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Microstructures and mechanical properties of carbon nanotubes reinforced pure aluminum composites synthesized by spark plasma sintering and hot rolling

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ABSTRACT

In this study, flat products of carbon nanotubes (CNTs) reinforced pure aluminum (Al) composites were fabricated by spark plasma sintering and hot rolling. The effects of sintering temperature and CNTs content on the microstructures and mechanical properties of the composites were investigated. It has been shown that the composite reinforced by 0.75 vol% CNTs and sintered at 630 °C has the best comprehensive mechanical properties, due to the combined positive effects of the good Al-CNTs interfacial bonding, full densification of the composite and the uniform dispersion of the CNTs. It has been found that the load transfer strengthening and dispersion strengthening of CNTs are the dominant strengthening mechanisms in the composites. The study provides a guidance for manufacturing the flat products of Al/CNTs composites with high strength and good ductility.

1. Introduction

Since their first discovery, carbon nanotubes (CNTs) have attracted extensive attention as reinforcements in composites due to the low density (1.2–2.1 g/cm^3) and excellent mechanical properties [1–3]. Based on the applicable literature about the composites reinforced by the CNTs published in the past two decades, it can be clearly seen that the majority of the investigations have been carried out on polymer based composites [4,5]. This might be attributed primarily to the relative easiness of polymer processing, which often does not require high temperature for consolidation as required by metal based composites. In spite of this, it is noted that the interest in CNTs reinforced aluminum (Al) based composites has been growing considerably, due to their promising application prospects in aerospace and automotive industries, where light weight combined with high stiffness and strength is desired. Powder metallurgy (PM) technique, including mixing reinforcements and Al powder, and subsequent sintering, has been the preferred route for fabricating the CNTs reinforced Al based composites [6–9]. Although the incorporation of different contents of the CNTs into the Al powder can be easily realized right now [10–12], the following sintering stage also plays a significant role on the high performance of the CNTs reinforced Al composites.

In general, the conventional sintering methods, such as hot press sintering, have shown obviously negative effects on the microstructures that required for high mechanical performance. On one hand, the relative high sintering temperature used in the conventional sintering for densification causes abnormal grain growth, which is detrimental to the mechanical properties. On the other hand, the high sintering temperature would promote the chemical reaction between the Al and the CNTs, and thus destroy the integrity of the CNTs. The formed brittle Al_4C_3 and imperfect CNTs tend to weaken the interfacial bonding and lower the load transferring efficiency from the Al matrix to the CNTs reinforcements. Recently, spark plasma sintering (SPS) technique became increasingly popular in the fabrication of metal based composites [13–16]. As a novel and rapid powder consolidation process, SPS offers several advantages over the conventional sintering process such as much faster heating and cooling rates [17–19]. Such characteristics can avoid CNTs damage and possible adverse chemical interfacial reactions, and thus can obtain a fine grain sized metal matrix. Additionally, the relative low sintering temperature and short holding time adopted in the SPS procedure can lower the fabrication cost.

Normally, the SPS is hard to obtain a strong bonding strength between the Al powder particles and completely eliminate the residual pores due to the short diffusion time, so it is necessary to strengthen the interfacial bonding and enhance the densification by post deformation. For example, Chen et al. [20,21] used hot extrusion as post-sintering

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processing to enhance the bonding strength between the Al powder particles and the dispersion uniformity of the CNTs in the Al matrix. The traditional trade-off tendency between strength and ductility in metal based composites was evaded in Al/CNTs composites owing to concurrent improvement of Al–Al grains and CNT–Al interfacial bonding by hot extrusion. Zare et al. [22,23] applied equal channel angular pressing (ECAP) as the post-sintering processing to consolidate the fabricated materials. The well-densified composites with only 2 vol% CNTs after eight ECAP passes exhibited an approximately 30% increase in the yield strength compared to the pure Al samples. Considering the increasing requirement of the flat products of Al and Al based composites instead of the bar products provided by hot extrusion or ECAP, it is necessary to pay attention to the microstructures and mechanical properties of the flat products of the Al/CNTs composites. In this paper, we conducted a systematic investigation on the relation between the microstructures and mechanical properties of the Al/CNTs composites fabricated by SPS and hot rolling. The effects of sintering temperature and the content of the CNTs on strengthening mechanism and fracture mode of the composites were discussed.

2. Experimental

Three types of the composites, referred to 0.75CNTs-590, 0.75CNTs-630 and 1.0CNTs-630, were fabricated by SPS and hot rolling using pure atomized Al powder as the matrix and the CNTs as the reinforcements. The details in sintering temperature and volume fraction of the CNTs in the composites were listed in Table 1. The morphologies of the CNTs (~10 nm in diameter and 3 μm in length, supplied by Sigma-Aldrich Co. LLC) and the Al powder were shown in Fig. 1a and b, respectively. It can be seen that severe agglomeration exists in the as-received CNTs, and the Al powder particles have an average size of 2 μm, ranging from 500 nm to 5 μm. Before mixing with the Al powder, the as-received CNTs were ultrasonically treated in ethanol for 1 h to decrease the agglomeration. Then the suspension of the CNTs was mixed with Al powder for 5 h using a 4 planetary ball mill. The rotation speed and the ball to powder weight ratio was the same as that in our previous work [24]. The mixed powders were then dried at 80 °C for 5 h in a vacuum oven to evaporate the ethanol. The stainless steel balls were removed from the mixed powders by 200 mesh sieve. The powder mixture was sintered by an FCT HPD 25/3 spark plasma sintering furnace at 590 °C or 630 °C for 0.5 h in an argon atmosphere, with the externally applied pressure of 30 MPa. Both the heating and cooling rates were 100 °C/min. The size of the sintered samples was 26 mm in diameter and 20 mm in height. These samples were sealed in stainless steel cans for pack hot rolling. The sealed cans containing the sintered samples were held at 550 °C for 0.5 h and were then hot rolled with a height reduction of 75% through 5 passes. Then the stainless steel coat was removed by wire-electrode cutting.

In order to indentify the possible chemical reactions involved in the fabrication process, the phases of the fabricated composites were characterized using a D/max2550pc X-ray diffractometer with Cu Kα radiation (k = 0.154 nm). The bulk densities of the as-sintered and as-rolled samples were measured by standard Archimedes method. The hardness of the samples was measured by Vickers hardness tester. Both the density and hardness results were averaged from at least five independent measurements. The microstructures of the mixed powders, as-sintered and as-rolled samples were investigated by SEM and TEM using an FEI Nova Nano230 scanning electron microscope and a Titan G2 60–300 transmission electron microscope (TEM). Mechanical polishing and ion thinning using a Gatan Precision Ion Polishing System at 2 kV were used to prepare the SEM and TEM samples, respectively. The tensile specimens with a cross-section of 4×3 mm^2 and a gauge length of 8 mm were machined by wire-electrode cutting from the rolled sheets along the rolling direction. The tensile tests were carried out using an Instron 3369 testing machine with a strain rate of 2.1×10^{-3} s^{-1}. The yield strength was determined as the 0.2 pct offset.

Table 1
The details of the sintering temperature and volume fraction of the CNTs.

Composite	Sintering temperature (°C)	Volume fraction of the CNTs (%)
0.75CNTs-590	590	0.75
0.75CNTs-630	630	0.75
1.0CNTs-630	630	1.0

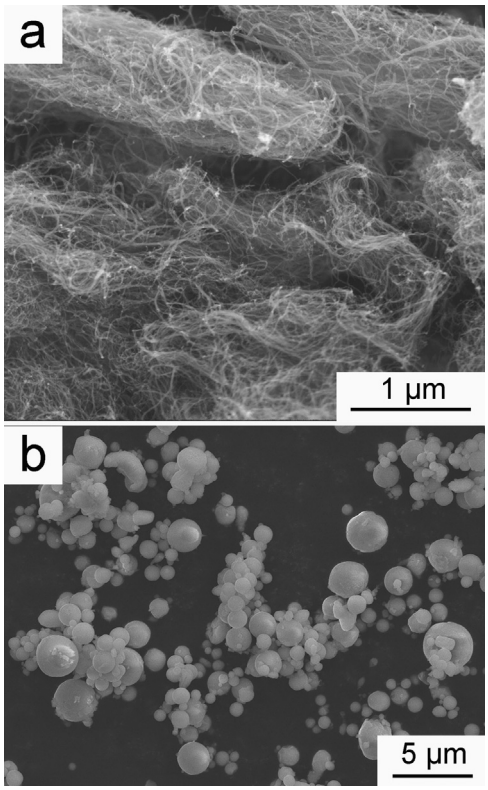

Fig. 1. SEM images of (a) as-received CNTs and (b) the Al powder.

3. Results

3.1. Morphology of the mixed powders and XRD of the rolled composites

Fig. 2a and b shows the SEM images of the ball milled Al powder with 0.75 and 1.0 vol% CNTs, respectively. From the enlarged image in Fig. 2a, it can be seen that the CNTs were uniformly attached to the surface of the Al powder. And as shown in Fig. 2b, the agglomeration of the CNTs appears in the Al powder mixed with 1.0 vol% CNTs. Comparing to the as-received Al powder, some spherical Al powder particles were transformed into irregular or flake shaped particles in both mixed powders. Fig. 3 shows the XRD patterns of the three composites after hot rolling. It can be clearly seen that only Al peaks exist in all the three composites, and no peaks corresponding to Al_4C_3 phase can be observed from the XRD patterns (the main Al_4C_3 peak is around ~55° [25,26]).

3.2. Hardness and density evolutions

Fig. 4a shows the bulk densities of the as-sintered and as-rolled composites. It can be seen that the density of the as-sintered 0.75CNTs-590 composite is substantially lower than that of the other two composites sintered at 630 °C. After hot rolling, the measured density of the composites increases substantially, as compared to that in the as-sintered state, and the 0.75CNTs-630 composite has the maximum

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