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Study of Al composites prepared by high-energy ball milling; Effect of processing conditions

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ABSTRACT

The present work deals with the synthesis of some Al-based composites prepared by mechanical milling and processing by powder metallurgy followed by the evaluation of process conditions as: type of additive, their concentration and milling intensity studying its effect on the characteristics of the powder composite and mechanical performance of the composite. Powder samples were microstructural characterized by electronic microscopy (SEM–TEM) and the mechanical response was followed by hardness and compressive tests. A pronounced effect on the mechanical response of the specimens was evident after the addition of reinforced particles and milling intensity. Microscopy studies showed a uniform dispersion of the reinforcing particles in the metallic matrix at nanometric scale and an important grain refinement of the Al matrix was confirmed. After processing, a 66% increase on the mechanical response was reached with 1% of additive complemented with short milling intensities.

1. Introduction

Today, there is a rising need for advanced materials in order to achieve a better performance for engineering materials that cannot be easily reached using conventional alloys. Thus, production and application of composite materials have increased in recent years [1]. Metal matrix composites (MMC) are engineering materials that contain at least two insoluble components: a metallic matrix (high ductility) and reinforcement particles (high stiffness) in the form of a particulate, short or continuous fiber with a well-defined interface between the matrix and the reinforcement phases [2]. These novel materials have the potential to provide tailored properties that are an average of the matrix and reinforcement properties for specific applications by adjusting the ratio of matrix and reinforcement [3]. Among many metals, aluminum (Al) has been preferred in many cases due to its relatively low density, reasonable mechanical properties, and good workability [4]. Due to its universal applications, AlMMCs are valuable alternatives for demanding industries as: transportation, aerospace, defense, etc., [5] because of the fact, that addition of small amounts of reinforcements particles in the metallic matrix, offers an important increase of mechanical properties compared to their unreinforced counterparts [6,7]. Some processes have been used for composites manufacture, which can coarsely be divided into three types: (a) liquid-state methods (pressure infiltration, stir casting, spray deposition and in-situ processing), (b) semi-solid state methods (compocasting) and (c) solid-state methods (powder metallurgy, mechanical alloying). The first method involves melting and casting processes, however, this method was unable to give good homogeneity of dispersions in metal matrix because of the high interfacial energy between the molten metal and dispersoid [8]. It is extremely difficult to obtain a uniform dispersion of components especially in viscose molten alloys in semi-solid techniques where particles have high clustering tendency [9]. It is possible to produce a fine and homogeneous distribution of hardening particles with a very fine particle size in the metallic matrix [2] using the solid-state or mechanical milling (MM) route (via high-energy ball milling), avoiding the reinforcement-particles clustering [10]. This is important due that fine dispersion of the strengthened phase in the Al structure has a preponderant effect on mechanical properties of prepared composites [11]. The amount that the dispersoids strengthen the final composite depends of their specific characteristics as: particle type, size, morphology, volume fraction and their physical distribution. Also, formation of good interface between matrix and reinforcement is very important [6] and plays a key role on the overall composite performance. The technological challenge is to increase the bonding strength of the interphase by controlling manufacture process based on some particle features as size, morphology and volume fraction. Oxides, carbures, nitrides as silicon carbide, boron carbide [12], titanium carbide [13] alumina [9,14] and hard metals such as titanium and tungsten have been used as reinforcement for aluminum composites [15] but for some

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applications, these materials are not appropriate due to their high cost, use of additives and complicated processing conditions. On the other hand, natural graphite (Gr) has received significant attention for its low chemical activity, low density, high melting temperature, excellent tribological properties, high availability and especially low cost. In order to improve the interfacial bonding between components, a metal coating with Cu, Ni, and Co, has been demonstrated to be a feasible method to solve this inconvenience [5], also can inhibit interfacial reaction under high temperatures during the fabrication process [16]. The present research deals with the synthesis of some Al–Gr composites, the effect of three different metals as Cu, Ni and Ag on the graphite and Al/MeGr interphase and the evaluation of process conditions as: type of additive, their concentration and milling intensity on the characteristics of the processed powders and their mechanical performance.

2. Experimental procedure

The studied composites were formulated using as raw materials pure powders of aluminum (99.5% purity and −44 μm, in size) and metallized graphite (MeGr) as reinforcing phase. Pure elemental powders of natural graphite (99.9%, −85 + 180), copper (99.5%, −100), silver (99.95%, −150) or nickel (99.8%, −44) were used for the MeGr preparation. Mixtures of Metal–Graphite powder mixtures (where Metal = Cu, Ag or Ni) with a concentration of 10 at.% metal were prepared and processed using a high-energy SPEX 8000M mill under an inert Ar atmosphere, selected milling intensities was 0, 1, 2, 4 and 8 h. The structure of Ni, Cu and Ag coatings was evaluated by X-ray diffraction (XRD) analysis. Al–MeGr composites preparation was carried out milling mixtures of pure Al powder and MeGr particles with a concentration of 1.0 (wt.%) during five time intervals (0, 1, 2, 4 and 8 h) as shown in Table 1 (due to space restriction, only the better mechanical results of each family are showed). Methanol was used as a process control agent in order to avoid excessive Al agglomeration. Pure Al samples were used as a reference for comparison proposes. Cold-consolidated forms were obtained from the powders using a two-step pressing method: pressing the powders in a circular die (900 MPa) in one direction and rotating the die to press the form in the opposite direction in order to obtain an ideal compaction level (this condition was calculated using density–pressure curve using a universal machine and plotted the curve searching for an asymptotic zone [17]. Sintering process was carried out with a heating ramp of 10 K/min until 723 K for 3 h under an Ar atmosphere. The morphology, size and metal-particle distribution of milled powder was examined using a JEOL-JSM 7201F SEM/EDS. X-rays diffraction (XRD) analysis was carried out using a Pan Analytical X'pert PRO X-ray diffractometer with Cu Kα radiation. Rockwell F hardness was measured on polished samples using a 1/16″ ball at a load of 60 kg and converted to Vickers scale. At least five hardness measurements were done and the average is reported. Compression tests were done at room temperature in an Instron Universal tester and 0.2% yield strength (σ_y) was measured.

3. Results and discussion

3.1. Microstructural characterization

The Fig. 1 shows the effect of milling on the general morphology of pure Al powder. As-mixed sample (Fig. 1a) presents semi-spherical particles, typical of gas-atomized metals. Milled products exhibit particles with higher size compared with an un-processed sample (0 h) due to Al ductile characteristics. After 4 h of milling (Fig. 1b), the metal particles are plastically deformed by high-energy collisions between grinding media and container. Morphology changes from spherical to irregular with large aggregates formed at the expenses of trapped minuscule particles are evident. Further milling (8 h) hardened large particles lead to fracture process activation (Fig. 1c), forming flake-like sub-particles on the material surface.

The Fig. 2(a–c) shows the morphology of pure Gr particles and the effect of milling on its physical characteristics. Contrarily to the ductile behavior of Al, Gr is fragmented into tiny pieces as a consequence of high impact forces and defoliation evidence is observed after 8 h of milling (Fig. 2c).

The Fig. 3a shows the morphology of CuGr metalized graphite particles; it is evident that particles are micrometric in size exhibiting a laminar structure. Bright zones correspond to zones with high metal concentration (Cu) surrounded by defoliated graphite layers, as high-magnification micrograph shows (bottom square). EDS analyses reveal the existence of an interphase composed of a mixture of Cu–Gr with a heterogenic composition creating an interlapping of elements as Shehata et al. described in their work [8]. In Fig. 3b a STEM micrograph shows the distribution of Cu and C in an isolated particle, graphite signal shows a rich distribution of carbon on the particle surface from one side to the center; while Cu signal is low reaching a maximum in the central part of the particle, indicating the presence of a metal core inside. Similar behavior was found with AgGr and NiGr particles. A possible mechanism involves the homogenization of spatial distribution of reinforcement particles at initial stages of milling by the control

Table 1
Nomenclature of metalized graphite and prepared composites.

Description	Nomenclature	Composition	Milling time (h)	Example
Milled Gr	Grxh	Pure graphite	x = 0, 1, 2, 4 and 8	Gr2 h[a]
Metallized Gr	MeGrxh	Graphite + Me Me = Cu, Ni, Ag.		CuGr4 h[b]
Composite Al–Gr	Al–1%MeGrxh–M4	Al + Metallized Gr Al + MeGrxh	M = 0, 1, 2, 4 and 8	Al–1%AgGr8 hM4[c]

[a] Gr milled 2 h.
[b] Cu/Gr milled 4 h.
[c] Al + 1% (wt.%) Ag/Gr milled 8 h and the mixture milled 4 h.

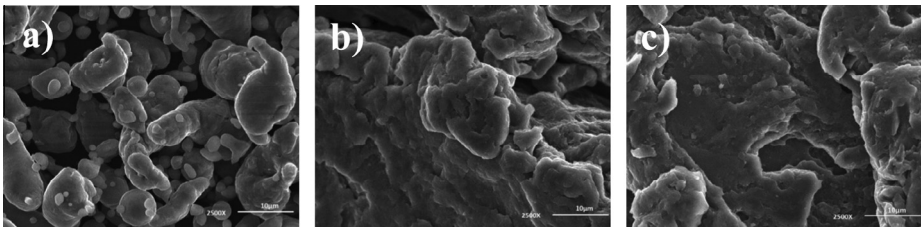

Fig. 1. SEM micrograph (2.5 Kx) of pure Al after (a) 0, (b) 4 and (c) 8 h of milling.