Influence of chemical surface treatment of basalt fibers on interlaminar shear strength and fracture toughness of epoxy-based composites

Seong-Ock Lee\textsuperscript{a}, Kyong Yop Rhee\textsuperscript{b,***}, Soo-Jin Park \textsuperscript{a,*}

\textsuperscript{a}Department of Chemistry, Inha University, 100 Inchon, Incheon, Republic of Korea
\textsuperscript{b}Department of Mechanical Engineering, Kyung Hee University, Yongin 446-701, Republic of Korea

\textbf{A R T I C L E   I N F O}

\textbf{Article history:}
Received 6 October 2014
Received in revised form 13 August 2015
Accepted 17 August 2015
Available online 24 August 2015

\textbf{Keywords:}
Basalt fibers
Acid-alkali treatment
Mechanical interfacial properties
Fracture toughness

\textbf{A B S T R A C T}

In our study, the effect of chemically treated basalt fibers on the mechanical interfacial properties of basalt fibers reinforced epoxy composites was investigated. The surface properties of the basalt fibers were determined by Fourier Transform Infrared (FT-IR) spectroscopy. The surface structure of the basalt fibers was analyzed by Atomic Force Microscopy (AFM). The acid and alkali chemical treatments led to significant changes in the surface characteristics of the fibers. The chemically treated fibers improved the mechanical interfacial properties, interlaminar shear strength (ILSS) and fracture toughness (KIC) of the composites. Composites treated with H\textsubscript{2}SO\textsubscript{4} had higher values of ILSS and KIC than the KOH-treated composites. These results are attributed to the improvement of interfacial bonding strength, which was caused by an increase of the surface roughness of basalt fibers.

© 2015 The Korean Society of Industrial and Engineering Chemistry. Published by Elsevier B.V. All rights reserved.

Epoxy resins have mainly been used as a matrix for fiber-reinforced composites because they have excellent mechanical properties, adhesion, water and chemical resistance. Fiber-reinforced composites are widely used in industrial applications. Recently, the use of basalt fibers in fiber-reinforced composites has been studied. Basalt fibers are fabricated from melted basalt rocks. Basalt fibers have higher elastic modulus, strength, and thermal resistance than glass fibers. Moreover, basalt fibers have excellent chemical resistance and thermal performance [1–8].

It is well known that the mechanical interfacial properties of fiber-reinforced composites are influenced by the adhesive strength and wettability of the fibers and matrix. However, the basalt fiber-reinforced composites exhibit low interface adhesion between basalt fibers and matrix due to the chemical inert surface of basalt fibers [9,10].

There have been many studies to improve the interfacial adhesion between basalt fibers and matrix. Kim et al. [11] investigated the effects of surface-treated basalt fibers on the interfacial adhesion between the fibers and epoxy resin. They found oxygen-plasma-treated basalt fibers improved the interlaminar fracture toughness of basalt/epoxy composites. Wei et al. [12] modified the surface of basalt fibers using organic/inorganic hybrid sizing, and found it increased the tensile strength and interlaminar shear strength of the basalt/epoxy composites.

In this paper, the effect of the surface chemical treatments on the interfacial adhesion between fibers and epoxy and the composites' impact resistance was investigated. Basalt fibers were treated with H\textsubscript{2}SO\textsubscript{4} or KOH solutions of varying concentrations, and compared with untreated fibers. Fig. 1 shows the FT-IR spectra of the chemically treated fibers, as well as the spectrum of the untreated fibers. The bands occurring at around 3400 cm\textsuperscript{-1}, 860 cm\textsuperscript{-1}, and 1030 cm\textsuperscript{-1} were attributed to metal-OH, Al-O, and Si-O-Si peak, respectively. The strong band at around 1320 cm\textsuperscript{-1} is assigned to Al-O. An absorption peak at about 470 cm\textsuperscript{-1} is due to Si-O-Si and Al-O-Al bending vibrations [13–16]. In acid and alkali solution, the Si-O-Si network is destroyed by OH\textsuperscript{-} and H\textsuperscript{+}.

The reactions are explained by the following equation:

\begin{equation}
\text{Si-O-Si + OH}^- \rightarrow \text{SiOH + Si-O-}
\end{equation}

\begin{equation}
\text{Si-O-Si + H}^+ \rightarrow \text{SiOH + Si-}
\end{equation}

Thus, an increase in the treatment concentration of fibers strengthens the surface OH peak intensity. Whereas, metal oxide peaks are weakened [17–19].
It is generally accepted that the mechanical properties of composites depend on the fiber-matrix interfacial adhesion. In our study, the degree of adhesion at the interfaces between basalt fibers and matrix resin was measured by short-beam flexural tests, with the ILSS of the composites determined by the following relationship:

$$\text{ILSS} = \frac{3P}{4bd}$$  \hspace{1cm} (3)

where $P$ is the breaking load, $b$ is the width of the specimen, and $d$ is the thickness of the specimen.

In addition, the composites' critical stress intensity factors, $K_C$, were determined. $K_C$ is a fracture toughness parameter that describes the state of stress near the tip of a crack as a function of the specimen geometry, crack geometry, and applied load, and is based on linear elastic fracture mechanics [22,23]. For the SENB tests, $K_C$ was determined according to ASTM E-399-78, using the following equation:

$$K_C = \frac{P \cdot L}{bd^{3/2}} Y$$  \hspace{1cm} (4)

where $P$ is the critical load for crack propagation (N), $L$ is the length of the span (mm), $b$ is the specimen width (mm), and $d$ is the specimen thickness (mm). $Y$ is the geometrical factor.
properties of the composites were improved by increasing the roughness of the basalt fiber surfaces, which most likely improved interfacial adhesion between the basalt fibers and epoxy resin. Therefore, the improvement in mechanical interfacial properties was caused by the enhanced interfacial adhesion between basalt/epoxy interfaces, which was induced by the chemical treatment of the basalt fibers.

Experimental

Materials

The fibers used in this study were commercial, untreated, roving-type basalt fibers (1200 tex, YJC Co., Ltd., Korea). The epoxy was diglycidyl ether of bisphenol A (DGEBA, YD-128, Kukdo Chemical, Korea). 4,4’-Diaminodiphenyl methane (DDM, TCI Co., Japan) was used as curing agent. To reduce the viscosity of DGEBA, Methylene ketone (MEK, OCI Company, Ltd., Korea) was used.

Surface treatment of basalt fibers

Prior to treatment, as received basalt fibers were dipped in acetone for 48 h to remove sizing agents. 150 g of desized basalt fibers were treated with 1.5 L of H2SO4 or KOH solutions of varying concentrations (0.5, 1, and 3 M) in 3 h. The treated basalt fibers were washed several times with distilled water and dried in an oven at 80 °C for 8 h.

Fabrication of basalt fibers/epoxy composites

100 g of DGEBA was mixed with 30 g of DDM at 70 °C. Then 20 g of MEK was added to the DGEBA/DDM mixture and stirred thoroughly. Basalt/epoxy laminates were made by continuous impregnation of fibers using the drum-winding technique for manufacturing prepregs, followed by hot pressing. Eighteen ply laminates of prepregs were made in a hot press (10 MPa, 150 °C, 150 min) employing the vacuum-bagging method.

Characterization

Infrared spectra of the chemically treated fibers were measured by Fourier Transform Infrared (FT-IR) spectroscopy (VERTEX 80V, Bruker). The fiber surface morphologies were analyzed using Atomic Force Microscopy (AFM, Nanoscope Multimode IVa, Bruker). The interlaminar shear strength (ILSS) was determined by the three-point short-beam bending test to estimate the interfacial adhesion strength of the composites, according to ASTM D-2344. An analytical expression for the critical stress intensity factor (Kc) was characterized by the Single Edge Notched Bending (SEN) test in the three-point flexural mode. Notches approximately half the thickness of the specimen were cut using a diamond-coated saw. The three-point bending test was conducted using a universal test machine (LRSK plus, Lloyd, UK), according to ASTM E-399.

Acknowledgements

This work was supported by the Industrial Strategic technology development 192 program (10050953) funded by the Ministry of Trade, Industry & Energy (MI, Korea).

References