

Article

Effect of High Temperature on Micro-Structure and Mechanical Properties of Fiber-Reinforced Cement-Based Composites

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Abstract: Synthetic fibers can effectively inhibit the formation and propagation of micro-cracks in concrete, significantly reducing the number and scale of cracks within the concrete matrix, thereby enhancing the concrete's crack resistance and seepage prevention capabilities. In this study, two types of synthetic fibers, polyvinyl alcohol (PVA) and polypropylene (PP), were incorporated into cement mortar to investigate their microstructural evolution at elevated temperatures and their influence on the mechanical properties of the mortar. Both fibers were added at a volume content of 0.5%. The mortar samples were subjected to the following temperature conditions: 20 °C (ambient), 200 °C, 400 °C, and 500 °C. The results indicate that the synthetic fibers employed in this study improved the tensile properties of the mortar at room temperature (20 °C). This enhancement persisted up to 400 °C, beyond which, at 500 °C, the mechanical properties of the fiber-reinforced mortar deteriorated significantly. At 400 °C, the tensile strength of the PVA group increased by approximately 16% compared to the unblended fiber group (JZ) and by about 45% compared to the PP group. After treatment at 500 °C, the tensile strength of mortar specimens in the PVA group and the PP group decreased by 36.47% and 24.14%, respectively, compared with that at 20 °C. The porous structure formed due to the high-temperature ablation of the synthetic fibers contributed to relieving the internal pressure within the mortar.

Keywords: synthetic fibers; fiber-reinforced mortar; high-temperature performance; micro-structure



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1. Introduction

In recent years, extensive research has highlighted that fires are a prevalent disaster affecting engineering structures [1,2]. During a fire, high temperatures can cause the decomposition of hydration products within cement-based composite materials, making their microstructure porous [3,4]. This change significantly reduces the mechanical properties of these materials, sometimes leading to complete failure [5]. More critically, the diminished load-bearing capacity of engineering structures due to fires poses a significant threat to human life and property. Therefore, cement-based composite materials must not only exhibit excellent mechanical performance but also possess exceptional high-temperature resistance to ensure stability and reliability under extreme conditions, such as fires.

The damage and degradation of cement-based materials at high temperatures are caused by various factors, with prominent mechanisms being steam pressure stress and thermal stress [6]. Besides internal damage, cement-based materials are also susceptible to bursting and spalling under high temperatures. The steam pressure stress mechanism indicates that free and bound water inside cement-based materials begins to evaporate and spread as temperature increases. The dense hardened cement paste prevents water vapor escape, generating steam pressure. When this pressure exceeds the material's tensile strength, it leads to high-temperature damage [7,8]. The thermal stress mechanism suggests

that the internal thermal expansion coefficient of cement-based materials is inconsistent, causing uneven heat transfer and resulting in a temperature gradient. This gradient generates thermal stress, deforming the material's structure, and when the thermal stress exceeds the material's critical tensile strength, it causes cracking and failure.

Due to the complex and random nature of internal pore cracks in cement-based materials, it is often necessary to incorporate the thermal cracking mechanism alongside the first two mechanisms for problem analysis [9]. According to this mechanism, cement-based materials are heterogeneous, with differing thermal properties between internal aggregates and cement mortars [10]. Continuous high temperatures damage the internal structure, and the thermal expansion mismatch between cement mortar and aggregate causes micro-cracks at the bonding interface [11].

Certain fibers can significantly enhance the internal bridging capacity of cement-based materials and increase their strength [12]. Among these, steel fiber is the most studied. Steel fiber, with its high tensile strength and melting temperature, can improve the strength and plastic deformation capacity of cement-based materials [13]. Synthetic fibers such as PP and PVA exhibit good dispersion and strong toughness, and their appropriate addition can impart excellent anti-shrinkage properties to cement-based materials [14]. Numerous tests have demonstrated the excellent mechanical properties of fiber-reinforced cement-based composites. Additionally, research on the high-temperature properties of these composites has made notable progress both domestically and internationally. Cavdar mixed three different types of polymer fibers—copolymerized polypropylene (CPP), homopolymer polypropylene (HPP), and aramid (AR)—into cement and compared the effects of high temperature on the resulting fiber-reinforced cement-based composites. The study found that the composites began to crack and break at 450 °C. The loss of flexural strength in the cement matrix without fiber was about 1.5 times greater than in the composite material with HPP and AR, and about three times greater than in the composite material with CPP [15]. Peng Yu et al. created fiber-reinforced cement-based composites by mixing PVA fiber and steel fiber, studying the effects of high temperature on their residual flexural strength and microstructure. The results indicated that increased temperature promoted the secondary hydration of fly ash, making the composites denser. At 400 °C, mass loss increased significantly. The gradual decomposition of hydration products such as calcium hydroxide led to an increase in internal defects [16]. Tian Ludan et al. investigated the morphological changes, mass loss rate, compressive strength, and flexural strength of fiber-toughened cement-based composites at high temperatures. The results showed that high temperature affected the mass loss rate of the specimens, but the fibers began to melt and volatilize around 300 °C. When the temperature exceeded 500 °C, the holes left by the melted fibers expanded, resulting in reduced material strength [17].

Relevant studies have shown that polypropylene fibers and polyvinyl alcohol fibers enhance the high-temperature mechanical properties of cement-based composites and can mitigate explosive spalling [18]. To further investigate the effect of fibers on the mechanical properties of fiber-reinforced cement-based composites after exposure to high temperatures, and to understand the evolution of their internal microstructure and mechanical properties, additional research is needed. This study uses polyvinyl alcohol fibers and polypropylene fibers as synthetic reinforcements in mortar specimens for high-temperature tests. Utilizing Scanning Electron Microscopy (SEM) and a tensile testing system, the specimens are subjected to a constant tensile load of 0.05 mm/min, while real-time changes in the microstructure are documented. In this paper, a mold is designed for the production of fiber tensile specimens. After temperature treatment, the specimen is placed on the tensile table equipped with SEM. In the process of testing the tensile strength of the specimen, the microscopic appearance of the specimen from the beginning of being stretched to breaking is recorded in real time, and the mechanical properties of each stage are analyzed. This paper explores the evolution of the internal microstructure and mechanical properties of fiber-reinforced mortar under high-temperature conditions.

2. Materials and Methods

2.1. Test Material

The experiment used P.O 42.5 cement produced by the Green Poplar Cement Plant in Yangzhou, with chemical composition shown in Table 1.

Table 1. Chemical composition of cement.

Ingredient (%)	SiO ₂	CaO	Al ₂ O ₃	Fe ₂ O ₃	MgO	SO ₃	Na ₂ O	Other
Content	5.10	45.20	43.90	0.7	1.70	0.28	0.70	2.42

The PVA fibers and PP fibers used in the experiment are depicted in Figure 1, and their basic physical properties are listed in Table 2.

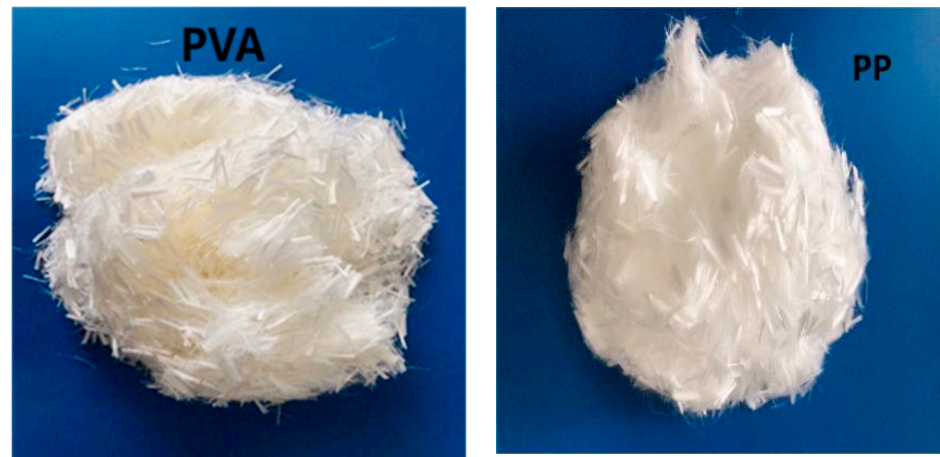


Figure 1. PVA fiber and PP fiber sample images.

Table 2. Basic physical properties of synthetic fibers.

Name	Diameter / μm	Strength /MPa	Length /mm	Elastic Modulus/MPa
PVA	17.26	813.31	13	12454.03
PP	31.12	510.42	12	4653.73

The sand used in the experiment was medium sand from Zone II, with basic performance indicators presented in Table 3.

Table 3. Basic performance indicators of sand.

Basic Properties	Apparent Density /(kg/m^3)	Clay Content/%	Fineness Modulus /M _x
Results	2620	1.2	2.6

2.2. Specimen Making and Temperature Treatment

2.2.1. Specimen Making

In material science, understanding the dispersion and orientation of fibers within fiber-reinforced mortar specimens is crucial, as these factors significantly influence the material's mechanical properties and overall performance [19]. Typically, the fibers in such specimens are distributed randomly, complicating experimental analysis and observation due to the unpredictability of fiber location and orientation [20].

To address this challenge and enhance the reliability of our experimental observations, this paper introduces a specialized approach involving the design and fabrication of custom

molds. These molds are tailored to align with both the unique requirements of the testing device and the specific arrangements of the fibers within the mortar. By standardizing the fiber positions, we facilitate more controlled experiments and generate more consistent, reproducible results.

Figure 2a illustrates the design of the custom mold, while Figure 2b details its precise dimensions. The design considerations for these molds include ensuring uniform fiber distribution, accommodating mechanical loading points, and allowing easy demolding without damaging the specimen. These molds not only enhance the precision of fiber placement within the mortar but also significantly improve the ease and accuracy of subsequent mechanical testing and analysis.

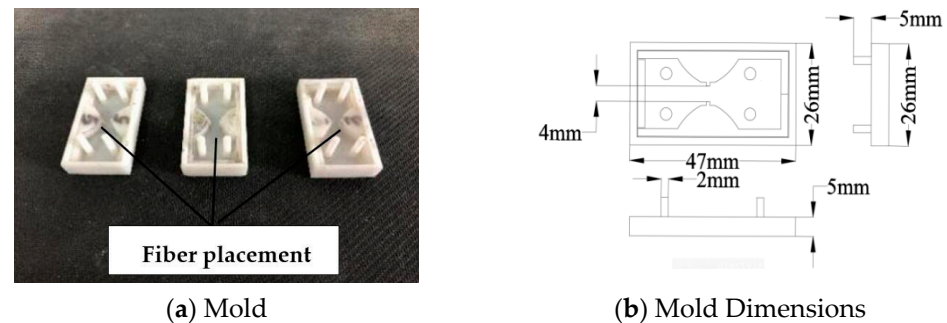


Figure 2. Microscopic test mold and mold dimensions.

By employing this innovative molding technique, our study aims to provide a clearer, more detailed understanding of the interactions between fiber orientation, distribution, and the mechanical properties of fiber-reinforced mortar.

The preparation of the specimens involves several meticulous steps to ensure their uniformity and quality:

1. **Mixing of Materials:** According to the standard “Synthetic fiber for cement concrete and mortar” (GB/T21120-2018) [21], the mass mix ratio of mortar is cement: sand: water = 1:1.5:0.5. This mixture is then thoroughly stirred for 3 min.
2. **Molding Process:** After mixing, the mortar is carefully poured into molds in two distinct layers to ensure even distribution and minimize air pockets, which can impact the mechanical properties. After the first layer is applied, fibers are strategically placed at precisely defined locations within the mold, as illustrated in Figures 1 and 2a. This placement is designed for experimental observation and does not reflect actual engineering conditions. To facilitate observation, 30 fibers were selected and placed in the fiber-doped sample. In order to avoid fiber clumping, the fiber is better dispersed for test observation. Select 30 fibers with tweezers and place them one by one parallel to the long side of the mold in the corresponding position (the position is indicated in Figure 2a). The sample group mixed with polypropylene fiber was named the PP group, the sample group mixed with polyvinyl alcohol fiber was named the PVA group, and the sample group without fiber was named the JZ group.
3. **Final Layer and Surface Finishing:** Following fiber placement, the second layer of mortar is applied. Care is taken to ensure this top layer fully encapsulates the fibers, promoting effective bonding within the matrix. The mortar surface is then smoothed to achieve a flat and even finish, essential for reducing imperfections that might affect subsequent testing.
4. **Curing of Specimens:** Once the specimens reach initial setting, they are placed in water at 20 ± 2 °C for 3 days. This water curing process is crucial for cement hydration, allowing it to harden properly and develop optimal mechanical properties. It also helps mitigate early-age thermal stresses that could lead to cracks.
5. **Demolding:** After the curing period, the specimens are carefully removed from their molds. This step is delicate, as improper handling can cause surface blemishes or structural defects.

These steps are structured to ensure each specimen is robust enough to undergo stringent testing protocols, providing reliable insights into the effect of fiber reinforcement under various thermal conditions.

In this comprehensive study, three distinct groups of specimens were meticulously crafted to investigate the influence of fiber compositions on the mechanical properties of mortar under various temperature treatments. The groups included a blank control, referred to as JZ, which contained no fibers and served as a baseline for comparison. The experimental conditions were represented by two additional groups: one infused with polyvinyl alcohol fibers (PVA) and the other with polypropylene fibers (PP).

2.2.2. Temperature Treatment

In this research, the thermal behavior of fiber-reinforced mortar specimens was carefully evaluated under a series of distinct temperature conditions. Specifically, temperatures of 20 °C, 200 °C, 400 °C, and 500 °C were chosen to analyze the thermal stability and performance changes of these engineered materials. The ambient temperature of 20 °C served as a baseline control group, providing a reference point to assess the impact of increased temperatures on the physical and mechanical properties of the specimens.

To systematically investigate the effects of high temperatures on the specimens, a controlled heating protocol was implemented. The temperature of the specimens was increased at a uniform rate of 5 °C per minute until reaching the predetermined target temperature (200 °C, 400 °C, or 500 °C). This gradual approach ensured consistent thermal exposure, minimizing thermal shock that could lead to uneven heating or damage. In particular, because the heating rates of different fire event vary, the heating rate used in this study is only valid for the specific materials and load conditions examined.

Once each target temperature was achieved, it was meticulously maintained for two hours. This hold period was crucial for allowing the specimens to fully acclimate to the elevated temperature, ensuring that any thermal-induced transformations in the material's structure or properties were fully developed. This step allowed for the assessment of the mortar's durability and structural integrity under sustained thermal stress.

Following this extensive conditioning at high temperatures, the specimens were allowed to cool passively within the furnace. This natural cooling process is vital as it mimics real-life scenarios where materials cool down at uncontrolled rates after being exposed to high temperatures. This stage was essential for observing the residual effects of thermal exposure on material performance, including potential degradation or strength loss. The specimens treated with the corresponding temperature will be sealed with plastic wrap for subsequent tensile testing.

Figure 3 in the documentation visually represents this meticulous temperature treatment methodology. After cooling, precautions were taken to preserve the condition of the specimens by sealing them in plastic bags, preventing any moisture or environmental contamination from influencing the results until further mechanical testing could be conducted. To minimize the influence of storage time on the test results, tensile tests were conducted on the second day after the heating process. Three specimens of each type were treated at the corresponding temperatures, and tensile tests were performed, with the results averaged.

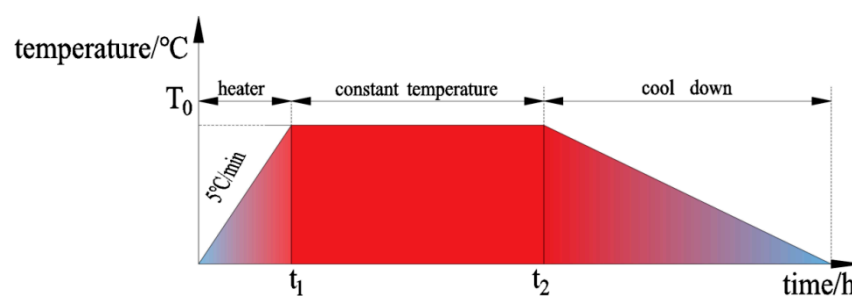


Figure 3. Schematic of the temperature treatment process.

2.3. Specimen Tensile and Real-Time Microstructure SEM

Using the SEM and its real-time loading test system, the effects of different fibers on the post-high-temperature tensile properties of concrete were analyzed based on changes in the load–displacement curves recorded by the tensile testing system and the patterns of microstructural changes in the specimens. Figure 4 shows the SEM and its real-time loading test system.

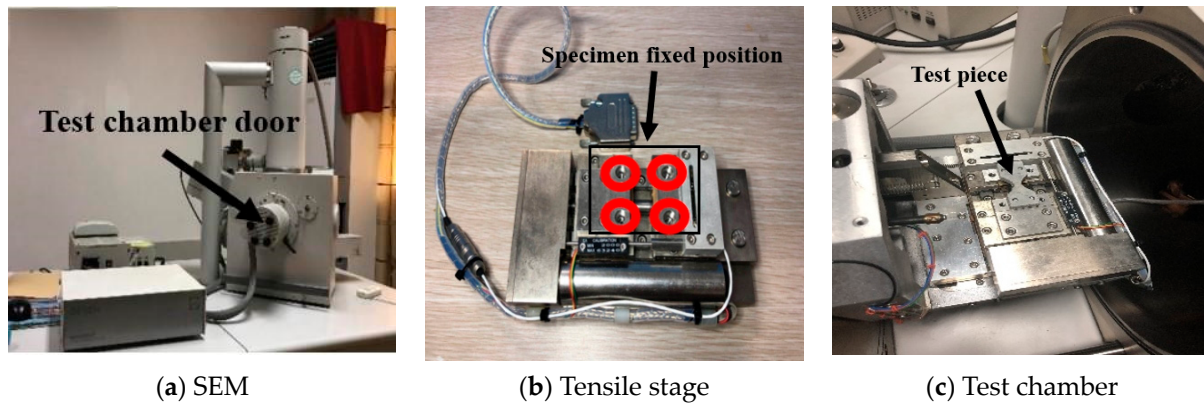


Figure 4. SEM and its real-time loading test system.

The process of specimen tensile and observation is as follows: (1) the tensile table is fixed to the SEM observation table; (2) reset the drawing part of the drawing table to the initial position; (3) put the circles at the two short ends of the test piece, respectively, on the corresponding positions of the drawing table (as shown in Figure 4b); (4) close the door of the SEM sample test warehouse, and extract the air until the vacuum is formed in the warehouse; (5) adjust the SEM lens to make the observation position clear to record the test process, start the tensile table to stretch until the specimen breaks.

3. Results and Discussion

3.1. Study on the Evolution of Mechanical Properties of Fiber Reinforced Mortar after High Temperature

Figure 5 shows that as the temperature increases from 20 °C to 500 °C, the area under the load–displacement curves for both PVA and PP specimens decreases. In contrast, in the JZ group, the load–displacement curve ceases to develop after reaching the peak load. This indicates that after high temperature treatment, the matrix material mixed with fiber has better tensile properties than the matrix without fiber [22]. Furthermore, Figure 5d shows that, compared to the JZ specimens, the addition of fibers improves the tensile strength of the specimens at all temperature levels. The PVA specimens consistently exhibit higher tensile strength than the PP specimens across all temperatures, suggesting that fibers with a higher elastic modulus consume more energy during the stretching process.

At 20 °C, the area under the load–displacement curves of the fiber-containing specimens is significantly larger than that of the JZ group, indicating that the introduction of fibers enhances the toughness and crack resistance of the specimens. During the material fracture process under tensile stress, fibers absorb energy, thus enhancing the tensile strength of the specimens. At 200 °C, as cracks develop during the stretching process and some fibers begin to melt near their melting point, reducing the number of effective fibers, the area under the load–displacement curve decreases. As the temperature continues to rise, the fibers gradually dissolve, and the failure mode of the fiber specimens becomes similar to that of the JZ group, showing characteristics of brittle failure.

Although the area under the load–displacement curves gradually decreases with increasing temperature, the tensile strength of PVA and PP specimens increases between 20 °C and 200 °C. This may be due to the release of bound water and other substances within the material, promoting the “secondary hydration” of cement particles and consequently

increasing the material's strength. From 200 °C to 400 °C, as fibers gradually dissolve and create numerous capillary channels, these channels provide space for the release of internal steam pressure, reducing the steam pressure caused by water vapor migration. Meanwhile, the water vapor generated during this stage continues to promote the hydration of internal cement particles, further increasing the strength. Therefore, there is a rebound phase in the strength of JZ, PVA, and PP specimens. However, the toughening effect of the fibers gradually diminishes during this phase, resulting in a decrease in the area under the load–displacement curves. At 500 °C, there is a declining trend in the tensile strength of the specimens, as substances like calcium silicate hydrate (C-S-H) gel and $\text{Ca}(\text{OH})_2$ within the specimens begin to decompose and dehydrate when the temperature exceeds certain limits, making the originally dense structure increasingly porous and leading to a reduction in strength. This finding aligns with the existing literature, which indicates that treating fiber-reinforced cement at the appropriate temperature can not only enhance its strength but also improve the interface properties between the fiber and the matrix, thereby enhancing the tensile properties of fiber-reinforced cement-based composites [23].

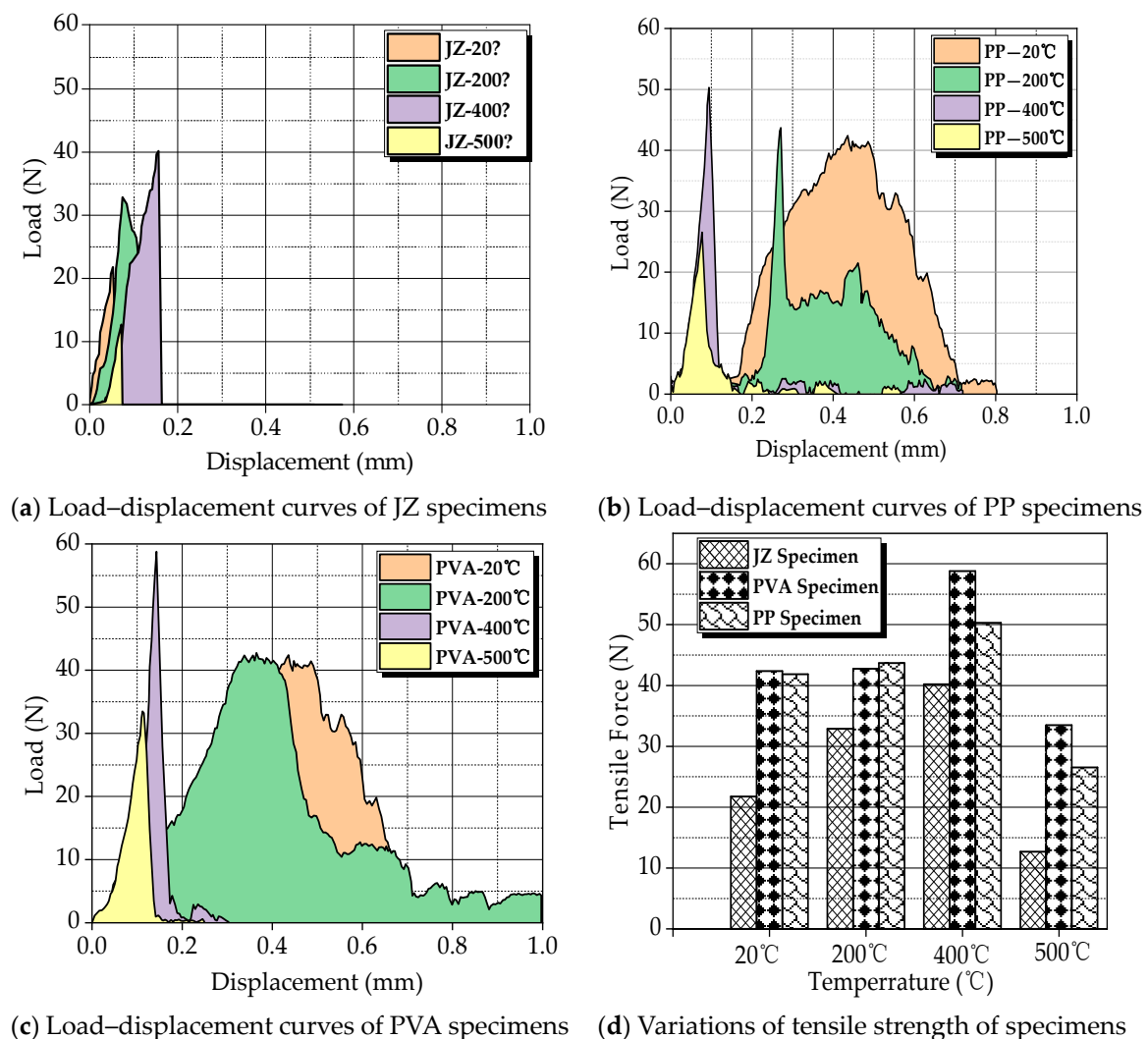


Figure 5. Tensile test results.

3.2. Study on Evolution of Microstructure and Mechanical Properties of Fiber Reinforced Mortar after High Temperature

Simple mechanical data analysis cannot provide a comprehensive understanding of the real-time microstructural evolution during the tensile process of fiber-reinforced mortar specimens subjected to different temperature treatments. Therefore, this study utilizes

environmental scanning electron microscopy to investigate the evolution of microstructure and mechanical properties of fiber-reinforced mortar after high-temperature exposure.

At 20 °C, the real-time microstructural evolution and mechanical curves for PVA and PP fiber-reinforced mortar specimens are shown in Figure 6 and Figure 7, respectively.

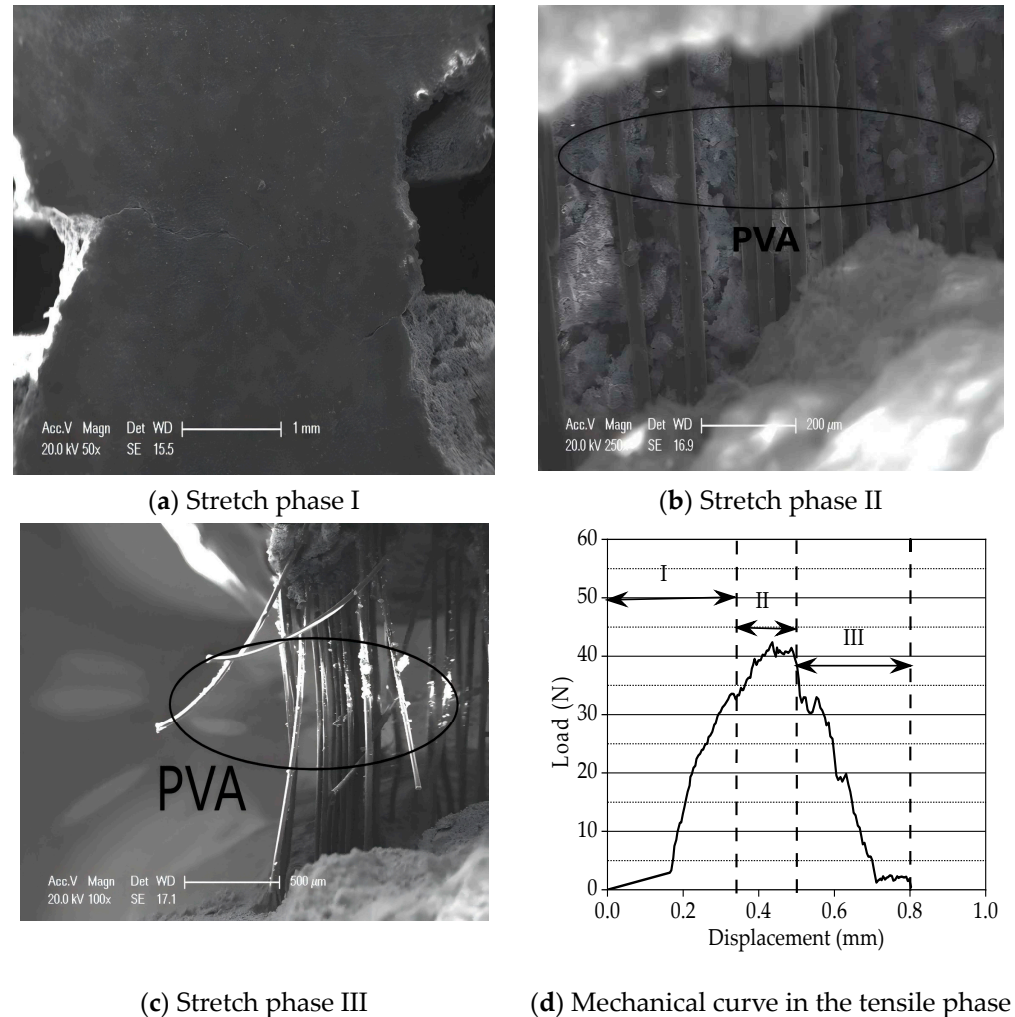
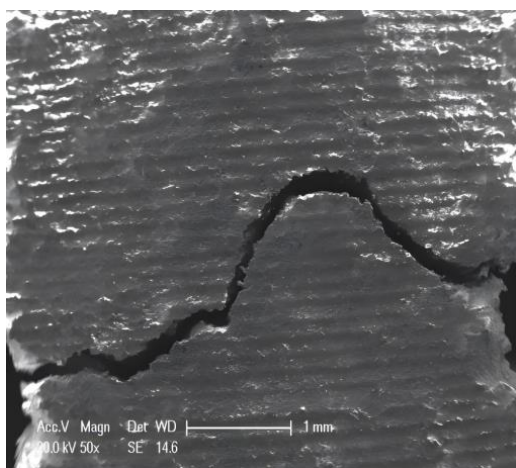


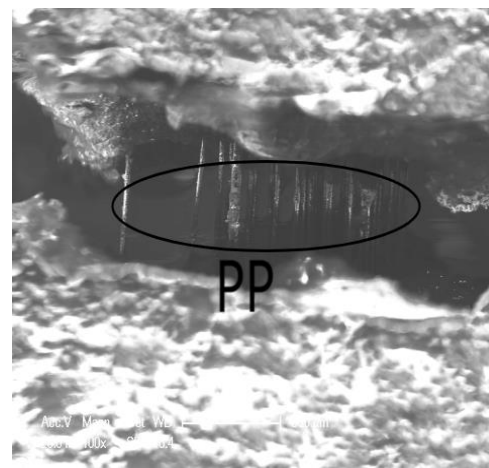
Figure 6. SEM diagram of uniaxial tensile process of PVA fiber specimens at 20 °C.

At 20 °C, the real-time microstructural evolution and mechanical curves for PVA and PP fiber-reinforced mortar specimens are shown in Figures 6 and 7, respectively. The real-time microstructural photographs of the tensile tests at this temperature indicate no thermal damage within the specimens. From phase I to II, the matrix mortar fractures, corresponding to the first peak in the mechanical curve. During phase II, both types of fibers begin to act as “bridges”, and the number of fibers involved in tension gradually increases, enhancing the tensile strength and raising the peak of the load–displacement curve. As the loading process continues from phase II to III, the fibers are progressively stretched, broken, or pulled out, consuming energy and increasing the area under the load–displacement curve, thereby enhancing the ductility of the fiber-reinforced mortar.

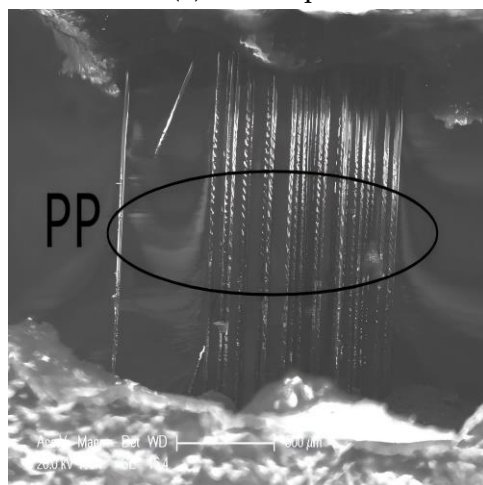
At 200 °C, the real-time microstructural evolution and mechanical curves for the PVA and the PP fiber-reinforced mortar specimens are depicted in Figures 8 and 9.



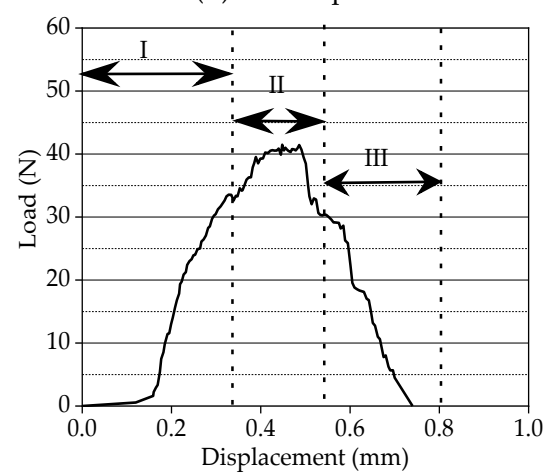
(a) Stretch phase I



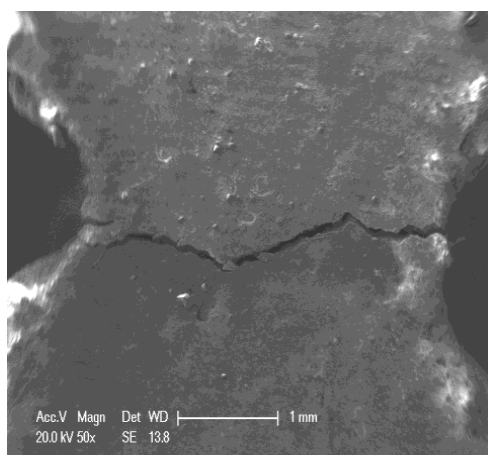
(b) Stretch phase II



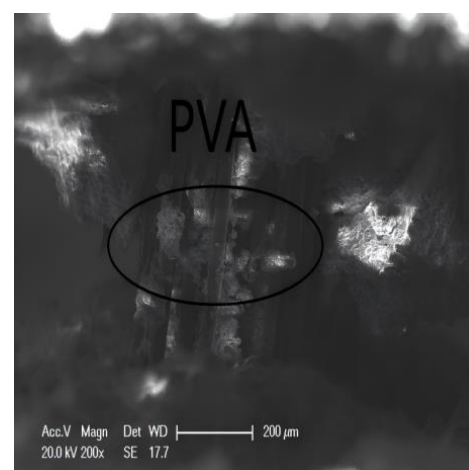
(c) Stretch phase III



(d) Mechanical curve in the tensile phase

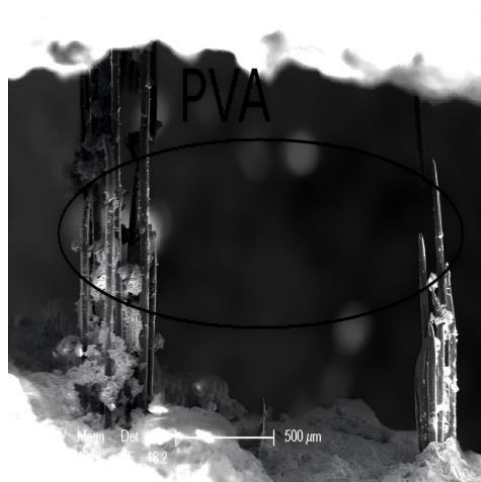
Figure 7. SEM diagram of uniaxial tensile process of PP fiber specimens at 20 °C.

(a) Stretch phase I

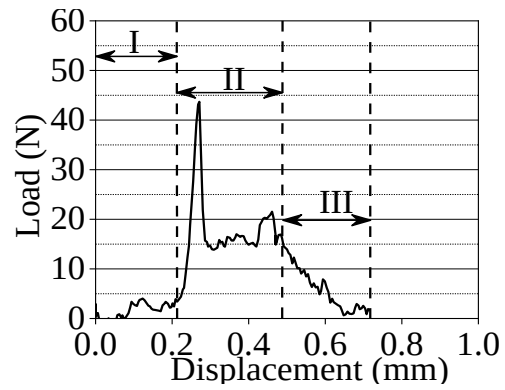


(b) Stretch phase II

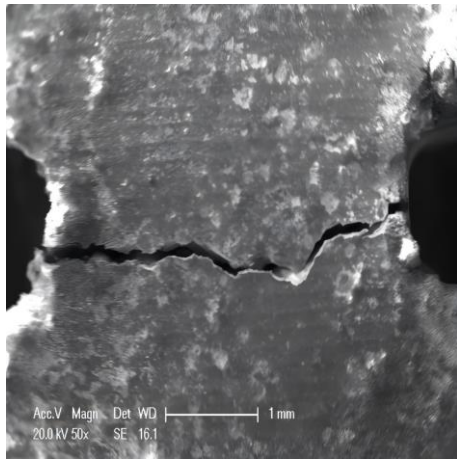
Figure 8. Cont.



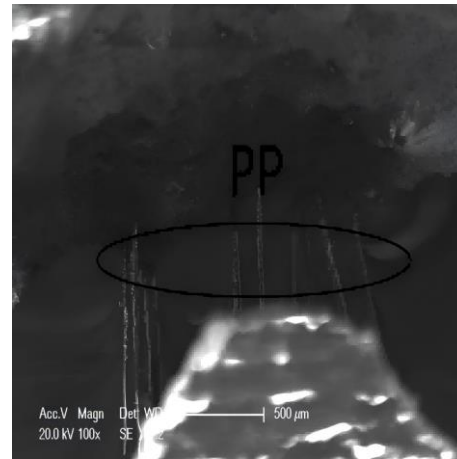
(c) Stretch phase III



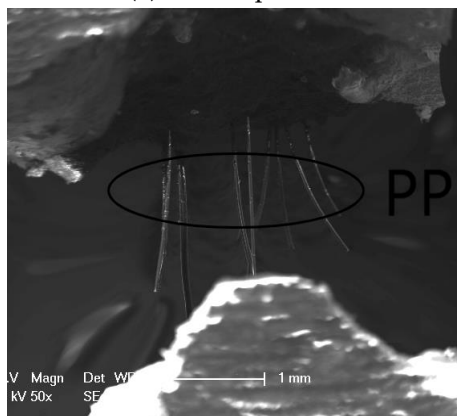
(d) Mechanical curve in the tensile phase

Figure 8. SEM diagram of uniaxial tensile process of PVA fiber specimens at 200 °C.

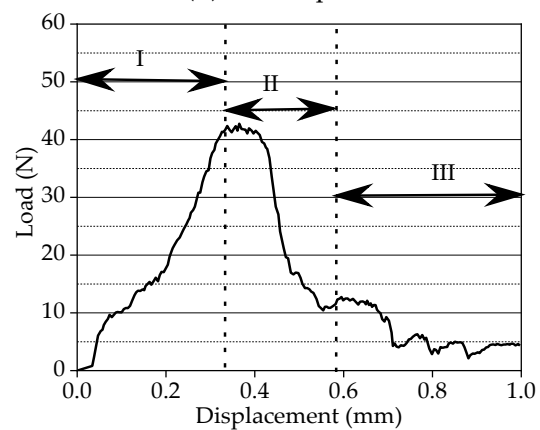
(a) Stretch phase I



(b) Stretch phase II



(c) Stretch phase III



(d) Mechanical curve in the tensile phase

Figure 9. SEM diagram of uniaxial tensile process of PP fiber specimens at 200 °C.

At this temperature, the real-time microstructural photos from phases II and III show a reduction in the number of fibers compared to those at 20 °C. This is because the melting point of both the PVA and the PP fibers is around 200 °C, leading to partial melting and a decrease in effective fiber count. Around 180 °C, the cement gel begins to dehydrate,

releasing and evaporating moisture [24]. The generated steam acts similarly to steam curing, continuing to provide conditions for the hydration of cement particles inside the fiber specimens, promoting further hydration of unhydrated cement particles, and thus enhancing the concrete strength [25]. Therefore, the tensile strength of the JZ specimens, as well as the PVA and the PP fiber-reinforced mortar specimens, is higher compared to 20 °C; although, the PVA and the PP fibers still delay the fracture of the specimens. Due to the decrease in fiber quantity caused by partial melting, the peak increase in the load displacement curve is relatively small, and the area under the curve is smaller compared to at 20 °C.

At 400 °C, the results of the experiment on the microstructure and mechanical properties of fiber-reinforced concrete after exposure to high temperatures are presented in Figures 10 and 11.

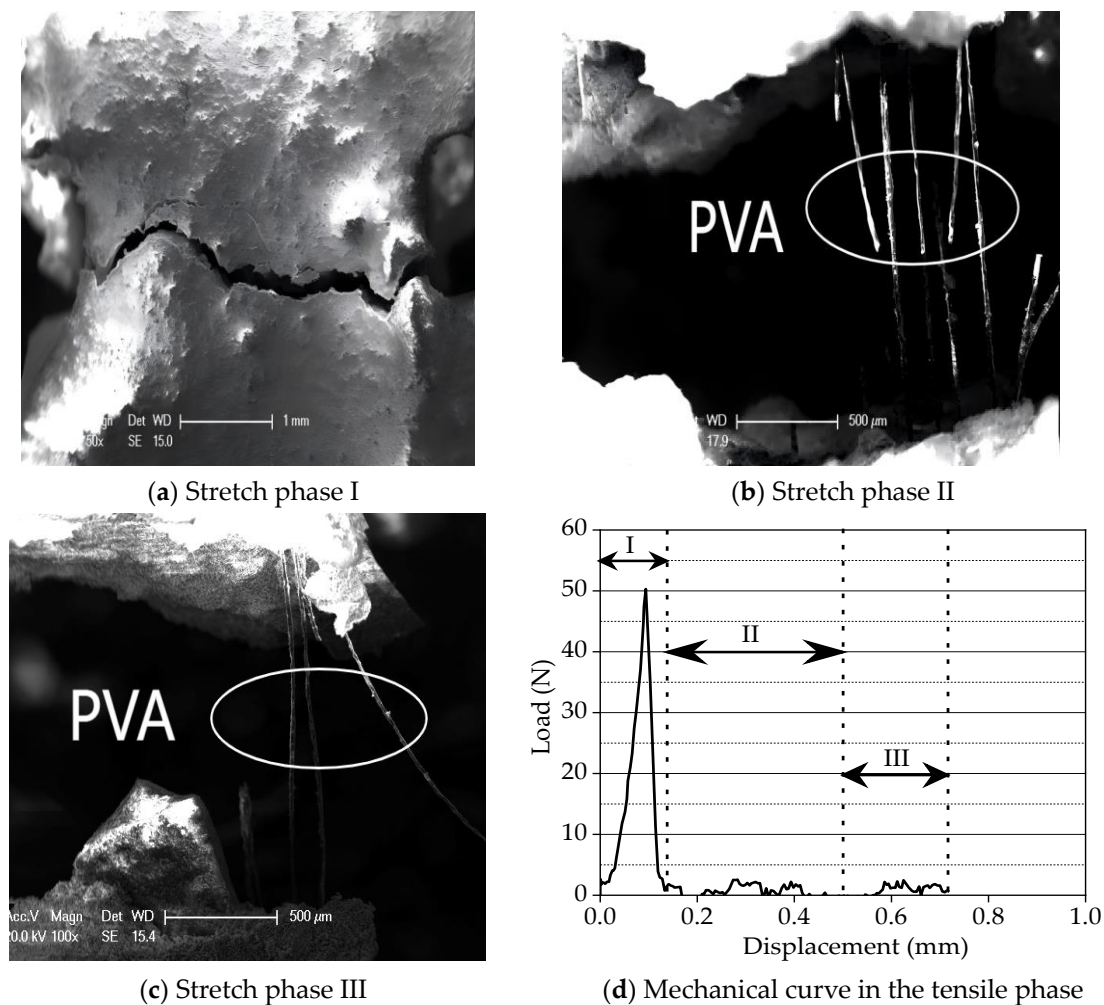


Figure 10. SEM diagram of uniaxial tensile process of PVA fiber specimens at 400 °C.

The SEM images from the uniaxial tensile process of the fiber specimens at 400 °C reveal uneven surfaces due to high-temperature calcination. A small number of fibers remain inside the specimens, as the cement matrix envelops some fiber surfaces, preventing complete dissolution. These fibers have smaller diameters compared to those at 20 °C and 200 °C. The mechanical curve from the tensile stage shows that the remaining fibers have lost their reinforcing and toughening effects, causing the load–displacement curve to decline sharply after peaking. Additionally, the steam generated by the internal cementitious materials continues to provide conditions for the hydration of incompletely hydrated materials inside the specimens, further enhancing their strength.

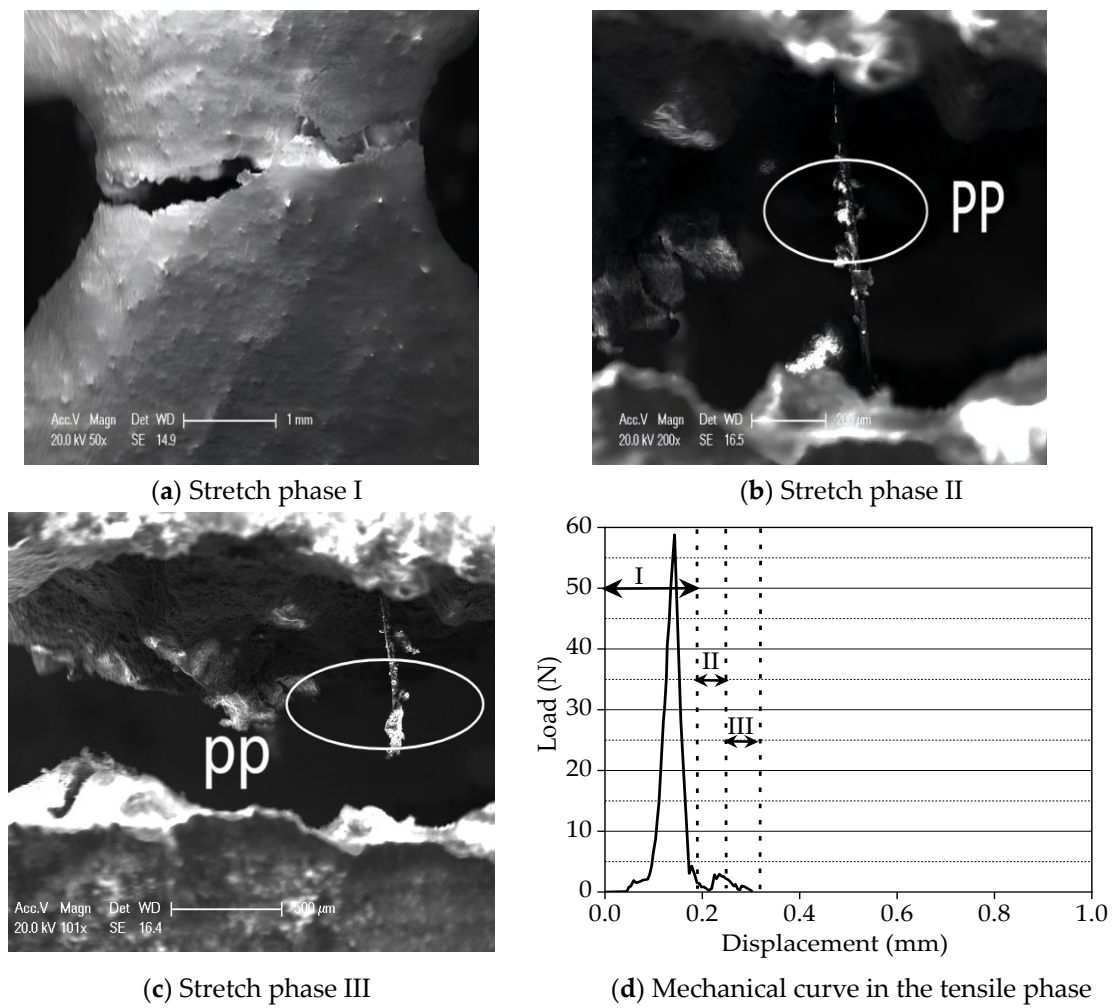


Figure 11. SEM diagram of uniaxial tensile process of PP fiber specimens at 400 °C.

At 500 °C, the real-time microstructural evolution and mechanical curves for the PVA and the PP fiber-reinforced mortar specimens are shown in Figures 12 and 13.

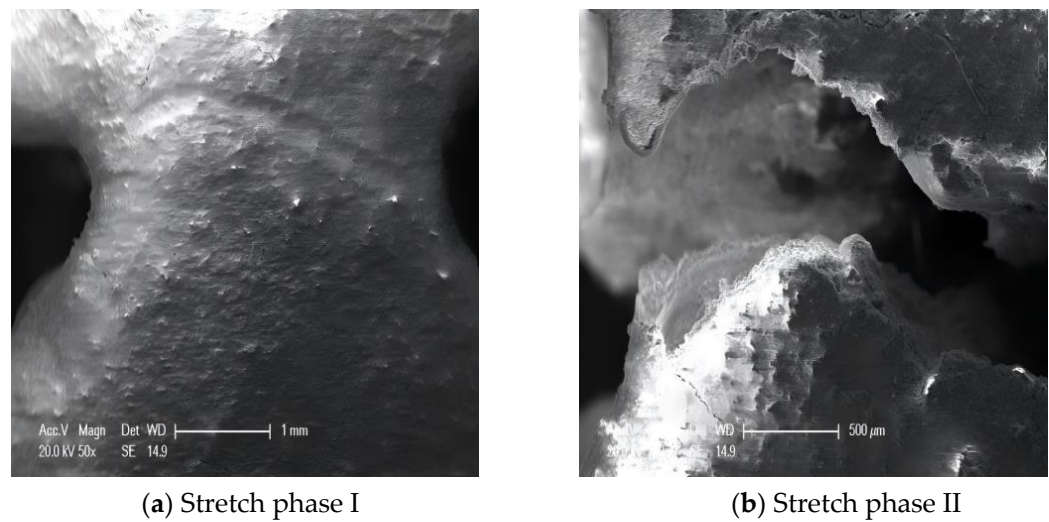
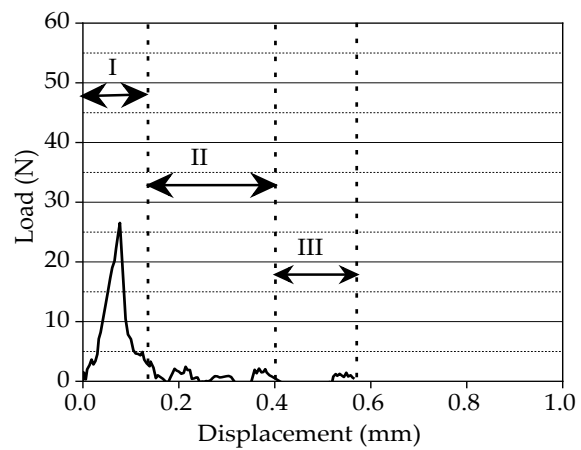


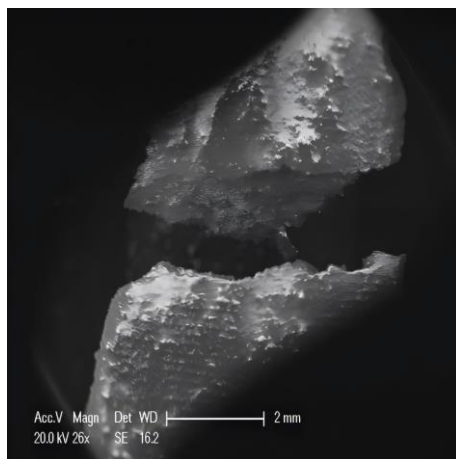
Figure 12. Cont.



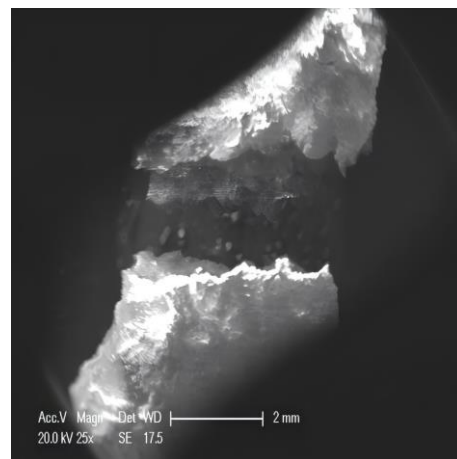
(c) Stretch phase III



(d) Mechanical curve in the tensile phase

Figure 12. SEM diagram of uniaxial tensile process of PVA fiber specimens at 500 °C.

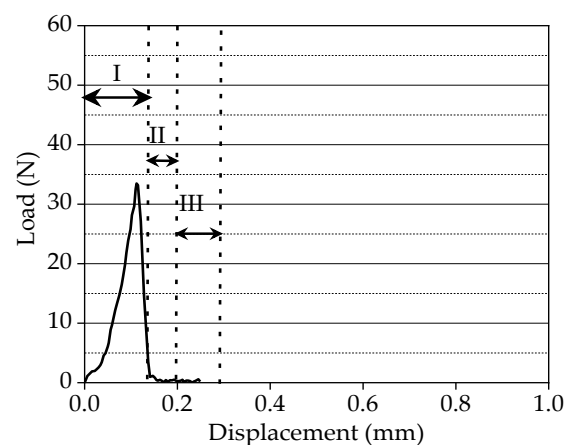
(a) Stretch phase I



(b) Stretch phase II



(c) Stretch phase III



(d) Mechanical curve in the tensile phase

Figure 13. SEM diagram of uniaxial tensile process of PP fiber specimens at 500 °C.

As temperatures rise, moisture gradually evaporates, and the fibers inside the fiber-reinforced mortar specimens progressively dissolve, increasing the number of capillary pores and microcracks [26]. At 500 °C, the real-time microstructural photographs of both fiber-reinforced mortar specimens show complete melting of fibers and the presence of

voids (as shown in Figures 2, 3, 4, 5 and 6c). The surfaces of phases I, II, and III exhibit clear signs of thermal damage. The tensile test process is shorter compared to other temperature ranges, but the tensile strength of the fiber-reinforced specimens is higher than that of the JZ group. This is because the JZ group has a higher density than the fiber group, and the fibers melt and form capillary channels in the fiber group, providing space for the release of steam pressure and reducing damage caused by steam stress inside the concrete [27]. The fibers in both the PVA and the PP fiber-reinforced mortar specimens melt due to high temperatures, causing the interior of the concrete specimens to lose the “bridging” action of PVA and PP fibers. Additionally, when temperatures reach 300 °C, the interlayer water of calcium silicate hydrate (C-S-H) and some of the chemically bound water of hydrated calcium aluminate in the concrete begin to be lost. Further dehydration occurs as $\text{Ca}(\text{OH})_2$ decomposes, causing the previously dense structure of the concrete specimens to gradually become loose, reducing strength. The mechanical curve from the tensile stage shows that the tensile strength of the PVA fiber-reinforced mortar specimen decreased by 36.47%, while that of the PP fiber-reinforced mortar specimen decreased by 24.14%. The trend of the load–displacement curve resembles that of the JZ group, displaying the characteristics of brittle material failure.

4. Conclusions

This paper investigates the mechanical properties of fiber-reinforced mortar after exposure to high temperatures, based on the theory of fiber reinforcement and the mechanisms of high-temperature degradation and damage in mortar. From the above research, the following conclusions can be drawn:

- (1) The tensile strength of the specimens increases and then decreases as the ambient temperature changes from 20 °C to 500 °C, with the most pronounced increase at 400 °C.
- (2) The tensile strength of the fiber-doped mortar specimens at all ambient temperatures is higher than that of the JZ group. At 400 °C, the tensile strength of the PVA group increases by about 16% compared to the JZ group and by about 45% compared to the PP group.
- (3) After high temperature treatment, the mechanical properties of PVA group were worse than those of the PP group. After treatment at 500 °C, the tensile strength of the mortar specimens in the PVA group and the PP group decreased by 36.47% and 24.14%, respectively, compared with that at 20 °C.
- (4) As the temperature rises, the fibers gradually melt, leaving pores inside the cement mortar. The bonding interface between the fibers and the cement matrix loosens, and cracks inside the cement mortar expand further, leading to significant internal damage and a decline in mechanical properties.

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