# Effects of glass fiber modified with calcium silicate hydrate (C-S-H(I)) reinforced cement

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**Abstract**. In this paper, calcium silicate hydrate (C-S-H(I)) and glass fiber modified with C-S-H(I) (SiF) at ambient temperature were synthesized. SiF and untreated fiber (OF) were incorporated into cement paste. Phase composition of C-S-H(I), SiF and OF was characterized by XRD. The surface morphologies were characterized by SEM. Flexural performance of fiber reinforced cement (FRC) at different curing ages was investigated. Results indicated that both SiF and OF could reinforce cement paste. SiF had a more positive effect on improving the flexural performance of FRC than OF. The strength of SiF reinforced cement was 11.48MPa after 28 days curing when fiber volume was 1.0%, 12.55% higher than that of OF reinforced cement. The flexural strength increased with the addition of fiber volume. However, the large dosage of fiber might cause a decrease in flexural strength of FRC.

**Keywords:** glass fiber, cement, flexural strength, modification

## 1. Introduction

Cement structures are the most widely used construction materials in contemporary development of the society. It has been applied to industrial floors, elevated slabs, overlays, dam, bridge, road and many other fields for their distinct advantages of high compressive strength, good environmental adaptability and so on [1]. However, shortcomings including poor toughness and brittle behavior have restrained its further development, leading to a deterioration of cement structures and shortened service life [2-5]. The incorporation of fibers has great potential for improving the defects of cement structures with great success obtained up to date [6-8]. Polymer fiber [9], glass fiber [10], nature fiber and steel fiber [11] have been applied to various fields and performed well, playing a positive role on the brittle behavior and poor toughness of cement structures. However, the interfacial adhesion between fiber and matrix is weak which will affect the toughening effect of the fibers. Effective methods have been put forward to improve the interfacial adhesion, including sizing [12], coating [13] and plasma technology [14]. But out-controlled process or complex operation are the main shortcomings of above all methods.

Tobermorite, a kind of C-S-H(I) which crystallizes from the system of $CaO$-$SiO_2$-$H_2O$ [15], has been a focus for the similarity with C-S-H phase produced in the hydration of Portland cement [16-18]. The mineral could be synthesized with silicon dioxide ($SiO_2$) and calcium oxide ($CaO$) as the starting

materials by hydrothermal reaction [19]. Lucie Galvankova [15] *et al*. synthesized C-S-H(I) at different reaction temperatures (170-190 $^{o}$C) and the $CaO/SiO_2$ (Ca/Si) ratio was set as 0.83. H Youssef investigated the influence of reaction conditions including time, Ca/Si ratios and temperature on the formation of C-S-H (I) [20].

In this paper, C-S-H (I) modified glass fiber (SiF) is prepared by CaO and $SiO_2$ and characterized by XRD and SEM. SiF and untreated glass fiber (OF) are incorporated into cement. The effects of fiber volume and fiber types to the flexural performance of fiber reinforced cement (FRC) are studied.

## 2. Materials and methods

Glass fiber chopped is bought from Taishan Glass Fiber Co.,Ltd., China with the length of 4mm and one bundle has about 500 filament fibers with an average diameter of 11μm. P.O42.5 cement is produced by Landscape Cement Group Co., Ltd. CaO powder is calcined from $CaCO_3$ (A.G., Sinopharm Chemical Reagent Co. formula) and $SiO_2$ is bought from Alfa Aesar (BET S.A= 350~ 410 $m^2/g$).

### *2.1. Synthesis of C-S-H (I) and Surface modification of SiF*

$SiO_2$, CaO and distilled water were added into a reactor in appropriate proportion and the mole ratio of Ca/Si was controlled at 0.5, 1.0 and 1.5. The reactor was shaken slightly to ensure the mixture fully mixed. Then, it was placed into an oven of 100 $^{o}$C for 48h. After the reaction, the mixture was poured into a beaker and dried at 60 $^{o}$C. The process of surface modified SiF was similar to that of C-S-H(I). The raw materials included glass fiber, $SiO_2$, CaO and distilled water. Reaction conditions were same as that of C-S-H(I). Ca/Si ratios were set as 0.5, 1.0, 1.3 and 1.5, respectively. The modified fibers were washed with distilled water to remove the unreacted calcium hydroxide (CH) and $SiO_2$ for 30 minutes. The washed fibers were then dried at 45 $^{o}$C for 48h.

### *2.2. Characterize of C-S-H(I) and SiF*

Synthesized C-S-H(I), OF and synthesized SiF were characterized by X-ray diffraction (XRD). Data was collected by X-ray diffractometer Empyrean (Pananalytical) on a D8 advance diffractometer system equipped with Cu Kα radiation (Bruker Co., Karlsruhe, Germany). The spectra of all synthesized phases were recorded in 2θ range of 10°-80° at the scanning speed of 0.3°/min. Surface morphologies of modified glass fiber were acquired by Quanta FEG 250 field emission scanning electron microscope (SEM) produced in FEI (American) whose resolution was 1.04 nm.

### *2.3. Preparation and testing of FRC*

**Table 1.** Details of samples' information.

| Curing ages (d) | Fiber volume (%) | | | | |
|---|---|---|---|---|---|
| | 0 | 0.50 | 0.75 | 1.00 | 1.25 |
| 3 | A0-0 | A0-1 | A0-2 | A0-3 | A0-4 |
| | A1-0 | A1-1 | A1-2 | A1-3 | A1-4 |
| 7 | B0-0 | B0-1 | B0-2 | B0-3 | B0-4 |
| | B1-0 | B1-1 | B1-2 | B1-3 | B1-4 |
| 28 | C0-0 | C0-1 | C0-2 | C0-3 | C0-4 |
| | C1-0 | C1-1 | C1-2 | C1-3 | C1-4 |

The water to cement ratio (w/c) of cement paste was 0.4 and fiber volume varied from 0.5% to 1.25%, respectively. Details of the samples' information (denoted as Xy-n) were listed in table 1. In table 1, X in Xy-n meant curing ages (A: 3d; B: 7d; C: 28d), y meant fiber type (0: OF; 1: SiF) and n signified fiber volume (1: 0.5%; 2: 0.75%; 3: 1.0%; 4: 1.25%). The size of FRC samples for flexural

performance was 80 mm × 20 mm × 20 mm. Water, cement and fiber were weighted, mixed, molded and demoulded. Samples were cured for 3 days, 7 days, 28 days at the relative humidity of 95% and the curing temperature of 20±2 ℃. The flexural performance was tested referenced to GB/T 7897.2-1987 by MTS (370 Load Frame, MTS Systems Corporation 14000 Technology Drive Eden Prairie. MN. 55344). The loading rate was 0.2 mm/min.

## 3. Results and discussion

### *3.1. XRD analysis of C-S-H(I), OF and SiF*

Figure 1 showed the XRD spectra of C-S-H(I) with different Ca/Si ratios. As was shown, there was only one intense peak in all samples at 2θ of 29.6°, indicating the formation of C-S-H(I) [20]. Also, the product of the hydrothermal reaction was C-S-H (I) in no matter with Ca/Si ratios. Figure 2 described the XRD spectra of OF and SiF. It has been observed that there only exists one broad peak in OF with a range of 10°~ 37°, amplifying its amorphous feature. As was known, glass was a kind of uncrystalline material and the results in figure 2 just reflected the amorphous properties of OF. A similar peak occurred in SiF, but a sharp peak was clearly visible at the position of 29.6° which is the same as C-S-H(I), indicating that the phase of C-S-H(I) could be synthesized on the surface of glass fiber referenced to figure 1.

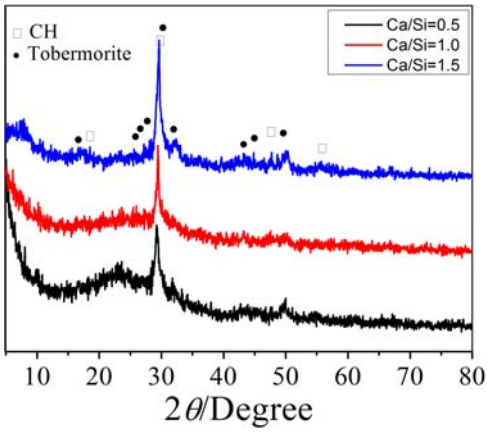


**Figure 1.** XRD spectra of C-S-H(I) synthesized with different Ca/Si ratios at 100 °C.

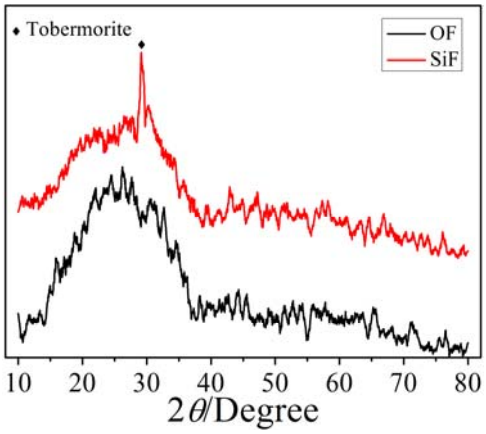


**Figure 2.** XRD spectra of OF and SiF.

### *3.2. EDS analysis of OF and SiF*

Figure 3 showed the surface morphologies and EDS analysis of OF and SiF. The surface of OF was smooth (figure 3(a)) and SiF was rough (figure 3(b)). The element of OF were consistent with SiF. Most notably, the element of Si had the most percentage and other element with lower percentage including Na, O and Ca (as shown in figure 3(c) and figure 3(d)). The atomic percentages of Si in OF and SiF were 20.02% and 32.54% and for the element of Ca, they were 1.89% and 5.04%, respectively. The atomic percentage of Si and Ca from SiF was higher compared with OF, indicating the formation of C-S-H(I) product on the surface of SiF as well.

### *3.3. Optimum conditions of SiF*

Figure 4 showed the surface morphologies of modified glass fiber with C-S-H(I) under varied Ca/Si. As we could see, when the ratio of Ca/Si was 0.5, no significant change was visible on the surface of glass fiber (as was shown in figure 4(a)). With the increase of the ratio of Ca/Si, more C-S-H(I) was synthesized. In figure 4(c), a visible but inhomogeneous nano material was synthesized on the surface of glass fiber and when the ratio of Ca/Si was 1.5. Coming to figure 4(d), a layer of homogeneous and

compact C-S-H(I) with the size of 350~400 nm was synthesized, indicating the method of C-S-H(I) modified glass fiber was feasible and the optimum ratio of Ca/Si was selected to be 1.5 herein.

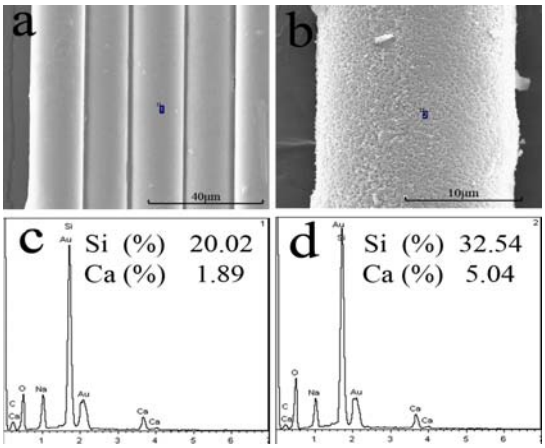


**Figure 3.** EDS analysis of OF and SiF (a, c: OF; b, d: SiF).

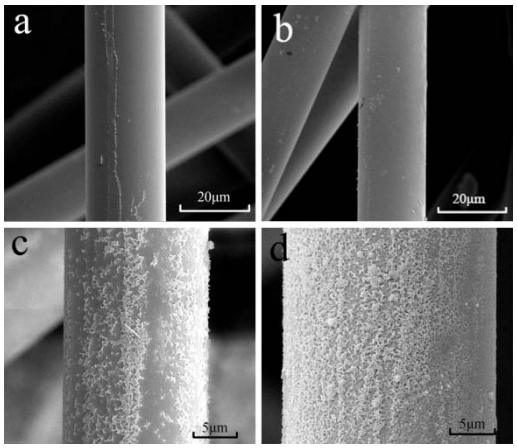


**Figure 4.** Surface morphology of glass fiber modified with C-S-H(I) (a: Ca/Si=0.5, b: Ca/Si=1.0, c: Ca/Si=1.3, d: Ca/Si=1.5).

### *3.4. Flexural performance of FRC*

Figure 5 showed the flexural strength of FRC at different curing ages. Basically, the incorporation of fiber could improve the flexural strength of FRC. Flexural strength increased with the increase of fiber volume and SiF had more advantage than OF. It could also be seen that excessive fiber may decrease the flexural strength of FRC. As was shown in figure 5(b), the flexural strength of B1-4 was 9.81 MPa, 28.24 % higher than that of B1-0 (7.65 MPa) and the flexural performance of B0-4 improved 21.3 % than that of B0-0.This may be attributed to the fiber reinforcing effect. FRC cracked under the action of load and propagates. Fiber who distributes randomly in brittle matrix created a three dimensional network. The fiber bridged the cracks and it must overcome the interfacial friction and interfacial bond between fiber and brittle matrix in the process of propagation. However, a negative effect may generate for a larger dosage of fiber and lead to a decrease of flexural strength of FRC. As was shown figure 5(c) that flexural strength of FRC increased with the addition of fiber volume until 1 % and then, a sharply decrease of flexural strength was visible when the incorporated fiber was 1.25 %. The strength of C1-4 and C0-4 declined down to 10.8 % and 9.42 % than that of C1-3 and C0-3, respectively. Such may be related to the poor dispersion of glass fiber in cement paste. More incorporated fibers implied a reduction in relative water consumption and led to inhomogeneous distribution of fiber even local agglomeration. Both SiF and OF had a positive effect on reinforcing cement no matter with the curing age, however, SiF had more advantage than OF.As was shown in figure 5(a), the flexural strength of A1-3 was 7.9 MPa, 9.6 % higher than A0-3.

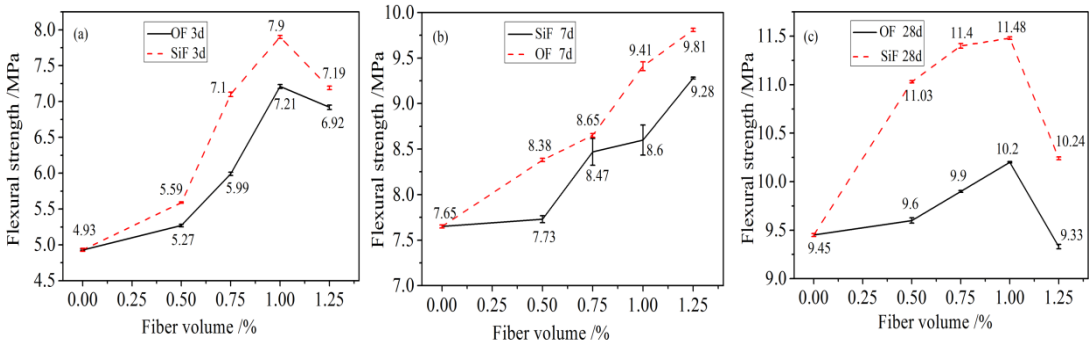


**Figure 5.** Flexure strength of FRC at different curing ages.

## 4. Conclusion

Synthesis of C-S-H(I) on the surface of glass fiber was successful and effective. The method was simple, and the complex experimental conditions and precision instruments were not required. After the modification, a homogeneous and compact layer of nano material was obtained on the surface of glass fiber when the Ca/Si ratio was 1.5. The results indicate that both SiF and OF can improve the flexural strength of FRC. To a certain extent, large dosage of fiber results in high flexural strength of FRC. However, it may also cause the decrease of flexural strength for a poor dispersion and local agglomeration. Compared to OF, SiF has a more excellent effect on the enhancement of the flexural strength of FRC, evidencing that the modification of glass fiber with C-S-H(I) is an effective manner to enhance mechanical properties. It is anticipated that such modified glass fibers can be a good candidate to promote the development of high-performance FRC in future.

## Acknowledgments

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