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Synthesis of nanostructured Al–Mg–SiO_2 metal matrix composites using high-energy ball milling and spark plasma sintering

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ABSTRACT

Mechanical alloying by high-energy ball milling is successfully used to produce a metal matrix composite of Al–Mg reinforced with amorphous silica particulate. Four different compositions are chosen with varying Mg content (0.5, 1, 2.5 and 5 by wt.%) by keeping SiO_2 content constant at 5 wt.% to make nanocomposites by high energy ball milling and microcomposites by mechanical mixing. No new phases are found in 20 h mechanically alloyed Al–Mg–SiO_2 metal matrix composite. XRD study showed Mg is completely dissolved into the Al matrix. XRD observation also showed decrease in crystallite size and increase in lattice strain with progress of mechanical alloying. SEM micrographs indicate decrease in particle size via fracture and cold welding phenomena. The powders are made in the form of cylindrical pellets of 20 mm diameter by Spark Plasma Sintering. X-ray diffraction analysis of the pellets obtained after sintering indicates the evolution of $MgAl_2O_4$ spinel structure along with Al_2O_3. Vickers hardness values observed for nanocomposites are more than twice as high as that of microcomposites.

1. Introduction

Metal matrix composites (MMCs) have been developed to meet the specific engineering properties which cannot be achieved by monolithic material. Different types of reinforcement in form of particulate, whiskers, or fiber have been used to alter the properties of MMC for specific application. One of the important composites which have received enormous attention is aluminum metal matrix composites (AMCs) reinforced with particulates. Different types of particulates such as SiC, Al_2O_3, AlN, TiB_2 and TiC dispersed in commercial Al alloy have been studied for interfacial effects to improve wetting and decrease degradation of reinforcement [1]. Processing techniques such as powder metallurgy, spray deposition and various casting techniques, namely, squeeze casting, rheocasting and compocasting [2] have been used to produce MMC. Ceramic reinforcement to matrix material can either be done by ex situ [3] or in situ [4] method depending upon the processing route. One of the important drawbacks of ex situ MMCs is the interfacial reaction between reinforcement and matrix resulting in poor wettability and bonding [5]. To overcome this, in situ process has been widely recognized because of its advantages such as formation of thermodynamically stable reinforcements in the matrix, clean reinforcement–matrix interfaces resulting in a strong interfacial bonding, finer particle size of reinforcement yielding better mechanical properties and potential for lower cost of production [4].

It is well known that properties of MMC are controlled by size and volume fraction of reinforcement and matrix material [4]. Enhanced mechanical properties are observed when dimension of the reinforcement is reduced to make it thermodynamically stable and homogenously distributed in matrix material [6]. Mechanical alloying (MA) processes have been widely used to produce nanostructured materials and MMC [7,8] followed by sintering to make bulk nanostructured MMC [9]. In the present investigation attempt is made to synthesize Al–Mg reinforced with amorphous silica particulate by varying Mg content (0.5, 1, 2.5 and 5 by wt.%) to form MMC. Spark plasma sintering (SPS) is used to form bulk microcomposites and nanocomposites. Vickers microhardness study is done on both microcomposites and nanocomposites to understand the effect of structure on hardness.

2. Experimental procedure

High energy ball milling is carried out in a planetary ball mill (Fritsch pulverisette P-5) at room temperature using WC vials and balls as milling media and toluene as process controlling agent (PCA) for the four different compositions as shown in Table 1. The materials used in this study are 99.7% pure Al powder, 99% pure Mg powder and 99.8% pure amorphous SiO_2 powder with a particle size of <45 μm (325 mesh). The milling speed and ball-to-powder weight ratio are maintained at 300 rpm and 10:1, respectively. Samples taken out of the vial at regular intervals of 5 h for X-ray diffraction (XRD) analysis using a *PANalytical X'pert-PRO* diffractometer with Cu Kα (λ = 1.54 Å) radiation. Single peak approximation method is used to determine crystallite size and lattice strain by drawing Williamson–Hall plot [10]

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Table 1
Composition profiles of alloy systems considered for study.

Sample identity	wt.% aluminum	wt.% magnesium	wt.% silica
Sample 1	94.5	0.5	5.0
Sample 2	94.0	1.0	5.0
Sample 3	92.5	2.5	5.0
Sample 4	90.0	5.0	5.0

taking correlation coefficient as 0.9. Diffraction pattern for annealed sample is used to correct instrumental broadening by assuming Gaussian line profile and measuring full width half maxima (FWHM). Acquisition conditions are 40 kV and 30 mA. Scans are obtained typically from 20 to 100° 2θ, with step size of 0.01° 2θ, with a count time of 10 s. The microcomposite (as mixed) and nanocomposite powder obtained after 20 h of MA are consolidated by spark plasma sintering (SPS) technique using SYNTEX Inc. Dr. Sinter-5000 series, MODEL: 625 SPS machine. Powders are consolidated to fully dense pellets of 20 mm diameter and 10 mm thickness using graphite punches and die. The SPS is carried out at 723 K (heating rate of 373 K/min) for 5 min under a vacuum of 10^{-2}–10^{-3} Torr. Uniaxial pressure of 50 MPa (max.) is applied to the powder mass throughout the SPS cycle when the desired sintering temperature is attained. The total processing time from loading to ejection of sintered pellet from the die of SPS machine is less than 30 min. Density of the SPS pellets are measured using distilled water as medium according to Archimedes principle. The density data revealed 99.2% of the theoretical density achieved by SPS. The Vickers hardness, HV, of the sintered micro and nanocomposites are determined by microindentation (Mitotoyo HM122) on sample surfaces polished down to 1 μm, applying loads of 50, 100, 200 and 300 g, respectively for 15 s of dwell time in each quadrant. Minimum 8 indentations are measured in order to have a representative mean value of the hardness. Microstructural characterizations of MA powders are investigated using JEOL JSM-6380A Scanning Electron Microscope (SEM) and Philips CM12 Transmission electron microscopy (TEM).

3. Results and discussion

XRD results of composition Al–0.5%Mg–5% SiO_2 are shown in Fig. 1(a) for milling time 0, 5, 10, 15, and 20 h. XRD pattern shows a decrease in peak intensity and an increase in peak broadening with progress of milling time. The Mg peak could not be detected in 0.5, 1.0 and 2.5%Mg samples (Fig. 1(a)–(c)) due to its small amount (i.e. less than 5 wt.%). In contrast, it could be detected in Fig. 1(d) where Mg content is 5 wt.%. After 5 h of milling, Al peaks intensity decreases and peak positions remain unchanged for all the compositions studied (Fig. 1(a)–(d)). This indicates the amount of Mg diffused into the Al is insignificant. After 5 h of milling, peak broadening is due to decrease in crystallite size and increase in lattice strain in the powder mixture. The variation in particle size as a function of milling time is studied using SEM as shown in Fig. 2(a)–(e). Increase in particle size is seen for the 5 h MA samples, which could be due to cold welding phenomenon resulting in formation of agglomerates [7] as clearly seen from Fig. 2(b). Due to severe plastic deformation of agglomerates further reduction in size is seen as shown in Fig. 2(c)–(e). At the initial stages of MA, Al and Mg existed as individual particles which appear to be of dimension of ~45 μm, and as the alloying progresses the less ductile Mg particles became embedded in ductile Al particles, resulting in the formation of a composite particle that enhances reduction of agglomerate size to ~10 μm (Fig. 2(c)). SiO_2 introduction in the Al–Mg alloy forms a diffusion couple [11]. The SEM images for initial and intermittent time interval clearly shows the progress of MA, which signifies repeated welding and fracture phenomena.

It is also seen from the XRD patterns that there is a continuous decrease in peak intensity with increase in peak broadening indicating reduction in crystallite size and increase in lattice strain with progress of milling. These observations are shown in Fig. 3(a)–(d). The sample with 0.5%Mg showed (Fig. 3(a)) that the average crystallite size of the Al phase decreased progressively with milling time

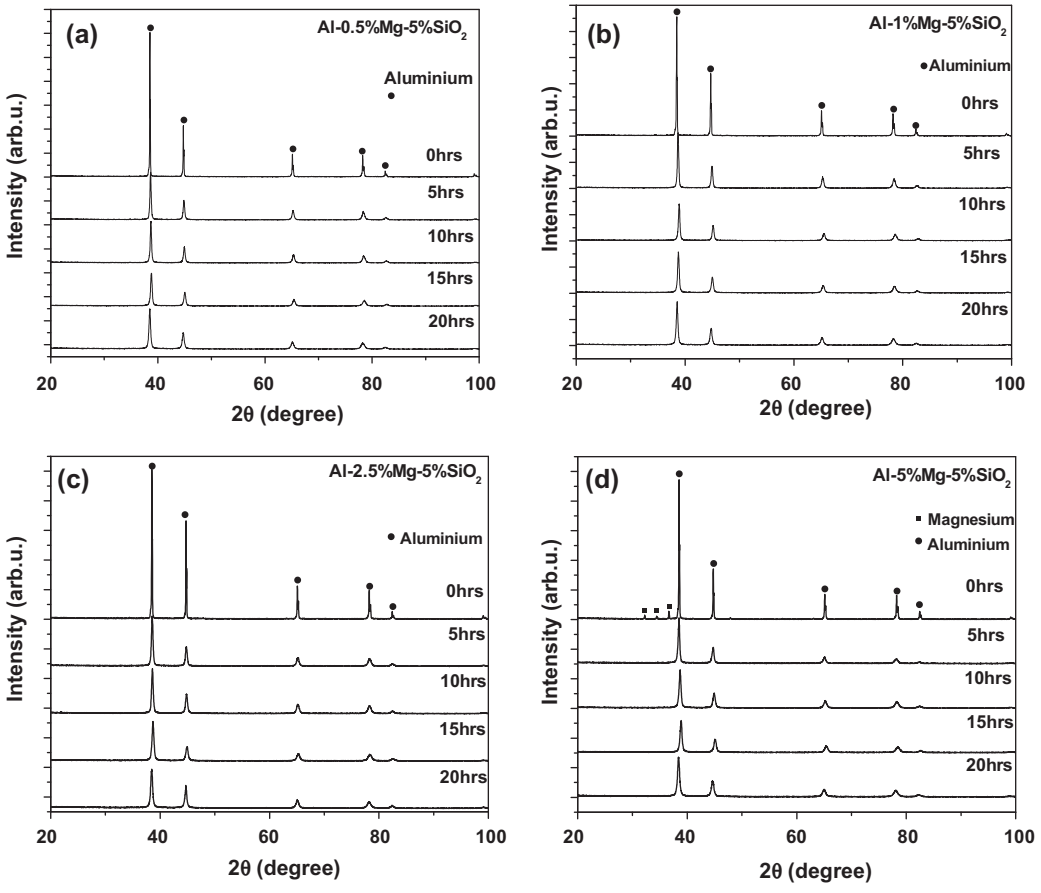

Fig. 1. XRD profiles of MA powders corresponding to varying Mg contents by wt (a) 0.5%, (b) 1%, (c) 2.5%, and (d) 5%.