Young's Modulus Enhancement and Measurement in CNT/Al Nanocomposites

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Abstract

Young's modulus is a critical parameter for designing lightweight structure, but Al and its alloys only demonstrate a limited value of 70–72 GPa. The introduction of carbon nanotubes (CNTs) is an effective way to make Al and its alloys stiffer. However, little research attention has been paid to Young's modulus of CNT/Al nanocomposites attributed to the uncertain measurement and unconvincing stiffening effect of CNTs. In this work, improved Young's modulus of 82.4 ± 0.4 GPa has been achieved in 1.5 wt% CNT/Al nanocomposite fabricated by flake powder metallurgy, which was determined by resonance test and 13.5% higher than 72.6 ± 0.64 GPa of Al matrix. A comparative study and statistical analysis further revealed that Young's modulus determined by tensile test was relatively imprecise (83.1 ± 4.0 GPa) due to the low-stress microplasticity or interface decohesion during tensile deformation of CNT/Al nanocomposite, while the value (98–100 GPa) was highly overestimated by nanoindentation due to the “pile-up” effect. This work shows an in-depth discussion on studying Young's modulus of CNT/Al nanocomposites.

Keywords CNT/Al nanocomposites · Young's modulus · Stiffening · Resonance test

1 Introduction

To date, carbon nanotubes (CNTs) have been widely used as reinforcements in metal matrix composites due to their exceptional modulus and strength [1]. In particular, CNT/Al nanocomposites have attracted lots of attentions, driven by the desire of weight reduction in the aerospace and automotive industries [2, 3]. Compared with aluminum

alloys, enhanced Young's modulus is the most advantageous feature of CNT/Al nanocomposites in lightweight structural design [4].

But unfortunately, Young's modulus of CNT/Al nanocomposites has been seldom reported so far. Even worse, there were large scattering among the limited results, making the stiffening effect of CNTs quite unconvincing. Such phenomenon was caused by two problems. One is CNT clusters and/or weak CNT/Al interface resulting from immature material fabrication in the early studies, which could be well settled with recently developed processing techniques, such as flake powder metallurgy [5], solution ball coating [6], and friction stir processing [7]. The other one is the uncertainty in measuring Young's modulus with different testing methods, including tensile test, nanoindentation, and resonance test. Generally, tensile test was the most popular method for measuring Young's modulus of CNT/Al nanocomposites due to its universality and convenience. But the values measured by tensile test distributed over a quite large range from 66.7 to 110.1 GPa in different studies [5, 8–20] as shown in Fig. 1a. Typically, the Young's modulus of 1.2 vol% CNT/Al nanocomposites fabricated by ultrasonic

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Fig. 1 Young's modulus measured by **a** tensile test, **b** nanoindentation from previous studies of CNT/Al nanocomposites

shaking were distinctly different in two studies (88.0 and 102.2 GPa, respectively [19, 20]). In addition, Young's modulus measured by nanoindentation also demonstrated large data scattering as plotted in Fig. 1b [18, 21–30]. As a comparison, resonance test was used only once in the study of CNT/Al nanocomposites so far [31]. Therefore, the uncertain and inaccurate measurement has become the biggest obstacle in studying Young's modulus of CNT/Al nanocomposite.

In this paper, tensile test, nanoindentation and resonance test have been applied to acquire a valid and credible Young's modulus of 1.5 wt% CNT/Al nanocomposite fabricated through flake powder metallurgy (Flake PM). Standard errors and deviations of as-measured Young's modulus were calculated to quantitatively compare the measurement accuracy and precision of each method. The Young's modulus of Al matrix was also measured through the same manner for reference. The stiffening effect of CNTs, possible causes of measurement inaccuracy and imprecision, improvement strategies, and suitable application situations were discussed.

2 Materials and Methods

2.1 Fabrication of CNT/Al Nanocomposite Samples

Aluminum alloy powders from the AA5083 series (about 4 wt% Mg, 0.5 wt% Mn and 0.1% Cr for the main alloying elements) with spherical shape and an average particle size of 35 μm were used as the Al matrix material. Multi-walled CNTs (99.9% in purity fabricated via catalytic vapor deposition with high-temperature graphitization treatment from TimesNano, China) of ~ 20 nm in diameter and 10 μm in length were used as the reinforcement. The raw aluminum powders were ball-milled with ball-to-powder weight ratio

of 20:1 and milling speed of 423 rpm for 4 h in flowing Ar atmosphere with water cooling. The as-prepared Al nanoflakes were first surface modified by polyvinyl alcohol (PVA) and then mixed with CNT suspension to get the 1.5 wt% CNT/Al composite powders, which were then heated in flowing Ar atmosphere at 400 $^{\circ}\text{C}$ for 2 h to remove the PVA. The CNT content of 1.5 wt% was chosen with the consideration of both uniform CNT dispersion and strong stiffening effect. More details can be found in our previous studies about Flake PM [5]. Then, the as-prepared 1.5 wt% CNT/Al composite powders were cold pressed into a cylinder of 39 mm in diameter by uniaxial pressure of 350 MPa. Subsequently, the green billets were sintered at 510 $^{\circ}\text{C}$ in vacuum for 2 h, followed by forging into a cylinder of 40 mm in diameter at 420 $^{\circ}\text{C}$ and extrusion at 450 $^{\circ}\text{C}$ into a rod of 8 mm in diameter. The Al matrix samples were also prepared through the same processing route.

2.2 Microstructure Characterization

A field-emission scanning electron microscope (SEM, Sirion 200 at an accelerating voltage of 5 kV) was used to characterize the morphology of the as-prepared CNT/Al composite powders. Raman spectrum was recorded with a microscopic laser Raman spectrometer (Senterra R200-L, Bruker Co. Ltd., German) in the backscattering geometry, and by using the 532-nm line of a diode-pumped solid-state laser in the spectral range from 400 to 2000 cm^{-1} . A wide-field confocal microscope (ZEISS Smartproof 5) and a field-emission transmission electron microscope (TEM, JEOL-2100F at an operating voltage of 200 kV) were used to characterize the microstructure of the as-fabricated CNT/Al nanocomposite. TEM samples were prepared by mechanical polishing to reduce sample thickness down to around 30–50 μm . Final ion milling of the punched disk samples, having 3 mm in diameter, was carried out by using a Gatan Model 691 precision ion polishing system.

Accelerating voltage was set to 5 kV and dual ion sources with milling angles of 10° was employed. Total ion milling time was in the range of 2–3 h to reach electron transparency.

2.3 Young's Modulus Measurement

Quasi-static tensile test, nanoindentation, and resonance test were applied to measure Young's modulus of the as-fabricated CNT/Al nanocomposite. Five dog-bone-shaped tensile specimens of 25 mm in gauge length and 5 mm in diameter were machined from the extruded rods with extrusion direction parallel to load direction. The preload was set as 10 MPa and the specimen was clamped by wedge gripping. Quasi-static tensile test was carried out on a Zwick Z100 tensile tester with a strain rate of 3×10^{-4} at room temperature. To obtain valid result, the Young's moduli within different proportional limits were calculated, which started from preload of 10 MPa to 50, 100, 150, 200, 250 and 300 MPa. Quasi-static tensile test was carried out on a Zwick Z100 tensile tester with a strain rate of 3×10^{-4} at room temperature. Five spots on polished surface were tested using nanoindentation under both static and continuous stiffness measurement (CSM) modes. To avoid mutual influence between different indents, a maximum indentation depth of 1 μm and a lateral spacing of 20 μm were set. The major difference between static and CSM modes is that modulus will be measured for only once during the loading of one static nanoindentation, while modulus was continuously measured at different depth in just one CSM nanoindentation. Detailed information of static and CSM modes can be found in Ref. [32]. Five cylindrical specimens with 60 mm in length and 6 mm in diameter were prepared for resonance test on ET-RT modulus tester from Nihon Tech. Three separate tests were run to obtain the average Young's modulus of one specimen. Young's modulus of the Al matrix samples was also measured by three methods for reference and the results are listed in Supplementary Material.

3 Results and Discussion

3.1 Morphology and Microstructure of CNT/Al Composite Powders and Nanocomposite

The as-fabricated CNT/Al composite powders were in flaky shape (Fig. 2a), and CNTs were uniformly dispersed on the surface (Fig. 2b). The powder shape was inherited from the ball-milled Al powder, which was plastically deformed due to the collision of milling balls and became

flaky [33, 34]. After compaction and extrusion of CNT/Al powders, the CNT/Al nanocomposite was well densified without obvious micropores as indicated by Fig. 2c. In Fig. 2d, the Raman spectrum revealed that no carbide formation was detected in the extruded sample. The intensity ratio of D band to G band (I_D/I_G ratio) is usually a good indicator of the quality of CNTs, which semiquantitatively represents the ratio of disordered carbon to graphite carbon. Lower ratio means better structure integrity of CNTs [35]. The slightly increased I_D/I_G ratio from 0.24 to 0.3 indicated well-reserved CNT structure after flake PM processing. In addition, (002) plane of CNTs with an inter-plane spacing of 0.34 nm can be clearly identified by the HRTEM (high-resolution TEM) in Fig. 2e, which also demonstrated well-bonded CNT/Al interface without interfacial defects or decohesion. The uniform dispersion and well-reserved structure of CNTs along with the tight CNT/Al interface assured good stiffening effect in the as-fabricated CNT/Al nanocomposite sample [36], which provides solid basis for further comparative study on measurement of Young's modulus.

3.2 Young's Modulus Measurement by Tensile Test

The engineering stress–strain curves of 1.5 wt% CNT/Al nanocomposite samples are plotted in Fig. 3a. Five samples exhibited similar ultimate tensile strength around 460 MPa and total elongations ranging from 3.8% to 5.4%. An obvious deviation from elastic linearity was present above tensile stress of 300 MPa on all curves, indicating the occurrence of macroscopic plastic deformation. Actually, for metal matrix composites, the stiffness and thermal mismatches between reinforcements and matrix can give rise to large point-to-point variations in stress, which usually leads to low-stress microplasticity [37] and a premature departure from elastic linearity [38, 39]. This phenomenon makes it difficult to determine an appropriate elastic range or linear portion for Young's modulus measurement [40]. In view of this, Young's modulus of different elastic ranges were calculated to obtain a valid value, which starts from preload of 10 MPa to proportional limits of 50, 100, 150, 200, 250, and 300 MPa. The as-measured result is given in Fig. 3b and summarized in Table 1.

For proportional limits above 200 MPa, the measured values are generally lower than the ones under 200 MPa, which is believed to be caused by the occurrence of microplasticity or possible interface decohesion above 200 MPa [41]. In the meantime, there were large scattering among the values measured under 100 MPa, which should be attributed to the initial curve fluctuation caused by

Fig. 2 SEM images of **a** flaky CNT/Al composite powders, **b** well dispersed CNTs on Al flakes. **c** Optical image of extruded nanocomposite. **d** Raman spectrum of raw CNTs and extruded nanocomposite. **e** HRTEM image of CNT/Al interface

Fig. 3 **a** Engineering stress–strain curves of 1.5 wt% CNT/Al nanocomposite samples; **b** Young's modulus measured within different elastic ranges (from preload of 10 MPa to different proportional limits of 50, 100, 150, 200, 250 and 300 MPa)

specimen clamping and alignment during initial loading. To minimize the influence of curve fluctuation at low stress and avoid microplasticity at high stress, 150 MPa is considered as an appropriate proportional limit, and the

smallest measurement standard deviation of 4.4 GPa also verified this point. Thus, the Young's modulus of 1.5 wt% CNT/Al nanocomposite measured by quasi-static tensile test would be taken as 83.1 ± 4.0 GPa.

Table 1 Young's modulus of 1.5 wt% CNT/Al nanocomposite samples measured by quasi-static tensile test within different elastic ranges (from preload of 10 MPa to different proportional limits)

Sample no.	Young's modulus (GPa) measured with different proportional limits					
	50 MPa	100 MPa	150 MPa	200 MPa	250 MPa	300 MPa
1	65.2	80.1	83.2	85.9	82.1	76.2
2	76.1	78.9	77.0	74.7	72.0	68.8
3	83.8	83.5	82.1	80.1	77.4	74.1
4	84.4	85.5	83.7	80.0	75.7	71.5
5	88.3	95.5	89.4	89.5	84.6	78.2
Avg.	79.6	84.7	83.1	82.0	78.4	73.8
SD	8.2	5.9	4.0	5.1	4.5	3.3

3.3 Young's Modulus Measurement by Nanoindentation

Load–displacement curves obtained by static nanoindentations and Young's modulus–depth curves obtained by CSM nanoindentations are shown in Fig. 4. The insert SEM images show the well-spaced triangle-shaped indents on the polished surfaces. In Fig. 4a, similar slopes of unloading curves indicate similar Young's modulus in each static nanoindentation. In Fig. 4b, severe fluctuations during initial loading were present in all curves, which were usually caused by the surface microroughness and oxidation layer at small indentation depth [32]. When indentation depth increased to ~ 100 nm, the curve became smooth and flat. Thus, the Young's modulus measured in one CSM nanoindentation was taken as the average value from depth of 100–1000 nm, and the details are listed in Table 2. Then, the as-measured Young's modulus of 1.5 wt% CNT/Al nanocomposite samples are 100.6 ± 0.7 and 98.0 ± 0.3 GPa for static and CSM nanoindentations, respectively.

Table 2 Young's modulus of 1.5 wt% CNT/Al nanocomposite samples measured by static and CSM nanoindentations

Method	Young's modulus (GPa)						
	Spot 1	Spot 2	Spot 3	Spot 4	Spot 5	Avg.	SD
Static	99.4	100.3	101.1	101.6	100.8	100.6	0.7
CSM	98.1	98.3	98.0	98.0	97.5	98.0	0.3

3.4 Young's Modulus Measurement by Resonance Test

The geometrical information and flexural vibration resonance frequency (f_f) measured by resonance test of CNT/Al nanocomposite samples are listed in Table 3. It should be mentioned that the f_f values of a single specimen measured in three separate tests were consistently the same, which indicated high stability and good repeatability of resonance test. In Eq. (1), L , d , and m stand for the length, diameter, and mass of the samples as listed in Table 3, respectively. T is an equipment coefficient, which is 1.05 in this study. Based on Eq. (1), the Young's modulus of 1.5 wt% CNT/Al nanocomposite was measured as 82.4 ± 0.4 GPa.

Fig. 4 **a** Load–displacement curves obtained by static nanoindentations, **b** Young's modulus–depth curves obtained by CSM nanoindentations of 1.5 wt% CNT/Al nanocomposite samples. The inset SEM images show the morphology of indentations

Table 3 Geometrical information, flexural vibration resonance frequency, and Young's modulus of 1.5 wt% CNT/Al nanocomposite samples measured by resonance test

Specimen no.	Length (mm)	Mass (g)	Diameter (mm)	f_f (kHz)	Young's modulus (GPa)
1	60.01	4.63	5.99	7.90	81.9
2	59.98	4.61	5.97	7.94	83.0
3	59.99	4.65	6.01	7.97	82.5
4	60.05	4.55	5.94	7.83	82.0
5	60.23	4.71	6.05	8.00	82.6
Avg.	—	—	—	—	82.4
SD	—	—	—	—	0.4

$$E = 1.6067 \times 10^{-9} \frac{L^3 m}{d^4} f_f^2 T. \quad (1)$$

3.5 Comparison of Measurement Accuracy and Precision

In supplementary material, the Young's moduli of Al matrix were also measured by tensile test, static/CSM nanoindentation, and resonance test through the same manner. The average measured values were 72.3, 90.7 (Static), 88.0 (CSM), and 72.6 GPa, respectively. The corresponding average values of 1.5 wt% CNT/Al nanocomposite were 83.1, 100.6, 98.0, and 82.4 GPa, which are 14.9, 10.9, 11.4, and 13.5% higher than those of Al matrix, respectively. Thus, the incorporation of CNTs indeed significantly enhanced the Young's modulus of Al matrix. However, the values measured by different methods were quite distinct, which still left some doubts on the stiffening effect of CNTs.

For finding out an appropriate measurement method and acquiring valid values of Young's modulus, standard errors (σ) and standard deviations (S) of as-measured Young's modulus were calculated with Eqs. (2) and (3) to quantitatively compare the measurement accuracy and precision of each method. The lower the values of σ and S , the higher the measurement accuracy and precision.

$$\sigma = \sqrt{\frac{1}{n} \sum_{i=1}^n (E_i - E_t)^2}, \quad (2)$$

$$S = \sqrt{\frac{1}{n} \sum_{i=1}^n (E_i - \bar{E})^2}, \quad (3)$$

where E_i is the measured Young's modulus in one test, E_t is the true Young's modulus of 1.5 wt% CNT/Al nanocomposite sample and $\bar{E}$ is the average value of Young's modulus measured by the same method.

Here, a widely acknowledged generalized shear-lag model [14, 24, 42] is applied to theoretically predict the Young's modulus of 1.5 wt% CNT/Al nanocomposite, which will be taken as E_t in following calculation. The equations are as follows:

$$E_c = (1 - V_{\text{CNTs}})E_{\text{Al}} + \left(1 - \frac{\tanh(nS)}{nS}\right)V_{\text{CNTs}}E_{\text{CNTs}}, \quad (4)$$

$$n = \sqrt{\frac{2E_{\text{Al}}}{E_{\text{CNTs}}(1 + \gamma_{\text{Al}})\ln(1/V_{\text{CNTs}})}}, \quad (5)$$

$$V_{\text{CNTs}} = \frac{w_{\text{CNTs}}/\rho_{\text{CNTs}}}{1 - w_{\text{CNTs}}/\rho_{\text{Al}} + w_{\text{CNTs}}/\rho_{\text{CNTs}}}, \quad (6)$$

where E_c , E_{Al} , and E_{CNTs} are the Young's modulus of nanocomposites, Al matrix, and CNTs, respectively. Here, E_{Al} is taken as 71 GPa according to ASM handbook [43]. E_{CNTs} was taken as 800 GPa according to experimental measurement by Demczyk et al. [44]. γ_{Al} is the Poisson's ratio of aluminum matrix, taken as 0.33. S is the aspect ratio of CNTs, taken as 500. V_{CNTs} is the volume fraction of the CNTs, w_{CNTs} is the weight fraction of CNTs. ρ_{CNTs} and ρ_{Al} are the density of CNTs and Al matrix, which are taken as 2.1 [45] and 2.66 [43] g/cm³, respectively. After this calculation, the theoretical Young's modulus is predicted as $E_c = 84.9$ GPa. The values of σ and S along with the fitted normal distribution curves are demonstrated in Fig. 5.

In Fig. 5a, static and CSM nanoindentation demonstrated considerable σ values of 15.8 and 13.1 GPa compared with 4.3 GPa of quasi-static tensile test and 2.5 GPa of resonance test, indicating significantly inaccurate measurement of Young's modulus using nanoindentation. Such inaccuracy should mainly be caused by the "pile-up" effect, which is the indented material being pushed out of testing surface and causes the underestimation of contact area. In nanoindentation test, the unloading slope at peak load, known as contact stiffness $S = dP/dh$, can be used to calculate the Young's modulus. Based on Sneddon relationships [46], a geometry-independent relation involving contact stiffness (S), contact area (A), and elastic modulus can be derived as Eq. (7):

$$S = 2\beta\sqrt{\frac{A}{\pi}}E_r \quad (7)$$

where β is a constant related with indenter geometry ($\beta = 1.034$ for a Berkovich indenter), A is the contact area and E_r is reduced elastic modulus, which accounts for the

Fig. 5 **a** Standard errors, **b** standard deviations, **c** fitted normal distribution of experimental Young's modulus measured by tensile test, nanoindentation and resonance test in 1.5 wt% CNT/Al nanocomposite

simultaneous elastic deformation in both sample and indenter. E_r is given by Eq. (8):

$$\frac{1}{E_r} = \frac{1 - \nu^2}{E} + \frac{1 - \nu_i^2}{E_i} \quad (8)$$

where E and ν are the Young's modulus and Poisson's ratio for the sample, respectively, and E_i and ν_i are the same quantities for the indenter. Seen from Eqs. (7) and (8), when a certain value of contact stiffness is measured, Young's modulus is inversely proportional to the contact area. In this way, significant overestimation of Young's modulus will be caused by the underestimation of contact area due to "pile-up" effect [47, 48]. In comparison, the Young's modulus measured by tensile test and resonance test (83.1 and 82.4 GPa, respectively) were much closer to theoretical value (84.9 GPa), which should be relatively accurate and valid.

In Fig. 5b, quasi-static tensile test exhibited much bigger S values of 4.0 GPa compared with 0.7 and 0.3 GPa of static and CSM nanoindentation and 0.4 GPa of resonance test,

indicating imprecise or unstable measurement of Young's modulus using tensile test. The feature of low-stress microplasticity or possible interface decohesion in CNT/Al nanocomposite are the major cause of low measurement precision of tensile test. Studies of other metal matrix composite material also suffer from the same problem, and efforts have been made to improve the measurement precision through intermittent unloading or fatigue, which only produced little effect unfortunately [40, 49]. Unlike tensile test, nanoindentation and resonance test are featured with small testing scale and tiny applied load, which made them much more precise for measuring Young's modulus of CNT/Al nanocomposite.

As indicated by the narrow span and accurate position of the fitted normal distribution curve in Fig. 5c, resonance test should be the most appropriate method for both accurate and precise measurement of Young's modulus in CNT/Al nanocomposite. The slight deviation of experimental Young's modulus (82.4 GPa) from theoretical prediction (84.9 GPa) should be attributed to two reasons: The Young's modulus of the CNTs used in this study was actually not as

high as 800 GPa, and CNTs were not perfectly aligned in the bulk material. For tensile test, in view of its low measurement precision, repetitive experiment is necessary to avoid accidental result and acquire valid Young's modulus. Although the absolute value measured by nanoindentation is usually overestimated, the feature of small testing scale can still be useful for comparative study on microscale elasticity in one or between different samples. Thus, in this study, the resonance test measured value of 82.4 ± 0.4 GPa will be taken as a valid and credible Young's modulus of as-fabricated 1.5 wt% CNT/Al nanocomposite, which is 13.5% higher than 72.6 ± 0.64 GPa of Al matrix.

In general, uniform CNTs dispersion and good CNT/Al interfacial bonding are necessary for enhancing Young's modulus through the incorporation of CNTs into aluminum matrix and flake powder metallurgy was considered as an effective method for achieving this goal [50]. Tensile test, static/CSM nanoindentation, and resonance test all identified Young's modulus increase by 14.9, 10.9, 11.4, and 13.5% in the Flake PM fabricated CNT/Al nanocomposite. The comparative study between different measurement methods reveals that resonance test is an appropriate method to both accurately and precisely measure Young's modulus of CNT/Al nanocomposite, which may assist in future studies on stiffening mechanisms.

4 Conclusions

In this work, 1.5 wt% CNT/Al nanocomposite was fabricated by flake powder metallurgy to study the stiffening effect of CNTs. Tensile test, nanoindentation and resonance test have been applied to acquire credible Young's modulus of the composite. Statistical analysis in standard errors and deviations of the as-measured values was conducted. The results revealed that:

- (1) According to resonance test and statistical analysis, the introduction of 1.5 wt% CNT could significantly enhance the Young's modulus of the aluminum matrix (72.6 ± 0.64 GPa) by 13.5% (82.4 ± 0.4 GPa) owing to the uniform CNT distribution and good interfacial bonding.
- (2) Tensile test demonstrated accurate but imprecise measurement of Young's modulus (83.1 ± 4.0 GPa) due to the low-stress microplasticity or possible interface decohesion during tensile deformation of CNT/Al nanocomposite, while nanoindentation significantly overestimated the value (98–100 GPa) due to the “pile-up” effect.
- (3) Resonance test demonstrated both accurate and precise measurement of Young's modulus due to tiny testing load and reliable determination of resonance

frequency, which is recommended for studying Young's modulus in such kinds of nanocomposites.

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