Synergistic Effect of Different Nanoparticles Hybridization on Mechanical Properties of Epoxy Composite

Tarek A. El-Melegy, N.S.M. El-Tayeb

Abstract— This paper is concerned with fabrication and mechanical characterization of different nano particles, i.e., Graphite (G), Silicon Carbide (SiC), and Aluminum Oxide or Alumina (Al_2O_3,) dispersed in epoxy composite in two primary scenarios. In the first scenario, the effect of dispersion of different weight fractions of single type of nano particles on the mechanical properties is investigated. While in the second scenario, the effect of hybrid dispersion of multiple nano particles is investigated. In order to optimize dispersion and particle-matrix adhesion, specimen were prepared using ultra-sonication. Experimental results demonstrate increase of 37% and 195% for $2wt\%$ of SiC dispersed in epoxy matrix in tensile strength and elastic modulus, respectively. An addition of $2wt\%$ of G shows an improvement of 85% and 32% in strength and modulus, respectively. For nanoparticle infusion of $3wt\%$ of Al_2O_3, enhancement of 132% and 200% in strength and modulus are yielded. A synergistic effect is concluded between particles of G and Al_2O_3. In this mixture, the maximum strength (56.6 MPa) is recorded at a G to Al_2O_3 ratio of 2:1. Likewise, a maximum enhancement in elastic modulus of 82% with respect to neat epoxy is recorded (0.84 GPa).

Index Terms— Epoxy, Nanoparticles, Mechanical properties, Ultrasonication, Nanoalumina, Tensile Strength

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1 INTRODUCTION AND BACKGROUND

Epoxy thermoset resins are characterized by: high stiffness, strength, low shrinkage during cure, excellent chemical resistance, dimensional stability and excellent adhesion with fillers [1],[2]. These properties have nominated epoxy resins for a range of applications such as protective coatings, medical devices, optical components, and structural composites [3],[4]. However, these applications have been limited by brittleness, susceptibility to fracture and easy crack propagation displayed by epoxy resins [2],[5],[6].

The current trend in recent years is to enhance the properties of regular engineering polymers, specifically epoxy, using various types of nanoparticle infusions. This infusion impacts various properties in polymers [7]. Nanosized particles are generally characterized by unique properties compared to larger-sized particles [8]. Some new properties arise due to use of nanosized particles (e.g. enhanced fire retardance, electrical conductivity) [8],[9]. Rahman and Padavettan explain how nanoparticles offer a large boundary surface which gives rise to properties as heat stability, reduced shrinkage, abrasion resistance and enhanced transparency [10].

The main challenge with the use of nano-sized particles to enhance epoxy resin properties is the achievement of sufficient and uniform dispersion of particles into the matrix in order to prevent agglomeration of particles for superior particle-matrix interface adhesion and interaction [11].

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Various techniques have been used in order to achieve adequate dispersion of nanoparticles into epoxy matrices. The techniques may be divided into mechanical and chemical techniques. Mechanical techniques include homogenization [12], disc milling, kneading, 3-roller milling (Calendaring) [13], ball milling [9], probe sonication [14], bath ultrasonication [15]. Whereas, chemical methods involve: in-situ polymerization of resin in the presence of nanoparticles [16], nanoparticle surface treatment using silane coupling agents [17],[18]. Functionalization of nanoparticles such as Carbon Nanotubes (CNTs) has been shown to produce enhancement in mechanical properties of epoxy based polymer composites [19].

Various researchers have shown that nanoparticle or CNT dispersion into polymer composites can enhance mechanical properties. Faleh, et al. [4] studied the effect of different weight fractions of multi-walled carbon nanotubes (MWCNTs) and silicon carbide nanopowders (SiC) on adhesive epoxy used in retrofitting structures (Araldite-420 and Sikadur-30 resins). This study showed that addition of 2wt% of MWCNTs to epoxy exhibited an improvement of about 17% in ultimate strength and 14% in stiffness of epoxy composite. On the other hand, addition of 1wt% of SiC to the epoxy gave 50% improvement in stiffness. One a different note, the authors noted a slight decrease in both strength and ductility and a boost in elastic modulus upon dispersing the nanoparticles by Ultrasonic mixing (UM) at 2-3 wt%, in the absence of nanoparticles.

Other researchers noted mean increase of 20-30% in mechanical properties as well as enhanced fatigue performance is obtained with only 1.5 wt% nano-silicon carbide (SiC) as deduced by Chisholm et al. [20]. Zhu et al. [21] report an increase in Ultimate Strength of 30-70% in functionalized SWCNT-reinforced epoxy composite with the addition of only 1-4 wt% of nanotubes.

A few researchers have extended the research so as to

investigate the synergistic effect of combining ranges of weight fractions of different types of particles on the mechanical properties. This effect has been observed in microscale filler combinations [22],[23] as well as in micro and nano scale filler combinations [24],[25]. Liu,et al. studied the synergistic effects of silica and rubber hybridization on Mode I fracture toughness (G_{IC}) and Stiffness (E) of epoxy-based composites [7]. A stiffness increase of 22% is noted with 20 wt% nanosilica. However, a 24% decrease is yielded for 15wt% of nanorubber. Combining both particles has not yielded any significant synergistic effects. It is worth noting this area of study requires further investigation into a wider range of hybrid systems of nanoparticles dispersed into epoxy composites.

Therefore, the objective of this paper is to select an adequate method for sufficient dispersion between the nanoparticles and the matrix to ensure adequate interface adhesion. Moreover, we aim to study mechanical effects due to dispersion of various types of ceramic nanoparticles into epoxy matrices, individually. Further, the study is extended to include the hybrid effect of combining types of nanoparticles together into the epoxy matrix in a multi-phase composite system. We aim to demonstrate the possibility of a synergistic effect.

2 MATERIALS AND METHODS

KEMAPOXY 150 is a local commercial epoxy that was purchased from Chemicals for Modern Buildings (CMB) [26]. It consists of resin A and hardener B. It is known for its adequate chemical resistance as well as mechanical strength. It is non-toxic upon curing as it is used in applications of: water tanks, food containers, repair mortar for concrete structures and protective coating for concrete floorings [26].

To study the effect of nanoparticles, three varieties of nanoparticles were acquired: Graphite (G), Silicon Carbide (SiC) and Aluminium Oxide (Alumina) (Al_2O_3). The particles were initially procured as micro-sized particles and ground to an average size of $50 - 70$ nm at the Centre of Metallurgical Research and Delmont Institute (CMRDI) [27]. The properties of the acquired particles are tabulated below:

TABLE 1

NANOPARTICLE REINFORCEMENT PROPERTIES [27].

Particles	Density (g/cm3)	Ultimate Tensile Strength (MPa)	Melting Temp. (°C)	Young's Modulus(GPa)
SiC	3.21	3440	2700	450
G	2.27	960	3500	150
Al2O3	3.99	1900	1740	435

Nanoparticles were proportioned into different weight fractions. A composition matrix was prepared including two sets of experiments. The first set comprises the dispersion of a single type of nanoparticles in different weight fractions (1%, 2 %, and 3%) into the epoxy matrix forming a binary-phase composite system. The second set comprises the dispersion of multiple nanoparticle types together in different weight fractions to form a multi-phase composite system. Compound B is weighed and then the nanoparticles are weighed and hand mixed into compound B because it is less viscous than compound A. The hand mixing is done for about $2 - 3$ mins. A flat stir piece was used to reduce gas entry into the mixture. The mix is shown below:
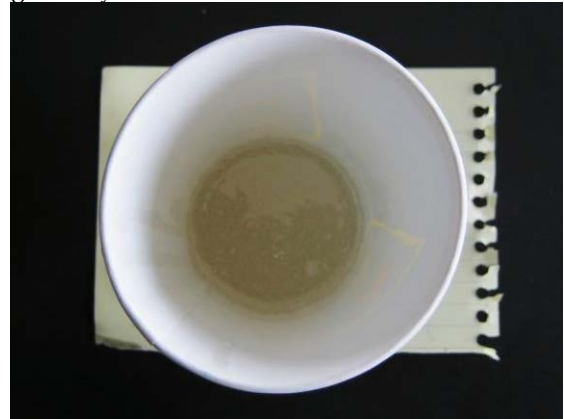

Fig. 1: Alumina particles mixed in Compound B Epoxy.

Afterwards, the mixture is sonicated in an Ultra-sonication Cleaner Bath (manufactured by FUNGILAB S.A) as shown in Fig. 2 The sonication is done at room temperature in 2 minute intervals with 10s breaks for a total of about 20 minutes. The mixture should not exceed 50°C to prevent modification of the chemical structure of both the nanoparticles and compound B of the epoxy. Therefore, the temperature was monitored and maintained in a water bath at $24 - 37$ °C.

Fig. 2: Ultra-Sonication Bath and Alumina Specimen (FUNGILAB S.A)

The mixture is then removed from the sonication and allowed to cool while Compound A is weighed. Compound A is weighed at the recommended ratio Of (A:B = 1:2). Sonication is continued for about 12 mins.
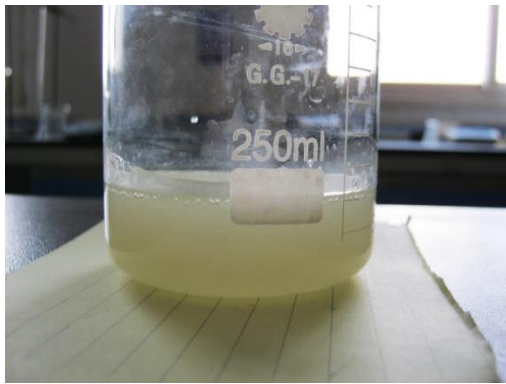
Fig. 3: Alumina-Epoxy prepared mixture

The mixture may be left for 1 min to cool off and release some of the entrapped bubbles as shown in Fig. 3. The mixture is then poured into a steel mold designed to accommodate 3 specimens. Specimen dimensions and geometry were specified as per ASTD D 638 [28]. The overall dimensions are: L 165 × W 19 × T 7 mm as shown below:
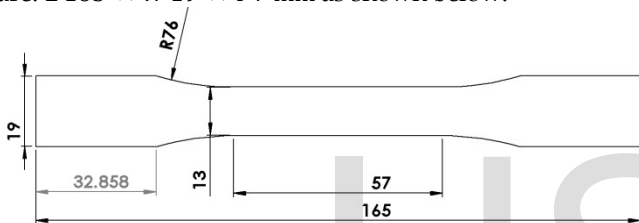

Fig. 4: Schematic Dawing of specimen.

The specimens are poured into the mold slowly and left to cure for 1 − 2 days. The specimen are then removed and left to reach full hardness and strength in about 5 more days (7 days total).

3 RESULTS AND DISCUSSION

The specimens were removed from the mold using a machined steel specimen (Fig. 5) with a taper to push the specimen out of the mold, and the extra epoxy due to meniscus formation was ground. A sample of finished specimen are shown in Fig. 6.

Fig. 5: Steel Mold and Dummy Steel Specimen

The samples were tested using a Universal Testing Machine (UTM) at a rate of 2 mm/min at room temperature. The deformation was recorded until failure (Fig. 7) using the stroke values produced by the machine. An attempt to use a strain gauge attached the specimen surface was concluded unsuccessful as the brittle fracture of epoxy would have been harmful to the strain gauge. At least three specimens were tested and the results were averaged.

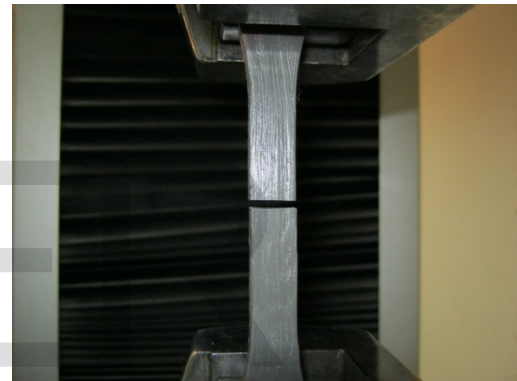Fig. 6: 1wt% G and 1wt% Al_2O_3 nanoparticle reinforced epoxy composite specimen.

Fig. 7: Fractured 1wt% SiC-nanoparticle reinforced epoxy composite specimen.

3.1 SiC-REINFORCED EPOXY

The Stress Strain curves resulting from testing various weight fractions of SiC reinforced epoxy composite of weight fractions from 1-3 wt% are shown in Fig. 8. The recorded tensile strength of the pristine epoxy is about 26 MPa. An elastic modulus of 0.46 GPa was deduced. The addition of 1wt% of SiC leads to improvement of (14%) in Strength and (84%) in Stiffness. Whereas, break strain decreases by (−40%) compared to neat epoxy. The addition of hard nano-SiC particles of strength about 3440 MPa acts as slip barriers for dislocations which oppose the deformations brought about by the load cell of the machine. For this reason, an increase in strength is observed with added particles. Additionally, the particles are hard and have a modulus of about 450 GPa. Added Stiffness therefore is also expected. Reduced ductility is indicated by the reduced strain at break owing the slip barrier event mentioned earlier.

Addition of 2wt% of SiC results in a greater enhancement in strength (37%) corresponding to 35.5 MPa is yielded. Conversely, there is a slight increase in Elastic Modulus (19%) . This effect is less than that observed in 1wt% of SiC. The observed Elastic Modulus Value is 0.55 GPa. A (10%)

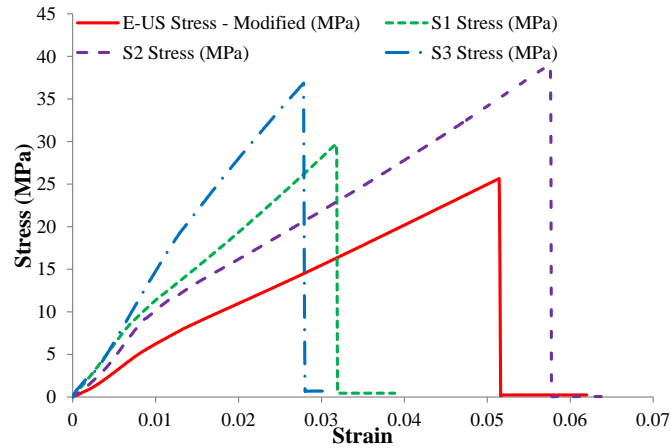increase in break strain is also noted.

Fig. 8: Effect of SiC dispersion on tensile properties of KEMAPOXY 150.

Addition of 3wt% of SiC results in an improvement in Strength over neat epoxy by (36%) yielding a value of 35.4 MPa. This value is slightly less than that of 2wt% SiC result.On the other hand, an exceptional improvement in Elastic Modulus is observed. (195%) improvement over neat epoxy with a value of 1.36 GPa is noted. This composition represents an opportunity of special interest. The reason for this enhancement should be investigated. Environmental and other testing conditions during this test should be further investigated. A decrease in break strain of (−48%) is observed.

3.2 G-REINFORCED EPOXY

Similar tests were conducted for graphite infused epoxy of particle weight fractions from 1-3 wt%. The results are shown in the Fig. 9. Adding about 1wt% G nanoparticles into the epoxy yielded a tensile strength of about 31.7 MPa. This constitutes an improvement of about (22%) compared to neat epoxy. Further, it is observed that there is an actual increase in elastic modulus of the carbon nanoparticle infused epoxy compared to neat epoxy. The elastic modulus obtained is 0.6 GPa indicating an enhancement of 31% over neat epoxy. The break strain achieved in this case is reduced from that of neat epoxy by (−38%). This is a peculiar finding that should be further investigated. However, this can be attributed to the brittle nature of the carbon nanoparticles which affect the properties of the overall composite. Note, however, that this decrease in strain is less than that exhibited by infusing 1wt% SiC.

The addition of 2wt% of G indicates further improvement in tensile strength (85%) yielding a value of 48.1 MPa. This improvement is greater than the previous specimen with 1wt% of G. Whereas, the improvement in elastic modulus seems to be similar in value to that with 1wt% of G, that is a (31%) improvement of value 0.61 GPa. Note however the increase in Break Strain by (54%) compared to neat epoxy.

The addition of 3wt% of G shows improvement in tensile

strength of (81%) over neat epoxy with a value of 47.1 MPa. This is however reduced improvement compared to the sample containing 2wt% of G. Furthermore, the improvement in elastic modulus seems to be relatively unchanged at (32%) and 0.61 GPa. Lastly, there seems to be an even greater increase in strain at break with increased percentage of (61%).

3.3 ALUMINA Al_2O_3-REINFORCED EPOXY

The results for addition of 1-3 wt% of Alumina into epoxy matrix are displayed in Fig. 10. The addition of 1 wt% of Al_2O_3 into epoxy leads to an exceptional improvement over neat epoxy in strength. This improvement is about 112% or double the strength with a value of 55.14 MPa. There is also noted improvement in elastic modulus of 100% over neat epoxy with a value of 0.92 GPA.
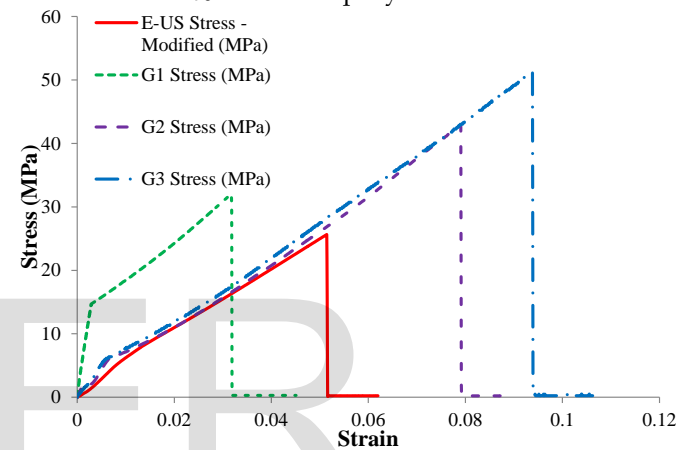

Fig. 9: Effect of G dispersion on tensile properties of KEMAPOXY 150.

Additionally, the sample withstands more strain before fracture with a break strain improvement of 21%. This indicates greater tensile toughness compared to neat epoxy. Alumina nanoparticles have a strength of 960 MPa and stiffness of 435 GPa. Therefore, enhancements are expected in both strength and stiffness.

Addition of 2 wt% of Al_2O_3 into epoxy leads to reduced improvement than before.
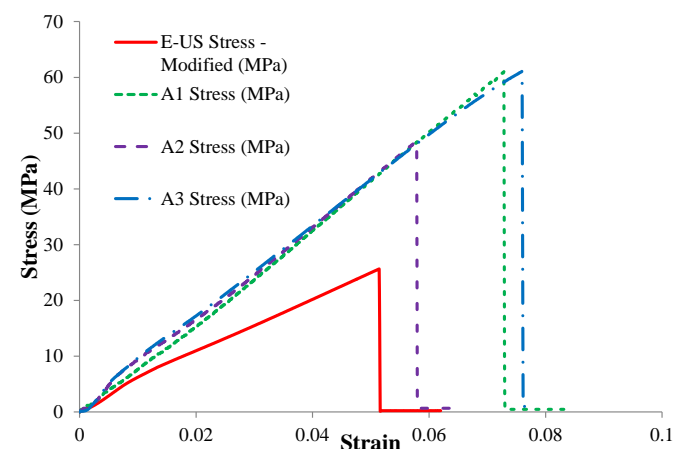

Fig. 10: Effect of Alumina dispersion on tensile properties of KE-

MAPOXY 150.

An improvement of (76%) with strength of 45.9 MPa and an improvement of (84%) in elastic modulus with a value of 0.85 GPa. Moreover, the break strain has a (7%) improvement indicating greater toughness than neat epoxy. However, this toughness is less than that of 1 wt% of Al_2O_3 as indicated by the areas under the curves shown above. Infusing 3 wt% of Al_2O_3 into epoxy yields the greatest improvement in strength of (132%) with magnitude of 60.3 MPa. Nevertheless, the improvement in elastic modulus is reduced than the previous formulations to just (76%) enhancement with a value of 0.81 GPa. Whereas, the greatest improvement in break strain of (51%) is observed. The graphs also indicate that this composite formulation yields the highest tensile toughness.

The results for the various nanoparticle reinforced epoxies are have been combined and charted in for different weight fractions in order to compare the enhancements yielded due to nanoparticle infusion. A general trend in strength enhancement can be deduced. The increase in particle weight fraction increases the strength enhancement over neat epoxy from $1-3$ wt% of any nanoparticle. An exception is noticed at 2 wt% Al_2O_3 where we see lower enhancement in strength (76%) than that due to 1 wt% Al_2O_3 which is at (112%). Further, maximum enhancement in strength over all nanoparticles is noticed at 3 wt% Al_2O_3 of 132% over neat epoxy
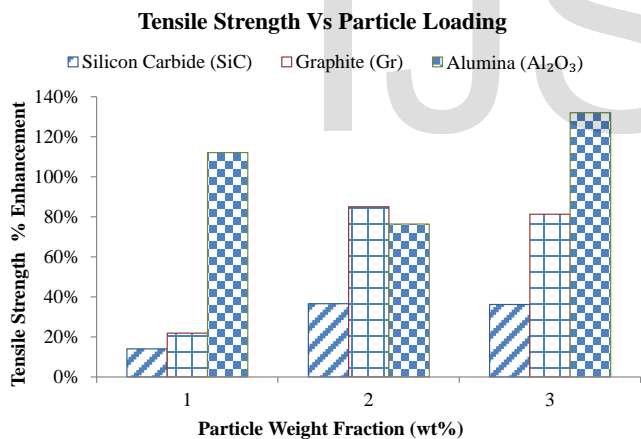

Fig. 11: Enhancement in Tensile Strength due to individual nanoparticle infusions in epoxy matrix.

Conversely, it can be noted that the least increase in strength is obtained at 2 wt% Gr of 22%.

Similar charts were plotted for enhancement in break strain due to the individual nanoparticle infusions in Fig. 12. The greatest enhancement in break strain is obtained at 3 wt% Al_2O_3 of 61% over neat epoxy. On the other hand, the curve shows actual deterioration in break strain in certain cases, compared to neat epoxy. The lowest value for break strain is obtained at 1 wt% SiC of -46% compared to neat epoxy.

It is also worth noting that addition of 2 wt% SiC is the only case that causes enhancement (10%) for SiC nanopar-

ticle infusions. The minimum improvement in break strain is obtained at 2 wt% Al_2O_3 of 7% compared to neat epoxy. A sample of the fractured specimen is collected in Fig. 13.
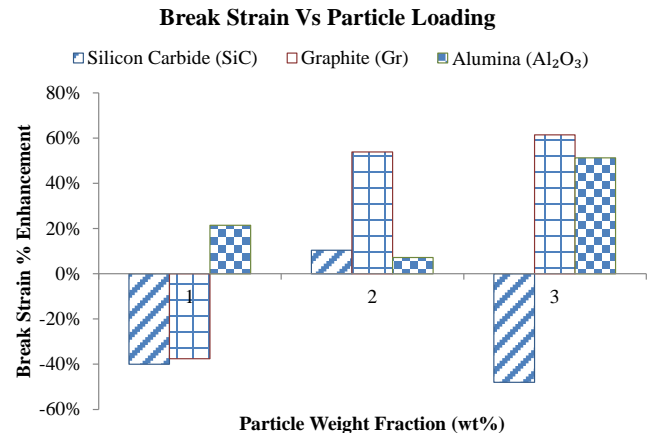

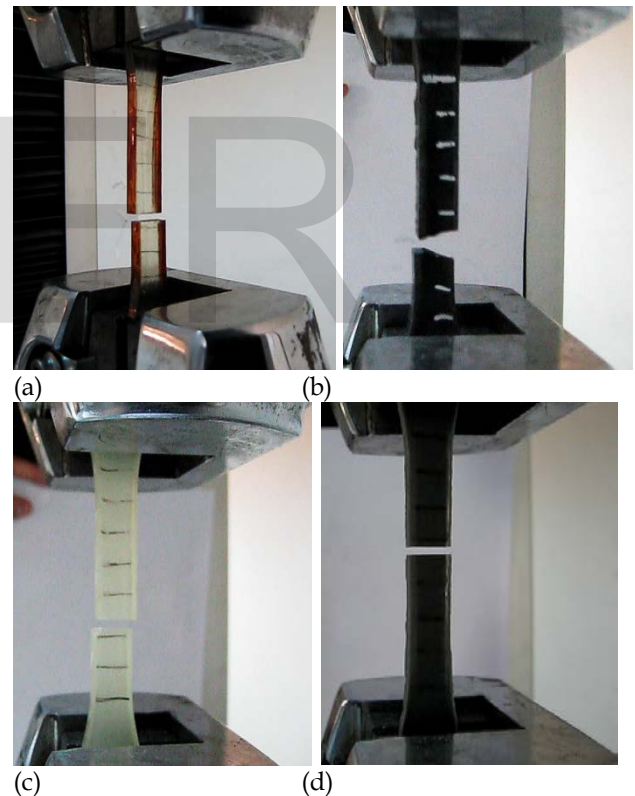Fig. 12: Enhancement in Break Strain due to individual nanoparticle infusions in epoxy matrix.

Fig. 13: Fractured Specimen on the Tensile Machine. (a) Neat Epoxy. (b) Graphite reinforced. (c) Alumina reinforced. (d) Silicon Carbide reinforced.

4 CONCLUSION

In this research, the effect of infusion of nanoparticles of Silicon Carbide SiC, Graphite G, and Alumina Al_2O_3 on the mechanical properties of commercial epoxy, KEMAPOXY

150 are investigated experimentally. Initially, the effect of individual nanoparticle dispersions is investigated individually when infused in epoxy. More importantly, the effect of hybrid/combined infusions of multiple nanoparticle types on the mechanical properties is investigated. Consequently, an adequate dispersion procedure was developed to maximize homogeneity of dispersions and consistence of results. This required the use of various resources and apparatus including: high precision scaling, and ultra-sonic mixing. The main results of the research are listed below:

- When nanoparticles of SiC are investigated individually, a maximum enhancement in tensile strength is observed for 2wt% of SiC. This enhancement (29.65 MPa) is 37% compared to neat epoxy (26 MPa). Similarly, maximum enhancement in elastic modulus is observed at 3wt% of SiC of 195% improvement 1.36 GPa over neat of 0.46 GPa.

- When nanoparticles of G are investigated individually, a maximum boost in tensile strength is also noted at 2wt% of Gr . This enhancement is (48.105 MPa) is 85% compared to neat epoxy. However, all weight fractions $(1 - 2 - 3 \, wt\%)$ of G exhibit almost the same improvement (0.61 GPa) in elastic modulus of 32% over neat epoxy.

- When nanoparticles of Al_2O_3 are investigated, a recorded maximum tensile strength (60.3 MPa) is observed at 3wt% of Al_2O_3 with enhancement of 132%. The elastic modulus is doubled (0.92 GPa) compared to neat epoxy at 1wt% of Al_2O_3, which is the recorded maximum.

The results from this research give more insight about the enhancement ability of epoxy in order to formulate nanoparticle infused composites more suited for structural applications.

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