Available online at www.scholarsresearchlibrary.com

Scholars Research Library

Archives of Physics Research, 2015, 6 (1):20-25
(http://scholarsresearchlibrary.com/archive.html)

Archives of Physics Research
Scholars Research Library
ISSN : 0976-0970
CODEN (USA): APRRC7

Development and study on thermal, electrical and mechanical properties of Epoxy/ZnO nanocomposites

E. Sumi, S. Virgin Jeba and A. Lesly Fathima

Research Department of Physics, Holy Cross College (Autonomous), Nagercoil, Tamil Nadu

---

ABSTRACT

The enhanced thermal, electrical and mechanical properties can be explored by polymer based nanocomposites. In the present work, initially nanocrystalline Zinc Oxide particle is successfully synthesized at room temperature by sol gel method using Zinc acetate and Oxalic acid. The material is characterized using XRD, to determine the grain size. The nanocomposites are prepared for pure, 1wt% 2 WT% and 3wt% by mixing and pouring the nanoparticles with the epoxy resin and appropriate amount of hardener into the mould. The obtained polymer sheet is characterized using thermal, electrical and mechanical studies to study glass transition temperature, AC conductivity, tensile and elongation properties of the ZnO/epoxy nanocomposites respectively.

Keywords: Zinc acetate, Oxalic acid, Sol Gel method, Zinc Oxide, Epoxy resin

---

INTRODUCTION

Nanocomposite is a matrix to which nanoparticles have been added to improve a particular property of a material. The advanced properties of the nanocomposite have caused the researchers to use this material in several fields. Polymer nanocomposites consists of a polymer or copolymer having nanoparticles or nanofillers dispersed in the polymer matrix [1-2].

The synthesis of polymer nanocomposites is an integral aspect of polymer nanotechnology. By incorporating the nanometric organic compounds the properties of polymers improved and hence this has a lot of applications depending upon the inorganic material present in the polymers. Because of their nanometer sizes, filler dispersion nanocomposites exhibit markedly improved properties, when compared to the pure polymers. These include increased modulus and strength, outstanding barrier properties, improved solvent and heat resistance [3-4]. The mechanical and the electrical properties of the polymer nanocomposites will obviously differ from that of component materials [5].

MATERIALS AND METHODS

Synthesis of ZnO nanoparticle:
In a typical experiment, 0.5M Zinc acetate dehydrate and 0.5M oxalic acid were dissolved separately in double distilled water and stirred constantly using magnetic stirrer. The solutions were stirred thoroughly until both the solutions are mixed together. To this solution 8M of ammonia solution is added drop by drop to maintain a pH of 8-9. Then the solution was ultrasonicated two times with distilled water and few drops of ethanol. After cleaning, the solution is heated for about 100°C for 3hrs, and then the sample is powdered by using agate mortar for 1 hour. The powdered sample is calcined for about 2 hours in the muffle furnace [6]. After calcination the sample was again grinded and used for characterization purposes.

Preparation of Neat epoxy polymer nanocomposites:
Commercially available ARALDITE LY 554 epoxy resin along with hardener HY 951 is used as matrix material in fabrication of different polymer nanocomposites.

60gm of epoxy resin and desired amount of hardener are taken in the separate beakers. The presence of air bubbles, moisture or other foreign matter in the polymer matrix can act as defects, so degassing of the polymer mix was carried out whenever required. So the epoxy resin and the amine hardener were ultrasonicated separately about 30 minutes for the removal of air bubbles. Then the hardener was added with the epoxy resin and mixed thoroughly. The resultant mixture was again ultrasonicated and then poured into the mould made of aluminium metal. The mould was left for curing inside a hot air oven at 100°C for 2 hours. Thus the neat epoxy polymer sheet is obtained.

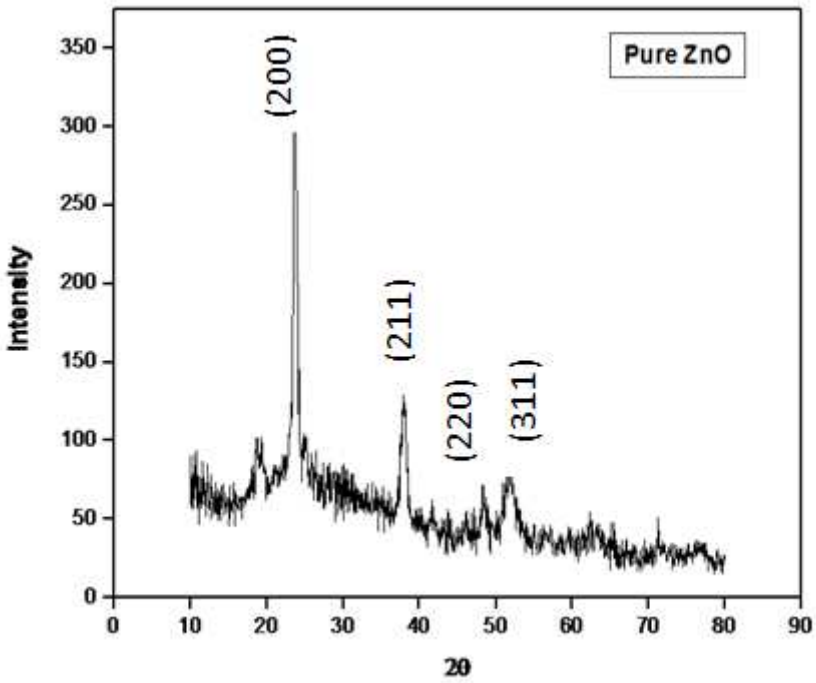
Preparation of ZnO/ epoxy nanocomposites:
Nanocomposites are prepared using a combination of two different processing techniques- mechanical mixing and ultrasonication.

Approximately 60gm of epoxy resin was poured with required quantity of filler particles and is slowly dispersed into the epoxy resin with continuous hand stirring. The mechanical mixer was then operated at 700 rpm and the beaker is sonicated for 60 minutes. Then appropriate amount of hardener (10 wt %) was taken into the beaker, mixed vigorously for few minutes and poured into the preheated aluminium mould. The mould was left for curing inside an oven at 100^{o}C for 2 hours [7]. Likewise, the polymer nanocomposite was prepared for 1wt%, 2wt% and 3wt% of ZnO nanofillers.

RESULTS AND DISCUSSION

X RAY DIFFRACTION ANALYSIS:
X- ray diffraction experiments were carried out with a XPERT-PRO diffractometer system with monochromated CuK_{α} (λ=1.54056Å) radiation. The grain size D is calculated from the prominent peaks using De-bye Scherrer's formula

$$D = K\lambda/\beta cos\theta$$



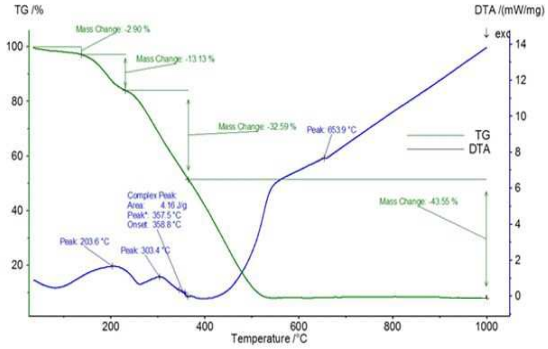
Using De-bye Scherrer's formula, the average grain size is found as 27.78nm

THERMAL ANALYSIS:
The thermal degradation characteristics of polymer/ metal nanocomposites were employed using STA 409 PL LUXX thermal analyser. TG/DTA curve of 3 wt% ZnO added nanocomposite is shown below.

Both neat epoxy and ZnO/ Epoxy systems have similar decomposition profiles and the degradation takes place in two stages. Initial weight loss (started at 100°C) was absorbed in the thermograms corresponding to evaporation of water molecules from polymer samples [7]. As evident from thermograms, the nanofiller have significant effect on thermal stability of polymers probably by preventing the decomposition of polymer backbone. The second step weight loss occurs due to the decomposition of polymer itself.

Table-1 : Thermal Analysis

Sample code	T_1 on set (T_{d1}°C)	T_2 on set (T_{d2}°C)	Glass transition temperature (°C)
Neat Epoxy	155	330	214
E+1Wt%	141	320	200.4
E+2Wt%	144	325	206.3
E+3Wt%	153	255	203.6



TG/DTA of epoxy +3 wt% ZnO

The TGA thermograms of Epoxy/ZnO nanocomposites system exhibits lower decomposition temperature behaviors compared to the Neat Epoxy. The thermal stability was not enhanced in Epoxy/ZnO polymer nanocomposites when compared with Neat Epoxy. From DTA analysis the glass transition temperature was determined and it was observed that the glass transition temperature of Epoxy/ZnO nanocomposite decreases when compared with Neat Epoxy. The change in Tg are due to the effects of nanoparticles only [7].

ELECTRICAL ANALYSIS

The capacitance (C) and dielectric loss factor (δ) measurements were carried out to an accuracy of ± 1% with Agilant 4284A LCR meter in the temperature range of 40-150^{0}C and with four different frequencies namely 1KHz, 10KHz, 100KHz and 1MHz. The observations are made while cooling the sample. Temperature is controlled to an accuracy of $\pm$ 0.5%. Air capacitance is also measured. The dielectric constant of the crystal is calculated using the relation,

$$\varepsilon = \frac{C}{C^{\circ}}$$

The A.C electric conductivity is calculated using the relation,

$$\sigma_{A.C} = \varepsilon_o \varepsilon_r \omega tan\delta$$

Effect of relative permittivity

The relative permittivity of the epoxy nanocomposites are less than that of the unfilled epoxy in the entire frequency range. The lowering of effective relative permittivity is due to mobilization of polymer chains in the epoxy nanocomposite. The more the filler, the more mobile are the polymer chains. On concluding, the relative permittivity increases as the temperature increases and the relativity decreases as the frequency increases [8].

Freq Hz	Dielectric Constant			
	Neat Epoxy	1 Wt %	2 wt%	3 Wt %
1K	6.105	4.128	4.128	4.441
10K	5.523	4.096	4.096	4.424
100K	5.214	3.339	3.339	4.404
1M	5.214	3.029	3.029	4.171

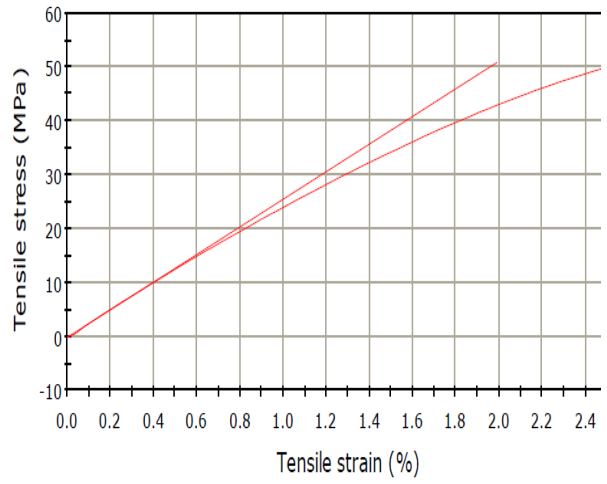
Effect of Tan delta

The tan delta values and A.C conductivity values of neat epoxy and ZnO/epoxy nanocomposites are presented in the table below. For neat epoxy and ZnO nanoparticle filled epoxy nanocomposites, there is a marginal decrease in tan delta values with increasing frequency for all filler concentration. This is due to the inability of charge carriers to traverse the thickness of the material at the higher frequencies [8].

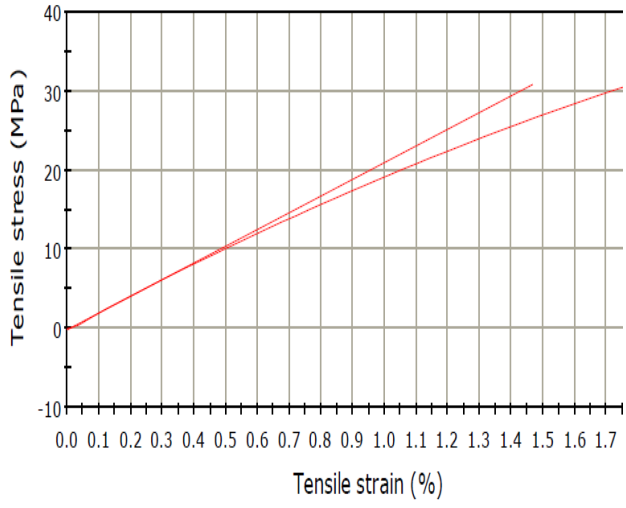
Freq Hz	Tan Delta			A.C Conductivity		
	Neat Epoxy	1 Wt %	3 Wt %	Neat Epoxy	1 Wt %	3 Wt %
1K	0.164	0.036	0.405	5.567	0.742	10.00
10K	0.155	0.021	0.272	4.759	0.437	6.690
100K	0.146	0.009	0.254	4.232	0.184	6.219
1M	0.132	0.005	0.212	3.826	0.101	4.916

MECHANICAL STUDIES:

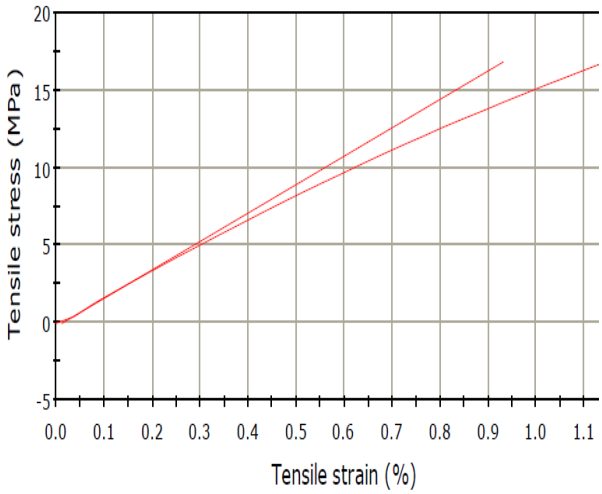
The tensile test measurements are carried out for neat epoxy and epoxy reinforced with ZnO nanoparticle



Stress-Strain Curve for Neat Epoxy



Stress-Strain curve for epoxy/1 wt% ZnO



Stress-Strain curve for epoxy/3wt% ZnO

Weight	Max load (N)	Extension at max load (mm)	Tensile stress at max load (Map)	Tensile strain at max load (%)	Modulus (GPa)
Neat Epoxy	1318.34	2.582	50.706	2.582	2.555
E+1wt%	1200.70	1.778	30.787	1.778	2.105
E+2wt%	843.155	1.101	16.215	1.101	1.763
E+3wt%	872.254	1.143	16.774	1.143	1.828

Tensile Properties

From mechanical studies tensile stress, tensile strain and modulus are found. Those illustrate that for neat epoxy the tensile properties are vastly better with maximum load than ZnO/epoxy nano composites. The decrease in the value is due to the formation of chain entanglement in the ZnO/epoxy matrix system which is due to the agglomerization and hence an unequal distribution of nanoparticle into the resin [10].

CONCLUSION

The ZnO nanoparticles are prepared by sol gel method. Neat epoxy and ZnO/epoxy nanocomposites have been synthesized by solution casting method. The synthesized polymer sheets are subjected to electrical and mechanical studies. The electrical analysis reveals that relative permittivity and electrical conductivity were decreased with the increase in frequency. The mechanical property characterization includes tensile properties for newly functionalized Zno/epoxy composites. The mechanical studies results that tensile strain, tensile stress and modulus are highly enhanced to neat epoxy when compared with ZnO/epoxy nano composites.

REFERENCES

[1] Arunkumar Lagashetty and Venkataraman (**2005**), “polymer nanocomposites”, resonance, july, pp 49-59.
[2] Zhaoyin Wen, TakahitoItoh, Takahiro Uno, Masataka Kubo and Osamu Yamamoto (**2003**), *Soild State Ionics*, 160,141-148.
[3] Xingyi Huang, Chonung Kim, Pingkai Jiang, Yi Yin and Zhe Li (**2009**), *Journal of Applied Physics*, 105, 014105.
[4] Kousik Dutta and S. K. De (**2007**), *Journal of Applied Physics* 102, 084110.
[5] ZhanhuGuo, Sung Park, H. Thomas Hahn, Suying Wei, Monica Moldovan (**2007**), *Journal of applied Physics* 101, 09M511.
[6] Bruna Martinello Savi, Larissa Rodrigues, Adriano Michael Bernardin (**2012**), " Synthesis of ZnO nanoparticles by sol-gel Processing", Ceramic and glass Materials group, Santa Catarina Extreme South University
[7] Ying-Ling Liua-Yuan Hsu, Wen-Lung Wei and Ru-Jong Jeng (**2003**), *Polymer* 44,pp.5159-5167.
[8] Santanu Singha and M. Joy thomas (**2008**), *IEEE Transactions on Dielectrics and Electrical Insulation* Vol. 15, pp1.
[9] I vlaeva, T Yovcheva, S Sainov, V Dragostinova and S Starvrev *Journal of Physics: Conference Series* 253, 012027 (**2010**).

[10] Ke Wang, Ling Chen, Jingshen Wu, Mei Ling Toh, Chaobin He, and Albert F.Yee (**2004**), "Epoxy Nanocomposites with Highly Exfoliated Clay: Mechanical properties and Fracture Mechanism".