Journal of Industrial and Engineering Chemistry 32 (2015) 153–156

Contents lists available at ScienceDirect

Journal of Industrial and Engineering Chemistry

journal homepage: www.elsevier.com/locate/jiec

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

Seong-Ock Lee [a], Kyong Yop Rhee [b,**], Soo-Jin Park [a,*]

[a] Department of Chemistry, Inha University, 100 Inharo, Incheon, Republic of Korea
[b] Department of Mechanical Engineering, Kyung Hee University, Yongin 446-701, Republic of Korea

ARTICLE INFO

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

Keywords:
Basalt fibers
Acid–alkali treatment
Mechanical interfacial properties
Fracture toughness

ABSTRACT

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 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_2SO_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.

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 interfacial adhesion between basalt fibers and matrix due to the chemically 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_2SO_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^{-1}, 860 cm^{-1}, and 1030 cm^{-1} were attributed to metal-OH, Al–O, and Si–O–Si peak, respectively. The strong band at around 1320 cm^{-1} is assigned to Al–O. An absorption peak at about 470 cm^{-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^- and H^+.

The reactions are explained by the following equation:

$$Si{-}O{-}Si + OH^- \rightarrow SiOH + Si{-}O{-} \quad (1)$$

$$Si{-}O{-}Si + H^+ \rightarrow SiOH + Si{-} \quad (2)$$

Thus, an increase in the treatment concentration of fibers strengthens the surface OH peak intensity. Whereas, metal oxide peaks are weakened [17–19].

* Corresponding author. Tel.: +82 328767234; fax: +82 328675604.
** Corresponding author. Tel.: +82 312012565; fax: +82 312026693.
E-mail addresses: rheeky@khu.ac.kr (K.Y. Rhee), sjpark@inha.ac.kr (S.-J. Park).

http://dx.doi.org/10.1016/j.jiec.2015.08.009
1226-086X/

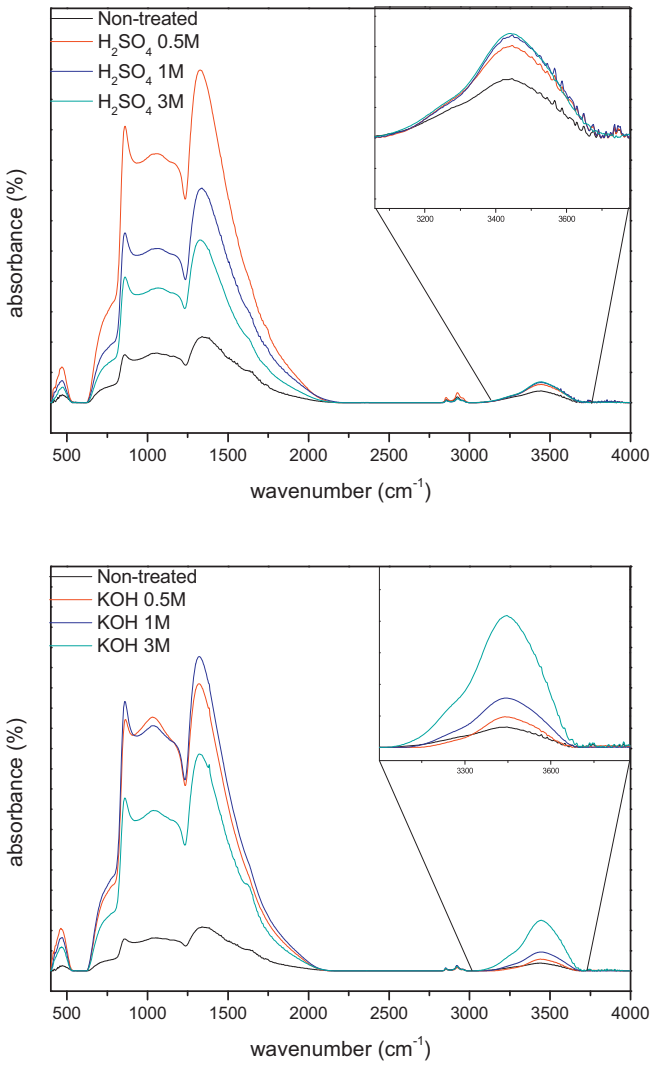

Fig. 1. FT-IR spectra of chemical-treated basalt fibers.

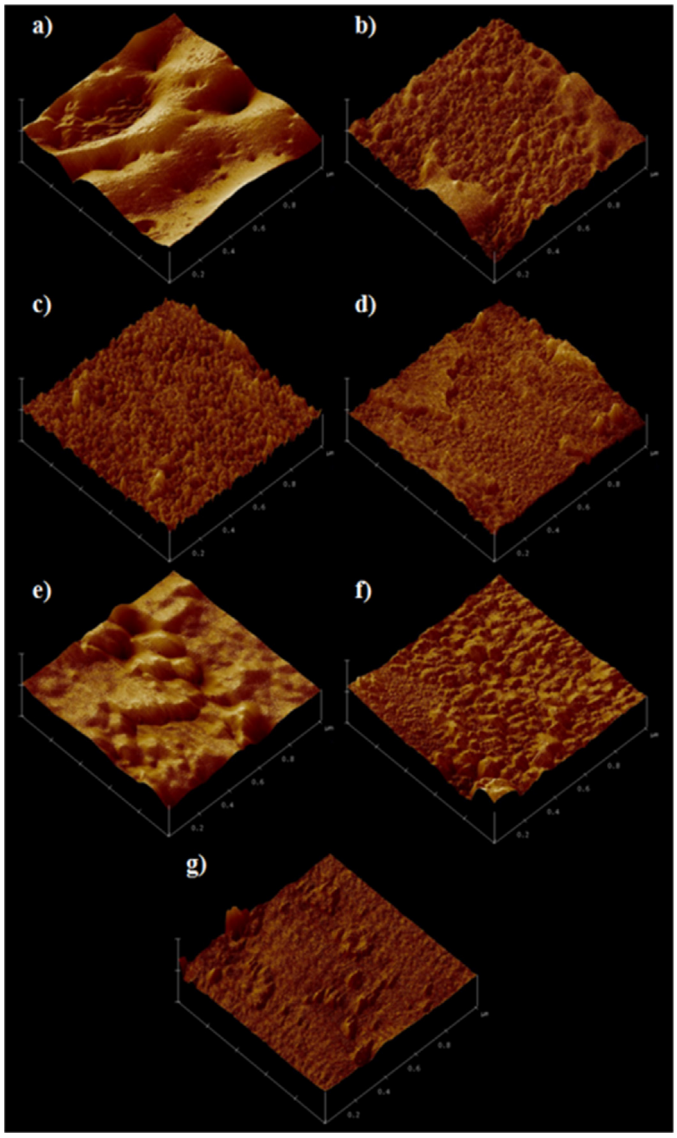

Fig. 2. AFM images of treated basalt fibers: (a) non-treated, (b) 0.5 M H_2SO_4, (c) 1 M H_2SO_4, (d) 3 M H_2SO_4, (e) 0.5 M KOH, (f) 1 M KOH, and (g) 3 M KOH.

Fig. 2 shows AFM images of the chemically treated fibers. It can be seen that the chemical treatment does change the surface morphology at the microscopic scale and the images illustrate how the roughening of the surface occurs. Therefore, we can expect that chemical treatment may lead to microetched formations on the surface of basalt fibers that result in improved interfacial adhesion between the fibers and epoxy resin. Whereas, corrosion of basalt fiber surfaces is progressed in high concentration solution. That decreases interfacial adhesion between the fibers and epoxy [20,21].

The results allow the characterization of surface states occurring in H_2SO_4 and KOH solution as schematically shown in Fig. 3. In the initial state, the non-treated fibers have slight surface flaws. In state A, Si–O–Si network is attacked by OH^- and H^+ ions. Then the fiber surfaces are assumed to include defects in the few areas. The fiber surfaces have a uniform structure because of OH^- and H^+ ions attack in suitable concentration solution (state B). In high concentration, the fiber surfaces are peeled by corrosion (state C). Surface treatment of the fibers effects roughness of the fiber surfaces. Thus, it influences the mechanical properties of the composites.

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:

$$\mathrm{ILSS} = \frac{3P}{4bd} \tag{3}$$

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_{IC}, were determined. K_{IC} 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_{IC} was determined according to ASTM E-399-78, using the following equation:

$$K_{IC} = \frac{P \cdot L}{bd^{3/2}} Y \tag{4}$$

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.