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Mechanical properties and tribological behavior of aluminum matrix composites reinforced with in-situ AlB₂ particles

Abstract

Aluminum matrix composites (AMCs) reinforced with in-situ AlB₂ particles have been fabricated using a combination of powder metallurgy, hot rolling and solution treatment. The effects of boron content (7 and 12 wt%), hot rolling and heat treatment parameters on the microstructures and mechanical properties of the composites were investigated by means of scanning electron microscopy (SEM), tensile test and micro-hardness measurements. The friction coefficient, wear behavior and scratch morphology of the AMCs and pure aluminum were also studied using scratch tests. The hardness and wear properties are higher in a case of composites when compared to unreinforced matrix material.

Keywords

situ, tribological, reinforced, behavior, composites, matrix, aluminum, particles, mechanical, alB₂, properties

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Mechanical properties and tribological behavior of aluminum matrix composites reinforced with in-situ AlB₂ particles

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Abstract: Aluminum matrix composites (AMCs) reinforced with in-situ AlB₂ particles have been fabricated using a combination of powder metallurgy, hot rolling and solution treatment. The effects of boron content (7 and 12 wt.%), hot rolling and heat treatment parameters on the microstructures and mechanical properties of the composites were investigated by means of scanning electron microscopy (SEM), tensile test and micro-hardness measurements. The friction coefficient, wear behavior and scratch morphology of the AMCs and pure aluminum were also studied using scratch tests. The hardness and wear properties are higher in a case of composites when compared to unreinforced matrix material.

Keywords: Aluminum matrix composites, AlB₂ reinforcement, Powder metallurgy, Wear

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1. Introduction

Aluminum matrix composites(AMCs) reinforced with ceramic particles have been extensively used in the aircraft, aerospace and automotive industries because of their high stiffness, strength to weight ratio, low coefficient of thermal expansion and good wear resistance [1-3]. The particulate reinforcements most often added to the pure aluminum or alloys include SiC, SiO₂, B₄C, TiB₂ and Al₂O₃ [4-9]. In previous studies, Khorshid et al. prepared the AMCs reinforced by two sizes of alumina particles by wet attrition milling and hot forward extrusion processes [10]. Haghighi et al. consolidated Al-5 vol.% nano-Al₂O₃ by equal channel angular pressing and extrusion and compared the physical and mechanical properties [11]. Vajargah et al. fabricated Al/TiB₂ nano composites via powder metallurgy route through a primarily cold isostatic pressing of aluminum powder containing different TiB₂ volume ratio and subsequent sintering at 650°C and 680°C [12].

Among all the reinforcements, boride particles are often chosen because of their lower density and the fact that their hardness is similar to other particles. Researchers reported that boride particulate reinforced AMCs can be fabricated via various processing techniques such as liquid state method, semisolid state method and powder metallurgy [13-16]. Toptan et al. produced AA1070 and AA6063 matrix B₄Cp reinforced composites by using titanium containing flux (K₂TiF₆) in order to overcome the poor wetting ability associated with the conventional casting [17]. Mazahery et al. worked on processing of Al–Si matrix composites reinforced with TiB₂ coated B₄C particulates and studied the wear behavior of the sol–gel coated B₄C particulate reinforced A356 matrix composites [14]. Topcu et al. prepared Al matrix composites reinforced with different B₄C ratios by powder metallurgy and investigated the mechanical properties of the composites [18]. Moreover, spark plasma sintering (SPS) method has been developed for sintering advanced ceramics and composite materials because SPS process allows preparation of bulk materials from mechanical alloyed powders using shorter sintering time at lower temperatures compared to hot

pressing and hot isostatic press processes [15]. In addition, the plasma generated between particles contributes to the elimination of surface impurities, leading to enhanced sintering and consolidation [19].

Unlike the many studies which have been done of the mechanical properties and structural characterization of B4C reinforced AMCs [14-18,20-27], little research has been done to examine the wear properties of AlB2 particle reinforced AMCs [28-31]. Actually, AlB2 particles are effective nucleation sites for aluminum alloys and the chemical reactivity between particles and melt is minimized, leading to excellent interfacial bonding with the matrix [32].

In the present work, AMCs reinforced with in-situ AlB2 particles are processed using powder metallurgy (mechanical alloying followed by spark plasma sintering) and hot rolling, the mechanical and wear properties of these particle-reinforced composites are investigated.

2. Experimental procedure

2.1 Fabrication of in-situ composites

The AMCs containing 7 and 12wt% boron powders were prepared using the powder metallurgy (PM) process and marked as Al-7wt%B and Al-12wt%B. The gas atomized aluminum powders (General Research Institute for Nonferrous Metals, 99.9% purity, 90μm average particle size) were blended with crystalline boron powders with equiaxial microstructure of average size 75μm (measured by LMS-30 laser particle size analyzer) in a high-energy ball mill for 5minutes. The dry-milling method was used in this process. The parameters of ball milling were: ball to powder weight ratio: 10:1; ball material: AISI 420 stainless steel; ball diameter: 6mm; speed: 1000rpm. 0.5wt% stearic acid powders were added into each batch to inhibit adhesion between the powder particles. Argon gas (99.9% purity) was put into a steel jar as protection because Al and B powders oxidize quickly in contact with oxygen. The powder mixtures were naturally air dried after milling.

The powder mixtures were sintered by spark plasma sintering (SPS) at 550°C at a heating rate of 40/min under a pressure of 50MPa (Dr.Sinter SPS-1050, Japan). The sintered samples were disks with 35mm diameter and 4mm height. The theoretical densities of the compacts were calculated from rule of mixtures given as Eq. 1 [33]:

$$\rho_{\text{theor}} = \rho_1 f_1 + \rho_2 f_2 + \rho_3 f_3 \quad (1)$$

where f_1 , f_2 and f_3 are the volume percentage of the boron, aluminum and AlB2, respectively, ρ_1 is the density of the boron (=2.34g/cm³) [34], ρ_2 is the density of the aluminum (=2.7g/cm³) [35], and ρ_3 is the density of the AlB2 (=3.19 g/cm³) [33]. The volume percentage of each phase was determined using ImagePro Plus software (Media Cybernetics). The actual densities of samples were determined using the Archimedes method.

The disks were heated to 450°C for 30min in an electric furnace chamber, hot-rolled at 400-450°C with each pass reduction of 10-15% and the finishing thickness was 2mm. The hot-rolled plates were maintained at 450°C for 2h, and then water-quenched to room temperature.

X-ray diffraction (XRD) investigations of the sintered, hot-rolled and heat treated samples were carried out using a Philips X'Pert Diffractometer with Cu K α ($\lambda=1.5406 \text{ \AA}$) radiation in the 2θ range of 20-100° at a rate of 4°/min.

The microstructures of the samples were observed using a scanning electron microscope (SEM)

equipped with an energy dispersive spectrometer (EDS, ZEISS ULTRA 55) and a 3D laser scanning microscope (VK-X100/X200 Series).

2.2 Uniaxial tensile and hardness tests

All specimens were polished before the tests using a polishing machine (Struers, TegraPol-21+TegraForce-5). The tensile tests were carried out using a universal testing machine (CMT4105) to investigate the tensile properties of the composites in the heat treated condition. Three specimens were used for each test and the average value was taken into account.

The micro-hardness of both pure Al and composites was measured using a LEICA-VMHT-30M Vickers tester with 100gf load.

2.3 Scratch test

The scratch tests were performed at room temperature using a Revetest scratch tester (CSM Instruments). The surface roughness of samples was measured to be about 0.05 μ m. In the present work, single-passed scratches were made using a diamond indenter with a spherical tip radius of 200 μ m. Normal load from 0.1N to 30N was applied progressively with a scratch length of 3mm. The scratch tests were carried out at two different sliding velocities, that is, 15 and 45mm/min. Frictional force was measured during the scratch tests and the friction coefficient is calculated by $\mu = F_t/F_n$, where F_t is the tangential force and F_n is the applied normal load of the indenter. The wear surfaces were observed under a 3D laser scanning microscope in order to analyze the wear mechanism and the wear area below the original surface was measured using a tactile profilometer.

The pure aluminum samples were prepared by casting and forging aluminum (the raw materials for atomized aluminum powders).

3. Results and discussion

Fig.1 shows the SEM photographs of the horizontal cross sections for binary Al-7wt%B and Al-12wt%B composites (sintered, hot rolled and heat treated, respectively). Multilayer composite structures were observed. According to the EDS results, as given in Fig. 2, it can be inferred that the ribbons embedded in the aluminum matrix are the agglomerations of boron and/or borides (see the mark in Fig. 1(a) and 1(d)). It is shown that the increase in boron content enhances the possibility of agglomerations, because the excess boron powder cannot mix sufficiently in a shorter time. To identify the phases of the composites, XRD investigations were conducted. Fig. 3 illustrates the XRD patterns of the composites under different conditions. Comparing experimental values with the standard values obtained from JCPDS card (PDF no. was marked in Fig. 3), the presences of Al, B and AlB₂ were detected. A certain amount of Fe contamination was also detected arising from the milling jar and balls. It is worth noting that the intensity of reflection peak around $2\theta=28^\circ$ increases after hot rolling and heat treatment for Al-7wt%B composites, but no obvious change is found for Al-12wt%B composites. According to the previous study, the peak heights can be used instead of integrated intensities when the percentage of each phase is calculated. The peak intensity ratio is proportional to the molar fraction ratio of the substances in a mixture [36]. It can be concluded that a slight formation of the AlB₂ phase takes place during hot rolling and heat treatment for Al-7wt%B composites. During the sintering process, some parts of the boron powders reacted with aluminum in-situ forming AlB₂ particles, and a particle reinforced Al matrix structure was generated. The lamellar AlB₂ particles are

around 5 μ m in length. The white dendrites of aluminum and dark AlB₂ flakes (arrows in Fig. 1) were distributed in the Al matrix. It shows a good interface bonding between AlB₂ particles and Al matrix. Some voids or porosity were observed in the sintered samples (the black spots in Fig. 1a and 1d), leading to low mechanical properties of the materials. After hot rolling, a majority of the AlB₂ particles showed a hexagonal morphology and became more uniform compared to that of sintered samples, as shown in Fig. 1(b) and (e). It can be seen that the distribution of the thickness of boron/boride layers is reduced and the internal voids are eliminated. This means that hot rolling can help to reduce the porosity and optimize the reinforcement distribution, as has been shown in previous research [37]. It is also stated that the flow of the aluminum matrix around the AlB₂ under high pressure can improve the bonding strength of AlB₂ and matrix. The AlB₂ particles can be seen to be coarser in both composites during heat treatment, as shown in Fig. 1(c) and (f). Fig. 1(c) reveals a uniform distribution of AlB₂ particles and lower agglomeration compared to Fig. 1(f). The figures also clearly show that a higher content of boron powder does not lead to a higher reinforcement volume. This is primarily attributed to the agglomeration of the boron particles in the structure which reduces the real reinforcement in the matrix [38]. The observation of microstructure is consistent with the XRD results, as shown in Fig. 3.

Fig. 1 SEM micrographs of composites (a) Sintered Al-7wt%B, (b) hot rolled Al-7wt%B, (c) heat treated Al-7wt%B, (d) sintered Al-12wt%B, (e) hot rolled Al-12wt%B, and (f) heat treated Al-12wt%B.

Fig. 2 EDS analysis results of composites (a) Sintered Al-7wt%B and (b) sintered Al-12wt%B.

Fig. 3 XRD patterns of the composites (a) Al-7wt%B specimens and (b) Al-12wt%B specimens.

The density values of the sintered compacts are shown in Table 1. The Al-12wt%B sample has higher relative density than that of the Al-7wt%B sample. In general, the sinterability of Al powders is relatively low due to the strong oxide scale on the surface of Al powders which prevents direct contact between the particles [39]. In the present study, the oxygen content was reduced by the SPS process as shown in Table 1. The specific surface cleaning effect of the SPS and high load pressure improved the contact between the particles by breaking the oxide scale on the surface of the particles.

Table 1 Some properties of the pure Al and composites

Sample	Density g/cm ³	Relative density/%	Oxygen content/%
Pure Al	2.583	95.66	-
Al-7wt%B powder	-	-	1.92
Al-7wt%B compact	2.268	85.29	1.47
Al-12wt%B powder	-	-	1.79
Al-12wt%B compact	2.261	85.31	1.35

The mechanical properties of the pure Al and the composites investigated are presented in Table 2. Each test was done three times and the average value is reported. Compared with the matrix material, the tensile and yield strengths of the Al-7wt%B composite increase significantly, whereas the elongation decreases. The Al-12wt%B specimens, however, exhibit lower tensile strength and elongation than those of the unreinforced material. It can be concluded that the excessive addition of boron powders in the matrix enhances the possibility of agglomeration, leading to poor performance of the composite. As can be clearly seen in Table 2, both the tensile and yield strengths of hot rolled composites are higher than the sintered composites. This is because that the large deformation improves the bonding strength between particles and matrix and allows the load to effectively transfer by AlB₂/Al interface. Furthermore, the composites with heat treatment exhibit the highest strength. Basically, the changing trend of elongation is opposite to that of strength.

Table 2 Tensile properties of the pure Al and composites

Samples	Tensile strength (MPa)	Yield strength (MPa)	Elongation (%)
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Pure Al	55.8 ± 2.1	15.6 ± 2.2	24.3 ± 0.9
Sintered Al-7wt%B	79.6 ± 3.8	32.5 ± 2.6	7.8 ± 0.6
Rolled Al-7wt%B	99.9 ± 3.2	60.6 ± 3.0	6.7 ± 0.7
Heat treated Al-7wt%B	145.7 ± 4.1	99.4 ± 3.9	3.7 ± 0.8
Sintered Al-12wt%B	36.4 ± 2.7	18.5 ± 2.3	9.3 ± 0.6
Rolled Al-12wt%B	41.6 ± 2.4	24.7 ± 2.0	11.1 ± 0.7
Heat treated Al-12wt%B	47.4 ± 2.3	31.2 ± 2.6	8.4 ± 0.8

The average micro-hardness values for the sintered, hot-rolled and heat treated composite samples were obtained, as shown in Fig.4. It can be seen that the AlB₂ particles enhance the hardness of the virgin aluminum sample (20.5HV in this study). This is due to the fact that the stiffer and harder AlB₂ reinforcement increases the constraint to plastic activity of the soft matrix during the hardness test. The hardness of the hot-rolled and heat treated samples also drops down gradually when compared to sintered samples.

Fig. 4 Vickers hardness with 100gf load for the composites under different conditions.

It is known that plastic deformation leads to work hardening for most polycrystalline metals but the stress can be relaxed when the strain increases to a certain extent. The strain softening occurs during hot working because of the dynamic recovery and dynamic recrystallisation [40,41]. As the stack fault energy of aluminum matrix is higher, the dislocations can be easier to move in the form of cross slip, making opposite dislocations eliminated. The dislocations can dynamically recover during the movement. In this case, the softening rate is higher than hardening rate, resulting in work softening. Accordingly, this softening counteracts work hardening. The interplay gradually decreases the work-hardening rate. A decrease in the hardness of the composites after heat treatment can be seen. In this study, the reduced hardness of the composites after hot rolling is ascribed to the higher strain softening rate because the grain coarsening during heat treatment decreases the role of grain refinement and the solid solution hardening is limited because of the low solubility of B in Al (0.022wt% at 659.7°C [42]). It is clear that the hardness of Al-7wt%B composites is higher than that of Al-12wt%B. This is because of the larger number of AlB₂ particles contained in Al-7wt%B samples.

Fig. 5 Coefficient of friction as a function of the applied normal force for all materials (a) Sintered Al-7wt%B compared with pure Al, (b) hot rolled Al-7wt%B, (c) heat treated Al-7wt%B, (d) sintered Al-12wt%B compared with pure Al, (e) hot rolled Al-12wt%B, and (f) heat treated Al-12wt%B

Fig. 5 shows the evolution of the coefficient of friction as a function of the progressive normal load for all samples. The coefficient of friction μ increases almost linearly with the load in all cases. This suggests that the reinforcements embedded in the matrix lead to a lower coefficient of friction, as shown in Fig. 5(a) and (d). It can also be seen that sliding speed has a little effect on the coefficient of friction. The results indicate, however, that the influence of speed is inconsistent with the coefficient of friction.

Fig. 6 Variation of scratch cross-section with normal load for all samples (a) Al-7wt%B samples compared with Al and (b) Al-12wt%B samples compared with Al.

As shown above, the hardness has some effect on the tribological behavior of the composites, but that is irregular. It is evident that the scratch cross-section tends to decrease during the increasing of the hardness for the Al-7wt%B composites. But this effect is not obvious for the Al-12wt%B composites.

It is clear from Fig. 6 that the unreinforced matrix materials worn much more than the reinforced composites, because the hard AlB₂ particles (HKnoop=9.6GPa) effectively resist the micro-cutting of the indenter [43,44]. The scratch cross-sectional area also increases linearly as the load increases for all materials. It is noted that Al-7wt%B samples experience a smaller wear area than the Al-12wt%B samples because of the higher wear resistance caused by the presence of more AlB₂ particles. The wear area is basically lower at the speed of 45mm/min and this is consistent with previous research [30]. There is no evident relationship between the coefficient of friction and wear behavior.

Fig. 7 Groove topography of matrix material and heat treated composites at a scratch speed of 45mm/min (a) Al, (b) Al-7wt%B and (c) Al-12wt%B.

Fig. 7 presents the groove topography of heat treated composites and Al at a scratch speed of 45mm/min. It is obvious that the progressively increasing load accelerates the plastic deformation in the sliding direction in all cases. The groove shows a slight difference under different conditions. The smooth topography of the Al-7wt%B composite in Fig. 7(b) is similar to that of pure Al (as shown in Fig. 7(a)), but the groove depth is shallower for the Al-7wt%B composite. This is because AlB₂ particles in the matrix change the stress state under the sliding conditions and influence wear behavior significantly. It can be seen that some fish-scales formed on the groove surface of the Al-12wt%B specimen because of boron powder agglomeration.

These loose areas have very low load-carrying capacity and hence are easily removed during scratch. In general, the scratch process is composed of rubbing, ploughing and plastic cutting [45]. For the composites studied in this work, some fractured particles could be observed on the groove surface.

4. Conclusions

The present study shows that:

- (1) Aluminum matrix composites reinforced with in situ AlB₂ particles were successfully fabricated by powder metallurgy followed by hot rolling.
- (2) The microstructure and mechanical properties of composites deteriorate after excessive addition of boron powder in pure Al. This can be attributed primarily to the less uniform microstructures of Al-12wt%B samples than those of Al-7wt%B.
- (3) Under the present scratch test conditions, the wear resistance of the pure Al is improved significantly due to the formation of in situ AlB₂ particles. The wear area and friction coefficient of the samples also increase almost linearly with the increase in the applied load.
- (4) The sliding speed affects the coefficient of friction and wear in this study but no regular change is found.

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