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Effect of milling time on dual-nanoparticulate-reinforced aluminum alloy matrix composite materials

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ABSTRACT

Carbon nanotubes (CNT) and nano-silicon carbide (nSiC)-reinforced aluminum (Al)-6061 alloy matrix composite materials were fabricated using high-energy ball milling and hot-pressing processes. The nSiC was used not only as a solid mixing agent to better disperse the CNTs in the Al powder, but also as a mean of inducing fine particle strengthening. The densification behavior of the dual-nanoparticulate-reinforced composites varied with the milling time. The crystallite sizes of Al in composites became significantly smaller when the milling time was increased. Moreover, the high-energy ball milling time significantly affected the microstructure and mechanical properties of the composites. We believe that the dual-nanoparticulate-reinforced composites can be used in a variety of applications as industrial component materials with precisely controlled properties.

1. Introduction

Engineering materials are continually required because they exhibit high performances and possess reliable characteristics and flexibility for multiple applications. Aluminum (Al) matrix composite materials have developed greatly since Duralumin was first fabricated by German metallurgists and then improved upon by American and Japanese scientists to become super and extra super Duralumin. These materials are widely used in many industrial fields, especially in automobile and aviation technology [1]. However, following this sensational materials development, only a few modified Al or Al matrix composite products have been introduced, such as Al cans and Al composite panels.

Recently, a new generation of Al matrix composites with highly anticipated materials properties has been introduced by combining Al and carbon nanotube (CNT) materials. Since they were discovered in 1991 [2], CNTs have been in the limelight as one of the strongest candidates for developing highly efficient next-generation materials due to their particularly high mechanical and electrical properties, chemical stability, and high thermal conductivity [3,4]. Many researchers are attempting to fabricate metal matrix composite materials reinforced with CNTs [5–8]. Kuzumaki et al. are pioneers of Al-CNT composite material fabrication, production composites with mechanical properties similar to those of pure Al bulk [9]. Esawi et al. have demonstrated the fabrication of Al-CNT composites by rolling and extrusion methods [10,11]. Liao et al. have produced Al-CNT composites by ultrasonic dispersion and spark plasma sintering processes and achieved an approximately 10% enhancement in tensile strength and Vickers hardness with respect to pure Al [12]. Deng et al. have also attempted to fabricate Al-CNT composite materials by a powder metallurgical route [13]. Bakshi et al. have tried thermal spraying coating of an Al-CNT composite powder to achieve better properties than those of ordinary coated materials [14].

Despite this enthusiastic line of research, CNT-reinforced Al matrix composite materials are still far away from being commercialized due to difficulties associated with the following issues: dispersing the CNTs, developing a suitable processing scheme and controlling the Al-CNT interface [5,15]. Our previous study demonstrated that the Vickers hardness of dual-nanoparticulate-reinforced Al matrix composites is eight times higher than that of pure Al bulk. Moreover, we observed that agglomerated CNTs could be well dispersed in an Al powder using nano-SiC (nSiC) as a solid mixing agent by mechanical ball milling and hot-pressing processes [16].

In this study, the effect of mechanical ball milling time on dual-nanoparticulate-reinforced Al6061 alloy matrix composite materials was investigated. CNTs, nSiC, and Al powders were

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mixed for five different milling times and then hot-pressed. The mechanical properties of the hot-pressed composites were measured using indentation and bending test equipments. The crystallite size and Raman spectra were rationalized in terms of the observed mechanical properties. Furthermore, microstructural analysis was performed to better understand the mechanical behavior of the dual-nanoparticulate-reinforced Al matrix composites as a function of the milling time.

2. Experimental procedure

Materials: Multiwalled CNTs (Baytubes C150P, Bayer Material Science, purity 99.5%, mean diameter: 20 nm, length: 10 μm) and gas-atomized Al6061 alloy powder (ECKA Granules, purity 99.5%, particle size below 63 μm) were used as starting materials. The SiC nanoparticles were produced in our laboratory by an inductively coupled plasma (ICP) process and is described in detail elsewhere [17]. The average particle size was between 20 and 30 nm.

Producing the composite powders: The powder composition was adjusted to Al6061 powder-1 vol% nSiC, then 6 wt% CNT was added to the mixture. The aluminum alloy powder and the nanoparticulate materials (CNTs and/or SiC) were mixed in a planetary ball mill (Retsch GmbH, PM400) for 30, 60, 120, 180, and 360 min in an argon atmosphere at 360 rpm using Ø10 mm balls, a 10:1 ball-to-powder weight ratio, and 20 wt% heptane as a process control agent. At the end of the process, the tight bowl containing the powder blend was transferred into a glove-box with a controlled inert argon atmosphere where the powder was passivated for almost 1 week. The milled powder was indeed in a highly activated energy state and could have easily oxidized/burned in contact with air.

Consolidation of the composite powders: After passivation, the blend was placed in a high-temperature steel mold. The mold was then heated up in air at the desired pressing temperature for 1.5 h and transferred rapidly (less than 5 s) to a uniaxial press (Walter+Bai AG, Switzerland). A pressure of 400 kN was then applied onto the composite powder for 4 s. The samples measured 30 mm in diameter and approximately 5 mm in thickness.

Characterizations: The density of the composites was measured by Archimedes's method according to ISO 3369:1975. The macro Vickers hardness of the composites were measured according to EN ISO 6507-1 with loads of 20 kg, applied for 15 s (220, GNEHM Härteprüfer AG and Paar MTH4 microhardness tester). At least five measurements were performed per sample. A four-point bending test was carried out using a Walter+Bai 150 kN servo-hydraulic test machine and DionSTAT software using a loading speed of 1 mm/min. The sample dimensions were approximately $3 \times 5 \times 22\ mm^3$. The composites' microstructure was observed by optical microscopy (Zeiss Axioplan light microscope), high-resolution cold field emission scanning electron microscopy (Hitachi, HRCFE-SEM S-4800), and high-resolution transmission electron microscopy (Jeol, HR TEM JEM-2200FS). The XRD patterns were measured using an X'Pert Pro diffractometer (PANAlytical) with Cu-K_α radiation ($\lambda = 1.54056$ Å, 45 kV and 40 mA) in the 2θ range of 20–80° using a linear detector (X'Celerator). A step size of 0.0167° and a scan rate of 0.05°/s were used. The crystallite size was calculated by the Scherrer equation [18]. Raman spectroscopy was performed on the bulks using a red He–Ne ion laser with a wavelength of 633 nm (Leica microsystems).

3. Results and discussion

The gas-atomized raw Al6061 alloy particles were highly spherical in shape and exhibited a wide distribution of sizes, as shown in Fig. 1a and b. The grains of the Al6061 alloy particles also showed a wide distribution of sizes ranging from 1 to 6 μm (Fig. 1c). The high-aspect-ratio CNTs were highly agglomerated, and many opened single CNT's tips were observed as shown in Fig. 1d and e. Parts of the CNT surfaces were covered with amorphous impurities. Fig. 1f shows the typical morphology of the nSiC particles fabricated by our own designed and modified inductively coupled plasma equipment [17]. The large size difference between the matrix particles and the reinforcement particulate materials make their mixing very challenging. However, it is very important to produce homogeneously well-mixed composite powders to create high-performance materials. Thus, high-energy ball milling was performed to disperse the CNTs in the Al6061 alloy powders together with the nSiC particles. The nSiC particles were used as a solid dispersion agent for the CNTs in the Al powders and play an important, synergistic role in enhancing the mechanical properties of such composites by, for example, inducing dispersion and fine particle effects [16]. Fig. 2 shows SEM micrographs of the dual-nanoparticulate-reinforced Al alloy composite powder as a function of the milling time. After only 30 min ball milling, several CNT and nSiC clusters were easily identified in the composite powders (Fig. 2a and b). The powder that was ball milled for 60 min showed many flake-like particles, but many CNT and nSiC clusters were still observed in the Al6061 alloy particles (Fig. 2c). These two powders showed a similar mean size distribution even though different ratios of particle shapes were observed.

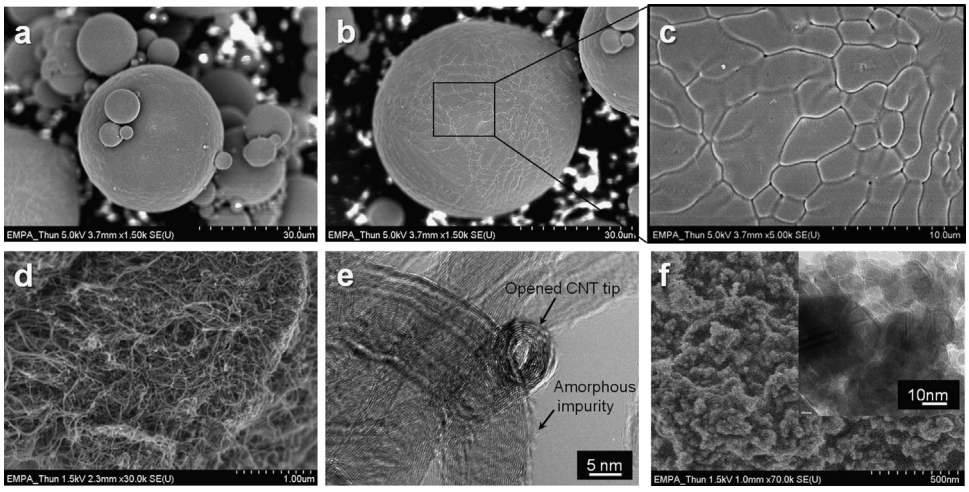

Fig. 1. Raw (a–c) Al 6061 alloy particle, (d and e) CNTs, and (f) nano-SiC particles.