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Carbon Nanofiber Concrete for Damage Detection of Infrastructure

Y.L. Mo and Rachel Howser Roberts

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1. Introduction

Fiber research in concrete construction is an ongoing field and the use of carbon nanofibers (CNF) is examined. Fibers improve brittle materials such as concrete by enhancing tensile strength, ductility, toughness, and conductivity. Short-fiber composites are a class of strain sensor based on the concept of short electrically conducting fiber pull-out that accompanies slight and reversible crack opening. For a fiber composite to have strain sensing ability, the fibers must be more conducting than the matrix in which they are embedded, of diameter smaller than the crack length, and well dispersed. Their orientations can be random, and they do not have to touch one another. The electrical conductivity of the fibers enables the direct current (DC) electrical resistivity of the composites to change in response to strain change or temperature, allowing sensing.

2. Nanotechnology in concrete

Despite the fact that nanotechnology is a relatively recent development in scientific research, the introduction of the concept is credited to Nobel Prize winner Richard Feynman from his 1959 lecture, "There's Plenty of Room at the Bottom" [1]. Feynman considered the possibility of direct manipulation of individual atoms as a powerful form of synthetic chemistry. Decades later, Feynman's concept morphed into the field of nanotechnology. According to the National Science Foundation and National Nanotechnology Initiative, the definition of nanotechnology includes three elements [2]:

- The size range of the material structures under consideration should be approximately 100 nanometers;

- The nanotechnology should have the ability to measure or transform at the nanoscale;
- There should be properties that are specific to the nanoscale as compared to the macro or micro scale.

Following this definition, in the past 25 years nanotechnology has expanded from Feynman's idea and now finds applications in fields ranging from medical devices to nano-reinforced concrete [3, 4].

To date, the awareness and application of nanotechnology in the construction industry are increasing; however, progress is uneven in the current early stages of its practical exploitation. Bartos [5] presents three reasons for this phenomenon:

- The nature of the construction industry differs greatly from the other industries doing research in nanotechnology. The final products coming from the construction industry are not mass-produced and require relatively long service lives, differentiating it from the products from the microelectronics, information technology, and automotive industries.
- Historically, there is a very low level of investment in construction research and development.
- Research in nano-related research and development requires very high initial capital investment

Despite these difficulties, there have been significant advances in nanoscience of cementitious materials with an increase in the understanding of basic phenomena in cement at the nanoscale. These include structure and mechanical properties of the hydrate phases, origins of cement cohesion, cement hydration, interfaces in concrete, and mechanisms of degradation [6]. A major nanotechnology application is to include nano-sized reinforcement in cement-based materials such as carbon nanotubes or nanofibers.

3. Fiber reinforced concrete

Concrete, composed of fine and coarse aggregates held together by a hydrated cement binder, is one of the most important construction materials and is used in diverse project areas including house foundations, high rise tower components, highways, and dams. Hydrated cement is a brittle material that is an order of magnitude stronger in compression than in tension. To compensate for this weakness reinforcement consisting typically of rebar or fibers are added to the concrete.

The use of fibers to reinforce brittle materials can be traced back to ancient times when straw and hair was added to mud bricks. The modern development of the use of fibers in construction began in the 1960s with the addition of steel fibers to reinforced concrete structures. This was closely followed by the addition of polymeric fibers, glass fibers, and carbon fibers in the 1970s, 80s, and 90s, respectively [7].

Fibers improve brittle materials such as concrete by enhancing tensile strength, ductility, toughness, and conductivity [8-13]. Fibers are typically used in two forms: short randomly

dispersed fibers in a cementitious matrix or a continuous mesh of fibers used in thin sheets. Here we will focus on randomly dispersed fibers used to arrest cracks. The cracking process within concrete begins with the onset of isolated nanocracks. These nanocracks grow together to form localized microcracks, which in turn grow together to form macrocracks. These macrocracks widen to form cracks visible with the naked eye. Fibers arrest these cracks by forming bridges across them. With increasing tensile stress, a bond failure eventually occurs, and the fiber will pull out of the concrete allowing the crack to widen. Fig. 1 shows the bridging action of fibers across micro and macrocracks in concrete.

Figure 1. Bridging Action of Fibers Across Micro and Macrocracks

4. Nanoreinforcement in cement-based materials

Since the discovery of carbon nanotubes (CNT) in 1991 [14], researchers have desired to implement the unique mechanical, thermal, and electronic properties of CNT and CNF in cement-based composites. Single-wall CNT (SWCNT), multi-wall CNT (MWCNT) and CNF are graphene ring-based materials with aspect ratios greater than 1000 and high surface areas [6, 15]. CNT and CNF have moduli of elasticity in the range of terrapascals and tensile strength on the order of gigapascal [6, 16, 17]. SWCNT consist of a single graphene sheet wrapped into a seamless cylinder, while, as the name suggests, MWCNT inhere of multiple concentric sheets of graphene wrapped around a hollow core. CNF are cylindric nanostructures with graphene layers arranged as stacked cones, cups, or plates. CNF are adventagious because their stacked structure presents exposed edge planes not present in CNT that intoduce increased surface area and better bond characteristics. Because of their structure, CNF are easier to produce and cost 100 times less than SWCNT [18]. Because of the increased bond surface and lower cost, CNF are more attactive than CNT for application in cement-based composites.

5. CNT and CNF dispersion

The majority of nanoreinforced composite research has been completed on polymers containing CNT or CNF [6, 19, 20]. One of the main reasons for this is because uniform dispersion is difficult in cement-based materials. Well dispersed CNF results in uniform calcium-silicate-hydrate (CSH) gel formation, which improves the structural and electrical properties of the concrete [21]. CNT and CNF are inherently hydrophobic and are attracted due to Van der Waals forces, causing the fibers to tend to agglomerate hindering their dispersion in solvents [17, 22-24].

Several solutions have been proposed to solve this issue including dispersing the fibers through milling, ultrasonication, high shear flow, elongational flow, functionalization, in addition to surfactant and chemical disperment systems [24]. These methods primarily fall into two categories: mechanical and chemical dispersion. The mechanical dispersion methods, such as ultrasonication, while effective in separating the fibers, can fracture them decreasing their aspect ratio. Chemical methods use surfactants or functionalization to make the fibers less hydrophobic, reducing their tendency to agglomerate. However, many of the chemicals used can digest the fibers causing the fibers to become less effective. The surfactants also often cause bubbles to form in the composite negatively effecting the strength of the material.

Gao et al [12] proposed a dispersion method specifically used for CNF/CNT dispersion in cement-based materials that eliminates the beforementioned drawbacks. In this method, a high-range water reducer (HRWR) is used to create a self-consolidating concrete (SCC). ACI Committee 237 Self-Consolidating Concrete offers the following definition for SCC [25]:

Self-consolidating concrete (SCC) is highly flowable, non-segregating concrete that can spread into place, fill the formwork, and encapsulate the reinforcement without any mechanical consolidation.

SCC is a product of technological advancements in the area of under-water concrete technology where the mixtures must ensure high fluidity and high resistance to washout and segregation. Okamura originally advocated SCC in 1986, and the first success with SCC occurred in 1988 [26]. The use of SCC has gained wide acceptance for savings in labor costs, shortened construction time, a better finish, and an improved work environment [27-30].

Advancement in SCC technology was primarily possible due to the introduction of new chemical admixtures that improved and controlled the SCC rheological properties. Better performing SCC mixes were produced on the advent of melamine, naphthalene, polycarboxylate, and acrylic based HRWR superplasticizers and viscosity modifying agents (VMA).

Gao et al [12] proposed using SCC because acceptable SCC is not only highly flowable, but it is also highly stable and homogenous on a macro scale. The Prestressed Concrete Institute (PCI) stipulates the following criteria for SCC [26]:

- Filling ability – The property that determines how fast SCC flows under its own weight and completely fills intricate spaces with obstacles, such as reinforcement, without losing its stability.

- Passing ability – the ability of SCC to pass through congested reinforcement and adhere to it without application of external energy.
- Stability – the ability of SCC to remain homogenous by resisting segregation, bleeding, and air popping during transport and placing as well as after placement.

Gao et al [12] studied SCC containing CNF to see if the same effect was present on the nano scale. In Gao et al's mixing procedure, HRWR, water, and CNF are mixed in a laboratory-grade blender. Simultaneously, fine aggregate, course aggregate, and cement are combined in a centrifugal mixer. The CNF mixture is then slowly added to the mixer to gain a homogenous mix. The fresh concrete was used to create cylinders that were tested in compression. After the test, pieces of the cylinders were observed under a scanning electron microscope (SEM). The SEM showed significant CNF clumping in specimens made of normal CNF concrete and uniform distribution in SCC containing CNF, as shown in Figs. 2 and 3, respectively.

Figure 2. Scanning Electron Microscope Image of CNF Clump in Normal Cement (1670x Magnification)

Figure 3. Scanning Electron Microscope Image of Well Dispersed CNF in a Uniform Self-Consolidating Cement (9410x Magnification)

6. Strain sensing ability of CNT/CNF cement-based materials

Smart materials are materials that sense their environment and respond to changes in strain, temperature, moisture, pH, and/or electric or magnetic fields. CNT/CNF composites qualify as smart materials since they can be used to measure strain and temperature [4, 13, 32-35]. There are two types of strain sensing, reversible and irreversible. The measurement of irreversible strain allows structural health monitoring, while the sensing of reversible strain permits dynamic load monitoring. Structural health monitoring is the process of implementing a damage detection and characterization strategy for engineering structures. Dynamic load monitoring can detect loads on structures as they are applied and removed in real time. These are important technologies because they gauge the ability of a structure to perform its intended function despite aging, degradation, or disasters. Typically, monitoring reversible strain is more difficult because it can only be monitored in real time. Additionally, reversible strain tends to be smaller than irreversible strain [31].

Strain sensing refers to the ability to measure an electrical or optical response corresponding to a strain. Chen and Chung [31] give the following requirements for a structural sensor:

- a. Wide strain/stress range of detection (from small strains up to failure)
- b. Response being reversible upon stimulus removal (necessary for repeated use of the sensor)
- c. Ease of measuring the response (without the need of expensive peripheral equipment)
- d. Presence of the sensor having no bad effect on the structural properties of the structure
- e. Chemical stability and durability
- f. Low cost

Current commonly used strain sensors include strain gages, fiber optic sensors, and piezoelectric sensors, which all suffer from high cost, poor durability, and the need for expensive peripheral equipment including electronics and lasers. Because of this, the use of sensors in civil structures is uncommon [31]. CNT/CNF composites could become a better option as a strain sensor because however, technology may provide a way to make them more cheaply in the future.

CNT and CNF cement-based materials exhibits properties necessary for reversible strain monitoring and electromagnetic interference (EMI) shielding. Short-fiber composites were found to be a class of strain sensor based on the concept of short electrically conducting fiber pull-out that accompanies slight and reversible crack opening. For a CNT/CNF composite to have strain sensing ability, the fibers must be more conducting than the matrix in which they are embedded, of diameter smaller than the crack length, and well dispersed. Their orientations can be random, and they do not have to touch one another [32, 33]. The electrical conductivity of the fibers enables the DC electrical resistivity of the composites to change in response to strain damage or temperature, allowing sensing [13, 32-35].

7. Carbon fiber cement and mortar self-sensing applications

Around the same time that CNT were discovered, researchers were adding carbon microfibers to cement-based materials and studying their mechanical properties. In 1992 while studying the mechanical properties of carbon microfibers dispersed in mortar, Yang and Chung [35] noted that the electrical resistivity of mortar containing these fibers dramatically decreased by up to several orders of magnitude.

This idea of electrically conducting concrete led to Chen and Chung proposing an intrinsically smart concrete containing carbon microfibers [8]. Chen and Chung prepared mortar cubes containing carbon microfibers and tested them cyclically. They discovered that the electrical resistivity of the concrete increased irreversibly upon compressive loading up to approximately 1/3 the compressive strength of the mortar. After this point, the resistance reversibly increased and decreased upon loading and unloading of the specimens. Chen and Chung concluded that carbon fiber reinforced concrete can serve as a smart structural material. Chen and Chung followed this experiment with a more detailed cyclic experiment on carbon fiber mortar under cyclic loads [31]. After this test, they concluded that the initial irreversible behavior is due to permanent damage associated with the fiber/matrix interface weakening. They attributed the reversible behavior to crack opening with fiber pull-out and crack closing with fibers pushing back in.

CNT are the most conductive fibers presently known and are, therefore, more ideal for electrical applications than their micro-scale counterparts [36, 37]. CNT and CNF are also attractive for use in cement-based composites because of strength and high aspect ratios [6, 16, 17]. Li et al proposed adding MWCNT to mortar for improved mechanical properties [14]. Li et al confirmed that the flexural and compressive strength of the concrete was enhanced, but they did not study the electrical properties. The same group later studied the electrical volume resistivity of cement paste containing CNT measured using the four-probe method [39]. They applied a cyclic compressive load to a 40.0 mm by 40.0 mm by 160.0 mm (1.575 in. by 1.575 in. by 6.30 in.) rectangular prism made of the material. The fractional change in the volume resistivity oscillated up to approximately 10% with the oscillation of the compressive load.

8. Damage detection of CNF concrete columns

Gao et al expanded the work on self-sensing cement-based materials by studying 152.4 mm by 305 mm (6.00 in. by 12.00 in.) cylinders made of concrete containing CNF [12]. Gao et al crushed the cylinders monotonically and studied the electrical resistance variation. They observed electrical resistance variations up to 80% and concluded that concrete containing CNF can be used for self structural health monitoring.

Howser et al continued Gao et al's work and extended it to a full scale reinforced concrete column containing CNF [4, 12]. A self-consolidating CNF concrete (SCCNFC) column was built and tested under a reversed cyclic load. The structural behavior and the self-sensing ability were examined. The results were compared to the structural and self-sensing ability of

a traditional self-consolidating reinforced concrete (SCRC) and a self-consolidating steel fiber concrete (SCSFC) specimen.

All of the columns were 508 mm (20.0 in.) tall with cross-sections of 305 mm by 305 mm (12.00 in. by 12.00 in.). Each specimen contained six #8 (25.4 mm or 1.00 in. diameter) rebar, which corresponded to 3.27% longitudinal steel by volume of concrete. The SCRC and SCCNFC columns contained #2 stirrups with a spacing of 120.7 mm (4.75 in.) providing transverse reinforcement of 0.287% by volume of concrete. Since the columns were designed to be shear critical, the maximum reinforcement spacing was chosen based on the ACI 318 specifications [25]. See Fig. 4 for the cross-section used for the SCRC and SCCNFC columns. SCSFC column contained no transverse reinforcement, as shown in Fig. 5. Each of the columns was rigidly connected to similar foundations. See Fig. 6 for the elevation view of the SCRC and SCCNFC columns and foundations. The SCSFC column is identical to that shown in Fig. 6, except it does not contain transverse reinforcement. Fig. 7 shows the experimental set-up.

Figure 4. Cross-Section of SCRC and SCCNFC Columns (dimensions in inches)

Figure 5. Cross-Section of SCSFC Column (dimensions in inches)

Figure 6. Elevation View of the Strong Axis of the Shear-Critical SCRC and SCCNFC Columns and Foundations (dimensions in inches)

Figure 7. Experimental Set-Up

The properties of the materials used for the three mixes were as follows:

- a. Cement: The cement used in all mixtures was ASTM Type III Portland cement.
- b. Fly Ash: Class C fly ash was used for the SCSFC mix and Class F fly ash was used for the SCRC mix.

- c. Coarse Aggregate: Crushed limestone with a maximum diameter of $\frac{3}{4}$ " was used in the SCCNFC column. River rock with a maximum diameter of $\frac{3}{4}$ " was used in the other columns.
- d. Fine Aggregate: Natural river sand with a fineness modulus of 2.71 was used in all mixes.
- e. High Range Water Reducer (HRWR): Glenium® 3200HES was used in the SCCNFC column and Glenium® 3400 HES was used in the other columns. Both chemicals were polycarboxylate admixtures from BASF Chemical Co.
- f. Viscosity Modifying Agent (VMA): RHEOMAC® VMA 450 was used in the specimens and also supplied by BASF Chemical Co.
- g. Steel Fibers: Dramix® ZP305 fibers were used in the SCSFC mix. This was a hooked fiber with a specific gravity of 7.85. The diameter of the fiber is 0.55 mm (0.0217 in.) and the length is 30 mm (1.18 in.) resulting in an aspect ratio of 55.
- h. Carbon Nanofibers: Pyrograf Products, Inc. PR-19-XT-LHT-OX fibers were used in this study. The specific gravity of the fibers was 0.0742. The diameter of the fibers was 149 nm ($5.87\text{e-}6$ in.) and the length was 19 μm ($7.48\text{e-}4$ in.) resulting in an aspect ratio of 128.

The mix proportions used for the three columns can be seen in Table 1. One percent fiber by volume was used for both of the fiber columns chosen based on literature review. It was discovered by Gao et al that CNF has an optimal dosage of approximately 1% by volume [12] [12]. It was found by many researchers that increased steel fiber increases concrete properties; however, after a percentage of 1% fibers by volume, the concrete becomes increasingly less workable, which could cause problems in construction such as honeycombing [39-41].

Material	SCRC Mix	SCSFC Mix	SCCNFC Mix
Cement	446 (752)	446 (752)	457 (771)
Fly Ash (Class C)	-	299 (504)	-
Fly Ash (Class F)	299 (504)	-	-
Fine Aggregate	937 (1580)	937 (1580)	898 (1514)
Coarse Aggregate (Limestone)	-	-	859 (1448)
Coarse Aggregate (River Rock)	491 (827)	491 (827)	-
Water	224 (377)	224 (377)	182 (307)
Glenium® 3400HES	2.81 (4.73)	2.81 (4.73)	-
Glenium® 7700HES	-	-	2.34 (3.94)
REHEOMAC® VMA 450	5.69 (9.59)	5.69 (9.59)	-
Steel Fibers	-	79.8 (134)	-
Carbon Nanofibers	-	-	3.23 (5.45)

Table 1. Mix Proportions in kg/m^3 (lb/yd^3) of Concrete

The main goal of testing the SCCNFC column was to prove that concrete containing CNF can be used as a sensor. To test the electrical properties of the concrete, wire meshes were constructed and embedded in each of the columns. The wire meshes were made of 12.7 mm (1/2 in.) hardware cloth with 14 gauge copper wire soldered to it. The wire extended outside of the column. The four probe method for measuring resistance was implemented, and the meshes were placed in the column as shown in Fig. 8. A power supply was attached to the top mesh that provided a current of approximately 31 V DC. An ammeter was attached to the bottom mesh and connected back to the power supply to complete a circuit. The current measured by the ammeter was recorded continuously during the tests by hand. Additional voltmeters were attached to the two middle meshes on both the north and south sides of the column to measure voltage. The voltage readings were also recorded continuously throughout the test.

Figure 8. Four Probe Method of Resistance Measurement

The first step of the load program was to apply an axial load that would remain constant during the course of the test. The axial load equaled one-tenth of each of the columns calculated axial capacity. The axial capacity is dependent on the compressive strength of the concrete, so the axial force for each specimen varied.

After the application of the axial load, a reversed-cyclic load was added using a 649 kN (146 kip) capacity actuator. The intended load path was to use force control to complete two cycles each of ± 89 kN (20 k), ± 178 kN (40 k), and ± 267 kN (60 k). A positive force denotes a push by the actuator while a negative force represents a pull. At the point of longitudinal steel yielding, the test switched to displacement control and completed two cycles each at a displacement

ductility of 2, 3, 4, etc. Once failure occurred, a descending branch on the load versus displacement curve was obtained in displacement control mode.

The load path followed for the SCRC column specimen can be seen in Fig. 9 with the first cracks, switch to displacement control and failure marked. The first crack on the south side of the column occurred at -178 kN (-40 k). The first shear crack formed on the column during the first -178 kN (-40 k) cycle at -178 kN (-40 k) on the west side. The column failed in shear and crushing of concrete at 276 kN (62 k). The west side of the column exhibited crushing of the concrete struts with large shear cracks. The east side exhibited local crushing at the actuator connection. The maximum displacement at the top of the column (drift) was 12.7 mm (0.50 in.).

Figure 9. SCRC Column Load Path

The load path followed for the SCSFC column can be seen in Fig. 10 with the first cracks and failure marked. The first shear and flexural cracks formed on the column during the second 178 kN (40 k) cycle at 178 kN (40 k) on the west and north sides, respectively. The second flexural crack formed on the south side during the second -178 kN (-40 k) cycle at -178 kN (-40 k). The column failed suddenly in shear and crushing at 347 kN (78.0 k) on the west side of the column before the rebar yielded. The maximum displacement was 8.38 mm (0.33 in.).

The actual load path followed for the SCCNFC column can be seen in Fig. 11. The pump shut down during the test, and the actuator unloaded during the fifth cycle of the test. The pump was turned back on and the test resumed. The first flexural crack formed on the column at 160 kN (36 k) on the east, west and north sides. The second flexural crack formed on the east, west and south sides at a load of -158 kN (-35.6 k). The column failed in the combined modes of shear and concrete crushing due to flexure at 298 kN (67 k) on the west side of the column. The maximum displacement was 10.16 mm (0.4 in.).

During each of the column tests, the electrical resistance was determined to check the self-sensing ability of the concrete. The electrical readings showed a great correlation between the peaks in the applied horizontal force, strain, and resistance plots for the SCCNFC column but

Figure 10. SCSFC Column Load Path

Figure 11. SCCNFC Column Load Path

little correlation between the resistance plots and the force or strain plots for the SCRC or SCSFC column. Fig. 12 shows the relationship between the SCRC column's horizontal force, LVDT strain, and electrical resistance versus time on the north side of the column. There is no relationship between the peaks and valleys in the electrical resistance and the load or strain on the north side of the column.

Fig. 13 shows the SCSFC column's force, strain, and resistance versus time on the north and south sides of the column, respectively. As shown by the grey vertical lines, there is not a relationship between the peaks and valleys in the resistance and load or strain until major cracking began to occur. After major cracking began to occur, the peaks and valleys in the electrical resistance began to correspond with the load and strain peaks and valleys. This point is shown by the dashed line in Fig. 13.

Fig. 14 shows relationship between the SCCNFC column's horizontal load, LVDT strain and electrical resistance versus time on the north side of column. As shown by the vertical lines in

Figure 12. SCRC Column Comparison of Horizontal Force, LVDT Strain and Electrical Resistance on North Side

Figure 13. SCRC Column Comparison of Horizontal Force, LVDT Strain and Electrical Resistance on North Side

the grid, there is very good correlation between the force, strain and resistance. On the north side of the column, the peaks and valleys matched up until the point that the column was greatly damaged.

Because of the strong correlation found between the horizontal load, LVDT strain, and electrical resistance verses time graphs for the SCCNFC column, the electrical resistance variation (ERV) was calculated and compared to the deflection at the top of the column. ERV is the measured electrical resistance minus the initial electrical resistance quantity divided by the initial electrical resistance. Fig. 15 shows the relationship between the ERV and deflection

Figure 14. SCCNFC Column Comparison of Horizontal Force, LVDT Strain and Electrical Resistance on North Side

at the top of the column for the first five cycles of the test. It is obvious from Fig. 15 that the column shows major damage at approximately a deflection of 2.03 mm (0.08 in.). This corresponds to the steel yielding in the SCCNFC column. This proves that SCCNFC can be used as a type of self-structural health monitoring system.

Figure 15. SCCNFC Column ERV versus Horizontal Deflection

9. Conclusions

Self-consolidating carbon nanofiber concrete (SCCNFC) follows the definition for nanotechnology set forth by the National Science Foundation and National Nanotechnology Initiative [2]. The size range of the carbon nanofibers (CNF) is approximately 100 nanometers, the SCCNFC is able to measure damage in the composite, and the CNF have properties that are specific to the nanoscale.

Well-dispersed CNF improves the strength and stiffness of concrete. Excess concentration leads to poorly dispersed CNF clumps inside the concrete and has a negative effect on both strength and electrical sensitivity. Highly workable and stable self-consolidation concrete (SCC) can maintain its workability and stability with the addition of fibers. SCC greatly increases the dispersion of carbon nanofibers (CNF) [12].

As proven by Gao et al [12] and Howser et al [4], SCCNFC can be used as a reversible strain sensor. In Howser et al's test [4], the peaks and valleys in the electrical resistance readings of the SCCNFC match the peaks and valleys of the applied force and the strain in the concrete. While the peaks and valleys in the electrical resistance readings of the self-consolidating reinforced concrete and self-consolidating steel fiber concrete specimens occasionally matched, there was not enough correspondence to safely assume that these concretes could be used as a reversible strain sensor. It was concluded that when an appropriate dosage of CNF is used, SCCNFC can be used for self-structural health monitoring.

Author details

Y.L. Mo and Rachel Howser Roberts

University of Houston, USA

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