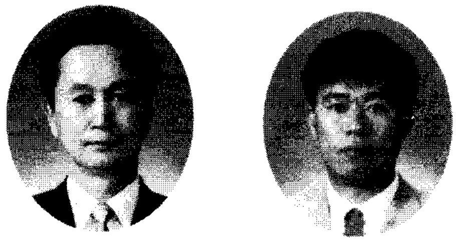
Mechanical Properties of Carbon-Fiber Reinforced Polymer-Impregnated Cement Composites



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ABSTRACT

A portland cement was reinforced by incorporating carbon fiber(CF), silica powder, and impregnating the pores with styrene monomers which were polymerized in situ. The effects of type, length, and volume loading of CF, mixing conditions, curing time and, curing conditions on mechanical behavior as well as freeze-thaw resistance and longer term stability of the carbon-fiber reinforced cement composites (CFRC) were investigated. The composite paste exhibited a decrease in flow values linearly as the CF volume loadings increased. Tensile, compressive, and flexural strengths all generally increased as the CF loadings in the composite increased. Compressive strength decreased at CF loadings above approx. 3% in CFRC having no impregnated polymers due to the increase in porosity caused by the fibers. However, the polymer impregnation of CFRC improved all the strength values as compared with CFRC having no polymer impregnation. Tensile stress-strain curves showed that polymer impregnation decreased the fracture energy of CFRC. Polymer impregnation clearly showed improvements in freeze-thaw resistance and drying shrinkage when compared with CFRC having no impregnated polymers.

Keywords : carbon fibers, cement composites, polymer impregnation, freeze-thaw, drying shrinkage

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1. INTRODUCTION

Although portland cement and its concretes have been used widely as major construction materials, problems such as long curing time, large shrinkage, and low tensile and flexural strengths, prevented its use in high-performance applications. There have been numerous attempts to improve the inherent problems in cementious materials by reinforcing them with various fibers.[1-10] Among the reinforcing fibers, carbon fibers(CF) are superior to other fibers in mechanical performance and chemical stability. These superior properties of CF are used in carbon-fiber reinforced cement matrices(CFRC) by yielding light weight, high strength, high durability and reliability, and more slender parts for construction applications. For these reasons, research and development in CFRC is now active and being paid more attention to.[11-19]

Polymer-impregnated concrete has gained wider applications, since Brookhaven National Laboratory and US Building Research jointly developed in late 1965.[20] Rio and Biagini[21] in 1975 improved concrete exhibiting compressive strengths of 282 MPa. Many papers on processing, basic science, and practical applications have been published.[22-31] More recently various high strength fibers, e.g. glasses, steel, polymers including CF, have been used in the authors laboratory.[16-18,32]

Polymer-impregnated CFRC with high strength, durability, and reliability were fabricated. The mechanical properties including drying shrinkage of the CFRC are presented in this paper.

2. EXPERIMENTAL INVESTIGATION

2.1 Materials

For CF, PAN-based CF from Korea Steel-Chemical Co.,Ltd and Pitch-based CF from Kureha, Japan, in 3, 6, 10, and 20 mm lengths(L_{CF}) were used.

The properties of the fibers are listed in Table 1.

For the matrix early strength cement from Ssang Yong Cement Co., South Korea, with silica powder from Gilae silica Co., South Korea, were used.

The properties of these materials are given in Tables 2 and 3.

For polymer impregnation styrene monomer(ST) obtained from Woosung Chemical Co., Seoul, South Korea with trimethylol propane trimethacrylate (TMPTMA) obtained from Mitubishi Rayon Co., Tokyo, Japan as the cross linking agent, azobis isobutyronitrile (AIBN) obtained from Otsuka Kagaku Co., Tokyo, Japan as the free radical initiator, and γ-methacryloxy-propyl trimethoxy silane(Dow Corning, Midland, MI, USA) as a coupling agent were used in a volume proportion of 88 : 11 : 1 : 1, respectively. The chemical formulas

Table 1 Properties of carbon fibers

Type of fibers	Fiber diameter (10^{-3}mm)	Specific gravity	Tensile strength (kgf/cm^2)	Modulus of elasticity (10^3kgf/cm^2)	Elongation (%)
Pitch-based carbon fiber	14.5	1.63	7,800	3.8	2.1
PAN-based carbon fiber	6.8	1.78	35,000	23.0	1.6

Table 2 Properties and composition of early strength cement

Specific gravity	Blaine (cm^2/g)	Setting time (h:min.)		Compressive Strength (kgf/cm^2)				SiO_2 (%)	Al_2O_2 (%)	Fe_2O_3 (%)	CaO (%)	MgO (%)	SO_3 (%)	Ig.Loss (%)	K_2O (%)	Na_2O (%)
		Initial	Final	1 day	3 days	7 days	28 days									
3.15	4,508	3:22	5:24	199	340	375	450	19.4	6.0	2.5	61.0	3.4	4.3	1.33	1.10	0.17

and physical properties of the chemicals are listed in Table 4.

Table 3 Properties and composition of silica aggregates

Chemical components (%)			Physical properties	
SiO_2	Al_2O_3	Fe_2O_3	Specific gravity	Particle size (μ)
92.5	3.81	0.51	2.60	7.1 (less than 80μ)

2.2 Mixing and Fabrication of Composites

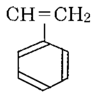
Mixing formulations of cement matrix are given in Table 5 An Omni Mixer (Chiyoda Technical & Industrial Co., Tokyo, Japan) of 30 liters capacity was used to mix cement, aggregates, admixtures and CF. The mixing times were 30 sec for dry blend, 3-5 min for a primary blend with water and admixtures, and 4.5~6 min for a secondary blend with CF in total of 10 min on average. CF were added during the secondary blend.

Curing of CFRC was begun 24 hr after casting the cement composites into different molds for different testings at 23 ±2℃ and 60±5% RH for air curing for 7 days. For autoclave curing, 180℃ and 1.01 MPa of pressure for 3hr were applied. Polymer impregnation of the cured CFRC was carried out after 28 days from the molding. The cured CFRC were contained in a pressure vessel first degassed at a pressure of 665±133 Pa for 1 hr followed by introducing ST monomer impregnant mixture given in Table 4. A pressure of 785±98 kPa was then applied to the vessel containing the CFRC for 3 hr followed by applying 1.96±0.09 MPa of pressure for 1 hr before completing the impregnation procedure. Polymerization of the impregnated ST monomers was carried out in water at 80℃ followed by air drying at 100℃ for 1 day.

2.3 Test Methods

All testing of CFRC specimens was carried out after 7 days from molding for air and autoclave cured specimens, and after 12 weeks from molding for polymer impregnated specimens. Three specimens were used for each data points. The average values within±10% are reported. Otherwise retesting using new specimens was carried out. The flow test was carried out by following the Korean Standard Method KS L 5105. Compressive and tensile specimens were prepared and tested following KS L 5105 and 5104. The dimensions of tensile specimens are 31×6×1.2cm in overall and the geometry of tensile specimen has been presented in a previous paper.[29] The compressive stress-strain relationship was obtained from cubic specimens of 5.08×5.08×5.08 cm and cylindrical specimens of 5cm diameter and 10 cm length with 3cm wire strain gauge attached to the middle of the specimen on a 100t Universal Testing Machine.

Table 4 Properties of chemical for impregnant

Type of Chemicals	ST	TMPTMA	AIBN	SILANE
Constitutional Formula		$CH_2OOC\text{-}C(CH_3)=CH_2$ $CH_3\text{-}CH_2\text{-}C\text{-}CH_2OOC\text{-}C(CH_3)=CH_2$ $CH_2OOC\text{-}C(CH_3)=CH_2$	$NC\text{-}C(CH_3)_2\text{-}N=N\text{-}C(CH_3)_2\text{-}CN$	$CH_2=C(CH_3)\text{-}C(=O)\text{-}OCH_2CH_2CH_2Si(OCH_3)_2$
Molecular Weight	103.15	337.8	165.1	249.2
Boiling Point (℃)	145.0	200℃/mmHg 185℃/mmHg	-	256
Specific Gravity (20℃)	0.910	1.065	1.128(25℃)	1.043
Flash Point (℃)	30.8	152	-	139
Viscosity (20℃, cP)	0.734	13.1	-	2.1

Tensile testing was done by using a 10t Instron Machine with a constant crosshead speed of 0.5mm/min and 3cm long wire strain gage attached on both sides of the specimen. The stress-strain curve was recorded on an X-Y recorder. Flexural strength of CFRC was determined by following the Japanese Standard Method JIS R 5201 using 4×4×16cm bar specimens under three-point loading.

Plate shaped specimens of 5×1.2×50 cm(b×t×L) were used to examine the relationship between flexural stress and deflection with 40cm span under three-point loading and 0.5mm/min crosshead speed.

Freeze-thaw resistance of CFRC was determined by following the rapid freeze - thaw method of ASTM C 666-2 under the temperature ranging from -18℃ to +10℃ of six to eight cycles in 1 day up to 300 cycles. For long-term changes of CFRC, drying shrinkage was determined by using 4×4×16 cm specimens and ASTM C 157 Comparator Method for CFRC cured under different conditions.

3. RESULTS AND DISCUSSION

3.1 Flow Values and Cf Loadings

As shown in Fig.1, the flow values decreased with increasing amounts of the short CF in the cement matrix. PAN CF showed slightly sharper reduction in flow values. This is believed to be due to the fact that PAN CF have about twice larger aspect ratio than that of Pitch CF. This agreed with the results that the longer the CF, the lower the flow values in Fig.1. This is because the longer the fibers, the more difficult to disperse because of the greater tendency for fiber-balling.

3.2 Compressive Strength and Modulus of Elasticity

Compressive strengths and moduli of CFRC formed by water to cement ratio (W/C) of 0.5, silica powder to

Table 5 Mixing proportion of cement matrices for CFRC

W/C (%)	S/C	CF Content (vol. %)	Unit weight (kgf/m^3)						
			Water	Cement	Silica powder	Carbon Fiber		Admixtures	
						Pan	Pitch	SP*	MC**
50	0.5	0	989	495	495	-	-	9.9	4.9
		1	979	490	490	16.3	17.8	9.8	4.9
		2	970	485	485	32.6	35.6	9.7	4.9
		3	960	480	480	48.9	53.4	9.6	4.8
		4	950	475	475	65.2	71.2	9.5	4.8
		5	940	470	470	81.5	89.0	9.4	4.7

* Superplasticizer(Mighty 150), ** Methy Cellulose(Viscosity 4,000cp)

cementratio(S/C) of 0.5, and using 10mm CF are given in Figs. 2 and 3 as well as listed in Table 6. The strengths and moduli did not increase with increase in the CF volume in the composites regardless of the kind of CF and curing method. However, the strengths clearly increased by approx. 1.8~2.3 times by impregnation of the polymer depending on the kind and volume loading of CF. The PAN-CF-reinforced composites exhibited superior properties to the Pitch CF composites, due to the superior strength of the as received PAN CF(Table 1). Pitch-based CF has a lower strength as well as a lower modulus because of its isotropic nature of the polymer chains in the fiber. However, isotropic Pitch CF are promising in CFRC applications because of their lower cost than PAN CF.

As shown in Fig.3, the compressive moduli decreased as the CF volume loading increased especially V_f above 3% for both air and autoclave curing. On the

Table 6 Mechanical properties of CFRC with W/C=0.5, S/C=0.5, and L_{CF}=10mm

Type of fiber	Fiber Volume (%)	Compressive strength* (kgf/cm^2)			Compressive modulus of elastic (kgf/cm^2)			Tensile strength (kgf/cm^2)			Flexural strength (kgf/cm^2)		
		Polymer impregnated	Auto-claving	In air	Polymer impregnated	Auto-claving	In air	Polymer impregnated	Auto-claving	In air	Polymer impregnated	Auto-claving	In air
PAN based CF	0	865(826)	466(390)	437(339)	2.32	1.43	1.23	77	47	39	180	115	84
	1	987(831)	468(387)	436(337)	2.33	1.52	1.24	115	74	56	352	221	182
	2	874(840)	470(385)	432(335)	2.32	1.42	1.21	141	87	64	430	255	229
	3	872(812)	462(383)	412(323)	2.29	1.34	1.18	149	99	67	484	279	238
	4	840(773)	437(355)	364(291)	2.27	1.28	1.09	157	97	64	492	273	233
	5	809(736)	383(302)	309(251)	2.28	0.99	0.76	160	90	59	498	248	205
Pitch based CF	0	865(826)	466(390)	437(339)	2.32	1.43	1.23	77	47	39	180	115	84
	1	870(830)	467(386)	414(316)	2.31	1.33	1.16	110	65	52	329	201	163
	2	866(819)	454(367)	394(306)	2.25	1.31	1.13	126	77	58	412	242	212
	3	861(789)	433(363)	364(282)	2.24	1.25	1.08	138	82	62	450	262	220
	4	814(743)	411(325)	328(249)	2.20	1.14	0.86	144	82	58	456	242	208
	5	750(685)	333(282)	263(220)	2.13	0.84	0.62	149	75	46	462	218	184

* Value in parentheses are from cylinder samples of 5×10cm

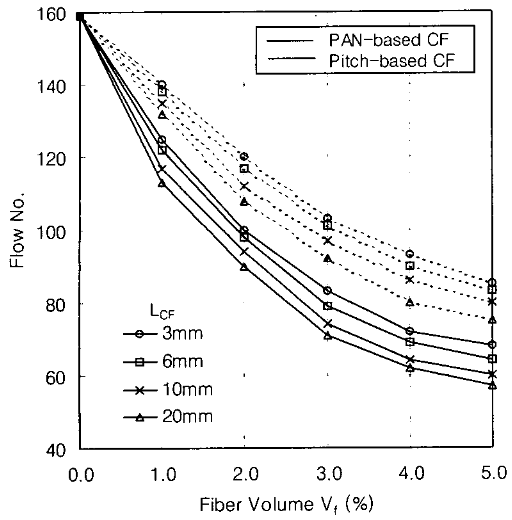


Fig.1 Flow values of CFRC as a function carbon fiber loading.

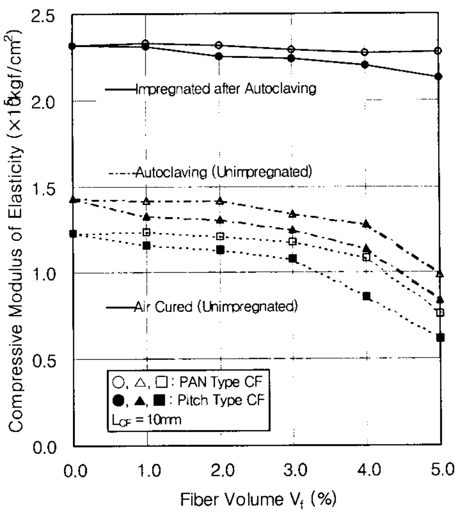


Fig.2 Compressive strength of CFRC versus fiber volume loading.

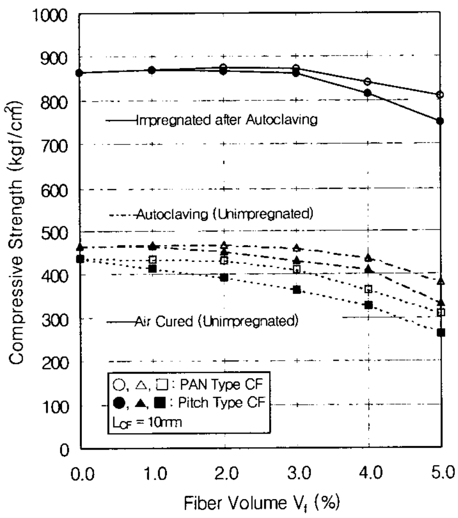


Fig.3 Compressive moduli versus fiber volume loading.

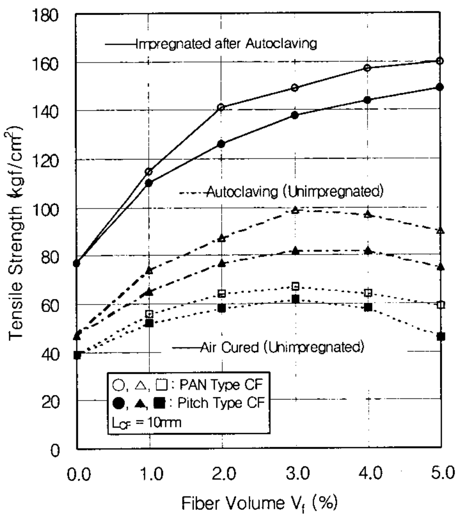


Fig.4 Tensile strength versus fiber volume loading

other hand, the impregnated polymer is shown to be effective for maintaining high modulus values with increasing the fiber loading. This must be from me reduction of the porosity of the CFRC by the impregnated polystyrene.

3.3 Tensile Behavior

Tensile strengths of CFRC having W/C=0.5, S/C=0.5 and L_{CF}=10mm as a function of CF volume loading is given in Fig.4 as well as in Table 6. The strength increased as the fiber volume loading increased in all cases as expected. Autoclave-cured CFRC exhibited higher tensile strengths than air-cured CFRC. The higher tensile strength with PAN CF

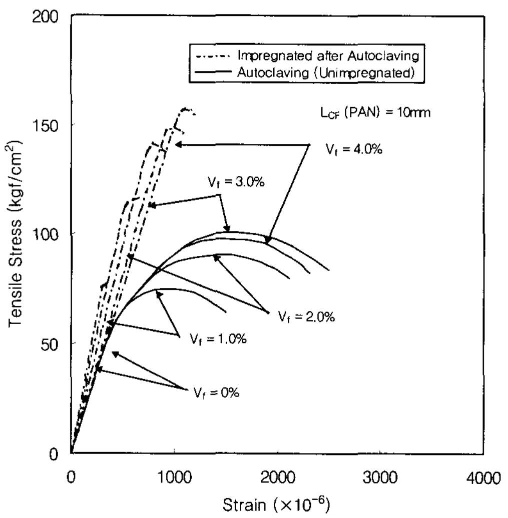


Fig.5 Tensile stress-strain curves of CFRC with 10mm PAN CF.

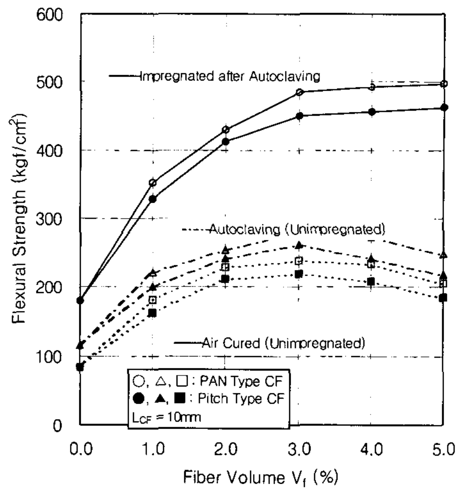


Fig.6 Flexural strengths versus CF volume loading.

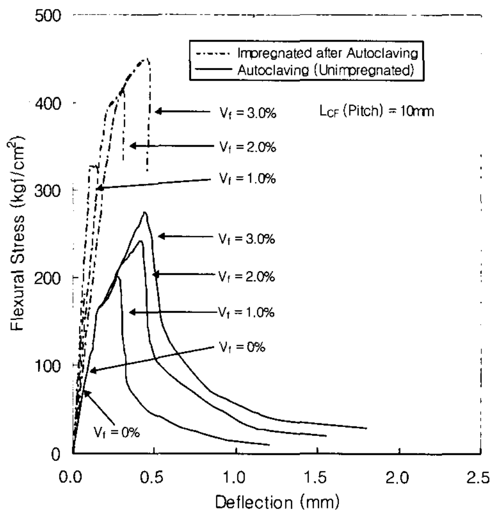


Fig.7 Flexural stress-deflection curves of CFRC with 10mm Pitch CF.

than with Pitch CF was also expected since PAN CF itself has a higher tensile strength and a higher aspect ratio than Pitch CF. Polymer impregnation after the autoclave curing increased the tensile strength by approx. 1.5 times to 149 kgf/cm^2 and by approx. 2.4 times from the air-cured CFRC at $V_f=3\%$. The tensile strength decreased when the V_f increased above 3% in the case of CFRC with no impregnated polymer. While the CFRC with impregnated polymer maintained the increasing trend of tensile strength as the CF volume loading increased up to 5%. As observed in a previous study[32], the increased fiber loading increased porosity as well as the increased inhomogeneous dispersion of the fiber in the matrix.

The increasing trend of tensile strength amounts of CF in the matrix increased the tendency for the fibers to agglomerate increased. As the amount of CF in the matrix increased, the tendency for the fibers to agglomerate increased. This tendency was also increased for increasing the fiber length but did not affect the standard deviations of the property values. It is believed that the impregnated polymers reduced the porosity of CFRC and increased the interfacial bounding between the fibers and the matrix.

As shown in Fig.5, polymer impregnation caused higher proportionality limit but much less ductility than the unimpregnated CFRC with PAN CF. The

Table 7 Test Results of Freeze-Thaw Resistance of CFRC

Test Series \ Cycles		0	10	30	50	70	90	110	130	150	170	190	210	230	250	270	300
Polymer impregnated	(a)	100	99.3	100.2	98.9	101.3	100.8	100.7	100.1	99.8	99.5	100.0	100.7	98.8	98.1	99.3	99.6
	(b)	100	99.4	99.3	98.8	98.7	98.3	98.0	97.5	97.4	97.9	97.8	98.4	98.9	99.0	98.5	97.3
Autoclaving	(a)	100	98.6	98.3	97.5	97.2	98.3	97.9	97.6	97.1	97.4	96.8	97.0	96.7	97.0	95.4	95.8
	(b)	100	98.4	97.4	97.0	96.7	96.5	96.0	94.9	94.7	94.9	95.2	95.1	95.0	95.2	94.7	94.2
In air	(a)	100	97.2	94.9	93.0	92.8	92.0	91.8	90.5	88.9	89.2	88.7	88.0	87.7	87.3	86.5	84.7
	(b)	100	97.6	95.8	94.0	92.3	91.2	89.5.	88.7	89.6	90.2	90.3	89.1	90.6	91.2	91.5	91.7

(a) Change of Relative Modulus of Elasticity(%), (b) Change of Weight(%)

Pitch-CF-reinforced CFRC showed similar stress-strain curves to the ones in Fig.5 except that with much lower tensile strength values (Table 6).

This proves a strengthening effect of the interfacial bonding between fibers and matrix by the impregnated polymers. It was also observed that much less fiber-pull out in fractured specimens of the polymer impregnated CFRC than in CFRC with no impregnated polymers.

3.4 Bending Behavior

Flexural strengths of CFRC are given in Table 6 and Fig.6. The strength increased as the V_f increased up to 3 vol.% of CF, then decreased in the case of CFRC without the impregnated polymers. For the polymer-impregnated CFRC, the strength continued to increase beyond 3 vol.% CF up to 5%. PAN CFRC show higher strengths than Pitch CFRC as in the other strength values discussed previously.

The cause of the decrease in flexural strength at fiber loading above 3% must be the same reason as for the tensile strength in the previous section. Polymer impregnation again improved the flexural strength markedly, approx. 1.6~2.5 times higher, by reduction in the porosity of CFRC and increase in the interfacial bonding between the fibers and the matrix.

This helped maintaining the increasing trend of the flexural strength beyond the 3% CF loading. Flexural stress-strain curves given in Fig.7 reveal that polymer impregnation reduced the fracture energy of the CFRC. This agrees well with the tensile behavior shown in Fig.5 and shows gain in strength sacrificed in toughness.

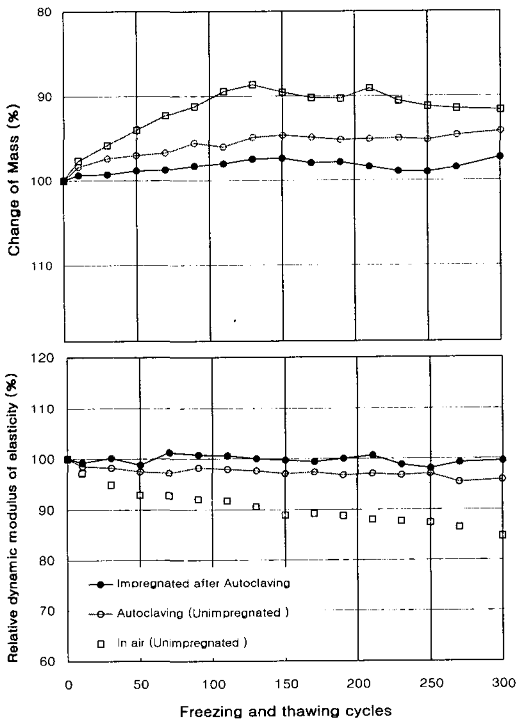


Fig.8 Change of mass relative dynamic moduli of elasticity as a function of freeze-thaw cycles for CFRC with 10mm Pitch CF at 3% loading.

3.5 Freeze-Thaw Resistance

Changes in mass and relative dynamic modulus of elasticity of CFRC as a function of freeze-thaw cycles up to 300 cycles are given in Table 7 and Fig.8 For polymer impregnated CFRC, approx. 1% decrease in modulus and approx. 3% reduction in mass at 300 cycles. This is a clear improvement of freeze-thaw resistance by the polymer impregnation. Air-cured CFRC showed the worst freeze-thaw resistance by exhibiting approx. 15% reduction in modulus and approx. 8% reduction in mass at 300 freeze-thaw cycles. This demonstrates the effectiveness of polymer impregnation in increasing freeze-thaw resistance of CFRC which is consistent with the other mechanical properties discussed previously.

3.6 Drying Shrinkage

Table 8 and Figs. 9~11 show the linear shrinkage of CFRC as a function of air-drying time for CFRC with various fiber loadings, kind of CF, and curing methods. As the CF loading increased, the drying shrinkage decreased in general. PAN CFRC exhibited somewhat smaller drying shrinkage than Pitch CFRC as the fiber loading increased. This is believed to be caused by a twice larger aspect ratio of PAN CF than Pitch CF. This agrees with the lower flow values by PAN CFRC in Fig.1.

Polymer impregnation is again shown to be effective in reducing the drying shrinkage by approx. 27~50%. at 6months Autoclave curing yielded smaller shrinkages by approx. 8.6~13.9 times than air curing at 6 months as shown in Figs. 11 and 12 Shrinkage of a CFRC cured by autoclaving followed by polymer impregnation exhibited only $1.5\sim2.8\times10^{-5}$ in 6 months of aging. This demonstrates the effectiveness of autoclaving and polymer impregnation for obtaining dimensionally stable CFRC.

Table 8 The Results of Drying Shringkage of CFRC ($\times10^{-5}$)

Test Series		VCF (%)	Testing Ages(days)							
			1	3	7	14	28	60	90	180
Polymer Impregnated after Autoclaving	Plain	0	10.1	13.1	13.7	14.9	18.1	22.5	24.3	27.5
	PAN -CF	1	6.7	8.8	9.9	11.5	13.3	14.6	15.8	17.0
		2	5.8	7.0	7.7	9.2	11.6	13.1	14.0	15.5
		3	2.1	4.1	5.1	5.9	7.0	7.9	8.6	9.6
	Pitch -CF	1	8.4	9.9	10.8	12.6	14.0	17.6	20.2	22.2
		2	7.0	8.6	14.9	11.3	13.1	14.3	15.3	16.5
		3	4.7	6.8	9.5	10.5	11.7	12.6	13.8	15.1
Cured in Autoclave	Plain	0	11.7	15.7	18.5	20.7	24.8	31.3	33.8	40.3
	PAN -CF	1	6.4	13.1	17.3	19.9	22.3	27.9	31.3	33.8
		2	5.6	8.7	10.1	12.2	15.3	17.3	18.8	21.3
		3	4.1	6.8	7.8	7.5	11.7	14.1	16.6	18.7
	Pitch -CF	1	8.8	14.5	16.7	18.1	22.1	25.4	29.6	32.4
		2	7.4	12.1	13.7	15.7	17.6	21.4	24.7	29.0
		3	5.7	8.3	9.6	11.1	13.4	16.6	19.1	22.3
Cured in Air	Plain	0	68.4	105.5	143.7	190.4	216.5	267.6	306.6	344.9
	PAN -CF	1	54.3	95.0	129.1	162.0	189.5	225.0	243.4	297.2
		2	36.8	80.3	180.3	135.7	162.0	213.2	235.2	284.4
		3	33.9	60.8	84.9	106.4	146.9	189.3	216.0	256.8
	Pitch -CF	1	58.7	103.3	138.2	168.9	203.9	234.3	257.1	320.6
		2	48.3	88.5	102.3	143.9	167.1	221.2	249.8	283.5
		3	37.4	74.1	116.1	137.9	187.8	211.5	234.2	271.4

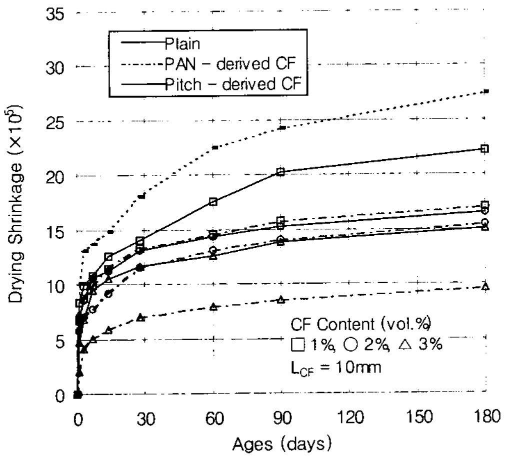


Fig.9 Relation between drying shrinkage and drying ages of CFRC impregnated in polymer after autoclaving

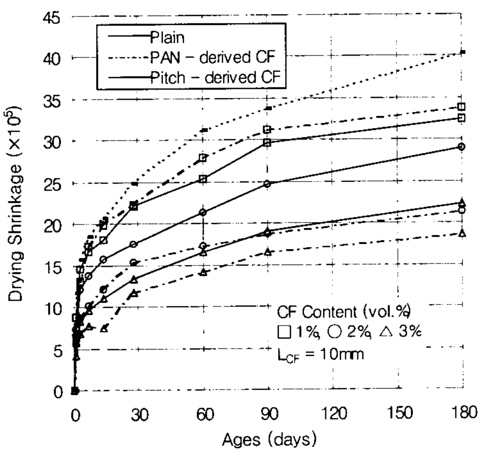


Fig.10 Relation between drying shrinkage and drying ages of CFRC for cured in autoclave

4. CONCLUSIONS

From this study, it was learned that the interfacial bonding between the fibers and the matrix as well as the curing condition influence the physical and mechanical properties of CFRC. The findings of this study are summarized below.

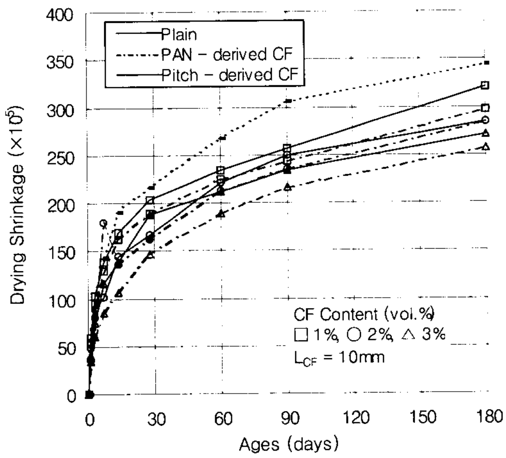


Fig.11 Relation between drying shrinkage and drying ages of CFRC for cured in air

(1) The flow values of CFRC decreased in a regular manner as the amount of CF increased as well as the increasing aspect ratio and the length of CF.

(2) The compressive strength decreased slightly by increasing addition of CF in CFRC. However, polymer impregnation increased the compressive strength by approx 1.8~2.3 fold as compared with unimpregnated CFRC. Essentially, the same behavior is shown for compressive modulus of elasticity of CFRC, except for those CFRC impregnated with polystyrene which exhibited virtually constant modulus of elasticity as the fiber volume loading increased.

(3) The tensile strength of CFRC increased up to 3-4% CF loading above which the strength decreased for CFRC having no impregnated polymers. The polymer impregnated CFRC did not show me decrease of tensile strength at CF loading above 3% up to 5% but showed approx. 1.5~3.2 fold higher strength than

CFRC having no impregnated polymers. PAN CF was more effective in increasing all the strengths (tensile, compressive, and flexural) than Pitch CF. Polymer impregnation is very effective in obtaining high-strength CFRC. The tensile stress-strain relationship of CFRC showed polymer impregnation of CFRC extended the linear limit to approx. 90% of the ultimate tensile strength. The high tensile strength of polymer impregnated CFRC was a compromise with a reduction in ductility of CFRC as compared with autoclave-cured CFRC.

(4) Flexural strength of CFRC increased as V_f increased up to approx. 3%. At CF above 3%, the strength decreased up to 5%. CFRC with PAN CF showed 4~14% higher flexural strength than those with Pitch CF. Polymer impregnation increased the strength of CFRC at V_f above 3% to 1.6~2.5 times those of unimpregnated CFRC.

(5) Polymer impregnation of CFRC reduced the weight change and deterioration due to freeze-thaw cycles up to 300 cycles. It maintained the relative dynamic modulus of elasticity constant under the freeze-thaw cycles.

(6) Drying shrinkage of CFRC was reduced by increased volume loading of CF in the composites. The linear shrinkage decreased by approx. 29~50% by polymer impregnation as compared with autoclave-cured CFRC with no polymers. The length change in 6 months was less than 2.8×10^{-4} for polymer-impregnated CFRC. Polymer impregnation is shown to be effective in rendering the dimensional stability of CFRC.

(7) In general, PAN CF is superior to the isotropic Pitch CF for reinforcing cement matrix. This superiority is obtained at the expense of the higher cost of PAN CF.

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