Journal of Alloys and Compounds 726 (2017) 358–366

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: http://www.elsevier.com/locate/jalcom

Microstructural evolution in ultrafine grained Al-Graphite composite synthesized via combined use of ultrasonic treatment and friction stir processing

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ARTICLE INFO

Article history:
Received 26 December 2016
Received in revised form
6 June 2017
Accepted 27 July 2017
Available online 29 July 2017

Keywords:
Ultrasonic treatment
FSP
Severe plastic deformation
Hardness
Recrystallization

ABSTRACT

An attempt was made to develop ultrafine grained Al-Graphite composites through friction stir processing (FSP). Al-Graphite composite was initially prepared using stir casting technique and it subsequently subjected to ultrasonic treatment prior to solidification. The results showed that the ultrasonic treatment during the composite preparation reduced the porosity, graphite particles' agglomeration and the graphite particles' size in the composite. The ultrasonic treated Al-Graphite composite was subjected to a single pass FSP at a constant tool rotational speed of 1600 rpm with three different feed rates, of 20 mm/min, 40 mm/min and 60 mm/min. The microstructure of FSPed composites within the stir zone exhibited a fine recrystallized grain structure due to the combined effect of severe plastic deformation and frictional heat generation during FSP. After the single pass of FSP, the grain size of the composite was reduced from 150 μm to 14 μm, 6 μm and 0.5–1 μm for the tool transverse speeds of 20 mm/min, 40 mm/min and 60 mm/min, respectively. Distribution of graphite particles in Al matrix was significantly improved after FSP process due to the strong stirring action of the rotating tool. Most importantly, microstructural analysis suggested that a substantial reduction in graphite particles' size could be achieved with the aid of FSP process. FSPed Al-Graphite samples exhibited superior hardness as compared to the unprocessed composites due to the uniform dispersion of fine graphite particles in ultra-fine grain structure. In a nutshell, ultrafine grained Al-Graphite composites with uniform dispersion of Graphite nano particles were produced using a single pass FSP process.

1. Introduction

Metal Matrix Composites (MMC) are one of the noteworthy novel class of materials which exhibit superior mechanical properties such as higher stiffness and hardness, good wear and erosion resistance [1]. Aluminum based MMCs are widely used in automobile and aircraft applications in virtue of its high strength to weight ratio, greater stiffness, improved heat resistance and good tribological characteristics [2]. Presence of hard reinforcements such as SiC in the aluminum matrix decreases the machinability and ductility of composites to a large extent which restricts its applications [3]. Graphite is a well-known high strength and low dense material which can be easily deformed due to its soft nature. Hence, incorporation of graphite in aluminum matrix is expected to improve the ductility and toughness of the Al based MMCs thereby their range of applications can be extended. Furthermore, the soft nature of graphite particles can offer an antifriction property to Al based MMCs which is essential for automobile parts such as bearings and pistons.

Currently, liquid metallurgy route is a preferred method for the production of Al MMCs because of its simplicity and lower cost. Moreover, it has the potential to scale up the technology for large scale applications. Agglomeration of reinforcement particles is one of the main drawbacks associated with Al based MMCs which is hard to avoid during solidification processing of the composites. Studies on Al MMCs suggest that the inhomogeneous distribution of reinforcement particles affects the mechanical properties such as

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http://dx.doi.org/10.1016/j.jallcom.2017.07.280
0925-8388/

strength [4–7] ductility [8,9], fatigue [10,11] and fracture behavior [12,13]. In recent times, ultrasonic treatment is reported as one of the effective methods to break the agglomerates of reinforcement particles and for the reduction of porosities of MMCs [14–16]. Recently, Christy et al. [16] have reported that the ultrasonic treatment of Al-2vol. % graphite composites has significantly reduced the agglomerates, porosities and the graphite particles' size.

Severe plastic deformation (SPD) is a promising technique to produce an ultra-fine grain structure in materials [17–19]. An investigation on high pressure torsion (HPT) processed Al–7 wt% Si alloy reveals that the ultra -fine grain structure has been obtained with an applied pressure of 8 GPa for 10 revolutions at room temperature [20]. It is reported that, Al-6061- 10 vol.% Al_2O_3 composites processing through high pressure torsion (HPT) has shown significant grain size reduction from ~35 μm to ~170 nm [21]. Ultra-fine grains with an average grain size of 230 nm have been achieved in Al-Al_2O_3/B_4C hybrid composites by using an accumulative roll bonding (ARB) technique [22]. Roohollah Jamaati et al. have found that, an ARB technique on Al/15 vol % Al_2O_3 composites has generated nano-grains with an average size of less than 100 nm [23]. Interesting finding such that, aluminum/copper multilayered composite which was fabricated via ARB process exhibits Cu and Al layers with an average grain size of 50 nm and 200 nm respectively [24]. The microstructural evaluation study of Al–7Si–Mg alloy by ECAP process has shown ultra fine structure grains after four passes [25]. Saravanan et al. [26] have fabricated ultra-fine grained Al-Graphite composite using equal channel angular processing (ECAP). Their research work on multipass (4 passes) ECAP of Al-Graphite composites has resulted grain refinement in submicron level (300 nm). FSP is an alternate route which can be used to fabricate ultrafine grained material successfully [27–29]. In the past few years, numerous aluminium alloys and their composites have been developed with ultrafine grain microstructure using friction stir processing [30–32]. A study of FSP on A6082/Al_2O_3 composites after four passes exhibits more uniform distribution of nano sized Al_2O_3 particles in ultra-fine grains of aluminium matrix [33]. Dolatkhah et al. have found that FSPed Al5052/SiC is attained an ultrafine grained structure with an average grain size of 0.9 μm from an initial average grain size of 243 μm [34]. Previous investigations suggest that the mechanical performance of the composites processed via FSP can be influenced by reinforced particle size, morphology and its distribution as well as grain size of matrix [35,36]. Thus, it is imperative to study the detailed microstructural analysis of FSPed composites. However, the studies on FSP of Al-Graphite composite are rather very limited [37–39]. Moreover, the information regarding microstructural evolution during FSP of Al-Graphite composites has not been reported in detail in the existing literature. Hence, the aim of the present research work is to develop an ultra-fine grained Al-Graphite composite with uniform distribution of graphite particles through the combined use of ultrasonic treatment and FSP. The microstructural evolution during ultrasonic treatment and FSP of Al-Graphite composite is also investigated.

2. Experimental details

Pure aluminum (99.8%, EC Grade Aluminum) and graphite powders with average particle size of 21 ± 4 μm were used to synthesize Al-5 vol.% graphite composite by stir casting technique. After adequate stirring of the melt, the preheated ultrasonic probe was dipped into the melt and sonicated for 5 min. A high power ultrasonic probe (Hangzhou Success, China), made of Ti-6Al-4V, was used to generate a 20 kHz and 2.5 kW power input for ultrasonic treatment process. After ultrasonic treatment, the composite melt was poured into a cast iron mold having the dimensions of 100 × 100 × 400 mm^3. For comparison purpose, the composite sample without ultrasonic treatment was also cast.

Samples of Al- 5 vol.% graphite composites with the dimensions of 100 × 100 × 6 mm^3, were electrode discharge machined (EDM) from the ultrasonicated sample and subjected to single FSP process at room temperature. FSP processing was carried with a tool made of high carbon high chromium D3 type steel with shoulder diameter, pin diameter and pin length of 18 mm, 6 mm and 3 mm, respectively. A schematic diagram of FSP tool consisting of cylindrical shoulder and a straight cylindrical pin is shown in Fig. 1. A single pass FSP was carried out at a constant tool rotational speed of 1600 rpm with different feed rates of 20 mm/min, 40 mm/min and 60 mm/min. Microstructure of ultrasonic treated and FSPed composites were analyzed using polarized light optical microscope (Carl Zeiss Axio Scope A1). The size and distribution of graphite particles in the Aluminium matrix were analyzed using Field Emission Gun Scanning Electron Microscopy (FESEM, Carl Zeiss, Sigma, UK) operated under Back Scattered Electron (BSE) mode. Grain structure and size of the graphite particles in the FSP processed samples were analyzed using JEOL JEM 2100 Transmission Electron Microscopy (TEM) operating at 200 kV. Samples for TEM analysis were prepared by standard methods involving mechanical grinding, polishing and dimpling followed by ion milling. Daksh HV100DT microhardness tester was used for hardness analysis in the FSPed samples. A load of 200 g and a dwell time of 10 s were used for measuring the hardness of the material. Minimum of 15 measurements were carried out at every region (HAZ, Transition and Stir Zone) and the average value was reported.

3. Results

Fig. 2 (a) and 2(b) compare the FESEM micrographs of stir-cast and ultrasonic treated Al-graphite composite. Microstructure of stir-cast composite shows a significant amount of porosity along with the agglomeration of graphite particles [Fig. 2(a)]. For the processing of Al MMCs using stir-cast technique, the molten aluminum is mechanically stirred to create a vortex, which draws the reinforcements together with the ambient gases into the liquid medium. Since, the interface energy for particle/gas interface is lower than the particle/metal interface, entrapped gases prefer to attach with reinforcement particles. This could be the possible reason for the presence of agglomerated graphite particles in the vicinity of porosity. In the case of ultrasonic treated composite, a uniform dispersion of graphite particles with significant reduction in porosity is observed [Fig. 2 (b)]. It is also noticed that (Fig. 2 b) the

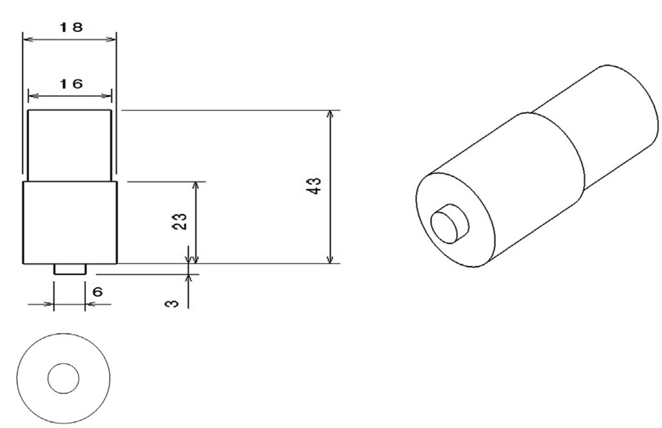

Fig. 1. Dimensions of the FSP tool.