

Dynamic Effect Analysis And Flexural Storage Modulus Optimization Of CFRP Hybrid Nano Composites Using RSM

Shailesh D. Ambekar, Vipin Kumar Tripathi, Huynh Tan Hoi

Abstract: In the last few years, due to the enhancement in the properties of CFRP with the presence of nano particles, the interest is increased in studying the use of CFRP hybrid nanocomposite. In this paper, the effect and evaluation of mixing of nano clay nanoZnO particles and carbon fiber plies angle orientation on dynamic mechanical properties laminates of CFRP were studied. A hand layup method followed by process of vacuum bagging was used to make the samples of composite. Dynamic mechanical analyzer (DMA) was used to inspect the dynamic mechanical properties of CFRP hybrid nanocomposite specimens with changing temperature. The response surface methodology is used for design of experiments by considering the percentage of weight of nanoclay, nanoZnO and fiber orientation angle as an independent variables and storage modulus due to the flexural loading is used as response. The optimization of the storage modulus is carried out with RSM response optimizer and optimum value of storage modulus is found.

Keywords: Dynamic mechanical analysis (DMA); Response surface methodology (RSM); hybrid nanocomposite; nanoclay; nanoZnO

1. INTRODUCTION

FRP hybrid nano composites combine two or more types of nano particles as additional reinforcement in a matrix. The composites mechanical properties are dependent on the properties of matrix, reinforcement fiber and the interaction of nano particles with them. So, making of FRP hybrid nanocomposites gives improved mechanical properties as compared to the conventional composites with reduced costs. Carbon fiber is most favorite reinforcement for composite structures due their capacity to advance the mechanical properties and tribological of polymer composites, also, carbon fiber has outstanding thermal stability, Young's modulus, superior strength, healthier thermal conductivity, and exceptional electrical properties than other fibers [1-3]. FRP composites are used in various applications where elevated mechanical properties are required, such as automobile, aerospace etc. Deca et al establish that there is improvement in the UV resistance and thermal stability in the polymers of wood with the inclusion of nanoZnO and nanoclay[4]. Rashmi, et al. and Li Chang et al. presented work on the improvement in the wear resistance of regular size and short carbon fiber composites in addition of nano particles. [5,6]. M.A. Shazed and A.R. Suraya showed that The CNT are also supportive in enhancement of mechanical properties of CFRP composites, when coating of h CNT applied with different fiber orientations [7]. As per David A. Hawkins et al presented that the significance of the nanoparticles on the upgrading the toughness of CFRP composites [8] The astonishing mechanical, thermal and electrical properties of carbon nano tubes and nanoZnO have provoked researches to make use of them as a filler in composite in order to perk up the properties of the parent matrix[9,10]. In the study by Declan Carolana et al discussed that the nanoparticles are eagerly transferable to a toughness

increase in the interlaminar fracture energy of the CFRP composite laminates [11]. With addition of nanoclay, the glass transition temperature can be increased as compared to neat CF/PPc composite demonstrating improved heat resistance and improved thermo mechanical properties with addition of organoclay as per the work of Mohamed H. et al [12]. Also many researchers have enlisted the effect of nanoparticles, fiber orientation etc, on the various carbon fiber composites by using dynamic mechanical analysis [13-18] M. B. A. Salam and M. V. Hosur et al. highlights on the effect of addition of multiwalled carbon nano tubes in carbon fiber, epoxy composites, which end results in improvement of the thermo mechanical properties of the composites[19,20]. Hasan Yavuz Unal et al. had worked on the Comparison of the Experimental Mechanical Properties experimentally and measurement by DMA of Nanoclay Hybrid Composites, The results of this study shows that the mechanical properties was positively affected by addition of nanoclay into epoxy resin[21]. Likewise it is also studied that carbon nano fibers, natural nano banan particles and nanoclay having stronger effect on the mechanical and viscoelastic properties of the composites as shown by researchers in their respective studies[22-24]. Sasan Nouranian, et al. have done the optimization of the nano composites for studying their effect in DMA[25]. Yasser Rostamiyan et al, [26-28] mixed epoxy resin with nanoclay as nanoparticle in the glass fiber reinforcement and studied its effects on mechanical properties, and found that new nanocomposite possesses improved mechanical properties like flexural strength, tensile and impact These studies are carried out using the response surface methodology for the design of experiments. R. Satheesh Raja et al have studied effective improvement in the the mechanical properties of GFRP composites with the implementation of micro fly ash. The results were statistically analysed using Central Composite Design (CCD) method [29]. So by the proof of researchers we can say that Central Composite Design can efficiently used to design the experiments, for analyse the results of experiments with respect to the mixing or combining of nano particles with epoxy and the FRP. Shailesh Ambekar and V.K Tripathi has done the

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optimization of the flexural strength by using the RSM and found that presence of the nanoparticles had positive effect on the static flexural strength [30]. V.K.Tripathi and N. S. Kulkarni worked on optimal design comprehensive procedure of a composite laminate taking various parameters [31]. U.V. Hambire and V.K.Tripathi have done the evaluation and optimization of dental composites for mechanical properties [32]. Pulak M Pandey and Gurminder Singh have studied effect of unit cell shape and strut size on flexural properties of ordered Copper foam [33]. There are many optimization algorithms which have been used in different problems. Guangran Zhang and E. H. Chimowitz have used a Markov Chain-Monte Carlo algorithm for Energy network optimization [34]. In this paper the effective dynamic analysis of the CFRP hybrid nanocomposites for the storage modulus under flexure is experimentally studied. The design of experiment is prepared by with CCD(Central composite design)the part of response surface methodology, which has been employed to develop the function of physical factors in terms of mathematical models to predict DMA performance of hybrid CFRP nanocomposite and also used to present a the optimum design of new CFRP hybrid nanocomposite. Experimental

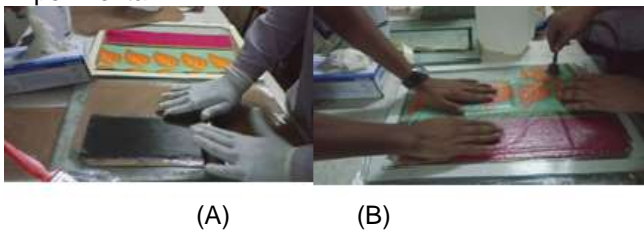


Fig. 1: (a) Hand Lay up of CFRP, (b) Vacuum Bagging

2.1. Details of Material

For the experiments in this evaluation CFRP epoxy composites treated as controlled sample. The unidirectional carbon fibrics having standard modulus 3K of 200 GSM ,code HCU200. The resin named Hinpox C, which is Bisphenol -A based liquid resin along with the amine modified hardener known as Hinpox hardener-B is a

colorless, and low viscosity liquid with stoichiometric ratio of A:B=10:3 was used. Resins, hardener along with carbon fiber ,supplied from the Hindoostan Mills Ltd. Mumbai, India. The nanoclay was montmorillonite, modified surface with layers of Aluminosilicate (approximately thickness of 1 nm), and Sp Gra. 2.6. Zinc oxide -ZnO nanoparticles, with Purity of 99.9%, SSA: 20-60 m²/g, with Particles Size: 30-50nm, Morphology: nearly spherical, True density: 6 g/cm³ density for Bulk: 0.28 - 0.48 g/cm³ were used, supplied from Nano Research Lab. Jamshedpur, (Jharkhand),India [30].

2.2. Fabrication of composites

CFRP hybrid nanocomposites laminates were fabricated by using hand layup method(as shown in Fig.1.(a))succeeded by process of vacuum bagging(as shown in Fig. 1.(b)). The epoxy resin system is a Diglycidyl ether of Bisphenol A (DGEBA) mixed with 1,3-phenylenediamine hardener at a ratio by weight of 100:30. The carbon fibers unidirectional of 200 gsm were used as main reinforcement in the matrix. The Montmorillonite nanoclay and Zinc Oxide nanoparticle were dried for overnight at 75°C in an oven before use for make appropriate for spreading in epoxy resin. The nano particle content was preferred in between the 1% to 5% for mixing in the epoxy resin and hardener. For the making of specimens, 14-16 nos. plies were used according to ASTM standards, accordingly the sub specimens are cut of the required size. For executing the mixing of the particles uniformly in the resin mixture, the ultra sonication of the mixture was done for 40 minutes at 40° C temperature, and then this mixture is magnetically stirred with 800 rpm for one and half hour. After completing the mixing treatment he hardener is mixed and this mixture is used for the preparation of the CFRO hybrid nanocomposites laminates by hand layup succeeded by process of vacuum bagging as shown in Fig.1. For process of vacuum bagging, pump was used for the period of 5 hours with a stable of 720 mm Hg vacuum pressure. And the specimen was put in the bag for 16 hours duration of curing at room temperature. After the curing, specimen was kept in an oven for process of post curing for five hours at 80°[30].

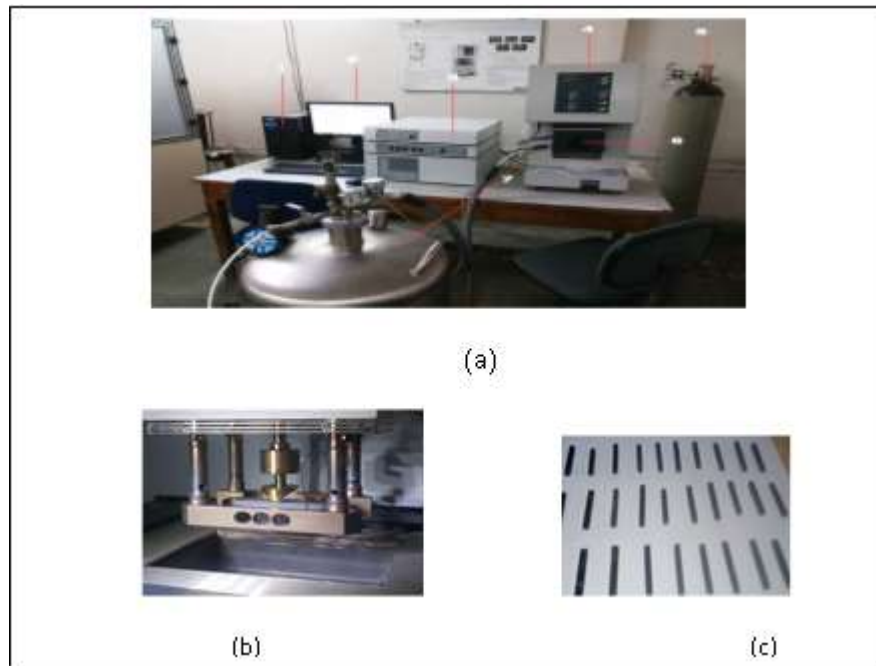


Fig.2: (a) DMA 242 E Artemis set up, (b) Specimen Holder in DMA setup, (c) Specimens of coded units

3. DYNAMIC MECHANICAL ANALYSIS (DMA)

DMA is an extensively used method to determine the effect of temperature on the properties of damping and various moduli of materials by application of sinusoidal force. Modulus is calculated in the 2 parts, these are storage modulus, E' and loss modulus, E'' . The relation of loss modulus to the storage modulus is known as mechanical damping (given by $\tan \delta$ is a dimensionless number) it gives the energy lost or can also be expressed in form of recoverable energy. The higher $\tan \delta$ value indicates that material has higher nonelastic strain, while lower δ shows that material is more elastic. Storage modulus elaborates the elastic behavior of the CFRP and which is proportionate to the energy stored in one cycle, while loss modulus gives the viscous properties of material, which is proportionate to energy dissipated in one cycle. The phase angle, δ which is the phase difference between the dynamic stress and strain of viscoelastic material under the effect of sinusoidal oscillation. This dynamic mechanical test is used to derive the viscoelastic and elastic behaviors of CFRP hybrid nanocomposites sample with the sinusoidal force application. The thermal analyses were completed by using a DMA242E analyser (Netzsch, Germany) in a mode of three-point-bending with the oscillation of 1 Hz (as shown in

Fig. 2.(a)). The storage modulus (E'), damping factor ($\tan \delta$) and loss modulus (E'') of each CFRP hybrid nanocomposite sample were determined under the subsequent conditions: a temperature range from 40°C up to 150°C at rate of heating of $3^{\circ}\text{C}/\text{min}$, as a cooling agent the liquid nitrogen is used by putting in the machine fixture (as shown in Fig. 2.(b)). The samples were sized in 40 mm in length and 4 mm in width, while the thickness were around 3.2 mm (as shown in Fig. 2.(c)).

4 .EXPERIMENTAL DESIGN

The experimental design was done by using the RSM, RSM (Response surface methodology) is the techniques for multidimensional mapping of response patterns for different levels of factors that are used to direct physical processes. RSM makes use of regression analysis of the data from several levels experiments carried out and can be applied to find approximate minimum or maximum points in response. In recent era, RSM played an important job in various fields like nanotechnology, biotechnology, etc. One of the abilities of RSM is used to develop a correct efficient model between output response (Y) and set of independent variables.

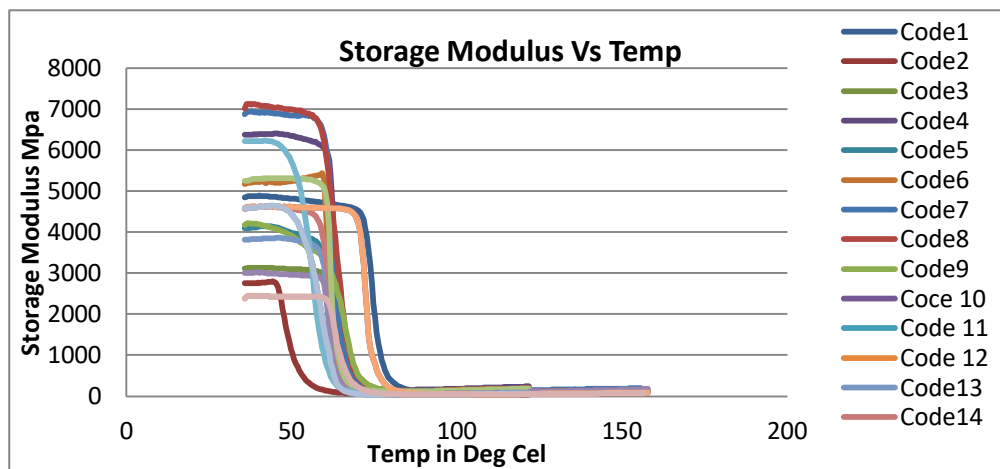


Fig. 3: Graph showing the effect of temp on storage modulus of the CFRP hybrid nano composites coded samples

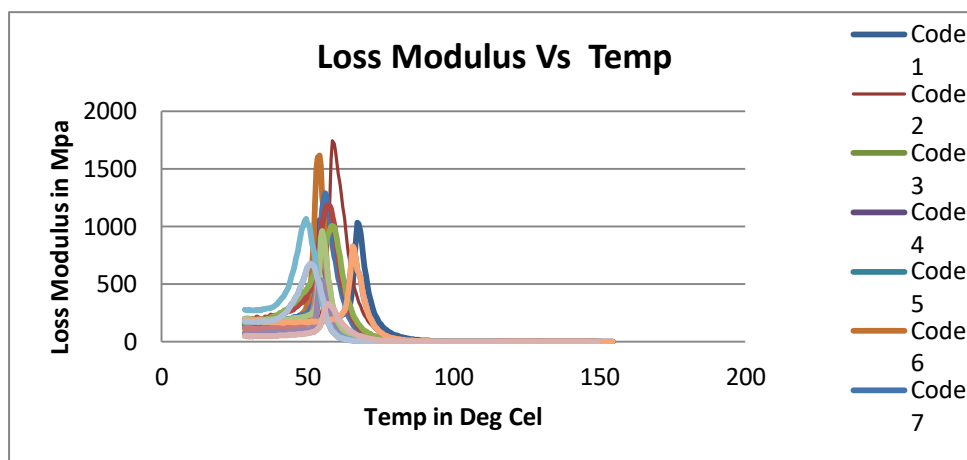


Fig.4: Graph showing the effect of temp on loss modulus of the CFRP hybrid nanocomposites coded samples

The following equation gives the value of Y

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \dots + \beta_k X_k + \epsilon$$

(1)

Where X_1, X_2, \dots, X_k are process independent variables and β_0 is constant coefficient, ϵ is the observed error and β_k is the coefficient factor in the system. This second order polynomial model, if there is form of curvature will be displayed as

$$Y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i < j} \beta_{ij} X_i X_j + \epsilon$$

(2)

In this experiment the software of Minitab was used to interpret and analyze the data processing and to obtain results. To design of experiments CCD was chosen. Here the input variables were nanoZnO wt%, nanoclay wt% and fiber orientation angle and the response was the storage and loss modulus[30].

Table 1: Actual and Coded Level of design parameters

Factors	Levels			Star Point $\alpha = -1.68179$	Star Point $\alpha = 1.68179$
	Low (-1)	Center(0)	High(+1)	- α	+ α
(A) wt% content of nanoclay	1.8	3	4.2	1	5
(B) wt% content of nano ZnO	1.8	3	4.2	1	5
(C) Fiber orientation angle	18.2°	45°	71.2°	0°	90°

Table 2: Experimental Design and corresponding responses

Code Sr. No	Coded Value A	Coded Value B	Coded Value C	Storage Modulus MPa	Loss Modulus MPa
1	1.681793	0	0	4846.46	1046
2	0	0	-1.68179	8016.13	1747

3	-1.68179	0	0	3109	476
4	-1	1	1	6372.54	1046
5	1	1	1	4090.56	784
6	-1	-1	-1	5175.47	1628
7	1	1	-1	6874	1259
8	-1	1	-1	7020.8	1187
9	1	-1	-1	4177.13	1010
10	0	0	0	4583	837
11	0	0	0	4575	680
12	0	0	0	2387	330
13	0	-1.68179	0	3816.73	545
14	0	1.681793	0	4571.76	742
15	0	0	0	4575	680
16	1	-1	1	5246.63	947
17	0	0	1.681793	3001.12	535
18	0	0	0	2387	330
19	-1	-1	1	6220.95	1069
20	0	0	0	4583	837
Controlled Sample				4928	2564

5. RESULTS AND DISCUSSION

5.1 Overall effect of temperature increase on modulus of storage and loss

In the Figure 3 and 4 it can be seen that how is the behavior of the storage and loss modulus of the coded specimen of CFRP hybrid nanocomposites. Storage modulus gives value for the stiffness of a viscoelastic material, which is proportionate to the energy stored during one loading cycle. It is around equal to the elastic modulus for a reversible deformation and rapid stress at low load. Loss modulus gives the value proportionate to energy dissipated in the form of heat during one loading cycle. It is vibration energy that which transformed during vibration and that cannot be recovered. Modulus value is expressed in MPa, It is found that the value of the value of storage modulus is increased upto 40% and the loss modulus is improved by almost 35% as shown in the table in the code number 2 sample. The detailed analysis of the values of storage modulus and its variance is calculated and studied the effects of various independent variables by using RSM.

5.2 Analysis and modeling for storage modulus using RSM

As the nanoclay wt%, nanoZnO wt%, and fiber orientation angle are considered as input variables which are noted as A, B and C respectively for ANOVA ie analysis of variance and the output response considered for analysis, was storage modulus of CFRP hybrid nanocomposites with nanoclay and nanoZnO as filler. In the stage, to examine

the effect of independent variables on consequent response, analysis of variance was conceded out by Minitab software. The actual and coded Level of design parameters is as per the Table 1.

5.2.1. Analysis of variance (ANOVA) and fitting regression model.

By considering the level of parameters as shown in the Table1, the corresponding experimental design and the values of response is displayed in the Table 2. Accordingly when the analysis of variance is carried out for the given set of the results, which is displayed in the Table 3. Then the regression equation derived from using the coefficients in the ANNOVA table 3 is given by Eqn.1. Fig5 (a) shows the graph of residual for normal probability, which demonstrate, is the collected data and allows the fits of distribution. When most of points in data fall nearby to the fitted line for distribution, the model is best suited for the said results. In this Fig 5 (a), the points plotted were falling nearer to the fitted line of distribution, so the distribution of storage modulus was closer to the normal distribution. Graph of residuals Vs fitted point values for ANOVA in Figure 5 (b) which indicates that the residuals were scattered randomly for the analysis, so it can establish that there is no any violation of hypothesis and proposed model was satisfactory [30].

Table 3: Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	9	28066520	3118502	2.96	0.053
Linear	3	5764463	1921488	1.83	0.206
A	1	6122	6122	0.01	0.941
B	1	676248	676248	0.64	0.441
C	1	5082093	5082093	4.83	0.053
Square	3	15383022	5127674	4.87	0.024
A*A	1	318475	318475	0.30	0.594
B*B	1	730914	730914	0.69	0.424
C*C	1	15142570	15142570	14.38	0.004
2-Way Interaction	3	6919035	2306345	2.19	0.152
A*B	1	215306	215306	0.20	0.661
A*C	1	14674	14674	0.01	0.908
B*C	1	6689055	6689055	6.35	0.030
Error	10	10527210	1052721		
Lack- of- Fit	5	4120661	824132	0.64	0.680
Pure Error	5	6406549	1281310		
Total	19	38593730			

Model Summary

S R-sq R-sq(adj)
1026.02 82.72% 68.17%

Regression Equation in Uncoded Units

$$\text{Storage Modulus} = 3833 + 21 A + 223 B - 610 C + 149 A^2 + 225 B^2 + 1025 C^2 + 164 AB - 43 AC - 914 BC \text{---Eqn.1}$$

From the regression analysis and the developed model and summary as well, it can be seen that is able to explain about 82% of the response, i e storage modulus

Figure 5 (c) gives the main effect plot of the input variables. These plots gives the significance of linear terms on response .It shows that the amount of storage modulus rapidly

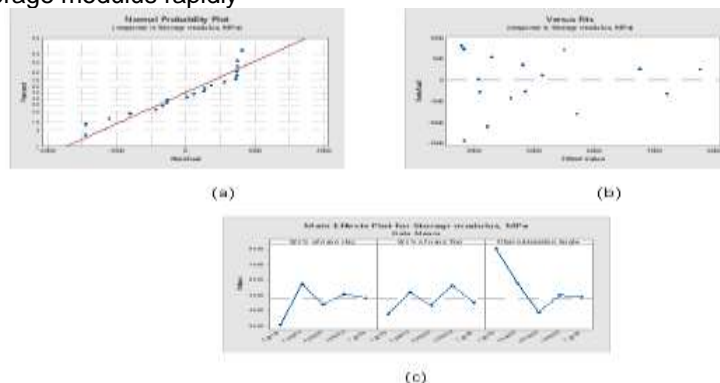


Fig. 5: (a) Normal probability plots for residuals, (b) Residual Vs fitted values (c) Main effect plot of designed factors for storage modulus

increased from 1% to 2% wt of the nano clay and get decreased between wt 2% to 3% of nanoclay and again gives increase trend in between 3% to 4.2%. Also it is observed the similar kind of trend in the case of storage modulus with the effective addition of nano ZnO While storage modulus is getting decrease with increase in degree of fiber orientation from 0⁰ to 45⁰, again it is get

increased from 45⁰ to 72⁰ and declines after this level. So it was observed from the main effect plot that there is direct effect of nanoclay and nanoZnO on storage modulus, while fiber orientation had reverse effect on storage modulus From plots, it can be consummate that all results were in approved with what observed in the ANOVA tables and fitted regression coefficients.

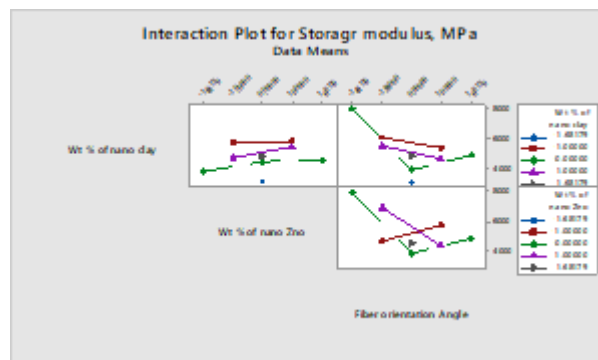


Fig. 6: Storage modulus interaction plot for data means

5.2.2 Plot of Interaction for storage modulus

Figure 6 gives the two way interaction between the input variable and its effect on the storage modulus as response. As per the left hand graph at corner, shows the interaction between weight % of nanoclay and nanoZnO on the storage modulus falls in the lower series of its range value, it deviates in the smaller range of value on Y axis. For interaction effects between weight % of nanoclay and fiber orientation angle effectively changes the storage modulus value at higher range. Similar kind of trend can be observed for the of interaction effect amongst the wt. percentages of nanoZnO and fiber angle of orientation in right hand corner graph.

5.3 Contour and surfaces graph plots for the storage modulus of CFRP hybrid nano composite

In the part of contour 2D plots and surfaces 3D graphs for the effect of various parameters on the storage modulus of CFP hybrid nanocomposites are explained storage modulus

by putting all other factors and parameters should be held constant or fixed at zero level. These graphs are useful for exploring the sound effects of factors for interaction plots and main effects on response.

5.3.1 Effect of nanoclay and nanoZnO on storage modulus of CFRP hybrid nanocomposites

Fig. 7 (a) and (b) shows the contour and surface graphs for nanoclay and nanoZnO and by considering value of fiber orientation angle fixed at 45° as zero level. The contour plot displays that the value storage modulus is decreasing with increase in nanoclay wt%. Similarly the value of storage modulus is increasing with the nanoZnO wt%, from the contour plot it can be seen that the maximum storage modulus can be obtained at highest content of both nano particles. As both the variables illustrate has clear effect on response at the same time and so that the interaction between these variables shows the effect on storage modulus.

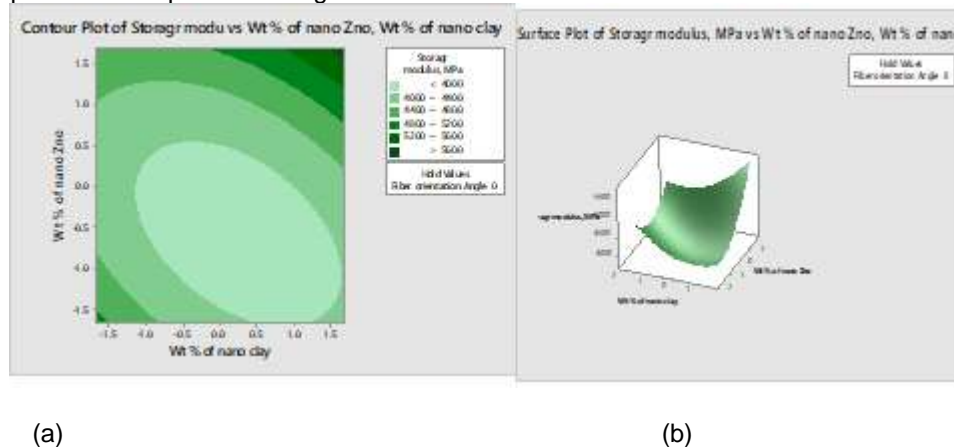


Fig. 7: (a) 2-D contour and (b) 3-D surface plots, for effect of nanoclay and nano ZnO on storage modulus

5.2.2 Effect of nanoclay and fiber orientation angle on the storage modulus

By fixing or holding the value of nanoZnO at zero level (ie at 3%), we can get read of contour graph and surface graph for fiber angle of orientation and nanoclay wt % as shown in Fig.8 (a) and (b) respectively. As shown in this plots, value of storage modulus is not much varied with

change in wt % of the nanoclay but get decreases with increased in value of fiber angle of orientation up to 45 degrees and again increasing towards the end. Furthermore it is noticeable that by altering in the worth of both the variables at the same time affect on storage modulus, after that there was a communal interaction between fiber angle of orientation and nanoclay% wt .

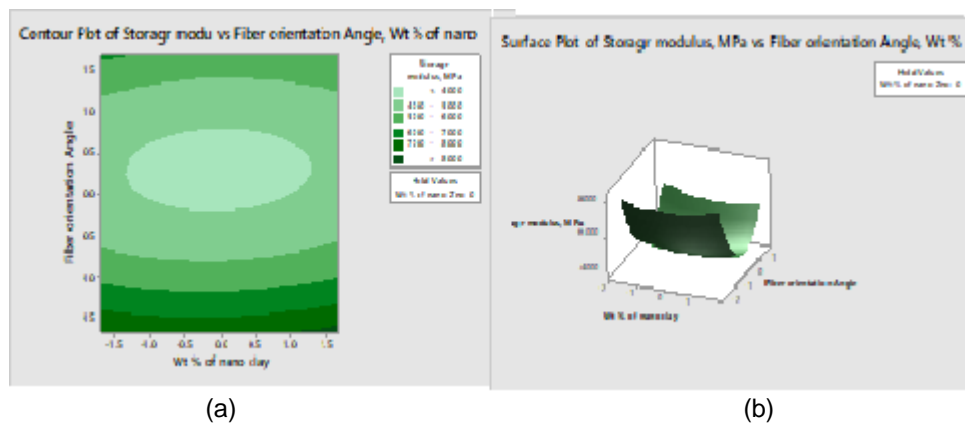


Fig. 8: (a) 2-D contour and (b) 3-D surface plots for effect of nanoclay and Fiber orientation angle on storage modulus

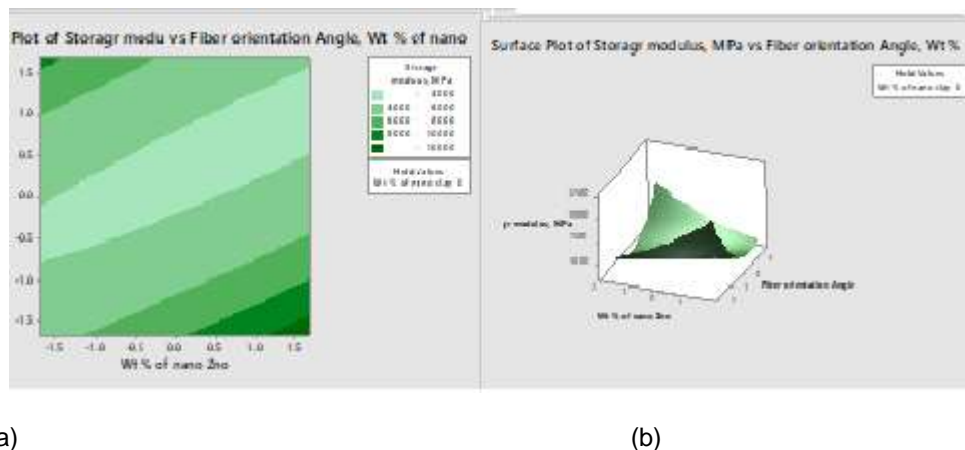


Fig. 9 : (a) 2-D contour and (b) 3-D surface plots, for effect of nano ZnO and Fiber orientation angle on storage modulus

5.3.3. Effect of nanoZnO and fiber orientation angle on storage modulus

Fig.9. (a) and (b) displays contour 2D graph and surface 3D plot for nanoZnO wt % with fiber orientation angle, by keeping nanoclay wt% was fixed at 3% as zero level.. As per the surface plot, storage modulus get decreases with increment in value of fiber angle of orientation, similarly the storage modulus increases with upgrading in wt % of nanoZnO. It can be seen that prudent values storage modulus can be obtain for the lower range of fiber angle orientation and higher nanoZnO wt %. The maximum obtained value for storage modulus was 8016.13 MPa which was in No. 02 coded level run with nanoclay = 0, fiber orientation angle = -1.68179 and nanoZnO = 0, the equivalent real values were nanoclay = 3%, fiber angle of orientation equal to 0^0 and nanoZnO = 3% respectively.

5.4 Optimization of storage modulus

In the part of optimization, the optimum value of flexural storage modulus was obtained by using Minitab response optimizer. For this step the highest value for flexural storage modulus was presumed at 12000 MPa, and consequent value got from the minitab was 11993.51 MPa and related values for nanoclay, nanoZnO and fiber orientation angle were 5wt%, 5 wt% and 0^0 respectively. Accordingly the specimens were fabricated and tested to get experimental

values of the flexural storage modulus for optimized combination and the average value of flexural storage modulus 11899 MPa was obtained.

6. CONCLUSION

In this current study the dynamic mechanical analysis of CFRP hybrid nanocomposites has been evaluated. In which the viscoelastic properties of the new CFRP hybrid nanocomposites are studied. The attempt is made in this work to obtain the optimum combination of the nanoclay, nanoZnO and fiber orientation angle of Carbon fabric, The Response surface methodology is used for design of experiments, modeling and the prediction of the optimum design. It is seen that the model presented was able to predict the 82% of the response ie storage modulus. The analysis of variance shows the direct effect of nanoZnO and nanoclay, fiber orientation angle on the storage modulus. While going through the graph of storage and loss modulus of coded samples, It is observed that the value of storage modulus is increased up to 40% and the loss modulus is improved by almost 35%. In the optimization stage the targeted storage modulus was 12000 MPa, obtained value from the software of Minitab was 11993 MPa, the actual obtained value after experiments was 11899 Mpa, which occurred at corresponding values for nanoclay, nanoZnO and fiber orientation angle were 5wt%, 5 wt% and 0^0

respectively The optimum value of the storage modulus is improved up to 90% or almost doubled from the controlled sample.

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Ethical Statement/Declaration by The Author

"I write on behalf of myself and all co-authors to confirm that the results reported in the manuscript are original and neither the entire work, nor any of its parts have been previously published. The authors confirm that the article has not been submitted to peer review, nor has been accepted for publishing in another journal. The author(s) confirms that the research in their work is original, and that all the data given in the article are real and authentic. If necessary, the article can be recalled, and errors corrected."

Yours Sincerely



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