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# HIGH STRAIN RATE SENSITIVITY OF GLASS/EPOXY/CLAY NANOCOMPOSITES

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**Key words:** Glass Fiber, Clay, Nanocomposites, High strain rate, Dynamic tension, Digital Image Correlation (DIC), Stress strain response.

**Summary:** *In general, composite materials are rate sensitive to their mechanical properties. In order to determine these properties, dynamic tensile tests are performed on drop mass test setup, which provides mechanical data at strain rates up to 1000 $s^{-1}$, filling the gap between conventional low speed instruments and split Hopkinson bar tests. The present research work is to study the effect of strain rate on the tensile behavior of glass/epoxy and its clay nanocomposites. The clay in terms of 1.5, 3 and 5 wt% are dispersed in the epoxy resin using mechanical stirrer followed by sonication process and glass/epoxy nanocomposites are prepared by hand layup technique followed by compression molding technique. Characterization of the nanoclay is performed using X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM). The Digital Image Correlation (DIC) technique is used for full field measurement of strain and strain rate using high speed camera. An array of 128 X 128 pixels is used to achieve 100,000 frames per second for obtaining dynamic strain. Stress strain measurements are reported for glass/epoxy and its clay nanocomposites over a range of strain rates from 0.0016 to 450 $s^{-1}$ and the variation of tensile modulus, strength and failure strain with strain rate are studied. The tensile modulus (18 GPa) and tensile strength (315 MPa) increase significantly up to 106% (37 GPa) and 67% (526 MPa), respectively at the highest strain rate for neat glass/epoxy composite. It is observed that the tensile modulus increases monotonically as the clay loading increases and 1.5 wt% clay loading found as optimal loading for tensile strength.*

## 1 INTRODUCTION

Composite materials are widely used in aerospace structures, automotive industry, and marine structures, and in many instances, they are exposed to high energy, high velocity dynamic loadings. The analysis and design of composite materials for such application prone to dynamic loadings, ranging from low-velocity impact to high-energy shock loadings, requires prior knowledge of high strain rate material properties. Development of dynamic failure criteria and numerical simulations also require systematic testing and characterization of the composite materials under quasi-static and high strain rate conditions. Hence, determination of dynamic mechanical properties of composites is necessary to prevent catastrophic failure at high loading rates and also to ensure weight efficient and structurally sound composite structures.

In recent years, layered silicates or nanoclay have received much attention as the outstanding reinforcement for various types of engineering polymers to improve mechanical, thermal, and fire resistance properties due to its high aspect ratio and unique intercalation/exfoliation characteristics. In the early 1990, the researchers at Toyota motor company [1] first reported that addition of small amount of layered silicate on nylon showed excellent mechanical, thermal and barrier properties in contrast to pristine nylon. Subsequently, many researchers investigated various types of polymer nanocomposites for further improvement of properties upon addition of nanoclay. Lan and Pinnavaia [2], [3] found that the presence of the organoclay in epoxy substantially increases the tensile strength and the modulus relative to the unmodified pristine polymer however failure strain of epoxy/clay nanocomposites is essentially same as the pristine matrix. It is observed that variables such as type of clay, the choice of clay pre-treatment method, the selection of polymer component, and the method of dispersion in the polymer influence the properties of the final nanocomposite. Although polymer/clay nanocomposites have been extensively studied for different types of polymers, very little information is available for continuous fiber reinforced polymer nanocomposites. Recently, researchers investigated the mechanical properties of epoxy/clay fiber reinforced nanocomposites and their findings prove that the mechanical properties of FRP can be enhanced with the addition of nanoclay [4]–[10]. In this paper, an attempt is made to study the effect of clay on dynamic loading of glass/epoxy nanocomposites.

However, limited amount of information is available on the effects of strain rate on the response of fibrous composites. Most of the dynamic work was conducted on lateral impact testing of composite laminates, but with less emphasis on constitutive properties characterization and development of dynamic failure criteria. Hence, it is essential to have a proper knowledge and understanding of the response of these materials over a wide range of strain rates. Dynamic tests on composite materials were first conducted by Rotem and Lifshitz [11]. They investigated unidirectional E-glass/epoxy composites under dynamic tensile loading and found that the dynamic tensile strength ($30\ s^{-1}$) is equal to three times the static one and that the dynamic tensile modulus is 50% higher than the static one. It is noted that the mechanical responses of fiber-reinforced polymeric composites are sensitive to strain rate. Armenakas and Sciammarella [12] studied the strain rate effects of UD glass/epoxy composites up to $500\ s^{-1}$. They found a linear increase of the elastic modulus with increase in strain rate and corresponding decrease in ultimate strength and strain, in contradiction with the previous study.

It is clear that a full characterization of the mechanical behavior of composite materials at high strain-rates is likely to require the use of a wider range of experimental techniques. Many test methods are commonly used in generating the rate dependent material data: the conventional screw drive load frame, servo-hydraulic system, impact tester and Hopkinson bar (or Kolsky bar) system. The conventional tensile test can provide strain rate dependent data up to $1\ s^{-1}$. Split Hopkinson pressure bar (SHPB), a widely used high strain rate testing technique, may be used to generate data at the high end up to and exceeding $1000\ s^{-1}$. Hamouda and Hashmi [13] discussed the experimental techniques employed for determining the mechanical behaviour of composite materials under impact loading at high strain rate in tensile, compressive and shear loading. Jacob et al. [14] reviewed the strain rate dependence of some mechanical properties of polymer composite materials. From the literature, it is noted that research work on medium strain rate of composites is limited. Also, the experimental techniques to generate tensile stress–strain data at the medium strain rates in the range of 1–100 $s^{-1}$ are not well established [15]. Two types of equipment have been used to generate data in this strain range: the high rate servo-hydraulic testing machine and the drop weight impact machine. The use of a servo hydraulic machine is common and convenient. However, the conventional hydraulic machine is limited to lower strain rates (< 10 $s^{-1}$), due to inertial effects of the load cell and grips. Hence, an attempt is made to use drop mass setup to achieve medium strain rates on tensile loading.

The drop weight impact test has many advantages; it is inexpensive, can accommodate different specimen geometries and allows easy variation of strain rate. However, the system is very sensitive to the contact conditions between the impactor and specimen and to spurious noise from ringing and vibrations. Lifshitz [16] investigated the tensile strength under dynamic loading of angle ply balanced glass/epoxy laminates using an instrumented drop weight apparatus. Failure stresses are found to be 20-30% higher than the static values. The failure strains and moduli are same for static and dynamic loadings. Groves et al. [17] investigated the high strain rate effects between 0.0001 $s^{-1}$ – 2660 $s^{-1}$ for continuous fiber polymer composites using combination of test machines and specimen geometries. They generated strain rates from 0 to 100 $s^{-1}$ using conventional hydraulic test machine and strain rates from 10 to 1000 $s^{-1}$ using acoustic drop tower with alignment fixture. They found unexpected, exponential-like increase in strength and modulus beyond strain rates of 10 $s^{-1}$ due to high-intensity stress waves, decrease in mobility and changes in fracture propagation for carbon/epoxy specimens. They generated strain rates from 1000 to 3000 $s^{-1}$ using split Hopkinson pressure bar. Okoli [18] conducted tensile, shear and three point bending tests to measure energy absorbed to failure of a material by instrumented impact tester on a woven glass/epoxy laminate at increasing rates of strain. A linear relationship obtained between absorbed/expended energy and the log of strain rate. An increase in tensile, shear, and flexural energy of 17, 5.9, and 8.5%, respectively, per decade of increase in the log of strain rate was observed. Shokrieh and Omidi [19] studied the behavior of unidirectional glass fiber reinforced polymeric composites under uni-axial loading at quasi-static and intermediate strain rates of 0.001–100 $s^{-1}$. The tests were performed using a servo-hydraulic testing apparatus equipped with a strain rate mechanism and a special jig and fixture. The experimental results showed that increase in tensile strength, modulus, strain to failure and absorbed failure energy of 52%, 12%, 10% and 53%, respectively in contrast to quasi-static testing. The visual inspection of the failed specimens showed significant changes in the fracture surface with increased strain rate. Brown et al. [20] reported the effect of strain rate on the tensile, shear and compression behaviour of a commingled E- glass/polypropylene woven fabric composite over a strain rate range of $10^{-3}$–$10^{2}$ $s^{-1}$ using an electro-mechanical universal test machine and a modified instrumented falling weight drop tower with specially designed fixtures.

Most of the high rate studies deal with uniaxial compression. Furthermore, it has been found that composite materials show different mechanical response in tension and compression under quasi-static and dynamic loading conditions. Compared with the compressive tests, tensile tests can provide more information on damage, fracture and failure behavior. Hence, an attempt is made to study the dynamic behavior of glass/epoxy/clay nanocomposites under tensile loading at mediuim strain rates.

Digital image correlation (DIC) techniques are widely accepted and commonly used as a powerful tool for the quasi-static and high strain rate experiments to obtain the in-plane strain field over the entire specimen surface. DIC is based on the principle of comparing speckle pattern structures on the surface of the deformed and the undeformed sample or between any two deformation states. The relevance of DIC for SHPB experiments was demonstrated in a recent study by Gilat et al. [21]. A comprehensive review of the digital image correlation technique is given by Sutton et al. [22] and Pan et al. [23]. Koerber et al. [24] investigated the strain rate effects on unidirectional carbon/epoxy composites at strain rates up to 350 $s^{-1}$ using split-Hopkinson pressure bar. They employed digital image correlation technique to obtain in-plane strain field for dynamic loading.

This paper discusses the application of a drop mass system and DIC for dynamic tensile characterization of glass/epoxy/clay nanocomposites. The results presented cover strain rates from quasi-static to several hundred per second.

## 2 SPECIMEN PREPARATION

Epoxy resin, diglycidyl ether of bisphenol A (DGEBA), and the curing agent triethylenetetramine (TETA) were supplied by Huntsman Ltd, India. E glass fiber, 610 GSM was procured from Sakthi Fibers, India. Clay, GARAMITE® 1958 (Alkyl Quaternary Ammonium Clay) was procured from Southern Clay Products, Inc., USA.

Nanocomposites containing the organo-montmorillonites were prepared by addition of clay ranging from 1.5, 3 and 5 weight%, respectively. The mould used for preparing nanocomposites is made of two rectangular glass plates having dimensions of 300 mm × 300 mm. Rubber beadings were used to maintain a 3 mm thickness all around the mould plates. Wax was used as a releasing agent. The epoxy resin was preheated at 60 °C in order to reduce the viscosity. The required amount of clay was added slowly while stirring using mechanical stirrer. The mixture was further stirred for 2 h. The curing agent TETA was added and the mixture was gently stirred in order to avoid the formation of bubbles. After degassing, the solution was cast in the mold.

The glass/epoxy nanocomposites were prepared by hand layup technique followed by compression molding technique. The epoxy organo-montmorillonites mixture containing different amount of clay addition 1.5, 3 and 5 wt% were fabricated by mechanical stirring. After adding the required amount of curing agent, a thin layer of epoxy/organoclay mixture was applied with a brush on an aluminum plate coated with a releasing agent. Then the epoxy/organoclay mixture was impregnated into the WRM glass fiber with the assistance of hand roller to ensure that all fibers were wetted. The laminates were cured at room temperature and kept in the compression molding machine for 24 h for complete curing.

## 3 CHARACTERIZATION TECHNIQUES

### 3.1 X-ray diffraction

X-ray diffraction analysis was performed on epoxy/clay nanocomposites using PHILIPS PW-1730 (CuKα radiation at a scan rate of 1°/min in 2θ ranges from 3 to 15°). The results show that the nanocomposites with 1.5 wt% and 3 wt% of clay exhibited no observable peaks, indicating that most of the clay particles in the nanocomposites were exfoliated or, the clay's interlayer distance (d-spacing) was more than 7 nm (Fig. 1). Only the highest clay concentration (5 wt%) showed a distinct peak correlating to a non-exfoliated structure.

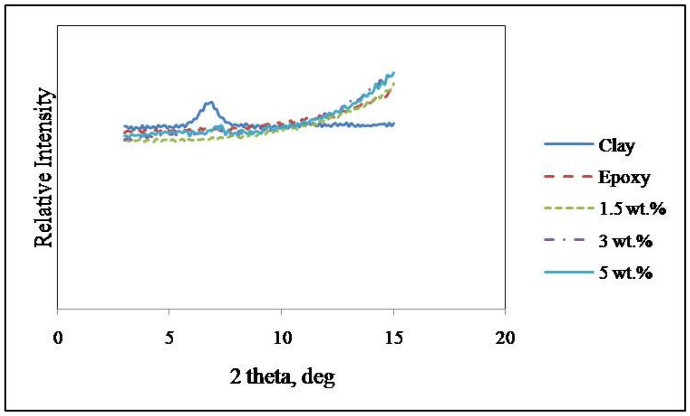


Figure 1: X-ray diffractograms of neat epoxy, nanoclay and epoxy/clay nanocomposites.

### 3.2 Scanning electron microscopy

SEM analysis was performed using Hitachi S-4800 Scanning Electron Microscope (SEM) at 5 kV accelerating voltage equipped with Energy Dispersive Spectroscopy (EDS) to identify the clay particles in the composites. The surfaces were sputter coated with a thin gold film to increase the conductivity for epoxy samples. The SEM micrographs in Fig. 2a shows the microstructure of layered silicate and Fig. 2b shows the dispersion of clay on epoxy at 5 wt%, which confirms clay agglomerates. Energy Dispersive X-Ray Spectroscopy (EDS) microanalysis technique confirms the presence of Silica, Magnesium and Alumina.

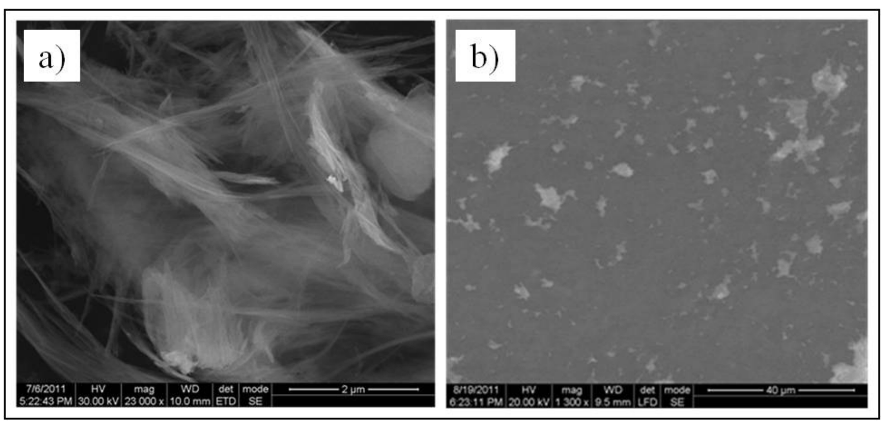


Figure 2: SEM micrographs of a) As-received nanoclay, b) 5wt% clay loading.

## 4 EXPERIMENTAL DETAILS

### 4.1 Drop mass test setup

Drop mass test setup (Fig. 3) is used for generating high strain rates from 10 to 1000 $s^{-1}$. A typical drop mass system usually consists of two long guide rods (drop tower) with a massive foundation, drop mass, and stop blocks. In the test, drop mass is raised by a motor to a pre-determined height through bearing assemblies and then dropped. The drop mass impact test has many advantages; it is inexpensive, can accommodate different specimen geometries and allows easy variation of strain rate. However, the system is very sensitive to the contact conditions between the impactor and specimen and to spurious noise from ringing.

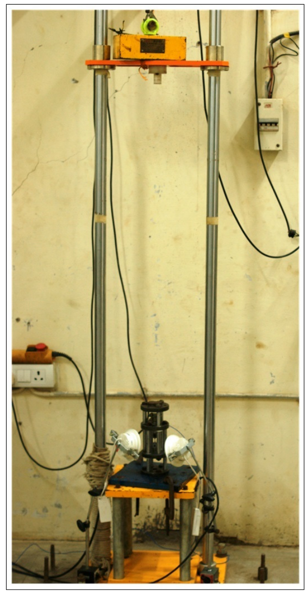

Figure 3: Drop mass test setup.

### 4.2 Digital image correlation

Digital image correlation is a non-contact strain measurement technique used to determine the displacement and full-field strain of a deformed specimen. In general, the implementation of 2D DIC method [23] comprises the following three consecutive steps, namely (1) specimen and experimental preparations; (2) recording images of the planar specimen surface before and during loading up to failure; (3) processing the acquired images using a computer program to obtain the desired displacement and strain information. A random or regular high contrast pattern (black-on-white speckle) is applied to the specimen surface using aerosol spray paint. In the current paper, a coarse speckle pattern (Fig. 4) was made manually for dynamic testing, considering the image resolution of high speed camera and VIC 2D image correlation software is used for measuring the deformation (strains) during quasi-static and dynamic experiments.

### 4.3 Quasi-static and dynamic testing

Quasi-static tensile tests were conducted in a 30 kN standard Instron 3367 test frame at a cross-head speed of 5 mm/min. This corresponds to nominal strain rate of 0.0016 $s^{-1}$, by dividing the stroke rate of the cross-head of the machine by the gauge length of the specimen. The Point grey 14 bit Grasshopper3 (2/3" Sony ICX625 sensor) camera with 5 MP resolution (2448 X 2048 $pixel^2$) corresponding to pixel size of 3.45 µm, coupled with a Edmund optics high resolution 35 mm FL f/1.8 lens was used to obtain the quasi-static specimen strain field. The camera was positioned at a distance of 15 cm away from the specimen surface. The acquisition rate of the camera was set to 15 frames per second (fps).

Figure 4: Speckle pattern on glass/epoxy specimen.

High strain rate tensile tests were conducted on the drop tower from heights of 0.5, 0.75 and 1 m, producing theoretical strain rates of 315, 385 and 445 $s^{-1}$, respectively. An impactor mass of 1 kg was used in the dynamic tensile tests. An in-house specimen fixture was designed and used for the dynamic tensile tests (see Fig. 5). The specimen is clamped at each end between steel grips. The top grip is directly bolted through the load cell to the fixed carriage. A moving carriage is supported by the lower grip and it is guided by three stainless steel rods. The drop tower striker imparts a load on the moving carriage which loads the specimen in tension through the lower grip as it travels downward. Load was measured with a PCB 208C04 Integrated Circuit Piezoelectric (ICP) sensor. A Phantom V611 high speed camera with 1 MP resolution, coupled with a SIGMA 50 mm f2.8 DG macro lens was used for the dynamic experiments and positioned at 15 cm away from the specimen surface. It is noted that the maximum frame rate depends on the area of interest for the Phantom V611 camera. Due to the smaller area of interest, the image size could be reduced to a resolution of 128 x 128 $pixel^2$, which resulted in a higher frame rate of 100,000 fps and a shutter speed of 9.5 μs. Two standard 12 W LED lamps on either side of the camera guaranteed an even illumination of the specimen surface.

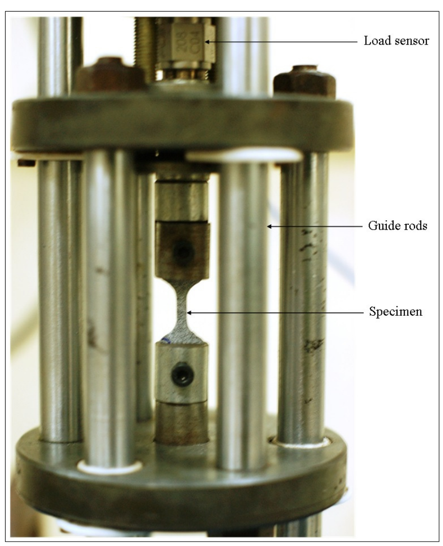


Figure 5: In-house specimen fixture assembly.

## 5 RESULTS AND DISCUSSION

Under quasi-static loading, the stress–strain response is approximately linear elastic up to the maximum stress point of 315 MPa followed by abrupt failure at a strain of 2.5%. Fig. 6 shows stress-strain response for glass/epoxy composite at static and dynamic loadings. At 0.75 m drop mass height corresponding to strain rate of 385 $s^{-1}$, the stress-strain response is different from quasi-static results. The stress strain behavior shows increasing slope and strength and decreasing strain with increasing strain rate.

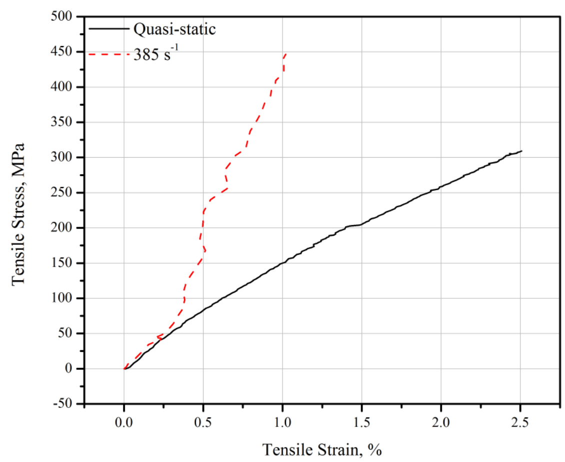


Figure 6: Stress-strain responses of neat glass/epoxy composites.

### 5.1 Effect of strain rate on tensile properties

The tensile modulus and strength are relatively sensitive to high strain rates, which can be explained by viscoelastic nature of epoxy matrix (Eyring principle), fibre–matrix interfacial properties and the time dependent nature of damage accumulation [20]. Table 1 summarizes the tensile properties of neat glass/epoxy composite at different strain rates. Fig. 7 illustrates the effect of strain rate on the tensile modulus and the strength. The tensile modulus (18.09 GPa) increases significantly up to 106% (37.31 GPa) at the highest strain rate for neat glass/epoxy composite. The tensile strength (314.9 MPa) increases up to 67% (526 MPa) at the highest strain rate for neat glass/epoxy composite. A similar trend of increase in tensile strength with strain rate is agreed with findings of Okoli and Smith [25].

| Drop mass height (m) | Tensile Modulus (GPa) | Tensile Strength (MPa) | Tensile Strain (%) | Strain rate ($s^{-1}$) |
|---|---|---|---|---|
| Quasi-static | 18.09 | 314.92 | 2.51 | 0.00167 |
| 0.5 | 28.58 | 421.67 | 2.27 | 315 |
| 0.75 | 34.80 | 476.96 | 1.94 | 385 |
| 1 | 37.31 | 526.08 | 1.80 | 445 |

Table 1: Tensile properties of neat glass/epoxy at quasi-static and dynamic loading

From the results, it is found that the stiffness of glass/epoxy/clay nanocomposites is more sensitive to strain rates compared to tensile strength. An average of 25% decrease in failure strain is found as strain rate increases for glass/epoxy/clay nanocomposites. Similar observations were found by Majzoobi et al. [26] nearly 50% decrease of failure strain for [±60º] glass/epoxy composite at 621 $s^{-1}$.

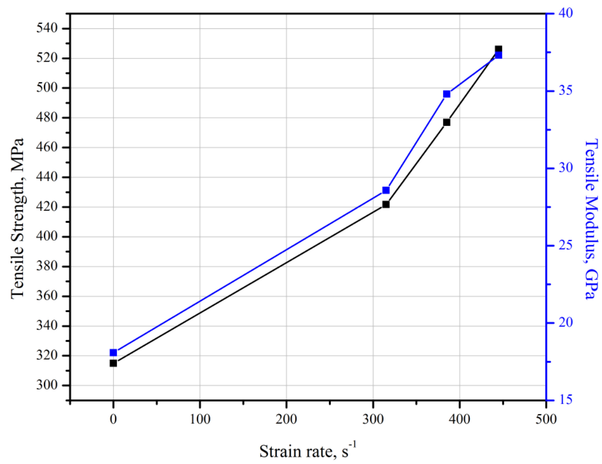


Figure 7: Effect of strain rate on tensile properties of glass/epoxy composites.

## 5.2 Effect of clay on tensile properties

It is found that the tensile modulus of the nanocomposites increases monotonically with increasing clay content. In general, the improvement in elastic modulus is attributed to the good dispersion of nanosize clay particles. Fig. 8 shows the effect of clay on tensile modulus at quasi-static and dynamic loadings. An increment of 15% in tensile modulus is achieved with addition of 3 wt% of clay in glass/epoxy at quasi-static loading. The larger the surface of the filler in contact with the polymer, the greater the reinforcing effect will be. This could partly explain why layered silicates, having an extremely high specific surface area (on the order of 800 $m^2/g$) impart dramatic improvements in modulus even when present in very small amount in a polymer. The good interfacial adhesion between the nano particles and the epoxy matrix restricts the mobility of polymer chains under tensile loading, which could be the reason for increase in tensile modulus. Similar findings are reported by many authors [10], [27]–[30]. It is also noted that the microstructures of glass fiber/epoxy/clay restrict the mobility of the polymer in the interface between the fiber and epoxy or between the clay and epoxy in the glass/epoxy/clay nanocomposites. This allows better stress transfer to the fibers and leads to an improved stiffness at low strain values [7]. The combined effect of high strain rate (445 $s^{-1}$) and clay (1.5 wt%) in tensile modulus is found to be 150%, when compared with neat glass/epoxy composite tested at quasi-static loading.

Similarly, tensile strength is increased with increasing clay content. Fig. 9 shows the effect of clay on tensile strength at quasi-static and dynamic loadings. An increment of 9% in tensile strength is observed with addition of 1.5 wt% clay at quasi-static loading. However, at higher clay loadings, the tensile strength is decreased. It is believed that higher clay concentration causes the formation of clay agglomerations. These clay agglomerates lead to stress concentration, which causes premature failure. At higher clay loadings, the epoxy/clay mixture becomes viscous and obstructs the degassing process, which might be the reasons for decrease in tensile strength. The combined effect of high strain rate (445 $s^{-1}$) and clay (1.5 wt%) in tensile strength is found to be 84%, when compared with neat glass/epoxy composite tested at quasi-static loading.

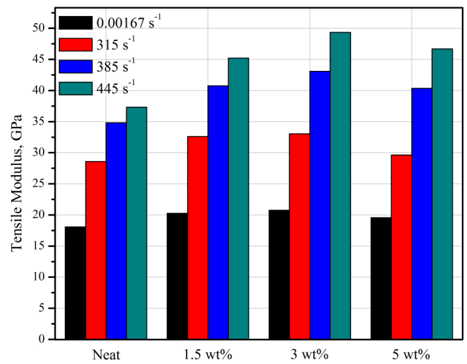


Figure 8: Effect of clay on tensile modulus of glass/epoxy/clay nanocomposites.

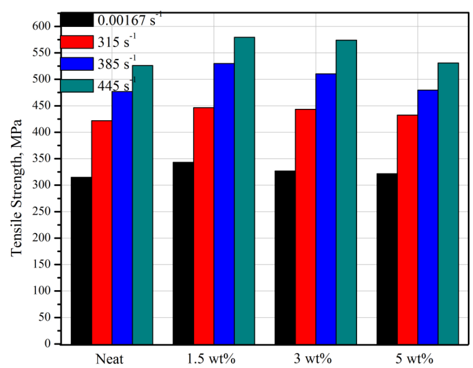


Figure 9: Effect of tensile strength on glass/epoxy/clay nanocomposites.

## 6 CONCLUSIONS

In this study, dynamic high speed tensile tests were conducted on glass/epoxy/clay nanocomposites, using a drop mass setup equipped with dynamic specimen fixture and high speed camera. Data from this instrument fill a gap between conventional, low-rate stress strain curves and SHB measurements. The effect of the strain rate on the tensile behavior of glass/epoxy composite is investigated at strain rates ranging from 0.0016 to 445 $s^{-1}$. Quasi-static and dynamic experiment results indicate that the tensile behavior of glass/epoxy/clay nanocomposites is dependent on the strain rate. When the strain rate increased from 0.0016 to 445 $s^{-1}$, the tensile strength is increased by 67% whereas the increase in tensile modulus is 106% for neat glass/epoxy composite. It is possible to detect general trends, which showed a significant increase in elastic modulus and tensile strength and a clear reduction in failure strain under dynamic loading. The presence of nanoclay has a significant effect on mechanical properties. Even at low weight fraction, an increase in modulus and tensile strength is observed on glass/epoxy/clay nanocomposites. Compared with the neat epoxy, the elastic modulus and tensile strength of the epoxy/clay nanocomposites are greatly improved by 16% and 9%, respectively.

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