Stress transfer efficiency in aligned multi-wall carbon nanotubes sheet / epoxy composites

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Abstract

Stress transfer efficiency for 60 walls multi-walled carbon nanotube (MWNT) was calculated by the shear-lag theory. The results indicated that stress transfer between walls is insufficient at several tens of nanometer from the end; however stress converged to the same value for adequate length. Effective young's modulus was also calculated and indicated that MWNT can be treated as short fiber for over 20 μm length (Aspect ratio > 400). The results implied that interfacial slippage or debonding area exists at the end of the MWNTs. Calculated results, including the alignment and interfacial strength between carbon nanotube (CNTs) and polymer, were compared to the mechanical

properties of aligned carbon nanotube sheet / epoxy composite to evaluate the validity of reinforcement mechanism of [MWNT](#). The experimental results were able to be explained reasonably by the numerical results.

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1. Background

A lot of experimental and theoretical researches for using carbon nanotubes (CNTs) as a reinforcement of polymer were conducted for the last two decades [1-12]. However, most studies for enhancing mechanical properties of CNTs/polymer composites were resulted in less increase compared with predictions.

The difference between theoretical prediction and experimental results was generally thought to be caused by length or alignment of CNTs, interfacial shear strength (IFSS) between CNTs and polymer, and dispersion of CNTs in the composites.

Length or alignment of CNTs are especially thought to be important factor. Li and Chou applied “microtome cutting” method to align CNTs unidirectionally and resulted in standard deviation of 15 degrees for arbitrary direction [13]. Furthermore, production method of long and aligned CNT sheet, which were vertically drawn from horizontally grown CNT array using SiO₂ or glass wafers, were established in recent years. This method has some advantages; this method is easy and simple to control the alignment of CNTs, and size of CNT sheets can be easily controlled. As told previously, alignment of CNTs is generally thought to be an important factor for enhancing mechanical properties of composites, which is the main reason that aligned CNT sheets are expected as ideal fillers for polymers. Several groups fabricated CNT bucky paper or aligned CNT sheet from CNT array to evaluate the mechanical properties of CNTs/polymer composites. The composites resulted in sharp increase of volume fraction, young’s modulus and strength [14-21].

Efficient production method of CNT arrays which can be easily drawn to fabricate CNT sheet is important factor for producing aligned CNTs sheet. Inoue et al proposed an efficient technique to grow up multi-walled CNTs forest with high quality using iron chloride as a precursor of catalyst in recent days [22]. They can grow up CNTs to the length of several millimeters in only 20 minutes by thermal chemical vapor deposition (CVD) method [23].

Our group focuses on manufacturing and evaluating the mechanical properties or fracture mechanism of aligned CNT sheet /Epoxy composites, of which aligned CNT sheet was produced by Inoue et al [24-27]. Stable production of CNTs/epoxy composite with easy control of the volume fraction of CNTs became enabled by applying hot-melt method using the uncured resin film as a matrix. Highest mechanical properties of our composites so far are the tensile strength of 239 MPa

and Young's modulus of 89.6 GPa by adding 32.8 % volume fraction of CNTs [25]. Tensile strength and Young's modulus are 4.4 times and 37.6 times higher than those of epoxy polymer, which value is extremely high in comparison with dispersed type of CNTs/polymer composites [28].

Our group previously focused on the effect of IFSS of multi-walled carbon nanotube (MWNT) and polymer and alignment of MWNTs in the composites. The results of 20 MPa in IFSS by nano-pullout method and $E_{eff} = 800$ GPa in alignment effect by evaluating actual alignment of CNTs in the composite were already obtained [26,29]. Therefore it was elucidated that IFSS is relatively weak but effective young's modulus is almost the same as those of theoretical predictions.

Whereas the interaction between walls of MWNT is also thought to be important factor, which we have not treated yet. When tensile stress was applied to the CNTs/polymer composites, it is thought that applied force is transferred to the MWNT from interface between most outer wall of CNTs and polymer, then into inner wall through the adjacent wall. L. Zalamea et al. calculated the stress transfer efficiency between walls of MWNT from shear-lag theory. The results elucidated that reinforcement efficiency of MWNT depends on the shear stress, diameter and length of MWNT [30]. However their calculations were restricted at most several walls of MWNT, and have not been applied to the actual diameter and length of MWNT. Therefore enhancement mechanism for mechanical properties of MWNT /epoxy composites is still ambiguous.

The main objective of this paper is to conduct quantitative discussions about reinforcement mechanism of the MWNT in the composites. Load transfer between walls of MWNT is calculated by shear-lag theory. The calculation results, including our previous study about theoretical calculations which took other factors into account [24-27], were compared with actual experiment results of aligned CNT sheet/epoxy composites [24,25] to explain the enhancement mechanism for mechanical properties of Aligned CNT sheet / epoxy composites.

2. Calculations for stress transfer efficiency between CNTs walls

2.1 Method of load transfer efficiency calculations

A shear lag theory is used for load transfer calculations of MWNT [30] and cap structure (capping effect) at the end of CNTs was neglected in this calculation. Model geometry is presented in Fig.1,

which is expanded to multi-walls. 60 walls (deducing from outer diameter of 50 nm and inner diameter of 10 nm for MWNTs used in this study [22,23], distance between walls (h_1) of 0.34 nm) of CNTs are assumed in this calculation. It is assumed that young's modulus (E) and shear modulus (G) of each wall is constant and defects or chemical bondings of each wall are ignored. Therefore MWNT can be modeled as continuous fibers which has interface between the walls. Tensile stress σ_0 was applied to most outer wall (boundary stress). Using the shear-lag theory, equilibrium for i th wall relevant with adjacent walls can be written in this equation;

$$\frac{d\sigma_i}{dz} = -\frac{2\pi R_{i-1}}{A_i} \tau_{i-1} + \frac{2\pi R_i}{A_i} \tau_i \quad (1)$$

here A_i is the cross sectional area of i th wall that calculated by following equation;

$$A_i = \pi(R_{i-1}^2 - R_i^2) \quad (2)$$

shear component τ_i can be written as;

$$\tau_i = G\gamma_i = G\left(\frac{u_i - u_{i+1}}{h_1}\right) \quad (3)$$

Here u_i is the displacement of each wall, γ_i is shear strain.

Strain ε_i can be written as;

$$\varepsilon_i = \frac{du_i}{dz} = \frac{\sigma_i}{E} \quad (4)$$

Eq. (1) was differenciated and coupled with eq. (2) ~ (4), then coalesced equations can be written as;

$$\frac{d^2}{dz^2} \begin{pmatrix} \sigma_1 \\ \sigma_2 \\ \vdots \\ \sigma_n \end{pmatrix} = \begin{pmatrix} \frac{2\pi R_1 G}{A_1 h_1 E} & -\frac{2\pi R_1 G}{A_1 h_1 E} & & & \\ -\frac{2\pi R_1 G}{A_2 h_1 E} & \frac{2\pi R_1 G}{A_2 h_1 E} + \frac{2\pi R_2 G}{A_2 h_1 E} & -\frac{2\pi R_2 G}{A_2 h_1 E} & & \\ & & \ddots & & \\ & & & -\frac{2\pi R_{n-1} G}{A_n h_1 E} & \frac{2\pi R_{n-1} G}{A_n h_1 E} \end{pmatrix} \begin{pmatrix} \sigma_1 \\ \sigma_2 \\ \vdots \\ \sigma_n \end{pmatrix} \quad (5)$$

boundary conditions are;

$$\frac{d\sigma_i(0)}{dz} = 0, \sigma_i\left(\frac{L}{2}\right) = 0, \sigma_1\left(\frac{L}{2}\right) = \sigma_0 \quad (6)$$

Stress distributions and shear stress distributions in every wall can be obtained from these equations. MATLAB 2012a (Mathwork, USA) was used and 4th order of Runge-Kutta algorithm for the boundary value problems were applied for the calculation. Based on the results, shear stress distribution of each wall can be obtained from eq. (5). Finally, effective young's modulus (E_{wall}) of MWNTs is estimated by the deformation of most outer wall;

$$E_{\text{wall}} = \frac{\sigma_0 L}{2 \times u_1(L/2)} \times \frac{A_1}{A_0 + A_1 + \dots + A_n} \quad (7)$$

Here $u_1(L/2)$ is a displacement of most outer wall and A_1 is the cross sectional area of most outer wall.

2.2 Results for calculating stress transfer efficiency

Stress distributions of each wall for various length of MWNT are summarized in Fig.2 (a) ~ (e). Stress of the each wall at the center is different while the length of MWNT is short (for the length of 2 nm ~ 200 nm). However stress converged to the same value for over 2 μm length. The results imply that stress transfer efficiency is sufficient for adequate length. However, it is also implied that insufficient stress transfer region exists for several tens of nanometer from the end.

Fig.3 is the normalized estimated young's modulus of MWNT in this study. Although effective young's modulus of MWNT is very small while the length is very short (as indicated in Fig.2 (a)~(c)), this value rapidly increased as the length became longer and resulted in converged value for over 20 μm length. The finalized value is about 0.96, which value implies that adequate length of CNTs can be recognized as hollow short fiber (deducing from outer and inner diameter of MWNT).

Fig.4 depicts the normalized shear stress distributions of each wall for the representative length of 2 μm . High shear stress area exists from several tens of nanometer from the end, indicating that area has almost no length dependency. On the other hand, shear stress of nearly zero was obtained at most area. Maximum shear stress is highest at the most outer wall, which value became smaller as heading for the inner wall. The maximum value of normalized shear stress was about 0.06 at the end of most outer walls, which value is proportion to the stress. Therefore it is thought that actual shear stress become higher than that obtained by the experimental value if the stress becomes higher [29]. The

results indicate that there happens interfacial slippage or debonding at the interface of MWNT walls, which causes the decrease of estimated young's modulus of MWNT. As told before, "capping effect" of CNTs is neglected in the calculation. Of course, this effect is important for discussing interfacial properties between CNTs and polymer and walls of CNTs, however if the aspect ratio is large enough (such as over several hundred micrometer), this effect can be neglected because there exists enough length for stress-convergence.

Finally, the dependency for shear modulus of MWNT was calculated. Fig.5 is the normalized shear stress of most outer wall for various shear moduli at the representative length of 2 μm . The results indicated that maximum shear stress became higher but shear transfer region became shorter when shear modulus became higher. This result also implies that there occurs interfacial slippage or debonding between the walls at the end of the MWNT, as explained before. Unfortunately actual interfacial slippage or debonding area could not be deduced from experimental or theoretical prediction. It is thought that other approaches will be indispensable to calculate these effects correctly, such as molecular dynamics, Finite Element Analysis or multiscale modeling [31-33].

3. Applying numerical calculation results to our previous studies

3.1 Brief Summary of Previous studies

Numerically obtained results were tried to apply for explaining previous experimental results. Aligned CNT sheets used in this study were produced from vertically aligned CNT arrays (forest). Synthesis method for producing vertically aligned MWNTs using iron chloride powders as precursor of a catalyst was established by Inoue et al. [22]. Aligned MWNT sheets were easily drawn from the MWNT forest. The length of MWNT was about 1 mm and diameter is 50 – 70 nm, as described before. Fig.6 shows a scanning electron micrographs (SEM) presenting the aligned CNT sheet.

The procedure of molding CNTs/epoxy prepreg is presented below; The B-stage cured epoxy resin which consists of bisphenol-A type epoxy/ novolac type epoxy/ aromatic diamine curing agent with release paper was obtained (Arisawa Mfg. Co., Ltd. Japan). A CNT sheet which is 20 mm in width and 45 mm in length was put on a polytetrafluoroethylene (PTFE) sheet (2 mm thickness), and covered with epoxy resin film with a release paper. For molding prepreg sheet, epoxy resin was impregnated into the CNT sheet at 90 °C for 3 min. Molded prepreg sheet is 25–40 μm in thickness,

which depends on the CNT areal weight. After peeling off a prepreg sheet from the release paper, the prepreg sheet was cured at 130 °C, 2.0 MPa for 1.5 h using the hot press [24]. The weight fraction of CNTs was measured by thermogravimetric analysis (TGA).

Mechanical properties of composites were evaluated by the tensile test on a screw-driven mechanical testing machine (Model 5966; Instron Corp., USA) under a constant displacement rate of 0.2 mm/min at room temperature. Specimen was with 3 mm in width and 45 mm in overall length. The longitudinal strain was measured using a non-contacting video extensometer (AVE; Instron Corp., USA). Two pieces of white marker were painted on the specimen as target markers of gauge length (10 mm). The strain was measured by tracking gauge marks placed on the specimen [26].

Young's modulus, tensile strength and fracture strain of composites are summarized in Table.1. The relationships between volume fraction of CNTs and young's modulus of composites, and between volume fraction of CNTs and ultimate tensile strength of composites are shown Fig.7. Both young's modulus and ultimate tensile strength increased. This is due to better alignment and less agglomeration of CNTs [24,25]. Effective young's modulus of MWNTs was calculated from obtained histograms by Eshelby/Mori-Tanaka theory. As shown Fig.8, the resultant value was obtained as $E = 800$ GPa, which value is higher than previous reported values and valid for theoretical predictions, as summarized in Table.2 [26,28,34-41].

3.2 Discussions

Numerical results were applied to explain the experimental results, especially focused on young's modulus. The results indicated that MWNT used in this study can be treated as hollow short fibers because they are ultra-long fibers. Moreover, inner diameter is small; which means that hollows can be ignored and MWNT can be treated as short fibers.

Therefore it is reasonable that our previous calculations (Eshelby/Mori-Tanaka theory [24]) for alignment effect, which indicated that estimated young's modulus of MWNTs are nearly same as theoretical values [25, 27, 34-41] and it means that experimental results can be reasonably explained by the numerical calculation results.

However strength appearance mechanism could not be explained reasonably by numerical or

microscopic factors. Fig.9(a),(b) are high magnification picture at the fracture surface of composites. Fig.9(a) depicts a microscopic observation results of CNTs at the fracture surface. A lot of sword-in-sheath fracture of CNTs are visible, which has already reported by previous studies [24,26]. Fig.9(b) shows a lot of CNTs exposure at the fracture surface, which length are in the range of 1.0 ~ 7.1 μm (average: 4.1 μm) and multiple fracture length is deduced to be 16.6 μm , which length is much shorter than actual length of CNTs and suggests multiple fracture of CNTs. This value is nearly same to the stress-converged length that obtained from previous calculation. The result also implies that interfacial strength between most outer wall and polymer has little effect on the mechanical properties of composites. The stress applied to the MWNTs can be easily calculated from exposed MWNT at the fracture surface. If the 20 MPa of interfacial strength between CNTs and polymer (from our previous research) is assumed [26], the obtained value is 3.3-12.7 GPa (in average) and 127-488 GPa (only most outer wall is considered and load transfer between walls is ignored), which is thought to have reached to the strength of MWNT [26]. Shear-lag and other previous calculation results could not explain these symptoms reasonably, which means that other factors should be taken into account to discuss the strength appearance mechanisms.

4. Conclusion

Several factors which were thought to dominate mechanical properties of MWNT/epoxy composites were considered to elucidate the reinforcement mechanism of the aligned CNT sheet/epoxy composites; especially focused on stress transfer efficiency. The results indicated that MWNT can be treated as short fibers for adequate length. Numerical calculations enabled to explain the mechanical properties of aligned CNT sheet /epoxy composites in case of ultra-long MWNT used in this study.

Future works are purposed for establishing detailed method of stress transfer estimation including interfacial slippage or debonding between each wall and elucidating the strength appearance mechanism of composites.

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Fig.1 Load transfer modeling for CNT (Shear-lag theory) [30].

Fig.2 Load transfer calculation results for various length of MWNT. (a) $L = 2$ nm, (b) 20 nm, (c) 200 nm, (d) 2 μ m and (e) 20 μ m.

Fig.3 Effective young's modulus of CNT for various length.

Fig.4 Interfacial shear stress distributions for each wall of MWNT ($L = 2 \mu\text{m}$).

Fig. 5 Shear stress distribution of most outer wall for various shear modulus of MWNT ($L = 2 \mu\text{m}$).

Fig.6 Microscopic picture of aligned CNT sheet obtained from SEM observation.

Fig. 7 (a) Young's modulus and (b) Ultimate tensile strength as function of CNT volume fraction.

Fig.8 Calculation results compared with experimental results (Under the assumption of 3-Dimensional distribution of CNT). The best fit young's modulus of CNT is $E_1 = 800$ GPa[27].

Fig.9 SEM micrographs showing (a) sword-in-sheath fracture of CNTs and (b) fracture surface of aligned CNT/epoxy composites after tensile testing [26].

Table

Table.1 Average mechanical property of aligned CNT/epoxy composites [24,25,27].

Specimen	Volume fraction (V_f)	Young's modulus (GPa)	Tensile strength (MPa)	Failure strain (%)
epoxy	0	2.5	54.7	3.05
Comp #1	4.5	18.8	97.4	0.503
Comp #2	10.5	27.8	153	0.545
Comp #3	21.4	50.1	181	0.36
Comp #4	29.6	73.4	193	0.22
Comp #5	32.8	89.8	217	0.237
Comp #6	42.3	82.9	319	0.404
Comp #7	15.3	42.9	230	0.526

Notice: Comp #6 resin was not impregnated well, and Comp #7 specimen was polished.

Table.2 Young's modulus and Strength of CNT reported by previous literatures.

Type of CNT	Young's modulus (TPa)	Measurement method
MWNT	0.40 - 4.15 (ave. 1.8)	CNT's vibration [34]
MWNT	1.28 (± 0.59)	Direct measurement [35]
MWNT	1	Bending observation [36]
MWNT	0.81 (± 0.41)	Direct measurement [37]
MWNT	0.27 -0.95	Direct measurement [38]
MWNT	0.91 (± 0.18)	Direct measurement [39]
MWNT	0.45 (± 0.23)	Direct measurement [40]
SWNT	1.19	Empirical force constant model [41]
MWNT	0.68	Eshelby/Mori-Tanaka theory (two dimensional) [25]
MWNT	0.8	Eshelby/Mori-Tanaka theory in this study (three dimensional)[27]