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Mechanical Properties and Microstructures of Nano SiC Reinforced ZE10 Composites Prepared with Ultrasonic Vibration

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Abstract. In the present investigation, SiC reinforced ZE10 alloy composites were fabricated by direct chill casting assisted with ultrasonic vibration. Two kinds of SiC with a size of 50 nm and 2 μm were chosen. For comparison, ZE10 alloys with and without ultrasound were also fabricated. The microstructures and the distribution of SiC were examined by optical microscopy and scanning electron microscopy. Neutron diffraction was also used to identify the second phases in the composites. In addition, mechanical properties such as hardness, creep and compression were evaluated. The results show that SiC was successfully introduced into the magnesium matrix. After the addition of SiC, the mechanical properties of the composites exhibit a slight decrease, which might be due to the grain coarsening.

Introduction

Magnesium based metal matrix composites reinforced with ceramic fibres and/or particles in micrometer scale have proven outstanding creep and wear resistance, and provide strength even at high temperatures. However, even a low fraction of nanoparticles can achieve better improvement of mechanical properties. The enhancement of mechanical properties is due to both particle strength effect (Orowan Strengthening) and grain refinement effect. Various nanoparticles reinforced magnesium alloys composites were reviewed in the literature [1]. Among them, SiC reinforced magnesium alloy composites were widely investigated due to the effective grain refinement and mechanical properties enhancement. Both micro and nano SiC particles show promising influence on mechanical properties of pure magnesium and various of magnesium alloys systems, such as AZ91 [2, 3], AM80 [4], Mg-(2,4)Al-Si [5], Mg-4Zn [6], Mg-6Zn [7]. All of them show a grain refinement and mechanical properties improvement even with some SiC clusters in the composites.

There are several ways to incorporate nano ceramics into magnesium alloys such as stir casting [8], disintegrated melt deposition[9], friction stir processing [10], powder metallurgy[11], and melt casting assisted with ultrasound [2, 5, 12, 13]. Among them, casting assisted with ultrasound is proven to be an effective and economical way to disperse the reinforcement uniformly in the composites. The promising dispersion of nano particles is due to the strong micro scale transient cavitation and acoustic streaming of ultrasonic wave [12]. Ultrasound also results in grain refinement in pure magnesium and AZ31 due to the cavitation-enhanced heterogeneous nucleation [14]. However, the grain size increases with the distance from the radiator.

ZE10 sheets exhibit a weak texture which leads to changes in the mechanical anisotropy as well as superior formability [15]. In this study, both micro and nano SiC were introduced into the ZE10 alloy with the assistance of ultrasound. The microstructures and mechanical properties of the as cast ZE10, ZE10 with ultrasound, micro SiC reinforced ZE10 composites (μm SiC/ZE10), and nano SiC reinforced ZE10 composites (nm SiC/ZE10) were investigated.

Experimental

The ZE10 alloy with a nominal composition of Mg-1Zn-0.5RE (RE=rare earth elements) and SiC reinforced ZE10 composites were cast using a permanent mold direct chill casting process. The chemical composition of ZE10 analyzed by optical emission spectroscopy (Spark Analyzer M9, Spectro, Germany) is shown in Table 1. The detailed casting process was described elsewhere [13]. ZE10 was molten and kept at 720°C, 1 wt. % of 2 µm SiC particles and 1 wt. % of 50 nm SiC were introduced in the melts, respectively. The melts of composites were stirred at 675 rpm for 5 min. The ultrasonic treatment was then operated for 10 min with a dipping depth of 20 mm in the melt. The ultrasound equipment (Hielscher, Germany) consists of an ultrasound generator UIP1500hd with a frequency of 20 kHz and a maximum power of 1.5 kW. For comparison, ZE10 alloys with and without ultrasound were also cast using the same process. Previous work shows that the casting is very dense [16], because the remaining melt is always above the solidified material, which leads to shrinkage feeding at every point. A constant flux of Ar-SF6 (5:1) cover gas was introduced on top of the melt during casting and ultrasound processing.

Table 1 Chemical composition of ZE10

Elements	Zn	La	Ce	Zr	Mg
Contents [wt. %]	1.39	0.19	0.31	0.046	Bal.

For microstructural investigations, all materials from the center of the castings were ground, polished with Oxide Polishing Silica (OPS, a colloidal silica suspension with an average grain size of 0.04 µm) suspension and etched with a solution of 8 g picric acid, 5 ml acetic acid, 10 ml distilled water, and 100 ml ethanol. Optical microscopy was performed with a LEICA DMI5000 M microscope. Grain sizes were determined using the line intercept method. The distribution of SiC in the composites was checked by a Zeiss Ultra 55 Scanning Electron Microscope (SEM) equipped with Energy Dispersive X-ray Spectroscopy (EDS). Neutron diffractometer STRESS-SPEC located at FRM II (Garching, Germany) was used to investigate the existence of SiC in the composites. The measured sample was a 10 mm^3 cubic. Measurement was performed using a wavelength of 0.165 nm by Ge monochromator with an incoming beam of Ø15 mm. The whole diffraction pattern (2-theta scan) ranging from 35° to 105° with 11 steps was collected in 20 min. Counting per step was using a 2D-aera detector. As both ZE10 and ZE10 with ultrasonic treatment samples contain no SiC, SEM and neutron diffraction were only performed on ZE10.

Hardness was evaluated using a Vickers testing facility under a load of 49 N. Two kinds of cylinders were prepared using the spark erosion technique. The cylinders with a diameter of 6 mm and a length of 15 mm were used for creep tests. The cylinders with a diameter of 11 mm and a length of 16.5 mm were prepared for room temperature compression tests. Creep tests were performed on ATS Lever Arm Test Systems under constant stresses of 100 MPa and a temperature of 175°C. The compression tests were done in a Zwick 050 testing machine at a stain rate of 1×10^{-3} s^{-1}.

Results and discussion

The optical microstructures of as - cast ZE10, ZE10 with ultrasonic treatment, µm SiC/ZE10, and nm SiC/ZE10 are shown in Fig.1. The grain sizes of as cast ZE10 and ZE10 with ultrasonic treatment are 106 ± 11 µm, and 109 ± 18 µm, respectively. The grain sizes are close, which indicates ultrasonic treatment has little effects on nucleation and grain growth of ZE10 alloy. The grain sizes of µm SiC/ZE10 and nm SiC/ZE10 are 210 ± 50 µm, and 225 ± 46 µm, respectively. In comparison with ZE10, grain sizes of the composites increase nearly 100 %, which is really different from previous knowledge. Both micro and nano SiC particles show a great potential in grain refinement of lots of magnesium alloys, such as AZ91 [2, 3], Mg-Al [17], Mg-4Zn [6],

Mg-6Zn [7], Mg-(2,4)Al-Si [5], et al. The grain coarsening of SiC addition (both 2 μm SiC and 50 nm SiC) might be due to the inactivation of Zr (ZE10 contains 0.046 wt. % of Zr, as shown in Table 1) in the composites. Zr tends to form stable compounds with Si, C, Al, Mn, Fe and O which will deteriorate its grain refining effect [18, 19]. During the powerful ultrasonic cavitation, instantaneous hot spots caused by bubble collapse provide high temperature of – 5000 °C, high pressure of – 1000 atm, as well as heating and cooling rates above 10^{10} K/s [20]. Thus, it is of high possibility that SiC discomposes to free Si and C, which induces the deactivation of grain refining effect of Zr.

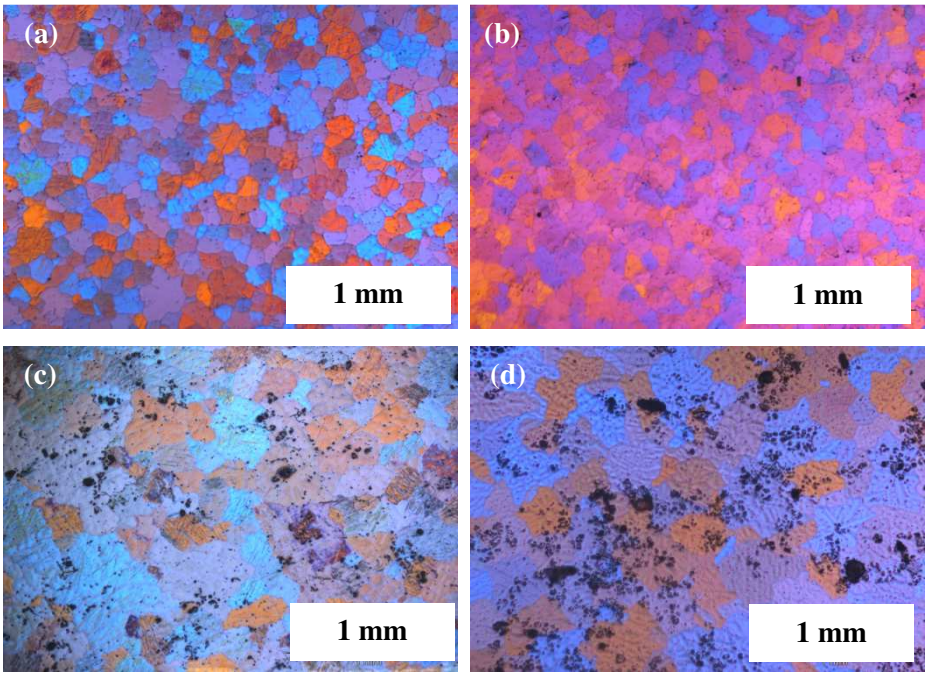

Fig. 1 Optical microstructures of (a) ZE10, (b) ZE10 with ultrasonic treatment, (c) μm SiC/ZE10, (d) nm SiC/ZE10

It is hard to characterize the SiC in the composites with conventional X-ray diffraction due to its less detectable volume mass. Thus neutron diffraction was introduced to analyse SiC in composites, the 2-theta diffraction pattern is plotted in Fig. 2. The patterns of both μm SiC/ZE10 and nm SiC/ZE10 show a small peak at 2θ = 46°, as marked with black arrow in Fig. 2 (a) which corresponds to SiC- (200) with a cubic structure (space group F-43m, a=0.4358 nm). The close up patterns between 44^0- 48^0 is shown in Fig.2 (b). The as – cast ZE10 show no such peak at 2θ = 46^0. This indicates both micro and nano SiC are successfully introduced to the composites with the assistance of ultrasound.

Fig.3 shows the SEM pictures of ZE10, μm SiC/ZE10, and nm SiC/ZE10. Mg-Zn-RE precipitates mainly distribute along the dendritic and grain boundary continuously. Small amount of this ternary phase can also be found inside the grains, as shown in Fig 3 (a) and (b). With the addition of micro and nano SiC, the Mg-Zn-RE precipitates still mainly present at the dendritic and grain boundary, but with a smaller size, as shown in Fig.3 (c-f). The particles in Fig.3 (c) and (d) tend to agglomerate to clusters. There are also different sizes of particle clusters in nm SiC/ZE10 composites. Furthermore, the clusters are not evenly distributed in the composites for both μm SiC/ZE10, and nm SiC/ZE10. The similar phenomenon can be found in other literatures [2, 3, 6, 7]. With the assistance of ultrasound, both micro and nano SiC clusters exist in μm SiC/AZ91 [3], nm SiC/AZ91 [2], nm SiC/Mg-4Zn [6], nm SiC/Mg-6Zn [7], et al.

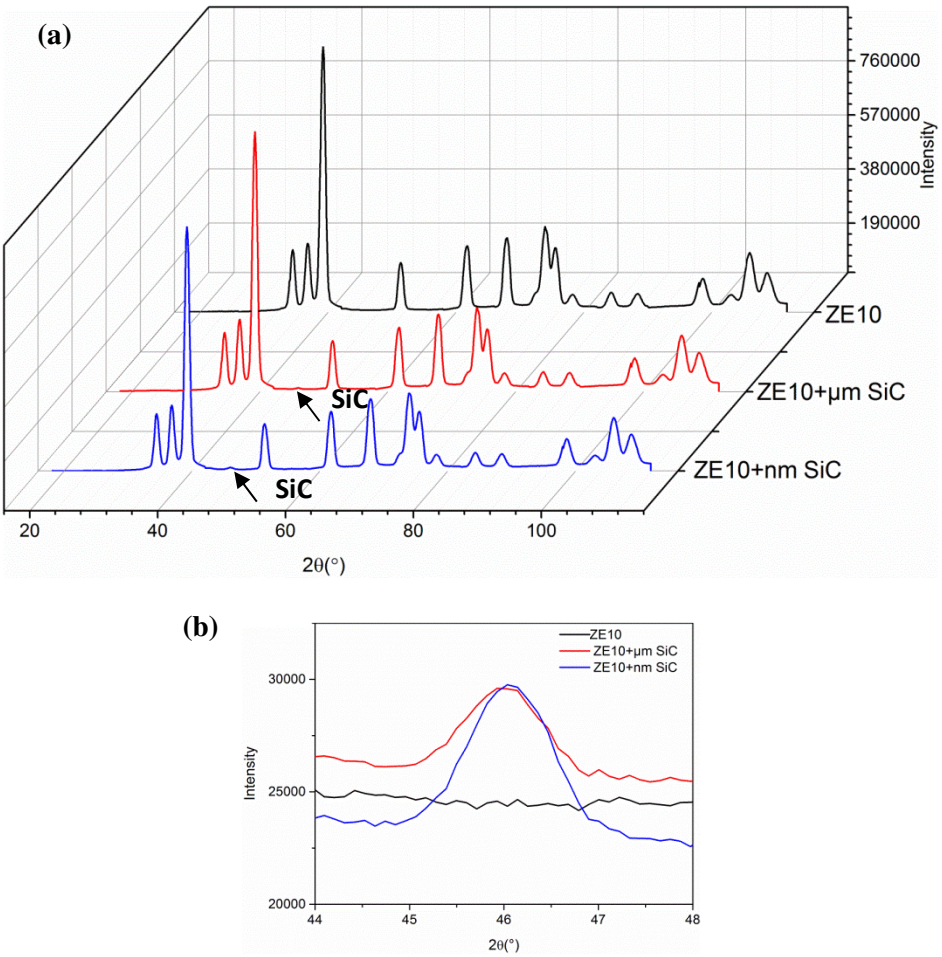

Fig. 2 (a) Neutron diffraction of ZE10 and the composites, (b) close up of the diffraction between 44 – 48 °

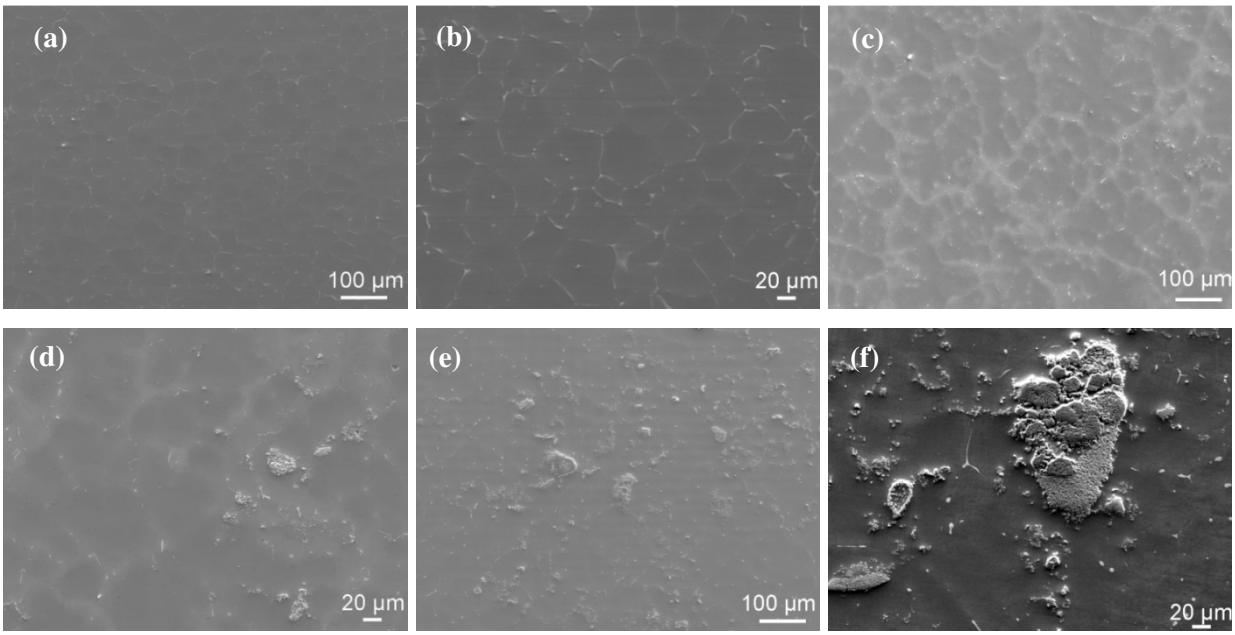

Fig. 3 SEM microstructures of (a) and (b) ZE10, (c) and (d) μm SiC/ZE10, (e) and (f) nm SiC/ZE10

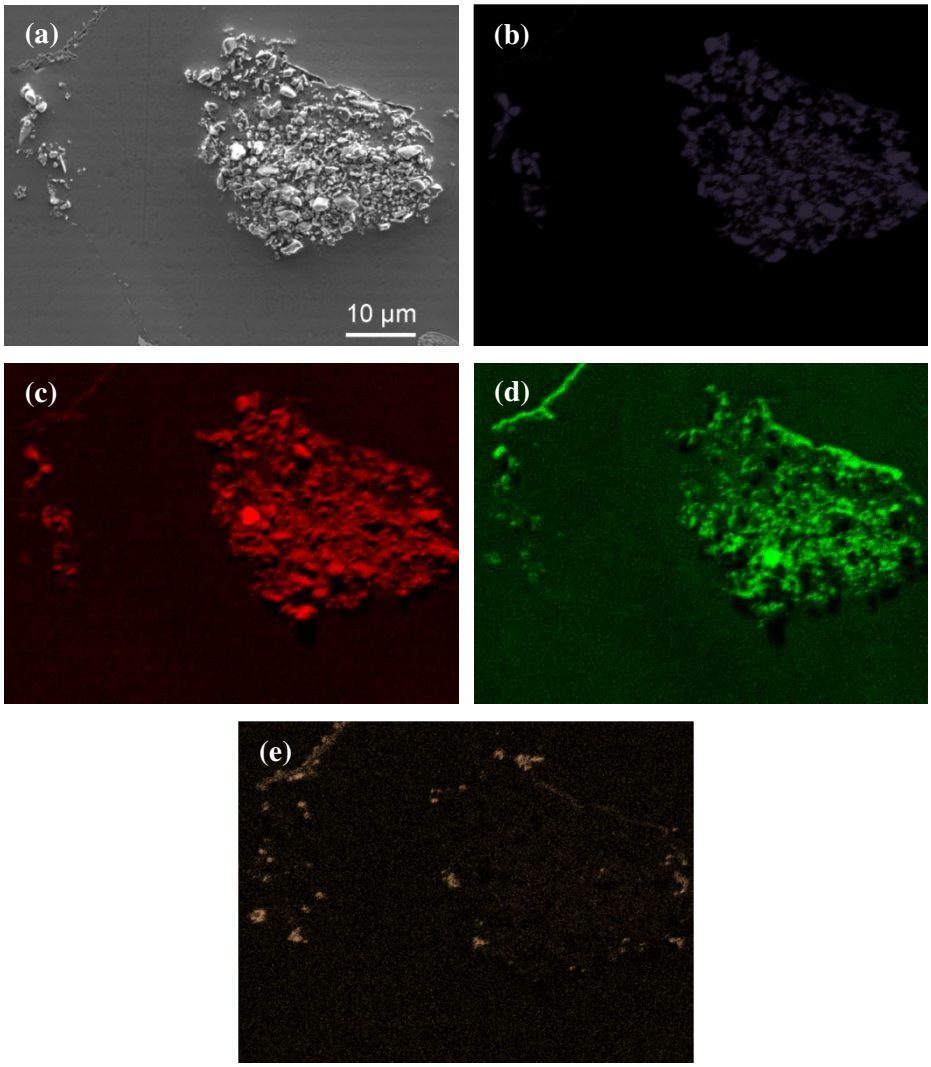

Fig.4 SEM microstructure of (a) μm SiC/ZE10, EDS mapping of (b) Si K, (c) C K, (d) O K, (e) Zr L

The clusters in the composites were checked by EDS mapping. The results of μm SiC/ZE10 and nm SiC/ZE10 are shown in Fig.4 and Fig.5, respectively. Combine the neutron diffraction and EDS results, both clusters in μm SiC/ZE10 and nm SiC/ZE10 are believed to be SiC particles. The high contents of oxygen on SiC clusters are due to two possible reasons: (1) the area contains SiC are easy to be oxidized during the preparation (grinding and polishing) of SEM samples; (2) remaining OPS (silica) from the polishing procedure. Zr can be found either near the SiC clusters or near the high oxygen containing area, which indicates there might be stable compound between Zr and Si, C, and/ or O.

The mechanical properties of these four alloys are listed in Table 2. The hardness of all alloys shows little difference. ZE 10 with ultrasonic treatment shows the highest hardness with a value of 40 ± 1.6 HV. This alloy also shows the best compression creep properties with a minimal creep rate of 2.2×10^{-7} s^{-1} under 100 MPa at 175 °C. The creep sample for μm SiC/ZE10 crashed after 2 hours of test which might be due to the uneven distribution of micro SiC clusters. Results of ambient compression tests also show a slightly deterioration on elongation and strength of both composites, and little difference of elongation and strength between ZE10 and ultrasonic ZE10. The decrease of mechanical properties of both micro and nano composites is due to the grain coarsening, and also may be due to the uneven distribution of SiC clusters in the composites. Detailed mechanism still needs to be investigated.

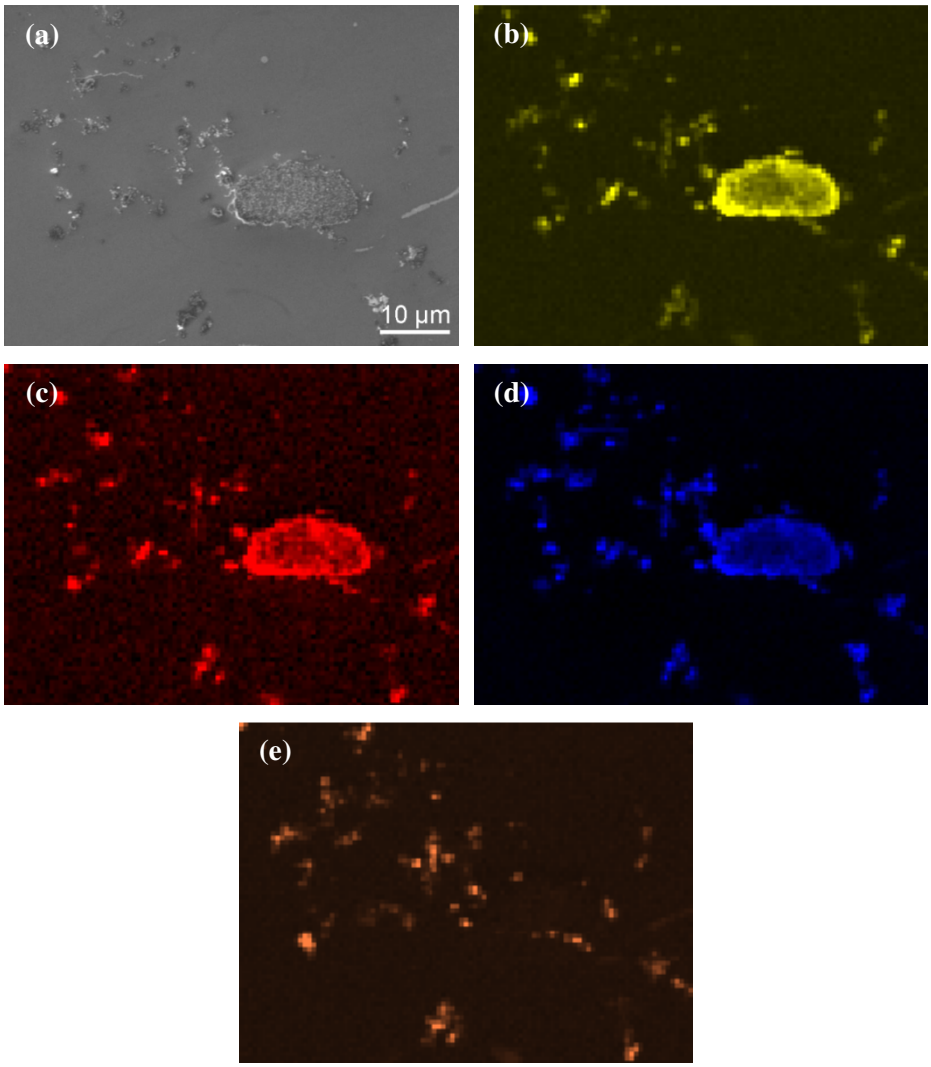

Fig.5 SEM microstructure of (a) nm SiC/ZE10, EDS mapping of (b) Si K, (c) C K, (d) O K, (e) Zr L

Table 2 Mechanical properties of ZE10, ZE10 with ultrasonic treatment, µm SiC/ZE10, nm SiC/ZE10

	Hardness [HV]	Minimal Creep rate [s^{-1}]	Rp 0.2 [MPa]	Rm [MPa]	A% [%]
ZE10	37.4±1.9	4.3×10^{-7}	99.4±6.5	338.2±5.5	16.4±0.6
ZE10-ultrasonic	40.0±1.6	2.2×10^{-7}	99.4±6.3	333.8±3.2	16.5±0.1
µm SiC /ZE10	37.1±1.8	failure	101.2±0.8	307.3±1.9	14.1±0.5
nm SiC /ZE10	38.3±2.8	8.6×10^{-7}	98.8±0.9	297.8±0.4	13.9±1.3

Summary

With the assistance of ultrasound, micro SiC and nm SiC are successfully introduced in ZE10 alloy, which is proven by neutron diffraction. The increase of grain sizes of μm SiC/ZE10 and nm SiC/ZE10 is due to the inactivation of grain refinement by Zr. Consequently, SiC is not a suitable grain refiner in the alloy systems which contain Zr, especially when the ultrasound is applied in the casting procedure. The mechanical properties (hardness, creep properties, and compression properties) of composites are slightly deteriorated due to the grain coarsening.

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