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Effect of CNT addition on cure kinetics of glass fiber/epoxy composite

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Abstract: In present time, developments in reinforced polymer composites have acquired preferential attention for high performance and high precision applications like aerospace, marine and transportation. Fibre reinforced polymer (FRP) composites are being substituted because of their low density, higher strength, stiffness, impact resistance, and improved corrosion resistance. Further laminated composites exhibit superior in-plane mechanical properties that are mostly governed by the fibers. However, laminated FRP composites suffer from poor out of plane properties in some applications. These properties can further be improved by the addition of Nano fillers like carbon nanotube (CNT), graphene and so on. Curing cycle plays a very important role in drawing out the optimum property of glass fiber/epoxy (GE) composite. It is expected that the cure kinetics can further be altered by addition of CNT due to its higher aspect ratio. The main objective of this work is to study the effect of CNT addition on cure kinetics of GE composite as multi-segment adsorption of polymer takes place on the CNT surface. In this study effects of curing parameters on mechanical properties and glass transition temperature of CNT embedded glass fiber/epoxy composite (CNT-GE) has been evaluated. For this study control GE and CNT-GE (with 0.1 wt. %) laminates were fabricated using hand lay-up technique followed by hot compression. The curing parameters that were considered in the present investigation were temperature (80°C, 110°C, and 140°C) and time (0.5 hr, 3 hr and 6 hr). For different combination of above mentioned temperature and time, samples of GE and CNT-GE composites were post cured. Mechanical properties were determined by flexural testing using 3 point bending fixture on INSTRON-5967 and thermal properties i.e. glass transition temperature (T_g) determined by Differential Scanning Calorimeter (DSC) to evaluate the effects of curing parameters. For CNT-GE samples, No much variation observed in flexural modulus with increase in post curing temperature and time, but swift increment was observed in flexural strength at 140°C with increase in post cure time. Elevation in T_g observed with increase in temperature and time duration of post curing; highest T_g noted at 140°C-6hr. Optimum post curing parameters for CNT-GE composite observed to be 140°C-6hr.

1. Introduction

When two or more materials are combined together the resultant material have improved properties as compared to the properties of original ingredient material, this resultant material is called as composite material. In recent time, due to increase in need for a material with combination of different properties, material having high strength with less weight or high corrosion resistance with less maintenance cost, fiber reinforced polymer(FRP) composite could be the answer. FRP composite is comprised of matrix polymer called epoxy and reinforcement which is glass fibers. Fibers have less diameter and long length resulting in higher aspect ratio which provides the composite, high strength and stiffness. Whereas epoxy provides shape and prevent these fibers from damage. Due to its high flexural strength, high flexural modulus and high corrosion resistance, it is the desired material for applications like space vehicles, automobile, marine and so on[1]. Fabrication of glass fiber/epoxy(GE) composite laminate goes through many different steps which decides the end properties. One of these steps is curing cycle, during which polymerization means cross linking of polymer takes place[2]. Once epoxy is polymerized it cannot be brought back to the initial condition, it changes from liquid to solid state permanently. Hardener is used to accelerate this cross linking. With correct selection of curing parameters; one can obtain the optimum properties of end product[3]. Two of the most important curing parameters are curing temperature and time duration which are



considered for the present study. Still during its life span these properties degrade with time. To improve these properties further, one method is to integrate second phase particulate in epoxy. Nano fillers like carbon nanotube(CNT), graphene and many more can be added for this purpose[4]. CNT have high aspect ratio which results in high specific surface area. Fiber/polymer interface is the weakest site in FRP composite, from where most failure initiate. Better interaction can be expected between CNT, polymer and fiber, resulting in improved interfacial bonding. It is possible that CNT addition may hinder the polymer-polymer bonding and promote polymer-CNT-polymer bonding and increasing its mechanical and thermal properties[5]. A relationship is observed between glass transition temperature(T_g) and degree of curing, hence differential scanning calorimetry(DSC) is done to evaluate T_g [6]. To evaluate changes in mechanical properties, flexural testing is done. It could be possible that CNT addition may affect the cure kinetics of the GE composite; to study this effect the current research is made.

2. Materials required and Experimental procedure

2.1 Materials required

Epoxy used for this study was diglycidyl ether of BisphenolA (DGEBA) and the hardener was Triethylene tetra amine (TETA), both were supplied by Atul Industries Ltd, Gujarat, India. The CNT used has diameter of ~10nm and length of ~5 μ m, purchased from sigma Aldrich. Glass fiber used is an E-type, 3 K plain weave and having 15 μ m fiber diameter, manufactured by Owens Corning, India.

2.2 Fabrication of CNT embedded GE composite

For the optimization of post curing temperature, laminates of glass fiber/epoxy (GE) composite and 0.1 wt. % of CNT filled glass fiber/ epoxy(CNT-GE) composite were fabricated. Initially CNT (0.1 wt. % of epoxy) was dispersed in acetone of 160mL and stirred for 0.5hr at a speed of 1000rpm. Then the epoxy was preheated for 0.5hr at a temperature of 70°C and stirred at a speed of 400rpm for better fluidity which facilitates the homogeneous dispersion of added CNT in the epoxy. Preheating of the epoxy was done; Parallely sonication of acetone + CNT suspension was done for 0.5hr to reduce agglomeration. After this acetone + CNT suspension was transferred to the flask which contains the preheated epoxy. Now the complete solution was stirred for 2.5hr at a temperature of 70°C and speed of 1000 rpm, to evaporate the acetone completely from the solution. Again sonication is done for 1hr followed by degassing for 18hr to remove the gases or air bubbles that are formed during the process of stirring and sonication. Laminate was fabricated by using hand layup method followed by hot pressing. Laminate were cut for the flexural testing as per the ASTM D7264standards using a diamond cutter.

2.3 Experimental method

2.3.1 Post curing schedule

Control GE and CNT-GE composite samples were post cured at temperatures of 80°C, 110°C and 140°C in a hot air oven for 0.5hr, 3hr and 6hr and then allowed to be annealed.

2.3.2 Flexural testing

This testing was performed on INSTRON 5967 UTM using 3 point bending fixture to evaluate the flexural strength. 1mm/min loading rate was used and test was performed at room temperature(25°C). Span length was kept equal to 16 x thickness of sample and overhang of 8 mm was provided.

2.3.3 Differential scanning calorimetry (DSC)

Differential scanning calorimetry is the technique used to study the effect of addition of CNT on the glass transition temperature(T_g) of the fiber reinforced composite. Change in the slope of the heat flow vs. temperature curve gives the glass transition temperature. This test was performed from 30°C to 180°C at heating rate of 5°C/min. T_g provides the range of temperature during which transition of epoxy from hard glassy state to soft rubbery state occurs.

3. Results and discussion

3.1 Effect of temperature and time on flexural strength and flexural modulus

Figure 1 shows the Variation in flexural strength with post curing time at different curing temperature for GE and CNT-GE samples. In figure 1 (a), at post curing temperature of 80°C no considerable changes observed in flexural strength with increasing curing time. Thermal energy required for better cross linking may not be available at this temperature for different time duration. Which is not the same case at post curing temperature of 110°C and 140°C, increment in flexural strength observed till 3hr of curing time, but for 6hr no notable change occurred at both temperatures. In figure 1(b), For CNT-GE samples increment in flexural strength can be observed as compared to control GE samples. Due to CNT addition, it promotes interfacial bonding which results in effective stress transfer from matrix to reinforcement. Energy supplied for the same reason goes on increasing with curing temperature and time. For CNT-GE composite maximum flexural strength noted at post curing parameters of 140°C-6hr.

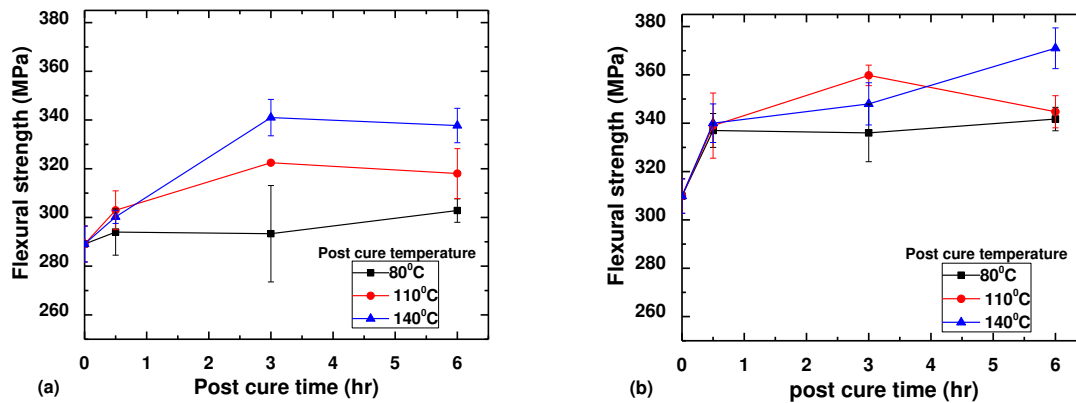


Figure 1. Variation in flexural strength with post curing time at different curing temperature a) for GE samples b) for CNT-GE samples

Figure 2 shows the Variation in flexural modulus with post curing time at different curing temperature for GE and CNT-GE composite. In figure 2(a) and 2(b), No notable change can be observed in flexural modulus for GE and CNT-GE samples, with increasing curing temperature and time. As flexural modulus is mostly governed by the glass fibers, further cross linking of the polymers does not have influence on overall flexural modulus of the composite.

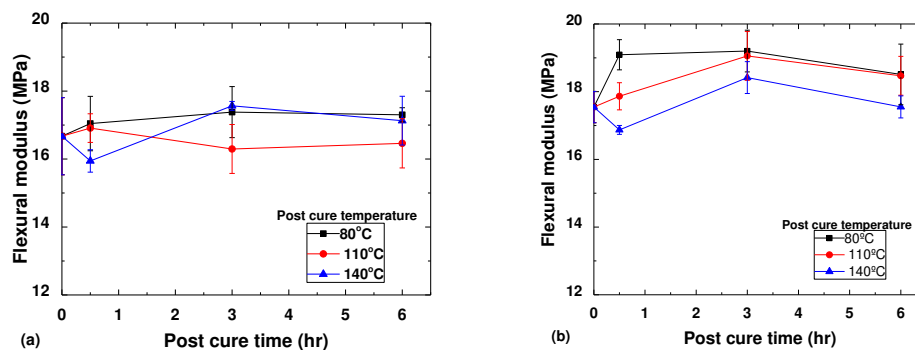


Figure 2. Variation in flexural modulus with post curing time at different curing temperature a) for GE samples b) for CNT-GE samples

3.2 Effect of temperature and time on Tg

Deviation in Tg with post curing time(hr) at various post curing temperatures can be observed in figure 3. Tg is acquired from the initiation of alter in slope of the heat flow against temperature. It was noticed that the Tg for GE samples is slightly higher than that of CNT-GE samples. CNT presence hinders the polymer-polymer chain interaction due to multi-segment adsorption of polymer on CNT surface, which hampers development of cross linking resulting in decreased cross link density of polymer-polymer chain, which leads to reduced Tg. In figure 3 (a) for GE samples, it can be observed that for all duration of post curing time, Tg is higher at post curing temperature of 140°C as compared to post curing temperature of 80°C and 110°C. It increases rapidly for all the temperatures till 3hr and after that the rate of increment decreases. Same goes for CNT-GE samples; there is an increase in Tg for all the temperatures till 3hr of post curing, but after 3hr to 6hr there is no considerable increment in Tg for post curing temperature of 80°C and 110°C. For post curing temperature of 140°C there is a continuous increase in Tg with increase in the post cure time showing no sign of saturation. Highest Tg noted for GE samples and CNT-GE samples was at post curing parameters of 140°C-6hr. The flexural strength for the CNT-GE samples are the highest for post curing at 140°C-6hr, confining better post curing and improved interfacial bonding.

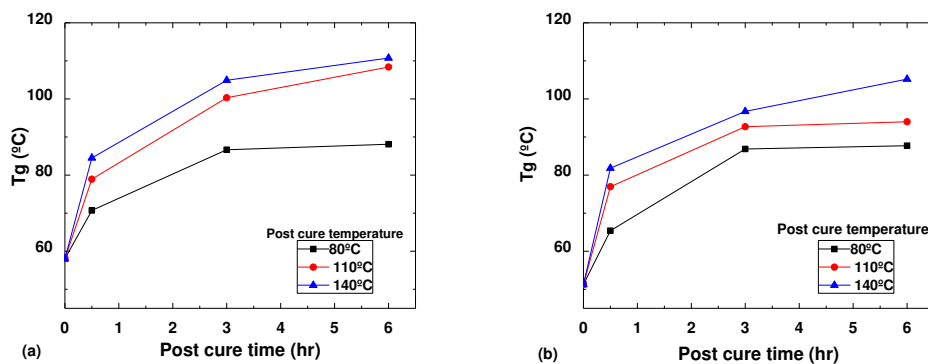


Figure 3. Deviation in Tg with post curing time at different curing temperature a) for GE samples b) for CNT-GE samples

4. Conclusion

The following conclusions can be drawn from the current study

1. Post curing at temperature 110°C and 140°C provides sufficient amount of activation energy for further cross linking of polymer which is not available at lower temperature like 80°C, that would be the reason for no considerable increment in the flexural strength and modulus, for the samples which are post cured at 80°C.
2. Optimum post curing parameters i.e. post curing temperature and curing time is observed to be 140°C and 6hr respectively for CNT-GE composite. Presence of CNT improved interfacial bonding resulting in increased flexural strength due to better stress transfer from epoxy to glass fiber. No considerable change in flexural modulus was observed as it mostly depends on glass fiber, not as much on cross linking of polymers.
3. Tg for CNT-GE samples observed to be lower than GE samples because of multi-segment adsorption of polymers on CNT surface, resulting in reduced polymer cross linking density which hinders the heat flow. Highest Tg value noted for GE and CNT-GE samples post cured at temperature and time duration of 140°C and 6hr respectively.

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