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Study on Sulfate Erosion Resistance of Basalt Fiber Concrete after Ultra-Low Temperature Freezing and Thawing

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Abstract

This study investigates the compressive and tensile properties of basalt fiber-reinforced concrete (BFRC) after ultra-low-temperature freeze-thaw cycles. Scanning electron microscope (SEM) analysis was conducted to examine the deterioration mechanisms caused by freeze-thaw cycles and sulfate erosion. The results show that compressive and tensile strengths increase with basalt fiber dosage. The optimal dosage is 0.2%. With longer exposure to sulfate erosion, both strengths decline significantly. Basalt fibers effectively bridge cracks, control expansion, enhance compactness, and improve concrete performance. Ultra-low-temperature freeze-thaw cycles and sulfate erosion cause rapid crack growth. Sulfate erosion produces crystallization products and expansive substances. These fill cracks, create pressure, and damage the internal structure. Freezing and expansion forces further enlarge voids and cracks. This provides space for expansive substances, worsening concrete deterioration and reducing its performance.

Keywords

Basalt Fiber Concrete, Ultra-Low Temperature Freeze-Thaw Cycle, Compressive Properties, Splitting Tensile Properties, Strength Deterioration Model

1. Introduction

In the northwest region of China, due to the high poster and cold winter, the local buildings are prone to suffer from the damage of freeze-thaw cycles and even ultra-low temperature freeze-thaw cycles. Due to the dual influence of regional environment and climate, soil and water resources contain a large number of chloride ions and sulfate ions [1], resulting in freeze-thaw damage and sulfate erosion will act together on the concrete building, which seriously jeopardizes the durability of concrete structure. In order to improve the resistance of concrete to sulfate attack and freeze-thaw resistance, adding fibers to modify concrete is a common method to enhance the durability of concrete [2]. Wang Jun [3] and others pointed out that carbon nanofibers can further enhance the resistance to freezing and frost by bridging cracks, controlling nanoscale cracks, nucleation, and optimizing the pore structure to improve the densification of concrete. Gong [4] and others found that polypropylene fibers can synergistically interact with the concrete's setting and hardening to effectively inhibit the cracks caused by the osmotic pressure and freezing and expansion pressures as well as the development of the concrete's internal pore space, thus enhancing the concrete's resistance to freezing and frost. Resistance to salt freezing. Xu Cundong *et al.* [5] added basalt fibers to concrete to improve compressive and flexural strength. The incorporation of basalt fibers can significantly slow down the rate of decline in mechanical properties of concrete under salt freezing conditions, but it is still difficult to completely prevent the destruction of concrete structure by salt freezing. In addition, some scholars have used plant fibers [6], waste plastic fibers [7], and waste tire fibers [8] [9] to produce more environmentally friendly concrete, which not only solves the problem of concrete resistance to sulfate attack and freeze-thaw cycle, but also has a positive environmental impact.

Basalt fiber is a kind of green and non-polluting modern inorganic concrete fiber made from basalt fusion, which has low cost and relatively simple preparation process. Its density is close to that of ordinary concrete, a feature that allows it to be better wrapped with concrete, effectively improving the mechanical properties of concrete [10]. Compared with metal fibers such as steel fibers, its cost is low and the preparation process is simple. Compared with organic fibers such as polypropylene, it has high tensile strength and modulus of elasticity. Its excellent high temperature resistance characteristics make it widely used in the field of fire protection and thermal insulation. For example, basalt fibers are used to make products such as fireproof cloth and fireproof clothing. This material is naturally the material of choice for fire protection and thermal insulation because of its excellent heat resistance, insulation properties and elegant appearance [11]. Therefore, in this paper, basalt fibers were selected to modify concrete in order to improve the mechanical properties of concrete, save costs and promote environmental sustainability.

Scholars have conducted extensive research on BFRC, but most of the research is on the sulfate erosion resistance under normal environment. However, few studies have been conducted on the sulfate erosion resistance after ultra-low temperature freeze-thaw cycles. Therefore, in this paper, the mechanical properties of BFRC after ultra-low temperature freeze-thaw cycles and subjected to sulfate erosion are investigated with fiber doping and erosion days as variables. And combined with SEM scanning electron microscopy, the damage mechanism of ultra-low temperature freeze-thaw cycle coupled with sulfate erosion is investigated. Finally, combined with the experimental data, the compressive and tensile strengths were analyzed, fitted, and a strength deterioration model of BFRC was established at room temperature, after freeze-thaw cycling from 25˚C to −80˚C, and after freeze-thaw cycling from 25˚C to −160˚C related to the number of days of sulfate erosion and basalt fiber dosage. This study aims to enable fiber concrete to adapt to more severe and complex environments, thus providing theoretical support for the development of fiber concrete in the field of ultra-low temperatures and further promoting the progress of this field [12].

2. Test Procedure

2.1. Test Materials and Mixing Ratios

The P.0 42.5 grade ordinary silicate cement produced by Huaxin Cement Co. was used in this test. The physical and chemical properties of the cement used in the test are shown in **Table 1**. The fine aggregate was natural river sand with a fineness modulus of 2.8, which belonged to medium sand, with a particle size of 0.35 mm to 0.5 mm, and the coarse aggregate was natural gravel crushed by a crusher, with a particle size of 6.0 mm to 24 mm. The basic mechanical properties of the aggregate used in the test are shown in **Table 2**. The water used was Wuhan tap water. The erosion used was anhydrous magnesium sulfate with 99.27% solubility produced by Wuxi Yatai United Chemical Co. The fiber used in the test was short-cut basalt fiber produced by Zhejiang Haining Anjie Material Co. The main parameters of this basalt fiber are shown in **Table 3**.

Table 1. Physico-chemical properties of the cements used.

Gelling Material	Ingredient (%)										Intensity (kg/cm)	Specific Surface Area (cm^3/g)
	CaO	SiO_2	Al_2O_3	Fe_2O_3	M_gO	SO_3	K_2O	Na_2O	MnO	TiO_2		
Cement	62.9	20.7	4.84	3.18	2.15	3.56	1.31	0.76	0.13	0.26	3.15	3413

Table 2. Basic properties of aggregates used.

Type of Aggregate	Main ingredients	Apparent density	Accumulation density	Water absorption rate (%)	Indicators of crushing	fineness modulus
Coarse Aggregate	$CaCO_3$	2.901	1.619	0.6	12	-
Fine Aggregate	SiO_2	2.635	1.615	0.9	-	2.8

Table 3. Main parameters of basalt fibers.

Calibre/μm	Lengths/mm	Intensity (g/cm^3)	Modulus of elasticity/GPa	Tensile strength/MPa
23	32	2.56	58	3732

In this test, reference to the "Specification for the Design of Plain Concrete Proportions" (GJ55-2011 [13]), the design of the reference proportion was carried out for plain concrete of C40 grade. Three kinds of fiber volume admixture (0, 0.1%, 0.2%) were also designed to be added into the base mix ratio respectively. The specific concrete mixes are shown in **Table 4**.

Table 4. Basalt fiber concrete mix ratio.

serial number	Cement (kg/m^3)	Water (kg/m^3)	Sand (kg/m^3)	Broken stone (kg/m^3)	Fiber Volume Admixture (%)
BF0	462	184	631	1127	0
BF1	462	184	631	1127	0.1
BF2	462	184	631	1127	0.2

2.2. Preparation of Specimens and Test Methods

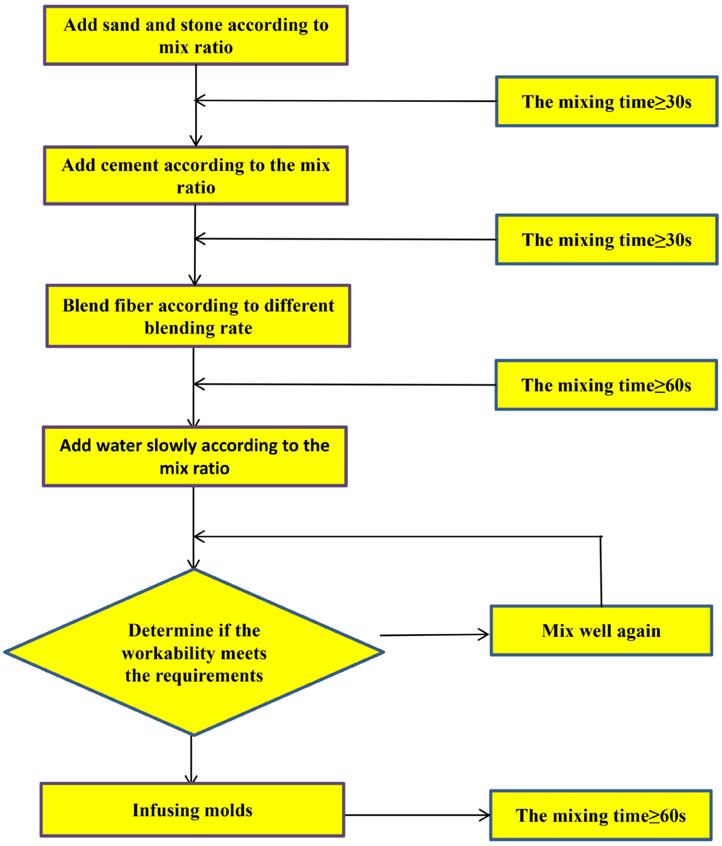


Figure 1. Detailed flow chart of fiber dry mix method.

1) Preparation of specimens: in order to the accuracy of the test and to be able to achieve the desired purpose, the size of all specimens was determined to be 100 mm × 100 mm × 100 mm with reference to the Test Method for Properties of Ordinary Concrete Mixe (GB/T 50080-2016) [14]. Distinguished from the conventional mixing method, in this test, the specimens of basalt fiber concrete were prepared by the fiber dry mixing method [15]. The specific operation procedure of this method is shown in **Figure 1**. After the specimens were prepared according

to the above method, they were placed in a constant temperature standard curing box for curing. The temperature of the equipment was set at 25˚C and the standard humidity was maintained at about 95%. After 28 days of curing, the specimens were removed and placed in a cool and dry place for natural drying.

Figure 2. Deep-cooling test chamber.

2) Specimen ultra-low temperature freezing and thawing method: In this experiment, we choose the deep cold test chamber independently developed by this group for ultra-low temperature treatment, as shown in **Figure 2**. The principle of this test chamber is using liquid nitrogen as a cooling medium, after filling the chamber with liquid nitrogen, the liquid nitrogen is vaporized in the environment and absorbs the surrounding heat to achieve the purpose of cooling. After the cooling is completed, the specimen is warmed back in the natural state. After the specimen temperature returns to normal temperature, the next round of cryogenic cooling is carried out in the same way. Considering that concrete is a material with obvious thermal inertia, its center temperature change will obviously lag behind the surface temperature in the cooling process. In the cooling process, the volume change of concrete shows the process of contraction, expansion and then contraction, which comes from the initial cooling process is mainly due to the cold of the concrete matrix and make the volume contraction, after the pore water inside the concrete will be iced, which will lead to the expansion of the volume of the concrete, and finally in the completion of the icing as the temperature continues to decrease, the volume of the concrete will be contracted again. The above process will lead to the development of cracks within the concrete, making the concrete performance decline. Too fast cooling rate will make the center of the concrete temperature hysteresis is more obvious, the greater the temperature difference will lead to more serious damage to the concrete. Too low a cooling rate will make the freeze-thaw cycle process too long, the consumption of liquid nitrogen is greater, making the cost of the test increased. Considering the actual working conditions of LNG storage tanks, liquid nitrogen will directly contact with concrete when injected, so the cooling rate of this test needs to be combined with the actual working conditions, so the cooling rate is not too slow. Therefore, we finally decided to set the cooling rate at 3˚C/min, and when the concrete is cooled

down to the target temperature, it needs to be kept at a constant temperature for more than 60min to ensure that the concrete is fully deformed at this temperature, and there is no obvious temperature difference between the inside and outside of the concrete. Temperature measurement was performed using a DT80G data acquisition instrument, and a platinum thermocouple was selected for the temperature sensor, the appearance of which is shown in Figure 3. Figure 4 is a graph of temperature change with time for an ultra-low temperature freeze-thaw cycle with a lower temperature limit of −160˚C.

(a) DT80G data acquisition instrument (b) Platinum thermocouples

Figure 3. Temperature control device.

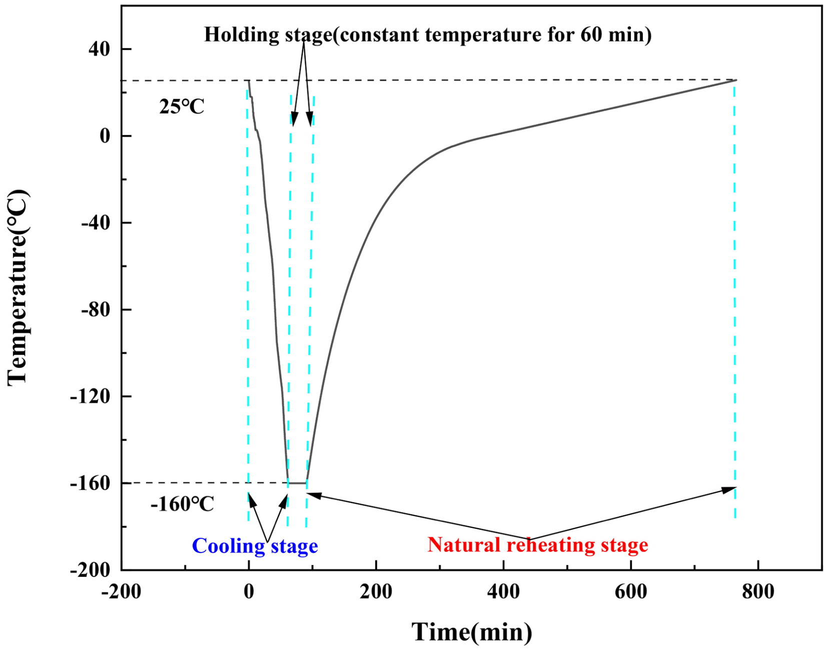


Figure 4. Temperature history of a complete ultra-low temperature freeze-thaw cycle.

3) Sulfate ion solution erosion method: The specimens were added to a 5% magnesium sulfate solution for sulfate erosion. All the specimens were divided into three groups in total. Among them, the first test group was a blank control group,

which was taken out after 0 days of immersion, the second test group was taken out after 60 days of immersion, and the third test group was taken out after 120 days of immersion.

4) Concrete compressive and splitting tensile test methods: compressive and splitting tensile tests were carried out according to the Standard for Test Methods of Physical and Mechanical Properties of Concrete (GB/T50081-2019) [16]. In the compressive test, the loading speed was set to 0.3 mm/min, while in the splitting tensile test, the loading speed was set to 0.5 mm/min. where the compressive strength was calculated according to Equation (1). And at the end of the compressive test, the values of three specimens in each group were averaged and multiplied by the size conversion factor of 0.95 to finally obtain the compressive strength of the specimens. And the split tensile strength was calculated according to formula (2). And at the end of the split tensile test, the value of each group of three specimens is averaged and multiplied by a size conversion factor of 0.85 to finally obtain the split tensile strength of the specimen.

$$f_{cu} = \frac{F}{A} \tag{1}$$

$$f_{ts} = \frac{2F}{\pi A} \approx 0.637\frac{F}{A} \tag{2}$$

In the formula:

f_{cu}—Cubic compressive strength of concrete (MPa).

f_{ts}—Splitting tensile strength of concrete (MPa).

F—Pressurized load at the time of destruction of the specimen (N).

A—the stressed area of the specimen subjected to pressure (mm^2).

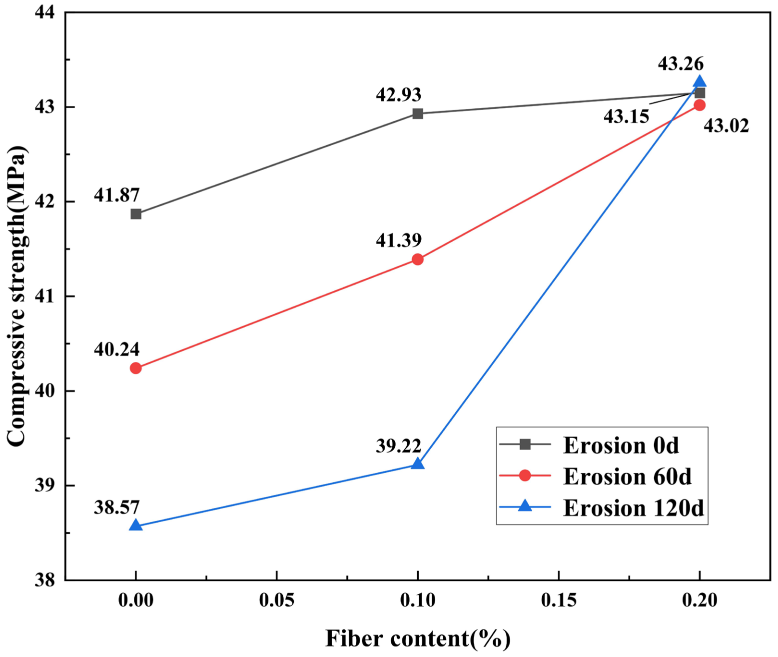
3. Test Results and Analysis

3.1. Effect of Different Influencing Factors on the Compressive Strength of BFRC

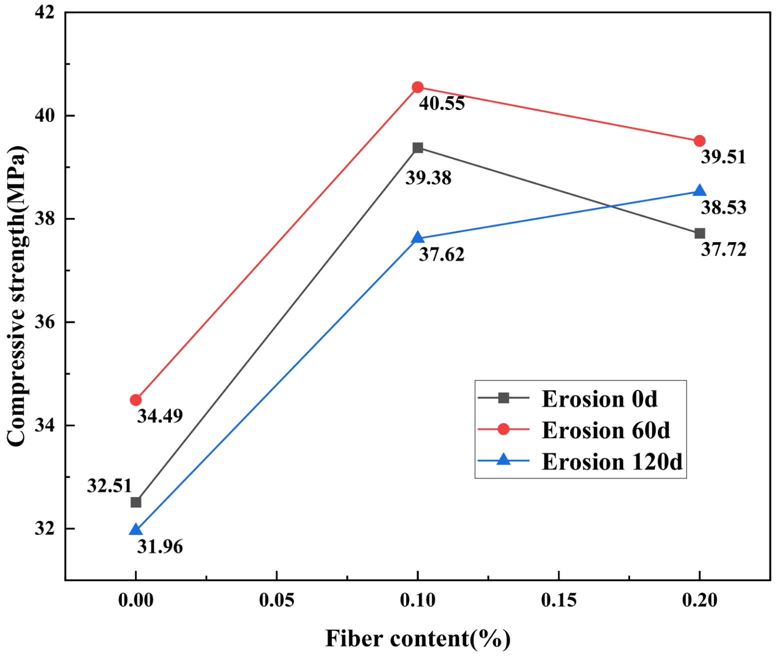
1) Effect of fiber dosage on BFRC compressive strength

The compressive strengths of specimens with different fiber dosages at room temperature and after undergoing three ultra-low temperature freeze-thaw cycles with temperature gradients from 25˚C to −160˚C are shown in **Figure 5**.

As can be seen in **Figure 5**, the compressive strength of the BFRC is enhanced with the increase in fiber dosage. This phenomenon is attributed to the fact that the addition of fibers enhances the internal densification of the specimen and fills its pores, thus significantly enhancing the compressive strength of the specimen [17]. The compressive strength of the specimens not subjected to sulfate attack at room temperature increased by 3.1% as the fiber dosage increased from 0% to 0.2%. The compressive strength of specimens subjected to sulfate attack after 120 d increased by 12.2% as the fiber doping increased from 0% to 0.2%. The compressive strength of specimens subjected to ultra-low-temperature freeze-thaw cycles and sulfate attack for 120 d increased by 20.6% when the fiber dosage was increased from 0% to 0.2%. The above data show that basalt fiber has an improvement effect on the compressive strength of specimens, and its improvement effect will be increased after sulfate erosion and ultra-low temperature freeze-thaw cycle. The reason is that the internal pores and cracks of the specimens subjected to sulfate erosion and ultra-low temperature freeze-thaw cycles will develop rapidly. More pores and cracks will give more space for fibers to play the role of filling and bridging, so that the compressive strength of the specimen is increased.



(a) At room temperature

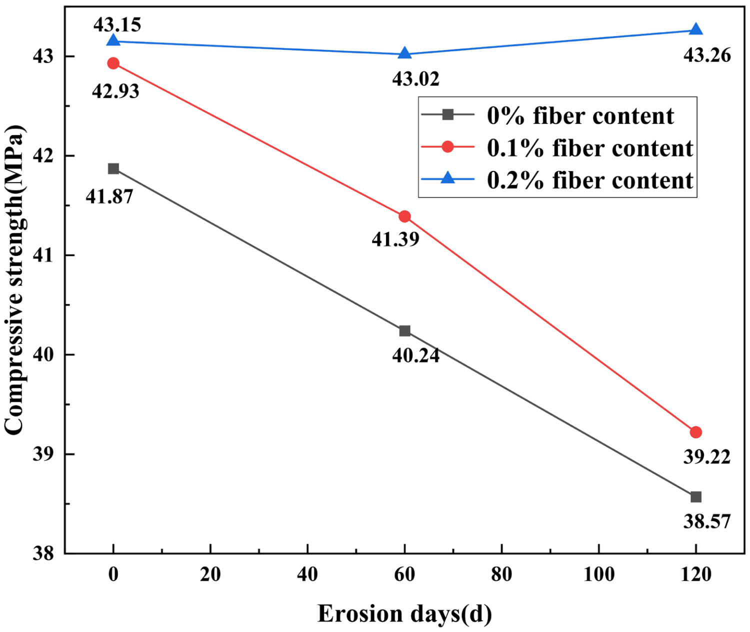


(b) 25˚C - −160˚C after freeze-thaw cycles

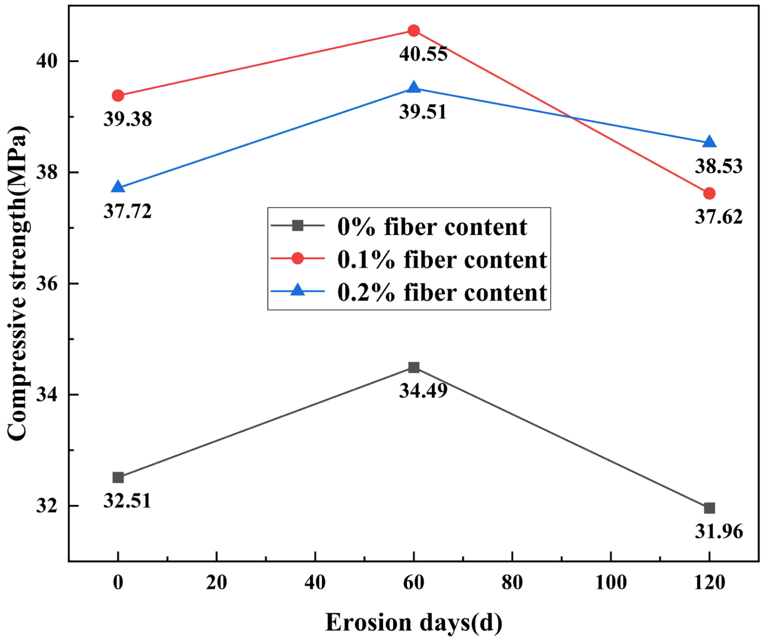
Figure 5. Effect of fiber dosage on compressive strength of BFRC.

2) Effect of erosion days on BFRC compressive strength

The compressive strengths of specimens with different erosion days at room temperature and after undergoing three ultra-low temperature freeze-thaw cycles with temperature gradients from 25˚C to −160˚C are shown in **Figure 6**.



(a) At room temperature



(b) 25˚C - −160˚C after freeze-thaw cycles

Figure 6. Effect of days of erosion on BFRC compressive strength.

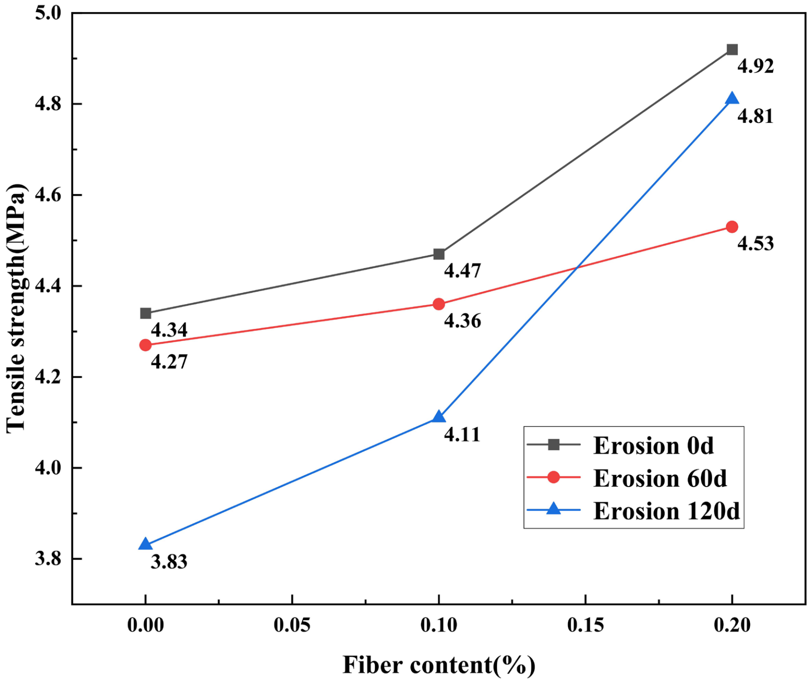
As can be seen in **Figure 6**, the compressive strength of BFRC at room temperature decreased with the increase in the number of days of erosion. In this case, the compressive strength of plain concrete experienced a decrease of 3.9% and 7.9% after 60 d and 120 d of sulfate erosion, respectively. The compressive strength of BFRC with 0.1% fiber admixture decreased by 3.6% and 8.6% after 60 d and 120 d of sulfate erosion, respectively. After ultra-low temperature freeze-thaw cycles, the compressive strength of plain concrete increased by 16% and then decreased after 60 d and 120 d of sulfate attack. The compressive strength of BFRC with 0.1% fiber dosage decreased by 13.5% after 60 d and 120 d of sulfate attack. The compressive strength of BFRC with 0.2% fiber doping decreased by 14% after 60 d and 120 d of sulfate attack. The compressive strength of BFRC after ultra-low-temperature freeze-thaw cycles showed an increasing and then decreasing trend with the increase in the number of erosion days. This phenomenon is mainly caused by two reasons. First, after the freeze-thaw cycle from 25˚C to −160˚C, the internal holes and cracks of the specimens develop more significantly. And the complex chemical reaction between sulfate and concrete will lead to the volume expansion of concrete and beside the generation of expansion stress. This leads to a short-lived increase in the strength of the concrete. However, over-expansion of the volume can cause cracking of the concrete leading to a reduction in the strength of the concrete [18]. Secondly, in the initial stage of sulfate attack, the sulfate within the concrete interacts with the cement hydration products to produce erosive substances that can fill the initial gaps in the concrete. With the continuous accumulation of erosive substances, microcracks and penetrating cracks gradually appeared in the concrete specimens, which led to the compressive strength showing a tendency of increasing and then decreasing [19]. The cracks and holes in the specimen at room temperature are relatively small, and the specimen is more likely to be over-expanded, so it eventually leads to a decreasing trend in the compressive strength of the specimen.

3.2. Effect of Different Influencing Factors on the Tensile Strength of BFRC

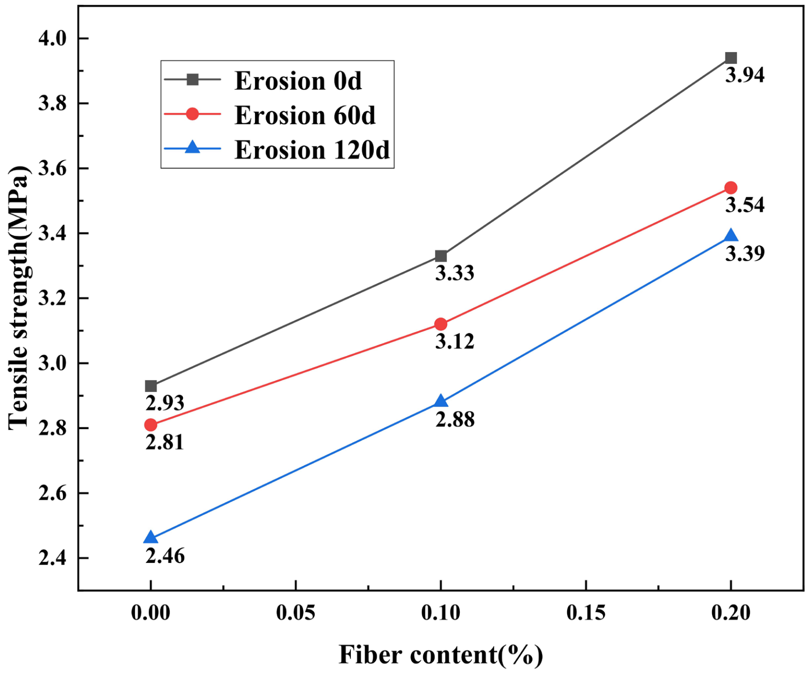
1) Effect of fiber dosage on BFRC tensile strength

The tensile strengths of specimens with different fiber dosages at room temperature and after undergoing three ultra-low temperature freeze-thaw cycles with temperature gradients from 25˚C to −160˚C are shown in **Figure 7**.

As can be seen in **Figure 7**, the general trend of the tensile strength of the BFRC specimens treated with sulfate erosion shows an elevated trend with the increase of fiber doping. The tensile strength of BFRC specimens subjected to erosion for 120 d at room temperature was enhanced by 7.3% and 25.6% when the fiber doping was increased from 0% to 0.1% and 0.2%, respectively. After freeze-thaw cycles from 25˚C to −160˚C, the tensile strength of BFRC specimens subjected to erosion for 120 d was increased by 17.1% and 37.8% when the fiber doping was increased from 0% to 0.1% and 0.2%, respectively. Obviously, the tensile strength of the specimens reached the maximum value when the fiber doping was 0.2%, which indicated that the fiber doping of 0.2% was the optimal doping. Basalt fibers have high modulus of elasticity and tensile strength, which enhance the tensile strength of the specimens by effectively bridging the cracks and strengthening the concrete at the microscopic level [20].



(a) At room temperature

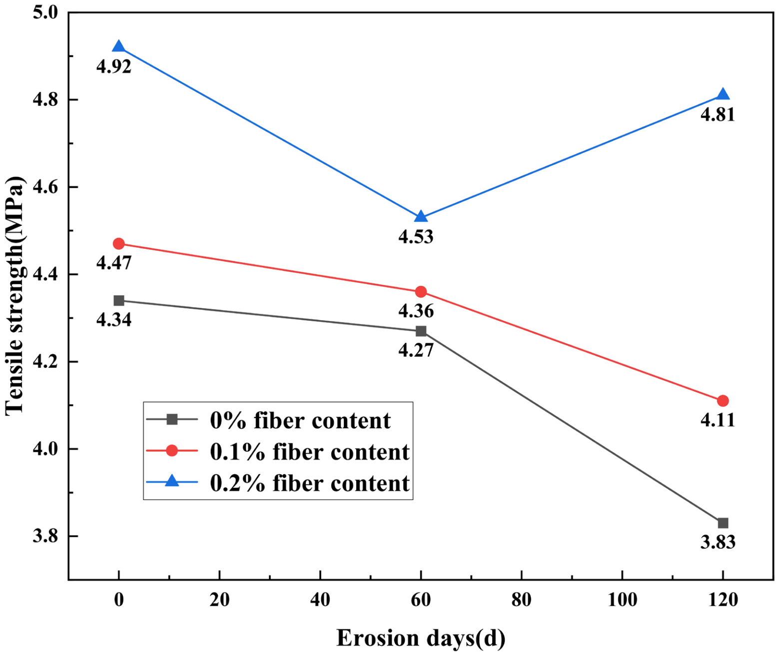


(b) 25˚C - −160˚C after freeze-thaw cycles

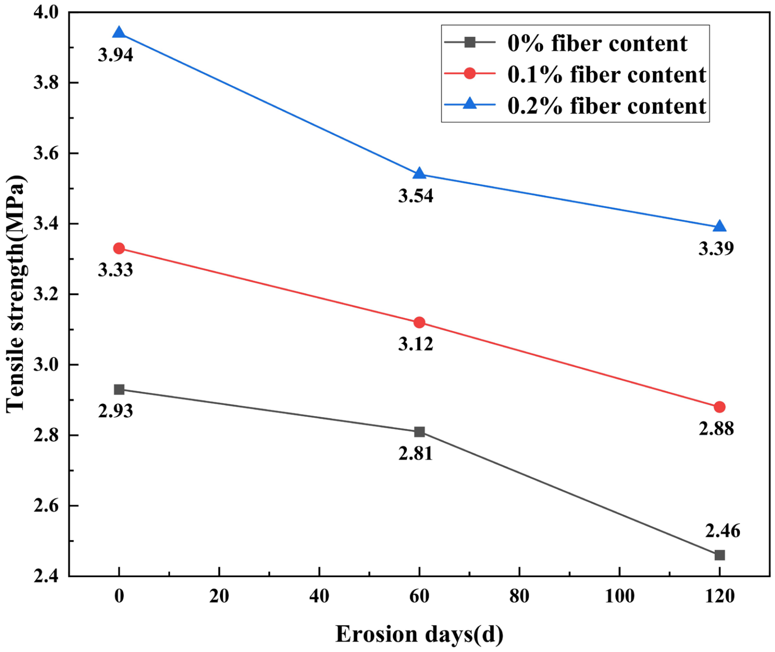
Figure 7. Effect of fiber dosage on tensile strength of BFRC.

2) Effect of erosion days on BFRC split tensile strength

The tensile strengths of specimens with different fiber dosages at room temperature and after undergoing three ultra-low temperature freeze-thaw cycles with temperature gradients from 25˚C to −160˚C are shown in **Figure 8**. As can be seen in **Figure 8**, the tensile strength of BFRC decreased with the increase in the number of days of erosion. The compressive strength of plain concrete subjected to 120 d of sulfate erosion at room temperature decreased by 11.8%.



(a) At room temperature

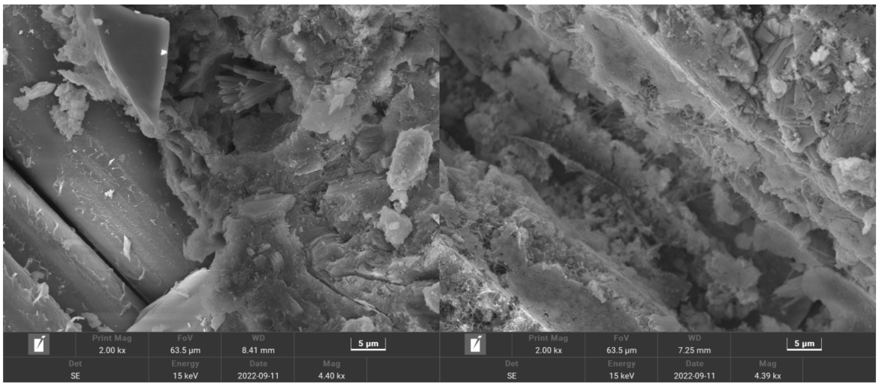


(b) 25˚C - −160˚C after freeze-thaw cycles

Figure 8. Effect of days of erosion on tensile strength of BFRC.

3.3. Microstructural Analysis of Eroded BFRC after Ultra-Low Temperature Freeze-Thaw Cycles

As shown in **Figure 9(a)**, large pores and cracks are clearly visible, indicating a loose structure and inherently low natural strength of the concrete. The chemical attack occurs when sulfate interacts with the hydration products of cement, leading to the formation of compounds such as caliche and gypsum. This process temporarily increases the density and overall quality of the concrete specimen. Conversely, physical erosion arises from the crystallization of sulfate, causing internal expansion. This leads to cracking, surface spalling, and a gradual loss of concrete quality.



(a) 0% BF, 25˚C (b) 0.2% BF, −160˚C

Figure 9. SEM image.

As shown in **Figure 9(b)**, the significant damage to the internal pore structure of specimens with a 0.2% fiber dosage after being subjected to ultra-low-temperature freeze-thaw cycles, ranging from 25˚C to −160˚C. A large number of cracks and pores are evident. Within the concrete structure, internal free water, adsorbed water, and capillary water in smaller pores freeze and expand due to the freezing and expansion forces. This phenomenon exerts pressure on the pore walls, causing microcracks in the concrete. As the lower temperature limit of the freeze-thaw cycles decreases, the pore sizes in the mortar gradually enlarge, and the cracks deepen.

This progression accelerates the infiltration of moisture from the external environment, amplifying the freezing and expansion effects and further intensifying concrete deterioration. Consequently, both the tensile and compressive strengths of the concrete gradually decline.

4. Conclusions

This paper investigates the mechanical properties of engineering structures subjected to an erosive environment after ultra-low temperature freeze-thaw cycles. Specifically, the paper focuses on the cubic compressive properties, splitting tensile properties and flexural toughness of BFRCs under extreme conditions, as well as the microstructural characteristics of BFRCs subjected to sulfate erosion after ultra-low temperature freeze-thaw cycles. The main conclusions drawn from the study are summarized below:

1) Basalt fibers can significantly improve the mechanical properties of concrete. The compressive strength of specimens subjected to erosion for 120 d after freeze-thaw cycles from 25˚C to −160˚C increased by 17.7% and 20.6%, and the tensile strength by 17.1% and 37.8% when the fiber dosage was increased from 0% to 0.1% and 0.2%, respectively. With the increase of sulfate erosion time, the decrease of tensile strength and compressive strength of the specimens became more and more significant, and the specimens subjected to erosion for 120 d after freeze-thaw cycles from 25˚C to −160˚C with 0.1% fiber doping had a decrease of 4.5% in compressive strength and a decrease of 13.5% in tensile strength.

2) The effects of ultra-low temperature freeze-thaw cycles and sulfate solution erosion on the internal deterioration mechanism of concrete were investigated. It was shown that the erosion products generated by the reaction of sulfate ions with concrete constituents are significantly expansive. In addition, the chemical reaction in the ionic solution produces crystalline salts and is accompanied by expansion stresses, which ultimately lead to cracking, surface spalling, and quality degradation of the concrete. After the ultra-low temperature freeze-thaw cycle, cracks and pores within the concrete develop rapidly, providing more space for the reaction of sulfate ions. This process further exacerbates the deterioration of concrete, resulting in a significant reduction in the mechanical properties of concrete.

The study focuses exclusively on cubic compressive and splitting tensile strength. It is worth noting that other critical properties, such as flexural strength and durability indicators (e.g., mass loss and scaling), are also significant. These aspects will be explored in future research.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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