Influence of Some Nano-Inorganic Oxides on the Mechanical Properties of Epoxy Based Nano Composites

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Abstract. Epoxy nano composites were prepared using CuO, Fe$_2$O$_3$, and MgO as additives. Some mechanical properties (Tensile strength, Elongation%, Impact and Hardness) were measured. These additives are capable to improve the mechanical properties for the resulting nano composites (in low percentages) such as tensile, impact, and hardness, while at the higher percentages the mechanical properties were reduced due to the agglomeration of the additives.

Key words: Epoxy Nanocomposites, Additive, Inorganic oxides.

1. Introduction

Materials that are needed for aerospace, underwater, and transportation applications are made of unusual combinations of properties. (1) These materials are called composites in which the characteristics of the finished product can be tailored to a specific engineering requirement by the good selection of matrix and the reinforcement type. (2, 3)

Composites have many advantages, e.g. their high strength and stiffness, low density, when compared with bulk materials, allowing for a weight reduction in the finished product. (4) Nanocomposites materials have emerged as suitable alternatives to overcome limitations of microcomposites and monolithics. (5)

Epoxy resins are thermosetting polymers easy to process, with high-performance, containing at least two epoxy groups per molecule. (6) They have been used extensively as high performance adhesive composite materials, due to their outstanding mechanical and thermal properties such as tensile strength, low creep, high thermal stability, and moisture resistance. (7) Epoxy nanocomposites demonstrate some advantages in both mechanical and dielectric properties compared with pure resin system and epoxy with micrometer-size fillers at a lower loading concentration. (8) The performance of the epoxy can be further improved by the use of fillers according to a unique application. (9)
2. Experimental Part

2.1 Chemicals

Epoxy resin type (Sikadur 52 A) was provided by Sika company located in Turkey, the hardener used was (Sikadur 52 B) with ratio of 2:1. Metal oxides nanopowder supplied by US Research Nanomaterials, Inc. company, which are magnesium oxide (MgO) has extra small size (40 nm) with specific surface area (SSA = 25 m$^2$/g), iron oxide (Fe$_2$O$_3$) is (20-40 nm) specific surface area (SSA = 40-60 m$^2$/g) and copper oxide (CuO) (25-55 nm) specific surface area (SSA: 13.98 m$^2$/g).

2.2 Preparation of Nanocomposites Specimens

Nanocomposites specimens were prepared using In-Situ polymerization method, glass template was prepared with dimensions (130×130×3) mm to be used for shaping the samples, the nano additives are added with different percentages (2, 3, 4, 5 and 6 %wt) to the epoxy resin gradually in a beaker and mixed manually using glass rod for 10-15 minutes. Homogeneous blend was obtained when the well mixed component were put in an ultrasonic bath for 23 minutes at 40˚C. The hardener was added and mixed well for 3 minutes and the mixture was molded in the glass template then left at room temperature for enough time to dry. The resulted sample was put in an oven at 50˚C for one hour then left for 2 weeks to complete curing. The samples were cut into multiple pieces based on testing devices requirement. The above process applied for all the percentages of each additive.

3. Results and discussion

3.1 Results of Tensile Properties According to ASTM: D638

Tensile strength is one of major tests been used to determine the enhancement in the mechanical properties for the nano-composites considering different percentages and types of additives. The analysis of stress and strain is done and determined with the tensile test from the measured load and deflection using the original specimen cross-sectional area.$^{(10)}$ Tensile strength of neat epoxy resin and its nanocomposites are tested and evaluated according to ASTM:D638. The results are listed in Table (1).

As the percentages of additives increase the tensile strength start improving up to 3 wt% of the additives from 25 MPa for the blank epoxy to 26.5 MPa in the case of 3 wt% CuO, 26 MPa for 3 wt% of Fe$_2$O$_3$ and MgO, while the tensile strength reduced as the percentage exceeds 4 wt% (degraded to 20 Mpa when
additive percentage increased to 6 wt%), this is due to accumulation of the additives above 3 wt%.

The tensile strength of nanocomposites, prepared from various polymers and inorganic particles, do not always increase. In some cases, the properties of nanocomposites are decreased by the addition of inorganic particles because of aggregation in polymer matrices at certain percentages (i.e. above 3 wt %). (11)

The optimal performance of nanocomposite may be achieved by proper dispersion of nanofillers in the resin. The dispersion of different nanomaterials into the epoxy matrix has been investigated by many researchers. (12, 13)

Table 1: Tensile strength values (Mpa) of epoxy nanocomposite

| Additive Type | Blank Epoxy | CuO | Fe₂O₃ | MgO |
|---------------|-------------|-----|-------|-----|
| Additive %    |             |     |       |     |
| 0             | 25.0        |     |       |     |
| 2             | 25.5        | 25.2| 25.5  |     |
| 3             | 26.5        | 26.0| 26.0  |     |
| 4             | 26.0        | 25.5| 25.7  |     |
| 5             | 24.0        | 23.0| 24.0  |     |
| 6             | 20.0        | 22.1| 22.0  |     |

Figure 1: Tensile strength results of epoxy nanocomposite.
After stirring the additives with epoxy, at high additive content more than 4 wt% the viscosity of nanocomposite series increased and is much more difficult to remove voids inside the aggregate in these samples.

### 3.2 Percentage of Elongation

The elongation characteristics are due to the fact that suitable microspores are formed to absorb the stress when the damage occurs and this effort increases the durability of the material.\(^{(14)}\) From the results of the elongation at break, it should be noted that the slight increase in ductility obtained for all the nanocomposite formulations indicating that the addition of the nanofiller does not induce any matrix fragility but increases its yielding behavior.\(^{(15)}\) The results of elongation-at-break are summarized in Figure (2) and the measurements are illustrated in Table (2). The values of elongation-at-break are found to be maximum in 3 wt% of nano-content (CuO, Fe\(_2\)O\(_3\), and MgO,) which are (7.54, 7.84, and 8.1 respectively). The increase of elongation-at-break might be due to the filling of nano particles into the amorphous region of matrix via uniform dispersion up to 3 wt% of nano additives. When the amorphous region of epoxy matrix had been completely filled by the nano particles, the sample achieved maximum tensile strength due to the load shearing by nano particles. The nano composite samples also showed the toughening behavior on addition of nano filler for the same composition, which led to enhance the elongation-at-break values. Beyond 3 wt% addition of nano additives in the epoxy matrix led to the agglomerate. This might be resulted the decrease in the tensile strength and the elongation-at-break.\(^{(16)}\)

| Additive % | Additive Type | Epoxy Blank | CuO | Fe\(_2\)O\(_3\) | MgO |
|------------|---------------|-------------|-----|---------------|-----|
| 0          |               | 6.9         |     |               |     |
| 2          |               | 5.70        | 6.37| 7.90          |     |
| 3          |               | 7.54        | 7.84| 8.10          |     |
| 4          |               | 6.32        | 5.82| 5.80          |     |
| 5          |               | 4.91        | 5.47| 5.00          |     |
| 6          |               | 4.19        | 5.25| 3.90          |     |

Table 2: Elongation % values of epoxy nanocomposites
3.3 Results of Impact Test According to ASTM: D256

Impact test is a method for determining behavior of materials when subjected to shock loading in bending, tension or torsion. Figure (3) shows impact properties of epoxy nanocomposites with different percentages of nano additives.

Increasing percentage of filler had improved the impact strength of epoxy resin; highest impact strength is observed at 3 wt% of (CuO and Fe$_2$O$_3$) and at 4 wt% of (MgO) which is (13.6 and 13.58) KJ/m$^2$ and (10.6) KJ/m$^2$ respectively as shown in Table (3) which contains the results for impact strength testing for the three different additives at variable percentages.

Further loading of nano additives in resin matrix led to a decrease in impact resistance. It is revealed that large quantity of nano filler leads to the agglomerate and caused the deterioration of toughness of cured epoxy nanocomposite films. It is observed that nano additives exhibit reinforcing capacities when added in small quantity. The improvement in the impact resistance might be due to load sharing by the nano particles and the intermolecular force of attraction between macro molecules of resin and nano particles due to hydrogen bonding, which enhanced the toughness of resin matrix at fully cured stage.
Table 3: Impact strength testing results (KJ/m²) for the epoxy nanocomposites

| Additive Type  | Additive % | Epoxy Blank | CuO | Fe₂O₃ | MgO |
|---------------|------------|-------------|-----|-------|-----|
| Epoxy Blank   | 0          | 7.69        |     |       |     |
| CuO           | 2          | 5.12        | 5.10| 7.60  |     |
| Fe₂O₃         | 3          | 13.60       | 13.58| 8.30 |     |
| MgO           | 4          | 8.97        | 8.33| 10.60 |     |
|               | 5          | 10.20       | 3.56| 9.30  |     |
|               | 6          | 6.94        | 3.50| 6.90  |     |

Figure 3: Results of impact strength testing (KJ/m²)

3.4 Results of Hardness Shore D

Hardness is a measure of a material’s resistance to localized plastic deformation (e.g., a small dent or a scratch). Early hardness tests are based on natural minerals with a scale constructed solely on the ability of one material to scratch another that is softer. \(^1\)
The effect resultes of nano particulates on the hardness of epoxy nano composites with respect to the varying concentration of nano additives are shown in Figure (4) and the experimental observation are shown in Table (4), from which it can be easily assessed about the trends of the hardness achieved by nano composite samples. That is, as the nano particles loading increased in the nano composite samples, the hardness Shore D value also increased and observed maximum value in the sample epoxy nanocomposites with filler loading of 3 wt% from CuO and MgO then start decreasing as the percentage increased, while it keeps increasing as the percentage of nano additive increased for Fe$_2$O$_3$. Hence, the samples with higher concentrations of nano particles show higher resistance to the indentation force therefore exhibiting higher Shore D values. 

This could be attributed to the inherent hardness of the additive particles compared with neat epoxy, which affects the overall hardness of prepared nano composite samples and become more prominent at higher concentration. But as soon as the loading of nano filler increased beyond 3 wt% of CuO and MgO the hardness value decreased. The decrement in the hardness could be attributed to the floccules of nano filler and developed a perforation in confined region within the nano composites. \(^{(16)}\)

| Additive Type | Epoxy Blank | CuO | Fe$_2$O$_3$ | MgO |
|---------------|-------------|-----|-------------|-----|
| Additive %    |             |     |             |     |
| 0             | 64.2        |     |             |     |
| 2             |             | 70.50 | 67.80      | 70.60 |
| 3             |             | 74.80 | 68.60      | 74.60 |
| 4             |             | 72.40 | 72.60      | 71.00 |
| 5             |             | 69.60 | 69.00      | 73.00 |
| 6             |             | 71.20 | 73.40      | 68.80 |
4. Conclusions

- The three nano additives are effective in improving the mechanical tensile, impact, hardness and elongation properties due to good dispersion of them into the epoxy resin especially at 3%.
- The nano additive percentage beyond 4% reduced most of the properties due to agglomeration of nano particles.

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