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Fatigue behavior of Epoxy/ SiO_2 NanocompositesReinforced with E- glass Fiber

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ABSTRACT

Fatigue behavior of Epoxy/ SiO_2nanocomposites with different volume fraction of SiO_2nano particles (1%,3%,5%,7% and10%) are reported , Atomic force microscopy techniques(AFM) was used with scanning probe microscope to measure the particles size of SiO_2nanoparticales.Minimum roughness of the composites was found for composites with 3% vol. fraction of SiO_2 nanoparticles. Epoxy/ SiO_2nanocomposites were reinforced with 6 layers of chopped mat E-glass fiber and their fatigue behaviourare reported .Fatigue test was carried under rotary bending method and the type of loading was sinusoidal wave with wave ratio R= -1 , and loading frequency 5Hz, which is believed to give a negligible temperature rise during the test.Values of fatigue strength ,fatigue life and fatigue limit of the tested composites from S-N curves are reported .The results show that addition of SiO_2nanoparticle enhance the fatigue values however more enhancement was obtained for Epoxy/ SiO_2nanocomposite reinforced with 6 layers of chopped mat E-glass fiber .

Key words: Fatigue, silica/ Epoxy Nanocompositeand E-glass fibers.

1.INTRODUCTION

Polymer nanocomposites show unique properties, combining the advantages of the inorganic nanofillers (e.g., rigidity, thermal stability) and the organic polymers (e.g., flexibility, dielectric, ductility, and processability) ,Inorganic nanofillersinclude nanotubes, metal oxides (e.g., SiO2, TiO2, Al_2O_3, Fe_3O_4), layered silicates (e.g., montmorillonite, saponite), metalic nanoparticles (e.g., Au, Cu), and semiconductors (e.g., PbS, CdS) have large surface area, leading to a dramatic increase in interfacial area so these nanofillers, even at very low concentrations, can strongly change the macroscopic properties of the polymer [1-4].

Compared to microparticles , nanoparticles have some unique features. Firstly, the much higher specific surface area can promote stress transfer from matrix to nanoparticles, Secondly; the required loadings of nanoparticles in polymer matrices are usually much lower than those of micro-fillers (typically 10–40 vol. % for the latter). Therefore, man y intrinsic merits of neat polymers, such as low weight, ductility, good processability, and transparency (e.g . for epoxy) will be retained after the addition of nanoparticles[5].

There are many important applications for polymer nanocomposites such as computer chip packaging (insulation), protective coatings, adhesives and advanced aerospace composites, based on their great strength, high temperature stability, good processability, and good chemical resistance. Also polymer nanocomposites show major improvements in mechanical properties, gas barrier properties, thermal stability, fire retardancy, insulating properties with good mechanical properties [6-8].

Fatigue concerns the damage of materials when subjected to cyclic loading at the stress level which is less than their ultimate static strength. Fatigue damage is known to be a slow process of which the development depends on the microstructure of the materials. For homogeneous materials, the fatigue behavior is often characterized by an early crack that dominatesthe damage development and lead to final fracture, for inhomogeneous materials, such as fiber-or particulate-reinforced polymers, thefatigue damage at an early stage is often diffuse in nature, as the crack can be initiated from multiple sites. In this case, the dominant crack may not be apparent until it is very close to the final fracture [9,10].Research on the fatigue behavior of nanocomposites basis for later understanding the complex fatigue behavior were performed, a number of these early works studied the effect of variation testing parameters on fatigue behavior. Many works have been conducted and reported in following.

Blackman et al [11] studied the fracture and fatigue behavior of nano-modified epoxy polymers and they showed the introduction of nano-silica particles into the epoxy polymer has increased both the initial toughness, as measured by the fracture toughness, and also significantly improved the cyclic-fatigue behaviour of the epoxy polymer. Further, design engineers would clearly prefer both the initial toughness and the long-term cyclic-fatigue properties to be significantly enhanced by the presence of the toughening phase in the epoxy polymer.

Manjunatha et al [12] studied the effect of rubber micro-particles and silica nano-particles on the tensile fatigue behaviour of a glass -fibre epoxy composite. It is clear that incorporation of either the CTBN rubber micro-particles or the silica nano-particles alone in the epoxy matrix have almost a similar beneficial effect on the fatigue performance of the GFRP composites. In addition to raising the fatigue limit by about 15%, these particles enhance the fatigue life of GFRP composite by about two to three times, when compared to the neat-resin matrix . Furthermore, the presence of both rubber and silica particles in the matrix to give a 'hybrid' modified GFRP results in further enhancement of the fatigue life particularly at the low stress ranges. Indeed, the fatigue limit raised by about 25% due to the presence of both these types

of particles. The suppressed extent of matrix cracking and reduced delamination growth rate in the composites based upon the modified matrices appears to be the main reasons for the observed enhancement of the fatigue lives of these GFRP composites.

Manjunatha et al.in another paper [13] showed that silica nanoparticles could increase the cyclic-fatigue performance of epoxy polymers, in addition to increasing the fracture toughness. Furthermore, this improvement in cyclic-fatigue performance has been found to be carried through to an improved performance of fibre-composite materials based upon such modified polymeric matrices.

In this work, Epoxy/ SiO_2nanocomposites**,** Epoxy/ SiO_2nanocomposites reinforced with 6 layers of chopped mat E-glass fiber wereprepared, and fatigue behaviorwere examinedand discussed in detail,The SiO_2nanoparticles have shown a positive effect on fatigue behavior of the nanocomposites.

2.EXPERIMETAL WORK

RAW MATERIALS

The raw materials used to prepare the samples are;Epoxy as a matrix (Nitofill, EPLV with Nitofill EPLV hardener from FosrocCompany),the weightratio of the epoxy resin to the hardener was 3:1 and gelling time 40 minutes at 35C°, mixed viscosity 1.0 poise at 35C° and density1.04g/cm^3.The fillers areSiO2 nanoparticles (Aerosil-200) from Aerosilpharma(hydrophilic silica) with mean diameter of 12nm, a specific surface area of 200±25m^2/g, Atomic force microscopy (AFM) was used(CSPM scanning probe microscope) tomeasurethe particles size of SiO_2nanoparticales, the particles size distribution is shown inFig.(1).

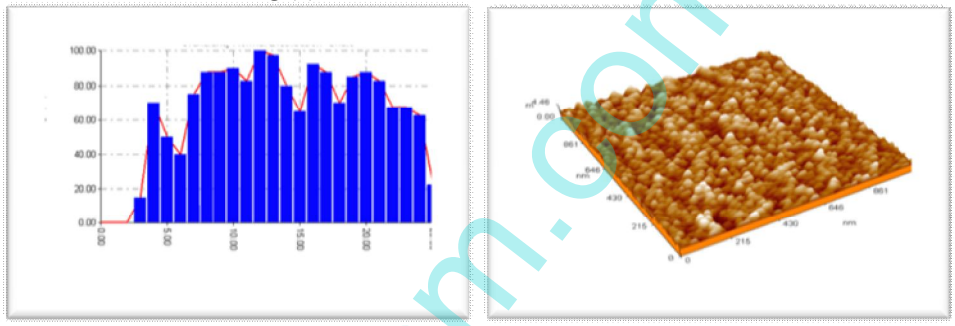

Figure1 AFM of silica nanoparticles

E-glass fibers typechopped mat as reinforcements of surface density300g/m^2. Table (1) shows some of the raw materials properties.

Table 1: Materials and some of their properties

Materials	Density g/cm^3	Particle Size (nm)	Surface area (m^2/g)	Purity%
Epoxy (EPLV)	1.04	----------	----------	----------
Nano-Silica (SiO_2)	0.05	12	200±25	99.8
E-glass fiber	2.55	----------	----------	----------

PREPARATION METHOD

Specimens of neat epoxy and its nanocomposites with different volume fraction (1%, 3%, 5%, 7% and10%)of SiO_2nanoparticales and others reinforced with six layers of E-glass fibers were prepared bymolding method.

The neat epoxy specimens were prepared by simple direct mixing of epoxy resin with the hardener. The nanocomposites were prepared in more complicated method such that;thenanoparticles were preheated at 120℃ for 2 h in order to eliminate possible absorbed water on their surface (which has been reported to increase the resins viscosity)and to prepare homogenous nanocomposites. An oil bath was used to heat up the mixture to desired 75℃temperatures so the viscosity of epoxy base is reduced. Proper mechanical stirring for 2h at this stage resulted better dispersion of nanoparticles, then the mixture was cooled to room temperature after that the hardener was added to the formulation and

mixed by mechanicalstirring for 10 minutes. The samples were left for 48 hours before pulling out from molds and left for 7 days before any test to get better curing conditions and kept then in vacuum chambers.
Fornonocomposite reinforced with E-glass fiber, they were prepared by pouring, the mixture of epoxy and SiO_2 nanoparticles on 6 laminates of the mat E- glass fiber uniformly using hand lay-up method, paint brush and aluminum notched roller to maintain uniformly mixture with the fibers sheet. The full curing of prepared sheets was done by leaving it for at least seven days before processing further.The nanocomposite sheets were cut to a certain dimension specimen according to the instrument manual specification by using circular saw.

FATIGUE TEST

Test samples for fatigue test cut from nanocomposites sheets were prepared with dimensions according to the instrument manual specification [14] as shown in Fig (2).

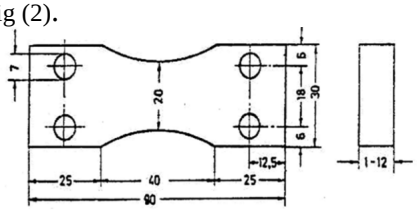

Figure2 Shape of fatigue test specimen with dimensions in mm [14].

The samples were tested at room temperature using alternating bending with stress ratio (R)$\sigma_{min}/\sigma_{max}$=-1 and loading frequency 5Hz, under constant displacement (U=20mm) the bending moment(m_b) of the stressed specimen can be calculated directly from the fatigue testing machine readings , from which bending stress (σ)can be determined from the following equation:

$$\sigma = \frac{m_b}{W} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where $W= \frac{h^2 b}{6}$

m_b =the bending moment (Nm)(was calculated from the standard curves between indication on dial gauge and the bending moment).
b = the width of specimen waist (20mm).
h = thickness of specimen.
Atomic force microscopy (AFM) was performed using CSPM scanning probe microscope to measurethe roughness of the surface specimenCSPM images surface roughness analysis of epoxy /SiO2nanocomposite with 1%, 3%and 10%SiO2 nanoparticales as shown in Figure (3) ,It was noticed that roughness of EP/SiO_2nanocomposites with1% volume fraction of SiO2nanoparticale was about 6.7nm .The roughness value of EP/SiO_2nanocomposites decreases to 2.36nmwith the increasing silica nanoparticles concentration until 3% vol. fraction of SiO_2 nanoparticles are reached, This behaviour could be due to good distribution and dispersion of nanoparticles with optimal concentration leading to overcome the problems of matrix structure (nanocracks, big agglomerations and weak resistance to loads) .Then the roughness of the surface of EP/SiO_2increase about 18% with increasing of concentration of silica nanoparticles to10% vol. fraction to 2.91nm,due to formation of some agglomerations .

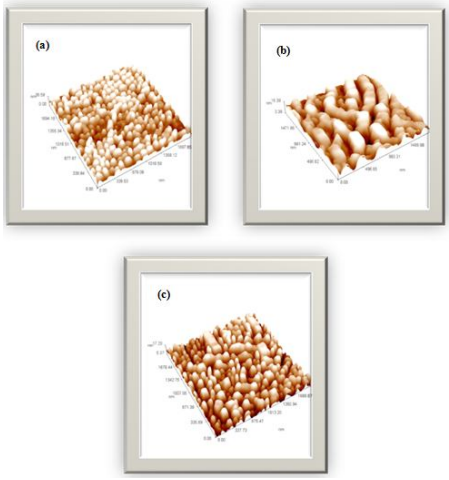

Figure3 Surface roughness of nanocomposies with[(a)1% ,(b) 3% and(c) 10%] vol. fraction ofSiO_2

3. RESULTS AND DISCUSION

The fatigue test showed that all specimens withSiO_2 nanoparticles suffer brittle fracture during the test course as shown in Fig.(4).

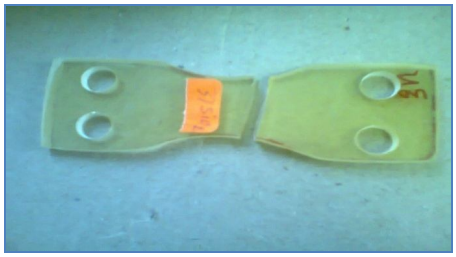

Figure4 The brittle fracture EP/3%SiO2nanocomposites

For the neat epoxy and its SiO_2nanocomposites , the maximum fatigue stress, and fatigue life were determined and listed in Table(2).

Table 2: Maximum fatigue stress and fatigue life of epoxy and EP/ SiO_2 nanocomposites.

Samples	Vol.% nano-SiO2	Maximum fatigue stress(MPa)	Fatigue life
EP	0	42.3	400
EP1S	1	52.5	1700
EP3S	3	67.4	2500
EP5S	5	53.3	5000
EP7S	7	44.9	7100
EP10S	10	53.7	19000

The results show variation in fatigue behavior of the neat epoxy and its nanocomposites, the neat epoxy specimens was fractured after about 400 cycles at notch area where it becomes location to stress concentration. This agrees with results reported by Harris [15]. It can be inferred from the table that the addition of silica nanoparticles has improved the maximum fatigue stress and fatigue life of the epoxy.EP3S nanocomposites with 3% nanoSiO2 show higher maximum fatigue strees where it increase about 39%, because nanoparticles tend to occupy small holes in the epoxy resin and act as a bridge to make more molecular interconnected resulting in reduction in total free volume as well as increase in cross-linking density [16]. At higher addition of SiO2 nanoparticles maximum fatigue stress decrease with increasing volume fraction of SiO2 nanoparticles fraction, but the results of maximum fatigue stress still higher than that of epoxy as shown in figure(5).

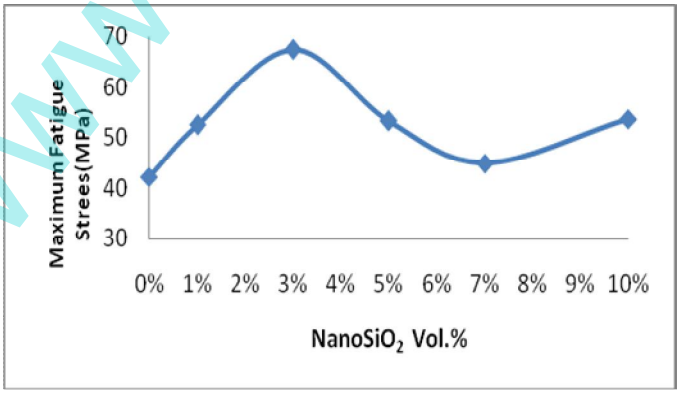

Figure5 Maximum fatigue stress vs. the volume fraction of $SiO_2$for Ep/SiO_2nanocomposites.

There are possible reasons of this decrement in maximum fatigue stress one would be the weak boundaries between nanoparticles and probable micronized trapped bubbles. The other responsible reason may be the effect of high amounts of nanoparticles on homogeneity in cross linking of the epoxy network. As the interfacial area of the particles is high their interaction with epoxy chain would cause the lower homogeneity in cross link density [17]. Finally, due to nanoparticles agglomeration lead to increase the distance (free volume space) between epoxy chains.The results of fatigue life that observed from table(2)and Fig.(6) is in good agreement with results recorded for the epoxy polymer containing nano-silica by Blackman et al [11].

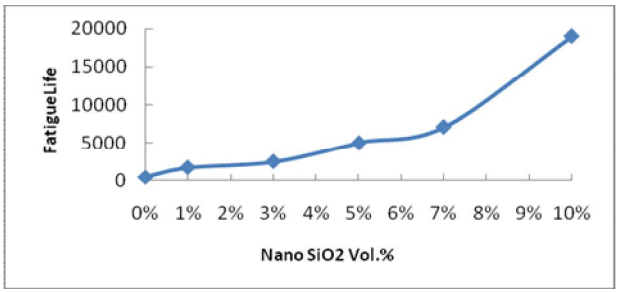

Figure7 Fatigue Life vs. the volume fraction of $SiO_2$for EP/SiO_2nanocomposites

Then bending stress (σ) verses number of cycles curves (σ -N) were plotted for the tested samples. Fig.(7) shows(σ -N) curves of the nanocomposites reinforced with 6 layers of E-glass fibers, the values of maximum fatigue stress, fatigue strength for $1*10^4$ cycles and fatigue limit are evaluated and listed in table (3).

The results show similar behavior to that observed for EP/ SiO2 nanocomposite, but with higher values due to E- glass fiber which enhance the fatigue of nanocomposites as shown in Figs(8-10),these results show good agreement with results of manjunatha.et al [12] which showedforfour different types of GFRP laminates with10 wt.% silica nano-particles exhibit higher fatiguelives, these particles enhance the fatigue life of GFRP composite by about two to three timesand raising the fatigue limit by about 15%, , when compared to the neat-resin matrix compositesand also in a good agreement withManjunatha et al[13] which showsThe fatigue life of the GFRP composite was increased by about three to four times due to the silica nanoparticles.

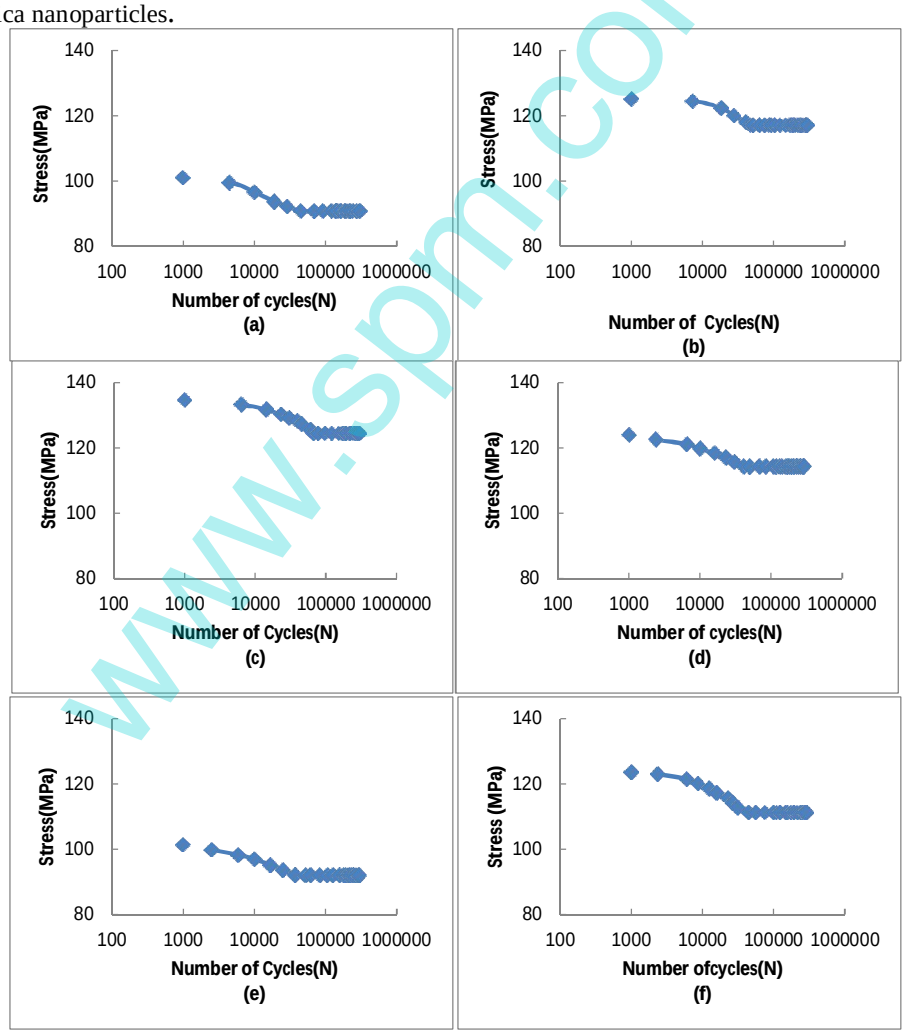

Figure 8 σ-N curves for(a) epoxy(b) 1%,(c)3%,(d) 5%,(e)7%and(f)10%Vol.fraction nano SiO_2

Reinforced with 6 layers chopped mat E-glass.

Table3: Maximum fatigue stress, fatigue strength and fatigue limit of Epoxy, EP/ SiO_2 nanocomposites reinforced with 6 laminates of E-glass.

Samples	Vol.% nano-SiO2	Max. Fatigue Stress(MPa)	Fatiguestrength (MPa)for1*10^4cycles	Fatigue Limit(MPa)
EPCM	0	102.5	96.67	90.8
EP1SCM	1	125.9	123.1	117.2
EP3SCM	3	136.2	132.2	124.5
EP5SCM	5	126.7	119.83	115.7
EP7SCM	7	103	97.08	92
EP10SCM	10	124.5	119.05	111.3

This is also agree with recent studies [18,19] indication that the inclusion of nanoparticles much improves the mechanical properties [18] and fatigue life [19] of continuous glass fiber reinforced EPS (GFRP).Under lower SiO2 content the dispersion of the particles in the epoxy matrix was good enough and fatigue limit increased with increased of the SiO2 contents.In many cases only EP nanocomposites filled with nanoparticles at low concentration are of practical interests because the introduction of nano fillers at high concentration will complicate the manufacture process and lead to high costs, the nanoparticles adhere to each other due to the inherent Vander Waals force between the particles resultinginnanoparticles agglomerates [20].

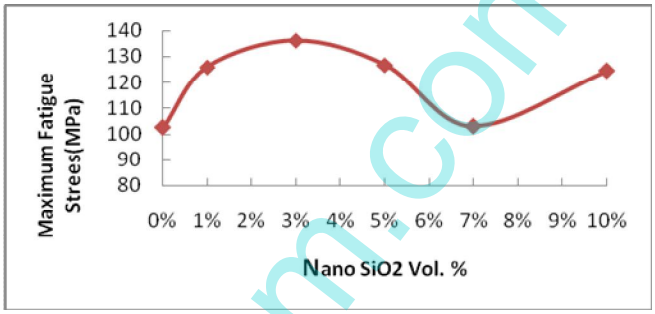

Figure8 Maximum fatigue stress vs. the volume fraction ofSiO_2 for EP/SiO_2nanocomposites reinforced with E-glass.

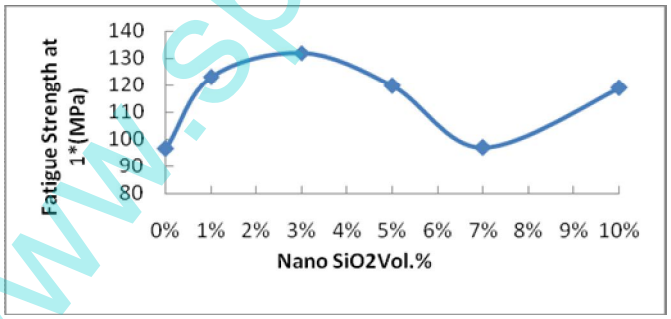

Figure 9 Fatigue strength vs. the volume fraction of$SiO_2$for EP/SiO_2nanocomposites reinforced with E-glass

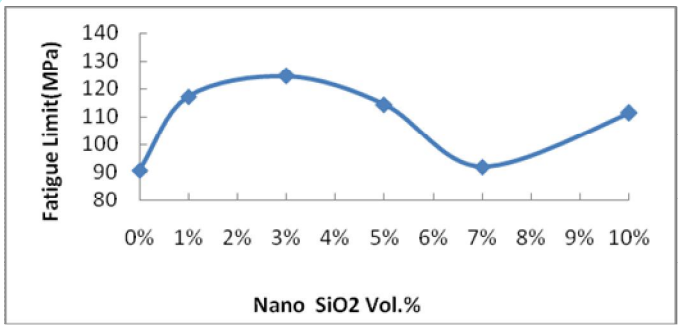

Figure 10 Fatigue limit vs. the volume fraction of$SiO_2$for EP/SiO_2nanocomposites reinforced with E-glass

EP/SiO_2nanocomposites reinforced with E-glass were examined to reveal the type of damage.The process of damage starts off with the initiation of micro cracks of matrix, which is an irreversible micro damage that occurs throughout the stressed region at the waist of the test specimen manifested by color whitening as shown in Fig.(11).

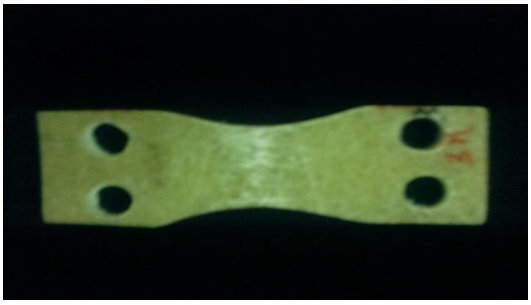

Figure 11 Photographs of tested specimens show damage resistance of3%Vol.$SiO_2$for nanocomposites reinforced 6 Layers of(CM)E-glass fiber.

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4. CONCLUSION

1-Based on the results obtained in this investigation, it is clear that the presence of silica nano-particles in the epoxy polymer reduces the brittle nature of the epoxy resin such that enhancing the maximum fatigue stress, fatigue life and roughness of the composite surface.
2-Better enhancement was observed for composite reinforced by E-glass fiber.
3-Highest fatigue results were found for EP/3% nano SiO_2 composite reinforced by 6 layers of E-glass fiber.

REFERENCES

[1] Zou, H.; Wu, S.S. andShen, J. " Polymer/silica nanocomposites: preparation, characterization, properties, and applications". Chem. Rev., *Vol.*108(2008) 3893-3957.
[2] Mark, J.E."Some novel polymericnanocomposites" Accounts Chem. Res., Vol. 12(2006) 881-888.
[3] Winey, K.I. andMoniruzzaman, M. "Polymer nanocomposites containing carbon nanotubes"Macromolecules, Vol. 39(2006) 5194-5205.
[4] Wei,L. M ; Tang, T and Huang B.T." Synthesis and characterization of polyethylene/clay–silica nanocomposites: a montmorillonite/silica- hybrid- supported catalyst and in situ polymerization. J. Polym. Sci. Pol. Chem.Vol. 42(2004) 941-949.
[5] H. Zhang, Z. Zhang, K. Friedrich and C. Eger "Property improvements of in situ epoxy nanocomposites with reduced interparticle distance at high nanosilica content" ActaMaterialia.Vol. 54 (2006) 1833–1842
[6] R. Sarathi, R.K. Sahuand P. Rajeshkumar"Understanding the thermal, mechanical and electrical properties of epoxy nanocomposites" Materials Science and Engineering A 445–446 (2007) 567–578.
[7] L. L. Zhai, L. GP and W. YW."Effect of nano-Al_2O_3 on adhesion strength of epoxy adhesive and steel"International Journal of Adhesion &Adhesives,Vol. 28 (2008) 23-28.
[8] R.P.Singh, M.Zhang and D.Chan"Toughening of a brittle thermosettingpolymer: Effects of reinforcement particle size and volume fraction"J. Mater. Sci. Vol. 37 (2002) 781-788.
[9] W.D.Callister "Materials Science and Engineering" John Wily and Sons, Inc, London2003.
[10] T.M.Dicka,P.B.Jar and J.R.Cheng"Predication of fatigue resistance of short-fiber-reinforced polymer" International Journal of Fatigue, Vol. 31(2009) 184-191.
[11] B.R.K.Blackman.A.J.Kinloch.J.Sohn Lee.A.C.Taylor.R.Agarwal.G.Schuenemen and S.Sprenger " Thefracture and fatigue behaviour of nano-modified epoxy polymers" MaterSci.,Vol.42 (2007)7049–7051 DOI 10.1007/s10853-007-1768-6.
[12] C.M.Manjunath.A.C.Taylor.A.J. Kinloch and S.Sprenger"The effect of rubber micro-particles and silica nano-particles on the tensile fatigue behavior of glass-fiber epoxy composite"J Mater Sci., Vol.44 (2009)342-345 DOI 10.1007/s 10853-3092-1.
[13] C.M. Manjunatha, "The tensile fatigue behavior of a glass fiber reinforced plastic composite using a hybrid toughened epoxy matrix" Journal of Composite Materials,Vol. 44(2010), 2095-2108.
[14] K.J. Majeed"Fatigue behavior of E-glass fibers/epoxy composites" PhD thesis, College of Science, University of Baghdad, Baghdad, IRAQ (2005).
[15] B. Harris "Fatigue in Composites" Woodhead Publishing Ltd, Cambridge England, (2003).

[16] N. S alahuddin, A. Moet, A. H iltnerandE. Baer"Nanoscale highly filled epoxy nanocomposite" Eur. Polym. J.,Vol. 38 (2002) 1477 – 1482.

[17] M.AL.Nikje, M.R.Khanmohammadi, A. B.Garmarudi and M.Haghshenas",Nanosilica reinforced epoxy floor coating composites : preparation and thermophysical characterization,"Current Chemistry Letters (2012).

[18] Uddin M.F. and Sun C.T."Strength of unidirectional glass/epoxy composite with silica nanoparticle – enhanced matrix"Composite Science and Technology,Vol.68, (2008),1637-1643.

[19] C.M.Manjunath.A.C.Taylor.A.J .Kinloch and S.Sprenger"the tensile fatigue behavior of silica nano-particles modified glass-fiber reinforced epoxy composite" Composite Science and Technology, Vol.70 (2010)193-199.

[20] G. Zhang, Z. Rasheva,J.Karger –Kocsis and T.Burkhart " synergetic role of nanoparticales and micro –scale short carbon fibers on the mechanical profiles of epoxy resin" eXPRESS Polymer Letters,Vol.5,No.10(2011),859-872 .