MICROSTRUCTURE STUDY ON AL-5%SiC NANOCOMPOSITE POWDERS

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Abstract: *In this study Al-5 (Vol) % SiC_p nanocomposite powder has been successfully synthesized by high-energy planetary milling of Al and SiC powders for a period of 25 h at a ball-to-powder ratio of 15:1. The changes of the lattice strain, the crystallite size of the matrix phase, and the nanocomposite powder microstructure with time have been investigated by X-ray diffraction (XRD), X-ray mapping, and scanning electron microscopy (SEM) analyses. The morphologies of the nanocomposite powders obtained after 25 h of milling have also been studied by transmission electron microscopy (TEM). The results showed that nanocomposite powders were composed of near-spherical particles and, moreover, the SiC particles were uniformly distributed in the aluminum matrix.*

Keywords: *Microstructure evolution, Al-SiC, nanocomposite, mechanical milling.*

1. INTRODUCTION

Metal matrix composites (MMCp) are engineering materials in which a hard ceramic component is dispersed in a ductile metal matrix in order to obtain characteristics that are superior to those of conventional monolithic metallic alloys [1- 5]. Among these materials, aluminum-based metal matrix composites (MMCs) are appropriate materials for structural applications in the aircraft and automotive industries because they are lightweight and have a high strength-to-weight ratio [6-12]. Uniform dispersion of the fine reinforcements and a fine-grained matrix improve the mechanical properties of the composite. Incorporation of ceramic particulates into the metallic matrix can be accomplished by several techniques, such as molten-metal routes or solid-state processing [13, 14]. It should be noted that Al-SiC composites are difficult to obtain by conventional melting-based methods due to the poor wettability between molten Al and the SiC. In addition, these methods usually lead to an undesirable reaction between the SiC and molten Al, which produces brittle phases of Al_4C_3 and Si [15, 16]. Mechanical alloying (MA), as originally developed by Benjamin, has been widely utilized to produce metal or ceramic-based composite powders with fine microstructures. The mechanical alloying process involves repeated welding and fracturing of a mixture of powder particles to produce an extremely fine microstructure [17- 27]. Recently, several research articles have focused on the production of composite materials, the effect of milling time on the mechanical properties of the products, and optimization of the milling parameters using the mechanical alloying method [28- 31]. As the milling time has considerable effect on morphology and other properties of the mechanically milled powder, this study was designed and undertaken to consider the structural evolution of nanocomposite powders during the course of milling. The current paper presents experimental results relating to the effect of milling time on the morphology, crystallite size, lattice strain, distribution of reinforcement phase, and microstructure of Al-SiC composite powders.

2. EXPERIMENTAL PROCEDURE

Aluminum (purity 99.9%) and SiC (purity >99.5%) powders were mixed to give a nominal composition of Al-5%(Vol)SiC. To avoid agglomeration and the cold welding of powder particles, stearic acid amounting to 1.5 wt% of the total powder charge was used as a process control agent. The preparation of the nanocomposite powders by mechanical milling

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Table 1. Crystallite sizes and lattice strains of Al-5%SiC milled powders after 5, 10, 15, and 25 h of milling.

Milling time (h)	Average crystallite size (nm)	Maximum lattice strain (%)
5	71	0.25
10	62	0.33
15	54	0.38
25	45	0.41

of the mixed powder was carried out using a high-energy planetary ball mill with a hardened stainless steel vial and 12 mm diameter hardened steel balls in an argon atmosphere. A rotational speed of 300 rpm and a ball-to-powder weight ratio of 15:1 were employed. In order to avoid oxidation of powders during the MA treatment, all handling was performed in a glove box in an argon atmosphere. Small amounts of the milled powders were collected for microstructure analysis at different stages during the MA process. A Philips XL-30 scanning electron microscope (SEM) coupled with an energy-dispersive spectrometer (EDS) for compositional analysis and a Philips CM200 FEG transmission electron microscope (TEM) were used to characterize the microstructures of the milled powders. X-ray investigations were performed using a Philips PW-1730 diffractometer with Cu-K_α radiation. To determine the crystallite size d and the lattice strain ε, the Williamson–Hall method was adopted. X-ray mapping was also carried out to observe the distribution of the different elements in the milled powders.

3. RESULTS AND DISCUSSION

3. 1. XRD Analysis

Figs. 1 (a)–(d) show the XRD patterns of the Al-5%SiC composite powders after 5, 10, 15, and 25 h of milling time. There was no formation of any other phase, but the X-ray diffraction patterns display increasing broadening of the crystalline peaks as a function of milling time. The XRD peaks were analyzed using the Williamson–Hall method [16] and the results are reported in Table 1. Of significance here is that as the milling time was increased, work hardening rate and matrix deformation were enhanced, and so the lattice strain increased and the average size of the aluminum phase crystallites decreased [16, 17]. Table 1 shows that the Al-5%SiC composite powders acquired the smallest crystallite size (45 nm) with the highest lattice strain (0.41%) after 25 h of milling. Furthermore, it is evident that mechanical milling led to a faster rate of change of the average grain size and lattice strain in the early stage of the MA process as compared to the later stages. A slight shift in the position of the

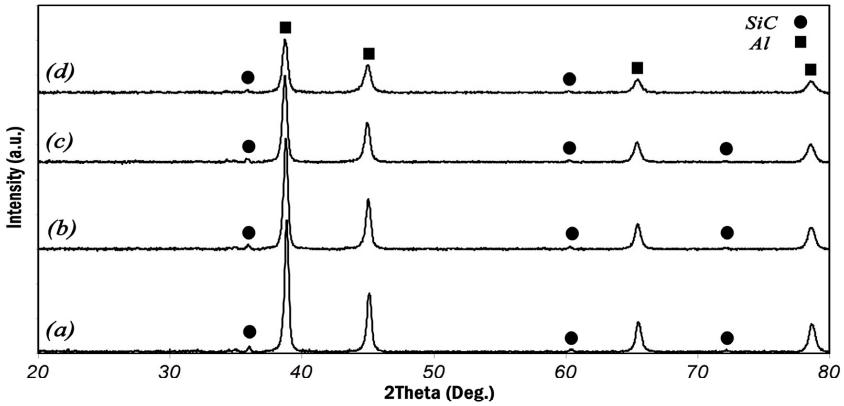

Fig. 1. X-ray diffraction patterns of Al-5%SiC milled powders after (a) 5 h, (b) 10 h, (c) 15 h, and (d) 25 h of milling time.

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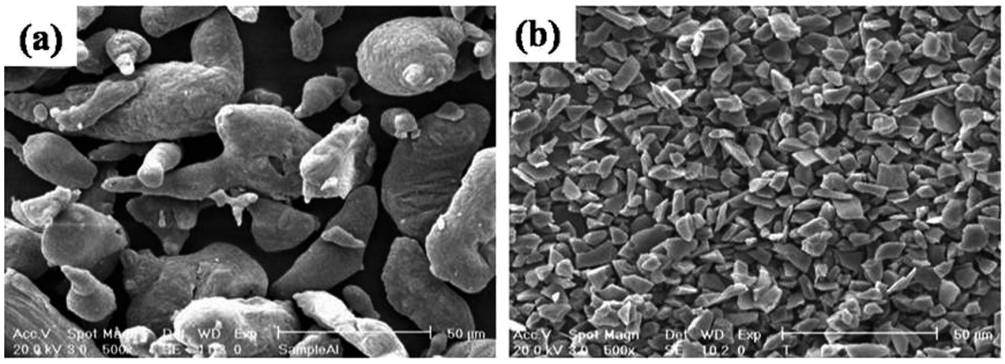

Fig. 2. Primary powders used: (a) Aluminum, (b) SiC

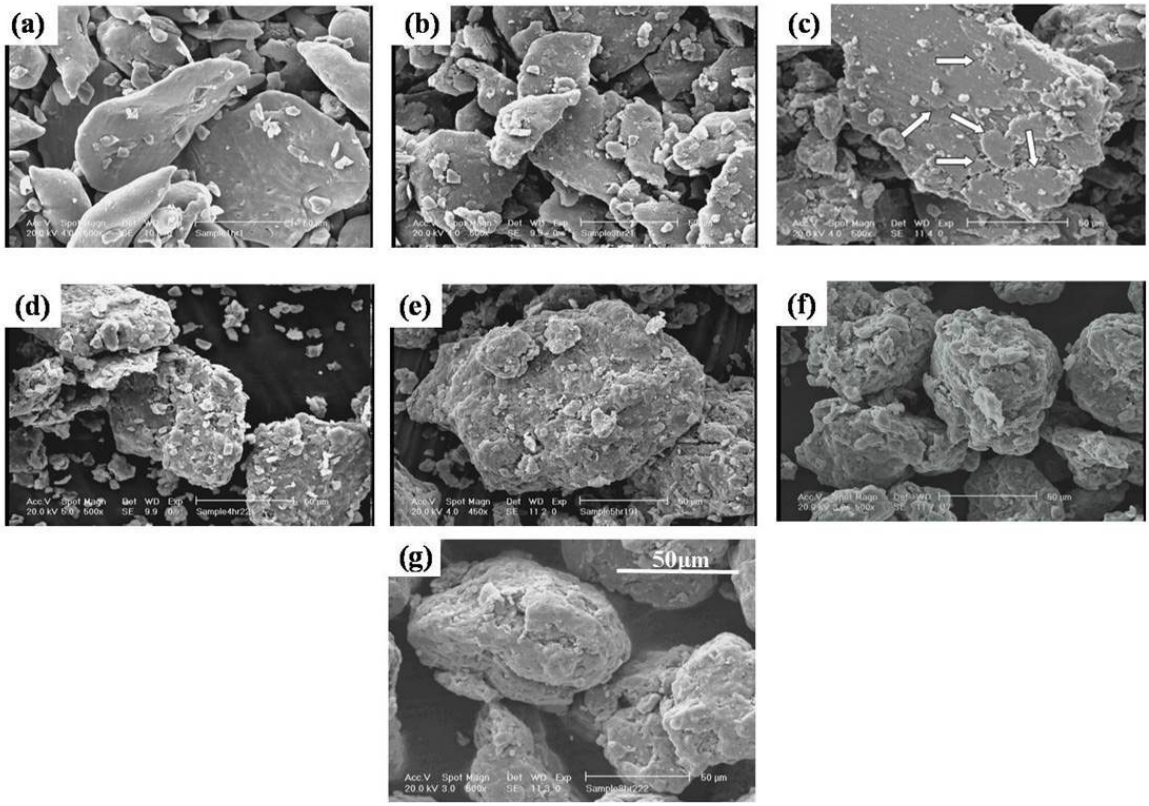

Fig. 3. SEM morphologies of Al–5% SiC after (a) 1 h, (b) 2 h, (c) 5 h, (d) 10 h, (e) 15 h, (f) 20 h, and (g) 25 h of ball-milling time.

XRD peaks was also noticed and could be related to the dissolution of impurities, mostly iron, in the lattice of the matrix phase [16].

3. 2. Microstructural Observations

Fig. 2 shows the particle morphologies of the Al and SiC initial powders. Fig. 3 shows the effect of milling time on the morphology of the Al/5%SiC powders, while Fig. 4 depicts X-ray map results showing the distribution of the elements Al and Si in the nanocomposite powders milled for 2 h and 5 h.

From inspection of these figures, it is clear that the powders obtained after the short milling time were bulky with random shape and size and the distribution of the reinforcing particle was not uniform (Fig. 4(a)). Also, the distance between

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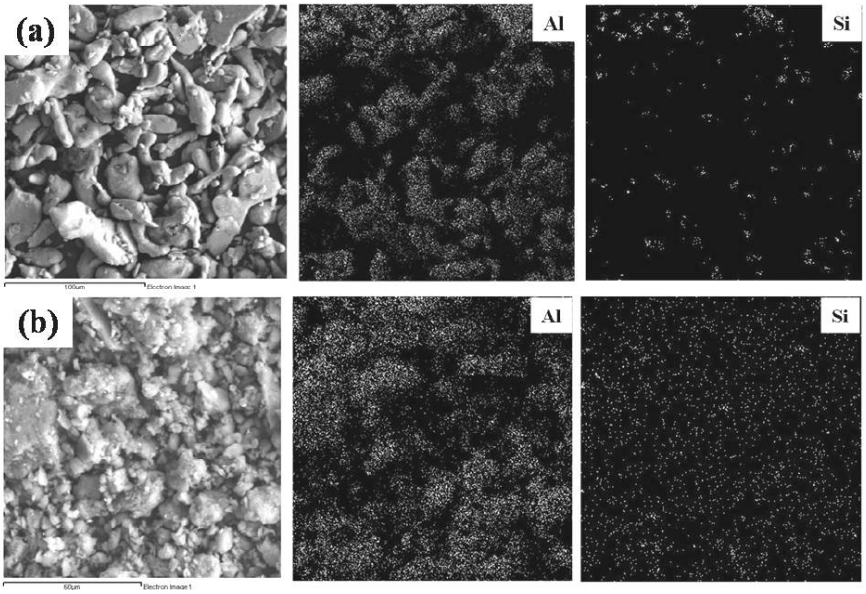

Fig. 4. X-ray maps showing the distributions of the elements Al and Si for the Al–5%SiC powders milled for (a) 2 h and (b) 5 h.

SiC particles was rather large (Fig. 3(a)). It can be seen from Figs. 3(b), (c) that the aluminum particles were first flattened by the cold working and shear forces generated by the ball-powder-ball collisions in the milling medium. Detailed inspection of the particle surface microstructure after 5 h of milling revealed the presence of SiC uniformly distributed on the surfaces of the flakes. Moreover, it is evident from Fig. 4 (b) that the dispersal of SiC in the aluminum matrix was greatly improved after 5 h of milling. Fig. 5 shows a broken SiC particle embedded in an aluminum matrix after 5 h of milling. Indentation of the hard reinforcing phase into the ductile matrix (Fig. 5) led to an increase of work hardening and therefore the increase of fracture rate of Aluminum matrix. As a result, the aluminum particles started to laminate and became finer. With increasing milling time, new flake surfaces were created and the particles welded together and agglomerated to form larger powders. Agglomerated powders have been shown and marked with arrows in Fig (3c). This occurred because of the tendency for decreasing surface free energy. On the other hand, increasing the MA time increases the hardness and this leads to fracturing of the agglomerated powders to form smaller particles [17]. However, there are differences in average particle size when different milling times are used, which indicates that milling time and other processing phenomena, such as competition between fracturing and agglomeration rate, have an influence on the morphology and size of the agglomerated powders (Figs. 3 (d)–(f)). It is clear that the increased milling time resulted in refining the SiC particles and decreasing the distance between them. Also, the aluminum particles were laminated and the broken SiC

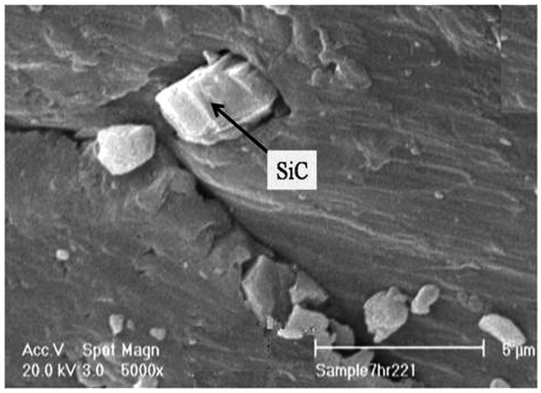

Fig. 5. SiC powder embedded within an aluminum matrix after 5 h of milling.

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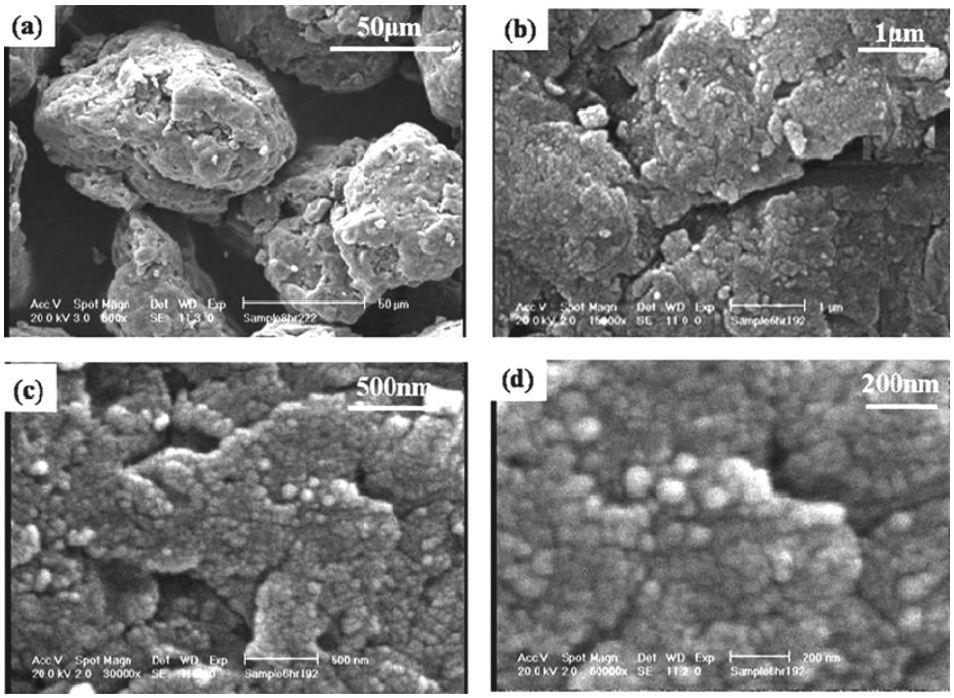

Fig. 6. SEM morphologies of Al–5% SiC after 25 h of ball-milling at different magnifications.

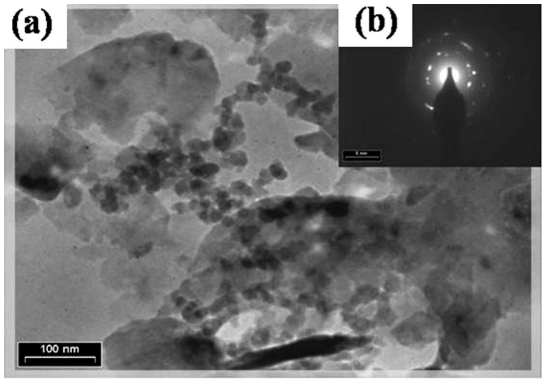

Fig. 7. (a) Bright-field image (BFI) and (b) selected-area diffraction pattern (SADP) of Al-5%SiC nanocomposite powder obtained after 25 h of ball-milling.

particles became embedded within the aluminum matrix lamina. Fracturing and agglomerating continued throughout the milling time competitively. Finally, a near-spherical nanostructured composite powder with a uniformly distributed reinforcing phase in a laminated aluminum matrix was obtained when fracturing and cold welding were balanced (Fig. 3(g)). The final powder particles were well-rounded and composed of a multitude of layers.

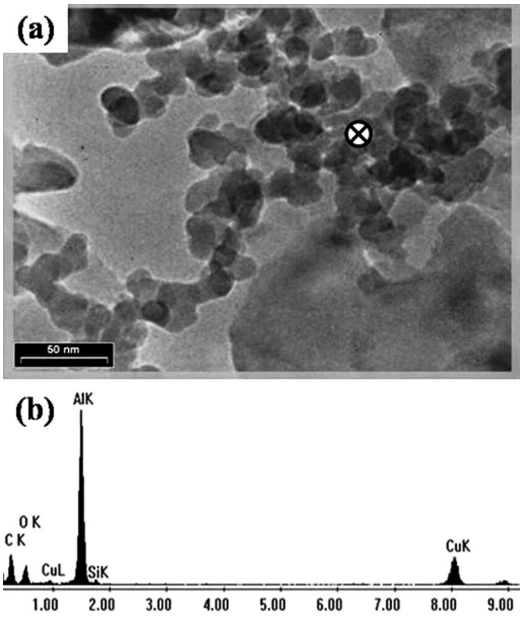

Fig. 8. (a) BFI of Al-5%SiC nanocomposite powder obtained after 25 h of ball-milling and (b) EDS analysis of the region marked in (a).

Fig. 6 shows the microstructure of the agglomerated spherical nanocomposite powder obtained after 25 h of MA (Fig. 3(g)) at different

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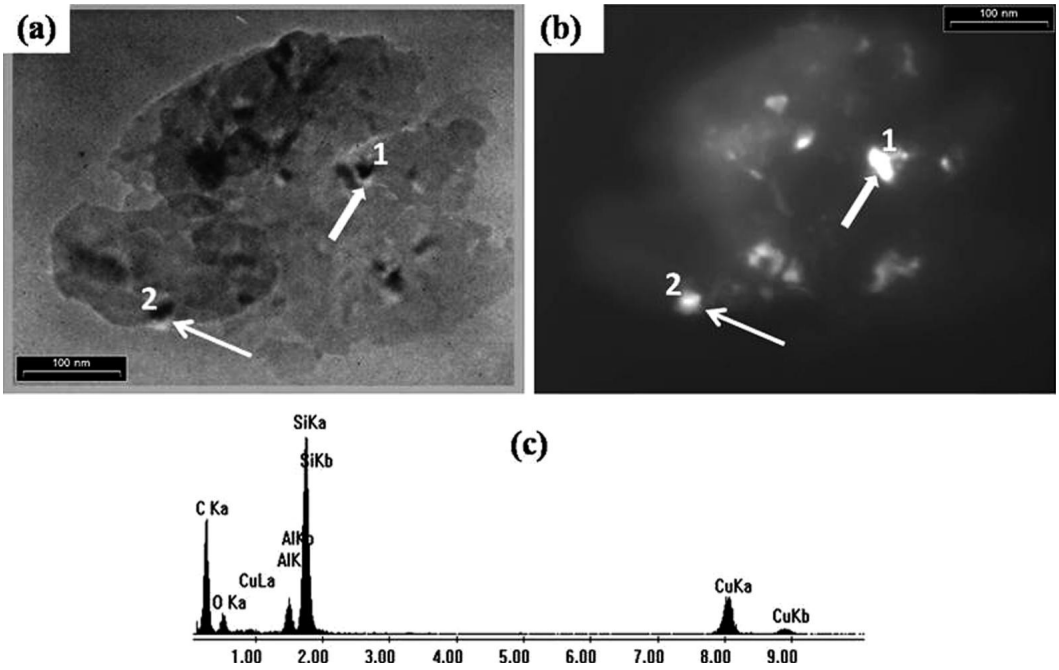

Fig. 9. (a) Bright-field image and (b) dark-field image of Al-5%SiC layered-structure powder obtained after 25 h of ball-milling, and (c) EDS analysis of the region marked by (1) in (a).

magnifications. It is obvious that the agglomerated powder included much laminate (Figs. 6(b), (c)) and contained embedded spherical nanoparticles (Fig. 6(d)).

Transmission electron microscopy (TEM) allowed a better understanding of the structure of the Al-5%SiC nanocomposite. Figs. 7 (a) and (b) show a bright-field image (BFI) and a selected-area diffraction pattern (SADP), respectively, of the nanocomposite powder obtained after 25 h of ball-milling. The bright-field image indicates that there are some nearly spherical nanoparticles and some layered structures of greater size (Fig. 7 (a)). The SADP shows a reasonably sharp ring?spot pattern, which is characteristic of diffracting polycrystalline components (Fig. 7 (b)).

Fig. 8 shows that the nearly spherical particles were approximately 5–20 nm in diameter. EDS analysis of the region marked in Fig. 8 (a) proved the existence of Al and SiC components. Bright- and dark-field images in conjunction with EDS analysis of 1, 2 points proved that the SiC nanoparticles, the numbered points, were appropriately distributed in the laminated matrix after 25 h of milling, as shown in Fig. 9. Dissimilar brightnesses of different points in the dark-field image (Fig. 9 (b)) can be attributed to different orientations and diverse crystallite structures of SiC nanoparticles.

4. CONCLUSIONS

- By using mechanical milling, nanometer-sized SiC particle reinforced Al–SiC composite powder can be synthesized.
- Progressive milling from 0 to 25 h changes the morphology of the powder particles from flaky to near-spherical.
- Morphological study has revealed that the agglomerated powders consist of many layers and that they contain embedded spherical nanoparticles.
- X-ray mapping and TEM analyses have indicated that the SiC nanoparticles are appropriately distributed in the metal matrix.
- It has been established that with increasing milling time, the lattice strain increases and the average size of the aluminum phase crystallites decreases. Moreover,

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mechanical milling improves the distribution of SiC throughout the aluminum matrix, thus enhancing the characteristics of the composite.

5. ACKNOWLEDGEMENTS

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