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# The Influence of Micro- and Nano-Filler Content on the Mechanical Properties of Epoxy Composites

MOHAMMED PARVEZ,SHAIKH NOOR, MD MUTTE KHAN

## Abstract

Inthisstudy,theinfluenceofmicro-andnano-fillercontentonthemechanicalpropertiesofepoxycompositeswasstudied.Thematrixmaterialisepoxy;themicro-fillersare$Al_2O_3$,$TiO_2$andflyashaddedin10wt%to30wt%byweightratio;thenano-fillersare$Al_2O_3$,$TiO_2$andclayaddedin 2.5 wt% to 10 wt% by weight ratio. Test samples were prepared using an open mould type die. Tensile, three-point bending and hardnesstestswerecarriedout.Thetensilestrength,elasticmodulus,elongationatbreak,flexuralstrength,flexuralmodulus,andthehardnessofthecomposite materials were obtained and evaluated. The results show that the tensile strength, flexural strength and elongation at the breakvalues of composites decreased while the tensile modulus and flexural modulus increased with the increasing micro- and nano-filler contentratio.

**Keywords:micro-filler;nano-filler;mechanicalproperties;epoxy;composite**

**Highlights**

- *Thispaperhighlightspossiblereasonsforvariationsinthemechanicalpropertiesofcompositesfilledwithnano-andmicro-fillertocomparethemechanicalpropertyofcompositescontainingvariousamountsofnano-andmicro-fillers.*
- *Smallamountsofnano-sizeparticlesinepoxyhaveastrongeffectonbothtensileandflexuralstrengthaswellasductility.*
- *Agglomerationproblemshavebeenobservedatahigheramountsoffillerratiosincomposites.*

## INTRODUCTION

Various fillers have been used to design the desiredpropertiesinthepolymer matrix. These fillersaremineralfillers,agriculturaland/orpuzzolanicwastes.Inthisfield,researchershavestudiedtheeffectsofparticleshape,fillertype,size,contentratio, and adhesion between matrix and fillers on themechanical properties of polymer composites. Zamanetal.[1] studiedthemicro-andnano-ZnO-filledpolypropylenecompositeswithfillerratiosbetween 2wt%to8wt%.Nano-filledcompositesshowedbetterresultsthanmicro-filledcompositesatthesame filler ratios. They stated that the dispersion ofparticlesisoptimalinthe5wt%fillercontentbecausethe morphology images and dispersion of nano-fillerswere better, which led to stronger interfacial adhesionbetweenmatrixandfillers.Gaoetal.[2] studiednano-$CaCO_3$-filledpolystyrenecomposites.Theyconcluded that fillers added stiffness to the polymercomposite, but increasing the filler ratio beyond thatpointcausedanagglomerationofparticles,whichdecreasedtheadhesionbetweenmatrixandfillersand caused a drop in the mechanical strength of thecomposite.Agubra etal.[3]investigatedthe effects ofdispersion methods on the mechanical behaviour ofnano-clay-filled glass fibre epoxy composites. Theystatedthatincreasingviscositycausesproblemsinthe homogeneousdispersionoffillersandresultsin theagglomerationofthefillerparticles.Lametal. studiedthehardnessvaluesofnano-clay-filledepoxycomposites.Theystatedthatthehardnessvalues of the composite is increased by adding nano-clayfillersuptoalimitandthendecreasedbecauseofclusters atthehighfillerratios.YasminandDaniel investigatedgraphite-filledepoxycompositeswith

Department of Mech
mohammedparvez124@gmail.com, Shaiknoor.jntu@gmail.com,mdmak.khan98@gmail.com
ISL Engineering College.
International Airport Road, Bandlaguda, Chandrayangutta Hyderabad - 500005 Telangana, India.

2.5 % to 5 % by weight filler ratios. They concludedthat the tensile strength and modulus of the compositeare increased by adding fillers, and an agglomerationof fillers occurred at a 5 wt% filler ratio. Sayer [6]usedceramicfillers,suchasSiC, $Al_2O_3$,and$B_4C$,intheglass-reinforcedepoxyresin.Heconcludedthat the elastic modulus and buckling load carryingcapabilityofcompositeswereincreasedbyadding ceramicfillers.Asi[7]studiedthemechanicalproperties of $Al_2O_3$-filled glass fibre-reinforced epoxycomposites.Heconcludedthatthetensilestrengthof composites decreased with the addition of $Al_2O_3$fillers. While the bending strength increased up to 10wt%fillerratioanddecreasedathigherratios.Yangetal. [8] studied the mechanical properties of rice huskflour-differentparticlesizeeffectsandreachedabetterdispersion with small particle fillers. Increasing theparticle size and the filler ratio caused difficulties inthehomogeneousdispersionoffillers,leadingtoweakad hesionbetweenmatrixandfillers.Imoisilietal.[11] investigatedcocoapod-filledepoxycompositeswith 2 3 5wt%to30wt%fillerratios.Theystatedthatthebestdispersi on of the filler was at the 5 wt% filler ratio.Furthermore,themechanicalstrengthvaluesdecrease dwhile modulus and microhardness values increasedwhen fillers were added. Ray et al. [12] studied themechanicalpropertiesofflyash-filledvinylestercomposites. They stated that the fly ash increased thestiffness and rigidity of composite, but the mechanicalstrengthwasreducedwithahighcontentratio.R ajaet al. [13] studied fly ash-impregnated glass fibre-reinforced polyester composites. The addition of 10wt%flyashimprovedthemechanicalpropertiesofthe composite. Chauhan and Thakur [14] investigatedthe filler size and loading effects on the mechanicalandtribologicalperformanceofcenosphere-filledvinylestercomposites.Theystatedthatthemechanica land tribological performance increased, and optimumvalueswereobtainedwitha6wt%fillerratio.Prak ashetal.[15]investigatedtheinfluenceofmicro-andnano-fillersonthemechanicalpropertiesofpultruded unidirectional glass fibre-reinforced epoxycomposite systems and concluded that the improvedmechanical properties indicate that the unidirectionalglassfibre-reinforcedepoxywithcombinedmicro-andnanofiller-filledcompositeisagoodcandidateforstructuralapplicatio n.Manjunathetal.[16]studied the effect of filler content on the performanceof epoxy/PTW composites and concluded that PTWadditions showed beneficiary effects on the density,hardness,andstiffnesspropertiesofcomposites;ho wever, strength properties and ductility were foundtodecreasewiththeincreasingcontentofPTW.Finall y, Sudheer et al. [17] gave a general review ofepoxycompositesandparticularlyonceriaepoxynanoco mposites.

Inthisstudy,theinfluenceofthemicro-andnano-filler content ratios of the mechanical propertiesof epoxy composites was studied. For this purpose,tensile,three-filledpolypropylenecomposites.Theystatedthat high filler ratios increased the interfacial area andmade the polymer composite brittle. Thus, the tensileandimpactstrengthofthecompositesdecreasedwhil e the tensile modulus increased. Ibrahim et al. [9]investigated oil palm ash-filled unsaturated polyestercompositeswith10wt%to30vol%fillerratios.Th ey observed an increase in the modulus and a decrease inthetensileandflexuralstrengthofthecompositeswithadd ed filler materials. Li et al. [10] studied rice brancarbon/nitrilerubber composites.They investigated

Materials

## 2EXPERIMENTAL

Inthisstudy,epoxyresinisthepolymermatrixmaterial.Mic ro-fillers,suchasaluminiumoxide($Al_2O_3$), titanium dioxide ($TiO_2$) and fly ash, wereadded at a 10 % to 30 % by weight ratio. Nano-fillers,suchasaluminiumoxide(AlO),titaniumdioxide ($TiO_2$) and nanoclay, were added at 2.5 % to 10 % byweight ratios. The materials used and their propertiesaregiven inTable 1.

pointbending,andhardnesstestswere carried out. Tensile strength, elastic modulus,elongationatbreak,flexuralstrength,flexuralmo dulus and the hardness of the epoxy compositeswereobtained and evaluated.

**Table1.***Propertiesofmaterialsused*

| Materials | Properties | Density | Supplier |
|---|---|---|---|
| Epoxy resin(MGSL285) | BisphenolA | 1.178 | Dost ChemiaCo. |
| Aluminiumoxide($Al_2O_3$) | 45µm | 3.90 | Eczacıbası |
| Titanium dioxide($TiO_2$) | 50µm | 4.00 | SintasPlastiCo. |
| Fly ash | 45µm | 2.00 | KutahyaCementCo. |
| Aluminium oxide($Al_2O_3$) | 40nm | 3.88 | GrafenChemiaCo. |
| Titanium dioxide($TiO_2$) | 10nm,anatase | 3.90 | GrafenChemiaCo. |
| Nanoclay(Montmorillonite modifiedwithTrimethyl stearylammonium) | Nanomer 10µm | 1.90 | Sigma Aldrich |

### CompositePreparation

In the composite material preparation process, the filler was dried in an oven at 70 ºC for 4 h. The epoxy resin was heated in order to reduce viscosity before mixing. Fillers were then added into the resin and mixed using mechanical stirrer for 2 h. During this stage, vacuum processes were used to remove the entrapped air. After that, the hardener (Curing agent MGS LH 285) was added and mixed manually. The vacuum process was

again applied. Afterwards, composite resin was poured into the open moulds. In the case of nano-filler, before the hardener was added, the resin was subjected to an ultrasonic method for 15 min. Finally, specimens in the moulds were tested at room conditions for 24 h, following which they were placed in an oven and heated at 60 ºC for 15 h and 80 ºC for 5 h for post-curing in order to cross-link.



## 3RESULTS

Standard tensile tests were performed using a Shimadzu test machine with a crosshead speed of 5 mm/min at room temperature (23 ± 1 °C). The tensile strength value was determined according to the ASTM D638-10 standard [18]. The tensile strength, elastic modulus and strain [%] values were obtained and evaluated. Three-point bending tests were carried out using a Shimadzu test machine according to the ASTM D790-10 [19] standard. The test specimen dimensions were 3.2 mm × 12.7 mm × 127 mm and the test speed was 2 mm/min. Flexural strength, flexural modulus, and elongation at break values were obtained and evaluated.

Flexural stress was calculated according to Eq. (1):

$$\sigma = \frac{3FL}{2bd^2}, \quad (1)$$

where $\sigma$ is the flexural stress [MPa], $F$ is the load [N], $L$ is the span [mm], $b$ is the specimen width [mm] and $d$ is the specimen thickness [mm].

Flexural modulus was calculated according to Eq. (2):

$$E_B = \frac{L^3 m}{4bd^3}, \quad (2)$$

where $E_B$ is the flexural modulus [MPa], $L$ is the span [mm], $b$ is the specimen width [mm], $d$ is the specimen thickness [mm], and $m$ is the slope of the linear region of the load-displacement curve.

Bending strain was calculated according to Eq. (3):

$$\varepsilon = \frac{6Dd}{L^2}, \quad (3)$$

where $\varepsilon$ is the strain [mm/mm], $D$ is the maximum displacement at the central point of the specimen, $d$ is the specimen thickness [mm], and $L$ is the span [mm]. Finally, the hardness test was carried out with a Barcol hardness tester according to the ASTM D2583-07 [20] standard.

All tests were repeated at least between 3 to 5 times, and the results were recorded and plotted, see Figs. 1 to 8.

Fig. 1 presents the relationship between the tensile strength and filler content of micro- and nano-epoxy composites. It is clear that the tensile strengths of micro-filled composites decreased with increasing filler content ratio. The results also show the reduced sensitivity of the strength of $Al_2O_3$ filled composites to the change in filler content ratios. In the case of nano-filler, the strength is in an increase up to 2.5 % filler content. This increase is about 8% and 12% for nano-$TiO_2$ and nano-$Al_2O_3$-filled composites, respectively.

Fig. 2 shows the variation of tensile modulus of micro- and nano-filler composites with filler content. In this figure, the tensile modulus of epoxy composites

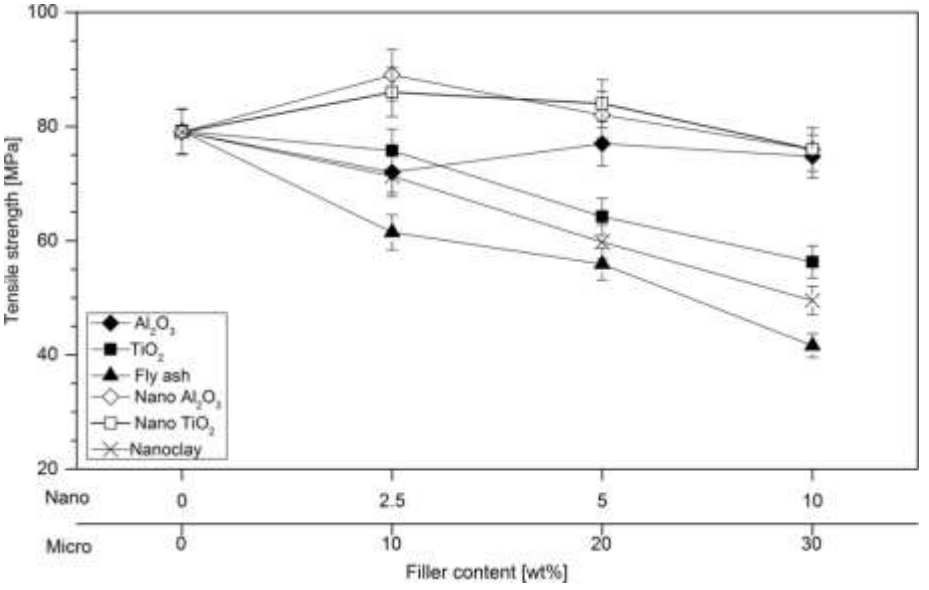


**Fig.1.***Therelationshipoftensilestrengthagainstfillercontentofmicroandnanoepoxycomposites*



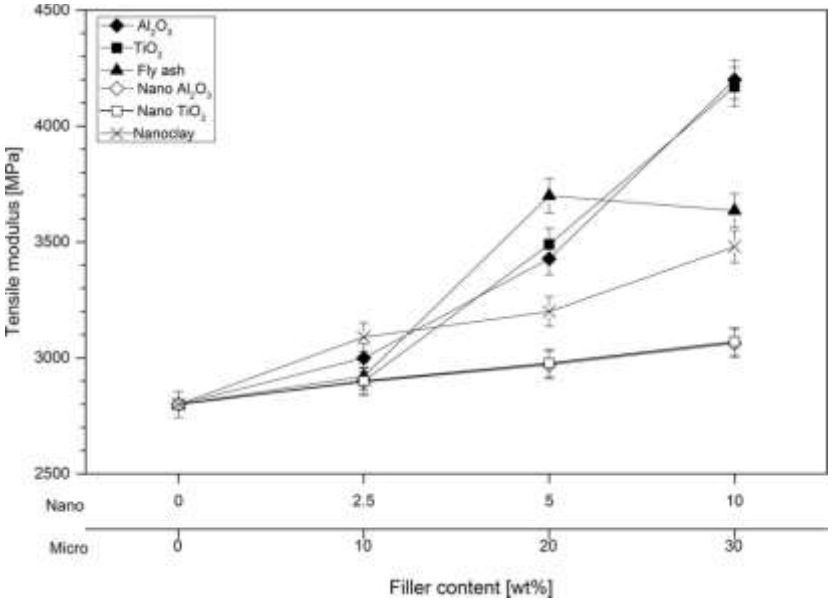


**Fig.2.***Therelationshipoftensilemodulusagainstfillercontentofmicroandnanoepoxycomposites*

increased with increasing filler content. Micro- andnano-fillers increased the polymer stiffness, and thisincreased its modulus. With a 200 % increase in fillercontent, there is a 50 % increase in the modulus forboth $Al_2O_3$- and $TiO_2$-filled epoxy composites. In thecase of nano-fillers, for a 300 % increase in nano-filler content, the modulus of nano-$Al_2O_3$, nano-$TiO_2$and nano-clay increased by 9 %, 9.6 %, and 24 %,respectively.

.3presentsthevariationofelongationatbreak with filler content for micro- and nano-epoxycomposites.Itisclearfromthisfigurethattheelongation-at-breakvaluesdecreasedwithincreasesof the filler content of the composite. The fillers gavethe matrix the brittle behaviour. For a 200 % increaseinmicro-$Al_2O_3$,$TiO_2$andflyashfillercontent,thedecreasesintheelongationatbreakare50%,66%,and65%,respectively.Fora300%increase

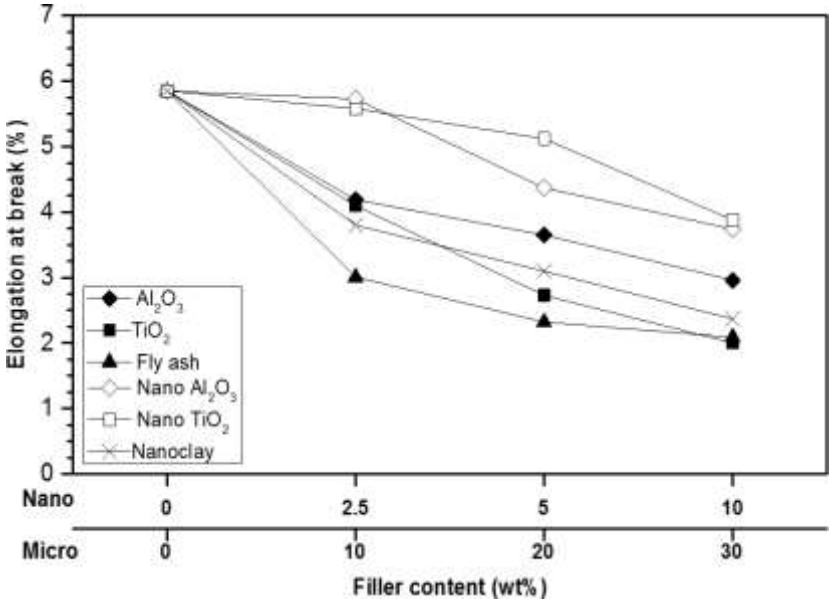


Fig.3.*Therelationshipofelongationatbreakagainstfillercontentofmicroandnanoepoxycomposites*

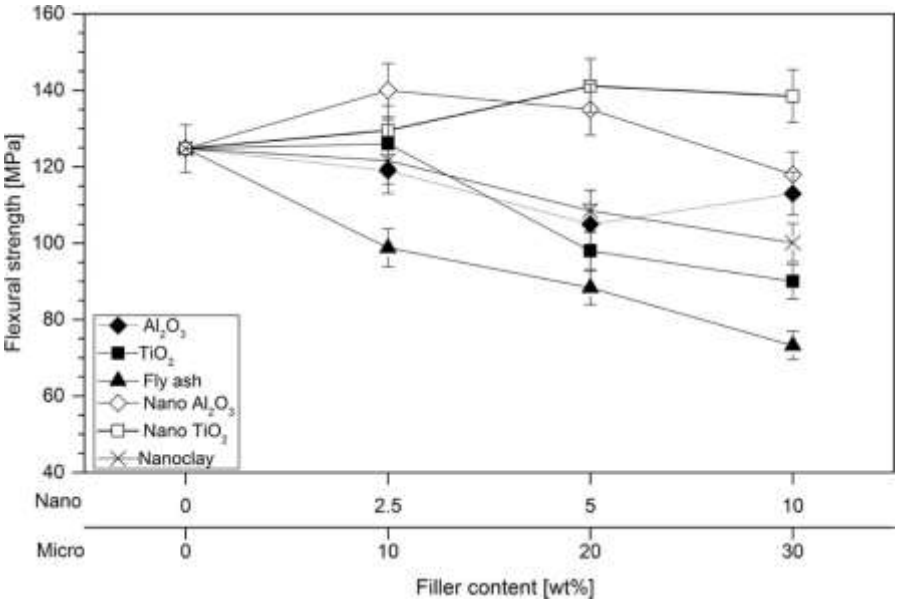


**Fig.4.** *Therelationshipofflexuralstrengthagainstfillercontentofmicro-andnano-epoxycomposites*

in nano-Al2O3, nano-TiO2 and nano-clay content, the decreases in the elongation at break are 37 %, 34 %, and 60 %, respectively.

Fig. 4 shows the flexural strength against filler content curves of micro- and nano-epoxy composites. In this figure, the flexural strengths of micro- composites decreased with increasing filler content, and this decrease ranged between 12 % to 45 %. The nano-composites follow an increasing trend up to
2.5 % to 5 % ratios, and then a decreasing trend at higher filler ratios; 2.5 wt% filled nano Al2O3 showed the highest increase at 12 %. The nano-TiO2-filled composite reaches a 13 % increase in flexural strength at 5 a wt% filled ratio. The nano-clay-filled composite follows a decreasing trend, and this decrease reaches 20 % at the 10 % filler ratio.

Fig. 5 shows the relationship between the flexuralmodulusfillercontentofmicro-andnano-epoxy

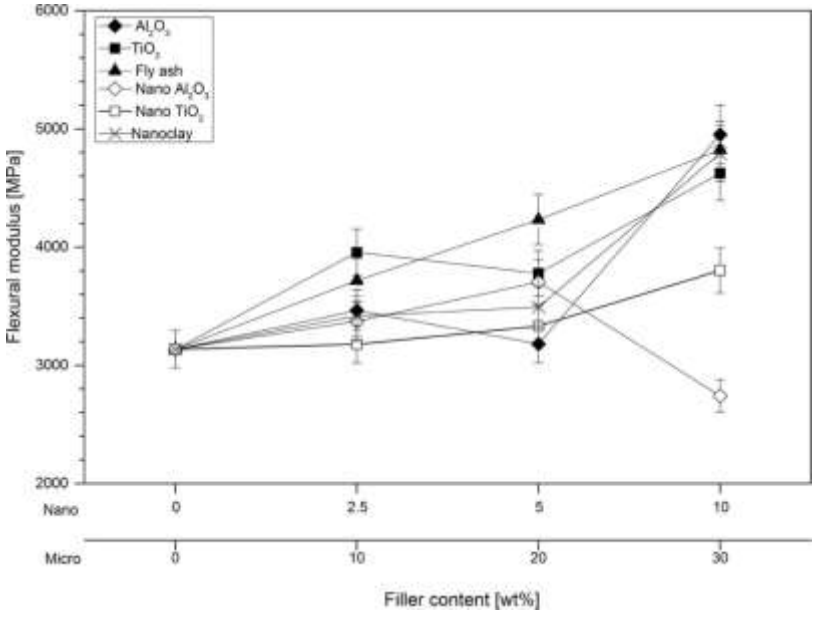

Fig.5.*Therelationshipofflexuralmodulusagainstfillercontentofmicroandnanoepoxycomposites*



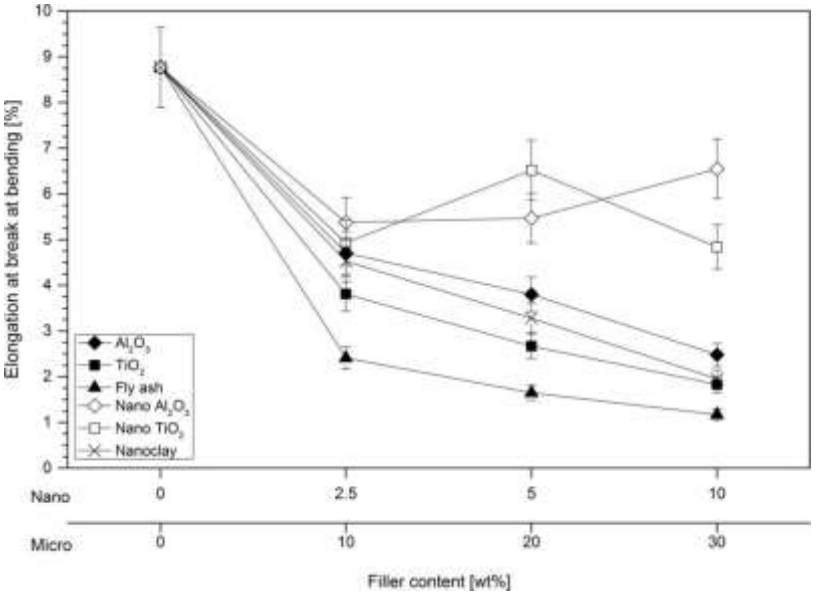


Fig.6.*Therelationshipofelongationatbreakatbendingagainstfillercontentofmicroandnanoepoxycomposites*

composites. As the fillers increased the rigidity and stiffness behaviour in the polymer composite, the flexural modulus increased with the increasing filler content. The highest flexural modulus values were reached with 30 wt% micro-filler content composites. For a 200 % increase in micro Al2O3, TiO2 and fly ash content, there are 57 %, 47 % and 53 % increases in the flexural modulus of the composite, respectively. For a 300 % increase in nano-filler content, there are 9 % and 45 % increases in nano-TiO2 and nano-clay epoxy composites flexural modulus, respectively. In the case of nano-Al2O3, there is an increasing and decreasing behaviour of 12 %.

Fig. 6 presents the variation of elongation at the break with filler content for micro- and nano- epoxy composites. As the presence of rigid fillers increased the matrix's brittle behaviour, which was reflected as reduced elongation at the break values of the materials, the figure shows that the minimum elongation at break values occurred at the maximum

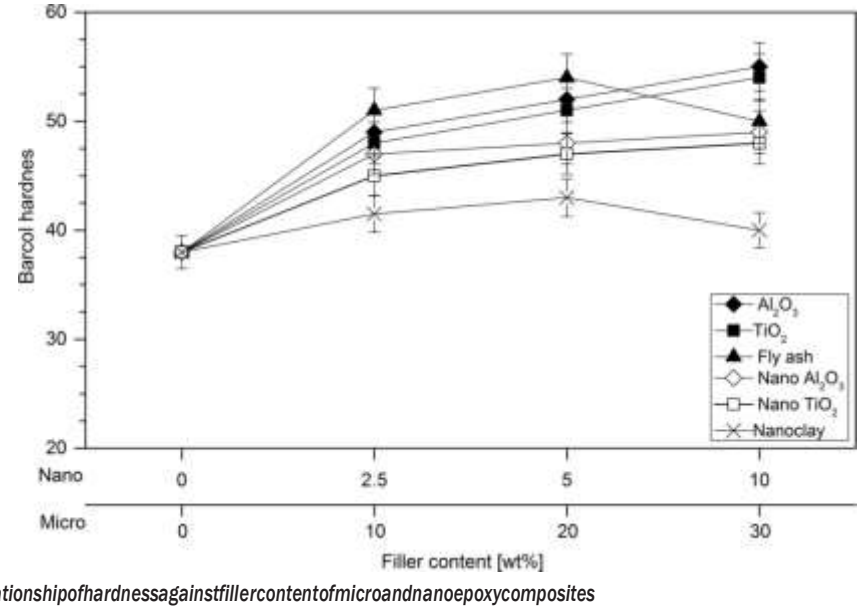


Fig.7.*Therelationshipofhardnessagainstfillercontentofmicroandnanoepoxycomposites*

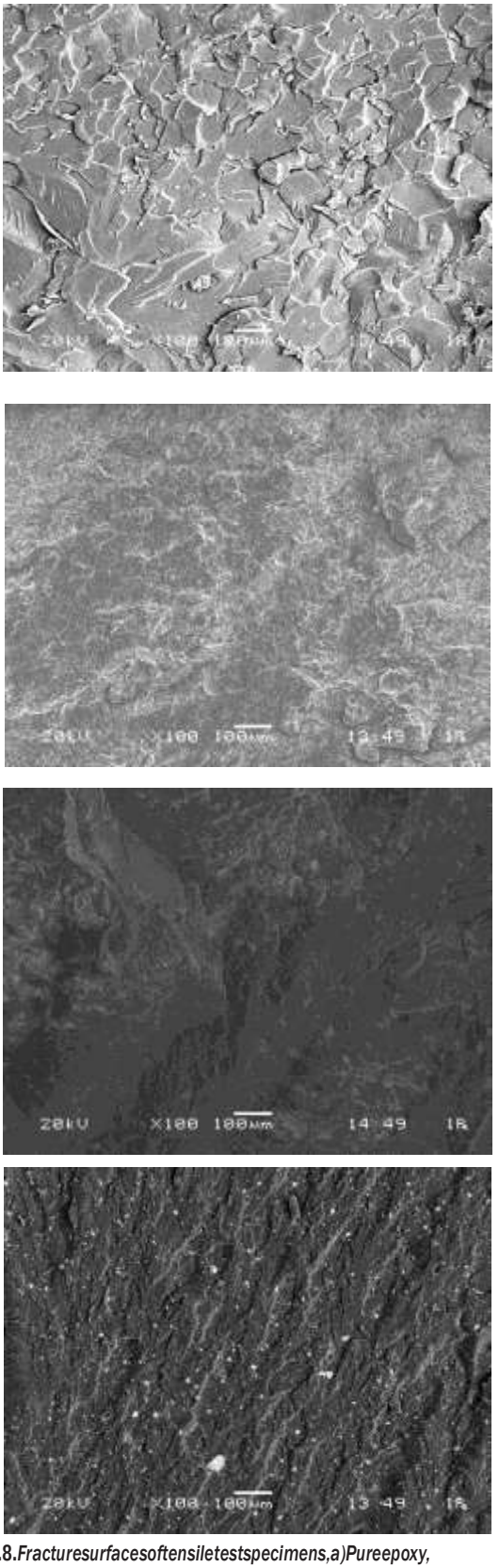


**Fig.8.** *Fracturesurfacesoftensiletestspecimens,a)Pureepoxy, b)20wt%Al2O3filled,c)20wt%$TiO_2$filled,andd)5wt%nano$TiO_2$filled*

filler ratio in the composites. For a 200 % increase inmicro $Al_2O_3$, $TiO_2$ and fly ash filler content, there aredecreasesintheelongationatbreakvaluesof72%,80% ,and87%,respectively.Inthecaseofnano-fillerfor a 300 % increase in filler content, there are 39 %,45%and78%decreasesintheelongationatbreakforna no-$Al_2O_3$,nano-$TiO_2$,andnano-claycomposites,respectively.

Fig. 7 shows the variations in the hardness ofmicro- and nano-epoxy composites with filler content.Itisclearthatthehardnessofthecompositesincrea sed withincreasingfillercontent.Again,therigidfillers increasedthehardnessoftheepoxyresin.Apart from fly ash and nano clay-filled epoxy composites,allcompositesfollowedanincreasingprofile withincreasing filler content. There is an increase of 30 %to50%inhardnessvaluewithanincreaseof200%to 300 % in filler contents.

Fig. 8 shows the fracture surface of the tensilespecimenforpureepoxy,20wt%$Al_2O_3$,20wt%Ti $O_2$and 5 wt% nano-$TiO_2$ epoxy composites. It is clearthatthefiller-filledcompositesfractureisabrittlemechanism-type failure.

## 4DISCUSSIONS

It is clear from the results that the tensile strengths of micro-filled composites decreased with increasing filler content ratios. This could be explained as the increasing filler content caused the weak adhesion between matrix and fillers and led to the decrease of the strength of the epoxy composite. In the case of nano-filler, the drop in strength is due to the non-homogeneous distribution of fillers at high filler ratios, which led to the agglomeration and caused stress concentration regions, leading to some drop in the strength. The decrease in nano-clay-filled epoxy composite strength could be explained by the agglomeration problem even at low filler content ratios.

The drop in elongation at break values of the composite with filler content could be explained as the elastic properties of the composite depend on the polymer matrix, which shows brittle behaviour in the presence of the fillers. This is because these fillers restrict the mobility of the polymer, and the higher the filler content is, the higher the brittleness of the composite is.

The decrease in the flexural strength of the composites could be explained by the agglomeration of the nano-fillers at higher ratios and by the presence of weak adhesive between the filler and matrix materials at high micro-filler contents. The increase in flexural modulus with the increasing filler content isbecause the fillers increased the rigidity and stiffnessbehaviour of the polymer composite.

Inmostcases,thefillerincreasedthehardnessof the composite. In the case of fly ash and nano-clayandahighfillercontentratio,asmalldropinhardnessv alue was observed. This could be explained by theweakness in the adhesion between the epoxy matrixandfillermaterials.Lametal.**[4]** explainedthatincreasing the filler ratio caused the increasing clusterin the nano clay filled composite and a decrease in thehardness value of the epoxy composite.

In general, it is clear from Figs. 1 to 7 that thefiller content has a significant effect and enhancementonmostofthemechanicalpropertiesofepo xycompositesbutthisisonlyeffectiveuptocertainfiller ratio levels. In the case of micro-fillers, overlyhigh levels of filler content cause a weakness in theadhesionforcebetweenthematrixandthefiller,while highlevelsofnano-fillersshowanagglomerationproblem.Hence,adroporw eaknessinthemechanicalproperties of the epoxy composite occurs.

## 5CONCLUSIONS

Fromthisstudythefollowingconclusionsarereached:

- Thetensilestrength,flexuralstrengthandelongation at break values of micro-filler $Al_2O_3$,$TiO_2$, fly ash and nano-fillers $Al_2O_3$, $TiO_2$, clayepoxycompositesdecreasedwithincreasingfill erratio.
- Thetensilemodulusandflexuralmodulusofmicro-filler $Al_2O_3$, $TiO_2$, fly ash and nano-fillers$Al_2O_3$,$TiO_2$,clayepoxycompositesincreased withincreasing filler content.

Ingeneral,thehardnessofthemicro-fillerAlO, $TiO_2$,flyashandnano-fillers$Al_2O_3$,$TiO_2$,clayepoxycompositesincreased withincreasingfiller content.

Epoxycompositesshowedbrittlebehaviourwiththeadditi on of thefiller.

Theproblemoftheagglomerationofthefillerispresent at

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higher nano-filler ratios
Theresultsareinagreementwiththeresultsreachedby[3],[4]and[13].