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Microstructures and tensile properties of nano-sized SiC_p/Al-Cu composites fabricated by semisolid stirring assisted with hot extrusion

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ABSTRACT

Al-Cu matrix composites containing nano-sized SiC_p were successfully prepared by dispersing nano-sized SiC_p/Al master alloy into the molten Al-Cu alloy with combined semi-solid stir casting and hot extrusion processes. Microstructures of nano-sized SiC_p/Al-Cu composites and their tensile properties were investigated. Results show that the α-Al dendrites became finer and better formed and nano-sized SiC_p was formed in the α-Al grains. With nano-sized SiC_p adding to the Al-Cu matrix, the tensile strength of the described composites increased remarkably especially under the temperature of 453 K, while their plasticity were still high. Nano-sized SiC_p particles had been uniformly dispersed in the Al alloy, yielding composites with high strength at elevated temperatures. The strengthening mechanisms of nano-sized SiC_p were also discussed. This will raise the probability of using Al matrix composites as lightweight materials for structural applications at elevated temperatures.

1. Introduction

Composites formed by combining different types of materials together are promising to stretch the performance boundary of monolithic materials [1,2]. Aluminum matrix composites (AMCs) reinforced by SiC_p particles have been a hot research issue in recent years mainly owing to their excellent properties such as high tensile strength and elastic modulus as well as good wear resistance, etc. [3–7]. Thus, such materials have potential applications in improving energy efficiency and system performance in some industries, such as automobile and aerospace. Commonly the dimension and distribution of the reinforcement have large effect on the properties of the composites. Compared with the traditional micron-sized SiC_p reinforced Al composites, the Al matrix composites reinforced by nano-sized SiC_p not only possess higher tensile strength, but also maintain good ductility, which entitles them with more competitive ability for applications [8]. However, for nano-sized reinforcements, although they have the potential to improve the strength of the material without sacrificing the plasticity, they are difficult to be uniformly dispersed in the metal matrix.

Prior studies reported that nano-sized particles tended to form micro-clusters, mostly because of the interattraction between nano-sized particles induced by the van der Waals force [9–12]. Therefore, the major obstacle for the usage of nano-sized particles is to make the nano-sized particles be more evenly dispersed in the metal matrix. Up to now, two major techniques for fabricating Al matrix composites having nano-sized particles have been developed, i.e., solid-state processing, such as high-energy milling, powder metallurgy, and nano-sintering, and liquid-state solidification processing, e.g., stir casting [13–20]. In these techniques, stir casting has some important advantages, such as low cost, capability of producing products with complex shapes, and processing simplicity [16]. However, uniformly disperse nano-SiC_p particles in the molten Al alloy is very difficult by using the stir casting method because of their poor wettability, attractive Vander Waals interactions and large surface-to-volume ratio. The non-uniform particle distribution and poor bonding strength at the SiC/Al interface would severely degrade the properties of the composites [16]. Therefore, in order to facilitate the dispersion of nano-sized SiC particles in the Al matrix, other processes which are in favor of the dispersion of nano-sized reinforcement in the matrix are needed.

In this study, the Al-Cu composites containing relatively uniform

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nano-sized SiC_p distribution were successfully fabricated by dispersing nano-sized SiC_p particles into the Al melts through semi-solid stir casting combined with hot extrusion processes. Such prepared process of the composites had several advantages. Firstly, the usage of the precursor powders which are fabricated by the mix of nano-sized SiC_p and Al alloy powders using mechanical ball milling will be benefit for the dispersion of nano-sized SiC_p in the matrix, on account of the disruption of the agglomerated nano-sized SiC_p clusters in advance. Secondly, a nano-sized SiC/Al master alloy is made from elemental powder mixtures and then subsequently added to Al melts. The nano-sized SiC_p particles will be fixed separation distance, and gradually released and dispersed in the melt by the master alloy method. Finally, liquid-state stirring and hot extrusion processes will remarkably promote the nano-sized SiC_p dispersion in Al matrix. Similar processes for the preparation of nano-sized SiC_p/Al-Cu composites materials have not been reported. In addition, the microstructures of nano-sized SiC_p/Al-Cu composites and their tensile properties were investigated, and meanwhile the enhancement mechanism was discussed. This work can provide guidance for future research on the fabrication and application of nano-sized SiC_p/Al-Cu composites.

2. Detail Experimental Procedure

The Al alloy having the composition of (wt%) 5.0 Cu, 0.8 Mn, 0.7 Fe, 0.5 Mg, 0.5 Si, 0.25 Zn, 0.15 Ti, 0.1 Cr and Al (balance) was used as the matrix in this study. The nano-sized SiC_p particles (99.9% purity, ~60 nm diameter) were used as the reinforcing material. If the agglomerated nano-sized SiC_p clusters were added into the melt Al alloy directly, it is difficult to break the clusters by semisolid stirring and disperse the nano-sized particles uniformly. Therefore, it is very important to break the particle clusters before dispersing them into the base alloy.

Fig. 1 is the schematic display of the preparation process for nano-sized SiC_p/Al-Cu composites. The precursor powders were prepared by mixing the calculated nano-sized SiC_p and Al alloy powders (99% purity, ~50 μm diameter) through using mechanical ball milling with ZrO_2 balls at the speed of 50 rpm for 50 h. A ball to powder weight ratio of 8:1 was used. The mixed powders were then pressed into cylindrical compacts of 28 mm in diameter and about 40 mm in height. The hot press sintering of the cylindrical compacts was carried out in a vacuum environment at 873 K for 20 min. Such sintered parameter was mainly selected from the optimized results of our experiment. Then nano-sized SiC-Al master alloy was dispersed into Al melt by stirring with a graphite stirrer at the speed of 500 rpm for 5 min at 973 K. The contents of nano-sized SiC_p/Al-Cu composites were controlled to 1, 2, and 3 wt%, respectively. After that, the liquid was filled with a pre-heated steel die. The Al alloy and the composites were homogenized for 10 h at 758 K in order to avoid segregations. The extrusion was carried out with the help of a 200 ton hydraulic press and the materials were extruded to the batten shaped samples at 773 K with the extrusion ratio of 16. Before the tensile test, the extruded samples were solution-treated at 773 K for 2 h, and then aged at 433 K for 18 h.

The microstructure of the composites was investigated by optical microscope (OM, Olympus PMG3, Japan), field emission SEM (FESEM, JSM6700F, Japan) and TEM (JEM-2100F, Japan). The tensile test samples were machined with a gauge cross section of 5.0 × 2.5 mm and a gauge length of 30.0 mm. A servo-hydraulic materials testing system (MTS, MTS 810, USA) was applied to the tensile tests at room temperature, while the elevated temperature tensile tests were carried out on a tester (CSS-2905, Changchun, China) at 453 K and 493 K. The constant strain rate of $3 \times 10^{-4} s^{-1}$ was used and each sample was measured at least three times.

3. Results and Discussion

Fig. 2(a–d) displays OM images of the as-cast Al-Cu alloy and Al-Cu composites reinforced with the nominal content of 1 wt%, 2 wt% 3 wt% nano-sized SiC_p. As shown in Fig. 2(a), the α-Al dendrites in the Al matrix are coarse with a size of about 200 μm. While in the various contents nano-sized SiC_p/Al-Cu composites, the α-Al dendrites are significantly refined due to the existence of nano-sized SiC_p, as shown in Fig. 2(b–d). With addition of 1 wt% nano-sized SiC_p, as shown in

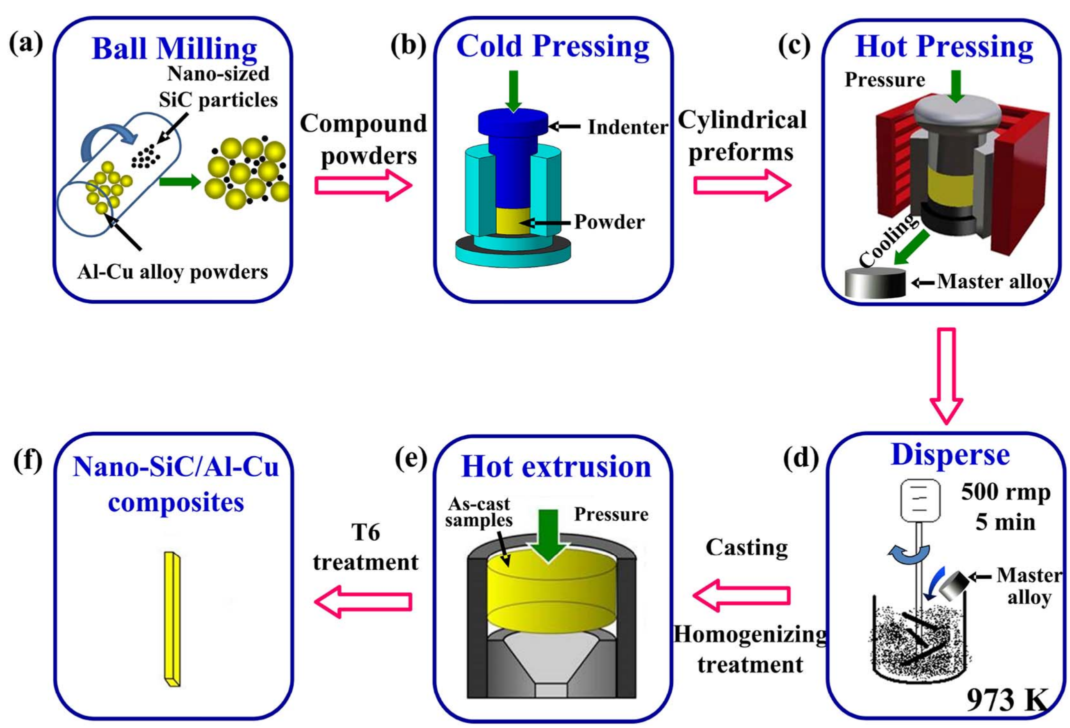

Fig. 1. Schematic display of preparation process, (a) ball milling dispersion of nano-sized SiC_p particles with Al alloy powders, (b) mixed powders were pressed into the cylindrical compacts, (c) preparation of nano-sized SiC/Al master alloy by hot press sintering of the compacts, (d) the SiC/Al master alloy was added into the molten metal with stirring the molten Al alloy, (e) hot extrusion process of the casted samples, (f) the samples of hot extruded nano-sized SiC_p/Al-Cu composites.

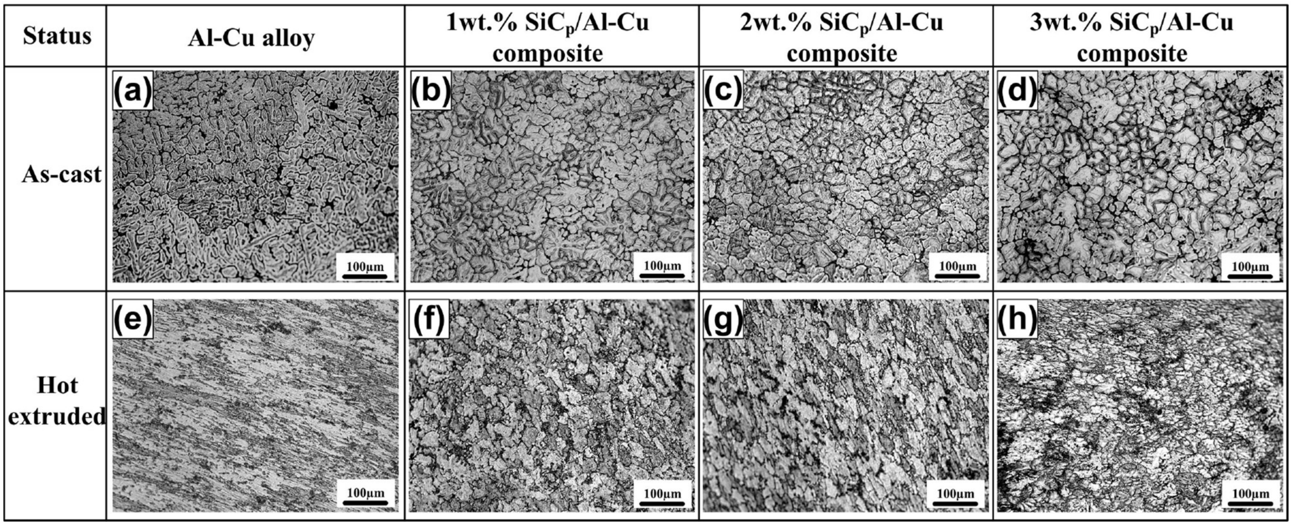

Fig. 2. As-cast microstructures of (a) Al alloy, and (b–d) the nano-sized SiC_p/Al-Cu composites with different contents of nano-sized SiC_p (b) 1 wt%, (c) 2 wt%, and (d) 3 wt%; the figs. (e–h) are corresponding to the hot extruded microstructures of (e) Al alloy, and the nano-sized SiC_p/Al-Cu composites with different contents of nano-sized SiC_p (f) 1 wt%, (g) 2 wt%, and (h) 3 wt%, respectively.

Fig. 2(b), the dendritic grains with small size are obtained, and the average grain size of α-Al decreases to about 100 μm, and further to 80 μm with equiaxed grain when 2 wt% nano-sized SiC_p is added (Fig. 2(c)). In the Al-Cu composite containing 3 wt% nano-sized SiC_p, as shown in Fig. 2(d), the α-Al is equiaxial crystal in shape with finer sizes about 75 μm, which increases the boundary concentration in the Al matrix. The increased boundary concentration could be beneficial to improve the composites' tensile strength since the grain boundaries usually play as a barrier to the dislocations motion. It is believed that a number of nano-sized SiC_p particles can provide heterogeneous nucleation sties for the α-Al crystal, and thus a more refined microstructure can be obtained. Meanwhile, other nano-sized particles have not been captured by solid-liquid interface, but been pushed away by the interface into the interdendritic regions. The "push" effect of the solid–liquid interface on other nano-sized particles gives rise to the retarded growth of α-Al dendrites.

Fig. 2(e–h) shows OM micrographs of the hot extruded Al alloy and Al-Cu composites reinforced with the nominal contents of 1 wt%, 2 wt % 3 wt% nano-sized SiC_p. After the hot extrusion, the α-Al dendrites are elongated or flattened, which will lead to a sharp increase in the dislocations, and the strength of the materials will be significantly improved. Compared with the as-cast microstructures, the α-Al grain after the hot extrusion process is obviously refined. In order to specify the grain sizes variation, statistic analyses of the grain sizes for 1 wt%, 2 wt % and 3 wt% nano-sized SiC_p/Al-Cu composites are respectively shown in Fig. 3. Clearly, the mean grain sizes of the described three composites are approximate 60 μm, 45 μm and 28 μm, respectively. As indicated in Fig. 2(e), the thick and brittle Al_2Cu phase in the grain boundaries of Al matrix alloy are broken into smaller particles, which make it difficult to distinguish the grain boundaries. However, in the microstructures of hot extruded nano-sized SiC_p/Al-Cu composites, as indicated in Fig. 2(f–h), the grain boundaries are still distinct which shows that the nano-sized SiC_p particles play an important role of grain boundary strengthening.

Fig. 4 shows the curves of engineering stress-strain for the extruded Al alloy and nano-sized SiC_p/Al-Cu composites under 298 K, 453 K and 493 K, and SEM images of the tensile fracture surface for the as-extruded Al-Cu alloy and the composites containing 2 wt% nano-sized SiC_p. Table 1 lists the detailed data of the tensile properties. As indicated, the yield strength and the ultimate tensile strength (UTS) of the composites (1 wt%, 2 wt% and 3 wt%) are improved significantly at different temperatures from 298 to 493 K. But tensile fracture strain decreases with the content of nano-sized SiC_p increasing. The higher the temperature is, the smaller the magnitude of the reduction. When the temperature was 298 K, for the Al-Cu composite reinforced by 1 wt% nano-sized SiC_p, its yield strength and ultimate tensile strength are 351 MPa and 482 MPa, which are 47 MPa and 19 MPa higher than those of the Al-Cu alloys, respectively. The tensile fracture strain decreases from 17.1% to 14.4%. With the nano-sized SiC_p content increase to 2%, the yield and tensile strength of the described composite increase to 373 MPa and 527 MPa, respectively, and tensile fracture strain decrease to 11.5%. With nano-sized SiC_p content further increased to 3%, the composite exhibits a little increase in the yield and ultimate tensile strength, but its tensile fracture strain greatly decreases to 7.8%. The similar phenomenon can be observed when the test temperature is 453 K and 493 K. Clearly, the strengths decrease with the increase of

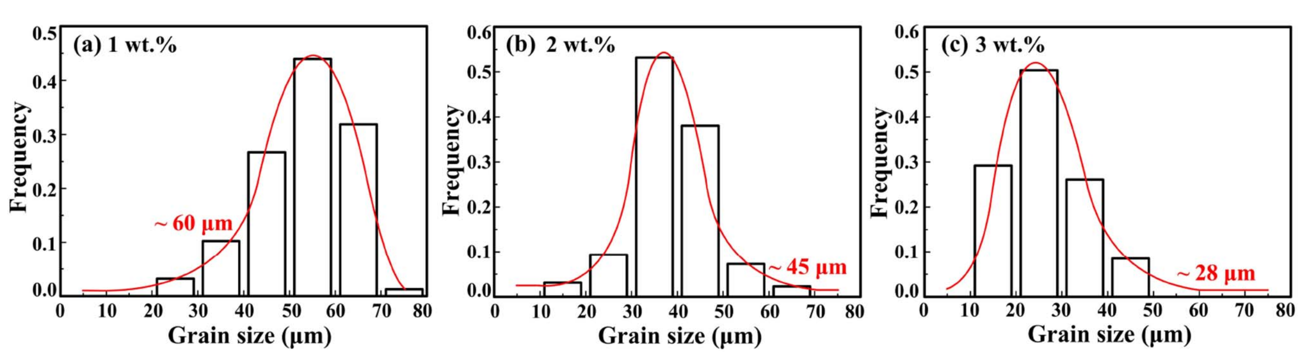

Fig. 3. Statistic analyses of the grain sizes for (a) 1 wt%, (b) 2 wt% and (c) 3 wt%.

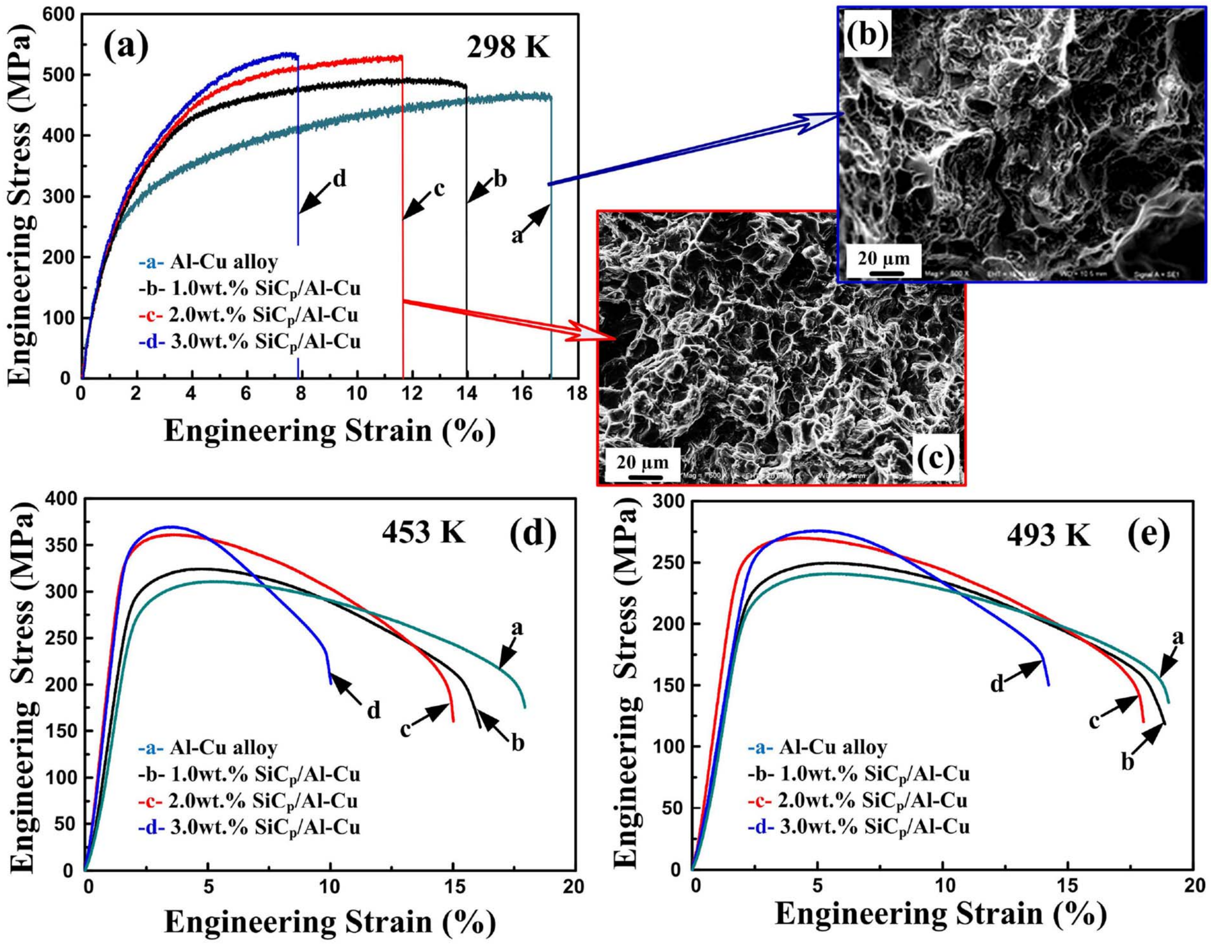

Fig. 4. Engineering stress-strain curves of the extruded Al alloy and nano-sized SiC_p/Al-Cu composites under (a) 298 K, (d) 453 K and (e) 493 K; SEM images of the tensile fracture surface of (b) Al alloy and (c) 2 wt% nano-sized SiC_p/Al-Cu composites.

the test temperature at the same SiC_p content. Although the composite having 3 wt% nano-sized SiC_p possesses the largest yield and ultimate tensile strength, it has the lowest fracture strain. Obviously, too many uniform distributed nanoparticles would deteriorate the plastic properties of the present composites. The 2 wt% nano-sized SiC_p/Al-Cu composite exhibits the best tensile properties, especially under high temperature conditions. Its strength is obviously improved with sacrificing very little in plasticity.

Fig. 4(b) and (c) exhibit the fracture surfaces of Al-Cu alloys and the composites containing 2 wt% nano-sized SiC_p particles. As shown in Fig. 4(b), the main fracture mode of the Al-Cu alloys is intergranular, and a few shallow dimples are found. However, the tensile fracture mode of the 2 wt% nano-sized SiC_p/Al-Cu composites are combined transgranular and intergranular. Many small and deep ductile dimples can be viewed on the fracture surfaces in Fig. 4(c), indicating that the transgranular fracture is dominated. Obviously, transgranular fracture could accommodate a heavier deformation. These changes also suggested that Al-Cu composites reinforced with nano-sized SiC_p possess better tensile properties than that of Al-Cu alloys.

Fig. 5(a) and (b) show TEM images of the distribution of nano-sized SiC_p in Al-Cu composites containing 1 wt% and 3 wt% nano-sized SiC_p particles. As shown, nano-sized SiC_p particles are well separated. With the increase in nano-sized SiC_p content, more particles can be detected in the matrix and their dispersions are significantly improved. Meanwhile, as shown in Fig. 5(c) the interface between nano-sized SiC_p and the matrix in 3 wt% nano-sized SiC_p/Al-Cu composite is clear, indicating the superiority of this fabrication technology. Also in Fig. 5(c), there are exhibited high density dislocations adjacent to the particles in the matrix. During the solidification process, the nano-sized SiC_p is in solid phase, and the α-Al dendrites forms and contracts in the solidification, which causes the space area near the ceramic particles to subject to severe compressive stress, resulting in a large number of dislocations. A higher magnification shows nano-sized SiC_p particle in Fig. 5(d). Fig. 5(e) shows the corresponding electron diffraction patterns obtained from the black areas in Fig. 5(d). The interface between nano-sized SiC_p and α-Al are shown in the HRTEM image of Fig. 5(f) and (g), which

Table 1
Data of tensile properties of the extruded Al-Cu alloy and its composites with different contents of the nano-sized SiC_p.

SiC_p contents (wt%)	298 K			453 K			493 K		
	$\sigma_{0.2}$ (MPa)	σ_b (MPa)	ε (%)	$\sigma_{0.2}$ (MPa)	σ_b (MPa)	ε (%)	$\sigma_{0.2}$ (MPa)	σ_b (MPa)	ε (%)
0	304_{-6}^{+8}	463_{-7}^{+8}	$17.1_{-1.3}^{+1.2}$	260_{-5}^{+6}	310_{-5}^{+3}	$18.1_{-0.7}^{+1.2}$	205_{-4}^{+5}	240_{-5}^{+4}	$19.2_{-0.8}^{+1.1}$
1	351_{-6}^{+12}	482_{-9}^{+15}	$14.4_{-1.2}^{+1.3}$	280_{-5}^{+13}	325_{-7}^{+16}	$16.2_{-0.8}^{+0.7}$	210_{-6}^{+9}	249_{-5}^{+9}	$17.9_{-0.6}^{+0.8}$
2	373_{-10}^{+13}	527_{-15}^{+14}	$11.5_{-0.7}^{+0.8}$	325_{-9}^{+7}	360_{-15}^{+6}	$14.9_{-0.7}^{+0.7}$	235_{-7}^{+8}	269_{-6}^{+7}	$17.1_{-0.5}^{+0.8}$
3	375_{-8}^{+6}	533_{-11}^{+9}	$7.8_{-0.5}^{+0.7}$	335_{-6}^{+7}	369_{-6}^{+7}	$10.3_{-0.6}^{+0.5}$	243_{-6}^{+8}	275_{-5}^{+6}	$13.2_{-0.7}^{+0.5}$

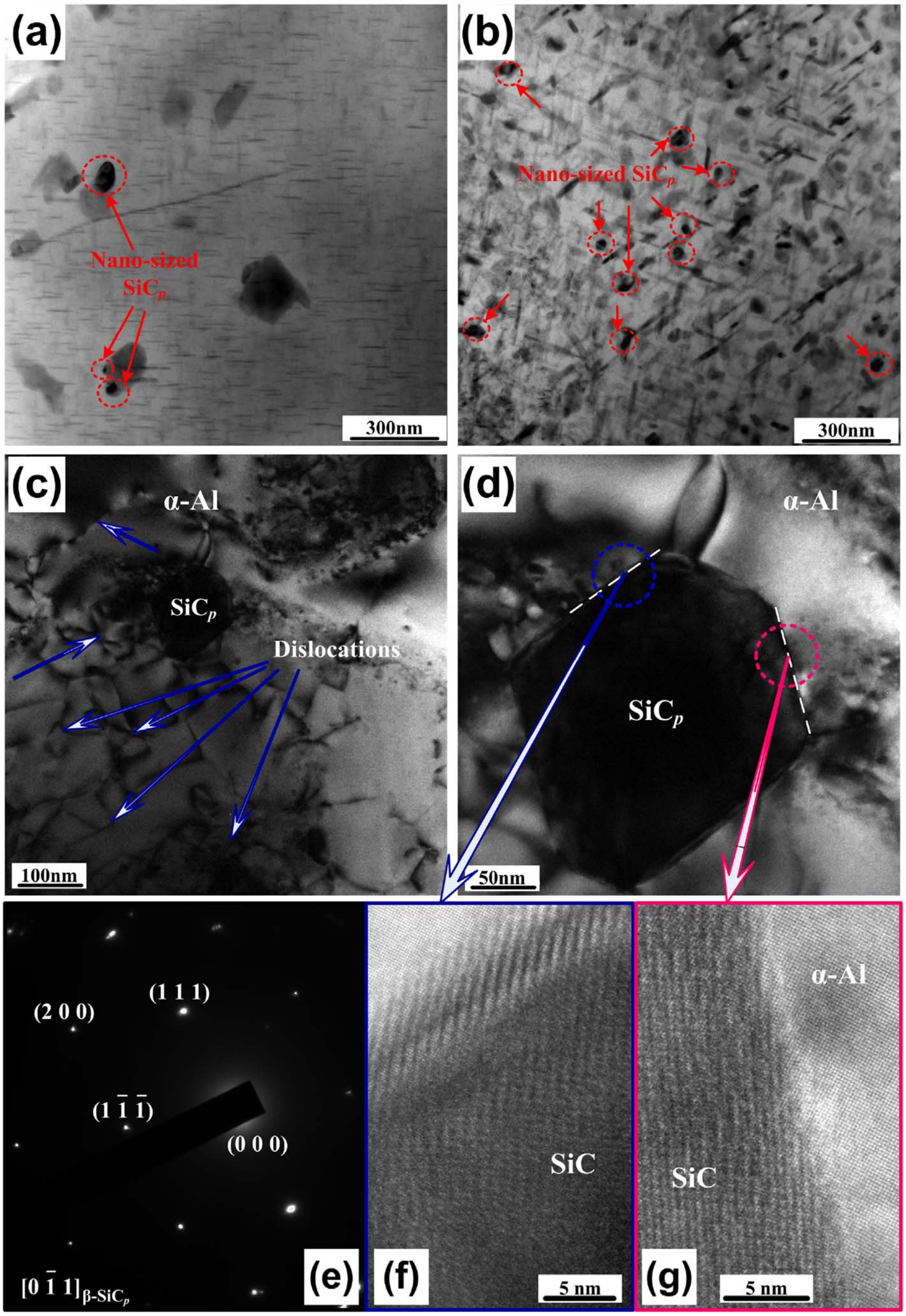

Fig. 5. TEM images show the distribution of nano-sized SiC_p in (a) 1 wt% and (b) 3 wt% nano-sized SiC_p/Al-Cu composite; (c) TEM micrographs of the 3 wt% nano-sized SiC_p/Al-Cu composite, (d) a higher magnification shows nano-sized SiC_p particle, (e) the corresponding SAED patterns obtained from black areas in (c), (f) and (g) HRFEM images of the interface between nano-sized SiC and α-Al matrix.

indicates that nano-sized SiC_p particle is perfectly coherent with α-Al. Intermediate phase has not been detected at the particle-matrix interface, indicating that reaction between nano-sized SiC_p and Al does not occur. The results imply that there is a strong interfacial strength between nano-sized SiC_p and α-Al matrix.

The significant improvement of the strength of nano-sized SiC_p/Al-Cu composites can be mainly attributed to the refined α-Al dendrites, the hindering of dislocations motion by nano-sized SiC_p particles, and the good interface combination as well as the suppression of the interfacial reactions. First, the grain boundary usually plays important roles as the barrier for the dislocations motion. Therefore, the increase in the grain boundary concentration is beneficial to improve the tensile strength. Moreover, the Hall-Petch relationship: $\sigma = \sigma_0 + k \cdot d^{-1/2}$ (where σ is the yield stress, σ_0 and k are the constants depending on material itself, and d is the average grain size) indicates that the yield strength increases with a smaller grain size of the matrix [21]. The refined α-Al dendrites will result in the increasing amount of matrix dendrite boundaries. The finer the dendrite is, the more tortuous the grain boundaries will be. Therefore, the crack propagation becomes more and more difficult and the composites can endure the large plastic deformation before fracture. Secondly, Orowan strengthening, caused by the interaction between dislocations and well-dispersed nano-sized SiC_p particles in α-Al matrix, is a strengthening mechanism significantly existed in nano-sized particles reinforced Al matrix composites. These dislocations are pinned down and accumulation inside the grains can lead to the enhancement of work hardening and fracture strain markedly, which have been proved by Liu et al. [22]. It is the main strengthening mechanism of the described composites under high temperature conditions, while the strengthening effect of grain boundaries at high temperature is weakened remarkably [23,24].

Finally, the reaction between the molten Al and SiC_p will produce Al_4C_3 which is a brittle and unstable phase. The presence of Al_4C_3

degrades the mechanical properties through crack propagation. In the present work, low sintering temperature (873 K) and short time stirring dispersion can suppress the interfacial reaction effectively, which is helpful to restrict the formation of the Al_4C_3 phase, as shown in Fig. 5. The strength and cracking resistance of the interface bonding are improved, which makes the occurrence of the cracks more difficult. With the content of nano-sized SiC_p increasing, more and more particles, which have not been captured by the solid-liquid interface, will gather at the interdendrite and the boundary of α-Al dendrite, which leads to more agglomeration of nano-sized SiC_p and higher concentration of the defects [14]. The cracks tend to propagate from those zones during the plastic deformation. In addition, more severe agglomeration of nano-sized SiC_p could lead to some particles not be completely wrapped up by the matrix. Those mentioned above will decrease the strengthening effect of nano-sized SiC_p particles in the Al-Cu composite. In a word, a combined interaction of the refined α-Al dendrites, the pinned dislocations motion by nano-sized SiC_p particles and good particle-interface results in the enhancement of the composites.

4. Conclusions

Nano-sized SiC_p/Al-Cu composites containing 1, 2, 3 wt% nano-sized SiC_p particles were successfully prepared by dispersing the nano-sized SiC_p/Al master alloy into molten Al-Cu alloy through stir casting assisted with hot extrusion. The α-Al dendrites in as-cast Al-Cu composites containing nano-sized SiC_p particles were strongly refined, of which the shape of α-Al changed from dendritic crystal with sizes about 200 μm to equiaxial crystal with much finer sizes about 75 μm. After hot extrusion process, the α-Al grains were further refined to approximately 28 μm for 3 wt% nano-sized SiC_p/Al-Cu composites. Meanwhile, the tensile strength of the present composites was improved when the contents of nano-sized SiC_p particles were arranged from 1 wt% to 3 wt %. Among them, the Al-Cu composites having 2 wt% nano-sized SiC_p displayed the best tensile properties, i.e., σ_s = 373 MPa, σ_b = 527 MPa and ε = 11.5%, σ_s = 325 MPa, σ_b = 360 MPa and ε = 14.9%, σ_s = 235 MPa, σ_b = 269 MPa and ε = 17.1% at 298 K, 453 K and 493 K, correspondingly. A combined strengthening effect of nano-sized SiC_p particles, the refined α-Al dendrites, good interface combination and suppression of interfacial reaction resulted in the significant improvement of the tensile strength.

Acknowledgments

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