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Evaluating the mechanical properties of E-Glass fiber/carbon fiber reinforced interpenetrating polymer networks

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Abstract

A series of vinyl ester and polyurethane interpenetrating polymer networks were prepared by changing the component ratios of VER (Vinyl ester) and PU (Polyurethane) and the polymerization process was confirmed with Fourier Transform infrared spectroscopy. IPN (Inter Penetrating Polymer Network - VER/PU) reinforced Glass and carbon fiber composite laminates were made using the Hand lay up technique. The Mechanical properties of the E-glass and carbon fiber specimens were compared from tests including Tensile, Compressive, Flexural, ILSS (Inter Laminar Shear Strength), Impact & Head Deflection Test (HDT). The IPN Reinforced Carbon fiber specimen showed better results in all the tests than E-Glass fibre reinforced IPN laminate with same thickness of the specimen, according to ASTM standards. It was found that the combination of 60%VER and 40%PU IPN exhibits better impact strength and maximum elongation at break, but at the slight expense of mechanical properties such as tensile, compressive, flexural, ILSS properties. The morphology of the unreinforced and reinforced composites was analyzed with help of scanning electron microscopy.

Keywords: *glass fiber, carbon fiber, morphology, IPN laminate, mechanical properties, Fiber Reinforced Plastics (FRP).*

1. Introduction

Nowaday's fiber Reinforced composites are widely used for many applications like structural, marine, aerospace, automobile, windmill blades etc., because of their high strength to stiffness, weight to stiffness ratio[1]. Especially in naval applications, its requirement is gone to the maximum extent of building the hulls and various structures. Many researchers have attempted with different fibers and resins ratios, to obtain the high toughened material[2]. Significant development and rigorous ageing tests have also been conducted over the FRP material, in order to understand, improve and investigate the material for different environmental conditions. It is very widely considered as the replacement for the steel and aluminum. Since it is used for structural and marine environments, many parameters have necessitated us to find its strength before it is finally deployed in to the field. It is very much essential for the user that the complete report on the material before it is been substituted as alternate material[3,4]. Epoxy is one of the predominant thermosetting resin in many decades and it is widely accepted as the FRP manufacturers to manufacture all sort of FRP products and dominates in the field of PMC, where as the cost makes the product too high[5]. The another very important polymer matrix material very extensively used in the field of composite manufacturing is vinyl ester, It is well known and accepted for its unique properties like corrosion resistance, impact resistance and cost, apart from that it exhibits good adhesion property between fiber and resin. Polyurethanes are another one of most versatile material and well known for his flexible nature, excellent tear resistance, lower price, abrasion resistance, shock absorption and elasticity property[5-7].

Normally FRP (Fibre Reinforced Plastic) manufacturers employ the chemistry to add more value to their products to make them competitive in the product in the field of use, but time and cost incurred for these phenomena is quite high. Instead of giving importance to their chemistry, if blend taken place between two resins, final resin improve the required property. Though this process diminishes the individual characterictics of the resin, it substantially improves many physical properties of the final product[8]. Taking all this factors in to account, the new beginning on interpenetration of polymer networks has been started. From the point of view, IPNs are special class of polymer networks Figure 1, bonded together with the permanent or physical entanglement not on covalent bonds.

Their isotropic morphology study and mechanical propeties like tensile, flexural, impact have completely being studied and reported in many publications. Despite there are many publications on IPNs based composite, reinforcement of fiber in polymer blends is limited[7-14]. In our present study, definite proportions of VER/PU IPNs are synthesised at room temperature and the same reinforced with the E-Glass Fiber (Silane Treated Glass Fibre to have better interaction with the resin) and Carbon Fiber, since both the fibers are advantageous in all the mechanical properties. At last the comparisons of the mechanical properties are made between E-Glass fiber and Carbon fiber[15].

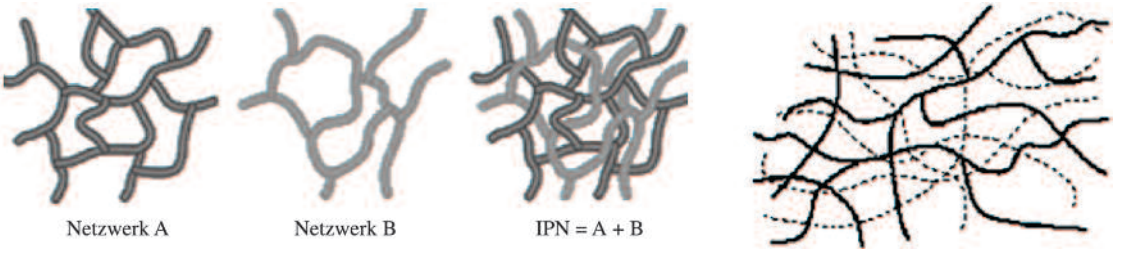

Figure 1. Structure of IPNs.

2. Experimental Setup

2.1 Materials

Two types of fiber were used for the comparsion purpose to fabricate the IPN reinforced composite material. Both fibers were purchased with the specification of plain woven fabric type; one is Woven E-Glass fiber (350 gsm) and another one Carbon fiber (350 gsm) both the fibers were used as the reinforcing materials in polyurethane/Vinyl ester interpenetrating polymer networks. Vinyl ester (VER), Polyol **(EMPEYOL E4000/2E4000, dihydroxy polyether polyol.) (PU)** were purchased from the firm MPL. Toluene (AR Grade, MERCK) & MEKP (Industrial Grade, catalyst) were used as received without any purification.

2.2 Preparation of VER/PU IPN

The polyurethane prepolymer was prepared by mixing 50 grams of polyol with 50 grams of toluene di isocynate (TDI) in toluene medium. The prepolymer was then mixed with Vinyl ester resin in varying proportions as given in Table 1. The catalyst (Methyl ethyl ketone peroxide, MEKP) and accelerator (Cobalt naphthanate) was added to the mixture and stirred continuously for 20 minutes.

2.3 Fabrication of VER and VER/PU IPN-Glass and carbon fiber composite

Plain woven carbon and glass fabric were cut into definite size and heated in the oven at 150°C to make it moisture free before processing. Calculated percentage of PU/VER resin was applied on the Plain woven glass fabric surface by a Hand lay – up technique, 5 layers were stacked successively in order to get about 3 mm thick composite. This was allowed to cure for 24 hours at room temperature and then was taken out of the mold and post cured at 80°C for 2 hours. The same kind of procedure as well was followed to obtain the carbon reinforced IPN composite, 100% VER – Glass fabric and 100% VER Carbon fibre composite. To measure the mechanical strength of E-Glass/Carbon IPNs, the samples were prepared around the ratio 60 wt% of fibers. There may the chance of formation of void content in the IPNs because of presence of carbondioxide, which was the by product of the isocyanate and presence of humidity in the air, during the course of study it might harm the strength of the specimen. So utmost care was taken during specimen manufacturing in such a way, before specimen manufacturing the polyol and the TDI was kept in the degassed chamber for the period of two hours to eliminate the dissolved air and water[16].

Table 1. IPN Formulation.

S.No	VER (grams)	PU (grams)	MEKP	Cobalt Naphthanate
1	100	0	1.5	1.0
2	100	10	1.5	1.0
3	100	20	1.5	1.0
4	100	30	1.5	1.0
5	100	40	1.5	1.0
6	100	50	1.5	1.0

2.4 Measurements

The FTIR spectra of IPN (PU 20%) composite were recorded between 400 – 4000cm-1 with a Perkin Elmer FTIR Spectrometer with KBr pressed pellet. Samples of VER/PU were kept on the ATR attachment and a minimum of scans were averaged with a resolution of 2 cm^{-1}. During the course of study, characteristic absorption peaks of functional group were detected and monitored. By using Universal Testing Machine (Instron Model) the mechanical properties of E-Glass/Carbon fibre reinforced IPN were investigated. The tensile test procedures were followed as per ASTM 3039/3039M-00[17-19]. The test piece was sized to the dimension of 250 mm × 25 mm × 3 mm (length x width x thickness) with end tabs at both ends, by maintaining the cross head speed of 1mm/min at room temperature. The tensile strength (σ), elongation at break (ε), and the modulus of elasticity (E) of the IPNs were found on each trail. Secondly, the Compressive strength was also analyzed according to the ASTM D 6641. The specimens were neatly polished at the sides and corners, rounded off with help of metallographic polishing machine to the size of 12 mm width by 140 mm length[1]. The tests were carried out with the cross head speed of 1.33 mm/min[20,21]. With the help of compressive strength test, the following results were calculated a) Compressive strength (σ) b) compressive strain (ε) c) compressive elasticity modulus (E= σ/ ε)[22]. Thirdly, Three point bending flexural test were also carried out as per ASTM D790-03[10] pertained to testing of plastics, the size of the flexural test pieces were 127 mm × 12.5 mm × 3 mm (length × width × thickness) with the cross head speed of 2 mm/min, span of 90 mm was also given[23].

The standard also states that the span of the specimen should be above 16 times the thickness of the specimen. Each ends were given the 10% of the span of the support. The Flexural Stress (σ_f), Flexural Strain (ε_f), Flexural Modulus of Elasticity (E_B) was calculated[24]. Apart from the above three tests, to get the Inter laminar shear strength value the

ILSS test were carried out as per ASTM D2344 standard. Initially the specimens were placed over the three point bending rig for a span length of 15 mm, following that the load was applied in the transverse direction with cross head speed of 1 mm / min. The specimen sizes were 18 mm × 6 mm × 3 mm (length × width × thickness). The Samples were fixed in the grips and the load were applied in such a way that the failure occurred at the mid-plane interface of the specimens[25,26]. To get the energy of failure value of the laminate, the impact strength was analyzed for all the proportions according to the standard ASTM D256-03, Izod Mode.

The dimensions of the samples were 63.0 mm × 12.8 mm × 3.2 mm (length × width × thickness)[27,28]. At last, the heat deflection temperature was investigated and measured by the HDT Tester, and the guidelines were followed as per the ASTM D648–01[29,30], with the loading pressure of 0.455 MPa, to the raising temperature of 2°C/min. Testing results were obtained from an average of five specimens. The fractured Specimens were examined using scanning electron microscope (SEM) model EVO MA15 to find out inter bonding of IPNs. Before to SEM evaluation, the specimens were coated with gold using plasma sputtering apparatus Edwards sputter coater model S150B. The inter laminar bonding strength of composite were determined at room temperature.

3. Results and Discussion

3.1 Infrared analysis

The Figure 2 shows FTIR spectra of 100% VER, 10% PU and 20% PU. From the spectrum (a) it is seen that the peak at 1618 cm^{-1} which is characteristic of – C = C – is seen clearly for 100% VER system. Addition of 10% PU and 20% PU prepolymer in to VER system does not show any marked change in the absorption bands indicating that IPN composed of VER and PU can be considered as ideal ones with negligible chemical bonds between the two networks[8,10,11].

3.2 Tensile strength results

The breaking stress on each specimen was calculated by dividing the break load by dividing the cross sectional area of the neat specimen. Though the breaking strength of the composite purely based on the modulus of fiber, the specimen with 100% of VER shown the better tensile stress strength in both carbon and E-glass reinforcement, this was because of the (hard segment) higher modulus value of VER. It shows the significance of VER brittleness property in to the system, the brittleness property of the VER also reduces the elongation at break considerably[9].

In Figures 3a, b, the strength of 100% VER shown as 854 MPa and 447 MPa respectively for carbon and E-glass fiber reinforcement. Also found that the strength of carbon fibre is twice that the value of E-glass fibre approximately for the same fiber volume fraction, the ratio was found to be in this study was 1.80 to 1.91[2]. The different componenet ratio of VER and PU shows the different mechanical properties. It was because of the higher elastic modulus of the PU content in the composite.The mechanical property of the IPNs are purely based on the strength and modulus of the reinforcing material used in the specimen manufacturing[8]. The E-glass fibre reinforced IPNs exhibits the explosive failure in the gage area, the reason was that the E-Glass fiber (polar fiber) possess very good hydrogen bonding and other polar interactions. Whereas the carbon reinforced IPNs shown Lateral failure in the gage area (pull-out fracture mechanism)[14]. The reason for pull out may be mainly because of the absence of bonding between fiber and matrix.

3.3 Compressive strength results

The compressive stress and compressive modulus of VER/PU IPN Glass fibre and VER/PU IPN Carbon fibre composites are shown in Figures 4a, b. From the figures

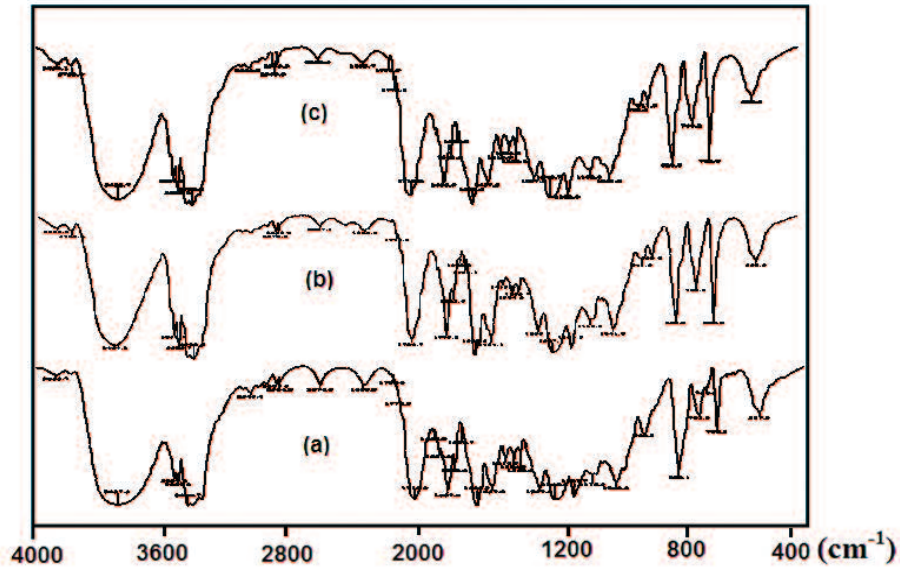

Figure 2. FT-IR spectra of IPNs (a) 100% VER; (b) 10% PU; (c) 100% PU.

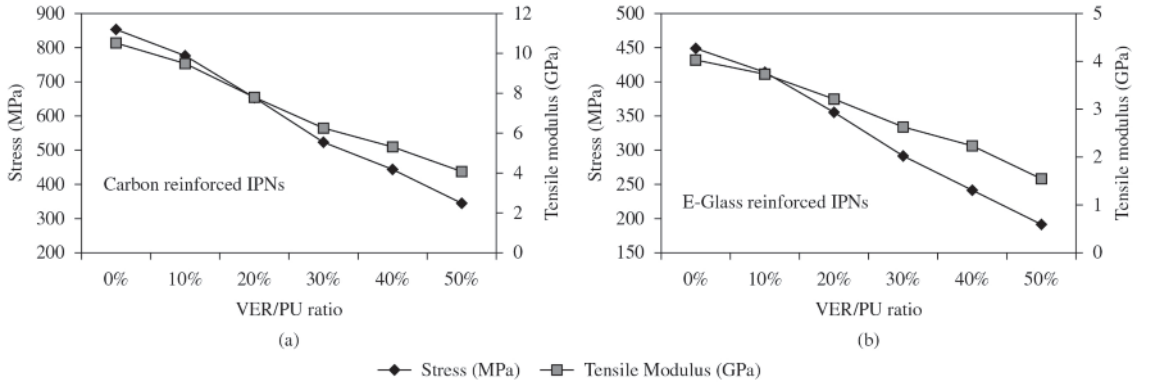

Figure 3. (a) Tensile Stress, Modulus diagram of Carbon Reinforced IPNs; (b) Tensile Stress, Modulus diagram of E-Glass Reinforced IPNs.

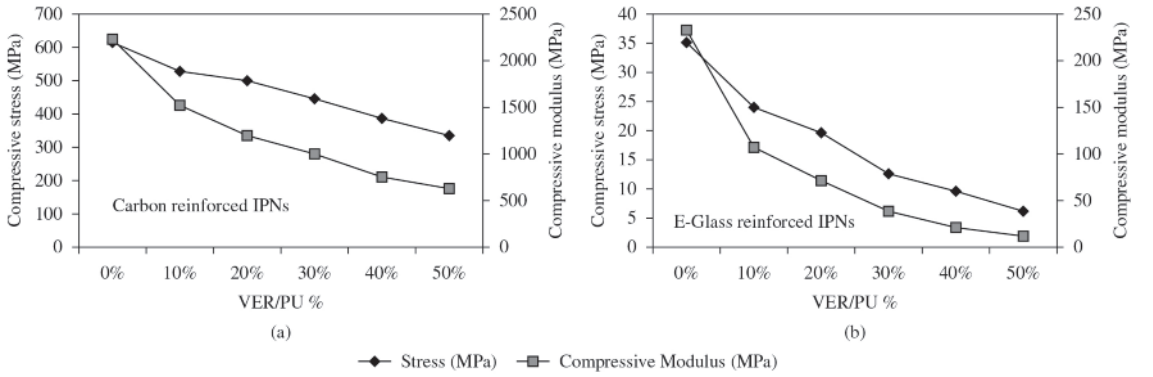

Figure 4. (a) Compressive Stress, Modulus diagram of Carbon Reinforced IPNs; (b) Compressive Stress, Modulus diagram of E-Glass Reinforced IPNs.

it is observed that the neat VER Glass fibre composite and carbon fibre composite has a compressive stress of 613 MPa and 35 MPa respectively. On increasing the PU content the compressive stress of both carbon fibre and glass fibre reinforced composite is found decrease by 47%, 23%, 55%, 33%, and 50% for 10%, 20%, 30%, 40%, and 50% of VER/PU Glass fibre composite, and 15%, 7%, 11%, 16%, and 15% for 10%, 20%, 30%, 40%, and 50% of VER/PU Carbon fibre composite. A similar decreasing trend in compressive modulus is observed with VER/PU Carbon fibre and glass fibre composite. The reason for this decreasing trend in compressive stress and modulus may be attributed to ether linkages present in PU Pre polymer which offers flexibility to the IPN formulation[23].

3.4 Flexural strength results

Figures 5a, b shows the flexural stress and flexural modulus of E-glass & Carbon Fibre reinforced composites with PU content of 0%, 10%, 20%, 30%, 40% and 50%. The following parameters have been extracted from the above diagram. (a) Flexural Stress (b) Flexural Modulus. As the value indicates in the tensile test, the flexural as well shows similar trend of tensile graphs. Adding of PU with VER matrix, markedly decreases the flexural stress and flexural modulus of the composite. The above results shows composite with higher PU significantly decreseases the flexural stress from 496 MPa to 248 MPa (E-Glass) and 957 MPa to 508 MPa (Carbon) respectively. Similar way the modulus value decreases from 930 MPa to 83 MPa (E-Glass) and 2870 MPa to 714 MPa (Carbon) respectively[8,14]. Nevertheless, the elongation of these two IPN reinforced composites were quite different, this mainly because of the strength of the fibre reinforcement. The PU acts as a plasticizer in the IPNs and lowers the stiffness value and leads to much increased strain to failure ratio.

3.5 Inter laminar shear strength results

Figure 6, illustrates the numerical value of the ILSS of IPN reinforce composite. It is interesting to note that ILSS measures how adequate, interfacial adhesion exist between the fiber and matrix and hence it is really a key factor in the knowing the performance of composites[25]. In this study the carbon fiber reinforced IPNs shows better strength than the E-glass reinforced IPNs. As if in the tensile strength study, it was observed that the ratio of Carbon to E-Glass reinforced IPNs were about 1.80 to 1.91. But whereas here (ILSS) the ratio of Carbon to E-Glass were about 1.25 to 1.35[2], this was because, the E-Glass fibers contains functional group like Hydroxyl groups on the surface of the fiber, it gives the better interfacial adhesion between the fiber and matrix[8]. Thus when cross linking completed after curing, a certain portions of the side chains were left and dangling in the system, so it creates free volume in the network and gives the plasticizing effect[8]. But It was recognized from

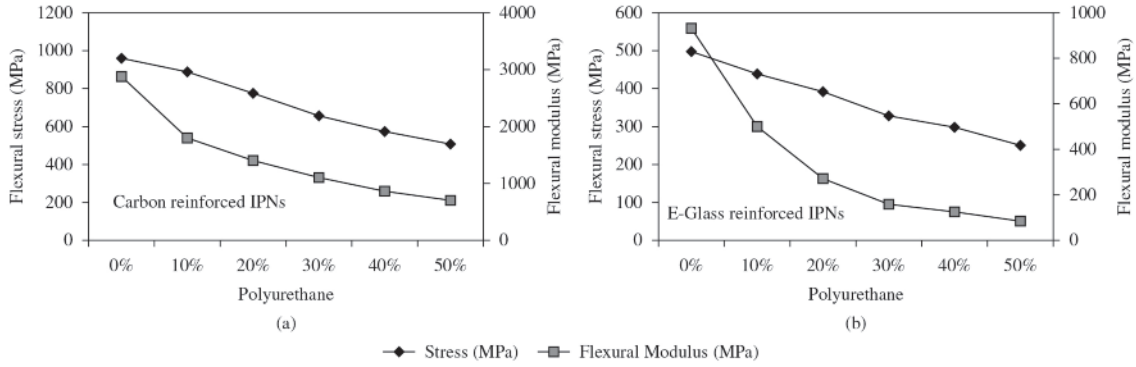

Figure 5. (a) Flexural Stress Vs Strain diagram of diagram of Carbon Reinforced IPNs; (b) Flexural Stress Vs Strain E-Glass Reinforced IPNs.

the above Figure 6 that, carbon fiber surface did not bond well to matrix resins, which resulted in poor shear and compression strength on composite.

3.6 Impact strength results

The Figure 7 shows the energy absorption characteristics of Carbon and E-Glass IPNs. The value of 0% PU falls in the range of 12 KJ/m^2 and 19 KJ/m^2 respectively. Normally VER possess excellent damping property because it contains either ethyl ester or butyl ester groups, which have good potential barrier and more mobility. As well its side ester group increases the distance between the molecules and offsets volume effect of the bulky groups[16].

The vinyl ester based composites showed the fiber pull out mechanism and rupture on fiber, after the impact test[18]. PU/VER IPNs shows higher impact values while doing the impact strength, the soft structure of PU forms the graft - IPN structures, and this soft structure increases the toughness property of reinforced composite (E-Glass/Carbon), this leads to high shear – rate fracturing[22]. This behaviorism is mainly because the permanent entanglement between the VER and PU. The soft property of PU influences to the maximum extent and affects the brittle property of VER, by the way it exhibits higher impact strength than the neat resin of VER[14,22].

3.7 Heat deflection strength results

The Figure 8 illustrates the Head Deflection Temperature of IPN based composite. The values reveal that the HDT value of the IPN reinforced composite decreases gradually by increasing the addition of PU in to the system. The HDT value of the composite decreases from 85°C to 47°C (carbon) and 78°C to 45°C (E-Glass) respectively for both the reinforcement, by gradually increasing the addition of PU. IPN reinforced composite with 0% PU exhibits a higher heat deflection temperature. Moreover it looses the HDT value by raising the PU significantly, irrespective of fiber reinforcement[13]. This phenomenon mainly because of the presence of interaction between hard segment and soft segment (HS-SS) in the composite, while raising the temperature the hydrogen bond present in the hard segment are getting disrupted. Increase in the temperature, eventually makes the hydrogen bond to become inactive; this leads to chain slippage in the hard segment, due to this PU looses its stiffness[7].

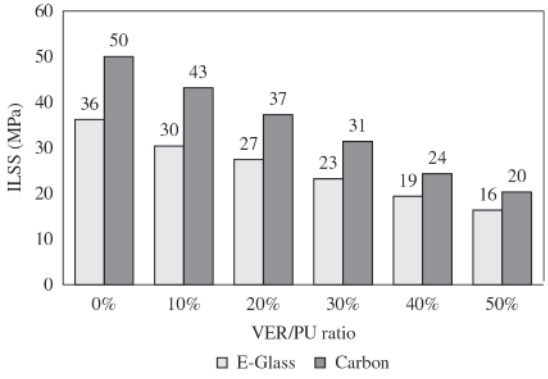

Figure 6. ILSS of Carbon/E-glass.

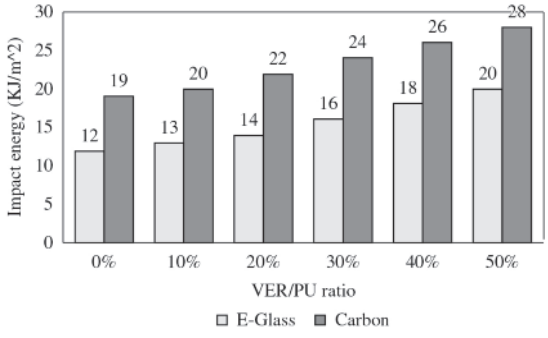

Figure 7. Impact Energy comparison of IPNs with reinforcement of Carbon/E-glass.

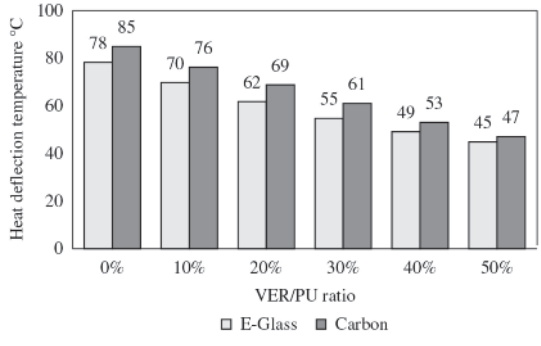

Figure 8. HDT strength of IPNs with reinforcement of Carbon/E-glass.

3.8 Morphological studies

After Impact test the fractured (images) specimens were analyzed by Scanning Electron Microscopy (SEM) to make our self thoroughly understand the interface behavior and enhancement mechanism. In the composite laminate, it was observed that there is a predominant mode of Shear Fracture (fiber matrix debonding) mechanism in all the specimens.

Figures 9a, b, c, d, e, f, and Figures 10a, b, c, d, e, f, shows SEM Images of 0%PU, 10PU%, 20PU%, 30PU%, 40PU% and 50PU% IPNs for both Carbon and E-Glass reinforcement. Bright phase resembles the VER and dark phase shows the PU, the dark phase of PU greatly contributes the better damping property by giving additional shearing action in the dispersed phase of IPNs[9,10].

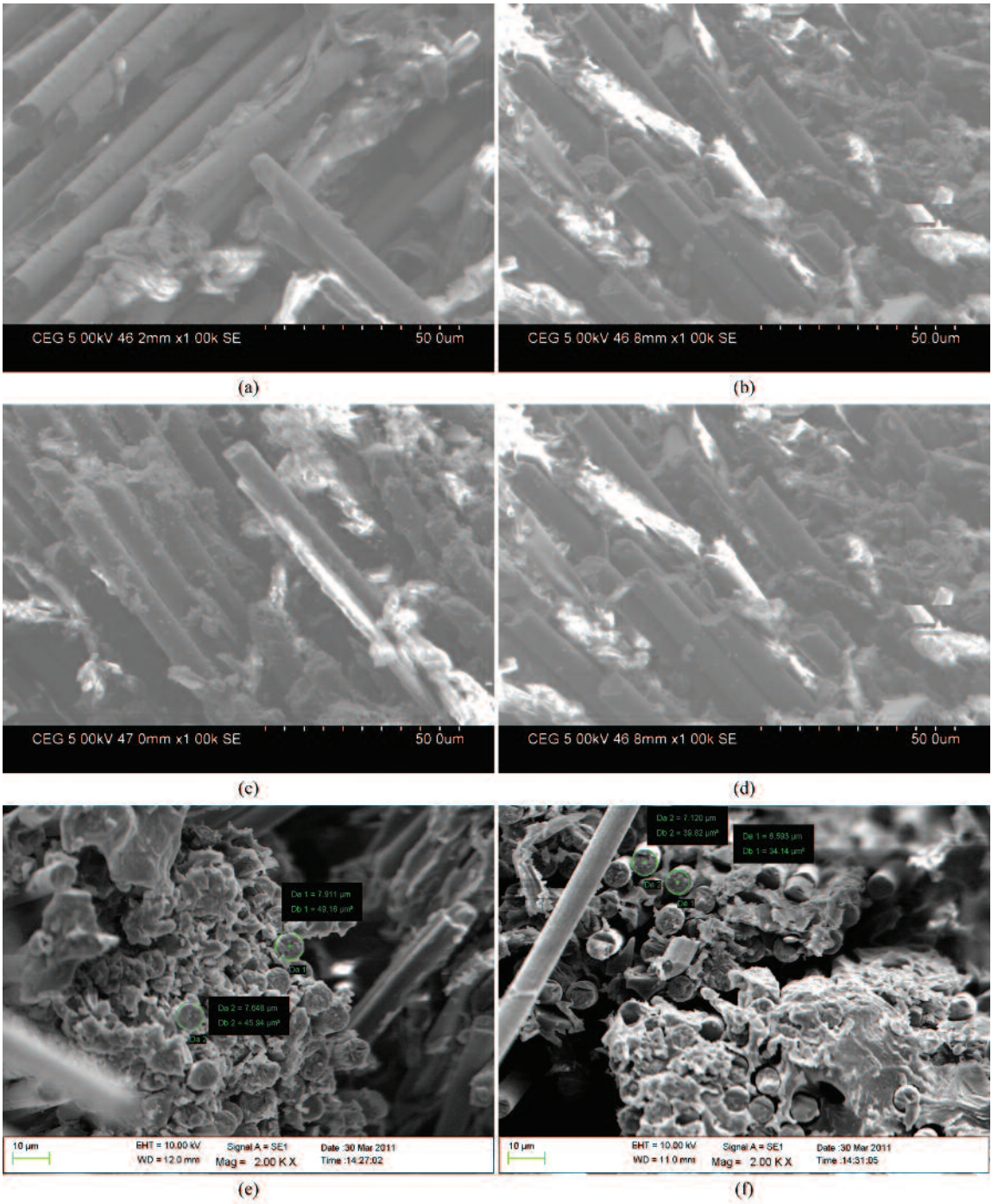

Figure 9. SEM Photographs of the (carbon) fractured surfaces for different compositions (a) 0% PU; (b) 10% PU; (c) 20% PU; (d) 30% PU; (e) 40%; (f) 50%.

Figure 9a shows the rupture in 0% PU resin composite, When percentage of PU varies from 0% to 10%, 20%, 30%, 40%, 50%, it shows in the form of different shade of color appearance in the SEM images, from bright phase to dark phase[15-16]. It is observed that in 9a, b, c, d, e and f, the dark phase increases, as much as the dark phase increases it proves that the evident of PU in the IPNs. Since PU has very good viscoelastic property it observes the maximum propagation of energy during the impact strength analysis[7].

As said in Carbon reinforced IPNs, E-glass reinforcement IPNs as well proves the same fracture mechanism like interfacial debonding in the fractured surface.

It is clearly observed that the addition of PU increases the damping properties of composite[7], the whole impact

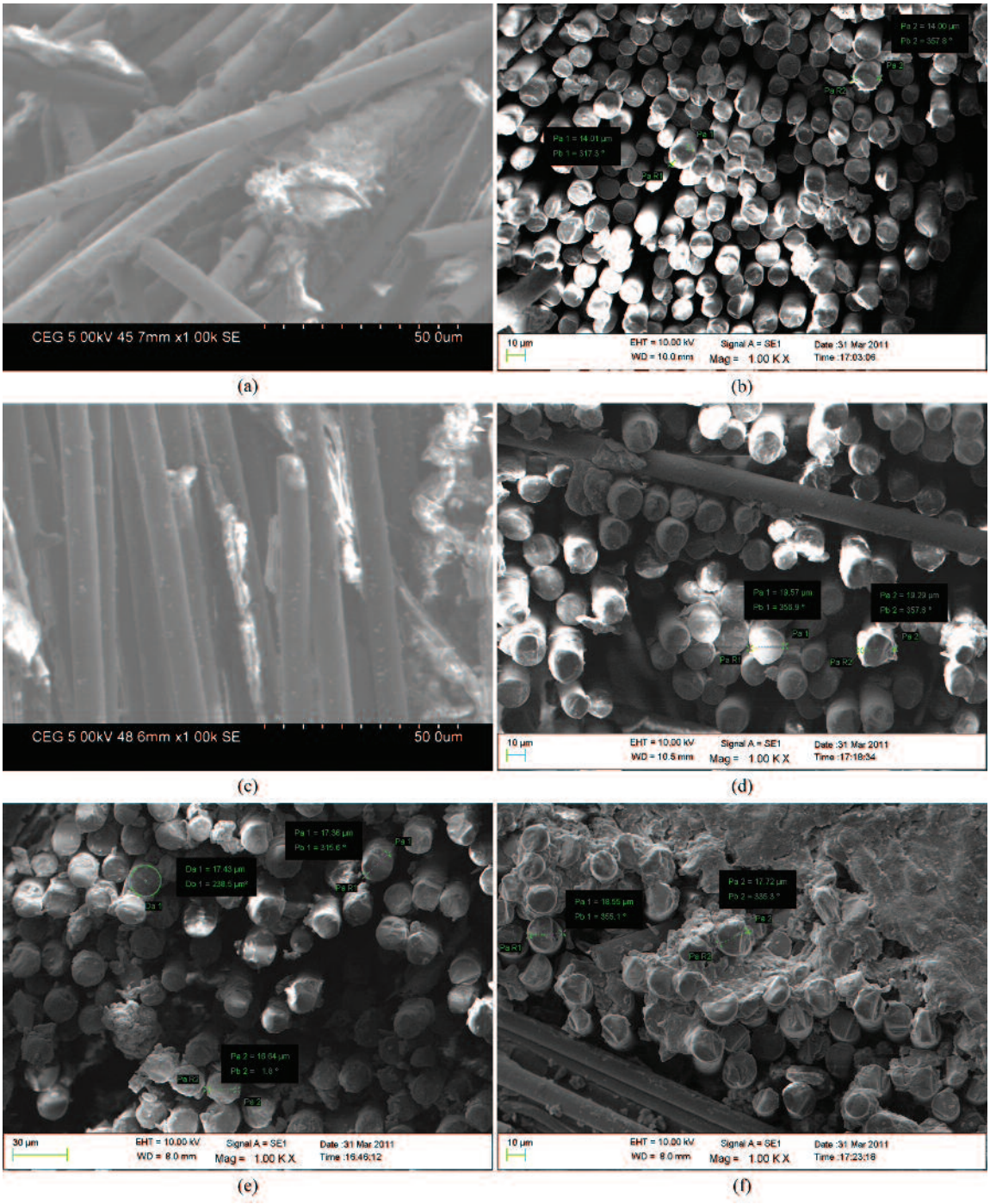

Figure 10. SEM Photographs of the (E-Glass) fractured surfaces for different compositions (a) 0% PU; (b) 10% PU; (c) 20% PU; (d) 30% PU; (e) 40%; (f) 50%.

strength of the composite can be improved by varying the proportionate of PU with VER. As the white phase increase in the images, (9b, c, d, e, f; 10b, c, d, e, f) the composite structure absorbs more amount of impact energy both in Carbon reinforcement and E-Glass reinforcement significantly. Though VER matrix gives the better impact energy transfer from fabric to matrix, the PU comprised IPNs exhibited far better results than the neat VER reinforced IPNs.

It is observed that E-Glass fibre reinforced IPN shows higher interfacial strength due to better wetting of E-Glass by the matrix resin. This may be explained due to the strong polar active sites present on the surface of glass fibre imparted by ionic sites of silicon and oxygen atoms and are responsible for the formation of strong intermolecular adhesion between E-Glass and matrix resin. This finding exhibited that the adhesion between the matrix and fiber, interfacial strength and impact energy transfer from fibre to matrix was largely improved.

4. Conclusion

The following conclusions were drawn from the study: Mechanical properties like Tensile, Flexural, Compressive stress and Modulus, ILSS and Thermo mechanical properties like HDT were found to decrease with increase in PU content, when compared to the neat VER – Glass fibre and VER – Carbon fibre composites. But the impact strength was found to increase tremendously with increase in PU content.

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