# Multi-scale Carbon-admixtures Enhanced Cementitious Anodic Materials for the Impressed current Cathodic Protection of RC Structures

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Received: 15 July 2015 / Accepted: 12 August 2015 / Published: 26 August 2015

Corrosion of the reinforcing steel is one of the most important metrics which severely deteriorate the durability of reinforced concrete (RC) structures. The anode performance is the critical factor to guarantee the reliability and serviceability of the impressed current cathodic protection (ICCP) system for RC structures. The multi-scale carbon-admixtures, i.e., carbon nanotube (CNT), carbon black (CB) and carbon fiber (CF), are used to enhance the conductivity of cementitious composites and then to prepare the excellent anode for ICCP system. The percolation threshold for each carbon-based cementitious composite is tested, and then the optimal formula with these three multi-scale carbon-admixtures is obtained. Furthermore, the conductive network formed by CNT, CB and CF in the concrete is analysed by the experiments of scanning electron microscope (SEM). Finally, the accelerated polarization experiments are carried out to investigate the serviceability of the cementitious composites have significant potential advantages to be used as the anodic materials for the ICCP system of RC structures.

**Keywords:** Reinforced concrete structures; Impressed current cathodic protection; Anode; Carbonbased cementitious composites; Multi-scale

# **1. INTRODUCTION**

Reinforced concrete (RC) structures are the most important structures in civil engineering [1-3]. In the past few decades, tremendous long-span bridges, skyscrapers, super dams and other immense harbor works have been built. Unfortunately, corrosion of the reinforcing steel is the predominant factor causing the durability deterioration of RC structures, especially in areas exposed to harsh environments, such as de-icing salt or coastal marine, etc [4, 5]. The durability deterioration of RC structures finally leads to very high repair costs, even sometimes much greater than the initial construction cost, and can result in the collapse of the structure directly in some extreme situations. Corrosion of reinforcing steel is being the major concern in civil engineering field [2, 6, 7].

Therefore, it is very urgent to find some effective approaches to prevent and control the initiation and development of the corrosion in the new and existing infrastructures. Over the past few decades, the active corrosion prevention and passive corrosion control methods have been investigated [8-11], e.g., corrosion inhibitors, cathodic protection (CP), electrochemical realkalisation (ERA) and electrochemical chloride removal (ECR), etc. The numerous research results indicate that CP is the only effective way to directly hinder the corrosion of RC structures. Compared with that of the sacrificial anode cathodic protection (SACP), the impressed current cathodic protection (ICCP) is more suitable, effective and promising to be used as an active corrosion control approach for its flexible characteristics on the electric distribution to guarantee the feasibility, effectiveness and reliability of corrosion control system of RC structures.

However, a large number of engineering practices [12-14] indicate that the conductivity and the anti-polarization performance of anode materials is one of the most significant metrics which severely influences the electric field delivery capability, and then severely degrade the serviceability of ICCP system. Over the last few decades, a variety of anode systems have been investigated for ICCP system [12, 15-18], including activated titanium mesh, metalized zinc, coke breeze asphalt, and conductive organic paints. However, these anode systems are either highly expensive, or suffering from durability problems. Developing better types of anodes is of great technological interests. In recent few years, the conductive layers, e.g., conductive polymer layers, conductive cement-mortar layers, are being investigated to be used as the anode systems for ICCP. Among these conductive layers, the carbon-based cementitious layers exhibit huge advantages because of the excellent compatibility with the concrete materials, convenience of installation and possibility of protection of places not easily accessible, etc.

Currently, the possibility of that the carbon-based cementitious composites used as the anode of ICCP has been confirmed [19-22]. However, especially with the presence of multi-scale carbon materials, there is a huge space to improve the conductivity of these carbon-based cementitious composites. On the other hand, the anti-polarization characterization needs to further elucidate. Therefore, we aim to explore the multi-scale carbon-admixtures enhanced cementitious anode for the ICCP system of RC structures in this study. It is worth also mentioning that there are some other multi-functions of this anode, such as pressure-sensibility, electrothermal effect and electromagnetic shielding effect, etc. Considering the length and scope of this paper, we only focus the anode function here, and we will discuss the other functions in the very close future. The contents of this paper are conducted as follows. The experimental details are introduced in section 2. In section 3, we discuss the performance of multi-scale carbon enhanced anodic materials in detail. We conclude this investigation in section 4.

## 2. EXPERIMENTS

#### 2.1 Raw Materials

The composition of carbon fiber (CF), multi-wall carbon nanotubes (MWCNT) and carbon black (CB) are listed in

Table 1. CF, CNT and CB are produced by Qiushi Chemical Co. Ltd. (China), Lishuo Composites Co Ltd (China). and Shenzhen Nano-carbon Co. Ltd (China), respectively.

Component	Diameter	Length	Purity	Surface	Conductivity
CF	7μm	3 mm	>93.0%	90~120 m <sup>2</sup> /g	$6.7 \times 10^2$ S/cm
MWCNT	10 nm~20 nm	5 μm~15 μm	>95.0%	$40 \sim 200 \text{ m}^2/\text{g}$	>100 S/cm
СВ	50~100 nm	_	>98.5%	$10 \sim 50 \text{ m}^2/\text{g}$	0.2 S/cm

Table 1. Property of CF, CNT and CB

The dispersants corresponding to CF and CNT are the methylcellulose (MC, mol. wt.: 40000~180000) produced by Tianjin Chemical Reagent Co. Ltd. (China), and polyvinylpyrrolidone (PVP, mol. wt.: 10000) produced by Tianjin Zhiyuan Chemical Reagent Co. Ltd. (China), respectively. The ordinary Portland cement (C, P.O. 42.5) produced by YaTai Group (China) is used to manufacture the testing specimens. The polycarboxylate superplasticizer is adopted as the water reducing agent (WRA), and the tributyl phosphate (TBP) is used as the deformer.

#### 2.2. Fabrication process of the specimens

Figure 1 presents the fabrication process of the carbon-admixtures enhanced cementitious specimens. Before the water-injection process, the carbon based admixtures, i.e., CB (the mass fraction from 0 to 6.0 wt. %), CF (the mass fraction from 0 to 1.2 wt. %) and CNT (the mass fraction from 0 to 3.0 wt. %), WRA and TBP are mixed together according to the corresponding mass ratio in Figure 1. For CF and CNT, the corresponding dispersants, i.e., MC and PVP, respectively, are also mixed during in this step. Then, the water is injected into the mixture mentioned above, and stirred by the glass rod during the injection process. After this, the mixture is dispersed by the corresponding devices, e.g., traditional mixer, ultrasonic cleaner and ultrasonic cell disrupter for CB, CF and CNT, respectively. Furthermore, the cement is mixed into the uniform suspension prepared by the dispersion process mentioned above, and re-dispersed by the mixer for 5 mins. Finally, the carbon-admixture enhanced cementitious is used to cast the specimens. The prepared specimens are cured in airtight condition for 24 hours, and then accelerated cured for 3 days by the heated water bath (60 °C). The size of all the specimens in this study is  $18 \times 20 \times 24$  mm<sup>3</sup>.



Figure 1. Fabrication process of the multi-scale carbon-admixtures enhanced cementitious anodic specimens.

#### 2.3. Testing of percolation threshold of the carbon enhanced cementitious composite

The percolation threshold is obtained based on the resistivity of the carbon enhanced cementitious composite, and the resistance of the carbon enhanced cementitious composite specimen is measured by the classic two-electrode method. To reduce the influence of the polarization on the measuring results, all the specimens are dried for 18 hours by the electric vacuum drying oven (50 °C). The surface of specimens is polished with 80, 240 and 600 grit silicon carbide paper. Then, the copper foils are bonded to the both ends of the specimen by the ZB2562® silver conductive adhesive (Resistivity:  $10^{-2} \sim 10^{-3} \Omega \cdot cm$ ). A standard resistor, which is close to the resistance of the specimen tested, is connected to the specimen in series. The current through the specimen is equal to that of the standard resistor. On the other hand, the voltage between the both ends of the specimen can be obtained. Thereby, the resistance of the specimen can be easily determined based on Ohm's law. The voltage of the resistance and the specimen is collected by the ADAM4117 card and AdamApax.NET Utility software at the same time.

# 2.4 Testing system of galvanostatic polarization curves of the multi-scale carbon-admixtures enhanced cementitious anodic materials

Figure 2 shows the galvanostatic accelerated polarization-experiment setup of the multi-scale carbon-admixtures enhanced cementitious anode. The classic three-electrode system is used to measure the potential of the anode. The graphite panel  $(30 \times 50 \times 3 \text{ mm}^3)$ , the saturated calomel electrode

(SCE) and the anode prepared are used as the counter electrode (CE), reference electrode (RE) and working electrode (WE), respectively. The size of the multi-scale carbon-admixtures enhanced cementitious anode here is  $18 \times 20 \times 24$  mm<sup>3</sup>, and it is cured in the heated water bath (60 °C) for 3 days after demoulding. To avoid the deterioration of the conductive wire connected to the anodic materials, the graphite rod ( $\Phi$ 3mm×25mm) is embedded into the anodic layer as the primary anode, and the conductive wire is directly connected with this graphite rod, and then the upper and lower surfaces of the specimen are sealed with epoxy resin. The three-electrode system is immersed into two kinds of the solutions in Table 2 which simulate the fresh-concrete pore solution and the contaminated concrete pore solution, respectively; there are three specimens in each solution, and namely, the  $1^{\#}$ ,  $2^{\#}$  and  $3^{\#}$ specimens and  $4^{\#}$ .  $5^{\#}$  and  $6^{\#}$  specimens of each type of multi-scale carbon enhanced cemnetitious anodic materials were placed in the solution I and the solution II, respectively. The solution is replaced one time for each 24 hours. The polarization current density of  $1200 \text{ mA/m}^2$  is taken during the test. and the standard resistor of 100  $\Omega$  is connected to the specimen in series to monitor the current in the circuit. When we monitor that the voltage between the both ends of the resistor is 0.22 V, the desired polarization current density is assured. The sampling frequency of the polarization potential is one time for each 3 hours.

Table 2. Two kinds of solution used to simulate the service condition of RC structures.

Solution	Composition (Unit: mol/L)	pН
Ι	Ca(OH) <sub>2, sat.</sub> + 0.2NaOH+0.6MKOH	13.5
II	Ca(OH) <sub>2, sat.</sub> +0.2NaOH+0.6MKOH+3.5 wt.% NaCl	13.5



Figure 2. Schematic plan of the accelerated polarization-experiment test.

#### 2.5 Microscopic analysis

At the end of the accelerated polarization experiment, all the polarized and unpolarized specimens are cut into the small blocks and the cubes  $(5 \times 18 \times 20 \text{ mm}^3)$  shown in Figure 3. SEM is used to analyze the conductive network of multi-scale carbon-admixtures dispersed in the cement matrix. The surface morphology of the specimens is simultaneously observed to evaluate the performance of carbon-admixtures after the polarization. In addition, we analyze the element distribution along the arrow 1, 2 and 3 illustrated in Figure 3 by the Energy-Dispersive Spectroscopy (EDS) with SEM, and then the content of sodium and potassium ions along the arrows can be obtained. The mentioned microscopic analysis is conducted by HELIOS NanoLab 600i FIB/SEM dual beam focused ion/electron microscope produced by the FEI Company in America.



Figure 3. The specimens prepared for EDS analysis.

#### **3. RESULTS AND DISCUSSION**

#### 3.1 Percolation threshold of the carbon enhanced cementitious composite

Figure 4 presents the change of resistivity versus the mass fraction of CF, CB and CNT. The resistivity of all the specimens decreases with increasing the content of carbon materials. There are the notable zones where the resistivity decreases sharply, and these notable zones are called the percolation transition zone. Thus, the percolation transition zones of these three carbon based cementitious composites are  $0.2 \sim 0.4$  wt. %,  $1.5 \sim 2.5$  wt. % and  $1.0 \sim 3.0$  wt. %, respectively.



**Figure 4.** The resistivity of the cement specimens with different carbon content. a), b) and c) exhibit the change of conductivity with CF, CNT and CB, respectively.

Tunneling effect theory can explain this percolation phenomenon [23]. When the content of carbon materials is in the percolation zone, the jumping of electron and electron hole between the particles of carbon based materials dominates in the conductivity of the composite, so the resistivity of the composite decreases dramatically and the conductivity of composite is highly sensitive to their contents added into cement matrix in this zone. When the content of carbon materials is lower than the percolation zone, the distance between the particles is further and it is very difficult for electrons and electron holes between the conductive particles to jump forming electric current. Accordingly, the conductivity of composite is mainly determined by the free-movement ions in cement matrix at this moment, e.g.,  $Ca^{2+} \ Na^+ \ SO_4^{2-} \ OH^-$ , and hardly influenced by the content of carbon based material. However, when the content of carbon materials exceeds the percolation zone, well physical contact between the particles is achieved in the cement matrix, and this good contact leads to that adding more carbon materials can't make the composite get the substantial increase of the conductivity.

3.2 Resistivity of the multi-scale carbon enhanced anodic materials

If the content of single carbon material is higher than that of the percolation zone in the cement matrix, the conductivity of the composite maybe not improved greatly. Therefore, we choose the contents in the percolation zone for each carbon material, e.g., 0.2 wt. %, 0.3 wt. % and 0.4 wt. % of CF, 1.5 wt. %, 2.0 wt. % and 2.5 wt. % of CNT, and 1.0 wt. %, 2.0 wt. % and 3.0 wt. % of CB.

From the geometric parameters of CF, CNT and CB shown in

Table 1, when these three kinds of carbon based materials are simultaneously added into the cement matrix, CF is the skeleton of the conductive network, and CB and CNT are the fillers to perfect the skeleton conductive network. Table 3, Table 4 and Table 5 show the resistivity of the composite mixed with CF, CB and CNT. We can find that the resistivity has a sharp fall after that CNT and CB is mixed into the cement matrix. When 0.2 wt. %, 0.3 wt. % and 0.4 wt. % of CF are solely added into the cement matrix, the resistivity is  $1.61 \times 10^7 \ \Omega \cdot \text{cm}$ ,  $1.01 \times 10^6 \ \Omega \cdot \text{cm}$  and  $1.85 \times 10^3 \ \Omega \cdot \text{cm}$ , respectively. The maximum and minimum resistivity of the corresponding multi-scale carbon enhanced anodic materials decreases to 10050  $\Omega \cdot \text{cm}$  and 21.66  $\Omega \cdot \text{cm}$ , 1190  $\Omega \cdot \text{cm}$  and 17.31  $\Omega \cdot \text{cm}$ , and 236.67  $\Omega \cdot \text{cm}$  and 11.94  $\Omega \cdot \text{cm}$ , respectively. These results show that CB and CNT can perfect the skeleton conductive network of CF in the cement matrix.

Figure 5 gives the SEM images of the multi-scale carbon-admixtures enhanced cementitious anode.

Figure 5 b) is the SEM image around the CF in

Figure 5 a); we can see plenty of CB particles and flexing CNTs around the CF in

Figure **5** b);

Figure 5 c) reveals the surface morphology of the CF where CB particles and flexing CNTs wind and attach on the surface of CF. According to

Figure 5 d), we find that CB particles can be chained well together by CNT. The results indicate that CB and CNT can fill the conductive network very well.

Table 3	. The resistivity	of the multi-scale	carbon admixtures	enhanced	cementitious	anodes	where the
n	nass fraction of	CF is 0.2 %.					

CB CNT	1%	2%	3%
1.5%	10050	192.11	106.28
2.0%	180.86	45.20	24.80
2.5%	104.39	42.18	21.66

**Table 4.** The resistivity of the multi-scale carbon admixtures enhanced cementitious andoes where themass fraction of CF is 0.3 %.

CB	1%	2%	3%	

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CNT			
1.5%	1190	117.90	21.33
2.0%	89.99	33.26	17.31
2.5%	63.30	30.38	18.00

**Table 5.** The resistivity of the multi-scale carbon admixtures enhanced cementitious anodes where the mass fraction of CF is 0.4 %.

CB CNT	1%	2%	3%
1.5%	236.67	35.03	14.51
2.0%	40.52	14.90	11.94
2.5%	33.02	16.02	15.90



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**Figure 5.** The conductive network of the multi-scale carbon-admixtures in cement matrix observed by SEM. a) and b) present the SEM image of CF selected randomly in the cementitious matrix and the SEM image of the distribution of the CNTs and CBs around the CF in the

Figure 5 a), respectively; c) and d) present the distribution of the CNTs and CBs at the surface of CF.

The fluidity of cement paste is quite poor when CB and CNT contents are both over 2.0 wt. % or CF content is over 0.4 wt. %. Taking into account the cost and conductivity of anodic materials simultaneously, CF02CNT20CB1 (CF02CNT20CB1 means that the content of CF, CB and CNT are 0.2 wt. %, 2.0 wt. % and 1.0 wt. %, respectively), CF03CNT15CB2 and CF04CNT15CB1 are adopted the reasonable multi-scale carbon enhanced anodic materials. Therefore, the following long-term polarization testing focuses on the three above mentioned mixture ratios.

#### 3.3 Long-term polarization performance

Figure **6** presents the change of the polarization potentials versus time, which is polarized by the constant current, i.e.,  $1200 \text{ mA/m}^2$ . According to these polarization curves, we can see clearly that the potential increase very slowly from tens to hundreds of hours, and then it suddenly reach the upper limit of the impressed voltage maintaining the constant density in a very short time. The change of the polarization potential here is determined by the resistance of the anode. Accordingly, the conductivity of the multi-scale carbon-admixtures enhanced cementitious anode is very stable at the beginning accelerating polarization process, i.e., from tens to hundreds of hours, and then the conductivity is deteriorated in a very short time. By comparison with the cementitious anode reported by the literatures [21, 22], we can find that the conductivity of the multi-scale carbon-admixtures enhanced cementitious anode can bear at the stable duration is much higher. Table 3 gives the stable durations of all the specimens with the polarization current of 1200 mA/m<sup>2</sup>; the average stable duration of CF03CNT15CB2 have the most excellent anti-polarization ability.

By the comparison of the polarization potential curves between the  $1^{\#}$ ,  $2^{\#}$  and  $3^{\#}$  specimens and  $4^{\#}$ ,  $5^{\#}$  and  $6^{\#}$  specimens in

Figure 6 a), b), c), we find that there is no difference about these polarization curves, and this indicates that the multi-scale carbon enhanced cementitious anodes are immune to the chloride ions.

When the specimens are immersed into the electrolyte solution, plenty of the ions in the solution, e.g., Na<sup>+</sup>, K<sup>+</sup>, OH<sup>-</sup> and Cl<sup>-</sup>, migrate into the specimens. Figure 7 illustrates the distribution of sodium and potassium ions along the arrows in the Figure 3. The contents of sodium and potassium ions reduce gradually along the arrows in the polarized specimens; however, this phenomenon doesn't occur in the unpolarized specimens. This indicates that the cations, e.g., sodium and potassium ions, are forced to move to the position of lower potential with the effect of the electric field. Similarly, we can deduce that the anions, e.g.,  $SO_4^{2-}$ , OH<sup>-</sup>, are forced to move to the position of higher potential.

Thus an internal anti-electric field would appear in the polarized specimen, and this causes a certain raise of the polarization potential.



**Figure 6.** Galvanostatic polarization curves of the multi-scale carbon admixtures enhanced cementitious composites with the constant current density (1200 mA/m<sup>2</sup>). a), b) and c) show the potential changes of the specimens CF02CNT20CB1, CF03CNT15CB2 and CF04CNT15CB1, respectively.



Figure 7. Distribution of the cations along the arrows in Figure 3. a) and (b) exhibit the content of Na<sup>+</sup> and K<sup>+</sup>, respectively. ("P" and "UP" denote polarization and unpolarization at the same circumstance, respectively, e.g., "UP1" denotes the cation distribution along the arrow 1 of the unpolarization specimen)

Figure 8 present the SEM of the polarized and unpolarized probe. From

Figure 8 a) and b), we can see clearly that the graphite rod bonds with the anodic material strongly in the unpolarized specimens; however, there is a big gap between the graphite rod and the anodic material in the polarized specimen. Thus, the deterioration of the interface between the graphite rod and the anodic material is one of the most important factors which lead to the rapid increase of the polarization potential. Compared the surface topography of CF in the polarized specimens under SEM, the surface of some polarized CFs is shown to be much coarser.

Figure 8 c) and (d) presents the surface topography of a CF selected randomly in the unpolarized and polarized specimens. Yue [24] pointed out that the content of the oxygen at the surface CF increased when CF were continuously electrochemically oxidized by applying current in salty solution. Momma [25] found that the resistivity of CF increased after electrochemical oxidation.



**Figure 8.** SEM images of the carbon-admixtures in cement matrix after the long-term accelerated polarization process. a) and b) present the change of the interface between graphite rod and anodic material in the polarized and unpolarized specimens, respectively; c) and d) show the surface topography of the CF in the polarized and unpolarized specimens, respectively.

Therefore, the conductivity of carbon-admixtures here becomes worse after the long-term polarization. This is one of the another major reasons leading to the increase of polarization potential.

According to the mentioned polarization mechanism, the polarization process of the anodic materials consists of two periods, i.e., the normal service period and the loss-service period; the normal service period is the stable duration of the polarization potential of the anodic materials, and the loss-service period is the mutation duration of the polarization potential of the anodic materials. For the stable duration, ations and anions could form an anti-electric field in the specimen; however, the weak anti-electric field has little influence on the serviceability of the anodic materials. For the mutation period, the electrochemical oxidization phenomenon of the carbon-admixtures and the deterioration of the interface between the anodic material and the graphite rod could result in the conductivity of the

anode a sharp increase, and this means that the anode could greatly increase the current consumption and severely degrade the electric field delivery capability of an ICCP system.

#### 4. CONCLUSIONS

The novel multi-scale carbon-admixtures enhanced cementitious composites are prepared and characterized to provide the critical anode for the ICCP system of RC structures. The conductive percolation transition zones of the cement based composites added with CF, CNT or CB are 0.2~0.4 wt. %, 1.5~2.5 wt. %, 1.0~3.0 wt. %, respectively. The optimum proportion of multi-scale carbon-admixtures enhanced cementitious anode is CF03CNT15CB2. The polarization mechanism of the novel anodic materials can be clearly revealed by the two-stage model. The accelerated service-life of CF03CNT15CB2 is more than 200h even under the extremely harsh polarization circumstances, and it is immune to CI<sup>°</sup>. The multi-scale carbon-admixtures enhanced cemential advantages to be used as the anodic materials for the ICCP system of RC structures.

#### ACKNOWLEDGEMENTS

This research is supported by grants from National Nature Science Foundation of China (Project No.: 51378156, 51008098 and 2011CB013604), the Ministry of Science and Technology (Project No.: 2011BAK02B01), the Fundamental Research Funds for the Central Universities (Project No.: HIT.BRETIII.2012 33) and the Natural Science Foundation of Heilongjiang Province of China (Project No.: E201222).

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