Comparison of Mechanical and Thermal Properties of Graphene-Epoxy Nano-Composite and Epoxy Resin

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Abstract: There has been growing interest in graphene and its remarkable properties since its discovery. Many attempts have been made to use graphene as a reinforcement material in composites rather than a bulk material; this is because of its high cost and manufacturing limitations. This paper speculates the use of Graphene powder to enhance properties of Epoxy Resins. It focuses on highlighting the development and basic construction of various types of composites and it presents the findings of an experimental development of Epoxy Nano-Composite reinforced with Graphene powder and also compares the result with non-reinforced Epoxy test samples. A detailed procedure is provided and conclusions are drawn on the interpreted results. Some suggestions, to get better results, are also highlighted.

1. Introduction

Graphene, a single layer thick sheet of carbon atoms, has sparked a lot of interest in the world of materials. This is because of its extremely good properties of thermal conductivity, electrical conductivity, mechanical strength and its light weight. The current manufacturing processes make direct application of this material unfeasible due to high costs. Thus it has been the efforts of many people to use graphene as a reinforcement in composite materials. Composite materials will take advantage of the strength and light weight characteristics of graphene while maintaining cost.

Graphene in its nano-powder form can be readily mixed and dispersed in polymer matrix. Epoxy, a type of a thermoset polymer, is widely used for making polymer composites. The aim of this paper is to demonstrate the properties of the enhanced Epoxy Nano-composite, reinforced with graphene in a given percentage, and to compare the percent enhancement with respect to non-reinforced Epoxy samples.

2. Composites Classification

Advanced Composites can be generally defined as a combination of two or more elements in a particular arrangement and ratio, such that the properties of the resultant material are enhanced as desired by the application. Composites, theoretically, are and can be of any formations and can be made with any number of elements. However, practically a few materials are used in conjugation with a variety of fillers or reinforcements to produce a wide variety of composites.

Composites, in its most simple form, contain two elements: a bulk material called the matrix and a reinforcing material. Depending on the type of matrix used, modern day composites can be divided into three major categories: Polymer matrix composites (PMCs), Metal matrix composites (MMCs), and Ceramic matrix composites (CMCs).

- Polymer Matrix Composites (PMC’s) – These are the most common and will be discussed here. Also known as FRPs (Fiber Reinforced Polymers) these materials use a polymer-based resin as the matrix, and a variety of fibers such as glass, carbon and aramid as the reinforcement.
- Metal Matrix Composites (MMC’s) - Increasingly found in the automotive industry, these materials use a metal such as aluminum as the matrix, and reinforce it with fibers such as silicon carbide.
- Ceramic Matrix Composites (CMC’s) - Used in very high temperature environments, these materials use a ceramic as the matrix and reinforce it with short fibers, or whiskers such as those made from silicon carbide and boron nitride.

3. Properties of Constituent Materials

In order to develop a Nano-composite with Graphene as a reinforcement element, Epoxy Resin was selected as a polymer matrix. It is one of the most widely used polymers in the industries and it has excellent mechanical properties. It further also has good curing time and properties. The following is the resin and hardener used: Epoxy Resin (Araldite LY 556\textsuperscript{*}), Hardener (Aradur HY 951\textsuperscript{*}). Graphene has remarkable mechanical properties because of its 0.142 nm long carbon bonds it shows an intrinsic tensile strength of around 130 GPa and a Young’s modulus of about 1TPa. It also has excellent force
4. Methodology

The method adopted while developing the Graphene based Epoxy composite, was devised after extensive research on various literatures which cited the properties and behavior of Graphene particles during different methods of manufacturing. A few major parameters were identified for good quality of composite: homogenous dispersion of Graphene in the epoxy resin, curing temperatures and post curing process parameters like temperature, percentage of Graphene by weight and the type of resin used. After careful analysis of different Epoxy resins and hardeners, and considering the desired applications of the thus developed composite (i.e. for aerospace structural applications) the combination of the resin Araldite® LY 556 and hardener Aradur HY 951 was used. This particular combination is widely used for industrial and structural applications. It further has good water resistant properties and forms excellent fiber composites due to its chemical structure and adhesive properties.

Expected increase in mechanical properties of the polymer by addition of Graphene was postulated. Since the composite was a particle reinforced composite, Research Grade Graphene was procured in the powder form. Agglomerated particles were observed and that was attributed to exposure to moisture. Hence, the required quantity of Gr powder (approximately 2.03 grams since 0.2% of Gr powder by weight was taken as the addition ratio) was weighed and placed in a crucible. The crucible was then placed in an oven (Memmert UF 450 Plus) for 3 hours at 160 °C. Afterwards weight was taken again and the dry Gr powder weighed at 1.8333g. Thus, moisture was removed and Gr powder was stored in an air tight packet till further use.

To breakdown all clumped dry particles and to obtain a uniform sized powder, the Gr powder was sieved in a sieved shaker with a sieve of hole-size 38 μm (microns). The shaking was done for 10 minutes with 10 second intervals at 1.5 mm amplitude of vibration. The collected powder was immediately stored in an air tight packet to avoid contact with moisture. The remaining coarse powder was mechanically grinded and sieve again with the same procedure.

Approximately, 300-400 ml sample of resin and hardener was required to form various test specimens. Respectively, 300.43 g of Resin was poured into a beaker. A 0.2 % concentration by weight of Gr powder was to be added as reinforcing material to the polymer. Accordingly, 0.6013g of Gr powder was measured and poured into the beaker containing the resin. Slight stirring is necessary while addition of Gr powder in order to avoid lumping.

After complete addition, the beaker was placed under a mixer and the resin was mixed using a standard vertical axis mixer (IKA RW20 Digital) at 265-270rpm for one hour. After the mixing, homogeneity was checked using visual inspection by dipping a glass rod and checking for lumps and uniformity in color and opacity to ensure uniform dispersion.

Molds—for forming the test specimens for tensile testing, compression and modulus testing and impact testing—compliant with ISO standards for test specimens were used. The entire mold assembly was cleaned with acetone and mold release agent was applied.

As mentioned in the data sheet of the resin used, the required proportion of the hardener was poured into a beaker and added to the resin and Gr powder mixture to start the reaction. The mixture was hand mixed thoroughly using a glass rod. Once the required consistency was obtained, the mixture was poured into different molds. An approximate gel time of around 30-35 minutes was observed. The castings were cured at room temperature (28 degree Celsius) for 24 hours. The molds were then moved into a hot air oven for post curing at 160 degree Celsius for 2 hours. After post curing the molds were removed and allowed to cool down till they reached room temperature. Test specimens were then taken out by slight tapping and stored in an air tight plastic bag till further testing began.

Standard procedures were followed for tensile, compression and hardness testing. DSC (To find Glass transition temperature) was also conducted to check the increase in thermal properties of the composite such as resistance to heat and creep etc. All results were noted and compared with the previously found values of the polymer and resulting increase or decrease % was calculated.

5. Comparative Results


The figures below show the graphs of all the tests conducted as per described.
Fig. 1: Tensile Test

Fig. 2.1: Compression Test

Fig. 2.2: Compression Test

Fig. 3.1: DSC(Glass Transition Temperature)

Fig. 3.2: DSC(Glass Transition Temperature)

Table 1: Comparative Test Results

<table>
<thead>
<tr>
<th>Test Conducted</th>
<th>Original Value</th>
<th>New Value</th>
<th>% change</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile Strength (kg/mm²)</td>
<td>6-7</td>
<td>3.0-4.0</td>
<td>33.33%</td>
</tr>
<tr>
<td>Compressive Strength at Yield Pt. (N/mm²)</td>
<td>110-115</td>
<td>120-125</td>
<td>9.09%</td>
</tr>
<tr>
<td>Ultimate Compressive Strength(N/mm²)</td>
<td>150-170</td>
<td>230-240</td>
<td>50%</td>
</tr>
<tr>
<td>Hardness</td>
<td>75-80</td>
<td>80-85</td>
<td>6.67%</td>
</tr>
<tr>
<td>Tg (Glass transition temperature) (°C)</td>
<td>95-100</td>
<td>125-130</td>
<td>31.57%</td>
</tr>
</tbody>
</table>

6. Inferences

It was observed that the tensile strength of the specimen decreased by approximately half of its original value. This was expected and attributed to the distribution of graphene in the resin matrix. Being immiscible, the graphene powder acted as an interference in the longitudinal continuity of the polymer, thus resulting in decreasing the overall tensile strength of the component. The graphene powder still had some agglomerated particles, which leads us to conclude that mechanical mixing is not the appropriate way to mix Nano-particles in a resin matrix; Ultra-Sonication method is suggested as it will achieve much more homogeneity and proper distribution of particle size.
The compressive strength of the specimen at yield point increased by a small margin. Proper mixing should increase this margin further. However, substantial increase in the ultimate compressive stress was observed. Also, no crack formation took place in the test samples; which leads us to conclude that the sample was flexible and had some amount of malleability.

The Shore-D hardness value of the sample increased by a small margin. Good surface finish was observed.

There was a significant increase in the glass transition temperature of the sample. This is dependent on the curing cycle as well, though enhancements were observed because of Gr powder addition. Proper mixing through sonication would achieve even better results in all of the above.

7. Conclusions

A higher compression strength without any noticeable crack development is a very advantageous quality desired in compressive load bearing structural components. The composite has, in general, a good strength to weight ratio.

The high glass transition temperature observed will serve as an excellent quality for application of this composite in high temperature applications, such as hot pipe lines, steam pipe-lines, Outer covering for components or machines with high temperature applications.

The molding properties of the composite were excellent and 90% of the mold area was filled without any additional help. The composite had a medium viscosity and thus can be easily casted into various shapes of different sizes. The resin is in liquid state at room temperature and is easy to handle.

It can be concluded that addition of graphene powder in such small quantity has caused noticeable changes in the mechanical properties of the polymer. With proper mixing using better techniques of manufacturing and processing at the Nano-scale, one can expect more enhancements.

8. References
