Scanning Electron Microscopy Study for Fracture Surface of Epoxy/Al_2O_3 Nanocomposites

Faez M. Hassan, Hassan Hadi Darwoysh

Abstract— The effect of Al_2O_3 nanoparticles 50 nm on some mechanical properties of epoxy resin was investigated (Young's modulus and Flexural strength). Moreover, this study shows the effect of using scanning electron microscopy on fracture surface. The nanocomposites were prepared by using three processing steps with different fraction volume of nanoparticles (1, 2, 3, 4, 5, 7 and 10 %). Flexural strength and Young's modulus of nanocomposites were increased at low fraction volume (Max. enhancement at 3%). However, at higher fraction volume, both Young's modulus and Flexural strength were decreased with increasing of ductility, and the mechanical properties were enhanced more than that of neat epoxy resin. Fracture surface topography shows rougher and lesser uniform surface compared with that neat epoxy resin, more than one crack propagation directions as well as river lines are lesser in length and crowded compared with neat epoxy resin. The current study shows that Al_2O_3 nanoparticles agglomerations are very obvious in the fracture surface.

Index Terms— Epoxy resin, Nanocomposites, Fracture surface topography.

1 INTRODUCTION

TTH epoxy/nanoparticles have many positively characteristics such as mechanical performance, dielectric behaviors and thermal stability properties as well as have many advantages of good corrosion resistance, adhesion to most substrate, good scratch resistance and excellent tribological properties. Several potential applications were leading to wide interest ing in this type of nanocomposites such as using sealants, paints, coating [1], [2], [3], [4]. The use of an additional phase (e.g. inorganic filler) to strengthen the properties of epoxy resin has been a common practice, where the nanoparticles can fill up the weak micro regions of resin to boost the interaction force at epoxy resin-filler interfaces.

Dramatic increases in the interfacial area between fillers and epoxy resin can significantly improve the properties of epoxy resin, so the reinforcement efficiency is strongly depend on particle size, dispersion of nanoparticles and fraction volume of nanoparticles in epoxy resin surface. Several techniques were used to have better dispersion of nanoparticles in epoxy such as sol-gel technique, in-situ technique, shearing mixing and ultrasonic homogenizer [4].

Recent researches [5], [6], [7], [8], suggest that ultrasonic homogenizer is the effective tool for the fabrication of epoxy/nanocomposites, but also every technique has disadvantage in fabrication such as in ultrasonic homogenizer which decreases the gelling time of epoxy resin, while shearing mixing leave the nanocomposites with several big agglomerations. Three steps technique were used to prepare nanocomposites, first shearing mixing gives good distribution without having good dispersion, but lead to decreases the needed time for using ultrasonic homogenizer (which is the second step), the gelling time still with acceptable range (i.e. enough time to molding the composite). Finally appling the third stage by using vacuum system to remove any bubble from the surface of composites [7].

- *Faez M. Hassan, Ph.D. in Physics Science in Al-Mustansiriya University, Baghdad, Iraq. E-mail: faizmohammed2007@gmail.com*
- *Hassan Hadi Darwoysh, Masters in Physics Science in Al-Mustansiriya University, Baghdad, Iraq. E-mail: hassanhadi.66@yahoo.com*

2 MATERIALS AND METHODS

2.1 Materials

Epoxy resin matrix used in this study is Nitofill, EPLV from Fosroc Company with Nitofill EPLV hardener. The mixing ratio 3:1, gelling time 40 minute at 35 C^o, specific gravity is 1.04 g/cm^3 and mixed viscosity is 1.0 poise at 35 C^o. The Al_2O_3 used in this study was produced by MTI with specific surface area 210 ± 25 m^2/g with average particle size < 85 nm while thedensity is 0.25 g/cm^3, the purity of Al_2O_3 ≥ 99.8 exposed for thermal treatment at 100 C^o for 30 minute to ensure discard of H_2O molecule that absorb by Al_2O_3.

2.2 Sample Preparation

The composites were prepared by mixing technique which consists of three steps. Firstly, the nanoparticles were weight and mix with epoxy resin under gloves box in nitrogen atmosphere. Then the Al_2O_3 nanoparticles and epoxy resin were mixed by shearing mixer for 15 minutes to have a good distribution. The second step was using ultrasonic Soniprep-150 MSE in 150 watt for 4 minutes to get good dispersion, and then let the sample container under vacuum to remove the bubbles.

The hardener mixed with Al_2O_3 nanoparticles/epoxy resin for 4 minute by ultrasonic homogenizer using ultrasonic may cause to decrease viscosity and increase epoxy resin temperature, the sample container should be putted in a cold water container to avoid high temperature which decrease time of gelling making the composite hard to mold, the third step was using vacuum system to remove the bubble before cast the composites in earlier prepared mold. The final product shape shows in Fig.1, where; (L) as specimen length, (D) as specimen

depth, (W) as specimen width and (Ls) as support span.

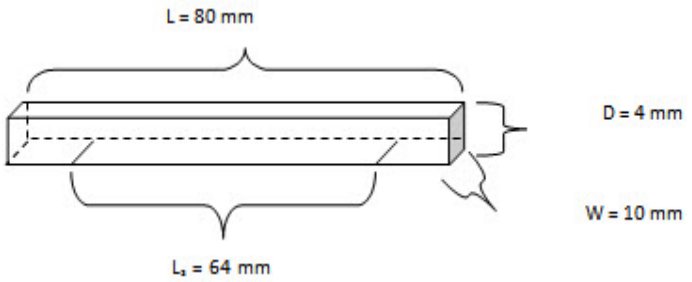


Fig. 1. Final Al_2O_3 nanoparticles specimen shape according to ASTM (D790-1984).

Concentration are expressed by volume fractions for, matrix Vm, and particle Vf, obtained from the volumes of individual components, Øm for matrix, and Øf for particles, the subscripts m, f represent the matrix and the particles components.

$$V_m + V_f = 1 \quad (1)$$
$$V_m = Ø_m / (Ø_m + Ø_f) \quad (2)$$
$$V_f = Ø_f / (Ø_m + Ø_f) \quad (3)$$
$$Ø_f = m_f / p_f \quad Ø_m = m_m / p_m$$

Where m, and ρ, are the mass and density of matrix and particles for the prepared composites respectively.

3 Characterization

All samples which are neat epoxy resin, epoxy Al_2O_3 nanoparticles/resin were subjected to the certain analysis. Three point bending analysis using (Instron 1122) was used to determine mechanical properties; Flexural strength and Young's modulus for nanocomposites. SEM technique using (Hitach 4400) also used to study the morphology of the fracture surface after examines the specimens with three point bending.

4 Results and Dicussion

Table 1, shows that the compositions, Flexural strength, and Young's modulus of nanocomposites (EP/Al_2O_3 nanoparticles) with 1, 2, 3, 4, 5, 7, and 10% as fraction volume for nanocomposites. The following equations were used to determine flexural strength and Young's modulus.

$$\sigma_f = 3PLs / (2Dw^2) \quad (4)$$
$$E_f = Ls^3 S / (4Dw^3) \quad (5)$$

Where (P) is the fracture load, (Ls) is the distance between the two support points, (w) is the width of the specimen, (S) equal to the slope of the tangent of the initial straight-line portion of load-deflection curve and (D) is the depth of the specimen.

From Table 1, Flexural strength of EP/nano Al_2O_3 increase with increased in fraction volume of nanoparticles of fumed Al_2O_3, maximum increment at volume 4%. The fraction of fumed Al_2O_3 behavior in nanocomposites is due to decreasing in space distance between chains crosslink caused by adding nanoparticles which are polar particles, creating van der-waals bonding between chains and particles lead to increase constrained between; particles/polymer chains, and polymer chains itself [9].

TABLE 1
THE COMPOSITIONS, FLEXURAL STRENGTH, AND MODULUS OF EPOXY/AL_2O_3 NANOCOMPOSITES

Sample EP nano A_2O_3	Max.Fraction Force (N)	Max. Deflection (mm)	Flexural Strength (Mpa)	Young Modulus (Gpa)
EP	96	6.2	67.76	1.43
1%	146.7	4.9	86.5	3.16
2%	151.99	5.01	88.9	3.12
3%	189.2	7.45	113.09	3.3
4%	187.3	8.77	112.8	3.2
5%	171.1	20.97	106.7	3.11
7%	154.9	4.89	89.5	3.5
10%	133.6	4.65	72.9	2.91

After volume 4%, the fraction of addition flexural strength begin to decrease, where increasing the addition of filler lead to increasing the constrained between polymer chains, decreasing the length of chains over certain critical length lead to decreasing flexural strength which is depend on chains length [9, 10], but flexural strength still higher than that of neat epoxy resin because of van der-waals bond which is weak bond but with huge numbers [11], see Fig. 2. Also it's obvious from Table 1, the stiffness (Young's modulus) of samples increase with increase of filler addition, this is because of particles agglomeration where it lead to increasing the constrained between polymer chains. This behavior has a good agreement with [5], [9], [11].

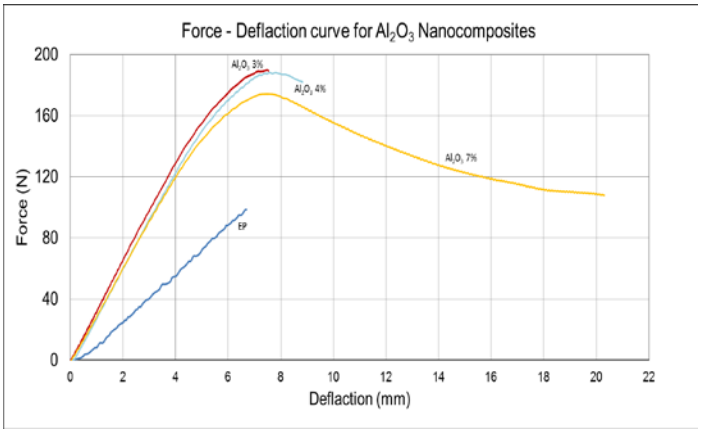


Fig. 2. Force-Deflection curves of pue epoxy and EP/AL_2O_3 nanocomposites.

5 SCANNING ELECRON MICROSCOPY

SEM technique was used to study the topography of the fractured surface after examination of the EP/ Al_2O_3 nanocompo-

sites specimens with three point bending test. SEM images magnified to 150X its original scale.

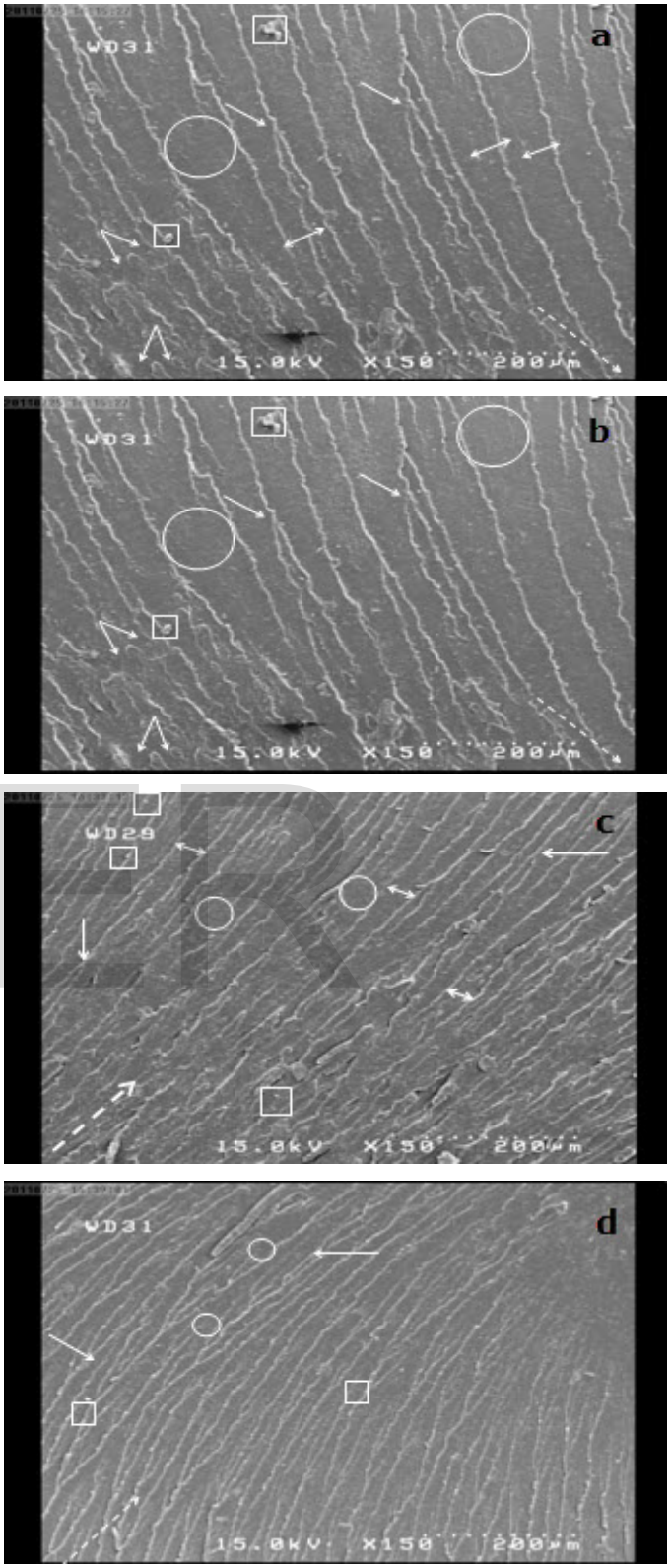
Fig. (3a) shows that SEM image of the topography of fractured surface of EP/Al_2O_3 nanocomposites with volume 1%. Fraction of Al_2O_3 nanoparticles, with following features; (1) more roughness with new pattern of fractured surface different from the epoxy specimen. (2) More rivers lines and crowded together as well as the separate between two river lines. (3) Alumina nanoparticles agglomeration begins to appear in the fractured surface. Small and sharp hyperbolic marks open in the direction of crack propagation.

Fig. (3b) is the SEM image of the topography of fractured surface of EP/ Al_2O_3 nanocomposites with volume 2%. Fraction of Al_2O_3 nanoparticles shows; (1) pattern of fractured surface just like the pattern appeared in fig. (3a) but the separate between two river lines was lesser. (2) Less flat area surface than area surface appeared in fig. (3a). (3) More Alumina nanoparticles agglomeration appeared in the fracture surface. Hyperbolic marks disappeared from the fractured surfaces which indicate that Al_2O_3 nanoparticles change the behaviour of epoxy matrix to fracture under load and new pattern of fractured appeared depending Al_2O_3 nanoparticles type (ship, size and nature).

Fig. (3c and 3d) are the SEM images of the topography of fractured surface of EP/Al_2O_3 nanocomposites with volumes 3% and 4% respectively. Fraction of Al_2O_3 nanoparticles shows that; (1) more roughness and less flat area surface. (2) More than one crack propagation direction appear obviously, more river lines, less long and crowded together compared with figures (3a and 3b), the distance between two river lines become closer, which dispersed stress and increase resistance to crack propagation. (3) Alumina nanoparticles agglomeration appeared in the fracture surface.

Moreover, fig. (3e) is the SEM image of the topography of fractured surface of EP/Al_2O_3 nanocomposites with volume 7%. Fraction of Al_2O_3 nanoparticles show that, (1) more roughly and less smooth area surface. (2) More than one crack propagation direction appeared, more river lines where the distance between two river lines become closer. Shorter river lines are crowded together compared with Fig. (3a and 3b). The behaviour of high speed brittle fracture appeared as big flat area surface appeared signed by (+). (3) Alumina nanoparticles agglomeration appeared in the fractured surface.

Finally, fig. (3f) is the SEM images of the topography of fractured surface of EP/Al_2O_3 nanocomposites with volume 10%. Fraction of Al_2O_3 nanoparticles shows, (1) more roughnes and less smooth area surface. (2) More than one crack propagation direction appear, more river lines, shorter river lines are crowded together compared with figures. (3a to 3e). (3) Small and sharp hyperbolic marks open in many directions begin to appear again indicating that nanoparticles effects begin to disappear and some of epoxy matrix properties begin to appear. (4) Alumina nanoparticles agglomeration appeared in the fracture surface.



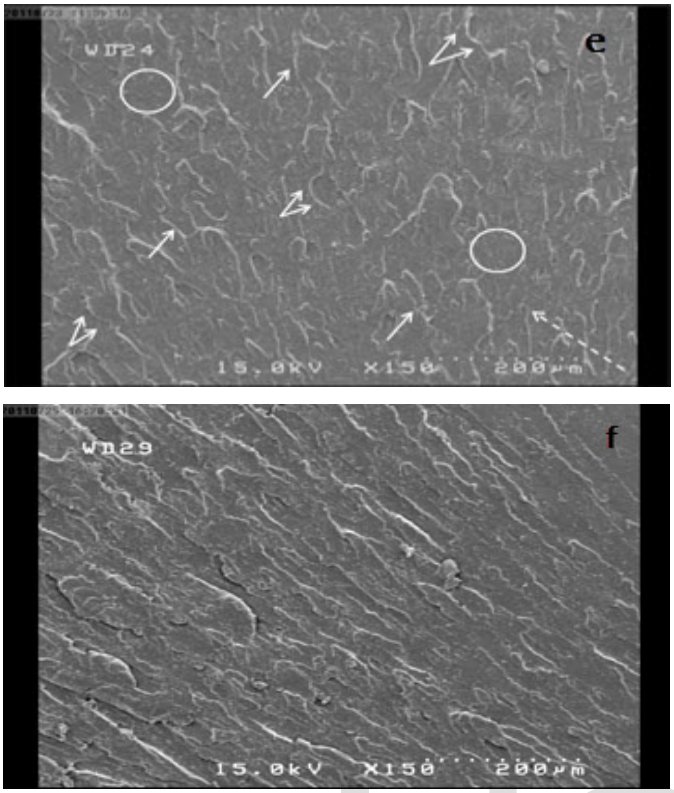


Fig. 3. Topography of fractured surface of EP/AL2O3 nanocomposites (3a. fraction volume 1%; 3b. fraction volume 2%; 3c. fraction volume 3%; 3d. fraction volume 4%; 3e. fraction volume 7% and 3f. fraction volume 10%).

6 CONCLUSION

The flexural strength of EP/nano Al_2O_3 increased with increasing fraction volume for Al_2O_3 nanoparticles, this behavior in nanocomposites is attributed to increasing in complicating chains crosslink caused by adding (because of van derwaals bond which is weak bond but with huge numbers of nanoparticles).

The stiffness (Young's modulus) of samples increased with increasing of filler addition, it's because of nanoparticles restrictions to the chains, decreasing in chains length and increasing in complicating the crosslink between polymer chains. Maximum stiffness appears at maximum flexural strength. Fracture surface topography shows more rough and lesser uniform surface compared with that of neat epoxy resin, more than one crack propagation directions, river lines are lesser long and crowded together compared with neat epoxy resin. Al_2O_3 nanoparticles agglomerations are very obvious in the fracture surface.

REFERENCES

[1] C. Chen, R. Justica, D. Schaefer and J. Baur, "Highly disperednanosilica-epoxy resins with enhanced mechanical properties," Polymer, vol. 49, pp. 3805-3815, 2008.

[2] J. Baller, N. Becker, M. Ziehmer, M. Thomassey, B. Zielinsski, U. Muller and R. Sanctuary, "Interactions between silica nanoparticles and an epoxy resin before and after during network formation", Polymer, vol. 50, pp. 3211-3819, 2009.

[3] R. Zhao, and W. Luo, "Fracture surface analysis on nano-SiO_2/epoxy composite", Materials Science and Engineering, vol. A 487, pp. 313-315, 2008.

[4] Y. Zheng, Y. Zheng, and R. Ning, "effects of nanoparticles SiO_2 on the performace of nanocomposites Materials Letters, vol. 57, pp. 2940-2944, 2003.

[5] A. Chatterjee, and M. S. Islam," Materials Science and Engineering, vol. A487, pp. 574-585, 2008.

[6] B. C. Kim, S. W. Park and D. G. Lee, 'Fracture toughness of the nano-particle reinforced epoxy composite", Composite Surface, vol. 86, 69-77, 2008.

[7] A. Chatterjee and M. S. Islam, "Fabrication and characterization of TiO_2–epoxy nanocomposite," Materials Science and Engineering, vol. A 487, pp. 547-585, 2008.

[8] J. Cho, M. S. Josh, and C. T. Sun, "Effect of inclusion size on mechanical properties of polymeric composites with micro and nano particles," Composites Science and Technology, vol. 66, pp. 1941-1952, 2006.

[9] C. S. Sipaut, N. Ahmed, R. Adnan, I. Ab. Rahman, M. A. Bakar, J. Ismail and C. K. Chee, "Properties and Morphology of Bulk Epoxy Composites Filled with Modified Fumed Silica-Epoxy Nanocomposites," Journal of Applied Sciences, vol. 7(1), pp. 27-34, 2007.

[10] T. H. Hsieh, A. J. Kinloch, K. Masania, A.C. Taylor and S. Sprenger, "The mechanisms and mechanics of the toughening of epoxy polymers modified with silica nanoparticles," Polymer, vol. 51, pp. 6284-6294, 2010.

[11] C. Chen, and A. B. Morgen, "Mild processing and characterization of silica epoxy hybrid nanocomposite," Polymer, vol. 50, pp. 6265-6273, 2009.