

Research Article

Preparation of Nano-SiO₂/Carbon Fiber-Reinforced Concrete and Its Influence on the Performance of Oil Well Cement

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Received 22 May 2019; Revised 17 July 2019; Accepted 23 July 2019; Published 15 September 2019

Guest Editor: Mohammad Jawaid

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In view of the oilfield well thin oil layer, small gap, and side drilling cementing after perforating and subsequent stimulation caused by the cement ring embrittlement (i.e., secondary channeling), the preparation of nano-SiO₂/carbon fiber-reinforced body and its influence on the performance of oil well cement were studied to improve the cement stone and enhance its adaptability to oil well pressure in this study. Carbon fibers were treated by liquid phase oxidation and a coupling agent, and the “grafting to” was used to bond nano-SiO₂ and carbon fibers. It was found that the mechanical properties of the enhanced cement stone are far better than those of the blank cement stone. The compressive strength and tensile strength of the enhanced oil well cement stone were increased by 25% and 26%, respectively, compared with those of the blank oil well cement sample; the modulus of elasticity was reduced by 29%. Finally, the enhancement mechanism of SiO₂/carbon fiber reinforcement on cement stone was explored by infrared, scanning electron microscopy, and XRD patterns. The deflection effect, pull-out effect, and bridging effect of crack were obtained.

1. Introduction

Oil well cement stones are brittle materials and inevitably will suffer from fracturing, perforation, and various subsequent operations during the mining process. In addition, the underground rock formation will have complex effects on it, which will result in the oil well cement stone producing fine cracks and tiny voids. These voids cause the sealing effect of the oil well cement stone to be invalid, which is not conducive to the production and development of the oil well [1–3]. Carbon fibers are high-strength, high-elastic materials that not only increases the strength of cement but also improves toughness [4–6].

However, the surface of carbon fibers is inert, and surface bonding strength is not strong enough to combine with other substances. This requires modification of the surface of carbon fibers to improve bonding force [7]. On the basis of many years of research, some relatively mature fiber modification methods have been obtained, among which the main methods are as follows: oxidation treatment [8, 9], surface

deposition [10–12], surface coating [13–16], grafting modification [17, 18], or the comprehensive application of the above methods [19–22].

The liquid phase oxidation method is the most commonly used method of oxidation treatment and refers to the addition of an oxidizing liquid such as concentrated nitric acid to carbon fibers. The surface of carbon fibers is capable of generating acidic groups such as carboxyl groups (-COOH) or hydroxyl groups (-OH), and a certain degree of the etching effect is exerted on the surface of carbon fibers to make the surface rough [23, 24]. Lu et al. [25] performed liquid phase oxidation of carbon fiber in a 65% nitric acid solution and evaluated the relationship between liquid phase oxidation time and a carbon fiber structure by LRS, XRD, SEM, and FTIR. The results show that the corrosion increases with the increase of oxidation time, but the nitric acid oxidation treatment does not change the overall structure of the carbon fiber.

The use of coupling agents for the treatment of carbon fibers has been extensive [26]. Due to the small number of

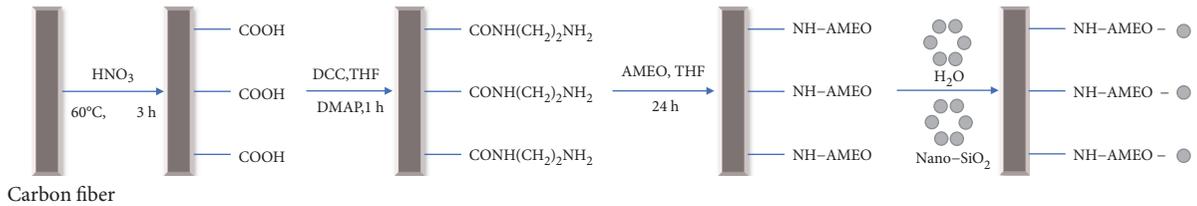


FIGURE 1: The processing steps of carbon fibers.

TABLE 1: Chemical composition of class G oil well cement.

Component	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	SO ₃	MnO ₂	Negligible component
Fraction (wt%)	22.7	3.39	4.81	65.5	0.9	0.37	1.21	0.09	0.25

TABLE 2: Mineral composition of class G oil well cement.

Component	C ₃ S	C ₂ S	C ₃ A	C ₄ AF
Fraction (wt%)	48~65	28~36	1~3	6~14

TABLE 3: Properties of carbon fiber.

Fiber length (μm)	100~1000
Fiber diameter (μm)	8~20
Tensile strength (MPa)	>3300
Bulk density (g/cm ³)	1.76
Elastic Modulus (GPa)	220

TABLE 4: Cement slurry formula.

Serial number	Name	Weight (g)
1	Cement	300
2	Water	132
3	Reinforcement	0.1, 0.2, 0.3, 0.4
4	G33S	3.0
5	CMC	0.6
6	Defoamer	Small amount

functional groups on the surface of carbon fibers, the effect of treatment with coupling agents is often less than ideal [19, 27]. Choim et al. [19] oxidized the fibers with carbon dioxide first, then treated with a coupling agent. The results show that the composite material reinforced with carbon fibers treated with nitric acid and then treated with a coupling agent is significantly stronger than the composite material reinforced with carbon fibers treated with a coupling agent alone.

The chemical grafting method is one of the grafting methods which chemically introduces reactive active sites on the surface of fibers and then initiates polymerization of monomers on the surface [28, 29]. In some cases, a certain amount of active groups such as -OH and -COOH are introduced on the surface of carbon fiber before the chemical grafting to improve the grafting effect [20, 21]. Guo-Liang et al. [22] used chemical grafting to modify the surface of carbon fibers. The carbon fiber was first pretreated with a mixture of nitric acid and sulfuric acid to form reactive functional groups on the surface, and then benzoyl peroxide (BPO) was used as the initiator that initiates polymerization of methyl methacrylate (MMA) monomers on the surface of carbon fibers. The chemical grafting methods can be further divided into "grafting from" and "grafting to." "Grafting from" is to initiate polymerization on the surface of carbon fibers by constructing initiators on the surface of carbon fibers. "Grafting to" is to synthesize polymer segments with reactive functional groups and then graft synthesized poly-

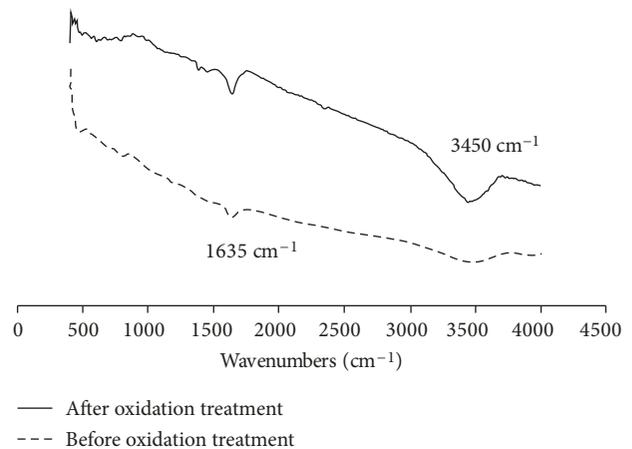


FIGURE 2: Infrared spectrum of carbon fibers before and after oxidation treatment.

mer segments onto the surface of carbon fibers by chemical reaction. The graft molecular structure and molecular weight are controllable, which are the most commonly used at present grafting methods [30].

The interaction between the nanoparticles in the matrix is more due to the same volume of nanoparticles which has more particles than ordinary particles, increasing the interfacial area and stress transfer between the matrix and the nanoparticles [31]. By chemically depositing or chemically grafting nanoparticle materials on the surface of inert carbon fibers, the surface area and surface activity can be improved, but the effect of chemical deposition is often inferior to that of chemical grafting [32].

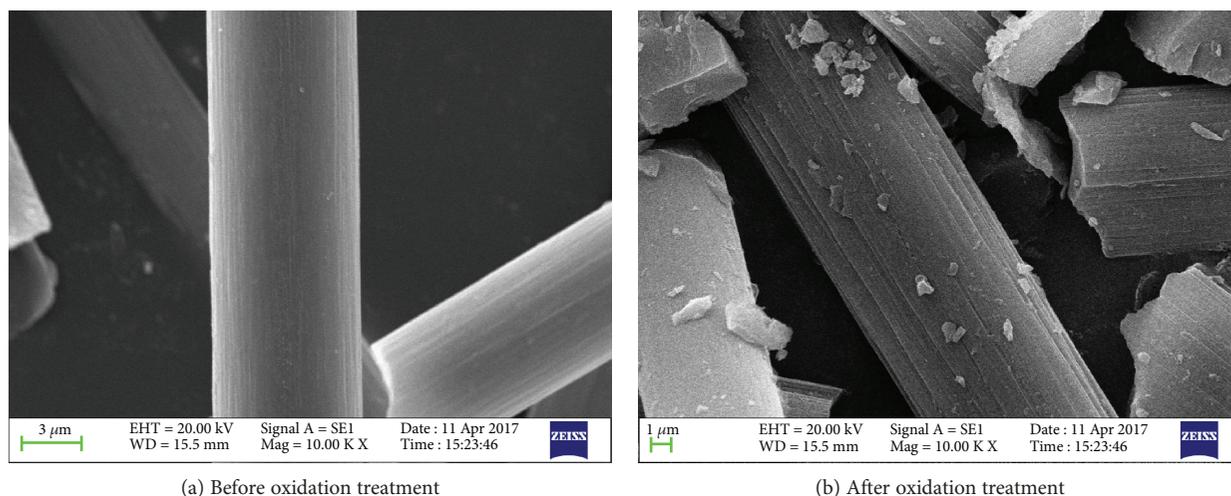


FIGURE 3: SEM image of carbon fibers before and after oxidation treatment.

As an inorganic filler with excellent performance, nano-SiO₂ is cheap and easy to obtain. In addition, nano-SiO₂ will provide sources of silicon for the hydration process of cement that is beneficial to the early strength development of cement [33, 34], so this study intends to construct nano-SiO₂/carbon fiber reinforcement through chemical bonding to enhance the toughening effect of the reinforcement at the interface between the carbon fiber and the cement matrix to better adapt to the working environment of the well. The effect of the oxidation is to introduce reactive acidic groups. 3-Aminopropyltriethoxysilane (AMEO) as a silane coupling agent was used to graft with nano-SiO₂. N,N'-Dicyclohexylcarbodiimide (DCC) and 4-dimethylaminopyridine (DMAP) could acylate the carboxyl group on the surface of carbon fibers to facilitate a connection with AMEO [35–37]. The processing steps of carbon fibers are shown in Figure 1.

2. Experiment

2.1. Experimental Materials

2.1.1. Cement. The cement material used in the study was silicate grade G oil well cement (Jiahua Special Cement, Leshan, Sichuan). The chemical composition is shown in Table 1, and the mineral composition is shown in Table 2.

2.1.2. Reinforcement Materials. In this study, PAN-based carbon fibers produced by Dalian Xingke Carbon Fiber Co. Ltd. were used, with a short fiber length of 100 μm (±2 μm) and a long fiber length of 1000 μm (±2 μm). The properties of carbon fibers are shown in Table 3. The nano-SiO₂ was purchased from the Changtai Micro-Chemical Factory, Shouguang City, Shandong Province; concentrated nitric acid and other chemical reagents (analytical grade) were from Chengdu Kelong Chemical Reagent Factory.

2.2. Experimental Methods

2.2.1. Preoxidation of Carbon Fibers. The surface of the carbon fiber is first washed with a mixed liquid of absolute ethanol and acetone reagent. Weigh 12 g of carbon fiber

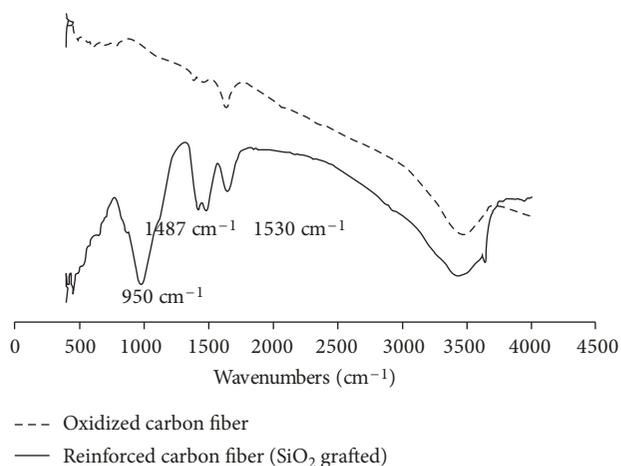


FIGURE 4: Infrared spectrum of the reinforcement and unreinforced carbon fibers.

and place it in a beaker, then weigh 100 mL of absolute ethanol and 100 mL of acetone into a beaker, stir well, and finally place the beaker in an ultrasonic cleaning machine (BL10-300, Shanghai Billion Instrument Co. Ltd.) for 1 h. The beaker was taken out, and the carbon fiber was washed with water and dried in an electric blast drying oven (101-2A, Beijing Zhongxing Weiye Instrument Co. Ltd.) for 10 h.

The carbon fiber was taken out and added to a beaker containing 200 mL of concentrated nitric acid, stirred well with a high-speed variable frequency stepless speed mixer (CJS-B12K, Qingdao Tongchun Petroleum Instrument Co. Ltd.), sealed with a plastic wrap, and tied with a rubber band. The beaker was placed in a water bath of 60°C (DZKW-4, Beijing Zhongxing Weiye Instrument Co. Ltd.) for 3 h.

2.2.2. Preparation of Reinforcement. In the experiment, the carbon fiber was subjected to a coupling agent coating treatment and grafted with nano-SiO₂.

The operation steps are as follows: The oxidized carbon fiber, DCC, and DMAP were added to tetrahydrofuran (THF), and the ultrasonic dispersion was carried out for

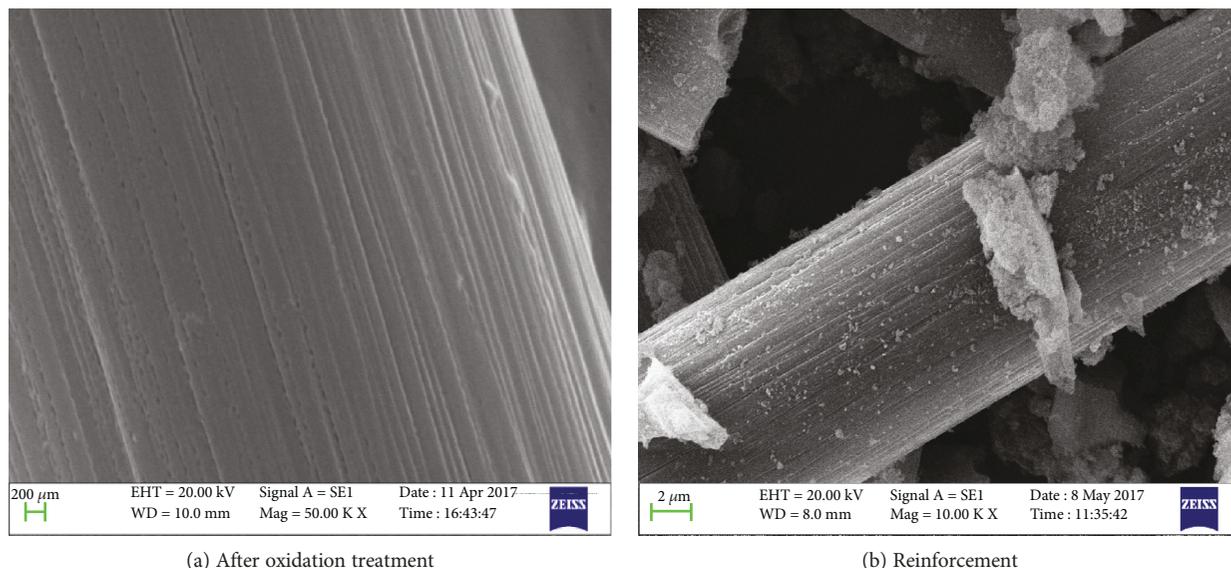


FIGURE 5: SEM image of the reinforcement and unreinforced carbon fibers.

1 hour, then AMEO was added, and the reaction was stirred magnetically for 24 hours. Finally, it is washed and dried with THF except water, and the dried product and silica are added to deionized water for ultrasonic dispersion for 1 hour and dried by suction filtration to obtain a reinforcement.

2.2.3. Preparation of Oil Well Cement Stone Samples. 0.1%, 0.2%, 0.3%, and 0.4% of the reinforcement were added to the cement slurry to prepare cement stone. The slurry formulation is shown in Table 4. In addition, the experiment also sets up a blank group of cement stone (the blank group is pure cement stone without any reinforcing material). The configured slurry was poured into various molds, and the molds were placed in a constant temperature water bath at 60°C for 3, 7, and 14 days of curing.

2.2.4. Testing Procedures. The cured rectangular specimens ($50.8 \times 50.8 \text{ mm} \times 50.8 \text{ mm}^3$) and the cured cylindrical specimens ($\Phi 50.0 \times 25.0 \text{ mm}^3$) were used to measure tensile and compressive strength by using electronic hydraulic testing machines (Haizhi Technology Co. Ltd., Beijing, China). Triaxial stress-strain curves were generated on a triaxial rock testing system (INT-2200, GCTS) by using a rectangular specimen ($40.0 \times 40.0 \times 160.0 \text{ mm}^3$).

To further analyze the phase composition, the type and microstructure of the hydration product have an effect on the mechanical properties of the cement stone. X-ray diffraction (Shimadzu, XRD-7000 Japan), scanning electron microscopy (SEM, JEOL JSM-6510LV, Japan), and thermal analysis (TG, JYJK-ELEC. Ltd., Beijing) were used for comprehensive analysis.

3. Results and Analysis

3.1. Infrared Analysis and SEM Characterization of Carbon Fibers. As shown in Figure 2, both lines have stretching vibration absorption peaks around 3450 cm^{-1} and 1635 cm^{-1} , which corresponds to the hydroxyl group (-OH) and car-

boxyl group (C=O), but the absorption peak of the carbon fiber increased after the oxidation treatment. This indicates that the number of oxygen-containing functional groups (C=O, -OH) on the surface of the carbon fiber was increased by acid oxidation treatment. An increase in aerobic functional groups could effectively increase the activity of the carbon fiber surface, enhancing its surface reactivity [38].

As shown in Figure 3, the surface of the fiber is more rough after concentrated nitric acid treatment, and many gullies and lines are formed. These gullies formed on the surface of the carbon fiber increase the specific surface area of the carbon fiber. The surface of the oxidized carbon fiber adsorbs some fine particles, which may be caused by a small portion of the carbon fiber being corroded by concentrated nitric acid.

3.2. Infrared Analysis and SEM Characterization of the Reinforcement. As shown in Figure 4, the reinforcement showed a new peak corresponding to the acylamino group (-CO-NH-) at 1530 cm^{-1} , indicating that the aerobic functional group on the surface of the carbon fiber did undergo acylation reaction [35–37]. A new strong Si-O absorption peak appeared around 950 cm^{-1} owing to the grafted SiO_2 carried a large amount of Si-O bonds. At 1487 cm^{-1} , a weak absorption peak of methylene (-CH₂) by a coupling agent appeared.

As shown in Figure 5, the main feature of the oxidized carbon fiber was that the surface of the fiber column has many obvious gullies. In the scanning electron micrograph of the reinforcement, the surface of the reinforcement was covered with a layer of granular material, and the shape of these materials is consistent with the shape of the nano- SiO_2 photographed in Figure 6, which can be basically determined to be nano- SiO_2 . The results of the analysis in Figures 4–6 show that nano- SiO_2 has been successfully grafted onto the surface of carbon fibers.

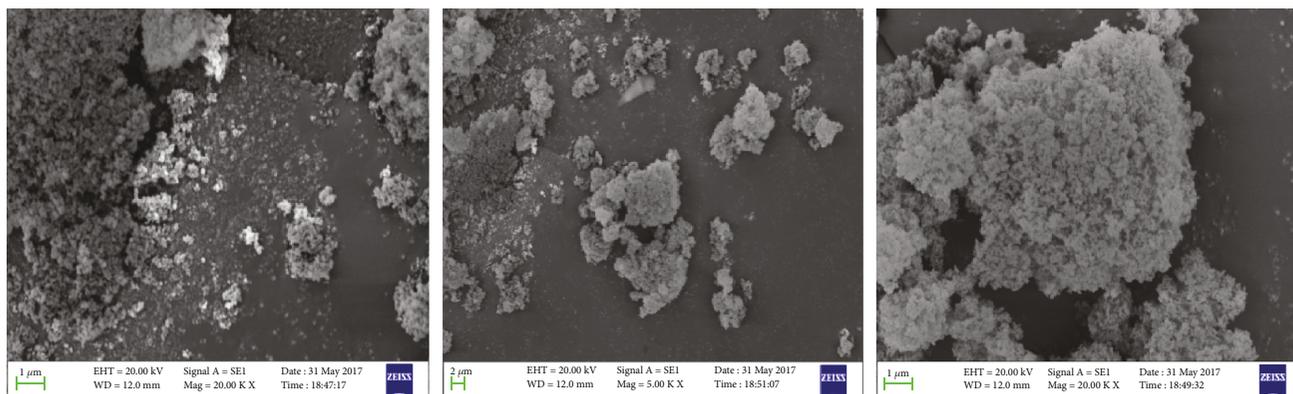
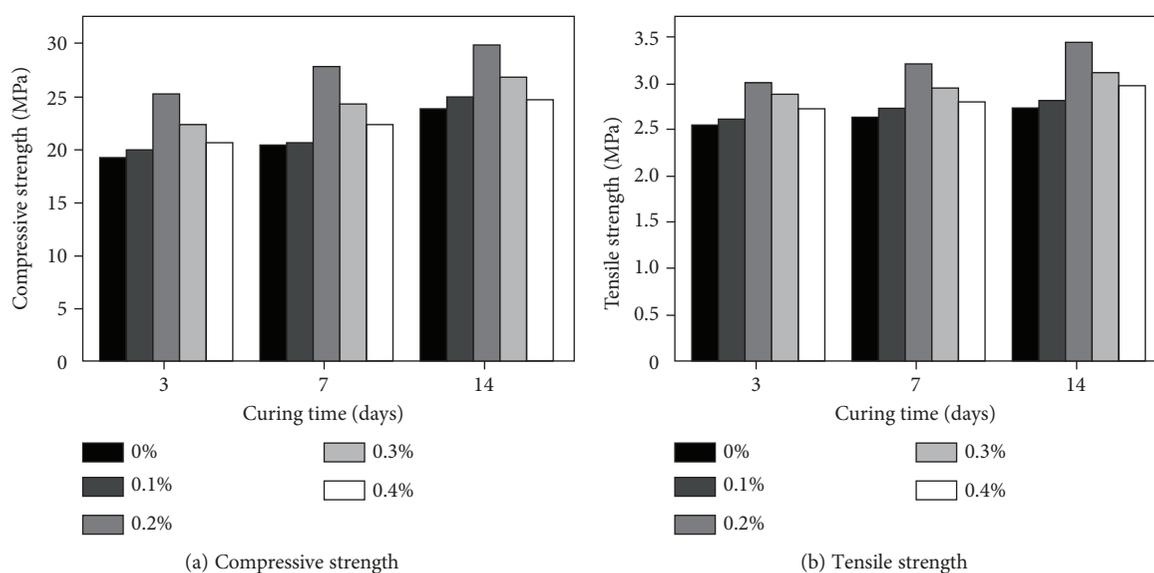
FIGURE 6: SEM pattern of SiO₂.

FIGURE 7: Strength of reinforced oil well cement stone.

3.3. Mechanical Properties of Cement Stone. According to Figure 7, when the curing time was the same and the dosage did not exceed 0.2%, the compressive strength and the tensile strength of the cement stone increase with the increase of the reinforcement in the oil well cement stone. When the amount reached 0.2%, the compressive strength and the tensile strength were maximized, and thereafter, the compressive strength and the tensile strength were gradually lowered [39, 40]. The strength development of the reinforced cement stone was very significant compared to pure cement stone and mixed cement stone, and it had a considerable enhancement effect when the curing time reaches 14 days. At 14 days of curing, the compressive strength and the tensile strength of the reinforcement cement stone increased by 25% and 26%, respectively. The extension of the hydration time contributes to the combination of the carbon fiber and the cement matrix, thereby better enhancing the reinforcing effect of the carbon fiber in the cement matrix [41].

The triaxial test was carried out under the conditions of a confining pressure of 20.7 MPa and a temperature of 60°C.

TABLE 5: Triaxial test results.

	Modulus of elasticity (MPa)	Differential stress (MPa)	Peak strain (%)
Before enhancement	6706.9	40.8	1.8
After enhancement	4736.1	50.0	4.1

The selected samples were cement stone with 0.2% reinforcement and blank samples (curing time was 7 days).

As shown in Table 5 and Figure 8, the oil well cement stone stress curve has an approximate straight line before reaching the maximum value. In this interval, the cement stone mainly shows elastic deformation. After the carbon fiber was added into the cement stone, the whole stress-strain process was prolonged, the ultimate stress of the cement stone was increased by about 25%, the elastic modulus was lowered by 29%, and the peak strain was increased by 144%. Therefore, the addition of reinforcements enhanced the ductility and toughness of cement stone [42].

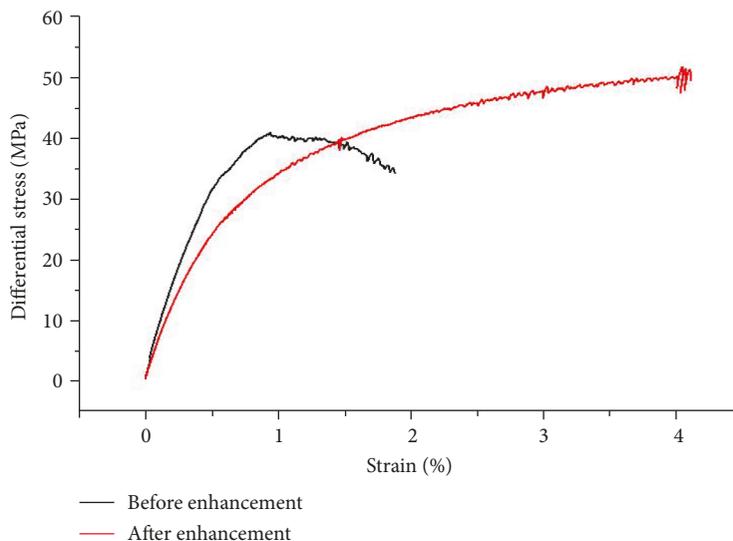
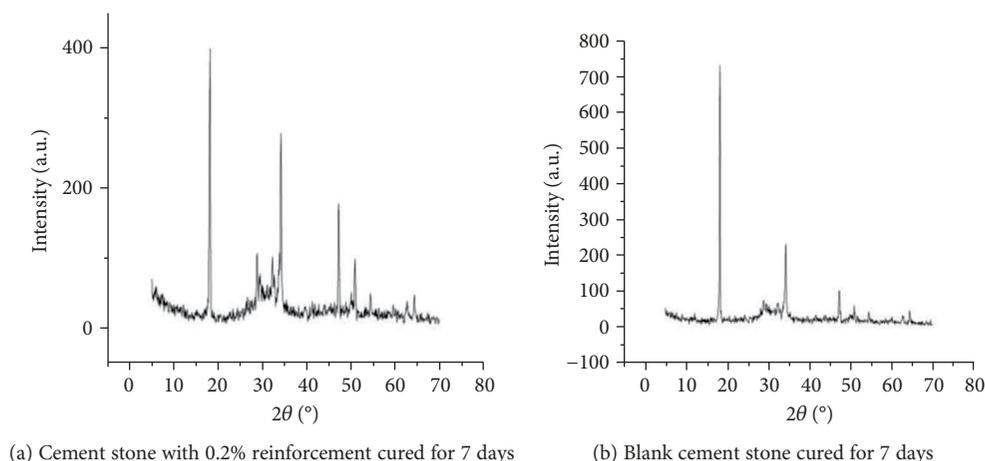


FIGURE 8: Stress-strain curves of cement stone.



(a) Cement stone with 0.2% reinforcement cured for 7 days

(b) Blank cement stone cured for 7 days

FIGURE 9: XRD pattern of cement stone.

3.4. Microscopic Characterization of Oil Well Cement Stone.

As shown in Figure 9, after the reinforcement was added, the type of the hydration product did not change significantly. The C-H bond corresponds to a sharp crystallization peak of $2\theta = 18^\circ$, 34° , and 47° , and the C-S-H bond was mainly concentrated at $2\theta = 30^\circ$ and 50° . However, the peak intensity corresponding to C-S-H was increased because nano-SiO₂ reacts with calcium hydroxide precipitated upon hydration to form C-S-H, which reduced the calcium hydroxide of the layered structure and increased C-S-H [33, 34]. Therefore, the grafting of nano-SiO₂ on carbon fibers will affect the early hydration process of cement stone and be beneficial to early strength.

The role of carbon fibers in the cement matrix is to delay and prevent the expansion of microcracks in the matrix and become the main carrier of the applied load [43, 44]. When the carbon fiber-reinforced cement stone is broken by an external force, if the plane of the crack

development is perpendicular to the carbon fiber (Figure 10(c), blue mark), the carbon fiber is pulled out from the cement stone and peeled off (Figure 10(c), red mark). The stripping process will dissipate a part of the energy, so the mechanical properties of the cement stone will increase [45]. It can be seen from Figures 10(a) and 10(b) that when the crack passes through the carbon fiber, the carbon fiber can crack at the tip by the bridging action of the carbon fiber in the cement stone. For bridging, there is a closed stress, which prevents the crack from continuing to expand to a certain extent and improves the mechanical properties of the cement stone [42].

4. Conclusions

- (1) Enhanced cement stone exhibits excellent mechanical properties. The compressive strength of the enhanced oil well cement stone was increased by

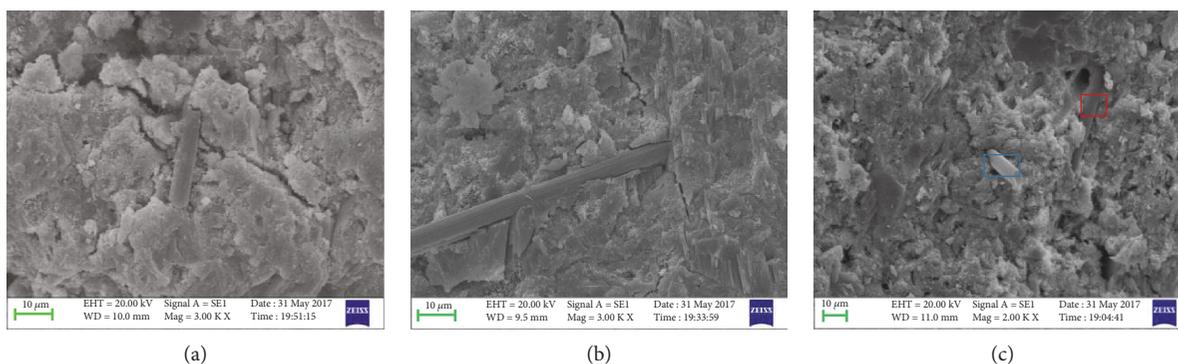


FIGURE 10: SEM pattern of cement stone with reinforcement.

about 25% compared with the blank cement, and the tensile performance was improved by about 26%. The ultimate stress of the cement stone was increased by about 25%, the elastic modulus was lowered by 29%, and the peak strain was increased by 144%

- (2) Carbon fiber successfully grafted nano-SiO₂ through a series of treatments; nano-SiO₂ grafted on the surface of carbon fibers has a certain influence on the hydration process of cement. The mechanism of carbon fiber reinforced cement stone is mainly the bridge effect of crack and the pull-out effect of crack

Data Availability

All data generated or analyzed during this study are included in this manuscript (and its supplementary information files). So the researchers can obtain all the data to verify the results, replicate the analysis, and conduct secondary analyses by reading our manuscript.

Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

Acknowledgments

The authors acknowledge the financial support provided by the National Science and Technology Major Project (2016ZX05045-001-007), Special Support Plan for High-Level Talents in Chongqing (CSTCCXLJRC 201911), and National Natural Science Foundation of China (51874254).

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