

Experimental Study on Improvement of Interfacial Bond Properties of Carbon Fiber Reinforced Cement Matrix Composites by Nano Silica

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Abstract. *The fiber reinforcement efficiency of carbon fiber reinforced cement matrix composites (CFRCM) is limited by the low permeability of mortar to internal filaments in the fibers, leading to premature failure of the composites due to low bond strength. In this paper, three kinds of nano-silica materials were used to improve the bond properties of carbon fiber bundles to cement-based matrix by coating and electrophoretic deposition. It is found that different methods have different positive or negative effects on the improvement of bond properties, and the effects are different under different embedded lengths. The modification principle is due to the high impregnation of nano silica particles on the fibers and the reaction of volcanic ash of the particles, which promotes the formation of calcium silicate hydrate gel inside the fibers. Relevant research needs further exploration.*

Keywords : *Cement-based composites; Carbon fiber; Nano silica-coating; Electrophoretic deposition; Interfacial bond properties.*

1 Introduction

Carbon Fabric Reinforced Cementitious Matrix (CFRCM) can improve the flexural(Wei et al. 2021), shear(Awani et al. 2016), and seismic(Koutas et al. 2015)bearing capacity of reinforced concrete structures. Although a large number of studies have shown the effectiveness of the CFRCM system reinforcement, the mechanical properties of CFRCM reinforcement are less effective compared to CFRP. This is due to the difficulty of the cement slurry to penetrate inside the carbon fiber bundles with hydrophobic properties, resulting in insufficient bonding between the fibers and the cementitious matrix(Schneider et al. 2019).

The bond properties between fibers and cement mortar matrix is one of the important factors that determine the mechanical behavior of CFRCM(Dvorkin and Peled. 2016). A lot of researches have been conducted to enhance the bond strength between carbon fibers and the matrix. The operation of oxidizing carbon fiber(Wang et al. 1998) is complex and requires dangerous acid-base chemicals; The functional groups generated by the plasma method(Zhao et al. 2020) on the surface of carbon fibers exhibits instability over time(Li et al. 2020). The more commonly used modification method is the inorganic coating method using nano-silica as the coating. Some studies have used sol-gel methods(Bompadre and Donnini.2022)or a suspension of nanosilica particles stirred with water to coat carbon fibers(Dvorkin and Peled 2016, Nadiv et al. 2017, Signorini et al. 2019, Zamir et al. 2019). In addition, nano-silica was deposited onto carbon fibers using electrophoretic deposition (EPD) (Li et al. 2020) to improve

the bond properties of fibers to cementitious matrix. While some of these studies is valid, others showed conflicting results. Therefore, such methods need to be further explored.

In this paper, to enhance the bond properties of carbon fibers in mortar matrix, three kinds of nano-silica coatings and electrophoretic deposition were used to modify carbon fibers, and then the effect of the modification was analyzed by pull-out test.

2 Experimental programme

2.1 Material properties

The properties of carbon fiber bundles, mortar ratio and compressive strength of cubic standard specimens are shown in Table 1 and Table 2, respectively.

The nano-silica provided by Beijing Huawei Rike Chemical Co., Ltd. is the white powder. It was mixed with deionized water and the liquid was fully stirred by a kitchen mixer for 10 minutes to finally form the nano-silica coating. The nano-silica provided by Hangzhou Wanjing New Material Co., Ltd. is the liquid. Their properties are shown in Table 3.

Table 1. Properties of carbon fiber bundles provided by the manufacturer

Filament number	Filament diameter (μm)	Density (g/cm^3)	Elastic modulus (GPa)	Ultimate tensile strength (MPa)	Ultimate tensile Strain(%)
12000	7	1.76	230	3530	1.5%

Table 2. The mix of mortar and compressive strength of cubic standard specimens

Portland cement (P.O 42.5) (g)	Medium sand (0.5-1mm) (g)	Fine sand (0.25-0.5mm) (g)	Water (g)	Redispersible latex powder (g)	superplasticizer (g)	Compression strength			
						14d		28d	
						Mean value (MPa)	COV	Mean value (MPa)	COV
100	133	67	45	1.5	0.15	22.97	0.08	30.13	0.06

Table 3. Properties of nano silica products

Type	Appearance	Partical size(nm)	Purity (%)	Content (%)	Solvent	PH	Supplier Manufacturers
NS-50	White powder	50	99	15	deionized water	-	Beijing Huawei Ruike Chemical Co., Ltd
VK-S01W	White paste	30 \pm 5	-	20	Water	5-7	Hangzhou
VK-S01B	Transparent liquid	15 \pm 5	-	30	Water	9-11	Wanjing New Material Co., Ltd

2.2 Specimen preparation and loading method

The coating process was as follows: the carbon fiber bundles were placed in an acrylic box and pressed with steel plates at both ends to make them in a tensioned state, and then the nano-silica coating solution was poured into the box (Figure 1); the carbon fiber bundles were soaked for 1 h and then taken out and dried. Electrophoretic deposition (Figure 2) process was as follows:

NS-50 at concentrations of 1% and 25% were used, and the specific operation referred to the literature of predecessors(Li et al. 2020).



(a)NS-50 (b)VK-S01W (c) VK-S01B
Figure 1. Modification test of nano silica coatings

After the surface treatment of the carbon fibers, the bottom layer of mortar was poured first and the carbon fiber bundles placed on the surface. Subsequently, the surface mortar was poured and fully vibrated. The specimens were overlaid and maintained for 48h after pouring, and sprinkled with water at room temperature after demoulding. The number of parallel test pieces for pull-out test in each group was five to seven. The Sanso Vertical UTM6503 electronic universal testing machine was used to load at a rate of 0.2 mm/min with a sampling frequency of 10 Hz, and the pull-out test setup is shown in Figure 3.

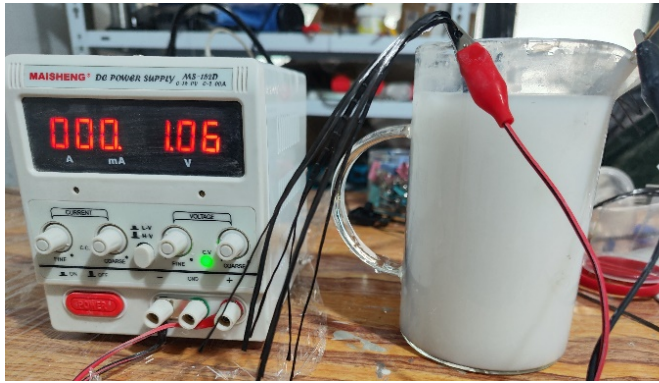


Figure 2. Electrophoretic deposition



Figure 3. The Pull out test of CFRCM bundles

3 Results and discussion

3.1 Test results

This work consisted of two series of tests, recorded as A and B in Figure 5 and Table 4; in test A, the specimens had a bond length of 6 cm and were maintained by water sprinkling at room temperature for 14 days; in test B, the specimens had a bond length of 3 cm and were maintained by water sprinkling at room temperature for 28 days. The nomenclature of the specimens is

“Modified Method - Coating Material - Bond Length - Maintenance Time”. Taking Coating-NS-50-L6-D14 as an example: it means that the specimen has been modified by the coating method and the modified material is NS-50; it has a bond length of 6cm and was maintained with water for 14 days. Figure 4 shows the load-displacement curve of each group of specimens.

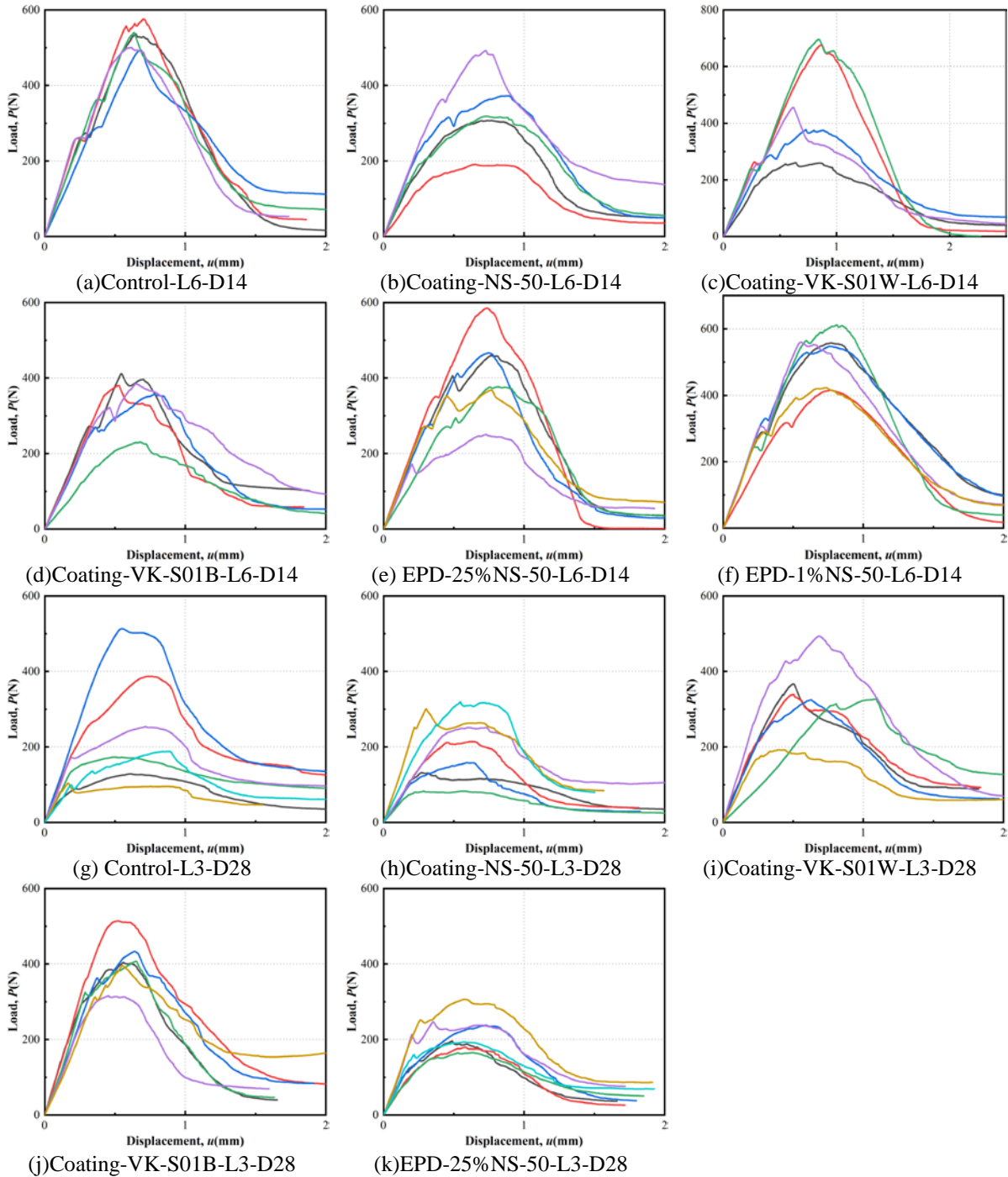


Figure 4. Load-displacement curves of each group of specimens

3.2 Discussion

With reference to relevant studies, the stiffness in the elastic rise phase of the curve, the peak load(Homoro et al. 2020), the average bond strength and the pull-out energy(Pi et al. 2021) can be used as important indicators to characterize the bond properties of the fibers-matrix interface. The stiffness K is the slope of the elastic rising section of the load-displacement curve, which is obtained by fitting. The average bond strength is calculated as follows:

$$\tau_a = \frac{P_{\max}}{nC_f l_e} \quad (1)$$

Where τ_a (MPa) is the average bond strength of the fibers calculated from the peak load, P_{\max} (N) is the peak load, n is the number of embedded fiber bundles in the specimen(1 in this study), C_f (mm) is the circumference of a bundle of fiber bundle (6 mm in this study), and l_e (mm) is the embedded length of the fibers(30 and 60 mm in this study).

The pull-out energy is defined as the mechanical energy consumed by the fiber bundle during a partial or complete pull-out test and can be calculated by integrating the area under the load-displacement curve in part or in full, respectively.

$$W_p = \int_0^{l_{\max}} P(u)du \quad (2)$$

Where W_p (N·mm) is the pull-out energy, $P(u)$ (N) is the pull-out load at displacement u , l_{\max} (mm) is the maximum value of displacement taken from the calculation of this paper(1.5 mm in this study), u (mm) is the slip value at the loaded end of the fibers.

Figure 5 and Table 4 show that the trends of stiffness, peak load and pull-out energy are almost the same for each group of specimens. According to Table 4, K , P_{\max} and W_p of EPD-1%NS-50-L6-D14 series specimens are close to those of Control-L6-D14 series specimens for group A specimens with a bond length of 6 cm. However, K , P_{\max} and W_p of all the remaining test groups are lower, which indicates that the bond properties of the modified specimens have deteriorated. The opposite phenomenon is shown for the group B specimens with a bond length of 3 cm. This is probably because the shorter the bond length, the lower the probability of uneven coating on the fiber and the better the modification will be. Compared to Control-L3-D28, K , P_{\max} and W_p of Coating-VK-S01W-L3-D28 specimens were improved by 8.72%, 36.43% and 27.84%, respectively; K , P_{\max} and W_p of Coating-VK-S01B-L3-D28 series specimens were improved by 30.90%, 64.92% and 40.93%, respectively. This indicates that the bond properties of the specimens modified by these two materials have been improved, but there is no significant effect on the improvement of ductility; K , P_{\max} and W_p decreased in all the remaining test groups, which represents a decrease in the bond properties.

The similar result has been reported(Zamir et al. 2019) for specimens modified by the coating method. The carbon fiber fabric was coated with silica fume with an average particle size of 450 nm mixed with water to form a suspension, but the tensile properties of the modified CFRCM specimens were lower than those of the control group, which contradicts the results of the literature(Dvorkin and Peled 2016, Signorini et al. 2019). The reason may be that carbon fabric, silica fume slurry and impregnation process were different from them. It was reported that carbon-glass fabrics were preimpregnated with silica fume powder with particle sizes of 50 nm and 200 nm(Dvorkin and Peled 2016)respectively, the former greatly improved the mechanical properties of specimens. The ultimate tensile strength of TRM specimens modified

by silica particles with a particle size of 150 nm increased by 31.7%(Signorini et al. 2019).

In addition, it was reported that the peak pull-out load and toughness of the specimen treated with 0.15um silica particles increased by 65% and 51% respectively(Nadiv et al. 2017); On the contrary, the peak pull-out load and the toughness of the specimen treated with 9nm silica particles decreased by 28% and 37% respectively. The reason was that during the drying process of the coating, the particles with smaller particle size presented a high-density agglomeration phenomenon, resulting in damage to the mechanical properties of the carbon fibers in the matrix. The mechanism that nano-silica can improve the bond strength between carbon fiber and cement mortar matrix is as follows(Dvorkin and Peled 2016, Signorini et al. 2019): silica particles can penetrate into the gap between carbon fiber filaments and react with calcium ions in cement paste to form pozzolanic reaction. This can promote the formation of hydrated calcium silicate in the fiber filaments, and make the fiber filaments and cement-based matrix form a good connection.

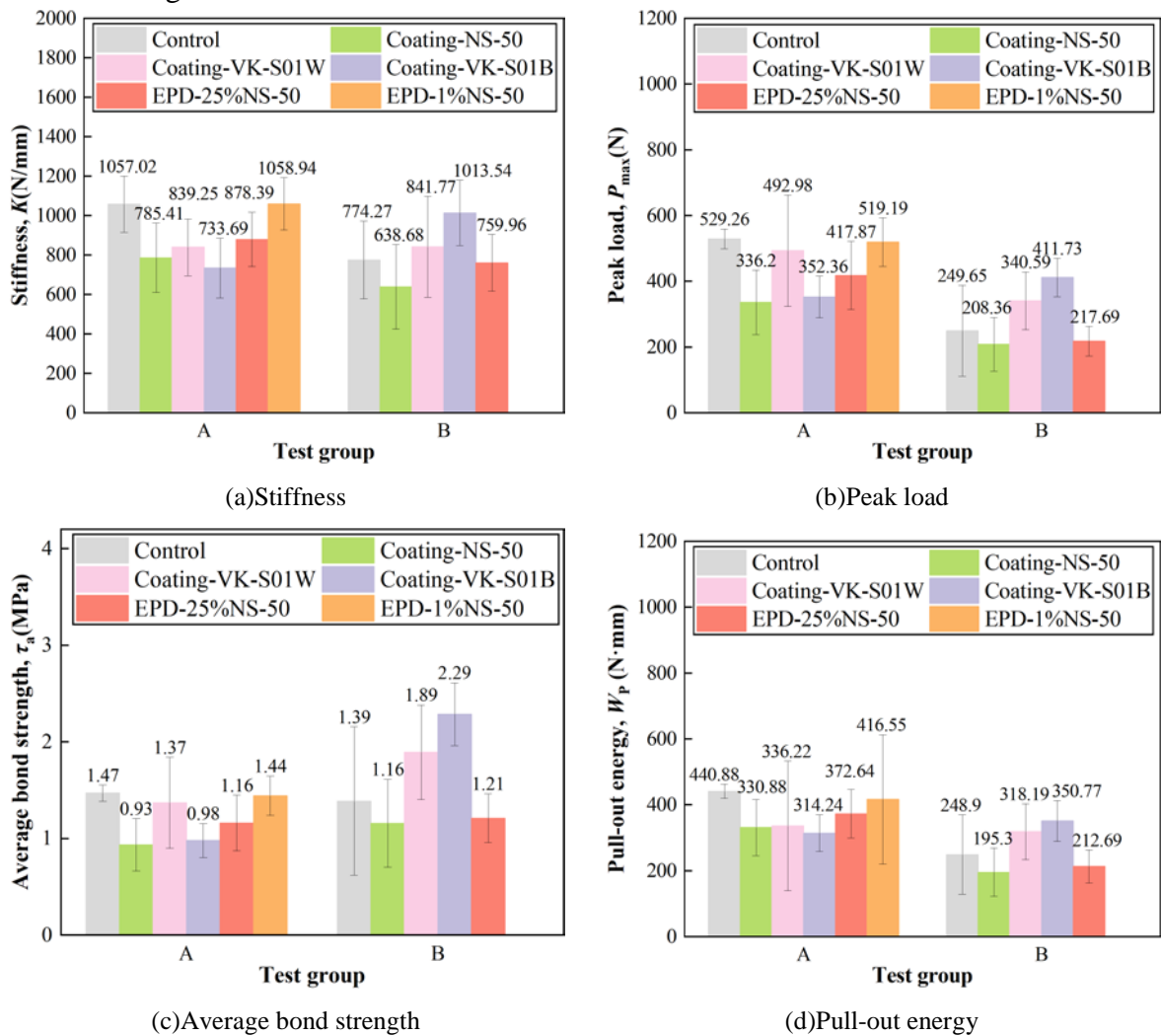


Figure 5. The bonding properties parameters of each group of specimens

Table 4. Improvement ratio of the bond properties parameters for each group of specimens

Test group	Specimens	K	P_{\max} and τ_a	W_p
A	Control-L6-D14	-	-	-
	Coating-NS-50-L6-D14	-25.70%	-36.48%	-24.95%
	Coating-VK-S01W-L6-D14	-20.60%	-6.85%	-23.74%
	Coating-VK-S01B-L6-D14	-30.59%	-33.42%	-28.73%
	EPD-25%NS-50-L6-D14	-16.90%	-21.05%	-15.48%
	EPD-1%NS-50-L6-D14	0.18%	-1.90%	-5.52%
B	Control-L3-D28	-	-	-
	Coating-NS-50-L3-D28	-17.51%	-16.54%	-21.53%
	Coating-VK-S01W-L3-D28	8.72%	36.43%	27.84%
	Coating-VK-S01B-L3-D28	30.90%	64.92%	40.93%
	EPD-25%NS-50-L3-D28	-1.85%	-12.80%	-14.55%

For the specimens modified by electrophoretic deposition, the research conclusion of this paper also contradicts the research of Li(Li et al. 2020). This may be due to the different parameters of carbon fiber, the type of silica, and the embedded length of carbon fiber. In addition, there may be differences in the preparation of test pieces.

To sum up, the current research conclusions on the modification effect of silica particles on carbon fibers are contradictory. Different materials, particle sizes, processes, and the uniformity of coating may present different effects. Related work needs to be further explored.

Further, although the curing period of the specimens in group A is shorter than that of the specimens in group B, it can still be found that the longer the bond length, the greater the peak load. However, Coating-VK-S01B group is an exception. According to Figure 4(c), the peak load of two specimens of Coating-VK-S01B series in group A is close to 700N, but the peak load of other specimens is lower. This may be due to the more dispersed carbon fiber and the lower synergistic stress performance caused by the immersion of the solution during the modification process. Due to the different bond lengths of group A and group B specimens, it is unreasonable to compare the bond properties between the two groups of specimens by K , P_{\max} and W_p , but by τ_a . According to Figure 5, it can be observed that τ_a shows inconsistency with the first three parameters. Compared with Control-L3-D28, τ_a has also verified that the bonding properties of the Coating-VK-S01W-L3-D28 and Coating-VK-S01B-L3-D28 series specimens have been greatly improved, even far exceeding all specimens of group A.

4 Conclusions

- The modification mechanism of silica particles lies in its pozzolanic reaction with cement mortar matrix, which makes the fibers and matrix form a whole and improves the bond properties between them.
- VK-S01W and VK-S01B have a greater improvement in the bond properties of specimens with a carbon fiber embedded length of 3 cm; conversely, they have a negative effect on the bonding performance enhancement of specimens with a carbon fiber embedded length of 6 cm. The phenomenon may be caused by the test process.
- In this paper, the use of other materials and methods also has a negative effect on the improvement of the bond properties. The related process needs to be improved.

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