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Structural Properties of Carbon Nanotube Reinforced Aluminum Matrix Composites

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Abstract—In this work, Al/Al chips–CNT composites were fabricated using powder metallurgy route involving mechanical ball milling, cold pressing and subsequently hot extrusion process without sintering step. Ball milled Al chips–CNTs with fixed amount of Al chips were used for homogenious distrubition. After preparing the Al chips coated CNTs, they were mixed with different amounts of Al powders in the range of 25–75 wt%. Structural properties of these Al/Al chips–CNT composites were evaluated. The distribution of CNTs was analyzed using a Scanning Electron Microscopy (SEM) equipped with Energy Dispersive Spectroscopy (EDS) analyzer. Microstructural analysis revealed that the CNTs on the Al chips were present throughout the extrusion direction and the uniform distribution of CNTs at the chip surface decreased by increasing CNTs percentages along with increasing wt% of Al chips. The results of the density and electrical conductivity test showed that an increase in the CNTs weight fraction resulted in a significant decrease of density and electrical conductivity of the composites with 50 and 75 wt. % of Al chips by comparing with the base alloy and the composite with 25 wt. % of Al chips, while addition of CNTs especially in the composites with 50 and 75 wt. % of Al chips caused increasing of the porosity in the composite structure.

Index Terms— Aluminum Chips, Carbon Nanotube, Composite, Microstructure, Powder Metallurgy.

1 Introduction

Carbon nanotubes (CNTs) have been regarded as ideal reinforcements for composites due to due to their high aspect ratio and outstanding mechanical stiffness [1]. The addition of CNTs can result in a significant improvement in mechanical properties of light composites suitable for the automotive or aerospace industries, where the fuel economy and weight reduction are the first priority [2]. Nevertheless, the theoretical potential of CNTs has not yet been demonstrated in metal matrix composites [3] due to difficulties in achieving homogeneous dispersion of CNTs with in metal matrices. Powder metallurgy (PM) has evolved into the most widely used technology for fabrication of CNT/Al composites, owing to its simplicity, flexibility and controllability [4].

In these regards, a two-step process was applied in this study. In the first step, a mechanical ball milling process was performed to pre-disperse CNTs on the Al chips.Then the Al chips with the well dispersed CNTs on their surface were mixed with pure Al powder because of the greater plasticity of pure Al. The composite mixture was subsequently consolidated by cold pressing and hot extrusion. The fact that extrusion processis a good way to achieve higher material performance and promote alignment of CNTs in the extrusion direction is remarkable. In this study, Al/Al chips–CNT composites were fabricated by the mechanical ball milling, cold pressing and hot extrusion process, and their density and electrical properties were investigated.

2 EXPERIMENTAL DETAILS

In this study, Al chips/Al and composite reinforced with 0.5, 1, 2 and 4 wt.% of CNTs were used. CNTs used were multiwall CNTs (MWNTs) synthesized by a catalytic carbon vapor deposition (CCVD) process, purchased from Grafen Chemical Industry, Turkey. According to the supplier, their average diameter and length were 9.5 nm and 1.5 µm, respectively. Before preparation process of composites, as-prepared CNTs were purified by washing with HNO_3/H_2SO_4 (1:1 by volume) for 30 minutes in an ultrasonic bath at 80 °C. The pretreated CNT was collected and washed to pH 5.0 by doubly distilled water. Afterwards, the pretreated MWCNTs were dried at 100°C in an oven for 24 hours.

AA6061 Al chips (~630 µm) produced by turning off an as-received cast bar without lubrication were ball-milled under argon atmosphere at 300 rpm for 3 hours with a ball to powder weight ratio (BPR) of 32 to coat the surface of Al chips with the pretreated MWCNTs. The Al chips were used as a media for observing the CNT’s movement during hot extrusion. To obtain good bonding [5], the present authors used Al powders as a good binder in recycling of Al chips containing CNTs through hot extrusion process. Commercial materials with low porosity and appreciable high strength can be prepared by hot extrusion after being processed by cold press. Since Al alloy powders can be usually consolidated by extrusion until the full density be reached, the CNTs-coated Al chips were then mixed with pure Al powder (97.7 % purity, about 8.6 µm in diameter). In order to consolidate the prepared mixtures, firstly they were filled into a cylindrical container with a diameter of 30 mm and pressed under 1600 MPa pressure at room temperature. Then, the cold pressed samples were consolidated by hot extrusion at 350°C with ram speed of 5 mm.s^{-1} and extrusion ratio of 9:1, resulting into cylindrical compact products with diameter of 10 ± 0.2 mm. The extrusion was performed without canning, degassing and atmospheric control [6].

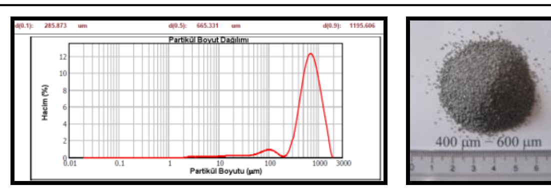

Fig. 1. Particle size and general view of AA6061 chips used in experimental works.

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In accordance with the standard metallographic procedure, which includes a grinding process up to 1200 mesh and a polishing process with alumina solution conducted on the base alloy and composites, microstructural characterization studies were conducted on metallographically polished base alloy and composite extruded samples using an Optical Microscopic (OM) and Scanning Electron Microscopy (SEM) equipped with Energy Dispersive Spectroscopy (EDS) analyzer. X-Ray Diffraction (XRD) analysis, density and electrical conductivity measurements. The experimental density measurements were determined by means of the Archimedes principle. The volume porosity of the samples that were fabricated under different conditions was measured by the Archimedes principle using the following equation:

$$\%Porosity = \frac{\rho_{Al-chips/Al} - \rho_{Comp}}{\rho_{Al-chips/Al}} \times 100 \quad (1)$$

where $\rho_{Al\text{-}chips/Al}$ is the density of the initial Al chips/Al without CNT and ρ_{Comp} is the density of the composites containing CNT. Electrical conductivities of the samples were measured based on eddy currents on sample surfaces utilizing a Hocking™ AutoSigma 3000 electrical conductivity meter. Prior to measurements, the sample surfaces were grinded with a 1200 mesh emery paper to obtain a flat surface. Mean values of ten measurements were taken to determine the electrical conductivities of the samples. Furthermore, the degree of CNT distribution in the composites was investigated by the SEM equipped with an EDS analyzer.

3 RESULTS and DISCUSSIONS

Figure 2 shows the microstructures of the base alloy and composites with different amounts of Al chips in the range of 25–75 wt%. All micrographs were taken from the plane parallel to the extrusion direction. CNTs were gradually embedded inside the Al chips through plastic deformation of the Al chips under the impact of the balls during mechanical ball milling process. In Figure 2, CNTs decorated the chip boundaries after extrusion. When the Al chip content was 50 and 75 wt. %, the entangled CNT clusters were adhered to the surfaces of the Al chips (Fig. 2).

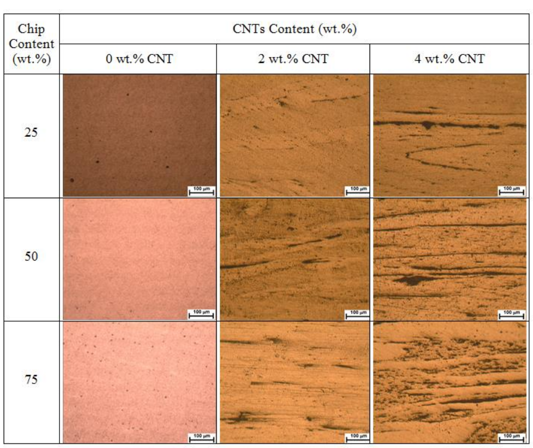


Fig. 2. Optical micrographs of the base alloy and composites with different amounts of Al chips in the range of 25–75 wt%.

The cross-sectional microstructures of the extruded rods with 25 and 75 wt.% Al chips using SEM are shown in Fig. 3. Chips in the composites exhibited a lamellar structure and the dark areas were detected around the chip surface even at lower magnification. Most of the dark areas were present throughout the extrusion direction because of the severe plastic deformation of the Al layers with recombination of CNTs during hot extrusion. Presence of coated CNTs between the layers was confirmed by carrying out EDS analysis as shown in Fig. 3. Possible interactions between CNTs and the Al matrix extruded at 350 ◦C were investigated using XRD, and no clear carbide phase was identified (Fig. 4). Figure 3 clearly illustrates that an increase in intensity of darker phase with increasing CNT content was also found due to entanglement of CNTs during ball milling process and higher CNT loading revealed network of CNT or porosity along the Al chips as shown in Fig. 3.

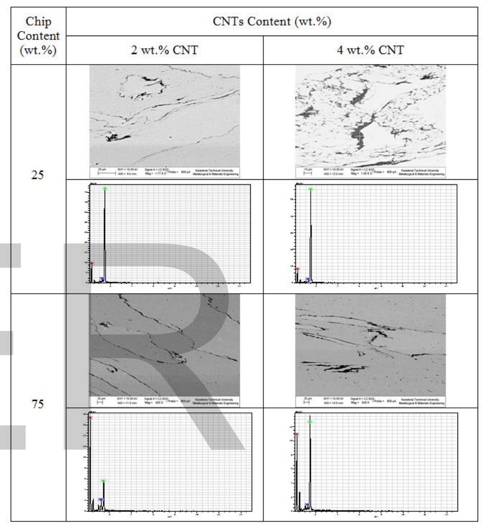


Fig. 3. SEM images and EDS results on the composites with 2 wt. % CNT and 4 wt. % CNT at fixed amount of Al chips.

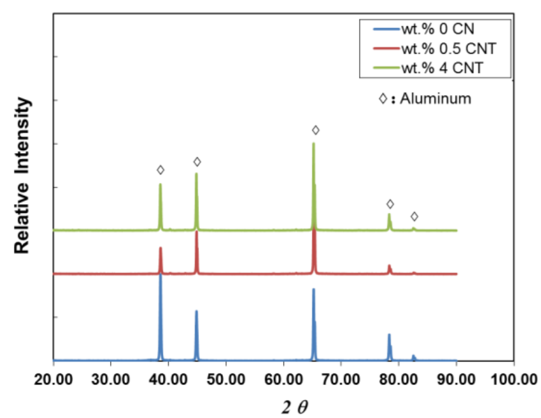


Fig. 4. XRD results of the composites with 75 wt. % of Al chips.

One of the main problems associated with the production of composites using the ball milling and hot extrusion processes may be attributed to the great difference in the sizes of the Al chips and CNTs [6]. When increasing the Al chip content to 75 wt.%, the porosity in the composite structure increased. (Fig. 5). After the hot extrusion, the CNTs could not redistribute within the matrix even though the chips elongated.

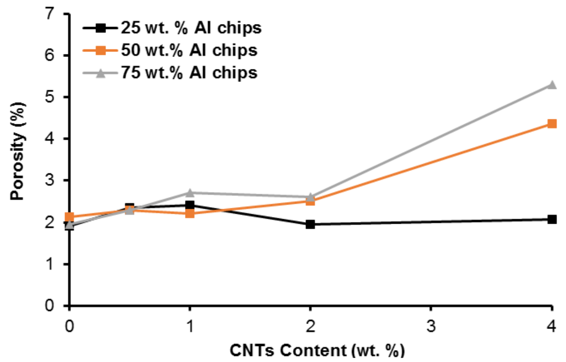
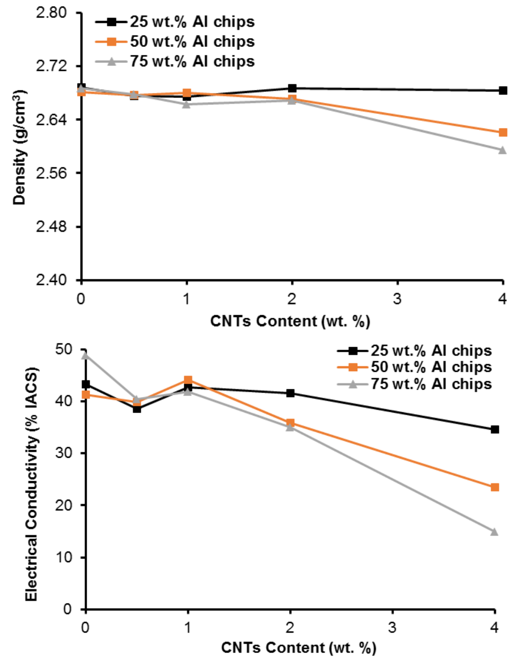


Fig. 5. The effect of CNTs content on the porosity of the composites with different amounts of Al chips in the range of 25 – 75 wt.%.

Figure 6 illustrates the changes in the density and electrical conductivity of the composites over base alloy prepared by the same route. It is evident from Fig. 6 that when the CNT content was exceeded 1.0 wt.%, the increasing CNT content in the composites with 50 and 75 wt. % of Al chips resulted in a lower electrical conductivity. On the other hand, the density of the composites decreased by increasing wt% of chips when the CNT content was exceeded 2.0 wt.%. This decrease in density can be explained by the presence of porosities in the composites with increasing the amount of CNTs along with decreasing soft and more easily deformable Al powder phase (Fig. 5). In metals or alloys, the electrical conductivity is mainly caused by free electrons. If the defects, such as the porosities, interfaces, particle boundaries and free surfaces remain in the composites, the electrical conductivity is adversely affected. It is further to add that if the smaller CNTs are agglomerated at the chip surface as evident from OM and SEM micrographs (Figs 2 and 3), they behave as a different phase at chip surface, which increases the scattering charge carriers between two chips and hence reduces the conductivity. As shown in Fig. 6, the decrease in the density and electrical conductivity of the composites with CNT addition may be attributed to the formation of the porosities and the agglomeration of smaller CNTs between the larger Al chips. Although the CNT has excellent electrical conductivity, these results are disadvantageous for the electron mobility [6].



(b)

Fig. 6. The effect of CNTs content on the a) density and (b) electrical conductivity of the composites with different amounts of Al chips in the range of 25–75 wt.

4 CONCLUSIONS

The following conclusions can be drawn:

1. The combination of mechanical ball milling, cold pressing-hot extrusion approach without sintering step for obtaining new CNT-reinforced Al composite provided a unique opportunity to produce light-weight metallic composites.

2. Microstructure characterization studies revealed that the CNTs on the Al chips were present throughout the extrusion direction and the uniform distribution of CNTs at the chip surface decreased by increasing CNTs percentages along with increasing wt% of Al chips.

3. Addition of CNTs to the base alloy caused a decrease in the density and electrical conductivity.

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