Research Article

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Effect of nano Al$_2$O$_3$ particles on the mechanical and wear properties of Al/Al$_2$O$_3$ composites manufactured via ARB

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Abstract: This study first tried to fabricate AA1060/Al$_2$O$_3$ composites via the stir casting and accumulative roll bonding process. Then, the effect of nano Al$_2$O$_3$ Vol% on mechanical, wear, and microstructural properties of these kinds of composites have been investigated. An excellent particle distribution through the aluminum matrix has been achieved after the fourth cycle. Then, mechanical properties, wear resistance, and microstructural properties have been investigated. The results showed that the strength of these composites was enhanced and the elongation of samples decreased by higher alumina Vol% contents. Also, there is a significant increase in wear resistance by increasing alumina content in the Al matrix through the stir casting process.

Keywords: accumulative roll bonding, mechanical properties, metal matrix composites, wear resistance, wear test

1 Introduction

Nowadays, aluminum-based composites are popular in many industries similarly as electrical, vessels, chemical, aerospace, etc. due to having excellent properties such as light density, high mechanical properties, high wear, and corrosion resistance [1–8]. Usually high hardness value of alumina particles makes it a desirable additive for utilizing as an emergent reinforcement in aluminum base composites [9–17]. Usually aluminum-based composites can be produced via several methods such as alloying laser techniques [18–20], plasma and high-speed oxygen fuel spray, and sputtering and high-energy milling techniques [21–23]. These techniques enhance the mechanical properties and wear resistance of Al-based composites by generating a hard coating on the Al matrix by sticking alumina particles through it. These particles are popular as reinforcing in MMCs due to having fantastic possessions such as high and abrasion resistance, degree of hardness, and melting point. To produce aluminum-based composites (AMMCs), stir casting usually is famous as a low-cost process for having the capability for high volume production and its simplicity. So, this process creates a low cavity (porosity) structure with an unvarying scattering of the additive particles through the metallic matrix [24–26].

Many forming processes through severe plastic deformation (SPD) processes are utilized to fabricate Al-based composites, such as accumulative roll bonding (ARB), cyclic extrusion compression [20], squeeze casting, spray forming [18,19], and powder metallurgy [27]. While cast MMCs usually have a desirable particle distribution through the metallic matrix, they have poor fracture toughness and ductility. So, processing of these kinds of composites via an SPD deformation technique is necessary [21]. So, these problems inspire the helping of manufacturing techniques such as ARB after the stir casting process, in this study. Saito was the first researcher who proposed the ARB for the first time in 1998 [23]. ARB is defined as cumulative rolling processes on multi-layered strips with the same primary dimensions which have been prepared. This process is not only a forming technique but also a bonding (as a branch of solid phase method of welding) process. For each ARB process, the reduction in thickness is 50% and will be repeated for all cycles. The main purpose of this study is to produce Al/alumina bulk composites by combined stir casting and ARB at 300°C and improve the mechanical properties with the homogenous spreading of
alumina particles. It was tried to produce Al/alumina composite with unvarying dispersed alumina particles in the Al matrix and estimate the wear, microstructural, and mechanical properties such as hardness, strength, and elongation.

2 Experimental procedure

2.1 The stir casting process

The fully annealed primary material aluminum alloy 1060 is used for fabricating the experimental samples. Alumina particles as additive particles with an average grain size of 50 nanometers are used as reinforcement with a purity of 99%. Before the casting process, the additive particles were preheated at 400°C for 5 hours to diminish their humidity. The alumina particles with 0, 2.5, 5 and 10 Vol% were put on the surface of the aluminum melt at 750°C, and this composition was rotated with a rotary speed of 650 rpm with the blowing of Argon gas to protect the melt from oxidation (Figure 1). The stir casting process allows the creation of a desirable bonding between aluminum and alumina particles which leads to decreasing the number of voids and cavities in the final composite matrix. The dimension of the final fabricated composite samples is 200 mm × 26 mm × 1 mm.

Brushing the surface of composite samples prior to the ARB process is necessary to create a desirable bonding between composite layers. Based on the film theory (one of the main theories to explain the solid phase method of welding), brushing the surface of composite samples before ARB allows to deliver a coarse surface which introduces a restricted shear during the forming process and disrupts the oxide layers. This disruption creates more cracks and allows the virgin metals to extrude along the cracks and toward the opposite composite layers which create metallic bonding [23].

2.2 ARB process

At the starting of the ARB process and to clean the contaminations and grease from the surface of composite samples, the samples were brushed and put in the acetone bath for 30 min. Then, the samples were heated at 300°C for 10 min, and the two strips were stacked against each other. In continuing, all the samples were rolled with a thickness reduction ratio of 50% (Figure 2). Then, the samples were halved and the surface preparation process was repeated for each cycle. According to Figure 3, adsorbed ions, greases, oxides, and dust subdivisions have surrounded the surfaces of samples. Using a 95 mm diameter stainless steel with 0.26 mm wire diameter and speed of 2,000 rpm, the sample strips were scratch brushed after degreasing in the acetone bath. So, surface cleaning before each cumulative rolling process is essential to generate an acceptable bonding.

To study the effect of Al2O3 Vol% on the wear and mechanical properties, The ARB was continued for up to four cycles to generate a desirable scattering of alumina subdivisions in the Al matrix. So, the ARB steps of the process are summarized in Table 1.

In order to set up the rolling process, a laboratory rolling machine with a capacity of 40 tones with a rotary speed of 60 rpm and a roll diameter 170 mm is used in this study. According to Figure 4 in order to supply the tensile test samples, ASTM standards E8M has been used. Also, standard ASTM-E384 with a 500 grams load for fifteen seconds was used as the Vickers hardness testing condition. The samples were prepared for wear test with 60 mm × 30 mm × 1 mm, and the test was performed by means of a “pin on disc” wear testing machine fitting into the fixture. The wear parameters were 500 s test time, 20 N load, and 1 m·s⁻¹ velocity. Wear tests were conducted using a “pin on disc” wear testing machine, with the 60 mm × 30 mm × 1 mm samples used. As the wear pin, a stainless steel pin containing 30 mm long and 8 mm in diameter was used. Also, the worn surface of samples was observed via scanning electron microscope (SEM).
3 Results

3.1 Alumina particles distribution

Figure 5 shows the SEM micrograph of composite layers after cycle No. 4. As shown in the figure, after the fourth cycle, the alumina particles have a uniform distribution inside the composite matrix, and because of this, for investigating the effect of alumina Vol%, all samples have been fabricated after four cycles of ARB. In other words, by increasing the cumulative rolling up to four cycles, the distance between alumina particles increases which leads to disruption of alumina clusters and generation of uniform particle scattering.

3.2 Mechanical properties

Having different alumina volume contents fabricated after four cycles, tensile tests have been carried out for MMC

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**Table 1: ARB process steps for fabrication of Al/alumina composites**

| No. of steps | Rolling temperature (°C) | No. of composite layers | Reduction in cycles (%) | Composite layer thickness (µm) | Final reduction (%) | Plastic strain |
|--------------|--------------------------|-------------------------|-------------------------|-------------------------------|--------------------|---------------|
| 1            | 300                      | 2                       | 50                      | 1,000                         | 50                 | 0.8           |
| 2            | 300                      | 4                       | 50                      | 500                           | 75                 | 1.6           |
| 3            | 300                      | 8                       | 50                      | 250                           | 87.5               | 2.4           |
| 4            | 300                      | 16                      | 50                      | 125                           | 93.7               | 3.2           |
samples (Figure 6). The flow stress rapidly reaches its maximum value by increasing the plastic strain. The enhancement of the mechanical properties was achieved by increasing the alumina content (Figure 6). According to Figure 7, the tensile strength of the ARBed MMCs is 127.75 and 178 MPa for monolithic samples and samples produced with 10 Vol% of alumina content registering 39.3% improvement. Strain hardening around the alumina particles during the rolling process activates the slip systems which generates further dislocations close the particles and decreases the flexibility of dislocations [19]. Also, because the ARB has been done in this study is a warm forming process, there is grain refinement during all the ARB cycles. Both of them lead to improving in the strength and decreasing in the elongation of samples which decreases the bonding strength.

The elongation of samples decreases considerably at higher Al2O3 contents which attributed to the weak adhesion of particles agglomerations to the Al matrix (Figure 7). Always, alumina particle layers contain voids affecting the elongation reduction. The harder particles block the motion of Al grains which increases the dislocation density [28–31]. All of these effects reduce the elongation and enhance the strength of the AA1060/alumina composites. According to Figures 6 and 7, higher volume fraction of particles leads to more strain hardening around the particles. So, the tensile strength increases while ductility drops. The ductility of the ARBed MMCs is 4.28% (monolithic sample) and 1.8% (10% alumina), as shown in Figure 7. As mentioned before, reduction of dislocation mobility and work hardening lead to the creation of this behavior. As mentioned before, porosities in alumina layers reduce the elongation value. So, the reasons (mechanism) for
enhancing the elongation are (i) growing the uniformity of alumina subdivisions inside the aluminum matrix, (ii) increasing the bonding between the Al matrix and alumina particles which decreases the voids among them, and (iii) decreasing the agglomeration and dispersing them inside the metal matrix.

The toughness value drops considerably from 4.2 J m\(^{-3}\) \times 10^4 (monolithic sample) to 2.7 J m\(^{-3}\) \times 10^4 (10% alumina), which is due to the less mobility of dislocations and stress hardening (Figure 8). So, increasing the number of alumina particles as a barrier against the grain’s movements in the AA1060 matrix decreases the elongation severely which reduces the toughness amplitude [27,32–34].

The hardness values of the samples after ARB increase (Figure 8). According to Figure 8, the average Vickers hardness amount increases from 157.0 up to 167.0 which is about 5.70% enhancement. With increasing the content of particles, the first reason for increasing the hardness via forming processes is the generation of a fine grain structure due to the amount of plastic strain. This phenomenon leads to the growth of dislocation density which impedes the movement of grain boundaries and leads to increasing the initial work hardening [21,31]. The materials gain a certain steady-state density of dislocations by increasing the ARB cycles up to four cycles. In stage 2, the limited work hardening around the alumina particles is the main factor in increasing the hardness value. So, higher values of alumina particles inside the composite matrix generate more strain hardening, and as a result, the hardness improves.
3.3 Wear test

Figure 9 shows the wear rate of composite samples versus alumina particles content fabricated via four cycles of ARB. As can be viewed in Figure 9, the most wear rate is for the monolithic composite sample (87 mm$^3$·mm$^{-1}$). The least value for the wear test belongs to the sample fabricated with 10% of alumina (47 mm$^3$·mm$^{-1}$). In the other words, the uniform distribution of alumina particles leads to the reduction of weight loss. This trend is due to the work hardening effect of the Al matrix and the dislocation strengthening mechanism around the alumina particles. Also, higher cycles lead to decreasing the porosities between alumina particles and aluminum base matrix which improves the bonding strength between them and decreases the wear rate [22].

Figure 9: Wear rate in sliding wear for Al/alumina composite vs alumina volume content.

3.4 Wear surface morphology

Figure 10 indicates the surface morphology of composite samples after wear testing. According to Figure 10, the wear rate of samples decreases at a higher number of cycles. Also, small wear tracks are the result of abrasive wear mechanisms (Figure 10). Finally, the result shows that the composite samples reinforced with 10% alumina particles have better wear resistance properties in comparison with monolithic samples. Based on the above-mentioned discussions and after the beginning of wear, growth of the grains occurred at the plastically deformed region (between the pin and the worn surface) due to high temperature. So, a coarse grain structure is formed under subsurface which had a strain incompatibility regarding the fine grain structure and non-equilibrium

Figure 10: Wear surface of the (a) monolithic and (b) Al/10 Vol% alumina composites.
ultrafine grains. In the other words, debris particles were formed when the strain incompatibility caused delamination of coarse grains as the subsurface of the wearing samples. Increasing the alumina Vol% decreases the width of wear groves and improves the wear resistance due to local work hardening of the aluminum matrix around the particles.

Figure 11 shows the SEM image of debris after the wear test of monolithic and composite samples fabricated with 10 Vol% of alumina particles. As mentioned before according to Figure 12, after the beginning of the wear between the worn surfaces and pin, growth of the grains occurred due to high temperature in the plastically deformed region with more growth in the subsurface zone than the middle thickness. So, under subsurface which had a strain incompatibility regarding the fine grain structure and non-equilibrium ultrafine grains a coarse grain structure is formed. In the other words when the strain incompatibility caused delamination of coarse grains as the subsurface of the wearing samples, debris particles were formed.

Figure 11: SEM micrographs of wear debris particles of (a) monolithic and (b) composite reinforced with 10 Vol% of alumina.

Figure 12: Schematics of the sliding wear of ARB composite samples vs different steps of deformation and recrystallization mechanism.
Also, these delamination and recrystallization processes occurred in repetitive rounds. Always, the generation of large particle debris in a shape is the result of this kind of wear mechanism. The initiation and nucleation of cracks on the sliding surfaces are the results of high shear stresses. So, this changes the shape of the loss of material from flakes to plates. So, the flows of surface material are toward the sliding direction which generates abrasive grooves under a higher applied load \[28–30\]. The debris size for the sample fabricated with 10% alumina content is less than that of the monolithic sample. This difference in the size of debris is the result of the local strengthening mechanism around the alumina particles which creates localized hardened regions around the alumina particles. Then, as a result, the size of debris decreases.

3.5 Fractography

To clarify the rupture mechanisms in the monolithic and Al/10 vol% alumina composite samples, an SEM study is conducted (Figure 13). Typically shear regions and deep dimples of the monolithic sample are the results of typically ductile fracture (Figure 13(a)). This type of fracture mode happens through the coalescence and formation of voids. By increasing the alumina content, the length of dimples decreases which has a particular effect on the fracture surface. Alumina substances sit on the cores and walls of dimples and are suitable sites for the propagation of micro-cracks \[21,31,35,36\].

4 Conclusions

At this point, the new AMMCs are made through four cycles of the ARB process by diffusing Al\(_2\)O\(_3\) volume content particles, the Phase conformation, mechanical properties and wear resistance of these kinds of composites, and microstructure properties were scrutinized. In summary, the following conclusions were drawn:

1. Fairly constant scattering of particulates in the Al matrix released by scanning electron microscopy. The strength, tensile toughness, and wear resistance of composites were enhanced due to the addition of alumina particles.
2. The results revealed that the composites with 10 Vol% of alumina particulates have better wear resistance properties compared to the monolithic sample.
3. Increasing alumina Vol% enhances the strength of composites. So, it reaches a maximum value of 178 MPa with 10 Vol% of alumina particles which is about 1.40 times more than that of monolithic composite.
4. The elongation for the composite sample fabricated via 10 Vol% of alumina was 1.79 and for the monolithic sample was 4.28 registering about 2.39 times decrease.
5. The average Vickers hardness amount of the samples improves from 157.0 to 167.0 for monolithic and 10% alumina samples registering a 5.7% improvement, respectively.

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Heydari Vini designed the experiments and conducted the approaches and Saeed Daneshmand classified the results and wrote the article text. All authors discussed the results and contributed to the final manuscript.

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