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Influence of various fluids on the interlaminar shear strength (ILSS) and impact behaviour of carbon/pei composites

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Composites can fail due to exposure to certain corrosive environments such as water, aqueous acids and bases, and organic solvents. The objective of this study was to determine the interlaminar shear strength (ILSS) and impact property changes on unidirectional carbon fibre reinforced polyetherimide (PEI) composites following exposure to various liquid environments at ambient and elevated temperature. The sample weights were increased for both of the samples with swelling effect. From the detailed investigation of the impact energies, it was observed that the crack initiation energy was decreased while the propagating energy was increased. Liquid environments with higher temperature affect material more remarkable compared to lower temperature. Also, remarkable decrease in ILSS values for all samples subjected to liquid environments was observed. By means of scanning electron microscope (SEM) studies the fracture mechanisms and morphology of the material after chemical corrosion was investigated. Remarkable morphological changes at the cross section of treated materials were observed. © *2006 Springer Science + Business Media, Inc.*

1. Introduction

A shortcoming of a number of polymer composite systems is their susceptibility to moisture degradation. Water can diffuse into composite either by planar diffusion or by anisotropic diffusion. In the later case, diffusivity of water in the fibre-direction of a graphite-epoxy composite was determined to be 10 times that of the value perpendicular to the fibres. This rapid diffusion along the fibre-matrix interface has been termed "water-wicking" and represents the main transport mechanism for conveying water to the interior of composites. Water-wicking in composites tends to occur preferentially along the interface, where wetting of the fibres is incomplete. Subsequent hydrolytic degradation of the matrix (e.g. SMC) can precipitate failure [1].

Composites can fail due to exposure to certain liquids such as water, aqueous acids and bases, and organic solvents [2–4]. Of particular interest is the influence of humid environment (moisture) on mechanical properties, particularly transverse static properties, and fracture behavior that is dominated by the matrix and interfacial properties. Depending on matrix phase chemistry, other fluids that are absorbed by the bulk matrix and by the interface phase can induce degradation. For most polymer composites, the absorption of moisture modifies the strength, stiffness, and delamination fracture behavior. Moisture induced property degradation manifests a strong correlation with the degree of sorbed moisture in the matrix. The rate of moisture sorption and solubility limits are governed by the matrix phase chemistry and by the fiber/matrix interface phase composition. Moisture related degradation is associated with chemisorption and reactivity characteristics of the permeating environment and matrix materials. Moreover, defects such as void, cracks, and other microstructural inhomogeneities associated with the fiber/matrix interface contribute to severity of moisture effects in resin matrix composites [1, 5, 6].

As more solvent diffuses in, the polymer begins to swell. Swelling of polymers is thus the result of organic

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liquids penetrating the amorphous regions of the polymer, leading to a reduction in the intermolecular attractive forces between polymer chains. Usually this action is a physical swelling effect, with no chemical reaction taking place and the molecular weight is preserved. In general, solvent pick-up in composites causes a reduction in matrix stiffness, which in turn leads to a decrease in compressive strength and shear strength [5, 7]. On the other hand organic fluids, such as gasoline, are more convenient environments which has a great potential of negatively affecting material microstructure and mechanical properties.

Plasticization of the bulk matrix and the interface phase by sorbed moisture influences the reduction in fracture toughness. Moisture induced plasticization causes softening of the resin and loss of strength. Moreover, moisture tends to weaken resin in vicinity of the fiber/matrix interface. The degrading effect of moisture in polymers composites is manifested by preferential cracking along the weakened fiber/matrix interface. Persistent fracture mechanisms appear to be interfacial cracking and debonding and microcrack growth and coalescence in the plasticized matrix material [1, 5].

Liquid environments affects all components of the composite, principally the matrix and the fibre matrix interface but also the fibre itself. The plasticization process involves interruption of the van der Waals' bonds between ethers, secondary amines and hydroxyl groups. Polymers with ketones and imides are more resistant to hydrolysis; they have fewer polar groups and this reduces their moisture sensitivity. Crosslink density may increase with increased concentration of secondary amino groups. This allows more freedom of motion and reduces the glass transition temperature. Plasticization reduces residual stresses and increases viscoelasticity. For many matrix materials, the effect of increasing moisture is very similar to an increase in temperature [1, 5].

The damage is believed to arise through environmentally induced stresses, which result from a moisture concentration gradient. This leads to a constraint on the outer surface which is trying to expand by swelling against the higher stiffness of the dry core and the generation of compressive surface stresses.

The effect of water and thermal strains on unidirectional glass-fibre reinforced laminates has been studied by jones *et al.* [8]. In addition, the performance propertie of cured polymer laminates have been found to be a significantly influenced by the presence of environmental moisture [9, 10]. Furthermore, moisture has been shown to have a detrimental effect on the impact behaviour of graphite-epoxy composites [11].

In various industrial applications PEI material has the potential to be exposed to many types of liquid environments. There has been growing interest in using fiber reinforced polymeric composites in offshore applications. The long-term durability of composites, however, must be characterized before their full potential can be realized in the aggresive environment of seawater, in which a composite structure is subjected to mechanical loading and moisture absorption. The deleterious effects of environmets, such as moisture, on static properties of composites have been well documented. However, research emphasizing the effects of moisture on fracture mechanisms and crack propagation behaviour is inadequate. However, persistent demands for use of composites in critical applications dictate that extensive characterization should be performed regarding external environmental effects on fracture aspects.

The objective of this study was to determine the interlaminar shear strength (ILSS) and impact property changes on unidirectional carbon fibre reinforced polyetherimide (PEI) composites following exposure to various corrosive environments at ambient and elevated temperature. Furthermore it is aimed to determine the relationship between ILSS and impact strength. Scanning electron microscope (SEM) investigations were done to understand the fracture morphologies after subjecting to chemical corrosives.

2. Experimental

Unidirectional carbon fibre reinforced polyetherimide (PEI) composites were kindly supplied by Ten Cate Advanced Composites (Nijverdal/Netherlands) in the form of hot pressed plaques. PAN based carbon fibre was manufactured by Amoco used in composite plaques (T300 12 K 309 NT type). The fibre volume content was 60%. Plaques were manufactured from 14 ply with a per ply thickness of 0.14 mm and the arial weight per ply is 222 g/m^2. The commercial code of the laminate is CD5150. Mechanical properties of the composite laminate and resin properties are given in Tables I and II. Also Fig. 1 represents the chemical structure of PEI.

The materials were subjected to four different liquid environments at two different temperatures. The liquid environments were saturated NaCl, 0.6 molar NaCl, triple distilled water (TDW) and gasoline. Samples were subjected to physico-chemical treatment in different environments at 20°C and 90°C. The average variation in the temperature during the experiments is ±0.5 both at 20°C and 90°C. Temperature of the environments was kept in controlled oven atmosphere samples were completely immersed into the liquid environments. In laboratory atmosphere (20°C, moderate humidity), samples were weighted by Scaltec SBC 31 balance with 0.0001 g accuracy before and after the testing period. Average weight of 10 samples was taken into account. Weight measurements were done daily and instrumented impact tests were performed on both as received (original) and 90 days treated samples.

Instrumented impact test is one of the important mechanical characterization techniques, which gives detailed information about the impact properties of the materials [2]. It is possible to monitoring both elastic and plastic energy absorption as a function of time or displacement. This is very important for composite materials, because it is possible to observe the energy absorption of delamination

TABLE I Mechanical properties of composite laminate

	−55°C	23°C	80°C	80°C*	Units	Method
Tensile strength (0°)	1583	1890	1728	1605	MPa	ASTM D3039
Tensile modulus (0°)	131	128	127	127	GPa	ASTM D3039
Tensile strength (90°)				26	MPa	ASTM D3039
Compressive strength (0°)	936	876	814	689	MPa	ASTM D3410
Compression modulus (0°)	120	119	120	123	GPa	ASTM D3410
In plane shear strength	121	104	94	89	MPa	ASTM D3518
In plane shear modulus	4395	3208	2744	2558	MPa	ASTM D3518
Flexure strength		1289	1072	1118	MPa	ASTM D790
Flexural modulus		99	100	97	GPa	ASTM D790

*pre-conditioned at 70°C/85% RH (1000 h).

TABLE II Resin properties

HDT 1.82 Mpa	196°C
Density (solid)	1.27 mg/m^3
MFR at 377°C, 6.7kg	9.0 g/10 min

during the impact. There is no possibility to investigate the liquid environments effects on the characterization of fracture in each step with conventional impact tests [12, 13]. On the other hand short beam shear test is one of the important tests for the investigation of interlaminar shear strength (ILSS) of the composites.

Impact tests were performed on an instrumented Ceast pendulum type tester (Resil 25) and test samples were prepared according to ISO 180 standards. Un-notched samples were used with the dimensions of 10 × 2 × 65 mm. Preliminary experiments were performed in order to find the appropriate falling angle, which was chosen as 50° in order to remove the inertial oscillations in the contact load between striker and sample. The strike range of the hammer, at 50° falling angle (izod hammer), was 1.08 kN. Hammer length and mass were 0.327 m and 2.508 kg respectively. Sampling time was 8 μs. Impact velocity was 1.51 m/s, and maximum available energy was 2.87 J. Before discussing these results it is important to understand the approach used in the analysis of force-time curves, which is critical in determining the impact characteristics of materials. Upon impact of the pendulum, the force rises sharply to a maximum value ($F_{\max}$) and then gradually decays to zero due to catastrophic failure. Total area under the force-time curve gives the impact energy for the system ($E_{\max}$). These curves can be divided in two regions. The first region is the crack initiation and the second is the crack propagation regions. The areas under each region give the energy for these processes, which are defined as energy for crack initiation (E_{i}) and energy for crack propagation (E_{p}). The spikes in the first region are due to inertial oscillations of the sample.

Fractured surfaces of the impact test samples were examined by scanning electron microscopy, (SEM), using a JOEL JSM-6335F field emission scanning microscope.

3. Results and discussion

The results of weight measurements are seen in Table III. Physically acting media can only affect the secondary valance forces. In these cases, the molecules of the media diffuse into the macromolecules and increase the distance between the polymer molecules. So, the sample weights were increased for all samples. Also at higher temperatures, as a result of increase in molecular mobility, there were 3 times higher increases in samples observed at 90°C compared to samples which were at 20°C [5]. Due to this effect the polymer material swells and softens. Media molecules wedged between the macromolecules have additional effects, which change the mechanical behaviour of the polymer. The intermolecular forces are lowered if media molecules diffuse in and the distance between the macromolecules increases, leading to a softened plastic [7].

The numerical results of instrumented impact tests of the samples are seen in Table IV. It is clearly seen from Fig. 2 that the immersed samples in saturated NaCl and gasoline environment has slightly lower $F_{\max}$ values compared to original samples. On the other hand, it is observed higher $F_{\max}$ at the samples which were immersed in 0.6 molar NaCl and TDW environments. Decreasing in $F_{\max}$ values were 2.40% and 1.92% in saturated NaCl and gasoline environments, respectively. On the other hand, increasing rates were 8.65% and 5.28% in 0.6 molar NaCl and TDW environments, respectively.

Fig. 3 shows that $F_{\max}$ values were dramatically decreased for all of the samples subjected to liquid environ-

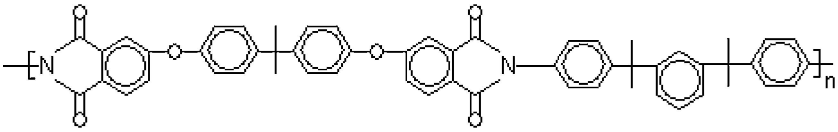


Figure 1 Chemical structure of Polyetherimide.

TABLE III The weight measurement results

	Temperature of the environments					
	20°C			90°C		
Liquid Environment	Initial weight (g)	After 90 days (g)	Rate of Increase (%)	Initial weight (g)	After 90 days (g)	Rate of Increase (%)
Saturated NaCl	14.9643	15.0433	0.52	14.3494	14.5955	1.71
0.6 molar NaCl	14.5770	14.6402	0.43	14.2918	14.4915	1.39
Triple distilled water	14.7571	14.8562	0.67	14.2532	14.4760	1.56
Gasoline	14.2694	14.3417	0.50	–	–	–

TABLE IV The numerical results of instrumented impact tests of the samples

	Temperature of the environments					
	20°C			90°C		
Liquid Environment	F_{max} (N)	E_{max} (J)	E_i (J)	F_{max} (N)	E_{max} (J)	E_i (J)
Original Sample	208	1.141	0.492	–	–	–
Saturated NaCl	203	1.131	0.445	166	1.152	0.251
0.6 molar NaCl	226	1.042	0.404	130	1.136	0.168
Triple distilled water	219	1.029	0.444	161	1.131	0.237
Gasoline	204	1.101	0.364	–	–	–

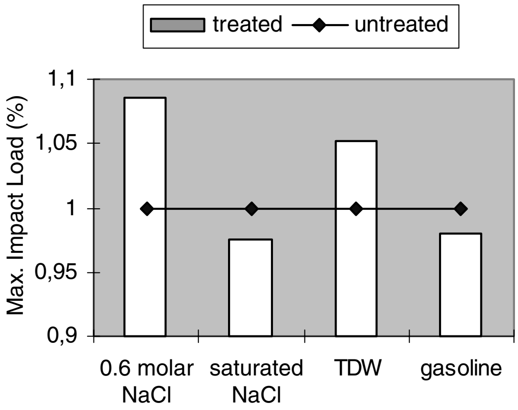


Figure 2 The variation of F_{max} for treated samples at 20°C.

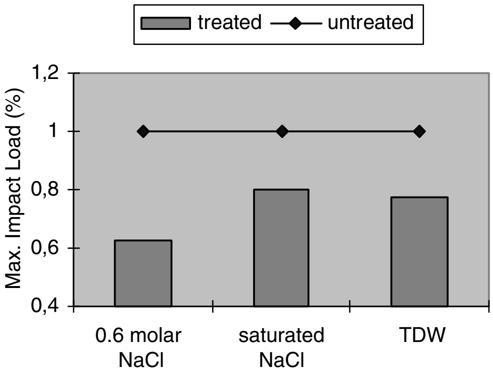


Figure 3 The variation of F_{max} for treated samples at 90°C.

ments at 90°C. There is a higher ability about the water diffusion at high temperature of 90°C happens by conveying the corrosive liquids along the fibre-matrix interface to the interior of the composite which leads to weaken the interface. In addition to this, the presence of liquids leading to increase the distance between the macromolecules, reduce the intermolecular attractive forces between the polymer chains and softens the matrix. It is seen from Fig. 4 that the crack initiation was formed at remarkably lower forces at higher environment temperature of 90°C. There were 20.19%, 37.50% and 22.59% decreases observed in NaCl, 0.6 molar NaCl and TDW, respectively. The most remarkable decrease was observed in 0.6 molar NaCl environments at 90°C after 90 days.

All of the E_{max} values (except saturated NaCl environment at 90°C) were decreased compared to original material. Most remarkable decreases were observed in 0.6 molar NaCl and TDW environments in E_{max} values as 9.8% and 8.6%, respectively. Decreases in E_{max} values of the other environments were approximately the same as seen from Fig. 5.

As provided in Fig. 4 Force-time curves of the corroded samples at 20°C shows a gradual increase until F_{max}, followed by a sudden small drop in load indicating of matrix cracking and debonding. After this point, the crack propagates rapidly in the structure and after a sharp drop samples were fractured with fibre fracture and pull-out. On the other hand, F-t curves of the corroded samples at 90°C shows remarkably different fracture behaviour. Cascade or stepwise fracture behaviour was seen for these samples. After reaching F_{max}, cracks were arrested at many points. This preventing the structure from the sudden failure, and it takes longer fracture times with higher energy absorption.

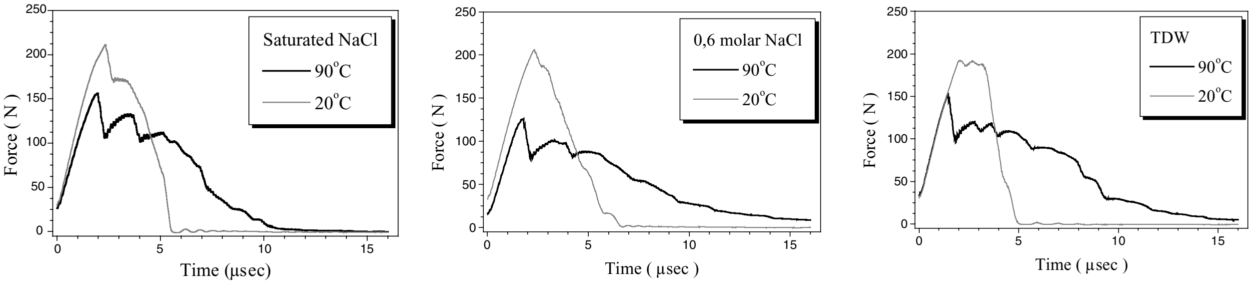


Figure 4 F-t curves indicating the decrease of F_{max} for samples treated at 90°C.

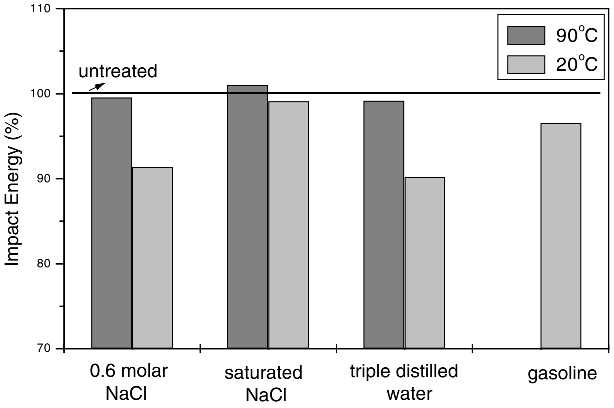


Figure 5 Comparison of the impact energies (E_{max}).

In addition to Fig. 4 it is possible to get differences in E_{max} values of the samples are seen in Fig. 5. E_{max} values were generally smaller compared to original samples. Also E_{max} values of fractured samples, which were immersed at 90°C environments, are higher compared to 20°C.

Fig. 6 shows the E_i and E_p values of the samples. Higher molecular diffusion rates at higher temperature (90°C) results in decrease in crack initiation energy as a result of fibre-matrix interfacial bonding, delamination strength. Finally, higher frictional loss between fibres, matrix and laminates gives higher crack propagating energy.

Decreasing in E_i values should be evaluated at the first step during the machine part design. After high temperature teratment (90°C), materials are fractured with lower F_{max} values and with lower E_i energies. High temperature environment makes the samples quite brittle with remarkable lower elastic shock absorbing ability.

Matrix swelling that occurs on absorption of moisture is analogus to the thermal expansion that occurs on heating. As a consequence, an analogus equationan be used to asses the swelling coefficients of the individual plies and to predict changes in the residual stress state during environmental conditions. The results of interlaminar shear strength (ILSS) are illustrated in Fig. 7. As seen in Fig. 7 there was a remarkable decrease in ILSS of all samples which were subjected to corrosive environments. Interlaminar shear strength was measured for original material as 85, 28 MPa. The most dramatical decrease in ILSS were observed in immersed samples in NaCl environments after 90 days. ILSS of this samples was measured as 63, 18 MPa which has a decrease of 39.2% compared to as received material.

On the other hand, lower ILSS values were observed at samples, which were treated at higher temperatures. It was observed that as similar to impact properties the

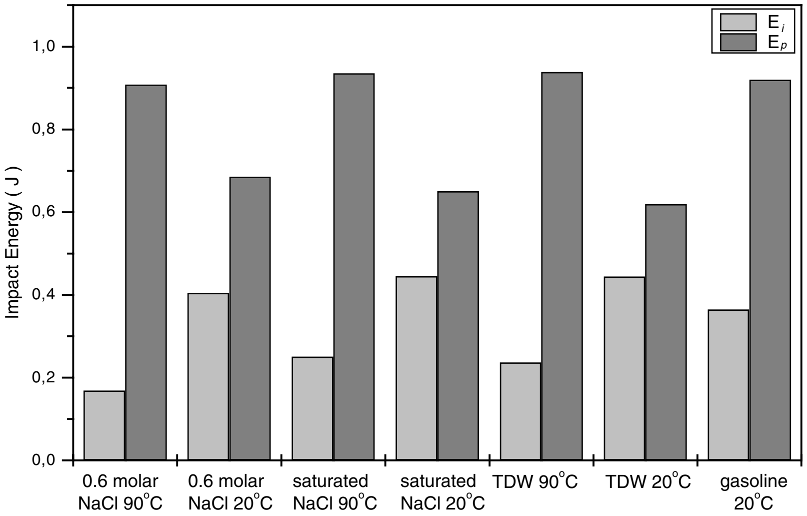


Figure 6 Analysis of the impact energy results.

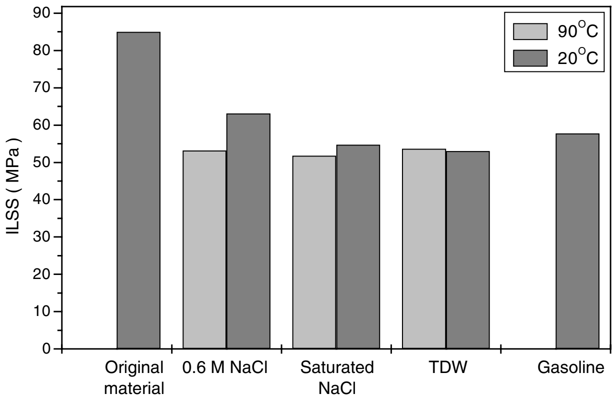


Figure 7 ILSS results of the samples.

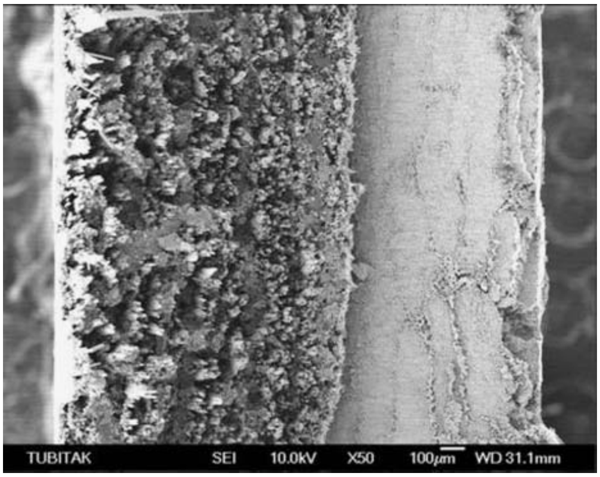


Figure 8 Fractured cross section of untreated sample.

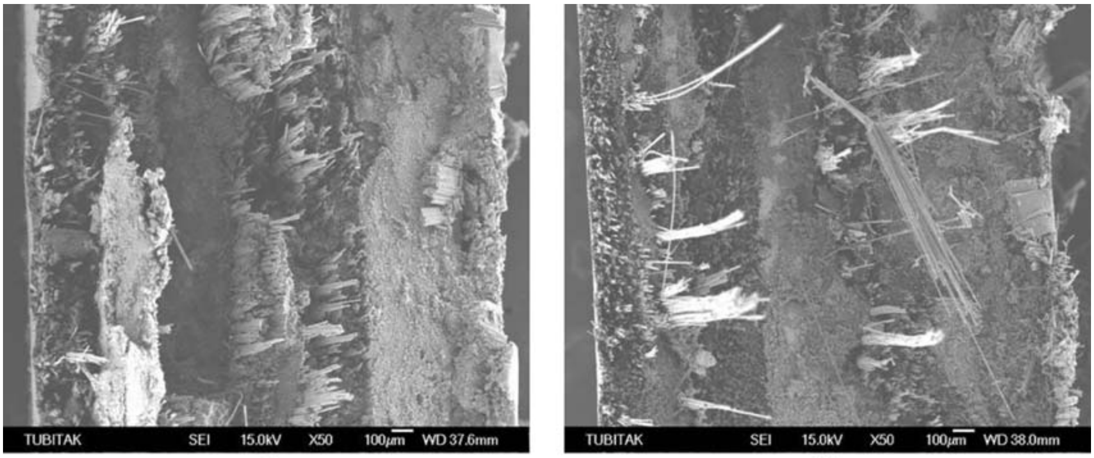
temperature of environment closely affect the ILLS properties.

It is known that many fibres affected from moisture morphologically. Especially there is many studies about mositure effects on mechanical properties of glass and kevlar fibres. On the other hand carbon fibres known that doesn't affected from moisture negatively. This knowledge claims the matrix and matrix-fiber interface about reduction in mechanical properties. The liquid environments penetrated to the interior of the composite by both along the fibre-matrix interface leads to weaken the interface and the matrix structure leading to increase the distance between the macromolecules, reduce the intermolecular attractive forces between the polymer chains and softens the matrix. This remarkably reduces the impact and interlaminar properties of the samples [1, 6].

Fractographic investigations were done by SEM studies. Fig. 8 illustrates the cross sections of fractured original sample. It is possible to observe the neutral axis, tensile zone and compression zones clearly. Neutral axis divides the fractured cross sections into two parts. The tensile zone is placed on the left hand side while the compression zone is placed on the right hand side. The original sample with a strong interface tends to show brittle-like planar fracture surfaces the brittle fracture morphology is clearly observed in Fig. 8 [14].

Fig. 9 illustrates the fractured cross sections of immersed samples in 0.6 molar NaCl environments. There was a remarkable morphological difference as compared to original sample (Fig. 8). There was not a evident neutral axis formation as observed in Fig. 8. Delamination between the plies (or laminates) was occur in the material. Each laminate was fractured individually with mixture of tensile-compression zones placed around the cross sections. As a result of liquid environment weaker interfaces were occur, which exhibit brush-like failures with protruding fibres. It is also possible to observe longer debonded, pulled out fibre bundles at the cross sections, which indicate the poor interfacial bonding and weakened zones between the fibre/matrix and interlaminar interfaces. Very long pulled-out fibres were observed at the samples subjected to 0.6 molar NaCl environments at 90°C. Also rougher fractured surface with high delamination, interlaminar and translaminar crack formations were observed.

Fig. 10 shows the boundary layer of the fractured untreated material surface of the sample at the tensile zone



(a) (b)

Figure 9 The micrographs show the pulled-out fibres for samples waited in 0.6 molar NaCl environments at (a) 20°C and (b) 90°C.

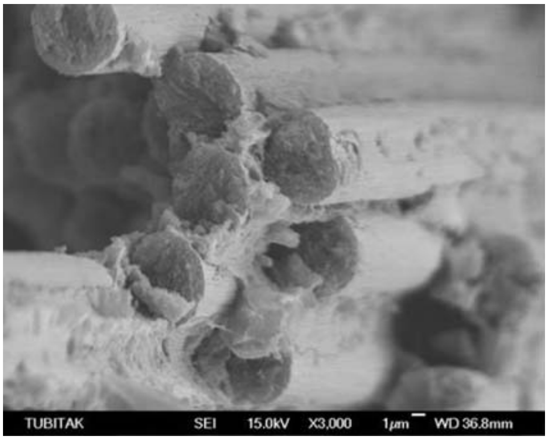


Figure 10 The boundary layer of the fractured untreated material surface at the tensile zone.

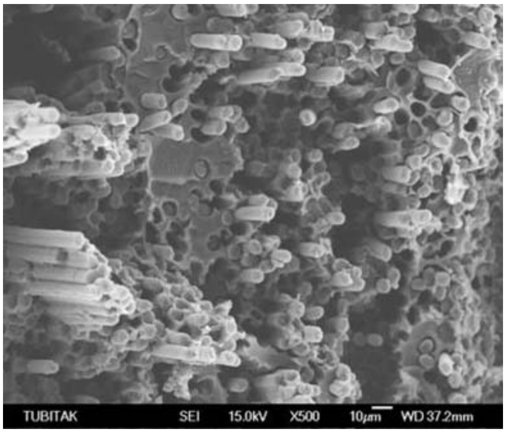


Figure 11 Micrograph of the fracture surface of immersed samples in 0.6 molar NaCl environment at 20°C after 90 days.

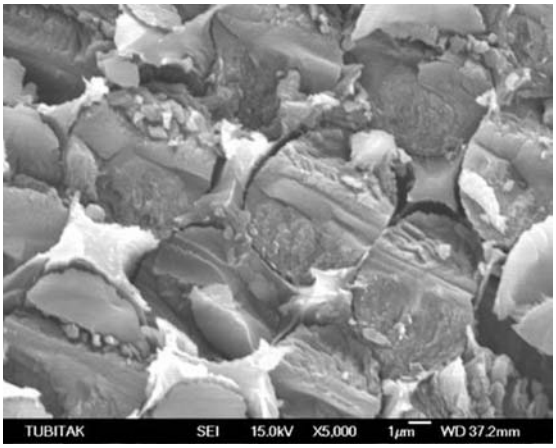


Figure 12 Micrograph of the fracture surface of original sample.

[14]. The fracture of a fibre bundle in the composite specimen with PEI matrix was observed. The fibres seem to be bonded very strongly to the matrix. The high interfacial strength between the fibre and matrix leads eventually to extensive transverse fibre fracture on the back face of impact rather than pulling-out of fibres, matrix cracking, etc. Fibres fractured in a brittle manner without any indication of yield or flow. As a result of strong fibre-matrix interfacial bond strength, the pull-out lengths show a very short stub of carbon fibre.

There are three basic failure modes in longitudinal compression. (1) Microbuckling of the fibers within the matrix in extensional or shear mode. (2) Shear failure of fibers, and (3) transverse tensile failure due to poisson's effect. This failure mode generally applies at low values of the fiber volume ratio. As in our material, shear fiber buckling produces tensile and compressive stresses in the fiber that create kink zones [1, 6].

There was a brittle fracture wrinkled pattern on the fracture surface of all fibres in the compression zone of untreated material (Fig. 12) [14]. In addition to shear fractured fibres, small fibre fragments as a result of micro buckling and the fracture of resin can be observed in Fig. 12. Fracture of the polyetherimide matrix was formed in irregular patterns including the matrix fragments of 0.5–5 μm sizes. The extensive fragmentation of the matrix can be attributed to compression and shear fracture from the impact loading and can be contrasted with the matrix fracture occurring under quasi-static loading conditions. Cracking through the fibres is also observed. Fig. 12 also shows the fibres at compression zone with shear bands on the surfaces also indicate the crack propagation direction.

As a result of immersion in 0.6 molar NaCl environment at 20°C after 90 days, there was a remarkable deformation in the fibre/matrix interface. It is possible to see many pulled long fibres, which indicate poor adhesion between the fibre/matrix interfaces as shown in Fig. 11. The length of pulled-out fibre is quite longer than fractured untreated material.

Due to transverse tensile failure due to poisson's effect, the first microcracks originating at the fiber-matrix interface are nearly normal to the interface, which indicates that failure is caused by the circumferential tensile stress in the matrix. For closely packed fibers, radial cracks occur at approximately 90° from the loading axis. As the load increases, interface cracks are formed in an area along the loading axis. These interface cracks are not immediately connected to the radial cracks developed earlier. In the last phase of damage development radial and interfacial cracks are connected to form a long continuous crack. Briefly the crack formation in compression zone of the composite can be summarized as [1, 6];

- Damage development in a transversely loaded composites.
- Initial radial cracks around closely packed fibres.
- Radial cracks around isolated fibers.
- Interfacial cracks.
- Interconnection of radial and interfacial cracks.

Fig. 13 illustrates the compression zone of materials, which was corroded in 0.6 molar NaCl environment at 20°C. Weakening at the interface resulted in insufficient

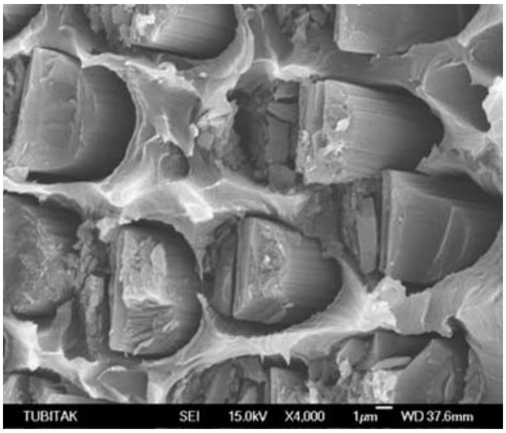


Figure 13 The weakness at fibre and matrix interfaces of immersed samples in 0.6 molar NaCl environment at 20°C after 90 days.

support of fibres by the matrix. Increased debonded fibre length caused more available fibres to be fractured as a result of crack formation as explained above. Also weakening after immersion in liquid environments results in remarkable plastic deformations and debonding around the fibres after mechanical loading.

4. Conclusions

Based on the nature of the composite material and the manufacturing process of the composite components, it is clear that their weak point is the poor interlaminar and intralaminar strength, which is mainly controlled by the properties of the matrix material. On the other hand, design with composites very often leads to design details that produce stress concentration sites and promote high interlaminar stresses known as edge effects. Then the ability of the material to withstand the formation and propagation of delamination is again a toughness-controlled parameter. The behavior of the composite laminate is mainly controlled by the matrix fracture properties and the fiber-matrix interface. In contrast, translaminar fracture type, where again the fiber-matrix interface plays an important role. In terms of fracture morphology, weak interfaces show extended fiber pull-out, giving an extremely irregular fracture surface. This is due to a physical correlation between the debonding and fiber bridging process. The degree of irregularity of the fracture serfaces may be used as a quality control factor. When the fibres are well bonded to the matrix (strong interface), debonding may not occur as the crack approaches the interface, but instead fiber failure in a brittle manner is expected.

The effects of various liquid environments and their temperatures on interlaminar shear strength (ILSS) and impact behaviour of carbon/PEI composite were investigated. The sample weights were increased for both of the samples with swelling effect. The ability of water diffusion into the interior of the composite was higher at higher temperatures.

Regarding hydrolysis, benzyl ethers are stable to acidic and basic media, and to oxidizing reagent. Imides are generally hydrolized in an alkali media, so we can say that hydrolising is not effective in our system [15]. The liquid environments penetrated to the interior of the composite by both along the fibre-matrix interface leads to weaken the interface and the matrix structure leading to increase the distance between the macromolecules, reduce the intermolecular attractive forces between the polymer chains and softens the matrix. This remarkably reduces the impact and interlaminar properties of the samples. The instrumented impact test results indicate that the maximum decreases in load happens at higher environment temperatures. But, the impact energy remains approximately the same except the samples subjected to TDW and 0.6 molar NaCl at 20°C. From the detailed investigation of the impact energy, it was seen that the crack initiation energy was decreased while the propagating energy was improved with increasing liquid temperature. Also, there was a remarkable decrease in ILSS results for all samples subjected to liuid environments. The micrographs were exposed the effect of environments leading to poor fibre-matrix interface.

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