Effects of fiber surface treatments on mechanical properties of epoxy composites reinforced with glass fabric

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Abstract In this study, effects of fiber surface treatments on mechanical behavior and fracture mechanism of glass fiber/epoxy composites were investigated experimentally. To change the composition of the glass and regenerate to the hydroxyl groups, activation pretreatment of heat cleaned woven glass fabric was performed using (v/v) HCl aqueous solution at different concentrations before silane treatment. The treatment of silanization of heat cleaned and acid activated glass fibers with γ-glycidoxypropyltrimethoxysilane were performed. In this work, short beam shear test has been conducted to determine the performance of the acid treatment and the silane treatment in terms of the interlaminar shear strength. The silane coating on the heat cleaned glass fibers increased the interlaminar shear strength of the composite. However, the silane coating on the acid activated glass fibers did not improve the interlaminar shear strength of the composite. In addition, the strengths of the glass fabric specimens in tension and flexure were investigated. When the glass fibers are first treated with HCl solution and then with silane coupling agent, the tensile strengths of the composites decreased significantly. Scanning electron photomicrographs of fractured surfaces of composites were performed to explain the failure mechanisms in the composite laminates broken in tension.

Introduction

Composite materials are composed of two or more components that differ in physical and chemical properties to provide desirable characteristics [1]. Mechanical characteristics of fiber reinforced polymer composites depend primarily on the mechanical properties of matrix and fiber materials, the surface of the fiber, and the nature of the fiber–resin bonding. These two last points are related to the surface properties of the fibers [2]. When using surface modification techniques such as silane treatments or plasma techniques (plasma treatment, plasma polymerization), it is well-known that compatibility between inorganic fillers and polymer matrices improves. Polycondensed thin films were prepared using silane coupling agents and plasma polymers by wet-chemical processes and plasma polymerization technique, respectively. Thus, it is possible to improve the adhesion between fiber and matrix [3–6]. However, plasma surface treatment and plasma polymerization as an alternative coating techniques have been mainly used for surface modification of fibers [5, 6]. In industry, silane coupling agents by wet-chemical process are applied for surface modification of glass reinforcements (fibers, particles) in order to form a functional interlayer. The silane molecule is a multifunctional one, which reacts at one end with the glass surface and at the other with the polymer matrix. [1, 3, 7–11].

Organosilanes have the general structure, $X_3Si–R$. R is a group which can react with the resin, and X is a group which can hydrolyze to form a silanol group in aqueous

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solution and, thus, react with a hydroxyl group of the glass surface. The trihydroxy silanols, $Si(OH)_3$, are able to compete with water at the glass surface by hydrogen bonding with the hydroxyl groups at the surface. When the treated fibers are dried, a reversible condensation takes place between the silanol and OH groups on the glass fiber surface, forming a polysiloxane layer which is bonded to the glass surface. Therefore, once the silane coated glass fibers are in contact with uncured resins, the R-groups on the fiber surface react with the functional groups present in the polymer resin, such as methacrylate, amine, epoxy and styrene groups, forming a stable covalent bond with the polymer [12]. This bond not only improves mechanical strength, but also the resistance to extreme environmental conditions, such as prolonged moisture exposure and thermal cycling [13].

With regard to silane treated glass fiber reinforced polymer composites, many studies have been performed to understand the relationships between the interfacial structure and the properties of the fiber/matrix composites [1, 7–9]. Park and Jin [9] examined the surface treatment of glass fibers with different concentrations to improve the interfacial adhesion at interfaces between fibers and matrix. They used the methacryloxypropyltrimethoxysilane (90%) containing aminopropyltriethoxysilane (10%) for the surface treatment of glass fibers. From the experimental results, the presence of coupling agent does lead to an increase of ILSS (interlaminar shear strength) of the composites, which can be related to the effect of increasing the degree of adhesion at interfaces among the three elements, i.e., fiber, matrix, and silane coupling agent. On the basis of experimental results, it was also reported that the mechanical interfacial properties of the composites decrease due to excess silane layer physisorbed onto the glass fiber at a given higher silane coupling agent concentration. Park and Jang [14] investigated the effect of the surface treatment of the glass fiber on the mechanical properties of glass fiber/vinyl ester composites. It is important to point out that the values of the flexural strength and the ILSS of methacryloxypropyltrimethoxysilane (MPS) treated glass fiber/vinyl ester composites increase up to 0.3% silane concentration and then decrease smoothly after the maximum point. Their results indicate that physisorbed MPS layers are formed on the chemisorbed layer by an excess amount above 0.3% concentration. This layer acts as a lubricant or deformable layer. Briefly, the interfacial adhesion and, therefore, the mechanical properties of the composites are mainly controlled by silane coupling agents.

However, the influence of pretreatment with dilute HCl acid solution of the glass fibers to regenerate the hydroxyl groups on the glass fiber surface prior to the silanization on the mechanical properties of the composites was not evaluated. According to González-Benito et al. [15], the acid activation of glass fibers greatly changes the surface composition and the hydration state of the glass fiber. Under acidic conditions, a great number of silanol groups are generated: although a substantial number of these silanols are of internal character, greater coating degrees can be achieved. There are only very few articles dealing with pretreatment of glass fiber prior to silanization. Olmos et al. [16] studied the effect of the nature of glass fiber surface in the water absorption of glass fibers/epoxy composites. Hydrochloric acid (HCl, 37 wt%) was used for the glass fibers surface activation before different silane coatings. The results obtained show that the presence of silanized fibers seems to induce changes in the process of water absorption of the epoxy resin, decreasing the relative gain of mass at equilibrium. González-Benito [17] investigated the curing process of an epoxy system at the interface formed with a silane coated glass fiber by using FTIR imaging. In the study, glass fibers were activated (hydroxyl regeneration) in a 10% (w/w) HCl aqueous solution for 1 h and silanized with a 1% (v/v) aqueous solution of 3-aminopropyltriethoxysilane (APTES). In the other work performed by González-Benito et al. [15], glass fiber has been treated by two different activation methods, reflux with neutral water and reflux with 10% HCl aqueous solution. The influence of different activation pretreatments of glass fibers on the structure of an aminosilane coupling agent layer was investigated. They concluded that acid treatments hydrolyze Si–O bonds, greatly changing the composition of the glass and regenerating silanol groups, some of them being of intraglobular nature.

In this study, effects of different fiber surface treatments (acid activation and silane treatment) on mechanical behavior and fracture mechanism of glass fiber/epoxy composites were investigated experimentally. To regenerate the hydroxyl groups of glass fibers, activation pretreatment of heat cleaned woven glass fabric was performed using (v/v) HCl aqueous solution at different concentrations. The treatment of silanization of heat cleaned and acid activated glass fibers with γ-GPS (γ-glycidoxypropyltrimethoxysilane) were performed. Mechanical properties of the composites have been investigated by tensile tests, short beam tests, and flexural tests. The short beam method was used to measure the interlaminar shear strength (ILSS) of laminates. The tensile and flexural properties of composites were characterized by tensile and three-point bending test, respectively. The fracture surfaces of the composites were observed with a scanning electron microscope (SEM).

Experimental details

Materials

Areal density of woven roving glass fabric, supplied by Metyx Telateks A.S. of Turkey was 300 g/m^2. The silane coupling agent was γ-GPS (γ-glycidoxypropyltrimethoxysilane) supplied by Dow Corning Corporation under the commercial name of Z-6040. The silane was 99.8% and was used as-received. Hydrochloric acid (HCl 37%) and acetic acid (CH$_3$COOH 100%) were of analytical grade purchased from Riedel-de Haën and also used without further purification.

The epoxy system Resoltech R1040 (unmodified liquid epoxy) and hardener Resoltech R1048 (hardener), both manufactured by Resoltech, France were chosen as the matrix. The composition for the epoxy resin system is specified in the product data sheet from the manufacturer to be (by weight): R1040 (78%) and R1048 (22%).

Surface treatments of glass fibers

The E-glass fibers were used in two forms for silanization: heat cleaned and acid activated glass fibers. As-received glass fibers were heat cleaned at 450 °C for 1.5 h to remove any organic substances, such as sizing or impurities, from the surface. Acid activation procedure can be summarized as follows: heat cleaned fibers were subjected to activation pretreatments with a hydrochloric acid aqueous solution (HCl 1% (v/v) and HCl 3% (v/v), separately) for 1 h at room temperature to regenerate the hydroxyl groups. After acid activation, all the samples were washed with distilled water several times until chloride-free, determined by an AgNO$_3$ test. Then, all the samples were dried at 110 °C for 1 h.

The silane treated fibers were obtained by treating the heat cleaned fibers and the acid activated fibers with γ-GPS by the following procedure: aqueous solutions were prepared by adjusting the pH of the distilled water to about 4.5 with acetic acid. The silane coupling agent was added to acidified water. The mixture was stirred for about 15 min before the silanes were hydrolyzed by dilute acetic acid solution. The concentration of silane aqueous solutions was 0.3% (v/v). It can be noted here that the used concentration in this study covers the range usually employed in the practical application of glass fiber reinforced plastics technology. After 15 min of the hydrolysis reaction of γ-GPS agent, the glass fabrics were immersed into the silane aqueous solution for 1 h. Then γ-GPS treated glass fabrics were dried for 30 min at 105 °C to drive the condensation of silanol groups at the surface and to remove traces of methanol from hydrolysis of the methoxysilane.

Composites preparation

The epoxy resin and hardener mixture were applied onto the as-received and the surface treated glass fabrics by a hand lay-up technique. Twelve layers were added successively in order to get about 3.5-mm-thick composite. The laminate was compressed thereafter, in a mold (25 × 35 cm^2) at a pressure of 100 bar, and the pressure was applied to the composite at room temperature for 150 min. After fabrication, the glass composites were cured at room temperature for 2 weeks before being tested.

Tensile strength testing

According to ASTM standard D-3039, tensile tests on composite sheets were performed in a Shimadzu AUTO-GRAPH AG-G Series universal testing machine with a video extensometer system (SHIMADZU Noncontact Video Extensometer DVE-101/201), with trapezium (advanced software for materials testing) for machine control and data acquisition. The specimens with length of 197 mm and width of 25 mm were prepared using a water jet cutter. Tensile tests were conducted at a constant crosshead speed of 2 mm/min at room temperature in air. At least six specimens were tested for each type of composite sheet to check for repeatability.

Flexure test

The flexural strength and modulus of the glass fiber/epoxy composites were measured using a three-point bending test according to ASTM D 790. The three-point bend fixture was manufactured by Shimadzu for use in a universal test machine running in compression mode. The tests were carried out with a span-to-depth ratio of 16:1 at a crosshead speed of 1.3 mm/min. Test specimens with length of 80 mm and width of 25 mm were prepared using a water jet cutter. At least six samples were measured and the results were averaged. The flexural strength (σ_f) and modulus (E_f) were obtained with the following equations:

$$\sigma_f = \frac{3PL}{2wt^2} \tag{1}$$

where σ_f denotes the flexural strength, P is the applied load at the moment of fracture, L is the span length, w is the width of the test specimen, and t is the thickness of the test specimen. For the case in which the three-point flexural specimen is not strain-gauged, the flexural modulus (E_f) can be determined from the slope of the initial straight-line part of the load-deflection curve by means of this equation:

$$E_f = \frac{L^3}{4wt^3}\frac{\Delta P}{\Delta \delta} \tag{2}$$

where $\Delta P/\Delta \delta$ represents the slope of the force–displacement curve and E_f the flexural modulus.

Short beam shear test

To determine the interlaminar shear strength of the composites, short beam shear tests were performed following ASTM D2344. A sliding roller three-point bending fixture, which included a loading pin (diameter 6.4 mm) and two support pins (diameter 3.2 mm), was used for the room temperature short beam shear tests. The test fixture was mounted in a 5-kN capacity, screw-driven load frame. Shimadzu AUTOGRAPH AG-G Series universal testing machine was used, with a cross-head speed of 1.3 mm/min, and at least six specimens were tested for each type of lay-up. The dimension of specimens was maintained at per ASTM standard for the three-point bend test. The length and width of the test specimens were 26.3 and 6.4 mm, respectively. Test specimens were cut from the laminates using water jet technique. The apparent interlaminar shear strength of composites was determined from specimens that were tested with a support span/sample thickness ratio of 5:1. The simply supported specimens allow lateral motion and a line load is applied at the mid span of the specimens. The apparent shear strength was then calculated as follows:

$$V = 0.75 \left(\frac{P_{max}}{wt} \right) \qquad (3)$$

where V is the apparent shear strength, P_{max} is the failure load, w and t are the width and thickness of the specimen, respectively.

Scanning electron microscopy (SEM) observation

The fracture surfaces of tensile-tested specimens were examined using the scanning electron microscope (JEOL JSM 6060) at accelerating voltage equal to 5 kV in the secondary electron mode. To reduce the extent of sample arcing, the samples were coated with gold by means of a plasma sputtering apparatus prior to SEM evaluation.

Results and discussion

Experimental results and discussions on the mechanical properties

Tensile test

Typical stress–strain curves obtained from the tensile tests are shown in Fig. 1. It is first noted that the tensile strength
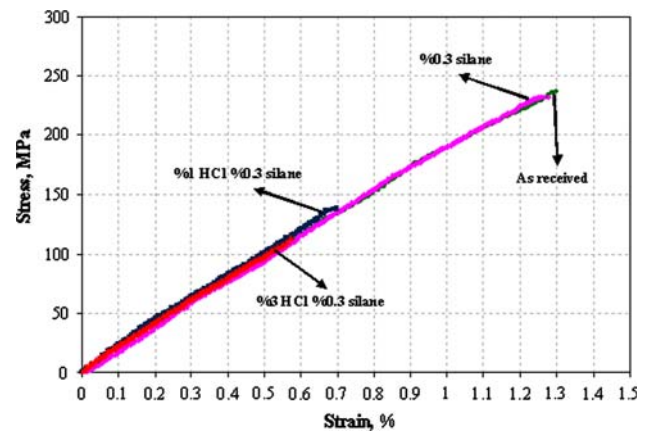

Fig. 1 Tensile stress–strain curves of laminates

and elongation at break vary considerably depending on the acid activation treatments of heat cleaned glass fibers prior to silanization. When the glass fibers are first treated with HCl solution and then with silane coupling agent, the tensile strengths of the composites seem to decrease significantly. It is known that the change in fiber strength has a direct influence on the mechanical properties of glass fiber composites because the fiber strength determines the ultimate load-bearing capacity of the composites. The decreased tensile strengths due to acid pretreatments may be attributed to the reduction of fiber strength. Table 1 demonstrates the mechanical properties of the composite laminates. As can be seen from Table 1, when the HCl concentration for acid pretreatment is increased to 3%, a further decrease of the tensile strength of the composite is noticed. The tensile strengths of the composites are decreased by approximately 41.5% for 1% HCl pretreatment + 0.3% γ-GPS treatment and 52% for 3% HCl pretreatment + 0.3% γ-GPS treatment in comparison with that of the as-received sample. The decrease in fiber strength is possibly owing to chemical corrosion of glass fibers exposed to hydrochloric acid. E-glass is comparatively sensitive to acid attack and the soluble components are leached and resulted in siliceous hydrated material [18]. Strength loss of the glass fibers is caused by an ion exchange reaction [19, 20]. The glass fibers will be chemically deteriorated as a consequence of non-siliceous ion leaching by ion-exchange reactions, i.e., the non-siliceous ions in the glass are replaced by the hydrogen ions in the acid [20]. Mechanism of the corrosion process is accepted, an ion exchange reaction, in which metal ions associated with the glass surface such as Ca^{2+} and Al^{3+} are replaced by H^+ from the acid medium [19]. It is believed that the acid treatments of the glass fibers prior to silanization may be responsible for the reduction in the tensile strength of the composites. It is thought that 3% HCl were strong and the fibers could be further damaged by

Table 1 Mechanical properties of glass fiber/epoxy composites (the data quoted are all average results taken from a minimum of six tests)

| Composite	Fiber volume fraction V_f (%)	Tensile modulus (GPa)	Tensile strength (MPa)	Elongation at break (%)	Flexure modulus (GPa)	Flexure strength (MPa)	ILSS (MPa)
As-received	39.3 ± 1.6	17.119 ± 0.85	236.263 ± 9.04	1.303 ± 0.12	19.762 ± 0.72	402.716 ± 12.8	27.978 ± 0.73
0.3% Silane	38.9 ± 1.5	18.660 ± 0.91	232.154 ± 10.1	1.277 ± 0.11	19.561 ± 0.91	467.834 ± 13.4	50.865 ± 1.10
1% HCl + 0.3% silane	39.1 ± 1.5	17.930 ± 0.86	138.288 ± 6.01	0.699 ± 0.06	20.347 ± 0.92	251.357 ± 10.3	26.723 ± 0.68
3% HCl + 0.3% silane	39.2 ± 1.6	18.339 ± 0.91	113.27 ± 5.05	0.572 ± 0.05	19.261 ± 0.87	184.446 ± 9.52	21.196 ± 0.57

The values after the (±) in all the tables refer the standard uncertainty of the measurement

occurring the interaction between the glass surface and HCl solution, thus resulting in lower tensile strength. On the other hand, González-Benito et al. [15] investigated the influence of different activation pretreatments of glass fibers on the structure of an aminosilane (γ-aminopropyl-triethoxysilane) coupling agent layer. According to their study, under acidic conditions, a great number of silanol groups are generated and greater coating degrees can be achieved. They found that the degree of silanization is the greatest for the acid activated samples. However, it is important to point out that the reduction in the tensile strength of the composites due to the chemical corrosion of glass fibers exposed to hydrochloric acid plays a vital role in the performance of composite materials.

However, in the absence of acid activation, there is a little difference (1.7%) in the tensile strength of the composite containing only silane treated glass fibers as compared to as-received glass fiber reinforced epoxy composite. It is interesting to note that although composite samples manufactured by acid activated fibers show small elongation at break values, all the test samples have almost similar tensile modulus. This indicates that all composites exhibit the same behaviors under tensile load.

Flexure test

Flexural strength–middle span deflection curves of laminates were given in Fig. 2. The flexural strengths of glass fiber/epoxy composites are presented in Table 1. It can be seen in Table 1 that the flexural strength of the composite is increased only with the silane treatment on the heat cleaned glass fiber. The flexural strength is enhanced by approximately 16% in comparison with that of the as-received sample. This may indicate the contribution of the silane treatment in terms of enhancement of fiber/matrix adhesion. The flexural strength of fiber reinforced composites depends on the adhesion between the fiber and the matrix, as well as on the strength of both constituents [6]. Wang et al. [7] investigated effect of interfacial mobility on flexural strength and fracture toughness of glass/epoxy
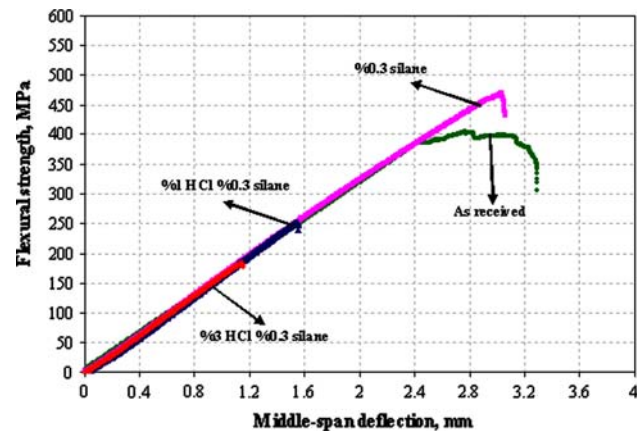
Fig. 2 Flexural strength–middle span deflection curves of laminates

laminates. Their results showed that the presence of silane coupling agent increased the flexural strength in both dry and wet samples. In their study, it was reported that the flexural strength increased probably due to the chemical bonding which occurs between the dissimilar phases. Shih and Ebert [21] also point out that stronger interfacial strength leads to an increase in flexural strength.

On the other hand, the flexural strengths of the composites are decreased with the acid activation treatments on the glass fibers. The flexural strengths of the composites are decreased by approximately 38% for 1% HCl pretreatment + 0.3% γ-GPS treatment and 54% for 3% HCl pretreatment + 0.3% γ-GPS treatment in comparison with that of the as-received sample. The remarkable reduction in the mechanical properties found in the composites may be mainly the result of glass fiber damage as mentioned above.

Interlaminar shear strength (ILSS) test

The results from the short beam shear tests of the composites show the relationship between surface treatments of the glass fibers and mechanical interlaminar properties of the composites (Table 1).

When the glass fibers are first treated with HCl solution particularly activated with 3% HCl solution and then with silane coupling agent, it can be seen that the ILSS values of the glass fiber/epoxy composites reduced as compared to that of composites containing as-received fibers. The interlaminar strengths of the composites are decreased by approximately 5% for 1% HCl pretreatment + 0.3% γ-GPS treatment of the glass fibers and 25% for 3% HCl pretreatment + 0.3% γ-GPS treatment of the glass fibers in comparison with that of the as-received sample (Table 1). It is clear from Table 1 that the composite prepared from γ-GPS treated glass fiber shows superior properties as compared to other composites. Kim and Mai [12] point out that a decrease in bond strength and other deteriorating effects are expected to occur if the surface treatment is excessive, leading to severe damage of the fiber. Jones and Betz [19] and Qiu and Kumosa [20] reported that glass fibers in acid solution will be chemically deteriorated and cause strength loss of the glass fibers. It is believed that the acid treatments of the glass fibers prior to silanization may be responsible for the reduction in the interlaminar shear strength of the composites.

As shown in Table 1, use of silane coupling agent, only 0.3% γ-GPS treatment, for surface treatment improves the interlaminar shear strength of glass fiber/epoxy composite in comparison with the as-received one. The improvement indicates that the adhesion at the interfaces between the fiber and the resin is largely increased. The interlaminar strength is enhanced by approximately 82% in comparison with that of the as-received sample. It is probable that the improvement of the interlaminar shear strength of glass fiber/epoxy composite may be due to the chemical interaction at the fiber–matrix interface.

SEM analysis

Figure 3a–d shows the SEM micrographs of the composite fracture surface obtained from tensile tests. As can be seen in Fig. 3a, it was found that the surfaces of as-received fibers were smooth and clean and the fibers were pulled out from epoxy matrix. There is no evidence or traces of matrix resin adhering to the fiber. Indeed, the fiber surfaces seem to be clean and free from any adhering polymer. It is clear that the interfacial adhesion between the fibers and the matrix is poor. This observation suggests an adhesive failure in the interface [22]. Therefore, the fracture mode of the as-received fiber reinforced composite is at the interface of the fiber/matrix and the interface structure cannot transfer stress effectively.

When the fibers had only the silane coupling agent surface treatment, the failure surface seem to be different from the previous case. Improved fiber/matrix adhesion was able to be seen in SEM photographs of fracture surfaces of the glass fiber/epoxy composite (Fig. 3b). A large amount of epoxy resin adhered to the fiber surfaces and formed a thick layer clearly indicate good interfacial interaction between the fiber and the resin and the cohesive failure at the matrix occurs.

Fig. 3 Representative SEM images of the breakage region for (**a**) as-received glass fiber reinforced epoxy composite; (**b**) 0.3% Silane treated glass fiber reinforced epoxy composite; (**c**) 1% HCl and 0.3% silane treated glass fiber reinforced epoxy composite; (**d**) 3% HCl and 0.3% silane treated glass fiber reinforced epoxy composite that were tensile-tested in machine direction
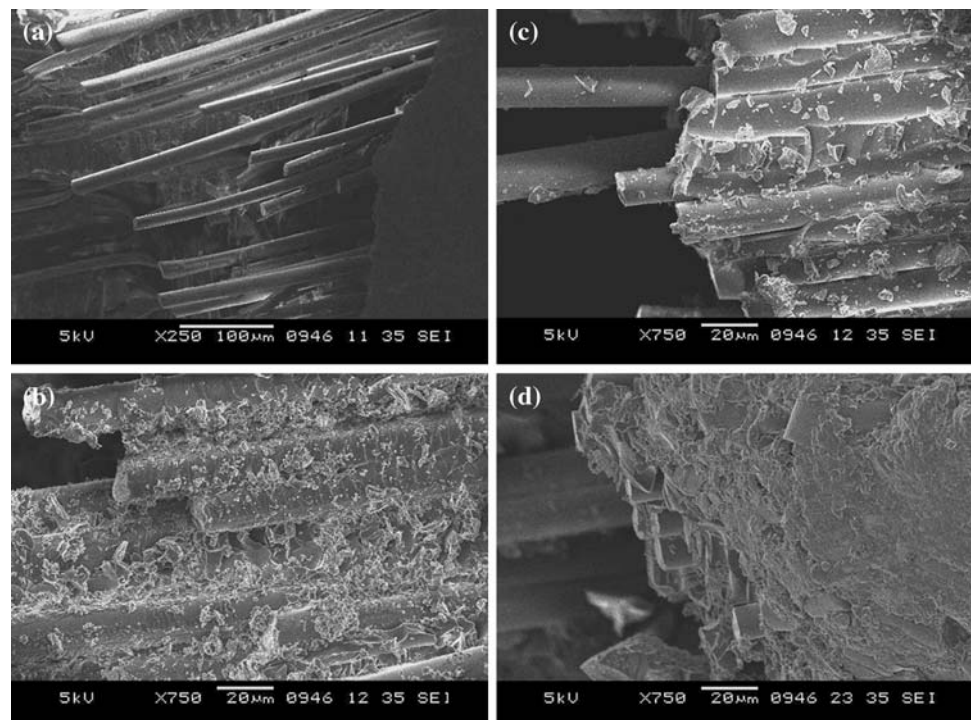
There are large amount of resin adhered on 1% HCl pretreated + 0.3% γ-GPS treated glass fiber surface (Fig. 3c) which shows to enhance the interfacial adhesion between the glass fiber surface and resin and the cohesive failure at the matrix occurs. For 3% HCl pretreated + 0.3% γ-GPS treated glass fibers (Fig. 3d), all fibers seem in epoxy resin after failure. The failure surface seems to be uniform. The cohesive failure at the matrix indicates an increase when the HCl concentration is increased up to 3%. This suggests the strongest interfacial interaction between fiber and matrix. On the other hand, as shown in Fig. 3c and d, it is emphasized here that the increase in fiber–matrix interfacial interaction did not cause the increase in ILSS values (Table 1). This is due to fact that when the surface treatment is excessive, this will lead to severe damage of the fiber and cause a decrease in ILSS of the composites.

Conclusion

The effect of the surface treatment of the glass fiber on the mechanical properties of glass fiber/epoxy composites was investigated. The tensile strengths, flexural strengths, and interlaminar shear strengths of the composites decreased due to HCl pretreatment of glass fibers prior to silanization. In this way, a decrease in the strengths is expected to occur if the surface treatment is excessive, leading to severe damage of the fiber. The tensile strengths of the composites are decreased by approximately 41.5% for 1% HCl pretreatment + 0.3% γ-GPS treatment and 52% for 3% HCl pretreatment + 0.3% γ-GPS treatment in comparison with that of the as-received sample. The interlaminar shear strength and the flexural strength of the composites are decreased by approximately 25 and 54% for 3% HCl pretreatment + 0.3% γ-GPS treatment in comparison with that of the as-received sample, respectively.

The presence of γ-GPS in the composite leads to increasing ILSS and flexural strength of the composite, which can be related to the effect of increased interfacial adhesion between glass fiber and polymer matrix. The interlaminar shear strength and the flexural strength are enhanced by approximately 82 and 16% in comparison with that of the as-received sample, respectively.

Briefly, the silane coating on the heat cleaned glass fibers has the beneficial effect on the adhesion between the fibers and the matrix and increase ILSS of the composite due to interaction between the fibers and the matrix which enhance the interfacial adhesion, whereas the silane coating on the acid activated glass fibers did not improve the interlaminar shear strength of the composite due to the fiber damage caused by HCl acid solution.

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