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ORIGINAL RESEARCH

# A novel approach to the uniformly distributed carbon nanotubes with intact structure in aluminum matrix composite

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## Abstract

Achieving a uniform dispersion of carbon nanotubes (CNTs) in metal matrix composites (MMCs) is a vital prerequisite for enhancing the mechanical properties of the samples. In this work, a novel strategy called little by little (LbL) adding was successfully adopted in order to achieve a uniform dispersion of CNTs. The Al composite powders reinforced with different amounts of CNTs (1, 2, 3, and 5 wt.%) were consolidated by spark plasma sintering (SPS). Based on scanning electron microscopy (SEM) as well as Raman spectroscopy, individual and uniform dispersion of CNTs without any serious structural damage was achieved in Al matrix. In this regard, the relative intensity ratio ($I_D/I_G$) of the raw CNTs was around 0.85 and that of LbL composite was ~ 0.9, indicating that the CNT structure experienced very little damage during the applied method. As a result of the uniform dispersion, there was no drastic reduction in mechanical property (microhardness) of the LbL-composites with increasing CNT content. Based on the tribological tests, it was found that the dominant wear mechanism of the LbL composite is abrasive wear accompanied by adhesion wear. Moreover, for the reference composites, produced by conventional wet milling process, the dominant wear mechanism was severe adhesive.

**Keywords** Aluminum · Carbon nanotubes · Metal-matrix composites (MMCs) · Raman spectroscopy · Uniform dispersion

## 1 Introduction

Carbon nanotubes (CNTs) have been considered as ideal nano reinforcements for strengthening metal matrix composites due to their one-dimensional structures with large aspect ratios and unique mechanical, thermal, and electrical properties [1–6]. In the past decade, increasing attention was particularly paid to CNT reinforced Al matrix composites (AMCs), because the light weight along with high strength made them interesting structural materials in aerospace and automobile industries, where the fuel economy and weight reduction are the first priority [7–10]. It is worth noting that Al and its alloys are the first option for the composite matrix reinforced by CNTs. As mentioned before, AMCs are being considered as a group of newly advanced materials for their light weight, high strength, high specific modulus, low coefficient of thermal expansion, and good wear resistance properties [7]. These composites are fast becoming favorite choice in numerous applications such as bearing sleeves, piston, gears, valves, and cylinder liners. In many of these applications excellent friction and wear performance are required [8–10]. On the other hand, because of the mentioned extraordinary mechanical, thermal and electrical properties of CNTs, they have been used to improve the properties of many different materials such as polymers, metals, and ceramics. Specifically, CNT-reinforced metal matrix composites possess a huge potential to be widely used in structural and functional applications, such as automobile, aerospace, sports, micro-electrochemical systems (MEMS), sensors, battery, and energy storage [11].

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However, theoretical potential of CNTs has not yet been achieved in metal matrix composites [11, 12]. In order to acquire high strengthening potential of nanotubes in AMCs, a homogeneous dispersion of un-bundled CNTs is an essential issue. Nevertheless, nanotubes naturally tend to self-assemble into clusters even after attempts are made to disperse them as a result of the strong attractive force due to their high specific surface area [13–15]. Although many studies have been made to deal with nanotube dispersion, it is still a great challenge to uniformly distribute CNTs into AMCs with small structural damages [13, 16]. Recently, high energy ball-milling (HEBM) has been widely used to disperse CNTs into the metal matrix composites. It has been reported that mechanical milling can produce a composite with homogeneous distribution of CNTs [17]. Despite the good dispersion that can be achieved by HEBM, the morphology and structure of CNTs is inevitably damaged during mechanical milling, which will be detrimental to the strengthening effect of CNTs [13]. In other words, severe structural damages including CNT shortening and crystal-structure changes seem to be unavoidable at the sacrifice of appropriate CNT dispersion [17–19]. Apart from HEBM, several approaches, such as in situ synthesis [20], spray-drying, and nano-scale dispersion (NSD) [21, 22], have also been reported in order to produce homogeneous CNT/Al composites through powder metallurgy (PM). However, they still failed to achieve uniform and individual distribution of CNTs with intact structure. Based on the previous studies, there is a dilemma for the applied methods to keep balance between homogeneous dispersion and maintenance of the structural integrity of nanotubes. In this regard, a novel and unique development of a production process which promotes a homogeneous dispersion of CNTs in the metal matrix without damaging them, is necessary for obtaining nanocomposites with excellent mechanical and physical properties. Moreover, a significant progress in the future manufacturing of CNT-reinforced metal matrix composites is strongly essential when compared with the previous methods.

Herein, we reported for the first time a novel strategy called little by little (LbL) adding through which a uniform distribution of CNTs with intact structure can be achieved in Al composites. SEM as well as Raman spectroscopy was used to evaluate the quality of dispersion and structural integrity of CNT. Furthermore, microhardness and pin-on-disc wear tests were utilized to examine the mechanical and tribological behavior of the resultant composites.

## 2 Experimental procedure

Multiwall carbon nanotubes (MWCNTs) with outer diameter of around 20–40 nm and purity of more than 95% were manufactured by US Research Nanomaterials. Flaky Al powders (101,056 Merck, purity > 90%) were also supplied in order to elevate the quality of CNT dispersion. Two techniques were used for preparing the powder mixture; typical solution ball milling for 1 h (named conventional method) versus novel method based on solution milling at 6 stages (named LbL). From now on, the novel applied method is called little by little and will be discussed later in details. In the solution (ethanol) milling, ball to powder weight ratio (BPR) and rotation speed were 20:1 and 300, respectively. The obtained composite powders (1, 2, 3, and 5 wt.% CNT/Al) were sintered at 500 °C, with a holding time of 10 min, a heating rate of 50 °C/min, and a pressure of 40 MPa in a Φ10 mm graphite mold, using a SPS apparatus installed at Ferdowsi University of Mashhad (Iran). Microstructures of the composites were studied by a scanning electron microscope (SEM, LEO 1450VP) equipped with energy dispersive spectrometry (EDS) analysis. A transmission electron microscope (TEM, LEO 912AB) with an operating voltage of 120 kV was also utilized. Raman spectroscopy (Teksan-Takram P50C0R10) was performed by using a 532-nm argon laser as the excitation source to evaluate the structural integrity of CNTs. In addition, a microhardness tester (Buehler model 1600.61025) with a load of 100 g and dwelling time of 10 s was carried out to examine the mechanical properties of the samples. The average values of 10 points were calculated and reported. Finally, in order to evaluate the tribological behavior of the composites, pin-on-disk wear test was performed at room temperature, using a normal load of 10 N and a sliding speed of 0.2 m/s. An abrasive SiC disc with a hardness of approximately 2500 HV was used as a counterpart. Prior to each test, surface of the samples was carefully polished and cleaned ultrasonically. The sliding distance was kept constant at 200 m.

## 3 Results and discussion

The applied approach, in this study, to produce CNT/Al composites was proposed to address the existing problems in this field as illustrated in Fig. 1a. Flake powder metallurgy (Flake PM) is a favorable technique in MMCs due to its low cost, flexibility, and ease of control. Generally, flake Al powder has a much higher apparent volume than spherical powders, which is beneficial for the uniform distribution of CNTs [16]. Since spherical Al powders have small absorbing surface areas for accommodating CNT and in order to make the morphology of Al powders more compatible with the one dimensional (1-D) CNTs, flaky Al powders were supplied (see Fig. 1b). As can be seen in Fig. 1b, the flaky Al powders with a thickness of lower than 800 nm, have a 2-D planar morphology providing a large flat surface area for CNT accommodation. In addition, TEM image of the raw CNT is shown in Fig. 1c.

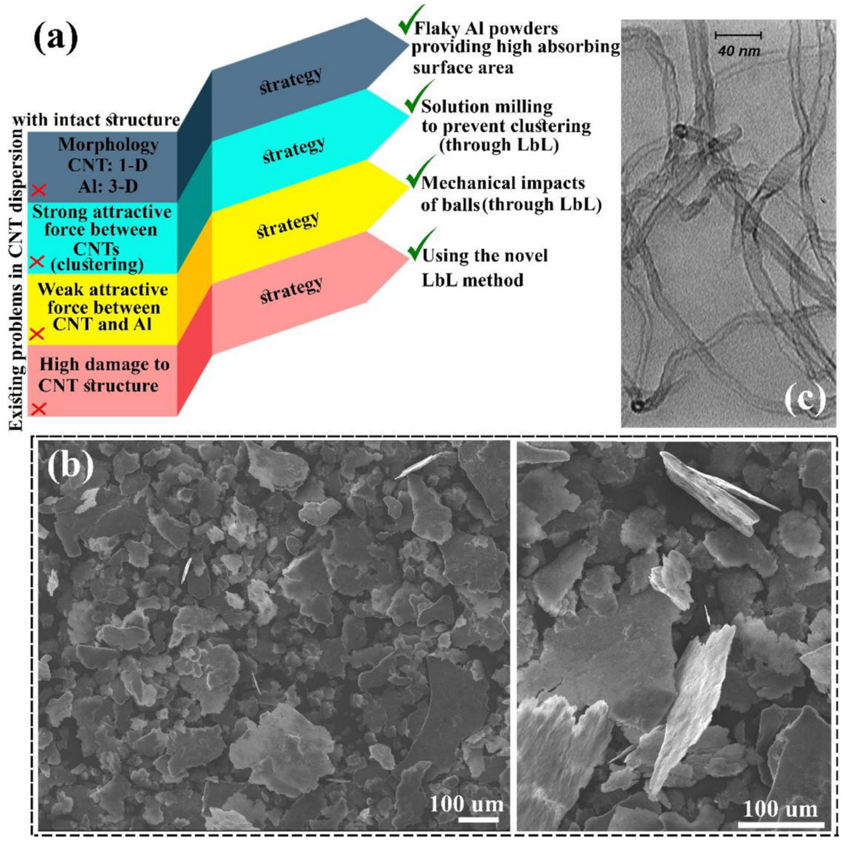


**Fig. 1** **a** Critical problems in CNT dispersion and the strategies for solving them through LbL method. SEM **b** and TEM **c** images of the raw flaky Al and CNT powders, respectively

Moreover, schematic illustration of the so-called little by little (LbL) method is shown in Fig. 2. In this method, the total amount of CNTs was divided into six portions being added through six sequential stages of 10 min milling with 10 min rest. This way, the first portion experienced 1 h milling while the last one was milled just 10 min, being designed to prevent damage of the CNTs structure. It should be mentioned that the total amount of Al was added to the milling vial at the first stage together with the first portion (1/6) of CNTs. Tip and bath sonication were also used for better dispersion of CNTs in ethanol prior to each stage. In this regard, a simple but effective approach was carried out to simultaneously resolve the mentioned problems in CNT dispersion (Fig. 1a). In order to better evaluate the LbL method, its CNT dispersion quality was compared with that of the conventional approach in CNT/Al composite system using the same processing parameters.

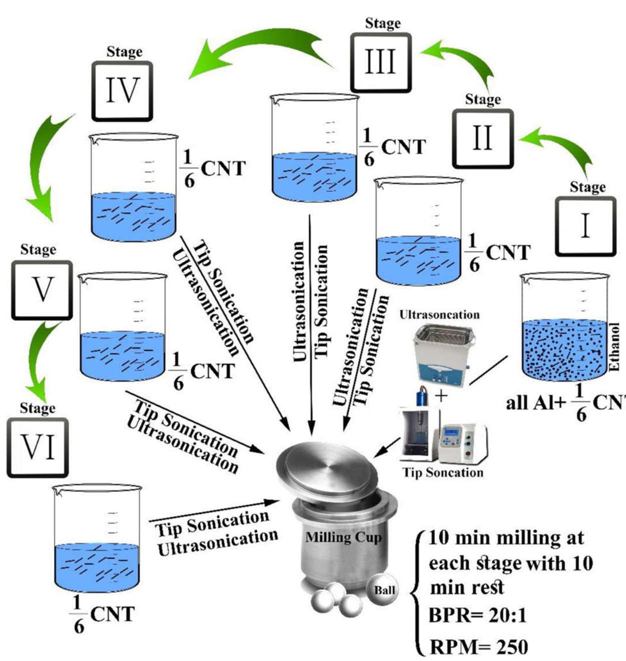


**Fig. 2** Schematic illustration of LbL method for CNT dispersion

Microstructures of the CNT/Al nanocomposites produced by different dispersion techniques are presented in Fig. 3. From the SEM images, a significant difference in the microstructure of the nanocomposites produced by LbL method (Fig. 3a–g) can be identified comparing with the reference (ref.) samples produced though the conventional technique (Fig. 3b–h). As can be seen, despite initial dispersion of CNTs in the reference composites, a huge amount of nanotubes is still agglomerated in the form of dark phases, which may originate from the clusters in the powder mixing stage. However, very small quantity of CNT clusters is found in the LbL samples in comparison with references (Fig. 3a–g). Therefore, as the applied method is changed from LbL to conventional process, dark areas clearly increase specially in

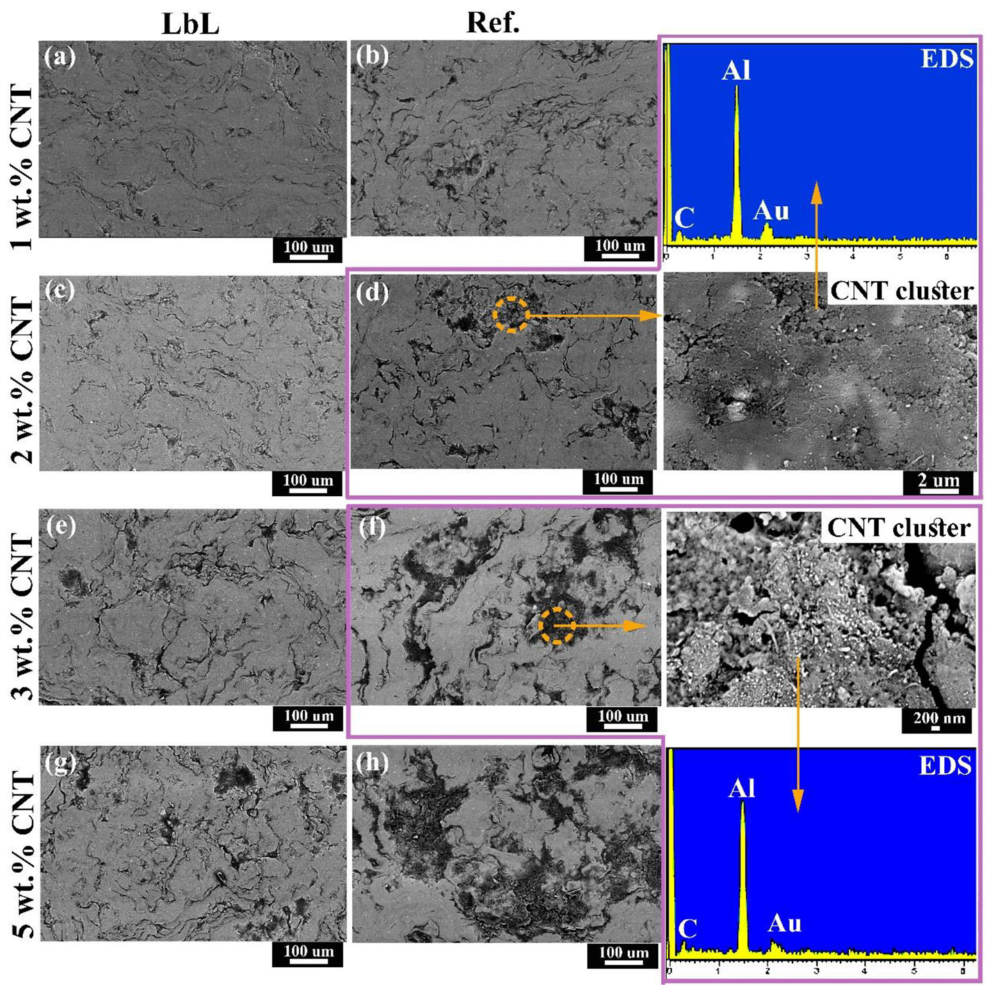


**Fig. 3** Typical SEM images of sintered Al nanocomposites containing different amounts of CNT produced by LbL (a, c, e, g) and conventional method (b, d, f, h). High magnification SEM as well as EDS analyses provided as attached images (d, f)

the case of 5 wt.% CNT/Al (Fig. 3h). Larger amounts of CNTs are strongly inclined to tangle together in the grain boundary of Al matrix, impeding the densification of the specimens, and thus leading to the decrease of the mechanical properties of the resultant composites [23]. It can be concluded that the applied method considerably affects the size of CNT clusters and dispersion quality. A higher magnification SEM image of a cluster is provided in Fig. 3 d and f along with EDS analyses as attached images, which further confirms the presence of CNTs in the dark areas.

Figure 4a reveals the morphology of CNT/Al powder mixture dispersed by the LbL process with CNT content of 2 wt.%. From this figure and also the magnified image of Fig. 4b, it is observed that CNTs are homogeneously dispersed in the matrix powder. An excellent dispersion of CNTs can be obviously seen on the flaky Al surfaces, including small flakes (Fig. 4b) and large Al flakes (Fig. 4c). Therefore, as can be seen, after applying the novel method, CNTs are almost disassembled and the individual ones have no distinct structural damage. Furthermore, the long tubular CNTs are visibly intact, showing the effectiveness of the applied technique (Fig. 4b). In conclusion, no CNT agglomerates are found in the Al matrix, which indicates the homogenous distribution of CNTs in the new method. On the other hand, highly agglomerated CNTs with the size of more than 5 μm can be obviously seen in Fig. 4d which belongs to the 2 wt.% CNT/Al reference powder sample.

It is worth mentioning that the dispersion stage is very important for reducing the number of clustering. Although short sintering duration of SPS effectively reduces CNT agglomeration, clusters formed in the previous processing stages, namely mixing and dispersing stage, could be carried over in the next step. It was found that applying LbL method reduces CNT clusters and finally results into more homogeneous dispersion of nanotubes. However, future studies need to be directed at other important CNT-MMC systems to prepare industry-acceptable process maps under powder metallurgy approach to achieve individual dispersion of CNTs without clustering. The novel applied method, in this study, controlled clustering of CNTs and contributed to the significant decrease of CNT agglomeration and clusters, which is helpful to eliminate internal defects and stress concentration as well as effective load transfer ability.

Structural integrity of CNTs can also be proved by Raman spectroscopy technique. The peaks located at around ~

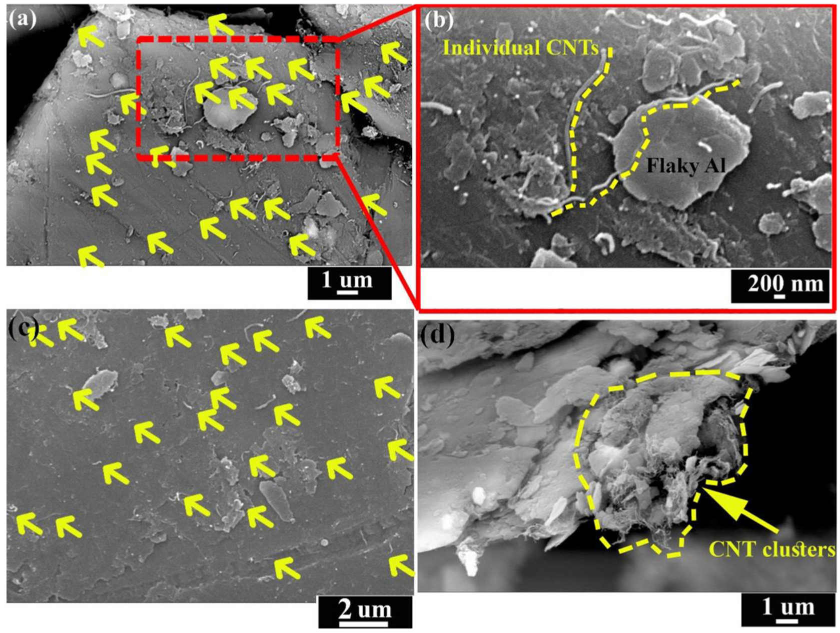


**Fig. 4** **a** Homogenous distribution of CNTs on flaky Al powder surface by LbL process with 2 wt.% CNT. **b** Magnified image of the marked area in section a. **c** Individual dispersion of CNTs on a large flake. **d** SEM image of a 2 wt.% CNT/Al powder mixture produce by conventional technique showing a large CNT cluster

1580 $cm^{-1}$ and ~ 1350 $cm^{-1}$, respectively, correspond to a typical G (graphite) and D (defect) bands [24]. The relative intensity between these two peaks is known to provide useful information regarding the quality of CNTs [25]. The Raman spectra of the raw CNTs as well as 3 wt.% CNT/Al composites produced by LbL and conventional process are shown in Fig. 5. As can be seen in Fig. 5, the relative intensity ratio ($I_D/I_G$) of the raw CNTs is approximately ~ 0.85 and that of LbL composite is ~ 0.9, indicating that the CNT structure experienced very little damage during the LbL processing in comparison with reference composite sample ($I_D/I_G$ = 1.1). Therefore, the possibility of keeping balance between uniform dispersion of CNTs and their structural integrity make LbL technique a promising route to prove the potential of CNT as a reinforcement in Al matrix as well as in other metal matrix composites.

In the case of mechanical properties of the LbL-composites, variations of the Vickers hardness with CNT content are approximately similar with that of the reference samples (Fig. 6a). In other words, with increasing the CNT content from 1 to 2 wt.%, the hardness values increase and thereafter decrease up to 5 wt.%. For a comparison, Vickers hardness of the pure Al is also added to the curve in Fig. 6a. However, unlike the reference samples, the variation of mechanical values of the LbL-composites is so smooth (~ 10%) while that of reference composites is around 55.5%. It indicates that applying the LbL method could control clustering of CNTs, and for this reason, there is no drastic decrease with increasing CNT content specially from 2 to 5 wt.%, in comparison with the reference samples (see Fig. 6a). Furthermore, the hardness values of LbL-composites are higher than that of reference-

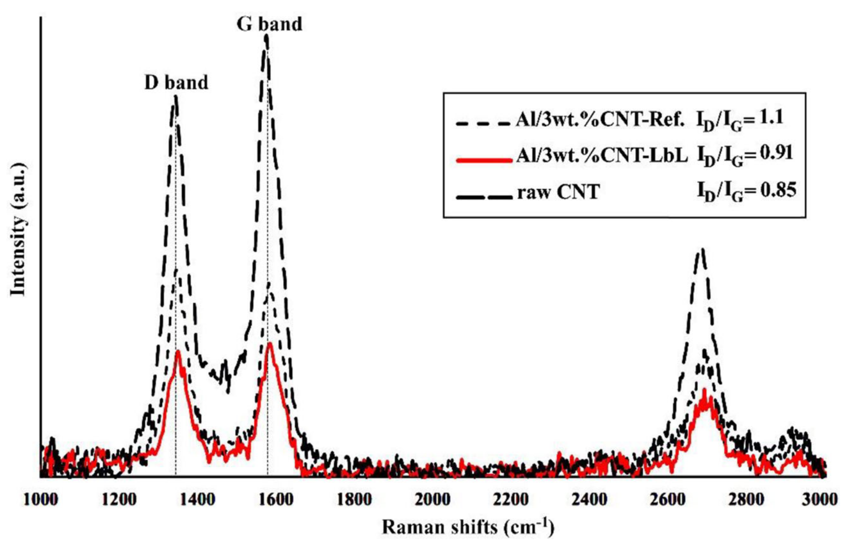


**Fig. 5** Raman spectra of the raw CNTs as well as composites reinforced with CNT produced by LbL and conventional methods

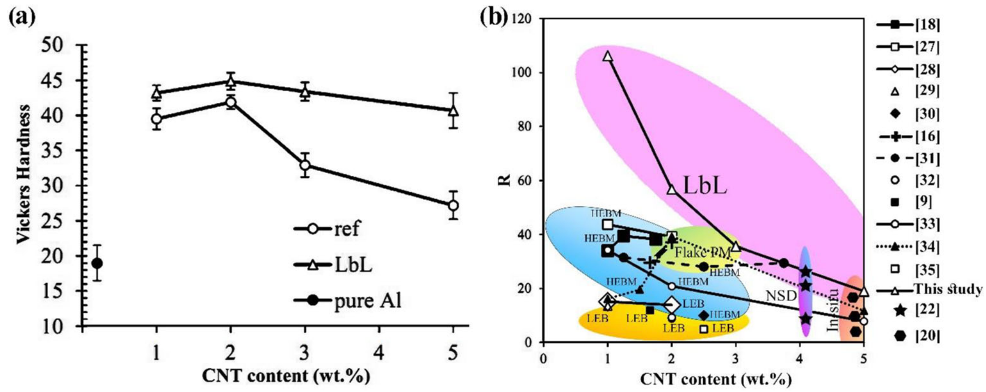


**Fig. 6** **a** Variation of microhardness (Vickers) of pure Al as well as LbL and reference composites as a function of CNT content, **b** the relationship of strengthening efficiency (R) and CNT content; comparison between this study and other publications

composites, revealing the effectiveness of the applied LbL method. As an instance, 2 wt.% CNT/Al LbL-composite exhibited ~ 136% and ~ 8% increase in Vickers hardness, compared with pure Al and 2 wt.% CNT/Al Ref-composite, respectively. Furthermore, comparing 5 wt.% CNT/Al composites produced by different methods, it is revealed that around 51% increase in the hardness can be achieved by utilizing the LbL method.

The strengthening efficiency (R) is generally used to characterize the strengthening effect of the reinforcement [26]. The advantage and superiority of the LbL method over other methods can be revealed by comparing the strengthening efficiency of CNTs, which is determined by $R = (\sigma_c - \sigma_m) / (V_{CNT}\sigma_m)$. Similar to the implication of yield strength, the microhardness is an indication of materials capability to resist plastic deformation. Therefore, similar formula could be proposed as follows:

$$\mathrm{R} = \frac{H_c - H_m}{V_{CNT} H_m} \tag{1}$$

$H_c$ and $H_m$ is hardness of composite and matrix, respectively. In addition, $V_{CNT}$ refers to volume fraction of CNT. The calculated data are also compared with the results of CNT/Al composites in other publications (see Fig. 6b). According to Fig. 6b, LbL-composites show the best strengthening efficiency of CNTs. It can be concluded that the applied method (LbL) could improve strengthening efficiency of CNT to the values higher than that of low energy blending (LEB), high energy ball milling (HEBM), nano-scale dispersion (NSD) as well as in situ and flake powder metallurgy methods with relatively better dispersion and less damage of CNT. The highest R of the Al composite containing ~ 2 wt.% CNT produced by flake powder metallurgy [16] was around 40, which is about 42% lower than the achieved value of R at the same amount of LbL-CNT in this study. The higher strengthening efficiency makes LbL-composite a promising material in the new class of high-performance MMCs. It can be concluded that CNT dispersion in the powder stage is very critical for obtaining high strengthening in the resultant composite. Using LbL technique leads to better distribution of nanotubes causing better CNT–matrix contact and load transfer, and finally, composite prepared by this method show better strengthening.

In order to evaluate the tribological behavior of the resultant composites produced by conventional and LbL methods, pin-on-disc wear test was carried out on the samples. SEM images of the representative worn surfaces of LbL and Ref composites reinforced with different amounts of CNTs (2 and 5 wt.%) are provided in Fig. 7a. As is obvious, the surface of the composites produced by LbL method, compared with the reference composites, has lower roughness and plastic deformations. However, in the case of composites produced by the conventional method, severe delamination and exfoliation can be observed. On the other hand, higher magnification SEM image of Fig. 7a reveals that the wear tracks on the surface of LbL composites are featureless and smoother along the sliding direction (S.D.) in comparison with the references. These visual and qualitative results of the SEM study are consistent with the mechanical behavior of the composites (see Fig. 6a) and will be further confirmed by comparing the tribological properties of LbL and Ref samples (Fig. 7b). Moreover, an obvious transition in the wear mechanism can be realized with changing the mixing method from conventional to LbL. Based on Fig. 7a, the dominant wear mechanism of the reference composites is severe adhesive wear with a significant exfoliation, while that of LbL composites is mild adhesive together with abrasive wear. Therefore, it can be concluded that the achieved uniform dispersion of CNT through LbL method extremely reduced the severity of the adhesion mechanism. Fig. 7b shows the variation of wear weight loss for the LbL and Ref composites as a function of CNT content (2 and 5 wt.%). As compared with Ref-CNT/Al composites, the tribological property is highly improved by applying LbL method. In this regard, a reduction of around 20% in wear weight loss was achieved for 2 wt.% and 5 wt.% LbL-CNT/Al composites compared with the reference samples. The better tribological results of LbL composites in comparison with the references could be attributed to the uniform distribution of CNTs which was achieved by LbL method. Figure 7c shows schematic illustration of flattening and unwrapping

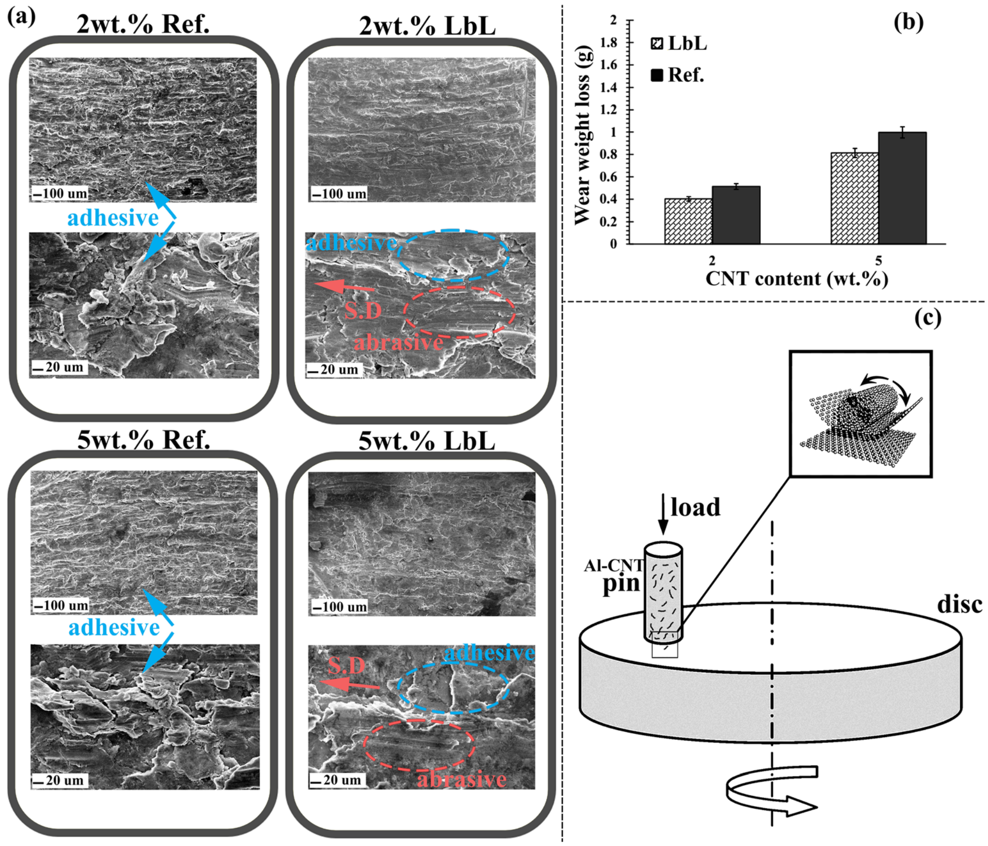


**Fig. 7** **a** SEM micrographs of the worn surfaces with varying CNT content for composites produced by different methods. Abbreviation of S.D. refer to the sliding direction. (b) Variation of wear weight loss as a function of CNT content and the applied method. (c) a schematic illustration, representing the flattening mechanism of CNT during wear test to form graphene-like lamella

mechanism of CNT during wear test. It is known that during friction, the well-dispersed nanotubes are removed from the surface of composites and transferred to the interface between the friction pair. Hence, CNTs can easily be rolled and then flattened to form graphene-like lamella, preventing close contact between the abrasive disc and the composites and therefore enhance the tribological behavior [36–39]. Finally, it can be deduced that the remaining CNTs with high strength, good ductility, and a cylindrical structure can act as a ball bearing during the friction process. Moreover, the easy sliding of their walls attached by weak van der Waals forces can probably lower the coefficient of friction.

As confirmed before, in the conventional method, the quality of CNT dispersion is strongly poor and large clusters are formed, specifically at higher amounts of CNTs (see Figs. 3 and 4). CNT clusters serve as a source of crack nucleation resulting in poor wear resistance of the composite [23]. In order to evaluate the quality of CNT dispersion in the both applied methods, worn surfaces of the composites were further studied by SEM at higher magnifications and the results are presented in Fig. 8. As can be seen in Fig. 8a, which belongs to 2 wt.% Ref-CNT/Al composite, CNT clustering was occurred in a micro-void located at the worn surface. Furthermore, the clustering become more aggravated in the reference composite reinforced by 5 wt.% CNT (see Fig. 8c). However, no CNT clusters are observed in the microstructure of the LbL-composites reinforced with 2 and 5 wt.% CNT, as shown in Fig. 8b and d, respectively. Individual dispersion of CNTs on the worn surface of 2 wt.% LbL-CNT/Al composite is marked by the arrows (see Fig. 8b). Figure 8 d shows a micro-crack which was produced during the wear test. In addition, high-magnification SEM image of the crack propagation path was shown in the inset of Fig. 8d. Notably, some individual CNTs which act as bridges across the crack can be observed in the figure. Therefore, CNT-crack bridging restrain crack propagation and play a toughening effect in the composite. The results show that LbL-CNTs act as bridges across micro-cracks which cannot be achieved by the conventional method (see CNT clustering in a micro-crack of 5 wt.% Ref-CNT/Al composite; inset of Fig. 8c). The fiber strengthening mechanism in bulk aluminum/CNT or other similar composite systems has always been inferred indirectly based on the post deformation fracture surface observation. The main evidences of fiber strengthening are deduced from the pull-out of CNTs and crack bridging.

In conclusion, LbL method has been proved to be successful in the dispersion of CNTs without serious damage in Al matrix and can be used in other nanocomposites. It is convincing that LbL technique would provide capable thoughts for further developments of MMCs in wide range of applications in the future.

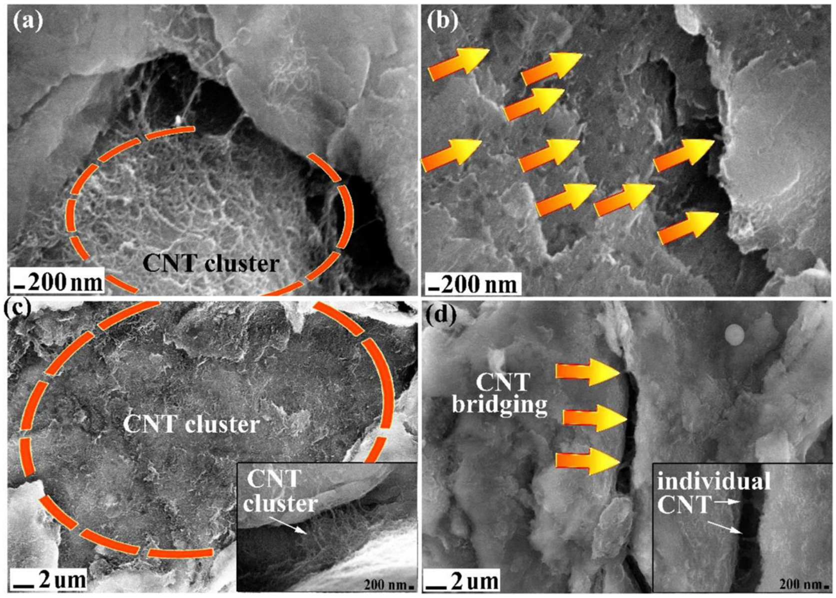


**Fig. 8** **a**, **c** CNT clustering at the worn surface of 2 and 5 wt.% CNT/Al Ref-composites, respectively. **b**, **d** Individual dispersion of CNTs at the worn surface of 2 and 5 wt.% CNT/Al LbL-composites, respectively. Insets show CNT clusters **c** and individual CNT bridging **d** at a crack located on the worn surfaces

## 4 Conclusions

A novel and effective approach, namely little by little (LbL) adding, for the uniform dispersion of CNTs in Al matrix was successfully adopted in this study. In order to evaluate the effect of CNT content, Al composite powders reinforced with different amounts of CNTs (1, 2, 3, and 5 wt.%) were consolidated through spark plasma sintering (SPS). According to the SEM images and Raman spectroscopy data, it was found that the individual dispersion of CNTs with intact structures can be achieved in Al matrix through LbL method. The relative intensity ratio ($I_D/I_G$) of the raw CNTs was ~ 0.85 and that of LbL composite was ~ 0.9, indicating that the CNT structure experienced very little damage during LbL. It was revealed that with increasing CNT content from 1 to 2 wt.%, the microhardness values increase and thereafter decrease up to 5 wt.% at both conventional and LbL methods. However, unlike the reference samples, the variation of mechanical values of the LbL-composites was so smooth (~ 10%) while that of reference composites was around 55.5% which could be attributed to the uniform dispersion of CNT by LbL technique. In addition, the hardness values of LbL composites were higher than that of reference-composites, revealing the effectiveness of the applied method. As an instance, 2 wt.% CNT/Al LbL composite exhibited ~ 136% and ~ 8% increase in Vickers hardness, compared with pure Al and 2 wt.% CNT/Al Ref composite, respectively. Furthermore, comparing 5 wt.% CNT/Al composites produced through different methods, it was shown that around 51% increase in the hardness can be achieved by utilizing the LbL method. Based on the tribological tests, it was found that the dominant wear mechanism of the LbL composite is abrasive wear accompanied by mild adhesion wear, while for the reference composites, the dominant wear mechanism was severe adhesive. Furthermore, a reduction of around 20% in wear weight loss was achieved for 2 wt.% and 5 wt.% LbL-CNT/Al composites compared to the reference samples. Finally, a successful development of a production process that promotes a homogeneous dispersion of CNTs in the matrix without damaging them is essential for obtaining nanocomposites with excellent mechanical and physical properties, and of course a significant progress in the future manufacturing of CNT/metal matrix composite when compared with the previous methods is strongly needed. Beside the mentioned advantages of uniform dispersion of CNT in the matrix (in comparison with reference samples) without scarifying structural integrity of the tubes, which were achieved by LbL method, very small clusters of CNTs were still observed in the microstructure. Therefore, future work needs to focus on maintaining the integrity while achieving individual dispersion of CNTs without any clustering.

## Compliance with ethical standards

**Conflict of interest** The authors declare that there is no conflict of interest.

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