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Srivastava, Shakun
Department of Mechanical Engineering, Pranveer Singh Institute of Technology

Srivastava, Anshu
Department of Applied Science and Humanities, Kanpur Institute of technology

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Determination of Halloysite Reinforced/Epoxy Nanocomposite Impact Strength by Experimental and Computational Procedures

Shakun Srivastava[1,*], Anshu Srivastava[2]
[1]Department of Mechanical Engineering, Pranveer Singh Institute of Technology, Kanpur, U.P., India
[2]Department of Applied Science and Humanities, Kanpur Institute of technology, Kanpur, U.P., India

E-mail: shakun227@gmail.com

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Abstract: The aim of the current research work is to examine the influence of various processing and fabrication parameters on the morphological, mixing, curing, and impact strength behavior on the basis of varied weight percentage of nanotubular halloysite reinforced fabricated nanocomposite by rule of mixture theory/ experimental results and finite element analysis. Experiments have been performed for impact properties, further finite element analysis (FEA) based software has been utilized to validate the impact mechanical property results. The finite element analysis simulation results are in good agreement to the experimental results obtained on developed nanocomposites. Field emission scanning electron microscope (FESEM) fractograph additionally supports the impact property results.

Keywords: Epoxy; HNT's; Impact testing; finite element analysis; FESEM

1. Introduction

In the past two decades, there has been a lot of study on nanofiller reinforcement epoxy matrix based composites with the goal of reducing the epoxy resins intrinsic brittleness. The epoxy matrix has been reinforced using a variety of nanofiller, including Nanotubular carbon (CNT's)[1)], clays of layered silicate[2)], Nano sized silica[3)] and graphene filler[4)]. Improvements in the epoxy matrix's mechanical, thermal, and electrical properties have been noted. Epoxy nanocomposites were able to be produced on a wide scale, but limitations including high cost, difficulties in mixing, and complex processing procedures prevented its mass production applications. The typical procedure for making epoxy nanocomposites comprises combining nanofiller with liquid epoxy resins, adding a hardener, moulding the mixture, and curing it to produce a cross-linked result.

The main impediment to the fabrication of epoxy matrix based nanocomposites with the enhanced mechanical characteristics has been attaining a good uniform distribution of nanosized fillers in the matrix epoxy resin. A well distributed epoxy/nanofiller nanocomposite has been produced using a variety of processes, including solvent assisted mixing[5)6)], high-shear technique of mixing[7)8)], roll milling technique[9)10)], ball milling technique[6)11)], ultrasonication technique[12)13)], and mechanical mixing technique[14)15)]. However, research is still needed to identify an effective and cost-effective mixing technique that would produce the optimal dispersion and ideal characteristics for a particular kind of nanofiller[16)]. The processing technique and mechanical characteristics of such nanocomposites are greatly influenced by the curing methodologies. The processing and qualities of epoxy nanocomposites must therefore be optimized, which requires an awareness of these problems.

Nanofiller incorporation has an impact on epoxy's curing behavior as well. Epoxies' curing reaction was stimulated by the incorporation of CNT's, which also reduced the curing's starting temperature and sped up overall curing while decreasing the degree of curing[17)18)] Epoxy/CNT systems' curing behavior may change if CNT's are modified[19)20)].

In addition to how CNT's interact with epoxy and the curing agent, CNT dispersion may also have an impact on how produced nanocomposites cure[17)]. Additionally, clay nanofiller were said to interfere with epoxy resins' ability to cure[21)22)]. Not only did the integration of particulate nanosized filler and its distribution affect the epoxy matrix resin's curing behaviour in layered silicate clays, but the epoxy resin's curing characteristics also had an impact on the state of dispersion (intercalation and exfoliation) of the nanosized clays[23)].

Halloysite nanotubes (HNT's), a type of clay material that naturally forms nanotubes and resembles the arrangement of multi-wall Carbon nanotubes, are one of the nanosized fillers. Epoxy matrix resins' izod impact hardness was significantly improved by the addition of HNT's[5)24)].

In addition to solitary HNT's, it was discovered that clusters of Nanotubular halloysite would also form in epoxy/HNT nanocomposites, which could harm the fabricated nanocomposites mechanical characteristics. In order to improve the dispersion of halloysite in epoxy matrix, several researchers used ball mill mixing technique, which further improved tensile strength, elastic modulus, and glass transition temperature (Tg)[25)26)]. Therefore, improving mixing techniques is necessary to achieve the optimal process-property balance.

Rubber polymers such as styrene butadiene rubber[27)], natural rubber[28)] and ethylene propylene diene monomer[29)] all had significant curing behaviour changes as a result of Nanotubular halloysite. Particulate HNTs' influence on the curing characteristics of epoxy matrix fabricated nanocomposites has not yet been thoroughly investigated.

The study of curing technique behaviour of epoxy matrix fabricated nanocomposites reinforced with particulate HNT’s can provide answers to some important queries in this polymer nanocomposite field, regarding effective mixing method, curing/hardener agent because it affects final characteristics and processability of fabricated nanocomposite.

In this study, epoxy matrix based halloysite nanotube reinforced composites were fabricated and ultrasonication mixing was employed to increase the uniform dispersion of particulate HNT/x (x=0 to 5 wt. %) in the epoxy matrix. The performance of the HNT’s/Epoxy nanocomposites was examined in relation to the effects of the characteristics of the epoxy matrix using triethylenetetramine curing agent.

Furthermore, the impact properties of epoxy nanocomposite were examined in relation to the effects of varied amount of particulate HNT’s. Microstructure of fabricated specimen confirms the increase in density in successive accumulation of HNT/x (x=1 to 5 wt. %) and shows the effectiveness of HNT/Epoxy nanocomposites.

The current work aims to fabricate epoxy-reinforced nanocomposites with halloysite nanotubes for a various applications, calculate impact strength through experiment and validate results by computational methods.

2. Materials and methods

2.1 Selection of composite materials

Nanotubular halloysite particulate filler (supplied by Sigma Aldrich, USA) is reinforced with Diglycidyl ether of bisphenol-A epoxy (DGEBA, LY 556) is a matrix material and triethylenetetramine hardener (TETA, HY 951) is used as a curing agent/hardener (provided by Ciba giegy ltd. Mumbai, India) for fabrication of different loading rate of HNT’s in specimens.

The parameters of the particulate halloysite reinforcing fillers and epoxy matrix are displayed in Tables 1 and 2, respectively.

Table 1 Characteristics of Halloysite Nanotubes

Nanotubular Halloysite (HNT’s)	
Category	Kaolinite
Formula	$Al_2Si_2O_5(OH)_4.nH_2O$
Crystal Symmetry	Monoclinic
Specific Gravity	2 To 2.65 (2.53 Used In Present Work)

Table 2 Properties of epoxy resin

Characteristics	Reason
High reactivity	Because of hydroxyl groups
Chemical stability	Due to ether linkages
Excellent adhesion to surfaces	Because of attractive forces between the epoxy resin and the surface of the substrate
Outstanding electrical resistivity property	Because of its high dielectric strength

2.2 HNT/Epoxy composite Preparation

The final characteristics of the fabricated epoxy matrix based halloysite reinforced nanocomposite are significantly influenced by the production method. For the development of varied wt. % of filler HNT/x (x=1 to 5 wt.%), specimens with dimensions of 150 mm x 150 mm x 3 mm, ultrasonication mixing approach (Rivotek sonicator 700 W, Model number XT 546 at 50% amplitude) is used for irradiation of mixture for 1 hour by ultrasonic waves for uniform morphology (Table 3).

Table 3 Samples designation and formulations

Sample Code	Description/ Reinforcement	Epoxy resin (g)	Curing Agent (g)
EH 0	Cured unmodified	100	10
EH 1	Cured 1 wt.% HNT	100	10
EH 2	Cured 2 wt.% HNT	100	10
EH 3	Cured 3 wt.% HNT	100	10
EH 4	Cured 4 wt.% HNT	100	10
EH 5	Cured 5 wt.% HNT	100	10

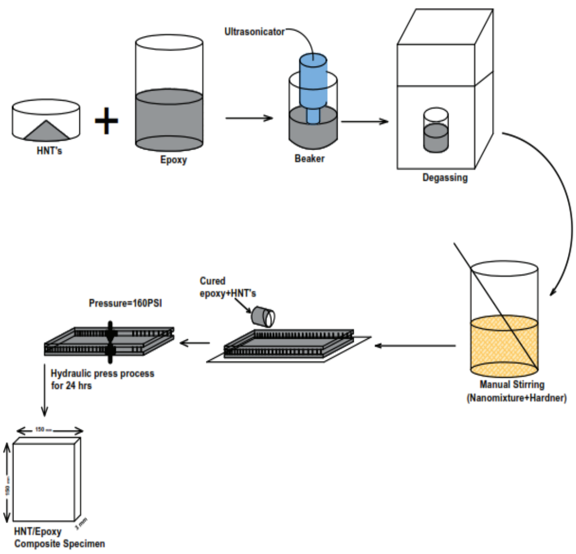

Fig. 1: fabrication steps of developed nanocomposites samples.

Further, the cured epoxy with halloysite have been poured in the steel mould and cured at room temperature (30 °C to 35 °C) in a hydraulic compression apparatus at 1600 PSI for 24 hours.

The manufactured sheets of specimens were shaped by contour cutting apparatus as per ASTM standards for different testing's (Fig. 1).

3. Mechanical Characterization

3.1 Impact Test

Impact properties were determined according to ASTM D256 method-A standard using an Impact Testing Machine (make: Tenuis Olsem, izod specimen) with pendulum capable of delivering the energy of 2.7 J at room temperature. The velocity of the striker at the moment of impact on HNT's / epoxy nanocomposite sheet is approx. 3.5 m/sec. For accurate results, five readings were carried out for every composite of varying halloysite nanotube HNT/x (x = 0 to 5 wt. %).

3.2 Modeling and finite element analysis approach

Simulation method has become in demand trend for characterization of composite materials and the findings of software must always validated by experimental work. For calculation of behaviour and strength of engineering structures and materials, the computer-based numerical technique has been used through Ansys that is one of the finite element analysis (FEA) software. Fabricated composite material assumes to be perfectly isotropic in nature.

3.2.1 Impact test modeling using inventor

For the finite element analysis modeling of impact test of the composites, a computer aided design model is generated using Inventor-2016 and saves as in Initial graphics exchange specification (IGES) format.

This file format is imported, generated and meshed into Ansys/ mesh module and simulation is done using Ansys/autodyne module for impact simulation at Ansys workbench platform.

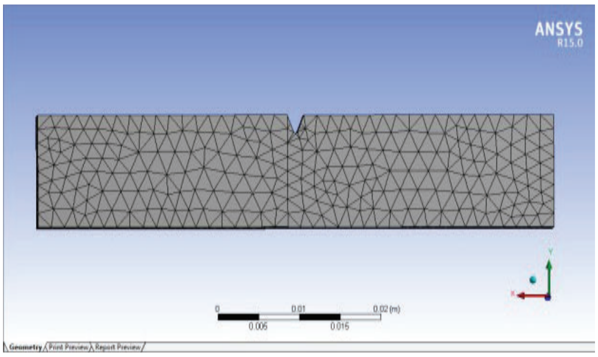

Fig. 2: Mesh generated of Izod impact test model

The elastic finite element model for impact test module linear and nonlinear structural mechanics applications have been simulated using explicit dynamics method for the problems involving high-velocity impact analysis.

Events which take place over very short periods of time are most suitably analysed and simulate using explicit dynamics and it consists of 914 nodes and 3575 square and triangular elements (Fig. 2).

4. Experimental Results and Discussion

The impact strength of the epoxy HNT/x (x = 0 to 5 wt. %) nanocomposites is shown in Fig. 3.

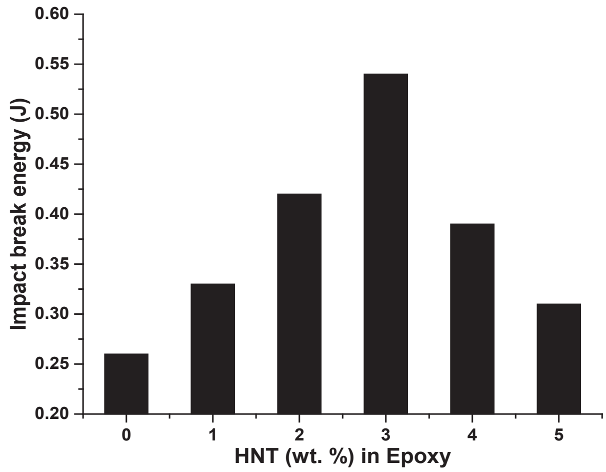

Fig. 3: Variation of Impact break energy as a function of halloysite loading

It is observed that adding HNT/x (x = 3 wt. %) into the epoxy increased its impact break energy from by approx. 3.5 times from 0.33 J for the unmodified epoxy to 1.19 J (Fig. 4).

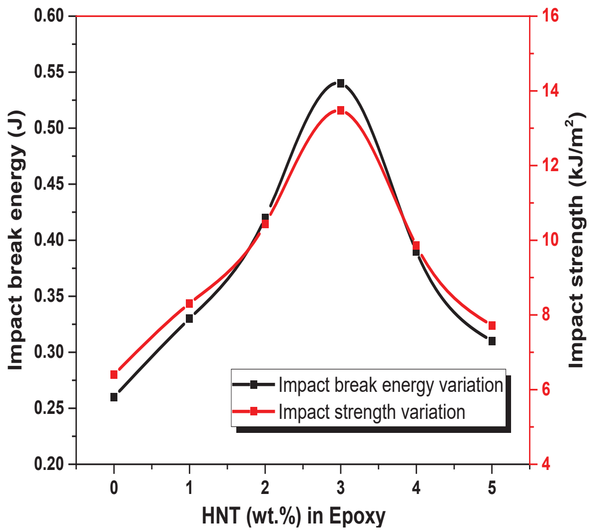

Fig. 4: Variation of impact strength and impact break energy as a function of halloysite loading

Improved interfacial interaction is responsible for increase of impact strength because of higher capacity to absorb energy during impact loading (Table 4).

Table 4 Impact strength of fabricated Samples

Sample Code	Impact strength (kJ/m^2)		
	Test value range		Mean value (with deviation)
	Min value	Max value	
EH 0	5.58	7.19	6.4 ± 0.82
EH 1	7.58	9.61	8.31 ± 1.30
EH 2	9.49	11.47	10.43 ± 1.04
EH 3	13.01	13.99	13.47 ± 0.52
EH 4	9.32	10.41	9.86 ± 0.55
EH 5	7.45	7.85	7.72 ± 0.27

Uniform distribution and proper dispersion of HNT's nanotubes in polymer matrix is responsible for applying high impact energy before failure[30].

Fracture mechanism can be related with Kirkendall effect as well as similar to the diffusion in solids. When the interface between two materials shifts as a result of variations in the atoms' rates of diffusion, it is known as the Kirkendall effect. It is also known as the high-temperature motion of a boundary layer between two elements.

It is worthwhile noted that the noticeable improvement in energy consumed during impact failure is comparable to the toughening effect of rubbers[31], but incorporating much lower reinforcement amount of HNT/x (x = 3 wt. %) the same toughening was achieved[6][32].

On further increasing the percentage (4 wt. % and 5 wt. %.) of HNT's particulate in cured epoxy polymer matrix (EH4 and EH5), coagulated micro-voids formed due to stress concentration zone near HNT's particulate, large deformation at a negligible change in load is observed due to the collapse of voids formed and coagulation of micro-voids.

5. Finite Element Analysis Simulation Results and Discussion

Figure 5 shows the energy absorption during impact failure of the HNT/Epoxy nanocomposites, graph distribution of full simulations for the geometrical models of the composites was performed in the non-linear explicit finite element code of LS-DYNA. Impact test results obtained from finite element analysis software given by ANSYS 15.0 for fabricated HNT/Epoxy composite specimens are as discussed[33][34].

Impact testing of EH3 composite model depicts the results during testing that the hammer has 2.7 J kinetic energy before striking the fabricated specimen model and after the impact, the hammer has 2.23 J energy.

This reduction of energy is due to absorption and consumption of kinetic energy during the fracture of the specimen model.

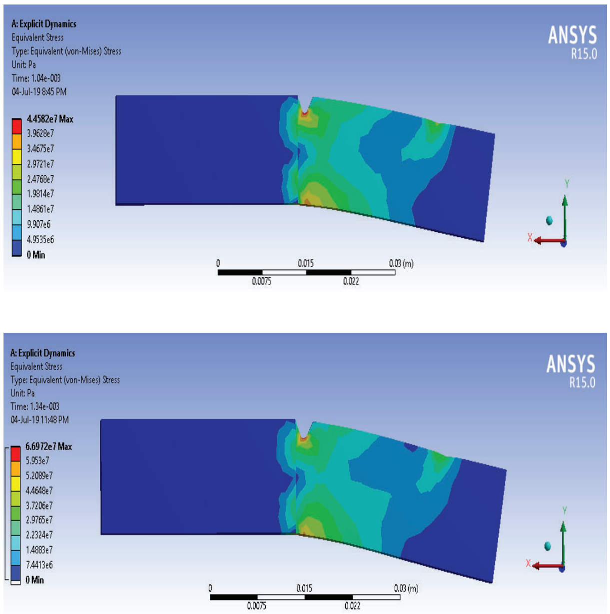

Fig. 5: FEM results and failure for composite in Izod impact test (0 wt% and 3 wt% HNT)

In the composites of epoxy/HNT/x (x = 0 wt. % to 5 wt. %), it may be demonstrated that with a step-wise increment of HNT's particulate in cured epoxy resin, the impact energy consumes to fracture composite system increases drastically.

This improvement is due to the higher amount of reinforcing HNT's particulate in epoxy which is responsible for absorbing the impact energy transferred through the epoxy matrix[35]. Compared results of experimental and finite element analysis simulation (Ansys workbench module-15.0) for the impact test or the composite is shown in table 5.

It is observed for impact fracture that the experiment energy needed for impact fracture is 0.54 J and Ansys workbench module autodyne solver value is predicted to be 0.4658 J for HNT/x (x = 3 wt. %) epoxy nanocomposite.

Table 5 Izod impact testing comparison results of experimental and finite element simulation

Sample code	Impact break energy (J) (Experimental)	Impact break energy(J)(Ansys simulation)	Error (%)
EH 0	0.26	2.7013-2.4332 = 0.2681	3.11
EH 1	0.33	2.7013-2.3849 = 0.3164	4.12
EH 2	0.42	2.7013-2.3237 = 0.3776	10.09
EH 3	0.54	2.7013-2.2355 = 0.4658	13.74
EH 4	0.39	2.7013-2.3008 = 0.4005	2.69
EH 5	0.31	2.7013-2.3701 = 0.3312	6.84

Whereas for the unmodified epoxy the observed values are 0.26 J for experimental stress and 0.2681 J for predicted autodyne solver value as shown in fig. 6.

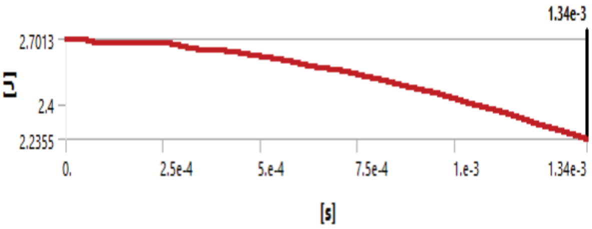

Fig. 6: FEM result (impact break energy) for EH3 composite

It is observed from the analysis of results that the impact test results for the finite element analysis method are in the same trend than the experimental results.

As the concentration of HNT is increased beyond 3 wt. % (EH4 and EH5) the deviation from proportional characteristics between experimental and computational impact strength analysis has been occurring, one possible reason is due to coagulation effect in microstructure owing to local concentration and micro void generation at less dense epoxy materials.

Fabrication defects like non-uniformity in matrix, voids, weak bonding strength of HNT's particulate with the epoxy resin in nanocomposite may be responsible for the difference in results. The highest error value, 13.74 %, was observed between experimental testing results and numerical analysis results for impact break energy.

6. F.E.S.E. Microscopic Observation

In support, FESEM micrograph evaluation for fractured tensile specimen's surfaces was carried out for HNT/ epoxy failure and its effect. Figure 7 shows the flaky type of morphology for the unmodified epoxy system.

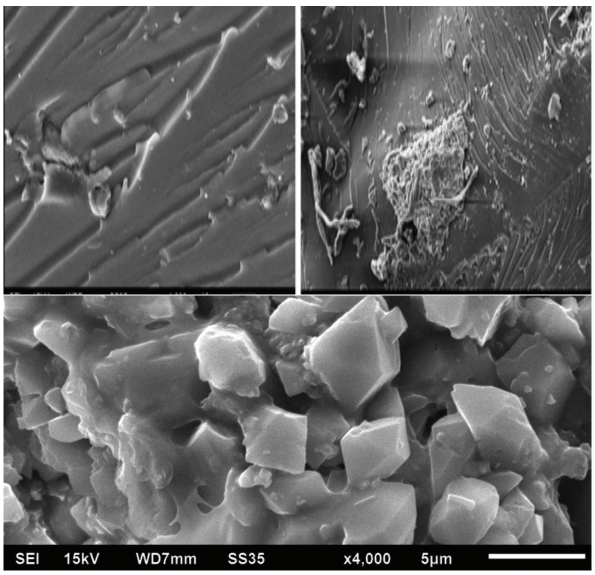

Fig. 7: Micrograph for HNT/x/Epoxy Composites: (a) x =0 wt% and 5 wt% (b) x=5 wt.%

FESEM micrographs (a and b) clearly indicate that the sample having 3 wt.% of HNT's in epoxy matrix shows the excellent dispersion of HNT's over the entire polymer matrix. Halloysite nanotubes are clearly visible on the polymer matrix. Scanning electron microscope is used to analyse the effects of halloysite nanotubes on fracture mechanism, responsible for propagation of cracks in HNT/epoxy nanocomposite.

FESEM images of cracks in modified epoxy nanocomposite. Massive micro cracking and shear yielding could absorb fracture energy and resist crack propagation, which results in a tough network in modified HNT's epoxy nanocomposite. The enhancement in various properties at this particular loading (3 wt. %) may be attributed to excellent properties of HNT's over entire polymer matrix are shown in fig. 8. Agglomeration of HNT's can be seen in samples have more than 3 wt. % nanotube and responsible for strength decrement.

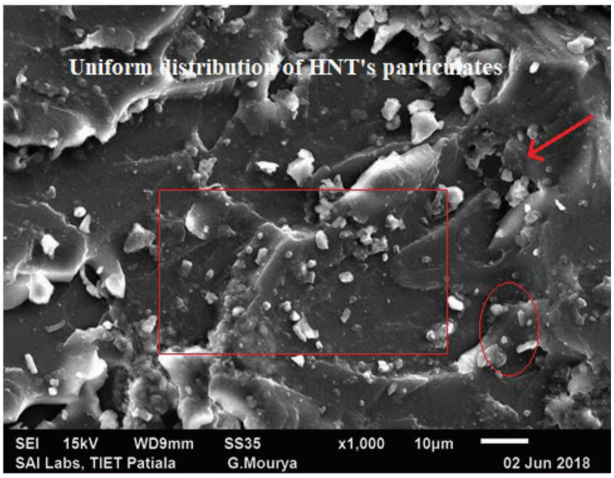

Fig. 8: Microscopic image of 3 wt. % HNT's loaded nanocomposite

Important findings from experimental results reveal that a 3 wt. % of halloysite nanotube inclusion in epoxy demonstrate better impact properties as compared to the near about composition values of HNT's in epoxy. The possible factors for these findings (for HNT ≠ 3 wt. %) may be the presence of small-scale micro voids and non-uniformity of a matrix of halloysite nanotube within the epoxy resin.

7. Conclusions

The conclusions drawn from the current research work are as follows:

1. A substantial increase in impact strength has been achieved for HNT/x (x=3 wt. %) nanocomposite which is about 110.46 % as compared to the neat cured epoxy specimen. Meanwhile, impact break energy improves up to 3 wt. % HNT's particulate reinforced composite and decreases for 4 wt. % and 5 wt. % loaded HNT/Epoxy composite.
2. Impact strength of developed nanocomposite

enhancement may be attributed to intrinsic toughening behaviour of HNT's particulate.

3. Microstructure analysis describes the effect of particulate reinforcement addition in the fabricated nanocomposite. From FESEM micrograph it can be concluded that in the absence of HNT's particulate fracture damage is more and insignificant amount of shear bend formation occurs, whereas damaged tensile fractured fibre appears.
4. A finite element analysis model applying Ansys design module and Ansys Autodyne module has been developed to simulate impact testing experimental results for fabricated HNT's particulate-epoxy matrix based composite specimens.
5. Simulation is done using explicit dynamics analysis systems were used for analyzing the specimen model for impact testing.
6. It is observed that the finite element analysis simulation results impact are in good agreement and in the same trend, when observed graphically, to the experimental results obtained on fabricated HNT/Epoxy nanocomposites.

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