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RESEARCH PAPER

Synthesis of Aluminum- CNTs Composites Using Double-Pressing Double-Sintering Method (DPDS)

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ABSTRACT

In this study, carbon nanotubes (CNTs) reinforced aluminum matrix was synthesized by DPDS for the first time. Planetary ball mill has been used to disperse of CNTs in Al matrix. The effects of the CNTs content and secondary pressing-sintering on properties of the nano-composites were investigated. Mechanical properties of aluminum metal matrix nano-composites samples were characterized using compression and micro-hardness measurements. Enhancements of about 52% in compressive strength and 64% in hardness were observed as compared to pure aluminum (matrix) fabricated under the similar condition by conventional sintering process. The morphology and phase analysis of composites have been studied by SEM, and XRD. This research work shows feasibility of manufacturing CNT reinforced metal composites by DPDS.

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INTRODUCTION

Metal matrix composite (MMC) is engineered combination of the metal (matrix) and hard particle (reinforcement) to get tailored properties. MMCs are either in use or prototyping for the space shuttle, commercial airliners, electronic substrates, automobiles, golf clubs, and a variety of other applications [1-7].

Incorporation of carbon nanotubes (CNTs) in polymer composites have been widely investigated since the discovery of CNTs by Iijima [8].In metal composites, however, the progress has been rather slow primarily due to challenges associated with CNT dispersion and high temperature processing used in the synthesis of metal composites. Carbon nanotubes (CNTs) have unique tubular structures with a few nanometer diameter that possess high elastic modulus (>1TPa) and high tensile strength (>30GPa) [9]. These exceptional mechanical properties combined with a low density make them an ideal reinforcement in Al matrix composites (AMCs) for the automotive and aerospace industries where weight reduction for decreasing fuel consumption is a priority [1, 9]. Agglomeration of CNTs [10] is detrimental to the mechanical properties of AMCs and is a major challenge that inhibits the development of these composites. Many methods have been used in an effort to reduce the agglomeration of CNTs: coating CNTs on the Al powder surface [11] in situ synthesis of CNTs in Al powders, [12] and mechanical milling (MM) of Al/CNT powder mixtures [13]. Furthermore, the mechanical properties of mechanical milling (MM) processed Al-CNT composite would be further improved by

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the Al grain refinement to the nano-scale due to intense straining and by the incorporation and dispersion of the oxide layer initially present on the surface of Al powders [10]. MM processed Al-CNT powders have been consolidated by conventional methods, e.g., hot pressing, [14- 15] hot extrusion, [16- 17] and spark plasma sintering [18- 19]. Since in these techniques densification occurs by heating at relatively high temperatures, an interfacial reaction between Al and CNTs may result in the formation of brittle Al_4C_3 phase [20]. Also grain growth of nano-crystalline Al may occur as well. Both have a detrimental effect on the mechanical properties of Al-CNT composites. The high porosity of sintered components leads to reduced mechanical strength and load capacity when compared with those properties of fully dense materials [21- 22]. In addition, it is clear that the development of high-quality composites demands improved mechanical properties. Therefore, powder metallurgists are continuously searching for new alternatives and mechanisms to improve mechanical properties and load support.

For a powder metallurgy (P/M) component to be considered for high performance application porosity must be reduced to a minimum; i.e. density must be maximized. In order to reduce porosity many methods have been explored, such as the hydro-pulsor technique [23], warm compaction [24], and high temperature sintering [25]. One of the more common methods to reduce porosity is known as "double-press double sintering" (DPDS), which decreases bulk porosity. Uniaxial die-pressing, due to its low manufacturing costs, is still the most traditional processing route. It also produces the closest tolerances in the finished parts, thus nearly eliminating post-sintering operations such as machining [26]. A potential alternative for improving the mechanical strength of composites is therefore the use of the double pressing/double sintering technique [27] developed by Hoeganaes [28]. The goal of this method is to increase the density of composites by two-fold pressing. According to German [29], reductions of 2–3% in porosity would result in up to a 20% increase in mechanical strength. The literature does not report the use of double pressing/ double sintering technique for the production of Al-CNT metal matrix composites (MMCs). On the other hand, there are a few papers that report its use in the production of sintered steels, and it is generally accepted that this technique induces higher densities and strengths. In fact, DPDS has been used by the automotive industry since1990s in highly demanding applications such as synchronizer hubs, crankshaft sprockets, steering column tilt levers and planetary gear carriers [30].

In the present research for the first time (as far as authors know) fabrication of fully-dense Al-CNT MMCs with uniform distribution of CNTs using DPDS processing is reported.

MATERIALS AND METHODS

The purity of aluminum powder was 99% (supplied by Khorasan Powder Metallurgy Co., Ltd.) and multiwall carbon nanotubes (MWCNT) (provided by Research Institute of Petroleum Industry, Tehran, Iran) had purity and density of 95% and 2.200g/cm^3 (diameter=10nm, length=30µm). TEM and SEM images of purified MWCNTs are shown in Fig. 1. It is observed that the CNTs had

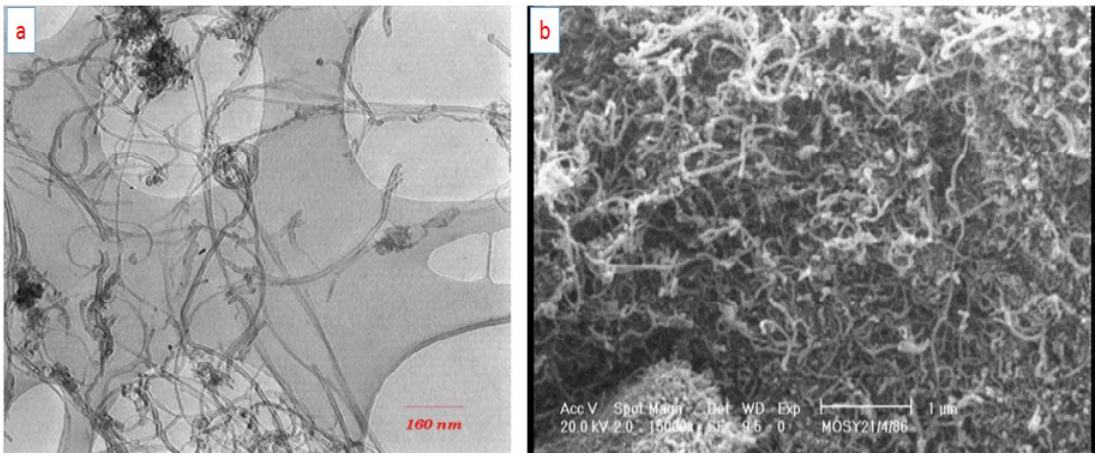

Fig. 1. TEM and SEM micrographs of MWCNTs: a- TEM and b- SEM.

a curvilinear morphology and twisted shape and were having nano-scale morphological features.

MWCNTs were mixed with aluminum in planetary ball mill containing stainless steel milling balls of 10mm and15mm diameter (giving initial ball to powder ratio (BPR) =20:1). Ethanol(2wt.%) was used as a process control agent (PCA) in order to minimize cold welding of Al particles as well as the strain hardening of the material at the higher energies involved during milling. It also prevents sticking of powders to the balls as well as walls of planetary ball mill during milling. The planetary ball mill was filled with argon and agitated using rpm of 200 and milling time up to 2hr (15minutes to an hour after the rest of milling was considered). They were all opened inside the glove box and samples were extracted. Nano-composite powder was then pressed in a uniaxial die of stainless steel under the pressure of 300MPa and then pre-sintered under argon atmosphere by heating at 500˚C for 45min. Before pressing of the samples for the second time, they were lightly sprayed with zinc stearate to reduce friction effects. Finally samples were sintered under argon atmosphere in 600˚C for 45min. Synthesized samples have size of 18mm in length and 12mm in diameter.

Vickers micro-hardness of the Al-CNTs composite samples were measured using HVS-1000A model micro-hardness tester, with a loading of 200g (1.96 N) and a dwell time of 15s (according to ASTM E384-05A). The Archimedes technique was used to measure the density of all samples. Compressive tests were carried out using a Zwick Royll-Z100 universal testing machine with an initial strain rate of 2mm/ min at room temperature (according to ASTM E9-89AR00).

Morphology of composites powders and fracture surfaces of failed compressive samples were examined using scanning electron microscope (SEM). XRD analysis of Al-CNTs samples were carried out using XPert (Philips PW 3710) Diffract meter (Co K_a radiation with λ = 1.789010A°) with voltage and current setting of 40kV and 30mA, respectively.

RESULTS AND DISCUSSION

Microstructure

The mixing process and its duration determine the effective dispersion of CNTs in the Al powder. SEM micrographs of the blended Al–CNTs powders are shown in Fig. 2. It seems to be homogeneous mixing of Al powder and CNTs at 8wt. %, and there is considerable agglomeration of CNTs at 12wt. %. The agglomeration of CNTs is one of the biggest

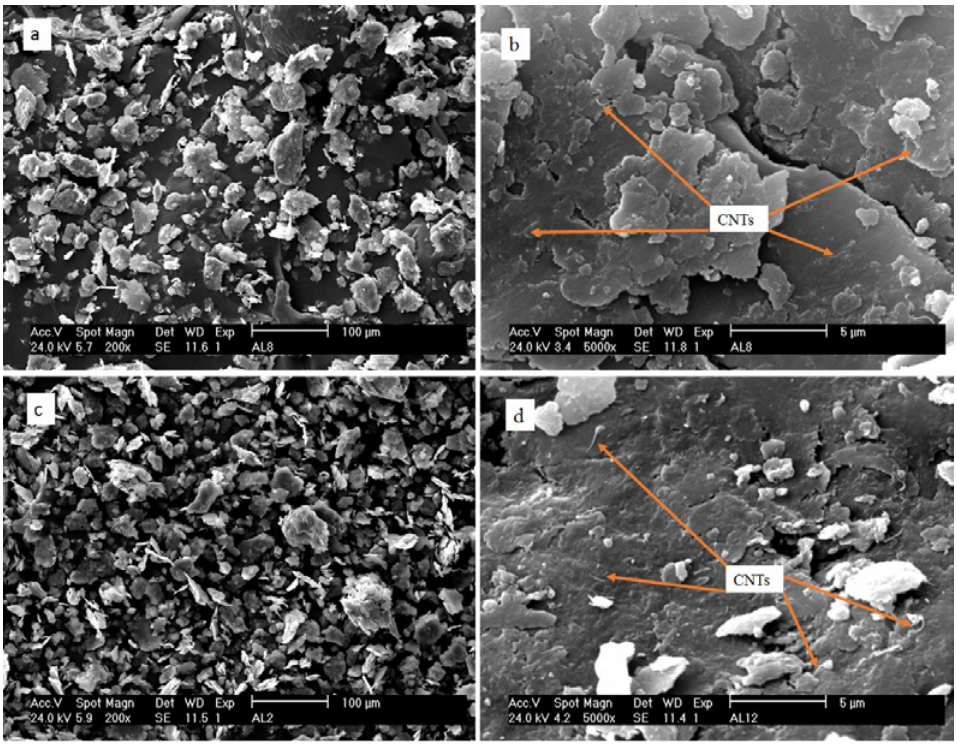

Fig. 2. SEM images of aluminum powder milled for 2 h with the addition of PCA (a, b) Al–8wt. % CNTs and (c, d) Al–12wt. % CNTs.

challenges for CNTs reinforced composites which lead to premature crack initiation and fracture in tension. For all the powders, CNTs were not good observed on the powder surface because CNTs were embedded within the aluminum powder during milling.

For metallographic samples SiC sandpaper number of 320p to 3000p were used. In order to view the microstructure of the samples by SEM, first samples were polished using 0.5 micron alumina suspension, felt and diamond paste. Finally samples were etched by a solution of 0.5 HF. Metallo-graphically polished cross-sectional views of the composites are shown in Fig.3. It shows smooth surface in samples having Al-8wt. % CNTs and (b) porosity in the samples having Al-12wt. % CNTs.

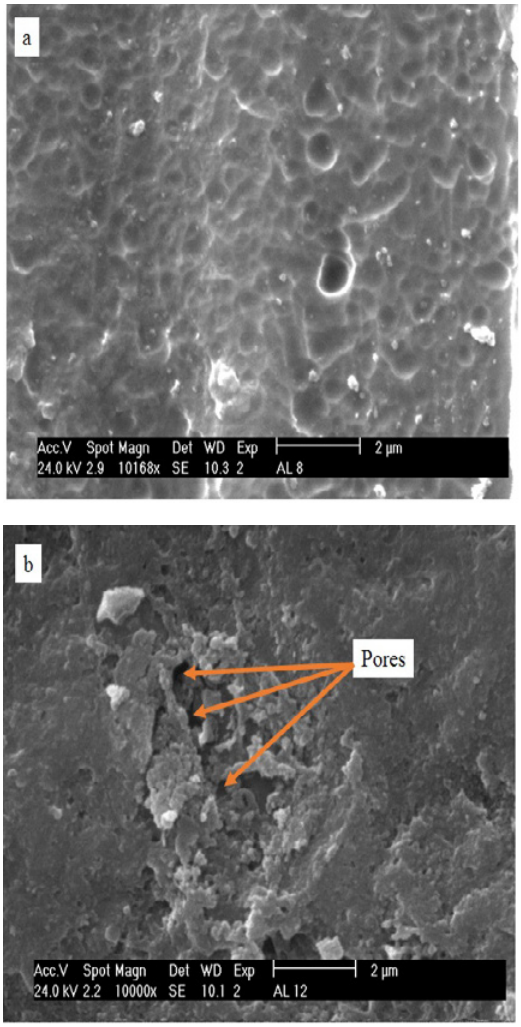

Fig.3. SEM micrographs of etched DPDS samples (a) Al–8 wt.% CNTs and (b) Al–12wt.% CNTs composites.

Fig. 4(a) shows CNTs pull-out on the compressive fracture surface of Al-CNTs MMCs specimen reinforced with 8wt. % CNTs. The pits on the fracture surface indicated that CNTs are pulled; on the other hand, the naked CNTs standing on the surface imply weak interfacial strength between CNTs and the Al matrix. Fig. 4(b) shows clusters of CNTs are also seen in the Al-12wt. %CNTs MMCs. The degree of CNTs clustering is higher in the Al-

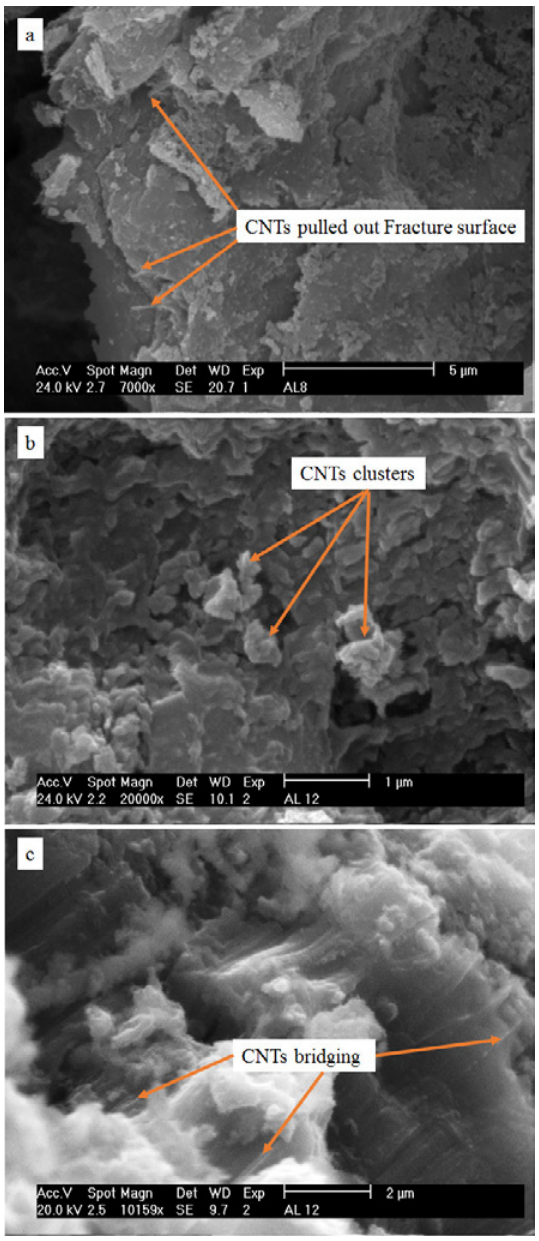

Fig. 4. (a) Fracture surface of Al–8wt. % CNTs nano-composite showing smooth surface of CNTs pulled out from the matrix and CNTs embedded in matrix (b) CNTs clusters in Al–12wt. % CNTs and (c) nanotube bridging.

12wt. %CNTs as compared to other samples. The surface tension of molten Al generates capillary forces which causes agglomeration of CNTs and the break- down of mesh structure leading to the formation of CNTs cluster. Of course, the amount of CNTs is also effective. Fig. 4(c) shows an image of carbon nanotubes bridging. When the nanotubes are line up perpendicular to the crack direction, their high strength prevents them from fracturing while the strength of the CNT– matrix interface is sufficient to inhibit pull out from the matrix. In this situation propagating cracks are forced to detour around the nanotubes.

X-ray analysis

The X-ray diffraction patterns of Al-CNTs composite powders, milled for 2h, are shown in Fig. 5, which shows five major peaks corresponding to FCC phase of Al. No observable peaks for CNTs are observed. Similar results were obtained for sintered and DPDS samples, which has been reported elsewhere [30]. The homogenous dispersion of CNTs within the matrix, unfavorable strain conditions and amorphous CNTs [31] could also be the reason for the absence of CNTs peaks. According to the X-ray diffraction (XRD) phase analysis results, the composite consists of only Al-phase even after 2h of milling. Furthermore, the diffraction lines corresponding to the aluminum were somewhat shifted. The peak shift generally occurs when CNTs are under a compressive state which is elicited by the high energy impact of milling media on the powders during the milling process [32, 33] and lattice strain increases. When molten aluminum reacts with carbon in CNT, there is a possibility of formation of aluminum carbide (Al_4C_3) at the matrix/reinforcement interface. The chemical reactions and the corresponding free energies have been represented by the equations [36]:

$$4[Al] + 3C = Al_4C_3\ ,\ \Delta G^f_{Al_4C_3} = -258.32\ + 0.0968\mathrm{T}$$

Here the square brackets [] represent that Al are in the molten Al and *T* the absolute temperature at which the reaction takes place. The thermodynamic properties of CNTs were assumed equal to graphite due to lack of the data for CNTs. This gives us a comparative analysis for reaction which might be correct for the CNT–alloy system as well, because for both graphite and CNTs the reacting planes are the same (0001) basal and (1 0 $\bar{1}$ 0) prism planes. The free energy of formation of Al_4C_3 is −35.7kJmol^{-1} at 2300K that certifies thermodynamic feasibility at the temperature experienced by in-flight particles. It is worthwhile to note that the peak corresponding to aluminum carbide (Al_4C_3), which is often seen in the composites prepared by the liquid metallurgy route, could not be found under current conditions. This can be attributed to intermittent stoppage of milling process at every 1h interval that did not allow temperature rise for the reaction to take place. Similar findings were also reported by Esawi et al. [32] in Al-CNTs milled powders. Of course if the vol. % of the carbide is high enough, then one could observe peaks corresponding to the other phases in the XRD pattern [34–38].

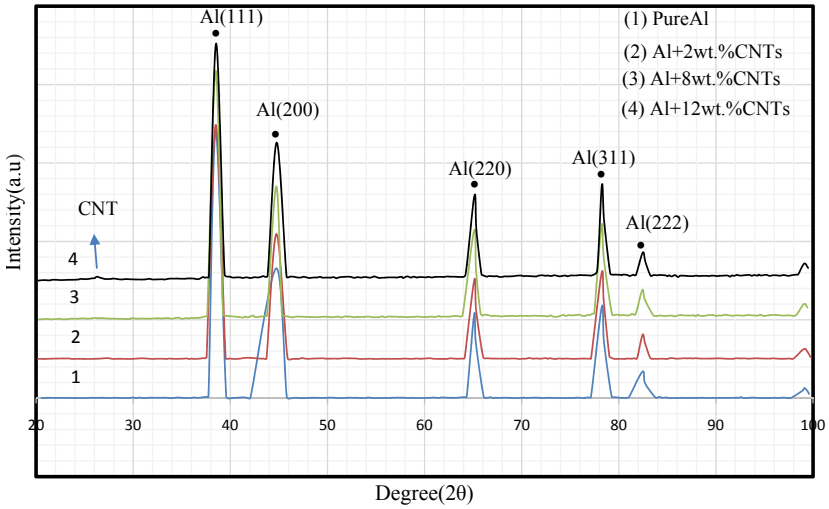

Fig. 5. XRD spectra obtained from the Al–CNTs nano-composites after milling process for 2 hours for different CNTs concentrations.

Mechanical properties

As it can be seen in Fig. 6 the measured density of samples after primary pressing and sintering decreases with increasing CNTs content. The main cause of reduction of density of nano-composites relative to the density of pure aluminum is because of increasing amounts of carbon nanotubes that have low density than pure aluminum. A drastic reduction of the measured density in the primary pressing and sintering is also could be due to the high porosity in the samples.

The effects of carbon nanotubes content and second pressing and sintering on measured density are shown in Fig.6. It is obvious that with addition of carbon nanotubes in second pressing and sintering, the measured density of the composites increase with increasing carbon nanotubes content, while large amount of carbon nanotubes try to reduce the relative density of the composites. This can be due to the fact that addition of carbon nanotubes could fill up the micro-voids resulting in increase of the density of CNTs–Al nano-composites, however, large amount of carbon nanotubes are prone to tangle together in blended powders of Al powders and carbon nanotubes. Reducing the density at CNTs content greater than 8wt%, could be due to the non-homogeneous distribution, agglomeration and cluster formation of CNTs, Which inhibit the diffusion of Al into the small spaces between

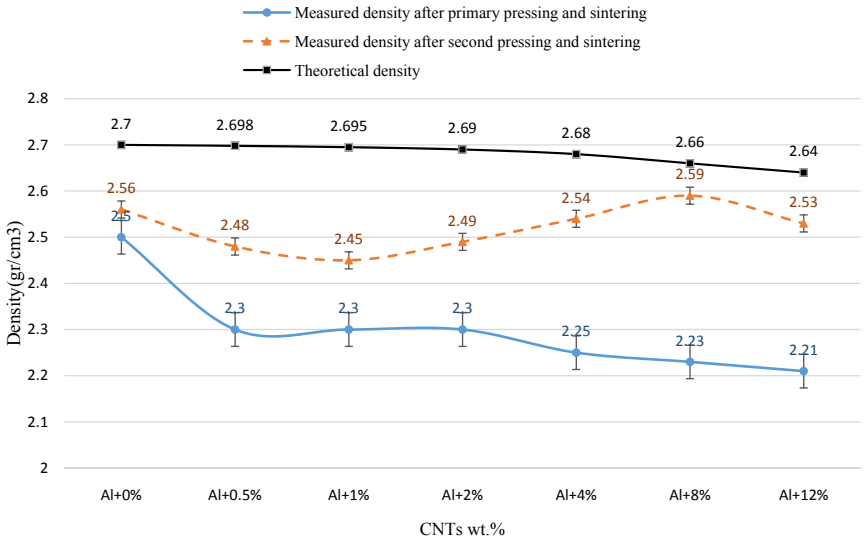

Fig. 6. Density of Al-CNTs nano-composites as a function of CNTs Wt.% at various conditions.

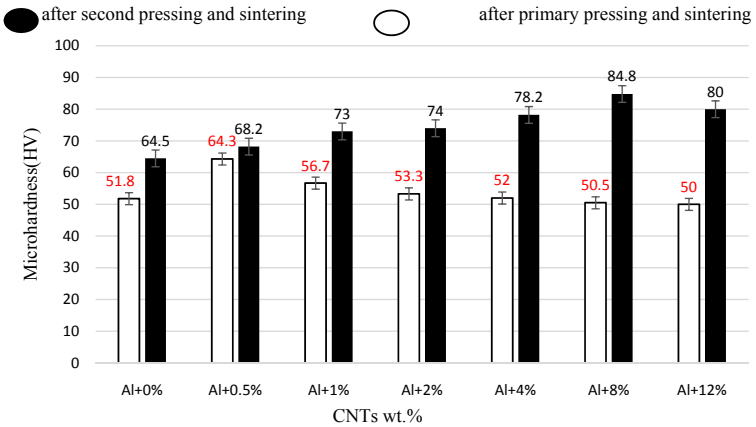

Fig. 7. Vickers micro-hardness of Al-CNTs nano-composites as a function of Wt. % CNTs, before and after second pressing and sintering.

the CNTs, resulting in a non-uniform distribution and the formation of pores during the sintering process. Thus leads to reduction in the measured density. Carbon nanotubes conglomeration not only impedes the densification of the specimens, but also becomes the defect source. Hence, the measured density of the composites decreases

Fig.7 shows that the average of Vickers micro-hardness values of the second pressed and sintered composites increased from about 51.8HV (for pure Al) up to 84.8HV (at 8wt. % CNT), equivalent to an increase of about 64%.When the CNT volume content is about 8wt. %, according to the theory of short fiber reinforced composites, the nanotubes distribute uniformly in the matrix, effectively inhibiting matrix deformation and producing a strengthening effect. At CNT volume contents greater than 8wt. %, the hardness of the composites decreased as the volume content was increased. This phenomenon is due to:

- Some agglomeration of the nanotubes was found when composites contain high CNTs volume content (Fig. 4(b)). In this state, the bulk material will have two types of interface, Al–nanotube and nanotube–nanotube. The strength of the nanotube– nanotube interfaces, getting up from agglomeration of the nanotubes, is very poor. Under applied load, this material would fail due to this poor interfacial bonding and cause a decrease in hardness.

- The density of the composites decreases with the increase of CNT during DPDS process (Fig. 6).

Fig. 7 also shows that the average of Vickers micro-hardness values obtained from second pressed and sintered nano-composites are higher than those obtained from nano-composites not subjected to second pressing and sintering. The second pressing and sintering process can increase the density of the nano-composite (Fig. 6) and cause deformation strengthening of matrix, as a consequence of which higher Vickers micro-hardness values are obtained.

Fig. 8 shows typical Compressive strength as a function of CNTs wt. % which obtained from compressive tests for sintered pure Al and Al-CNTs nano-composites. These data demonstrate that the compressive strength increased from 184.69MPa (pure Al) to 280.50MPa (for composites containing 8wt. % CNTs), about a 52% increase. Therefore as a result of this experimental work, it can be concluded that efficiency of CNTs depends on dispersion, volume fraction of CNTs, and the CNT-Al interfacial strength.

The hardness and compressive strength values of the Al-CNTs were higher than those of Al without CNTs (Fig 7 and Fig.8). Three reasons could be considered for higher hardness and compression strength of the Al-CNTs composite including: the first reason is increment of CNTs phase as the higher dislocation motion produced by CNTs. Secondly the matrix of Al-CNTs MMCs contains higher dislocation density as result of dislocation generation due to differences in thermal expansion coefficient of aluminum and CNT which causes thermal mismatch(or Orowan looping) stresses resulting in increased dislocation

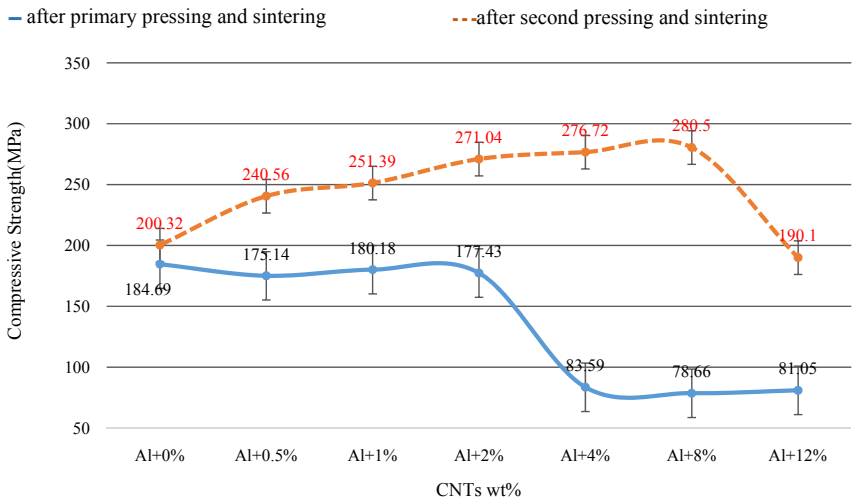

Fig. 8. Compressive strength of the Al-CNTs nano-composites as a function of wt.% CNTs, before and after second pressing and sintering of the samples.

density and in turn causes increment of Al-CNTs MMCs micro-hardness and compressive strength. Restriction in dislocation motion due to lower grain sizes of reinforcement also responsible for the higher hardness and compressive strength. The third reason is that generally the Al-CNTs mechanical properties are strongly influenced by the nature of interface between the CNTs phase and aluminum ductile matrix, as this interface controls the efficiency of the load transfer from the Al matrix to the CNTs. Further TEM studies are under way to find out possibility of any faults at interface regions between the matrix and CNTs.

CONCLUSIONS

Al-based composites reinforced up to 12wt. % MWCNTs were successfully fabricated by double-pressing double-sintering (DPDS) technique. The hardness and compressive strength increases with increasing CNT content to 8wt. % MWCNTs. The composite with 8wt. % CNTs content exhibits increment of about 64% and 52% in hardness and compressive strength respectively as compared with the hardness and compressive strength of aluminum (matrix) fabricated under the similar condition by conventional sintering process. Finally, it can be concluded that the DPDS method is an appropriate and the newly used method for the synthesis of Al-CNT composites.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interests regarding the publication of this paper.

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