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Original Research Paper

Mechanical properties of Al–Mg/Al_2O_3 nanocomposite powder produced by mechanical alloying

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ABSTRACT

The effect of milling time on the microstructure and mechanical properties of Al and Al–10 wt.% Mg matrix nanocomposites reinforced with 10 wt.% Al_2O_3 during mechanical alloying was investigated. Steady-state situation was occurred in Al–10 Mg/10Al_2O_3 nanocomposite after 20 h, due to solution of Mg into Al matrix, while the situation was not observed in Al/10Al_2O_3 nanocomposite at the same time. For the binary Al–Mg matrix, after 20 h, the average crystallite size was 25 nm. Up to 20 h, the lattice strain increased to about 0.67% for Al–Mg matrix.

By milling for 10 h the large increase in microhardness (190 HV) for Al–Mg matrix nanocomposite was caused by grain refinement and solid solution formation. From 10 to 20 h, slower rate of increasing in microhardness, and at 20 h micro hardness reach (201 HV).

1. Introduction

Interest on powder metallurgy (P/M) aluminum metal matrix composites (MMCs) is increasing, since there is a potential field of applications in aerospace, chemical, transportation, structural and automotive industries. P/M aluminum MMCs have improved strength, high elastic modulus, increased wear resistance, low density, and high stiffness over conventional base alloys [1,2]. High energy ball milling is a simple and useful technique for attaining a homogeneous distribution of the inert fine particles within a fine grained matrix [3]. During ball milling two essential processes occur, cold welding between the different particles and fracturing of the cold welded particles due to high energy collision [4]. The cold welding minimizes the diffusion distance between the atoms of the different components. The fracturing of the welded particles impedes the clustering of the particles promoting the transfer of the high ball collision energy to all particles and produces new, clean surfaces without oxide layers accelerating the diffusion [4,5]. The most important advantage of this method with respect to other alloying methods is feasibility of addition of alloying elements for improvement of mechanical and physical properties of alloys.

Ceramic nano-particles have received great attention owing to their property advantages over conventional coarse grained counterparts [6–8]. Among various reinforcements, Al_2O_3 is one of the most widely used dispersoids in Al-based composites. There are some publications about the production of this type of composite using the mechanical milling method. They are successfully incorporated microscale Al_2O_3 particulates in pure Al matrix using the mechanical milling process [9–13]. However, there are only few works on the effect of the addition of nanometric particulates on the structural and morphological behavior of Al powders.

Al–Mg alloys as matrix are interesting because of their high specific strengths and good corrosion resistances [14,15]. Mechanical alloying has been used previously for the preparation of a range of Al–Mg alloys with bulk Mg concentrations varying from 3 to 70 at% [14–16]. The equilibrium solid solubility of Mg in Al at room temperature is only about 1 at% [17], indeed, the solubility of Mg in aluminum has been increased substantially by the mechanical alloying process [18].

Therefore, the objective of present work is the investigation of effect of milling time on morphological, microstructural and microhardness changes of Al/10 wt.% Al_2O_3 and Al–10 wt.% Mg/10 wt.% Al_2O_3 powder mixtures during the mechanical alloying process.

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2. Materials and experimental procedure

2.1. Materials

Commercial pure aluminum powder with an average particle size of 80 μm and 99.5% purity, Al_2O_3 (99.5% purity 80 μm size) and Mg powder of 99.98% purity with average particle size of (100 μm) were used as raw materials for composite fabrication.

2.2. Ball milling

Pure powder were milled to form Al–$x$$Al_2O_3$, ($x$ = 5, 10 wt.%) composites in a Fritsch planetary ball mill, while confined in sealed 250 ml steel containers rotated at 250 rpm for 20 h. in order to study the effect of Mg add, pure Al was milled with Mg and Al_2O_3 for different milling time 2, 5, 10, 15 and 20 h to form Al–xMg/10AL_2O_3 nanocomposite. The container was loaded with a blend of balls (φ = 10, 20 mm). The total weight of the powder was about 25 g and the ball to powder mass ratio was about 20:1. Stearic acid (3 wt.%) was used as the process control agent to prevent excessive cold welding of powder particles.

2.3. Morphological analysis

The powders produced after milling were investigated by using SEM Model Quanta 250 FEG (Field Emission Gun) attached with EDX unit (Energy Dispersive X-ray Analyses), with accelerating voltage 30 kV. Morphology, size and particle distribution were examined of the milled powders were quantified by visual basic software using several SEM images and their morphology was characterized by scanning electron microscopy.

2.4. Structural evolutions

X-ray diffraction (XRD) patterns were carried in a Rigaku-DXR 3000 X-ray diffractometer using Cu Kα radiation (λ = 0.15406 nm) at 30 kV and 30 mA settings. The XRD patterns were recorded in the 2θ range of 20–70° with a step size of 0.02° and a scanning rate of 1.5 degs./min.

The crystallite size and lattice strain of milled aluminum powders were estimated by XRD peak broadening using William–Hall method as follows [19]:

$$B\cos\theta = \frac{.9\lambda}{D} + 4\varepsilon\sin\theta \quad (1)$$

where B, λ, θ, D and ε are full width at half maximum (FWHM), the wave length, peak position, crystallite size and lattice strain, respectively.

2.5. Microhardness

Microhardness measurements using a Vickers indenter at a load of 250 mN and dwell time of 5 s was conducted on the powder in order to determine hardness of the powder. For measurement of microhardness, the powder mixtures were cold pressed under 25 MPa. Prior to indentation, the surfaces of the samples were polished using a sequence of increasing grit sand paper followed by a series of diamond pastes.

3. Results and discussion

3.1. Morphology

Fig. 1 shows the SEM micrograph for Al–10 Mg/10Al_2O_3 nanocomposite powders mechanically alloyed for different milling Times. At the beginning of milling (from 2 to 5 h), the powders are still soft and cold welding predominates. Consequently, the particles size increases Fig. 1(a and b) Particles shape has become flattened due to cold working effects during milling. Fig. 1c shows that after 10 h of milling, particles have become more rounded in shapes and finer in average size. That's may be because of the predominant processes at this stage are cold working and fracturing with probably some recovery. Powder particles after 15 h seem equiaxed with almost the same size, but some coarse particles still remain as shown Fig. 1d, after 20 h of milling a balance is established between the cold welding and fracturing events and a steady-state situation is obtained as shown in Fig. 1e. It means that the distribution of Al_2O_3 particles in the Al–Mg matrix is very uniform at this stage [13]. And Fig. 1f shows the high magnification of Fig. 1e and also, the average particle size of Al_2O_3 particles after 20 h milling. The cold worked structure of Al matrix with high dislocation density provides suitable conditions for diffusion of bigger Mg atoms (Mg atoms have a larger atomic radius than Al atoms) along dislocation lines into Al lattice [5]. Additionally, the decreased particle size during milling reduces the diffusion distances between particles and facilitates diffusion. Diffusion is further aided by the increased defect density and a local rise in temperature (at Al and Mg interfaces). The combination of these effects would permit sufficient diffusion to occur in the interfacial regions of the nanocrystalline grains to form solid solutions [20]. Fig. 2 shows the effect of Mg add on the powder shape after 15 h milling as discussed earlier.

3.2. Structural analysis

X-ray diffraction patterns of Al–10 Mg/10Al_2O_3 powder mixtures for different milling times are shown in Fig. 3. With increasing the milling time to 20 h the Al_2O_3 lines reduced in height due to the reduction of alumina particles to submicron sizes and/or the low volume fraction of the Al_2O_3 phase [21]. Also Mg peaks disappeared completely, which indicates the formation of the FCC supersaturated solid solution of Al–Mg ($Al(Mg)_{ss}$)due to the diffusion of Mg atoms into the Al lattice. This was inferred by the clear shift of the Al peaks toward lower angles as shown in Fig. 3b, due to the solution of the larger Mg atoms into the Al matrix. Moreover, neither the presence of Al–Mg intermetallics nor unalloyed Mg was revealed from the diffraction pattern, which suggests completed solid solution formation. The results were in good agreement with previous findings [16,18,22,23], in which only Al–Mg solid solution was present in alloys up to 30 at.% Mg. Furthermore milling to 20 h leads to broadening of the $Al(Mg)_{ss}$ peaks and decreasing of their intensities, which indicate reduction in crystallite size and accumulation of heterogeneous strain in the materials.

Fig. 4 shows the effect of milling time on the crystallite size of composite powders. Up to 10 h, the crystallite size decreases rapidly to about 70 and 43 nm for Al/10Al_2O_3 and Al–10 Mg/10Al_2O_3 composites respectively and from 10 to 20 h slow grain refinement occurs. Increase of crystal defects such as point defects and dislocations was caused by Plastic deformation of powder particles during milling [19,24]. Moreover for each milling time, the crystallite size for Al–Mg matrix was lower than for Al. It can be due to the formation of Al–Mg solid solution with higher work hardening effect than pure Al which leads to the crystallite size reduction during milling and facilitation of nanostructure formation. The decrease in the crystallite size with milling time is attributed to dislocation generation caused by severe plastic deformation [20,25]. Also in Al–Mg systems, the Mg concentration has a strong effect on the defect structure. With increasing nominal Mg content of the powder mixture the dislocation density increases [5].

Fig. 5 showing the effect of milling time on the lattice strain of composite powders. Up to 10 h the lattice strains increase rapidly