Characterization of Graphene Polymer Composite

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Abstract *Nanotechnology is used in numerous fields, wherever the application varies from engineering to cosmetics. Nano scale products and materials exhibit at least one novel or superior property due to its nano scale size.*

Graphene Epoxy Composite and Graphene Polyester Composite specimens are prepared with five different percentage ratios of graphene i.e. (0%, 1.5%, 3%, 4.5% and 6 %.). Specimens were prepared by Ex-situ process & as per ASTM standards for Tensile (D638) and Compression (D695–85) test specimens. The total amount of matrix (Epoxy and Polyester) used for Tensile and Compression for Graphene Epoxy Composite test specimens is 150 grams and for Graphene Polyester Composite test specimens is 130 grams which is constant for every percentage variation in graphene and tests are conducted.

The Tensile strength(1.5%) and Compression strength(4.5%) is more in Graphene Epoxy Composite when compared to Graphene Polyester Composite. The results reveal that Graphene Epoxy Composite is having better results than Graphene Polyester Composite.

*Key Words: Graphene1, Epoxy2, Polyester3, and Ex-situ Process**4 etc…***

1. INTRODUCTION

Nanotechnology is precisely defined as characterization, production and application of devices and systems at nanometer scale, manipulating their shape and dimensions in a controlled way.. These nano scale products and materials exhibit at least one novel or superior property due to its nano scale size.

The properties of materials differ at atomic and subatomic levels when compared to the material at a much larger scale (Macro & Micro). In Macro & Micro (large) size materials the average of all quantum forces act upon the atoms affecting the material properties, but when the materials are made smaller and smaller, these average forces will no longer act upon the atoms. The nano size of the materials contributes high surface area and quantum effects to the materials, which improves the materials by enhancing their reactivity, strength and electrical properties. Nano science is a part of nanotechnology, which helps to study the physical properties of materials and products at atomic, molecular and micro molecular level depending on the material dimension. Nanotechnology combined with nano science controls the matter at nanometer scale and takes all the existing fields to Nano scale level [1].

1.1 Nanomaterial

Particles with less than 100nm (1 micron) measurement are termed as nano particles (at least in any one dimension). Nano particles have high relative surface area per unit volume, which makes them more chemically reactive than that of macro sized particles of the same material. The materials designed from the nano particles (nanomaterials) show new or enhanced size of the material properties contrasted with ordinary materials [2].

1.2 Nano-Graphene

The investigation of materials features such as its composition, structure, and different properties like physical, electrical, magnetic, etc. Nanoparticles characterization is important to build up comprehension and control of nanoparticles amalgamation and applications. The particle size and size distribution of nanoparticles can be resolved utilizing various economically accessible instruments. Instruments can be utilized for the investigation of dry powders and powders scattered in suspension. There are two fundamental systems for characterizing molecule size. The molecule size and size distribution of Graphene nanoparticles, as basic properties have been determined by Scanning Electron Microscopy (SEM), X- Ray Diffraction (XRD) [3].

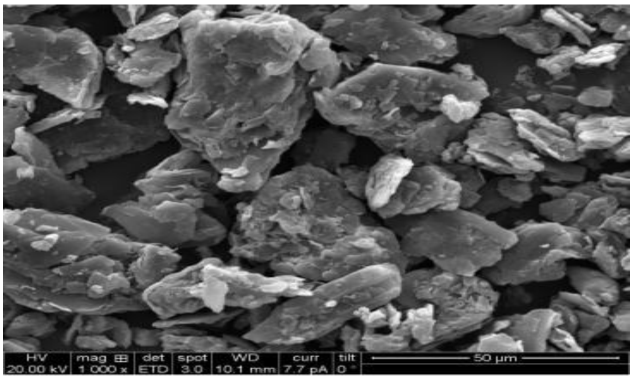


Fig -1: SEM image of nano Graphene

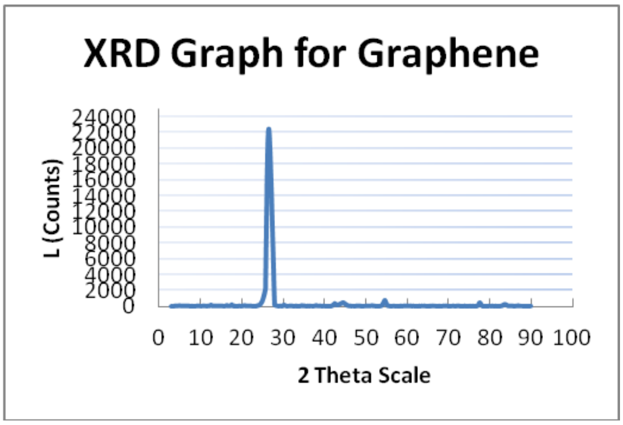


Chart-1: XRD image of Nano Graphene

Table -1: Technical parameters of Graphene

Parameters	Values
Bulk density	0.241g/cc
Diameter average X&Y dimensions	10-20 micron
Thickness average Z dimensions	3-6 nm
Purity	96-99%
Number of layer	Average number of layer 1-3
Surface area	323-600 m^2/g

1.3 Epoxy

Lapox L-12 is a Liquid epoxy resin based on bisphenol-A with medium viscosity which can be mixed with different hardeners for making glass fiber strengthened composites. The decision of hardener relies on upon the handling system to be utilized and on the properties needed to the cured composite [4].

Hardener K6 is a low viscous liquid at room temperature. It is regularly utilized for hand layup applications. Being somewhat receptive it gives a short pot life and fast cure at room temperature. Laminates can be subjected to working temperatures of 1000^0 C.

Table-2: Lapox L-12

Parameter	Value	Unit
Epoxide Equivalent	182-192	gm/eq
Epoxy value	5.2-5.5	eq/kg
Viscosity at 25^0 C	9000-12000	mPa.s

Table-3: Hardener K-6

Parameter	Value	Unit
Epoxide Equivalent	182-192	gm/eq
Epoxy value	5.2-5.5	eq/kg
Viscosity at 25^0 C	9000-12000	mPa.s

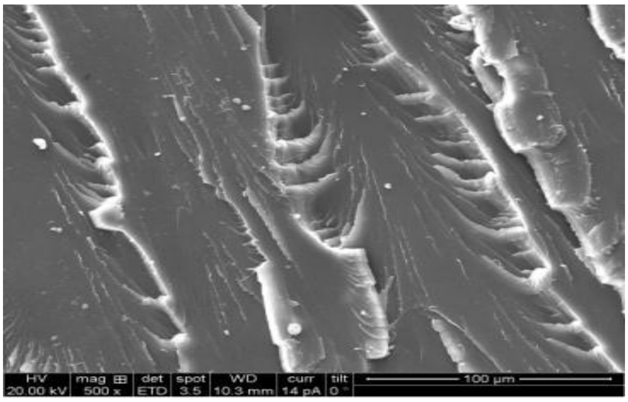


Fig-2: SEM image of Epoxy Composite

1.4 Polyester

Polymers are substances whose particles comprise of an extensive number of units of a few types: the units themselves, comprising of various atoms, are generally referred to as the fragments of the polymer. In the polymerization of blends of two monomers, the structure of every macromolecule contains units of both monomers. Such a polymer is called copolymer and the procedure of its combination is called copolymerization [5].

The subsequent copolymers were of low atomic weight (8,000-10,000 g/mol), hard, crystalline solids and susceptible to transformation from the liquid state to fibers which could be extended underneath their dissolving point with an extreme increase in strength.

Table-4: Polyester

Property	Typical value
Appearance	Blue Opaque Liquid
Viscosity	1600-2000 cP
C&P Viscosity	15, 30, 45, 60, 75, 90 minute versions available
Geltime	3210-250 cP
Density	1.10 g/cm
Volatile Content	40-44 %
Flash Point	31°C
Shelf Life	6 months

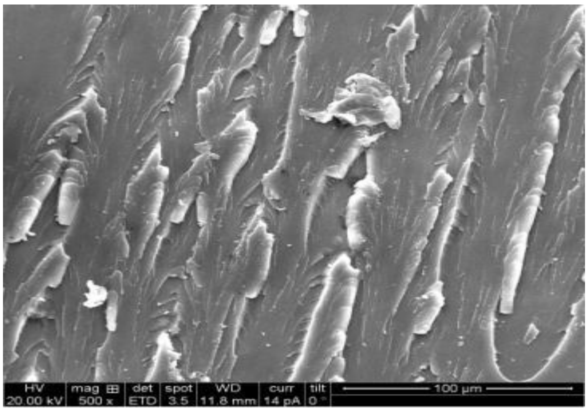


Fig-3: SEM images of Polyester Composite

2. EX -SITU PROCESS:

Ex - situ procedures are largely and easily adopted systems with minimum cost, where nanoparticles, are added or blended or mixed externally, to a monomer or resin, and cured by polymerization [6].

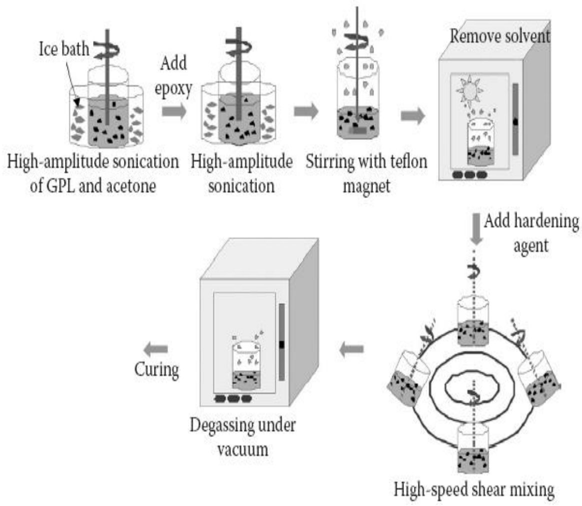


Fig -4: Ex -Situ Process

3. SPECIMEN PREPARATION OF EPOXY GRAPHENE COMPOSITES (0%, 1.5%, 3%, 4.5%, 6 %.)

Epoxy Graphene Composite specimens are prepared with five different percentage ratios of graphene i.e. (0%, 1.5%, 3%, 4.5% and 6 %.) of epoxy. Specimens were prepared by Ex-situ process, which includes many steps.

In step one, A known quantity of graphene with different percentage was taken in a first beaker and an acetone of 30 ml is poured into a first beaker which is constant for each different percentage, later for better mixing of graphene and acetone beaker is subjected to magnetic stirring by using magnetic stirrer to get a perfect mixing.

In step two, To achieve the standards as per ASTM the total amount of material (Epoxy) required for Tensile and Compression test specimens is 150 grams, which is constant for every percentage variation in graphene, is taken in a second beaker and it was kept on heater for heating the epoxy. By doing this viscosity of the epoxy reduces and generation of bubbles is minimized. The Epoxy is heated for approximately 60^o – 70^o C for 10 min and latter a constant temperature of about 40^o C is maintained for next 30 min with a continuous stirring with a glass rod.

In step three, The heated epoxy is allowed to attain to the room temperature later the graphene and acetone mixed liquid solution which is present in beaker one is poured in to the beaker two which consists of heated epoxy and stirred continuously for 30 min, so that all the acetone which is present in the Graphene-Epoxy matrix is evaporated. After the evaporation of acetone in the Graphene-Epoxy matrix hardener is added to it with a ratio of 1:10 of epoxy.

In step four, The evaporated acetone in the Graphene-Epoxy matrix with hardener is poured to the rectangular aluminum mould, the inner part of the mould is coated with Silicon grease as a realizing agent, The composite which is poured into the mould is allowed to solidify for 24 hours. The dried and hardened specimens are removed from the moulds which are ready for machining as per ASTM standards. The same procedure is repeated for different percentage of Graphene is tabulated.

4. SPECIMEN PREPARATION OF POLYESTER GRAPHENE COMPOSITES (0%, 1.5%, 3%, 4.5%, 6 %.)

Polyester graphene composite specimens are prepared with five different percentage ratios of graphene i.e. (0%, 1.5%, 3%, 4.5% and 6 %.) of polyester. Specimens are prepared by Ex-situ process with slight changes compared to Epoxy Graphene Composite. The viscosity of Epoxy is higher it is very difficult to mix Graphene. If we start mixing the grapheme directly to the epoxy there may be chances of including bubbles and also formation of lumps with these two reasons the method adopt for epoxy is different than that of Polyester.

To achieve the standards as per ASTM the total amount of material (Polyester) required for Tensile and Compression test specimens is 130 grams, which is constant for every percentage variation in graphene. Polyester and Graphene is measured and poured into beaker and stirred continuously for 2 min. As the viscosity of Polyester is less it is very easy to mix Graphene. 10 grams of accelerator is added to the mixture containing Polyester and Graphene immediately 5 grams of catalyst is also added into the beaker stirred for 1 min which are constant for every percentage of specimens.

Graphene-Polyester matrix with accelerator and catalyst is poured to the rectangular aluminum mould, the inner part of the mould is coated with Silicon grease as a realizing agent, and the composite which is poured into the mould is allowed to solidify for an hour. The dried and hardened specimens are removed from the moulds which are ready for machining as per ASTM standards. The same procedure is repeated for different percentage of Graphene

5. MACHINING PROCESS:

Machining is Processes in which a piece of raw material is cut into a desired final shape by a controlled material-removal process. Milling is a cutting process that uses a preparing cutter to expel material from the surface of a work piece. The preparing cutter is a spinning cutting instrument, regularly with different cutting focuses [7]. Tensile and Compression example's of both epoxy graphene and polyester graphene composite of distinctive proportions are machined to ASTM Standard. The Tensile specimen is machined by standard D638, and Compression specimen is machined by D695- 85.

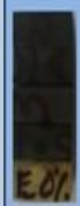
Type of test	Specimen before tests		Specimen Dimensions (mm)
	Epoxy Graphene	Polyester Graphene	
Tensile ASTM D638			
Compression ASTM D695-85			

Fig-5: Machined specimens

6. TENSILE

Tensile tests are performed for a many reasons because the outcomes of Tensile tests are utilized as a part of selecting materials for designing applications. Tensile properties as often as possible are incorporated in material particulars to guarantee the quality. Tensile properties regularly are measured in course of envelopment of new materials and procedures, so that diverse materials and procedures can be thought of. At last, Tensile (ductile or brittle) properties are utilized to predict the characteristics of a material under types of loading other than uniaxial tension.

6.1 Tensile specimen

Consider the normal tensile specimen which has enlarged ends or shoulders for holding. The critical part of the specimen is the gauge length. The cross-sectional range of the gage length is decreased with respect to that of the rest of the specimen so that deformation and failure will be restricted in this distinct length only. The gauge length is the region over which estimations are made and is focused inside of the reduced section. The distances between the ends of the gauge area and the shoulders are sufficiently incredible so that the larger ends don't constraint deformation inside of the gauge length, and the gage length should be great with respect to its diameter [8].

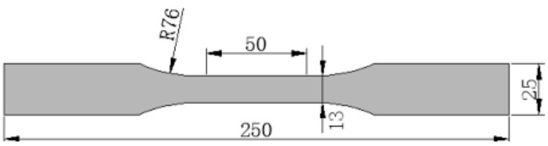


Fig-6:Tensile specimen

7. COMPRESSION

A Compression test decides the attributes of materials under compression loads. The specimen is compacted and deformation at different loads is recorded. Compressive stress and strain was ascertained and plotted as a stress strain diagram which is utilized to focus on elastic limit, proportional limit, yield strength and yield point. Compression Tests are off to a greater significance, because it serves to ascertain the diverse material properties that are pertinent to hot and cold metal forging utilized for distinctive metal shaping applications. It is imperative to locate the suitable load for the forging operation. Load relies on the type materials and flow stresses [9].

7.1 Compression specimen

Compression test specimen is prepared according to ASTM standards D695- 85 [10].

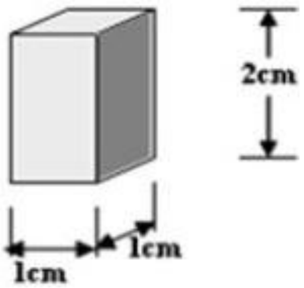


Fig-7: Compression specimen

8. TESTED SPECIMENS

Type of test	Epoxy Graphene Specimen		Specimen Dimensions (mm)
	Before Test	After Test	
Tensile ASTM D638			
Compression ASTM D695-85			

Fig-8: Epoxy Graphene specimen

Type of test	Polyester Graphene Specimen		Specimen Dimensions (mm)
	Before Test	After Test	
Tensile ASTM D638			
Compression ASTM D695-85			

Fig-9: Polyester Graphene specimens

9 .SEM IMAGES OF EPOXY GRAPHENE COMPOSITE

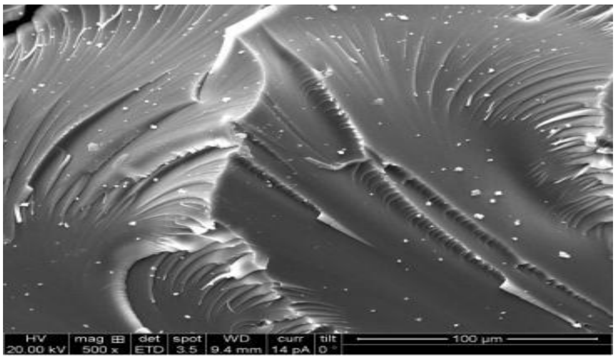


Fig-10: 500 x magnification

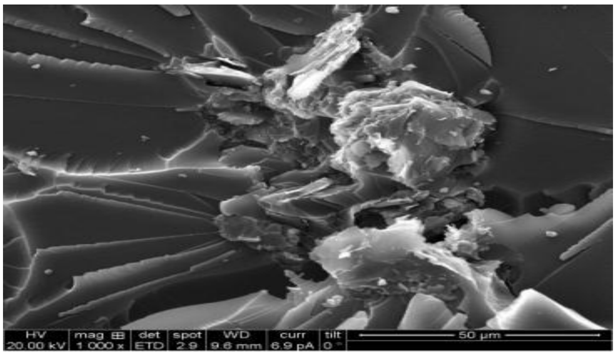


Fig-11: 1000 x magnification

10. SEM IMAGES OF POLYEMER GRAPHENE COMPOSITE

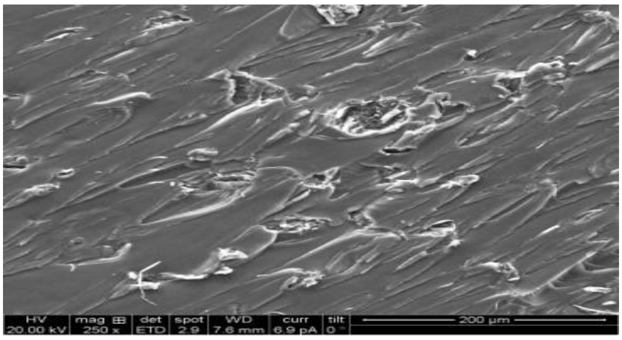


Fig-12: 200 x magnification

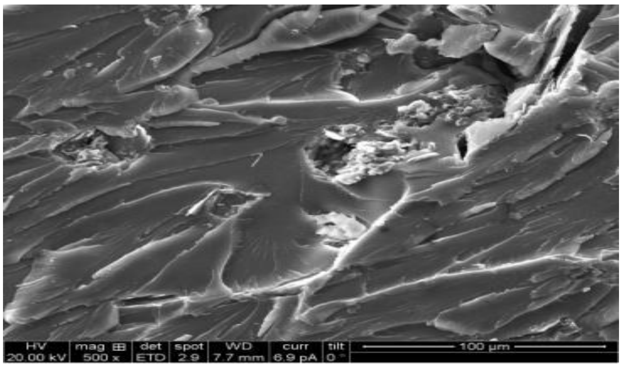


Fig-13: 500 x magnification

11. TENSILE TEST RESULTS

The Tensile test is performed in the Universal Testing Machine (UTM) and results are evaluated to ascertain the rigidity of composite specimens [11]. The test prepared overlays were subjected to elastic loads on a PC controlled Universal Testing Machine and the tests were performed at a steady crosshead rate of 5 mm/min. In this work the Tensile strength is characterized as a ultimate strength at which the complete fracture of the specimen occurs and the relating loads is the critical load or the most extreme load a material can with stand. The tests were closely observed and directed at room temperature [12]. The test results of the Polymer Matrix Composite mixed with Epoxy Graphene Composite and Polyester Graphene composite are tabulated in Table 5 and Table 6.

Table-5: Epoxy Graphene Composite (Tensile)

No. of (%)	Peak Load (Kg)	Peak Disp. (mm)	Eng. UTS (Kg/sq mm)	Reduction area (%)
0%	156.0	4.099	2.690	0.076
1.5%	179.0	5.355	3.320	0.097
3%	177.0	4.990	3.153	0.091
4.5%	113.0	5.244	2.121	0.096
6%	134.0	4.591	2.430	0.085

Table-6: Polyester Graphene Composite (Tensile)

No. of (%)	Peak Load (Kg)	Peak Disp. (mm)	Eng. UTS (Kg/sq mm)	Reduction area (%)
0%	167.0	9.233	3.002	0.157
1.5%	159.0	8.407	2.849	0.009
3%	145.0	5.473	2.813	0.099
4.5%	100.0	3.700	1.928	0.069
6%	140.0	5.020	2.034	0.085

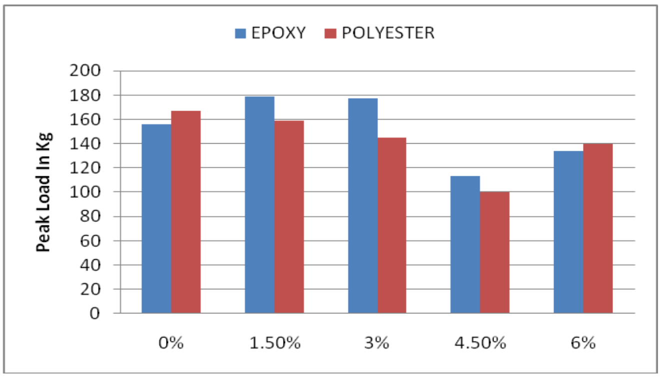


Chart-2: Comparison of different percentages of Graphene in Epoxy, Graphene in polyester Composite (Tensile)

12. COMPRESSION TEST RESULTS

A Compression test decides the qualities of materials under varying loads. The specimen is packed and twisting at different load. The regularly used specimens for polymer composites are in rectangular cross section. This test is led according to ASTM D-695-85 standard. The Compression test is performed in the all Universal testing machine (UTM) and results are dissected to ascertain the Compression quality of composite examples [13]. The prepared samples were subjected to Compression test on a PC controlled Universal Testing Machine.. The test outcomes are in indicated in Table 7 and Table 8

Table-7: Epoxy Graphene Composite (Compression)

No. of (%)	Peak load (N)	Cross Head travel at peak (mm)	Comp. Strength (N/mm^2)
0%	10920.00	1.9	104.56
1.5%	5640.00	4.5	52.35
3%	5160.00	1.9	49.47
4.5%	11640.00	8.0	111.23
6%	7320.00	6.2	70.10

Table-7: Polyester Graphene Composite (Compression)

No. of (%)	Peak load (N)	Cross Head travel at peak (mm)	Comp. Strength (N/mm^2)
0%	8760.00	1.9	83.25
1.5%	7560.00	2.2	73.78
3%	7500.00	5.6	70.26
4.5%	6900.00	3.7	65.50
6%	7450.00	2.0	69.84

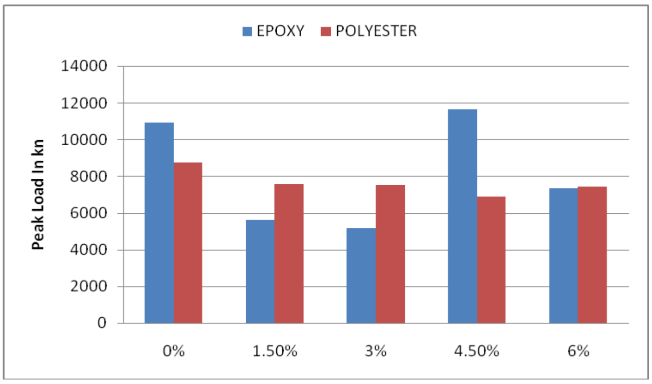


Chart-3: Comparison of different percentages of Graphene in Epoxy, Graphene in polyester Composite (Compression)

13. CONCLUSIONS

The Tensile strength is more in 1.5% of Epoxy Graphene Composite (peak load is 179 kg,) and 1.5% Polyester Graphene Composite (peak load is 166 Kg,) as shown in the Chart-2 and in Table-5 and 6. Founds to be highest when compared to other Percentages. Hence from above results and discussions Tensile strength is maximum in Epoxy Graphene Composite when compared to Polyester Graphene Composite.

The Compression strength is more in 4.5% of Graphene polymer Composite (peak load is 11640 KN , Compression Strength is 111.23 N/mm^2) and In Polyester Graphene Composite the variation is very very less in addition of Graphene is respect in the Chart-3 and Table 7 and 8

Graphene Composites is making interest for scientist to discover and to utilize its property for many applications like defense, medical, aerospace and civil applications etc.

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Dr C Anil Kumar is a Professor in Dept of Mechanical Engineering KSIT Bengaluru. To the credit I had 10 International Journals & 13 Conferences Proceedings.