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Preparation and study of light transmitting properties of sulfoaluminate cement-based materials

Yue Li [a,b], Jiaqi Li [c,*], Hui Guo [a,b]

[a] The Key Laboratory of Urban Security and Disaster Engineering, MOE, China
[b] Beijing Key Lab of Earthquake Engineering and Structural Retrofit, Beijing University of Technology, China
[c] Department of Civil and Environmental Engineering, University of California, Berkeley, United States

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ABSTRACT

Polymethylmethacrylate (PMMA) optical fibers with two types of diameters were uniformly arranged in sulfoaluminate mortar by different methods. Light transmitting sulfoaluminate cement-based materials (LTSCM) were prepared. Optical and mechanical properties of LTSCM were studied. The compressive strength decreased linearly with increasing volume fraction of optical fiber in LTSCM. Compressive strength of specimen after 80 °C water bath was larger than that of specimen under standard curing conditions. According to CCD technology, brightness of optical fiber in different positions of LTSCM can be exhibited clearly. Light transmitting performance of LTSCM was tested by optical power method. Transmittance of specimen decreased with increasing spacing between the detector and specimen. Optical power increased with increasing number of fiber and with increasing diameter of fiber. Transmittance of fiber was weakened due to water bath at high temperature. Voids existed between the fiber and matrix.

1. Introduction

Light transmitting cement-based material (LTCM) is a new type of light transmitting material [1,2]. Its light transmitting properties depend on a large number of optical fibers, which transmit light through the cement-based product. It can greatly enhance the lighting effect of buildings, reduce the energy consumption of architectural lighting, and promote building energy saving. Nowadays, LTCM is usually used as building envelope, such as exterior walls of museum and opera house. In the World Expo Shanghai 2010, light transmitting cement-based materials were used as exterior walls of Italian Pavilion. In addition, LTCM also has artistic value, and can be used as decorations in exhibition hall and museum [1].

A few studies concerning components, preparation and properties of LTCM have been reported. (1) From the standpoint of material components, the matrix materials of LTCM are primarily Portland cement or mortar [3–8]; the types of optical fiber are mainly glassy fiber [3–6] and organic fiber [7,9,10]. (2) From the standpoint of preparation, literature [6] reported that parallel optical fibers were arranged in matrix; literatures [7,9,10] reported the application of optical fabric technology in preparation of LTCM. However, the studies described above have not solved the problem – the irregular arrangement of fibers in matrix. Literatures [11–13] introduced that molds were used to prepare LTCM, while the embedment of optical fiber was difficult, and the workload was heavy. (3) The properties of LTCM, including physical, mechanical, and light transmitting properties were studied. In the previous work of the authors [3–5], LTCM was composed of Portland cement mortar and glassy fibers. Parallel fibers were arranged in mortar uniformly. Compressive strength was tested, light transmitting properties were measured by spectrophotometer, and the microstructure of LTCM was observed by SEM. The results showed that completed LTCM had high light transmittance. In the range of 520–630 nm wavelength, the light transmittance of LTCM reached, or even exceeded that of 70g A4 printing paper. With increasing volume fraction of optical fiber, its light transmittance gradually increased, while its compressive strength decreased. The compressive strength at 28 days was less than 25 MPa.

Previous studies demonstrated that main performance of LTCM depends on the characteristics of matrix materials, type of optical fiber, and its arrangement. In existing papers, matrix materials are all made from Portland cement. Sulfoaluminate cement has many advantages, such as high early strength, high ultimate strength, and low alkalinity, compared with Portland cement. In addition, the representation form of light transmittance in previous work

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* Corresponding author at: Department of Civil and Environmental Engineering, University of California, Berkeley, CA, United States.
E-mail address: Jiaqi.li@berkeley.edu (J. Li).

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was relatively simple, and different test methods need to be developed. Thus, the main object of this paper is to study the preparation of LTSCM, including the components of raw materials, arrangement method of optical fiber, molding process and curing. Moreover, the physical, mechanical and optical properties of LTSCM are discussed, and its microstructure is analyzed. This study aims to promote research and engineering application of this type of material. The matrix is composed of sulfoaluminate cement, two methods are designed to produce the spatial form of optical fibers. Strength performance of LTCM is studied, and two test methods, which provide the references for design and preparation of the materials, are used to evaluate its transmitting properties.

2. Materials and methods

2.1. Raw materials

Cement: The high early strength sulfoaluminate cement used in this work was from BJX CO., LTD, Tangshan, China. Its physical properties and chemical composition were given in Tables 1 and 2, respectively.

Aggregate: The sand used in this study was ISO standard sand based on Standard ISO 679:1989 (ISO Standard Sand CO., LTD., Xiamen, China). The particle size distribution of sand was continuous grading of 0.06–0.6 mm.

Optical fiber: The optical fibers used in this study were PMMA fibers (JT, Nanjing, China) with diameters of 1.0 mm and 0.5 mm, respectively. The tensile strength of the fiber with diameter of 1.0 mm was 65.73 MPa, and its elongation capacity was 10%. The tensile strength of 0.5 mm fiber was 78.47 MPa, and its elongation capacity was 20%. The coating of the fiber was fluororesin. The loss rate of fiber at 650 nm wavelength was less than 350 dB/km. Its bending diameter was eight times greater than the diameter of the optical fiber. The operating temperature was from −20 to 70 °C.

Table 1
Physical properties of cement.

Density (g/cm^3)	Blaine surface (g/cm^3)	Water requirement for standard consistency (%)	Compressive strength (MPa)		Flexural strength (MPa)	
			1d	3d	1d	3d
2.87	427	24.4	49.0	52.9	8.8	9.4

Coupling agent: A-151 silane coupling agent, and its component is vinyltriethoxysilane.

Water reducer: F0X-8H polycarboxylic acid powder with water reducing rate of 30%.

Antifoaming agent: P764 antifoaming agent powder. The admixtures were from China Building Materials Academy.

2.2. Testing methods

2.2.1. Test method for strength and micro-properties

Compressive and flexural strength tests for hardened pure cement mortar and LTSCM were carried out in accordance with China national standard GB175-2007 (title: Common Portland Cement). The consistency of high flowing cement mortar was carried out in accordance with China national standard GB/T2419-2005 (title: Test method for fluidity of cement mortar). In compressive strength test, the orientation of optical fibers parallels to the length of specimen, and is perpendicular to compression. In flexural strength test, the optical fibers parallel to the load.

The pore structure of specimen was tested by full-automatic mercury penetration porosimetry (Quantachrome PoreMaster 60GT). Sample under standard curing conditions for 28 days was shaped into 2.5–5.0 mm particles by using a sharp knife. The particles were classified into two sets. Sample A was a particle without fiber; Sample B was a particle that had one fiber. The microstructures of Sample A and B under standard-curing conditions for 28 days were observed by SEM (QUANTA-FEC250 scanning electron microscope).

2.2.2. Preparation of LTSCM

2.2.2.1. Preparation of sulfoaluminate cement mortar. Based on lots of trials, the mix proportion of sulfoaluminate cement mortars with high consistency, high strength, and high compactness was determined. The water–cement ratio was 0.35. The dosage of water reducer and antifoaming agent was 0.7% and 0.2% of cement content by mass, respectively. And the mass ratio of sand to cement was 0.8. The consistency of mortar was 150 mm, and its compressive strengths at 3 days and 28 days were 54.2 MPa and 69.8 MPa, respectively.

2.2.2.2. Embedment of optical fibers and casting. Optical fibers were immersed in silane coupling agent solution with volume concentration (volume ratio of solid to water) of 20%. After 2 min, they were air-dried. Then, two methods can be used to fasten optical

Table 2
Chemical composition of cement.

Loss (%)	CaO (%)	SiO$_2$ (%)	Al$_2$O$_3$ (%)	Fe$_2$O$_3$ (%)	MgO (%)	SO$_3$ (%)	R$_2$O (%)	Na$_2$O (%)	K$_2$O (%)
6.82	37.18	16.40	25.83	1.09	1.82	9.22	0.59	0.16	0.31

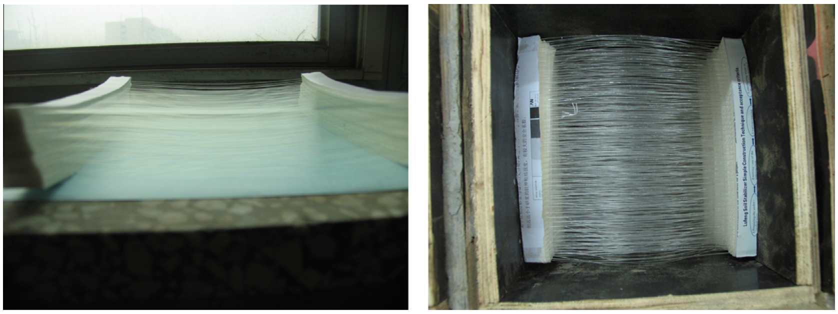


Fig. 1. Fastening method and casting mold (Method 1).

Fig. 2. Fastening method and casting mold (Method 2).

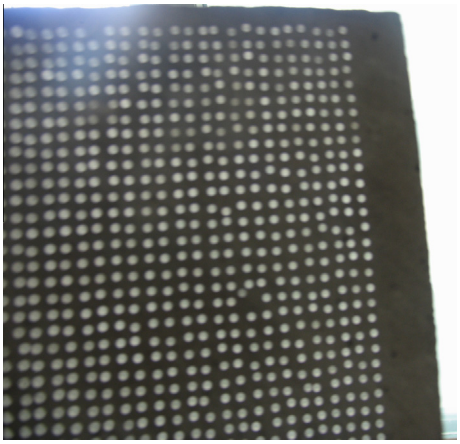


Fig. 3. Diced LTSCM specimen.

fibers. Method 1 (Fig. 1): fibers were cut into the same length. 2 mm-thick tapes were used to fasten the two ends of parallel optical monofilaments. The centroid distance of fibers was 4 mm. Then, the parallel fiber sets were overlaid by other sets to form a 3 cm-height piece. The pieces were put into a mold separately, and were cast by sulfoaluminate slurry. Method 2 (Fig. 2): optical fibers were wrapped around the rotating steel cylinder with notches, and two steel rods were used to fasten the two ends of each parallel optical fiber set on the die. Finally, the die was put into the mold, and the bulk was cast with slurry.

2.2.2.3. Curing and slicing. The cement mortars containing optical fibers under standard curing conditions (temperature 20 ± 2 °C, RH 95 ± 5%) for 28 days were sliced into two types of specimens, as shown in Fig. 3. Bulks with dimensions of 4 cm × 4 cm × 16 cm were used to test compressive strength; and slices with size of 4 cm × 8 cm × 1 cm were polished and used to measure optical properties. In addition, some of these two types of specimens would be immersed in 80 °C water for 3 days, then mechanical and optical properties were tested. The objective of the test was to check the appearance of aging of optical fiber by accelerated test.

2.2.3. Testing method for light transmitting properties

2.2.3.1. CCD test. CCD (Charge-coupled Device) is a type of semiconductor device, it can transfer optical image to digital signal. CCD lattice, namely the set of planar image through *X* and *Y* directions, is usually used in digital camera. In this test, CCD images of optical fibers in LTSCM were obtained from DCU224M – CCD camera. The actual testing facilities were shown in Fig. 4, and simulated optical path was given in Fig. 5. LTSCM was irradiated by parallel white light source at wavelength of 390–780 nm. The transmitted light was gathered via the convex lens. Then images were formed in the CCD camera, and they were directly shown as photographs in computer.

2.2.3.2. Testing method for optical power. The light transmitting properties of LTSCM were measured by optical power meter, which was used to measure the optical power of optical energy passing along optical fibers, to measure absolute optical power, and to evaluate the transmission quality of optical fibers. The equipment includes a stable light source, a detector and a display. The diameter of the effective surface of the detector is 1 cm. In this study, the optical power meter was used to measure the optical power of LTCM in various distances between LTCM and the light source. Simulated and actual tests optical path were shown in Figs. 6 and 7, respectively. The light source was white light.

3. Results and discussion

3.1. Results and analysis of strength

The fastening methods of optical fiber and mix proportion of mortar, as described in Section 2.2.2, were used to prepare LTSCM. The number of optical fibers in unit area of section was

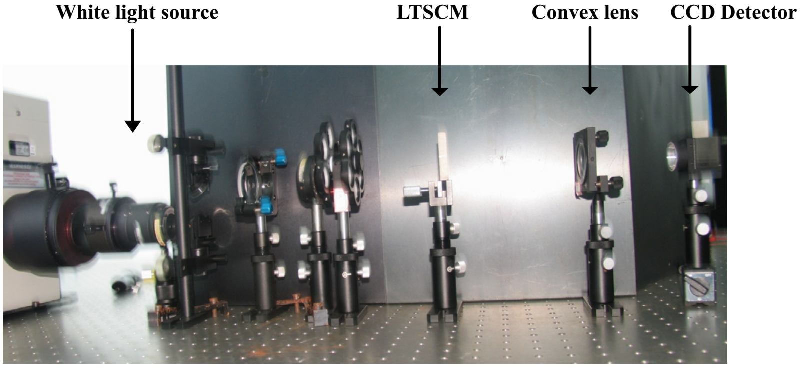


Fig. 4. Optical path of CCD test.

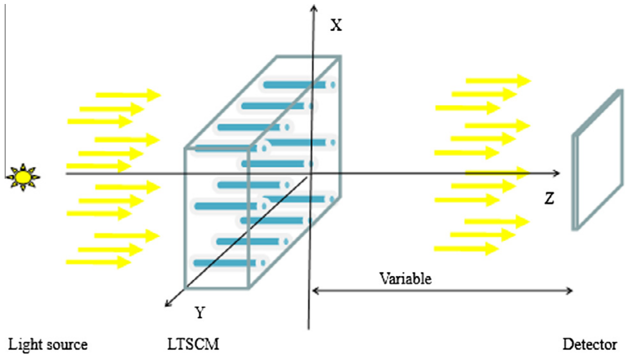


Fig. 5. Simulated optical path of CCD test.

uniform. There were about 625 fibers in a 10 cm × 10 cm section. The strengths of the specimen and volume fractions of fibers were illustrated in Table 3.

From Table 3, it can be seen that: (1) Whether the fibers were introduced or not, the strengths of LTSCM were relatively high. The compressive strengths were larger than 60 MPa, and the flexural strengths were greater than 9 MPa. These were much higher than the values of the study about Portland cement-based light transmitting materials with parallel glassy fibers [3–5]. (2) For standard curing specimens, the compressive strength of LTSCM was lower than that of the specimen in absence of optical fiber. Compressive strength of the specimen decreased linearly with increasing volume fraction of optical fibers. For instance, the compressive strength of specimens containing 1.23% fiber and 4.91% fiber decreased by 5.6% and 9.4%, respectively. The decrease in flexural strength of specimen containing 1.23% fiber was very small. While, the flexural strength of the specimen containing 4.91% fiber decreased by 21.2%. This is because that optical fiber is made of soft plastic with low elastic modulus. For compressive strength test, the fibers could be regarded as defects, such as pores, in cement mortar. The greater addition of fibers leads to a larger decrease in strength. For flexural tests, the adhesive performance of the interface between the matrix and fiber of 1.0 mm diameter was worse than that of interface between matrix and 0.5 mm fiber. The large volume fraction of fiber and its parallel arrangement lead to the great decrease in flexural strength. (3) In terms of aging test, compressive and flexural strengths of specimen after 80 °C water bath were larger than those of specimen under standard curing conditions. The main reason is that, water bath at high temperature accelerates the hydration of sulfoaluminate cement, and improves the development of matrix strength.

3.2. Results and analysis of light transmitting properties

In previous studies [3–5], UV–visible spectrophotometer was used to measure the light transmitting ratio of LTCM, however this method is more suitable for testing light transmittance of liquid, and is not suitable for measuring that of fiber. LTCM should be cut into a thin slice with thickness of 5 mm. In order to measure the light transmittance, the cross section of the slices should be polished. The deviation of thickness of specimen and the toughness degree of the surface could greatly affect the results. Moreover, this

Table 3
Strengths of specimens containing optical fibers with different diameters.

Code	Diameter of fiber (mm)	Volume fractions of optical fibers (%)	Compressive strength at 28d (MPa)	Flexural strength at 28d (MPa)	Curing condition
1	0	0	69.07	11.8	Standard curing
2	0.5	1.23	65.22	11.6	Standard curing
3	0.5	1.23	67.83	11.8	Aging
4	1	4.91	62.56	9.3	Standard curing
5	1	4.91	65.21	9.7	Aging

Aging: 80 °C water bath for 3 days.

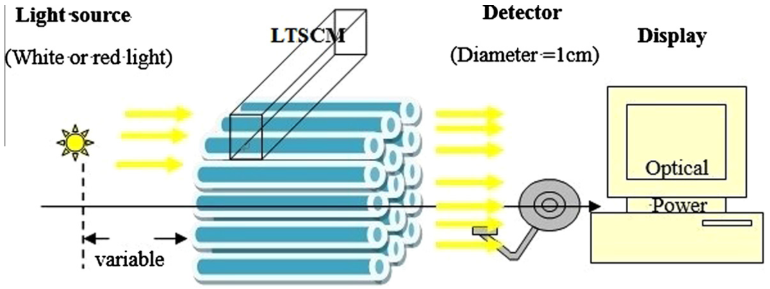


Fig. 6. Simulated optical path.

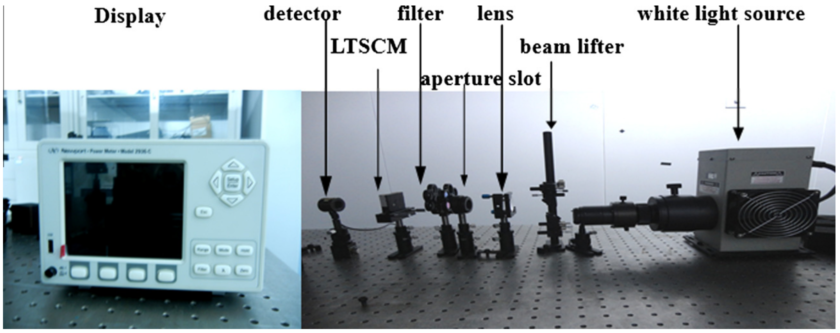


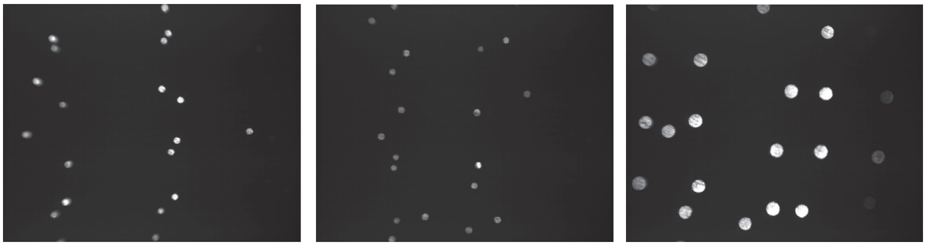
Fig. 7. Actual optical path.

method cannot test the light transmittance of single fiber. The problem can be solved by CCD method. Furthermore, optical power meter was used to measure the absolute optical power and relative loss of optical power in fiber. In optical fiber system, the application of optical power meter is very basic, and is similar to that of multimeter in electronics. Thus, the combination between CCD method and optical power meter is able to fully represent the optical properties of LTSCM.

3.2.1. Results and analysis of CCD tests

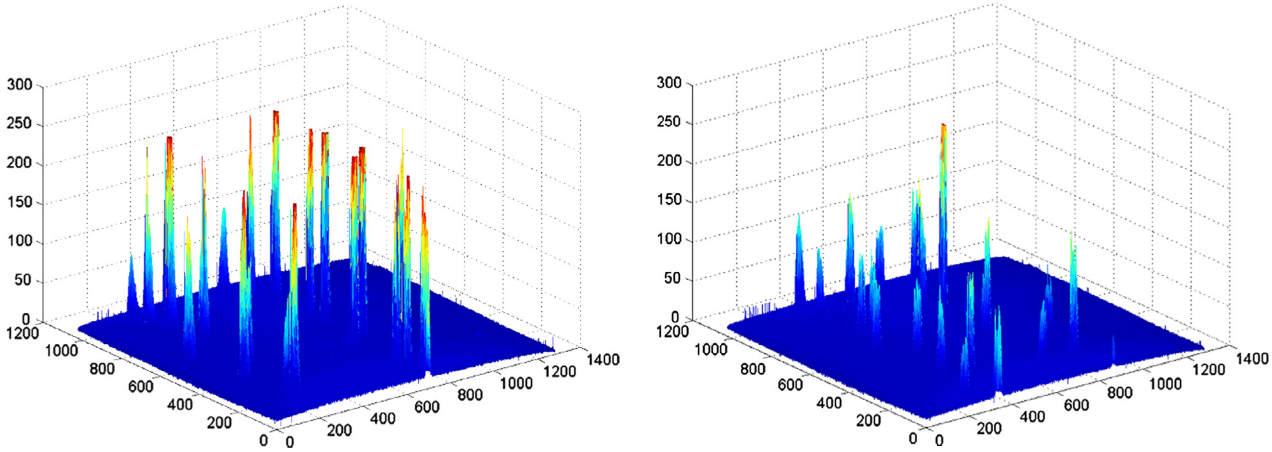
Fig. 8(a)–(c) shows CCD photographs of Sample 2–4 in Table 3.

Fig. 8 illustrates that the numbers of optical fibers in CCD photographs were all about 20. Although the numbers of fibers were mostly the same, different fibers presented various luminances. With regard to standard curing Sample 2 and 4, the number of bright optical fibers was approximately half of the total numbers of fibers in the testing region. Because the volume fraction of fiber (4.91%) in Sample 4 was higher than that of Sample 2 (1.23%), the brightness of the testing region in Sample 4 was much stronger than that of Sample 2. Sample 3 was a photograph of a specimen with fiber volume of 1.23% after aging. Compared with Sample 2, the number of bright fibers was obviously less, and its light transmitting performance of entire region was lower, which illustrates that water bath at high temperature leads to a great decrease in light transmittance of optical fibers.

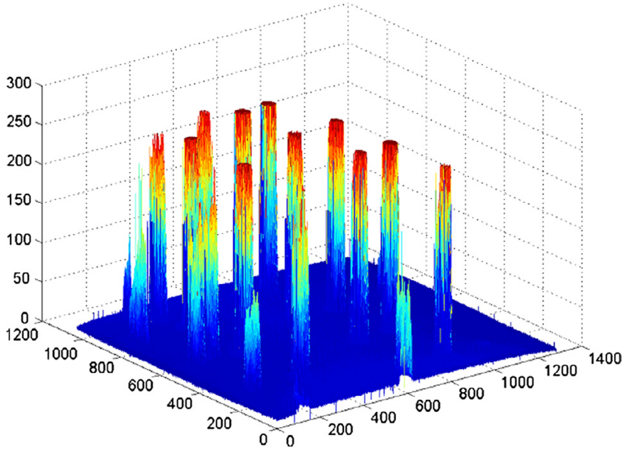


(a) Sample 2 (b) Sample 3 (c) Sample 4

Fig. 8. CCD testing results of LTSCM containing fibers with different diameters.



(a) Sample 2 (b) Sample 3



(c) Sample 4

Fig. 9. Gray level simulation of CCD images from Fig. 8.

The continuous gray values (black–gray–white) were classified into 256 gray levels, and the range was 0–255 (from black to white). Gray level of each optical fiber in CCD images from Fig. 8 can be analyzed by MATLAB software, as shown in Fig. 9. From Fig. 9, the lightest one, Sample 4, had more fibers and higher gray value; while the darkest one, Sample 3, had fewer fiber and lower gray value.

3.2.2. Results and analysis of light intensity

The light transmitting properties of the specimens in Table 3 were tested by optical power meter. In the test, make sure that the effective surface of the detector can detect 1–3 fibers. The results were shown in Figs. 10 and 11.

Fig. 10 represents optical powers of Sample 4 in Table 3, it presents the results at different distances between the specimen and detector. It can be seen that, with increasing distance between the detector and specimen, the optical power decreases gradually. It can be attributed to the diffraction of the light of each optical fiber with an increasing spacing, which leads to a larger light spot and a subsequent decrease in incoming optical power on effective surface of the detector (its diameter is 1 cm). For the same distance, the optical power increases gradually with increasing number of detected optical fibers in the detector. This is due to superposition of light intensity of multiple light.

Fig. 11 presents the optical power of specimens with different types of fibers, which are at a distance of 300 mm from the detector. It can be seen that, for specimens with the same number of optical fibers, the optical power of specimen with 1 mm fiber is larger than that of specimen with 0.5 mm fiber. The reason for this is that the transmitting area of the specimen with optical fiber of larger diameter is larger. In respect of the specimens containing fibers with diameters of 0.5 mm, the optical power decreases significantly after water bath. This is mainly because the highest working temperature of PMMA optical fiber is 70 °C, during water bath at 80 °C for 3 days, thermal chemical aging occurs, it leads PMMA to oxidative degradation, which significantly increases absorption loss in electron transfer of short wavelength, finally loss in short wavelength of PMMA rises sharply [14–15]. Thus, light transmitting properties of optical fiber are affected.

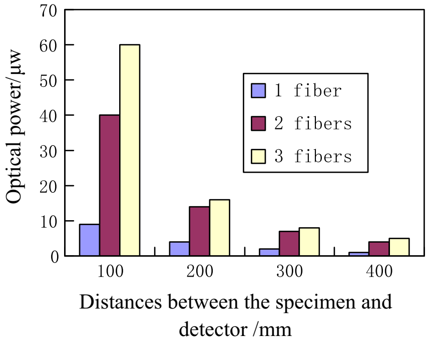


Fig. 10. Optical powers of sample 4.

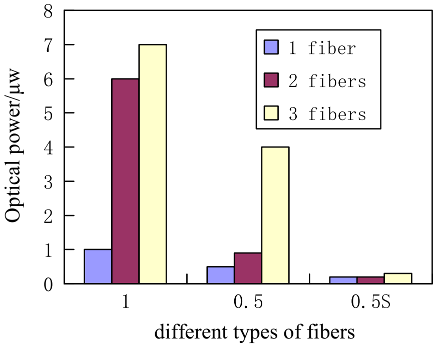


Fig. 11. Optical powers of specimens at the same distance.

Based on study of LTCM regarding the mechanical and optical properties, the following aspects should be assured, so that its wide application can come true. (1) The matrix of LTSCM should be made of high strength, and high consistency cement mortar. The arrangement of optical fibers could be parallel or special designed. In order to achieve good light performance, the volume fraction of fiber should be larger than 4%. (2) The idea application of this type of materials includes products of landscape and architecture, such as decorative plates and blocks, and curved plates, so that the creativity and imagination and be achieved. It should be noticed that, if PMMA fibers were used, the highest working temperature of the LTCM should not be higher than 70 °C. Otherwise it could result in the aging of the fiber, and weakness of optical properties.

3.3. Microstructure analysis of LTSCM

A large number of studies demonstrate that porosity, pore diameter, pore distribution, pore size, and orientation of pores have important effects on strength of mortar and concrete [16–23]. The pore structure of hardened cement determines its compactness, porosity, permeability and mechanical properties [24–26]. The pores can be classified into four major types: harmless pore (diameter < 20 nm), less harmful pore (diameter, 20–100 nm), harmful pore (diameter, 100–200 nm), and much harmful pore (diameter > 200 nm) [27].

The pore structures of Sample A (without fiber) and Sample B (with a fiber) were tested by mercury intrusion technique. Pore distribution curves of Sample A and B were given in Fig. 12. And their characteristic parameters of pore structure were shown in Table 4.

Table 4 illustrates that the most probable pore diameter of Sample B was 2.47 nm larger than that of Sample A, the porosity of Sample B was 0.99% larger than that of Sample A, and Sample B had more harmful pores, the diameter of which is larger than 100 nm. The addition of the single fiber increased the volume fraction of the harmful pores by 1.23%. In addition, considering that the porosity of optical fiber was approximately zero, while the porosity of matrix of Sample A was 14.31%, it can be inferred that a large number of gaps formed around the fiber–matrix interface. The increase in porosity and the number of harmful pores affected

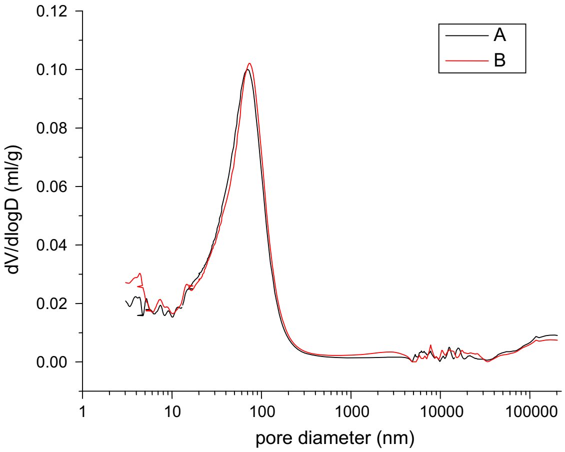


Fig. 12. Pore distribution curve.

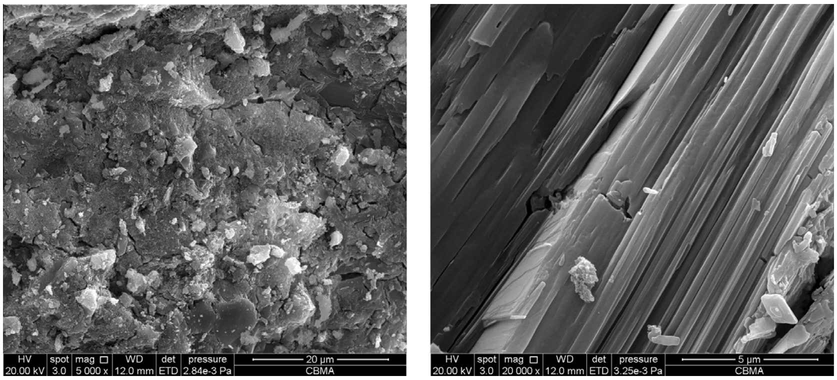


Fig. 13. SEM images of matrix of Sample A.

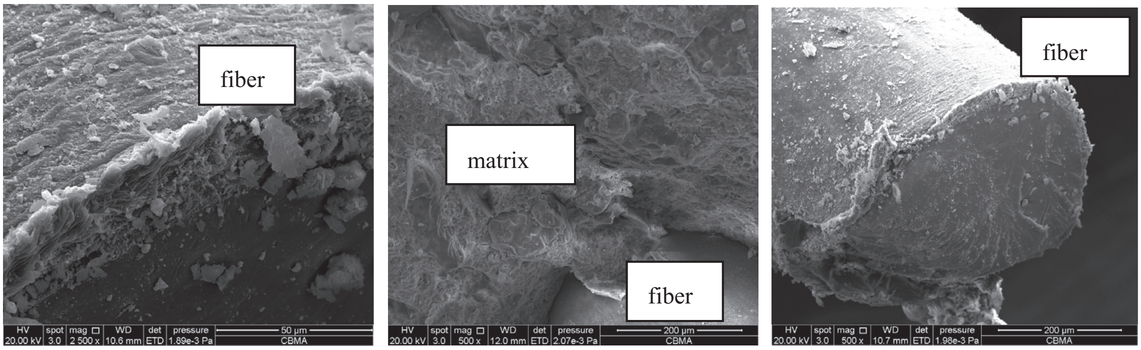


Fig. 14. SEM images of interface between optical fiber and mortar (Sample B).

Table 4
Characteristic parameters of pore structure of LTSCM.

Code	Median pore diameter (nm) (by volume method)	Most probable pore diameter (nm)	Porosity (%)	Pore distribution%			
				200–200,000 (nm)	100–200 (nm)	20–100 (nm)	0–20 (nm)
A	58.14	70.47	14.31	11.40	10.80	57.28	20.52
B	59.94	72.94	15.30	11.84	11.59	54.26	22.31

the properties of LTSCM, such as compressive strength. Table 3 illustrates the compressive strength of sulfoaluminate cement mortar decreased by 9.43%, after the introduction of 1 mm-diameter fibers.

SEM analyses of Sample A and B were shown in Figs. 13 and 14, respectively. Fig. 13 illustrates that the matrix of LTSCM was compact, its porosity was relatively small, and calcium sulfoaluminate hydrates occurred. Fig. 14 shows that PMMA fiber was composed of fluororesin coating, which is much thinner than the coating of glassy fiber [3–5] and PMMA core. The fracture section of the fiber is rough although it looks very smooth by the naked eye, which means the surfaces of the entire fibers actually are rough. Since the surface of optical fiber was dealt with silane coupling agent, there was coupling material on the surface. The molecule of silane coupling agent has two types of groups with different chemical properties. The general formula for a silane coupling agent typically is $YSiX_3$. Y is a non-hydrolyzable group, and X is a hydrolyzable group [28]. The groups of the molecule can react with organic and inorganic materials [29]. Therefore, Silane coupling agents have the ability to form a durable bond between organic and inorganic materials [30–32]. However, in this work obvious gaps can be detected in a well-marked interface between fiber and matrix, which means that coupling agent cannot work very well on PMMA-fiber–matrix interface. Thus the modified coupling agent, which can deal with interface between PMMA-fiber and cement matrix, needs to be developed.

4. Conclusions

(1) Taking into account both optical and mechanical properties, the optimal composition of the LTSCM is described below: The matrix material is composed of calcium sulfoaluminate cement mortar. The water–cement ratio is 0.35. The dosages of water reducer and antifoaming agent are 0.7% and 0.2% of cement content by mass, respectively. And the mass ratio of sand to cement is 0.8. Optical fibers with diameter of 1 mm are used, and its volume fraction should be larger than 4%.

(2) Compressive strength of specimens containing optical fibers is smaller than that of specimens without optical fibers. Compressive strength of specimen mainly decreased linearly with increasing volume fraction of optical fibers. Compressive strength of specimen after 80 °C water bath was larger than that of specimen under standard curing conditions.

(3) According to CCD technology, brightness of optical fiber in different positions of LTSCM can be exhibited well. The Gray level of each optical fiber in CCD images can be plotted by MATLAB software. Thus, light transmitting performance of different optical fibers can be shown.
(4) Entire light transmitting performance of LTSCM can be tested by optical power method. Light transmittance of specimens decreases with an increasing distance between the detector and specimen. For the same distance, optical power increases with increasing number of fibers. With regard to the same number of optical fibers, optical power of specimen with fibers of large diameter is larger than that of the specimen with fibers of smaller diameter.
(5) Light transmittance of fiber is significantly reduced due to water bath at 80 °C.
(6) Pore structure tests show that the introduction of optical fiber results in lots of gaps on fiber–matrix interface. SEM analysis also demonstrates the result described above. After dealing with a coupling agent, coupling material was covered on the surfaces of fibers, but coupling agent could not work very well on PMMA-fiber–matrix interface.

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