Electrical Resistivity Measurement of Cement-Based Binders Using Embedded Four-Terminal Probe Method

Tsung-Chin Hou[1], You-Hua Chiou[1], Huang-Hsing Pan[1], and Chao-Hsun Chang[2]

[1]*Department of Civil Engineering, National Kaohsiung University of Applied Sciences, Kaohsiung, Taiwan*

[2] *Department of Construction Engineering, National Kaohsiung First Univ. of Science and Technology, Kaohsiung, Taiwan*

E-mail: tchou@cc.kuas.edu.tw

ABSTRACT: This paper uses embedded four-terminal probe method to study the electrical resistivity of cement binders. There are four kinds of materials being studied including plain cement, 15% weight of fly ash, 30% weight of slag, and 1% volume of graphite mixed binders. Resistivities of the specimens are measured under static and loading states. For the static tests, the measured resistivities and polarization histories are used to detect the hydration degree and age of the cement-binders. The experimental results show that the volumetric resistivities grow with the materials' age due to hydration process. The induced polarization effects are prominent at the early age (1 day) but moderate after 7 days. It is also found that the saturation time of polarization would decrease with the age of cement binders. For the loading tests, resistivity measurements are shown to be capable of identifying the occurrence of critical damages under compression and flexure. By adding conductive particles such as graphite powders into the cement mixtures, electrical resistivity can be lowered.

1. INTRODUCTION

Concrete is a composite material composed of cement, water, aggregates, and other additives. Although the mechanical strengths and composing ratio are higher of aggregates, cement binders still play a critical role that governs the overall performance, such as compressive and flexural strengths of concrete. Since concrete stands a major part of most civil infrastructures, it is necessary to develop certain practical techniques that could provide promising understanding of concrete elements' compositions, chemical properties, and the corresponding physical behaviors.

Electrical measurement has shown to be efficient and economic an approach for detecting concrete's physical conditions such as temperature, stress, strain, and cracking, etc [1]. In this paper, we employed electrical resistivities of concrete under constant electric fields (DC) to study its material properties. Compared with other physical parameters, electrical resistance is relatively a quantity simple to acquire. The measured resistance is then converted into resistivity that could represent the scaleless material parameter. Electrical measurement also ensures the structural integrity of the objects and thus, belongs to one of the non-destructive examination (NDE) techniques.

Instead of using conventional four-terminal approach with which probes are installed on objects' surfaces or perimeters [2-3], we have adopted embedded four-terminal probe method to obtain the electrical resistivity of cement binders. The purposes of using this probing approach are to provide the concrete specimens a more homogeneous electric field for electrical measurement while causing no significant deterioration to the structural integrity. The static tests studied the hydration degree and concrete age by observing the polarization characteristics. Similarly, the loading tests examined the changes of resistivity with respect to stress, strain, and the occurrence of critical damage.

2. EXPERIMENTS

2.1 Measuring Techniques

There are four approaches for resistance measurements in fields: two-terminal direct current method, two-terminal alternating current method, three-terminal direct current method, and four-terminal probe method [4]. Among those, four-terminal probe method has shown to be of less measuring inaccuracy by offering the electric current through the two outmost electrodes and measuring the voltage responses from the two inner electrodes [5]. In recent years, researchers have applied this method for resistance measurements of cementitious materials by either wiring copper probes along the objects' surfaces [1,6], or embedding metal plates into the objects' interiors [7].

In this paper, we have adopted the four-terminal probe method by embedding round copper sticks as terminals into cement binder specimens, as shown in Figure 1. The purposes of using this probing approach are to provide the specimens a more homogeneous electric field for electrical measurement while causing no significant deterioration to the structural integrity. Therefore, this approach can then quickly deploy into fields for practices.

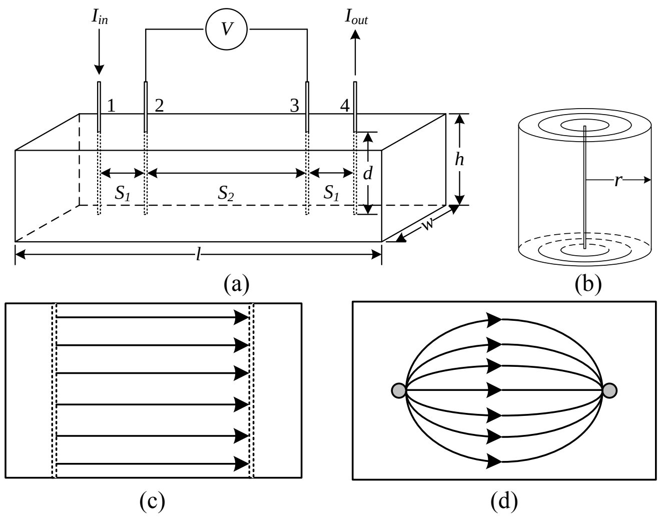


Figure 1 (a) embedded four-terminal probe method, (b) the induced electric field at electrode 1 and 4, (c) current paths side view, (d) current paths top view

2.2 Finite Boundary Resistivity

A commonly used approach for computing electrical resistivity of a finite boundary object is Smits equation [8]. However, this equation is applicable for surface-contact electrodes with a semi-infinite conducting field, which is not the case of this study. Therefore, we have modified Smits equation to obtain a finite boundary resistivity (FBR) equation for the embedded probe method. The modification is based on the following assumptions:

1. current moves along the latitude of probe array, as shown in Figure 1(c) and (d)
2. current density is evenly distributed along the probe longitude
3. the interior electric field of each probe is neglected
4. potential between the inner electrodes only depends on the resistivity of the conducting medium

With a current, I, injected at a certain electrode, the generated current density, j, at distance, r, from the electrode is:

$$j = I / 2\pi r d \tag{1}$$

where d is the embedded length of the electrode. The corresponding electric field of the electrode can then be expressed as:

$$E(x) = j / \sigma(x) = \rho(x) \cdot I / 2\pi rd \quad (2)$$

where $\sigma(x)$ is the electrical conductivity of the material and $\rho(x)$ is the resistivity; $\rho(x) = 1/\sigma(x)$. Accordingly, the potential function, $V(x)$, at distance, r, from the electrode is:

$$V(x) = \int E(x)dr = \rho(x)I / 2\pi d \int 1/rdr = \rho(x)I / 2\pi d \cdot \ln r + C \quad (3)$$

Assuming the resistivity is constant along the current paths, $\rho(x) = \rho$, electrode 1 is subjected to current input, and electrode 4 is subjected current output, the electric potential can thus be expressed as:

$$V = \rho I / 2\pi d \cdot (\ln r_4 - \ln r_1) + C \quad (4)$$

where r_4 is the distance from electrode 4 and r_1 is the distance from electrode 1. Therefore, the potential difference, ΔV, between electrode 2 and 3 is:

$$\Delta V = V_2 - V_3 = \rho I / \pi d \cdot \ln((S_1 + S_2) / S_1) \quad (5)$$

where S_1 and S_2 can be referred in Figure 1. With the measured voltage of the inner electrode pair, the electrical resistivity of the conducting medium can then be determined.

$$\rho = \Delta V / I \cdot \pi d / \ln((S_1 + S_2) / S_1) \quad (6)$$

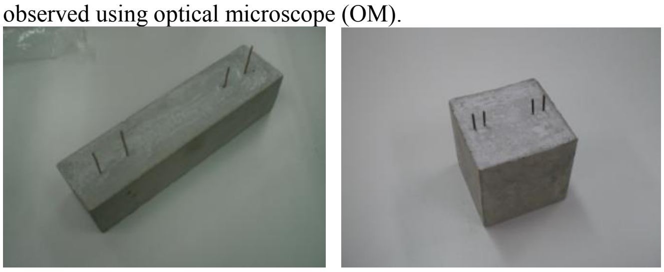
2.3 Experimental Preparation

There were four groups of cement-based binders being studied in this paper, including plain cement (Group 1), 15% weight of fly ash (Group 2), 30% weight of slag (Group 3), and 1% volume of graphite (Group 4) binders. CNS 61 Type I Portland cement, F grade fly ash, and ASTM C989 standard slag were selected and used in the preparation of specimens, respectively. The particle size of graphite powder is 30nm in diameter, and is supplemented with specified dispersant. Specimens of the dimension (height by width by length) 50x50x50mm and 40x40x160 were prepared and cured in sealed bag at room temperature for 1, 7, 14, 21, 28 days before tests. The mixing proportion for all groups is summarized in Table 1.

Table 1 Mixing proportion by weight (%)

w/b	0.4				
group #	water	graphite	cement	fly ash	slag
1	40		100		
2	40		85	15	
3	40		70		30
4	40	1(v%)	99 (v%)		

As for the metal probes, round copper sticks of 1mm in diameter were used. The electrodes are installed with S_1 = 20mm, S_2 = 80mm, and d = 40mm for the rectangular specimens; S_1 = 5mm, S_2 = 30mm, and d = 50mm for the cubes. In this study, all the probes were installed upon the casting work of cement mixtures; however, it is expected that post-instrumentation of the electrodes can rapidly be applied to existing concrete structures. Samples of the completed testing specimens are shown in Figure 2. Keithley 2611 System SourceMeter was employed for both current supply and voltage measurements. Porous structures of the binder specimens were also observed using optical microscope (OM).



(a) (b)

Figure 2 Prepared (a) rectangular and (b) cubic binder specimens

3. RESULTS AND DISCUSSION

3.1 Static Test – DC Resistivity

Rectangular specimens were used for static DC resistivity test. Electrical measurements were carried at various curing age (1, 7, 14, 21, 28 days) with direct current of 10mA as the input. As expected in Figure 3, resistivity grew with age due to hydration effects of the binders, such as reduction of ionic concentration, vaporization of water, formation of porous structures, etc. After 1 day of curing, resistivities of the four binders stand close still, but grew individually with different rates after 7 days. Among those, group 2 and 3 shows relatively larger resistivity values than plain cement binders. The 28-day resistivity of group 3 has reached 107.45kOhm-cm, which is 3.3 times the value of group 1 (32.52kOhm-cm). Similarly, the 28-day resistivity of group 2 is about 2.1 times the value of group 1. These consequences were attributed to the replacement of cement by fly ash and slag, which would reduce the concentration of hydroxyl ions (OH^-) of the binders and thus, shrink the size of pore distribution [9-10]. The OM images taken at 28 days of curing age (Figure 4(a), (b), and (c)) have further confirmed this observation.

In addition to the higher growing rates of resistivity, Figure 3 also illustrate that both the resistivity of group 1 and 4 appear to stabilize after 28 days; while group 2 and 3 keep rising continuously. Although our results indicate that fly ash and slag may cause similar effects on resistivity growth of cement binders, further investigation in this phase is required. It should also be mentioned that highly conductive graphite was supposed to reduce the resistivity of cement binders; however, this effect was not significant as shown in Figure 3. Large porous structures caused by the addition of dispersant were found within the binder specimens (Figure 4(d)) and thus, restrained the influences of conductive graphite powders.

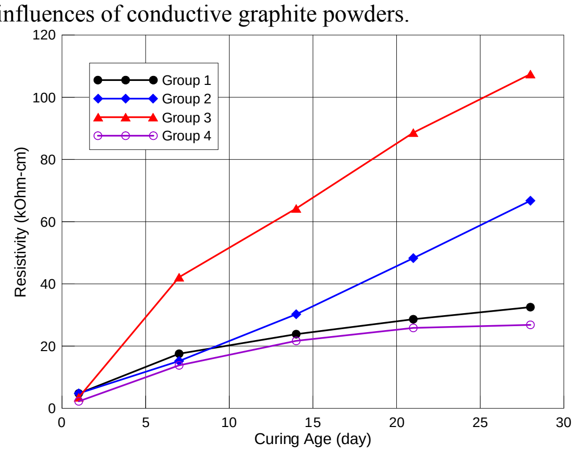
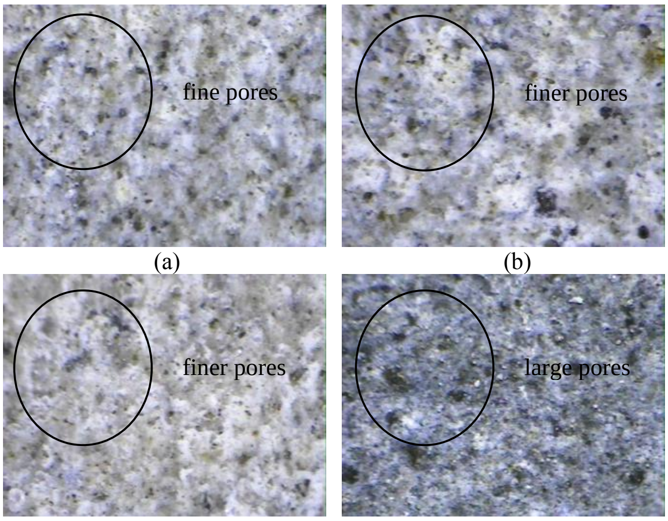


Figure 3 Effect of curing age on electrical resistivity



(a) (b) (c) (d)

Figure 4 OM images (×300) of (a) plain cement, (b) 15% fly ash, (c) 30% slag, and (d) 1% graphite binders

3.2 Static Test – Dielectric Effects

Since cement-based materials are known to be dielectric, ionic polarization would occur more or less when exposed to an electric field. Similar to DC resistivity, this inherent nature has also been proposed for self-sensing applications of cement-based materials [11]. In this test, resistivities of each specimen were recorded both before and after saturation of polarization. DC current of 10mA was supplied to each specimen for 3600 seconds in order to ensure a complete saturation of polarization was reached. The initial and polarized resistivities for both rectangular and cubic specimens at 1, 7, 14, 21, and 28 days of curing age were presented in Figure 5.

For plain cement binders, polarization has resulted in a similar amount of resistivity shift (1-3kOhm-cm) through the curing age. This phenomenon may suggest that under the specified curing condition (room temperature in sealed bags); hydration process is regularly affecting the ionic concentration as well as free water of cement binders. Similarly, the steady shift of resistivity can also be observed in group 2 specimens (5-7kOhm-cm) except for the first day, where the applied electric excitation has resulted in much larger shift of resistivity. Fly ash replaced certain amount of cement, restrained the hydration of C_3A and C_3S at very early age, and resulted in vast amounts of free water [12]. The excessive free water is thus responsible for the significant polarization.

A steady shift of resistivity can also be observed for group 3 binders before 14 days (4-8kOhm-cm). After that, the effect appears to be nullified as applied electric excitation caused no polarization effect. The reason remains unclear while the high growth rate of initial resistivity indicate that the ionic concentration of slag-cement binders is lower than fly ash-cement binders after 21 days of curing age. For group 4 binders, an increasing resistivity shift with respect to curing age was observed. The addition of conductive graphite particles did reduce and stabilize the electrical resistivity. Our results have suggested that the hydration of cement was not affected by graphite powder while the supplemented dispersant caused large porous structures trapping most ions at late curing age. As a result, these trapped ions then reacted to the constantly applied electric excitation (DC current), leading to a slight increasing of polarization effect after 14 days.

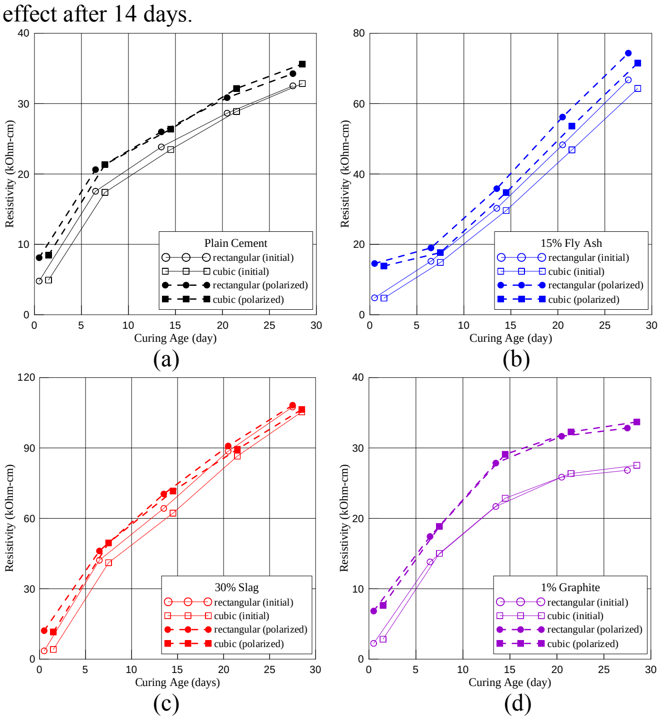


Figure 5 Effect of polarization on resistivity shift of (a) plain cement, (b) 15% fly ash, (c) 30% slag, and (d) 1% graphite binders

It should be mentioned that the measured electrical resistivities of cubic binder specimens were very close to the value of rectangular specimens. This observation has further validated the applicability of using finite boundary approach for computing electrical resistivity of embedded four-terminal probe method. Although there still exists a slight discrepancy (either initial or polarized resistivity) between rectangular and cubic specimens, it is highly anticipated that this approach can rapidly be deployed for field practices.

3.3 Static Test – Saturation of Polarization

When direct currents were constantly applied to cement-based materials, electrical resistivity would grow with charge time until all the ions were fully dipoled. The duration required for polarization saturation varied with compositions and mixing proportion of cement-based materials [13]. Table 2 has listed the saturation time of all the binder specimens investigated in this study. The duration of polarization saturation reduced with the curing age; while also varied with respect to material composition and specimen geometry. The reduction of saturation time is primarily associated with the completion of hydration process and pore distribution. As the pore structures were maturely formed, determined conduction paths were then served as shortcuts for those ions within pore water to quickly polarize under external electric fields.

Table 2 Saturation time of polarization (sec)

group #	specimen type	1 day	7 days	14 days	21 days	28 days
1	rectangular	1500	1200	1000	700	300
	cubic	1500	1300	1100	800	400
2	rectangular	1300	1200	1000	400	300
	cubic	1500	1300	1100	500	300
3	rectangular	1500	1200	800	700	500
	cubic	1400	1300	800	500	300
4	rectangular	1000	900	800	700	600
	cubic	1100	1000	900	700	700

Unlike the resistivity growth discussed previously, fly ash and slag seemed to cause no significant effects on saturation time of cement binders. The inclusion of conductive graphite particles, on the other hand, appeared to effectively shorten the saturation time before 14 days; while the duration stayed at 700 seconds after 21 days. Since graphite powders did not cause severe disturbances to the hydration process of cement, the large pores formed in between 14 to 21 days may be responsible for this. It should be mentioned that a reported saturation time for plain cement binder with w/b = 0.35 is 200 seconds [13], which is very close to the value of group 1 specimens (300 seconds, plain cement binders with w/b = 0.4) in this study. Based on our limited results, the geometry and size effects on saturation time remained unclear.

3.4 Loading Test – Compression

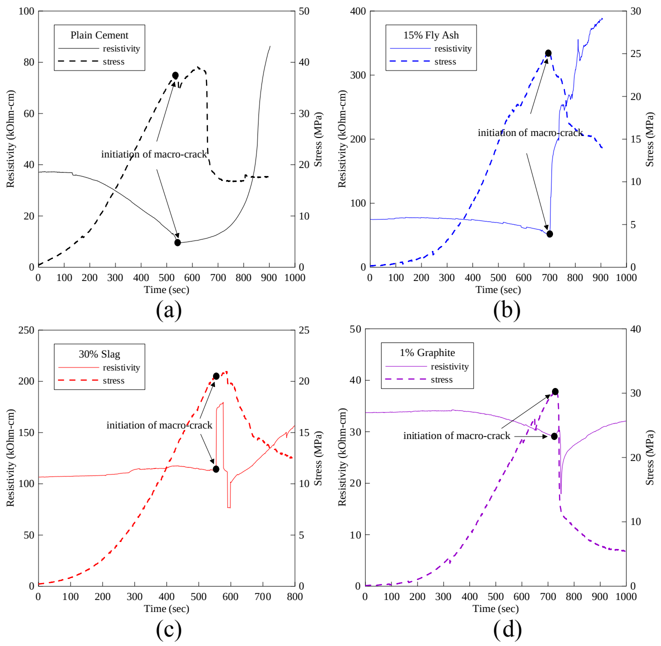


Figure 6 Effect of compression on electrical resistivity of (a) plain cement, (b) 15% fly ash, (c) 30% slag, and (d) 1% graphite binders

Cubic specimens were used for the uniaxial compression test after 28 days of curing. Each specimen was supplied with a 10mA DC current for quite a few minutes. This will ensure the saturation of polarization was reached before any mechanical load was applied. The four electrodes were arranged parallel to the loading direction of MTS machine, and the electrical measurements were recorded with a sampling rate of 1Hz during the loading test. Figure 6 shows the test results of all the four binder cubes. In each case, resistivity was shown to precisely capture the initiation of macro-crack with the occurrence of sharp fluctuation. Resistivity then grew with another rate as the macro-crack propagated. Since the locations and propagation of macro-cracks were not controllable in prior, the growth of resistivity would thus develop into various patterns, as shown in the figure. Nevertheless, electrical resistivity measurement using embedded four-terminal probe method was shown to provide cement-based materials a self-sensing capability.

It must be noted that as the compression began to load onto the cubes, a slight decreasing of resistivity can also be observed. This was primarily caused by the compressive strain which would shorten the distance between pores within the materials, and thus decrease the electrical resistivity. It can also be observed that there exists certain ratio (gage factor, GF) in between the percent change of electrical resistivity and the compressive strain of cement binders. However, further study is required for quantifying the gage factors with respect to different type of materials at specific strain levels.

3.5 Loading Test – Flexure

Similar to the cubes used in compression test, rectangular specimens cured for 28 days were employed for the flexure test. In this test, the location and propagation of macro-crack can be predicted in prior; the four electrodes were then placed perpendicular to the loading direction with the inner pair cross the potential location of macro-crack. Saturation of polarization is also completed before three-point flexural loading was applied. Figure 7 shows the effect of flexure on electrical resistivity of all the four cement binders. In each case, resistivity was again shown to precisely capture the occurrence of macro-crack with a sudden increasing of resistivity. This result was expected since the failure crack would locate at the center of the specimen and thus, block the current paths.

Unlike the effect of compression, flexural strain appeared to be of subtle influence to the electrical resistivity. As shown in Figure 7, resistivity experienced only very few amount of growth before macro-crack was quickly generated. The combined tensile and compressive strain has caused a more complicated mechanism of electric conduction. Therefore, we suggested a further study of four-terminal probe method to the effect of flexure on cement resistivity.

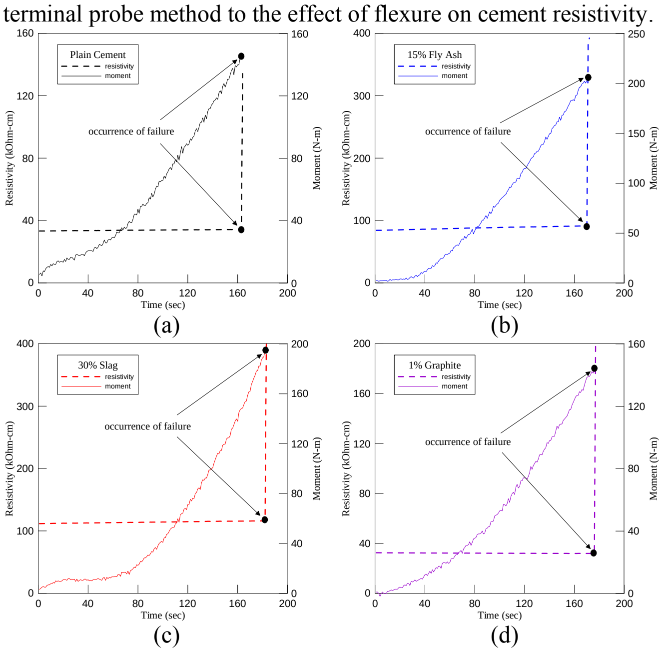


Figure 7 Effect of flexure on electrical resistivity of (a) plain cement, (b) 15% fly ash, (c) 30% slag, and (d) 1% graphite binders

4. CONCLUSION

Based on our investigations on electrical resistivity measurement of cement-based binders using embedded four-terminal probe method, the following conclusion can be made:

1. Replacement of cement by certain amount of fly ash and slag would significantly increase the binder resistivity. On the other hand, inclusion of conductive graphite particles would slightly reduce the binder resistivity.
2. Polarization effect of cement binders can be nullified by slag at the late age (28 days); while be amplified by fly ash at the early age (1 day). Graphite powder does not directly affect the polarization of cement binders.
3. The duration of polarization saturation reduced with the curing age; while also varied with material composition and specimen geometry. Fly ash and slag caused no significant effects on saturation time of cement binders.
4. Resistivity measurement using embedded four-terminal probe method can precisely capture the initiation of macro-crack of cement binders under compression. This can also be used to identify the flexural failure of cement binders.
5. There exists certain gage factors in between the percent change of electrical resistivity and compressive strain of cement binders. However, this relationship is not significant in the case of flexural strain.

5. REFERENCES

[1] Wen, S. and Chung, D. D. L., “Damage monitoring of cement paste by electrical resistance measurement,” Cement and Concrete Research, Vol. 30, 2000, pp.1979-1982

[2] Gowers, K.R. and Millard, S.G., “Measurement of concrete resistivity for assessment of corrosion severity of steel using Wenner Technique,” ACI Materials Journal, Vol. 96, No. 5, 1999, pp.536-542

[3] Millard, S.G., “Reinforced concrete resistivity measurement Techniques,” Proceedings, Institute of Civil Engineers, Part 2, 1991, pp.71-88

[4] Whiting, D. A., and Nagi, M. A., “Electrical resistivity of concrete - a literature review,” PCA R&D Serial No. 2457, Portland Cement Association, 2003

[5] Wenner, F., “A method of measuring earth resistivity,” Bulletin of the Bureau of Standards, Vol. 12, No. 4, 1916, pp. 469-478

[6] Hou, T. C. and Lynch J. P., “Conductivity-based strain monitoring and damage characterization of fiber reinforced cementitious structural components,” Proceedings of SPIE 12th Annual International Symposium on Smart Structures and Materials, 2005

[7] Chiarello, M. and Zinno, R., “Electrical conductivity of self-monitoring CFRC,” Cement and Concrete Composites, Vol. 27, 2005, pp.463-469

[8] Smits, F. M., “Measurement of sheet resistivities with the four-point probe,” The Bell System Technical, 1958, pp.711-718

[9] Ehtesham, S. and Rasheeduzzafar, H., “Corrosion resistance performance of fly ash blended cement concrete,” ACI Materials Journal, Vol. 91, No. 3, 1994, pp.264-271

[10] Hope, B.B. and Ip, A.K., “Corrosion of steel in concrete made with slag cement,” ACI Materials Journal, Vol. 84, No.6, 1987, pp.525-531

[11] Wen, S. and Chung, D. D. L., “Cement-based materials for stress sensing by dielectric measurement,” Cement and Concrete Research, vol. 32, 2002, pp.1429-1433

[12] Baweja, D., Roper, H., and Sirivivatnanon, V., “Corrosion of steel in marine concrete: long-term half-cell potential and resistivity Data,” Proceedings, Third ACI International Conference on Concrete in Marine Environment, SP-163, 1996, pp. 89-110

[13] Wen, S. and Chung, D. D. L., “Electric polarization in carbon fiber-reinforced cement,” Cement and Concrete Research, Vol. 31, 2001, pp.141-147