

Effect of Al₂O₃ and SiO₂ Nanoparticle on Wear, Hardness and Impact behavior of Epoxy composites

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Abstract

A growing need for materials with good tribological and mechanical properties led to prepare, characterize and measure wear, hardness and impact properties of epoxy (EP) with different weight percentage of SiO₂ and Al₂O₃ nano particles (0.5%, 1%, 1.5%, 2%, 2.5%, 3%). Silica and alumina nanoparticles have average diameter size 16.69 and 46.73 nm respectively measured by scanning probe microscope. A combination of two different techniques (high shear mixing followed by ultrasonication) was used to prepare the nanocomposites. Fourier transform infrared (FTIR) was used to characterize the nanoparticles (Al₂O₃ and SiO₂). Wear rate, hardness and impact energy properties were measured for EP/ Al₂O₃, EP/ SiO₂ nanocomposites. Decreasing in wear values with addition of nanoparticles (especially nano Al₂O₃) were observed. Approximately 30% and 80% increasing in hardness values and impact energy were observed. An enhancement in whole measurement for all composites was observed, yet (EP/ Al₂O₃, EP/ SiO₂) composites with 2.5% nano Al₂O₃ show in general better hardness and impact results.

Keywords: Nanoparticles, Nanocomposites, wear, Impact, hardness.

Introduction

Polymeric composites have become main part of industrial applications due to their excellent thermal, mechanical and tribological properties apart from their easy process ability and low cost. Such materials are good candidates to form a special class of engineering tribo- materials due to their unique properties such as wear resistance, self lubricant, impact resistance, corrosion resistance and ease of fabrication. They are utilized in numerous applications including seals, cutting tools, bearings and artificial prosthetic joints. For such applications and others, polymer matrix must withstand high mechanical and tribological loads, so fillers, fibers or particles are added to enhance its properties. Wear is defined as damage to a solid surface, generally involving progressive loss of material, due to relative motion between that surface and contacting substance or substances. The five main types of wear are abrasive, adhesive, fretting, erosion and fatigue wear, which are commonly observed in practical situations. The inherent deficiency of polymers could be altered successfully by using various special fillers (micro to nano-sized particles) [1,2].

Polymer nanocomposites, have been widely employed to replace the traditional metals and ceramics in microelectronic packaging, coatings, aerospace, automotive, food packaging and biomedical applications. This is mainly because of their adequate strength, lightness, versatility, ease of processing and low cost. By addition of lubricating and/or reinforcing fillers, the tribological properties of polymers are generally improved. Nanoparticles reinforced polymer composites have attracted more and more attentions. The extremely high specific surface area facilitates creating a great amount of interphase in composite and a strong interaction between the fillers and the matrix. Thus, the nanocomposites always have unique properties resulting from the nano-scale structures. Some nano-inorganic particles are demonstrated to have the ability in reducing the friction and improving the wear resistance of polymers. The addition of nanoparticles, such as SiO₂, Al₂O₃, Si₃N₄ and TiO₂, in epoxy is also for purposes of processing ability and mechanical properties [3, 4]. Fillers can improve tribological properties of polymers. One of the fillers that is used to reinforce polymers is SiO₂. Because of its rigidity and high stability, nano sized silica powders have widely been used to improve properties of polymer matrix composite materials [5].

The present paper focuses on the wear and mechanical (impact energy and hardness) behavior of the Epoxy nano composites with different wt% of nano-sized SiO₂ and Al₂O₃ particles.

Experimental Part

Materials

High pure grades of nano Al₂O₃ [46.73nm] and nano SiO₂ [13.69nm] are supplied from Sigma Aldrich. Scanning probe microscope (Angstrom –Advanced Ltd, USA) was used to measure the nano average diameter as shown in Figures (1) and (2).

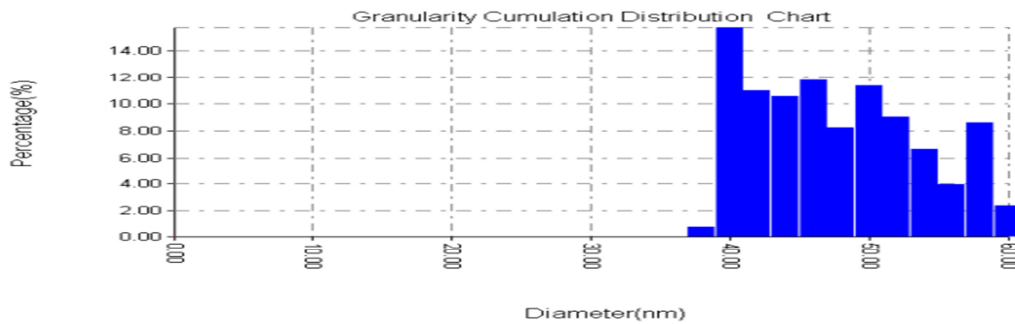


Fig (1): Scanning Probe Microscope of nano Al_2O_3 .

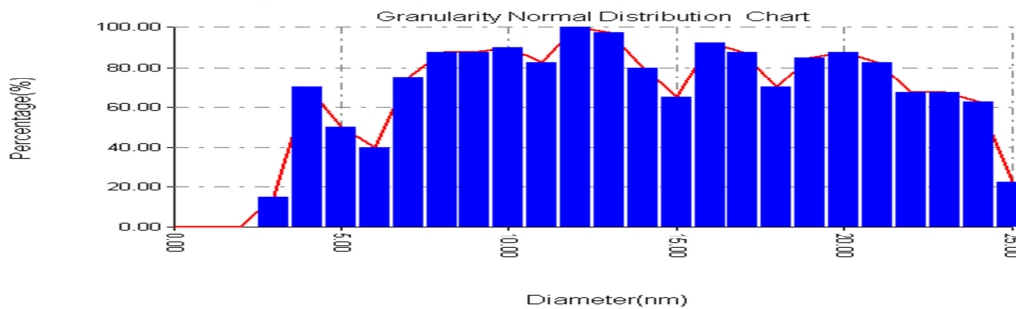


Fig (2): Scanning Probe Microscope of nano SiO_2 .

Epoxy resin matrix used that was Nitofill, EPLV from Fosroc Company with Nitofill EPLV hardener.

Preparation method

Two different steps were used to prepare EP/nanocomposites with different weight percentage of nano particles (Al_2O_3 , SiO_2). The first step includes high shear mixing for fixed time followed by ultra sonication for one hour.

Epoxy resin and hardener were taken into different two beakers and both degassed to remove trapped air bubbles and moisture. The nanoparticle was weighed by Sartorius BL 210S ($d = 0.1$ mg) and manually mixed with epoxy resin under a glove box in a nitrogen atmosphere to avoid interaction of silica nano particles with any unwanted particles from the environment, especially interaction with water vapor because this type of interaction increases particle agglomeration and decreases any interaction (chemical or physical) of particles with the polymer chain in the matrix. Then the nano silica and epoxy resin were mixed by a shearing mixer at 800 rpm for 15 minutes to have a good distribution. The second step was using an ultrasonic homogenizer, Soniprep-150 MSE 150 watt, for 4 minutes to get a good dispersion, and then let the sample container under vacuum to remove bubbles. The hardener was mixed with nano silica and alumina/epoxy resin for 4 minutes by an ultrasonic homogenizer, using ultrasonics may cause a decrease in viscosity and an increase in epoxy resin temperature, then the sample container should be put in a cold water container to avoid high temperature which decreases the time of gelling, making the composite hard to mold. The third step was using a vacuum system to remove the bubbles before casting the composites in the earlier prepared mold identically to ASTM [6].

Samples (EP, Six EP/ Al_2O_3 , and Six EP/ SiO_2 nanocomposites) with different low weight percentages are classified in Table (1).

Table (1) EP and EP/nanocomposites

<i>Samples</i>	<i>Symbols</i>
<i>100%EP</i>	<i>EP</i>
<i>99.5EP/0.5 n Al₂O₃</i>	<i>A 0.5</i>
<i>99EP/1 n Al₂O₃,</i>	<i>A 1.0</i>
<i>98.5EP/1.5 n Al₂O₃,</i>	<i>A 1.5</i>
<i>98 EP/2 n Al₂O₃,</i>	<i>A 2.0</i>
<i>97.5EP/2.5 n Al₂O₃,</i>	<i>A 2.5</i>
<i>97EP/ 3n Al₂O₃,</i>	<i>A 3.0</i>
<i>99.5EP/0.5 n SiO₂</i>	<i>S 0.5</i>
<i>99EP/1 n SiO₂</i>	<i>S 1.0</i>
<i>98.5EP/1.5 n SiO₂</i>	<i>S 1.5</i>
<i>98 EP/2 n SiO₂</i>	<i>S 2.0</i>
<i>97.5EP/2.5 n SiO₂</i>	<i>S 2.5</i>
<i>97EP/ 3n SiO₂</i>	<i>S 3.0</i>

Experimental techniques

1-Wear Test

Wear test was carried out in a pin-on-disc type friction and wear monitoring test rig (supplied by DUCOM) as per ASTM G 99. The counter body is a disc made of hardened ground steel hardness (269HB) with speed 500 rpm and track radius 6 cm. The specimen is held stationary and the disc is rotated while a normal load is applied through a lever mechanism. Series of test are conducted with sliding velocity of 12 cm/sec under normal loading of 20N. The material loss from the composite surface is measured (ΔW) and the wear rate is given as

$$\text{WR} = \Delta W / S \cdot D \dots\dots\dots(1)$$

WR: Wear rate (Kg/cm) , $\Delta W = W1-W2$: Weight difference(g)

And the sliding distance $S \cdot D = 2 \pi r n t \dots\dots\dots(2)$

Where:

t: is the test time (sec) r: radius of center rotating n: number of cycle (500 rpm)

2- Impact Test

Charpy impact test machine was used to measure the absorbed energy of the samples and all tests were carried out at room temperature. The standard charpy specimens with , 10 mm× 10mm × 55mm were prepared .

3- Hardness test

Shore D hardness was used to measure the surface hardness , the indenter was attached to a digital scale that is graduated from 0 to 100 unit the usual method was to press down firmly and quickly on the indenter and recording the maximum reading as the shore D hardness measurement were taken directly from the digital scale reading. Hand –operated durometer was used to measure the surface hardness (shore D) of epoxy pure and composites (epoxy with SiO₂ and epoxy with Al₂O₃). Shore hardness of the samples were measured as per ASTM D2240. Hardness results are average of four measurements taken from different parts of the samples.

Results and Discussion

1-Wear test

The results of wear tests are shown in Table (2) . Wear rate results decrease five time nano Al₂O₃ weight percentage to 3% . This is because of presence of ceramic powders of SiO₂ which have high hardness [3, 6]. This improvement in wear rates is more obvious for 3%SiO₂ comparing to pure Epoxy, which emphasis the role of SiO₂ in improvement of wear rate.

neat epoxy and Al₂O₃ and/ SiO₂ filled epoxy nanocomposites are shown in Table (2) it was observed that the wear rate values decreased with increasing filler loading for all the samples. Figure (3) shows wear rate as a function weight percentage. It may be noted that the resistance to wear is larger in Al₂O₃ filled epoxy compared to the SiO₂filled. The SiO₂ being much harder than the matrix epoxy .This is probably due to the fact that epoxy can easily remove at sliding surfaces (contact area) but in the composite case the ceramic nano particles act as a rough surface relative to the counter face against which they slide. Thus, the improvement in the tribological behavior of nanometer- filled epoxy composite is related to the improved characteristics of the transfer film. The results agree well with the reported by Kishore and Kumar about increasing wear rate by addition Al₂O₃ [7].

Table (2) Wear rate of EP and its nanocomposites

<i>Samples</i>	<i>K(g/cm)</i>
<i>EP</i>	$1.1*10^{-7}$
<i>A 0.5</i>	$2.4*10^{-8}$
<i>A 1.0</i>	$2.1*10^{-8}$
<i>A 1.5</i>	$1.6*10^{-8}$
<i>A 2.0</i>	$1.1*10^{-8}$
<i>A 2.5</i>	$2.0*10^{-8}$
<i>A 3.0</i>	$2.1*10^{-8}$
<i>S 0.5</i>	$4*10^{-8}$
<i>S 1.0</i>	$3.1*10^{-8}$
<i>S 1.5</i>	$2.7*10^{-8}$
<i>S 2.0</i>	$2.1*10^{-8}$
<i>S 2.5</i>	$2.0*10^{-8}$
<i>S 3.0</i>	$2.6*10^{-8}$

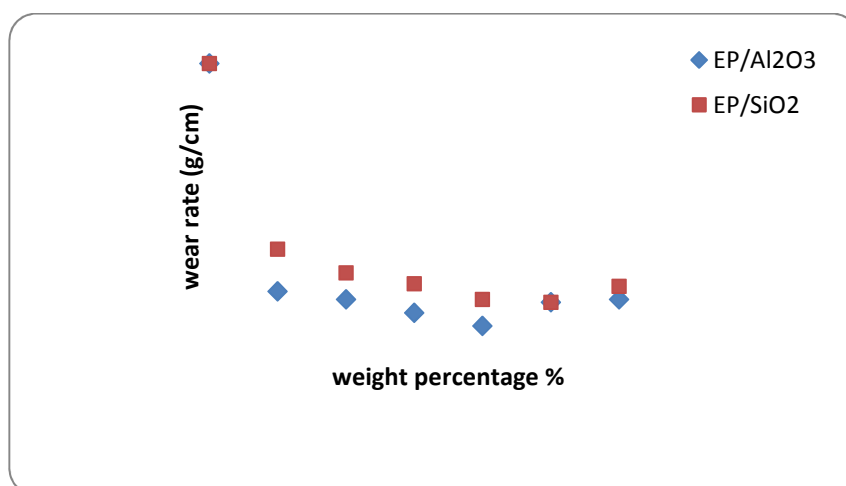


Fig. (3) Wear rate as a function weight percentage of nanoparticles

2-Charpy Impact test

Charpy impact energy values of different composites recorded during the impact tests are given in Table (3). It shows that the resistance to impact loading of epoxy composites improves with addition of nano Al_2O_3 compared to the unfilled and EP/SiO₂ nanocomposites. An increasing in Gc could be due to inducing plastic deformation around epoxy matrix caused by nanoparticles [8]

In general it is noted that the impact energy is directly proportional to the weight content of the reinforcement material as shown in Table (3). In fact, there is almost a constant rate of increase in impact energy with the increase of reinforcement material content. It is also noted that the material toughness of alumina composites are significantly higher than those of silica composites with equivalent reinforcement content. This is justified by the reports that the physicochemical interaction between the particle and the matrix plays a significant role in the obtained composites. Moreover the strong chemical bonding improves the mechanical properties of the composites such high increase in Gc values (88%-80%) could be due to generation of more interfacial surface due to nanoparticles with high module lead to absorb higher mechanical stresses. Nanoparticles act as strong stress concentrators which resist crack propagation [9]

Table (3) Impact energy of EP and its nanocomposites

Samples	Gc(kJ/m ²)
EP	9.07
A 0.5	12.1
A 1.0	13.2
A 1.5	14.5
A 2.0	14.9
A 2.5	15.6
A 3.0	17.4
S 0.5	11.2
S 1.0	11.8
S 1.5	12.5
S 2.0	13.6
S 2.5	14.8
S 3.0	16.4

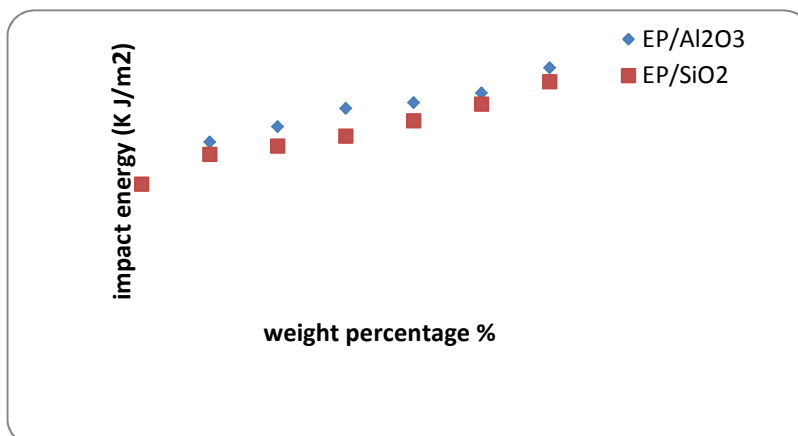


Fig.(4) impact energy as a function of weight percentage of nanoparticles

3- Hardness test:

Table (4) lists the experimental measurements of Shore-D hardness of neat epoxy and different wt% of Al₂O₃ and SiO₂ epoxy nanocomposites. From the table, it can be observed that the incorporation of nanofillers into epoxy showed significant improvement in the hardness of composites. It can be observed that the hardness of composite improves more than (70 %) by addition of 3% Al₂O₃ nano powders. The hardness improves gradually by addition of more powders. This could be due to bond strength achieved between Al₂O₃ and Epoxy. Therefore, this development in hardness can support the improvement of wear resistance that was achieved in this investigation. Al₂O₃ has a much greater surface hardness because of its ceramic nature. Therefore, the contribution of Al₂O₃ into epoxy increased the hardness. Figure (5) the hardness values increase with increasing weight percentage of nanofiller (Al₂O₃ and SiO₂) and hence the nanoparticles might impose better resistance against epoxy segment motion under indentation [5]. Figure (5) shows an enhancement of increase in hardness values represent the ability of materials to stand firm to the local surface deformation [7, 8, 9].

SiO₂ have high hardness and by increasing wt% of SiO₂ nano particles hardness values increase by about 60% and EP/SiO₂ nano composites show higher hardness than EP/Al₂O₃ comparable hardness values were reported by Nikje et al [10] for nanosilica reinforced epoxy were nano SiO₂ was treated by coupling agent.

Table (4) Hardness of EP and its nanocomposites

Samples	Hardness
EP	51
A 0.5	64
A 1.0	69
A 1.5	72
A 2.0	78
A 2.5	83
A 3.0	85
S 0.5	61
S 1.0	66
S 1.5	70
S 2.0	77
S 2.5	78
S 3.0	81

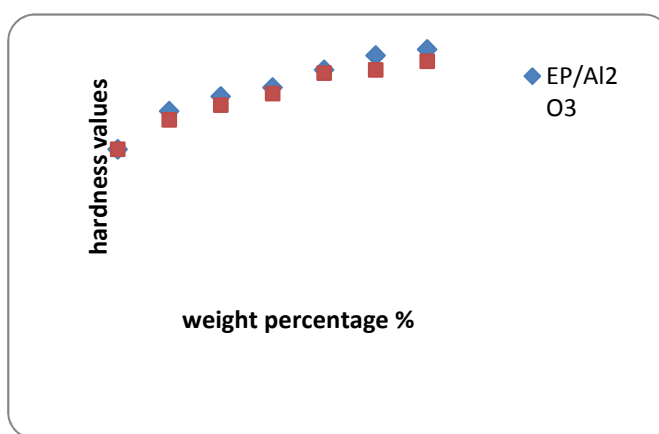


Fig.(5) Hardness values as a function of weight percentage of nanoparticles

Conclusions

Addition of low weight percentage of Al_2O_3 , and SiO_2 nano particles to EP decrease wear rate and increase impact energy and hardness of EP/ SiO_2 and EP/ Al_2O_3 nanocomposites .

EP/ Al_2O_3 nanocomposites in general show highest wear resistance, impact, and hardness values especially for composites with 3% wt percentage of Al_2O_3 nanoparticles.

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