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Durability of Glass Fiber Reinforced Cement Composites:

Effect of Silica Fume and Metakaolin

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The primary concern for glass fiber reinforced cement composites (GFRC) is the durability of glass fibers in the alkaline environment of cement. Despite the use of improved alkaline-resistant glass fibers (AR-glass) and pozzolanic materials such as silica fume and fly ash, durability concerns still exist. This report presents an experimental investigation on the hot-water durability of glass fiber reinforced cement composites. Hot-water durabilities of AR-glass fiber reinforced composites in blended cement matrix were compared for their flexural and tensile performance. The different matrices selected were (a) cement; (b) cement + 25% metakaolin; and (c) cement + 25% silica fume. Specimens after normal curing of 28 days were immersed in a hot water bath at 50°C for up to 84 days and then tested in flexure and tension. The results indicate that the blended cement consisting of synthetic pozzolan metakaolin significantly improves the durability of GFRC composite. ADVANCED CEMENT BASED MATERIALS, 1997, 5, 100–108. *© 1997 Elsevier Science Ltd.*
KEY WORDS: Cement, Composite, Durability, Flexure, Glass fibers, Metakaolin, Pozzolan, Silica fume, Tension, Toughness

Durability of glass fibers in the alkaline environment of cement is a major concern [1–5]. Despite the use of improved alkaline-resistant glass fibers (AR-glass) and pozzolanic materials such as silica fume and fly ash, durability concerns still exist. The long-term properties of glass fiber reinforced cement (GFRC) composites have shown a reduction in strength and ductility with time. The cement hydration in GFRC composites, immersed in water or exposed to weathering, results in lime crystals and calcium silicate hydrates (CSH) penetrating the fiber bundles, filling the interstitial spaces between glass filaments, thereby increasing the bond to individual glass filaments. This embrittlement results in lack of ductility of GFRC composites with aging [6].

Pozzolanic materials such as silica fume and fly ash react with lime, both neutralizing it and creating a cementitious binder that itself further improves the matrix strength. However, silica fume with very fine particle size (<0.1 μm) may penetrate the filament space, and the free lime deposition mechanism is exchanged for even harder, more damaging CSH within the fiber bundle.

Research has indicated that when synthetic pozzolan metakaolin (a calcined china clay produced by the thermal activation of kaolin in air at 750–800°C [$2SiO_2$, Al_2O_3] is used, lime entirely disappears by 28 days [7]. Metakaolin particles are also perhaps too large to penetrate the interstitial spaces of the fiber. Thus, addition of metakaolin may result in significant improvements in long-term properties. This study was conducted to examine this possibility.

Experimental Program

Test Series and Mix Proportions

Table 1 presents the details of mix ingredients and their proportions. Higher amounts of water superplasticizer, and polymer were necessary for silica fume and metakaolin matrix to achieve comparable workability. Specimens were fabricated at Pont-à-Mousson SA, France, and were tested at the ACBM Center, Northwestern University, U.S.A. The test series included: (a) plain mortar, PLAIN; (b) control, GFRC (C); (c) metakaolin, GFRC (MK); and (d) silica fume, GFRC (SF). The cement used was a U.S. white portland cement, the silica fume was Elkem air densified microsilica conforming to ASTM C-1240, metakaolin was Englehard Metamax conforming to ASTM C-618, the polymer was Forton acrylic polymer, the superplasticizer was Sikament 10, and the AR-glass fiber was Cem-FIL. Spray-up process was used to obtain GFRC samples. The matrix mix ingredients were mixed in a high shear mixer. The mortar and chopped glass fibers were then sprayed into a mold in four crossed layers. The composite was then roller compacted to ensure compliance with the mold

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TABLE 1. Details of test series and mix proportions

Mix Ingredients	Test Series			
	PLAIN (kg)	GFRC (C) (kg)	GFRC (MK) (kg)	GFRC (SF) (kg)
Cement	100	100	100	100
Sand	100	100	100	100
Metakaolin	—	—	25.0	—
Silica fume	—	—	—	25.0
Water	27.2	27.2	44.0	45.9
Polymer	9.8	9.8	12.3	12.3
Superplasticizer	1.8	1.8	3.0	3.0
Glass fibers	—	5.0% by weight of composite	5.0% by weight of composite	5.0% by weight of composite

face, impregnation of the fiber by slurry, and removal of entrapped air. The rolled surface was finally troweled smooth. The sample boards were demolded after 24 hours. The specimens were cut, stored in a controlled chamber at 20°C and 50% relative humidity (RH) for 9 days, and then sent from Pont-à-Mousson SA, to the ACBM Center, where they were stored at 20°C and 100% RH for 28 days. One set of specimens (minimum of three per test) was tested for flexural and tensile performance before being exposed to hot water aging.

At least three specimens were tested for flexural and tensile performance for a given test and aging condition. Additional specimens were tested when necessary to derive statistically reliable results.

Hot Water Aging

To evaluate the long-term performance of GFRC composites, the specimens after normal curing of 28 days were immersed in a hot water bath. The temperature of the water bath was maintained at 50°C. One set of specimens was taken out after 28 days and the other after 84 days of hot water aging. These sets were tested for flexural and tensile performance.

Flexural Tests

The flexural test (third-point loading) was performed according to ASTM C-947 [8]. Figure 1 shows the flexural test set-up. The support anvils were designed to eliminate any possible torsion in the specimen; in addition, both the supports and the two loading points were designed to eliminate any possible in-plane forces. Thus, only the vertical-line loads are applied to the specimen. A digital closed-loop hydraulic testing machine (MTS) was used for flexural tests. The load-point as well as mid-point deflections were measured. The mid-point deflection was used as a feed-back signal to accurately obtain the load-deflection behavior. The test was continued until the post-peak load reached 5% of the maximum load. The dimensions of the specimen were approximately 50 mm × 10 mm × 225 mm. The actual dimensions of the specimen were measured and used in all the calculations. All the specimens were tested with the "mold" face down on the two bottom supports.

The bend-over point corresponds to the point where the linearity of the load-deflection curve changes. Stress and strain corresponding to the limit of proportionality (LOP) and failure (MOR) were calculated according to ASTM C-947 and the European code [9]. The modulus of elasticity in flexure was calculated based on the deflection and load values at the bend-over point as well as one third of the maximum load point. The toughness of the specimen was obtained based on the area under the load-deflection curve up to the point where the post-peak load reached 5% of the maximum load.

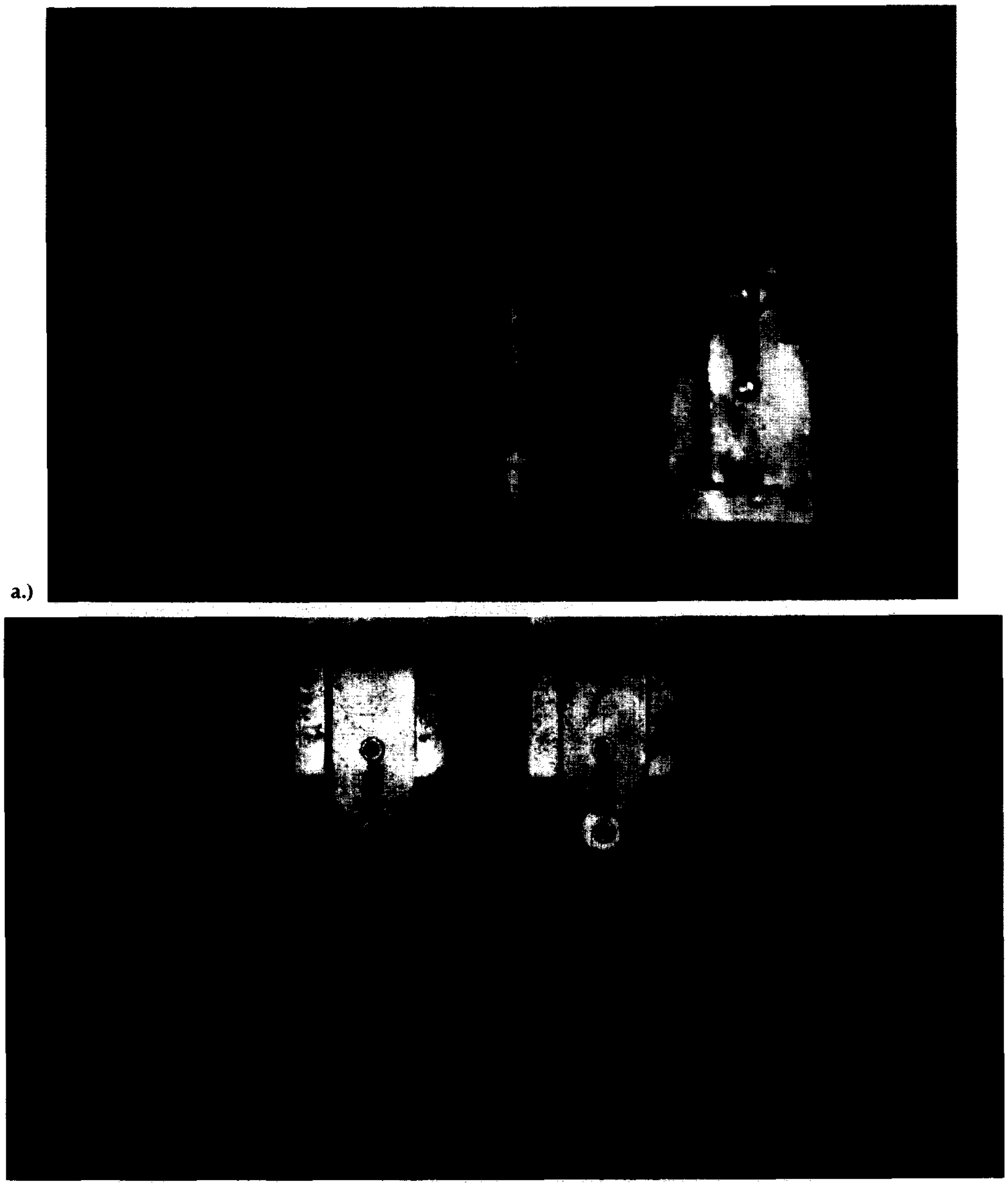
Tensile Tests

The uniaxial tensile test was performed on notched specimens using a digitally controlled closed-loop hydraulic testing machine (MTS). This test provides a better understanding of the material behavior. The provision of notches is helpful in obtaining a stable post-peak response [10]. The specimen dimensions were approximately 25 mm × 10 mm × 225 mm. The ends of the specimen were mounted with thin steel plates on either side to make them stronger and avoid any damage due to the grips. Two notches of length 6.25 mm were cut from either side (left and right) at the mid-point of the specimen. The crack mouth opening displacement was then used to measure the displacement. Two extensometers of 12.5-mm gauge length were used to measure the displacement, and the average displacement was used as a feed-back signal to obtain steady post-peak behavior. Figure 2 presents the test set-up for direct tensile testing.

Results and Discussions

Flexural Performance

Figure 3 presents the aging effects on the flexural load-deflection behavior for PLAIN and different GFRC



a.)

b.)

FIGURE 1. Flexural test set-up. (a) Specimen before loading. (b) Specimen at failure.

composites. An overall look at these graphs indicates that flexural performances of unaged GFRC composites are comparable. However, with aging, GFRC (C) and GFRC (SF) show significant deterioration, while GFRC (MK) continued to perform better. This will be discussed in detail.

Flexural stress and strain at LOP are presented in Tables 2a and 2b; and at failure in Tables 3a and 3b and Figure 4. Flexural stress and strain at MOR for unaged GFRC composites are much higher compared to PLAIN mortar. However, with aging, the behavior of GFRC composites changes depending on the matrix mix compositions. GFRC (C) and GFRC (SF) show significant reduction in flexural stress (50%–60%) and strain (75%–90%) with aging (84 days of hot water aging). GFRC (MK) performed much better and showed only a relatively minor drop in strength (15%) and toughness (20%) with aging.

It should be noted that the strains at failure are only approximate, as they were calculated based on the deflection at failure using equations valid for elastic materials (Table 2c). This is true only for linearly elastic materials. From the load-deflection curves it is quite clear that the behavior of GFRC composites is nonlinear. In addition, once cracks are formed, strains are localized and are not homogeneously distributed. As a result, calculated values of strains are dependent on the geometry of the specimen as well as the testing arrange-

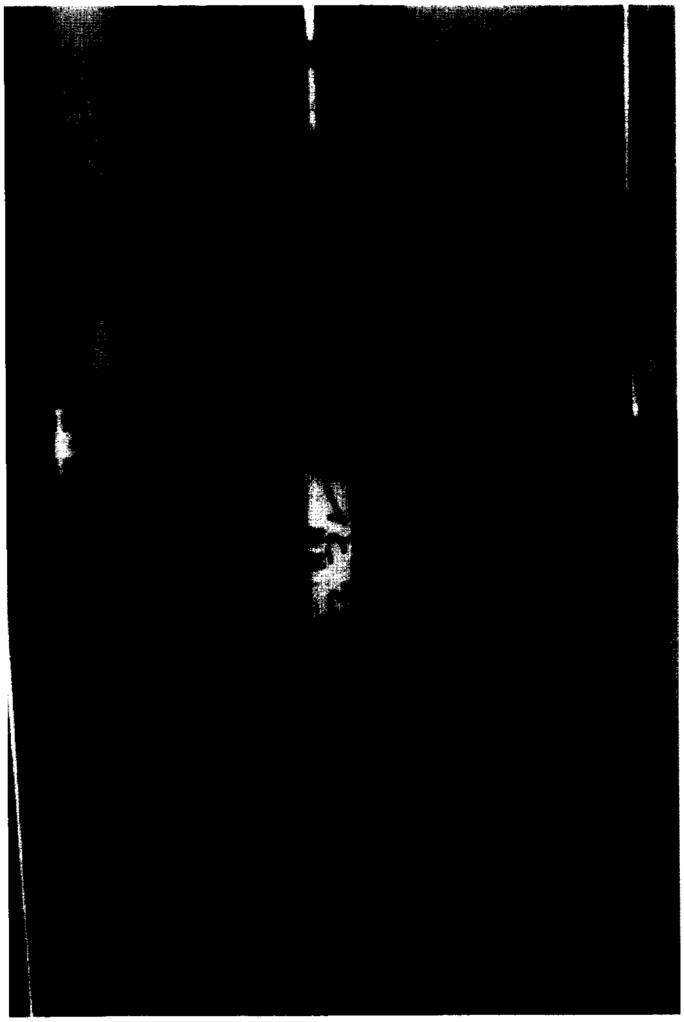


FIGURE 2. Tensile test set-up.

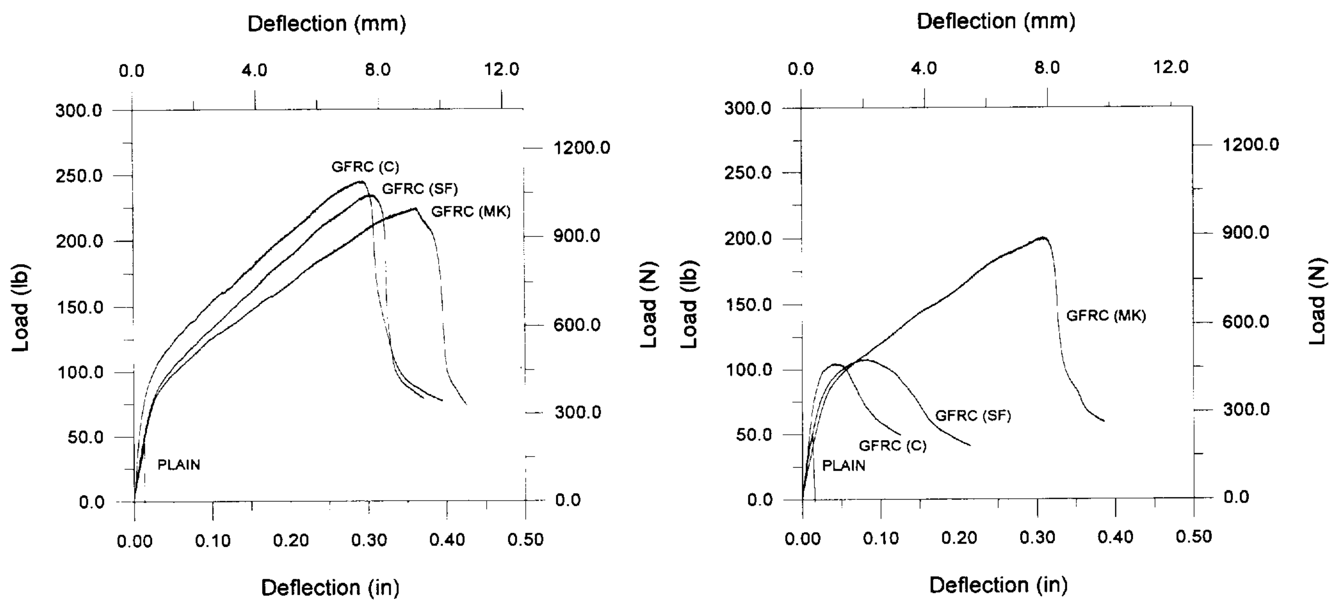


FIGURE 3. Flexural load-deflection behavior. (a) Unaged (normal curing, 28 days); (b) 84 days aged (hot water, 50°C).

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TABLE 2a. Flexural strength test results: Stress

		Flexural Strength (MPa)					
		Elastic (LOP)			Failure (MOR)		
Test Series		Unaged	28 Days Aged	84 Days Aged	Unaged	28 Days Aged	84 Days Aged
PLAIN	Average	7.44	7.89	8.31	7.44	7.89	8.31
	Std. Dev.	0.921	0.384	2.068	0.921	0.384	2.068
GFRC (C)	Average	13.60	13.76	12.72	30.15	20.21	14.91
	Std. Dev.	1.718	1.301	1.649	1.830	1.366	2.177
GFRC (MK)	Average	14.03	13.07	13.05	34.30	30.84	29.49
	Std. Dev.	1.052	0.896	4.339	1.462	1.837	6.566
GFRC (SF)	Average	10.63	12.04	9.85	28.00	20.20	12.12
	Std. Dev.	1.791	2.433	0.979	3.049	2.243	1.358

TABLE 2b. Flexural strength test results: Strain

		Flexural Strain (%)					
		LOP			MOR		
Test Series		Unaged	28 Days Aged	84 Days Aged	Unaged	28 Days Aged	84 Days Aged
PLAIN	Average	0.042	0.036	0.052	0.042	0.036	0.052
	Std. Dev.	0.0029	0.0024	0.012	0.0029	0.0024	0.012
GFRC (C)	Average	0.131	0.094	0.065	1.144	0.465	0.116
	Std. Dev.	0.0200	0.0130	0.0086	0.048	0.084	0.029
GFRC (MK)	Average	0.177	0.101	0.117	1.248	1.015	1.006
	Std. Dev.	0.0120	0.0110	0.013	0.076	0.1190	0.1300
GFRC (SF)	Average	0.150	0.165	0.119	1.275	0.646	0.294
	Std. Dev.	0.011	0.0370	0.0280	0.1120	0.1070	0.1390

TABLE 2c. Flexural strength test results: Deflection at mid-point

		Deflection at Mid-Point (mm)					
		LOP			MOR		
Test Series		Unaged	28 Days Aged	84 Days Aged	Unaged	28 Days Aged	84 Days Aged
PLAIN	Average	0.329	0.287	0.365	0.329	0.287	0.365
	Std. Dev.	0.019	0.019	0.086	0.019	0.019	0.086
GFRC (C)	Average	0.823	0.624	0.451	7.202	3.113	0.808
	Std. Dev.	0.103	0.070	0.075	0.564	0.576	0.223
GFRC (MK)	Average	1.325	0.756	0.871	9.346	7.636	7.315
	Std. Dev.	0.096	0.073	0.217	0.591	1.187	0.634
GFRC (SF)	Average	0.990	1.073	0.759	8.403	4.243	1.873
	Std. Dev.	0.032	0.177	0.165	0.468	0.632	0.854

TABLE 2d. Flexural strength test results: Modulus of elasticity

		Modulus of Elasticity (GPa)					
		Based on Bend-Over Point			Based on 33% of Maximum Load		
Test Series		Unaged	28 Days Aged	84 Days Aged	Unaged	28 Days Aged	84 Days Aged
PLAIN	Average	20.63	25.08	18.89	22.62	47.97	22.20
	Std. Dev.	2.549	1.873	3.999	3.891	15.21	5.626
GFRC (C)	Average	11.98	17.01	22.64	18.09	30.54	28.74
	Std. Dev.	1.254	1.296	3.001	1.858	2.356	3.737
GFRC (MK)	Average	9.161	14.92	12.58	13.33	18.67	15.59
	Std. Dev.	1.075	0.645	3.068	2.506	0.956	5.213
GFRC (SF)	Average	8.224	8.639	9.968	10.04	13.56	15.86
	Std. Dev.	1.850	2.052	2.458	1.924	2.876	4.095

TABLE 2e. Flexural strength test results: Flexural toughness

Test Series		Flexural Toughness (N-m)		
		Unaged	28 Days Aged	84 Days Aged
PLAIN	Average	0.035	0.039	0.055
	Std. Dev.	0.009	0.011	0.018
GFRC (C)	Average	8.670	3.207	1.123
	Std. Dev.	1.121	0.273	0.288
GFRC (MK)	Average	7.559	5.949	5.366
	Std. Dev.	0.451	0.564	0.902
GFRC (SF)	Average	7.563	3.299	1.578
	Std. Dev.	1.185	0.538	0.709

TABLE 3b. Tensile strength test results: Strain

Test Series		Tensile Strain (%)		
		Unaged	28 Days Aged	84 Days Aged
PLAIN	Average	0.040	0.004	0.005
	Std. Dev.	0.028	0.001	0.005
GFRC (C)	Average	1.788	0.560	0.375
	Std. Dev.	0.501	0.236	0.135
GFRC (MK)	Average	1.857	1.239	1.339
	Std. Dev.	0.255	0.610	0.204
GFRC (SF)	Average	1.626	1.063	0.689
	Std. Dev.	0.940	0.303	0.473

ment. It will be shown later that the values calculated from flexural specimens do not correspond to those from the uniaxial tensile test.

Flexural modulus of elasticity values of PLAIN and GFRC composites are presented in Table 2d and Figure 5. Flexural modulus of elasticity was calculated based on two methods. In the first method, the linear load-deflection curve up to the bend-over point was used. Since the load-deflection curve did not show a clear bend-over point in some cases, we decided to obtain modulus of elasticity values based on the curve up to one third of the maximum load. The values of modulus of elasticity obtained based on the load-deflection curve up to one third of the maximum load always showed higher values. However, the trends were similar. The flexural modulus of elasticity depends mainly on the amount of polymer and water present in the matrix. This is clear from the lower values of modulus of elasticity for GFRC (MK) and GFRC (SF) composites, which contained higher amounts of polymer and water.

The toughness of the specimen was obtained based on the area under the load-deflection curve up to the point where the post-peak load reached 5% of the maximum load (Figure 6). Flexural toughness values of PLAIN and GFRC composites are presented in Table 2e and Figure 7. All unaged GFRC composites showed comparable values of toughness. However, with aging, there was a reduction in toughness, but the amount of reduction depended on the matrix. GFRC (C) and GFRC (SF) showed a considerable reduction in flexural toughness (80%–90%) when aged in hot water for 84 days. The composites containing metakaolin, GFRC (MK), performed more satisfactorily. The drop in flexural toughness with aging was relatively small.

TABLE 3a. Tensile strength test results: Stress

Test Series		Tensile Strength (MPa)		
		Unaged	28 Days Aged	84 Days Aged
PLAIN	Average	0.394	0.416	0.523
	Std. Dev.	0.085	0.128	0.187
GFRC (C)	Average	12.68	5.704	4.503
	Std. Dev.	1.673	0.462	1.120
GFRC (MK)	Average	12.18	11.49	13.42
	Std. Dev.	1.110	2.911	3.133
GFRC (SF)	Average	12.29	6.988	4.627
	Std. Dev.	1.598	2.001	0.301

Tensile Performance

Figure 8 presents the aging effects on the tensile stress–strain behavior for PLAIN and different GFRC composites. Tensile stress–strain curves provide a better understanding of the material behavior. An overall look at these graphs indicates that the tensile performances of unaged GFRC composites are comparable. However, with aging, GFRC (MK) performed better compared to other GFRC composites. This will be discussed in detail.

Tensile stress and strain at failure are presented in Table 3 and Figure 9. Tensile stress and strain of GFRC composites are significantly higher compared to PLAIN mortar at all ages. This is a clear indication that the addition of glass fibers (5%) significantly improves the tensile stress-strain behavior of cement composites. However, among the different GFRC composites investigated in this research, the matrix plays a significant role on the durability of GFRC composites. GFRC composites with silica fume show moderate drops in tensile strength (43%) and strain at failure (35%) when subjected to hot water aging up to 28 days. However, with continued aging (84 days of aging), GFRC (C) and GFRC (SF) show comparable values of stress and strain at failure, which indicate that the presence of silica fume does not significantly influence the aging behavior of GFRC composites at later ages. GFRC (C) and GFRC (SF) show a significant drop (60%–65%) in tensile strength and strain at failure (55%–80%) when aged for 84 days. The significant improvement in the durability

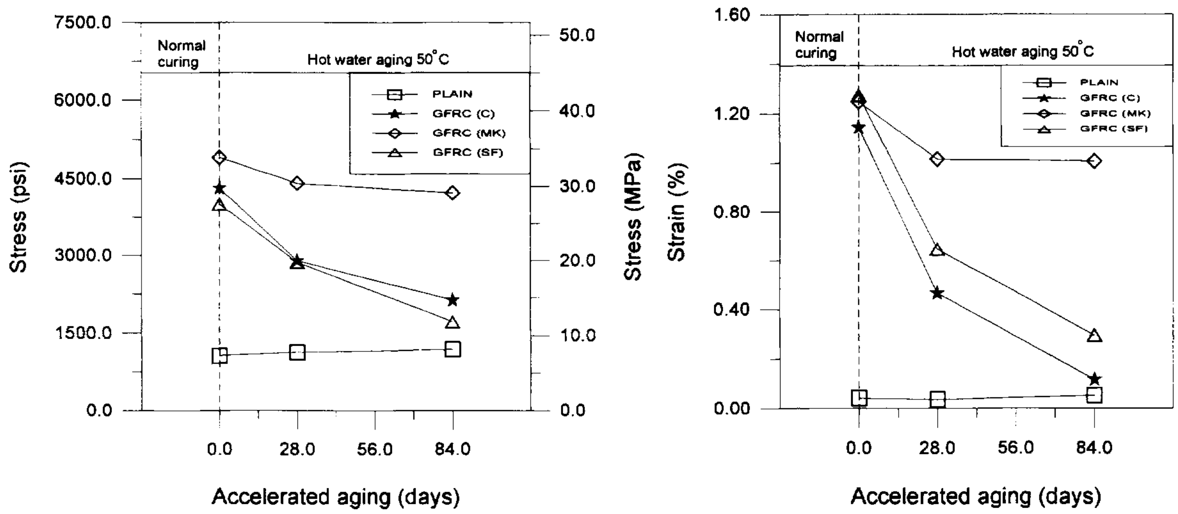

FIGURE 4. Flexural stress and strain at failure (MOR). (a) Stress; (b) strain.

of GFRC composites is provided when metakaolin is present in the matrix. GFRC (MK) showed a small drop in tensile strength (5%) and strain at failure (30%) after 28 days of hot water aging. However, with continued aging (84 days), there was no decrease in either the tensile strength or the tensile strain at failure.

The values of flexural strains (Figure 4) and tensile strains (Figure 9) are not comparable. This is because the flexural strain values were calculated based on the assumption of elasticity as recommended by the pertinent American and European standards [8,9]. However, the response of GFRC composites is not always linear. In addition, as a result of strain localization after cracking, the calculated value of strain is highly gauge dependent [10]. Even for plain concrete, only the notched tensile test can give meaningful results [11].

Summary and Conclusions

1. The addition of glass fibers to the cement matrix significantly improves the flexural and tensile behavior of cement matrix. However, the performance of these composites with aging depends on the matrix mix ingredients.

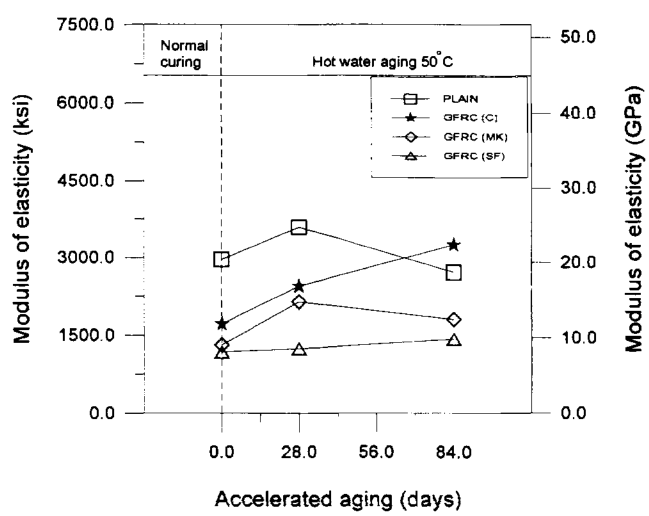

FIGURE 5. Flexural modulus of elasticity based on limit of proportionality (LOP).

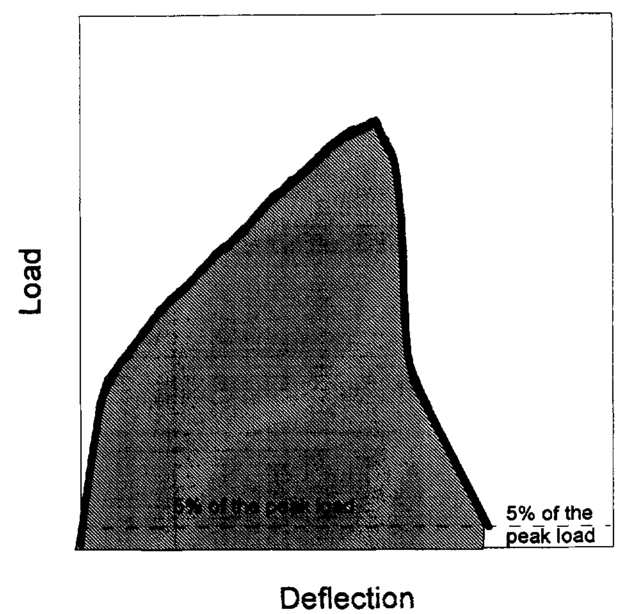

FIGURE 6. Area selected for the flexural toughness calculation.

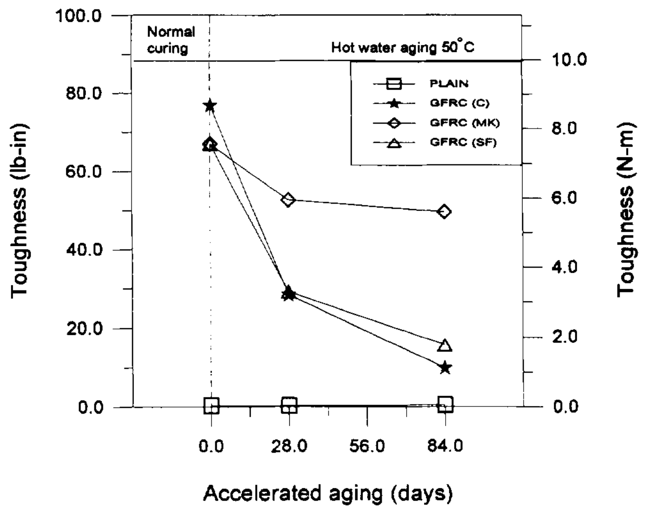

FIGURE 7. Flexural toughness based on total load-deflection curve.

2. The addition of silica fume did not improve the behavior of the composite with aging. This may be due to the fine particle size.
3. Synthetic pozzolan significantly improved both the flexural and the tensile behavior of aged GFRC composites. With aging, the GFRC composite that contained metakaolin showed only a relatively minor drop in flexural strength, strain, modulus of elasticity, and toughness. The tensile strength did not show any decrease, whereas the reduction in strain at failure was relatively small. This is a major improvement in the durability of GFRC composites.
4. The practice of evaluating ductility of GFRC composites based on elastically calculated strain values can be enormous.

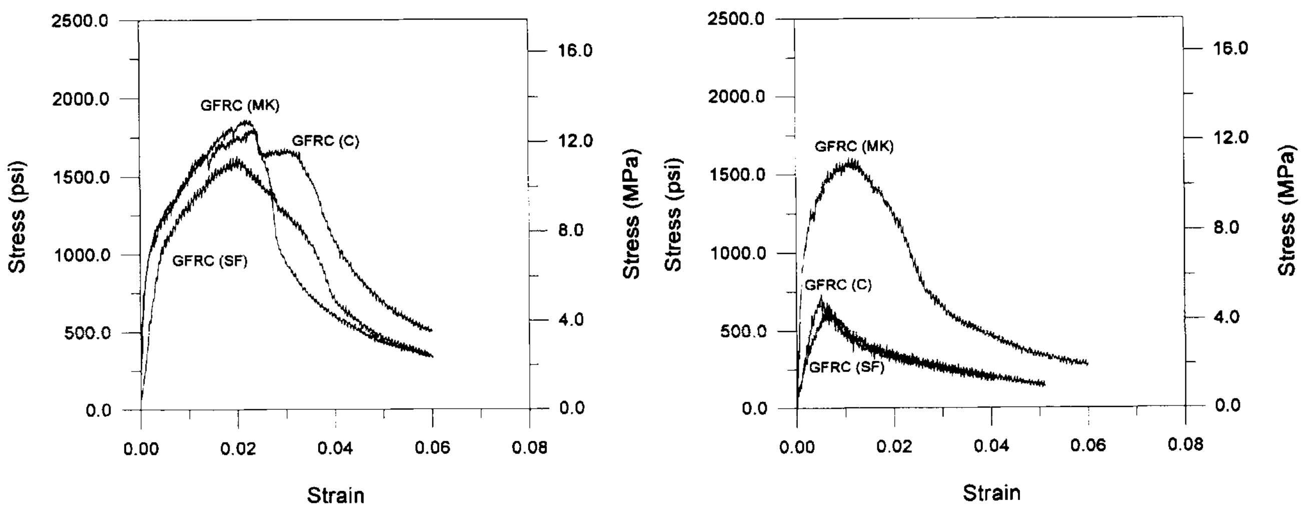

FIGURE 8. Tensile stress-strain behavior. (a) Unaged (normal curing, 28 days); (b) 84 days aged (hot water, 50°C).

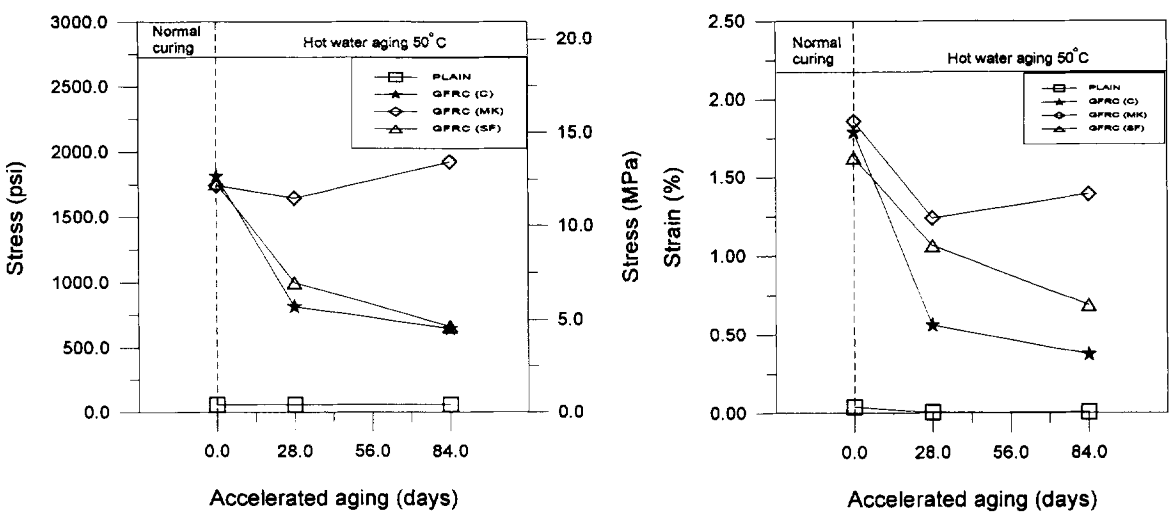

FIGURE 9. Tensile stress and strain at failure. (a) Tensile strength; (b) strain at maximum load.

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