Damage Assessment in Glass Fiber-Epoxy Matrix Composite under High Velocity Impact of Ice

Sh.Dolati[1], A.Fereidoon [2], A.R.Sabet [3]

Abstract

This study investigated the influence of nanoclay on the impact damage resistance of glass fiber-epoxy composites under high velocity ice impact loading. Addition of 0.5 wt. % nanoclay into epoxy was shown to improve damage resistance compared to composite plates having no nanoclay platelet. The glass fiber-epoxy composites containing nanoclay brought about substantial improvement in ice impact damage resistance and damage tolerance in the form of smaller damage area. Delamination followed by high velocity ice impact constituted major damage mode in the specimens tested.

Keywords: Nanoclay; High Velocity Ice Impact; Damage Extension

1. Introduction

Glass fiber composites are among advance engineering materials that present high modulus-weight ratios, high strength-weight and are widely used in the aerospace, military, sporting goods and automobile industries. Epoxy resins are engineering class of polymer matrix used for their high strength and stiffness, high temperature resistance, thermal stability, low creep, good adhesion, and excellent process ability. However, cured epoxy resins have a low toughness with mostly brittle behavior when subject to impact so to be able to develop the resin without giving in to its existing properties various methods have been adopted such as addition of toughening agents [1] and introduction of various type of nano sized materials in to the resin system. Nano material reinforcements, such as carbon nanotubes and nanoclays are among nanoparticles that have been studied in the past decades to improve the fracture toughness of epoxy resins [2].

Last few years, polymer-clay nanocomposites have been studied due to their capacity to improve the properties of the polymer resin. These polymers display a complex rheological behavior due to their dispersed structure in the matrix [1,2]. Thus, to earn fundamental understanding of nanocomposite dispersion, characterization of their internal structure and their rheological behavior is crucial [1,2].

Nanoclays have been studied as reinforcement in epoxy systems. Clay has a layered structure in which these layers are bonded together by van der Waal's forces. It has been reported that the dispersion of nano platelets have an important role in determining the mechanical and physical properties of the composite such as fracture behavior and toughening mechanisms [3]. Previous studies [4,5] showed that complete exfoliation encourages strength and stiffness, whereas intercalation is key to improved fracture toughness of the nanocomposites. A balance between an intercalated and exfoliated structure may be beneficial to improve both the modulus and toughness [6]. In addition to mechanical properties, clay–epoxy nanocomposites have also indicated an array of property improvements with low clay contents, including thermal stability, reduced moisture and gas permittivity and superior flame retardancy [7 - 9].

The effect of nanoclay on the mechanical and thermal properties of S2 glass-epoxy composites was showed that by adding 1wt.% of nano silicate, the interlaminar shear strength, flexural strength and fracture toughness were improved by 44%, 24% and 23%, respectively[10]. In another study has been exhibited flexural testing of glass fiber-

1- Department of Mechanical Engineering, Islamic Azad University, Semnan, Iran.
2- Department of Mechanical Engineering, Semnan University, Semnan, Iran.
3- Department of Composite, Iran Polymer and Petrochemical Institute, Tehran, Iran.

Corresponding author: Sh. Dolati, Department of Mechanical Engineering, Islamic Azad University, Semnan, Iran.
Email: afereidoon@semnan.ac.ir

reinforced composites with nanoclay modified matrix improved the modulus and strength, respectively, by 6% and 27% [11].

Amongst the material variables, the mechanical properties of fibre and matrix, interface properties and fibre configuration play important roles in determining impact damage resistance and damage tolerance of the composites [12, 13]. The presence of small amounts of titanium dioxide and Cloisite30B nanoclay, into an unsaturated polyester matrix improved the impact properties of the nanocomposites through the reduction of subcritical cracks in the matrix [14]. Hayes et al. investigated the nanocomposites containing nanoclay as the matrix material to produce hybrid nanoclay-fibre reinforced polymer composites with aim to improve the mechanical and fracture properties [15,16]. The incorporation of nanoclay improved energy absorption of glass fibre composites by about 48% when 20J energy was applied to it [17].

A few studies have been reported on the low velocity impact of laminated and sandwich composites [17,18] and More wide research work is needed on the properties that have not been investigated to establish sufficient database for development application of the hybrid composites [19]. This study aims to present substantial improvements in fracture resistance of the composites under high velocity ice impact loads. The study compares experimental test results on glass fiber-epoxy composites containing nanoclay to that text samples without any nanoclay. Special focus has been placed on understanding effect of nanoclay on fracture mechanisms, damage area and orientation of fiber.

2. Experimental

2.1. Materials and fabrication of composite laminates

Composite and nanocomposite laminates were made using E-glass woven roving cloth with areal density of 400 g/m^2 here after referred to WR (supplied by Camelyaf Turkey) as the main reinforcement. An epoxy resin (CO-207, supplied by Mokarrar materials engineering company Iran) was mixed with hardener (HA-11, supplied by same company) at a ratio of 100:15 by weight. The nanoclay selected for this study is Cloisite30B (supplied by Southern Clay Products USA). This nanoclay is a natural montmorillonite mineral modified with a quaternary ammonium salt. The nanoclay was dried in an oven at 150 °C for 1 h prior to use. Mixing epoxy and 0.5 wt. % nanoclay was conducted at a shear rate of 2000 rpm for 1h using a high speed shear mixer (Heidolph, Germany). The mixture was subjected to sonication using an ultrasonicator (Hielscher, Germany) at an ultra-high frequency for 30 min to further disperse the clay, while resin temperature was maintain cold using a cold water bath. The mixture was degassed in a vacuum oven and followed by addition of curing agent. Two, four and six ply composite and nanocomposite laminates were prepared by hand lay-up with lay-up of [WR], [±45°], [WR/WR], [±45°/±45°], [WR]3s and [±45°]3s on a steel mould plate which was cured at room temperature for 24 h and followed by post-cure at 80°C for 8h in a vacuum hot press.

2.2. High velocity ice impact tests

Impact tests were performed on 150 mm × 150 mm square samples at room temperature. High velocity ice impact tests were conducted using a gas gun (see Figure. 1). The gas gun consists of 1.75 m long smooth barrel with inside diameter of 21 mm, a fast acting high pressure release valve, a breech unit, a rupture disk unit, a supply gas vessel, a 500 ml gas reservoir for each shut release, a target holder and one projectile velocity measuring units [20]. Initial velocity of ice projectile was measured after it was motivated from the gun barrel using a chronograph F-1 model from Shooting Chrony.

A cylindrical ice impactor (see Figure. 2) of 11.7 gr in weight with a 20.5 mm in diameter, 40 mm length and high velocity ice impact 140 m/s was impacted on the centre of specimen whose edges were firmly fixed.

Since ice projectile are strongly dependent on temperature, after the ice projectile was removed from the freezer, weighed, and then directly placed into the pipe. For all specimens' tests, three specimens for each composition were tested.

2.3. Ice impact damage measurement

Damage area for all specimens after impact tests were determined using a back light marking as well as getting help from whitening phenomenon associated with brittle fractures of composite and nanocomposite laminates. For all specimens, scanning was performed by direct photo scanning of both sides with a flatbed scanner at 300 dpi. Damage areas were measured by ImageJ software [21].

The objectives of this experiment was to observe and measure the impact damage and damage modes behaviour on composite and nanocomposite laminates by hail ice impact and to quantify the effect of parameters such as variation in orientation of layers, thickness of specimens, the effect of nanoclay in laminates. As *seen* in the photos, the ice cylindrical crushes into fine powder immediately upon contact and continue to fail at the platen face during the impact event [22].(see Figure. 3)

2.4. Characterization

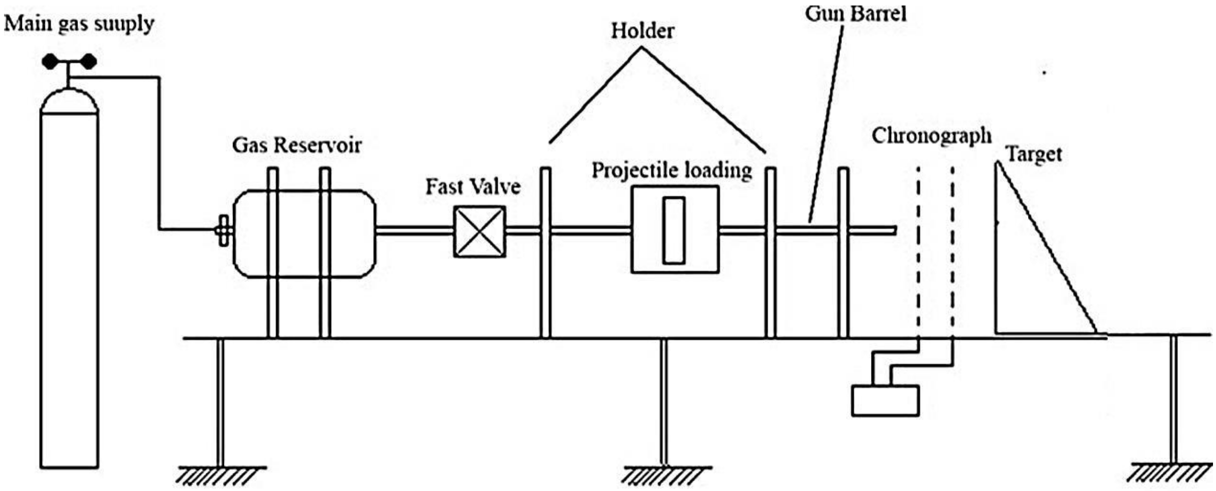

Fig. 1. Schematic representation of high velocity impact testing device (gas gun).

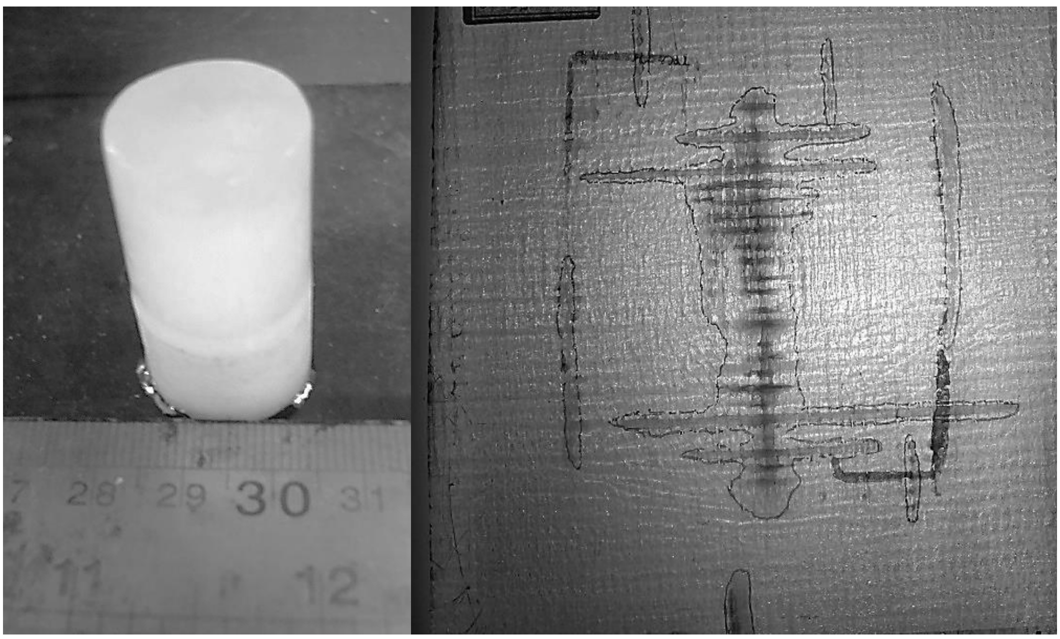

Fig. 2. (a) Moulded ice projectile. (b) Dark area showing damage extension area after impact.

X-ray diffraction (XRD) were obtained using a Philips XPERT XRD system equipped with CuKa radiation at the generator voltage of 40kV and Generator current of 40mA (λ=1.5405A°). The Bragg's law, λ=2dsinθ, was used to calculate the crystallographic spacing (d-spacing).

Transmission electron microscopy (TEM) observation was performed on an EM900 using an accelerating voltage of 80 kV. TEM was performed on ultra microtomed specimen prepared using a LEICA microtome equipped with a glass knife mounted on 200 mesh copper grids.

3. Result and Discussion

3.1. Modes of ice impact damage

Damage area for all composite and nanocomposite laminates specimens were measured and damage modes were investigated. Figure. 4 describes the damage modes at impact energy of 114.66J. Note that all of damage modes were not particularly observed in progression for composite and nanocomposite laminates.

Impact damage tends to be located within a ‘‘fir-tree’’ shaped zone (Figure. 5) where the delamination increases from the impacted surface [23]. Delamination is started at almost every dissimilar lamina interface and tends to be oriented in fibre direction of the lamina [24]. Delamination failure caused by interlaminar stresses is the most important failure mode observed in composite structures.

Since the ice impact dissipated on specimens, the laminate specimens with nanoclay differ from the laminate specimens without nanoclay in that the delamination are much lower due to presence of nanoclay. Several of specimens with and without nanoclay (Figure. 4) by hail ice impact, fiber splitting and fiber fracture were observed that it is due to lower thickness and upon impact energy.

3.2. Damage area measurement

The damage area in the specimens has been calculated based on the above method. This area includes all the damages that have been entered into the specimen, which may be included all the various modes of damage, such as delamination, fiber splitting, matrix cracking, and fiber fracture.

Figures 6-8 presents the damage area vs. fiber orientation and stacking sequence

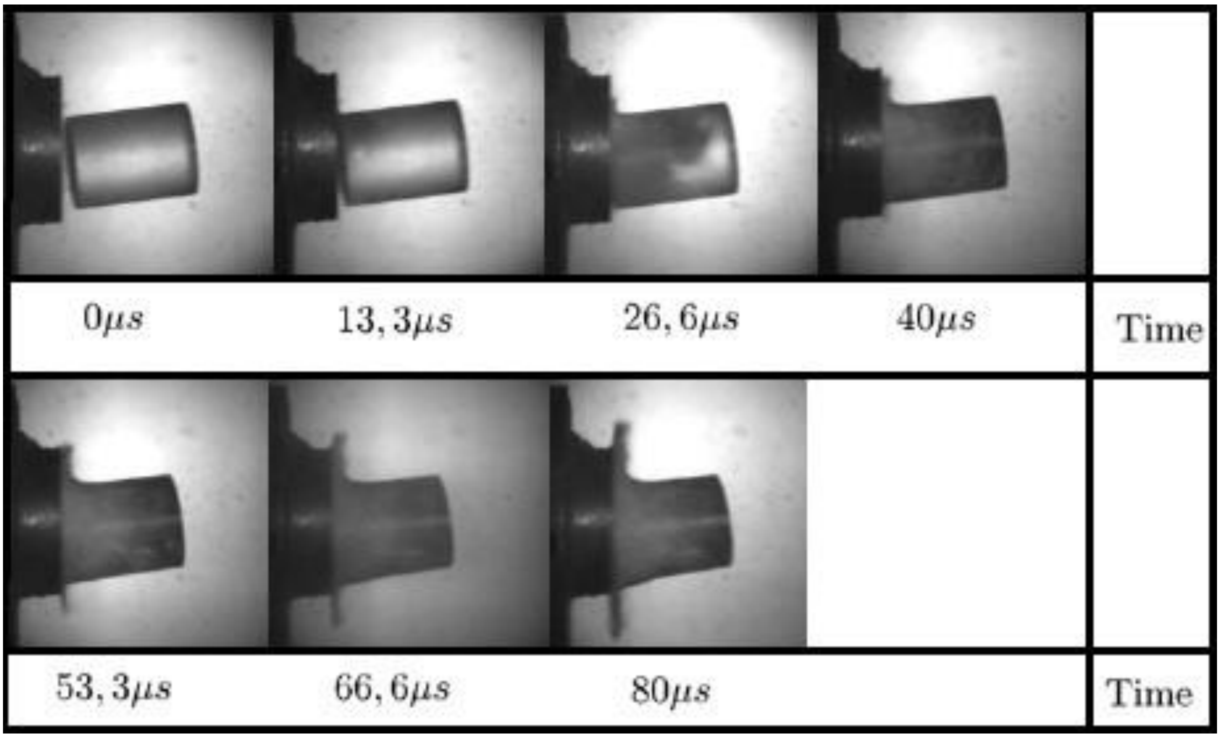

Fig. 3. The failure history for a cylindrical ice [22]

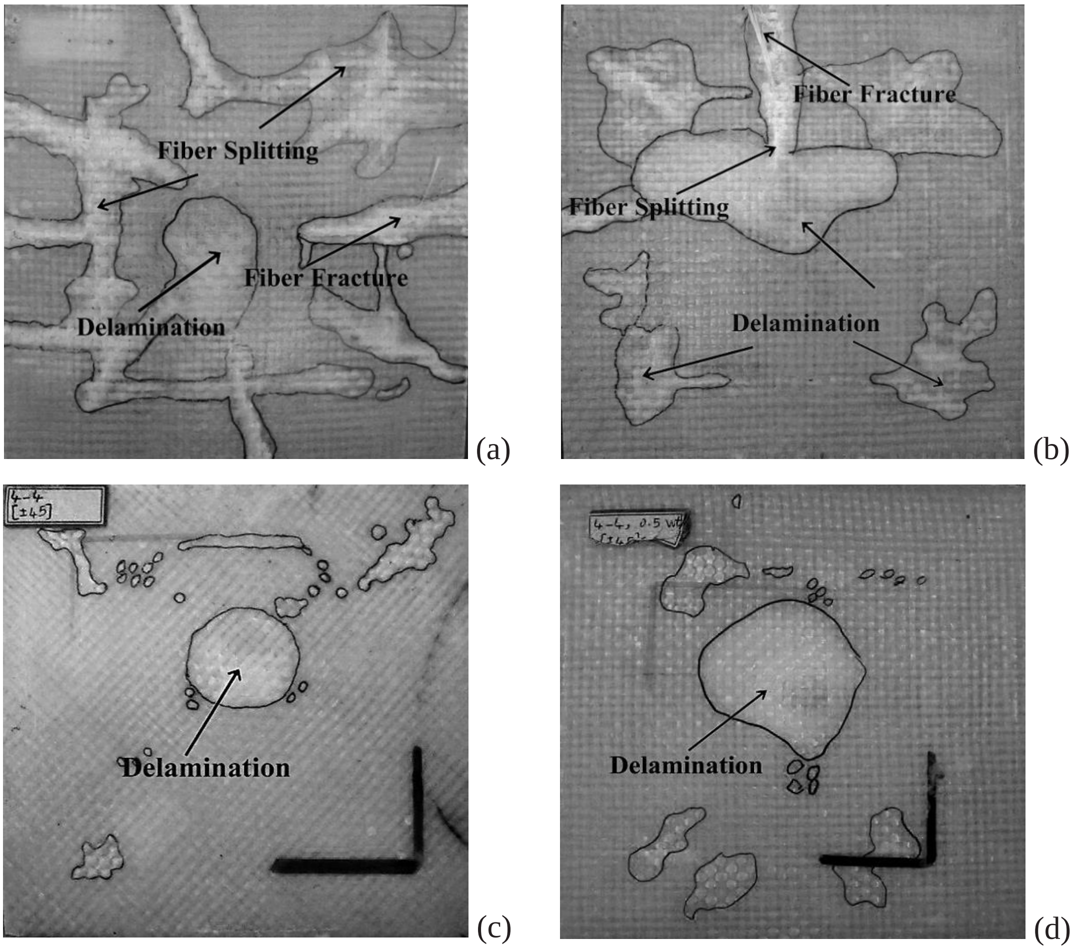

Fig. 4. Damage extension for (a) Sample [WR/WR] two layered, (b) Sample [WR/WR] two layered with nanoclay, (c) Sample [±45°/±45]s four layered, (d) Sample [±45°/±45°]s four layered with nanoclay.

containing two, four and six layers.

Decreasing in thickness of laminates were be two layers, indicating that the more damage created and to reduce the resistance of the laminates (to note that the incipient impact energy were almost constant). As shown, the laminate with ±45 for all of laminates thickness exhibited the higher damage resistance compared with corresponding laminates with 0/90 reinforcement. This behaviour may be attributed to the effect of transverse shear stresses are being involved with the intralaminar cracks and transverse shear stresses and delamination are recognized to be related to the laminate stacking sequence [25]

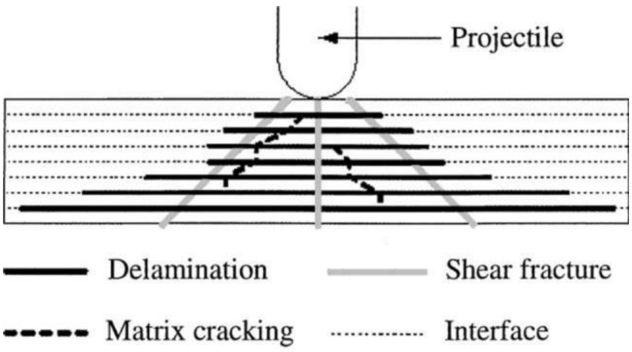

Fig. 5. Illustration of impact damage in a laminate. [23]

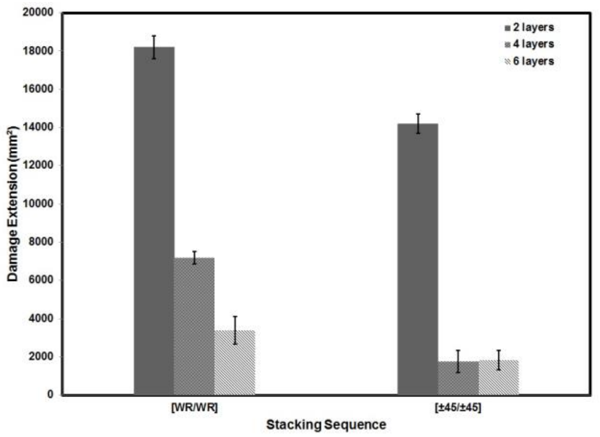

Fig. 6. Damage extension vs. different stacking sequence of two, four and six layered composite panels under hail ice impact.

so transverse shear stresses are involved to increase the ice impact resistance in ±45 laminates than 0/90 laminates.

It should be noted that nanoclay has a strongly effect on damage area in both the front and back faces (see Figures. 7, 8). The composite laminates containing 0.5 wt. % nanoclay had the smallest damage area amongst all laminates studied, whereas the damage areas of those containing neat resin. The higher damage resistance of the laminates with 0.5 wt. % nanoclay is related with a higher absorbed energy and higher interlaminar fracture resistance than the other laminates, so it is accepted that the strengths of nanocomposites constitute the state of nano filler dispersion.

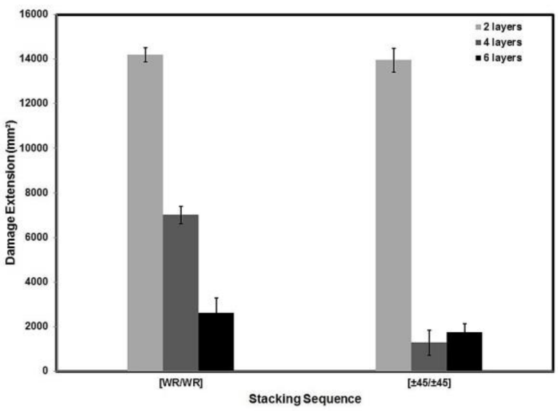

Fig. 7. Damage extension vs. different stacking sequence of two, four and six layered nanocomposite panels under hail ice impact.

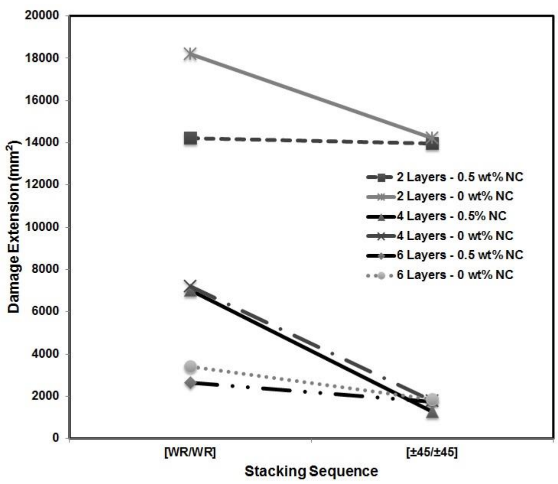

Fig. 8. Comparison of damage extension between with and without nanoclay (NC) samples under single hail ice impact.

3.3. X-ray diffraction analysis

In this work, the quality of the dispersion of nanoclays in the resin was evaluated from rheology tests knowing that a high level of exfoliation of the nanoclay platelets. This rheological behavior shows the ability of the nanoclays to "interact" with the polymer matrix, its surface treatment. X-ray diffraction (XRD) and transmission electron microscopy (TEM) were used to study the distance between the nanoclay platelets and investigation on dispersion morphology nanoclay platelets in epoxy resin.

Figure.9 shows the XRD patterns for epoxy resin-Cloisite30B nanocomposites at 0.5wt. % clay contents, as compared with pure nanoclay (Cloisite30B). For epoxy resin-Cloisite30B nanocomposite with 0.5wt. % content of Cloisite30B, the interlayer spacing has increased from 16.44A° of original Cloisite30B to amount 34.77A°, indicating the great extent of intercalation of the Cloisite30B layers by epoxy resin. While nanocomposites containing 0.5 wt. % nanoclay Cloisite30B showed almost no peaks, indicating an exfoliation of Cloisite30B in epoxy resin. These results show exfoliation and intercalation of nanocomposites containing 0.5wt. % Cloisite30B. This is attributed to strong miscibility between epoxy resin and Cloisite30B [23].

In Figure.10, present TEM microgargh of specimen with 0.5 %wt. nanoclay, in this micrograph, the dark lines are individual silicate layers and with area indicate epoxy

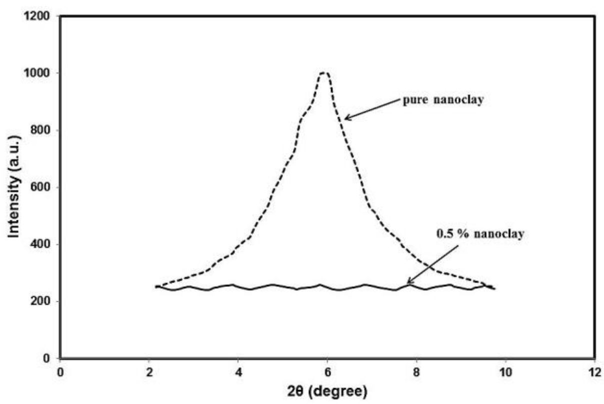

Fig. 9. XRD patterns of Epoxy-Cloisite30B nanocomposites and pure Cloisite30B.

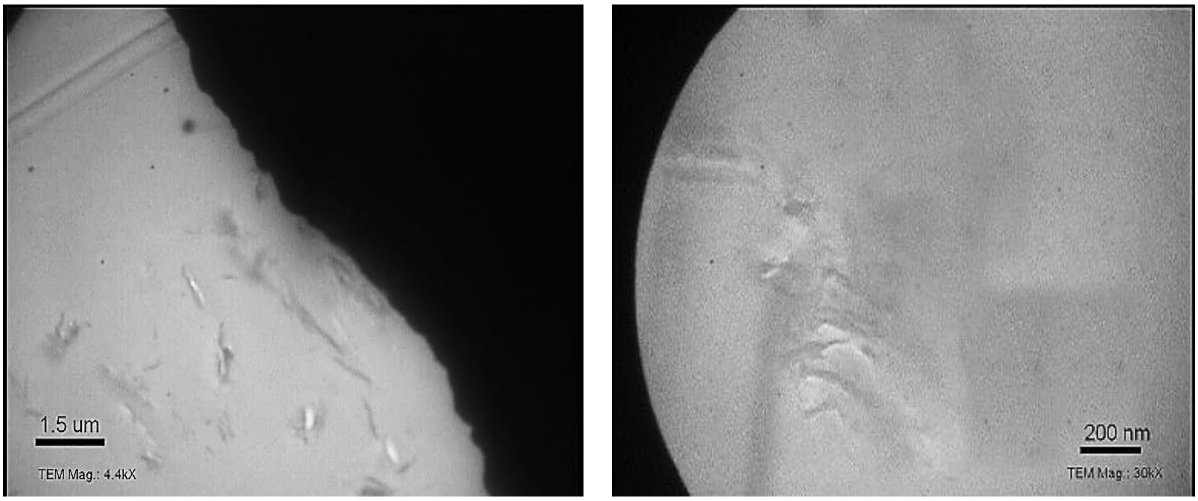

Fig. 10. TEM micrograph of specimen containing 0.5 wt.% nanoclay.

resin. It can be seen clay layers irregularly separated to with relative exfoliation resulting in well dispersion of polymer nanocomposites. TEM photograph showed that as the size of the nanoclay platelets increases and the distance between the platelets is also increased indicating exfoliation of nanoclays in epoxy matrix. Therefore, a well-dispersed and homogeneous blend showed and the mechanical performance of the nanoclay composites is increased.

4. Conclusions

To access this study ice impact damage on the composite laminates containing nanoclay modified epoxy matrix under high velocity impact loading. The position impact properties as evaluate of damage tolerance of composite were studied for application industry by and by. From the results, the following conclusions can be highlighted:

Specimens containing 0.5 wt. % of nanoclay showed lowered damage extension compared to nanoclay free laminates.

Increase in laminate thickness resulted in lower damage extension.

Laminates with ±45 fiber orientation in both nanoclay containing and nanoclay free specimens produced lower damage extension compared with 0/90 woven roving reinforcements.

Addition of nanoclay improved the shear strength in laminate layers giving rise to a higher resistance to fibre under high velocity ice impacts. The laminates containing nanoclay tended to fail mainly by delamination.

XRD diffraction analysis and transmission electron microscopy confirmed intercalation and exfoliation of nanoclay particles in epoxy resin matrix.

Delamination, fiber fracture and fiber splitting constituted major damage mode in all specimens.

References

1. S. Ullah, K. Iqbal, A. Munir, J-kyo Kim: *Composites Part A*, 42 (2011), 253.
2. F. Bensadoun, N. Kchit, C. Billotte, F. Trochu, E. Ruiz: *J.Nano.Mat*, 2011 (2011).
3. T. Lan, T. Pinnavaia: *Chem. Mater*, 6 (1994), 2216.
4. W.J. Boo, Liu, H.J. Sue: *J. Mater. Sci. Technol*, 22 (2006), 829.
5. A.S. Zerda, A.J. Lesser: *J. Polym. Sci. Part B: Polym. Phys*, 39 (2001), 1137.
6. C. Zilg, R. Mulhaupt,J. Finter: *Macromol. Chem. Phys*, 200 (1999), 661.
7. A. Yasmin, J.J. Luo, J.L. Abot, I.M. Danial: *Compos. Sci. Technol*, 66 (2006), 2415.
8. J.K. Kim, C.G. Hu, R.S.C. Woo, M.L. Sham: *Compos. Sci. Technol*, 65 (2005), 805.
9. J.K. Kim, C.G. Hu: *Compos.Inter*, 12 (2005), 271.
10. A. Haque, M. Shamsuzzoha, F. Hussain, D. Dean: *J. Compos. Mater*., 37 (2003), 1821.
11. X. Kornmann, M. Ress, Y. Thomann, A. Necola, M. Barbezat, R. Thomann: *Compos. Sci. Technol*, 65 (2005), 2259.
12. K. Iqbal, S.-ullah Khan, A. Munir, J.-kyo Kim: *Compos. Sci. Technol*, 69 (2009),1949.
13. J.K. Kim, D.B. Mackay, Y.W. Mai: *Composites*, 24 (1993), 485-.

14. J.C. Lin, L.C. Chang, M.H. Nien, H.L. Ho: *Compos. Struct.*, 74 (2006), 30.
15. J.F. Timmerman, B. Hayes, J.C. Seferis: *Compos. Sci. Technol*, 62 (2002), 1249.
16. N.A. Siddiqui, R.S.C. Woo, Jang-K Kim, C.C.K. Leung, A. Munir: *Composites Part A*, 38 (2007), 449.
17. A.F. Avila, M.I. Soares, A.S. Neto: *Int. J. Impact Eng*, 34 (2007), 28.
18. M.V. Hosur, F.H. Chowodhury, S. Jeelani: *J. Compos. Mater*, 41 (2007), 2195.
19. M.F. Lai, J.K. Kim: *Polymer*, 46 (2005), 4722.
20. A. R. Sabet, M. H. Beheshty, H. Rahimi: *Polymer composites*, (2008) 932.
21. http://rsbweb.nih.gov/ij/download.html (accessed 2011).
22. A. Combescure, Y. Chuzel-Marmot, J. Fabis: *Int. J. Solids Struct*,. 48(2011), 2779.
23. M. A. Will, T. Franz, G. N. Nurick: *Compos. Struct.*, 58 (2002), 259.
24. S.A. Hitchen, R.M.J Kemp: *Composites*, 26 (1995), 207.
25. S.H. Dolati, A. Freidoon, A.R. Sabet: J. *Compos. Mater.*, published online 26 April 2013.