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Investigation of microstructure, hardness and wear properties of Al–4.5 wt.% Cu–TiC nanocomposites produced by mechanical milling

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ABSTRACT

The present work deals with studies on the manufacturing and investigation of mechanical and wear behavior of aluminum alloy matrix composites (AAMCs), produced using powder metallurgy technique of ball milled mixing in a high energy attritor and using a blend–press–sinter methodology. Matrix of pre-mechanical alloyed Al–4.5 wt.% Cu was used to which different fractions of nano and micron size TiC reinforcing particles (ranging from 0 to 10 wt.%) were added. The powders were mixed using a planetary ball mill. Consolidation was conducted by uniaxial pressing at 650 MPa. Sintering procedure was done at 400 °C for 90 min. The results indicated that as TiC particle size is reduced to nanometre scale and the TiC content is increased up to optimum levels, the hardness and wear resistance of the composite increase significantly, whereas relative density, grain size and distribution homogeneity decrease. Using micron size reinforcing particulates from 5% to 10 wt.%, results in a significant hardness reduction of the composite from 174 to 98 HVN. Microstructural characterization of the as-pressed samples revealed reasonably uniform distribution of TiC reinforcing particulates and presence of minimal porosity. The wear test disclosed that the wear resistance of all specimens increases with the addition of nano and micron size TiC particles (up to 5 wt.%). Scanning electron microscopic observation of the worn surfaces was conducted and the dominant wear mechanism was recognized as abrasive wear accompanied by some delamination wear mechanism.

1. Introduction

Aluminum-based alloys are widely used as aerospace and automotive components, because of their high specific strength, stiffness and formability. However, both pure Al and Al alloys possess poor wear resistance. On the other hand, Al alloy matrix composites are known to offer better wear resistance and bulk mechanical properties. These composites are synthesized by liquid or powder metallurgy routes. The wear performance of these composites is a subject of strong interest, especially for their potential application in automobile components including cylinder block, piston and brake disks [1]. The wear test is usually conducted under sliding wear conditions using pin-on-disk or block-on-ring tests. Though high volume fractions of hard reinforcements are favored for wear resistance, the wear rate of the counter-body is found to be greatly enhanced by the abrasive action of the reinforcements [2]. Carbides, oxides, nitrides and different intermetallics compounds have been used extensively as reinforcing particulates for AMCs [3]. In specific, SiC and Al_2O_3 were the commonly used ceramic reinforcements and recently NiAl, Ni_3Al and intermetallics composite have been shown to improve the wear resistance of aluminum and magnesium alloys to a level similar to that of SiC reinforced composite, whilst reducing counterface wear rates [4–6]. TiC was not investigated enough as a ceramic reinforcement to Al alloy matrix nanocomposites, however it has been receiving much attention lately for its high melting temperature (3160 °C), low thermal coefficient of expansion, extraordinary hardness, excellent wear and abrasion resistance [7,8]. A decrease of the reinforcement particle size from micrometric to nanometric scale, brings a superior increase in mechanical strength of the composite, but the tendency of particle clustering and agglomeration also increases [9–11]. It is important to note that a homogeneous distribution of the reinforcing particles is essential for achieving the improved properties [12]. Mechanical alloying (M/A) via ball milling has been successfully employed to improve particle distribution throughout the matrix [13–15].

Mechanical alloying is solid-state synthesis process that consists of repeated cold-welding, fracturing, dynamic recrystallization and mechanically activated inter diffusion among the powder particles. A high energy ball mill offers indeed supplementary degrees of freedom in the choice of possible routes for synthesizing

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new materials and appears further as an attractive method of synthesis in view of its potential for large scale production [16]. To obtain an absolute nanocrystalline alloy powder, materials from initial mixtures of elemental powders in a grain size range of micrometers with appropriate weight proportions are charged into a high energy ball mill container. By applying the proper milling condition such as suitable process control agent (PCA), ball to powder ratio (BPR) and time of milling, the specific alloy will be achieved in solid state. Recently, a number of $Al_{65}Cu_{20}TM_{15}$ or $A_{l50}TM_{40}Si_{10}$ (TM = early transition metals like Ti, Nb and Zr) alloys have been developed by mechanical alloying with amorphous and/or nanocrystalline matrix and in situ nano-intermetallic phase/compound dispersion [6,17,18]. These composites have exhibited nearly 2–3 times greater wear resistance and hardness than the conventional microcrystalline and age hardenable Al alloys [19–21]. The general characteristics and mechanisms of mechanical milling and alloying have been studied, but the influence of milling parameters on mechanical properties of composites is not yet fully known [22]. Wear behavior of these novel mechanically alloyed Al alloys or composites has not been investigated fully [2].

The present study has focused on the manufacturing of MMC's using a nanocrystalline alloying matrix for the desired composite with the composition of Al–4.5 wt.% Cu, which according to the previous study was prepared through mechanical alloying using a high energy attritor ball mill to produce the nanostructure matrix powder of the composites [23]. Different concentrations of nano and micron size TiC particles as reinforcement were added to the matrix to improve the hardness and wear resistance of the MMCs, proper distribution of the reinforcing particles in the matrix is very important in blending process which is measured through precise EDX analysis [4,6], then the effect of different weight fractions of reinforcement on the wear resistance, hardness, relative density and microstructure of a new generation nanocrystalline matrix composites are investigated. An effort has also been made to understand the operative wear mechanism.

2. Experimental procedure

Pre-alloyed powder of the composite matrix produced by mechanical alloying technique with nominal stoichiometry of Al–4.5 wt.% Cu (M) and particle size in range of 10–20 μm with a flake like morphology, typical of mechanical alloyed particles were used. The chemical composition of the matrix is shown in Table 1, according to previous study to produce the alloying matrix, pure Al and Cu elemental powders with average particle size of 150 and 40 μm respectively were subjected to mechanical alloying in an attrition ball mill. Milling was operated for 70 h at the rotational speed of 270 rpm with 12:1 ball to powder weight ratio (BPR) and 1.5 wt.% stearic acid was used as the milling process control agent (PCA), using stainless steel vial (AISI 316) and hardened chromium steel balls (6 and 10 mm diameter with the ratio of 1:2). The powder in the vial was protected with high purity argon gas and the milling tank was cooled with a fix flow of cold water through a rotating system. Samples for analysis were removed by interrupting the ball mill at various intervals (each 10 h). Alloying behavior, solution and crystallite size evolution during milling were determined by X-ray diffraction (XRD) analysis using a Philips (30 kV and 25 mA) diffractometer with Cu Kα radiation (λ = 1.5405 Å). All XRD experiments were done with a step size of 0.02° and a time per step of 0.1 s. Crystallite size of powders was evaluated using the Williamson–Hall and Sherrer methods [23,24]. Since both Al and Cu are ductile materials, the dominant mechanism that occurs through mechanical alloying of these elements is indeed ductile–ductile which results in formation of lamellar structure of repeated flattened layers of aluminum and copper in each grain of the powder particles [24].

In order to study the effect of reinforcing particulate amount and size on the hardness, sliding wear behavior and the microstructure of the composites, different weight percent of nano-size (0%, 0.5%, 1%, 3%, 5% and 7 wt.%) and micron size (0%, 5%, 7% and 10 wt.%) TiC particulates were added to the prepared alloy matrix powder and blending was performed for 2 h in a planetary ball mill at nominal room temperature and at a rotation speed (cup speed) of 400 rpm with 12:1 ball to powder weight ratio using tungsten carbide (WC) coated vials and hardened chromium steel balls in four different sizes (20, 15, 10, 6 mm diameter) under Ar atmosphere and applying 1.5 wt.% stearic acid as the milling PCA [24]. The microstructure and distribution of the milled TiC powders and the compacts were studied by CamScan MV3200 scanning electron microscope and supplemental EDX.

The same equipment was employed to characterize the milled powder morphology and the topology of the surfaces of compact samples. The as-received and milled powders were consolidated into compacts using cold and hot unidirectional pressing. First, 4 g of powders were measured and poured into in a cylindrical tool steel die and were cold pressed. Fig. 1a shows the scheme of the die with an inner diameter of 10 mm. Powder mixture was sustained for 10 min in the pressure of 650 MPa then according to Fig. 1b by adjusting an auto-controlled power of the heating system, the compacted samples were hot pressed at 400 °C under pressure of 500 MPa for 80 min. Then they are allowed to cool to room temperature and stripped off the die for characterization and mechanical tests. Density values of compact samples were measured applying three different methods of geometrical, Archimedes and theoretical approaches. Vickers microhardness method was used to determine the hardness of consolidates using a Mitutoyo microhardness tester. The VHN measurements were conducted at 50 gf for 10 s of dwell time. A minimum of five indentations were measured per condition.

The dry wear tests were performed according to ASTM standard G99 [25] on a unidirectional pin-on-disk aperture wear tester with a counter face of AISI 52100 steel which was hardened to 62 HRC at ambient temperature, the cylindrical shape pins with the dimensions of 10 mm in diameter and 15 mm in length were prepared by machining the consolidated samples for the tests. Prior to wear tests, the pins and disks were polished/grinded with 800 grit SiC paper to a 1 μm finish. Samples were cleaned thoroughly using an ultrasonic device with acetone and dried prior and after the tests and then weighed to determine the wear loss using an electronic balance (GR200-AND) with an accuracy of 0.1 mg. All data on wear were measured from the pins. The volume loss of samples, according to Eq. (1), was calculated through the mass loss divided by the Archimedes density of the materials and the friction coefficient (μ) values were measured by a load cell equipped with the pin-on-disk apparatus and the wear rate values of the samples were calculated for all samples according to Eq. (2).

The morphology of the worn surfaces was analyzed using similar SEM, the same test parameters were used for all samples: 10

Table 1
Chemical composition of the alloying matrix.

Materials	Si	Fe	Cu	Mn	Mg	Cr	Ni	Zn	Ti	Al
Chemical composition (wt.%)	0.01	0.012	4.542	0.003	0.002	0.02	0.001	0.01	0.004	Base