Microstructural Investigation of Long-Term Degradation Mechanisms in GFRP Dowel Bars for Jointed Concrete Pavement

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ABSTRACT: Steel dowel bar is used to transfer loads in concrete pavement slab. However, once the steel dowel bar corrodes, it may cause faults, such as joint freezing in concrete pavement, level differences resulting from spalling or decreased efficiency of load transfer, etc., which are the same problems experienced by typical reinforcing steel. This study evaluated the applicability of glass fiber-reinforced polymer (GFRP) dowel bar as a substitute for steel dowel bar. A microstructural analysis was conducted to examine the decrease in durability of GFRP dowel bar exposed to deterioration environments. To analyze the deterioration mechanism of GFRP dowel bar, scanning electron microscopy was employed and the porosity was measured by the gas absorption method. It was concluded that the longer the GFRP dowel bar was exposed to deterioration environments, the more the interlaminar shear stress decreased. This result was validated by the microstructural analysis. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 108: 3128–3137, 2008

Key words: dowel bar; glass fiber-reinforced polymer; degradation; concrete pavement

INTRODUCTION

Fiber-reinforced polymers (FRPs) have gained lots of attention as substitutes for steel reinforcing rods in concrete because of their durability. Currently, many different studies on the applicability of FRPs are ongoing in various fields. Among these fields, new research using FRPs as a load transfer element in concrete pavement structures has been gaining attention. The dowel bar commonly used for load transfer in concrete pavement slab is made from steel. However, once the steel dowel bar corrodes, it may cause faults, such as binding due to lockout of the dowel bar in concrete pavement, level differences resulting from spalling or decreased efficiency of load transfer, etc., which are the same problems experienced by typical reinforcing steel.[1–3]

A typical dowel bar is more vulnerable to corrosion when it is used to transfer loads on the joints of concrete pavement compared with when it is used as a reinforcement in concrete structures. As shown in Figure 1(a), the reinforcing steel used to increase the tensile strength of concrete structures is protected by the concrete surrounding it and, consequently, is not directly exposed to external conditions unless critical faults such as concrete cracks occur. However, the dowel bar used as a load transfer element in concrete pavement is more likely to corrode since it may be directly exposed to external conditions when the materials between pavement slabs and their joints are collapsed [Fig. 1(b)].[4]

When corrosion occurs in a dowel bar, freezing is likely to be caused by the volume expansion, followed by fractures due to the curling of the concrete pavement slabs (Fig. 2).[5] To solve the problems caused by the corrosion of steel dowel bar, many studies on the performance and feasibility of FRP dowel bar are being undertaken. In particular, glass fiber-reinforced polymer (GFRP) dowel bar is considered as a potential solution with high industrial productivity.[6–9]

GFRP dowel bar can be easily damaged by the capillary water of concrete, which typically has high alkalinity (pH 12.4–13.7) compared with other FRP composites.[10,11] Thus, an adequate durability study on the material should be performed so that GFRP dowel bar can be applied as a load transfer element and reliable life span data of concrete pavement can be obtained.

The deterioration of GFRP dowel bar occurs first when free hydroxyl ions (OH^-) and H_2O molecules diffuse through the matrices of the GFRP dowel. The resins widely used for FRP composites include polyester, vinyl ester, and epoxy. Weak adhesion of the polyester or the vinyl ester results in serious deterioration, as described in reaction 1, when OH^- penetrates into the structure[10]:

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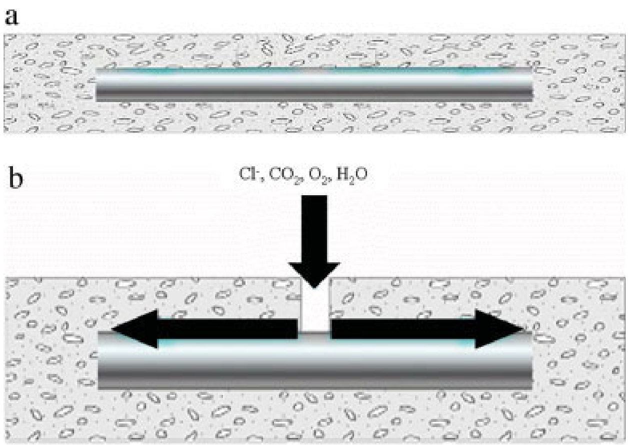
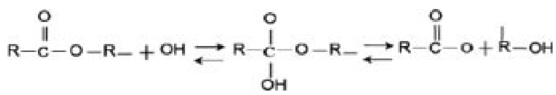


Figure 1 Installed steel rebar and dowel bar in concrete. (a) Steel rebar in concrete structures. (b) Dowel bar in concrete pavement slab. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]



(1)

Meanwhile, the matrix formed by ester-free vinyl ester is hardly deteriorated by hydroxyl ions compared with a polyester matrix. The epoxy matrix has a molecular structure different from that of the ester group, making it more resistant against OH^-. The failure of the matrix is only due to expansion and plasticization mechanisms.[11]

In water or alkaline solution, the deterioration mechanisms of the glass fibers essentially depend on leaching and etching. External alkaline ions leaching into the glass fibers give rise to the most important chemical reaction that dissolves the glass fibers into water (reaction 2)[12]:

$$\mathrm{Si-ONa} + \mathrm{H_2O} \rightarrow \mathrm{Si-OH} + \mathrm{NaOH} \quad (2)$$

The other critical reaction is etching, in which hydroxyl ions break Si—O—Si bonding as described by reaction 3:[12]

$$\mathrm{Si-O-Si} + \mathrm{OH^-} \rightarrow \mathrm{-Si-OH} + \mathrm{Si-O^-} \quad (3)$$

The deterioration at the interfaces between the fibers and the matrices involves a much more complex mechanism.[13] The interface is a nonhomogeneous region with a thickness of about 1 μm. This layer is weakly bonded and is most vulnerable to deterioration.[13]

The three dominant deterioration mechanisms included matrix osmotic cracking, interfacial debonding, and delamination.[14]

This study investigated the deterioration characteristics of GFRP dowel bar in different simulated environmental conditions experienced by concrete pavement. An accelerated test method was applied to examine the long-term deterioration of the GFRP dowel bar within a limited period. Analyzing the results involved microstructural analysis using scanning electron microscopy (SEM) and gas absorption to look into the deterioration of the glass fibers and the matrix resin.

EXPERIMENTAL PLAN AND METHODS

GFRP dowel bars

FRP dowel bar has higher tensile strength and corrosion resistance than steel dowel bar. FRP dowel bar is manufactured by arranging fibers unidirectionally, joining them with polymer resin and, finally, engineering the shape through a pultrusion process. Its mechanical properties depend on the direction of the fibers and it is typically weak against bending and shear loads. To overcome the weaknesses, measures such as varying the space between dowel bars and increasing their cross-sectional area have been used. As such, the cross-sectional area of FRP dowel bar is larger than general reinforcing bars. When considering FRP dowel bars, the functionality and economic productivity accomplished by increasing the section area should both be considered. Glass fiber is one of the FRP dowel bar component is considered to have the highest applicability and was chosen for the present study. The properties of glass fiber are listed in Table I. Vinyl ester resin was chosen as polymer matrix. Typical polymers used as matrix material include epoxy, vinyl ester, and polyester. Because the polyester matrix is easily damaged by OH^- ions, it is not suitable for FRP dowel bar material. Although vinyl ester is also affected by OH^- ions, ester-free commercial products have dramatically decreased adverse effects. Based on previous research and data with respect to the conventional FRP reinforcing material, this study used an economical vinyl ester product with good durability.

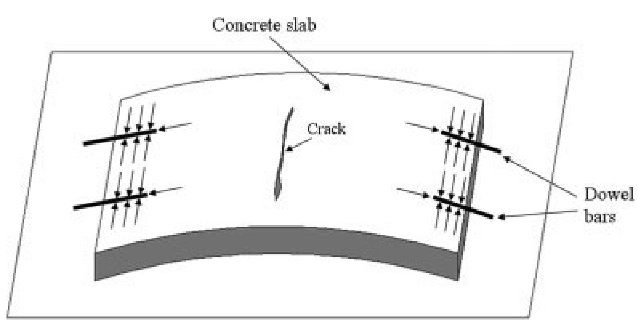


Figure 2 Concrete pavement slab curling.

TABLE I
Mechanical Properties of the Glass Fibers and Matrix

Mechanical properties	Vinyl ester resin	E-glass
Yield stress (MPa)	90	1,890
Elastic modulus (GPa)	3.4	71
Ultimate strain (%)	5.2	2.64
Fiber density (g/cm^3)	–	2.62
Fiber diameter (10^{-6} m)	–	16.5

The properties of the vinyl ester used in the study are described in Table I.

The GFRP dowel bar used in the study contained 80% glass fiber and 20% vinyl ester resin, and was manufactured through a pultrusion process. The cross-sectional area of the GFRP dowel bar was elliptical. The load transferred from the upper side tended to be concentrated on the top and bottom of the dowel bar, which were in contact with the concrete. The resulting stress concentration at the concrete surfaces in such an arrangement can cause fractures in the pavement. Studies aiming to solve this problem by increasing the load supporting areas of the top and bottom have found that an elliptical section area is desirable in terms of stress distribution. Therefore, this study employed GFRP dowel having an elliptical section whose ratio of semimajor axis to semiminor axis was 54 mm/34 mm (long axis/short axis). The dimensions of the GFRP dowel bar are listed in Table II.

Accelerated aging

To evaluate the durability of GFRP dowel bar, three different conditions were prepared to match those in actual concrete pavement structures. The accelerated deterioration environments for the tests were as follows. By mixing 1% NaOH, 0.16% KOH, and 1.4% $Ca(OH)_2$, a solution was prepared with a pH of 12.6, which is a typical value measured in concrete pavement. Temperature was considered as another factor since the GFRP dowel bar becomes more brittle when the alkaline solution warms up. In summer, the temperature of concrete pavement is likely to reach 70°C, and alkaline water may come into contact with the bar after rainfall. The influence of temperature was considered because higher temperatures induce accelerated aging of the bar.

Accordingly, two alkaline solutions at 70°C and at 20°C were used at the same time. Exposure times in the alkaline solutions were 20, 40, 60, and 120 days. Second, 3% NaCl solution was prepared to simulate a seaside road vulnerable to damage from salty seawater. The temperature of the NaCl solution was set at 20°C. Because alkaline solutions are known to have a major effect on GFRP, we can determine which environment has the greater effect by comparing the results of NaCl and alkaline solutions at a room temperature of 20°C and seeing if this also holds true for higher temperatures. Third, an anti-icing agent condition, created with anti-icing agents used for snow-removal work, was reconstructed by making an equivalent solution. The dowel bars arranged around joints are affected by the anti-icing agents in winter. For the purpose of examining the effects of anti-icing agents, a GFRP dowel was soaked in 4% $CaCl_2$ solution, which is the most widely used anti-icing chemical. The exposure times for NaCl and $CaCl_2$ solutions were 20, 40, and 80 days. The exposure temperature of 20°C was chosen because the temperature of the pavement is less than 20°C when using anti-icing agents.

TEST METHODS

Interlaminar shear stress test

Dowel bar made of GFRP is used to support against bending or interlaminar shear stress rather than ten-

TABLE II
Shape and Volume Fraction of the GFRP Dowel Bar

Shape of the GFRP dowel bar	Dimension (mm)			Composition ratio (%)	
	Long axis	Short axis	Applied diameter	Glass fiber	Vinyl ester
	50	34	31	80	20

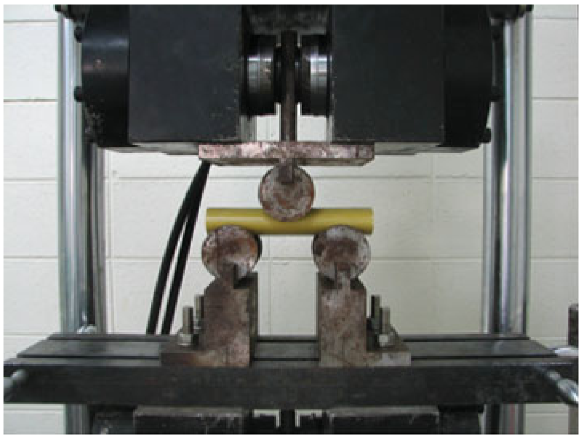


Figure 3 Setup for apparent interlaminar shear stress test. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

sion. GFRP materials have much better tensile strength than steel reinforcements, but have relatively poor resistance against lateral loads called shear forces. This is due to the fact that GFRP dowel bar is manufactured by a pultrusion process in which the fibers are arranged unidirectionally and bonded using polymer matrix. This study adopted ASTM D 4475 (standard short-beam test method for apparent shear strength of pultruded reinforced plastic rods)[15] to investigate the interlaminar shear properties of GFRP dowel bar. Although the results obtained in accordance with ASTM D 4475 were not directly applicable to dowel bar design, the method is widely used to compare and analyze interlaminar shear stress depending on the shape and size of GFRP dowel bar. According to the test, specimens should be supported at two supporting points and the load should be applied at one loading point. The supporting point is positioned at a distance from the loading point equal to 1.5 times the diameter, and the supporting and loading points should be more than 1/2 the diameter. Figure 3 illustrates the interlaminar shear stress test setup. The test specimens were stored in a room with a constant temperature of (23 ± 2)°C and a constant humidity of (50 ± 5)% for 40 h prior to the test to make sure they had the same internal and external conditions. The test was conducted with a displacement speed of 1.3 mm/min using a UTM (ESH testing limited, model 250) with 250 kN of maximum capacity. The results were determined in accordance with eq. (4):

$$S = 0.849\frac{P}{D^2} \qquad (4)$$

where, S is the interlaminar shear stress (MPa), P is the maximum load (kN), and D is the diameter (mm).

Measurement of porosity

The porosity analysis was conducted by exposing the GFRP dowel bar to durability deterioration environments followed by a gas absorption analysis. Vinyl ester resin may expand due to permeation by chemical solutions when it is exposed to various environments. This expansion may change the density of the GFRP dowel bar. If the vinyl ester resin does expand, this will cause cracks in the resin or at the interface of the glass fiber and the resin. The occurrence of cracks can be determined by measuring the change in the pores after exposing the bar to the potential damaging environments.

Decreased durability was attributable to the aging of glass fiber and polymer matrix caused by micro crack and pore developed by various kinds of adverse factors. The durability deterioration factors were investigated under deterioration conditions using gas absorption methods. Throughout the experiment, nitrogen gas was used as an adsorbate for the gas adsorption pore measurement, and a temperature of −196°C was maintained with liquid nitrogen. The adsorption isotherm

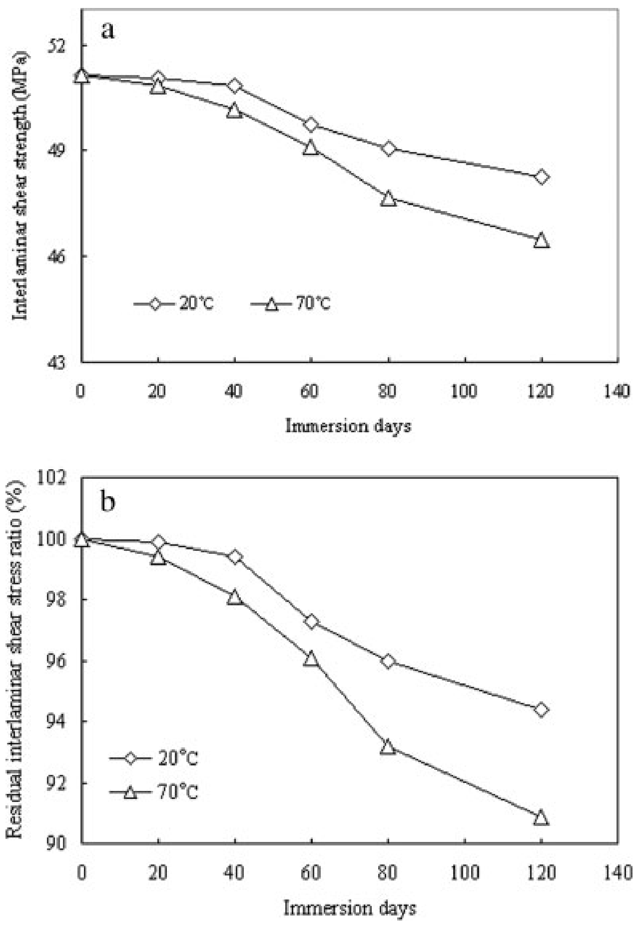


Figure 4 Interlaminar shear properties of GFRP dowel bar after immersion in alkaline solution. (a) Interlaminar shear stress; (b) Residual interlaminar shear stress ratio.

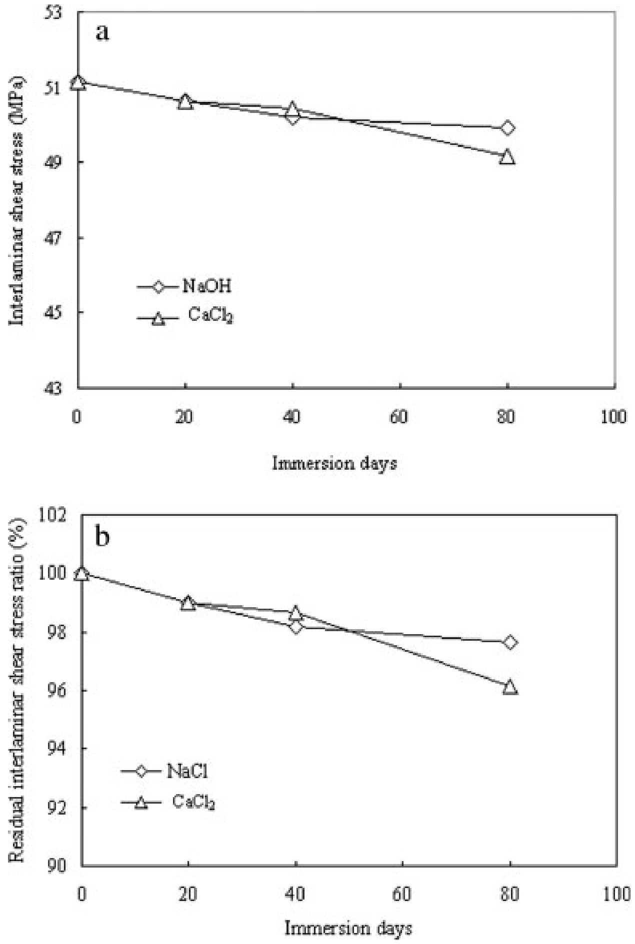


Figure 5 Interlaminar shear properties of GFRP dowel bar after immersion in NaCl and $CaCl_2$ solutions. (a) Interlaminar shear stress; (b) Residual interlaminar shear stress.

was measured for various gas pressures in the range 1–760 mmHg. The range of the air gap size was determined based on whether the gas was absorbed, and the size distribution of the air gap was estimated using the adsorption isotherm. The method for calculating the air gap volume was similar to that used to establish the adsorption volume. The size distribution of the pores was calculated with the Kelvin formula.

$$r_K = \frac{-2\gamma V_m}{RTl_n(P/P_0)} \quad (5)$$

where, γ is the surface tension of the nitrogen at the boiling point ($-196°C$; 8.85 ergs/cm^2), V_m is the mole volume of the liquid nitrogen (34.7 cm^3/mol), R is the amount of gas (8.314×10^7 ergs/deg/mol), T is the boiling point of nitrogen ($-196°C$), P/P_0 is the relative pressure of the nitrogen, and r_K is the kelvin radius of a pore.

SEM investigation

SEM displays or records a magnified picture on a cathode ray tube screen (Braun tube screen) by scanning electronic rays of 1–100 nm over the surface of a specimen in the *x*-*y* directions and detecting signals such as second electrons, reflected electrons, penetrated electrons, visible rays, ultra-red rays, X-rays, and internal electromotive forces. This method allows several qualitative and quantitative analyses regarding microstructural observations and element distributions. In this study, a SEM analysis was used to look into the deterioration process of GFRP by observing the deteriorated surface.

The GFRP dowel bar sample for the scanning electron microscope (SEM) analysis was prepared as follows. The 10-cm GFRP dowel bar was immersed in each solution, and then the middle 10 cm was cut out using a precise micro-diamond saw. The surfaces were then examined after polishing.

TEST RESULTS

Interlaminar shear stress

GFRP dowel bar soaked in alkaline solution lost its strength as the soaking time increased; the decrease rate of interlaminar shear stress was greater at 70°C

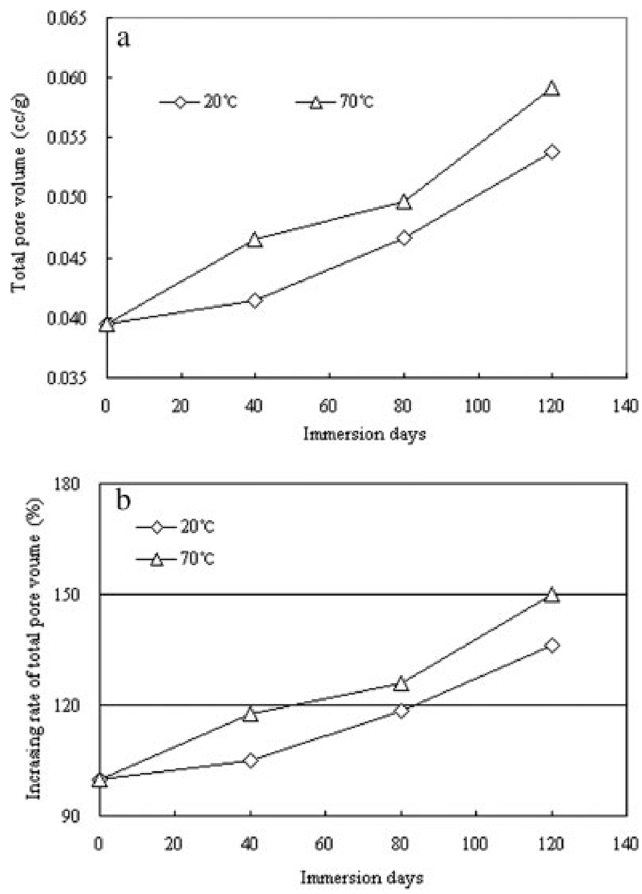


Figure 6 Gas absorption test results after alkaline solution immersion. (a) Total pore volume; (b) Increasing rate of total pore volume.

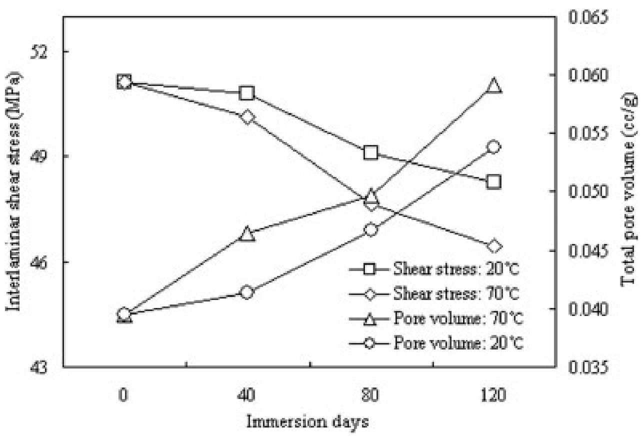


Figure 7 Total pore volume and interlaminar shear stress versus alkaline solution immersion days of GFRP dowel bar.

than 20°C (Fig. 4). When soaked in the alkaline solution at 70°C, the measured residual stress at 20, 40, 60, 80, and 120 days were 99.4, 98.1, 96.1, 93.2, and 90.9%, respectively. At 20°C, the residual stress was 99.9, 99.4, 97.3, 95.9, and 94.4%, respectively, compared with the control specimen.

The results of the interlaminar shear stress tests after soaking the specimens in NaCl and $CaCl_2$ solutions are shown in Figure 5. Exposure to both NaCl and $CaCl_2$ solutions did not have significant effects on interlaminar shear stress. The residual interlaminar shear stress in the NaCl condition at soaking times of 20, 40, and 80 days were 98.9, 98.2, and 97.7%, respectively. The values measured in the $CaCl_2$ condition were 98.9, 98.6, and 96.1%, respectively.

With the same exposure time, the interlaminar shear stress after soaking in NaCl and $CaCl_2$ solutions were compared. When soaked for 80 days, the residual interlaminar shear stress after treatment with alkaline solutions at 70 and 20°C were 93.2 and 95.9%, respectively. After 80 days in NaCl and $CaCl_2$ solutions, the residual interlaminar shear stress decreased to 97.7 and 96.1%, respectively. Accordingly, the interlaminar shear stress of the GFRP dowel bar was governed by alkalinity more than by reactions with the other solutions tested. However, the environmental influences did not appear to be important since discrepancies were not noticeable. Even so, when the bar was exposed to a high tem-

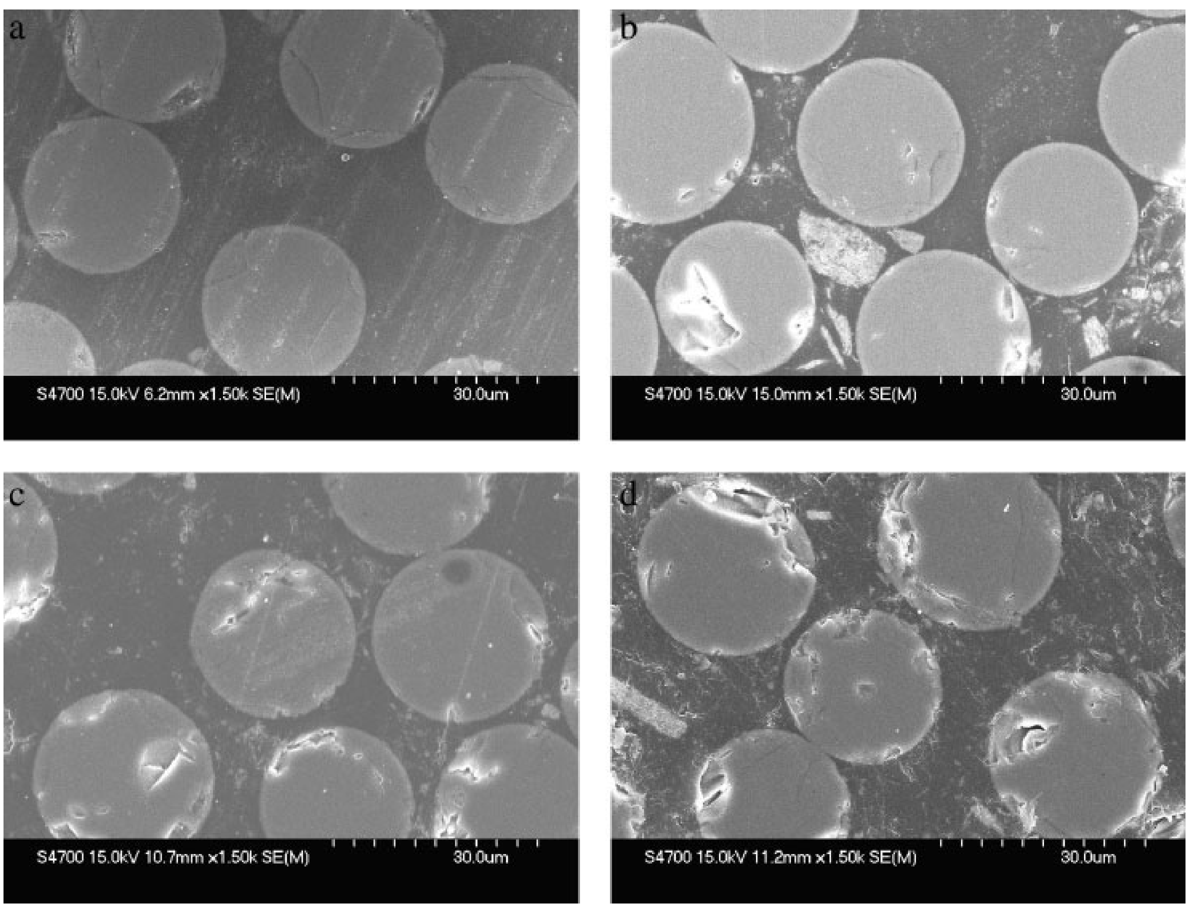


Figure 8 SEM investigation after alkaline solution immersion at 20°C. (a) Control (0 days immersion); (b) After 40 days immersion; (c) After 80 days immersion; (d) After 120 days immersion.

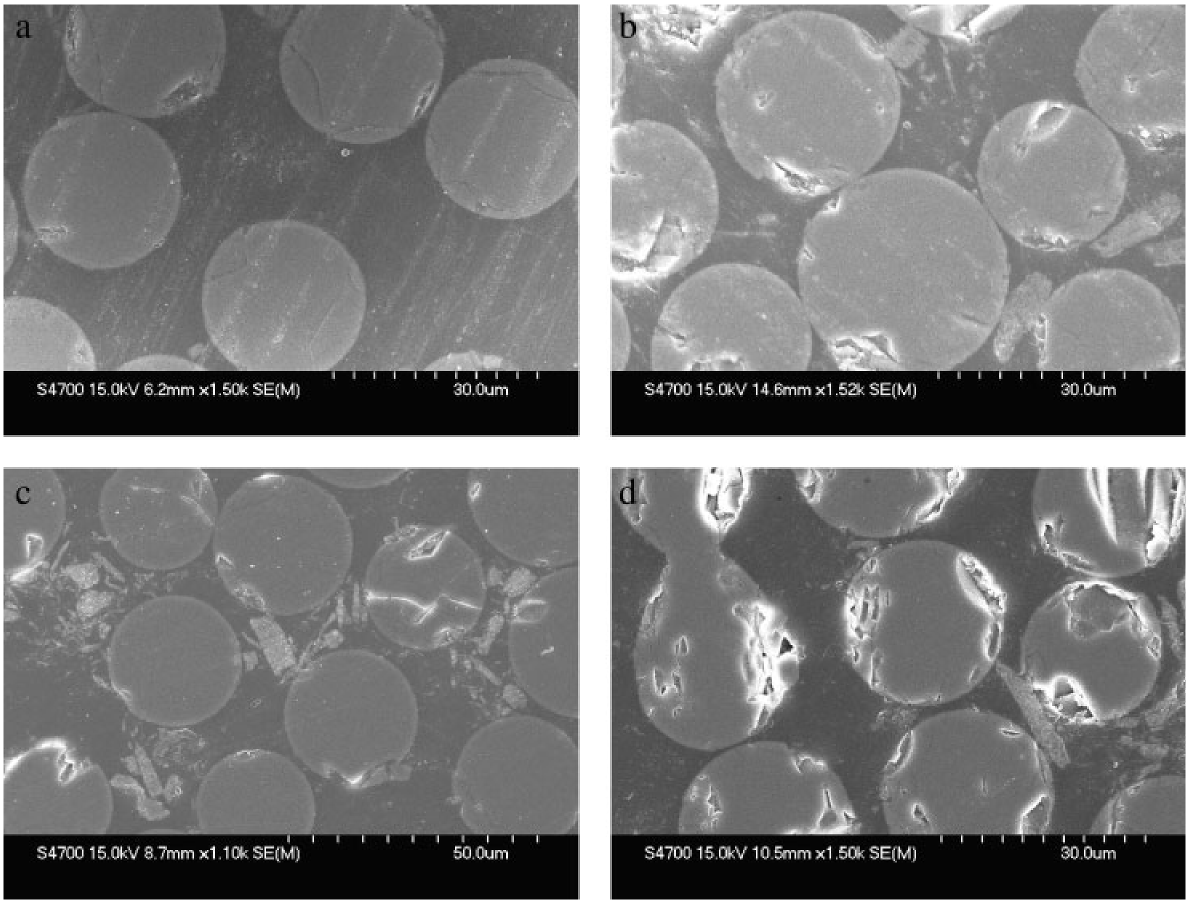


Figure 9 SEM investigation after alkaline solution immersion at 70°C. (a) Control (0 day immersion); (b) After 40 days immersion; (c) After 80 days immersion; (d) After 120 days immersion.

perature alkaline solution, the interlaminar shear stress decreased slightly. This occurred because the deterioration of the polymer matrix and interface of the glass fiber was considerable, and this affected the interlaminar shear stress without any visible impact, as the SEM images indicate.

Porosity measurements

The results of gas absorption test for porosity measurements in alkaline condition are shown in Figure 6. It was concluded that a longer soaking time led to a larger porosity; the increase in porosity was greater at 70°C than at 20°C. When soaked in alkaline solution at 70 °C, the rates of porosity increase at 40, 80, and 120 days were 17.7, 25.8, and 49.9%, respectively, compared with the control specimen. Likewise, the rates at 20°C were 4.8, 18.2, and 36.2%, respectively. Additionally, the interlaminar shear stress of the GFRP dowel bar decreased as the porosity increased. Figure 7 shows the correlation between the increase in total pore volume and the interlaminar shear stress with immersion days. The increase in porosity of the GFRP dowel bar indicated an increase in gaps between glass fibers and vinyl ester matrices; thus, the interlaminar shear stress reduction can be traced by examining porosity.

SEM investigation

Figures 8 and 9 present the results of the SEM micrograph of GFRP dowel bar after alkaline solution immersion of 20 and 70°C , respectively. As the immersion time grew longer, the glass fiber showed increasing effects, but the polymer matrix did not, and little effect was seen on the interface between the two. Since the GFRP dowel bar was exposed to the alkaline solution at 20°C, some cracks and collapses were found in the glass fibers by Day 40. By Day 80, the cracks and collapses had developed further, and deterioration became substantial by Day 120. The SEM analysis showed that the levels of deterioration at 20 and 70°C were similar, but the rate was much faster at 70°C. Deterioration of fibers and polymer resin was observed at Day 40, becoming successively more severe on Days 80 and 120.

The results of the SEM analyses and porosity measurements were similar to each other; these

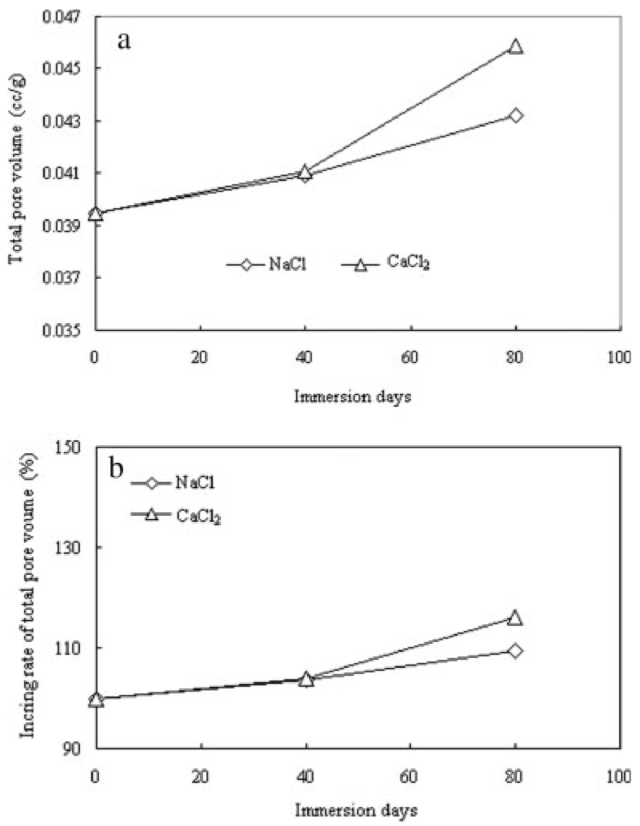


Figure 10 Gas absorption test results after NaCl and $CaCl_2$ solution immersion. (a) Total pore volume; (b) Increasing rate of total pore volume.

methods can provide clues as to the origins of decreases in the interlaminar shear stress. Figure 10 shows the total pore volume and the increases in total pore volume of the GFRP dowel bar after soaking in NaCl and $CaCl_2$ solutions, which represent sea and anti-icing agent conditions, respectively. Up to 40 days, there was no significant change in total pore volume, whereas a large change in porosity appeared by Day 80. The tendency was more distinct in $CaCl_2$ solution than in NaCl solution.

In Figure 5, which presents the results of interlaminar shear stress testing, there is no significant variation in the stress up to Day 40, but a distinct stress change was detected on Day 80. It is postulated that the internal structures of the GFRP dowel bar do not experience any major change up to Day 40 but that by Day 80, the internal total pore volume begins to increase, which leads to the decrease in interlaminar shear stress. The increase in the total pore volume of the GFRP dowel bar indicates that fractures had started to develop in the vinyl ester polymer matrices and the glass fibers. However, the result of the interlaminar shear stress decrease was not major since fractures on the interface of the glass fiber and polymer matrix were rare.

SEM results shown in Figure 11 and 12 verify this mechanism. Slight damage on the polymer matrices were observed in addition to the deterioration of the glass fibers up to Day 40 compared with the control specimen. This tendency developed more seriously after 80 days of exposure. Although the deterioration of the specimens in either NaCl solution or $CaCl_2$ solution was similar up to Day 40, longer exposure up

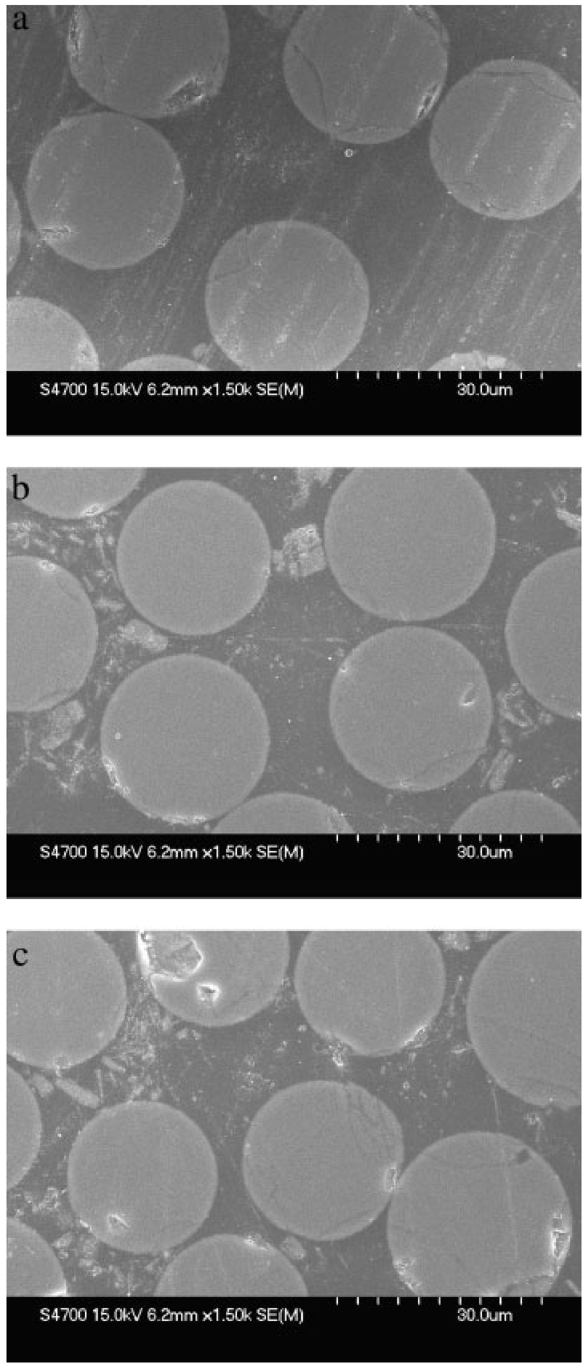


Figure 11 SEM investigation of GFRP dowel bars after NaCl solution immersion. (a) Control (0 days immersion); (b) After 40 days immersion; (c) After 80 days immersion.

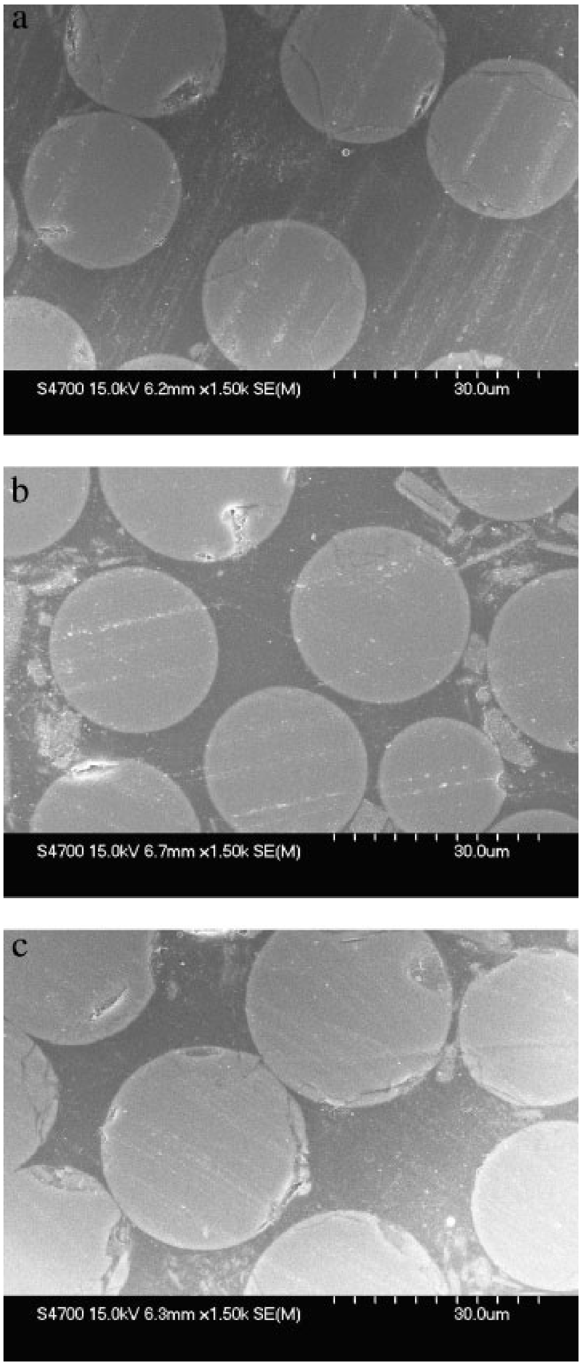


Figure 12 SEM investigation of GFRP dowel bars after $CaCl_2$ solution immersion. (a) Control (0 days immersion); (b) After 40 days immersion; (c) After 80 days immersion.

to Day 80 showed accelerated deterioration in the $CaCl_2$ solution.

SEM results after 80 days of exposure were analyzed to compare the effects of alkaline, NaCl, and $CaCl_2$ solutions. In terms of deterioration within the same time period, alkaline conditions caused more severe deterioration than sea or anti-icing agent environments (Figs. 8, 9, 11, and 12). In particular, the deterioration of the glass fibers was distinct in the alkaline solution at 70°C. Aging of the vinyl ester resin was not frequently observed in this study, although other methods of analysis such as density measurement, differential scanning calorimetry (DSC), and Fourier transform infrared spectrometry (FTIR) would be necessary for more accurate results. In other words, the penetrating hydroxyl ions did not contribute greatly to the deterioration of the vinyl ester resin, which means that little coupling occurred between the ester group molecules and hydroxyl ions. However, other methods of analysis such as density measurement, DSC, and FTIR are necessary to obtain more accurate results.

CONCLUSIONS

This study used a microstructural analysis to evaluate the deterioration when GFRP dowel bar was applied as a load transfer element in concrete pavement slabs. Exposure conditions included alkaline, NaCl and $CaCl_2$ solutions, which represented possible conditions experienced by the concrete pavement slabs. The analysis results are summarized as follows.

- The major decrease in the interlaminar shear stress after exposure of the GFRP dowel bar was evident for the alkaline solution but was not noteworthy compared with the results of exposure to the $CaCl_2$ and NaCl solutions. However, the higher temperature resulted in a lower rate of decline in the alkaline solution.
- A significant correlation was observed between the measured interlaminar shear stress and the total pore volume of the GFRP dowel bar. As the total pore volume of the bar increased, the interlaminar shear stress decreased. This was attributable to the fractures occurring in the glass fibers and polymer matrices. However, the decrease in the interlaminar shear stress was not important since fractures generated in the interface of the glass fiber and polymer matrix were rare.
- According to the SEM analysis, the fractures in the GFRP dowel bar were generated mainly at the glass fibers. The most serious damage appeared in the alkaline condition, followed by $CaCl_2$ and NaCl solutions. But, the results are not significant. In the alkaline condition, higher temperature accelerated the deterioration. Identical tendencies were observed by SEM imaging.
- The SEM analysis suggested that the deterioration of the GFRP dowel bar occurs in glass fibers rather than polymer matrices. Since the vinyl ester resin used for the polymer matrices contains ester groups, it is likely to deteriorate by reaction with hydroxyl ions. However, the vinyl

ester resin tested in the study was a commercial product designed to be used for civil engineering structures and had a minimal ester group content. Thus, the deterioration of the polymer matrices by the chemical reactions with the hydroxyl ions was not significant. Therefore, the analysis methods such as density measurement, DSC, and FTIR are necessary for more accurate results.

- A microstructural analysis on GFRP dowel bar made of E-glass fiber and vinyl ester resin revealed that the main deterioration occurred at the glass fibers. The polymer matrices by the chemical reactions with the hydroxyl ions were not significant.

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