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Mechanical Properties of Epoxy/TiO<sub>2</sub>-SiO<sub>2</sub> Hybrid Nano-Composites Khalid R. Al-Rawi

## Mechanical Properties of Epoxy/TiO<sub>2</sub>-SiO<sub>2</sub> Hybrid Nano-Composites

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## **Abstract**

In this paper, we attempt to improve the mechanical properties of epoxy resin by adding  $TiO_2$  and  $SiO_2$  with varying compositions (0, 1, 2, 3, and 4 wt %) respectively. Also epoxy hybrid nanocomposites containing different shape nano fillers of  $TiO_2$ :SiO<sub>2</sub> composites, with ratio (1:1), were prepared with varying compositions (0, 1, 2, 3, and 4 wt%). The mechanical properties of composites such impact , wearing, and fatigue are investigated as mechanical properties.

Keyword: Nanocomposite, Hybrid Epoxy/ TiO<sub>2</sub> -SiO<sub>2</sub>, Mechanical properties.

الخصائص الميكانيكية لمتركبات الايبوكسي/ ثاني أوكسيد التيتانيوم-ثاني أوكسيد السيليكون النانوية

الهجينة

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قسم الفيزياء - كلية العلوم للبنات-جامعة بغداد

الخلاصة

في هذا البحث تمت المحاولة لتحسين الخصائص الميكانيكية لراتنج الايبوكسي من خلال اضافة ثاني أوكسيد التيتانيوم و ثاني أوكسيد السيليكون وبتراكيز مختلفة (0,1,2,3,4 ) % على التوالي.كذلك تم تهيئة وتحضير متراكبات للايبوكسي هجينة من خلال استخدام نوعين مختلفين من المضافات وهما ثاني أوكسيد التيتانيوم و ثاني أوكسيد السيليكون وبنسبة ( 1:1) حيث حضرت بالتراكيز (0,1,2,3,4 ) % . وتمت فحص ودراسة الخصائص الميكانيكية مثل الصدمة والبلى والكلال.

كلمات مفتاحية: متراكبات نانوية ، هجينة ، ايبوكسي / ثاني أوكسيد التيتانيوم-ثاني أوكسيد السيليكون ، الخصائص الميكانيكية.



## **Introduction**

The field of nanotechnology is one of the most popular areas for current research and development in basically all technical scientific. This obviously includes polymer science and technology and even in this field the investigations cover a broad range of topics. Nanotechnology is now recognized as one of the most promising areas for technological development in the 21st century. In new materials research, the development of polymer nanocomposites is rapidly emerging as a multidisciplinary research activity whose results could broaden the applications of polymers to the great benefit of many different industries. Polymer nanocomposites (PNC) are polymers that have been reinforced with small quantities (less than5% by weight) of nano-sized[1]. High performance polymer composite materials are used increasingly for engineering applications under hard working conditions. The important factors influencing their performance are the molecular architecture, conditions of curing, the ratio of the epoxy resin and the curing agent [2]. The use of an additional phase (e.g. inorganic fillers) to strengthen the properties of epoxy resins has been a common practice, Epoxy resin is a thermoset resin with good thermal and environmental stability, high strength and wear resistance [3] .Epoxy resins are used in a variety of applications since their properties, such as thermal stability, mechanical and engineering response, low density and electrical resistance, can be varied considerably. [4]Particles smaller than tens of nanometers in primary particle diameter (nano particles) are of interest for the synthesis of new materials because of their low melting temperature point, special optical properties, high catalytic activity, and unusual mechanical and thermal properties compared with their bulk material counterpart interest in the development of nanocomposites consisting of organic polymers and (TiO<sub>2</sub>) or (SiO<sub>2</sub>) nanoparticles are growing[5].Organic-inorganic hybrid materials, especially polymer matrix composites with inorganic nanoscale building blocks, have drawn the widespread attention of researchers owing to the promise of combining the superior mechanical and thermal properties of inorganic phases with the flexibility and processibility of organic polymers [6]. The comprehensive performances of the composites depended on many factors, such as the intrinsic properties of the polymers, the methods of processing technology of the composites, the dispersion of the nanoparticles in the polymer matrix, and



the interfacial compatibility between nanoparticles and the polymer matrix. The recent investigation has shown that the epoxy/nanocomposites demonstrate some advantages in both mechanical and dielectric properties compared with pure resin system and epoxy with micrometer-size fillers at a lower loading concentration (1-10 wt%). In the case of micro composites, nanocomposite materials can be classified, according to their matrix materials, in three different categories Ceramic Matrix Nano composites (CMNC); Metal Matrix Nano composites (MMNC) and Polymer Matrix Nano composites (PMNC) [7]. Epoxy resins modified with inorganic particles such as carbon, TiO<sub>2</sub>, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, clay and soon have shown improved performances [8]. For inorganic/organic composites, the size of particles and the interfacial adhesion have great effect on the properties of the resin matrix. The well dispersed inorganic fillers in polymer matrices and compatibility between inorganic and organic phases are important to achieve an overall good performance [9, 10]. For inorganic/organic composites, the size of particles and the interfacial adhesion have great effect on the properties of the resin matrix. The well dispersed inorganic fillers in polymer matrices and compatibility between inorganic and organic phases are important to achieve an overall good performance[11]. The application of nanometer materials to the thermosetting resin for property modification is a promising channel. Compared to conventional inorganic/polymer composites that need over 30 wt% loading of microscale fillers, the same level of enhancements may be achieved with less than 10 wt% loading of well-dispersed nanoscale inorganic additives .[12].

Aim of work: to improve the mechanical properties of epoxy resin.

#### Materials

**Experimental** 

Epoxy resin was a FOSROC Co. product (nitofill EP L-V), Jordon. The density of epoxy resin was 1.04 gm.cm<sup>-3</sup>, with viscosity of resin about 12000cp at  $25^{\circ}$ c. TiO<sub>2</sub> (20nm) and SiO<sub>2</sub> (50nm) nanoparticles were provided by Degussa comp.Nano-SiO<sub>2</sub> and Nano-TiO<sub>2</sub> is likely to agglomerate easily because of its high surface energy

#### Preparation and Curing Procedure of EP/TiO<sub>2</sub> –SiO<sub>2</sub>

Prepare EP/TiO<sub>2</sub> and EP/SiO<sub>2</sub> nanoparticles composites by different concentration, and Prepared EP/TiO<sub>2</sub>:SiO<sub>2</sub> nanoparticles with ratio 1:1 as hybrid nanoparticles composites. Concentration of hybrid nanoparticles (0%,1%, 3%,4% and 5% )of resin respectively, were dispersed in the epoxy by using a ultrasonic stirrer, and mixed for 90 min at 50 °C . A mixture of all EP/TiO<sub>2</sub>, SiO<sub>2</sub> materials was degassed in vacuum at 70°C for about 20 min. The resulting mixture was then cast into a mold at room temperature. All samples were cured at 70°C for 2h to satisfy a full curing.

#### **Characterizations and Measurements**

The impact strength was measured on a tester of type MODEL (XJJ series), specimen without notch. In order to evaluate the dynamic toughness, the Charpy impact value measured using a standard impact fracture energy measurement, ASTM - (6110), Laryee, China Origin. The Charpy impact value is expressed by a following equation.

#### $E=M.(\cos\beta-\cos\alpha)$

E: Impact fracture energy (J), M: Impact force torque (N .m), $\beta$ : pendulum raising angle after samples breaking,  $\alpha$ : actual measurement value of pendulum rising angle.

The wearing characteristic was assessed by the weight loss, *W*, which was calculated by the following equation

#### $W = W_1 - W_2$

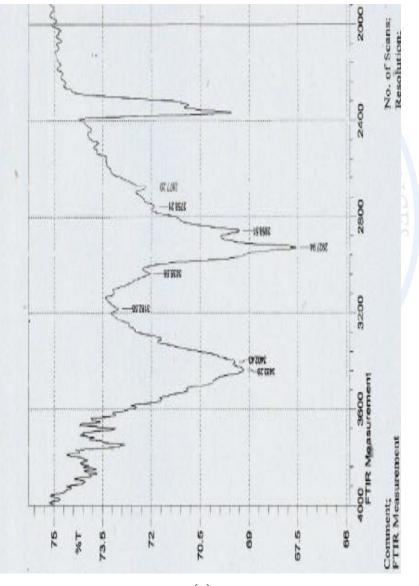
Where  $W_1$  and  $W_2$  are respectively the weight of a sample before and after its test. The fatigue tests were performed according to ASTM-D3479 specimens using an HI-TECH LIMITED Model No.:HSM 19,SER.No.E280 computer controlled loading frame. The applied load was sinusoidal with a frequency of 2 Hz, with 2 mm deflection a maximal load of (*P*max) 7 N and a stress factor of (*R*) 0.2. specimens were tested from the composite and the and reinforced hybrid composite on room temperature. All fatigue specimens were tested using the same machine. The machine cycles the specimens to failure and the number of cycles-to-failure was recorded by computer data acquisition system.





## **Results and Discussions**

The FT-IR spectra of EP, EP/TiO<sub>2</sub>:SiO<sub>2</sub> (1:1) hybrid nano composites material were illustrated in Fig.1. From Fig.1a, we can see that the hydroxyl-stretching band of epoxy resin appears at 3433 cm<sup>-1</sup>, and In Fig.1b (EP/TiO<sub>2</sub>:SiO<sub>2</sub> (1:1)) there is the absorption peak at 3429 cm<sup>-1</sup>. The characteristic of –OH stretching, which is the unreactive Ti OH and Si OH groups in inorganic networks .



(a) Fig.1, (a) FT-IR spectra of EP



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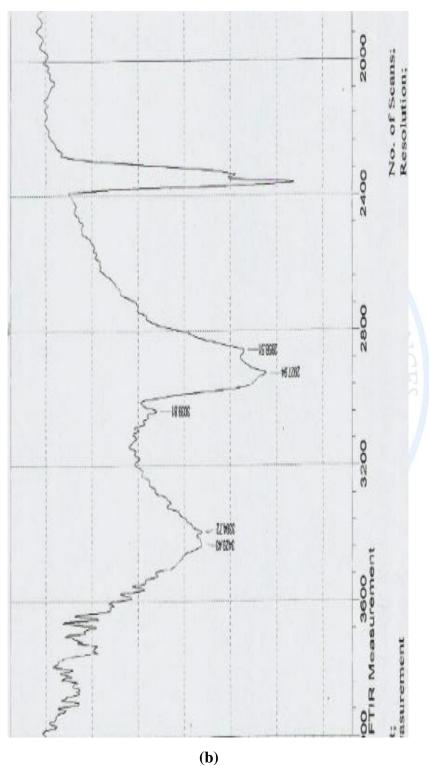


Fig.1(b),FT-IR EP/TiO<sub>2</sub> : SiO<sub>2</sub> 1:1 hybrid nano composites material



Fig.2 shows the effect of different weight percents of nano  $TiO_2$  and  $SiO_2$  fillers content on impact resistance of the EP/  $TiO_2$  and EP/  $SiO_2$  composites. It can be seen that the impact resistance increased at first and then decreased with the increase of  $TiO_2$  and  $SiO_2$  content. It reached the maximum when the  $TiO_2$  content was about 4wt% and  $SiO_2$  content was about 1wt%. Fig.2 shows the effect of ratio  $1:1 TiO_2$ -SiO<sub>2</sub> content on impact resistance of the EP/  $TiO_2$ -SiO<sub>2</sub> composites. It can be seen that the impact resistance increased at first and then decreased with the increase of  $TiO_2$ -SiO<sub>2</sub> content. It reached the maximum when the  $TiO_2$ -SiO<sub>2</sub> content. It reached the maximum when the  $TiO_2$ -SiO<sub>2</sub> content. It reached the maximum when the  $TiO_2$ -SiO<sub>2</sub> content. It reached the maximum when the  $TiO_2$ -SiO<sub>2</sub> content was about 2wt%. Since impact strength reflects the energy consumed before fracture, the results given demonstrated that the nano particles in the composites were able to induce plastic deformation of the surrounding matrix polymer to a certain extent under the condition of high strain rate [13].

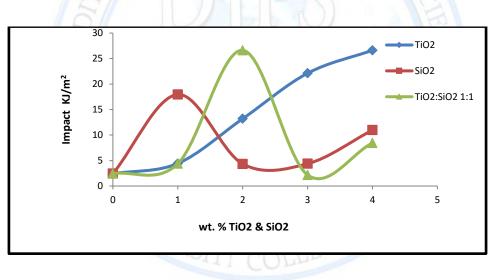


Fig. 2 The relation between impact strengths and TiO<sub>2</sub>, SiO2 contents

Fig. 3 shows the relationship between the wear loss and the  $TiO_2$  and  $SiO_2$  content respectively and shows the EP/ $TiO_2$ -SiO<sub>2</sub> composites of ratio 1:1. It can be seen the wear loss decreases with the increasing of the SiO<sub>2</sub>-TiO<sub>2</sub> contents. It is lower more than 90% than that of pure epoxy when TiO<sub>2</sub> content is 4%, SiO<sub>2</sub> content is 2%, and SiO<sub>2</sub>-TiO<sub>2</sub> content is 4%.



This phenomenon indicates that the improvement of wear resistance of epoxy polymers are able when increase the interfacial interaction between nanoparticle and matrix.

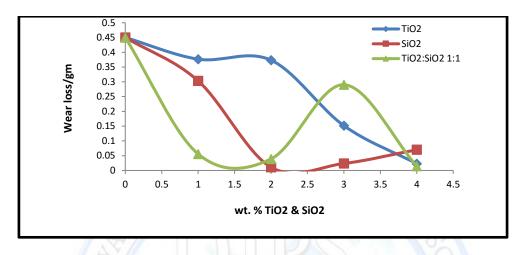


Fig. 3 The relation between wear loss and TiO<sub>2</sub>, SiO<sub>2</sub> contents

Fig. 4 shows the relationship between the number of cycles and the  $TiO_2$  and  $SiO_2$  content respectively and show the EP/ $TiO_2$ -SiO<sub>2</sub> composites of ratio 1:1. It can be seen the number of cycles increases with the increasing of the  $TiO_2$  contents with maximum value at 4%, while at the EP/SiO<sub>2</sub> composites of show the maximum value at 2% its upper than  $TiO_2$ . It can be seen the number of cycles decreases with the increasing of the  $SiO_2$ -TiO<sub>2</sub> contents show the EP/ $TiO_2$ -SiO<sub>2</sub> composites of ratio 1:1 lower than the case of SiO<sub>2</sub>. This phenomenon indicates that the SiO<sub>2</sub> filler improvement of fatigue resistance of epoxy polymers is able when the content is 2%.





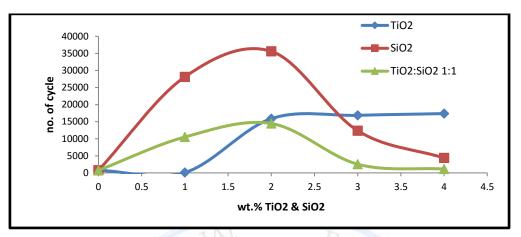


Fig. 4 The relation between No. of fatigue cycles and TiO<sub>2</sub>,SiO<sub>2</sub> contents

Fig.5 show Atomic Force Microscopy (AFM) observation uniformity and three-dimensional surface profile of epoxy-TiO<sub>2</sub> nanospheres in the nanocomposite . Fig.6 show Atomic Force Microscopy (AFM) observation uniformity and three-dimensional surface profile of epoxy-SiO<sub>2</sub> nanospheres in the nanocomposite .

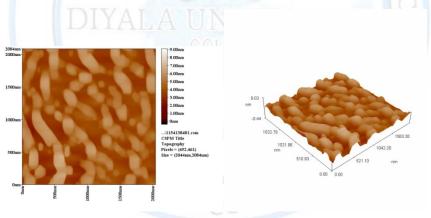
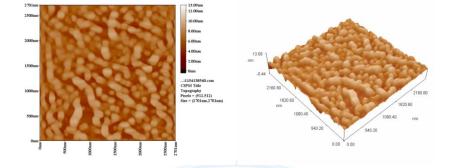


Fig. 5: AFM micrograph showed uniformity and a three-dimensional surface profile of TiO<sub>2</sub> nanospheres in the epoxy nanocomposite.



# Fig. 6: AFM micrograph showed uniformity and a three-dimensional surface profile of SiO<sub>2</sub> nanospheres in the epoxy nanocomposite.

## **Conclusion**

1-The impact resistance of epoxy had increased due to the addition of the nano filler of  $TiO_2$  nano partical.

2-The higher impact resistance and wearing resistance of epoxy hybrid nano Composites at concentration 2% of ratio 1:1 TiO<sub>2</sub>:SiO<sub>2</sub> hybrid.

3-The above experimental results indicate that thisTiO<sub>2</sub>:SiO<sub>2</sub> with ratio 1:1 a good filler material for polymer hybrid nano composite materials.

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