Journal of Alloys and Compounds 509 (2011) 2221–2226

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: www.elsevier.com/locate/jallcom

Structural and morphological evaluation of Al–5 vol.%SiC nanocomposite powder produced by mechanical milling

S.A. Khadem*, S. Nategh, H. Yoozbashizadeh

Department of Materials Engineering, Science and Research Branch, Islamic Azad University (IAU), Tehran, Iran

ARTICLE INFO

Article history:
Received 22 July 2010
Received in revised form 28 October 2010
Accepted 29 October 2010
Available online 10 November 2010

Keywords:
Mechanical milling
Nanostructured materials
Al–SiC nanocomposite powder

ABSTRACT

Al–5 vol.%SiC nanocomposite powder is produced by using attrition milling in this investigation. The particle size distribution has been determined based on the standard deviation and the coefficient of variation. The results show that the addition of hard SiC particles accelerates the milling process, leading to faster work hardening rate and fracture of the aluminum matrix. Furthermore, Al becomes smaller crystallite size during ball milling of Al powder in the presence of SiC particles. Finally, the scanning electron microscopy (SEM) analysis and the X-ray diffraction (XRD) experiments are used to investigate the effect of milling time on the structural changes of Al–5 vol.%SiC nanocomposite and pure aluminum powder.

1. Introduction

Nowadays, there are potential applications for nanocomposite materials due to superior mechanical properties (strength and ductility with attendant workability) of them in comparison with monolithic materials. However, the method of producing and amount of components are vital challenges of researchers in this field. In recent years, nanocomposite materials (when the reinforcement is of nanometer dimensions, typically <100 nm in size) have received the serious attention of researchers in view of their much better properties than those of either the monolithic material or the composite containing a coarse-grained reinforcement phase [1]. Aluminum alloys have reasonable strength and ductility with suitable workability. Therefore, these alloys have been widely used for synthesizing of nano-composites with different nano-sized ceramic particulates such as Al_2O_3 [2]. Aluminum-based metal matrix composites (MMCs) are ideal materials for structural applications in the aircraft and automotive industries due to their light weight and high strength-to weight ratio [3]. Ceramic nano-particles have received great attention owing to their property advantages over conventional coarse grained counterparts [2]. Among various reinforcements, SiC is one of the most widely used dispersoids in Al-based composites [4–9]. There are some publications about the production of this type of composite using the mechanical milling method. Different materials and methods have been used in this research in comparison with similar studies. Parvin et al. [4], El-Eskandarany [7], Shokuhfar and Dashtbayazi [8] and Kamrani et al. [9] successfully incorporated microscale SiC particulates in pure Al matrix using the mechanical milling process. However, there are only few works on the effect of the addition of nanometric particulates on the structural and morphological behavior of Al powders.

Thus, in this research pure Al nanostructure and Al–5 vol.%SiC nanocomposite powder is produced by mechanical milling. A statistical method is used to estimate the particles size distribution and steady state condition of milling.

2. Experimental procedures

High purity aluminum powder with mean particles size of 20 μm produced by nitrogen gas atomization and β-SiC powders with particles size of 25–55 nm were also used as the starting materials (Table 1). Fig. 1 shows SEM micrograph of the aluminum powder with spherical particles shape. Fig. 2 shows TEM micrograph of nano-size SiC particles. 2 wt.% of stearic acid as a process control agent (surfactant) was added to the powder mixture to control the particles size of composite powders during the milling process.

The milling was performed under high purity argon gas atmosphere in an attrition mill using a hardened stainless steel vial and hardened steel balls of 5–6 mm in diameter. The ratio of balls to powder weight and rotational speed were 20:1 and 350 rpm, respectively. The powders produced after different stages of milling were examined using a Leo440i scanning electron microscope (SEM) operating at a voltage of 15 kV.

The size distribution of the milled powders was quantified by visual basic software using several SEM images and their morphology was characterized by scanning electron microscopy. X-ray diffraction (XRD) patterns of powders were taken in air atmosphere using a Store (model stadi mp) X-ray diffractometer (30 kV and 25 mA) with Cu Kα radiation (λ = 0.15406 nm). The grain size of milled aluminum powders was estimated by XRD peak broadening using William–Hall method as follows [10]:

$$B\cos\theta = \frac{0.9\lambda}{D} + 2\eta\sin\theta \quad (1)$$

where B, λ, θ, D and η are full width at half maximum (FWHM), the wave length, peak position, crystallite size and lattice strain, respectively.

* Corresponding author. Tel.: +98 9126351075.
E-mail address: khadem@iau-saveh.ac.ir (S.A. Khadem).

0925-8388/$ – see front matter © 2010 Elsevier B.V. All rights reserved.
doi:10.1016/j.jallcom.2010.10.188

Table 1
Specification of nano size SiC particle.

Properties	Purity	Dissociate silicon content	Oxygen content	Crystallographic form	Average particle size	Specific surface area	Apparent density	True density	Morphology	Color
SiC	≥99.0%	≤0.20%	≤0.61%	Cubic	≤50 nm	≥90 m^2/g	0.05 g/cm^3	3.22 g/cm^3	Nearly spherical	Green

The coefficient of variation and standard deviation from the average particles size was used as a criterion for the estimation of particles size distribution. A low standard deviation indicates that the data points tend to be very close to the mean, whereas high standard deviation indicates that the data are spread out over a large range of values.

In the case where $x_1, x_2, \ldots, x_i, \ldots, x_N$ are a set of N data, with each value having the same probability and μ is mean value of these data, the standard deviation is:

$$\sigma = \sqrt{\frac{1}{N}\sum_{i=1}^{n}(\chi_i - \mu)^2} \quad (2)$$

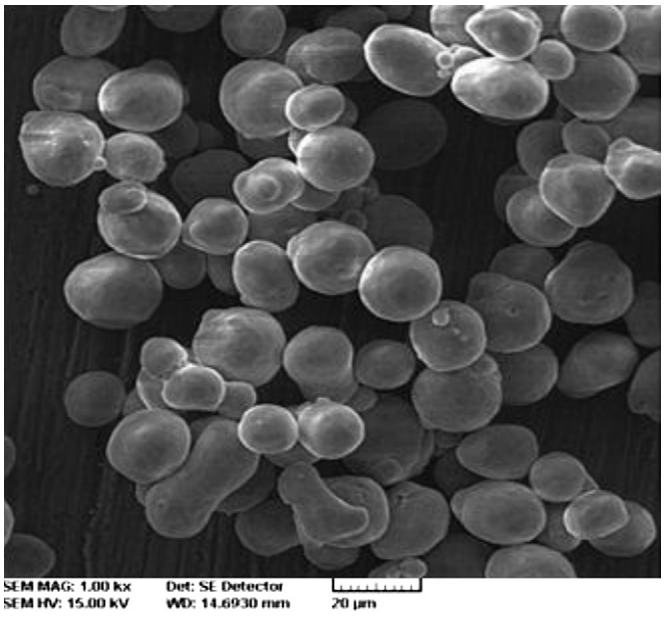

Fig. 1. SEM micrograph of microscaled pure aluminum powder.

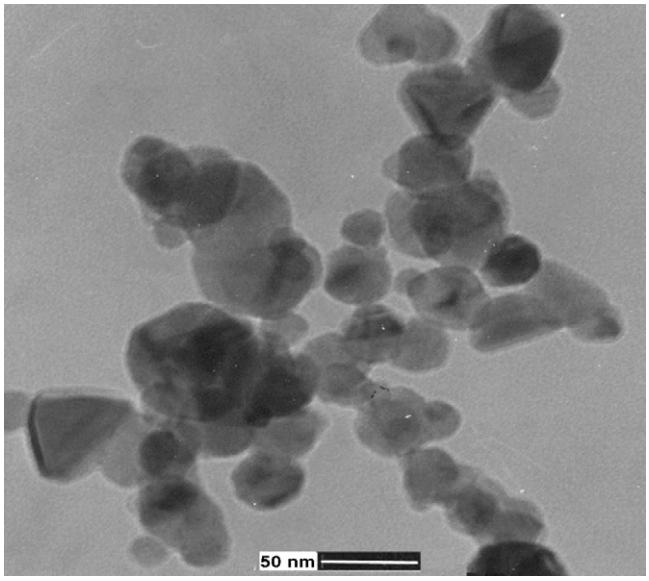

Fig. 2. TEM micrograph of nanoscaled SiC nano particle.

In probability theory and statistics, the coefficient of variation (c_v) is a normalized measure of dispersion of a probability distribution. It is defined as the ratio of the standard deviation σ to the mean μ.

This is only defined for non-zero mean, and is most useful for variables that are always positive. It is also known as unitized risk [11].

3. Results and discussion

3.1. Particles size distribution

Fig. 3 shows the variation of average particles size versus milling time for pure aluminum and Al–5 vol.%SiC powder mixture. It is observed that for pure aluminum a maximum average particles size is obtained after in around 8 h of milling time. But for Al–5 vol.%SiC powder mixture samples the trend shows a decrease in particles size as milling time increased. The effect of milling time on the particles size of ductile powders has been studied in the case of pure and composite powders [12–15]. In all cases a similar trend in powder particle size was observed, i.e. an initial increase in followed by a decrease and then steady state. This behavior can be attributed to the cold welding of initial ductile particles followed by work hardening and thus the fracturing of powder particles. When the rate of cold welding and fracturing processes equals, the steady state is achieved. There is a maximum value in the particles size of pure aluminum after 7 h of milling approximately. The steady state condition is achieved between 25 and 35 h as shown in Fig. 3. The addition of 5 vol.%SiC has a strong effect on the variation of particles size of the composite powder. In this case powder particles tend to decrease continuously during ball milling due to the effect of nano sized SiC particles on the welding behavior of the aluminum particles. In comparison to micrometric particulates, extremely small nanoscale SiC particulates smears the surface of aluminum particles. Under this circumstance, matrix particles are surrounded by nanoscale SiC particles preventing cold welding to take place. This outcome is in consistent with results performed by Razavi et al. [15]. They have shown that in the case of nanocompos-

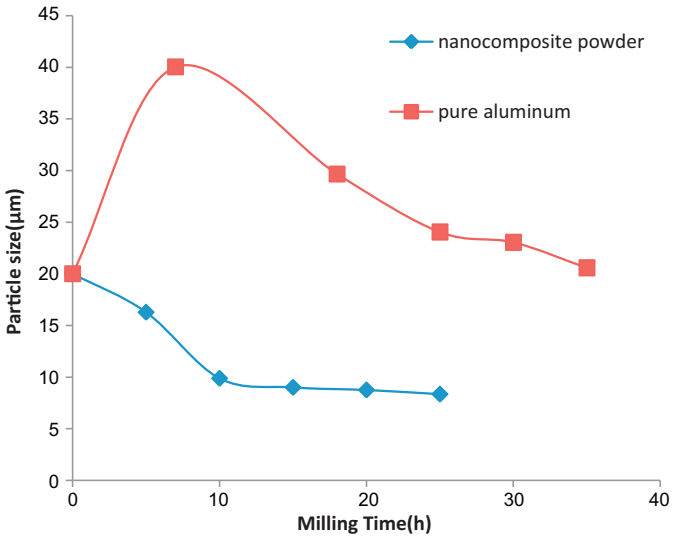

Fig. 3. Effect of milling time on the average particle size.

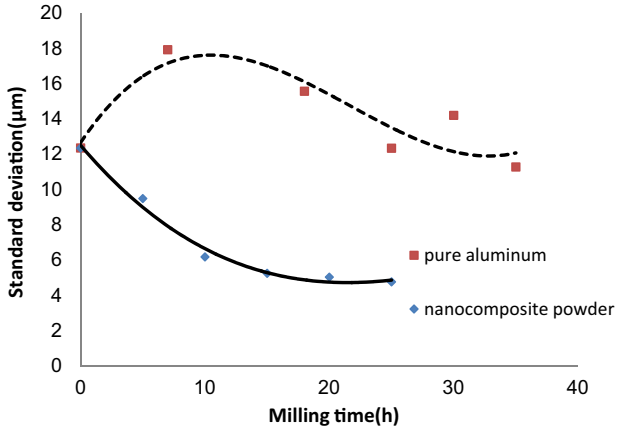

Fig. 4. Effect of milling time on the standard deviation for pure and nanocomposite powder.

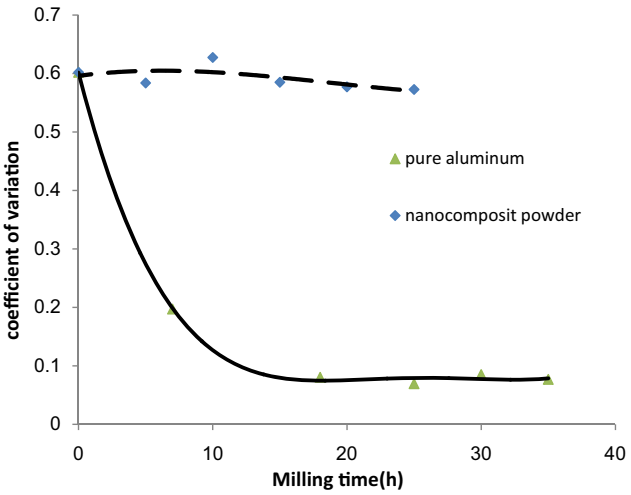

Fig. 5. Effect of milling time on the coefficient of variation for pure and nanocomposite aluminum powders.

ite powders, longer milling time is required to achieve steady-state condition compared to microcomposite powder. Another possible reason could be the local deformation of the aluminum particles in the vicinity of hard ceramic particles during the milling process [12]. The local deformation increases the work hardening rate and the hardness of the powders and decreases their cold welding.

Particles size distribution can be evaluated using standard deviation. Fig. 4 shows standard deviation for pure aluminum and Al–5 vol.%SiC powder mixture versus milling time. As can be seen the maximum is obtained after 7 h milling time for pure aluminum and after 25 h there is a little variation in the standard deviation. As seen in Fig. 4 size distribution of Al–5 vol.%SiC powder mixture decreases with an increase in the milling time. Fig. 4 also shows that for pure aluminum the maximum variation in the distribution of particles size is observed after 7 h of milling time. This behavior

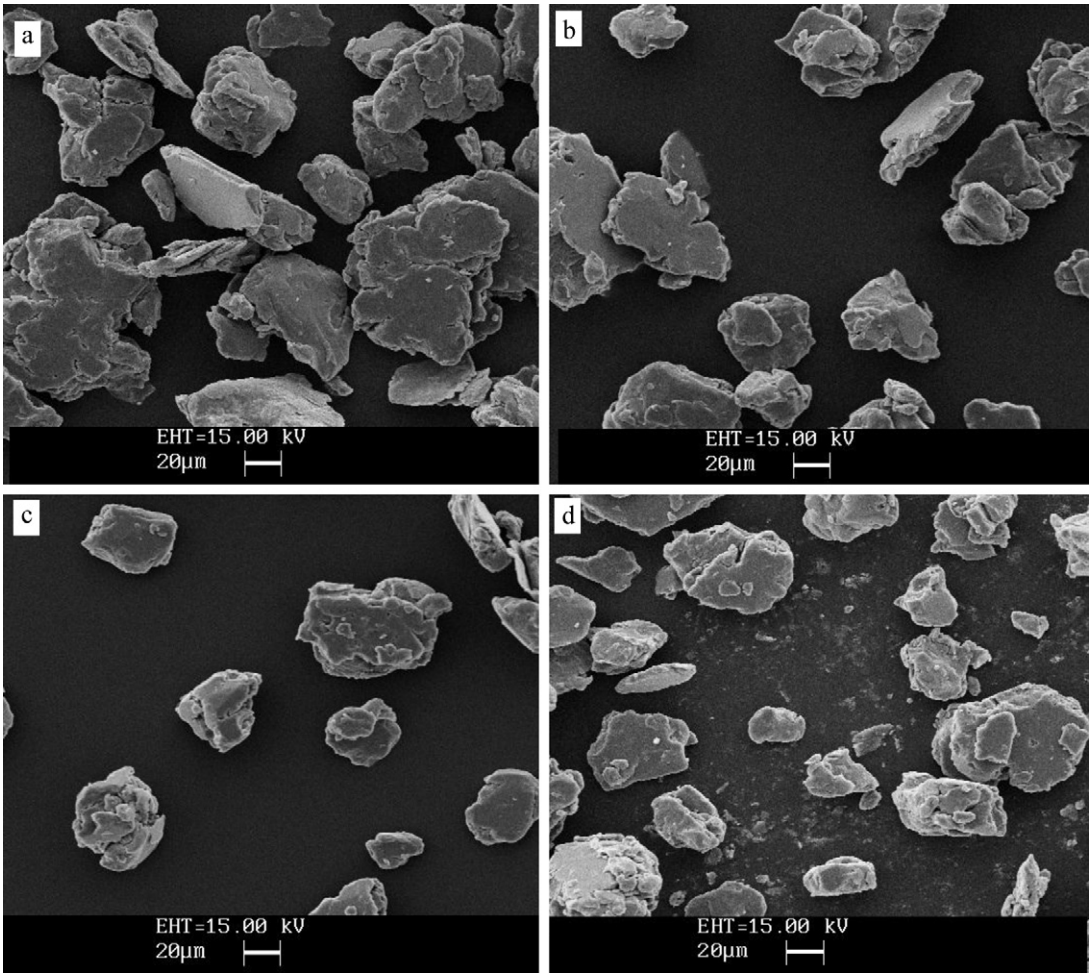

Fig. 6. SEM micrograph of pure aluminum at various milling time (h) of 7 (a), 18 (b), 25 (c), and 30 (d).

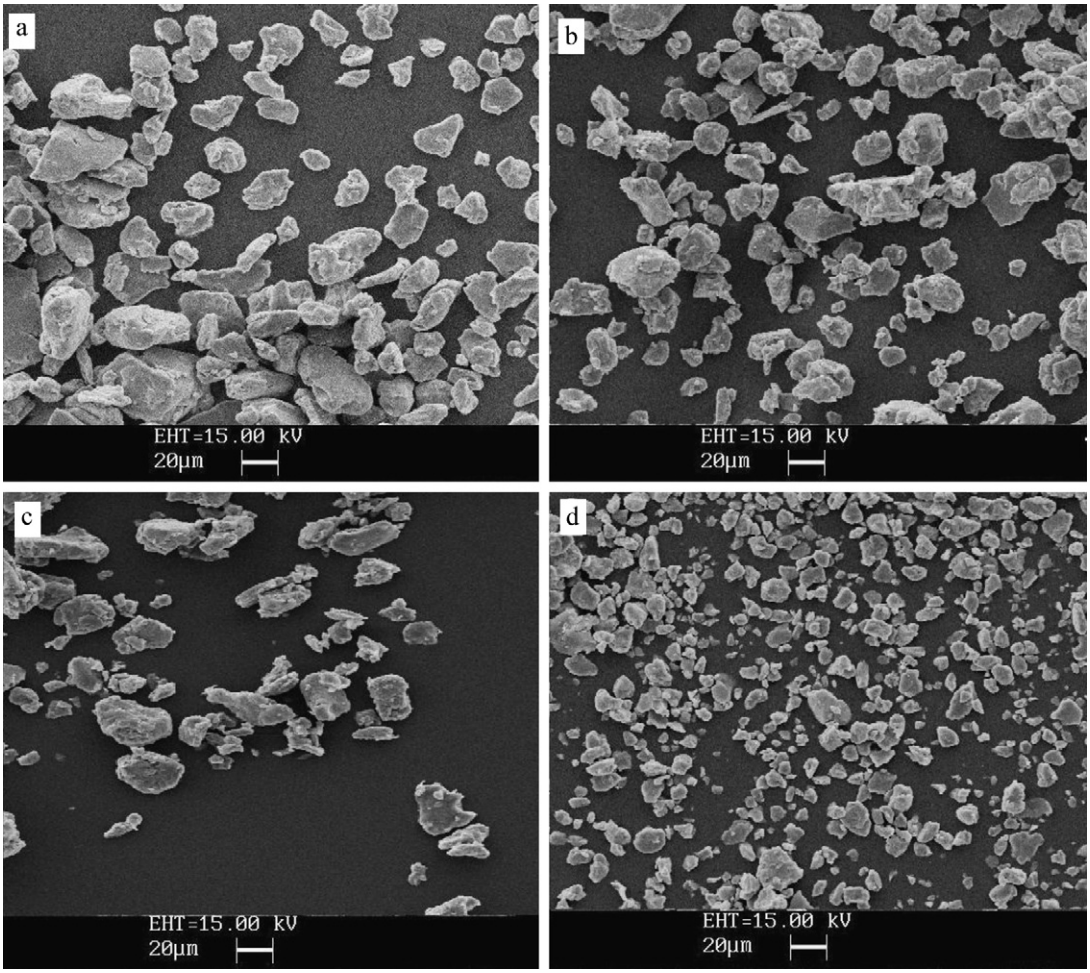

Fig. 7. SEM micrograph of Al–5 vol.%SiC (nm) at various milling time (h) of 5 (a), 10 (b), 15 (c), and 20 (d).

is attributed to the cold welding and fracturing of particles during ball milling.

Fig. 5 shows the coefficient of variation for pure aluminum and Al–5 vol.%SiC powder mixture versus milling time. It is observed that increasing milling time leads to a decrease in the coefficient of variation for pure aluminum. This variation, however, is very small for the Al–5 vol.%SiC powder mixture. It can be concluded that the variation of powder particles size in pure aluminum decreases with milling time. On the other hand milling process causes that particles size move toward a narrow range of variation and this variations are very small for nanocomposite powder and therefore variations of relatively the coefficient of variation are low for Al–5 vol.%SiC powder mixture.

3.2. Morphology

The change in morphology of powder particles was studied by SEM. As can be seen in Fig. 1, the initial Aluminum particles exhibit a spherical shape with narrow size distribution. After 7 h milling, the initial particles were deformed and a change from spherical to irregular shape was noticed (Fig. 6). The average size of particles also increased as explained above.

It seems that the cold welding is the dominant mechanism during milling due to the ductility of aluminum powders before 7 h of process. On the basis of our observations, the size of aluminum particles size is about 40 μm at this time. The aluminum particles become work hardened by increasing milling time. Accordingly, the fracture can be occurred at this time as reported by other researchers [4,7,12–14]. Fig. 4 indicates that the average particles size decreases after 18 h of milling. As seen in the previous section addition of nano sized SiC particles to Al powder had a strong effect of mechanical milling behavior. Fig. 7 shows the micrograph of Al–5 vol.%SiC nanocomposite powder at various milling time. It

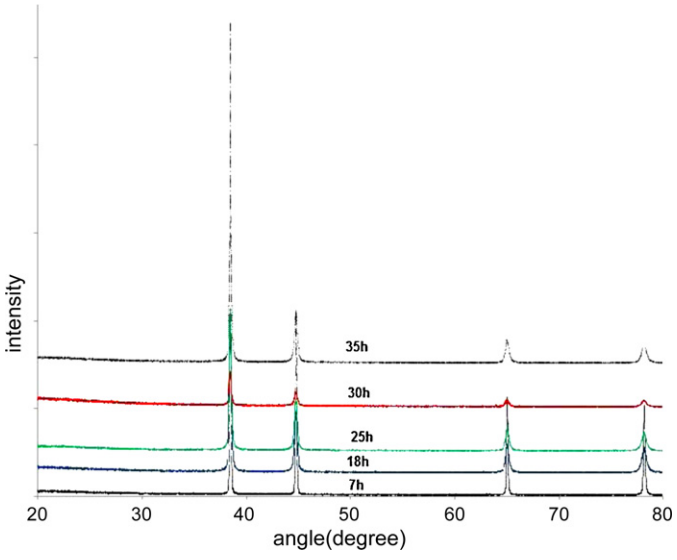

Fig. 8. XRD patterns of pure aluminum powder milled for 7, 18, 25, 30, and 35 h.

can be seen that particles shape and sizes are different from pure aluminum powder milled.

3.3. Structural evolutions

Figs. 8 and 9 show the XRD patterns of pure aluminum and Al–5 vol.%SiC powder mixture at various milling time. The XRD analysis of peaks has been carried out by the Williamson–Hall method. Fig. 10 shows that for Al–5 vol.%SiC powder mixture the average grain size is below 100 nm after 2 h of ball milling but for pure aluminum crystallite is achieved after 10 h. Mechanical milling of soft aluminum powder is accompanied by sub-micron structural changes. Severe plastic deformation of the particles can lead to grain refining, accumulation of internal stress, change of the lattice parameter, and formation of cell structure [12,13].

Fig. 11 reveals the value of latice strain versus milling time obtained by Williamson–Hall method (η in Eq. (1)). The lattice

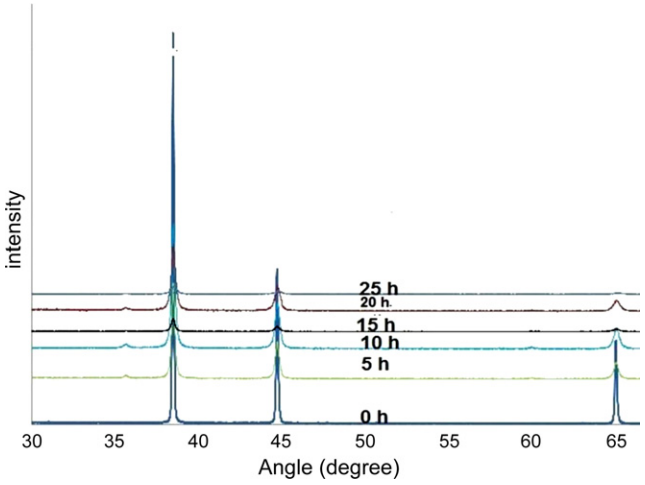

Fig. 9. XRD patterns of Al–5 vol.%SiC powder mixture milled for 0, 5, 10, 15, 20, and 25 h.

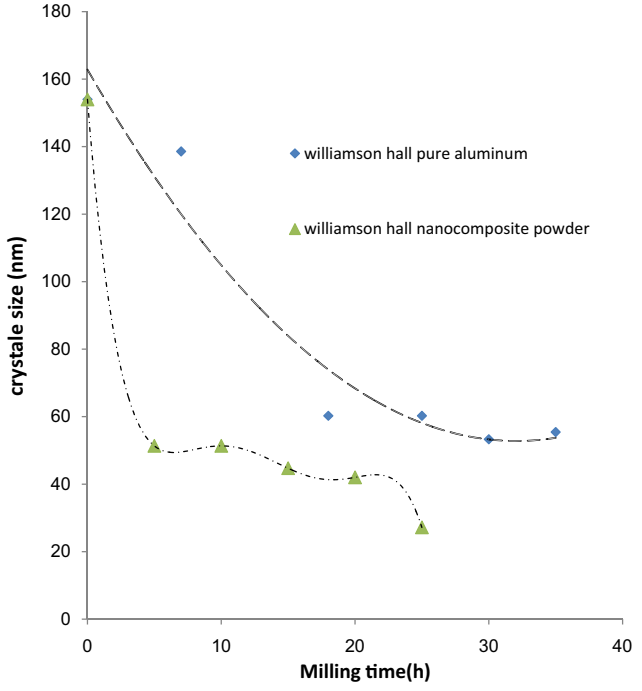

Fig. 10. Grain size of pure and nanocomposite aluminum powder versus milling time.

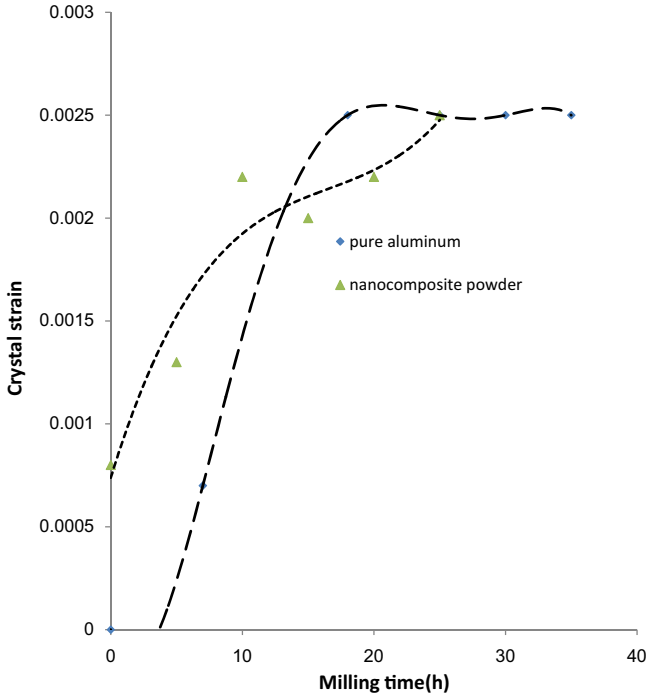

Fig. 11. Lattice strain of pure and nanocomposite aluminum powder versus milling time.

strain increases with milling time due to the lattice distortion. The effect of nano particles causes that crystal strain in nanocomposite powder increase more rapidly related to pure aluminum. The lattice strain curve has a maximum [12,13] which is ascribed to the grain size reduction and its effect on strain reduction. After a short milling time, severe plastic deformation brings about a deformed lattice with high density of dislocations. Further milling causes a nano-crystalline structure, in which dislocations reach readily to the grain boundaries, reducing the total level lattice strain [12].

4. Conclusions

It was found that the addition of nano size SiC has a great influence on the morphological characteristics powder mixture, and also work hardening of the powder so decreases the time taken to reach a steady state. This is based on the observed correlation between particles size and standard deviation versus milling time, explained by the morphological and microstructural evolution of the powder particles.

Acknowledgements

The authors would like to acknowledge the financial support of the Iranian Nanotechnology initiative (INI). The authors also would like to thank Dr. M. H. Enayati, Dr. Zakeri, Dr. Zangane, and Mr. H. Hafizpour for their ongoing help and advice.

References

[1] Suryanarayana, J. Alloys Compd. (2010), doi:10.1016/j.jallcom.2010.09.063.
[2] M. Khakbiz, F. Akhlaghi, J. Alloys Compd. 479 (2009) 334–341.
[3] B. Prabhu, C. Suryanarayana, L. An, R. Vaidyanathan, Mater. Sci. Eng. A 425 (2006) 192–200.
[4] N. Parvin, R. Assadifard, P. Safarzadeh, S. Sheibani, P. Marashi, Mater. Sci. Eng. A 492 (2008) 134–140.
[5] B. Xiong, Z. Xu, Q. Yan, B. Lu, C. Cai, J. Alloys Compd. (2010), doi:10.1016/j.jallcom.2010.09.171.
[6] Y. Saberi, S.M. Zebarjadb, G.H. Akbaria, J. Alloys Compd. 484 (2009) 637–640.

[7] M.S. El-Eskandarany, Mechanical Alloying for Fabrication of Advanced Engineering Materials, William Andrew Publishing, New York, 2000, p. 332.
[8] A. Shokuhfar, M.R. Dashtbayazi, Mater. Sci. Forum 553 (2007) 257–265.
[9] S. Kamrani, R. Riedel, S.M. Seyed Reihani, H.J. Kleebe, J. Compos. Mater. 44 (2010) 313–326.
[10] C. Suryanarayana, Prog. Mater. Sci. 46 (2001) 1–184.
[11] A. Azar, M. Momeny, Statistics and Its Application in Management, Samt Pub., Iran, 2004, pp. 27–56.
[12] S.S. Razavi Tousi, R. Yazdani Rad, E. Salahi, I. Mobasherpour, M. Razavi, Powder Technol. 192 (2009) 346–351.
[13] J.B. Fogagnolo, M.H. Robert, E.M. Ruiz-Navas, J.M. Torralba, J. Mater. Sci. (2004) 127–132.
[14] J.B. Fogagnolo, F. Velasco, M.H. Robert, J.M. Torralba, Mater. Sci. Eng. A 342 (2003) 131–143.
[15] Z. Razavi Hesabi, A. Simchi, S.M. Seyed Reihani, Mater. Sci. Eng. A 428 (2006) 159–168.