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Effect of CuO solid particles on thermal behavior of multilayer epoxy- based glass fiber composite

Abstract

In this research work, two types of composites' specimens are prepared by using $CaCO_3$, Al_2O_3, MgO, TiO_2 and CuO as functional fillers with epoxy as matrix and glass fiber as reinforcement. Initially three composite specimens are fabricated where TiO_2 particle of varying wt (4gm, 8gm, 12gm) is used with fixed wt of epoxy and glass fiber (120gm and12gm, 212gm). Similarly another three composites' specimens are prepared where CuO is used instead of TiO_2. In both the cases equal manual compression pressure is used. Thermogravimetric analysis (TGA) and scanning electronic microcopy (SEM) analysis of the prepared specimens are done with the help of calorimetric and Mountains®8 software respectively. Due to addition of TiO_2 and CuO it is observed that the melting temperature (Tm) and glass transition temperature (Tg) are elevated which indicate degree of composite crystallinity established by the strong interfacial interactions of CuO than that of TiO_2 particles and the amorphous region of the chain. As a result thermal stability of the composites prepared with CuO is enhanced over TiO_2. Particle scanning of the composites by Mountains®8 software analysis shows that with increasing projection area and mean diameter of functional filler particles, surface outlines of the composites are smoothen which has positive correlation with CuO particles in the composites.

Keywords: composite, glass fiber, epoxy, thermal behavior, tga, fillers

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Md Shahinoor Alam
Department of Mechanical Engineering, Dhaka University of Engineering and Technology, Bangladesh

Correspondence: Md Shahinoor Alam, PhD Research Student, Department of Mechanical Engineering, Dhaka University of Engineering and Technology, Gazipur-1707, Bangladesh, Tel +8801715009804, Email majshahin4282@gmail.com

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Introduction

Due to design flexibility and improved thermal stability, composites have already occupied a strong position in modern industries. Tremendous work has been done for the thermosetting polymers[1–4] and their composites[5–7] over the decades. Epoxy resins are one of the highly used polymer matrices for their versatile applications in the engineering field.[8–10] Thermal characteristics of polymer composites have got significances in structural design and applications like dimensional solidity and material nature at high temperature, load bearing capacity at certain temperature, end to end temperature transformation etc.[11] Many researchers have studied the influencing factors of composites' properties[12–14] and lot of modifications have been done on it to improve the thermal behavior in industrial applications. Micro and nano particle filer materials are now being used in epoxy matrix to manufacture composites with intended characteristics and enhanced performance. Due to addition of filler particles in it heat deflection temperature of epoxy is increased as well.[15] Latent heat storage capacity of phase change material (PCM) composites with support material remains consistent but variation in loading is observed due to particle shape, size micro structure and molecular interaction between PCM and support material.[16] Kinetic parameters of thermal decomposition can be interpreted to reduce chemical process in thermal analysis and are mainly used to find out material composition and to foresee thermal stability of composites.[17] In the initial condition without modifier polymer will have limited utilization due to their thermal decomposition and low shear stress which can be enhanced by adding functional fillers with matrix.[18] In composite manufacturing, it is very much essential to measure the melting and glass transition temperature of the material to ensure its proper environment for usages without any effect on it. Thermo plastics are used to increase the toughness of thermosetting resins for their high glass transition temperature.[19–21] Thermogravemetric analysis (TGA) is an important thermal analysis technique which has a wide application in polymer materials' characterization.[22] Through thermal analysis, properties of polymeric materials are found out as a function of temperature.

An epoxy based glass fiber composite when modified with CuO functional filler develop less thermal stress and high glass transition temperature in it resulting improved mechanical properties (impact strength: 957.008 J/m, tensile strength: 141.870 N/mm^2 and flexural strength: 214.683 N/mm^2).[23] Composites fabricated in a sandwitch form with the help of carbon fiber and polyvinyl chloride demonstrate weak strength and enhanced susceptibility to damage when experience huge penetration from a source in an aggressive environment (-70^{0}C).[24] Unstitched composites of carbon fiber reinforced polymer woven laminates experience considerable delamination and fiber damage when impacted by 6.7 J/mm in an extreme temperature (-70^{0}C). On the other hand, stitched composites of polyimide laminates (T650-35) demonstrate similar pattern detrimental effect when impacted by 13.4 J/mm at 23 and -70^{0}C.[25] The principal potential benefits of exploiting modified composites from the similar nature of fiber are to change the thermal, mechanical and physical properties to suit end use application. Therefore, the modification of the polymer matrix is essential to use it for intended purposes. Changing properties of the base materials by reinforcing fillers to the polymer matrix is very populous.[26] Filler materials addition in epoxy resigns reduces the cost and improves the thermal properties of it significantly.[27,28] Against this background, the aim of this research work is to investigate the influence of CuO particle on thermal behavior of multilayer epoxy based glass fiber composites modified with $CaCO_3$, Al_2O_3, MgO and TiO_2 or CuO.

Experimental

Composite preparation

By using hand lay-up technique initially three composite specimens are prepared with components weight as mentioned in Table 1 & Figure 1. Similarly another three composite specimens are

prepared taking components' weight as mentioned in Table 2. In the experiment 2 CuO is used instead of TiO_2 which is used in earlier case (experiment 1). Light compression pressure of similar load is applied in both the cases to fabricate the required composites.

Table 1 Components weight in experiment-1

Specimen name	Fiber slabs arrangement	Components wt							
		Matrix material wt (gm)		Fiber glass wt (gm)	Filler particle wt (gm)				
		Epoxy (LY556)	Araldite (HY951)		$CaCO_3$	Al_2O_3	MgO	TiO_2	Total
S1	G/ G/ G/ G/ G	120	12	212	4	4	4	4	16
S2	G/ G/ G/ G/ G	120	12	212	8	8	8	8	32
S3	G/ G/ G/ G/ G	120	12	212	12	12	12	12	48

Table 2 Components weight in experiment-2

Specimen name	Fiber slabs arrangement	Components wt							
		Matrix Wt (gm)		Fiber glass wt (gm)	Filler particle wt (gm)				
		Epoxy (LY556)	Araldite (HY951)		$CaCO_3$	Al_2O_3	MgO	CuO	Total
S4	G/ G/ G/ G/ G	120	12	212	4	4	4	4	16
S5	G/ G/ G/ G/ G	120	12	212	8	8	8	8	32
S6	G/ G/ G/ G/ G	120	12	212	12	12	12	12	48

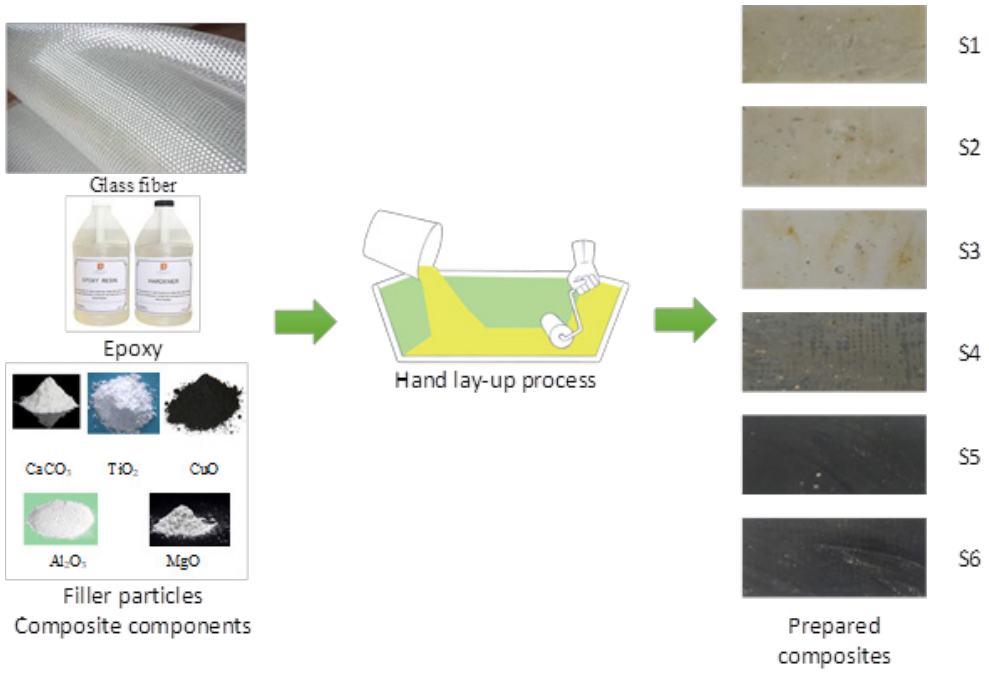

Figure 1 Preparation process of sample composites.

Test procedure

Thermal gravimetric (TG) test: In purging nitrogen of TGA instrument (SDT650 serial No 0650-0180), thermogravematric analysis is done within temperature range from 50ºC to 1000ºC at a heating rate of 5ºC per minute. Thereafter, the samples concerning differential scanning calorimetric (DSC) and thermo gravimetric (TG) measurements acquired from a trial specimen for measuring heat deflection temperature (HDT) by getting it cut at a perpendicular direction from the glass mat. 25–45mg weighing sample is taken for all the specimens in each case of TGA. From thermal reaction in the instrument, the analytical data of heat flow or weight loss in percentage is plotted along y-axis against temperature along x-axis in °C. These are known as DSC and TGA curves respectively and are used for differential thermal analysis and required interpretations.

Scanning electron microscopy (SEM) test: The electronic microscopic images of fabricated composites are taken with the help of SEM machine of model Hitachi SU-1510 by preparing specimens according to ASTM standard D 5299. These micrographs are analyzed using Mountains®8 software to observe surface morphology of the prepared composites (Figure 2).

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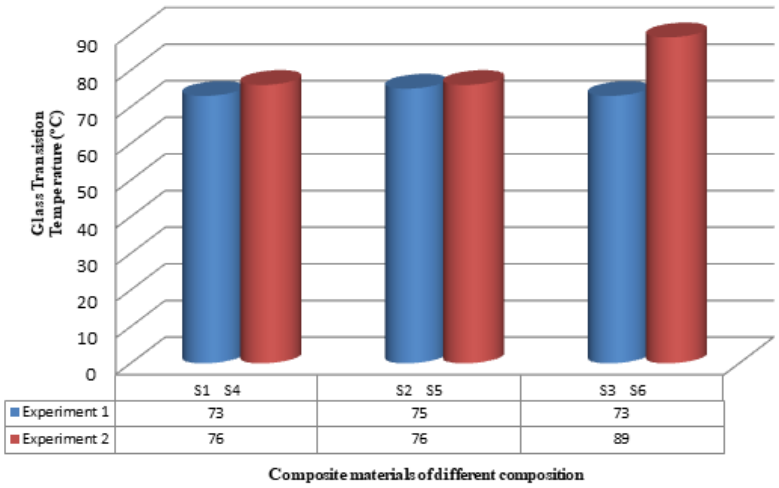
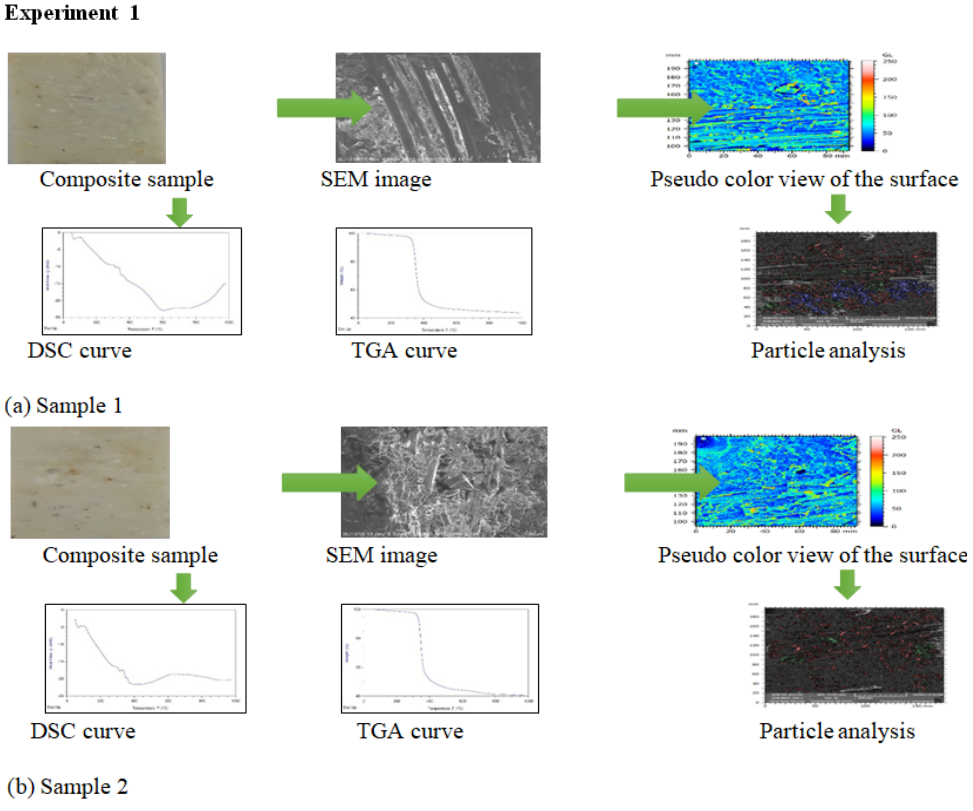


Figure 2 Comparison of glass transition temperature between the specimens of experiment1 and experiment 2.

Results and discussion

TG analysis

Due to physical change or chemical reactions there may be a change of temperature in the sample composites or visa-vis. This change of temperature is responsible for mass loss of composites. Thermal analysis generates different curves which assist us to find out important information regarding the composition and their stability in prepared composites. Unique thermogram of a particular material ensures its own testimony. DSC and TGA curve of the thermogram have got many important interpretations in analyzing fabricated composites (Figure 3 & Figure 4). Initial inflection point or starting of decomposition temperature (275ºC), first inflection point, second inflection point, subsequent inflection point as well as final temperature (Table 3) are very symbolic to analyze as well as characterize the prepared composites. Experiment condition has direct influence on reaction temperature and interval. Therefore it is difficult to determine exact values. Generally in amorphous condition, melting (Tm) is seen in a crystalline polymer and glass transition (Tg) to polymer only. A polymer may be amorphous as well as crystalline. So it is very evident that a sample will show both glass transition and melting temperature (Table 4). If CuO filler particles are added with other specified material the glass transition temperatures of the prepared composites (S4, S5, and S6) are enhanced (Table 4 & Figure 4). On the other hand if TiO_2 is added the glass transition temperature of the prepared composites (S1, S2 and S3) are deteriorated (Table 4 & Figure 3). As a result the thermal stability, physical properties as well as mechanical properties of the composites fabricated with CuO will improve significantly than other type (Figure 2).



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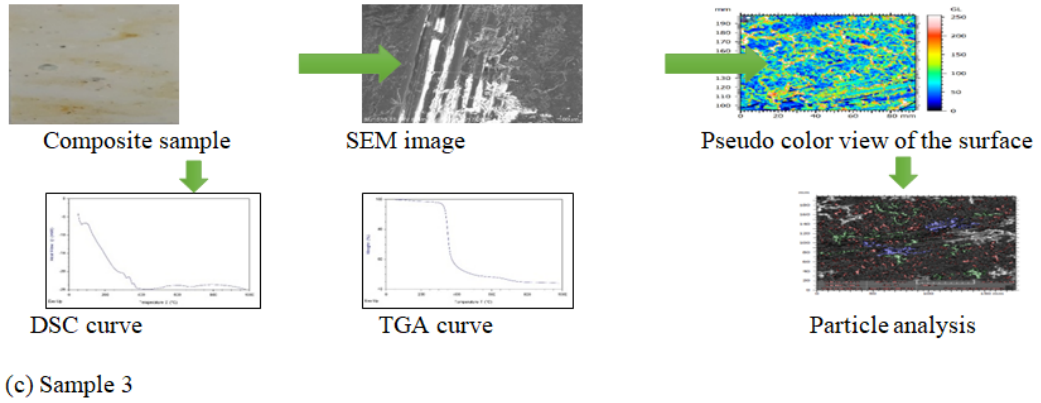
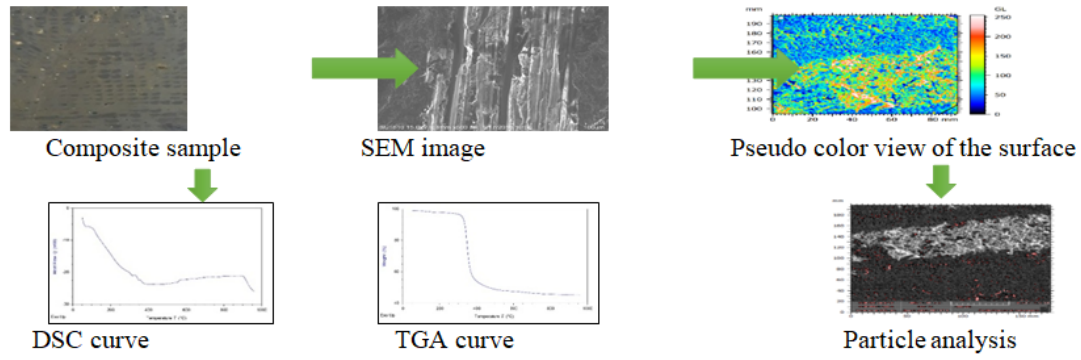
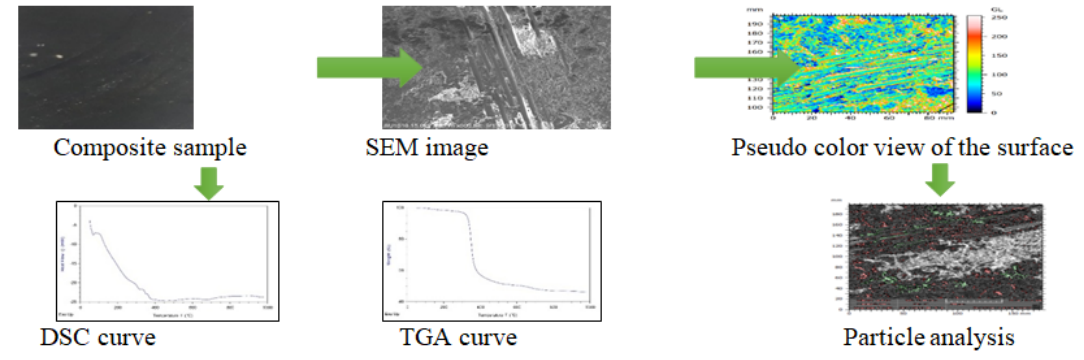


Figure 3 SEM and TGA Analysis of experiment 1 specimens.

Experiment 2





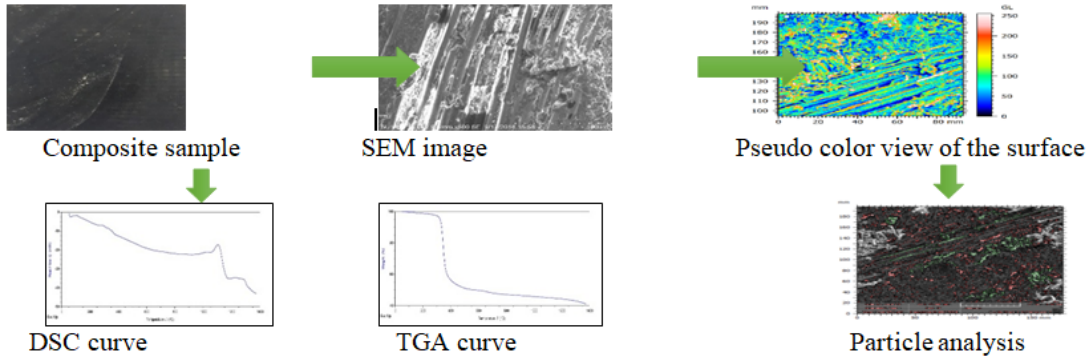


Figure 4 SEM and TGA Analysis of experiment 2 specimens.

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Table 3 Mass loss change in percentage form different inflection point of prepared composites

Specimens	First inflection point (°C)	Second inflection point (°C)	Mass loss change in percentage from initial inflection point (275°C) to first inflection point	Mass loss change in percentage from first inflection points to second inflection point
S1	580	830	51.35	2.50
S2	590	840	54.20	3.25
S3	590	820	50.25	4.50
S4	575	820	47.50	2.50
S5	580	820	39.40	2.50
S6	580	900	48.10	4.00

Table 4 Glass transition temperature and initial mass loss of prepared composites

Specimens	Glass Transition Temperature (° C)	Mass Loss Change in Percentage from 50° C to initial inflection point (275° C)
S1	73	1.65
S2	75	1.80
S3	73	1.75
S4	76	1.90
S5	76	1.60
S6	89	1.90

Volatility and inflection point are two important considerations to determine the temperature range regarding the mass loss in prepared composites. At 275 ºC temperature, a persistent and reasonable wt loss (1-2%) is found in all the cases. On the other hand the smooth change in the gradient of slope with 580ºC to 590ºC temperature range could properly establish the point of inflection (Table 3 & Figure 3 & Figure 4).

SEM micrograph analysis with Mountains®8 software

The continuity of fiber, uniform distribution of filler particle and their synergism as well as the morphology of composites can be investigated well by analysis the SEM images through Mountains®8 software. Important changes in the major structure and homogeneity in filler particle distribution were seen in all the sample composites. However, when the composites are reinforced with CuO filler particles, noteworthy shift in the microstructure were noticed (Figure 4). SEM images of the composite specimens are studied with the help of Mountains®8 software and illustrated in Figure 2 & Figure 3 & Table 5. Pseudo color view of the surface and particle analysis in threshold detection method (Table 5) demonstrate that the sample composites S1, S2 and S3 are less efficacious than that of sample composites S4, S5 and S6. Again when the mean equivalent diameter and projected area of functional particles are increased, the interfacing and adhesion of particles with fibers are improved and smooth outlines of the composite surfaces are found (Figure 3 & Figure 4). However, composites prepared with CuO attributes better morphology than that of composites prepared with TiO_2. Few irregularities are also seen in some composite samples which may be caused due to voids in it and insufficient bonding and adhesion among matrix, fibers and fillers (Sample S2). Lack of adhesion and stress transformation between fiber laminate and filler material is found in the microstructure of composites prepared with TiO_2 than composites prepared with CuO. This indicates improved thermal stability of the composites reinforced with CuO functional fillers which is also resolved by TG analysis.

Table 5 Particle parameters of sample composites through threshold detection method

Sample composites	Mean projected area (mm^2)	Mean equivalent diameter(mm)	Mean form factor
S1	1.519	0.7176	0.7673
S2	0.6075	0.5537	0.7803
S3	1.671	0.7484	0.7700
S4	0.5068	0.5485	0.7837
S5	1.085	0.6571	0.7721
S6	1.245	0.7032	0.7687

Conclusion

The adhesion, stress transformation and synergism among fibers, matrix and filler particles are well recognized in composites prepared with CuO than that of TiO_2. The glass transition temperature of composites is also increased due to CuO particle addition. These ultimately enhanced the thermal stability and surface morphology of the composites which are fabricated with CuO filler particles.

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Institute of Radiation and Polymer Technology (IRPT), Atomic Energy Research Establishment, Savar, Dhaka.

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Bangladesh Ordnance Factories (BOF), Gazipur, Dhaka.

Conflicts of interest

The author declares that there is no conflict of interest.

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