



Formation and heat treatment of kinetic sprayed nanocrystalline Al coatings reinforced with multi-walled carbon nanotubes: The relationship between microstructural features and physical properties



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ARTICLE INFO

Article history:

Received 6 August 2015

Revised 14 January 2016

Accepted in revised form 20 January 2016

Available online 22 January 2016

Keywords:

Metal-matrix composites (MMCs)

Multi-walled carbon nanotube (MWCNT)

Kinetic spraying (cold gas dynamic spraying)

Heat treatment

Electrical properties

Mechanical properties

ABSTRACT

Nanocrystalline (nc) Al matrix composites reinforced with multi-walled carbon nanotubes (MWCNTs) (1.0 and 3.0 vol.%) were fabricated by mechanical attrition and consolidated using a kinetic spraying process, followed by heat-treatment at a homologous temperature of $0.67 T_M$ for 1–4 h. Microstructural features including thermal stability of the nc structures, the interface between MWCNTs and the Al matrix, and the MWCNT structures of as-sprayed and heat-treated MWCNT/nc Al composite coatings were characterized and related to their corresponding mechanical and physical properties. The relationship between microstructural features and properties of the composite coatings was investigated.

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1. Introduction

Multi-walled carbon nanotubes (MWCNTs) have proven to be versatile reinforcing agents for metal matrix composites. The unique physical and mechanical properties of MWCNTs including their high thermal [1] and electrical [2] conductivity, superior elastic modulus [3], and tensile strength [4] serve to improve the properties of various metal matrixes (i.e. Al [2,5–10], Cu [11], Ni [12], and Ti [13] as well as bulk metallic glasses [14]). In the case of Al, ductility is extremely good but strength is poor, compared to a variety of other metals. In order to improve the mechanical and physical properties of Al, numerous studies have been performed related to the fabrication of Al matrix composites and the evaluation of their properties using Si [15], SiC [16], B₄C [17], and Al₂O₃ [18] particles and precipitation [19]. Especially, homogeneously dispersed MWCNTs can be used to improve the mechanical properties of Al and its conductivity through the use of high aspect ratio and nano-sized (diameter: 20–60 nm) MWCNTs. MWCNTs possess relatively inferior properties, compared to single-wall carbon nanotubes (SWCNTs). However, the industrial practicality of MWCNTs is much better than that of SWCNT in terms of price. Hence, research with regard to MWCNT/Al composite has recently grown steadily. Since uniformly dispersed MWCNTs in metal matrix have been an issue [20], several techniques including the sintering of

solid mixtures [2,9], melt stirring [10], and thermal spraying [6] have been applied toward the fabrication of MWCNT/Al composites. However, these techniques are limited due to either localization or the destruction of MWCNTs. In our earlier works, MWCNT/Al composite powders were subjected to high velocity oxygen fuel (HVOF) spraying [25,26]. The loss and destruction of embedded MWCNTs and the innate defects of lamellar structure degraded the measured properties of the composite coatings [25,26].

On the other hand, mechanical ball milling has emerged as a fabrication technique for MWCNT/Al composites since uniform dispersions and strongly embedded MWCNTs have been achieved and demonstrated via this process [5,21]. Mechanical ball milling can also be used to transform micro-size grained Al to nanocrystalline (nc) Al [16,17,22–24]. Applying the technical merits of mechanical ball milling, the dispersion of enfolded MWCNTs and the formation of an nc Al matrix were simultaneously achieved in this study. In general, nc metals are characterized by their high strength, ductility, and toughness compared to conventional polycrystalline metals, since a relatively large volume fraction of atoms located in the grain boundaries yields a large physical property difference to nc materials [22–24]. Thus, synergetic effects from a combination of MWCNTs and nc Al matrix are expected to result in improved mechanical and physical properties.

Additionally, kinetic spraying (KS) (or cold gas dynamic spraying) is appropriate to fabricate a deposit of thermally-sensitive materials (i.e. MWCNT/nc Al composite), which can be damaged by the thermal spraying heat source. KS is a coating technique in which deposition

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proceeds through the solid-state bonding of micro-size particles. Fine particles are accelerated to supersonic velocities by the drag force of a supersonic gas flow and the particles are successively impacted onto a substrate. Due to the high kinetic energy of the impacting particles, particles can experience a severe plastic deformation under an extremely high strain-rate (1.0×10^6 – $0.5 \times 10^9 \text{ s}^{-1}$) [27–30]. In this state, heat energy converted from plastic deformation is not transferred into the external system, as it is accumulated within the deforming particle under adiabatic conditions. This adiabatic heating induces shear instability at the interface (i.e. drastically increases the strain and temperature) [27–30]. Since no significant thermal energy is supplied to the composite powder during KS, transformation of the Al matrix nc structure and thermal damage to the MWCNTs does not occur.

Several papers exist which are related to the investigation of CNT-involved composite deposition using kinetic spray process [31–36]. With regard to recently published studies, matrix materials were primarily Cu as opposed to Al, which was appropriate to be deposited via KS process [31–33]. In these studies, CNT-involved Cu matrix composite were fabricated yielding good heat-transfer property, when applied to industrial products (i.e. boilers, plate heat exchangers, etc.). The deposition state of CNT-involved Cu matrix composite and their microstructure was examined. On the contrary, KS deposition of Al matrix CNT composite was rarely investigated relative to the case of Cu. S.R. Bakshi et al. reported on MWCNT–Al composite layers fabricated by KS process [34,35]. The deposition states of the agglomerated composite powder were primarily investigated with focus on the mechanical response of MWCNTs to supersonic impact. Microstructural evolution and related properties were not profoundly reported. On the other hand, in our earlier study, the strengthening mechanism of kinetic sprayed MWCNT–Al composites with a correlation between microstructure and mechanical properties being elucidated [36].

Although several studies with regard to CNT-involved composite have been conducted, there are still many areas worth investigating to further develop CNT-involved composite in the field of kinetic spraying. In this study, MWCNTs/nc Al composite coatings were fabricated and heat-treated at a homologous temperature of $0.67 T/T_M$ for 1–4 h. Microstructural features (nc grain stability, the interface between the MWCNT and Al matrix, and MWCNT states) of the as-sprayed and heat-treated composite coatings were characterized, and compared to a pure Al coating counterpart. Microstructural features were significantly related to the mechanical and physical properties of the as-sprayed and heat-treated composite coatings. Strengthening mechanisms and the conductivity contribution of the composites were discussed as related to the obtained microstructural features. This study serve to provide an improved understanding of microstructures and properties of composite coatings, opening up a variety of potential applications for MWCNT/nc Al composite coatings.

2. Experimental

2.1. Composite powder preparation

MWCNTs and pure Al (purity: 99.9%) powders were used for the fabrication of composite powders. The MWCNT diameters were approximately 20–50 nm as shown in Fig. 1a. Fig. 1b shows a HREM image of the outer walls of a MWCNT where the MWCNT possess the typical bamboo structure. The outer wall interspacing was 3.34 Å, which was identical to that of the basal plane in graphene. The MWCNTs did not exhibit any other phases such as amorphous carbon or impurities. The pure Al powder was spherical with a particle sizes range of $-82 + 23 \mu\text{m}$ (mean: $60 \mu\text{m}$) (Fig. 2a and d).

The MWCNTs and Al powders were blended for 24 h via blending machine. The blended quantities of MWCNTs were 1.0 and 3.0 vol.%. The blended powders were mechanically ball-milled using a high-energy planetary ball milling machine. Stainless steel balls (5 mm diameter) were used as the ball milling media. The ball to powder weight ratio was 15:1. Ball milling was performed for 10 h with a rotational speed of 200 rpm in a stainless steel container. Fig. 2b and c show the morphologies of the fabricated MWCNT/ns Al composite powders with 1.0 and 3.0 vol.% MWCNTs, respectively. The morphologies were irregular and rock-shaped with the size of the composite powders ranging $-100 + 20 \mu\text{m}$ (mean: $65 \mu\text{m}$).

2.2. KS and heat treatment

In this study, a commercially available KS system (KINETIC 3000, CGT) with a de Laval type tungsten carbide nozzle was utilized. Equipment and kinetic coating process details have been previously published [27–30,37–39]. In this study, three process conditions (C1, C2, and C3) were employed to evaluate the deposition behavior of MWCNT/nc Al composites at different impact velocities. Details of the KS process parameters are summarized in Table 1. Al 1050 alloy was used as the substrate material. Prior to deposition, the substrate surface was polished.

KS aluminum coatings were heat-treated at $350 \text{ }^\circ\text{C}$ ($0.67 T/T_M$) for 1, 2, 3, and 4 h using a vacuum furnace (vacuum degree: 10^{-2} Pa – 10^{-4} Pa) to protect the coatings from oxidation. The heating rate was $5 \text{ }^\circ\text{C min}^{-1}$ and the samples were cooled within the furnace.

2.3. Analysis

The microstructures of the KS coatings were analyzed with a field emission scanning electron microscope (FE-SEM) (JSM-7500F, JEOL). To clearly observe the microstructure of the coating cross section,

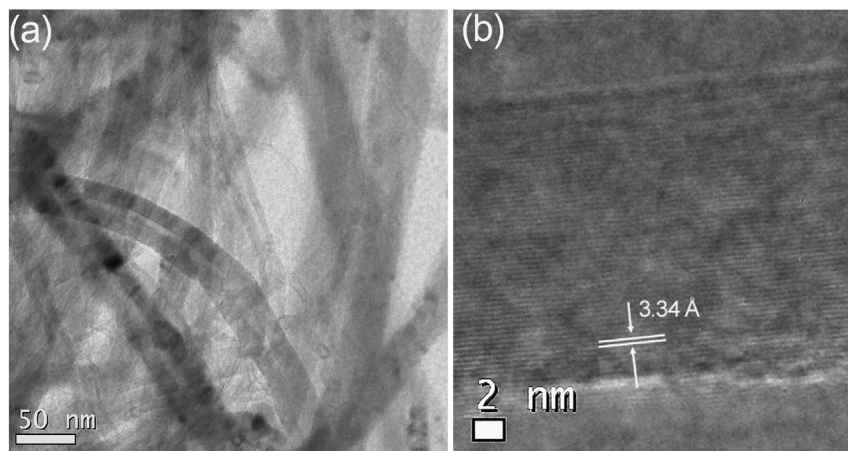


Fig. 1. (a) TEM BF image of initial MWCNTs, and (b) HREM image of MWCNT walls.

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