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Strength enhancement by shear-flow assisted dispersion of carbon nanotubes in aluminum matrix composite

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ABSTRACT

A large shear flow under high-ratio differential speed rolling promoted the formation of a novel microstructure in which carbon nanotubes were uniformly dispersed and directionally aligned in the aluminum matrix. As a result, the processed composite exhibited a high yield stress (~400 MPa) and good ductility (~10%).

1. Introduction

Carbon nanotube (CNT) reinforced metal matrix composites (MMCs) attract great attention these days, but improving the properties of MMCs is difficult because achievement of uniform dispersion of CNTs and good interfacial bonding strength between the CNTs and surrounding metal matrix is challenging. Ball milling technique has been found to be effective in CNT dispersion inside the metal powders. In many cases, the compacts sintered from the ball milled powders are subjected to post-sintering deformation processes such as rolling, extrusion, equal channel angular pressing and high pressure distortion [1–4] to enhance the bonding between the CNTs and the matrix, and the homogeneous dispersion of CNTs in the matrix.

The present work focuses on using a combination of ball milling and high-ratio differential speed rolling (HRDSR) as synthesis method of producing the CNT reinforced aluminum matrix composites with high quality dispersion of CNTs. The HRDSR technique emerges as a new severe plastic deformation method with an advantage regarding its suitability for fabricating the large-scale sheets with ultrafine grained microstructure [5,6].

2. Material and methods

The Al-CNT powders were fabricated by mechanically adding pure aluminum powders (99.5% in purity, 100–150 μm in diameter) and 3 vol% multi-walled carbon nanotubes (MWCNTs) (~20 nm in diameter and approximately 10–15 μm in length) in an attrition mill at 400 rpm for 6 h under an argon atmosphere. The ball-to-powder weight ratio was 15:1. The ball-milled powders were placed in a copper tube with an inner diameter of 26 mm and degassed for 1 h at 673 K; then, the ends of the tube were sealed by mechanical pressing. The tube sample was held at 723 K for 20 min and then subjected to symmetric rolling for a thickness reduction to 2 mm in 8 passes. After removal of the copper tube layer, the composite (with thickness of 1.2 mm) was subjected to HRDSR for a single pass, reducing the final thickness to 0.2 mm. The roll temperatures were maintained at 473 K throughout the entire rolling process. In HRDSR, the speed ratio between the upper and lower rolls was set to 2, and no lubricant was used. For comparison, powders without MWCNTs were also prepared under the same milling conditions and subjected to the same rolling procedure.

Densities of the HRDSR-processed pure aluminum and the HRDSR-processed composite were measured by the Archimedes method; they were 99.1% and 98.3% of the calculated values, respectively. To check the possible formation of aluminum carbides during the processing, X-ray diffraction (XRD, Rigaku, Ultima IV) with a Cu Kα radiation source was used. The microstructures of the pure aluminum and the composite were examined using a field-emission TEM (JEM 2001F) operated at 200 kV. Focused ion beam milling was used to observe the normal direction (ND)–rolling direction (RD) and the RD–transverse direction (TD) sections of the composite. Electron back-scattering diffraction (EBSD) analysis was performed on the ND–RD section of the composite using a scanning step size of 70 nm. Raman spectroscopy was undertaken to

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examine the possible structure damage of CNTs during ball milling or HRDSR. Tensile tests were conducted at an initial strain rate of 1×10^{-3} s^{-1}. Tensile specimens with a gauge length of 10 mm were cut out along the RD.

3. Results and discussion

The Raman spectra of the initial CNTs, the composite powders and the HRDSR-processed composite are shown in Fig. 1(a). A relative intensity (I) between the D- and G- bands ($=I_D/I_G$), which provides information about the quality of the internal CNTs [7], was 0.68 for the initial CNTs. This value was increased to 1.48 after ball milling, but further increase did not occur during the subsequent rolling process (1.41). These results imply that in the process of ball milling, CNTs were broken, but they were not further damaged during the rolling process. The XRD patterns of the composite powders and the HRDSR-processed composite are shown in Fig. 1(b). There were no detectable peaks other than those assigned to aluminum in either the powders or composite, indicating that a reaction between the CNTs and aluminum did not occur appreciably during ball milling or rolling. The insets in Fig. 1(a) and (b) reveal that CNTs are well dispersed inside the ball-milled powders and that interface between CNT and the matrix in the composite has a good bonding, respectively.

Fig. 2(a) and (b) shows the TEM micrographs (observed on the RD–TD section) of the HRDSR-processed pure aluminum and composite, respectively. Comparison of the two microstructures indicates that the addition of CNTs refined the aluminum matrix as well as changed the matrix grain morphology. The microstructure of the pure aluminum (Fig. 2(a)) is heterogeneous and consists of small cells outlined by dense tangles of dislocations and equiaxed (sub)grains containing a high density dislocations in their interiors. The size of typical (sub)grains was in range between 0.5 and 2.5 μm. The HRDSR-processed composite (Fig. 2(b)) reveals the much finer and more homogeneous microstructure. Its microstructure consists of elongated grains with length of 200–500 nm and thickness of 100–200 nm. The mean grain size determined using image analysis software was 320 nm. Fig. 3 shows the TEM micrograph (observed on the RD–TD section) of the composite prior to its subjection to HRDSR. Most of CNTs are found in grain interiors (some are indicated by arrows) and they are randomly distributed without preferred orientation. This configuration of CNTs was, however, drastically changed after HRDSR, as shown in Fig. 2(c)). Many CNTs are loosely clustered to form the bands aligned in the direction of the RD and they are preferentially located near or on the boundaries of grains. The CNTs in a band are individually separated without entanglement. The similarity of the distance between the CNT band and thickness of grains suggests that the matrix grain size is determined by the formation and distribution of CNT bands. Fig. 2(d) shows the TEM micrograph of the HRDSR-processed composite observed on the ND–RD section. As in the RD–TD section, the CNTs are highly aligned to the RD and formation of CNT bands is evident. The grain size and interspacing between the adjacent CNT bands are also comparable. In some grains, CNTs are found in their interiors. Shear banding, which occurs intensively and uniformly throughout the sample thickness during HRDSR [6], is believed to have played an important role in achieving the ultrafine-grained microstructure with high-quality dispersion of CNTs. During shear banding, the CNTs initially randomly distributed in the powders move and become aligned to the flow lines almost parallel to the RD. Accumulation of dislocations occurs near the CNTs as they effectively hinder the dislocation motion. The accumulated dislocations turn into grain boundaries via a dynamic recovery process, resulting in the formation of a bamboo-like microstructure followed by its splitting into smaller grains. The uniform dispersion of CNTs is also expected to help to preserve the ultrafine grained structure at the high processing temperatures by increasing its thermal stability.

Fig. 4(a) and (b) shows the EBSD image (observed on the ND–RD section) of the HRDSR-processed composite and the EBSD phase map for graphite, respectively. It is apparent that CNTs are concentrated at grain boundaries and their distribution at grain boundaries is uniform. In some grains, CNTs are found in their interiors (marked as A), which agrees with the TEM observation (Fig. 2(d)). A schematic for the CNT distribution in the matrix of the HRDSR-processed composite drawn based on the TEM and EBSD observations is shown in Fig. 4(c).

Fig. 5 shows that the true stress–true strain curves of the HRDSR-processed aluminum and composite. The yield stress (YS) of the composite (392 ± 3 MPa) is higher than that of the pure aluminum (293 ± 2 MPa) by 34%. It is worthy of noting that the HRDSR-processed composite exhibits the YS value similar to that of the 4 vol% MWCNT reinforced aluminum composite with the grain size of 74.3 nm (about 390 MPa) [8], which was fabricated by hot extrusion of the ball-milled powders, despite its having a lower CNT content and a larger matrix grain size. This result may be regarded as an indication of achievement of higher quality dispersion of CNTs during HRDSR. To explain the gain of strength by addition of CNTs, the modified shear lag model for composites reinforced by short fibers, developed by Nardone and Prewo [9], was used. According to the model, the increase in strength due to

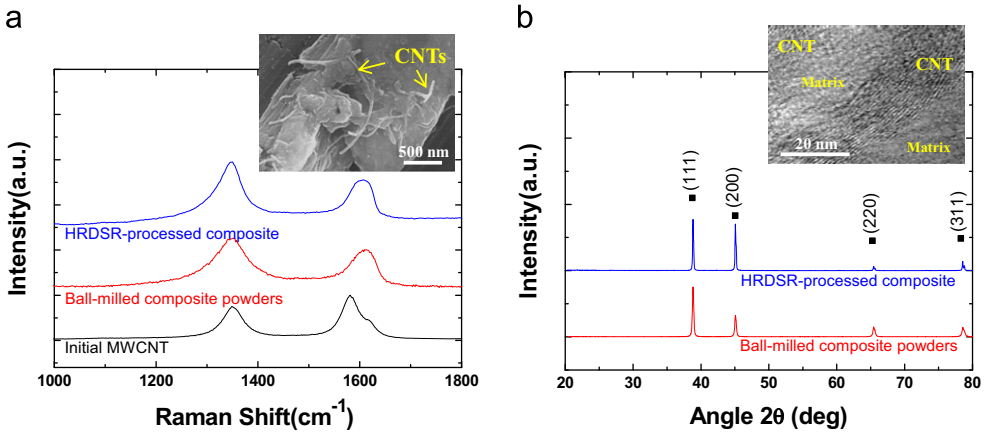

Fig. 1. (a) Raman spectra and (b) XRD of the composite powders and the HRDSR-processed composite. The insets in (a) and (b) shows the CNTs in the interior of the composite powders and the interface between CNT and the matrix in the HRDSR-processed composite, respectively.