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Interfacial characterization in carbon nanotube reinforced aluminum matrix composites

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ABSTRACT

In this work, the effects of the sintering parameters, such as temperature and the techniques used (HP and SPS), on CNT/Al composite interfaces are studied. The major role of the native aluminum oxide (Al_2O_3) layer covering the aluminum grains is highlighted. It is shown that, for a sintering temperature below 620 °C, the amorphous Al_2O_3 layer prevents the reaction between aluminum and carbon. For greater sintering temperatures, the breaking of the oxide layer due to its crystallization leads to the formation of aluminum carbide (Al_4C_3) by reaction between aluminum and the CNT. The Al_4C_3 crystals grow perpendicularly to the matrix grain boundaries by thermally activated diffusion of the carbon atoms coming from the CNT. It is also demonstrated that, by limiting the sintering time, which is the case in SPS, it is possible to limit the growth of the Al_4C_3 crystals and thus to preserve the CNT.

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1. Introduction

Innovative composite materials obtained by advanced fabrication processes are developed in order to respond to the requirement of new applications. Aluminum (Al) matrix composite reinforced with Carbon Nanotubes (CNT) is a good example and the growing number of investigations [1] emphasizes the interest generated by this metal matrix composite. The association of the low density of aluminum (Al) (2.6989 g/cm^3) with the great mechanical, electrical and thermal properties of carbon nanotubes (CNT) (Young's modulus of about 1 TPa, tensile strength >100 GPa [2], thermal conductivity of 3000/m·K [3] and electrical resistivity of 10^{-8}–10^{-6} Ω/m [4]) is expected to lead to the conception of a very promising material for many fields such as aeronautic and automotive applications. However, the achievement of physical properties improvement by addition of CNT in metal is not systematic. Because these materials are prepared through powder metallurgy route, the CNT are mainly located at matrix grain boundaries. These interfaces will then play a major role in the properties of the composites. Concerning the mechanical properties, Bakshi et al. identified the most significant parameters which affect the strengthening efficiency of CNT [1]. As the main strengthening mechanism is load transfer between the matrix and the reinforcement, uniform CNT dispersion at matrix grain boundaries and a strong bonding between the CNT and the aluminum grains are required to ensure effective load transfer. The properties of the CNT–matrix interface are also very important for the electrical transport properties of CNT composites [5]. Indeed, CNT at matrix grain boundaries can act as a conductive path for enhanced electrical [6,7] and thermal conductivities [8]. Therefore, controlling the evolution of the interfaces during the thermal treatment is crucial. In particular, strong bonding between CNT and aluminum is necessary to take advantage of the CNT physical properties. The eventual formation of a new phase at the CNT–Al interface has been investigated and several studies emphasize that reaction between Al and CNT can occur during consolidation resulting in the formation of aluminum carbides (Al_4C_3) [9–11]. Therefore, it seems obvious that the transformation of CNT, which are distributed at matrix grain boundaries, into Al_4C_3 will affect the interface bonding and bulk composite physical properties [5,12]. Several studies show that the formation of Al_4C_3 could affect the CNT/Al composite mechanical properties: by increasing the shear stress of the interface and the tensile strength [13–15] or on the contrary by promoting fracture [16–20]. In addition, the formation of Al_4C_3 results in the decrease of the CNT amount in the material, which would be detrimental to the expected properties.

Al_4C_3 crystals have been observed in composites synthesized through solid (*i.e.* powder metallurgy) and also liquid state routes. The presence of Al_4C_3 at the interface has been reported in samples sintered at temperatures below the aluminum melting point (660 °C) by Hot-Pressing (HP) [21,22], Spark Plasma Sintering (SPS) [23,24] and hot extrusion [25]. When the processing temperature is higher than 660 °C, the reaction between Al and CNT is enhanced. The CNT degradation is

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more intense leading to the production of nucleation sites that can react with liquid aluminum [9]. Kang et al. specify that the reaction between CNT and liquid aluminum occurs simultaneously with the CNT oxidation during thermal spray consolidation [26]. It is also noticed that the Al_4C_3 shape depends on the processing route. Deng et al. fabricated CNT/Al composite by hot-pressing at 500 °C in which Al_4C_3 crystals with block shape were found [15]. Esawi et al. observed Al_4C_3 nanorods in CNT reinforced Al matrix composite produced by hot extrusion at 500 °C [27]. Kwon et al. observed Al_4C_3 with various shape depending on the sintering process: tube and dumbbell shaped in sample sintered by SPS (600 °C) followed by hot extrusion (450 °C) and fine needles after a heat treatment at 800 °C [9,13]. Some authors observed a thin transition layer of Al_4C_3 between CNT and Al matrix in a composite annealed at 850 °C [15,28]. The various Al_4C_3 shapes prove that nucleation and growth mechanisms depend on several parameters (*i.e.* temperature, holding time and consolidation techniques) and no clear explanation is proposed in the literature.

This paper focuses on the effects of the sintering temperature and the consolidation technique on the composite interfaces. Boundary zone in samples prepared by HP and SPS are observed and analyzed. As a result, a mechanism of the interface evolution in CNT/Al composites including the formation of Al_4C_3 is proposed.

2. Experimental procedure

Commercial aluminum (Al) powder (average particle size of 5 μm) with a purity of 99% (main impurities: Fe: 0.69 at.%, Si: 0.14 at.%, O: 0.16 wt.%) and MWCNT (purity 90%, diameter 9.5 nm and length 1.5 μm), supplied by Nanocyl (Belgium), produced *via* Catalytic Chemical Vapor Deposition (CCVD) process, were used in the present study (see Fig. 1a and b). In order to produce 0.5 wt.% CNT/Al composite powder with uniform CNT distribution as shown in Fig. 1c, CNT were mechanically and chemically treated to disentangle them before being dispersed into Al powder. At first, a carbon nanotube suspension was obtained by high speed stirring. In the second step, CNT were oxidized with nitric acid (HNO_3 16 vol.%) and finally, their surface was chemically modified with a surfactant (Sodium dodecyl sulfate) to achieve the disentanglement. Then, disentangled CNT were dispersed into the raw Al powder using a slurry based process. For that, CNT and Al powder were mixed together in ethanol. The slurry was successively sonicated for 3 h and ball-milled for 1 h at low speed (250 rpm) to complete the dispersion. Finally, the CNT/Al composite powder was filtered and dried in an oven under vacuum at 110 °C for 48 h.

The CNT/Al powder was placed in a carbon mold with a diameter of 20 mm and sintered using two different powder metallurgy techniques: hot-pressing (Centorr Furnace Vaccum Industries/Load Cell 200KNS INSTRON®) and Spark Plasma Sintering (model 515S-SYNTEX, Sumitomo Coal Mining Co. Ltd., located at the regional SPS platform facility hosted by ICMPE, Thiais, France). For both sintering techniques, the sample temperature was controlled by a thermocouple placed in the mold, closed to the sample. The study of the sintering temperature effect on the composite grain boundaries was conducted with samples hot-pressed at

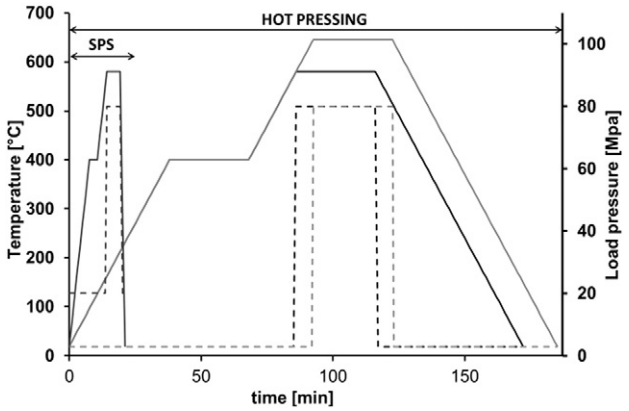

Fig. 2. HP and SPS sintering cycles; the temperature is in continuous lines, the pressure in dotted lines.

different temperatures. The powder was first debinded at 400 °C under high vacuum before being sintered from 580 °C to 645 °C under a load pressure of 80 MPa in nitrogen atmosphere. The heating rate was 10 °C/min and the holding time was 30 min for both sintering and debinding steps. The investigation of the sintering technique effect on the composite interfaces consists in comparison of samples sintered at the same temperature (580 and 600 °C), by HP and by SPS. No sample was heated by SPS above 600 °C to completely avoid the risk of aluminum local melting. SPS technique, which is well-known for its high heating rate, allows the application of sintering cycles greatly shorter than those applied in hot-pressing as shown in Fig. 2. After debinding at 400 °C during 5 min, the sample was sintered by SPS at 580 or 600 °C during 5 min with a heating rate of 50 °C/min under argon atmosphere. In order to avoid any temperature overtaking, the heating rate was greatly reduced before the sintering dwell.

The interfaces were observed using Field emission Scanning Electron Microscope (FESEM, Hitachi S-4700). These observations require appropriate polishing procedure. Thus, the composite surface was prepared by grounding with SiC paper and polishing with diamond paste (from 6 μm to 0.25 μm). The samples were etched with Keller's etchant for 8 s to reveal the matrix grain boundaries and finally coated with chromium to reduce eventual charging phenomena. X-ray diffraction (XRD) analysis was performed in order to check if any phase transformation occurred during heat treatment. A Bruker D8 Advance diffractometer was used for collecting X-ray diffraction data. The Cu Kα lines were used as a source. All the patterns were recorded for 2θ values from 28° to 68° with a step size of 0.02° and the acquisition time for each step was fixed to 2 s. CNT–Al interfaces were examined and analyzed using Transmission Electron Microscope (TEM, FEI Tecnaï 200 and Philips CM30) equipped with an Energy Dispersive Spectroscopy (EDS) detector. Thin specimens were carefully prepared by mechanical polishing and ion beam milling (Gatan, PIPS) for that purpose. The Al_4C_3 growth was investigated by *in situ* TEM observations conducted thanks

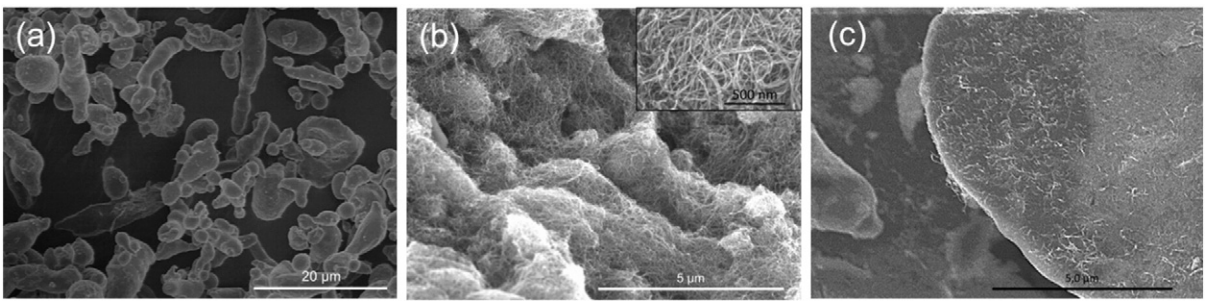

Fig. 1. (a) Raw aluminum powder, (b) CNT and (c) CNT/Al composite powder used to prepare the composite materials.

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