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Impact of Carbon Nanotubes on Polymer Nano composites

G Satish[1], V V S Prasad[2], K Ramji[3]
Research Scholar, Department of Marine Engineering, Andhra University, Visakhapatnam, 530003, India [1]
Professor, Department of Marine Engineering, Andhra University, Visakhapatnam, 530003, India [2]
Professor, Department of Mechanical Engineering, Andhra University, Visakhapatnam, 530003, India [3]

***Abstract*— Carbon nanotubes have been the focus of considerable research due to the excellent mechanical, electrical and magnetic properties. In the present work fifteen specimens of polymer composites is fabricated, multi walled carbon nanotubes (MWCNT's) are considered as filler material for proposed volume based quantity and for different orientations of glass fiber for every specimens. The effect on the mechanical properties such as hardness, tensile and flexural strength was studied and graphs are plotted for experimental data. Scanning electron microscopy shows the homogeneous dispersion of carbon nanotubes and agglomeration on glass mat. It was found that the mechanical properties of these composites were significantly improved by incorporating a small percentage of MWCNT's compared to the neat composites. The experimental results are compared with statistical results and found to be in good agreement.**

***Index Terms*— Multi walled carbon nanotubes, carbon nanotubes, glass mat, mechanical properties.**

I. Introduction

Sumio Ijima invented carbon nanotubes (CNTs) in 1991[1]. The researchers focused on this material for the reason that they have unique structural, mechanical, electrical, thermal and other materials- related properties of carbon nanotubes [2]-[4]. It gives tremendous opportunities for the development of new material. Carbon nanotubes is the key component of the nano technology, as it controls the matter on nanoscale and its effects on properties of composite materials. CNT's have been used in many potential applications such as nanoelectronic and photovoltaic devices [5],[6], super conductor [7], electromagnetic actuator [8], and nano composite materials [9], [10].

A polymer composite consists of a polymer matrix with nano-sized filler. Many different types of filler have been tested and are also used commercially to improve the properties of polymers. In this context, CNTs is one of the most interesting filler for future applications. Because of their extraordinary properties and large aspect ratio, minute amount of CNTs can have a large influence on the properties of a composite [11-13]. The carbon nanotubes are insoluble in any organic solvent and difficult in dispersion in a polymer matrix due to strong vander waals interaction. Also carbon nanotubes tend to form into aggregates and it is a negative effect on the properties of composite. In general, there are several methods for dispersion of CNT/polymer composites.

They can be summarized as magnetic stirring, shear mixing, reflux, most commonly used ultra-sonication [12], solution mixing, melt blending, In situ polymerization, latex technology and layer-by –layer deposition [14].

Solution mixing is one of common method for the fabrication of CNT/polymer laminated composites because it can be accepted for small sample size [15-17]. Blending of solution involves in three methods; dispersion of CNT's in a solvent by mechanical mixing, magnetic agitations or sonication. Carbon nanotubes can also dissolve in polymer resins at room or elevated temperature [14]. Melt blending is commonly used method to fabricate CNT/polymer nano composites. Thermo plastic polymers, such as polypropylene [18], polystyrene [19], poly (ethylene 2,6-naphthalate)[20] can be processed as matrix materials in this method. The reinforcement of CNT in polymer composites, proper dispersion and interfacial adhesion between the CNTs and polymer matrix has to be guaranteed. The effect of CNT dispersion and functionalization on the properties of CNT/polymer nanocomposites[14]. In situ polymerization is an efficient method for uniform dispersion of CNT's in a thermo setting polymers. The advantage of this method is that covalent bonding can be formed between functionalized CNT's and polymer matrix, hence much improved mechanical properties of composite through strong interfacial bonds [14].

The fabrication of polymer nanocomposites mostly done through resin transfer moulding and hand lay-up method. The phenomena like void formation, wetting, degree of curing, resin flow, processing time and reinforcement ratio effect on the composites. Hand lay-up method is a reliable process; it is by its nature very slow and labor-intensive. Resin transfer moulding is another method used for complex shapes at relatively at high production rates [24], [25].

It should be noted that nanocomposite materials are significant in the emerging field, many studies are being made to devise new processing methods that can produce nanocomposites with unique structure and properties for required and for the specified applications [21-23].

In the present study, for three different orientations and for the proposed percentages of carbon nanotubes composites are fabricated. By using magnetic stirrer, the proposed percentages of CNT are mixed and are followed by hand layup technique. The mechanical properties are investigated.

II. Experimental Details

A. Materials

The fiber chosen were woven roving glass mat for fabricating specimens. Polyester, epoxy, phenolic and vinyl ester are mostly used as thermoset resins, the matrix materials used is LY556 (Bisphenol-A-Diglycidyl-Ether) epoxy resin and hardener is HY951(Tri-ethylene-Tetramine). Multi walled carbon nanotubes are selected as the filler material for the high tensile modulus and strength, low aspect ratio, low density, high flexibility, excellent thermal and electrical properties. These are allotropes of carbon, composed of two or more concentric cylindrical shells of graphene sheets with diameter in the nano meter scale.

B. Preparation of composite specimens

The surface is cleaned. For the proposed percentage (0%, 1%, 3%, 5% & 7%) of multi walled carbon nanotubes is uniformly dispersed in the matrix material by stirring process. Fabrication process was done for three different oriented specimens: in $0^0/90^0$ oriented specimen, the fiber direction is same for the five layers; in $0^0/45^0$ oriented specimens the alternate layer fiber are directed towards positive 45 direction and in $0^0/135^0$ oriented specimen, the alternate layers fiber are directed towards 135 direction, showed in fig. 1-3. The polymer matrices are poured uniformly and air bubbles were removed carefully with a roller. It was kept under pressure and cured for 24 hours. The length, width and thickness of each specimen were approximately 300mm X 300mm X 3mm respectively.

Fig 1: specimen containing 0% MWNT's for 0/90 deg orientation.

Fig 2: specimen containing 1% MWNT's for 0/90 deg orientation.

Fig 3: specimen containing 7% MWNT's for 0/90 deg orientation.

C. Mechanical Testing

After the fabrication of specimens, the tensile strength of the composite was measured with a universal testing machine in accordance with the ASTM D638 procedure at a cross head speed of 2mm/min. The flexural test was performed on the same machine, using the three point bending fixture according to ASTM D790 with the cross head speed of 2mm/min. The hardness tests were performed on the Rockwell hardness machine in accordance with the ASTM D785 using M scale.

D. Statistical Procedure

In statistical modeling, regression analysis is a statistical process for estimating the relationship among variables. The focus is on the relationship between a dependant and one or more independent variables. A form of regression analysis in which data is fit to a model expressed as a mathematical function. A non linear regression must generate a line (typically a curve) as if every value of Y was a random variable.

A non linear regression analysis was done to establish a relationship of tensile strength, flexural strength and hardness with respect to the experimental data of specimens [26]. In order

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to calculate error and to compare between the experimental data and the regression based data for each individual specimen graphs are plotted. Analysis was performed using Mini Tab software.

III. Results And Discussions

A. Morphological properties

Scanning electron microscopy is used to investigate the dispersion of carbon nanotubes and surface morphology of the composite. The voids and pores may lead to stress concentration points and leads to premature failure of the composite during loading. As the cracks got initiated, the matrix materials were lost, mostly in the form of resin particles. The SEM evidence in fig 4 and 5 supports this explanation. Therefore, studies of the surface topography on composite provide information on the interfacial adhesion that exists between the fiber and matrix material when reinforcing agent is incorporated. Homogeneous distribution of reinforcing agent MWCNT's leads to improvement of strength and toughness due to strong interaction between MWCNT's and matrix materials. The well dispersion of MWCNTs may have an effect of physical cross linking points, thus increase the tensile strength. The increase in tensile strength is observed for MWCNTs percentage up to 5%, the reduction in the tensile strength observed for MWCNTs percentage higher than 5%. It should be rather associated with aggregation, which increased with increasing MWCNTs percentage [24],[25]. Due to aggregation, the bonding between the fiber and matrix material becomes weak. Hence by loading on the composite, stresses leads the composite to failure in fig 6. Fig. 7 shows the fracture generated in specimen, when placed in tensile experiment. The fig 8 & 9 shows the orientation of the fiber in the composite at an angle 45 deg and 90 deg respectively.

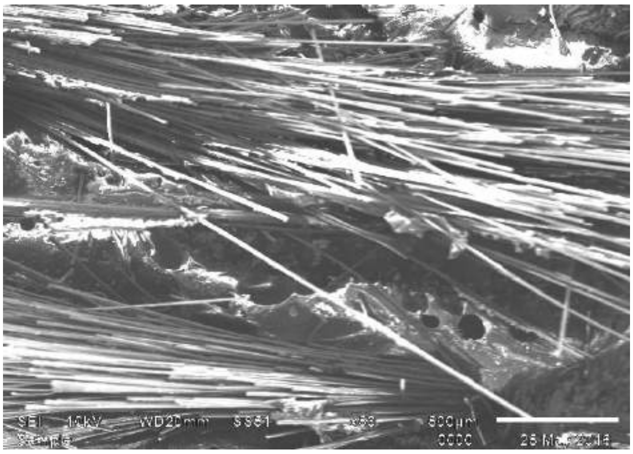

Fig. 4: SEM image showing matrix cracking and voids effect.

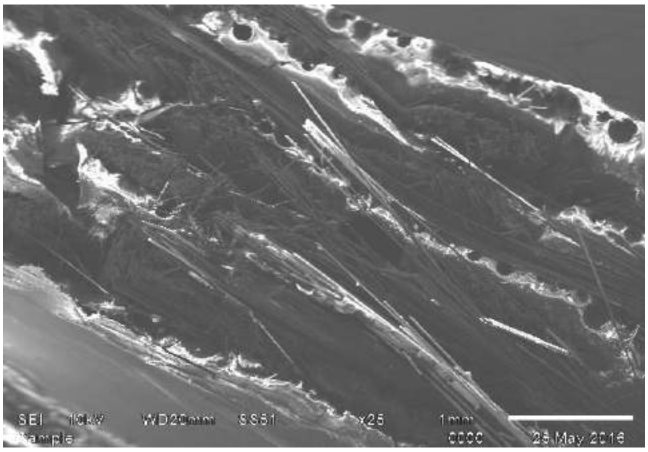

Fig. 5: SEM image showing fracture running along fiber-matrix material and flexural tested specimen.

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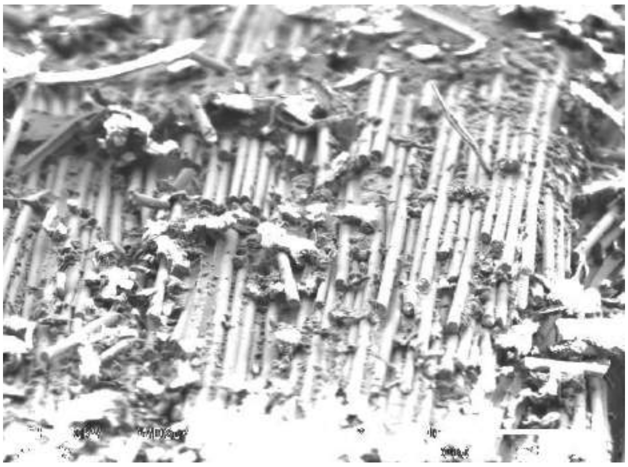

Fig. 6: SEM image showing agglomeration for 7% MWNT's for 0/90 deg specimen.

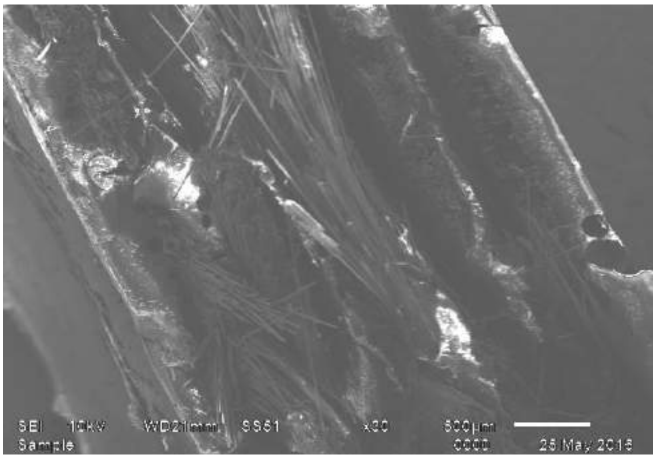

Fig. 7: SEM image of the fracture surface of the tensile tested specimen for 0/90 deg specimen.

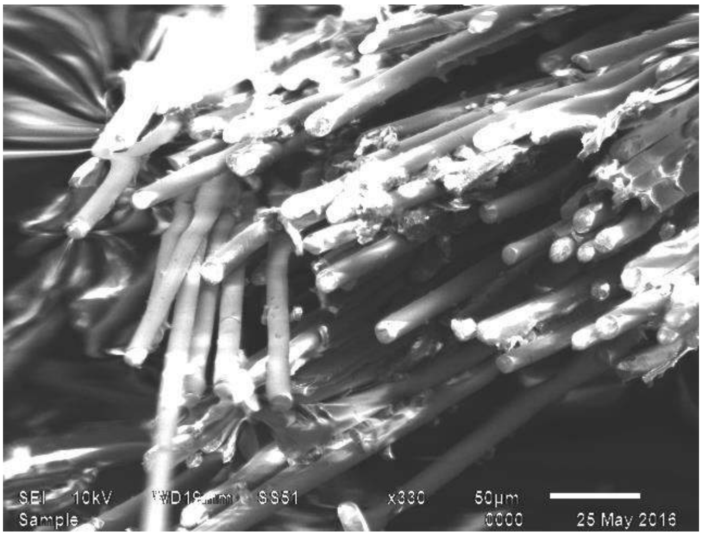

Fig. 8: SEM image showing orientation of fiber for 0/45 deg specimen.

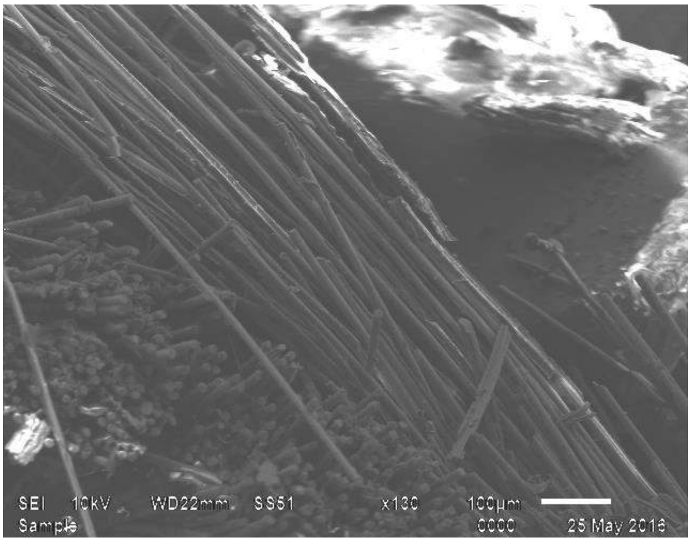

Fig. 9: SEM image showing orientation of fiber for 0/90 deg specimen.

B. Mechanical Properties

Fig. 10-12 shows the load and displacement with time represents the curves for highest values of tensile stresses from three different orientations of specimens. Among the three orientations, the maximum value of tensile strength and load withstand ability is obtained for 0/90 deg orientation specimen with 5% of carbon nanotubes. The curves are almost linear as shown in fig 10-12. The tensile properties of the composite increases with increase in percentage of carbon nanotubes because it is main load bearing element.

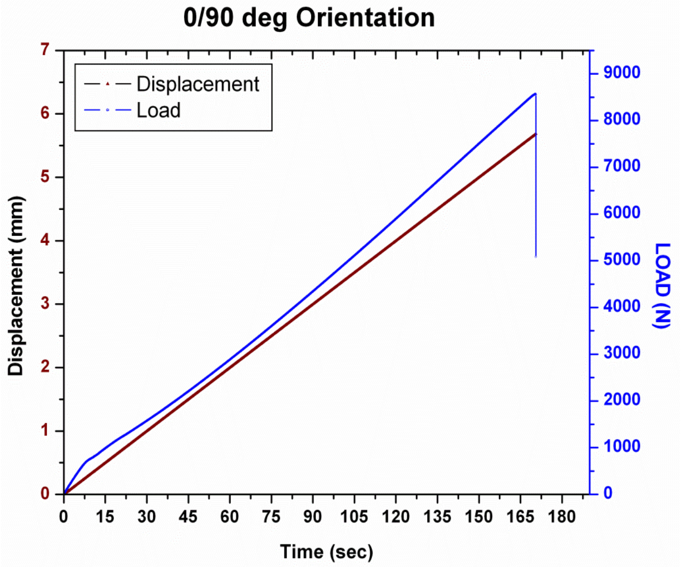

Fig.10: Load and Displacement curve for 0/90 deg oriented specimen.

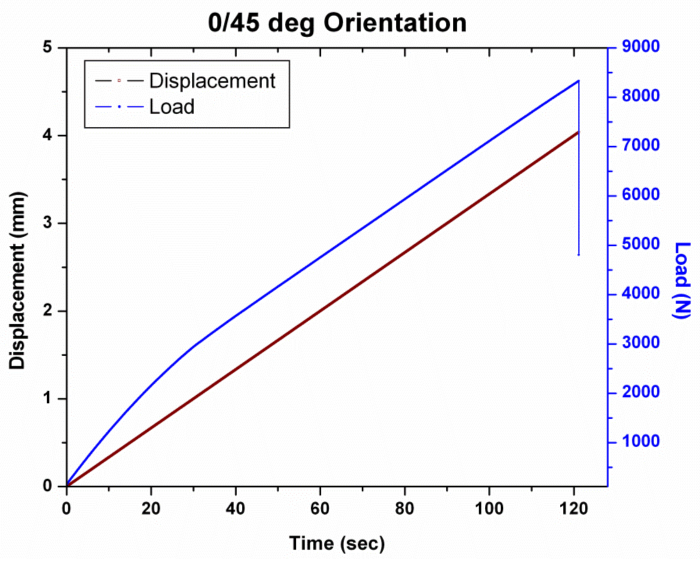

Fig.11: Load and Displacement curve for 0/45 deg oriented specimen.

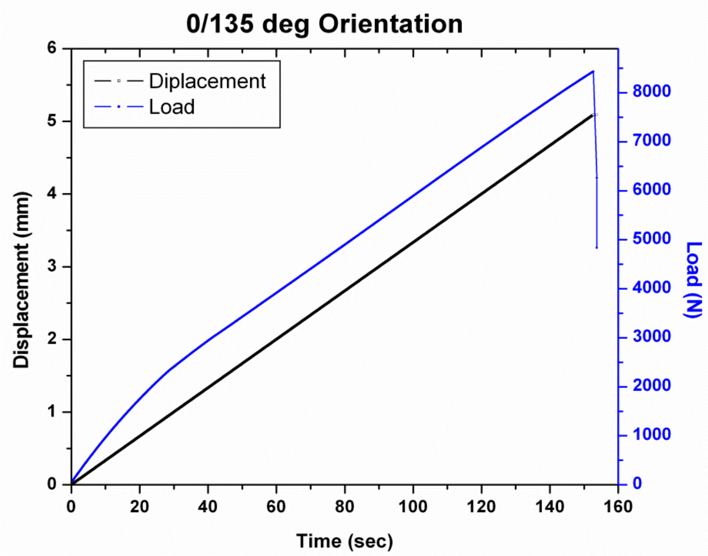

Fig.12: Load and Displacement curve for 0/135 deg oriented specimen.

C. Effect of Tensile Strength

Tensile strength is the limit of a material for withstanding tensile stress without causing damage to the material and resists the expansion of the material. The improvement in the tensile strength is due to strong interaction between carbon nanotubes and polymer matrix, which leads to good dispersion of carbon nanotubes in polymer matrix. When the percentage of CNT above 5%, the dispersion of carbon nanotubes is poor and agglomerates takes place. Hence the tensile strength decreases.

Fig. 13-15 shows the effect of carbon nanotubes on the tensile strength for three different orientations. For $0^0/90^0$ orientation, if 1% of filler material is added, it is improved by 1.3% of strength. If 3% is added, it is improved by 45.1%. If 5% is added 50.82% improvement is seen. Beyond 5% level the tensile starts decreasing. The highest strength is obtained for at 5% in the three orientations. In similar way for 0/45 degrees and 0/135 degrees orientated specimens the tensile stresses varies.

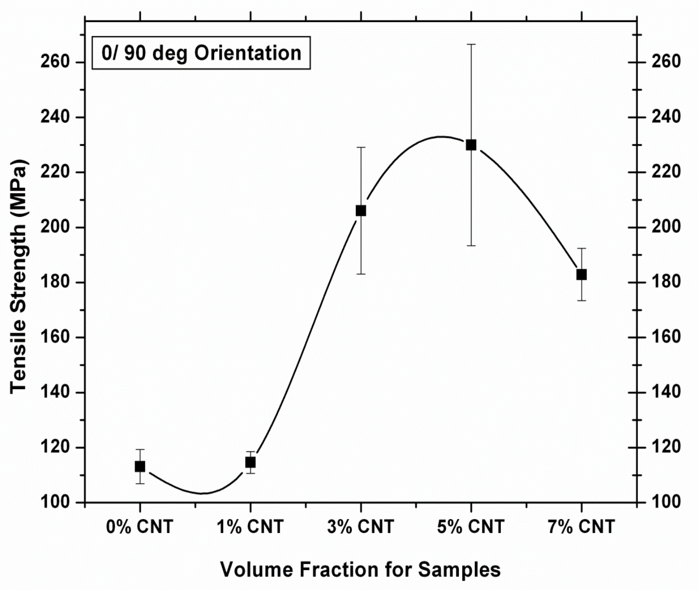

Fig. 13: Variation of tensile stresses.

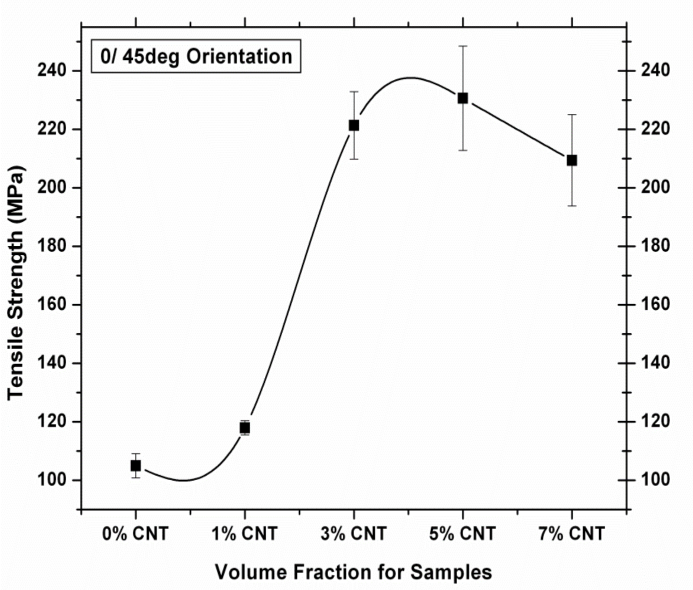

Fig. 14: Variation of tensile stresses.

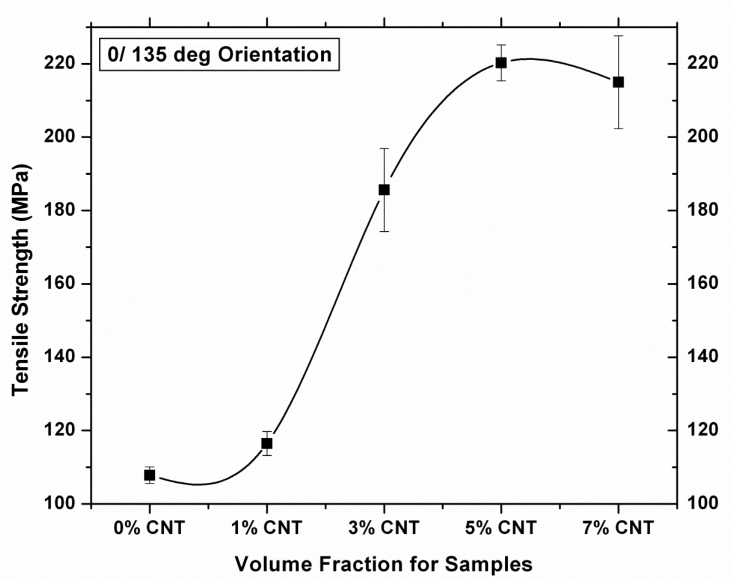

Fig. 15: Variation of tensile stresses.

D. Effect of Flexural Strength

Another one of the most important and widely measured properties of composite material is flexural strength. The flexural strength represents the highest stress experienced within the material just before it yields in a flexural test.

Fig. 16-18 shows the effect of carbon nanotubes on the flexural strength for three different orientation specimens. For $0^0/90^0$ orientation, if 1% of filler material is added, it is improved by 5.2% of strength. If 3% is added, it is improved by 15.5%. If 5% is added 4.7% improvement is seen. Beyond 3% level the flexural strength starts decreasing. The highest strength is obtained for at 3% in the three orientations and in the same way for other specimens.

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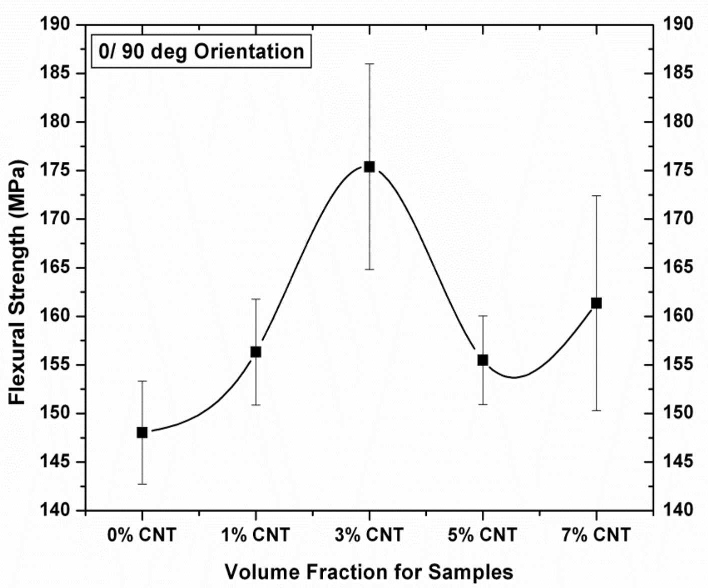

Fig. 16: Variation of flexural stresses.

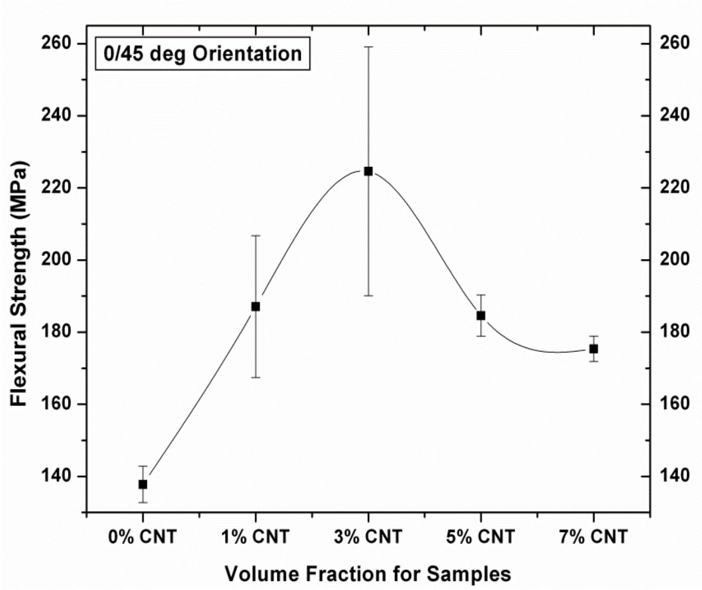

Fig. 17: Variation of flexural stresses.

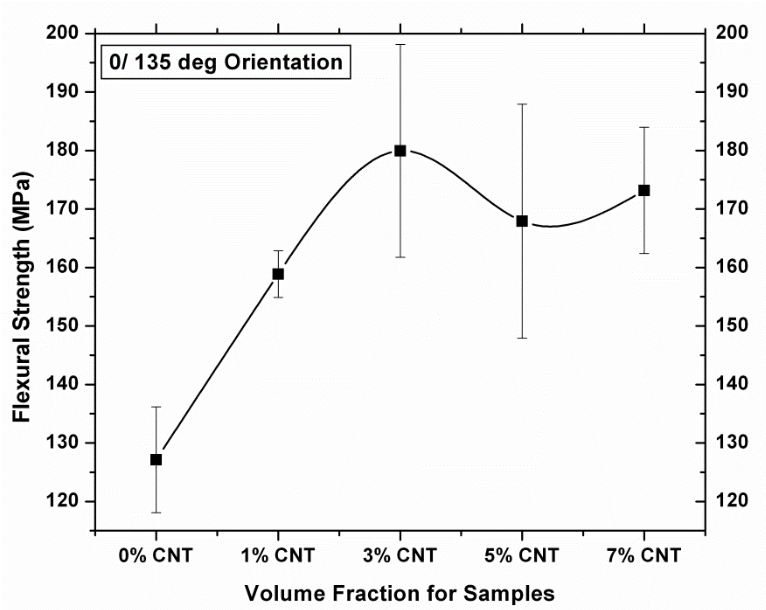

Fig. 18: Variation of flexural stresses.

E. Effect on Hardness

Hardness is a characteristic of a material and it defines as resistance to indentation in specimens. The hardness of carbon nanotubes based composite increases with increasing percentage of carbon nanotubes upto 5%. However it decreases when CNT% increases above 7% due to the agglomeration of CNTs.

Fig. 19-21 shows the effect of carbon nanotubes on the hardness for different oriented specimens. For $0^0/90^0$ orientation, if 1% of filler material is added, it is improved by 1% of

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strength. If 3% is added, it is improved by 2.55%. If 5% is added 4.97% improvement is seen. Beyond 5% level the hardness starts decreasing. The highest hardness is obtained for at 5% in the three orientations.

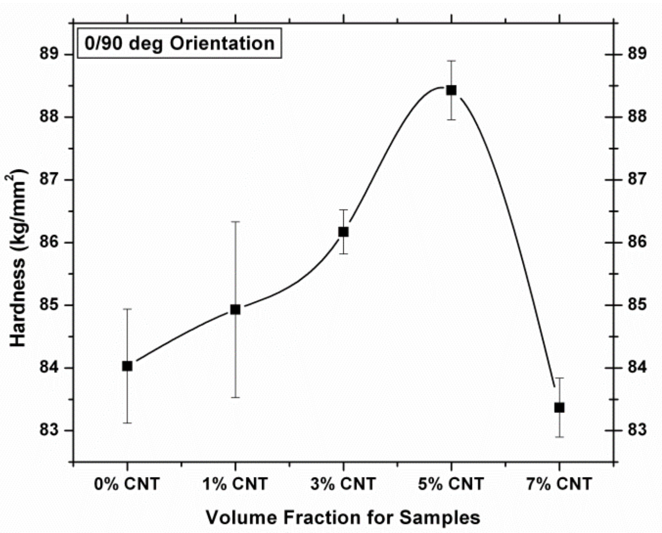

Fig. 19: Variation of hardness.

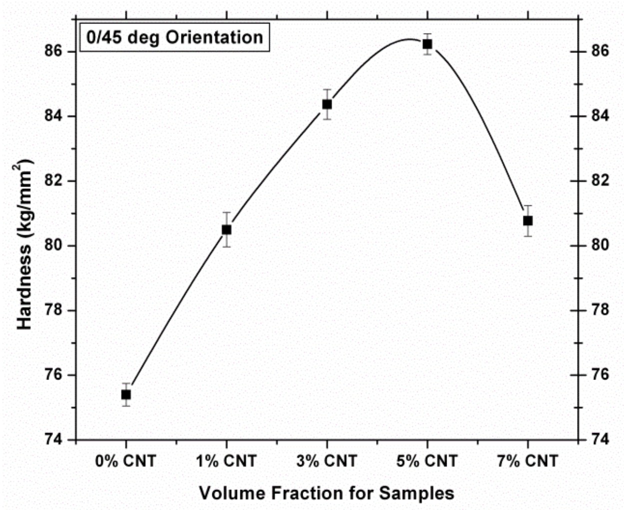

Fig. 20: Variation of hardness.

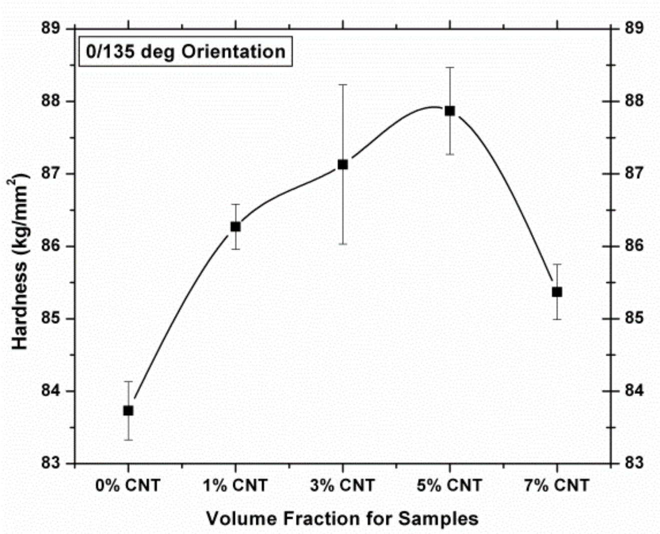

Fig. 21: Variation of hardness.

F. Development of Regression Model

In statistics, regression analysis is the technique for the modeling and finding analysis of numerical data consisting of values of dependent variables(response variables) and one or more independent variables(predictors).several prediction techniques have been used to model the mechanical properties of composite materials based on different parameters.

Regression model is most useful method in determining the mechanical properties of CNT-reinforced polymer composite. A positive effect is achieved, so we are considering tensile and flexural strength data for statictical prediction using regression model. The experimental data was used to generate a mathematical model using regression analysis.

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By using that equation, an error can be found by placing the experimental values in that equation. By comparing the experimental values and regression values an error can be found for tensile stress, flexural stress and hardness.
Regression equation is formulated to get the relation between response/dependent variables i.e., tensile/flexural strength/hardness (Y) and the input parameters i.e., volume percentage (X).

For tensile strength

It was observed that while considering tensile strength of the composite for three different orientations, the regression model was found to be in agreement with experimental results. Figs. 22-24 represent the comparison between the experimental and regression values of tensile strength values.

The regression equations for tensile strength were developed as:

For 0/90 deg specimens: $Y = 90.23 + 57.45 (X) - 5.722 (X)^2$ (1)
For 0/45 deg specimens: $Y = 92.31 + 51.35 (X) - 4.857 (X)^2$ (2)
For 0/135 deg specimens: $Y = 91.09 + 46.27 (X) - 4.301 (X)^2$ (3)

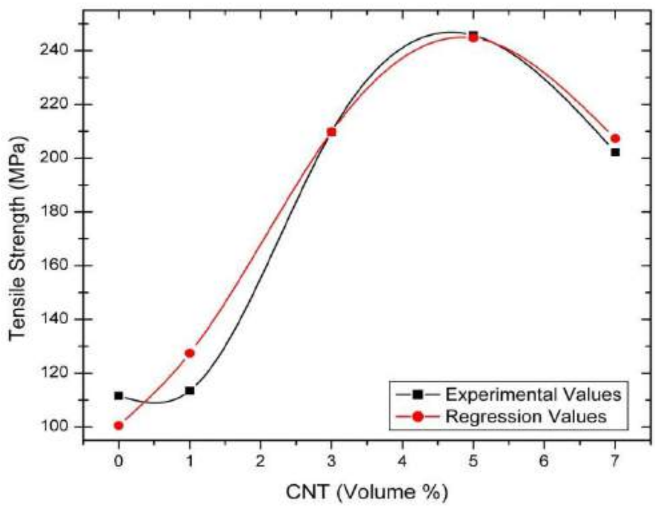

Fig. 22: Comparison of experimental and regression tensile strength values for $0^0/90^0$ orientation specimen.

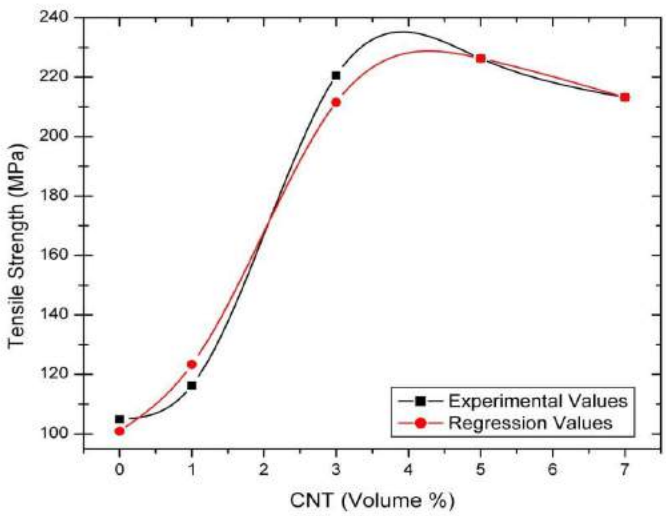

Fig. 23: Comparison of experimental and regression tensile strength values for $0^0/45^0$ orientation specimen.

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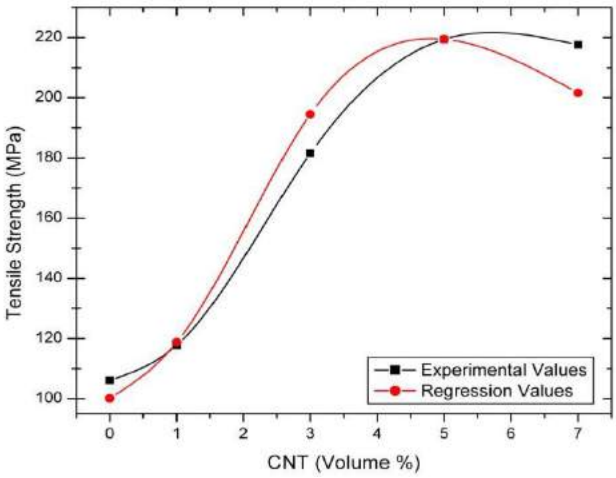

Fig. 24: Comparison of experimental and regression tensile strength values for $0^0/135^0$ orientation specimen.

For flexural strength

It was observed that while considering flexural strength of the composite for three different orientations, the regression model was found to be in agreement with experimental results. Figs. 25-27 represent the comparison between the experimental and regression values of flexural strength values.

The regression equations for flexural strength were developed as:

For 0/90 deg specimens: $Y = 145.3 + 12.00 (X) - 1.737 (X)^2$ (4)

For 0/45 deg specimens: $Y = 150.4 + 40.02 (X) - 5.983 (X)^2$ (5)

For 0/135 deg specimens: $Y = 130.7 + 16.97 (X) - 2.051 (X)^2$ (6)

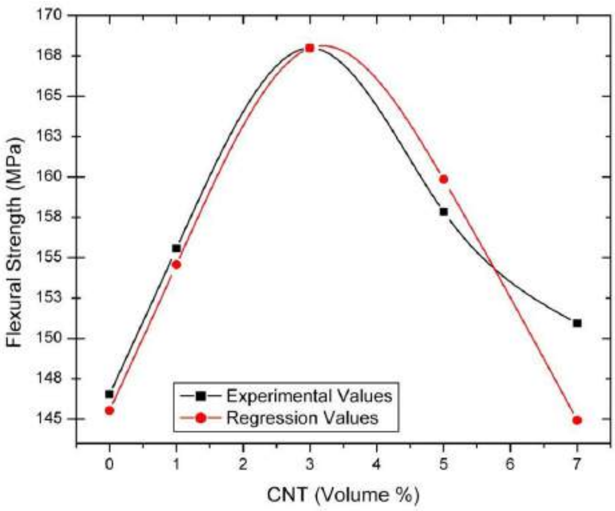

Fig. 25: Comparison of experimental and regression flexural strength values for $0^0/90^0$ orientation specimen.

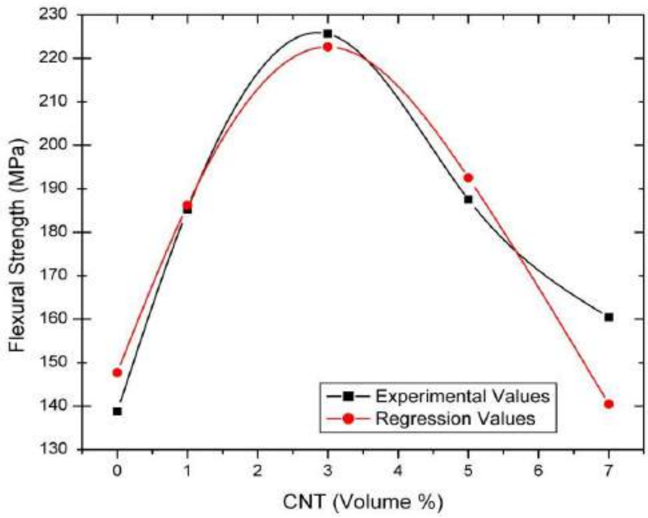

Fig. 26: Comparison of experimental and regression flexural strength values for $0^0/45^0$ orientation specimen.

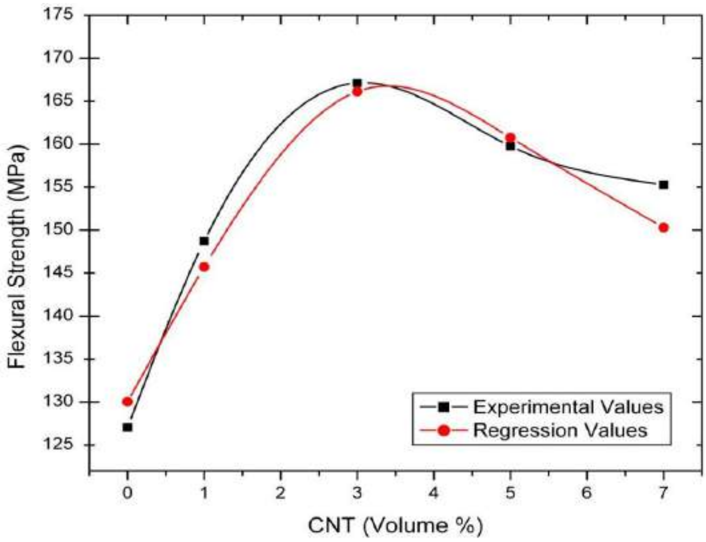

Fig. 27: Comparison of experimental and regression flexural strength values for $0^0/135^0$ orientation specimen.

For Hardness

It was observed that while considering hardness of the composite for three different orientations, the regression model was found to be in agreement with experimental results. Figs. 28-30 represent the comparison between the experimental and regression values of flexural strength values.

The regression equations for flexural strength were developed as:

For 0/90 deg specimens: $Y = 82.94 + 2.253\,(X) - 0.2563\,(X)^2$ (7)
For 0/45 deg specimens: $Y = 83.60 + 2.022\,(X) - 0.2496\,(X)^2$ (8)
For 0/135 deg specimens: $Y = 75.57 + 4.975\,(X) - 0.5998\,(X)^2$ (9)

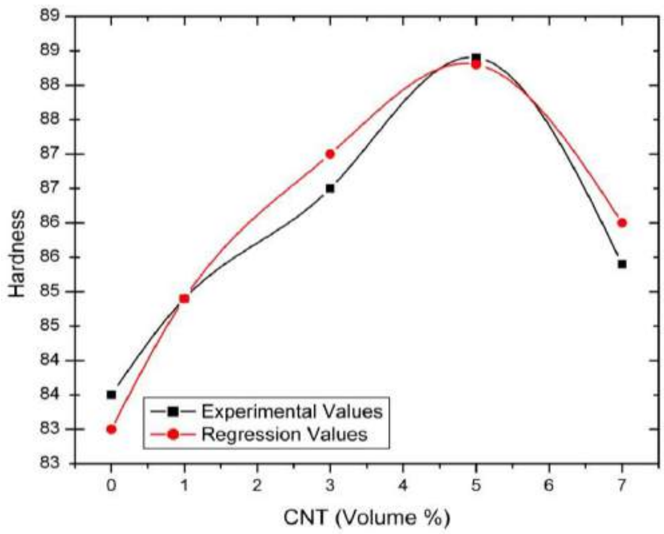

Fig. 28: Comparison of experimental and regression hardness values for $0^0/90^0$ orientation specimen.

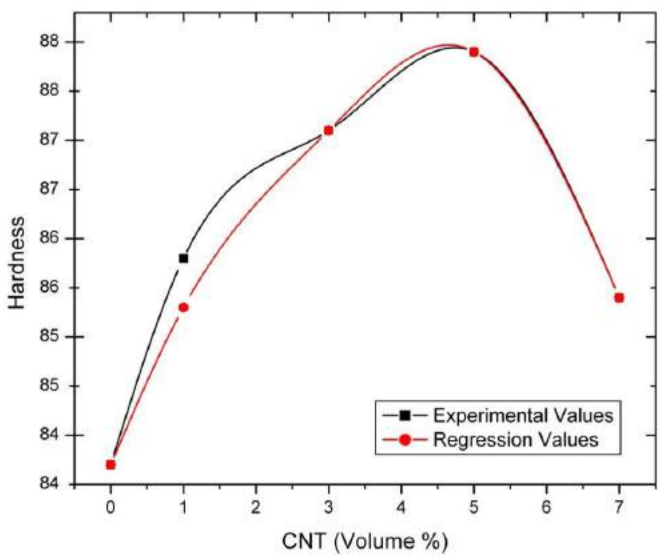

Fig. 29: Comparison of experimental and regression hardness values for $0^0/45^0$ orientation specimen.

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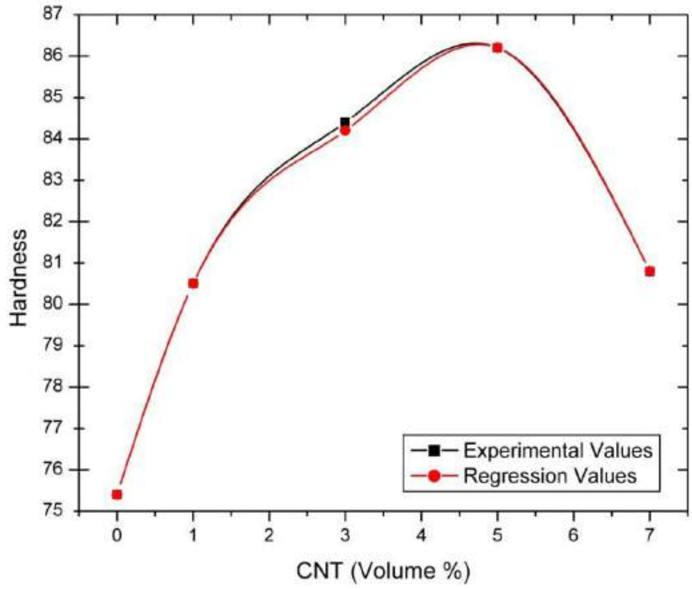

Fig. 30: Comparison of experimental and regression hardness values for $0^0/135^0$ orientation specimen.

The square residual values (R^2) of tensile and flexural strength for proposed percentage of multi walled carbon nanotubes were found to vary between 0.97 to 0.98 respectively in the regression model. As R^2 of 1.0 represent that the regression curve perfectly fits the data. R^2 is a statistic that give information about the fit of curve for collected experimental data. In regression, the R^2 coefficient of determination is the measure of how well the regression line approximates the real data points. The fig 15 to 20 shows the comparision between the experimental and predicted strength values. The table 1 represents the experimental values and regression values for tensile,flexural and hardness properties for all specimens.

TABLE I: MECHANICAL PROPERTIES DATA OF EXPERIMENTAL AND REGRESSION ANALYSIS.

	Tensile Strength (MPa)			Flexural Strength (MPa)		Hardness	
Orientation	**MWCNT%**	**Exp. data**	**Reg. data**	**Exp. data**	**Reg. data**	**Exp. data**	**Reg. data**
0-90 deg's	**0**	111.5	100.5	146.54	145.54	84.0	83.0
	1	113.42	127.42	155.57	154.57	84.9	84.9
	3	209.77	209.77	167.98	167.98	86.2	87.0
	5	245.73	244.73	154.85	159.85	88.4	88.3
	7	202.28	207.28	156.93	144.93	83.4	86.0
0-45 deg's	**0**	104.91	100.91	136.74	147.74	83.7	83.7
	1	116.31	123.31	185.26	186.26	86.3	85.3
	3	220.51	211.51	225.62	222.62	87.1	87.1
	5	226.32	226.32	184.54	192.54	87.9	87.9
	7	213.15	213.15	171.44	140.44	85.4	85.4
0-135 deg's	**0**	106.13	100.13	125.03	130.03	75.4	75.4
	1	117.76	118.76	161.71	145.71	80.5	80.5
	3	181.56	194.56	167.11	166.11	84.4	84.2
	5	219.44	219.44	159.76	160.76	86.2	86.2
	7	217.57	201.57	159.30	150.30	80.8	80.8

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IV. Conclusion

The tensile, flexural and hardness properties of carbon nanotubes based composite are studied. The mechanical properties are increasing continuously due to the addition of carbon nanotubes as filler material. The studies are carried out for both, the orientations of the glass mat and also by varying the percentage of carbon nanotubes of composite. By adding some percentage of CNT in the composite, we could get better mechanical properties due to interfacial interaction between carbon nanotubes and polymer matrix and then decrease due to agglomeration. It is demonstrated by the present study that the composite can be improved by adding the percentage of carbon nanotubes.

A relationship between experimental and predicted (regression model) values are determined to be strong by the high R^2 values which were obtained. This means that a good relationship can be expected. The comparison between experimental and predicted values showed that they were in good agreement.

To fabricate advanced composites, collective efforts are required from various fields of material science. Uniform dispersion, interaction between filler material and matrix, temperature and pressure are the sensitive conditions which affects the properties of composites. If we could control these parameters, an advanced material of desired properties would come to existence.

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