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Mechanical and Microstructural Response of Aluminum Composites Reinforced with Ceramic Micro-Particles

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Abstract: Aluminum matrix composites have recently taken an important role in advanced applications because they have a good combination of physical and chemical properties. For this reason, in this work, aluminum composites, with additions of ceramic particles (mullite or tungsten carbide), were manufactured in order to determine the effect of those particles on the mechanical properties and microstructure of aluminum. The manufacture of the composites was carried out by means of powder metallurgy. We studied composites with additions of 0.5 and 1 vol.% of the respective ceramic. Composites were sintered at 580 and 601 °C, which corresponds to 88 and 91% of the melting point of aluminum, respectively. Observations in SEM, together with EDX analysis, confirm that mullite particles are located at intragranular and transgranular positions of the aluminum matrix, while tungsten carbide particles were found mostly at intragranular areas of the matrix. From the analysis of the studied ceramics, it was found that with the use of mullite, there are greater improvements in the hardness and elastic modulus of the manufactured composite.

Keywords: aluminum matrix; powder metallurgy; mechanical properties



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1. Introduction

The use of aluminum matrix composites (AMC's) with unique properties is growing day by day in different industries, due to their high resistance-weight ratio, good corrosion resistance, and high thermal and electrical conductivity [1–3]. These composites initially substituted some cast iron and bronze alloys, but owing to their poor wear resistance, they were subjected to many experiments in order to understand their wear behavior. The results of these experiments have been reported by several researchers during the last three decades [4–6]. Scientists have found that the incorporation of different types of reinforcement materials, such as ceramics, metals, and intermetallic particles, have proved to be an effective way to increase the wear properties of the aluminum matrix. This is how AMC's have been manufactured with the addition of different compounds that are intended to be the reinforcers of the metallic matrix, thus the AMC's have been manufactured with SiC/B₄C [7], SiC/Al₂O₃ [8], TiC [9], alloys of Fe-Cr-Al/Y₂O₃ [10], FeC/Cr₂C [11]. Likewise, they have been processed through very different methods, such as stir casting [12], powder metallurgy [13], spray atomization and co-deposition [14], plasma spraying [15], and squeeze-casting [16]. These composites have been manufactured to produce lightweight components with high, specific mechanical properties such as wear resistance, thus they have been applied in the automotive and aeronautical industry where the reduction of weight, wear, friction, and low coefficient of thermal expansion are important [1]. A situation to consider is that, to date, the production of the AMC's has been conducted through very sophisticated methods, using a special aluminum alloy [8,10,11] or using very special reinforcing materials, such as graphene [17–19]. Depending on the route of processing and according to the findings in the literature, the incorporation of

non-metallic ceramic reinforcements in aluminum increase the mechanical properties of AMC's, resulting advantages such as: hardness, strength, fatigue, fracture, degradation, and corrosion resistance, due to the small grain size and homogeneity of their microstructures [20]. This work aims to study the effect of small volume fraction additions of tungsten carbide or mullite on the mechanical properties of pure aluminum, processed by powder metallurgy techniques, which represents a combination of conditions not studied so far.

2. Materials and Methods

The AMC's were prepared using the following precursor powders: Aluminum (99.9%, 1 μm , Sigma-Aldrich, St. Louis, MO, USA), Mullite- $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ (99.9%, <10 μm , Sigma-Aldrich) and Tungsten Carbide-WC (99.9%, <100 nm, Sigma-Aldrich). The amounts of the ceramic added were 0, 0.5, and 1 vol.%, whereas the batch of each studied system was 25 g. The powders were mixed and grounded in a planetary type of mill (Retsch, PM100, NJ, USA), using ZrO_2 balls of 3 mm diameter as the grinding medium, in a ratio of 11:1 ball weight:weight of the powder, grinding was done at 300 rpm for 0.25 h. With the resulting powders from the milling, cylindrical samples of 20 mm in diameter by 3 mm in thickness were manufactured at a pressure of 185 MPa, with the help of a uniaxial press (Montequipo, Tlalnepantla de Baz, Mexico, LAB-30-T). Then, the pressed samples were sintered in an electric furnace (Carbolite, Sheffield, UK, RHF17/3E) at 581 $^\circ\text{C}$ and 601 $^\circ\text{C}$ for 1 h at atmospheric pressure. The heating rate was 8 $^\circ\text{C}/\text{min}$, after the sintering time was reached, the furnace was turned off with the samples inside for their gradual cooling. The density of the samples was determined by the Archimedes method. The microstructure of the sintered samples was analyzed using scanning electron microscopy (JEOL, Akishima-shi, Japan, 6300). The SEM was equipped with an X-ray dispersive energy analyzer (Hitachi, Tokyo, Japan, SU9000 UHR FE-SEM). The mechanical properties of the obtained specimens were evaluated by microhardness using a Vickers microdurometer (Wilson Instruments, Campbellford, ON, Canada, S400); the elastic modulus was determined by the ultrasound method, using an ultrasonic sensor (GrindoSonic, Leuven, Belgium, A-360). The flexural strength at 3 points was determined in a universal testing machine (Instron, Norfolk County, MA, USA). Finally, the surface fractures were analyzed using a stereoscope (Olympus, Tokyo, Japan, SZ61). The values reported for all measurements are the average of the measurements made on 5 different samples for each system studied.

3. Results and Discussion

3.1. Morphological Analysis of Powders

Figure 1 shows pictures of the different study samples resulting from the grinding stage. In Figure 1a which corresponds to the control sample, an agglomeration of a large number of particles can be observed. For the case where WC is added (Figure 1b,c), although agglomerates formation is observed they are smaller, which indicates that somehow the presence of WC helps to prevent them from forming. This is perhaps because the WC affects the electrical properties on the surface of the aluminum, which causes the electrostatic forces to decrease in this area, thus avoiding the formation of large agglomerates, as has been suggested in the literature [21]. In Figure 1b,c, the particles with a lighter coloring belong to the WC and are easily distinguishable by the density difference of both components WC and Al. Likewise, samples with mullite (Figure 1d,e) show the behavior described for WC, although mullite seems to form larger agglomerates than those formed by the WC reinforcing, as it is difficult to distinguish mullite from aluminum because their densities are closer. In general, all the images show that the particles acquired the shape of flakes due to the type of grinding carried out and the strong ductility of the aluminum, which seems to have been laminated during the mechanical action of grinding.

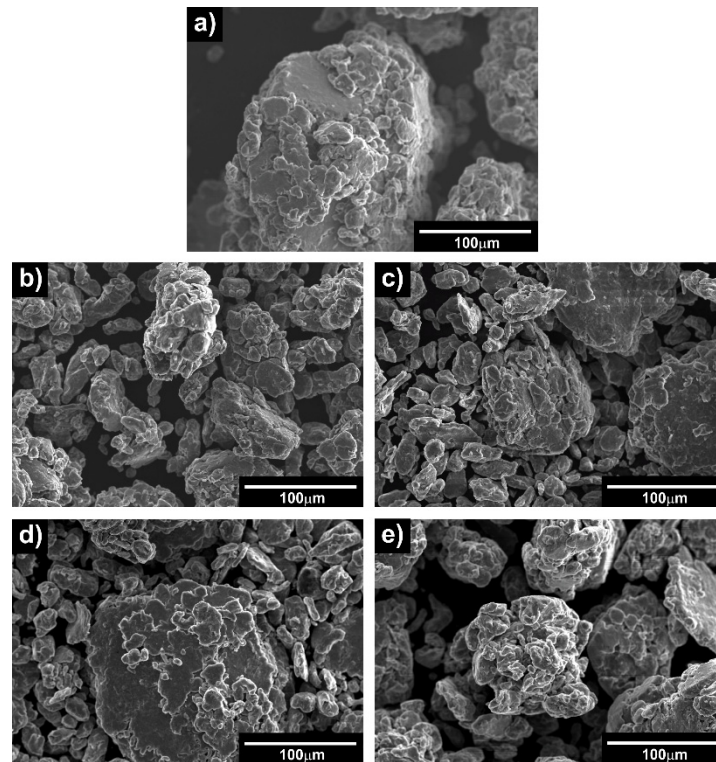


Figure 1. Micrograph of the powders after the grinding stage for the studied compositions: (a) 100 vol.% Al, (b) Al/0.5 vol.% WC, (c) Al/1.0 vol.% WC, (d) Al/0.5 vol.% mullite and (e) Al/1 vol.% mullite.

3.2. Size Distribution of Powders

Figure 2 shows the results of the particle size distribution performed on the 5 samples obtained after the milling stage. In this figure, it is observed that approximately 50% of the powders in all the samples have sizes smaller than 50 μm . However, during grinding, powders were obtained with varying sizes according to the composition of the samples. For the samples with additions of WC (Figure 2a), it can be seen that when a greater amount of reinforcement is added (1 vol.% WC), the particle size decreases (average of 48 μm), but it does not have a uniform distribution, while the addition of 0.5 vol.% WC has a better size distribution, where the average particle size is 101 μm . Pure aluminum presented very large particle sizes, with an average of 126 μm , formed by agglomerates. In the case of the sample with mullite additions (Figure 2b), the size distribution is more homogeneous. In this case, the average particle size was 41 μm for the sample with 0.5 vol.% mullite and 89 μm for the sample with 1 vol.% mullite.

3.3. Density

Figure 3 shows the results of the measurements of the relative density on the samples evaluated through the Archimedes' principle. In this figure, it can be noted that the samples that best density are those that contain mullite, as those samples with 1 vol.% mullite have a better densification. With regard to the samples with WC, the densification of the samples is not significant, although the samples with 1 vol.% WC densifies more than that which contains 0.5 vol.% WC. With respect to the 100% Al sample, the relative density reached by this is greater, while in the case of samples with WC, the density of these samples was lower than that of pure aluminum. On the other hand, the sintering temperature does not have an important effect on the densification of the samples, since in all cases similar densities are obtained regardless of the sintering temperature.

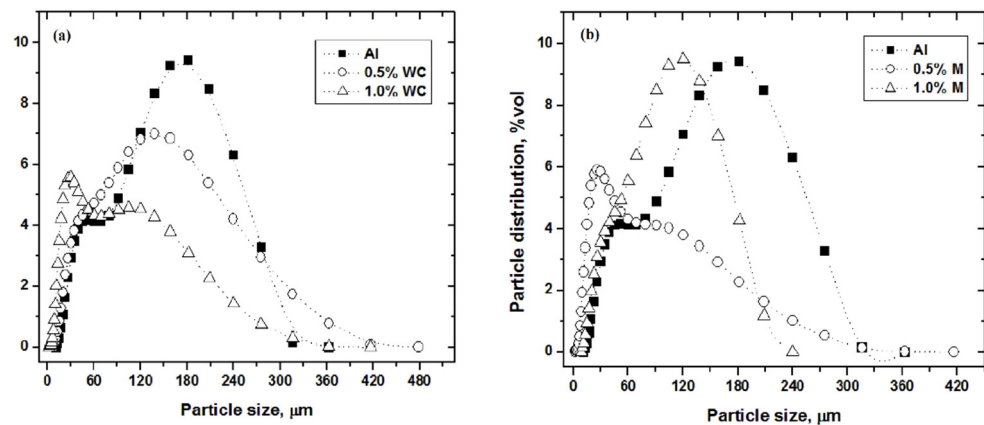


Figure 2. Distribution of particle sizes in powder samples after the grinding stage. (a) System with additions of WC, (b) System with additions of mullite.

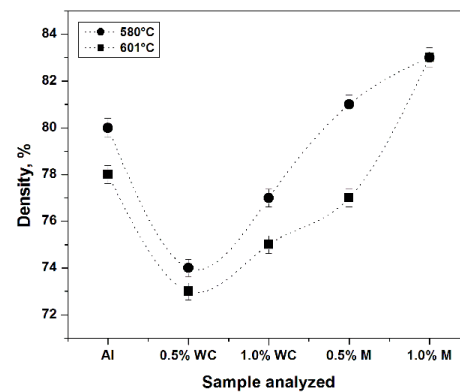


Figure 3. Results of the relative density reached by the samples with different compositions and sintered at different temperatures.

As can be seen in these results, very low densification values were achieved, it is well known that densification of powdered materials is a process of pore elimination whereby the porosity of these sintered alloys decreases with the increase of the time and correct sintering temperature. In this sense, the sintering conditions, mainly the times, were probably insufficient to achieve a better densification of the materials. Another factor to consider is the compaction pressure, where it is surely necessary to increase this pressure in order to achieve higher green densities. Finally, the better densification in the samples with mullite compared to those containing WC, is due to the difference in densities between these ceramics and Al, this gap being greater between Al and WC than between Al and mullite.

3.4. Microstructure

Figure 4 shows the microstructures of the samples sintered at 601 °C for 1 h. In all samples, very homogeneous microstructures in terms of shape and grain size can be observed. In the sample with WC contents, the presence of large pores is evident, which helps to explain the lower density reached by these samples. The smallest clear points present at inter and intragranular zones correspond to the reinforcing material added in each sample. This is verified with the EDX analyses performed on these points and shown in Figure 5 for the sample with WC and mullite, respectively, observing in Figure 5a the presence of the W as part of the WC, and Si in Figure 5b as a component of the mullite.

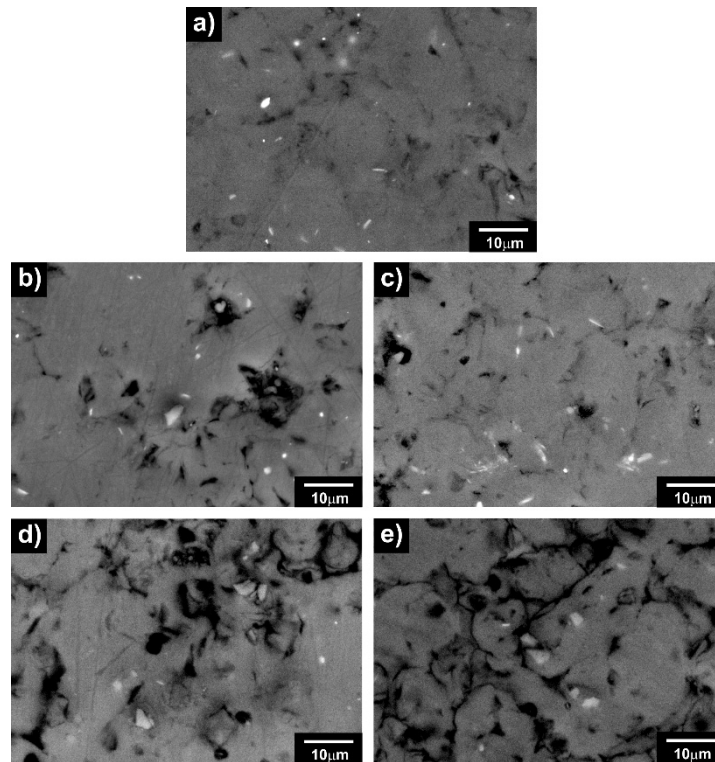


Figure 4. Microstructures of the samples sintered at 601 °C, during 1 h. (a) Pure aluminum, (b) Al-0.5 vol.% WC, (c) Al-1 vol.% WC, (d) Al-0.5 vol.% Mullite and (e) Al-1 vol.% mullite.

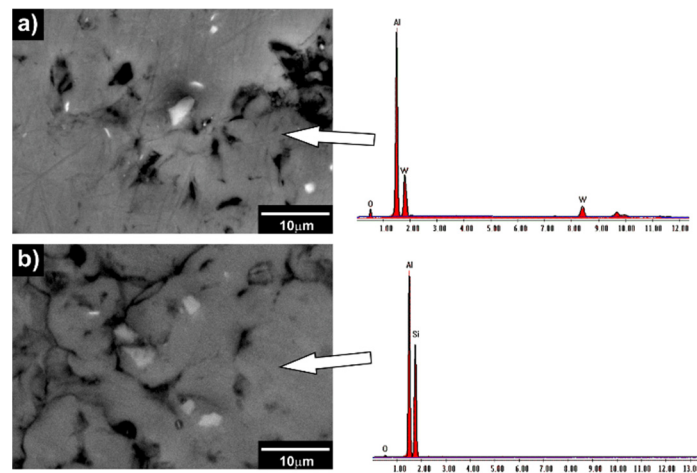


Figure 5. EDX made in samples sintered at 600 °C for 1 h, (a) composite of the matrix Al-0.5 vol.% WC and (b) composite of the matrix Al-0.5 vol.% mullite.

3.5. Mechanical Properties

3.5.1. Microhardness

Figure 6 shows the results of the microhardness measurements made in the study samples. In this figure it is observed, as expected, that when adding ceramic particles of greater hardness than aluminum, this property will increase, being that the increase in hardness is greater for the sample with WC. Likewise, the increase in the amount of reinforcing material manifests itself as an increase in the hardness of the material. Again, it is observed that the sintering temperature does not have an important effect on the final hardness of the samples.

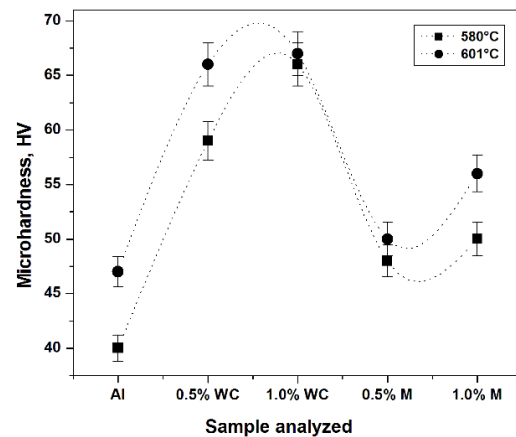


Figure 6. Results of the microhardness evaluated in the samples with different compositions and sintered at different temperatures.

3.5.2. Elastic Modulus

Figure 7 shows the elastic modulus measured in the study samples. The WC and mullite ceramic materials have a greater elastic modulus than aluminum, this condition is reflected in Figure 7, where the samples with additions of WC and mullite reached a final elastic modulus greater than those of pure aluminum. The elastic modulus of the samples with WC is greater because the elastic modulus of the WC is greater than that of the mullite. The behavior of the curves is similar, observing that the temperature does not significantly affect the final value of the elastic modulus.

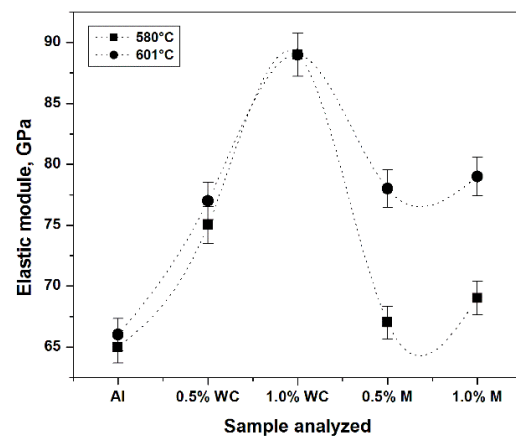


Figure 7. Results of the elastic modulus evaluated in the samples with different compositions and sintered at different temperatures.

3.5.3. Bending Strength

Figure 8 shows the results of bending resistance measurements. In this figure, it is appreciated that when adding a ceramic compound of greater hardness and lower ductility than the aluminum matrix, there is a decrease in the mechanical strength of the composite obtained, mainly in the sample with WC due to the strong fragile nature of this compound. For the case of mullite additions, likewise, there is a loss of strength in the material. However, what is sought when adding ceramic materials of great hardness compared with that of a soft and ductile metal such as aluminum is to increase the hardness and stiffness of this metal beyond its mechanical strength.

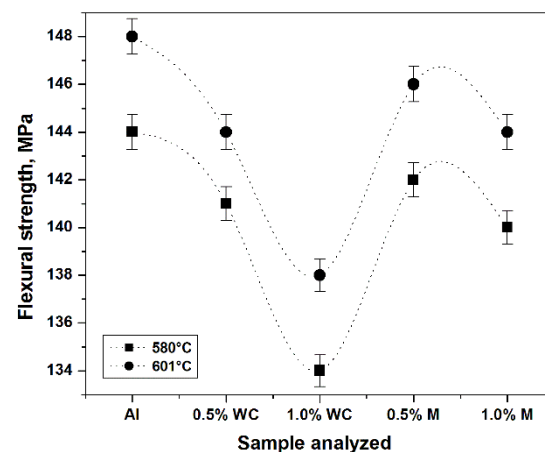


Figure 8. Results of the flexural strength measurements of the samples with different compositions and sintered at different temperatures.

3.5.4. Fracture Surfaces

Figure 9 shows images of the fracture surface of samples without reinforcement and with 1 vol.% WC or mullite used as reinforcing materials. By means of these photographs, it can be observed that in all samples the fracture had a fragile behavior, since there is a complete detachment of grains. This is caused by the processing route that does not allow a solid conforming, since according to the results of evaluated densities, the samples have a certain porosity, as the presence of these pores is where the cracks have their origin.

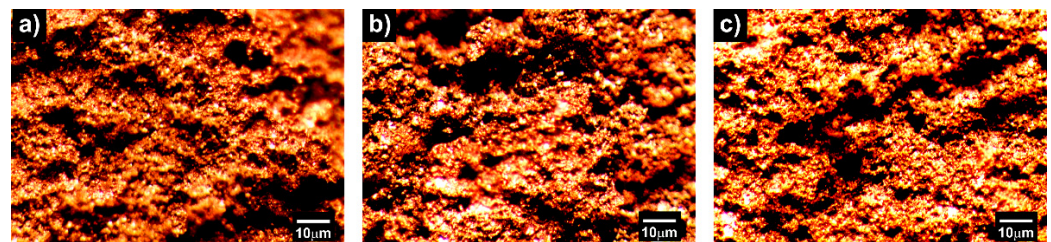


Figure 9. Images taken with a stereographic microscope from the fracture surface area of the composite (a) Pure aluminum, (b) Al-1 vol.% WC and (c) Al-1 vol.% mullite.

From the results of the mechanical properties evaluated in the composites obtained here, we have that the reinforcement of ductile metals by means of ceramic particles, materials that are hard and brittle in nature, coupled with powder metallurgy processing, can only be reflected in increases in the hardness and elastic modulus of the metal, since the microstructures present only the distribution of the two phases in a homogeneous way, without the formation of solid solutions. Therefore, it is not feasible to achieve increases in the strength and ductility of the resulting materials through this type of process and reinforcement, a situation that has been well documented by other researchers [22].

4. Conclusions

- Aluminum-based composites can be effectively hardened by dispersing mullite particles and tungsten carbide, through powder metallurgy techniques that involve the combination of mechanical grinding, cold compaction, and pressureless sintering. The results showed that reinforced ceramics provided aluminum increments in hardness and stiffness properties.
- The reinforcement of ductile metals by means of ceramic particles, materials that are hard and brittle in nature, coupled with powder metallurgy processing can only be reflected in increases in the hardness and elastic modulus of the metal, since the microstructures present only the distribution of the two phases in a homogeneous way, without the formation of solid solutions. Therefore, it is not feasible to achieve

increases in the strength and ductility of the resulting materials through this type of process and reinforcement

- Microstructure observations indicate that mullite particles are located at intragranular and transgranular positions of the aluminum matrix, while tungsten carbide particles were found mostly at intragranular areas of the matrix.
- This processing methodology, with favorable results in terms of increasing the mechanical properties of aluminum, can be a simple and inexpensive processing alternative, which makes it attractive to scale the process at the industrial level.

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