Scientific paper

PCM-Concrete Interfacial Tensile Behavior Using Nano-SiO₂ Based on Splitting-Tensile Test
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Abstract
The purpose of this study is to improve the interfacial performance between the concrete and polymer cement mortar (PCM) by using nano-SiO₂. This study examined the bond properties of the inclusion of nano-SiO₂ in the PCM based on splitting-tensile tests. In addition, the bonding mechanism was investigated with SEM. The results demonstrate that the inclusion of 2% nano-SiO₂ in the PCM is beneficial to compressive strength and microstructure so as to obtain good interfacial bond strength as a repair layer mortar. The results also show that the increase in the surface roughness, the improvement of substrate concrete strength, vertical casting, and wet saturated interface state facilitate bond strength and change the failure mode of nano-SiO₂ PCM/concrete composite specimens. At that time, the interfacial strength is predominantly influenced by the interface roughness and the old concrete strength through one-way analysis of variance. The results of SEM present that the interface of nano-SiO₂ PCM/concrete composite specimen is more compact than that of the PCM/concrete composite specimen due to the transformation of harmful Ca(OH)₂ into more C-S-H gels and the formation of a better polymer film structure at the interface. As a result, mixing nano-SiO₂ into PCM accompanied by adopting effective treatment method of surface and higher compressive strength of substrate concrete is significantly beneficial to bond strength.

1. Introduction
As a typical organic-inorganic composite, polymer cement mortar (PCM) has superior properties over ordinary cement mortar, including better workability, increased flexural strength, decreased water absorption, developed impermeability (Zhong and Chen 2002; Zhong et al. 2002; Yang et al. 2009), high resistance to frost (Mirza et al. 2002), low rate of shrinkage (Wang and Wang 2010) and long-lasting durability (Al-Zahrani 2003; Ohama 1995; Sakai 1995). In light of previous experimental research (Park et al. 2009; Chung 2004; Sakakibara et al. 2004), it has been considered that PCM is a widely used cementitious repairing material due to its considerable adhesive property with concrete. Hassan et al. (2001) reported that PCM was regarded as a very compatible overlay repairing materials with concrete in comparison to other repairing materials, on account of the formation of polymer films. The phenomenon of polymer modification process is based on the fact that the polymer particles deposit or condense on the surface of the hydrated cement and aggregate and gradually form a film, which will eventually form an interpenetrating network structure with the cement hydration product (Zhao et al. 2018). The coalescence of polymer particles modifies the process of cement hydration, resulting in the decrease in porosity and permeability, the enhancement in flexibility and adhesive strength, the optimization in the microstructure of the mortar and concrete (Khan et al. 2018).

However, research had shown that the incorporation of polymer tended to exert a negative influence on both flexural and compressive strength and exhibited a limited contribution of the tensile strength of cementitious materials (Barluenga and Hernández-Olivares 2004; Rashid et al. 2019). Moreover, although PCM is perceived to have the good adhesive property as for a repair material, study had demonstrated that there still existed a weak link at the interface of substrate concrete/PCM in the composite structure (Zhang et al. 2011, 2012; Satoh and Kodama 2005). Owing to the weak bond at the concrete/PCM interface, what measures can be adopted to improve this interface effectively to avoid premature debonding failure is now an issue gaining considerable attention.

Therefore, studies increasingly demonstrated that the addition of polymer and silica fume, simultaneously improved the bond strength significantly (Gao et al. 2002; Alessandra and Sichiari 2006, 2007). The composite mortar, which combines polymer with silica fume,
has been proven to have synergistic effects, facilitating the acquisition of higher bond strength and superior durability (Jiang et al. 2017). Besides, the incorporation of silica fume into PCM can improve the chemical bonding at the interface of concrete/PCM composite and this enhancement is more obvious on condition that substrate concrete has a higher roughness (Mizan et al. 2020).

Compared with silica fume, nano-SiO2 has the advantages of larger specific surface area and higher activity. At present, it has been widely applied in concrete and gradually put into use in polymer concrete. The current research revealed that it was incorporating nano-SiO2 in high performance concrete that greatly improved the compressive strengths and bond strengths of paste–aggregate interface compared to incorporating silica fume, especially at early ages (Qing et al. 2007). This is mainly because that nano-SiO2 which has a higher pozzolanic activity can not only provide much more nucleation sites for hydration products than silica fume at early ages, but also it can react with Ca(OH)2 crystals rapidly, decrease the orientation of Ca(OH)2 crystals and reduce the large-sized Ca(OH)2 crystal gathered at the interface, so that the interface performance is improved more effectively than silica fume (Madadi et al. 2007; Madhawanthi et al. 2017). However, although nanomaterials have been introduced into PCM and this new composite has been proved to possess good mechanical properties, present studies about various properties on PCM modified by nano-SiO2 are still very limited.

Since there is the absence of information about the bonding performance of PCM modified by nano-SiO2 as an overlay mortar, it is important for repair process to study the bonding property between the old concrete and this new overlay PCM. Therefore, the aim of this research work is designed to study the bonding strength between PCM modified by nano-SiO2 and old concrete by splitting tensile test, exploring the effect of the roughness of interface, compressive strength of old concrete, moisture state of interface, and the way of pouring concrete on bond strength of composite specimen. Tensile strength is extremely important to the interfacial bond strength of composite specimens, and it is related to the interfacial shear strength to some extent (Ma et al. 2009). Moreover, this study also aims to investigate the microscopic bonding mechanism of substrate concrete and the new repairing material to explore how nano-SiO2 reacts on the interface.

2 Materials and methods

2.1 Materials and mix proportion of substrate concrete

Substrate concrete was fabricated by mixing cement, natural sand, gravel, fly ash, water- reducer, and tap water. The cement used in this study was P.O 42.5 Portland cement and technical characteristics are shown in Table 1. The indicators of cement is in agreement with GB 175 (SPC 2007). Table 2 presents the technical characteristics of fly ash. The indicators of fly ash meet the requirement of GB/T 18736 (SPC 2017). The coarse aggregate was crushed limestone with a nominal maximum size of 31.5 mm and apparent density of 2650 kg/cm3, while natural sand with 2.68 fineness modulus and apparent density of 2600 kg/cm3 was river sand. The aggregate used in the test meets the requirement of JGJ 52 (CA & BP 2007). The water reducer was a liquid polycarboxylic acid with the density of 1.05 g/cm3. In this research, three types of normal concrete that were applied to old substrate concrete were studied. Compressive strength of substrate concrete was 20 MPa, 30 MPa, and 40 MPa at 28 days and concrete with three strength grade was termed as NC20, NC30 and NC40, respectively. The mix proportion and compressive strength of substrate concrete are shown in the Table 3. Concrete design mix was carried out according to JGJ 55 (CA & BP 2011).

2.2 Materials used for polymer cement mortar (PCM)

PCM was fabricated using cement, ISO standard sand,
fly ash, water-reducer, defoamer, polymer and tap water. The used cement was P.O 52.5 Portland cement. Commercially available PCM was ethylene vinyl acetate (EVA) re-dispersible latex powder. The technical characteristics of EVA are given in Table 4. The water reducer was polycarboxylic acid in powder form. The defoamer was organosilicon defoamer.

2.3 Materials used for composite mortar

For the purpose of increasing the interfacial bonding strength of PCM-concrete, nano-SiO$_2$ was used to modify PCM in this experimental work. The study used a commercial nano-silica, in powder form, with an average primary particle size of 20 nm, corresponding to a surface area of 250 m$^2$/g. In this work, PCM modified by nano-SiO$_2$ with 1%, 2% and 3% dosage was mainly investigated as a repair overlay. The microstructure observed by transmission electron microscopy (TEM) of the nano-SiO$_2$ is shown in Fig. 1. In addition to nano-SiO$_2$, the other raw materials used to produce composite mortar are identical to those used to produce PCM.

2.4 Mix proportions of PCM and composite mortars

The mix proportions and compressive strengths of two types of mortar are listed in Table 5, which shows that the compressive strength of two types of mortar meets the requirements of JG/T 289 (SPC 2010).

2.5 Surface preparation

Above all, roughness is considered an important factor on performance of bonding between old concrete and new repair mortar layer. In this research, three different methods for the treatment of the surface of the substrate concrete to form three different levels of roughness. Therefore, three different roughness types were discussed. These types can be summarized as the roughness formed by a cutting surface (C), artificial chiseling surface (AC), and splitting surface (S) as shown in Fig. 2. The splitting surface is acquired by splitting the existing concrete in the middle by splitting tensile test, and then removing coarse aggregate and hardened cement paste that remained loose and cracked on the fracture surface. The artificial chiseling method is considered convenient to operate, simple to use, and lower to cost. This method used a steel drill and hammer to remove the free mortar and cement stone on the concrete surface until the coarse aggregate was exposed, to form a randomly uneven surface.

The last method was to use a rock cutter to cut a 100×100×50 mm$^3$ for substrate concrete specimen. The cut surface served as the bonding surface that is smoother and level than the two types of bonding surface that

| Type of concrete | Cement (kg/m$^3$) | Sand (kg/m$^3$) | Stone (kg/m$^3$) | Fly ash (kg/m$^3$) | Water reducer (kg/m$^3$) | Water (kg/m$^3$) | Apparent density (kg/m$^3$) | Compressive strength (MPa) |
|-----------------|-----------------|----------------|-----------------|-----------------|-------------------------|----------------|-----------------------------|-----------------------------|
| NC20            | 291             | 802            | 1108            | 73              | 5.5                     | 175            | 2420                        | 27.5                        |
| NC30            | 308             | 833            | 1060            | 78              | 6.9                     | 170            | 2430                        | 34.1                        |
| NC40            | 347             | 814            | 1037            | 87              | 7.8                     | 165            | 2440                        | 42.7                        |

Table 3 Mix proportions of substrate concrete.

| Performance | Solids content (%) | Apparent density (g/L) | Appearance | Steady state material | Film-forming Temperature (°C) | Particle size (μm) |
|-------------|--------------------|------------------------|------------|-----------------------|------------------------------|-------------------|
| Index       | 99±1               | 540±50                 | White powder | Poval             | 4                            | 0.5 to 8          |

Table 4 Technical characteristics of re-dispersible EVA latex powder.

| Samples | Cement (g) | Water (g) | Sand (g) | Fly ash (g) | Nano-SiO$_2$ (wt%) | EVA (wt%) | Water reducer (wt%) | De-foamer (wt%) | Compressive strength (MPa) |
|---------|-------------|-----------|----------|-------------|--------------------|----------|--------------------|----------------|----------------------------|
| PCM     | 720         | 24        | 1200     | 80          | 0                  | 4        | 0.15               | 0.1            | 51.0                       | 59.1                       | 65.8                       |
| 1% Nano-SiO$_2$ PCM | 712         | 240       | 1200     | 80          | 0                  | 4        | 0.28               | 0.1            | 55.0                       | 65.6                       | 69.0                       |
| 2% Nano-SiO$_2$ PCM | 704         | 240       | 1200     | 80          | 0                  | 4        | 0.45               | 0.1            | 57.5                       | 67.3                       | 72.4                       |
| 3% Nano-SiO$_2$ PCM | 696         | 240       | 1200     | 80          | 0                  | 3        | 0.8                | 0.1            | 56.0                       | 65.1                       | 71.0                       |

Table 5 Mix proportions and compressive strength of PCM and composite mortar.
mentioned above. Thus, in this study, qualitative analysis based on the visual inspection way was adopted to define the roughness level instead of the quantitative assessment that most researchers generally used. In addition, the moist state of the substrate concrete surface may affect the interfacial bond strength of composite specimens. Before pouring PCM or nano-SiO₂ PCM, the bonding interface was processed to be in a wet saturated state (WE) or dry state (D) to allow the influence of the state of moistness of the interface on the bond strength of the composite specimens to be studied. The wet saturated state refers to the substrate concrete specimens being soaked in water for 24 hours before being taken out and put into the mold. When there is no visible water at the bonding interface, a new repair layer mortar is poured. The dry state means that specimens are placed in a dry and well ventilated environment for two weeks, as close to the natural dry environment as possible, and the humidity at the bonding surface is below 10% RH before pouring new mortar layer.

2.6 Preparation and splitting-tension test of composite specimens

At first, old concrete specimens with the size of 100×100×100 mm³ were created using a mold. The specimens underwent standard curing for 56 days. These old concrete specimens were made into the 100×100×50 mm³ specimens by cutting or by splitting tensile test. The cut or split old concrete specimens were put into the mold, and then PCM or composite mortar was poured into the rest space of the mold and vibrated to form 100×100×100 mm³ bonded specimens. Two types of pouring methods were adopted, vertical casting (VC) and parallel casting (PC), respectively. After 3 days, 7 days and 28 days age of standard curing in the mold, splitting-tension testing on the bonded specimens was conducted using a universal testing machine according to GB/T 50081 (CA & BP 2002) standard. The principle and loading schematic diagram is shown in Fig. 3. The splitting tensile strength of bonded specimens is obtained from Eq. (1), which is also based on above-mentioned standard:

$$f_u = \frac{2F}{\pi A} \times 0.85 \times 0.541 = \frac{F}{A}$$

where $f_u$ denotes the splitting tensile strength of bonded specimens, F refers to the failure load, A means to the area of bonded specimens, and the $f_u$ is correct to 0.01 MPa. Each set of composite specimens was tested for three times, and the average value of three independent experiments was taken as the final result of splitting tensile strength.

2.7 Parameters studied

The studied parameters in this study were the effect of compressive strength of substrate concrete, the roughness of interface, the moisture state of interface and the way of pouring concrete on the interface of substrate concrete and nano-SiO₂ PCM. In addition, in order to compare with nano-SiO₂ PCM overlay repair materials, composite specimens of NC30 substrate concrete and PCM were also studied. The details of the test specimens are shown in Table 6.

2.8 Microstructure test

After curing for 28 days, the test sample was cut out from the interface of nano-SiO₂ PCM/concrete and PCM/concrete composite specimens with an area of about 1 cm². The prepared testing samples cut from composite specimens should be kept dry. The specimens were sprayed with gold before observing to enhance electrical conductivity. Scanning electron microscopy (SEM) was used to observe the microscopic morphology and micro-cracks of the interface under different magnification. Figure 4 shows test samples without
spraying gold for SEM. Besides, SEM was used to observe the microscopic morphology of PCM and three kinds of nano-SiO₂ PCM at 28 days.

3. Results and discussion

3.1 Micromorphology of PCM and nano-SiO₂ PCM

Morphology of PCM and nano-SiO₂ PCM was characterized with SEM after 28 days in Fig. 5. It can be seen that PCM is loose and porous with some cracks as compared to nano-SiO₂ PCM. This also means that the addition of nano-SiO₂ promotes cement hydration and improves the microstructure of PCM. It can also be seen from the Fig. 5 that there are small holes in 1% nano-SiO₂ PCM and 3% nano-SiO₂ PCM, but there are few defects in 2% nano-SiO₂ PCM. Due to the difference in microstructure, the strength of PCM incorporated 2% nano-SiO₂ can be improved the biggest. Owing to good microstructure and compressive strength, PCM modified by 2% nano-SiO₂ was adopted as the repair layer mortar in this research.

3.2 Interfacial splitting-tension bond strength

Table 7 describes the splitting-tension bond strength of composite specimens at different ages. Table 7 and Figs. 6(a) to 6(e) illustrate the influence of different factors on bond splitting tensile strength of composite specimens. Figure 6(a) shows splitting-tension bond strength test results of nano-SiO₂ PCM/concrete composite specimens in different roughness levers after 3 days, 7 days and 28 days. Based on these test results, the surface roughness of substrate concrete significantly affects bond strength. It is shown that bond strength of composite specimens increases with the increase of surface roughness. In the case of NC30 composite specimens, the increase percentage were 46.5%, 45.9% and 20.4% from the roughness of cutting surface to the roughness of artificial chiseling surface at 3 days, 7 days and 28 days, respectively. At that time, when the roughness of artificial chiseling surface transforms to the roughness of the splitting surface, the increase percentage of bond strength were 19.2%, 11.7% and 9.5% at 3 days, 7 days and 28 days, respectively. This result may be due to the higher interaction as the interface roughness increases. The higher interaction reduces relative autogenous shrinkage between old substrate concrete and nano-SiO₂ PCM, which is beneficial to promote the bond strength (Momayez et al. 2005). Besides, it is the increasing interface roughness that makes a larger contact area between new overlay mortars and old concrete. This generates the greater mechanical interaction force and van der Waals force at the interface, forming a greater macro bond strength.

Figure 6(b) presents the results of splitting-tension
bond strength of nano-SiO$_2$ PCM/concrete composite specimens for the case of different compressive strengths of old concrete after 3 days, 7 days and 28 days. From these test results, it can be concluded that bond strength is significantly affected by the compressive strength of old concrete, increasing with the rising of old concrete compressive strength at different ages. This may be attributed to decreased autogenous shrinkage of higher strength composite that is formed by higher compressive strength of substrate concrete (Diab et al. 2017). The higher bond strength profits from decreased autogenous shrinkage of composite. Besides, together with the increase of the strength of base concrete, its elastic modulus increases (CA & BP 2010) and the difference of elastic modulus of composite specimens is improved accordingly (Hu 2014). Meanwhile, the increase of bond strength is relatively significant when concrete compressive strength of old concrete increases from 30 MPa to 40 MPa. As Fig. 5(b) shows, when substrate concrete compressive strength increases from 30 MPa to 40 MPa, the increase percentage of bond strength was 12.1%, 16.7% and 18.1% at 3 days, 7 days and 28 days, respectively, which is higher than that of increases from 20 MPa to 30 MPa.

Figure 6(c) shows the effect of concrete pouring way on splitting-tension bond strength after 3 days, 7 days and 28 days. It can be seen that the cohesive effect of the vertical casting method is better than that of the parallel casting method for composite specimens whatever substrate concrete compressive strength is high or low. The difference in the bond strength is the result of forming holes and segregation on the uneven part of the bonding surface and not acquiring continuously distributed adhesive strength along the vertical direction of the composite specimens if parallel casting concrete is adopted. These reasons make the continuity of the interface become worse, resulting in lower bonding strength. In vertical casting, why the higher interface bond strength is that the overlay mortar can sink more easily and bubbles near the bonding surface can rise and discharge, making the contact between concrete old and new mortar more compact. Apart from this, it is confirmed that the higher substrate concrete strength is, the lager is the difference of bond strength that is caused by two types of casting methods. As shown in Fig. 5(c), it would have acquired a higher rate of increase which is 8.8%, 13.9% and 9.6% at 3 days, 7 days and 28 days, respectively, under the higher substrate concrete, such as 40 MPa. Meanwhile, if the lower substrate concrete such as 25 MPa is adopted, the lower rate of increase is more accessible at 3 days, 7 days and 28 days, which is 5.5%, 7.7% and 7.7%, respectively.

Figure 6(d) shows the effect of interface moisture state on splitting-tension bond strength at 3 days, 7 days and 28 days. It is seen that the bond strength of the nano-SiO$_2$ PCM composite specimens at the wet saturated state is higher than that of the drying state in three substrate concrete compressive strength cases. The prime reason is that when the interface is in a drying state, the substrate concrete will absorb water from the overlay repair material, leading to incomplete hydration of repair mortar near the interface. However, if there is excessive moisture on the surface, the pores of the base concrete surface will be blocked and the contact between the repair overlay material and the old substrate concrete will be obstructed. Therefore, it is realized that the best state of bonding surface is the wet saturated state. Moreover, Fig. 5(d) also presents that if the strength of the base concrete is relatively low, such as 25 MPa, the bond strength of the composite specimens in the two interface moisture condition is not significantly different. The bond strength in a wet saturated state is 3.3%, 5.6%, 5.2% higher than that of a drying state at 3 days, 7 days and 28 days, respectively. However, when the strength of the base concrete reaches up to 40 MPa, the increase of bond strength is 5.8%, 6.9%, and 8.0% at 3, 7 and 28 days, respectively, in the two different interface moisture conditions.

Figure 6(e) shows the splitting tensile strength of NC30-S-D-PC (PCM) and NC30-S-D-PC composite
specimens at 3 days, 7 days and 28 days. It can be seen from Fig. 5(c) that the bond strength of NC30-S-D-PC composite specimens is higher than that of NC30-PCM composite specimens at various ages. This indicated that the nano-SiO$_2$ inclusions as overlay repairing material can more effectively enhance the bond strength of composite specimens. Given that the structure of the interface transition zone is considerably related to bonding strength, the reason for the increase of bonding strength may be that the nano-SiO$_2$ incorporation optimizes the structure of the interface transition zone between the new mortar and the old substrate concrete and is more beneficial to acquire denser structure of the interface transition zone which exists more chemical bonding (Mizan et al. 2020). In addition, the addition of nano-SiO$_2$ increases the strength of PCM, and the elastic modulus increases accordingly, which may be reduce the difference between the elastic modulus and that of C30 base concrete and lead to the increase of bond strength. This is because the smaller difference of elastic modulus, the smaller the stress concentration, which has a significant negative influence on bond strength at the interface (Pan 1999).

3.3 The failure mode of the composite specimens at 7 days

The obtained tensile strength ($f_{et}$), average tensile strength, and the failure modes of 12 composite specimens groups under different parameters at 7 days are shown in Table 8. The fracture modes are classified according to the position of the interfacial fracture of the specimens after the splitting tensile tests. The possi-

![Fig. 6 The influence of (a) Different surface roughness levels, (b) Different substrate concrete compressive, (c) Different casting methods, (d) Different interface moisture states and (e) Different repair overlays on the bond strength of interface.](image-url)
ble failure modes of the composite specimens are classified in Fig. 7. In this experiment, four types of fracture modes were found, including pure adhesive interface fracture (I), concrete cohesion fracture (C), interface-concrete mixed-mode fracture (I-C), and interface-PCM mixed-mode fracture (I-P), as shown in Fig. 8.

In this study, composite specimens with low roughness, such as roughness of artificial chiseling surface and the roughness of the cutting surface, showed pure adhesive interface (I) fracture. For example, this fracture mode (I) was observed in specimens NC30-C-D-PC and NC30-AC-D-PC. In this failure mode, as shown in Fig. 7(a), the failure surface is relatively straight along with the interface of the bonding composite specimen, even with tiny cracks in the concrete or PCM layer. The composite specimens with low compressive strength of substrate concrete tended to generate concrete cohesion fracture (C). For example, this fracture mode (C) was observed in specimens NC20-S-D-PC and NC20-S-WS-PC. In this fracture mode, the fracture generated in the substrate concrete cohesion layer completely with the vast majority of concrete attached to the PCM side, as shown in Fig. 7(b). Whereas, composite specimens which owned high substrate concrete compressive or low substrate concrete compressive with vertical casting gave rise to mixed-mode failure (I-C), such as specimens NC20-S-D-VC, NC30-S-D-PC, NC30-S-WS-PC, NC30-S-D-VC and NC40-S-D-PC. As shown in Fig. 7(c), the failure fracture was predominantly found at the interface, full of concrete attached to the PCM side. When the specimens possessed high substrate concrete compressive strength with the interface saturated state or vertical casting, the mixed-mode failure mode (I-P) appeared in these composite specimens. Specimens NC40-S-WS-PC and NC40-S-D-VC showed this typical failure mode. In addition, NC30-S-D-PC (PCM) bond composite specimen also belonged to this fracture mode. In this fracture mode, the failure fracture was mainly observed at the interface and some PCM adhered to the concrete substrate, as shown in Fig. 7(d).

Based on the discussion of the above fracture modes, it may be considered that as the surface roughness increases, the failure surface transfers closer to the concrete cohesion (C) layer on condition that the low compressive strength of substrate concrete is used and that to the mixed-mode (I-C or I-P) if the high one is used. The improvement of old concrete compressive strength also alters the failure mode. The composite specimens with the low substrate concrete strength are more likely to produce concrete cohesion fracture (C), while the high ones are more inclined to produce mixed-mode failure (I-C). Comparing with dry surface state and parallel casting, composite specimens with saturated surface state and the vertical casting for the case of high compressive strength of substrate concrete can acquire higher bond strength, leading to the transformation of failure mode from I-C fracture to I-P fracture. However, comparing with NC30-S-D-PC (PCM) composite specimen, the mixing of nano-SiO$_2$ in the PCM, as NC30-S-D-PC sample shown, tends to shift the failure mode from mixed-mode (I-P) to the mixed-mode (I-C).

### 3.4 One-way analysis of variance (ANOVA)

Since the above-mentioned factors have been known to influence the bond interface strength of nano-SiO$_2$ PCM/concrete, three equal repeated tests were conducted for each influencing factor with different levers on the bonding strength of interface, and univariate ANOVA that denotes a variance model of only one dependent variable was performed to analyze. We assume

![Fig. 7 Classification of failure modes of composite specimens.](image-url)
that ‘A’ is an influencing factor on the bonding strength of interface: ‘A’ has r different levels A₁, A₂, ... Aᵣ, the test results under Aᵢ is denoted as Xᵢ, which is supposed to obey the normal distribution $\text{N}(\mu_i, \sigma^2)$ and are represented as $X_i \sim \text{N}(\mu_i, \sigma^2)$, $i=1, 2, \ldots r$. All values of $X_i$ are mutually independent. Nᵢ independent tests were conducted under level Aᵢ to obtain samples $X_{i1}, X_{i2}, \ldots X_{in_i}$, among which $X_{ij}$ is supposed to obey the normal distribution $\text{N}(\mu_i, \sigma^2)$ and are represented as $X_i \sim \text{N}(\mu_i, \sigma^2)$, $i=1, 2, \ldots r; j=1, 2, \ldots n_i$. Similarly, all values of $X_{ij}$ are mutually independent. In this example, each level is considered as a group. The relevant calculation principles, parameters, and formulas are shown in Tables 9 and 10, and significant analyses are shown in Table 11. Based on these equations, the F test can be carried out for factor A supposing that $H_0 = u_1 = u_2 = \ldots = u_r$.

In this section, based on the selected test results and the criterion for the test of significance, a one-way ANOVA is performed for each factor of influence on the bond interface strength of nano-SiO₂ PCM/concrete at 7 days. Based on our calculations and analysis, as shown in Table 12, according to F value of the table, it is indicated that 4 types of influence factors and levels are highly significant effect to the bond strength. Therefore, four factors were ranked according to their degree of influence on the substrate concrete and new overlay material interfacial bond, concluding that the surface roughness is ranked first, followed by the old concrete strength, pouring way, and interface state. And thus it is concluded that the utilizing higher old concrete and surface roughness can remarkably improve interfacial bond performance. Apart from that, the saturated interface state and vertical casting can also moderately improve the interface bond strength.

### 3.5 Micromechanical investigation

JSM-6700F scanning electron microscope (SEM) was used to observe the microstructure of the interface of PCM/concrete or nano-SiO₂ PCM/concrete composite specimens. Figure 9 shows SEM images of the microstructure of the PCM bonding sample after 28 days. It can be seen from Fig. 9(a), the microstructure of the three-phase system of the bonding sample which comprises old concrete cement paste, ITZ and PCM at lower magnification is very clear.

Comparing with the PCM matrix below, it is indicated that loose microstructure and obvious holes are found in ITZ. AS seen from the Fig. 9(b), a large amount of Ca(OH)₂ crystals with large size is mainly enriched and a few loose C-S-H gels are found in ITZ. Similarly, as shown in Fig. 9(c), it is mainly found that a large variety of AFt crystals is in the pores with a few surrounding C-S-H gels and Ca(OH)₂ crystals. It can also be seen clearly from Fig. 9(c) that the connection between hydration products is not dense. As seen from Fig. 9(d) at higher magnification, in addition to the large-sized and block Ca(OH)₂ crystals, and a few
C-S-H gels, a small quantity of large rod-shaped AFt crystals and fragmentary polymer particles which have been formed a film is observed. Some hydration products intersperse between the polymer films, which contributes to the increase of bond strength.

Meanwhile, the SEM image of the microstructure of nano-SiO$_2$ PCM bonding sample at 7 days is shown in Fig. 10. At lower magnification, the ITZ of the three-phase system is also clearly visible. It can be seen from the comparison of Figs. 9(a) and 10(a), the ITZ of composite mortar has no obvious defect, and the compactness of microstructure is better than that of PCM after curing for 28 days. As shown in Fig. 10(b), Ca(OH)$_2$ crystals which have been transformed into thin and laminar crystals and more C-S-H gels are found in ITZ. As seen from Fig. 10(c), at higher magnification, the hydration products appear to be dense, with thin Ca(OH)$_2$ crystals and a large number of C-S-H gels interwoven with polymer films. Similarly, Fig. 10(d) at higher magnification shows that the fine AFt crystals and a great many C-S-H gels are generated and the film formation of polymer particles is more obvious in ITZ. Comparing with PCM bond sample, composite mortar bond sample can form more continuous polymer films in ITZ. They are effectively connected to hydration products in pores and distributed among hydration products, which can further fill the gap between the holes and cement hydration products in ITZ, making ITZ denser. This indicates that the nano-SiO$_2$ with high pozzolanic activity can react with Ca(OH)$_2$ rapidly, generating a large amount
of C-S-H gels at the interface and promoting polymer film formation. The ITZ bond strength of nano-SiO₂ PCM sample is improved mainly due to the transformation of harmful Ca(OH)₂ into a large amount of C-S-H gels and the formation of a better polymer film structure.

4 Conclusions

The following conclusions can be made from this experimental investigation:

1. 2% nano-SiO₂ PCM is adopted as the repair layer mortar due to good microstructure and mechanical properties compared to the 1% nano-SiO₂ PCM and 3% nano-SiO₂ PCM.

2. Composite specimens of NC30 substrate concrete/PCM and composite specimens of NC30 substrate concrete/2% nano-SiO₂ PCM were simultaneously studied with regard to bond strength and failure mode. The results reveal that the inclusion of nano-SiO₂ in the PCM can increase the interfacial bonding strength of composite specimen. Besides, the mixing of nano-SiO₂ in the PCM tends to shift the failure mode of composite specimens from mixed-mode (I-P) to the mixed-mode (I-C) at 7 days.

3. The interfacial roughness, substrate concrete com-

Fig. 9 Scanning electron microscopy images of ITZ of PCM bonding sample at the different magnifications shown below. (a) 200, (b) 5000, (c) 10000 and (d) 20000.

Fig. 10 Scanning electron microscopy images of ITZ of nano-SiO₂ PCM bonding sample at the different magnifications shown below. (a) 200, (b) 5000, (c) 20000 and (d) 20000.
pressive strength, the method of concrete pouring and the interface moisture state affect the bond strength and failure mode of nano-SiO$_2$ PCM/concrete composite sample in splitting-tension tests. The roughness of natural fracture surface, high strength of substrate concrete, vertical casting and wet saturated interface state facilitate bond strength development. Compared with the roughness of artificial chiseling surface, composite specimens adopting the roughness of the splitting surface make bond strength increase by 19.2%, 11.7% and 9.5% at 3 days, 7 days and 28 days, respectively. When compressive strength of substrate concrete increases from 30 MPa to 40 MPa, the increase percentage of bond strength was 12.1%, 16.7% and 18.1% at three different ages, which is all higher than that of from 20 MPa to 30 MPa. Higher strength of substrate concrete, such as 40 MPa, using vertical casting or wet saturated treatment of interface enables bond strength to enhance by 8.8%, 13.9% and 9.6% at three different ages and 5.8%, 6.9%, and 8.0% at three different ages, respectively. Meanwhile, as the surface roughness increases, the failure surface transfers closer to the concrete cohesion layer (C) on condition that the low compressive strength of substrate concrete is used and that to the mixed-mode (I-C or I-P) if the high one is used. The improvement of old substrate concrete strength also alters the failure mode. When the interface moisture state is achieved to wet saturated state or vertical casting method is adopted for the case of high substrate concrete compressive, the failure mode is transformed from I-C fracture to I-P fracture.

(4) Through one-way analysis of variance, it is concluded that the interfacial strength is predominantly influenced by the interface roughness and the strength of old concrete, while interface moisture state and pouring way also exert a positive impact on the splitting tensile strength. Therefore, utilizing a higher compressive strength of substrate concrete and surface roughness can remarkably improve bond strength at the interface of nano-SiO$_2$ PCM/concrete.

(5) Through the microscopic test of SEM, the results presented that the interface of the nano-SiO$_2$ PCM/concrete composite specimen was more compact than PCM/concrete composite specimen and has no obvious defect. The bond strength of nano-SiO$_2$ PCM sample is improved mainly due to the transformation of harmful Ca(OH)$_2$ into a large amount of C-S-H and the formation of a better polymer film structure.

In conclusion, as a novel repairing material, good splitting tensile bond strength is achieved by mixing 2% nano-SiO$_2$ into PCM, especially in case that compressive strength of substrate concrete is higher and splitting surface of substrate concrete which has higher interface roughness is used. Besides, the modification mechanism of nano-SiO$_2$ in this novel composite is also revealed in this study, which can provide an indication for engineering application of nano-SiO$_2$ in polymer cement-based repair materials.

Acknowledgments
This research was supported by the National Natural Science Foundation of China (Grant Nos. 51168031, 51868044).

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