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Mechanics of Advanced Composite Structures

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The Structural and Mechanical Properties of Al-2.5%wt. B₄C Metal Matrix Nano-composite Fabricated by the Mechanical Alloying

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PAPER INFO

Paper history:

Received 3 April 2015

Received in revised form 13 June 2015

Accepted 13 June 2015

Keywords:

Mechanical properties

Al/B₄C nano-composite

Mechanical alloying

ABSTRACT

In this study, aluminum (Al) matrix reinforced with micro-particles (30 μm) and nano-particles (50 nm) boron carbide (B₄C) were used to prepare Al-2.5%wt. B₄C nano-composite and micro-composite, respectively, using mechanical alloying method. The mixed powders were mechanically milled at 5, 10, 15 and 20 hrs. The XRD results indicated that the crystallite sizes of both the micro-composite and nano-composite matrix decreased with increasing milling time, showing 55 nm and 40 nm, respectively. Mechanical testing results showed an increase in the flexural strength from 98 to 164 and 115 to 180 MPa, and an increase in the hardness from 60 to 118 and 75 to 130 HV for micro-composite and nano-composite, respectively. The results indicate that the strength and hardness of the nano-composite are higher than those of the micro-composite due to the presence of the fine particles.

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1. Introduction

In direct comparison with the corresponding monolithic alloys, Aluminum Metal Matrix Composites (AMMCs) offer a combination of (a) higher stiffness-to-density ratio, (b) better elevated temperature properties and (c) improved wear resistance. These composites are applicable particularly to the structural, wear, aerospace and transportation industries. The size of the reinforcement particles in the particulate aluminum MMCs can vary from around 10 nm up to 500 μm or larger. The composites with a fine and uniform dispersion of particles in the range of 10 nm to 1 μm are referred to as "nano-composites". The mechanical properties of nano-metric dispersion strengthened MMCs are far superior in comparison with those of micro-metric counterparts with a similar volume fraction of particulates. However, a homogeneous distribution of the reinforcing particles is essential for achieving the improved properties in the composites. The powder metallurgy (P/M) techniques are

known to contribute to the good distribution of the reinforcement particles, without the segregation phenomena typical of the casting processes [1].

The mechanical alloying method which is used in the production of composites and nano-composite material is a powder metallurgy process. This method allows the preparation of powder mixtures and homogeneous materials. The biggest problem in the production of MMCs by liquid-phase method is that there is not enough wettability of reinforced particles with matrix materials and it is not used more than 30% of volume fraction of reinforced particles [2]. The desired amounts of reinforcement material can be used in the powder metallurgy that is a solid phase process. One of the most important advantages of the mechanical alloying is providing homogeneous distribution of reinforcing particles within the metal matrix [3,4].

The ceramic particles such as SiC and Al₂O₃ are the most widely used materials for the reinforcement of aluminum [5]. The Boron carbide (B₄C) is one of the most promising ceramic materials due to

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its attractive properties, including high strength, low density (2.52 g/cm^3), extremely high hardness (the third hardest material after diamond and boron nitride), good chemical stability and neutron absorption capability [6-8]. Due to its high hardness, B_4C could be an alternative to SiC and Al_2O_3 as a reinforcement phase in AMCs for the applications where a good wear resistance is a major requirement. Shorowordi et al. investigated wear behavior of Al- B_4C and Al- SiC composites fabricated by stir casting method under the same conditions [9]. They observed that the wear rate and friction coefficient of Al- B_4C were lower than those of Al- SiC . Lee et al. fabricated aluminum matrix composite reinforced with B_4C particles and SiC particles through the same route (pressureless infiltration method) and under the same conditions in order to compare the effect of the reinforcement type on the tensile properties of the composites [10]. They reported that the strength of the Al- B_4C composite was greater than that of the Al- SiC composite. Moreover, Al- B_4C composites have been used in nuclear industries due to the specific ability of the B10 isotope to capture neutrons [11].

In the present study, aluminum matrix reinforced with 2.5%wt. of B_4C nano-particles is produced via mechanical alloying (MA) and cold pressing. The mechanical and structural properties of the bulk nano-composites such as crystal size, lattice strain of matrix alloy and micro-hardness in different periods have been studied.

2. Experimental Procedure

2.1 Materials

The commercial aluminum (Al) powder and boron carbide (B_4C) particles are used as raw materials in order to fabricate the micro-composite and nano-composite. The morphology of Al spherical powders (99.7 % purity) is shown in Fig. 1. As can be seen, as-received Al powders have the particle size of $5 \mu\text{m}$. The SEM micro-graph of as-received B_4C powders (>99.5% purity) is shown in Fig. 2. It can be seen that some B_4C nano-particles (50 nm) have been conglobated together, as shown in Fig. 2 (a) and B_4C micro-particles ($30 \mu\text{m}$) have almost regular shapes, as shown in Fig. 2 (b).

2.2. Fabrication of the Composites

The Al and B_4C powders are mechanically alloyed under high purity argon gas in a planetary milling apparatus. The Milling parameters are selected according to Table 1. It should be noted that Stearic acid ($\text{CH}_3(\text{CH}_2)_{16}\text{CO}_2\text{H}$) is added as the process control agent (PCA) to prevent the agglomeration and contamination of the powder mixture dur-

ing the milling process [12]. The composite powders are compacted by the cold pressing at a pressure of 200 MPa. Then, as-milled powders are heated up to 580°C for 2 hrs. Hereafter, the specimens that used $30 \mu\text{m}$ and 50 nm B_4C as reinforcement are denoted as micro-composite and nano-composite, respectively.

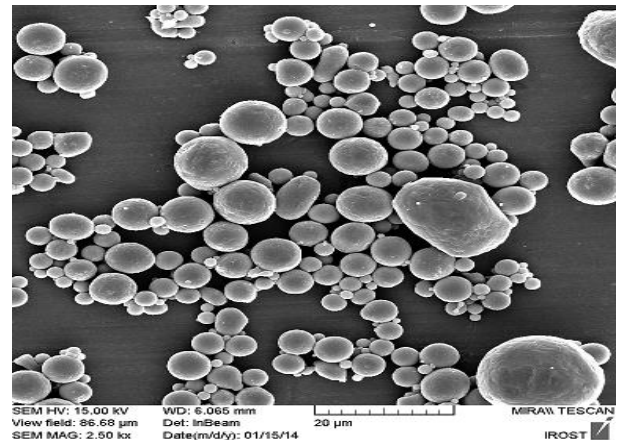


Figure 1. The SEM micro-graph of as-received Al powder

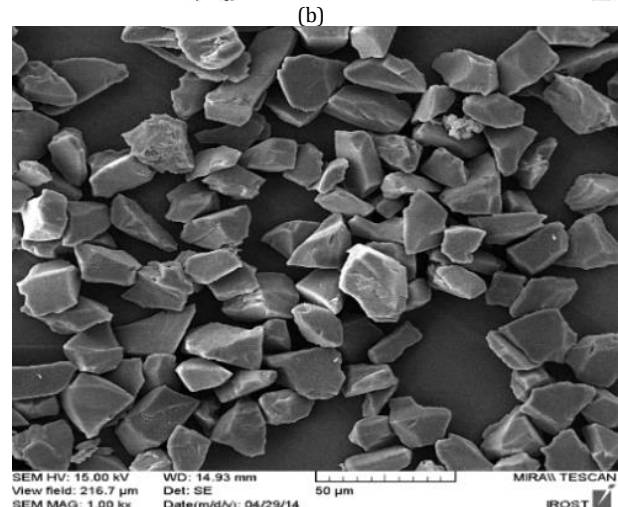
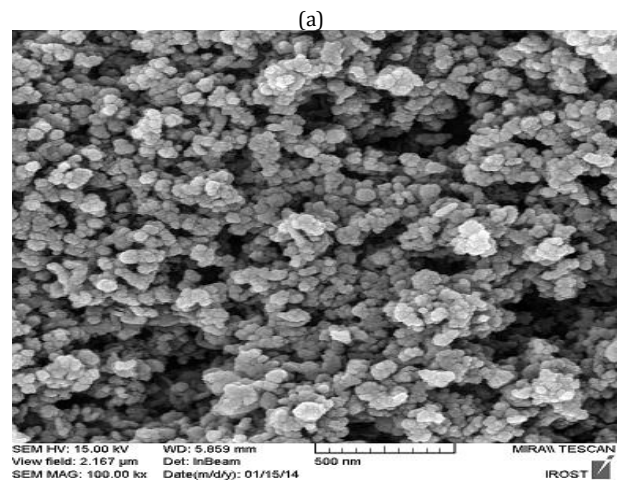


Figure 2. The SEM micrograph of as-received B_4C powder particles as (a) nano sized B_4C , (b) micro sized B_4C

Table 1. The milling parameters

Ball to powder ratio	Rotational speed	Milling time
10:1	270 rpm	5,10,15,20 hrs

2.3. Examination of the Microstructures and the Mechanical Properties

The microstructure characterization of the milled powders at different stages during the MA process is conducted, using a MIRA\TESCAN scanning electron microscope (SEM). The X-ray diffraction (XRD) analysis is performed by X-ray diffractometer using CuK α radiation at 30 kV and 25 mA. The crystallite size and the lattice strain of the milled aluminum powders are estimated by XRD peak broadening using Williamson-Hall equation as follows [13]:

$$\beta_s \cos \theta = 0.9 \lambda / D + 2\epsilon \sin \theta \quad (1)$$

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

The instrumental broadening (β_i) is removed by applying the following equation:

$$\beta_s = \beta_e - \beta_i \quad (2)$$

where β_e is the FWHM of the measured XRD peak.

The flexural strength and the hardness test of two kinds of composites were carried out, using standard strength and Vickers hardness test machine. The hardness test was done at a load of 10 kg.

3. Results and Discussion

3.1. Starting Microstructure

Fig. 3 shows the SEM micro-graphs of Al/B $_4$ C powders mixture, at different milling times, shown in two different magnifications. As shown in this figure, the powder particle size decreases as the milling time increases, which is probably due to the two opposing factors of cold welding and fracturing of powder particles [13]. While cold welding increases the particle size, fracturing reduces the size. In the early stages of the milling (5 hrs), as shown in Fig. 3 (a) and (b), the powder particles are still soft and cold welding predominates. Consequently, the particles have larger size compared to other powders that are milled at higher milling time. It has been shown that particle shape has become flattened due to the cold working effects during the milling [14]. As the milling time increases to 10 hrs, as shown in Fig. 3 (c) and (d), the particles appear in irregular shape having high aspect ratio. Also, many small and irregular particles with relatively low aspect ratio exist. After 15 h, a progressive decrease in the aspect ratio of irregular particles can be observed in Fig. 3 (e) and (f).

Longer milling time (20 hrs), provides a balance between welding and fracture, with the morphological transformation from laminar to equiaxed particle morphology, as shown in Fig. 3 (g) and (h). Some B $_4$ C particles are entrapped in the Al matrix during the mechanical milling and form clusters. These B $_4$ C clusters provide easier propagation of the cracks in Al matrix under the cyclic loading during the milling. Also the cold working induced during MA process, intensifies the initiation and propagation of cracks within powder particles. These cracks would propagate through the matrix alloy and finally fracture of the aluminum particles. These fresh fractured surfaces with B $_4$ C particles on them, would weld into other surfaces. With the repeated fracturing and the cold welding processes that take place during the energetic ball milling, B $_4$ C particles are eventually distributed uniformly within the Al matrix [15].

3.2. Structural Analysis

Fig. 4 shows the XRD patterns of the Al-2.5%wt. B $_4$ C nano-composite powders after 5, 10, 15, and 20 hrs of milling time. Due to the low content, the fine size and a limited scattering factor of B $_4$ C, its XRD peaks are not appeared. As can be seen in this figure, the significant phenomenon is peak broadening which occurs due to a decrease in the grain size and an increase in the lattice micro strain [16].

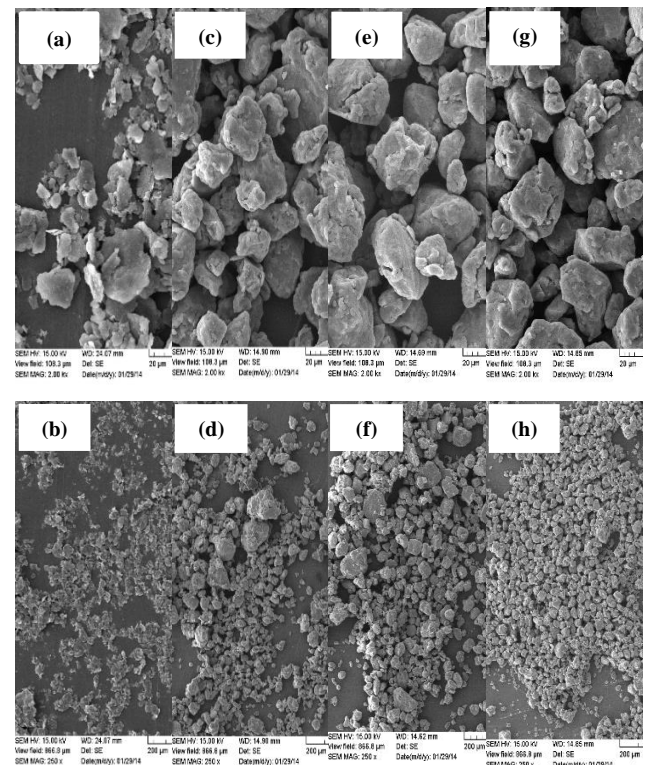


Figure 3. The morphology of nano-composite powder after (a) and (b) 5 hrs, (c) and (d) 10 hrs, (e) and (f) 15 hrs and (g) and (h) 20 hrs milling time

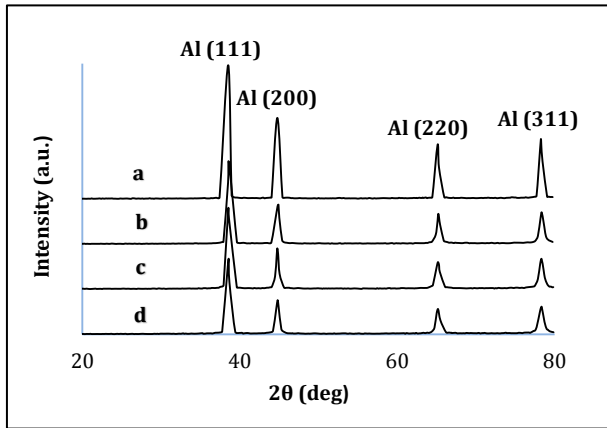


Figure 4. The X-ray diffraction patterns of Al-2.5%B₄C nano-composite powders after (a) 5 hrs, (b) 10 hrs, (c) 15 hrs and (d) 20 hrs of milling time

As shown in Fig. 5, the crystallite size decreases by increasing the milling time, down to 55 nm and 40 nm for micro-composite and nano-composite, respectively. On the other hand, the effect of the milling time on the lattice strain of the examined powder particles is presented in Fig. 6. It has been indicated that the lattice strain of the powder particles shows an increasing tendency. The reason of this variation can be referred to this fact that with increasing the milling time, severe deformation on powder particles is applied, leading to increase the crystalline defects such as point defects, dislocations and so on [17].

3.3. Mechanical Properties Evaluation

Figure 7 shows the flexural strength value of two kinds of composites as a function of the milling time. As shown in this figure, the flexural strength increases with increasing the milling time. It has been reported that the increase in the composite strength is influenced by a few factors such as the milling consequent deformation and work hardening; the grain refinement and sub grains production because of an increase in the dislocations density; an increase in the dislocations density because of the difference in the thermal expansion coefficient of the aluminum and boron carbide [18]. On the other hand, high sintering temperature (580°C) and the difference in thermal expansion coefficient of aluminum and boron carbide produce thermal stress [18]. The stress disappears by dislocations production and causes an increase in dislocations density. This is important for the strength enhancement.

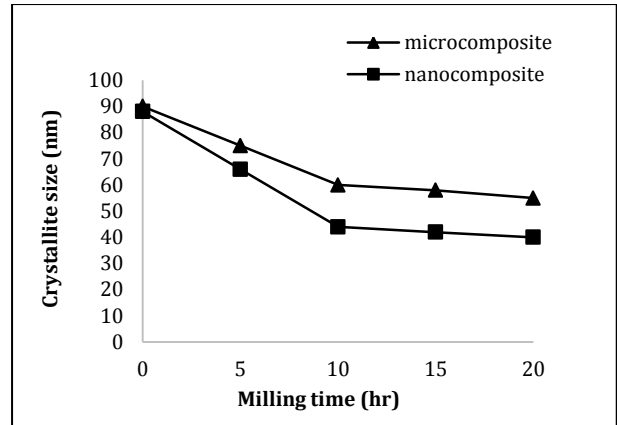


Figure 5. The change of the crystallite size for the microcomposite and nano-composite

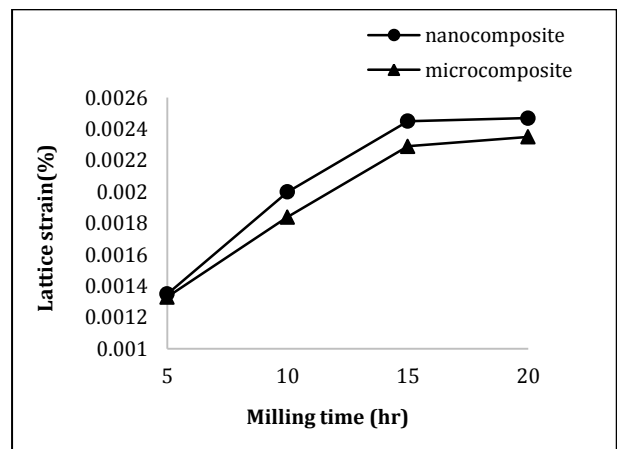


Figure 6. The effect of the milling time on the lattice strain of Al-2.5%wt. B₄C microcomposite and nano-composite

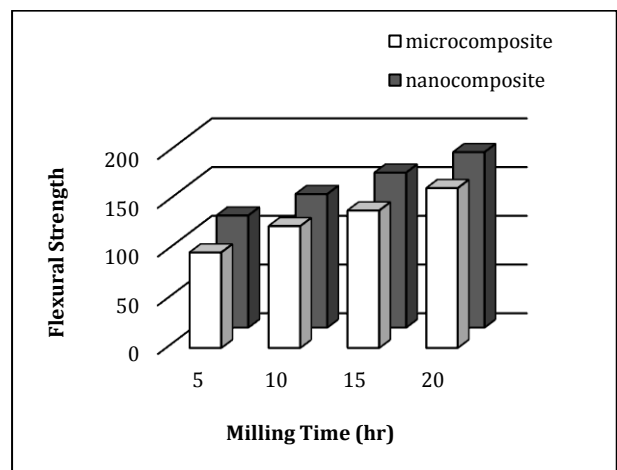


Figure 7. The variation of the flexural strength of the microcomposite and nano-composite with the milling time

Also, the inhibition of dislocation movement by carbide particles can contribute to the increment of strength. It can be said that the nano structured Al matrix produced by MA, influences the strength according to the famous Hall-Petch equation, as shown in Eq. (3) [19], and Tabors empirical relationship [20]:

$$H = H_0 + KD - 1/2 \quad (3)$$

Where H_0 and K are appropriate constants associated with the hardness measurement and D is the grain size.

The micro-hardness value of two kinds of the specimens as a function of the milling time is shown in Fig. 8. The same tendency as strength is observed for hardness value. As can be seen, the hardness value of the specimens increases with increasing the milling time. On the other hand, it can be seen that the hardness value and strength of nano-composite are higher than the micro-composite specimen. The difference between the two specimens is probably due to the Orowan strengthening mechanism, which in turn is due to the presence of nano-particles B_4C in the nano-composite specimen. The Orowan strengthening effect is produced by the interaction of fine particles and dislocations. The fine non-shearable ceramic reinforcement particles pin the crossing dislocations and promote dislocations bowing around the particles (Orowan loops) under external load [21,22]. The Orowan effect can be expressed by the following expression [23]:

$$H_{OROWAN} = 3\sqrt{3} \frac{Gb}{\lambda} \quad (4)$$

Where G and b are shear modulus of matrix and Burger's vector, respectively and λ is the interparticle spacing between dispersoids.

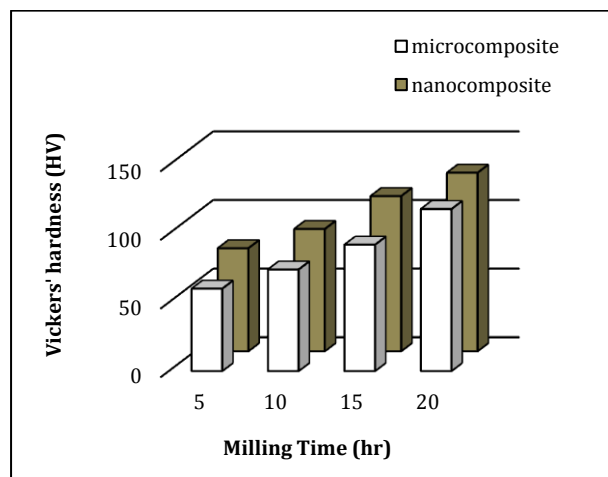


Figure 8. The variation of Vickers' hardness of the micro-composite and nano-composite with the milling time

4. Conclusions

The Al-2.5%wt. B_4C micro-composite and nano-composite were produced by mechanical alloying method. The structural evolution and mechanical properties of two kinds of specimens during the ball milling stages were investigated.

During the mechanical alloying, the crystallite size decreases with increasing the milling time. After 20 h milling, the crystallite sizes of the micro-composite and nano-composite were 55 and 40 nm, respectively. The mechanical properties of the specimens were studied using hardness and flexural strength tests. The flexural strength of the micro-composite and nano-composites increased with increasing the milling time and its values after 20 hrs milling were 164 and 180 MPa, respectively. The same tendency as the strength was observed for the hardness value. The values of micro-hardness of the micro-composite and nano-composite after 20 hrs milling were 118 and 130, respectively.

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