Experimental study on the strain self-sensing properties of nano carbon black modified resin based CFRP bars

Dan Liu¹,², Ronggui Liu¹, *, Shaofeng Zhang¹

¹Faculty of Civil Engineering and Mechanics, Jiangsu University, Zhenjiang Jiangsu, China
²Architectural Engineering Institute, Jinling Institute of Technology, Nanjing Jiangsu, China

*Corresponding author: liurg@ujs.edu.cn

Abstract. In order to explore the influence of nano carbon black content on the piezoresistive effect of resin based CFRP bars, 2%, 4%, 6%, 8%, 10% nano carbon black modified resin were used as matrix, and the roller extrusion process was adopted to prepare CFRP bars. The initial resistivity, the response of resistance change and the relationship between sensitivity change and strain under static tensile test were compared and analyzed. The results show that the strain sensitivity of CFRP bars can be significantly improved, and the discreteness of test data can be reduced by adding uniformly dispersed carbon black into the resin matrix. The optimum content of carbon black is 6%, the sensitivity is 25.4 in the strain range of 0 ~ 1.2%, and the linear relationship between resistance change rate and strain is satisfied, and the determinable coefficient is 0.985. When the content of carbon black is more than 6%, the effect of modification on the sensing properties of CFRP bars is not obvious.

1. Introduction

Carbon fiber reinforced polymer (CFRP) is one of the ideal materials for major engineering projects to meet the needs of high performance, fatigue resistance, corrosion resistance, and long service life for its excellent performances such as high specific strength, high specific modulus, good designability, lightness in weight, and high corrosion resistance [1, 2]. In addition, CFRP material is a polymer conductive composite material, and the self-sensing function of physical quantities such as strain and temperature can be realized by establishing the correspondence between environmental change factors (such as force field, strain field, temperature field, etc.) and the electrical signal response of CFRP.

Experimental studies have shown that the resistivity characteristic quantity representing the strain self-sensing function of CFRP bars has a large dispersion due to the anisotropy of the composite material [3], and resistance change rate response curve under the external load has a large fluctuation [4]. Therefore, exploring the self-sensing characteristics and sensing performance enhancement methods of CFRP composites in the form of bars is a key problem to be solved at present. By incorporating a certain amount of conductive fillers such as carbon powder, carbon nanofibers, and nickel powder into the CFRP resin matrix, the internal conductive network circuit of the composite can be increased, which can significantly improve the discrete characteristics of the measured values and increase the sensing sensitivity [5,6].
Carbon black nanomaterials are zero-dimensional nanomaterials with superior conductive properties and are an ideal matrix conductive modified filler [7]. There are relatively few studies on the use of nano-carbon black as a modifier for the sensing properties of CFRP bars.

In this study, resin-based modified CFRP bars with different nano carbon black doping were prepared by roll extrusion process. The effect of carbon black doping on the initial resistivity of CFRP bars and the real-time response of the resistivity and sensitivity indexes of CFRP bars under tensile load with the strain of the bars themselves were analyzed, which can provide reference for further application of self-aware CFRP bars in practical engineering.

2. Materials and Methods

2.1. Materials

The carbon fiber is HF10-3K type carbon fiber yarn. The main indexes of carbon fiber are shown in Table 1. The main indexes of epoxy resin and mass ratio are shown in Table 2. The carbon black type is N550, and the particle size is about 77nm.

Table 1. The main parameters of CFRP

| Category  | Tensile strength /MPa | Carbon content /% | Elongation /% | Resistivity /Ω·cm⁻¹ |
|-----------|-----------------------|-------------------|---------------|---------------------|
| HF10-3K   | 3562                  | 95.6              | 1.5           | 1.4953×10⁻³        |

Table 2. Epoxy resin main parameters index

| Category          | Component A | Component B |
|-------------------|-------------|-------------|
| Density (g/cm³, 25°C) | 1.1~1.2     | 0.9~1.0     |
| Viscosity (cps, 25°C) | 1000~1300   | 10~50       |
| Quality ratio     | 100:30      |             |

2.2. CFRP bar specimen preparation

After several tests, resin B component has better flowability than A. And the dispersion of carbon black in component B is better than that in component A under the same mixing conditions.

2.2.1. CFRP bar specimen preparation

Therefore, firstly, the corresponding mass fraction of carbon black and component B were mixed and stirred at high speed with a high speed stirrer for 15min, then both amounts of component A were added and stirred at high speed for 5min to obtain the carbon black modified epoxy resin matrix.

In this test, the mass of carbon black blended into the total mass of the resin matrix was 2%, 4%, 6%, 8%, 10%, and the doping amount of 0% was set as the control group. Carbon black-modified CFRP bars were prepared by roll extrusion process (Figure 1). The sample capacity of the specimens with different doping amounts were 10. The diameter of the test CFRP bars was controlled between 7.8 and 8.3 mm.
After each group of CFRP bars was completely cured, a standard specimen length of 500 mm was intercepted.

In order to prevent the hydraulic chuck of the MTS testing machine from damaging the CFRP bars during the loading test, the steel anchorage needs to be installed at both ends of the bars. The form of anchorage is the internal tapered bonded anchorage, and the pull-out test confirmed that this anchorage can effectively anchor the CFRP bars in the tensile test. The anchorage and CFRP bars were glued with a homogeneous mixture of Lica-300 reinforcing epoxy resin and 0.5-1mm quartz sand in the ratio of 2:1. The anchor is to be maintained for 14 days after the filling.

The distance between the middle measuring electrode of CFRP bar is 150mm, and the outer electrode for power supply and the measuring electrode are 50mm apart. The electrode material is 0.02mm thick and 8mm wide purple copper foil, and the lead wire is connected with the electrode by tin soldering, and the electrode and CFRP bar are firmly bonded by conductive silver paste. After 48h curing of the conductive silver paste, the surface of CFRP bars is evenly coated with 2~3mm thick epoxy resin protection layer by brush.

2.3. Test equipment and test methods
To eliminate the effect of contact resistance on test accuracy, the measurement circuit uses a four-electrode method [8]. A constant current source is used to supply power to the two electrodes near the end with a rated current of 400 mA. The real-time voltage value of the input is automatically collected by the computer through the CFRP TEST SYSTEM data acquisition system, which is homemade and improved by the group. The voltage measurement range is 0~10V, and the test accuracy is ±0.1% FSR with 12-bit resolution.

After the specimens were fabricated, the initial resistance of CFRP bars with different carbon black doping was measured by the above method under the same ambient temperature conditions.

The static tensile test of carbon black modified CFRP bars was carried out using MTS testing machine. The whole test is recorded with a camera for measuring the elongation of the CFRP tendon micrometer real-time changes, conversion can be obtained during the loading of the CFRP tendon real-time strain. The real time resistance of CFRP bars was recorded automatically by the CFRP TEST SYSTEM data acquisition system.

3. Results and Discussion
The initial resistivity distribution, the resistivity-strain and resistivity-change-strain relationships of modified CFRP bars can be obtained by analyzing and processing the measured data.

3.1. Effect of carbon black doping on the initial resistivity of CFRP bars
The initial resistivity distributions measured for six groups of modified CFRP bars with different carbon black doping are shown in the box plot of Fig. 2.

It can be seen that the initial resistivity distribution of CFRP specimens with 0 carbon black doping is scattered, and the initial resistivity values span a wide range of sizes and the data are very discrete. In the process of increasing the mass percentage of carbon black doping to 10%, the mean values of initial resistivity of CFRP bars decreased by 1.59%, 1.72%, 2.36%, 2.19% and 1.99% compared with the control group, and the standard deviation of each group decreased by 59.32%, 69.10%, 91.33%, 87.38% and 86.81% compared with the control group.
The main reason for this phenomenon is that the measured average value of CFRP mass in the CFRP bars prepared by roll extrusion process is 63.4%. There are a large number of contact points between carbon fiber filaments in CFRP bars, and a stable conductive network has been formed inside the tendons. The addition of carbon black can not significantly increase the effective conductive circuit inside the CFRP tendons.

However, the dispersion of the initial resistivity of the CFRP bars was significantly reduced with the increase of the carbon black doping mass fraction. After the carbon black doping exceeds 6%, it is no longer possible to optimize the initial resistivity value dispersion by continuing to increase the carbon black doping.

The main reason is that the carbon black is evenly dispersed in the matrix at the weak inter-fiber contact position inside the bars. And increasing the carbon black content can effectively supplement the weak inter-fiber contact position inside the bars. After the carbon black doping reaches the threshold, the conductive network at the weak position is already connected by the carbon black particles, so the dispersion degree does not change significantly.

3.2. Effect of carbon black doping on the rate of change of resistance of CFRP bars

The response curves of the rate of change of resistance with strain for CFRP bars with different carbon black doping in the whole process of static tensile test are shown in Figs. 3. The distribution of the slope of the curves in the figure can be divided into three variation intervals, which are consistent with our previous theoretical research results [9].

In Figure 3 (a), the data curves of the two control groups have a low overlap in the first interval, indicating a high degree of dispersion of the initial resistance values. And the curves in the second stage
basically overlap, indicating that the internal conductive network tends to stabilize after the stretching of the bars in the second stage.

Comparing Fig. 3 (a) and (b), when the matrix is mixed with 2% and 4% carbon black, the overlap of the test curves in the first zone is higher and the dispersion is improved compared with the control group. However, in the second section, the two data curves changed from the overlap line of the non-doped carbon black group to an open line. The main reason for this is that the carbon black doping is easier to combine with the fiber contacts during the small strain phase, but the total amount of carbon black doping cannot completely connect the intermittent fiber contacts, and the conductive network formed inside the different bars is also contingent, so the two curves cannot completely overlap. It can be inferred that the overlapping curves in the second section of Fig. 3 (a) are accidental, and this inference is confirmed by further tests on other non-carbon black-doped CFRP bars.

When the carbon black doping is 6%, the curves are more coincident in both the first and second stages, and the curves in the third stage also change from non-coincident to coincident. However, the overlap between the resistivity curves tends to decrease when the carbon black doping is further increased. It can be seen that the threshold of carbon black doping in CFRP bars could be in the range of 4% to 8% as shown in Figure 3(b) and (c). And the lowest dispersion between the groups is obtained when the carbon black doping is 6%. With the increase of the carbon black doping, the value of the rate of change of resistance for the same strain increases continuously.

3.3. Effect of carbon black doping on the sensitivity of CFRP bars

In order to study the variation of sensitivity K with strain for CFRP bars with different carbon black doping, the data of the first section in the above test were fitted with endpoint translation lines, and the trend lines of each scattered point were plotted. The slope of the trend lines in Figs. 4 is the sensitivity of CFRP tendon.

Figure 4. Resistance change rate-strain fitting diagram for different CB doping modified CFRP bars

The distribution of the data points in Fig.4(a) is more discrete, which is consistent with the previous analysis. Comparing Fig. 4 (a), (b) and (c), with the increase of carbon black doping, the dispersion of the experimental data was significantly improved and the slope of the fitted straight line (the sensitivity) was slightly increased.

The mean value of sensitivity K increased by 2.1 and the coefficient of determination increased by 0.15 from 0% to 2% of carbon black doping. The value of K increased by 0.82 and the coefficient of determination changed by 0.01 from 2% to 4%. This result shows that 2% and 4% carbon black incorporation gives almost the same sensitivity improvement.

When the carbon black doping was increased to 6%, the average sensitivity value was 25.4, the highest among the six groups, and the coefficient of determination was 0.985. When the carbon black doping was increased, the sensitivity K value decreased compared to the 6% doping group, and was higher than that of the 4% group.
The variation of sensitivity $K$ with the carbon black doping is shown in Fig. 5. It can be seen that the sensitivity coefficient of CFRP bars reaches the maximum when the carbon black doping is 6%. The above experimental results show that the sensitivity of CFRP bars can be effectively improved by adding a certain amount of carbon black conductive phase in the matrix.

![Figure 5. Initial resistivity of CFRP bars modified with different carbon black doping](image)

The sensitivity $K$ increases and then decreases with the increase of carbon black doping from 0% to 10%, and the threshold value of carbon black doping is between 4% and 6%. The effect of increasing the carbon black doping on the continued improvement of sensitivity is no longer obvious, and this conclusion is also consistent with the experimental results of literature [8].

4. Conclusion

The doping of carbon black with a certain mass ratio in the CFRP tendon matrix can significantly reduce the initial resistivity dispersion, but the effect on reducing the initial resistivity value of the tendon is not obvious.

With the increase of carbon black doping, the rate of change of resistance at the same strain level is improved, the sensitivity response was increased, the dispersion of data is reduced, and the optimal doping amount of carbon black is between 4% and 6%.

The CFRP bars modified with nano carbon black have three intervals of resistance change versus strain, and in the strain range of 0–1.2% (the first and second intervals), the relationship is approximately linear, indicating that the CFRP bars can achieve good strain self-sensing.

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