Mechanical Study on Al-based Composites Synthesized by Mechanical Milling and Hot Extrusion

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Abstract

Al-based composites were fabricated by solid-state route and were characterized by optic and scanning electron microscopy in order to follow their microstructural evolution. Composites were prepared using powder metallurgy techniques in order to obtain samples to carry out mechanical tests on hot extruded and machined samples. Microstructural characterization reveals that, by milling, a homogeneous dispersion of insoluble particles into Al matrix is obtained; this produced an important improvement on hardness and strength compared with the reference. Milling intensity and particle concentration have an important effect on the mechanical properties of composites.

Introduction

Aluminum and its alloys have a wide diversity of industrial applications because of their corrosion resistance. The interest to increase aluminum strength for applications in demanding industries [1], where the light weight of these alloys would be an advantage over the parts made of cast iron [2], has motivated the study of reinforced aluminum composites. For different ceramic/metal composite systems the incorporation of a ductile metal phase like aluminum is known to improve mechanical properties compared to monolithic ceramic performs, due to the fact that metal is more ductile, metal phase provides a significant resistance to crack propagation [3]. The principle of the strength enhancement in these materials lies in introducing of high strength dispersed phase into the metal matrix [4], without losing the benefit of low density [5]. Aluminum composites can be prepared by dispersing insoluble particles like carbides, oxides, nitrides, silicon, graphite, etc. into the matrix by using techniques in a solid or liquid state [1]. On the other hand, graphite (C_g) has been recognized as high strength, low density material, due its high strength to mass ratio, has been used as reinforcement material in polymer-based composites [6]. Another advantage is its structural stability at high temperature [5]. The amount that the dispersoids strengthen the composite depends on particle type, size, morphology, volume fraction and distribution. The extent to which the particles withstand dissolution in the matrix and coalescence is an important factor of composite strengthening. Some difficulties are encountered in production of these composites by liquid route [2]. Three well-known facts are: (i) gasification of carbon, which initiates below the melting temperature of pure aluminum, (ii) reaction between aluminum and carbon to form aluminum carbide, an unstable compound with very poor mechanical and thermal properties, and (iii) poor wettability at the Al/C_g interface. The last one negatively influences the final properties of the prepared composite by porosity increase [7].

With the advent of mechanical milling (MM), it became possible to integrate a very fine distribution of hardening particles into the metal matrix by solid-state powder processing, which otherwise would be difficult or even impossible with most material techniques [8]. This process starts with dry, high energy milling of the matrix powder with dispersoids, producing a homogeneous composite with fine microstructure and good distribution of dispersed particles [4]; additionally high dislocation density and small subgrain size in the matrix can be obtained [9].

Powder metallurgy (PM) is a technology capable of providing competitive components at low cost with high material efficiency [10]; basically it consists of mixing elements or alloy powders, compacting the mixture in a die, and sintering the compacts to just below their melting points in a controlled-atmosphere furnace to bond the particles. The porosity concentration can be eliminated or reduced by subsequent hot extrusion or rolling [9]. However, used raw materials only consist in simple powder mixtures. Powder mixing is a critical step [11] since it controls the distribution of particles and porosity, both of which influence the composite mechanical behavior. Some variables like reinforcement size-shape and type of matrix can induce agglomeration, this can be a cause of low performance [12]. If reinforcement particles are homogeneously distributed [13] using mechanical milling (MM) in the first stage, it can contribute to achieve a good microstructural components distribution with a decrease in their grain and particle size [14] and increase on the mechanical response. Furthermore, solid-state processing minimizes reactions between matrix and reinforcement, which can enhance the bonding between reinforcement particles and matrix [15].

The used method in this work lies with introducing graphite particles into the aluminum matrix by MM and PM. The aim is the Al-based composite synthesis and the study of the effect of graphite concentration on mechanical properties (strength and hardness) of consolidated, sintered and hot extruded composite materials. The effect of different milling conditions on the graphite distribution into Al matrix is presented and discussed as a result of microstructural and mechanical characterization.

Experimental procedure

Al-based composites were produced following next sequence:

Components: Alfa Aesar powders of Al (99.5% purity and -325 in size), Cu (99% purity and - 325 in size) and graphite (99.9% purity and -20+84 mesh), were used as raw materials.

Reinforcement preparation: metallized graphite (MG) was prepared by milling a mix of graphite-copper with 15 at.% of Cu, using a SPEX 8000M device in Ar atmosphere during 4h.

Al based composite synthesis: composites were prepared by mixing Al powder with prepared MG particles in concentration of 0.0, 0.5 and 1.0 wt.% of Cu-MG with the nomenclature showed in Table 1. Then, the as-mixed powders were milled in a ZOZ CM01 Simoloyer mill for 4 milling intervals (1, 2, 4 and 8h) under Ar atmosphere. Milling device and media were made of stainless steel, Methanol was used as process control agent to avoid excessive aluminum agglomeration. Pure Al samples (without Cu-MG addition), milled and un-milled were used as reference materials for comparison proposes.

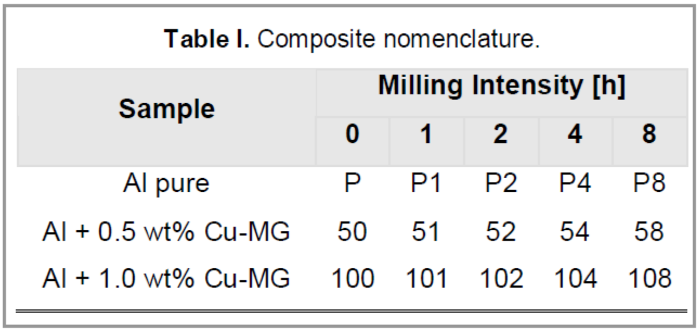


Table I. Composite nomenclature.

Sample	Milling Intensity [h]				
	0	**1**	**2**	**4**	**8**
Al pure	P	P1	P2	P4	P8
Al + 0.5 wt% Cu-MG	50	51	52	54	58
Al + 1.0 wt% Cu-MG	100	101	102	104	108

Powder characterization: In order to accomplish the microstructural observations, a portion of powder composite samples were prepared using standard metallographic techniques and studied with an optic and scanning electron microscope (JEOL-JSM 7401F).

Mechanical Testing: Hardness tests were executed in compacted and sintered samples with a Wilson Rockwell hardness tester (average of five measurements was considered) in Rockwell F scale and converted to Brinell. Compression tests were carried out in an Instron universal tester at constant displacement rate of 0.0333 mm/sec, yield stress was measured at elastic limit and maximum stress was measured at arbitrary condition of 20% strain. Four samples with the best mechanical performance (compression tests) were then hot extruded by using an indirect extrusion method (extrusion ratio 16:1) and mechanically tested by tensile assay, yield stress was measured at elastic limit.

Results and Discussion

Morphological analysis

Fig. 1 shows some micrographs of 1.0%-CuMG composite. At the beginning un-milled powders exhibit a predominantly spherical morphology with a moderately broad size distribution typical of gas atomized metals. Later it is evident a notable increment in the particle size, particles were severely deformed plastically changing their morphology from spherical to flattened, forming large aggregates, cold welding is the predominant phenomenon [11], causing the large-sized particle formation. Next fracture process arise, work hardened fragile flakes appear forming finer particles in comparison with initial stages of milling. Then, welding and fracture mechanisms reach equilibrium and the formation of particles with randomly oriented interfacial boundaries. The final stage is characterized by the steady state process, in which the microstructural refinement can continue, but the particle size and size distribution remain approximately the same [16].

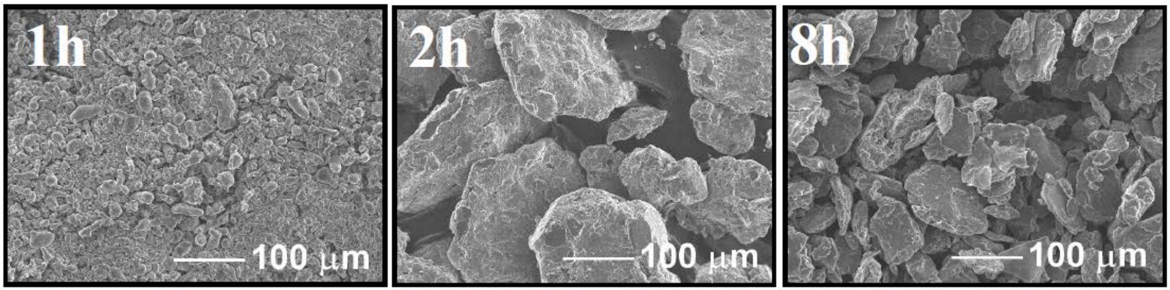


Fig. 1. SEM micrographs on 1.0%-CuMG particles with 0, 2 and 8 hours of milling.

Since the size distribution of powders tended to decrease with further milling, it is clear that both, the matrix and reinforcement particles were fractured. Small particles were embedded over the matrix surface. With further milling, the surface fractured again and a new surface was exposed and covered by free particles.

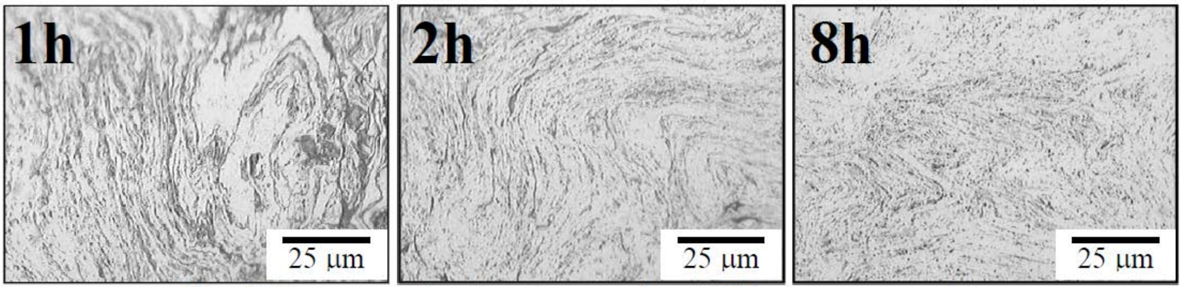
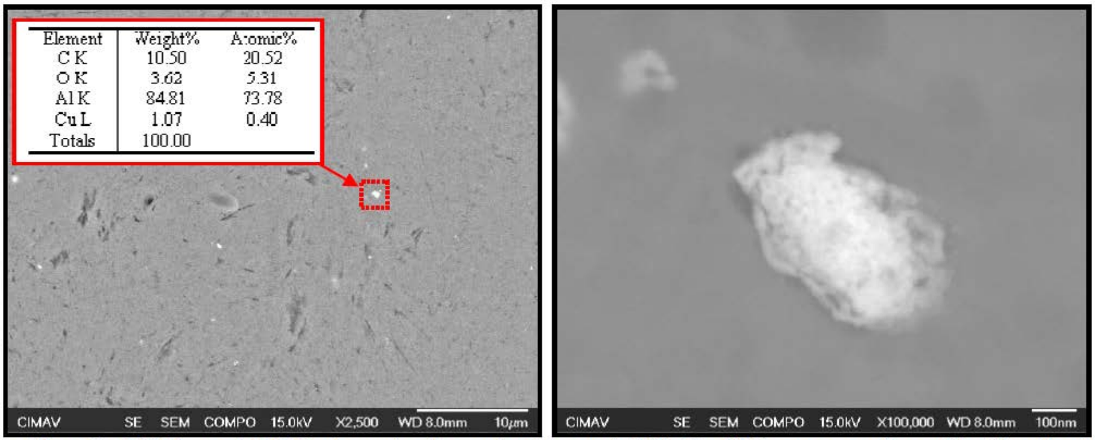


Fig. 2. Transversal optic micrographs at 500x (same sample).

This process was repeated and then the fragmented particles were captured by welding particles and confined to welding lines, thus obtaining a lamellar configuration (Fig. 2). Repeated processes of fracture of the composite particles and convolution resulted in a uniform distribution of the CuMG particles in a layer fashion and good bonding between the layers [8], which contributes to increase the composite strength as it will be presented later.

The reinforcement particles are dispersed into the matrix and have a notable content of C and Cu (EDS analysis). Even though the high solubility of Cu in Al, particles remain insoluble as remnant and these present a nanometric size grouped in form of cluster as Fig. 3 shows. SEM studies complemented with micro analysis on Cu-MG particles (bright spots) embedded between Al matrix layers shown a homogeneous and fine dispersion of the reinforcement into the metallic matrix, this can contributes to the improvement of the compression strength, because of the homogeneous stress dispersion is associated with a favorable microstructural arrangement [8].



Element	Weight%	Atomic%
C K	10.50	20.52
O K	3.62	5.31
Al K	84.81	73.78
Cu L	1.07	0.40
Totals	100.00	

Fig. 3. SEM and EDS chemical analysis on 1.0%-CuMG composite with 4h of milling.

Mechanical properties on sintered composites

Un-milled and milled composite powders were cold pressed at 950 MPa, in form of cylindrical samples (6.25 mm in diameter) and sintered for 3h at 823 K under a protective argon flow (150 cm3/min), in order to carry out the mechanical assays. Mechanical results are showed in Fig. 4a, it is clear that milled-Al samples present a better performance on stress-strain curves compared with un-milled Al, due mainly to work hardening [17]. From Table 2, it is evident that milling induces an important increase on the yield and maximum strength (compared with pure Al)

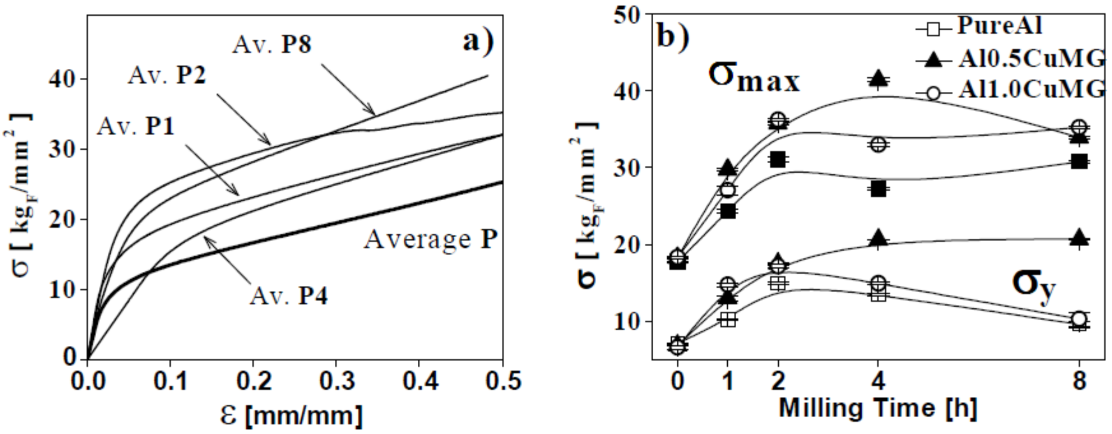


Fig. 4. a) Stress-Strain curves of un-doped Al samples. b) Yield and maximum strength values found in sintered composites as a function of milling intensity and additive content.

In contrast to good experimental values found in the present study, Son et al. [8] established that addition of graphite decreases the compressive strength of the composites, due possibly to low contact area between matrix powders. Fogagnolo et al. [18] mentioned that reinforcement clustering, cracks in the reinforcement surface or poor bounding between matrix and reinforcements can also deteriorate the composite strength. High concentration composites (1.0%Cu-MG) presented a modest performance probably due to the presence of free enforced particles which segregated forming agglomerates and diminishing the final properties [19] or by matrix saturation effect [8]. Fig. 4b shows a comparison of σ_y and σ_{max} in synthesized composites, the increment in both properties is notable. Cu-MG concentration and milling intensity have an important effect on the mechanical performance in tested composites. It indicates a synergic effect of metal graphite addition and milling intensity. The optimum point was obtained with 4h of milling and low concentration (0.5%Cu-MG) of reinforcement particles, like Esawi et al. [20] found.

Table 2. Compression test results and hardness measurements in the composites after sintering.

	Milling Time [h]	σ_y [kg/mm^2]	σ_{max} [kg/mm^2]	**Brinell Hardness**
Pure Al	*0*	7.12 ± 0.05	17.71 ± 0.04	-----
	1	10.17 ± 0.07	24.31 ± 0.22	-----
	2	14.91 ± 0.16	31.05 ± 0.33	*Under 55 HB*
	4	13.46 ± 0.18	27.18 ± 0.21	-----
	8	9.60 ± 0.44	30.71 ± 0.15	-----
Al-050% Cu-MG	*0*	6.84 ± 0.05	18.24 ± 0.04	-----
	1	13.01 ± 0.31	29.72 ± 0.20	*Under 55 HB*
	2	17.50 ± 0.18	35.71 ± 0.14	*55 ± 1*
	4	20.62 ± 0.01	41.34 ± 0.24	*66 ± 1*
	8	20.66 ± 0.05	33.87 ± 0.13	*63 ± 1*
Al-1.00% Cu-MG	*0*	6.58 ± 0.28	18.31 ± 0.20	-----
	1	14.72 ± 0.25	27.02 ± 0.63	-----
	2	17.14 ± 0.23	36.17 ± 0.14	*59 ± 1*
	4	14.90 ± 0.17	32.95 ± 0.22	*Under 55 HB*
	8	10.29 ± 0.92	35.20 ± 0.13	*Under 55 HB*

Macro hardness determinations were done instead μ-hardness test due the fact that Cu-MG particles have different hardness values compared with metal matrix; a punctual measure could induce a high scattering in results depending the localization of indentation point. Hardness measurements in compacted and sintered composites (Table 2) show that mechanically milled Al samples (2h) are harder than the as-mixed sample; the differences between composites with same Cu-MG addition and different milling time were significant. The differences in the microstructure between the as- mixed and the milled powders explain the variation in their hardness and confirm the effectiveness of the process. The interactions between the reinforcing particles and the matrix are responsible of the improvement on mechanical properties of synthesized composite. So the quality of the bond Al/C_g is limited by the process of integration of the particles into the metal matrix. Besterci [21] concluded that volume fraction of carbide phase $A1_4C_3$ are in good agreement with achieved mechanical properties and the best strengthening was obtained with carbon types with a high transformation rate to Al carbide content and low subgrain size. On the contrary, Fogagnolo et al.[18] concluded that the hardness differences between the milled powders are much smaller compared with as-received powder. O. Yilmaz et al. [9] concluded that the presence of hard dispersed particles will cause additional strain hardening and the strength of the composites increases with the volume percentage of particles in the composite. Complement studies showed that is true, but higher particle concentration (>5%) decreases the strength of the products by matrix saturation effect.

Mechanical properties on extruded composites.

Based on the best mechanical performance results of previous mechanical compression tests, 75 grams of milled samples **P2**, **54**, **102** and reference **P** (un-milled pure Al) were compacted, sintered and hot extruded (773 K) using an indirect extrusion method with an extrusion ratio of 16:1, obtaining a bar of 1 cm in diameter. Based on E8-ASTM (Standard Test Methods for Tension Testing of Metallic Materials), three samples of each condition were machined in order to carry out the tensile tests in a Universal Instron Machine at room temperature. In Fig. 5a some average tensile curves from 3 tests in each sample are presented. Curves show that mechanically milled Al sample

(P2) is tougher than the as-mixed sample (in good agreement with compression results) and the differences between samples are significant. Fig. 5b shows a plot with σ_y and σ_{max} results and they are compared with some commercial alloys Al-1350 [22] mechanically treated, small circles are yield strength and small squares represent UTS reported values for this alloy, between them are the type of mechanical hardened treatment used. It is evident the effect of hardening by Cu-MG addition and mechanical processing on the mechanical behavior of the material.

The final hardness of the composite is due to two factors: the effect of the milling process on the matrix alloy, and the effect of the reinforcement particles. The analysis of these results demonstrates that the effect of the mechanical alloying process (milling intensity) is greater than reinforcement concentration. The best composite (54 sample) presents an increased 50% of σ_y and 200% of ultimate tensile strength (UTS) compared with reference sample (P). Differences between the reference sample (P) and the others composites are perceptible in UTS region.

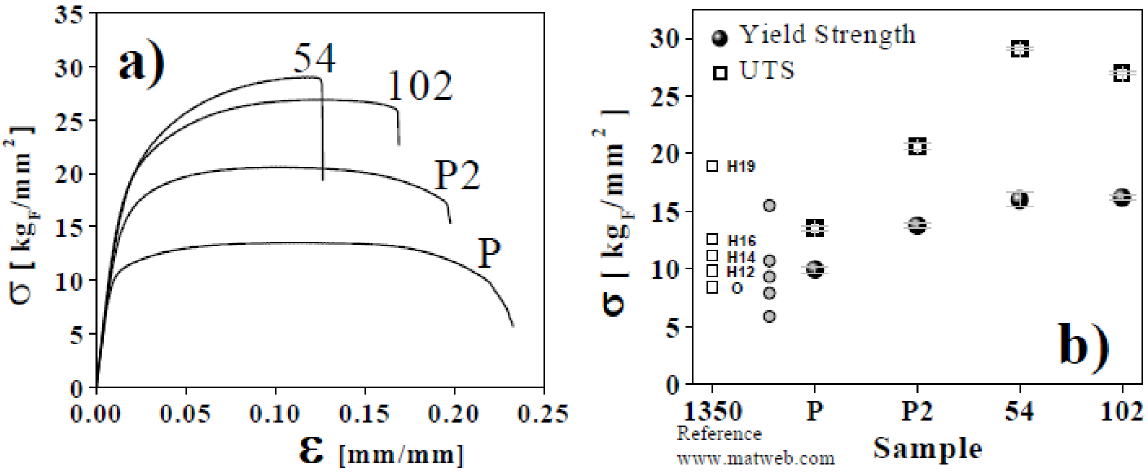


Fig. 5. a) Average Stress-Strain curves in tensile essay. b) Yield and UTS found in composites compared with a commercial 1350 alloy.

Summary

Reinforcement particles present sub-micrometric size and were homogeneously distributed into the aluminum matrix leading an important effect on the mechanical performance of the prepared composites. Additive concentration has an important effect on mechanical properties of composites and it has a synergic effect with milling intensity. Low concentration Al Cu-MG composite with 4h of milling (sample 54) was the best option as strengthening condition with an increase of 41% (UTS) compared with pure milled sample (P2). Pre-milling process increases the mechanical properties of Al-based composites prepared by powder metallurgy technique.

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