CARBON-FIBER REINFORCED CEMENT BASED SENSORS

by

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ABSTRACT

The addition of carbon fibers has proved to be one of the most effective ways of improving the electrical conductivity of ordinary cement pastes. Numerous studies have been conducted toward using this property for measurement of strain, temperature change and chloride penetration in concrete.

In the present study, a small carbon fiber reinforced cement specimen with rectangular cross section was positioned in a 60 Hz, \pm 10 V AC circuit with a data acquisition system in order to monitor the changes in its electrical resistivity under the influence of different parameters. The parameters studied included fiber content, water-cement ratio, moisture content, alkali concentration, temperature, compressive loading, and flexural loading. The influence of method of curing the specimen on its electrical resistivity over 28 days was also studied.

It was seen that a high fiber fraction and low moisture content makes the specimen act like a pure resistor with negligible capacitance or inductance associated with it. Air curing was preferred over moist curing because the increase in resistivity over time was lesser for air-cured specimens. It was also observed that electronic conduction was dominant over electrolytic conduction in a mix proportion with high fiber volume fraction and low water to cement ratio. The influence of temperature on the electrical resistivity of pastes with large amount of fiber was barely significant. The resistivity was found to steadily decrease under compressive loading and then increase during the formation of macro-cracks.

It has long been established that carbon fiber reinforced cement paste specimens have the ability to monitor its own state under various conditions by exhibiting a variation in its resistivity values. However no work has been carried out to show how such a specimen would behave if embedded in a concrete member. For this reason, the resistivity versus load behavior of such specimens embedded in concrete cylinders and beams under compressive and flexural loading were also studied. In flexure, resistivity of an embedded specimen showed a slight increase few minutes prior to the formation of the first crack in the beam. There was a steep increase in resistivity on further widening of the crack leading to believe that in flexural members this sensor may perform well as a

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crack sensor than as a strain sensor. It was observed that the sensor would more accurately mirror the strain in the concrete member it is attached to in its resistivity readings, if it is a resilient material with high yield strength and low modulus of elasticity.

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INTRODUCTION

Structural health monitoring (SHM) is the most modern development in the field of civil engineering. Innovations like fiber-reinforced polymers are being increasingly used to strengthen and repair civil infrastructures. But instead of repairing damages it is wiser to prevent these damages from happening and causing causalities. SHM involves constant monitoring of a structure's condition or change in its condition through the implementation of electrical and electronic systems like strain gauge, fiber optic sensors, transmitting devices, data acquisition systems, etc. These developments have led to the coinage of a new term – "civionics"[1] Civionics is to civil engineering what avionics is to aerospace engineering.

Commonly used corrosion detection devices in bridges and structures cost well over 1000 USD. Hence it is costly to install a large number of such systems in a structure. The need for an inexpensive method for monitoring infrastructure insitu as it responds to various stresses, temperature variation, changes in ionic concentration within the system, and vibrations have been the key factor in the development of self-monitoring materials. Self-monitoring materials can be considered as smart materials. However, in contrast to smart materials such as optical fibers, piezoelectric sensors, etc., self-monitoring materials are themselves structural materials [2]. A self-monitoring structure should be able to sense the changes in strain, stress, temperature and chloride content that take place within itself and respond in a particular manner without the need for embedded sensors. Such materials are also known as intrinsically smart structural materials [2]. The study of their responses helps understand the behavior of the structure and even foresee damages that might occur. Presently various fiber optic sensors and strain gauges are embedded in the structure to monitor these responses. But with intrinsically smart structural materials there are a number of advantages like [2]:

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- > low cost,
- > great durability,
- large sensing volume, and

> absence of mechanical property degradation due to the embedding of sensors.

Dry cement paste is a semiconductor or insulator with resistivity of the order 10⁴ohm-cm. Conductivity of a cement based specimen depends on the pore structure and the chemistry of the pore solution [3]. Introduction of carbon fibers into cement paste matrix significantly increases the electrical conductivity enabling one to monitor changes in resistivity under varying degrees of strain. There are also other factors that bring about variation in electrical resistivity of the paste like change in temperature, presence of chlorides, shrinkage, etc. Hence the measurement of variation in resistivity helps one to monitor the response of sensor embedded structures under these varying conditions.

The objective of this research was to study several factors that affect resistivity of a carbon fiber reinforced cement paste (CFRCP) specimen by subjecting it to different conditions that exists in field like temperature variation, chloride infiltration, and loading. The results were then examined to propose modifications to make a more effective sensor. Tests were also aimed at determining the best mix proportion for the sensor material. One major topic that this report focused on is the way in which such a sensor responded to indirect loading. For instance, three small CFRCP sensors were cast in a 10 cm diameter cylindrical concrete specimen along its longitudinal axis and the whole unit loaded to 75% of the ultimate strength of concrete. The effect of loading and distance from point of application of load were studied. Similarly a CFRCP specimen was cast into a beam and subjected to 4-point loading to study the effect of bending.

This thesis consists of 10 chapters. In Chapter 2, a review of the literature in the field of resistivity of carbon fiber reinforced cement specimens and concrete is given. In Chapter 3, the experimental program including the materials as well as the method used is described. The effect of various factors like temperature variation, presence of chloride ions and loading (direct and indirect), are discussed in detail in Chapters 4 to 8. In Chapter 9 the final results of the experiments are detailed and modifications are proposed. Chapter 10 suggests few recommendations for future research.

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LITERATURE REVIEW

2.1 CARBON FIBERS

Use of carbon fibers as reinforcement in concrete is gaining momentum because of the need for superior structural and functional performance. Carbon fiber reinforced mortar exhibits superior tensile strength, flexural strength, tensile ductility, flexural toughness, impact resistance, and freeze thaw durability [4]. Drying shrinkage is also reduced by the addition of fibers [4]. Short carbon fibers approximately 5mm in length are used more than continuous fibers because short fibers can be added directly into the concrete where as continuous one cannot be and short fibers are less costly than continuous ones [5]. However continuous fibers have an advantage over short fibers of providing better bond with the cement matrix because of their high aspect ratio. Since the interface bond between carbon fibers and cement matrix is crucial in determining the performance, surface treatment methods like using ozone, silane, SiO₂ particles or hot NaOH solution are often employed to overcome this drawback of short carbon fibers. Ozone and silane treatments improve the wettability by water because of the oxygen containing functional groups in the interfacial layer created by the treatment [6]. These treatments also increase the active specific surface area [5], the number of bonding sites on the fiber surface [7], and the surface roughness [7]. Admixtures such as latex, methylcellulose and silica fume also help the bond.

Increase in fiber volume fraction enhances the properties of concrete. However too great a fiber volume fraction can deteriorate the concrete properties because high fiber content produces large amount of air voids which in turn decreases the compressive strength of concrete. Hence a proper dispersing agent and addition of superplasticizer become necessary when using fiber volume fractions greater than 3%[8]. When compared with the conventional steel fiber composites with 0.3 - 1.0 diameter steel fibers, at equal fiber volume fractions, the carbon-cement composites have almost three orders of magnitude more fibers, with the number of fibers per square unit exceeding a few

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thousands [8]. Most of the studies conducted so far on the electrical resistivity of carbon fiber cement mortar have used an optimum fiber content of 0.2 vol. % [9],[10],[11].

To fully utilize the advantages that carbon fibers offers as a construction material it has to be properly dispersed. Using silica fume of about 15% by weight of cement assists dispersion. The reduction in porosity by the addition of silica fume also presents better chances of improving bonding [4]. Methylcellulose of about 0.4% by weight of cement is added along with silica fume to enhance dispersion as well as workability. Addition of methylcellulose produces foam and so a defoamer must also be used [4]. In general, the slump of carbon fiber reinforced cement tends to decrease with increasing carbon fiber content. Therefore, certain amount of water reducing agent should be added in order to maintain the mortar at a reasonable flow value.

Instead of methylcellulose, latex may be used as a dispersant. But due to its less effectiveness a large amount (20% of the weight of the cement) is required. This increases the cost [4]. In spite of its inefficacy as a dispersant, latex is known to improve the flexural strength, flexural toughness, impact resistance, frost resistance and acid resistance [12]. The fact that the ease of dispersion increases with decreasing fiber length is a major incentive for using shorter carbon fibers.

The advantages of adding carbon fibers cannot be restricted to these. Researchers have discovered that the properties of carbon fibers can be exploited to a much greater extent. These discoveries are believed to be a curtain raiser for a great breakthrough in civionics. The functional properties brought about by the addition of carbon fibers are [5]:

- 1. Strain sensing ability for smart structures.
- 2. Damage sensing ability.
- 3. Temperature sensing ability
- 4. Thermoelectric behavior.
- 5. Thermal insulation ability to save energy for buildings.
- 6. Vibration reduction [13].
- 7. Electrical conduction ability to facilitate cathodic protection of embedded steel and to provide electrical grounding or connection.

8. Radio wave reflection/absorption ability for electromagnetic interference or EMI shielding, for lateral guidance in automatic highways, and for television image transmission.

The dual characteristic of carbon fibers of being electrically conducting and having small diameter makes it the best choice for use in smart materials. Carbon fibers are conductive and more resistant to alkaline environment than glass or polymer fibers, which are non-conductors. Steel fibers are conductive but they have a large diameter ($\geq 60\mu m$) when compared with carbon fibers (15µm).

Although carbon fibers are thermally conductive, addition of carbon fibers decreases the thermal conductivity of concrete due of the formation of air voids [14]. The electrical conductivity of carbon fibers is higher than that of the cement matrix by about 8 orders of magnitude, whereas the thermal conductivity of carbon fibers is higher than that of the cement matrix by only one or two orders of magnitude [5]. As a result, the electrical conductivity is increased upon carbon fiber addition in spite of the increase in air void content, but the thermal conductivity is decreased upon fiber addition.

2.2 METHODS OF MEASURING ELECTRICAL RESISTIVITY OF CONCRETE

Any one of the three methods mentioned below can be used to measure the electrical resistivity of concrete.

2.2.1 Two-Probe Resistivity Measurement Technique

In this method a known D.C/A.C current (I) is applied between two electrodes embedded in concrete at a specific distance and the voltage (V) is measured or vice versa. The resistance (R) is determined by using Ohm's law:

$$R = V/I$$

The resistivity is obtained by multiplying the measured resistance by a conversion factor, called the cell constant (A/L):

$$\rho = R \frac{A}{L}$$

where A is the specimen cross-sectional area and L is the length of the specimen.

2.2.2 Four-Probe Resistivity Measurement Technique

This is one of the most commonly used methods for measuring electrical resistivity. This method was originally developed by Wenner (1916) to measure earth resistivity. This measurement setup consists of four electrodes, which are equally spaced, and a small alternating current is applied between the outer electrodes. The potential is then measured between the inner electrodes. The resistivity is determined using the following formula:

$$\rho = \frac{2.\pi.a.V}{I}$$

where,

 $\rho = \text{resistivity (ohm \cdot cm)}$

a = distance between inner electrodes (cm)

V = voltage (volts)

I = current (amperes)



Fig.2.1: Four-Probe Method

A conducting liquid is used to improve the contact between the concrete and the electrode tips. However the presence of steel rebars too near to the electrode tip might become a source of error. This is because rebars conduct current much better than concrete; they will disturb homogeneous current flow. While measuring with four electrodes over bars at 10 or 20 mm depth, errors can be made by as much as a factor of 2. So none of the measuring electrodes should be placed above rebars.

Even though electrical resistivity can be measured using the four-probe method, ohmmeter measurements are normally made with just a two-point measurement method. However, when measuring very low values of ohms, in the milli- or micro-ohm range, the two-point method is not satisfactory because test lead resistance becomes a significant source of error.

2.2.3 Disc Method

Disc (one electrode) method involves an electrode disc placed on the concrete surface over a rebar and measuring the resistance between the disc and the rebar. It requires a connection to the reinforcement cage and full steel continuity. The method is illustrated in Fig.2.2. The resistance can be converted to resistivity using a cell constant that depends on the cover depth, which varies over the surface and the rebar diameter. Precise calculation of the cell constant is not possible, because the exact current flow cannot be predicted. For maximum precision, the cell constant can be determined empirically using concrete slabs of known resistivity. Alternatively, the cell constant can be estimated. For usual cover depths, disc and bar diameters being 10 -50 mm, the cell constant is approximately 0.1 m. So the resistivity measured using a small disc electrode is approximately [15]:



Fig.2.2: Disc Method

2.3 PROBLEMS ASSOCIATED WITH DIRECT CURRENT MEASUREMENTS

Although direct current (D.C.) can be used for the measurement of electrical resistivity of concrete, its use is restricted due to the effects of polarization. Concrete conducts through the movement of ions in pore solution. When chemical reactions take place at the electrodes and hydrogen and oxygen gases are liberated and get deposited around the electrodes in the form of a thin film a polarization potential or back e.m.f. is created[16]. Current cannot be accurately measured because of these polarization effects.

However in most of the experiments conducted by Chung et. al. [2] [17] [10], direct current has been used because it is believed that polarization is appreciable only when the resistivity measurement is continuously performed over a long time (D.D.L. Chung, Composite Materials Research Laboratory, SUNY, Buffalo, NY, USA, .pers. comm.). If so, it can be corrected using the formula [16]:

$$I = \frac{V - V_p}{R}$$

where V = applied voltage

 V_p = polarization potential

It follows that if DC measurements are made, at least two different values of the applied voltage, V, have to be used to determine the two variables V_p and R.

AC provides both resistance and reactance information and AC is relevant to data acquisition by wireless methods. However AC measurement also requires more expensive electronics than DC measurement.

2.4 THE NEED FOR CEMENT BASED CARBON FIBER SENSORS

There are mainly four reasons for which cement based carbon fiber sensors are being developed. They are:

- 1. To keep a track of strains and stresses developed in a structure under the influence of dynamic and static loading.
- 2. To determine the temperature variation of a structure.
- 3. To determine the chloride content of the structure and hence the possibility of rebars to corrosion.

Many studies have been carried out to determine the response of carbon fiber reinforced cement-based composite to compressive and tensile forces, chloride content, and temperature variation. What is required is to compile these findings together with a deeper understanding of the mechanisms involved, develop a cement-based carbon fiber sensor, which can display the values of electrical resistivity of the concrete into which it is integrated. These values can then be calibrated to determine the stresses, strains, chloride content, and temperature of the structure at different locations enabling engineers to get first-hand knowledge of the behavior of a structure and act quickly in case of an emergency.

2.5 STRAIN MONITORING ABILITY

In one of the earliest studies of concrete as a self-monitoring material Chen and Chung [18] studied how electrical resistivity varies during crack generation or propagation. They observed that concrete's volume resistivity decreased during crack closure. On application of stress up to failure, the following observations were noted:

For mortar without fibers

- 1. It was observed that in plain mortars with no fibers the changes in volume resistivity were insignificant or negligible up to fracture.
- 2. The resistivity increased abruptly when the mortar fractured; this is because of the widespread cracking which accompanied fracture and the fact that the mortar was more conductive than air.

For mortar with fibers

- 1. All mortar with fiber showed an increase in volume resistivity on compression up to fracture.
- 2. It was seen that fiber-reinforced mortar containing latex, a polymer, exhibited a higher resistivity when compared with fiber-reinforced mortar containing either silica fume or methylcellulose. This was attributed to the insulating property of latex.
- 3. For all mortars with fibers, the resistivity decreased when the mortar fractured.

Next, on application of a cyclic compressive loading equal to 1/3 of the fracture stress, the following observations were made:

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For mortar without fibers

1. All mortars without fibers showed no change in the resistivity during the stress cycling.

For mortar with fibers

- 1. All mortars with fibers exhibited:
 - (i) Irreversibly increasing resistivity during the first loading The irreversibly increasing resistivity during the first loading is attributed to flaw generation.
 - (ii) Reversibly increasing resistivity during unloading in any cycle The reversibly increasing resistivity during unloading in any cycle is attributed to crack opening, which was hindered under compressive loading.
 - (iii) Reversibly decreasing resistivity during the second and subsequent loadings - The reversibly decreasing resistivity during the second and subsequent loadings is attributed to the crack closure under compressive loading.

2.6 DAMAGE SENSING ABILITY

If the mechanism of interface degradation is considered then in a composite material with carbon fibers resistivity increases during damage. However interface degradation occurs only during the first cycle of fatigue testing. Hence it is not possible to monitor resistivity variation in more advanced stages of damage. We notice a noticeable increase in resistivity when cracks are plentiful and damage occurs. This limits us to obtain results only in the very initial cycles of fatigue testing. Even during the first cycles, damage can be monitored only after the cracks are plentiful.

In experiment conducted by Chung [10] using self-monitoring material it was noticed that when damage occurs (probably in the form of micro cracks in the case of a brittle matrix such as cement) it increases the chance of adjacent fibers to touch each other hence decreases the electrical resistivity of the material. In this way it is possible to monitor the slight damage mechanics as long as up to 350 cycles. Testing was performed under cyclic loading (tensile or compressive) at stress amplitudes equal to 0.3 0.5 and 0.70 of the fracture stress. For compressive testing each cycle took 38.1 seconds and for tensile testing it took 52.2 seconds.

The following points were noted:

- 1. $\Delta R/R_o$ decreased during loading and increased during unloading.
- 2. $\Delta R/R_o$ had a baseline which monotonically decreased as cycling progressed.

This decrease is because of the adjacent fibers coming in contact with each other. The decrease was limited to a certain number of cycles only probably because the damage was stabilized. The number of cycles up to which $\Delta R/R_o$ decreased depended mainly on the stress amplitude. It occurred only in the early fatigue life, i.e., 5.8% of compressive fatigue life at stress amplitude of 0.07 of fracture stress, or 9.2% of tensile fatigue life at stress amplitude of 0.07 of fracture stress. The baseline decrease was not linear with cycle number. This decrease provides an indication of the extent of damage in the regime of slight damage.

 $\Delta R/R_o$ baseline increased slightly during few cycles prior to fracture providing an indication of the impending fracture. However due to the slightness of this increase the warning is not reliable. This slight increase is attributed to cracking. At fracture, $\Delta R/R_o$ greatly increased due to cracking. It can be concluded that damage results in an irreversible resistivity change.

MATERIALS AND METHODS

3.1 MATERIALS

3.1.1 Cement

The cement used was a Type 10 Normal Portland Cement (fineness of blaine 448 m^2/kg). The chemical composition of the cement is given in Table 3.1.

C3S (%)	C ₂ S (%)	C3A (%)	C ₄ AF (%)	CaO (%)	SiO ₂ (%)	Al ₃ O ₂ (%)	Fe ₂ O ₃ (%)	MgO (%)	SO3 (%)	Loss on ignition (%)	Alkalies (%)
63	13	6	11	65.4	21.1	4.44	3.68	0.9	2.7	1.61	0.38

 Table 3.1: Chemical Composition of Cement

3.1.2 Silica Fume

Densified silica fume with specific gravity 2.27 from Norchem, Inc. was used. The chemical composition of the silica fume used is presented in Table 3.2.

	SiO2 (%)	Al ₃ O ₂ (%)	TiO ₂ (%)	P2O5 (%)	Fe ₂ O ₃ (%)	CaO (%)	MgO (%)	Na2O (%)	K ₂ O (%)	SO3 (%)	Loss on ignition (%)
Average data	93.32	0.71	0.001	0.09	0.89	0.83	0.56	0.10	0.62	0.25	2.84

Table 3.2: Chemical Composition of Silica Fume

3.1.3 Carbon Fiber

Pitch-based carbon fibers from Conoco-Phillips, Inc. were used. The properties of the fiber are presented in Table 3.4.

Table 3.3: Properties of Carbon Fibers

Diameter (µm)	Length (average)	Tensile strength	Modulus of elasticity	Density (kg/m ³)
		(IVIFa)	(GPa)	
9-11	5	2100	232	1900

3.1.4 Superplasticizer

The high range water reducer used was a polycarboxylate-based superplasticizer with specific gravity 1.08 (Glenium 3000 NS, Master Builders).

3.1.5 Mix Proportions

Mix proportions given in Table 3.4 were used for making specimens for different sets of experiments. Silica fume added at a relatively high dosage in all mixes acted as a dispersant as well as a densifier. Superplasticizer was added to obtain the desired workability.

Water/cementitious ratio, by weight	Silica fume/cement ratio, by weight	Carbon fiber (vol. %)
0.3		0
0.3		1
0.3	0.2	3
0.3		5
0.4		5
0.5		5

Table 3.4: Mix Proportions

3.2 METHODS

3.2.1 Mixing Procedure and Specimen Preparation

The mixing was performed using a Hobart mixer (4.5 L) and the following mixing sequence was employed.

- 1. The superplasticizer was added to the water.
- 2. The silica fume and cement was mixed thoroughly in the mixer.
- 3. The fibers were gradually introduced into the silica fume cement mix.
- 4. Once ³/₄ of the fibers were added, water was slowly introduced and the mix was allowed to form into slurry.
- 5. A small amount of water was kept aside and the left over amount of fiber was added.
- 6. Finally after making sure that the constituents have mixed well the remaining amount of water added and mixed once again for few minutes.



Fig.3.1.a: Specimen for Resistivity Measurements with 30 mm Inter-Electrode Spacing



Fig.3.1.b: Specimen for Resistivity Measurements with 70 mm Inter-Electrode Spacing

Prismatic specimens were cast in two sizes with dimensions $15 \times 25 \times 50$ mm and $15 \times 25 \times 110$ mm. Two copper electrodes of thickness 0.35 mm and width 10 mm were inserted through the entire depth of the specimen as shown in Fig.3.1.a and Fig. 3.1.b. The inter-electrode spacing was 30 and 70 mm for the 50 mm and 110 mm length specimens, respectively. For each experiment a set of four specimens were cast for each dimension and the graphs were plotted for the average of their results. Twenty-four hours after casting, the specimens were carefully demoulded and electrical measurements were begun. The measurements were made up to an age of about 28 days. After each measurement, the specimens were immediately transferred to the curing room maintained at $23\pm2^{\circ}$ C and 75% relative humidity.

3.2.2 Measurement Technique

The two-probe resistivity measurement technique was adopted. Electrical resistivity measurements were made by applying a known AC voltage of $\pm 10V$ across the electrodes using a function generator at a frequency of 60 Hz. This frequency was selected because it was observed that at higher values there was a great fluctuation in the resistivity reading, owing to the incongruence in the frequencies of the excitation signal and sampling rate of the system. The reason for selecting an AC voltage has been cited earlier in Chapter 2. Due to the high percentage of fiber volume present in the specimen it was seen that the specimen behaved more like a pure resistor. Fig.3.2 and Fig.3.3 show the setup used for resistivity measurement.

A known resistance of 100 ohms and the specimen are connected in series in the circuit (Fig.3.2). Since the current flowing through the entire circuit is constant, based on Ohm's law the following equation holds true:

$$\frac{V_R}{R} = \frac{V_X}{X}$$

where,

 V_R = voltage across known resistor R,

R = known resistor = 100 ohms,

 V_X = voltage across unknown resistor, and

X = unknown resistor.

The value of X is thus calculated by the software DASY Lab 8.0 and the resistances versus time values are plotted in the display unit. Resistivity ρ is calculated as follows:

$$\rho = X \frac{A}{L}$$
 Eq. 3.1

where,

X = resistance of specimen, now known

A = cross-sectional area of specimen

L = gauge length = inter electrode spacing



Fig.3.2: Simplified Circuit Diagram for Resistivity Measurement



Fig.3.3: Setup for Resistivity Measurement

An inter-electrode spacing of 70 mm is known to produce almost constant values of resistivity [16]. However resistivity reading was also taken for a 30 mm inter-electrode spacing for comparison. The specimens are identified as 70 mm and 30 mm which actually refers to its inter electrode spacing.

INTERNAL FACTORS AFFECTING ELECTRICAL RESISTIVITY OF CARBON FIBER REINFORCED CEMENT PASTE

All systems in this universe are in a state of entropy. There are always elements at work that bring about changes in a system. In a system like a carbon fiber reinforced cement paste (CFRCP) specimen, there are various factors that play a critical role in changing its resistivity. This chapter enumerates the internal factors that affect the electrical resistivity of such a system.

There are basically two mechanisms of electrical conduction in moist specimens: electronic and electrolytic [19]. Electronic conduction is through the motion of free electrons in the conductive phases, e.g. carbon fibers, and electrolytic conduction is through the motion of ions in the pore solution. The conductivity of CFRCP can be attributed to mainly three media: carbon fibers, the pore solution within the matrix and the physical interface between the fibers and matrix.

Carbon fibers are known to reduce the electrical resistivity of cementitious composites. The conductivity of the mix is directly proportional to the volume fraction of carbon fibers and can be visualized as shown in Fig.4.1. The plot of resistivity vs. time (Fig.4.2 and Fig.4.3) for mixes with different fiber fraction volume further asserts this fact.



Fig.4.1: Percolation Theory[20]

The conductivity in a CFRPC system follows the phenomena of percolation theory. Percolation theory is basically a geometrical theory that describes the structure of random particles or filaments in a matrix as a function of their volume fraction [20]. It postulates that it is only when the volume fraction of the particle or filament exceeds a certain critical value that the particle or filament can come into contact and form clusters. As a result, electrical conduction can occur due to the connection of the clusters. Percolation threshold thus is the critical fraction of lattice points that must be filled in order to create a continuous path of nearest neighbors from one side to another. Thus the electrical conductivity of the specimen also largely depends on the fiber's aspect ratio and material.

A higher volume fraction ensures that there exists more fiber-to-fiber contact thus allowing easy passage of current. Moreover a high volume percentage and low w/c ratio makes the specimen act like a pure resistor with negligible capacitance and inductance values.



Fig.4.2: Resistivity as a Function of Percentage of Fiber Volume for 30 mm Specimens



Fig.4.3: Resistivity as a Function of Percentage of Fiber Volume for 70 mm Specimens

The influence of w/c ratio on the resistivity of cement paste is quite significant as can be seen from the graph in Fig.4.4 and Fig.4.5. On comparing different combinations of fiber percentage and w/c ratio, it can be seen that the set with 5% volume of carbon fiber and 0.3 w/c ratio offers the least resistance. The combination of 5% fiber volume and 0.5 w/c ratio has lesser resistivity than 5% fiber volume and 0.4 w/c ratio. This can be attributed to the large amount of pore solution available in the capillary pores of the mix with 0.5 w/c ratio. However the combination of 5% fiber volume and 0.3 w/c ratio has a lesser resistivity value than the one with 0.4 w/c ratio and is lower than even the mix with 0.5 w/c ratio. This is because the former most has larger amount of fines and lesser pores with respect to both the latter ones thus allowing denser packing and more fiber-to-fiber contact. Moreover the large amount of superplasticizer that was added to obtain the desired workability can bring about finer packing of different phases. A lower w/c ratio can also assist in better contact between the electrode and matrix. It can be concluded that on decreasing the w/c ratio from 0.5 to 0.3 the conduction transfers from electrolytic to electronic and fiber-to-fiber contact starts playing a prominent role.



Fig.4.4: Resistivity as a Function of w/c Ratio for 30 mm Specimens



Fig.4.5: Resistivity as a Function of w/c Ratio for 70 mm Specimens

An interesting observation was made during the curing phase. On comparing the resistivity of two sets of specimen, one moist cured and the other air cured, it was noticed that the increase in resistivity over time was higher for the moist cured set, (Fig.4.6). This is quite contrary to that of plain concrete where the increase in resistivity with time is greater during air curing [21]. The increase in resistivity during moist curing in concrete is primarily due to the increased hydration of cement and filling of pores with hydration products, while for air curing resistivity increases due to loss of moisture from the concrete[21].

Few explanations that can be given for the contrary behavior of carbon fiber reinforced cement paste are:

- 1. The moist samples are more susceptible to the effects of capacitance and inductance than air-dried samples thus giving readings, which are not purely resistance readings.
- 2. There could be some effect of corrosion on the copper electrode over the days although none was visible to the naked eye.
- 3. Water, which is a bad conductor of electricity may have an adverse effect on the resistivity values when present in large amounts in a sample.



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EXTERNAL FACTORS AFFECTING ELECTRICAL RESISTIVITY OF CARBON FIBER REINFORCED CEMENT PASTE

Few of the external factors that affect the resistivity of a CFRCP specimen are temperature variation, presence of chloride ions and loading. This chapter discusses the effect of temperature variation and chloride ions on the variation in resistivity. The effects of compressive and flexural loading will be discussed in later chapters.

Brameshuber et. al [22] summarizes the phenomenon of electrical conduction in cement pastes. Change in resistivity during temperature variation and chloride penetration is a result of the electrolytic conduction of cement paste. The electrolytic conductivity of cement paste essentially depends on the chemical composition of the pore solution and the pore structure (paste composition, compaction and curing) as well as the paste moisture and temperature. Figures 5.1.a and 5.1.b show the ionic conduction in the hardened cement paste.



Fig.5.1.a: Capillary Pore Distribution of Hardened Cement Paste in Current-Free State G: gel, C: continuous capillary, D: dead end or closed capillary[22]



Fig.5.1.b: Capillary Pore Distribution of Hardened Cement Paste Under the Effect of an Applied Electrical Field [22]

The hardened cement paste can be classified into cement gel, open pores (e. g. capillary pores), closed pores and dead end pores. The pore solution contains both positive and negative ions (essentially Na⁺, K⁺, Mg⁺⁺, Ca⁺⁺, OH⁻, Cl⁻, SO₄⁻) that are distributed evenly within the hardened cement paste without being affected by external currents. The positive ions (cations) move to the cathode and the negative ions (anions) to the anode when an electrical field is applied via the two electrodes. The charge carriers in the closed pores or dead ends are blocked and act as condensers without affecting the ohmic resistance of the paste [22].

In a water-saturated system, electrolytic conduction generally takes place via the liquid phase of the pore solution of the cement paste. In semi-moist systems the ionic conduction takes place via the monomolecular water film adsorbed on the pore walls. The system acts as an insulator when it becomes very dry .i.e. the conductivity drops to lower values.

5.1 TEMPERATURE VARIATION

Ion migration in electrolytes is a frictional movement in a medium. The internal friction (viscosity) decreases with an increasing temperature, and thus the resistivity too. Consequently there is an increase in the resistivity when temperature decreases due to the restricted movement of the ions.

In the experiment conducted in this study both 30 mm and 70 mm specimens were subjected to a temperature change ranging from -20° C to 65°C. Care was taken to

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Fig.5.2: Variation of Resistivity with Temperature for 30 mm Specimens



Fig.5.3: Variation of Resistivity with Temperature for 70 mm Specimens

prevent loss of moisture from the samples. An entire cycle of temperature variation was carried out, starting at room temperature, increasing it to 65°C, then decreasing it to -20°C and finally bringing it back to the room temperature.

The specimens were maintained for a period of 2 hours at each temperature. It was seen that as the temperature increased, the resistivity decreased and vice versa. The initial and final resistivities at room temperature were not the same indicating a 'drift'. There was a 23.5% increase in resistivity in the 30 mm sample and a 2.3% decrease in resistivity in the 70 mm sample. This contradictory behavior could not be explained, however smaller difference in the initial and final resistivity values in the 70 mm sample indicates that a greater inter-electrode spacing is more suitable in such measurements. There are also chances that the resistivity values did not indicate the exact value corresponding to a given temperature because the core temperature might have been different from the surface temperature.

In general, even though the resistivity showed variation with change in temperature the change was not very conspicuous. A reason for this could be the high fiber volume fraction (5%), increasing the fiber-to-fiber contact and low w/c ratio thus causing the electronic conduction to take precedence over the electrolytic conduction.

If this sensor were to be implemented in the field it is also necessary to record the ambient field temperature and relative humidity, so that the resistivity changes due to these factors can be compensated and any further changes in resistivity can be interpreted as being caused by changes in stress level [23].

5.2 PRESENCE OF CHLORIDE IONS

Ingression of chloride ions increases the alkalinity of mortar thus making concrete highly susceptible to corrosion. In order to assess the effect of chloride ions on the electrical resistivity of CFRCP sample, two sets of mixes were prepared. One set was prepared using plain water and the other was prepared using 0.55 M NaCl solution i.e. 32.175 gms NaCl/L of water added.

Na⁺	+	Cl	\rightarrow	NaCl
23 gms		35.5 gms		58.5 gms

If 35.5 gms Cl⁻ is present in 58.5 gms NaCl solution then there is 19.525 gms Cl⁻in 32.175 gms NaCl/L of water added. The variation in resistivity over 28 days of air curing was plotted as shown in Fig. 5.4. A decrease in the resistivity was observed in samples with NaCl solution. Table 5.1 shows a comparison between the resistivity values at 28 days .

	Resistivity at 28 days (ohm-cm)	Percentage difference (%)
30 mm sample w/o NaCl	69	52
30 mm sample with NaCl	33	52
70 mm sample w/o NaCl	46	26
70 mm sample with NaCl	34	

Table 5.1: Comparison of Resistivity Values for Specimens with and without NaCl

Once more, it was seen that the variation in resistivity was lesser for the 70 mm interelectrode spacing.



Fig.5.4: Variation of Resistivity in the Presence of Chloride Ions

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COMPRESSIVE LOADING

Concrete depicts a brittle behavior in tension. Addition of fibers has known to increase the tensile capacity of concrete. However the maximum strain achieved prior to failure is very low when compared with strain reached under compressive forces. If one is to use a CFRCP as a sensor, then loading plays the pivotal role in its working mechanism. The reaction to loading of such materials has been discussed in detail by Chung [2], Fu[10, 11], Wang [11]. However, in this chapter the load versus resistivity graph has been discussed once again as a preface for the following chapters.

Fig.6.2 and Fig.6.3 show the resistivity versus load response over time of the specimens with 30 mm and 70 mm inter-electrode spacing in compression. A diagram of the experimental setup is shown in Fig. 6.1







Fig.6.2: Variation of Resistivity Under Compressive Load for 30 mm Specimens

The load was applied till failure at a rate of 0.1 kN/sec. Initially as load increases there was a steady fall in the resistivity value. This can be attributed to the increase in contact between carbon fibers by the closing of microcracks and damage as well as due to a decrease in the distance between the matrix and the copper plates. Then there was a relatively flat portion, which indicated that the closing of original micro cracks and opening of new ones reached a dynamic balance[24]. Gradually as the load increased it was seen that the resistivity too started increasing. This increase in the resistivity was a clear indication of formation of micro cracks within the matrix, which created distance between the fibers and weakened the fiber-matrix interface. Finally as the load reached the full capacity of the specimen there was a sudden ascend in the resistivity value. This can be attributed to the complete failure of the matrix thus resulting in air pockets within the matrix. The resistivity of air being higher than that of the matrix, increases the resistivity of the specimen.



Fig.6.3: Variation of Resistivity Under Compressive Load for 70 mm Specimens

However when used as a strain sensor the resistivity changes before the formation of macro cracks are relevant or in other words the resistivity changes that are reversible on load removal are the values that can really tell what is happening in the structure. During the experiment it was noted that the initial resistivity changes only in the range of 5 - 10 % of ultimate load was irreversible. The resistivity changes during load values higher than 10 % of maximum load and up to 45 % of maximum load were reversible to a great extent. This contradicts with the findings of Chen and Chung [18] where they noticed an irreversible change in the resistivity values in the initial phase of loading due to flaw generation and then a reversible decrease in the resistivity values till larger cracks were formed. The reason for this could be the low resistivity of the specimen itself, which makes the resistance changes too small to be conveniently detected. Figures 6.4 and 6.5 compare the variation of resistivity versus strain and stress versus strain in the 30 mm and 70 mm samples respectively.



Fig.6.4: Comparison of Resistivity vs. Strain and Stress vs. Strain for 30 mm Specimens



Fig.6.5: Comparison of Resistivity vs. Strain and Stress vs. Strain for 70 mm Specimens

In the stress vs. strain plot it can be seen that the slope of the resistivity in the elastic range in greater than the slope in the strain hardening and softening regions. Thus indicating that such a sensor can more efficiently monitor strains in the elastic range of compressive stress application than in the post crack period.

RESPONSE OF EMBEDDED SENSOR TO CYCLIC LOADING

If a CFRC sensor were to be used in the field it is assumed that it will never be under the direct application of load. In fact, it will show resistivity changes depending on the strain it suffers through the transfer of load from the structural member in to which it is integrated. In order to study the response of the sensor towards indirect loading three 30 mm specimens were cast along the longitudinal axis of a concrete cylinder with a diameter of 10 cm and height of 20 cm as shown in Fig 7.1. The sensors were cured for 28 days prior to placing them inside the cylinders. The cylinders were then further cured for 28 days. The cylinders were subjected to cyclic loading. The load during each cycle was gradually increased to 75 % of the ultimate capacity of the cylinder (40 MPa).





Resistance readings were taken across each sensor during loading. R_U , R_M and R_L represent the resistance across the upper, middle and lower sensors. Resistivity was calculated using Eq. 3.1. Three such cylinders were tested and the average value of variation in resistivity with load of each sensor was plotted. Fig 7.2 shows the variation in resistivity in each sensor with change in load. The behavior of the specimen under

indirect cyclic loading was found to be similar to that under direct compressive loading. Resistivity decreased in the initial phase till it reached a relatively flat phase after which it increased. Mostly the upper-most sensor displayed this characteristic behavior. The middle one was slower in displaying this trait and by the time the cylinder was loaded to 75% of the ultimate load, the resistivity hadn't changed. The lowest one lagged behind the most. Even though it did not show an appreciable decrease in resistivity when the resistivity of the uppermost sensor was at its lowest, it definitively exhibited an increase in the resistivity towards the end, indicating that micro cracks were beginning to form at higher load values. It was assumed that there exists a perfect bond between the CFRCP sensor and the concrete matrix. And so it was expected that the upper and lower specimens would strain to the same extend and hence their resistivity curves would also be similar. However, the variation in resistivity decreased with increase in distance from top. This was observed in all the samples tested. From Fig. 7.2 it was seen that the change in resistivity varied in the following manner:

Rate of change in resistivity in upper specimen > rate of change in resistivity in middle specimen > rate of change in resistivity in lower specimen.



Fig. 7.2: Variation of Resistivity Under Cyclic Loading

It is also likely that the area of contact between the carbon fiber specimen and concrete created a weak interface in the matrix. Thus there was not sufficient bond strength between the sensor and the concrete to allow transfer of shear stress, which would have led to an equivalent amount of straining in the top and bottom specimens. The specimens were mainly affected by the stress transferred from above through the column of concrete lying directly above it. Hence most of the straining was brought about by the compressive stress rather than the shear stress. The lowermost specimen showed an increase in resistivity towards the end of the loading phase indicating that the increasing compressive force was causing it to fail.

The results of this experiment also indicate that if the senor is placed far from the point of application of load, unless there is a perfect bond between the sensor and concrete matrix, considerable amount of straining is required before it can show changes in the resistivity reading. This may also be an artifact related to specimen preparation where there is a possibility that the sensors might have angled off from the longitudinal axis at the time of vibration.

RESPONSE OF EMBEDDED SENSOR TO FLEXURAL LOADING

To further study the influence of loading, a sensor was embedded in a polypropylene fiber reinforced concrete (PFRC) beam with dimensions 10 cm x 10 cm x 35 cm. The fiber used in these beams was a self-fibrillating fiber with the following particulars, volume fraction: 1%, fiber length: 54 mm, aspect ratio, l/d: 360, and density: 900kg/m³. The resistivity of the fiber reinforced concrete was found to be 809 ohm-cm at an age of 28 days. Prior to this the same experiment was tried with a plain concrete beam. However owing to the very brittle nature of concrete when the crack formed in the beam it traveled across the sensor breaking it into two. On using fiber reinforced concrete the progress of crack into the depth of the beam was delayed and hence it was possible to subject the specimen to flexural bending for a longer period of time. The variation in resistivity was more clearly visible as shown in Fig. 8.2. The sensor was embedded 2 cm below the face of casting. The beam was subjected to four-point loading and the sensor was placed on the downside as shown in Fig. 8.1.



Fig.8.1: Setup for Flexural Loading

On comparing the compressive and flexural responses of the specimen it was seen that in flexure there was no decrease in resistivity in the initial phase like in compression. Few minutes prior to the first formation of crack in the beam there was a gradual increase in the resistivity as shown in Fig. 8.2. This indicated a transfer of load from the matrix of the beam to the sensor. Accompanying the sudden decrease in load during the formation of the first crack in the beam there was a sudden increase in the resistivity value by approximately 52% of the initial value but lesser than the resistivity of the PFRC beam (809 ohm-cm). Thus indicating that the crack propagated to the surface of the sensor but was arrested by the fibers surrounding it thus only creating micro cracks with in the matrix of the fiber specimen.

If the specimen had split into two then the percentage increase in resistivity would have been much greater as was observed in the experiment with plain concrete. As the crack in the beam widened, the resistivity increased steeply indicating further straining of the sensor. Finally there was again a sudden increase in resistivity reaching a value that was almost equal to the resistivity of the fiber reinforced concrete, which indicated that the CFRCP specimen had partially damaged.

The bending stress in the PFRC beam was calculated assuming the beam to be a homogeneous material and that the stress across the cross-section of the beam is constant. The bending stress versus deflection and resistivity versus deflection plot in Fig. 8.3 gives a better picture of what happens during the formation of crack in the beam. It can be seen in Fig. 8.3 that at the formation of the first crack there is a sudden decrease in load. Corresponding to this decrease is a relatively flat portion in the resistivity graph. This is very similar to the behavior of the sensor under compressive loading where there was a flattening of the resistivity graph prior to the formation of the micro cracks. This flat region was earlier explained to correspond to a phase in which a dynamic equilibrium is reached between the closing of old cracks and opening of new. It is at the end of this flat region that the sensor starts getting considerably damaged.

In a practical application, the steel reinforcement would act as a crack arrestor. Due to the fact that it showed a considerable variation in resistivity in the post crack period such a sensor can be more effective as a crack sensor rather than a strain-sensor under flexural loading.



Fig.8.2: Variation of Resistivity Under Flexural Loading



Fig.8.3: Comparison of Resistivity vs. Deflection and Bending Stress vs. Deflection Under Flexural Loading

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CONCLUSIONS

This study was aimed at laying the groundwork for the possible implementation of carbon fiber reinforced cement paste specimen as a sensor to monitor strain, temperature variation and chloride penetration.

It was found that the electrical resistivity of both plain cement pastes and carbon fiber reinforced paste when continuously cured, increased over time. This can be attributed to the overall change in the microstructure of the paste due to hydration. An increase in fiber volume fraction at constant w/c ratio decreased the electrical resistivity significantly.

A mix proportion with w/c ratio of 0.3 and a fiber volume fraction of 5% had lower electrical resistivity than specimens with higher w/c ratio and lower fiber volume fractions. It was found that w/c ratio did not significantly affect the electrical resistivity of samples with higher fiber volume fraction. It could be because the carbon fibers provided the primary conduction path as opposed electrolytic conduction through poresolution in connected capillary pores. It is possible to take advantage of this large fiberto-fiber contact for solely monitoring strain since the effect of moisture and temperature on resistivity is relatively insignificant. While using such a sensor in the field it is recommended to coat the specimen with a thin layer of water resistant material to prevent an unnecessary increase in the moisture content in the sensor.

It was seen that the electrical resistivity decreased with increase in temperature and vise versa. It is believed that at higher temperatures, the viscosity of the pore solution decreases thus enabling easier movement of ions. There was a considerable decrease in the electrical resistivity in the presence of chloride ions. The net change in resistivity when subjected to temperature change and due to the presence of chloride ions was lower for sensors with a 70 mm electrode spacing than for sensor with 30 mm electrode spacing.

On direct compressive loading it was seen that during the elastic range resistivity decreased continuously. This clearly indicated that the fibers were coming in contact with

each other. The resistivity then flattened and continued in this phase till the formation of the first visible crack after which it increased suddenly. On an average, the resistivity changes between 10% and 45% of the ultimate load capacity of the specimen were found to be reversible. Since the slope of the resistivity was always the steepest in the elastic region during compression, it can be said that the sensor works most effectively in the elastic region. In the load vs. resistivity experiment, the 70 mm electrode spacing samples showed more reproducibility in results as compared to the 30 mm electrode spacing samples.

In the experiment where the sensors were embedded in a cylindrical concrete specimen the weak interface between the concrete surface and the carbon fiber specimens played a detrimental role in the transfer of stresses. Most of the straining in the specimens seems to have been due to the compressive force transferred through the column of concrete lying directly above it. The changes in resistivity seem to be highly dependent on the distance from point of load application, which is not appreciable in a strain sensor. Hence it is necessary to increase the sensitivity of the sensor to stress and improve its bond with concrete.

Under the influence of flexural load, the sensor showed a very gradual change in resistivity few minutes prior to the time the beam reached its peak load. On formation of the first crack and on its widening, there was a significant increase in the resistivity. Hence the specimen can be made to act as a good crack sensor if the transfer of stress from the structural member to the carbon fiber specimen is not abrupt.

RECOMMENDATIONS FOR FUTURE RESEARCH

There is a wide range of potential that can be tapped in the field of sensing using carbon fiber reinforced cement based sensors. The biggest advantage of this system is its low cost and straightforward mechanism. The development of such a low-cost sensor can significantly increase the number of structures being monitored through out the world. However a lot of work has to be done in creating a stable sensor out of this system.

During the experiments it was observed that sensors made from the same mix showed significant variation in resistivity. Such a behavior is unacceptable in any commercial application. One has to be extremely careful when making the mix and be attentive that the fibers disperse uniformly throughout the mix. The contact between electrodes and the matrix was of major concern. This problem was finally resolved by creating a rough surface on the electrode for better bonding.

Another major issue is, since these sensors have been developed for multi purpose sensing, whether it is possible to differentiate between the changes in resistivity that each external factor brings about. One method would be to calibrate the resistivity in these sensors for particular values of strain, chloride content and temperature. However this is not an easy task because the physical and chemical property of the sensor itself changes with time. Since it is the process of hydration that causes the chemical reactions in the sensor amount of cement used has to be kept minimal. Developing a more stable medium in which the fibers are dispersed would be of great advantage.

Based on the observations made during this research it is highly recommended that the thickness of the sensor be kept to a minimum. Decreasing the thickness can help in improving the sensitivity of the CFRCP specimen. However the brittle nature of carbon fiber poses as a limitation in achieving a very thin sensor. Efforts should be addressed to the development of a resilient material with high yield strength and a low modulus of elasticity if the sensor has to show changes in its resistivity value that mirrors the strain in the concrete member in to which it is integrated. The effect of steel rebar on the resistivity readings in such a sensor is not known. Experiments that can validate the usefulness of these sensors in steel reinforced structural element can make researchers and investors sit up and take notice.

The results of these experiments are based on work conducted in laboratory environment. Hence future work is required where these sensors are made to function under a combination of different environmental factors that bring about changes in resistivity.

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