

# The Reinforcement of Carbon Nanotubes in Epoxy based CFRP Composites

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**Abstract**— Epoxy or polyepoxide is a thermosetting epoxide polymer that cures (polymerizes and cross-joins) when blended with a curing operator or "hardener". Most basic epoxy resins are created from a response amongst epichlorohydrin and bisphenol-A. Epoxy tar is most generally utilized as a lattice for cutting-edge composites because of their unrivaled warm, mechanical and electrical properties; dimensional solidness and synthetic protection. Epoxy surface coatings are among the most broadly utilized modern completes and give unrivaled attachment, adaptability and consumption protection when connected to metallic substrates. Epoxy gums are likewise utilized with different curing specialists, diluents and properties.

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## I. INTRODUCTION

Carbon nanotubes (CNTs) possess exceptional mechanical, electrical and thermal properties, making them ideal fillers for polymer nano-composites for various structural and functional applications. Factors influencing the properties of CNT nano-composites have been extensively studied and the development of nano-composites with much improved mechanical and functional properties have been reported. Incorporation of CNTs into a polymer matrix along with long fibre reinforcements to produce hybrid composites has also attracted significant attention in recent years. The mechanical and fracture properties were improved after addition of small quantities of carbon nanotubes to the matrix. It is also well established that the quality of composite components depends on the processing route adopted for fabrication. The aspect of producing CNT-CFRP has not been given due attention. The aim of this topic is to develop CNT containing CFRP hybrid composites for specialty applications. For high end structural applications composites are processed and by similar process of prepregging the epoxy-based CNT-CFRP hybrid composites are produced. The prepreg manufacturing involves, i) alignment of continuous fibre bundles or tows in the longitudinal direction, ii) continuous wetting, or impregnation, of tows using a polymer resin by passing them through a resin bath, iii) maintaining the uniform thickness of resin using a device called the doctor blade, and iv) collecting the impregnated fibres on a take-up spool. In this study, special focus has been placed on studying the effects of resin type and CNT content on various critical parameters in a solvent less prepregging process and also

the in variations of epoxy and prepreg during curing. Crucial mechanical properties, like strength, modulus, interlaminar shear strength and torsional shear properties of the resulting hybrid composites were evaluated. These are significant, especially for structural applications, like specialty sports goods and the main body of wind turbine blade.

### 1.1. Composite Materials

Any material which is made up of two or more different materials with a clearly distinguished boundary between them either prepared from naturally available organic ingredients or synthetically developed materials is called as composites. This material is selected for testing based upon its feasibility for following applications.

- Aviation
- Ballistic
- Bulletproof jackets & helmets
- Spacecraft
- Marine
- Automobiles

All the above mentioned are challenging applications & requires advanced materials of very high strength as well as low weight as possible i.e. high strength per unit weight. For this peculiar requirement carbon fibers and carbon nanotubes are most suitable among all available options. Due to this reason the resin is reinforced with carbon fibers & carbon nanotubes to prepare the composite materials of very good strength and sustainability in any physical environment. It also provides considerable weight reduction as compared to the metals.

Basically fiber reinforced composite materials are of 3 types:

- Polymer based composites
- Metallic based composites

• Ceramic based composites

Polymers generally contain groups of epoxides called as epoxy having number of molecules embedding in it. An epoxide group is an oxirane structure, a three-member ring with one oxygen and two carbon atoms. The reaction with amines, acids, amides, alcohols, phenols, acid anhydrides, or mercaptans cures few epoxide groups through polymerization by which thermosetting resins like epoxies are produced. The polymers are available in a variety of viscosities from liquid to solid.

**1.2 Functions of The Matrix**

- Bonding strength: The matrix binds the fibers together, holding them aligned in the important stressed directions. Fibers helps the composite to take compression, flexural and shear forces as well as tensile loads. The strength of composites embedding short fibers depends on the presence of the matrix as the load-transfer medium, and the efficiency of this load transfer on quality of the fiber/matrix bond.
- Fiber isolation: Fibers acts as separate entities if are separated from each other. Reinforcing fibers have with high variable strengths due to brittleness. These fibers materials are stronger as well as there is the additional benefit that the fiber aggregate does not fail catastrophically. Here, the fiber bundle strength is closer to monolithic rod of equivalent load-bearing ability. If fibers are separated from each other then and then only these cracks cannot pass unimpeded through sequences of fibers in contact, which would result in completely brittle composites.
- Environmental protection: The matrix should protect the reinforcing filaments from mechanical damage (e.g. abrasion) and from environmental attack. GRP materials do not suit to this as glass fibers permits diffusion of water and the environmental damage that results is aggravated by stress. In order to counter the damaging nature of alkaline cement having matrix alkali-resistant glasses including zirconium have been developed. In composites like MMCs or CMCs, fibers are subjected to oxidation attack at higher temperature, so matrix safeguards the fibers.
- Crack stoppage: A ductile matrix will provide a means of slowing down or stopping cracks that might have originated at broken fibers. Conversely, a brittle matrix may depend upon the fibers to act as matrix crack stoppers.
- Toughness: The toughness of composite can be increased with the matrix as its quality of grips on fibres is also good.
- Flexibility: Many matrix materials are weak as well as flexible as compared to common reinforcing filaments and their strengths and moduli are often neglected in calculating composite properties. Inherent shear stiffness and

compressional rigidity of metals in MMC's are important to determine the nature of the composite in shear and compression as they are structural materials in their own right.

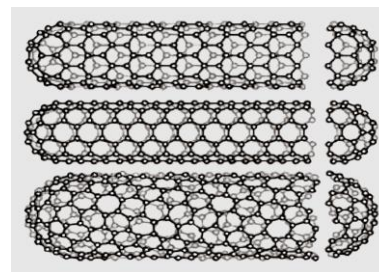
**1.3 Parameters Affecting Composites:**

**Fiber parameters affecting mechanical performance of composite:**

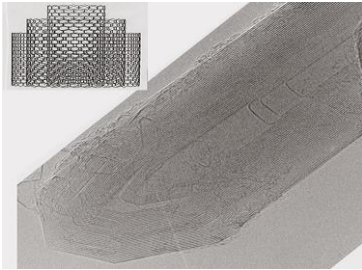
- Length: The fibers are of two types long as well as short. Long, continuous fibers have easy orientation, high impact resistance, low shrinkage, improved surface finish and dimensional stability compared to short fibers which have low cost, can be easily work and have fast cycle time fabrication procedures with fewer flaws and therefore have higher strength.
- Orientation: Fibers give very high stiffness and strength with respect to given direction. Fibers have high stiffness and strength in mat when oriented in more than one direction. However, it cannot match the stiffness and strength of unidirectional composites for the same volume of fibers per unit volume of the composite.
- Shape: Mostly fibers are circular in shape. Hexagon and square-shaped fibers having high strength and packing factors are also possible but they are difficult in handling and processing
- Material: Material of the fiber directly influences mechanical performance of a composite. Fibers should have high elastic moduli and strengths. This have been most important factors in graphite, aramids, and glass dominating the fiber market for composites.

**1.4 Carbon Nanotubes**

Carbon nanotubes (CNTs) are part of nanomaterial made up entirely of carbon in which multi-walled carbon nanotubes Industries focuses mostly on (MWCNTs). MWCNTs form a tubular shape by superimposing multiple layers of graphite.



*Fig.1.1. Sketch of three different SWNT structures as microscopy*



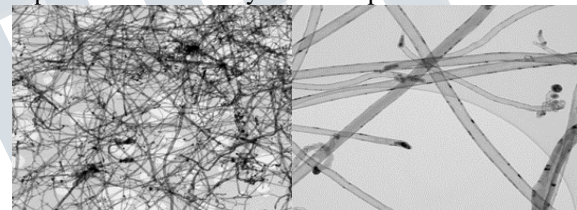
**Fig.1.2.High resolution transmission electron**

Such cylindrical graphitic polymeric structures can be used in major applications in electronics, optics and other fields of materials science due to their improved properties. CNT's have electrical and thermal conductivity, strength, stiffness, and toughness. It strengthens the structure, accessing solubility, dispersion, commissioning imaginative applications in materials, electronics, chemical processing and energy management, to name just a few. Usually industry is interested in, the electrical conductivity (as conductive as copper), their mechanical strength (Up to 15 to 20 times stronger than steel and 5 times lighter) and their thermal conductivity (same as that of diamond and more than five times that of copper). All these properties in combination provides a whole new variety of useful and beneficial applications.

## II. MATERIALS AND PREPARATION OF CNT MODIFIED MATRICES

The multi walled carbon nanotubes (NK-50 supplied by Nano Carbon, Korea) used in this work were of bamboo-like structure and were produced by a vapor grown method. The outer diameters ranged between 40-60nm and the lengths were about 20um. Typical TEM images of these CNTs are presented in Fig. 2.1. The rheological behaviors were studied through two different epoxy resins; one (EP1) was made from Epon 828 (supplied by Shell), a DGEBA epoxy, and the other (EP2) was Araldite LY556 (supplied by Huntsman). The main reinforcement used was carbon fiber roving (Pyrofil TR 30S, supplied by Mitsubishi Rayon, Japan) . Araldite LY556 resin system was eventually selected for making prepregs, which was composed of Araldite LY556, Aradur 5021 and hardener XB 3471 (all supplied by Huntsman), which were mixed in the ratio of 100:25:12 parts by weight. Natural as well as operating CNTs were used for this. The functionalisation method was a combination of processes chosen based on extensive studies reported previously. The first of all CNTs were oxidized in a UV/O<sub>3</sub> chamber (Jelight 144AX-220) for 30 min, next treated by a surfactant. In order to improve the dispersion in the resin the CNTs were treated by using

polyoxyethylene phenyl ether (Triton X-100, supplied by VWR international, UK) with the critical micelle concentrations (CMC) value of 0.2mM at 25°C. The procedure adopted for the treatment was basically similar to that reported previously. A desired amount of CNT was dispersed in acetone containing 10CMC of surfactant, equivalent to a Triton weight to acetone volume ratios of approximately 12.5 mg/1000 ml. The mixture+ was subjected to sonication in a bath (Branson 150) for 120 min. A desired amount of epoxy (EP1 or EP2), heated to 75°C to lower the resin viscosity, was added into the suspension of as-treated CNT/acetone to obtain 0.5wt% or 1.0 wt% CNT in the epoxy matrix. The initial dispersion was achieved by ultra-sonication at 60 °C for 30min, followed by degassing in a vacuum oven at 70°C overnight to ensure complete removal of acetone. The CNTs were mixed with high speed shear for 30 min to enhance dispersion. Two different speeds, 3000 and 4000 rpm, were used to study their effects on dispersion and viscosity of the suspensions.



**Fig.2.1. TEM micrographs showing morphologies of CNTs**

### 2.1 Rheological Studies and Curing Behaviour

Rheological studies were being used in order to select a suitable matrix material and identify optimal prepregging parameters. Types of resin blends containing 0wt%, 0.5wt% and 1.0wt% CNT with addition of epoxy resins were prepared. The viscosity changes were measured on an oscillatory rheometer (TA 300, TA instrument) using a parallel plate geometry (with 40mm flat plate and 300 μm in gap) was used of this purpose. By varying the viscosity evaluation of effects of shear mixing speed, type of epoxy and CNT functionalization was done. To determine a suitable temperature for the prepreg process Temperature sweep was also performed for the finally chosen resin system; the viscosity measurements were taken over a temperature range from room temperature to 50°C. Curing behaviours were determined by conducting Differential scanning calorimetry (DSC) analysis on CNT nanocomposites as well as hybrid composite prepregs.

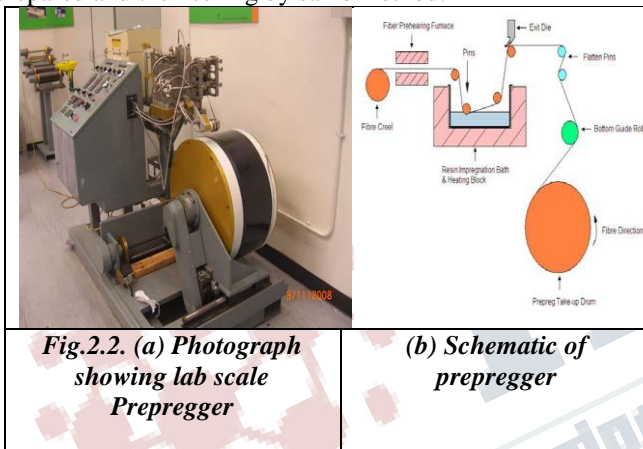
### 2.2 Prepreg Process and Composite Fabrication

Lab-scale prepregger was used for preparation of CNT-CFRP hybrid composite prepregs. (Model 40 Research Tool Corp., USA). A photograph and a schematic of the prepregger system are presented in Fig.2.2. Based on the

outcome of the rheological studies, the temperature of the resin bath was set at 37°C, which was optimised to maintain the viscosity of the CNT-resin mixture within the required limits (15~20 Pa s) and to achieve good wetting of the carbon fibre tow. The flattening pins and dies were also maintained at the same temperature. Prepregs were obtained with a consistent resin mass fractions of 60±5%.

**2.3 Fabrication of Specimens and Mechanical Tests**

The flexural properties and the inter laminar shear strength (ILSS) of the hybrid composites were determined according to the specifications, ASTM standards D790 and D2344, respectively. 2.5 mm thick laminates consisting of 9 layers of unidirectional prepregs were fabricated by hand lay-up and curing in a vacuum hot press at 90°C for 8 hours. Specimens of 12.7 mm wide x 70 mm long were cut from the cured composite plates. ILSS specimens were prepared by first cutting 5mm thick unidirectional laminate, secondly prepared and then curing by same method.

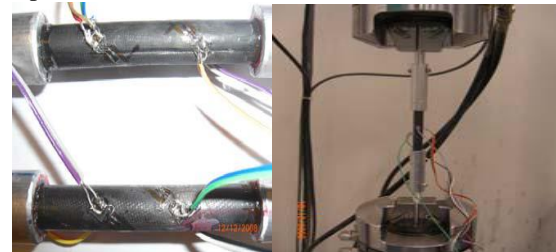


**Fig.2.2. (a) Photograph showing lab scale Prepregger**

**(b) Schematic of prepregger**

In-Plane shear moduli of the hybrid composites were determined using tubular specimens with a length of 100 mm and internal and external diameters of 6±0.2mm and 10±0.2mm, respectively, as shown in Fig. 2.2. Eight unidirectional, 90-degree, layers of prepreg were wrapped over a Teflon rod, which were then pressed in an outer mould consisting of identical two halves with internal longitudinal grooves with a diameter of 10.5mm in order to fabricate specimens. The tubes were wrapped with vacuum bags along with porous peel plies and breeder, which were then cured as described in the previous section; ends of composite specimens were bonded into aluminium tubes as the fixtures for gripping, Fig.2.3, according to the specification, Standard MIT-STD-375 (ASTM D5448). A gauge section length of 60mm was obtained after fixing. Two pairs of uniaxial strain gauges were bonded in a “fish bone” manner, each placed on the opposite sides near the two ends of the tubes. The specimens with the end tabs were

gripped on a universal testing machine (Instron Model 1125) and loaded at an angular velocity of 0.05 rad/min. At least three specimens were tested for each set of conditions.



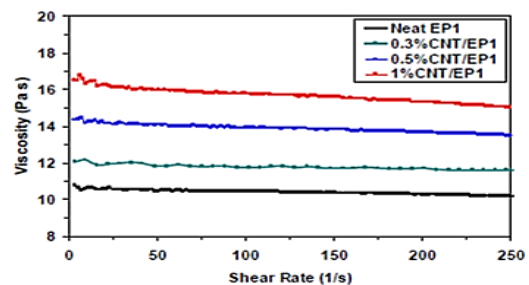
**Fig.2.3. Torsional shear specimens and test set-up.**

**III. RESULTS AND DISCUSSIONS**

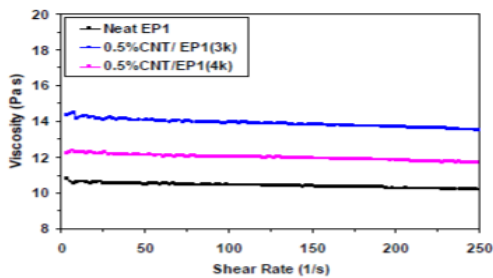
**3.1 Rheological Properties**

**3.1.1. Effects of CNT Content and Mixing Speed on Viscosity**

Viscosity is the single most critical parameter for processing of prepregs. It must fall within certain limits (1.5~ 2.4 Pa s) for processability and good wetting of fibres. A series of experiments was conducted to identify optimal processing conditions with improved CNT dispersion while maintaining the viscosity within the limits. Several important parameters, such as the effects of CNT content, mixing speed, type of epoxy and CNT functionalisation, were evaluated. Fig.3.1. shows the variation of viscosity as a function of the shear rate of untreated CNTs dispersed in an epoxy (EP1) using a high speed mixer at 3000rpm. The viscosity of the suspension was consistently higher for the nano-composites than for the neat epoxy, and it consistently increased with increasing CNT content. The overall responses of the CNT nano-composites were similar to that of the neat epoxy in terms of the Newtonian behaviour. However, small decreases in viscosity with increasing shear speed were observed for all materials studied. This observation was well expected due to shear thinning, which is in turn caused by localised shear-induced heating and a small decrease in aggregate size.

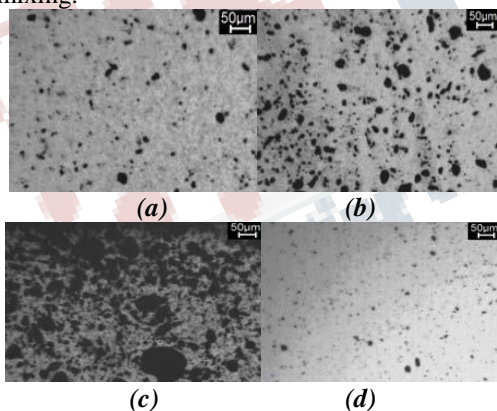


**Fig.3.1. Effects of CNT content and shear rate on viscosities of resins with and without untreated CNTs.**



**Fig.3.2. Variation of viscosity of untreated 0.5wt% CNT/EP1 suspension as a function of shear rate showing the effects of mixing speed.**

The viscosities measured for the 0.5wt% untreated CNT/epoxy suspension after mixing at a higher speed of 4000rpm are presented in Fig.3.3. At higher mixing speed there was significant reduction in viscosity showing the efficiency of high speed shearing in breaking up the CNT aggregates. This is further confirmed by the optical microscope images of the CNT/epoxy suspensions, containing different CNT contents and after dispersion at different mixing speeds, as shown in Fig.3.3. The aggregates of different sizes are clearly seen depending upon the processing parameters used: the aggregate size grew with increasing CNT content, and it was the largest for the 1.0wt% CNTs/epoxy suspension. Mixing at a higher speed significantly improved the dispersion with smaller aggregate sizes, thus further studies were made below using high speed mixing.

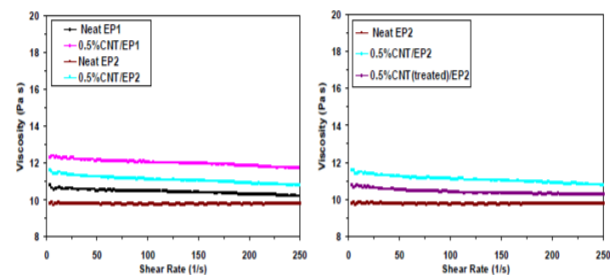


**Fig.3.3. Dispersion state of untreated CNTs in epoxy matrix (EP1):(a) 0.3wt% CNT/epoxy ; (b) 0.5wt% CNT/epoxy ; (c) 1.0wt% CNT/epoxy mixed at 3000rpm; and (d) 0.5wt% CNTs/epoxy mixed at 4000rpm**

**3.1.2. Effects of Epoxy Type and CNT Functionalisation on Viscosity**

To further study the effects of epoxy type, both untreated and functionalised CNTs were dispersed in the EP2 resin system using the same procedure and dispersion speed of

4000rpm. Fig.3.4. (a) shows the variation of viscosity for different materials. It should be noted that the EP2 resin system without CNT has a lower viscosity (9.8 Pa s) than the EP1 resin (10.8 Pa s), so were the corresponding nanocomposite suspension containing 0.5wt% untreated CNTs. By using the same procedure 0.5wt% CNTs functionalised by a surfactant were dispersed in EP2 resin thus evaluating effects of functionalization. A further reduction in viscosity was observed compared to the CNT/EP2 nanocomposite suspension. The effects of surfactant treatment on viscosity of nano-composite suspension were significant, the viscosity was decreased to 10.7 Pa s at a low shear rate and increasing the shear rate did not much reduce the difference. The reduction in viscosity enhances dispersion of CNTs in the epoxy matrix, so the importance of functionalisation of CNT reinforcements for processability of Prepregs was proved repeatedly. The hydrophobic octyl group of the surfactant interacted with CNT through adsorption while the hydrophilic segment could link with epoxy through hydrogen bonding resulting in improvement of CNT dispersion.



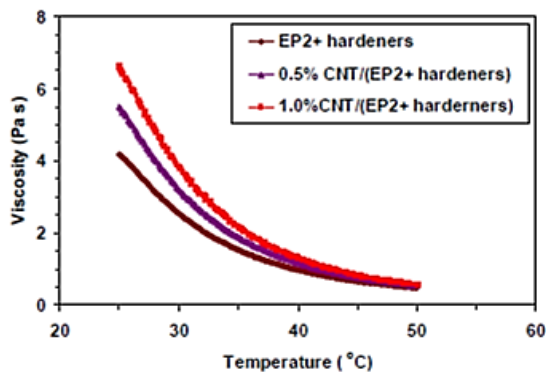
**Fig.3.4. Variation of viscosity of 0.5wt% CNT/epoxy suspensions as a function of shear rate: (a) effects of type of resin; and (b) effect of CNT functionalization**

**3.2 Thermomechanical Properties and Curing Behaviours**

**3.2.1 Curing Natures of Neat and CNT Modified Resins**

Various studies reported that CNTs could act as a catalyst for epoxies and initiate early stage curing. A large decrease in activation energy is not very desirable for a prepreg process as this may shorten the process time available for making prepregs and decrease the shelf life of prepregs. The DSC thermograms of the neat epoxy and nano-composites obtained at a ramping rate of 10°C/min are given in Fig.3.6, and important kinetic parameters determined from these curves are given in Table 1. The thermograms of neat epoxy and CNT nano-composites exhibited two exothermic peaks, representing two curing reactions, which were well expected as the resin system used (Araldite LY556, Aradur 5021 and hardener XB34) contained a co-curing agent in addition to the main hardener. The main exothermic peaks for both the

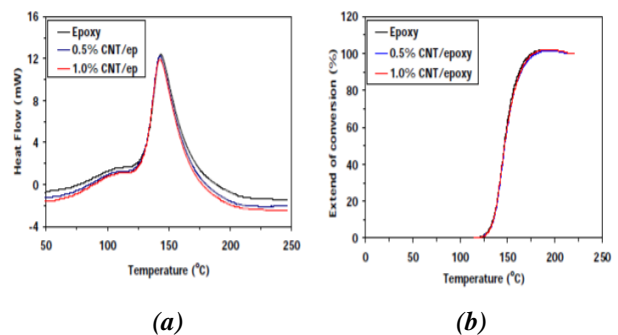
neat resin and the CNT nano-composites started at the same temperature, about 131°C (Table 1). The exothermic peak for the 1.0wt% CNT/epoxy was marginally lower than the neat epoxy or 0.5wt% CNT/epoxy, indicating catalytic behavior for nano-composites with a high CNT content. The heat gradient ( $\Delta H$ ) of 1.0wt% CNT nano-composite is significantly lower than that of the neat epoxy and the 0.5wt% CNT nano-composite, suggesting a reduction in the degree of cure. It is envisaged that the presence of a large amount of CNT nanoparticles physically hindered the mobility of the polymer chains and the cross linking process. To further study the curing reaction mechanisms and any possible catalytic effect of CNTs, the fractional extent of conversion of nano-composites are plotted against temperature as shown in Fig.



**Fig.3.5. Effect of temperature variation on viscosities of epoxy resins with and without CNT incorporation**

### 3.2.2 Curing Behaviour of Prepregs

The same experimental conditions were used for evaluating the thermo mechanical characteristics of the uncured CNT-CFRP composite prepregs. The curing parameters evaluated using thermograms are given in Table 2. The 1.0wt% CNT-CFRP composite prepreg exhibited a lower curing onset temperature,  $T_{onset}$ , a lower peak temperature,  $T_p$ , and a lower heat of reaction,  $\Delta H$ , than the neat CFRP and 0.5wt% CNT-CFRP hybrid composite prepregs. The variation of exothermic peaks in response to varying ramp rates of 5, 10 and 20°C/min were recorded. The activation energies were calculated from the slope obtained by plotting  $\log_{10}$  (heating rates) against the inverse of corresponding peak temperatures ( $1/T_p$ ) and are presented in Fig. As expected, the  $E_a$  values decreased consistently with increasing CNT content (Table 2) as a reflection of increased catalytic effect because of carbon nanotubes. The DSC thermograms measures the glass transition temperatures,  $T_g$ , of the prepregs which was cured at 90 °C for 8 hours as shown in Fig.



**Fig.3.6. Dynamic DSC thermograms of curing for neat epoxy and CNT nanocomposites: (a) plot of heat flow; and (b) Extent of conversion as a function of temperature**

**Table 1: Effects of CNT content on onset temperature,  $T_{onset}$ , peak temperature,  $T_p$ , time to peak temperature,  $t_p$  and heat of reaction,  $\Delta H$**

Sample	$T_{onset}$ (°C)	$T_p$ (°C)	$t_p$ (min)	$\Delta H$ (J/g)
Epoxy	131	144.0	1.26	262
0.5wt% CNT/epoxy	131	143.6	1.24	261
1.0wt% CNT/epoxy	131	143.2	1.19	254

Both CNT-CFRP hybrid composites showed a lower  $T_g$  than the neat CFRP without CNTs, where the reduction was significant for the hybrid composites containing 1.0wt% CNT as a result of a lower degree of cure. The physical hindrance of CNTs and CNT agglomerates impaired the mobility of the active groups in epoxy and the curing agent, leading to lack of curing and requiring a longer post-cure time for the 1.0wt% CNT-CFRP hybrid composites.

### 3.3. Mechanical Properties of Hybrid Composites

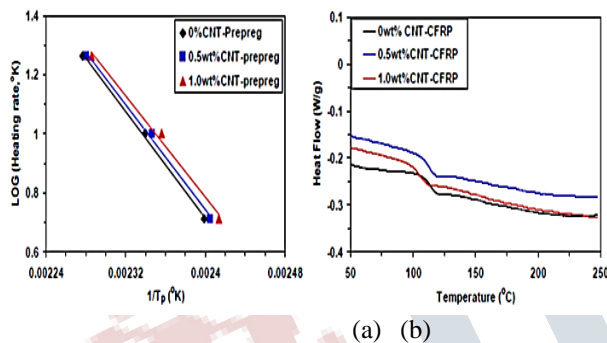
#### 3.3.1 Flexural Test

The flexural properties of CFRP and CNT-CFRP hybrid composites are shown in Fig.3.8. Since the carbon fibre content, stacking sequence and curing parameters were all kept the same, With in the data scattering, both the flexural strength and modulus increased marginally with the addition of 0.5wt% CNT. This was expected because the epoxy matrix contained well dispersed, functionalised CNTs. A further increase in CNT content to 1.0wt% however, was rather detrimental to the flexural strength and modulus, most probably due to potential agglomeration of CNTs a high content of CNTs.

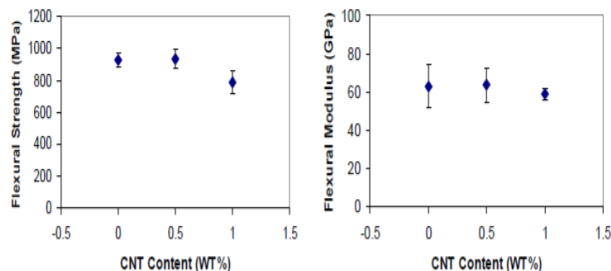
Table 2: Effects of CNT addition on onset temperature,  $T_{onset}$ , peak temperature,  $T_p$ , time to peak temperature,  $t_p$ , heat of reaction,  $\Delta H$ , activation energy,  $E_a$  and glass

transition temperature,  $T_g$ , for CFRP prepregs with and without CNTs.

Sample (°C)	Tonset (°C)	Tp (°C)	tp (min)	$\Delta H$ (J/g)	EaTg (KJ/mol)
CFRP prepreg 114.3	136	151.1	1.17	182.3	82.2
0.5% CNT/CFRP prepreg 112.8	136	150.1	1.15	174.1	80.7
1.0% CNT/CFRP prepreg 104.4	135	149.0	1.11	153.2	78.0



**Fig.3.7. Plot of Log (heating rate) vs. peak temperature for uncured prepregs; (b) thermograms of cured CFRP composite prepregs obtained at a heating rate of 100C@min.**

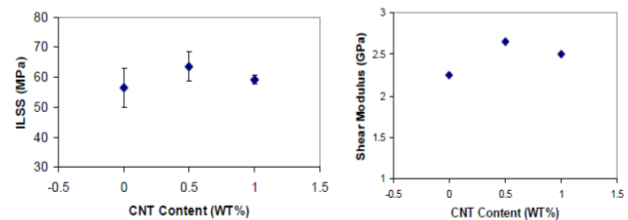


**Fig.3.8. Flexural Properties of CNT-CFRP hybrid composites as a function of CNT content**

### 3.3.2 Inter laminar Shear Strength and In-Plane Shear Modulus

The inter laminar shear strengths (ILSS) of CFRP and CNT-CFRP hybrid composites were measured using the short beam specimens and the results are shown in Figure . The ILSS increased by 11% with the addition of 0.5% CNT, but it decreased with a further increase in CNT content to 1.0wt%.

The shear modulus was measured by torsional loading of the tubular specimens. Torsional rigidity is considered one of the most important characteristics of many specialty structural composite components, such as golf shafts and turbine blades for wind mills. The shear modulus values presented in Fig. 3.9b show a trend similar to the ILSS against the CNT content with the highest value corresponding to about 0.5wt% CNTs. The improvements in torsional modulus against the neat CFRP were 18% and 11% respectively for the 0.5wt% and 1.0wt% CNT contents.



**Fig. 3.9. Shear properties of CNT-CFRP hybrid composites.**

## IV. CONCLUSIONS

Epoxy-based CFRP composites with and without carbon nanotubes(CNTs) in the matrix were prepared through a prepregging process. The mechanical properties that are directly relevant to specialty applications of CNT-CFRP hybrid composites, including the in-plane and torsional shear properties were characterised. The following can be highlighted from the study:

- The flexural strength and modulus of the composites were marginally improved after addition of 0.5 wt.% CNTs, while a further increase in CNT content to 1.0 wt.% was not beneficial due to potential poor dispersion of CNTs.
- The inter laminar shear strength of 0.5 wt.% CNT-CFRP hybrid composites increased by 12% compared with the neat CFRP composites.
- The torsional properties were measured using tubular specimens made from carbon fiber prepregs. The torsional modulus and strength of the 0.5 wt.% CNT-CFRP hybrid composites increased remarkably by 17% and 19.5%, respectively.
- The SEM examination of the fractured surface indicates that the improvements in the in plane and torsional shear properties are directly related to enhancement of both the interfacial adhesion between the modified matrix and carbonfibers, and the matrix strength/modulus due to the functionalized CNTs.

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