The Effect of Surface Treatment of Glass Fiber on the Mechanical Properties of Epoxy Composite

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1. Introduction

Nowadays, polymeric composites are widely used in many industries. Among them, glass fiber reinforced polymer has the paramount importance due to its mechanical, chemical, and physical properties. It is well known that the properties of composites not only rely on each reinforcement and matrix properties but also depend on the nature of interphase region which are significantly controlled by chemical bond and load transfer between fiber and polymer. Surface treatment of glass fibers is one of the ways of enhancing the interfacial adhesion and affinity between fiber and polymer matrix.

The inorganic glass fiber and organic polymer are incompatible, introducing a third material such as coupling agents to form a chemical bond and improve interfacial adhesion. Organofunctional silanes coupling agents have been effective for glass fiber chemical modification. However, the acid treatment cause to generate hydroxyl groups on the surface of glass fibers to react with silane coupling agents more efficiently. The acid activation of glass fibers prior to silanization, changes the surface of fibers and increases the amount of -OH groups on the surface. The hydrolyzed groups Si-OH in silane tend to react with Si-OHs in the glass fiber surface to form Si-O-Si bonds.

This research studied the influence of different fiber surface treatments in mechanical properties. Interactions between glass fibers and silane coupling agents were characterized by FTIR. Mechanical properties of the composites were investigated by short beam shear strength (ILSS). The topology of the surface of glass fibers were observed with scanning electron microscope (SEM).

2. Experimental

E-glass fibers used in this study were supplied by STA Co. Thermoseting bisphenol-A (2040 Epolem) and amine system hardener (2047 Epolem) were prepared by Axson Co. Resin and hardener were mixed in ratio of 100:32 to prepare polymer as matrix of composite. The silane coupling agent, APTES (γ -amino propyl triethoxy silane), supplied by Aldrich-Sigma. Hydrochloric acid (HCl 37%) and acetic acid (CH₃COOH 100%) were used without further purification.

Acid treatment of E-glass fibers done by HCl (10%, v/v) for 3 hours at room temperature. After acid activation, samples were washed with distilled water several times to

remove chloride compeletly -, as determined by $AgNO_3$ test. Finally they were dried at 110 °C for one hour. The silane treated fibers were obtained by treating as-received fibers and acid activated. Aqueous solutions were prepared by methanol (95 wt.%) and distilled water (5 wt.%) and the silane concentration of 0.5 (v/v). After the silane coupling agent was hydrolyzed at pH=4 for 1 hour with acetic acid, the fibers dipped in the solution for 30 minutes and dried at 110 °C for 1 hour. Table 1 shows UGF, GF, AA, GFTS, and AATS preparation.

As received and treated glass fibers as reinforcment of epoxy were used to prepare composite by vacuum infusion processing. The epoxy resin and hardener mixture in stoichiometric ratio were applied to the fibers. The curing schedule of all composite samples was 24 hours at room temperature, 2 hours at 40 °C and 16 hours at 70 °C.

Table 1. Sample code and conditions

Surface treated Sample	Acid Activation	Silanization treatment	Composite Sample
UGF	-	-	Co-UGF
GF	-	-	Co-GF
AA	HCl 10%	-	-
GFTS	-	γ-APS	Co-GFTS
AATS	HCl 10%	γ-APS	Co-AATS

3. Results and Discussion

FTIR Spectroscopic Measurement

FT-IR spectroscopy was used to observe the effect of acid activation and silane coupling agents on the glass fiber surface. Figure 1 exhibits the FTIR spectrum of glass fiber (GF), acid activated fibers (AA), silanized glass fiber (GFTS), and acid activated silanized fibers (AATS). It was shown that the peak at 1042.02 cm⁻¹ corresponds to the stretching vibration of Si-O-Si bonds. After acid activation, this peak shifted to 1082.42 cm⁻¹. The peak at 3400-3500 cm⁻¹ is related to the OH stretching vibration. After acid activation, OH peak broadens were compared to GF spectrum. Therefore, the content of Si-OH on the surface increases as spectrum shows. After silanization of fibers and acid activated fibers the band of OH at 3400-3500 cm⁻¹ reduced. therefore, an interaction takes place between silane coupling agent and glass fiber.

The degree of adhesion at the interface between a fiber and a matrix can be measured in terms of the interlaminar shear strength (ILSS). Table 2 shows the results from short beam shear tests of the composites. As seen, surface treatment leads to an increase of ILSS of the composites, which can be related to the effect of increasing the degree of adhesion at interfaces among the fiber, matrix, and

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silane coupling agent. The interlaminar shear strength of Co-GFTS increases about 18% compare to GF samples. The composite silanization treatment glass fiber composite showed the highest values interlaminar strength, with acid activation glass fiber composite, however, the enhancement was not significant. It is believed that excessive surface treatment prevents increasing bond strength.

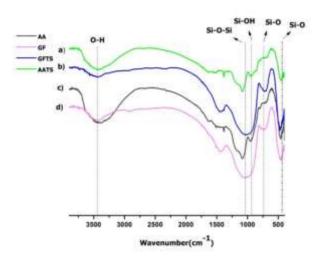


Fig. 1. The FTIR Spectrum (a) AATS (b) GFTS (c) AA (d) GF samples

Table 2. Short beam shear strength result

Composite	Thickness (mm)	Width (mm)	Force(N)	ILSS (MPa)
Co-UGF	1.8	12.34	318.58	10.75±0.32
Co-GF	1.95	12.31	350.06	10.91 ± 0.22
Co-GFTS	2.25	12.49	475.11	12.69 ± 0.42
Co-AATS	2.02	12.74	379.49	11.11±0.24

Comparing the SEM micrographs of AATS and GFTS samples in Figure 2, the coating layer was observed on the surface of glass fiber. The silanizated glass fibers are noticeably thicker, however, the silanizated fibers after HCl activation treatment display cracks along the fiber surface.

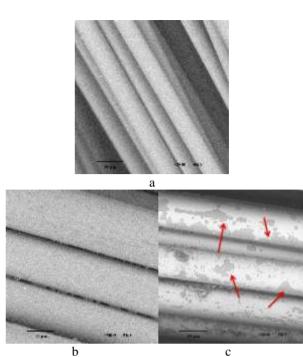


Fig. 2. Scanning electron micrographs a) GF, b) GFTS, c) AATS

4. Conclusion

The effect of surface treatment of the glass fibers on the mechanical properties of glass fiber/epoxy composites was investigated. The mechanical properties of glass fiber/epoxy composite material are strongly dependent on molecular structure of coupling region. The surface treatment of glass fibers led to increasing ILSS of the composite. The enhancement of 18% for silanized glass fiber composite compare to non-treated glass fiber composite was found in short beam shear test.