

# Study the Mechanical Properties and Fracture Structure by Scanning Electron Microscopy for Epoxy/Silica Nanocomposites

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## Abstract

The effect of nano silica particles (Fused silica) 12nm on some mechanical properties of epoxy resin was investigated ( Young's modulus, Flexural strength). Also study the effect on fracture structure by using scanning Electron Microscopy. The nano-composites were prepared by using three steps process with different volume fraction of nano-particles (1, 2, 3, 4, 5, 7, 10, 15, and 20 vol. %). Flexural strength and Young's modulus of nano composites were increased at low volume fraction (max. enhancement at 4 vol.% ). However at higher volume fraction both Young's modulus and flexural strength decrease. Moreover, above, the mechanical properties are enhanced more than that of neat epoxy resin. Fracture structure 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. Silica nano-particles agglomerations are very obvious in the fracture surface.

## 1. INTRODUCTION

Epoxy/nano fused silica composites have many positively characteristics such as mechanical performance, dielectric behaviors, thermal stability properties, and also have many advantages of good corrosion resistance, adhesion to most substrate, good scratch resistance, and excellent tribological properties. Several potential applications was leading to wide interest in this type of nano-composites such as using in sealants, paints, coating [1-5]. The use of an additional phase (e.g. inorganic filler) to strengthen the properties of epoxy resin has been a common practice, where the nano-particles 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

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reinforcement efficiency is strongly depend on particle size, dispersion of nano-particles and volume fraction of nano-particles in epoxy resin structure. Several techniques were used to have better dispersion of nano-particles in epoxy such as sol-gel technique, in-situ technique, shearing mixing and ultrasonic homogenizer [4]. Recent research [6-11] suggest that ultrasonic homogenizer is the effective tool for the fabrication of epoxy/nano-composites, but also every technique has disadvantage in fabrication such as in ultrasonic homogenizer decreases the gelling time of epoxy resin, while shearing mixing leave the nano-composites with several big agglomerations. Three steps technique was used to prepare nano-composites, first shearing mixing gives good distribution with out having good dispersion, but lead to decreases the needed time for using ultrasonic homogenizer (which is the second step) so the gelling time still with acceptable range (i.e. enough time to molding the composite), apply the third stage of using vacuum system to remove any bubble from the structure of composites [8].

## **2. MATERIALS AND METHODS**

### **2.1 Materials**

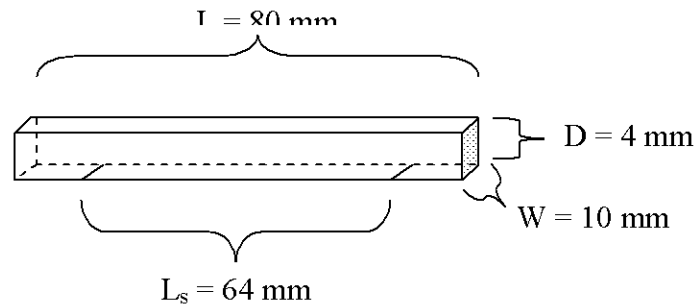
Epoxy resin matrix used that was Nitofill, EPLV from Fosroc Company with Nitofill EPLV hardener. The mixing ratio 3:1, gelling time 40 minute at 35 °C, specific gravity 1.04 g/cm<sup>3</sup> and mixed viscosity 1.0 poise at 35 °C. the used fused silica was Aerosil-200 produced by Aerosil pharma (hydrophilic silica) with specific surface area 200 ± 25 m<sup>2</sup>/g, average particle size 12 nm while density 0.05 g/cm<sup>3</sup>, the purity of fused silica ≥ 99.8, exposed for thermal treatment at 100 °C for 30 minute to ensure discard of H<sub>2</sub>O molecule that absorb by fused silica. Micro silica was Mallinckrodt 2847 produced by Cambridge lab, particle size 100 µm density 2.4g/cm<sup>3</sup> also exposed for thermal treatment at 100 °C for 30 minute.

### **2.2 Sample preparation**

The composites were prepared (with volume fraction prepared according to equations (1-3)) by mixing process which consists of three steps. Firstly, the nano-particle was weight by Sartorius BL 210S (d = 0.1 mg) and manually mix with epoxy resin under gloves box in nitrogen atmosphere to avoid interact of silica nano particles with any unwanted particle from the environment specially interaction with water vapor because this type of interaction increase particles agglomeration and decrease any interaction (chemical or physical) of particles with polymer chain in the matrix. Then the nano silica and epoxy resin were mixed by shearing mixer at 800 rpm for 15 minutes to have good distribution. The second step was using ultrasonic homogenizer, Soniprep-150 MSE 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 nano silica/epoxy resin for 4 minute by ultrasonic homogenizer, using ultrasonic may

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cause to decrease viscosity and increase epoxy resin temperature then 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 identically to ASTM (D790-1984) specification. All the above steps were done for micro composites. The final product shape show in Fig.1 where; (L) as specimen length,(D) as specimen depth, (W) as specimen width and ( $L_s$ ) as support span.



*Fig. 1.* Final nanocomposite specimen shape according to ASTM (D790-1984),

Concentration are expressed by volume fractions for, matrix  $V_m$ , and particle  $V_f$  obtained from the volumes of individual components,  $\phi_m$  for

matrix, and  $\phi_f$  for particles, the subscripts  $m$ ,  $f$  represent the matrix and the

particles components [12].

$$V_m + V_f = 1, \quad (1)$$

$$V_m = \phi_m / (\phi_m + \phi_f), \quad (2)$$

$$V_f = \phi_f / (\phi_m + \phi_f), \quad (3)$$

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$$\phi_f = m_f/\rho_f, \quad \phi_m = m_m/\rho_m,$$

Where  $m$ , and  $\rho$ , are the mass and density of matrix and particles for the prepared composites.

## 2.3 Characterization

All samples; neat epoxy resin, epoxy resin/nano-particles SiO<sub>2</sub>, epoxy resin/micro-particles SiO<sub>2</sub> were subjected to the following analysis; Three point bending analysis using (Instron 1122) was used to determine mechanical properties; Flexural strength and Young's modulus for nano/micro composites. SEM technique using ( Hitach 4400 ) was used to study the morphology of the fracture surface after examine the specimens with three point bending.

## 3. RESULTS AND DISCUSSION

### 3.1 Three point bending analysis for samples

Table 1, shows compositions, Flexural strength, and Young's modulus of nano-composites (EP/12 nm SiO<sub>2</sub> particles) and micro-composites (EP/100 μm SiO<sub>2</sub> particles), with 1, 2, 3, 4, 5, 7, 10, 15, and 20% as volume fraction for both nano and micro composites. The following equations were used to determine Flexural strength  $\sigma_f$ , and Young's modulus [13, 14] .

$$\sigma_f = 3PL_s / (2Dw^2) \tag{4}$$

$$E_f = L_s^3 S / (4Dw^3) \tag{5}$$

Where (P) the fracture load, (L<sub>s</sub>) 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.

Table 1: The compositions, flexural strength, and modulus of nano-composites and micro-composites

Composition	Force (N)	Deflection (mm)	Flexural strength Ep/nano SiO <sub>2</sub> MPa	Young's modulus Ep/nano SiO <sub>2</sub> GPa
EP	98.52	6.7	67.4	1.42
EP/1% SiO <sub>2</sub>	150.1	4.61	85.71	1.47
EP/2% SiO <sub>2</sub>	159.47	4.91	90.46	1.57
EP/3% SiO <sub>2</sub>	155.92	4.82	88.23	1.77
EP/4% SiO <sub>2</sub>	174.37	5.61	96.68	3.04
EP/5% SiO <sub>2</sub>	124.95	7.39	74.9	2.71
EP/7% SiO <sub>2</sub>	132.55	5.62	73.3	2.65
EP/10% SiO <sub>2</sub>	128.65	5.7	70.1	2.66
EP/15% SiO <sub>2</sub>	123.44	5.9	73.13	2.70
EP/20% SiO <sub>2</sub>	128.46	3.75	76.3	2.70

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From Table 1, Flexural strength of EP/nano SiO<sub>2</sub> increase with increased volume fraction of nanoparticles of fumed silica, maximum increment at 4% Vol. fraction of fumed silica, this 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 [15,16].

After 4% Vol. 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 [17], 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 [18] see Fig.2.

Also its 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 Sipaut *et al*, (2007), Chen, *et al* (2009), Chatterjee *et al* (2008) and Hsieh (2010) [19].

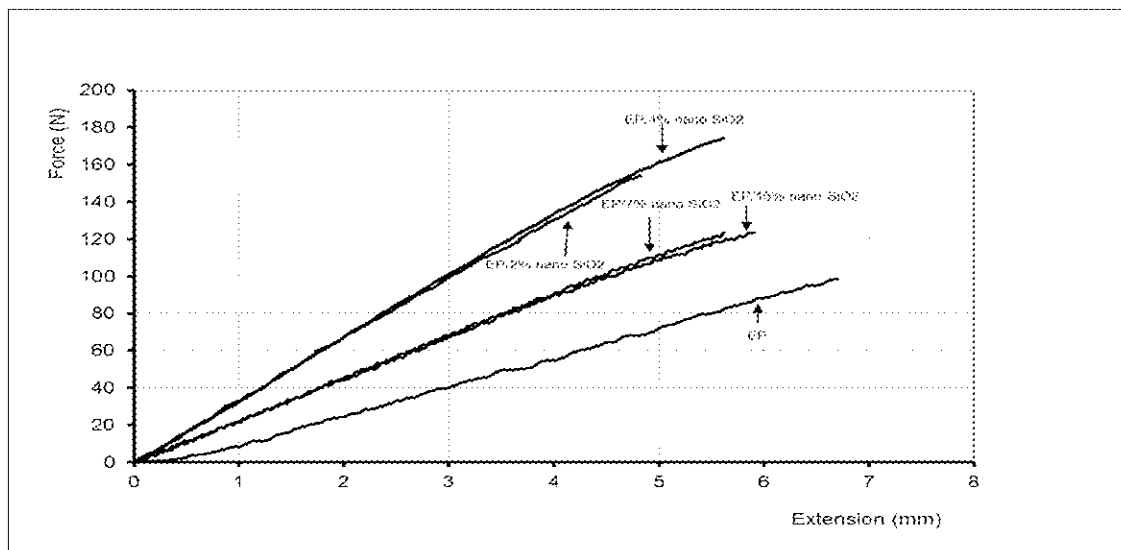


Fig. 2: Flexural strength, and Young's modulus of nano-composites

### 3.2 Scanning Electron Microscopy Image of Silica Nanocomposite Fracture Structure Topography

Figures (3A – 3I) show SEM image of the Fracture structure topography of neat epoxy resin and nanocomposites magnified to 60,000 times its original scale. Figures (4-9A – 4-9I) show structure nanocracks (signs by arrows) created

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because of polymerisation begin at numerous regions and speeds, according to the nature of chemical reaction of resin and hardener, which generated regions that polymerized faster than others applying various stresses on polymer structure creating nanocracks (which is nano-defect generate at early stage of polymerisation). Figure (3A) of (neat epoxy resin) shows SEM image of the Fracture structure topography of neat epoxy resin with nanocracks of average width equal to 22 nm, average length equal to 200 nm and average crack distribution equal 12 per  $\mu\text{m}^2$ . Figures (3B, C) show SEM image of the Fracture structure topography of nanocomposites (1, 2 vol.% fraction) have nanocracks of good agreement mode with figure (3A) but less average width, length and distribution.

Figures (3D, 3E) show SEM image of the Fracture structure topography of nanocomposites with (3, 4 vol.% fraction of  $\text{SiO}_2$  nanoparticles) have different mode of nanocracks comparing with (figure 3A, 3B, 3C). The nanocracks almost vanish from the structure because of silica nanoparticles, which have good adhesion force arising from the polar of  $\text{SiO}_2$  particles. Figures (3F, 3G, 3H, J and I) show SEM image of the Fracture structure topography of nanocomposites with (5, 7, 10, 15, 20 vol.% fraction of  $\text{SiO}_2$  nanoparticles), have a good agreement mode of nanocracks with figures (3A, 3B and 3C). In addition, figures (3F, 3G, 3H, 3J and 3I) show nanoparticles agglomerations (signs by circles) because of high volume fraction of nanoparticles no good dispersion occurs.

### 4. Conclusion

The Flexural strength of EP/nano  $\text{SiO}_2$  increased with increasing volume fraction for fumed silica nanoparticles, this behavior in nano-composites is attributed to increasing in complicating chains crosslink caused by adding (because of van der waals bond which is weak bond but with huge numbers) nanoparticles. The stiffness (Young's modulus) of samples increase with increase 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 structure 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. Silica nano-particles agglomerations are very obvious in the fracture surface.

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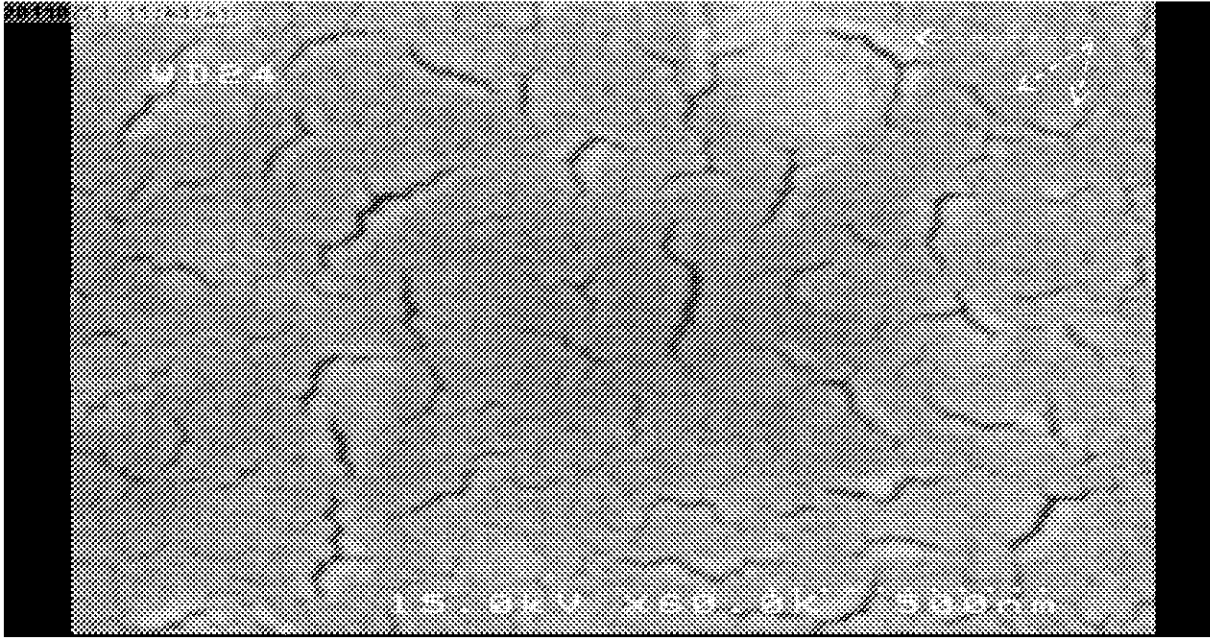
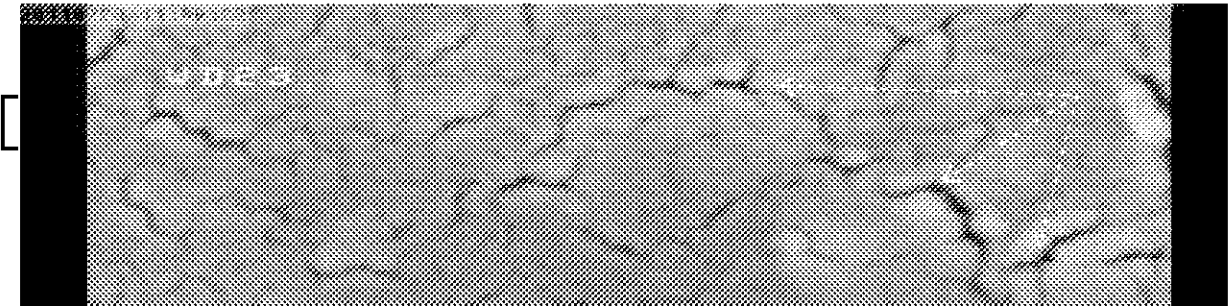


Fig. ( 3A). Fracture structure topography of neat epoxy resin.



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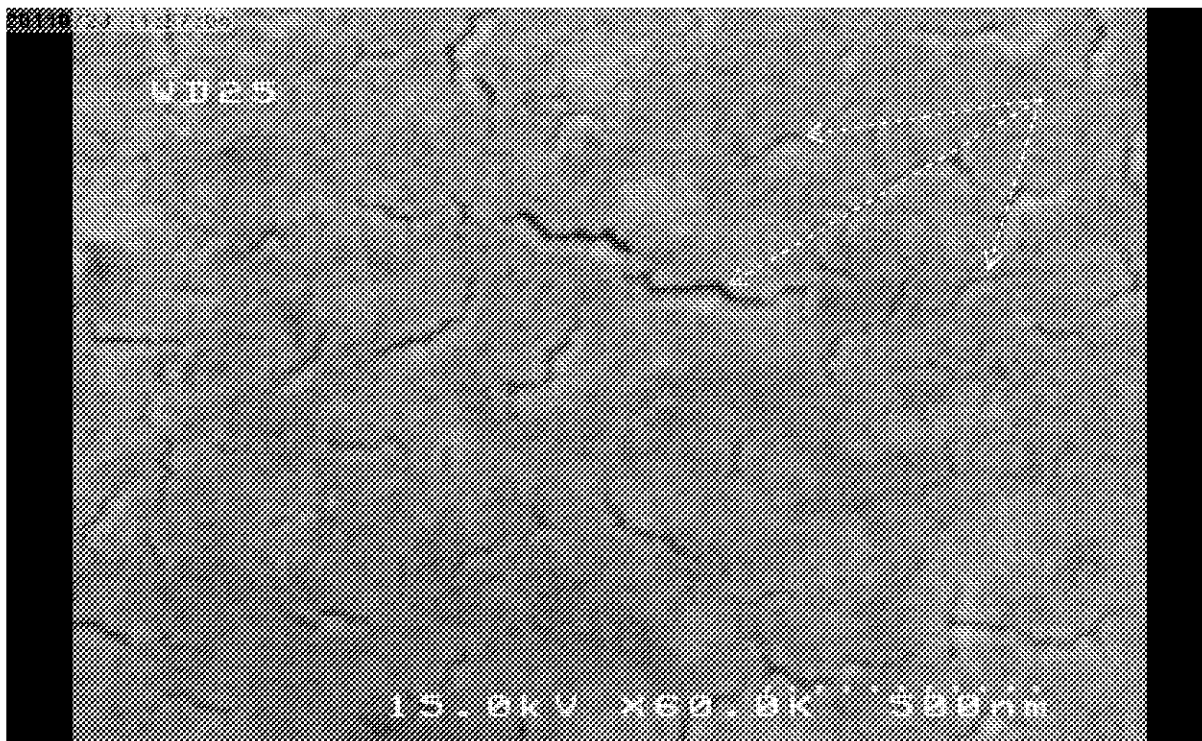


Fig. ( 3C). Fracture structure topography of nanocomposies (EP/ 2 vol.% nano SiO<sub>2</sub>).

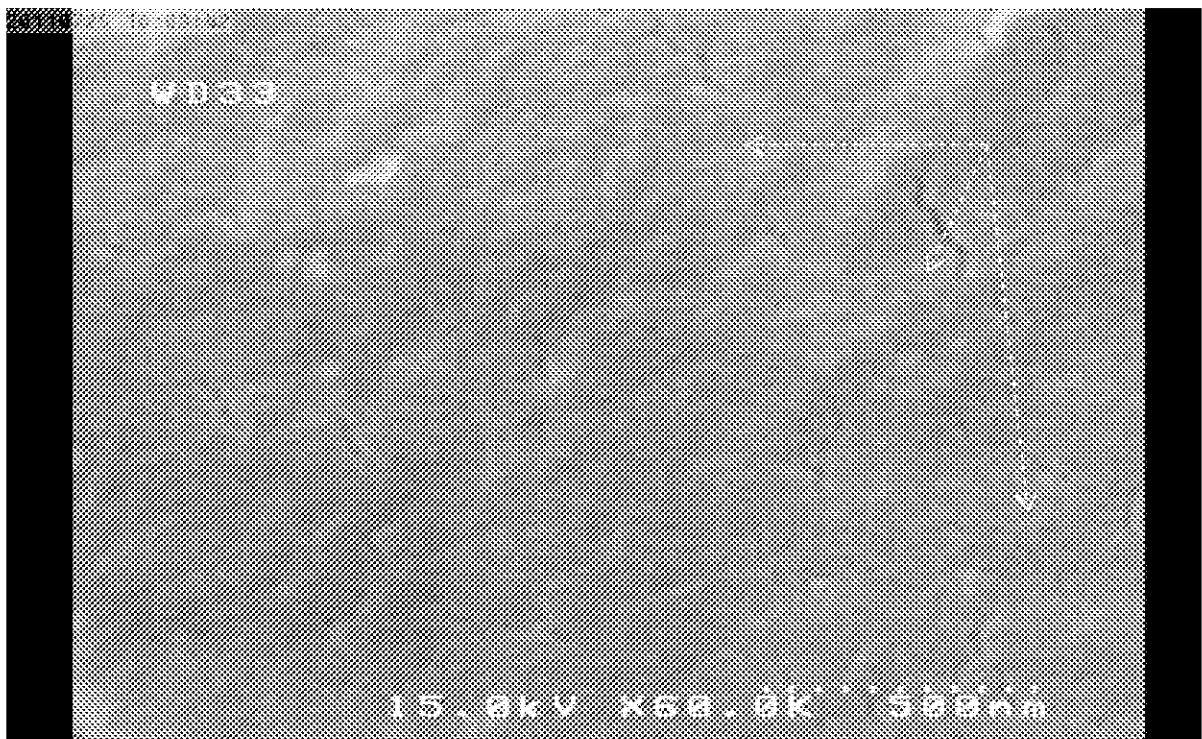


Fig. ( 3D). Fracture structure topography of nanocomposies (EP/ 3 vol.% nano SiO<sub>2</sub>).



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Fig. ( 3E). Fracture structure topography of nanocomposies (EP/ 4 vol.% nano SiO<sub>2</sub>).

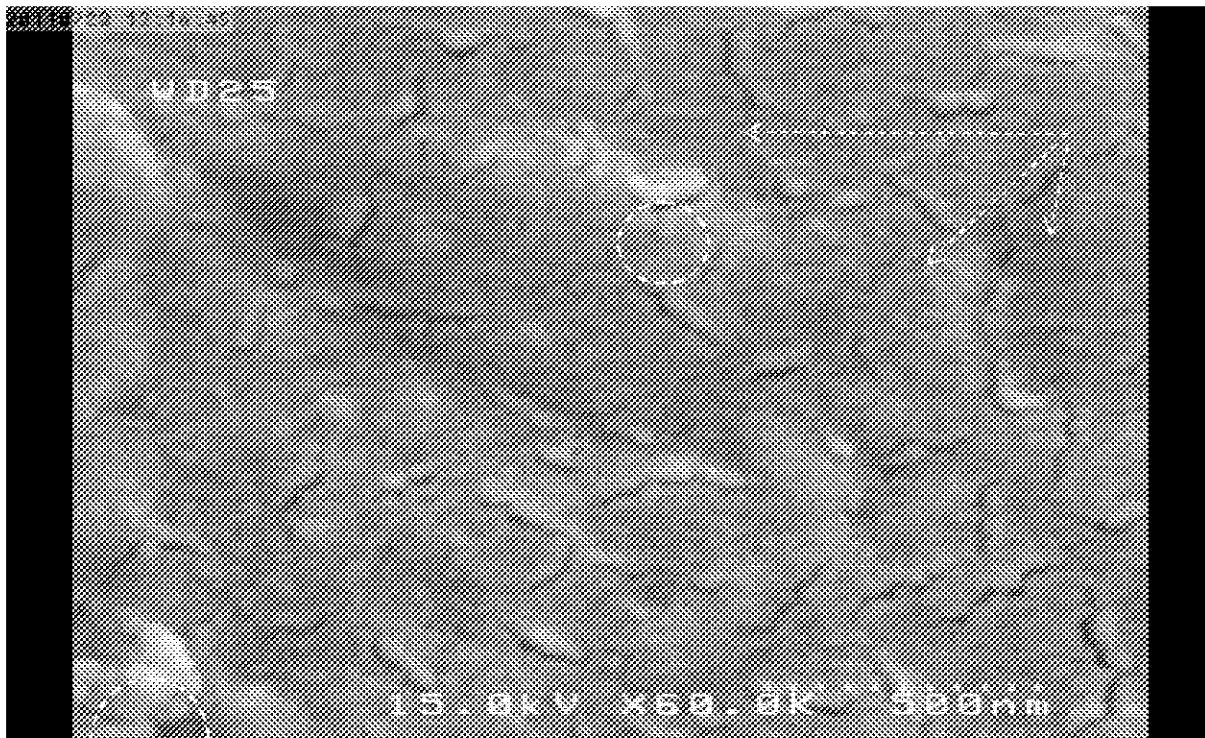


Fig. ( 3F). Fracture structure topography of nanocomposies (EP/ 5 vol.% nano SiO<sub>2</sub>).

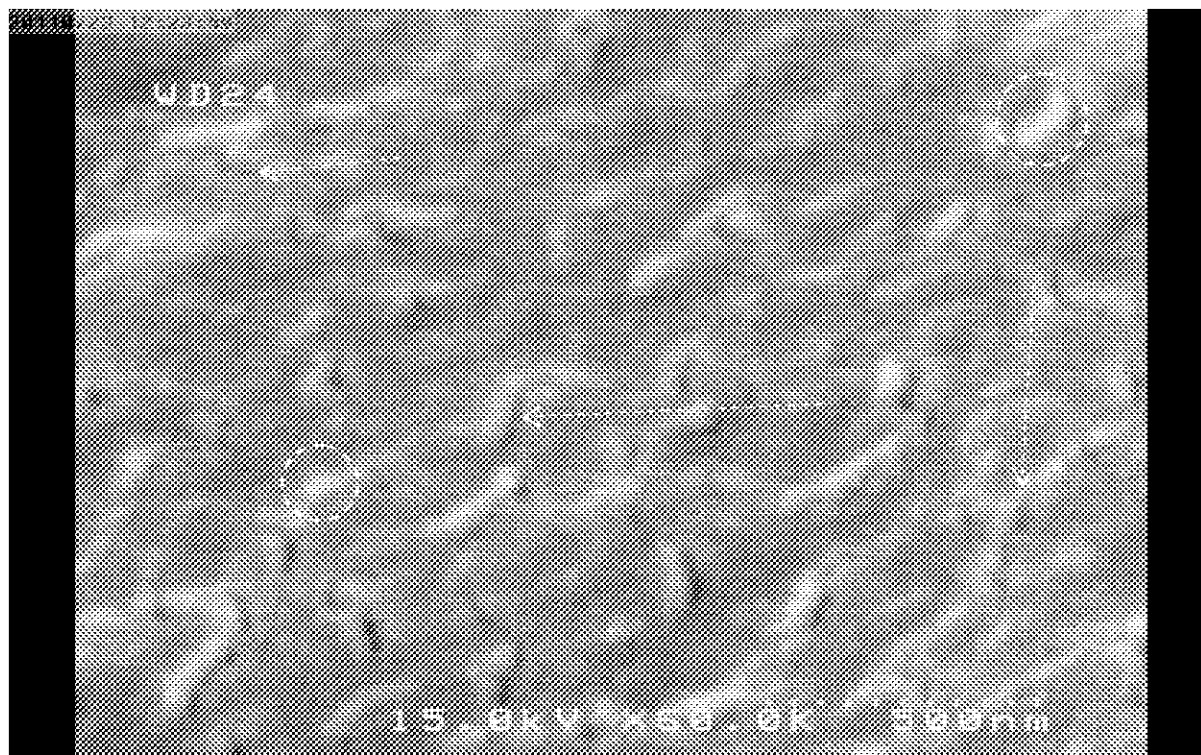


Fig. ( 3G). Fracture structure topography of nanocomposies (EP/ 7 vol.% nano SiO<sub>2</sub>).

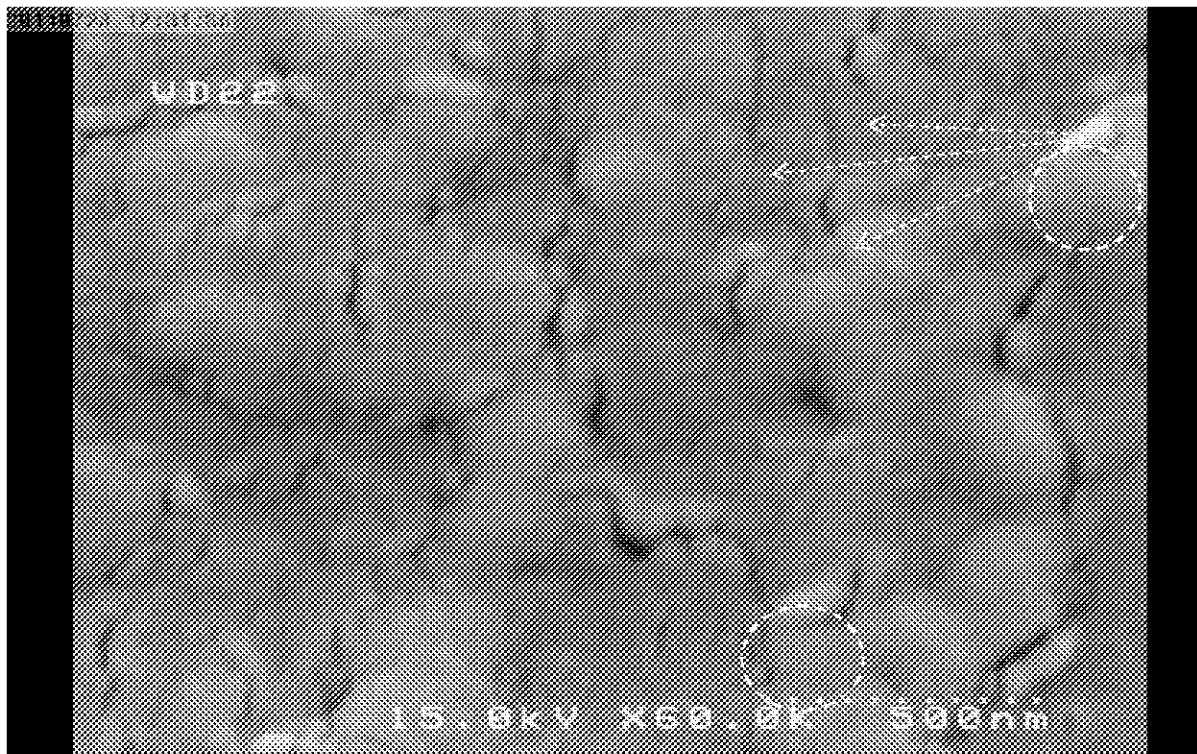


Fig. ( 3H). Fracture structure topography of nanocomposies (EP/ 10 vol.% nano SiO<sub>2</sub>).

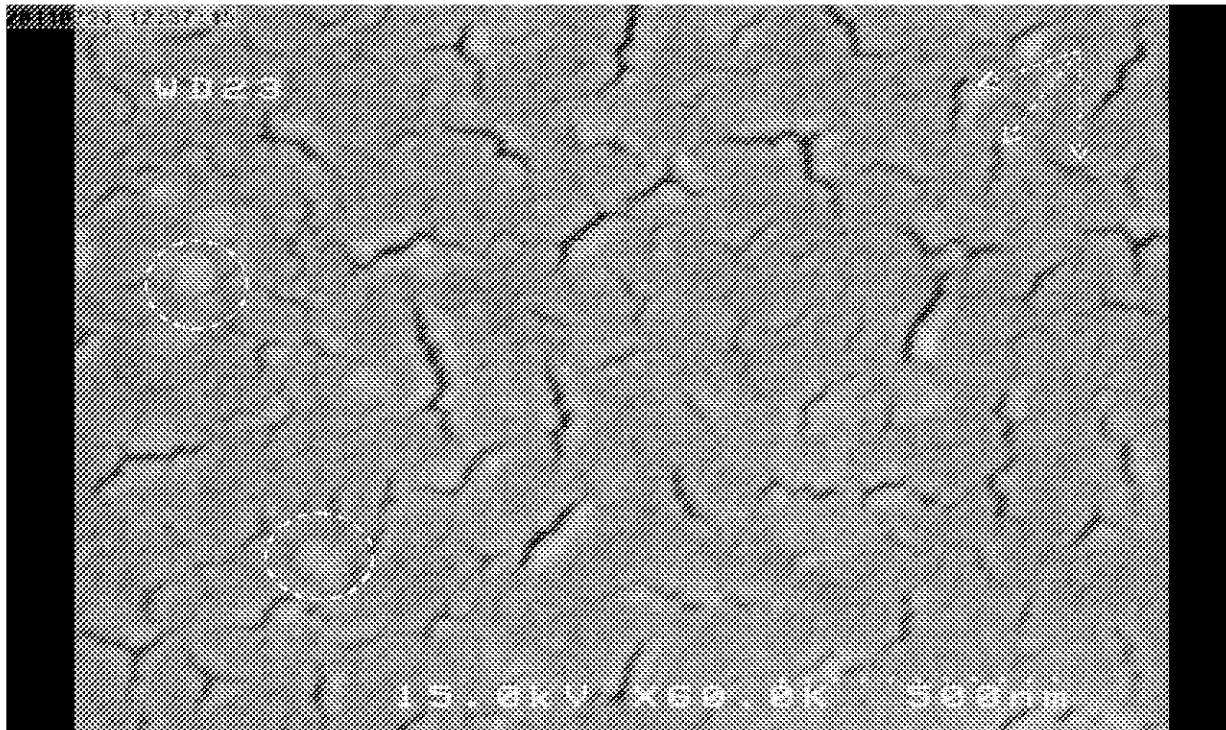


Fig. ( 3J). Fracture structure topography of nanocomposies (EP/ 15 vol.% nano SiO<sub>2</sub>).

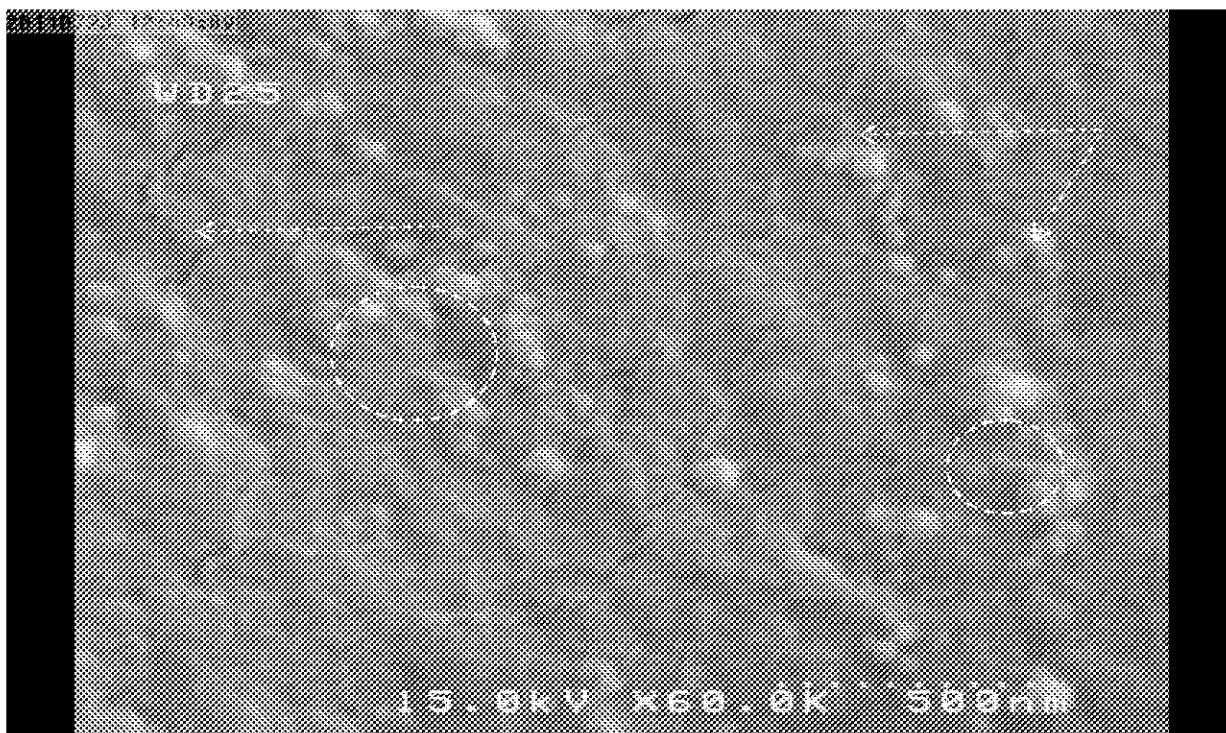


Fig. ( 3I). Fracture structure topography of nanocomposies (EP/ 20 vol.% nano SiO<sub>2</sub>).

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## دراسة الخصائص الميكانيكية والتصدع التركيبي باستخدام مسح المجهر الإلكتروني

### لمتراكبات الايبوكسي / سيلكا النانوية

مصطفى حسام سعيد ، قسم العلوم ، كلية التربية الأساسية ، الجامعة المستنصرية

### الخلاصة

تمت دراسة تأثير المضافات النانوية لجسيم السيليكا (  $\text{SiO}_2$ - 12 nm ) لمادة الايبوكسي على الخصائص الميكانيكية ( قوة الانحناء، معامل المرونة) و على شكل تركيب الكسر باستخدام المجهر الإلكتروني. حضرت المتراكبات النانوية باستخدام طريقة من ثلاث مراحل الخلط الميكانيكي والخلط بالامواج فوق الصوتية واستخدام تقنية التفريغ ولعدة نسب حجمية (1، 2، 3، 4، 5، 7، 10، 15، 20%) حيث وجد ان قوة المتانة ومعامل المرونة يزداد بزيادة نسب الاضافات للمتراكبات النانوية وخصوصا في النسب ذات الاضافات القليلة حيث وصلت اعلى نسبة تحسن في الاضافة 4% وفي نسب الاضافات الاعلى ينخفض التحسن معى بقاء الخصائص الميكانيكية للمتراكبات اعلى من الخاصائص الميكانيكية لمادة الايبوكسي. كما ان شكل تركيب الكسر ازداد تعقيد وقلة المساحات المسطحة مقارنة بشكل تركيب الكسر لمادة الايبوكسي كما قد ظهر اكثر من اتجاه لانتشار الكسر على عكس شكل تركيب الكسر في مادة الايبوكسي. كما ظهر واضحا وجود التكتلات للجسيمات النانوية لمادة السيليكا على سطح تركيب الكسر.