Materials Characterization 140 (2018) 172–178

Contents lists available at ScienceDirect

Materials Characterization

journal homepage: www.elsevier.com/locate/matchar

Microstructure evolution and properties of graphene nanoplatelets reinforced aluminum matrix composites

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ARTICLE INFO

Keywords:
Aluminum metal matrix
Graphene nanoplatelets
Microstructure
Interface
Tensile strength
Electrical conductivity

ABSTRACT

Aluminum matrix composites reinforced with graphene nanoplatelets (GNPs) were prepared by continuous casting and subsequent rolling. Al-GNPs master alloy, prepared by ball milling and cold pressing, was added into Al melts to fabricate the composites. Microstructure evolution of the composites were observed using scanning electron microscopy (SEM), transmission electron microscopy (TEM) and electron back scattered diffraction (EBSD). GNPs distributed uniformly in the as-cast specimen and then transformed into fibers in the rolling direction after deformation and finally a lamellar structure formed in the composites. Raman spectra showed that the structural damage of GNPs mainly came from ball milling. The interface between GNPs and Al was well combined in general while a few microcracks were observed, which reduced the ductility of the composites and no adverse aluminum carbide (Al_4C_3) was detected at the interface. Stacking faults were observed interior of the aluminum grain, which may due to the existence of plentiful interface introduced by GNPs. Fracture observation revealed that the load transferred from Al matrix to GNPs. The ultimate tensile strength of Al-0.2 wt% GNPs composites was about 36.8% higher than that of pure Al with the same casting and rolling process, which should result from the lamellar structure and load transfer, while the conductivity of the composites decreased slightly, indicating that interface scattering between Al and GNPs is very limited. The investigation results show that Al-GNPs composite is potential for high strength and high conductivity application.

1. Introduction

Graphene with excellent mechanical properties and electrical performance shows great potential as reinforcement of metal matrix composites to obtain high strength and high conductivity. Graphene reinforced aluminum matrix composites are promising materials requiring high strength coupled with high conductivity, especially for aluminum wires and cables for power transmission. Previous studies demonstrated that the addition of few-layer graphene [1], graphene oxide [2], or graphene nanoplatelets (GNPs) [3,4] has significant improvement in mechanical properties of Al [5,6], Mg [4], Ti [7,8] and Cu [9] if the reinforcement was uniformly distributed. However, it is difficult to disperse graphene in molten aluminum because of its large specific surface area and poor wettability between graphene and aluminum melts. Ball milling is an effective way to distribute particles in the metal matrix [10] and was employed in the previous researches on graphene/Al composites combined with hot pressing [11,12], hot rolling [13] and mechanical alloying [14]. However, all these methods are complex and costly, which limits their industrial application. Liquid metallurgy was used to be considered impossible due to the large density difference and adverse interfacial reaction between carbon and aluminum melts at high temperature. Much focus has been put into the researches on casting methods, such as stir casting, pressure infiltration and induction smelting [15], however, satisfactory results on industrial scale have not been reported yet.

In the present study, ball milling and cold press were also used, not for composites but for Al-GNPs “master alloy”, which was added into aluminum melts as the GNPs carrier. GNPs were chosen because of its easier producing process and maintaining the characteristics of two-dimensional materials meanwhile [5]. Continuous casting and subsequent rolling were employed for the fabrication of Al-GNPs composites. Microstructure evolution, mechanical properties and electrical conductivity of the composites were investigated to reveal the distribution and contribution of GNPs in the composites.

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https://doi.org/10.1016/j.matchar.2018.04.007
Received 13 November 2017; Received in revised form 1 March 2018; Accepted 6 April 2018

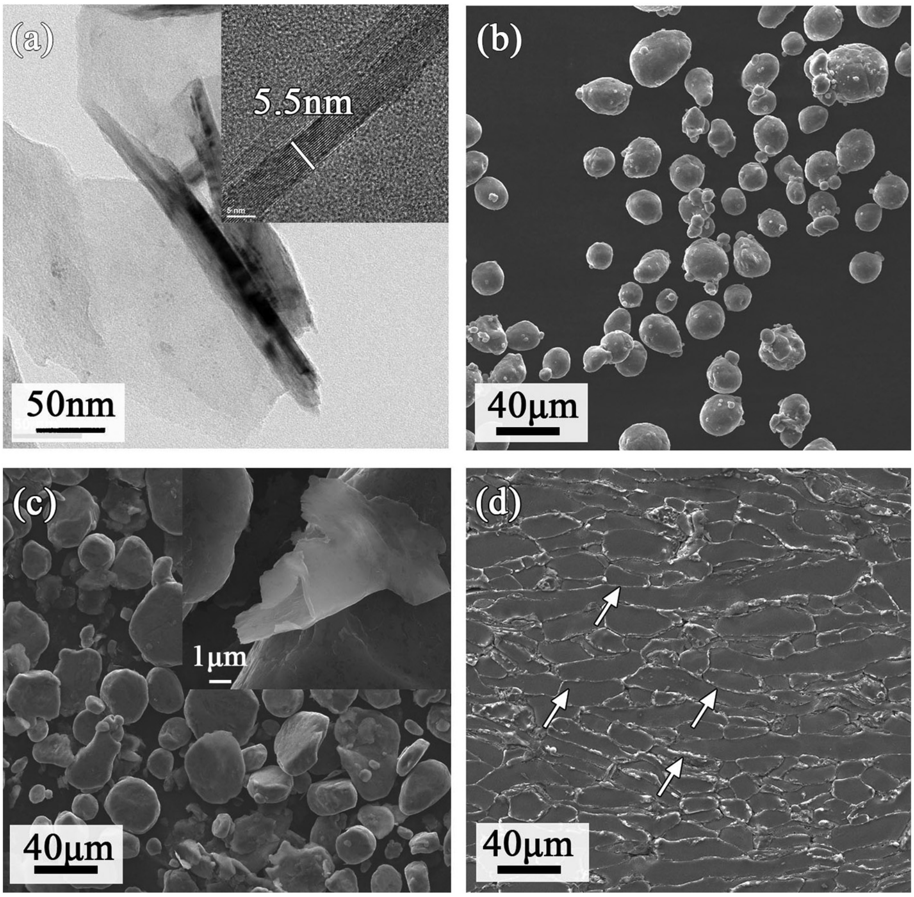

Fig. 1. (a) TEM image of overlapped GNPs powders before milling, (b) SEM image of spherical Al powders before milling, (c) ellipsoidal Al powders with GNPs coated after milling and (d) mixed powders after SPS showing the uniform distribution of GNPs.

2. Experimental

Aluminum powders (99.99% in purity) and GNPs powders were used to fabricate Al-GNPs master alloy. Fig. 1(a) shows the image of GNPs powders obtained by TEM. The GNPs are overlapped due to high surface energy. High resolution TEM (HRTEM) observation revealed that the thickness of GNPs was about 5.5 nm, which corresponds to about 15 stacking layers, as shown in the insert in Fig. 1(a). Al powders with 2 wt% GNPs were ball milled for 4 h under argon atmosphere, with a speed of 300 rpm, and the ball-to-powder ratio of 5:1. Fig. 1(b) and (c) show SEM images of the Al powders and milled Al-GNPs powders, respectively. Spherical Al powders with the average size about 30 μm in diameter (Fig. 1b) transformed to ellipsoid (Fig. 1c) after ball milling. However, GNPs could hardly be distinguished by SEM because it is transparent. Few graphene could still be seen attached to the surface of aluminum powders, as shown in the insert in Fig. 1(c). A small portion of the Al-GNPs powders were consolidated by spark plasma sintering (SPS) with the pressure of 50 MPa at 500 °C. It is worth noticing that SPS was not used for the preparation of composites, but just to make clear the distribution of GNPs. As shown in Fig. 1(d), there are no obvious agglomerations and the surface of each aluminum powder is surrounded by GNPs after ball milling, which clearly indicated that GNPs should distribute almost uniformly after milling. The mixed Al and GNPs powders were then cold pressed into compacts with the size of 28 mm in diameter and 25 mm in height. The cold pressed mix powder was called Al-GNPs master alloy and was added into aluminum melts heated with electromagnetic induction furnace with a mass proportion of 1: 9. Finally, Al-0.2 wt% GNPs composite rods with a diameter of 9.5 mm were obtained by continuous casting and subsequent rolling. 9.5 mm pure Al rods were also prepared for comparison with the same casting and rolling process.

Microstructure of the Al-GNPs composite rods were observed by SEM (JSM7600F) equipped with energy-dispersive spectroscopy (EDS), TEM and HRTEM (JSM2100F) and EBSD (FEI NOVA NanoSEM). Raman spectroscopy (LabRAM HR Evolution) was used to investigate the structure of GNPs. The specimens for SEM, Raman and EBSD analyses were cut from the longitudinal section of the 9.5 mm rods. Mechanical and electrolytic polish in a solution of 10% perchloric acid and 90% ethanol at room temperature were used for specimen preparation. Samples with a diameter of 3 mm for TEM observations were reduced to around 40 μm by mechanical polishing and then further thinned by ion milling with a Gatan Model 691 precision ion polishing system.

Tensile specimens were machined along the rolling direction with the size of Φ9.5 × 200 mm. The average tensile strength of ten tests was recorded using Zwick/RoellZ020. The microhardness of polished samples was measured using a Digital Metallic Vickers Hardness tester with a 200 g load and 10s dwell time. At least ten points were measured to obtain an average value. Five conductivity measurements were recorded using a FD-102 eddy current conductivity tester.

3. Results and discussion

3.1. Microstructure

The microstructure of as-cast Al-0.2 wt% GNPs composites observed by SEM showed no significant difference with that of pure Al. However, it is interesting to find that fibers arrange along the rolling direction after continuous rolling, as shown in Fig. 2(a). To determine the chemical compositions of these fibers, EDS line scan across the fiber was conducted, as shown in Fig. 2(b) and (c). The intensity of element