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The Effect of Addition Graphite Filler on Mechanical Properties of Epoxy Material

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Abstract

The current study focuses on the effect of addition graphite filler on mechanical properties of epoxy material. The tensile and bending samples preparation of pure epoxy samples and epoxy with graphite powder with different weight ratio (0.5%, 1%, 1.5%, and 2%wt) using ultrasonic dispersion method. The results showed that the tensile strength and bending resistance were improved at (0.5%, 1%, 1.5%wt) and decreased at (2% wt).The decrease in tensile strength and bending resistance may result from using less sonication time or low ultrasonic energy. The impact test results were also improved with graphite addition and decreased at ratio (2% wt). The decrease in impact test due mechanism of graphite clusters to act as stress concentration sites to produce gaps that absorb some deformation work and prevent development of fissures in the resin matrix. There is an increase in hardness values until 1,5% wt of graphite. In addition of, the distribution of graphite can also affect this result where the penetrant was just on the surface.

Keywords: Epoxy, graphite;bending test; impact, hardness; tensile strength; SEM morphology

1. Introduction

Since the past two decades, research into composites has led to the development properties of materials not only the parent materials but also of classic micro composites [1, 2]**.** Composite materials are hybrid materials where various fillers/ reinforcements with at least one dimension in the micro-nanometer scale incorporated with different matrices [3, 4]. The dispersion of the various filler within the polymer matrices leads to large interfacial contacts between the organic-inorganic phases, which generated an interfacial material that has a quite different morphology, and also has properties that are excellent to those of the bulk polymer phase[5, 6]. As a result, considerable improvements in the properties of composites may recognize even at minimum level of filler loadings [7-9].

The chemical structure of epoxy resins are basically based on chemical groups e.g. epoxides. The simplest formulation of epoxide is ethylene oxide (C_2H_3O). Epichlorohydrin (C_3H_5OCl) is a much more widely used epoxide for producing epoxy resins [10] . Additives of particles with a small amount (<5 wt%) to a matrix can probably increase the strength, dimensional stability, and resistance to thermal degradation without increasing of the weight or processability of the composite. This idea of infusion of particles into a polymer by low volume or weight fraction forms the basis of structural composites synthesis [11].The key point in fabrication of composite processes is the polymer-particle interface. The symmetric dispersion of filler particles produces larger interfacial area per unit volume between the surface of an element and a matrix polymer[12]. Because of its low shrinkage during cure, good cross-

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linking, low viscosity, high strength, low creep, low cost and good adhesion to many substrates [13].

The injection of nanoparticles into the polymer showed the stability of the resins used in industrial applications. The presence of nanomaterials acts as a filler for the voids generated during the casting process, which leads to improved mechanical properties. According to a number of researches, 1.5% of the nanomaterials are capable of improving mechanical properties by 20-30%, such as the properties of tensile, bending, shear, fatigue and others. In addition, a lot of research uses nanomaterials in weight ratios from 1% to 5% to obtain an appropriate improvement in mechanical properties at the lowest possible cost. Sometimes the increase in filler ratio has an adverse effect on the mechanical properties due to agglomeration in the matrix materials, which leads to the appearance of heterogeneous regions in the crystal structure of the composite material [14, 15] **.**

A study by Hamad A. Al-Turaif which focused on the effect of different size of nanoparticle titanium oxide (TiO_2) on the tensile strength property of epoxy resin by the same weight fraction (1.0,3.0,5.0and10.0%wt.), for three different size (17,50 and 200 nm). The composite was characterized by tensile, flexural test, followed by scanning electron microscopy (SEM) and X-ray photoelectron spectroscopy (XPS) of the fracture surfaces. The best result at each weight fraction, found that an enhancement tensile strength property in the epoxy composite proportional increasing amount nanoparticles at big size, but with small size tensile strength has other route, the best was at 3%wt., and then decreased by the additional amount. Young modulus, improvement proportional when increasing filler TiO_2, as same behaviour of tensile strength, then decreased [16]. A. Mirmohseni and S. Zavareh studied tensile strength of nanocomposite by using three different nanoparticles with an epoxy (ABS, clay,TiO_2), also used different weight fraction for each types of nanoparticles. The result indicated that the tensile strengths of nanocomposite increased by 64% for hybrid(clay+ABS+TiO_2) compared pure epoxy, also effect addition of three type modifiers was observed with,(ABS) has good effect on tensile strength, a dispersion of (ABS,TiO_2) in epoxy resin was improved morphological of the nanocomposite [17].Aidah Jumahat et al. used the spherical shape silica (SiO_2) nanoparticles, have a mean particle size of 20 nm into epoxy with weight fraction of silica content (5, 13 and 25)%wt. used static uniaxial tensile test to investigate the tensile stress–strain response and tensile properties of unmodified and nanomodified epoxy polymers, concluded that the best enhancement tensile strength increased proportional with increasing SiO_2 about 24.37%, compared to the pure polymer. An improvement in young modulus was observed when increasing percentages to obtain better at 25%wt. by 38.18% compare with pure epoxy[18]. M. Conradi et al. studied tensile strength for two sizes of spherical silica nanoparticles (SiO_2), (30 and 130) nm with an epoxy at a fixed volume fraction 0.5%vol. Initially there are pre-treated of the silica fillers with diglycidyl ether of bisphenol A (BADGE) in order to prevent agglomeration. Using a three point bending to study the mechanical properties of the composite and they got that the tensile strength increases about 30% for the 130 nm, while increasing tensile strength about 60% for the 30 nm when the silica/epoxy composites were compared to the pure epoxy. The increasing tensile strength is proportion inversely with increasing size of silica. The modulus was seen to enhance for 130nm SiO_2 by 15.38% compare pure epoxy, therefore, the increasing Young modulus is proportion inversely with increasing size of silica [19]. Halil Burak Kaybal et al improved tensile strength property of epoxy by using silica particles by two step, first step, mechanical mixer and second step ultrasonic to disperse the SiO_2 nanoparticles into epoxy resin with five different weight percentage (0-5)%wt. The best tensile strength was obtained by 45% in 3%wt.of SiO_2. In these cases, if SiO_2NPs were added with weight fraction more than 3%wt., no more enhanced was investigated due to aggregations [20]. M.S. Goyat et al studied tensile strength of epoxy reinforced by (TiO_2) nanocomposites (diameter ~48 nm) with weight fraction was(0.5-20) % to an epoxy resin by optimized ultrasonic assisted dispersion TiO_2 nanoparticles(0.5-20)%wt. into epoxy resin. The result indicated best increasing tensile strength at 10%wt. by 27% compare with epoxy, then decreased when increasing amount of add particles to resin [21] . Dain N. Contantinescu et al. studied the influence of silica nano-powders on

tensile strength property of epoxy nanocomposites, the two kinds of SiO_2NPs were used; one has particle size about (8-22) nm and, but the other has particle size about (220-550) nm, by two ways were prepared samples, first way, keeping the mixture under a vacuum of 30mbar for 2 hours at room temperature for degassing. The results show that the ultimate strength pure epoxy is 71.37MPa when the weight percentage were (0.3, 1.0 and 3.0) %, the ultimate strength were (83.02, 85.74 and 85.07) MPa respectively. Second way, same procedure of first way one, while add hardener after vacuum, then mixed by for about 2 minutes, they got result ultimate strength pure epoxy is 90 MPa. When the weight percentage were (0.1, 0.3 and 0.5) %, the ultimate strength were (82.86, 77.38 and 57.95) MPa respectively[22].

The aim of this research is to study some mechanical properties of epoxy reinforced with graphite filler through ultrasonic dispersion method (Probe tip). The percentage of graphite addition was 0.5%, 1%, 1.5% and 2%wt to epoxy to find out the various mechanical properties, such as tensile test, bending test, impact test and hardness test as well as SEM morphology.

2. Experimental Work

2.1 Materials Used

Epoxy type in this study was Sikodor-52 supplied from Sika Egypt (Egypt). The Sikodor-52 is two component, low viscosity liquid, based on high strength epoxy resin and their advantage such as solvent free, suitable in both, dry and damp conditions, shrinkage-free hardening, high mechanical and adhesive strength, and very low viscosity. In addition, the filler material was synthetic graphite (As-received flakes) was purchased from Sigma-Aldrich Company in the form of powder of particle size < 20 μm and density of 1.9 g/cm^3. All the chemicals and solvents were used without any further purification. The Table 1 shows the properties of an epoxy resin.

Table 1

Properties of epoxy resin

Density at 20 ˚C	Comp.(A+B) 1.1Kg/m^3 (mixed)
Coefficient of thermal expansion	89*10 per ˚C (from -20 ˚C to 60 ˚C)
Mechanical strengths (at 20 ˚C and 65% r.h)	Tensile strengths (25 N/mm^2)
	Flexural strength (50 N/mm^2)
Modulus of elastic	1060 N/mm^2

2.2 Preparation of epoxy/graphite Composites

Before the mixing of graphite with epoxy resin, the graphite powder was firstly dried in a furnace at temperature 50 ˚C. Then, different weights ratio of graphite were added (0. 5, 1, 1.5 and 2 %wt). Thus, in the mixing procedure, the mechanical mixing process was firstly used in the composites preparation. Meanwhile, the ultrasonic machine (probe machine) was also utilized to achieve in this mixing process. The composite epoxy materials are placed in the ultrasonic device for 10 minutes for the purpose of dispersion. The ultrasonic machine was MSK-USP-3N type of 300W ultrasonic processor with a quality sound-proof chamber and stainless steel elevating platform. It is designed for disperse liquid with a small quantity, homogenize liquid phase from co-precipitation and mix multi chemical in one solution more efficiently than any other method. After mixing was finished, the mold was prepared and cleaned after being coated with wax to facilitate samples removal from the mold cavity. Then, left at ambient temperature until the samples are hardened.Then, samples were removed from the rubber mold .Finally; all samples were cleaned with a different foil from silicon carbide to eliminate surface defects. The mold is made of silicon rubber according to the international standard ASTMD -638 with Standard dimensions of mold (260*166*13 mm) [23]. Figure 1shows the sonication probe was used for mixing process.

Fig. 1. sonication probe used for mixing process

3. Characterizations

3.1 Mechanical Test

Universal testing machine type (WDW-LOOE) is one of the important machines to conduct different mechanical tests. This machine used to conduct different mechanical tests such as tensile, compression and bending test. For example, the tensile inspection was carried out on dumb-bell samples according to the ASTM D638-02 using tensile machine type(WDW-LOOE Inc., China) with model WDW-5E microcomputer controlled electronic universal testing machine". The dumb-bell samples with 2mm thickness cut off from the molded layers using a Wallace die cutter. The speed test, that used, was 5 mm/min at 25± 3ºC with 5 specimens were tested for each case.

Bending test was also used for the purpose of detecting bending strength for the samples of epoxy and its composites. This test used here was the mode of three-points bending test. In this test, the samples were placed on two points of focus and from the top there is pointed head representing the fulcrum in the center of the sample and then applied force on the sample. The bending resistance test samples were used in standard dimensions as the sample (124*9*4 mm), according to ASTM D790 [24] .

3.2 Impact Test

In this test the impact type was Charpy impact test. This pendulum is connected to a circular disk inserted with special gradients to calculate the amount of energy spent to break the sample. The sample was placed in the place assigned to the device so that the direction of the pendulum is perpendicular to the sample width. The pendulum is then released to the gravity function to break the sample, where the potential energy of the pendulum becomes a kinetic energy that loses part of it in the sample fraction. The samples were tested at laboratory temperature. The test was conducted according to ASTM 4812 – 05 test method [25].

3.3 Hardness Test

Hardness of the specimens was tested using Hardness-meter Shore A tester" TH200 (Beijing time technology Ltd). The test was performed for at least 5 readings on different places at room temperature for each specimen.

3.4 SEM Test

The morphological features of epoxy/ graphite composites were observed using SEM (inspect S50 SEM, Japan). The cryogenically fractured surface of this nanocomposite was coated with thin film of gold or carbon in order to make them conductive with incident electrons beam thus better SEM image.

4. Results and Discussion

Figure 2 shows the results of tensile samples of pure epoxy with different percentages of graphite. It was observed that an increase in tensile strength of composite samples with graphite at (1.5%wt) reach to 50MPa compared with percent weight fractions (0.5, 1 and 2%wt) of graphite compared to pure epoxy. The best improvement percentage in tensile strength is reach to 65% at (1.5%wt) as shown in the figure 3. This increase in the tensile properties may be due to the fact that the graphite particles acted as filler for the voids in the pure epoxy, so the tensile properties increased in the ratio of 1.5%wt. As for the reason for the decrease in properties by 2% wt, some authors think that there is aggregation of graphite particles that led to the presence of gaps in other areas that have become weak as mentioned in [26].

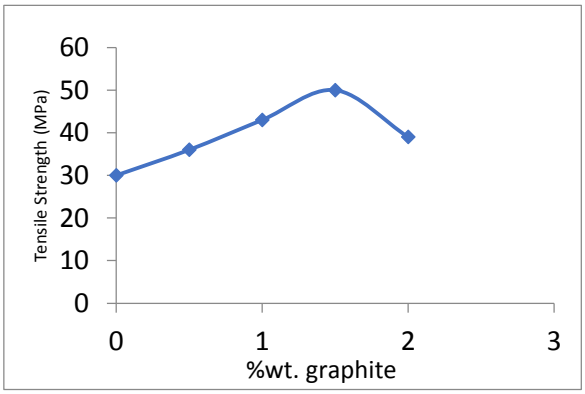

Fig.2. Tensile stress for pure epoxy with different percentages of graphite.

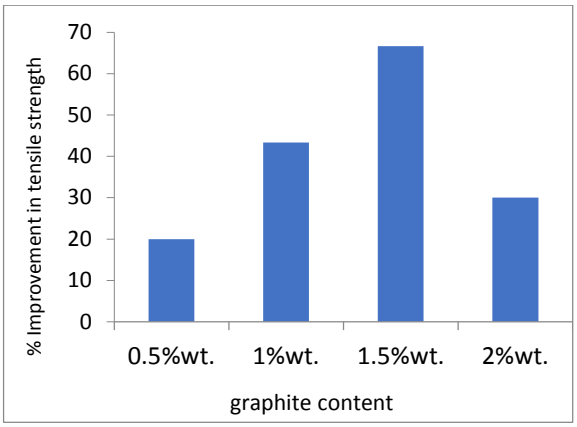

Fig.3. Improvement percentage in tensile stress for epoxy with different percentages of graphite.

When adding a little percentage of graphite, the tensile strength increased, otherwise adding more the percentages of the graphite, the tensile strength decreased. Low ultrasonic energy or time may be insufficient to dispersion graphite, leading to graphite aggregation. This matches with other studies the graphite platelet/epoxy composites higher tensile strength with the addition ratio lower while ratio decrease with the addition graphite platelets high compared to the pure epoxy. The lower strength was seen at higher graphite platelet concentration. The cluster of platelets when the composites are under load, the platelets in the cluster may produce a high stress concentration and cause premature failure [27, 28].

Figure 4 shows the results of bending strength of samples epoxy with different percentages of graphite The bending resistance of the pure epoxy samples was observed to increase with the graphite at loading (1.5%wt) then reduce at loading (0.5, 1, and 2% wt). The best improvement percentage was occurred in 1.5%wt which reach to 57% as shown in Figure 5. This decrease in the bending strength due to crack formation took place in the test samples, which leads us to conclude that the samples are not flexible. Adding of particle resulted in a decrease in the bending strength of the samples. This was due to the poor dispersion of the particles in the matrix. This matches with other studies effect added of nanocarbon black particle [29].

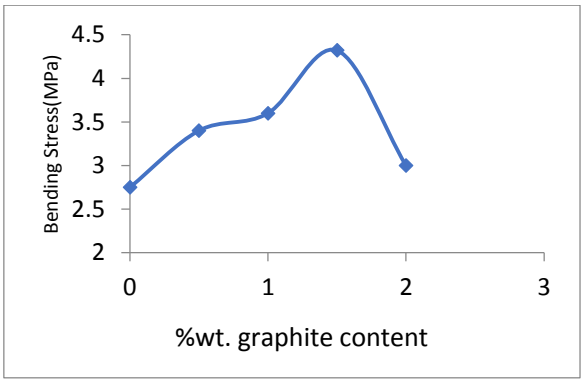

Fig.4. bending strength for pure epoxy with different percentages of graphite.

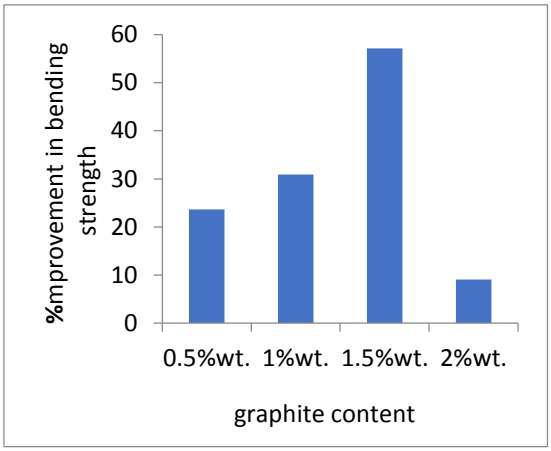

Fig.5. Improvement percentage in bending stress for epoxy with different percentages of graphite.

Figure 6 Indicates the results of impact test, The results of the impact strength test for composite materials showed higher impact strength at (1.5%wt) than other concentrations. The observation of showed that the value of the impact strength decrease after the ratio of 1.5%wt. The best improvement percentage was fall in 1.5%wt which reach to 85% as shown in Figure 7.

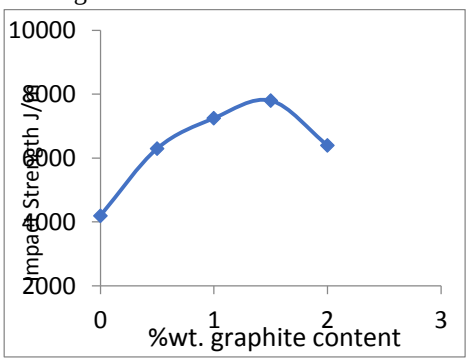

Fig. 6. Impact test for pure epoxy with different percentages of graphite.

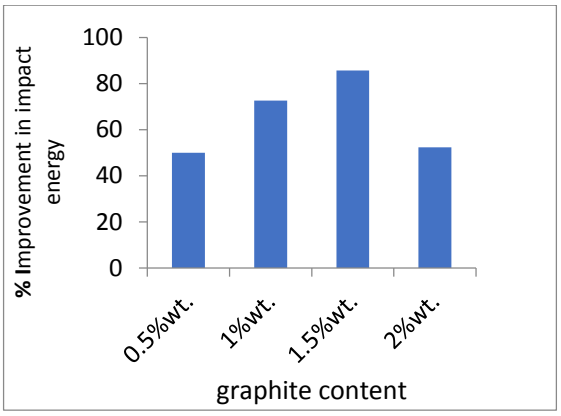

Fig. 7. Improvement percentage in impact for epoxy with different percentages of graphite.

Figure 8 exhibited the results of hardness test. It was conducted for epoxy with different percentages of graphite samples. There is little increase in hardness values with increasing the percentage of graphite up to 1.5%wt. with the best improvement in hardness was closet to 1.5% as shown in the Figure 9.

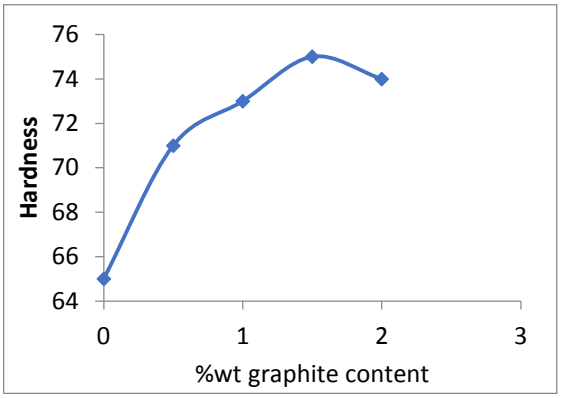

Fig.8. Hardness of epoxy with different percentage graphite.

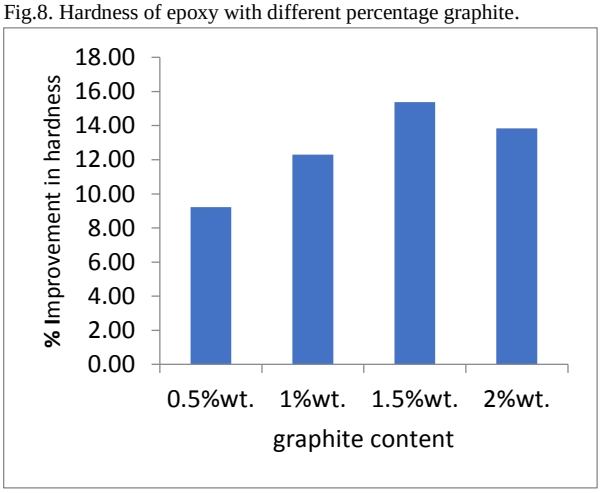

Fig. 9. Improvement percentage in hardness for epoxy with different percentages of graphite.

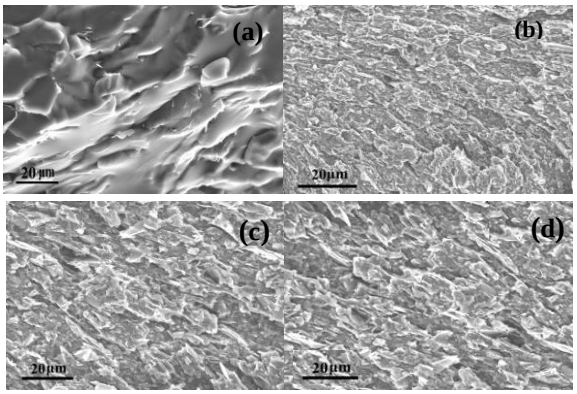

Fig. 10. SEM images of epoxy composites reinforced by graphite particle at loading (a) 0.5%wt, (b) 1%wt, (c) 1.5%wt and (d) 2%wt.

Figure 10 shows the SEM images of epoxy filled with graphite particles. As can be seen, the micro graphite flakes distributed within epoxy matrix, owing to the strong interaction between the epoxy chains and graphite surface[30]. Some voids in 0.5% and 1% wt. of graphite were noticed. To evaluate the status of dispersion and distribution of graphite within epoxy, SEM observation was carried out. However, as the loading of graphite increased to 2%wt, several aggregations occurred due to insufficient mixing process to achieve the higher dispersion [1, 31]. This observation was also suggested by previous literature by the author R. Baptista who fund that increasing graphite tended to form small agglomerates as well as several pores presenting in the epoxy matrices [22].

5. Conclusions

The current study has concluded that:

1-Increased tensile strength of epoxy samples with increase in graphite filler at (0.5%, 1%wt , 1.5%wt and 2%wt) compared with neat epoxy. The best improvement in tensile reach to 67% was carried out in 1.5%.wt.

2-Increased bending strength of epoxy samples reinforcement by (0.5%, 1%wt , 1.5%wt and 2%wt) compared with neat epoxy. The best improvement in bending reaching to 57% was occurred in 1.5%.wt.

3-Impact strength test for composite materials showed higher impact strength at (1.5% wt) than other concentrations. The best improvement in bending reaching to 85% was occurred in 1.5%.wt.

4-Hardness test results increased up to 1.5%wt. then reduced with the increase of graphite ratio at 2%. The best improvement in bending reaching to 15% was occurred in 1.5%.wt.

Conflicts of interest

Not applicable

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