ENHANCED THERMAL PROPERTIES OF TiO_2 NANOFILLER IMPOSED EPOXY COMPOSITES

A.Lesly Fathima, Department of Physics, Holy Cross College (Autonomous),

Nagercoil – 629 004, Tamil Nadu, India.

Email: leslysat@gmail.com

Abstract- Nanocomposites are new materials made with fillers which have nanosize. The purpose of this study is to analyze the thermal properties of epoxy resin with titanium dioxide nanoparticles. Titanium dioxide nanoparticles are prepared by sol-gel method by using the compounds Titanium tetra isopropoxide and acetic acid. The prepared Titanium dioxide nanoparticles are characterized by PXRD and the grain size in nanoscale is confirmed. The sheets of neat epoxy resin and epoxy with addition of TiO_2 are primed by solution casting method. The developed polymer is subjected to thermal studies. The inception decomposition temperature increases as the percentage of nanofiller increases**.** The glass transition temperature value is considerably increasing with the increase in amount of TiO_2.

Keywords: Sol-gel, Titanium dioxide, Solution Casting method, Epoxy, Polymers, Nanocomposites, TGA/DSC

1. INTRODUCTION

Polymer nanocomposites have attracted increasing attention in recent years because of their significant improvement of physical and chemical properties over the matrix polymers. The addition of just a few percent by weight of nanoparticles can result in significant improvement in thermal, dielectric and mechanical properties. Many studies have been carried out on the incorporation of rigidinorganic nanoparticles, which is a promising approach to improve both stiffness and toughness of plastics simultaneously [1–6].The effects of inorganic fillers on properties of the composites strongly depend on filler size and shape, type of particles, the fraction surface characteristics and degree of dispersion [7-8]. Various nanoscale fillers including montmorillonite, silica, calcium carbonate and some metal oxides have been reported to enhance the mechanical properties, thermal stability, electrical properties, gas barrier properties and flame retardancy of the polymer matrix [9-11]. Among various metal oxide fillers, nano-sized zinc oxide (ZnO), titaniumdioxide (TiO_2) and cerium oxide (CeO_2) fillers have attracted considerable attention because of the unique physical properties as well as their low cost and extensive applications in diverse areas [12-15].

2. EXPERIMENTAL DETAILS

2.1 Synthesis of TiO_2 nanoparticles

The nanopowder is prepared by sol-gel method. 1M of TTIP is mixed in 4M of acetic acid. The mixture is stirred for one hour using magnetic stirrer. To this mixture 10M of double distilled water is added dropwise. During the addition of water this mixture is transformed to gel. The obtained gel is kept for 24 hours. After aging of 24 hrs, gel is dried in an oven at 200°C. The soild crystals formed are ground by an agate mortar. The fine powder is calcined to 600°C in a muffle furnace for 2 hours.

2.2 Preparation of pure epoxy sheet

ARALDITE Epoxy resin (EP103) and hardener (HY- 956) are used in this study to form pure and TiO_2(1wt%, 3wt%) added epoxy/nanocomposites.Epoxy resin of 60gm and hardener of 6gm are poured into beakers separately. To remove the air bubbles, both are to be ultrasonicated for 30 minutes. After the completion of this process, the hardener is added and it is mixed with hand stirring. Finally it is ultrasonicated to remove any gas bubbles generated during the mixing process. After degassing, the mixture was poured into the mould. Then the mould is placed in an oven at 100°C to cure for 2hours. Thus neat epoxy sheet is obtained.

2.3 Preparation of Epoxy/TiO_2 polymer nanocomposite sheets

The TiO_2nanofillers (1 wt%) are dispersed into 60gm of epoxy resin, and both are mixed by a high speed mechanical mixer (at 600 rpm). It is then ultrasonicated to remove the gas bubbles. After the completion of the degassing process, the 6gm of hardener is added into epoxy/nano filler slowly with hand stirring. The mixture is ultrasonicatedfor another 30 minutes to remove any gas bubbles generated during the mixing process. After degassing, the mixture is poured into the mould. Then the mouldis placed into the oven at 100°C for 2 hours. The same procedure is repeated for 3 wt% nanofiller dispersed epoxy nanocomposite [16]. The photograph of developed polymer sheets is shown in Fig.1.

Fig.1: Photograph of developed polymer sheets

3. RESULTS AND DISCUSSION

3.1 Powder X- Ray Diffraction Analysis

The PXRD analysis was performed using XPERT-PRO diffractometer system with monochromatedCuK$_\alpha$ (λ=1.54056Å) radiation. PXRD pattern of synthesized titania nanoparticles is reported in Fig.2. The X-ray diffraction spectrum confirms that the synthesized Titania particle is in anatase crystalline phase. The data obtained is in good agreement with standard JCPDS card no.21-1272. The crystallite size D is estimated from the Debye Scherrer's formula

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

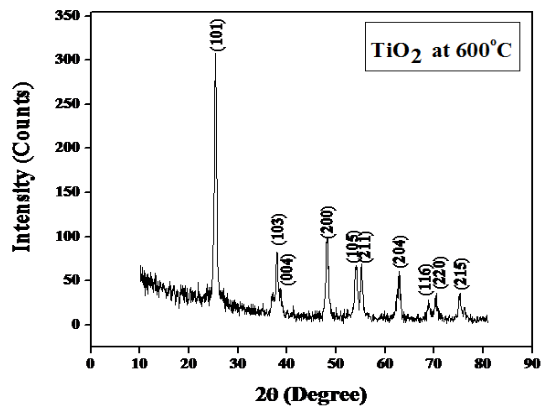

Fig.2: PXRD pattern of TiO_2 nanoparticles

The crystallite size of synthesized TiO_2 particle is found to be 15.98nm and this confirmsthat the prepared TiO_2 particle is in nanoscale.

3.2 Thermal analysis

The thermal properties are analyzed using thermogravimetric analysis and differential scanning calorimetry. The thermal analysis is performed using NETZSCH STA 449F thermal analyzer. 5 mg of dried material is heated from 20^{o}C to 600^{o}C at a scan speed of 10^{o}C/min. The thermogravimetric graph of epoxy/TiO_2nanocomposites is shown in Fig.3.

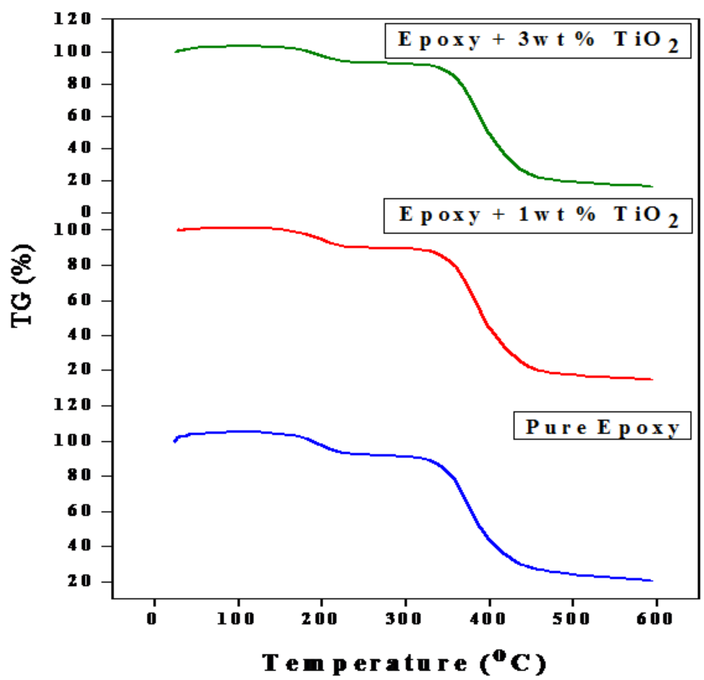

Fig.3: Thermogravimetric curve of pure and nanofiller (1 wt%, 3 wt%) added

Epoxy nanocomposites

ISSN 2091-2730

The inception decomposition for pure epoxy and nanofiller (1 wt%, 3 wt%) incorporated epoxy occurs at temperature 154ºC, 156ºC and 159ºC respectively. The inception temperature increases as the percentage of nanofiller increases [17]. The second decomposition temperature for pure,1 wt% and 3 wt%nanofiller added epoxy nanocomposites are 313ºC, 321ºC and 328ºC respectively. The major weight loss at temperatures higher than 310ºC is due to the effective dispersion of nanoparticles with epoxy resin. The temperature corresponding to 5% initial mass loss ($T_{5\%}$) for pure,1 wt% and 3 wt%nano TiO_2 added epoxy composites are 201ºC, 211ºC and 226ºC respectively. The TG variation with temperature designated the thermal stability of nanocomposites.

The differential scanning calorimetrycurves are shown in Fig.4. The glass transition temperature of neat epoxy is 71ºC which exactly matches with the reported value [18]. The Tg value of 1 wt% and 3 wt% TiO_2 added epoxy nanocomposites are 75ºC and 78ºC respectively. The glass transition temperature value is considerably increasing with the increase in amount of TiO_2. The observed increase in Tg of the nanocomposites may have primarily attributed to the resistanceto polymer chain mobility introduced by the presenceof nanoparticles in the epoxy matrix, which becomes comparatively more effective with increased homogeneity in dispersion of the particles in the matrix. The presence of particles restrains the mobility of polymer chain and reinforces the effect of cross-linking becauseTiO2 nanoparticles act as physical cross-linkers by increasing the apparent cross-link density.

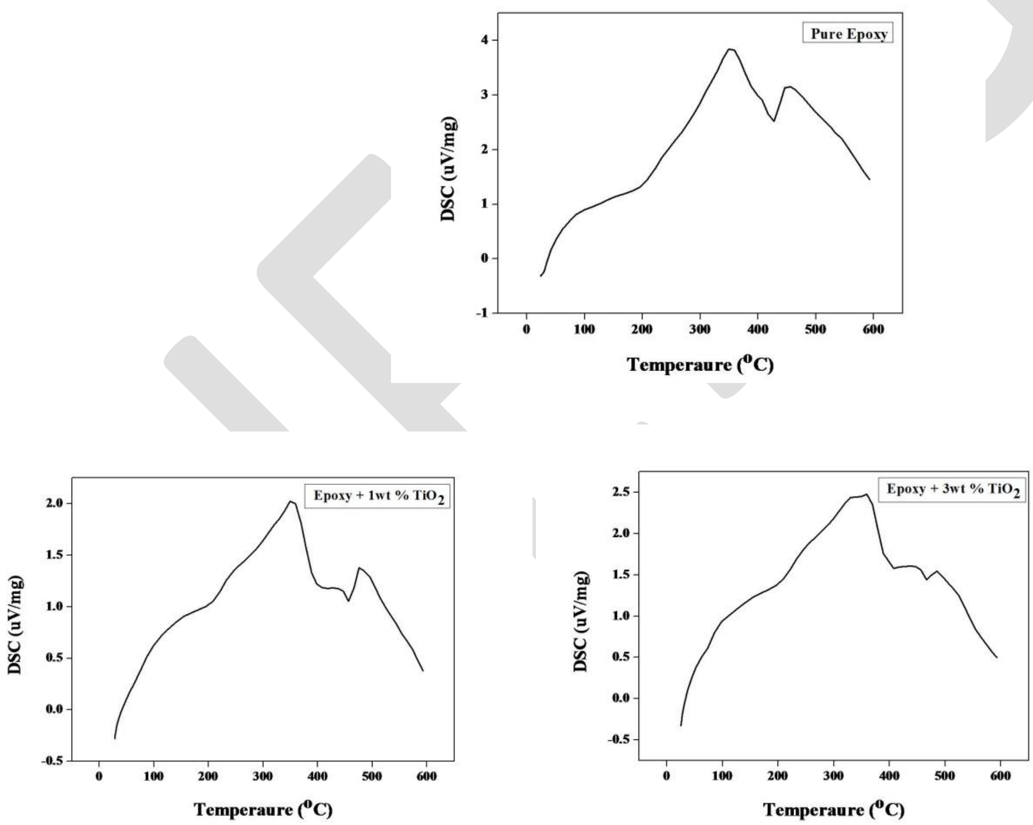

Fig.4: Differential Scanning Calorimetry curves of pure and TiO_2 (1wt%, 3wt%)added

Epoxy nanocomposites

4. Conclusion

Titanium dioxide has been prepared by sol-gel technique using titanium tetra isopropoxide and acetic acid. The powder X-ray diffraction spectrum confirms that the synthesized Titania particle is in anatase crystalline phase. The crystallite size of synthesized TiO_2 particle is found to be 15.98nm. Neat and nano filler added epoxynanocomposites are synthesized by solution casting method. An increaseof about 7ºC in the glass transition temperature (T_g) and significant improvement in thermal stability of epoxy/TiO_2nanocomposites are achieved with 3wt%nanoparticles loading in epoxy matrix, which is attributed to the homogeneous dispersion of nanoparticles inthe epoxy matrix.

REFERENCES:

1. MohagheghianMajid, Ebadi-Dehaghani Hassan, AshouriDavoud, MousavianSaman. "A study on the effect of nano-ZnO on rheological and dynamic mechanical properties of polypropylene" Experimentsand modelsComposites: Part B 42,2038–2046, 2011
2. Sun T, Chen F, Dong X, Han CC. "Rheological studies on the quasi-quiescent crystallization of polypropylene nanocomposites" Polymer, 49:2717–27, 2008
3. Zhang QX, Yu ZZ, Xie XL, Mai YW. "Crystallization and impact energy of polypropylene/CaCO3 nanocomposites with nonionic modifier" Polymer,45:5985–94 2004
4. Luyt AS, Dramicánin MD, Antic´ Zˇ, Djokovic´ V.Morphology, "mechanical and thermal properties of composites of polypropylene and nanostructured wollastonite filler" Polym Test, 28:348–56, 2009
5. Saminathan K, Selvakumar P, Bhatnagar N. "Fracture studies of polypropylene/ nanoclay composites. Part I: Effect of loading rates on essential work of fracture" Polym Test, 27:296–307, 2008
6. Zhao H, Li RKY "A study on the photo-degradation of zinc oxide (ZnO) filled polypropylene nanocomposites" Polymer, 47:3207–17, 2006
7. Haydar U. Zaman1, Park Deuk Hun1, Ruhul A. Khan, Keun-Byoung Yoon. "Morphology, mechanical, and crystallization behaviors of micro and nano-ZnO filled polypropylene composites" Journal of Reinforced Plastics and Composites, 31(5) 323–329, 2012
8. Chan CM, Wu J, Li JX and Cheung YK. "Polypropylene/ calcium carbonate nanocomposites" Polymer, 43:2981–2992, 2002
9. Galgali G, Agarwal S and Lele A. "Effect of clay orientation on the tensile modulus of polypropylene– nanoclay composites" Polymer, 45: 6059–6069, 2004
10. Alexandre M and Dubois P. "Polymer-layered silicate nanocomposites: preparation, properties and uses of a new class of materials" Mater SciEng R, 28:1–63, 2000
11. Motha K, Hippi U, Hakkala K, Peltonen M and Ojanpera V. "Metallocene-based functionalized polyole- fins as compatibilizers in polyolefin nanocomposites" J ApplPolymSci, 94: 1094– 1100, 2004
12. Chatterjee A. "Effect of nanoTiO2 addition on poly (methyl methacrylate): an exciting nanocomposite" J ApplPolymSci, 116: 3396–3407, 2010
13. Li YJ, Duan R, Shi PB and Qin GG. "Synthesis of ZnO nanoparticles on Si substrates using a ZnS source" J Cryst Growth, 260: 309–315, 2004

14. Zeng D, Xie C, Zhu B, Song W and Wang A. "Synthesis and characteristics of Sb-doped ZnO nanoparticles" Mater SciEng B, 104: 68–72, 2003
15. Yang Y, Chen H, Zhao B and Bao X. "Size control of ZnO nanoparticles via thermal decomposition of zinc acetate coated on organic additives" J Cryst Growth, 263: 447–453, 2004
16. Annlin Bezy.A, Lesly Fathima.A, "Effect of TiO_2 nanoparticles on mechanical properties of epoxy resin" International journal of Engineering Research and General science,3(5), 2015
17. Anand Kumar Gupta, Balakrishnan V.R and Tiwary S.K. "Synthesis of insitu generated ZnO incorporated PI high temperature resistive NC films: FTIR,AFM, XD, Microhardness and micromechanical analysis" International Journal of Polymer Technology, 1:181-188, 2009
18. Ghosh P.K, AbhishekPathak, Goyat M.S and SudiptaHalder. "Influence of nanoparticle weight fraction on morphology and thermal properties of epoxy/TiO2 nanocomposite" Journal of reinforced plastics and composites, 31(17): 1180-1188, 2012