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# Effective Evaluation of Elastic Properties of a Graphene and Ceramics Reinforced Epoxy Composite under a Thermal Environment Using the Impact Hammer Vibration Technique

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**Abstract**: This paper presents an evaluation of the mechanical properties of nanocomposites when a lower concentration of nanoparticles graphene and ceramics are mixed with epoxy to determine the damping and stability characteristics of hybrid epoxy, using vibration techniques to extract accurate results. The effectiveness of the Impact hammer vibration technique is validated with mechanical testing such as three-point bending in terms of Young's modulus of the nanocomposite. The graphene nanocomposite carries nanoparticle 1 wt.% of epoxy, while the ceramic nanocomposite carries 3 wt.% of epoxy. It is observed that the reduction in frequency under a thermal environment is significantly less for graphene and ceramic reinforced hybrid nanocomposites, whereas the reduction in pure epoxy under a thermal environment is high. Thus, the results show that the addition of nanoparticles to composites shows improvement in the mechanical and thermal stability of elastic properties. The elastic properties obtained from the vibrational analysis are more consistent and economical than the three-point bending test for the evaluation of hybrid nanocomposites.

Keywords: graphene; ceramics; epoxy resin; elastic properties; vibration technique

## 1. Introduction

Composites are widely used in the field of aerospace, automotive and other highperformance structural applications due to their high stiffness and strength-to-weight ratios. They are a cheaper lightweight option than conventional materials such as metals. Despite the several advantages of using composites, they have several drawbacks, including high stress and strain development under load conditions. However, research has shown that the addition of nanoparticles in the polymer matrix is considered a highly effective technique to improve the mechanical properties of composites. Amendola et al. [1] investigated that the addition of nanoparticles in the polymer matrix results in nanocomposites with enhanced thermal and mechanical properties. It was shown that there is a good agreement with the matrix and high surface-to-volume ratio of the fine nanoparticles, which is the main reason behind the enhancement of the mechanical properties of the nanocomposites [2–4]. Firsov et al. [5] used filler in nanocomposites due to its improved physical properties such as high aspect ratio, low electrical resistivity, high thermal conductivity, high strength and elastic modulus. K B Kanchrela et al. [6] observed that the use of Yttria-stabilised zirconia (YSZ) nanoparticles improved some mechanical properties of glass fabric composites. Kaushal Kumar et al. [7] found that the use of TiO<sub>2</sub> nanoparticles in an epoxy composite increased its tensile strength. T S Muthu Kumar et al. [8] found an increase in the thermal stability and tensile strength of the polymer matrix composites due to the presence of small coffee bean powder. Taqui ur Rehman et al. [9] observed that the use of fillers  $SiO_2$ ,  $TiO_2$ 



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and TiO<sub>2</sub>@ SiO<sub>2</sub> as fillers in Zepoxy have minimal value LC (leakage current) and PD (partial discharge) for the best insulation performance. P Venkateshwar Reddy et al. [10] investigated the mechanical properties of a Prosopis juliflora fibre reinforced hybrid composite, which increased when using  $Al_2O_3$  as filler material. Wei et al. [11] investigated that the addition of graphene nanoparticles at lower concentrations (0.3%) showed increased tensile strength (12.6%) and increased flexural strength (10%). Srivastava et al. [12] investigated composites with graphene at lower weight ratios and with high aspect ratios, which improved the tensile strength by almost 30%. It was concluded that the mechanical properties of composite material can be increased with the addition of fillers such as graphene. Nanocomposites with lower concentrations of ceramic nanoparticles have high thermal conductivity and lower electrical conductivity, which is useful for industrial insulation and electrical packaging. Unnikrishnan [13] investigated the low concentrated epoxy-based nanocomposites with thermoplastic and particulate fillers. The toughening process increased fracture toughness and impact resistance.

## 1.1. Composite Made of Natural Fibre/Filler

Natural fillers may be preferred when bio-composites are required, according to P K Jagadeesh et al. [14]. These composites are referred to as renewable and eco-friendly composites. Up to a certain weight percentage, natural fillers perform well, but if you add more, the qualities of composite materials may suffer. When combined with hydrophilic fibres and hydrophobic matrices, fillers improve adhesion behaviour. The fillers can be added following the demands of the material's qualities, but they are typically added following the type of composite application.

As a more environmentally friendly, biodegradable, and renewable resource than petroleum-based synthetic polymers, biopolymers were suggested by A. Vinod et al. [15]. However, compared to synthetic polymers, the mechanical properties of materials are unsatisfactory and need additional exploitation. These days, adding plasticisers, nanofillers, and coupling agents to biopolymers and biopolymer blends is one of several approaches for improving the properties and structural integrity. Commercially available biopolymers include TPS, PVA, PLA, PHBV, Chitosan, epoxidized plant oils, and polysaccharides. However, these materials have significant drawbacks, including gas permeability, moisture sensitivity, short shelf lives, low mechanical strength, and susceptibility to bacteria and fungi. This is because the structural and physical characteristics of biopolymers can be specifically tailored by using nanoparticles as fillers.

According to MR Sanjay et al. [16], natural fibre composites have similar tensile strength, impact strength, interlaminar shear strength, thermal, water absorption, and tribological properties to synthetic fibre composites. However, several factors affect the properties of composites, including the type of resin used, the origin of the fibre (fruit, stem, leaf, etc.), the type of reinforcement used (powder form, short fibre, continuous fibre), the fibre orientation (unidirectional or multi-directional), the manufacturing method used (hand layup, compression moulding, injection moulding, etc.), the crystallinity index and crystallite size of the fibre, the chemical functional groups present in the fibre, and volume and weight (raw or surface treated).

## 1.2. Significance of Nano Filler

Ganapathy et al. [17] filled the fibres made from the aerial roots of banyans with graphene. To create better epoxy composites, he described the appropriate ratio of graphene powder to banyan fibres. He noted that the unfilled epoxy composite had a flexural strength of 155.51 MPa and tensile strength of 27.93 MPa, while the strongest hybrid composites in terms of tensile strength (40.6 MPa) and flexural strength were those that contain 4% graphene (163.23 MPa).

The impact of  $Al_2O_3$  nanofillers on the mechanical, wear, and hardness properties of basalt/epoxy laminate composites were discovered by Vinay et al. [18]. By using the hand layup process, composite laminates of basalt/epoxy with varying amounts of  $Al_2O_3$  nanofillers were created. According to ASTM standards, mechanical properties such as tensile strength, interlaminar shear strength (ILSS), flexural strength, impact strength, and hardness were examined. Flexural strength and ILSS were shown to increase for small percentages of nanofillers, whereas tensile strength declined for larger percentages of fillers, and hardness increased for larger percentages of fillers. As the content of nanofillers increased, the wear rate gradually decreased.

Cissus quadrangularis stem fibre (CQSF)/epoxy resin particulate with and without coconut shell ash (CSA) powder underwent mechanical characterisation by Jenish et al. [19]. The hand lay-up method was used to build the base material from epoxy and 30 wt.% CQSF with 40 mm fibre length, and CSA was added separately at 2.5, 5, 7.5, and 10 wt.%. The tensile test SEM image of the CQSF/epoxy with 5 wt.% CSA filler composite showed less matrix breakage and fibre/matrix bonding, which boosted the tensile strength of the composite material. At 10 wt.% CSA, the impact strength (20.03 J/cm<sup>2</sup>) and hardness (98 HRRW) values were greater in the CQSF/epoxy resin composite, indicating that impact and hardness steadily rise as CSA filler content rises.

In this paper, a lower concentration of the nanoparticles was maintained with 1 wt.% of epoxy in the case of graphene nanocomposites and 3 wt.% of epoxy in the case of ceramic nanocomposites. To evaluate the properties of the nanocomposites, vibration techniques (ASTM E1876-15) were conducted. The validation of the elastic properties such as Young's modulus, Shear modulus and Poisson's ratio was carried out by comparing the results obtained from the above two methods.

#### 2. Materials and Methods

In this study, a thermoset type of polymer matrix material epoxy LY556 was used. Araldite hardener HY 917 was used as a curing agent. Graphene nanoparticles and stoichiometric spinel (MgAl<sub>2</sub>O<sub>4</sub>) precursor prepared by solid-state synthesis (0.4 wt.%) were used as reinforcement. Pure Isopropanol (2-Propanol),  $C_3H_8O$ , M.W 60.10 and Acetone extra pure AR grade,  $C_3H_8O$  and M.W 58.08 were used to ease the sonication process by the addition of a volatile liquid. The lower concentration of nanoparticles was maintained with 1 wt.% of epoxy in the case of graphene nanocomposites and 3 wt.% of epoxy in the case of ceramic nanocomposites. The flow chart for preparation of Epoxy/Graphene Composite is shown in Figure 1.



Figure 1. Flow Chart for preparation of Epoxy/Graphene Composite.

The epoxy composite samples mixed with epoxy resin LY556 were combined with hardener Araldite HY917 in a 10:1 ratio. The solution was manually stirred for 5 min and then poured into dies. To cure the samples, they were kept at 75 degrees for 1 h. The samples were machined according to the requirement of the testing apparatus. The graphene nanocomposites were prepared by a powder process. The sonication process was carried out while the samples were prepared to properly disperse the nanoparticles in the resin system. Failure to carry out the sonication process can severely affect the mechanical properties. The Sonication process was conducted in a sonicator with a titanium probe running at 1 Amp, 0.1 mV for 1 h. Sonicated solution was mixed with graphene nanoparticles manually. For preparing the sonication was carried out in 3 intervals of 1 h. The solution was then kept in the oven to evaporate acetone from the solution. To prepare solid samples, the solution was mixed with hardener Araldite HY917 in a 10:1 ratio and then poured into the die for the curing process. The solidified samples were machined according to the requirement of the testing apparatus.

#### 2.2. Sample Preparation of Ceramics/Epoxy Nanocomposites

The ceramic nanocomposites were prepared by the powder process. Sonicated solution was mixed with ceramic nanoparticles manually. For preparing the sonicated solution, epoxy was heated and then mixed with a solution of isopropanol and ceramic powder. Sonication was carried out for 30 min; the solution was then kept in the oven to evaporate isopropanol from the solution. To prepare solid samples, the solution was mixed with hardener Araldite HY917 in a 10:1 ratio and then poured into the die for the curing process. The solidified samples were machined according to the requirement of the testing apparatus. Figure 2 shows three different developed samples of pure Epoxy, Epoxy/Graphene and Epoxy/ceramic.



Figure 2. (a) Pure Epoxy sample (b) Epoxy/Graphene sample (c) Epoxy/ceramic sample.

#### 3. Material Characterisation

Characterisation of materials was carried out to identify the mechanical properties of the nanocomposites. It helps in the proper evaluation of its capabilities. The three-point bending test and the Impact hammer test were conducted to evaluate the elastic properties.

## 3.1. Three-Point Bending Test-ASTM D7264

The three-point bending test (based on ASTM D7264) was conducted [20] using an Universal Testing Machine (Manufacturer INSTRON, Model 8801, Indian Institute of Technology, Dhanbad, India). The specimens were tested at room temperature under a uniform strain rate of 1 mm/min and the span to depth ratio was maintained to be greater than 16:1. The maximum flexure stress, maximum flexure extension and elastic modulus were obtained from the test as tabulated in Tables 1–3.

Sample	Epoxy (Mpa)	Graphene/Epoxy (Mpa)	Ceramic/Epoxy (Mpa)
1	4315.7	4470.9	4724.08
2	4685.9	5185.1	4866.4
3	4086.4	4361.5	-
Mean	4362.7	4672.5	4795.2

Table 1. Young's Modulus Values of Epoxy and Nanocomposites.

Table 2. Elastic properties of epoxy and Nanocomposites.

<b>Elastic Properties</b>	Epoxy	Graphene/Epoxy	Ceramic/Epoxy
Young's modulus (GPa)	3.99	4.156	4.34
Shear modulus (GPa)	1.511	1.6	1.72
Poisson's Ratio	0.320	0.298	0.261

Table 3. Epoxy sample readings at varying temperatures.

<b>Elastic Properties</b>	45 Degrees	60 Degrees	75 Degrees
Poisson ratio	0.205	0.166	0.161
Shear modulus (GPa)	1.41	1.2	1.073
Young's modulus (GPa)	3.4	2.8	2.492

#### 3.2. Impact Hammer Test-ASTM E1876-15

To validate the improvements, we conducted impact hammer testing as used in [21] to explore the changes in elastic modulus along with damping characteristics. By measuring the resonant frequencies in a different configuration, Young's modulus, Shear modulus and Poisson's ratio were calculated using the ASTM E1876-15 [11]. The test samples were created according to the ASTM E1876-15, where B/t > 5 and L/t > 20 (B = breadth, L = Length & t = Thickness). The samples were placed on the specific fixtures and a contact accelerometer was placed to read the vibrations. The electrical signals were then transferred to software DeweSoft, (Version 2020, creator, Dewesoft, Indian Institute of Technology, Dhanbad, India) which converted them into vibrational signals to highlight the resonant frequency. Figure 3 shows the different positions of the testing fixture for evaluating elastic constants Young's and Shear moduli in bending and torsion modes of vibrations.

The Dynamic Young's modulus using standards.

$$E = 0.9465 \left(\frac{mf_f^2}{b}\right) \left(\frac{L^3}{t^3}\right) T_1 \tag{1}$$

$$T_1 = \left[1.000 + 6.585 \left(\frac{t}{L}\right)^2\right] \tag{2}$$

where: E = Dynamic Young's modulus, Pa; m = mass of the bar, g; b = width of the bar, mm; L = length of the bar, mm; t = thickness of the bar, mm; f<sub>f</sub> = fundamental resonant frequency of bar in flexure, Hz; T<sub>1</sub> = correction factor for the fundamental flexural mode to account for the finite thickness of the bar.



Figure 3. (a) Out of Plane frequency. (b) Torsional frequency setup.

Dynamic shear modulus used formulas provided under ASTM standards.

$$G = \frac{4 Lm f_t^2}{bt} R \tag{3}$$

$$R = \left[\frac{1 + \left(\frac{b}{t}\right)^2}{4 - 2.521\frac{t}{b}\left(1 - \frac{1.991}{e^{\pi\frac{b}{t}} + 1}\right)}\right] \left[1 + \frac{0.00851n^2b^2}{L^2}\right] - 0.060\left(\frac{nb}{L}\right)^{\frac{3}{2}}\left(\frac{b}{t} - 1\right)^2 \quad (4)$$

where G = Dynamic shear modulus, Pa;  $f_t$  = fundamental torsional resonant frequency of bar, Hz; n = the order of the resonance, here n = 1.

Poisson's ratio used formulas provided under ASTM standards.

$$u = \left(\frac{E}{2G}\right) - 1\tag{5}$$

where  $\mu$  = Poisson's ratio; *E* = Dynamic Young's Modulus. Pa; *G* = Dynamic Shear Modulus, Pa.

## 4. Results and Conclusions

The Young's modulus, Shear modulus and Poisson's ratios were calculated and tabulated after obtaining the resonant frequencies. The effect of the nanoparticles on the elastic properties has been discussed. There was an increase in Young's modulus due to the inclusion of nanoparticles in the matrix, as shown in Table 1 measured from UTM for Young's modulus, and Table 2 measured from vibration techniques for all the elastic constants. The inclusion of graphene nanoparticles showed an increase of 7.1%, while the inclusion of ceramic caused an increase of 10.4%.

Similar results to that of the three-point bending test were produced from the impact hammer test, with an error of about 8%. However, the values from this test are much more dependable as the number of iterations is more. The ceramic nanocomposites showed the highest improvement in Young's modulus (8%) and shear modulus (13%), while Graphene nanocomposites showed less of a decrease in Poisson's ratio due to lower brittleness.

#### 4.1. Elastic Properties of Nanocomposites under Thermal Environment

To evaluate the thermal stability of the elastic properties, the entire set-up was transferred to an industrial oven. The temperature was increased slowly and the resonant frequencies were recorded. The elastic properties were then evaluated from the formulas provided in the ASTM standard. The trends of Young's modulus, Shear modulus and Poisson's Ratio is shown in Figure 4 and their corresponding values are given in Tables 4 and 5.



Figure 4. (a) Trends of Young's modulus (b) Trends of Shear modulus (c) Trends of Poisson's Ratio.

<b>Elastic Properties</b>	45 Degrees	60 Degrees	75 Degrees
Poisson ratio	0.298	0.284	0.268
Shear modulus (GPa)	1.54	1.51	1.41
Young's modulus (GPa)	3.998	3.880	3.578

 Table 4. Graphene/Epoxy sample reading at temperatures.

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<b>Elastic Properties</b>	45 Degrees	60 Degrees	75 Degrees	
Poisson ratio	0.244	0.186	0.163	
Shear modulus (GPa)	1.62	1.33	1.18	
Young's modulus (GPa)	4.053	3.161	2.744	

The selected nanocomposites were tested up to 75 degrees, as the flash point for epoxy LY556 is 80 degrees. The inclusion of nanoparticles showed constantly better properties, even at elevated temperatures. The graphene nanoparticles showed the best performance under thermal conditions and, on average, lost 5% of their Young's modulus and a 4% decrease in Shear modulus. This is because graphene tends to disperse easily under high temperatures in a low viscosity system. Even though the ceramic lost up to 20% of its properties, its values were higher than that of pure epoxy composites.

## 4.2. Validation Conclusions

The investigation of elastic properties was carried out by performing a three-point bending test and an Impact hammer test. The results from the three-point bending test were investigated. There was an increase in Young's modulus with the addition of graphene nanoparticles (an increase of 7.1%), while the addition of ceramic nanoparticles showed an increase of 10.4%. Nanocomposites showed lower maximum flexural loads (7% decrease) as the addition of nanoparticles increases the brittleness of the composites. The graphene nanocomposites showed the lowest stress values (6% decreases) compared to that of pure epoxy composites. This is due to the dispersion of graphene in the epoxy matrix, forming a perfect continuous structure. The results from the Impact hammer test were investigated. The ceramic nanocomposites showed the highest improvement in Young's modulus (8%) and shear modulus (13%), while graphene nanocomposites showed less of a decrease in Poisson's ratio. This is because nanoparticles tend to disperse easily and form a continuous system in a low viscosity medium. The results from the three-point bending test and Impact hammer test were compared and given in Table 6.

Testing Method	Epoxy (GPa)	Graphene/Epoxy (GPa)	Ceramic/Epoxy (Gpa)
Three-point Bending test	4.36	4.67	4.79
Vibrational hammer test	3.99	4.15	4.34
Percentage difference (%)	9.2	12.5	10.4

Table 6. Comparison between Three-point bending and Vibrational hammer test.

There was a difference of 10%–12% between the final results but the impact hammer test was considered to be a more dependable test, as Young's modulus values after several iterations were found to be more consistent when compared to the three-point bending test results. As the Impact hammer test is non-destructive, a detailed analysis of elastic properties under varied thermal conditions was also possible. At elevated temperatures the graphene nanocomposites showed only a 5% decrease in Young's modulus and a 4% decrease in Shear modulus. Even though the ceramic lost up to 20% of its properties, its values were higher than that of pure epoxy composites. This is because graphene and ceramic nanoparticles tend to disperse easily under high temperatures in a low viscous system. Thus, the results show that the addition of nanoparticles to composites shows improvement in the mechanical and thermal stability of elastic properties. The Impact hammer vibration test can be carried out to efficiently investigate the elastic properties of the composites under varied thermal conditions.

## 5. Conclusions

Two different samples of nanocomposites have been developed with graphene/1 wt.% epoxy and ceramic/3 wt.% epoxy. The mechanical properties of the developed nanocomposites are compared in terms of young's modulus, shear modulus and Poisson's ratio at a temperature of 45 degrees, 60 degrees and 75 degrees. Further three-point bending tests and Impact hammer tests were carried out to compare the elastic properties of both the developed nanocomposites. The inclusion of graphene nanoparticles showed an increase of 7.1%, while the inclusion of ceramic caused an increase of 10.4%. At an increased temperature, the graphene nanoparticles showed the best performance under thermal conditions and, on average, lost 5% of their Young's modulus and there was a 4% decrease in Shear modulus, even though the ceramic lost up to 20% of its properties. The results from the three-point bending test shows lower maximum flexural loads (7%) decrease), as the addition of nanoparticles increases the brittleness of the composites. The graphene nanocomposites showed the lowest stress values (6% decreases) compared to that of pure epoxy composites. The results from the Impact hammer test showed the highest improvement in Young's modulus (8%) and shear modulus (13%), while graphene nanocomposites showed less of a decrease in Poisson's ratio. There was a difference of 10%–12% between the final results of the three-point bending test and impact hammer test. However, the impact hammer test was considered to be a more dependable test, as Young's modulus values after several iterations were found to be more consistent when compared to the three-point bending test results.

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