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Microstructure and mechanical property of multi-walled carbon nanotubes reinforced aluminum matrix composites fabricated by friction stir processing

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ABSTRACT

Aluminum matrix composites reinforced by different contents of multi-walled carbon nanotubes (MWCNTs) were fabricated by friction stir processing (FSP). The microstructure of nano-composites and the interface between aluminum matrix and MWCNTs were examined using optical microscopy (OM) and transmission electron microscopy (TEM). It was indicated that MWCNTs were well dispersed in the aluminum matrix throughout the FSP. Tensile tests and microhardness measurement showed that, with the increase of MWCNT content, the tensile strength and microhardness of MWCNTs/Al composites gradually increased, but on the contrary, the elongation decreased. The maximum ultimate tensile strength reached up to 190.2 MPa when 6 vol.% MWCNTs were added, and this value was two times more of that of aluminum matrix. Appearances and fracture surface micrographs of failed composite samples indicated that the composites become more and more brittle with the increase of the MWCNT content.

1. Introduction

Since the discovery of Iijima in 1991 [1], carbon nanotubes (CNTs) have attracted more and more attentions for their good electrical and thermal characteristics, extraordinary strength, as well as their high elastic modulus values [2,3]. These properties are favorable to the applications of CNTs in aerospace and automotive industries where energy saving becomes increasingly important. Both the near-perfect structure and the strong sp^2 bonding between the C–C bonds contribute to an elastic modulus of CNTs as high as 1 TPa and a tensile strength as high as 50 GPa [4,5]. All these make CNTs an ideal strengthening additive in fabricating composites. Aiming at getting higher specific strength and thermal conductivity, as well as lower coefficient of thermal expansion, a number of investigations have been carried out to fabricate CNTs/metal matrix composites. Many methods have been explored in previous researches, including powder metallurgy [6–8], spark plasma sintering [9,10], plasma spray forming [11,12] and semi-solid powder processing [13]. However, all these methods are faced with a key challenge which is how to obtain a uniform dispersion of CNTs in the matrix, especially when the content of CNTs is high [14,15].

Friction stir processing (FSP) is a solid-state forming process which was developed based on the principles of friction stir welding (FSW) techniques invented by The Welding Institute (TWI) in 1991 [16,17]. During FSP, a rotating tool pin is plunged into the surface of metal part to be processed and then traverses along predetermined paths. Heat is generated through the friction between the rotating tool pin, the shoulder and metal surface. The temperature of metals does not reach its melting point and only plastic deformation occurs in the processing zone. After the moving of tool pin, recrystallization occurred in the severely deformed metals, resulted in the refined and homogeneous grain structure in the stirred zone [18,19]. If strengthening additives are introduced into holes or grooves on the surface of the metal matrix before FSP, they will be involved into the stirred zone by the stirring of the tool pin and will be dispersed into the metal matrix. For this reason, FSP is a good way to incorporate strengthening additives into metal matrix to fabricate bulk composites. Recently, FSP has been applied to fabricate metal matrix composites reinforced with CNTs [20,21]. For example, Morisada et al. [22] inserted MWCNTs into the grooves which were premachined on the AZ31 plates, and then subjected the plates to FSP. A 50% improvement in hardness of matrix was found after incorporating MWCNTs by FSP. However, the tensile strengths of composite were not reported.

In the present study, FSP was used to fabricate aluminum matrix composites reinforced with MWCNTs. And the microstructure of nano-composites and the interface between aluminum matrix and carbon nanotubes were investigated. In addition, the influences of the MWCNT content on mechanical properties of composites as well as its strengthening mechanism were discussed.

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2. Experimental methods

Plates of A1016 commercial aluminum alloy with a desired size of 300 mm × 100 mm and a thickness of 5 mm were used as the substrate materials. Holes with the same depth but different diameters, i.e. six holes with a depth of 3.5 mm and a diameter of 0 mm, 2 mm, 4 mm, 6 mm, 8 mm and 10 mm respectively were drilled by a drilling machine on the surface of the aluminum plates. After filling the holes with various quantities of MWCNTs, these plates were processed by FSP. The volume fraction of CNTs in each CNTs/Al composites was calculated and the FSP samples were denoted as 0 vol.% MWCNTs/Al, 1.6 vol.% MWCNTs/Al, 2.5 vol.% MWCNTs/Al, 4.4 vol.% MWCNTs/Al, 5.3 vol.% MWCNTs/Al and 6 vol.% MWCNTs/Al, respectively. The MWCNTs used in this experiment were obtained from commercial suppliers and were used without any further purification. The diameters and lengths of the MWCNTs were ranging from 10 to 40 nm in diameters and 10–20 μm in length as shown in Fig. 1.

The FSP procedure was schematically shown in Fig. 2. The FSP machine used in this experiment was self-designed and modified from a horizontal-type milling machine. The fixed pin tool was 12 mm in diameter and 7.8 mm in length. The shoulder diameter was 26.8 mm, and a tilt angle of 2° was applied on the fixed pin tool during FSP. The pitch distance was 0.3 mm. During FSP, the forward velocity of the rotating pin was kept as a constant of about 30 mm/min, and a rotational speed of 950 rpm (rotation per minute) was used. Five passes on the same position were conducted on each plate to achieve the uniform distribution of MWCNTs in matrix material.

Microstructure in cross section of each sample was observed by optical microscopy (OM) after etched with Keller's reagent (1 ml HF + 1.5 ml HCl + 10 ml nitric acid + 87.5 ml distilled water). Microstructure of MWCNTs/Al composite, specially the distribution of MWCNTs in Al matrix, was examined using a JEOL 2010 transmission electron microscopy (TEM) operating at 200 keV. TEM samples were prepared by twin-jet electro-polish in a solution of 25 vol.% of HNO_3 and 75 vol.% of methanol at a temperature of −30 °C. The voltage was set as 12 V. Vickers microhardness of the MWCNTs/Al composite sample was measured using a HX-1000 model microhardness tester, with a loading of 100 g and a dwell time of 10 s. Tensile tests were carried out using a WDS-100 universal testing machine with an initial strain rate of 2 mm/min at room temperature. The fracture surfaces of failed tensile samples were examined using a FEI QUANTA200 scanning electron microscope (SEM).

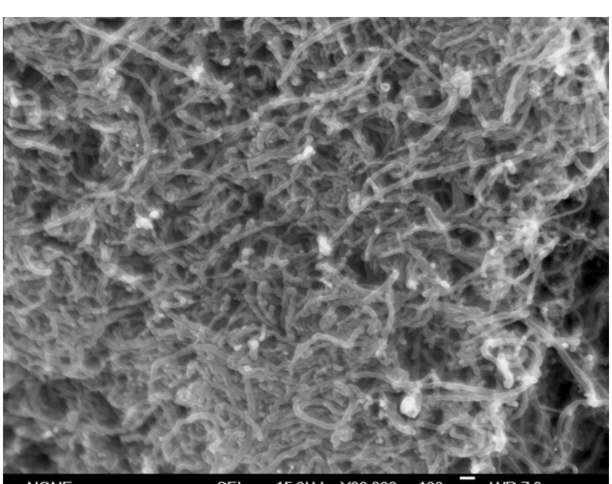

Fig. 1. SEM micrograph of MWCNTs used as the reinforcement in this experiment.

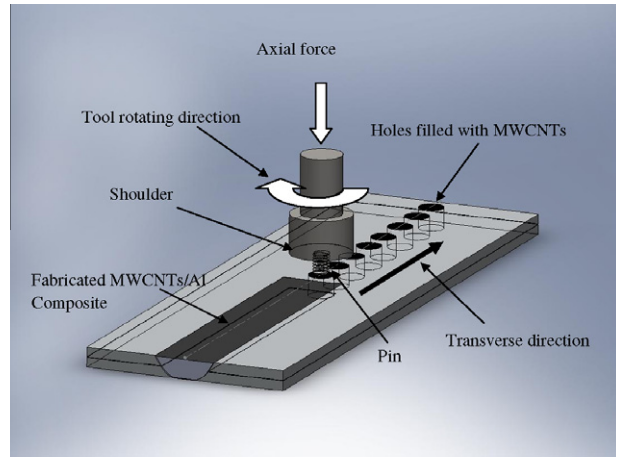

Fig. 2. Schematic of the FSP process of MWCNTs/Al composites.

3. Results and discussion

3.1. Microstructure and MWCNTs survivability

The macrostructure of the friction stir processed area in the sample containing 4.4 vol.% MWCNTs is shown in Fig. 3a, in which four different regions are observed. These regions include unaffected base metal (BM), friction stir zone (FSZ), shoulder deformation zone (SDZ), and thermo-mechanically affected zone (TMAZ), as marked as A, B, C and D in Fig. 3a respectively. It is obvious that the grain size of the FSZ is much finer than that of the BM as shown in Fig. 3b. In the FSZ, as is shown in Fig. 3c, homogeneous and fine equiaxed grains are formed, which is attributed to the dynamic recrystallization of highly deformed grains during FSP [23]. As is shown in Fig. 3d, elongated grains are found in the SDZ, which indicates that recrystallization also occurs in this region for the highly plastic deformation and heat influence caused by the friction of the shoulder and the substrate surface. Layer band structure is formed in TMAZ, which can be shown in Fig. 3e. The layer band structure is formed due to the impact of the thermodynamic cycles, rather than the direct driving of tool pin in this region during FSP.

As shown in Fig. 4, TEM observations indicate that the MWCNTs could be singly dispersed in the aluminum matrix due to intense stirring effect of the rotating threaded pin during FSP. The high magnification TEM image of MWCNTs/Al interface reveals a good interfacial compatibility and wetting exists between the hydrophilic MWCNTs and the aluminum matrix. Intermediate compounds or nano-pores are not detected in the interface. A strong interface between the MWCNTs and aluminum matrix, which can transfer the load effectively, is a significant factor influences the mechanical properties of the composites. Regions with high dislocation density and ultrafine grains (50–100 nm) are detected in composites, which are induced by severe deformation and the dynamic recrystallization of metals during FSP. Moreover, the ultrafine grains are also discovered in other additive reinforced aluminum matrix composites [24,25], and this is considered to be beneficial for the improvement of the mechanical properties of composites. The appearance of high density of dislocations also indicates that the recrystallization process have not been completely finished in the FSZ. Breakages of the MWCNTs are also visible which might be caused by the high compressive forces during FSP, as shown in Fig. 4d.

3.2. Microhardness

Microhardness variations of the FSP MWCNTs/Al composites reinforced with different MWCNT contents have been evaluated