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COMPARISON OF CALCULATED PREDICTION MODEL RESULTS WITH EXPERIMENTAL STRENGTHS OF CARBON NANOTUBE REINFORCED ALUMINUM MATRIX COMPOSITE MATERIALS

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ABSTRACT

Industrial type multi-wall carbon nanotube reinforced aluminum matrix composites are successfully fabricated by vacuum assisted infiltration of molten aluminum alloy into the carbon nanotube containing preform. Compressive mechanical properties of these composites are investigated and compared with the calculation results of aforementioned prediction models. Thermal mismatch model is the most matching model for the yield strength of composites due to consideration of thermal expansion differences between reinforcement and matrix material in the model which affects punching of dislocations in the matrix. Furthermore, shear lag model gives the closest results for the ultimate compressive strength values by considering interfacial bonding parameters.
Keywords: Carbon nanotubes, metal matrix composites, shear lag, thermal mismatch, Halpin-Tsai equations.

KARBON NANOTÜP TAKVİYELİ ALÜMİNYUM MATRİSLİ KOMPOZİT MALZEMELERİN DENEYSEL DAYANIM DEĞERLERİ İLE TAHMİN MODELİ SONUÇLARININ KARŞILAŞTIRILMASI

ÖZET

Endüstriyel tip çok cidarlı karbon nanotüp takviyeli alüminyum matrisli kompozitler, ergitilmiş alüminyum alaşımının carbon nanotüp içerikli preform içerisine vakum destekli sızdırılması (infiltrasyonu) ile üretilmektedir. Üretilen kompozitlerin basma kuvveti altındaki mekanik özellikleri incelenmiş ve en çok tercih edilen tahmin modelleri ile yapılan hesaplamalar ile karşılaştırılmaktadır. Isıl uyumsuzluk (thermal mismatch) modeli, matris malzemesi ile takviye elemanı arasında varolan ısıl genleşme farkının yaratmakta olduğu matris malzemesinde dislokasyon yığılması etkisini dikkate aldığından, basma yükü aldında elde edilen akma gerilmesi sonuçlarına en yakın değerleri vermektedir. Ayrıca, kayma gecikmesi (shear lag) modeli arayüzey bağı parametrelerini dikkate aldığından maksimum basma gerilmesi değerlerine en yakın sonuçları vermektedir.
Anahtar Sözcükler: Karbon nanotüpler, metal matrisli kompozitler, kayma gecikmesi, ısıl uyumsuzluk, Halpin-Tsai denklemleri.

1. INTRODUCTION

Carbon nanotubes are the strongest and stiffest materials discovered in terms of tensile strength and elastic modulus, respectively.

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Simulation studies of Yakobson [1] and experiments of Falvo [2] demonstrate a remarkable "bend, don't break" response of individual single-wall carbon nanotube (SWCNT) to large transverse deformations [3]. Young's modulus of a cantilevered individual multi-wall carbon nanotube was measured from the amplitude of thermally driven vibrations observed in the TEM as 1.0 to 1.8 TPa. Apart from that, TEM-based tensile and bending tests gave more reasonable modulus and strength of multi-wall carbon nanotube (MWCNT) of 0.8 and 150 GPa, respectively. MWCNTs and SWCNT bundles may be stiffer in bending compared to tensile strength because in tension, bundles are weaker due to "pull-out" of individual nanotubes. The stress–strain curves suggest that the load is carried primarily by MWCNT on the exterior surface of the tubes, from which breaking strengths from 11 to 63 GPa were revealed in the studies of Yu et. al.[4] On the other hand, the mean value of tensile modulus was between 0.27 – 0.95 TPa.

The mechanical behavior of the composite is related with the load sharing between matrix and reinforcement [5]. The proportion of the load carried by the reinforcement is independent of the overall carried load, but composite strength depends on factors such as volume fraction of reinforcement, shape, orientation, etc. Many studies were carried on about the strengthening mechanisms of CNT reinforced aluminum (Al) matrix composites [6]. Some of these studies compared the experimental data with the prediction model results for evaluating only tensile strength and elastic modulus of these composites [7, 13 - 15].

This study presents the experimental compressive strength data of vacuum infiltrated CNT/Al composites compared with the various prediction models. These three models are Halpin-Tsai equations, shear lag theory and thermal mismatch theory for yield strength. The objective is to determine the most matching models for compressive strength prediction of vacuum infiltrated CNT/Al composites.

2. MATERIALS AND METHOD

In this study, fabrication of CNT /Al composites by vacuum assisted infiltration method with using CNT and CNT-Al powder preform reinforcement is realized successfully. Firstly, preforms are produced. Next, one of the most used metal matrix materials; 6063 aluminum alloy is infiltrated into these preforms by using vacuum assisted casting machine. After fabrication, compressive test specimens are prepared according to TS 206 [16] standard. Compressive test studies are continued according to TS 206. On the other side, calculations are made for three different models and modifications of two. Finally, compressive test data are compared with the model results.

2.1. Experimental Procedure

Industrial type MWCNTs (10-30 nm diameter; ~2.1 g/cm^3 true density) with a purity rate of over 85% are provided from Chengdu Organic Chemicals Co. Ltd. and chemically functionalized with 65 wt% HNO_3 by reflux of dispersion at ~125 °C with the help of heating magnetic stirrer for 48h. The resulting sample is neutralized by washing, filtered and dried in drying-oven at 40 °C for 48 hours.

Preforms are prepared with functionalized CNTs, Al powder, Poly Vinyl Alcohol (PVA) as a pore forming agent and colloidal silica (10 - 20 nm grain size; ~1.20 g/cm3 density) as binding agent. CNT preforms are fabricated with and without aluminum powder content in two different CNT ratios and ingredients of these groups are listed in Table 1.

CNTs or CNTs-Al powders were mixed with colloidal silica and PVA solution. This mixture is poured into a flexible mold to fabricate preforms. Next, these preforms are dried at room temperature for 48 hours. Finally, dried preforms are cured at 600°C to evaporate PVA and sintered at 1000 °C for 3 hours with a heating/cooling rate of 10 °C/min under vacuum environment.

For vacuum assisted infiltration, wax pattern is prepared and mounted to the rubber flask base and finally assembled to the flask. Then, gypsum-bonded casting investment is prepared by mechanical mixing and poured into the flask gently. After solidification, wax is removed and finally mold is cured with a heating step rising from room temperature to 720 °C before casting process.

Table 1. Contents of the fabricated preforms

Main Group Code Name	Subgroup Code Name	Carbon nanotube (g)	Al powder (g)	20% PVA (mL)	Colloidal Silica (mL)
PC	P2C	2	-	8	2
	P6C	6	-	8	2
PCA	P2C10A	2	10	8	2
	P4C8A	4	8	8	2
PA[R]	P12A[R]	-	12	8	2

[R] Refence preform to see the mechanical effect of Al powder to the composite material

Following the preparation of preforms and mold, molten 6063 Al alloy is prepared by melting in electric furnace at 800°C. Just before the casting process, preform is placed close-fit into the mold by using a lancet. Finally, casting operation is realized. Details of the fabrication study are given in a previous publication [17].

After fabrication, compressive test specimens are prepared according to TS 206 [16], standard of "Compression Testing of Metallic Materials" as medium size testing specimen (10x30 TS206) and tests are carried out with a class 1 calibrated electro mechanic testing machine with 5 mm/min testing speed and elongation is measured from the crosshead movement. Usually, in a compressive test, the strain increases with the sample deformation without reaching a limit and ultimate compressive strength is specified with a slope starting from a specified offset point like determination of yield point. In our compressive test, maximum stress value is certain to decide ultimate compressive strength like the study of Li et. al. [18].

2.2. Prediction Models

The discussion of elastic properties and strengths of metal matrix composites begins with the comparison of the rule of mixture type predictions and the experimental data. Finally, micromechanical approach or equations like Halpin-Tsai are adopted to calculate metal matrix composite mechanical properties as an approximation. Our three main models are explained below.

2.2.1. Halpin-Tsai Equations

Halpin, Tsai and Kardos have empirically developed equations that giving quite satisfacroty results compared with the micromechanical equations [19]. Equations are useful for discontinuous fibers oriented in the loading directions. The Haplin-Tsai equation to:

$$\frac{p}{p_m} = \frac{1+\xi\eta\, V_f}{1-\eta\, V_f}\,, \eta = \frac{\frac{p_f}{p_m}-1}{\frac{p_f}{p_m}+\xi} \tag{1}$$

Where p represents the moduli such as $E_{//}$, σ *or* G; p_m and p_f are the corresponding matrix and fiber moduli, respectively; and ξ is a measure of the reinforcement, which depends on the boundary conditions such as fiber geometry, distribution and loading conditions.

For fibers of unit aspect ratio, the Halpin-Tsai equation is given as [20]:

$$\frac{p}{p_m} = \frac{1+\eta\, V_f(2+40V_f^{10})}{1-\eta\, V_f} \;; \qquad \eta = \frac{p_f-p_m}{p_f+p_m(2+40V_f^{10})} \tag{2}$$

Cox has found orientation factor parameter α to account for the randomness of the discontinuous fibers [15]. If the fiber length is much smaller than the thickness of the specimen like our products, the fibers are assumed randomly oriented in three dimensions; and the parameter α = 1/6 is used for the calculation. The modified equation is given as below:

$$\frac{p}{p_m} = \frac{1+\xi\eta\, V_f}{1-\eta V_f}\,, \eta = \frac{\frac{\alpha p_f}{p_m}-1}{\frac{\alpha p_f}{p_m}+\xi} \tag{3}$$

2.2.2. The Shear Lag Model

The Shear Lag model is derived from the transfer of load from the matrix to a discontinuous aligned reinforcement where the fiber end is neglected assuming perfect interfacial bonding exists [5]:

$$p_c = p_m V_m + p_f V_f\left(\frac{1-\tanh{(qs)}}{qs}\right), q = \left[\frac{2p_m}{p_f(1+\gamma_m)\ln(1/V_f)}\right]^{1/2} \tag{5}$$

where γ_m is the Poisson ratio of the matrix material.

2.2.3. Thermal Mismatch

Dislocation strengthening is a mechanism occurs when dislocations are formed to accommodate a misfit between the reinforcement and the matrix during straining. It can also occur through the relaxation of thermal residual stresses coming from the composite fabrication called the "thermal mismatch".

As given in Arsenault's model [21] of thermal mismatch, the corresponding increase in strength of the composite can be calculated from:

$$\Delta\sigma = \beta G \rho^{1/2} b\ ,\quad \rho = \frac{4V_f\varepsilon_{therm}}{b(1-V_f)}\left(\frac{1}{t_1}+\frac{1}{t_2}+\frac{1}{t_3}\right) \tag{6}$$

where β is a constant equal to 1.25 and *G* is the shear modulus of the matrix, ρ is the dislocation density generation is found to be: where ε_{therm} is the thermal strain, *b* is the Burger's vector and $t_{1\text{-}2\text{-}3}$ are the reinforcement dimensions.

3. RESULTS AND DISCUSSION

Starting with the matrix material specimens, compressive tests are applied to composite material specimens with the same parameters as mentioned in TS 206 [16]. Typical engineering stress-strain curves of compression tests specimens are given in Figure 1. In addition, average 0.2% yield strength and ultimate compressive strength of all specimens are given in Figure 2 with standard deviations. Composites are named according to the preforms used for the fabrication of these composites as:

i. Al: 6063 Al matrix material
ii. P2C: 0.25 wt.% carbon nanotube / 6063 Al composite
iii. P2C10A: 0.25 wt.% carbon nanotube - Al powder / 6063 Al composite
iv. P4C8A: 0.50 wt.% carbon nanotube - Al powder / 6063 Al composite
v. P6C: 0.75 wt.% carbon nanotube / 6063 Al composite
vi. P12A: Al powder / 6063 Al reference

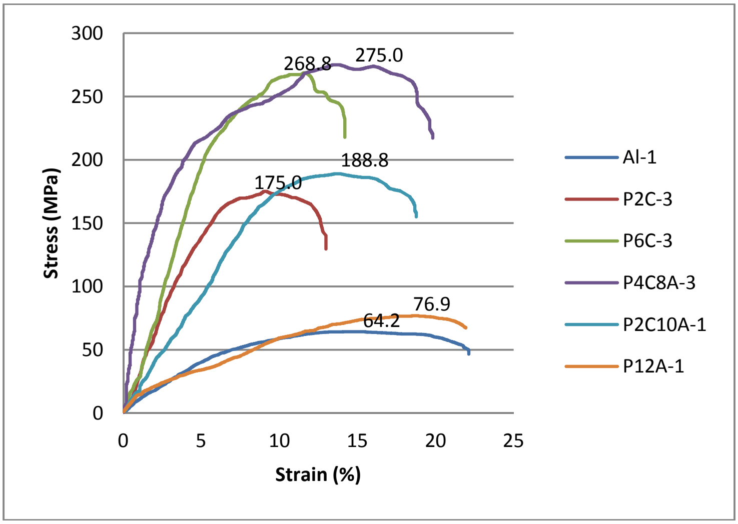

Figure 1. C. Typical stress-strain curves of matrix material and different type of composite materials (ultimate compressive strength values are written on curves)

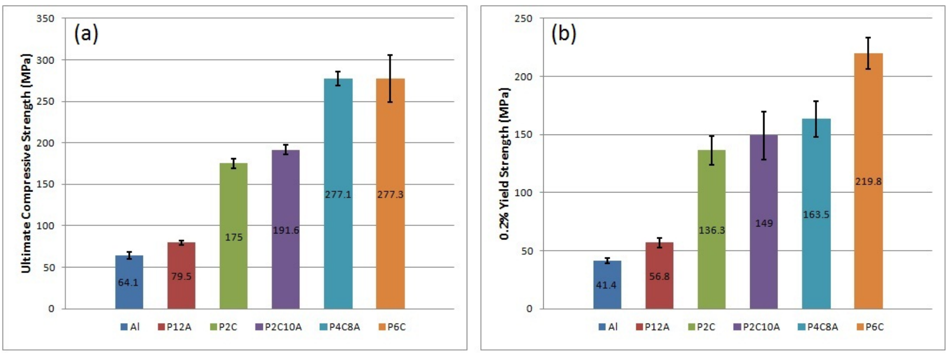

Figure 2. D. Comparison of avarage (a) ultimate compressive strength (b) 0.2% yield strength

In order to theoretically evaluate the possible gain in mechanical properties with CNT addition, the prediction models associated to the strengthening mechanisms presented in section 2.2 are used. The yield stress is calculated with shear lag model, Halpin-Tsai and modified Halpin-Tsai equations and thermal mismatch model. Furthermore, shear lag model is customized for hybrid composite and effect of Al_2O_3 ingredient is shown in addition to the strengthening effect of carbon nanotubes. As known from the PhD thesis of Poirier [5], the strengthening effect of Al_2O_3 below the amount of 1 vol% is fairly weak. Considering the Al_2O_3 amount of our aluminum containing preform reinforced composites, incremental yield strength will be below 10 MPa. Due to these reasons, this weak effect of Al_2O_3 is shown with a single model (Shear Lag with Al_2O_3) by considering possibility of maximum Al_2O_3 amount of 1 vol%.

Yield strength and ultimate compressive strength predictions are made and graphics are plotted by using parameters given in Table 2. According to the calculations and experimental results, model – experimental data comparison graphics for % 0.2 yield strength and ultimate compressive strength are shown in Figure 3.

Table 2. Parameters for strengthening calculations

Parameter	Values	
	Yield Strength	**Ult. Compressive Strength**
Strength of 6063 Al: σ_{6063Al}	41.4 MPa	61.4 MPa
Strength of CNT: σ_{CNT} [22]	100 GPa	120 GPa
Poisson ratio: γ_{6063Al} [23]	0.33	0.33
Shape factor of CNTs: s_{CNT} [14]	100	100
Strength of alumina: σ_{Al2O3} [24]	5500 MPa	5500 MPa
Shape factor of alumina: s_{Al2O3}	5	5
Avarage length of CNTs: λ_{CNT}	20 μm	20 μm
Avarage diameter of CNTs: d_{CNT}	20 nm	20 nm
Orientation factor of CNTs: α_{CNT} [15]	0.166667	0.166667
Thermal strain: ε_{therm} [14]	0.009718	-
Burgers vector: b [5]	0.286 nm	-
Dimension of CNTs: t_{1-2-3}	20 nm	-
Constant for CNTs: β [14]	1.25	-
Shear Modulus of Al: $G_{6063\ Al}$ [23]	25.8 GPa	-

Thermal mismatch is the first model that has the most matching values for carbon nanotube reinforced 6063 aluminum matrix composite material. Carbon nanotubes have a coefficient of thermal expansion of $\sim 10^{-6}\ K^{-1}$; while commercial purity aluminium presents a much greater coefficient of thermal expansion of $23.4 \times 10^{-6}\ K^{-1}$. Thus, in carbon nanotube/6063 Al composites, there exists a significant coefficient of thermal expansion mismatch between the matrix material and the carbon nanotubes which would result in prismatic punching of dislocations at the interface, leading to work hardening of the matrix [14]. The generated dislocation density depends on the reinforcement surface area. Carbon nanotubes have an advantage due to their small diameter. The dislocation density generation is likely to be higher, which in turn would result in increased strengthening. In parallel with the mentioned circumstances, yield strength of the facricated carbon nanotube reinforced composites are increasing in direct proportional to the carbon nanotube ratio. The differential between the model and experimental results are related to the parameters which are decided to be selected as avarage values for carbon nanotube dimentions. Otherwise, the yield strength would be never as high as the value calculated with the thermal mismatch model. The reason is the agglomeration of carbon nanotubes in the matrix. This condition reduces the surface area of carbon nanotubes. According to the reduction of the surface area, the dislocation density therefore the yield strength decreases.

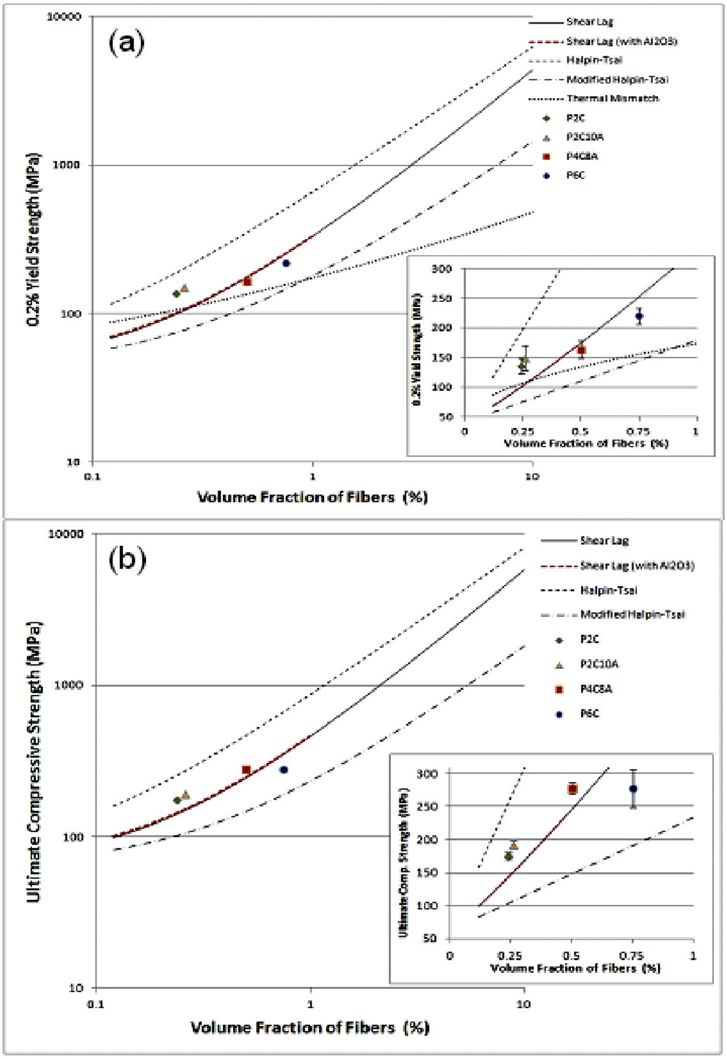

Figure 3. Comparison of experimental and calculated data of CNT /Al composites (a) Predicted 0.2% yield strength – volume fraction (b) predicted ultimate compressive strength – volume fraction of carbon nanotube curves calculated with different models.

The Halpin-Tsai equation is a semi-empirical methods by which the yield strength of composites can be calculated for fibers of unit aspect ratio. In modified Halpin-Tsai equation, the geometry factor , which depends on the boundary conditions such as fiber geometry, distribution and loading conditions is incorporated into the model. The modified Halpin-Tsai model demonstrates the significant effect of reinforcement geometry alone on the strength properties of a unidirectionally oriented and orthotropic composite at both constant volume fraction and packing geometry; changing from a sphere to a long fiber gives an order of magnitude or more increase in strength for both a unidirectional and a randomly distributed reinforcement depending on the constituent [25]. Experimental results don't reach to the amount of Halpin-Tsai equation for carbon nanotubes of unit aspect ratio; because the effect of shape factor is too high for used carbon nanotubes. It is seen that, the experimental results are above the modified Halpin-Tsai prediction curve. This relation is also related to the avarage carbon nanotube dimention and length parameters. It seems that our carbon nanotubes have higher length/diameter ratio *c* than the accepted parameter for the calculation. Furthermore, the length/diamater ratio will be even more higher than estimated value considering the agglomeration of carbon nanotubes.

Shear Lag model is the most matching model among all prediction models. This model comprises the transfer of load from the matrix to the reinforcement by the interfacial shear stress. Thus, the strength of the reinforcement is directly utilized. Strengthening effect of high aspect ratio reinforcements up to s = 100 such as multi-wall carbon nanotubes to composites are preferably predicted with this model [14]. Wetting is a necessary condition for interfacial shear stress transfer for carbon nanotube/aluminum composites. Positive effect of functionalization of carbon nanotubes is observed from the matching of experimental results and shear lag model. Approximate effect of Al_2O_3 content of composite is also shown for shear lag model where any notable difference cannot be seen.

Similiar results for compressive strenght of fabricated composites can be seen from the graphic of predicted ultimate compressive strength – volume fraction of carbon nanotube fibers curves calculated with different models given with experimental ultimate compressive strength results of fabricated carbon nanotube reinforced composites (Figure 3(b)). It can be seen from Figure 3 (b) that average ultimate strength of P6C composite is incongruously below the shear lag curve. The reason of this condition is the agglomeration of carbon nanotubes in the matrix. Inherently, agglomeration of nanotubes causes the decrease of the mechanical properties. However, shear lag model gives the closest results for the ultimate compressive strength values by considering interfacial bonding parameters. Model results will be more synchronized for both yield and ultimate compressive strength predictions by determining the parameters compatible with the actual data. For this reason, accurate measurement of reinforcement dimensions is vital. In real, it is impracticable to determine all true sizes and orientations of randomly distributed reinforcements from the fabricated composites. Furthermore, wetting of CNTs is a changeable factor that considerably affects the real strength of composites.

4. CONCLUSION

The addition of functionalized industrial type multi-wall carbon nanotubes increased the yield and ultimate compressive strength of 6063 aluminum matrix composites in direct proportion to the nanotube ratio. This circumstance can be explained with the strengthening mechanism models for composite materials such as rule of mixtures, shear lag model, thermal mismatch and Halpin-Tsai equations.

Thermal mismatch model is the most matching model for the yield strength of composites due to consideration of thermal expansion differences between reinforcement and matrix material in the model which affects punching of dislocations in the matrix. Furthermore, shear lag model gives the closest results for the ultimate compressive strength values by considering interfacial bonding parameters. Model results will be more synchronized by determining the parameters compatible with the actual data. For this reason, accurate measurement of reinforcement dimensions is vital. In fact, it is impossible to determine all true sizes and orientations of randomly distributed reinforcements from the fabricated composites.

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