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Thermal and Mechanical Properties of Woven Glass Fiber Reinforced Epoxy Composites with Carbon Nanotubes Grown in-Situ.

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ABSTRACT
Woven glass fiber/Multi-walled carbon nanotubes (CNTs) reinforced epoxy matrix was investigated for toughness enhancement by in-situ growth of CNTs on the glass fiber surface.Woven glass fibers were uniformly coated with an iron acetate catalyst using spin coating techniqueand CNTs were grown on the fibers using chemical vapor deposition (CVD) technique.Scanningelectron microscopy (SEM) studies have confirmed that CNTs were grown uniformly around these non-carbon fibers. Flexure test confirmed that the growth of 0.62 wt.% CNTs increased flexure strength and modulus of the glass fiber reinforced epoxy matrix by 16.5% and 13.2%, respectively. In addition,the in-situ growth of CNTs increased flexure toughness and ductility by 30.58% and 8.6%,respectively, compared to the baseline properties of the glass fiber reinforced epoxy composites. Thermal analysis also presented an increase in both thermal stability and glass transition of CNTs grown epoxy composite compared to the baseline composite. The mechanical and thermal performance is the resultant effect of modified fiber/matrix interface and interfacial stress transfer.
Keywords: Chemical vapor deposition; In-situ growth; Stress transfer; Interfacial bonding

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I. Introduction

Woven glassfibers are attractive candidatesin the continuous fiber reinforced composites due to their corrosion and impact resistance [1] and ductile characteristics[2]. However, glass fiber reinforced composites are susceptible to premature failure or low-velocity impact damage due to lower interfacial strength between the fiber and the matrix [3]. Sincethe glassfiber epoxy composite is a combination of inorganicand organicmaterials, acompatible coatingisgenerally applied on the fibers, which playsan important role in controlling the interface properties [4].A considerable number of studies have been reported onthe improvement inthe interfaceproperties [5,6], which are directly related to strength and toughness of the composites.Several other studies are also reported on densifications [7,8] and toughening[9-14]of matrix by nanoparticle additions to improve the toughness of the fiber reinforced composites. However, those mechanisms are not sufficient for good stress transfer between fiber and matrix [15]. In order to improve the stress transfer, it is essential to introduce a stress bridging mechanism between the fiber and matrix.A bridging mechanism developed by direct growth of CNTs on fiber surfaces has been found to have considerable effect on the interface bonding, stress transfer and damage tolerance [16-18].Studies suggest that,CNTs attached to the surface of the fibers interlock the polymer chains andcreates a physically strong adhesion between two phases. In-situ synthesis allows aligned and controlled growth of CNTs on fiber, whichtoughens fiber-matrix interphase, creates bridging and eventually increases the toughness of the composite.A number of articles described the direct growth of CNTs on carbon fiber [16-20] and improvement in the flexural and tensile properties of the fiber reinforced composites. However, there areonly few literatures to shed light on the toughening of glass fiber reinforced composites using the controlled growth of CNTs. CNTs growth morphology associated with the non-homogeneity between silica rich glass fibers and CNTs [21] are considered as the deficiency in direct growth of CNTs on the glass fiber surface.Although, some researchers described the CNTs growth mechanism on the glass fibers[22,23], those were not adequate in describing the morphology of grown CNTs, the interactions between the CNTs and the fibers and the effect of CNTs growth on the mechanical performance of the glass fiber reinforced composites.

A number of other variables can influence flexural strength. Interface plays a critical role in stress transfer between fiber and matrix. The types of chemical compounds in the surface coating of glass fiber are among those who influence the surface conditions. Epoxy Silane is the typical coating on the surface of E-glass for the

application with epoxy resin. Silane coupling agents consist of epoxy group which makes reaction with epoxy matrix. A. T. Dibenedetto et al [24] reported improved transmission of interfacial shear stress between fiber and matrix compared to uncoated fiber. Also J.G Iglesias [25] reported the effect of cross linking density and interpenetrating network of Silane coupling on interfacial strength between glass fiber and epoxy. The current research therefore aimed at investigating the effects of interface modifications simultaneously with fiber surface modification and direct growth of CNTs.

This research was initiated to demonstrate that the direct growth of CNTs reduces the premature failure and increases strength of glass fiber reinforced composites. In order for this demonstration a controlled in-situ growth of CNTs was investigated on unidirectional woven glass fiber using atmospheric pressure chemical vapor deposition technique. The sizing ofthe fiber glass surface was first modified by controlled heat treatment and then CNTs were grown. Ahybrid composite [26]was fabricated using thisCNTs grown and neat wovenglass fiber. The growth mechanism and weight fractions of CNTs were evaluatedand the resulted fiber reinforced composites were tested for mechanical strengthand thermal stability and degradation. These studies were supported by the morphology of CNTs growth on the woven glass fiber.

II. Experimental

2.1. Materials

Plain weave unidirectional E- glass fiber, style no 7721 (purchased from a Fiberglass site and manufactured by Hexcel USA) was chosen for this investigation. Approximately 95 vol. % of the fiber makes up the warp (roll length) direction of this fabric.Unidirectional woven glass fiber was used in the laminated composites to strengthen in one direction and save weight in the less critical orientation. The density and ply thickness are 2.05 g/cc and 0.17 mm respectively. The Breaking strength is approximately 61.294 MN/m (350 lb_f/in). A typical unidirectional fabric is shown in Fig.1. Epoxy SC-780 (supplied by Applied Poleramic Inc.) was chosen as the matrix material. This is a low viscosity, toughened epoxy system prepared from a mixture of Bisphenol A and F with a small portion of reactive diluents. The Curing agent for this epoxy was a mixture of aliphatic and cycloaliphatic amines. This epoxy is specifically designed for a room temperature vacuum assisted resin curing process, which is compatible with glass fiber [28].

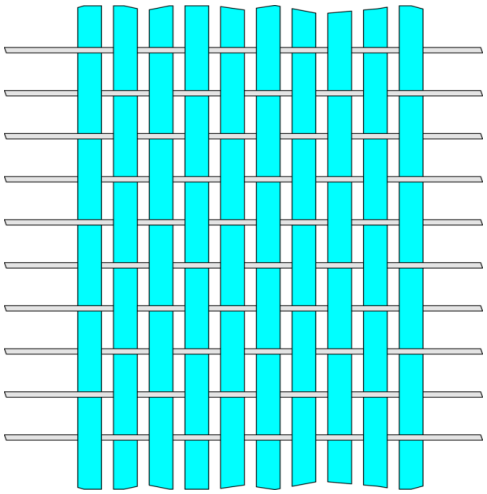
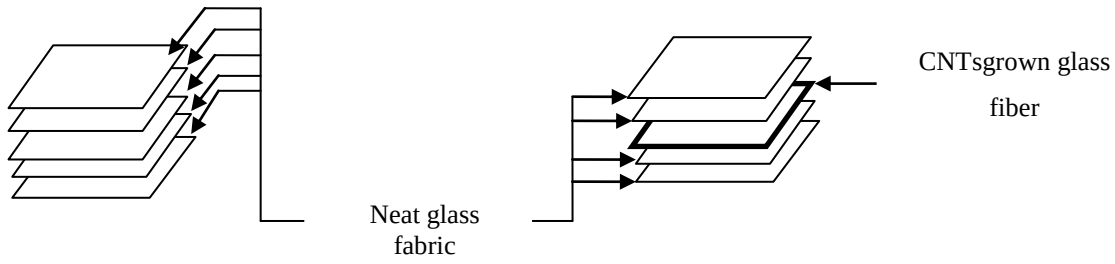


Fig.1.A schematic of an unidirectional woven glass fabric

2.2. Fabrication of the woven glass fabric reinforced laminated composite

Fabrication of woven glass fiber reinforced epoxy composite is a three step process: (1) Coating the fiber using solution based catalyst, (2) Growing CNTs on the fiber surface and (3) Lamination. The woven glass fiber was coated with iron (II) acetate catalyst using a Laurel spin processor (supplied by Laurel technologies Inc., USA). CNTs growth was performed using theatmospheric pressure CVD furnace (Easy Tube 3000, Ronkonkoma, NY, USA).The growthtemperature was kept lower than the glass transition temperature of the glass fiber. The catalyst was reduced to metal particles using hydrogen and then the growth of CNTs took placewithethylene introductionat a feed rate of 0.15 L/min at 600 ° C for 30 minutes.Details on the complete fabrication process are found in previous works published elsewhere [27]. For the investigation, two laminated systems were prepared which are neat and CNTsgrown woven glass fiber reinforced epoxy composites.The neat composite was fabricated with 5 layers of un-sized fabrics. The CNTsgrown woven glass fiber reinforced composite was fabricated in a laminated hybridization technique [26] withone layer with grown CNTsstackingin the middle of four un-sized layers,which isshown in Fig.2.



(a) Neat Panel (b) CNTs Panel

Fig.2. Layouts of (a) Neat (b) CNTs grownglass fabric reinforced panels

2.3. Testing procedure

Thermal stability and weight loss of the neat and CNTs grown woven glass fiber reinforced epoxy composites were investigated using Mettler Toledo TGA/SDTA 851 Thermo gravimetric analyzer (TGA). A sample of about 12–15 mg was placed in an Alumina crucible and heated from 25 to 800 °C with a heating rate of 10 °C/min in a nitrogen atmosphere. Glass transition (T_g)temperature was investigated using Differential scanning calorimetry (DSC). Aclosed Aluminum pan having about 12–15 mg of specimen and a closed empty pan were placed inside the DSC heater and heated from 25 to 400 ºC with a heating rate of 10 ºC/min in a nitrogen atmosphere. For this investigation, the differences in the heat flux from the pan with specimen and the empty pan were measured against the temperature changes.

Flexural strength and modulus were investigated using a Zwick/Roell testing machinein accordance with three point bending method (ASTM D790-02) [29]. Flexure strength was measured using the following equation

$$\sigma_f = \frac{3PL}{2bd^2} \quad (1)$$

Where P is the load applied, L is the span length; b and d are the width and thickness of the specimen, respectively. L/d ratioof the specimen was maintained 32 in order to minimize the effect of in plane shear. At least five specimens with the width of 12 mm were prepared for each type of composites.

Flexure toughness was essentially calculated from the measured stress-strain profile from the flexure test using the following equation-

$$E_f = \int_0^{\epsilon} \sigma_f \, d\epsilon \quad (2)$$

Where,ϵ represents strain rate at the failure stress due to flexure loading.

The microstructure of the CNTs grown woven glass fiber surface wasinvestigated using JEOL JSM 5800 Scanning electron microscopy(SEM). The woven fibers were coated in gold using a Hummer VII ion beam sputter coater to enable imaging. To obtain high resolution images beam energy of 10 KV was applied on the samples (around 2 mm thick) inside the vacuum chamber which was maintained at 1.5E-3 Torr pressure.

III. Results and discussion

With one ply of CNTs grown woven glass fiber, the amount CNTs in the CNT grown woven glass fiber composite was found as 0.62 wt. %. Fibers in both neat and CNT gown systems were foundroughly between 68.5 wt. % and 72.3 wt. %, which are typical for a glass fiber reinforced composite. The results include the analysis thermal history of neat and 0.62wt. % CNTs grown woven composites while they were post heated. Flexural strengths of both composites (neat and 0.62wt.% CNTs reinforced) were evaluated and the results were co-related with thermal response, growth morphology of CNTs and its impact on potential modifications in the interphase zone between fiber and matrix.

3.1. Thermal analysis

Thermogravimetric analysis (TGA) of neat and CNFs grown woven glass fiber reinfoced epoxy composites are shown in Fig.3. From the weight loss curves it is found that the thermal degradation of neat woven glass fiber reinforced epoxy composite started earlier than the CNTs grown woven glass fiber epoxy composite. From the Differential thermal gravimetry (DTG) of the TGA it is found that the peak weight loss in the CNTs grown glass fiber reinforced composite occurred at approximately 5 ºC higher temperature (355 °C vs 350 °C) than in the neat composite. The DTG plots also represented that the weight loss peaked at 3 temperatures (at 300,350 and 600 ºC) in the neat system.Multiple peaks were also found in the DTG plot of neat glass system elsewhere [30]. The peak degradation of CNTs grown woven glass fiber composite was found to have only at one temperature (355 ºC).This observation indicates that the CNTs grown woven glass fiber compostie has stable degradation at reasonably higher temperaure than the neat woven glass fiber composite.

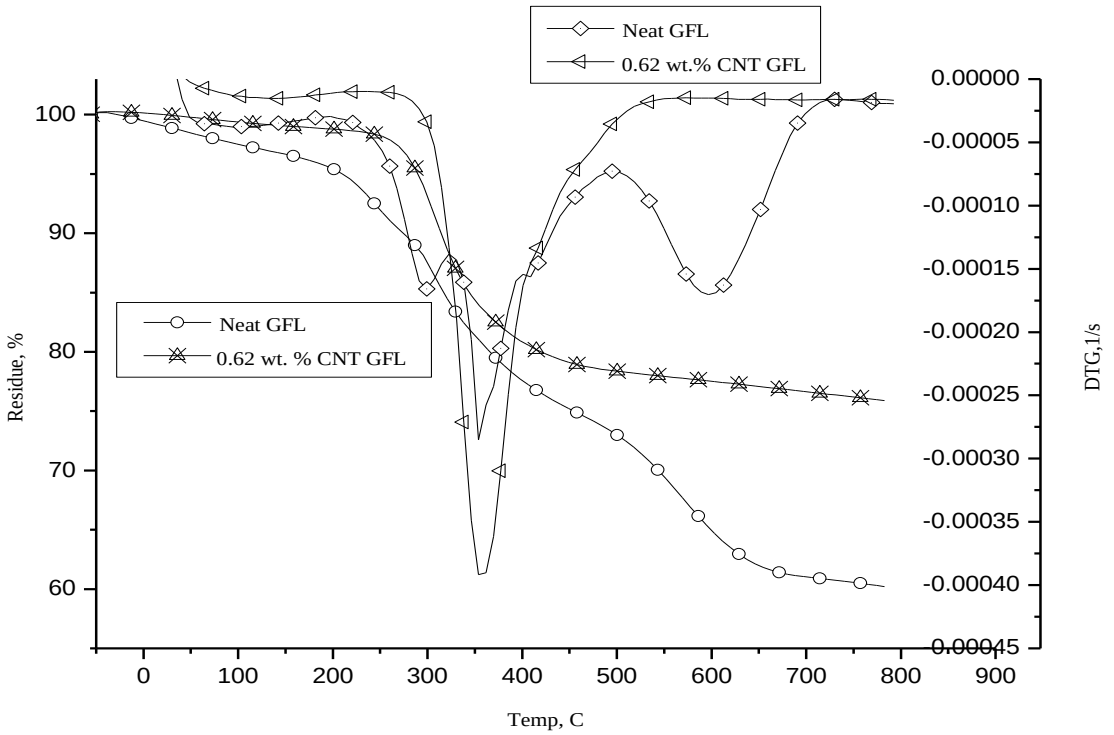


Fig.3. TGA of neat and CNTs grown epoxy-glass fiber composites

DSC analysis of neat and CNTs grown woven glass fiber reinforced composites are shown in Fig.4. The DSC analysis apparently did not show any significant change in the heat flux profile between the neat and CNTs grown system, however,a minor increase in the glass transition temperaturewas observed in the CNTs grown woven glass fiber compared to the neat one. The results suggest that the contentof CNTs in the composite are small to make any changes in phase transformation or glass transition.

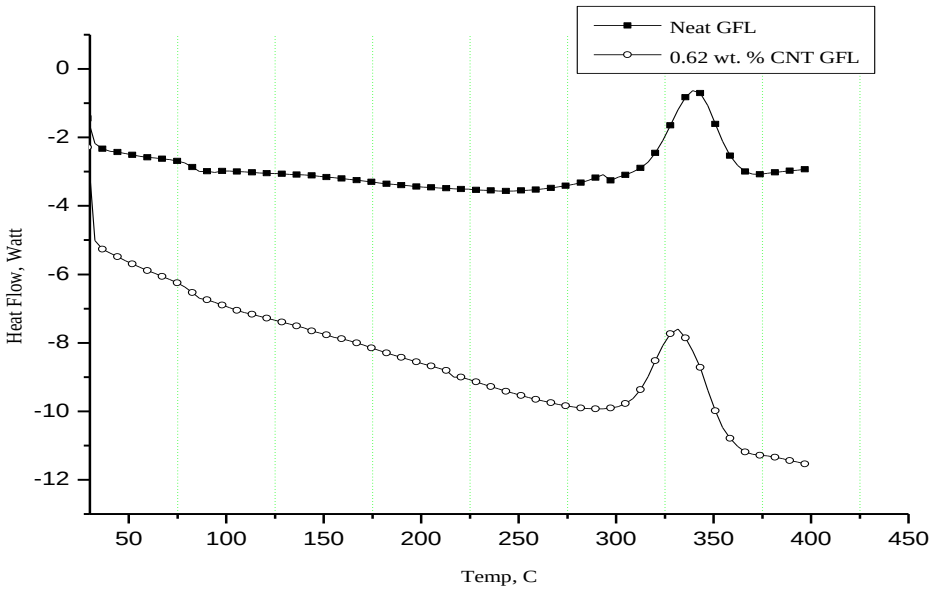


Fig.4. DSC of neat and CNTs grown epoxy-glass fiber composites

3.2. Flexural analysis

Flexural analyses of neat and CNTs woven glass fiber composite are shown in Fig.5. It is found that, flexural strength of 0.62 wt. % CNTs grown woven glass composite is approximately 526± 42.2 MPa, which is around 16.6 % higher compared to neat laminate (451±53 MPa). This increase in flexure strength indicates the improvement in mechanical performance of CNTs grown woven glass composite due to increase in the frictional resistance in the fiber matrix interface. Flexural modulus of the CNTs grown composite is also found to increase by approximately 14.5% (17±1.2GPa vs 15±1.3 GPa), compared to neat composite.Theincrease in the modulus indicates the increase in the interfacialstrength between fiber and matrix. A complete statistical data on the flexural analysis in presented in Table 1.

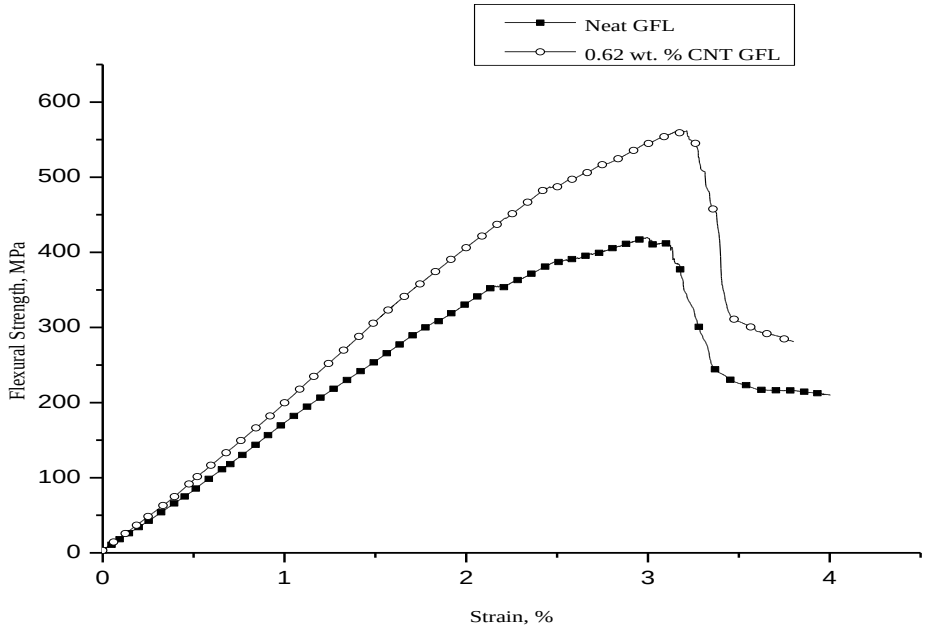


Fig.5. Flexure strength vs. strain of neat and CNT composite

Table.1. Flexural strength-strain relationship of the glass fiber reinforced composites

Sample Type	σ_f (MPa)	σ_f (Avg) (MPa)	S.D. (MPa)	E_f(GPa)	E_f (Avg)(GPa)	S.D. (GPa)	ϵ_f(Avg) (%)
Neat	414 419 518 501 404	451.2	53.1	14.27 15.68 15.98 16.61 13.16	15.14	1.3	3.00
0.62 wt.% CNT GFL	545 468 490 562 563	526.1	42.2	18.43 15.86 15.87 17.94 17.67	17.15	1.2	3.20

In a traditional continuous fiber reinforced composite, thefiber and matrix inter-phasearea plays important roles in controlling the load transfer between the fiber and matrix. In many cases the compatible coating [31] on the fiber alters the bonding characteristics between fiber and matrix, which reduces the interfacial strength.Since CNTs have extremely large surface area,the growth CNTs on the fiber surface reduces the mobility and alters the curing mechanism of the matrix in the interphase, which increases interfacial strength. The layer with grown CNTscreates a bridging effect between the fibers and the matrix [32] that helps restrict the sliding of the layers under tension or compression and thus increasesflexural strength. This improvement in both strength and modulus implies that growth of CNTs on the multiple layers of the laminates in a controlled manner would deliver better composites with outstanding mechanical properties.

Failure strain of the CNTsgrown woven glass composite also increased around 8.62 % compared to the neat one, which indicates a good balance between fiber matrix bonding and stress transfer. Flexure toughness was calculated from the area under the stress-strain plot using equation (2) and the values for the neat and CNTsgrown woven glass composites were found as 753.7kJ/m^3 and 984.3kJ/m^3, respectively. This result shows a 30.58% increase in flexure toughness in the CNTs laminate compared to neat laminate. In addition, the failure strain of the aligned CNTs grown woven glass composite also found to increase around 8.62 % compared to the neat one. These increases in the toughness and failure strain indicatean overall improvement in the ductility, delamination/crack resistance and failure strength. Therefore, the results strongly suggest our initial claim that, controlled growth of CNTsreduces the well-known premature failure of the glass fiber reinforced polymer composite.

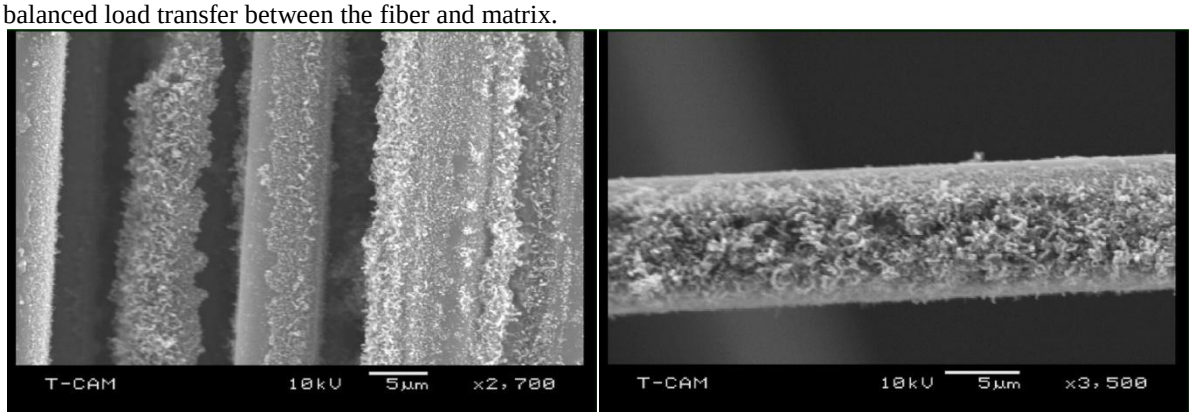
While CNTs growth operation is performed in the CVD chamber, high temperature treatment resulted in the potential alteration of the fiber surfaces. Fig 6 shows the transformation of the fiber surface appearance due to treatment at 600 ºC. The surface became gray due to the reaction occurred at ~600 °C in hydrogen environment which resulted in saturated Silane. It is possible that hydrogen can increase chemical activity of the coupling agent and result into strong interfacial bonding [24]. Also the bonding can increase between the fiber and the grown CNTs through theCVD process in the radial direction on the fiber surface. The resulted increase in interfacial strengthincrease toughness due to the increase in the energy of separation of the CNTs. More investigation is necessary to separate out the effect of hydrogen on the crosslinking of the coupling agent and their ultimate effect from the effect of CNTs growth.

(a) (b)

Fig.6. Surface of woven glass fiber (a) As received condition (b) Heated at 600 °C in hydrogen

3.3. Analysis ofmicrostructure

SEM micrograph of the CNTs grown woven glassfabrics shown in Fig.7. This figure shows that CNTs were grown in a radial direction around the surface of 10 µm diameter unidirectional glass fibers.The high resolution (3500×) image (Fig.7 (b)) of single fiber represents the densely populated CNTs around the fiber. Similar growth mechanism was found our previous works published elsewhere [27] where carbon fiber was used as the substrate. In bothstudies, CNTs grew uniformly over the fiber surface;not the space between the fibers (approximately 5 µm wide) which happened due to the absence of the catalyst. Previously presented increase in the flexure strength and toughness(See Fig.5) matches with this uniform growth result which suggests a balanced load transfer between the fiber and matrix.



(a) (b)

Fig.7. SEM images of CNTs (a) on the woven glass fabric (b) on single glass fiber

The mechanism of the strength and ductility transformation in the CNTs grown woven glass fiber composite can be modeled as shown in Fig.8. A single glass fiber reinforced matrix under uniform tensile stress is presented in this model. In a neat fiber reinforced composite the interfacial shear stress ($\tau(x)$) developes gradually on the interface and reaches the steady state until the critical length of the fiber is reached. In the case of CNTs grown woven composite, the radially grown CNTs result in the gradual reduction of interfacial shear stress along the length of the fiber toward the center of the composite. Therefore a resultant parabollic distribution of shear stress is developed along the length of the fiber, which increase the load carrying capacity of the interphase and the composite.

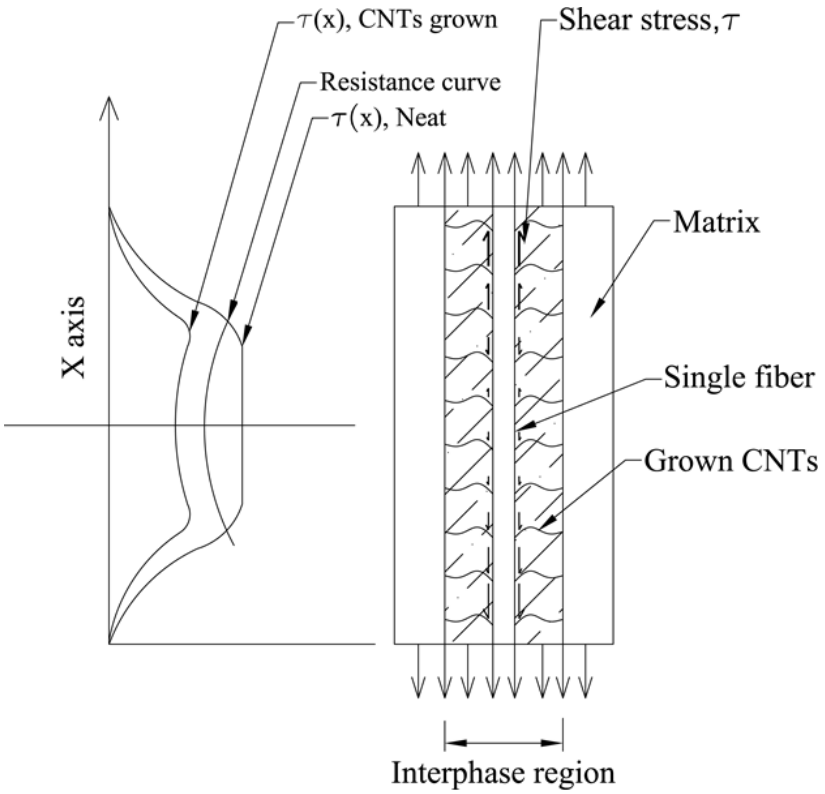


Fig. 8. Interphase model with a CNTs grown single fiber and matrix. The stress profile shows how the fiber matrix interfacial shear stress is generated along the fiber length.

IV. Conclusions

A controlled growth of CNTs was investigated on the woven glass fiber and the effect of this growth on the mechanical properties of woven glass fiber/epoxy composite was evaluated. CNTs grown woven glass fiber composite exhibited more inter-laminar resistance between the fiber and matrix which was revealed by ~17% and ~15% increase in flexural strength and flexural modulus, than the neat samples, respectively. The chemistry and appearance of the fiber surface was modified due to heat treatment at high temperature, which resulted in increased interfacial bond between fiber and CNTs. Uniform growth of CNTs was observed inthe microscopic analysis,which supported the evidence of a balanced fiber-matrix composite.A quantitative model was developed based on the growth of CNTs on the woven glass fiber to enumerate the development of improvedinter-phasewhich resulted in gradual reduction in the shear stress apart from the loading plane.

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