# Processing and Charecterisation of Epoxy/Carbon Black Nanocomposites by Twin Screw Extrusion

**R.M. Kulkarni[1*], G.B. Rudrakshi[1] and H.N. Narasimhamurthy[2]**

[1]Department of Mechanical Engineering, BEC Bagalkot, Karnataka, India
[2]RVCE, Bangalore, India
*Corresponding author e-mail: rmktg@rediffmail.com

**Abstract-This paper presents the experimental investigation of the influence of process parameters of twin screw extrusion on the dispersion, electrical, thermal and mechanical properties of epoxy/CB nanocomposites. Four factors, namely, loading level of carbon black in epoxy, screw speed; temperature and number of passes were selected for the design of L18 OA layout. Response factors such as electrical resistivity, glass transition temperature, microhardness and impact strength of epoxy/CB nanocomposites were studied. From TGA, the addition of CB to epoxy resulted in improved thermal stability. ANOVA of the experimental results showed that CB loading had significant influence on all the responses.**

**Keywords: Carbonblack (CB), orthogonal array (OA), thermo-gravimetric analysis (TGA), DOE: design of experiment, analysis of variance (ANOVA).**

## I. INTRODUCTION

Epoxy (LY556) has extensive application in aerospace and automotive industries for structural applications. However, it's insulating nature results in local heating and premature degradation of the structures and electronic components due to the accumulation of electrostatic charge on their surface. To avoid electrostatic charging of an insulating matrix, an electrical conductivity greater than 108 S-cm-1 is required. Conducting epoxy can be used in various applications including electromagnetic radiation shielding, electrostatic discharge (ESD) protection and electro-packaging applications to avoid electrical charges and electrical magnetic field from disturbing the communications due to its superior structural property.

Electrical conductivity can be achieved by incorporating highly conductive fillers, such as CB particles, carbon fibres, metallic fillers or intrinsically conducting polymers in thermosets and thermoplastics [1-6, 8-13]. Graphite and CB also have advantage of being compatible with many polymer systems. Though incorporating carbon nanotubes (CNT) and carbon nanofibers (CNF) in plastics exhibits better mechanical and electrical properties [8-13], the automobile and aerospace industries cannot rely on these materials for mass production due to high costs.

The nanofillers are dispersed in polymeric resins by several mechanical methods like ultrasonication, magnetic agitation, high speed stirring, shear mixing, etc. The improvement in the end properties due to the addition of CB is dependent on filler quality, amount of CB, and the dispersion technique adopted [1-5]. Twin screw extrusion

used for dispersing nanoclay in vinylester yielded superior results [6-7]. Several authors investigated electrical behavior of different grades of epoxies and had found that the percolation threshold varies between 0.5 and 4 % nanofiller content. Th. V. Kosmidou et al investigated the effect of dispersion of carbon black in epoxy (DGEBA/TETA) on electrical and mechanical properties. In both with and without post cured cases, at lower filler contents, Tg increased up to maximum value (at about 0.7 wt % filler). Typical dielectric behavior was observed below 1% CB. At higher CB contents conductivity increased significantly.

Though electrical property improvements of different grades of epoxy are reported, such studies involving LY556 which has extensive application in automobile and aerospace industries are scarcely available and also parametric study of twin screw extrusion for processing epoxy (LY556)/CB nanocomposites is not reported. Hence, the research was mainly focused on parametric studies of twin screw extrusion for dispersing CB in LY556.

## II. EXPERIMENTAL

### A. Materials

The resin used in this study was Bisphenol based epoxy of grade LY556 supplied by Huntsman, Hindustan Ciba-Geigy Ltd. The curing agent for this resin was Hardener HY951. Nanofiller used for fabrication of nanocomposites were carbon black N220 grade supplied by Philips Carbon Black Ltd.

### B. Design of Experiments

CB was dispersed using ultrasonication followed by twin screw extrusion (Alpha 18, Steer Engineering, Bangalore). Six levels for CB loading and three levels each for number of passes, temperature and screw speed were selected. Based on the factors and levels, L18 Orthogonal array was selected. The physical layout for the designed experiments is shown in Table1.

| Expt. No. | CB (%) | No. of passes | Temp, ($^0$C) | Screw speed, (rpm) |
|---|---|---|---|---|
| 1 | 0 | 5 | 5 | 75 |
| 2 | 0 | 10 | 15 | 100 |
| 3 | 0 | 15 | 30 | 125 |
| 4 | 2 | 5 | 5 | 100 |
| 5 | 2 | 10 | 15 | 125 |
| 6 | 2 | 15 | 30 | 75 |
| 7 | 4 | 5 | 15 | 75 |
| 8 | 4 | 10 | 30 | 100 |
| 9 | 4 | 15 | 5 | 125 |
| 10 | 6 | 5 | 30 | 125 |
| 11 | 6 | 10 | 5 | 75 |
| 12 | 6 | 15 | 10 | 100 |
| 13 | 8 | 5 | 10 | 125 |
| 14 | 8 | 10 | 15 | 75 |
| 15 | 8 | 15 | 5 | 100 |
| 16 | 10 | 5 | 30 | 100 |
| 17 | 10 | 10 | 5 | 125 |
| 18 | 10 | 15 | 15 | 75 |

### C. Nanocomposite preparation CB / Epoxy specimens

The CB was dispersed by wt. 0, 2, 4, 6, 8 and 10 % in epoxy resin using ultrasonicator 37 kHz for 1 h using tip sonicator and later by twin screw extrusion as per DOE. The screw extruded Epoxy/CB gelcoat was mixed with hardener by 11:100 ratios as per manufacturer's recommendation and cured at room temperature for 24 Hours.

## III. RESULTS AND DISCUSSIONS

### A. Electrical Conductivity, TG and Mechanical properties of epoxy / carbon black

At concentrations above 2 wt% CB, the resistivities were observed to be low and decrease marginally with increasing CB content. The temperature, numbers of passes and screw speed had very little effect on the conductivity when compared to that of CB loading in Fig-1.
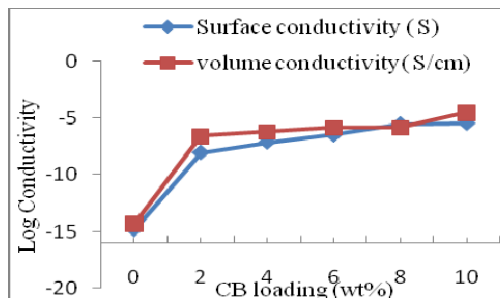


Fig. 1 Volume and surface resistivity for epoxy/CB nanocomposite

### B. Microhardness

Figure 2 shows that the microhardness has increased with increased CB content in the material and attained maximum value at 10 wt% CB. The micro-hardness increased with increase in number of passes. The maximum microhardness was found in 10 wt% CB loading which showed 30% increase in micro-hardness when compared to that of epoxy without any reinforcement. The increase of the CB content resulted in increase of the number of high strength reinforcements inside the composites, thus increasing their micro-hardness property.
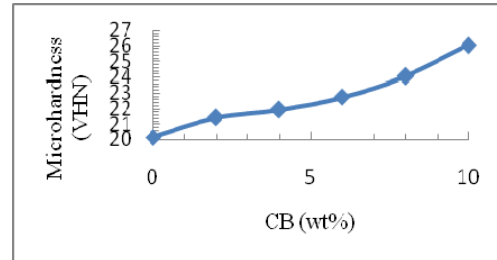


Fig. 2 Microhardness for epoxy / CB nanocomposite

### C. Tg of epoxy/ CB nanocomposites

Differential Scanning Calorimetry was used to determine the Tg of the nanocomposites using the measurement of heat flow versus change in temperature. Figure 3 shows the variation of Tg with increase in CB loading. The Tg was highest at 10% wt CB loading and resulted in 37% increase in Tg. The number of passes and temperature had very little effect on Tg but the same decreased with increase in screw speed. The level 1 of number of passes resulted in reduced Tg when compared to other two levels.
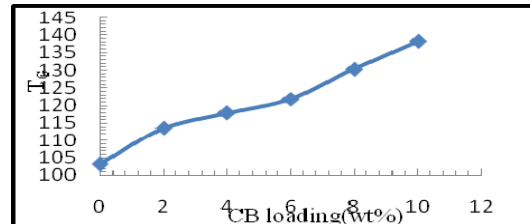


Fig. 3 Tg for epoxy / CB nanocomposite

### D. Impact strength of epoxy/ CB nanocomposites

Figure 4 shows the variation of impact strength of the nanocomposite with CB content. The impact strength was maximum at 10 wt% processed at 15 $^0$C, 15 passes and 75 rpm and showed 50% increase in impact strength at this combination.
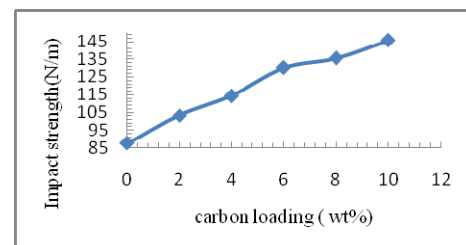


Fig. 4 Impact strength of epoxy / CB nanocomposite

## IV.  SIGNAL-TO-NOISE RATIO

To determine the effect each variable on the output, the signal-to-noise Ratio needed to be calculated for each experiment conducted. Taguchi recommends analyzing data using the S/N Ratio that will offer two advantages; it provides guidance for selection of the optimum level based on least variation around on the average value, which closest to target, and also it offers objective comparison of two sets of experimental data with respect to deviation of the average from the target.

Experiment 17 has the highest S/N Ratio with respect to both volume conductivity and surface conductivity and resulted in the best performance with least resistivity in both case. The S/N Ratio for the response microhardness is highest for the combination experiment 18 and has the best performance Glass Transition Temperature and impact strength.

## V. ANALYSIS OF VARIANCE (ANOVA)

ANOVA is mainly carried to analyze the statistical significance of different factors at different levels on the response variables. It is performed based on the DOE for all S/N Ratios .MINITAB module was used to perform ANOVA choosing General linear Model (GLM). This module was used to analyze the effect of factors on the responses and their significance on the responses of designed experiment. It has been performed for 0, 2,4,6,8 and 10 wt% epoxy/CB nanocomposite to examine the effect of process parameters on the twin screw extrusion Frmm Analysis of variance it is evident that CB is the most significant factor followed by number of passes, Temperature and Screw speed respectively for influencing the conductivity, microhardness, Tg and impact strength.

## VI. GREY RELATIONAL ANALYSIS

Grey analysis was carried out to optimize the parametric combination of twin screw extrusion for processing epoxy/CB nanocomposites. The multiple performance characteristics included conductivity, microhardness, Tg and Impact strength.

In Grey Relational Analysis, normalization of experimental data was performed. Linear normalization of the experimental results was performed in the range between zero and unity. The normalized data processing corresponds to larger the better type for all the responses.

$$\xi(k) = \frac{\Delta_{min} + \xi\Delta_{min}}{\Delta_{oi}(K) + \xi\Delta_{max}}$$

$\xi$ = distinguishing coefficient between zero and one.
In this study $\xi$ value is taken as 0 .5
$\Delta_{min}$ = smallest value of $\Delta_{oi}$
$\Delta_{max}$ = largest value of $\Delta_{oi}$

It has determined that the combination 10 wt% CB, 15 passes, $15^{o}$C temperature and 75 rpm screw speed had the highest value of grey relational grade and thus gets the highest rank. This indicates that this factor combination of experiment 18 is the optimal.

## VII. CONCLUSION

Epoxy/CB nanocomposites were fabricated using ultrasonication and twin screw extrusion to study the effect of CB loading on the electrical, thermal and mechanical properties of the nanocomposites. The effect of process parameters on the responses such as volume and surface conductivity, microhardness, Tg and impact strength were analyzed.  Based on the experimental results the following conclusions were drawn:

S/N Ratio analysis and ANOVA revealed that CB loading was the most significant factor on the resistivity, microhardness, Tg and impact strength. The resistivity decreased as loading of CB increased. A percolation threshold at less than 2 wt% was obtained in both volume and surface resistivity. Resistivity decreased with increase in number of passes and decrease in temperature. The screw speed had little effect on resistivity.

The microhardness, Tg and impact strength increased with increase in CB loading and number of passes and with decrease in temperature. The screw speed had little effect on Tg and impact strength.The Grey Relational Analysis helped to arrive at the best parameter combination 10 wt% CB loading, 15 passes, 150 C and 75 rpm, to achieve the best responses of resistivity, microhardness, Tg and impact strength collectively). The resistivity decreased by 1.0E10, microhardness increased by 29.96 %, Tg increased by 35.92 % and impact strength increased by 69.23% at the best parameter combination as per Grey Relational Analysis.

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