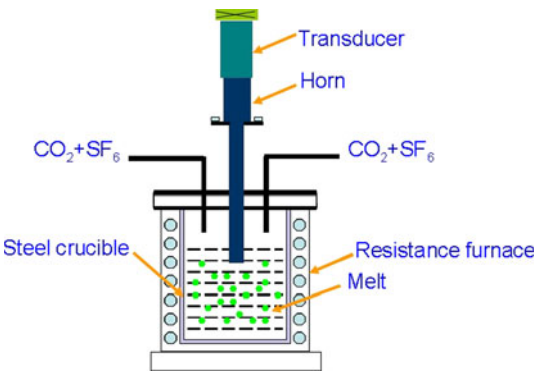

Fig. 1 Schematic of experimental setup for ultrasonic vibration of magnesium melt

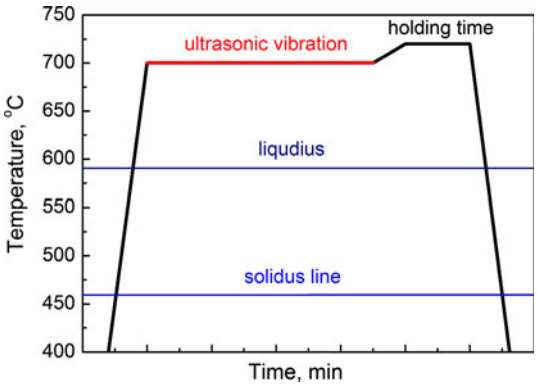

Fig. 2 Schematic illustration of the temperature–time sequences for ultrasonic vibration

the melt for about 20 mm. The melt temperature for ultrasonic vibration was also controlled at about 700 °C. The melt was then ultrasonically processed at 350 W power level for 20 min before the ultrasonic probe was removed from the melt. After ultrasonic vibration, the melt was elevated to a pouring temperature of 720 °C, and cast into a preheated steel mold (450 °C) and allowed to solidify under a 100 MPa pressure. The volume content of SiC particles in the SiCp/AZ91 composites was 1 and 3%, respectively. It should be noted that ultrasonic vibration was not used during the solidification process but in molten composite. For comparison, an AZ91 alloy ingot without ultrasonic vibration was also cast under the same conditions.

Optical microscopy (OM), scanning electron microscopy (SEM), and transmission electron microscopy (TEM) were employed to study microstructure modification of the matrix alloy and reinforcement distribution introducing by ultrasonic vibration. Samples for microstructure analysis were prepared by the conventional mechanical polishing and etching using 4% oxalic acid. Microstructural features of the SiCp/AZ91 composite were identified using energy dispersive spectrophotometric (EDS) analysis. Specimens for TEM were prepared by grinding–polishing the sample to produce a foil of 50-μm thickness followed by punching 3-mm-diameter disks. The disks were ion beam thinned. Tensile test was conducted at room temperature and at a rate of 0.5 mm/s.

Results and discussion

Microstructures

Figure 3 shows OM micrographs of as-cast AZ91 alloy and SiCp/AZ91 composites. The microstructure of the as-cast AZ91 alloy without ultrasonic vibration consists of primary α-Mg and eutectic phase β-$Mg_{17}Al_{12}$ as shown in Fig. 3a. The phase β-$Mg_{17}Al_{12}$ in the form of plates is located

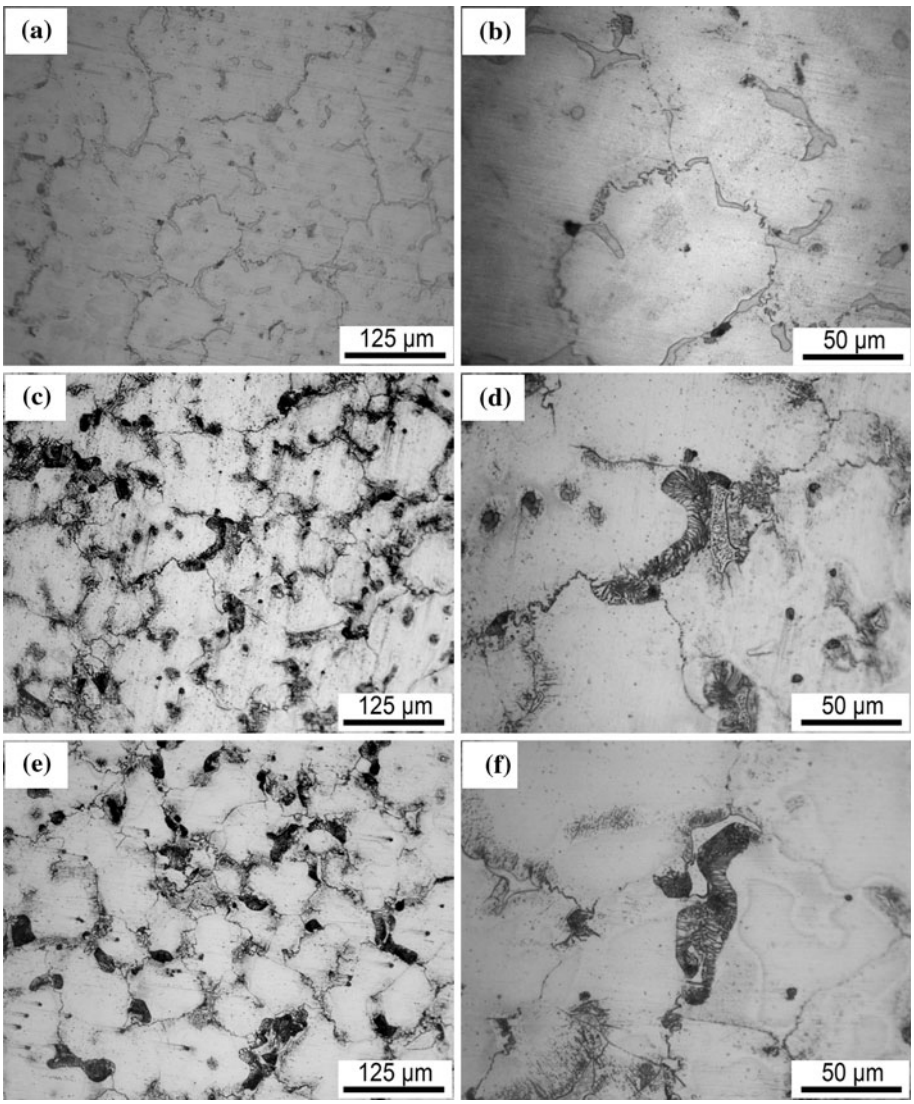

Fig. 3 OM micrographs of **a** AZ91 alloy, **c** 1 vol% SiCp/AZ91 composite, **e** 3 vol% SiCp/AZ91 composite; higher magnification of **b** AZ91 alloy, **d** 1 vol% SiCp/AZ91 composite, and **f** 3 vol% SiCp/AZ91 composite

mainly at grain boundaries. With the addition of SiC particles, as shown in Fig. 3c, e, grains of matrix in both 1 and 3 vol% SiCp/AZ91 composites are obviously refined. The grains of matrix in the 3 vol% SiCp/AZ91 composite demonstrate marginal refinement compared with that of 1 vol% SiCp/AZ91 composite. At higher magnification, the morphology of $Mg_{17}Al_{12}$ were significantly changed as show in Fig. 3b, d, f. Most of the phase β-$Mg_{17}Al_{12}$ in the SiCp/AZ91 composites is changed to fine and oriented lamellae along the grain boundary. This change may improve the mechanical properties of SiCp/AZ91 composites.

Figure 4 shows the SEM micrographs of the 1 vol% SiCp/AZ91 composite. It can be seen from Fig. 4a that there exists a lamellar plates and a relatively spherical phase in the 1 vol% SiCp/AZ91 composite according to their morphology, whose typical SEM images are shown in Fig. 4b at higher magnification. Figure 5 presents the results of EDS analysis of the regions marked in Fig. 4b. It can be clearly seen that the dominant intermetallic phase in the 1 vol% SiCp/AZ91 composite is $Mg_{17}Al_{12}$ as shown in Fig. 4a. Because the EDS peaks for SiC particle (area A) inevitably include compositional information of the matrix alloy, it is evident that Si peak corresponds only to composition of SiC particles compared with the compositional information of the matrix in the 1 vol% SiCp/AZ91 composite.

Figure 6 shows the SEM micrographs of SiC particles along the grain boundaries in the SiCp/AZ91 composites. It is found that most of the SiC particles distribute at grain boundaries in both 1 vol% SiCp/AZ91 composite and 3 vol% SiCp/AZ91 composite. There are some SiC agglomerations in the composites. The SiC particles inside the grains are little. It can be attributed to "push" effect of the solidification front on particles. Although ultrasonic vibration can be also employed to fabricate microparticles-reinforced magnesium matrix composite, the processing

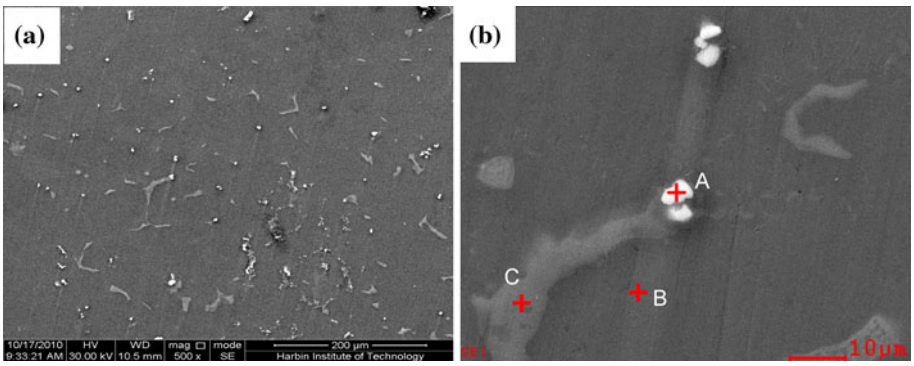

Fig. 4 SEM micrographs of 1 vol% SiCp/AZ91 composite: **a** under lower magnification; **b** under higher magnification

parameters were not optimized. Further improvement in the dispersion of particles is desired.

Figure 7 shows the TEM micrographs of SiC particles in the 1 vol% SiCp/AZ91 composite. It can be seen that inside the particle clusters as shown in Fig. 6a, SiC particles are separated by magnesium. At higher magnification, as shown in Fig. 7b, no interfacial activity is observed in the interface between a SiC particle and the AZ91 alloy matrix. This indicates that the SiC particles bond well with the alloy matrix.

The microstructure of materials usually depends on the nucleation stage and on the subsequent growth condition. Although there are many submicroscopic particles of insoluble solid impurities in the melt, the number of active particles is insufficient for effective heterogeneous nucleation. In order to achieve the grain refinement, the sufficient nuclei are essential [17, 18]. During the ultrasonic vibration, when the melt is subjected to local random compression–expansion cycles, if the local pressure in the melt becomes less than its vapor pressure during the half-period of expansion, an ultrasonic cavity is formed. The ultrasonic cavity continues to grow until collapses during the half-period of compression, thus, producing a high intensity shock wave in the melt [19]. Under the action of high intensity of shock, the insoluble solid impurities including SiC particles throughout the melt become active and involve in the solidification of process as nuclei, which lead to the heterogeneous nucleation easily upon a slight undercooling.

However, the final grain size is determined not only by the nucleation, but also by the growth condition. If the dissipation of latent heat is slow, the initial nuclei are unstable and remelt during the later solidification. Therefore, a rapid extraction of latent heat as well as the sufficient nuclei is necessary for refining the microstructures. In the present study, the SiCp/AZ91 composite melt was elevated to a pouring temperature of 720 °C after the ultrasonic probe was removed, as a result part of the initial nuclei introduced by ultrasonic cavitation may remelt. On the other hand, the temperature of the steel mold for casting was higher leading to a slow extraction of latent heat during the later solidification process. In addition, the "push" effect of the solidification front on the insoluble solid impurities include SiC particles may cause the nuclei clusters at grain boundaries. Consequently, the microstructures with grain refinement of matrix, as well as the refined phase $Mg_{17}Al_{12}$ along the grain boundaries in the SiCp/AZ91 composite solidified from the melts as shown in Figs. 3 and 4.

Tensile properties

Figure 8 shows yield strength (σ_{YS}, 0.2% proof stress), ultimate tensile strength (σ_{UTS}, the ultimate tensile strength), and elongation to fracture of as-cast AZ91 alloy and SiCp/AZ91 composites. It can be seen from Fig. 8 that the ultimate tensile strength, yield strength, and elongation to fracture of the SiCp/AZ91 composites are simultaneously enhanced compared with that of the as-cast AZ91 alloy. The massive increase in elongation to fracture is different from what is observed in the composites using traditional fabrication methods, such as stir casting. The improvement of the tensile strength of the SiCp/AZ91 composites could be attributed to the refinement of α-Mg grains, the $Mg_{17}Al_{12}$ phases, as well as the addition of SiC particles present in the microstructures as shown in Figs. 3 and 4.

According to the classic Hall–Petch equation: $\sigma_y = \sigma_0 + K_y d^{-1/2}$, where σ_y is the yield strength, σ_0 and K_y are material constants, and d is the mean grain size. The value of K_y is dependent on the number of slip systems. It is higher for HCP metals than for FCC and BCC metals [20]. Since Mg is HCP, the grain size affects the yield strength significantly. As shown in Fig. 3, more grain boundaries associated with finer α-Mg grains would obviously result in better mechanical properties. In the as-cast AZ91 alloy without ultrasonic vibration, as shown in Fig. 3a, b, the phase $Mg_{17}Al_{12}$ constitutes a continuous brittle phase at the grain boundaries of α-Mg dendrites which decreases the tensile strength. Figure 3b, d

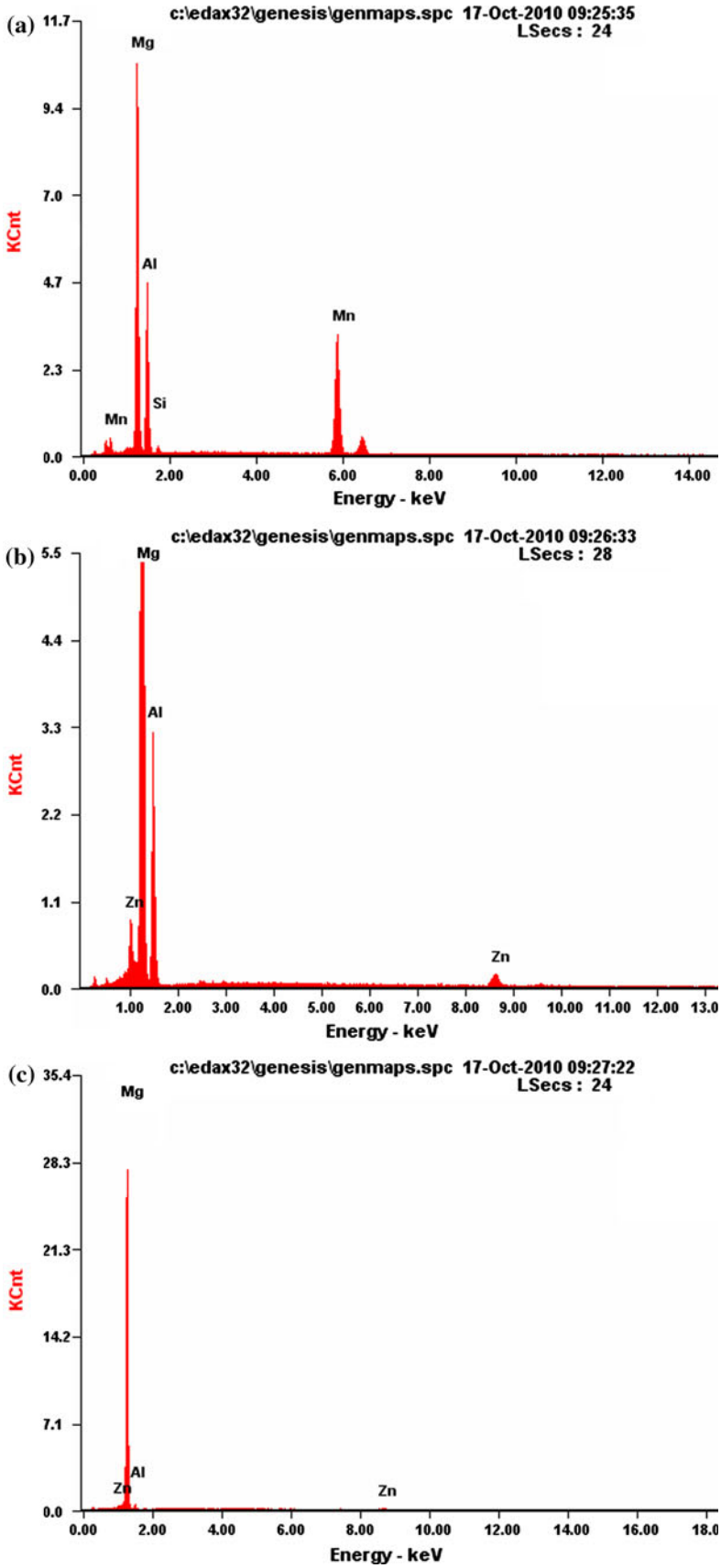

Fig. 5 EDS spectrum of phases in the 1 vol% SiCp/AZ91 composite: **a** region A; **b** region B; **c** region C marked in Fig. 4b

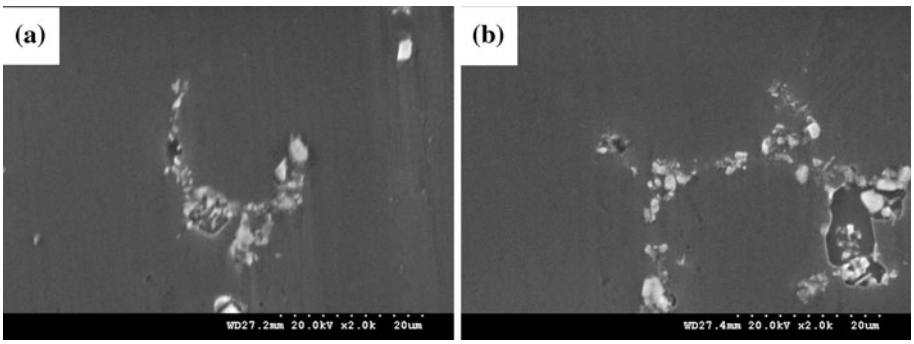

Fig. 6 SEM micrographs of SiC particles along the grain boundaries: **a** 1 vol% SiCp/AZ91 composite; **b** 3 vol% SiCp/AZ91 composite

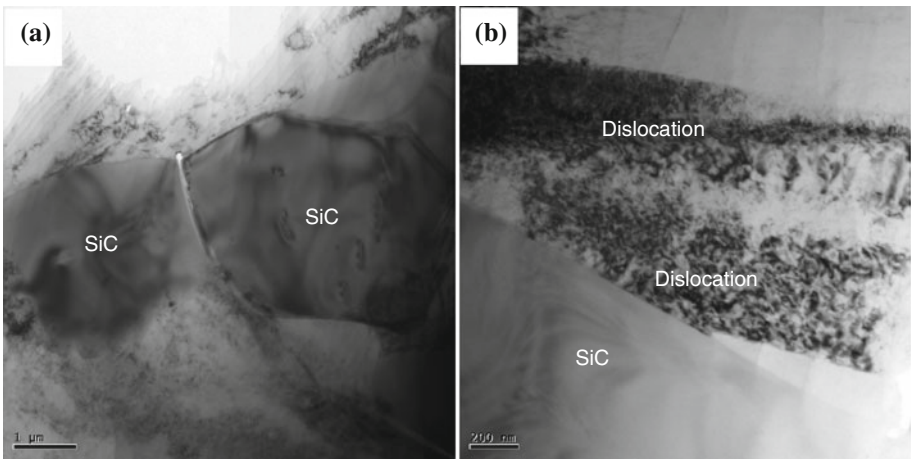

Fig. 7 TEM micrographs 1 vol% SiCp/AZ91 composite: **a** low magnification of SiC particles; **b** higher magnification of SiC particles

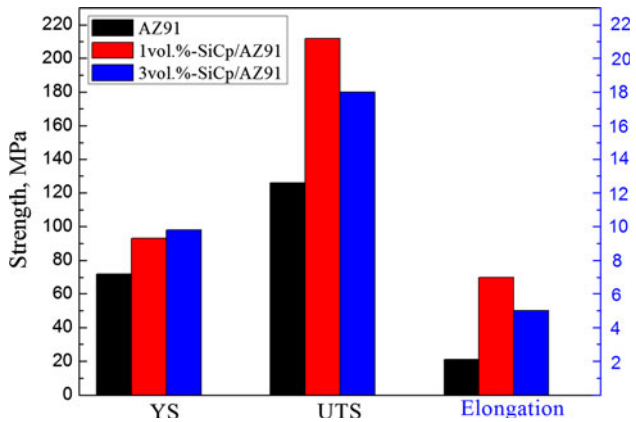

Fig. 8 Tensile strength of AZ91 alloy and SiCp/AZ91 composites

illustrates the transition of the phase $Mg_{17}Al_{12}$ from massive to lamellar in the SiCp/AZ91 composites which will contribute to the improvement of the tensile strength.

On the other hand, as shown in Fig. 7, the dislocations near the interface between the SiC particles and AZ91 alloy matrix (due to the different coefficient of thermal expansion between the matrix and the reinforcement particles) also contribute to the mechanical properties of the composite. However, in the 3 vol% SiCp/AZ91 composite, the ultimate tensile strength and elongation to fracture decrease due to more SiC clusters along the grain boundary of matrix compared with the 1 vol% SiCp/AZ91 composite (Fig. 6). Thus, it is thought that discontinuity and refinement of the phase $Mg_{17}Al_{12}$, the refinement of α-Mg grains and the addition of the SiC particles should play a major role in enhancement of the tensile properties of the composite in our study as shown in Fig. 8.

Conclusions

(1) Ultrasonic vibration can be successfully utilized to fabricate magnesium matrix composites reinforced with micro SiC particles. The ultimate tensile strength, yield strength, and elongation to fracture of the SiCp/AZ91 composites are simultaneously enhanced compared with that of the as-cast AZ91 alloy.

(2) With the addition of SiC particles grains of matrix are refined, while the sphericity and distribution of the phase $Mg_{17}Al_{12}$ along the grain boundary are changed to fine and oriented lamellae along the grain boundary in the SiCp/AZ91 composites compared with that of the as-cast AZ91 alloy.

(3) Most of the SiC particles locate at grain boundaries in both 1 vol% SiCp/AZ91 composite and 3 vol% SiCp/AZ91 composite. Inside particle clusters some SiC particles may be still separated by magnesium at higher magnification.

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