Development of Cement-Coir Carbon Fiber Composites with Damage Self Detection Capability

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Abstract

Research on the manufacture of cement-carbon composite materials using carbon fiber from coconut coir fiber has been performed. Carbonization was carried out at two phases. First, it was carbonized at a temperature of 400°C for 300 min and continued by the second phase at a temperature of 800°C for 60 min. The structures of carbon fiber was measured using X-Ray Diffraction (XRD) while the sample surface analysis was carried out using Scanning Electron Microscope (SEM) and the electrical conductivity of samples was measured using LCR (Inductance Capasitance and Resistance) meter. Three carbon types (carbon without treatment, carbon soaked in 10% and 20% solution of potassium hydroxide (KOH)) were used to manufacture cement-carbon composites. Three levels of carbon content of 0.5, 0.75 and 1.0% by weight of cement were used. Results showed that the cement-carbon composite added with soaked carbon in 20% solution of KOH at 1.0% carbon content has the best properties of compressive strength (24.94 ± 1.24 MPa), modulus of rupture (MOR) (5.23 ± 0.47 MPa) and damage self-detection (load at the first crack = 21.04 N).

Keywords: coconut coir fiber, carbon, cement-carbon composites, potassium hydroxide, damage self-detection.

Introduction

Cement composites are very important materials for infrastructure such as buildings, roads, and bridges. In fact, this material is always associated with damage. Therefore, it is necessary to detect the damage to avoid accident. One of the detection technologies of building damage that has been developed is by using optical fiber as a sensor that is connected with a system of damage monitoring.

The study of damage detection of buildings in terms of its material has been widely done. Yao *et al.* (2003) and Chen *et al.* (2004; 2005) made cement-carbon fiber composite using electrical conductivity of carbon fibers with ability to detect damage. Good electrical conductivity of the material is due to percolation. Percolation is a state in which carbon fibers of the adjacent contact produce a continuous flow of electricity which causes electrical conductivity rises. This material is sensitive to changes in the load so that it can detect this change (Wen and Chung 2007).

The addition of certain amounts of carbon fiber will increase strength and electrical conductivity of composites. Yao *et al.* (2003) added carbon fiber as much as 0.2 ~ 1.2% by volume fraction. Chen *et al.* (2004, 2005) made cement-carbon composite with silica fume and carboxy methylcellulose (CMC) as a dispersing.

Carbon fibers in the market are available in two types in the forms of polyacrylonitrile (PAN) and isotropic pitch. Carbon has high strength, electrical conductivity and expensive. An alternative material with a lower price but has ability that is close to commercial carbon fibers is required.

Carbon fiber derived from wood and natural fibers can be used as an alternative to commercial carbon fibers. Coconut coir fiber (*Cocos nucifera*) has a plentiful availability. These fibers have higher lignin content than other natural fibers. High content of lignin will cause the fibers to have high strength (Khalil *et al.* 2006).

Research manufacture of carbon from wood and natural fibers has been carried out. Preparation of carbon from Japanese cedar wood by carbonization temperature of 700°C has caused the cell wall seemed increasingly clear (Ishimaru *et al.* 2007). Nishimiya *et al.* (1995) stated that the charcoal from sugi wood became a conductor when carbonized at a temperature of 800°C, due to reduced compounds in the secondary cell wall.

Girgis *et al.* (2002) and Pari *et al.* (2006) activated the charcoal product by using materials such as KOH, NaOH, H_3PO_4 and $ZnCl_2$. Activation was conducted at a temperature of 800°C by flowing water vapor, nitrogen gas or CO_2. The main effect of this activation is expansion of the pores of charcoal by eliminating hydrocarbon compounds or tar coating its surface.

This study aimed to determine the pattern of changes in the structure and electrical conductivity of coconut coir carbon fiber, both without treatment and by soaking in KOH solution. It also aimed to determine the effect of soaking solution of KOH treatment on the strength and capability of damage self-detection of composite materials.

Materials and Methods

Materials

Materials used in this study were coconut coir fibers, potassium hydroxide (KOH), portland cement, sand, water, silica fume densified type (bulk density around 650 ~ 700 kg/m^3), and technical grade CMC.

Production and Characterization of Carbon from Coconut Coir Fiber

Production of carbon of coconut coir fiber was conducted in a carbonization furnace with 5 kg capacity, with 400°C in temperature for 300 min and cooled for 12 ~ 24 h. Carbon produced then divided into three: carbon without any treatment, carbon treated with 10% KOH, and soaked in 20% KOH. Carbon was then reheated on activation furnace with 300 g capacity at 800°C for 60 min. Carbon was then analyzed for its structure pattern and performance using XRD (Shimadzu 7000 series-40 kV) and SEM (JSM 6360 LA-20 kV).

Measurement of Electrical Conductivity of Coconut Coir and Coconut Coir Carbon

Electrical conductivity measurement of coconut coir and coconut coir carbon was done using LCR meter. The value of the conductivity is:

$$\sigma = \frac{D}{R \times A}$$

With σ is electrical conductivity, D is length of the piece of material (measured in metres, m), A is the cross-sectional area of the specimen (measured in square metres, m^2), and R is the electrical resistance of a uniform specimen of the material (measured in ohms, Ω).

Production of Cement-Coconut Coir Carbon Composites

Three kinds of carbon fibers were used in this research, those were carbon fibers without treatment, carbon fibers soaked in 10% KOH and carbon fibers soaked in 20% KOH. The fiber length was 5 ~ 10 mm and the ratios to cement weight were varied into 0.5, 0.75 and 1%. Other materials involved in this research were sand with 0.595 ~ 0.841 mm in size, 10% silica fume and 0.5% CMC of cement weight. Ratio of water to cement was 0.62 and sand to cement was 1.0.

Production of cements involved four steps. Firstly, 30% of total water, CMC, carbon fibers and silica fume were stirred well using mixer. Secondly, the mixed materials were put on a mortar mixer which was filled with cement and sand inside while during this stirring process the rest of water (70%) was added slowly. Thirdly, after all composite materials were well dispersed, it was put on 25 x 25 x 300 mm metal molding. Lastly, composites were conditioned at room temperature for 24 h, pulled from its mold and soaked in water for 28 days.

Test of Cement-Coconut Coir Carbon Fiber Composites

Tests conducted for the composite were compressive strength (ASTM C116-90), modulus of rupture and self-damage detection (ASTM C293-94). To determine the effect of immersion of carbon fibers in aqueous KOH to the strength of the composite, statistical analysis was performed using a factorial design in complete randomized design (CRD).

Self-damage detection test was conducted to show the relation between accepted load and electrical conductivity of samples. Schematic of the test (Figure 1) is refers to ASTM C293-94 about Flexural Strength Test with Center Point Load Method.

Results and Discussions

Analysis of Structure Pattern of Carbonized Coconut Coir Fiber

Table 1 shows the XRD analysis of the structure of coconut coir, carbon and the three other carbons with 800ºC carbonization temperature for 60 min.

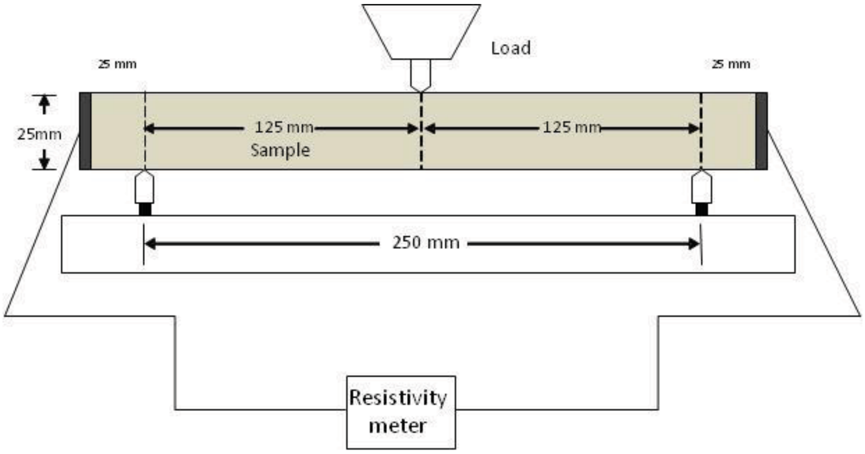

Figure 1. Schematic of test samples of cement-carbon fiber composites.

Table 1. Structure of coconut coir and its carbon fibers.

Materials	X (%)	θ_{002} (°)	d (nm)	θ_{100} (°)	d (nm)	Lc (nm)	N	La (nm)
Coconut coir fiber control	16.36	22.31	0.40	22.31	-	-	-	-
Carbonized coconut coir fiber at temperature of 400°C, 300 min	37.30	22.93	0.39	44.05	0.21	2.27	5.86	19.88
Carbonized fiber at 800°C,60 min, untreated	51.74	23.68	0.38	44.04	0.21	1.60	4.27	5.08
Carbonized fiber at 800°C, 60 min, soaked in KOH 10%	53.34	23.80	0.37	44.02	0.21	1.61	4.30	2.88
Carbonized fiber at 800°C, 60 min, soaked in KOH 20%	52.31	23.64	0.38	44.08	0.21	1.44	3.83	4.05

Note : X : degree of crystallinity
θ : diffraction angel
d : distance of aromatic layer
Lc : height of aromatic layer
N : number of aromatic layer
La : width of aromatic layer

Degree of crystallinity of coconut coir fiber was lower compared to coconut coir carbon at carbonized temperature of 400 and 800°C. The changes were due to the intencity displacement on diffraction angle and the formation of new diffraction angle. Thus, it showed the differences between crystal structure of coconut coir and its carbon.

Degree of crystallinity on the carbon fiber at carbonized temperature of 800°C was higher than the degree of crystallinity on the carbon fiber at carbonized temperature of 400ºC. This was caused by distance of aromatic layer (d), height of aromatic layer (Lc) and width of aromatic layer (La) of the high temperature carbon which were lower than those of carbon at carbonized temperature of 400°C so that the structure of the crystal was arranged more regularly.

Carbon soaked in KOH and carbon without treatment had similiar structure pattern. Carbon fiber soaked in 20% KOH solution treatment and carbon fiber without treatment had different Lc and La value. This was showed by the presence of carbon structure changes due to KOH soaked treatment.

Electrical Conductivity of Coconut Charcoal from Fiber

Coconut coir charcoal with carbonization at temperature of 400°C had an electrical conductivity of 0.0000032 S m^{-1}, which is smaller than that of the carbon fibers that were produced at higher temperature. Carbon fibers carbonized at temperature of 800°C without treatment and 10% KOH soaked treatment had electrical conductivity values of 111.83 and 112.05 S m^{-1} while the electrical conductivity of carbon fiber soaked in 20% KOH was 136.195 S m^{-1}. This shows that the immersion of carbon fiber in a solution of 20% KOH has an effect in reducing substances that exist around the pores so it can conduct electricity better.

Appearance of carbon fiber surface transversely using the SEM can be seen in Figure 2.

Based on Figure 2 it seems that the pores of coconut coir fiber were not apparent. Pores are evident when fiber was carbonized at temperature of 800°C. This happens because heating causes degradation of compounds of middle lamella and secondary cell wall that form component which produce gases, liquids and solid substances such as charcoal (Vigouroux 2001).

Compression Strength and Modulus of Rupture of Composites

Compression strength and modulus of rupture of cement-carbon composites are shown in Table 2.

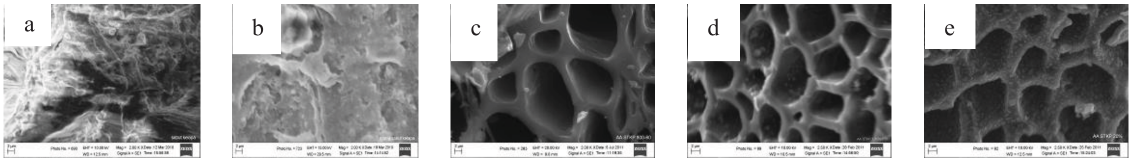

Figure 2. Surface appearance of (a) coconut coir fiber, (b) charcoal carbonized at 400°C, (c) carbonized at 800°C without treatment, (d) carbonized at 800°C soaked in 10% KOH and (e) carbonized at 800°C soaked in 20% KOH.

Table 2. Compression strength and modulus of rupture of cement-carbon composites.

No.	Materials	Compression Strength (N mm^{-2})	Modulus of Rupture (N mm^{-2})
1	Control (cement-sand)	30.88 ± 2.63	6.63 ± 1.21
2	Untreated carbon fibers - 0.5%	21.47 ± 3.99	4.25 ± 0.53
3	Untreated carbon fibers - 0.75%	20.34 ± 2.16	4.56 ± 0.11
4	Untreated carbon fibers - 1.0%	17.35 ± 2.05	4.52 ± 0.29
5	Soaked in 10% KOH - 0.5%	18.06 ± 0.69	4.61 ± 0.42
6	Soaked in 10% KOH - 0.75%	20.82 ± 2.11	4.51 ± 0.26
7	Soaked in 10% KOH - 1.0%	15.77 ± 0.07	4.55 ± 0.17
8	Soaked in 20% KOH - 0.5%	17.72 ± 4.81	4.58 ± 0.40
9	Soaked in 20% KOH - 0.75%	19.56 ± 2.91	4.83 ± 0.45
10	Soaked in 20% KOH - 1.0%	24.94 ± 1.24	5.23.± 0.47

Note: - carbon fibers percentage based on cement weight
- values are mean ± standard deviation of each of the three samples tested

Cement composites with carbon fibers soaked in 20% KOH and carbon fiber content of 1.0% had the highest values of compressive strength and modulus of rupture compared to other composites. Based on advanced statistical analysis using Dunnet test, it can be seen that the strength of carbon fiber composites soaked in 20% KOH with fiber content of 1.0% is not significantly different from controls. This shows that the addition of carbon fibers in the composite does not affect the strength of the composite.

Testing of Self-Damage Detection of Cement-Carbon Composite

Testing of the ability of self-damage detection in composite was conducted at the same time by performing two measurements. The changes of load and simultaneous electrical conductivity can be observed and recorded. The relationship between given load with the change in electrical conductivity of control samples (cement-sand) can be seen in Figure 3.

Based on Figure 3 we can see that giving load on control samples did not lead to increased electrical conductivity. The electrical conductivity values of control sample before and while given load was in the range of 10^{-4} S m^{-1}.

The relationship between load provided with the electrical conductivity of carbon fiber-cement composites without treatment can be seen in Figure 4.

Based on Figure 4 we can see that giving load on cement composites with carbon fibers without treatment, resulting in an increase in electrical conductivity of the composite at the time of occurrence of cracks. For the composite with carbon fiber content of 0.5, 0.75 and 1.0% by weight of cement, an increase in the time has given successive load of 45.00, 43.44 and 42.19 N. The maximum conductivity values of composites at the time of receiving the load to levels of carbon fiber of 0.5, 0.75 and 1.0% were 0.011456, 0.010939 and 0.018334 S m^{-1}. Based on these data we can say that the greater content of carbon fiber used the greater electrical conductivity of the composite and the earlier it can detect damage to the load given to it.

The relationship between the load provided by the electrical conductivity of cement composites - carbon fiber soaking solution of KOH 10% can be seen in Figure 5.

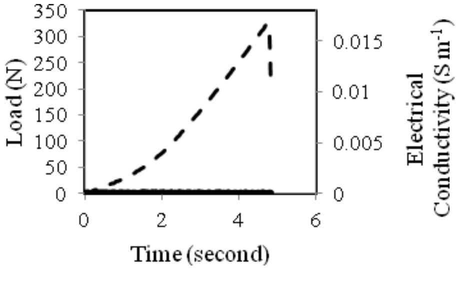

Figure 3. The relationship between given load and electrical conductivity of control composites.
– – – Load, —— Electrical conductivity.

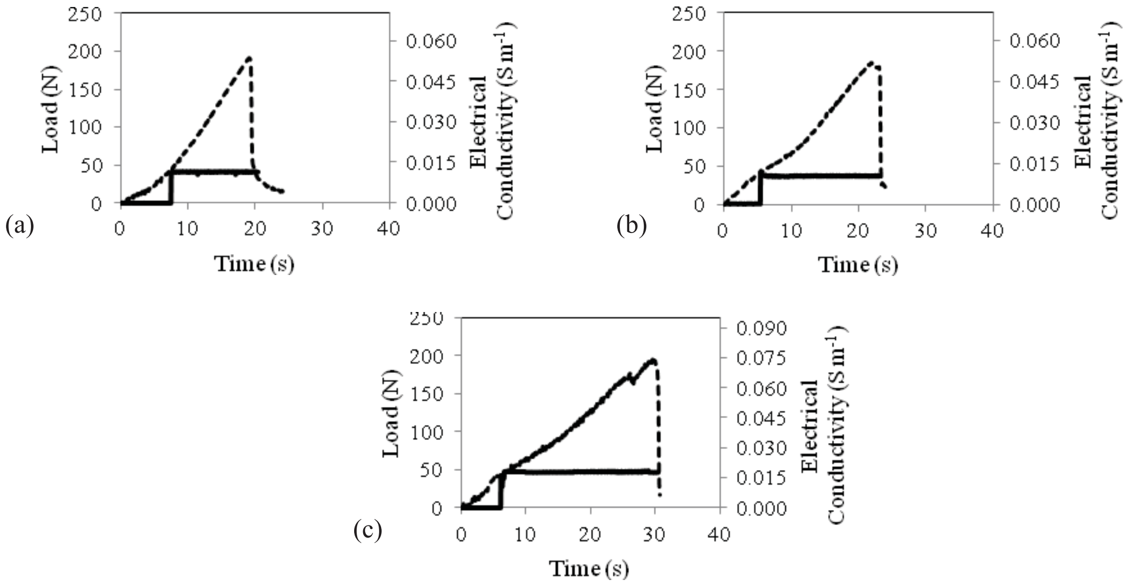

Figure 4. The relationship between load and electrical conductivity of cement composites with carbon without immersion at carbon content of (a) 0.5% (b) 0.75% and (c) 1.0% by weight of cement.
– – – Load, ——— Electrical conductivity

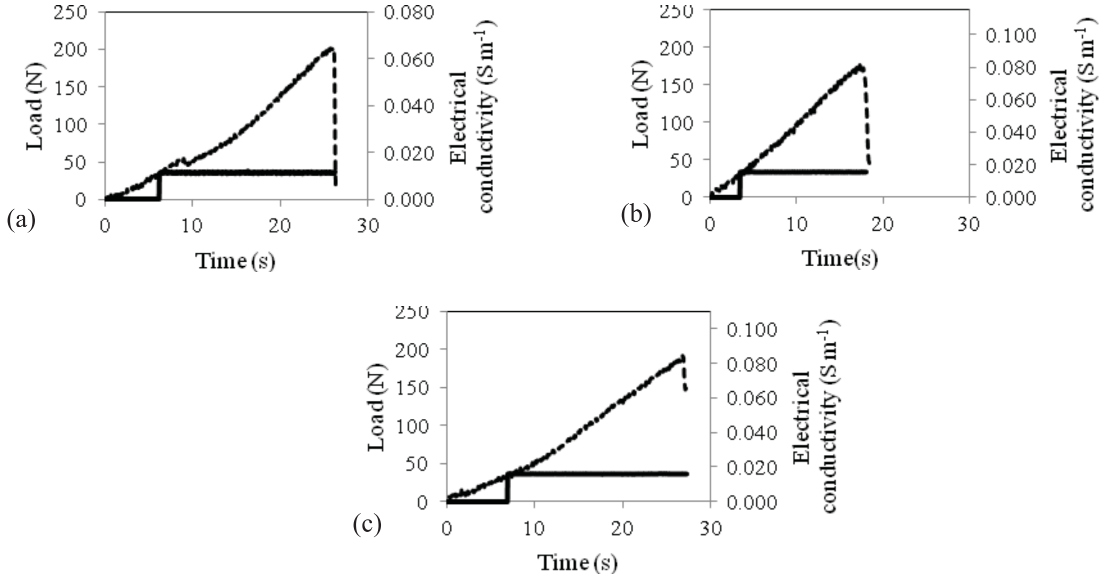

Figure 5. The relationship between load and electrical conductivity of cement composites with carbon fibers soaked in 10% KOH at carbon content of (a) 0.5% (b) 0.75% and (c) 1.0% by weight of cement.
– – – Load, ——— Electrical conductivity.

Based on Figure 5 we can see that the giving load on cement composites with carbon fibers soaking solution of 10% KOH, resulting in increased electrical conductivity of the composite when the occurrence of cracks. Successively for composites with carbon fiber content of 0.5, 0.75 and 1.0%, increase in conductivity occurs when the load is given to a sample of 36.72, 33.59 and 32.81 N. The maximum value of the electrical conductivity of the composite at the time of receiving loads for the content of carbon fiber of 0.5, 0.75 and 1.0% were 0.012151, 0.015466 and 0.015832 S m^{-1}, respectively.

The relationship between load and electrical conductivity of cement composites - carbon fiber soaked in 20% KOH solution can be seen in Figure 6.

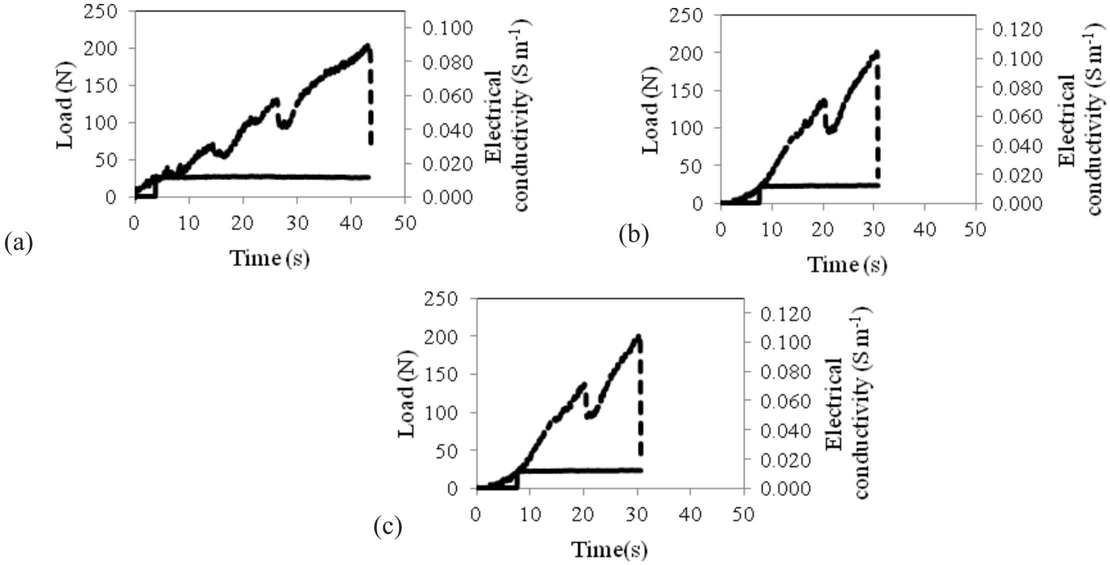

Figure 6. The relationship between load and electrical conductivity of cement composites with carbon fibers soaked in 20% KOH at carbon content of (a) 0.5% (b) 0.75% and (c) 1.0% by weight of cement.
– – – Load, —— Electrical conductivity

Based on Figure 6 we can see that giving load on the composites with soaked 20% KOH solution carbon fiber increased the value of electrical conductivity when the occurrence of cracks. Respectively for composites with carbon fiber content of 0.5, 0.75 and 1.0%, increased conductivity occurs during given load of 25.0 , 23.44 and 21.04 N. The maximum electrical conductivity of the composite value at the time of receiving the load in carbon fiber content of 0.5, 0.75 and 1.0% were 0.012291, 0.012474 and 0.01288 S m^{-1}, respectively.

Based on test data, cement composite with carbon fiber soaked in 20% KOH solution can detect early damage in comparison with other carbon fiber composites. This was indicated by the smaller load provided at the time of its electrical conductivity increases.

Conclusions

The pattern of the carbon structure of coconut coir carbon fibers carbonized at 800°C is different from that carbonized at 400°C. This is due to the changes of crystal structure due to high temperature heating. In addition, the appearance of the surface and its electrical conductivity becomes higher after carbonized in high temperature.

Composites with carbon fiber soaked in KOH solution gave the self detection damage better than cement composites with untreated carbon fibers. Composites with carbon fiber soaked in 20% KOH solution with a content of 1.0% by weight of cement had a compressive strength value (24.94 ± 1.24 N mm^{-2}), modulus of rupture value (5.23 ± 0.47 N mm^{-2}) and best self-damage detection (detection of damage to the load at 21.04 N) compared with other carbon-cement composites.

Composites of cement-carbon fiber soaked in 20% KOH solution with 1.0% carbon content can be used for further study. In addition, it is necessary to do research on the morphology of the composite at the time of the increase of its electrical conductivity or in the event of detection of the damage themselves.

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