The Korean Journal of Ceramics, **6**[2] 201-205 (2000)

Damage Detection in Fiber Reinforced Composites Containing Electrically Conductive Phases

Soon-Gi Shin and Hideaki Matsubara

Japan Fine Ceramics Center, 2-4-1 Mutsuno, Atsuta-ku, Nagoya 456, Japan

(Received February 23, 2000)

Fiber reinforced plastic (FRP) composites and ceramic matrix composites (CMC) which contain electrically conductive phases have been designed and fabricated to introduce the detection capability of damage/fracture detection into these materials. The composites were made electrically conductive by adding carbon and TiN particles into FRP and CMC, respectively. The resistance of the conductive FRP containing carbon particles showed almost linear response to strain and high sensitivity over a wide range of strains. After each load-unload cycle the FRP retained a residual resistance, which increased with applied maximum stress or strain. The FRP with carbon particles embedded in cement (mortar) specimens enabled micro-crack formation and propagation in the mortar to be detected in situ. The CMC materials exhibited not only sensitive response to the applied strain but also an increase in resistance with increasing number of load-unload cycles during cyclic load testing. These results show that it is possible to use these composites to detect damage and/or fracture in structural materials, which are required to monitor the healthiness or safety in industrial applications and public constructions.

Key words: Self-diagnosis, Composite, Resistance, FRP, CMC

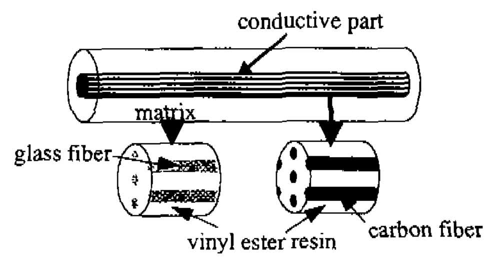
I. Introduction

There are many demands and requirements for quantitative methods to evaluate the health and safety of important constructions that have suffered a serious disaster or overloading. Recently, in situ monitoring techniques utilizing structural materials with the ability to diagnose their own health, so-called self-diagnosis materials or intelligent materials, have received increasing attention. Some methods of fracture detection in fiber reinforced plastics (FRP) that have been embedded in structural materials as reinforcement have been proposed.[1-6] Muto et al. reported that the changes in electrical resistance in carbon-fiber/glass-fiber-reinforced plastic (CFGFRP) composite could be used as a measure of fatal fracture.[1] Although the CFGFRP composite showed appreciable resistance change in the strain range above 0.7-1.5% due to fracture in the carbon fibers, the detection limit is too large to detect local damage in structural materials. The acoustic emission method and fiber optic sensors have been researched with the aim of using them to monitor the health of materials in service, but these systems are expensive. It is therefore necessary to develop self-diagnosis materials that are both more sensitive in the small strain region and are simple in concept and design.

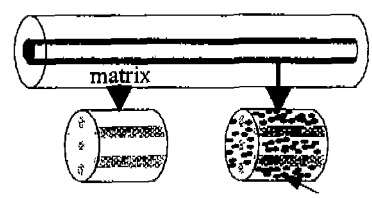
In the present work, electrically conductive composites containing continuous paths of conductive particles were designed and fabricated. Similar structures were formed in the FRP composites and the ceramic matrix composites (CMC). The detection ability of these materials was evaluated by measuring the changes in electrical resistance with applied strain.

II. Experimental Procedure

Fig. 1 is a schematic drawing of the structural design for conductive FRP which basically consists of vinyl ester resin (Showa High Polymer Co., Ltd. RIPOXY R-804) and glass fiber (Asahi glass fiber Co, Ltd. ER2220). The carbon fiber



(a) Carbon-Fiber/Glass-Fiber Reinforced Plastics (CFGFRP)



(b) Carbon-Particle/Glass-Fiber Reinforced Plastics (CPGFRP)

Fig. 1. Schematic drawing of the structural design for CFG-FRP (a) and CPGFRP (b).

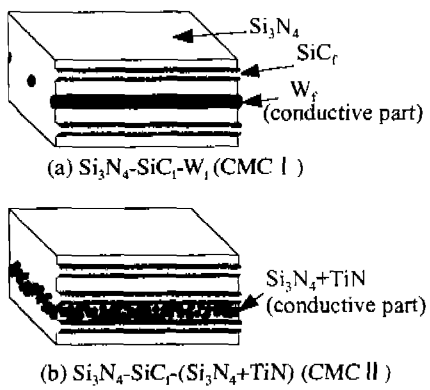


Fig. 2. Schematic drawing of the structural design for CMC containing W fiber (a) and TiN particles (b).

(pitch-based C_f, Toho Rayon Co., Ltd. BESFIGHT UM63) introduced in replacement of some of the glass fibers forms conductive paths and enhances the strength in the length direction of the composite. Carbon particles (graphite, SEC Co., Ltd. SPG5) dispersed in a part of each matrix created conductivity paths with a percolation structure. The composites containing carbon particles are referred to hereafter as carbon-particles/glass-fiber-reinforced plastics (CPGFRP). The preparation details of the composites and reinforced concrete specimens have been described in another paper.[7]

Fig. 2 shows the structural design for the CMC materials. The composites were fabricated by the filament winding method using Si_3N_4 particles (Ube Industries Co., Ltd. SN-COA) as the matrix and SiC fiber (SiC_f, ϕ15 μm, Nippon Carbon Co., Ltd. NL-401) as the reinforcement for strengthening and toughening. A portion of the fibers was replaced with W fiber (W_f, ϕ30 μm, Nippon Tungsten Co., Ltd.). Conductive particles of Si_3N_4 with 40 vol% TiN (mean diameter <2.0 μm, Japan New Metals Co., Ltd.) were dispersed in a part of the matrix. These composites were hot pressed under 40 MPa at 1773 K in N_2 atmosphere for 1 hr. Sintered specimens were cut into bars of 3×4×45 mm^3 for the bending test, with either conductive fiber or particles located 0.5 mm from the tensile side. The components of each composite are listed in Table 1.

The self-diagnosis ability of these materials was evaluated

Table 1. Components of Each Composite

Component \ Composite	CPGFRP (vol%)	GFGFRP (vol%)	CMC I (vol%)	CMC II (vol%)
Carbon powder	0.3	–	–	–
Carbon fiber	–	0.3	–	–
Glass fiber	45	45	–	–
Vinyl ester resin	bal.	bal.	–	–
W fiber	–	–	0.02	–
TiN powder	–	–	–	0.13
SiC fiber	–	–	40	40
Si_3N_4 powder	–	–	bal.	bal.

by simultaneous measurement of stress and electrical resistance change as a function of applied strain during a tensile loading test or bending test. Electrical resistance measurements were made by attaching a power source (Hioki Electric Co., Ltd. 7020 type) supplying a constant current to the fiber/powders within the composite via copper wires. The voltage drop across the specimen was measured using a digital multimeter (Yokogawa Electric Co., Ltd. 7561 type), and the resistance recorded simultaneously with the strain (via strain gauges attached to either side of the specimen) by a data-logger (Keyence Co., Ltd. NR-110 type). The entire system was controlled from a computer interface.

The resistance change was defined as relative change in resistance $(R-R_0)/R_0$, indicated by $\Delta R/R_0$ in which R_0 denotes initial resistance. Two types of loading were applied: normal tensile or bending until specimen fracture, and cyclic loading-unloading test below the maximum stress level. Three specimens were used to confirm reproducibility, and representative data from each test are reported in the following section.

III. Results and Discussion

3.1. Fracture detection of FRP composites

Fig. 3 shows the changes in electrical resistance and applied stress as a function of applied strain in tensile test for CFGFRP and CPGFRP. The stresses in both specimens increased linearly in proportion to the strains until fracture of all the carbon fibers or glass fibers. The CFGFRP showed

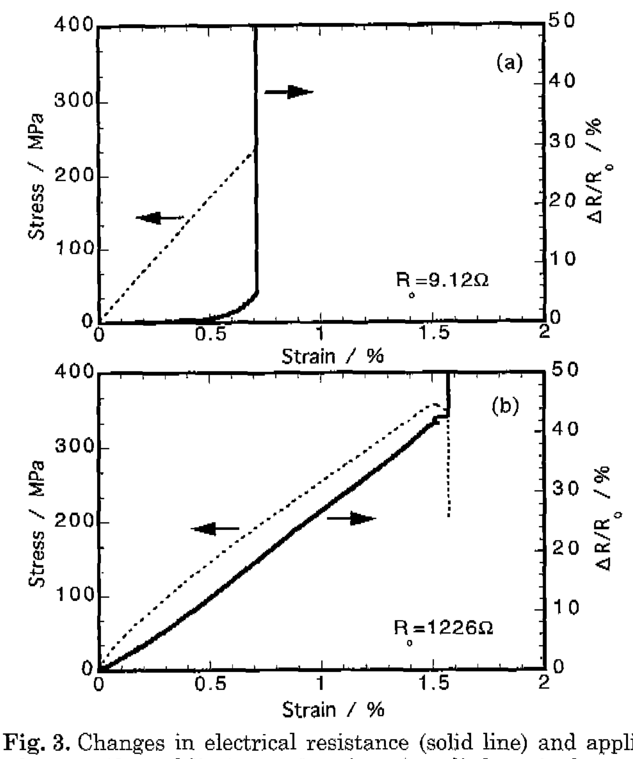


Fig. 3. Changes in electrical resistance (solid line) and applied stress (dashed line) as a function of applied strain for tensile tests of (a) CFGFRP and (b) CPGFRP.

a slight change in resistance below 0.5% strain and an extremely large change around 0.7% strain; namely, the resistance of CFGFRP exhibited a non-linear response to applied strain as shown in Fig. 3(a). The initial resistance, R_0, for CPGFRP is higher than that for CFGFRP because of poor electrical contact between carbon particles in the percolation structure. It can be seen that the CPGFRP exhibited a linear increase in resistance from the small strain region up to fracture in the composite as shown in Fig. 3(b). The change in resistance to applied strain commenced at around a strain of 0.01% (100 μm strain) or less. Comparing Fig. 3 (a) with (b) shows that the CPGFRP possesses higher sensitivity in the small strain region and has a wider detectable strain range than CFGFRP.

These results mean that the percolation structure of carbon particles enables more sensitive and versatile monitoring than the carbon fibers. The sensitivity of CPGFRP was attributed to local breakage of electrical contacts and/or to the rearrangement of the particle structure under applied tensile stresses.

Fig. 4 shows resistance change and applied strain as a function of time in a cyclic loading test for CPGFRP. Specimens were loaded and unloaded cyclically under a progressive increase in strain. It can be seen that the resistance change corresponds well with change in strain. It is worthy to note that the resistance change decreased but did not completely return to zero in the unloaded state. The residual resistance remained after the application of above 0.2% strain, and increased with maximum previous strain applied. Fig. 5 shows the maximum resistance change during loading, indicated by ΔR_{max}, and the residual resistance change after unloading, denoted by ΔR_{res}, as a function of maximum previous strain applied. The change in residual resistance correlated closely with previous maximum strain, indicating that the CPGFRP has the ability to memorize maximum deformation or damage inflicted in the past.

The microstructures of CFGFRP after loading-unloading cycles inducing 0.6% strain and 2.1% strain were observed by SEM as shown in Fig. 6(a) and (b), respectively. It can be seen that the number of microcracks in the matrix increased with increase in applied strain. Although the CPGFRP returned elastically to its original shape after loading, the percolation structure did not return reversibly to its initial state because of microcrack formation in the matrix. This irreversible change in percolation structure of the conductive phase is partly responsible for the appear-

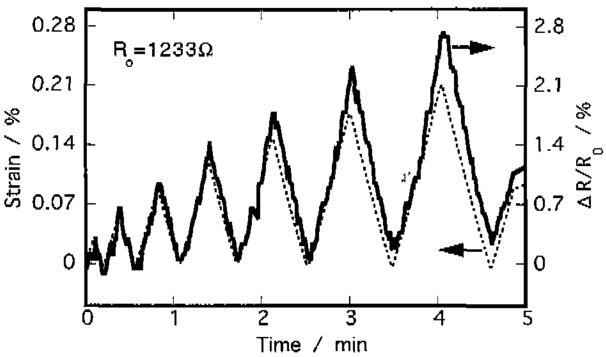


Fig. 4. Changes in electrical resistance (solid line) and applied stress (dashed line) as a function of time during a cyclic loading test for CPGFRP.

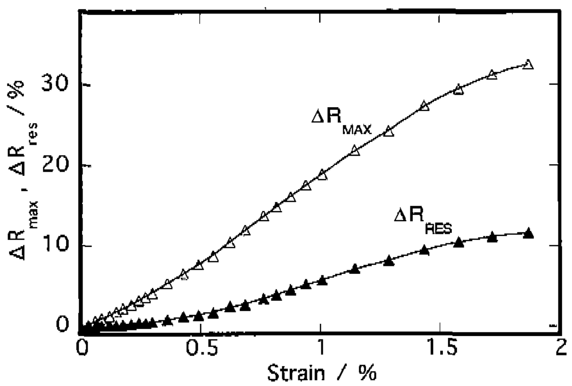


Fig. 5. Maximum resistance change at loading state (ΔR_{max}) and residual resistance change at unloading state (ΔR_{res}) as a function of applied strain during a cyclic loading test for CPGFRP.

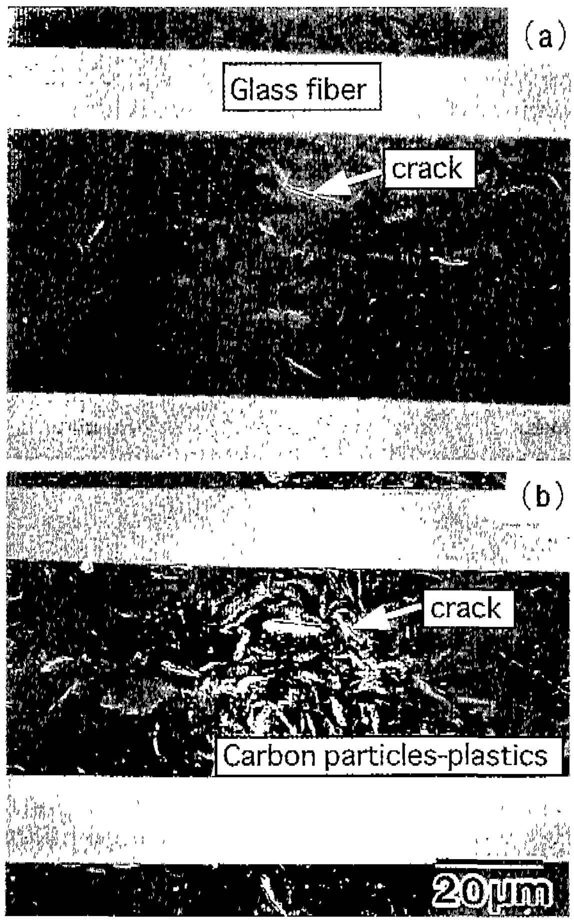


Fig. 6. SEM photographs of CPGFRP (polished side-section of conductive part) after unloading at (a) 0.6% strain and (b) 2.1% strain during a loading test.

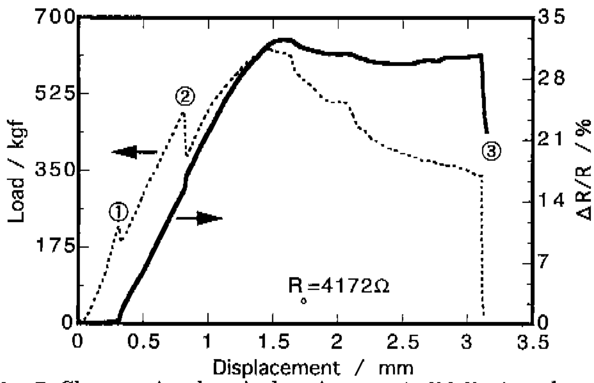


Fig. 7. Changes in electrical resistance (solid line) and applied load (dashed line) during a bending test for CPGFRP embedded in concrete.

ance of residual resistance over a wide strain range.

3.2. CPGFRP composites embedded in concrete

The CPGFRP was embedded in the tensile side of concrete specimens in order to demonstrate its fracture detection ability. Fig. 7 shows the applied load and the resistance change of CPGFRP as a function of displacement during bending. The load-displacement curve shows discontinuous changes at points ① and ②, which correspond to crack formation and crack propagation in the concrete specimen, respectively. It can be seen that the resistance of CPGFRP started increasing simultaneously with crack formation and a discontinuous change in resistance appeared in response to crack propagation. On removing the load, the resistance dropped abruptly (point ③) as a result of the release of residual elastic strain in the composite, re-connecting some of the particles in the percolation structure. As the carbon powder structure did not return completely to its original state, however, a large residual resistance remained.

These results demonstrate that the CPGFRP has the ability to detect minute crack formation/propagation and record the loading history of concrete based structural materials.

3.3. Fracture detection ability of CMC

The dependence of applied stress and change in resistance on strain for CMC are shown in Fig. 8. The composite was made conductive by adding W fiber or TiN particles. Both composites displayed non-linear responses of resistance change to applied strain and fractured at about 0.2% strain. The CMC I with W fiber showed a slight change in resistance in the small strain region and then a drastic change when it finally fractured. The CMC II containing TiN particles exhibited a distinct change in resistance from small strain to fracture of the composite as shown in Fig. 8(b). These results also suggest that the change in electrical conductance of the particle percolation structure in a ceramic matrix is advantageous for the detection of fracture in CMC materials.

Fig. 9 presents the variation of resistance for the CMC II during a cyclic bending test as a function of the number of

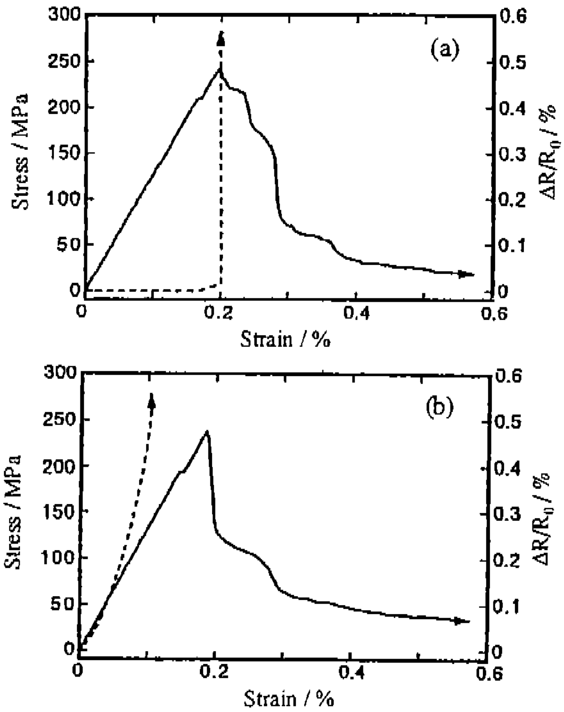


Fig. 8. Changes in electrical resistance (dashed line) and applied load (solid line) as a function of applied strain in bending tests for (a) CMC I and (b) CMC II.

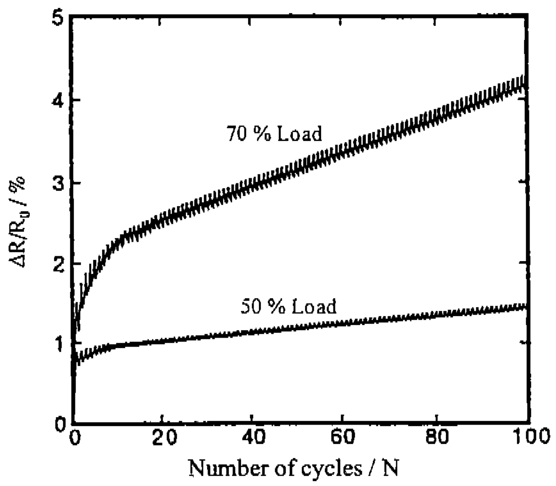


Fig. 9. Change in electrical resistance of CMC II during cyclic bending tests.

cycles. The stress applied during loading was kept constant at 50% and 70% of maximum stress (250 MPa) for the CMC. The residual resistance after unloading rapidly increased up to 10 cycles. SEM photographs (Fig. 10) showed that this was due to the microcracks in the matrix disconnecting the conduction paths formed by TiN particles. It should be noted that the residual resistance increased proportionally with increasing number of loading cycles after 20 cycles, which was more distinct for cyclic loading under 70% of maximum stress.

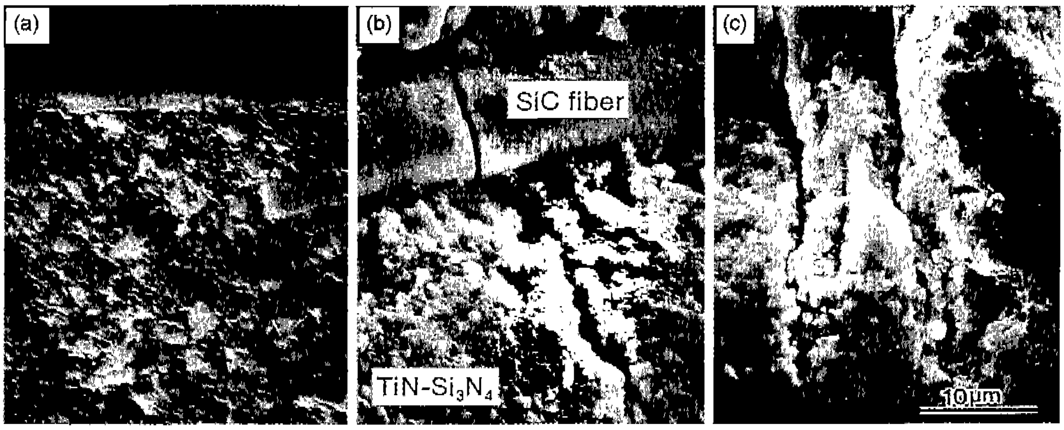


Fig. 10. SEM photographs of fractured surface of conductive parts of CMC II. (a) before test, (b) after bending and (c) after cyclic bending (100 cycles).

This result suggests that the CMC containing TiN particles has the ability to reveal cumulative fatigue of the composite by measurement of the residual resistance.

IV. Conclusions

The fracture detection ability of two types of conductive composites has been investigated. The results obtained were as follows:

1. Compared with composites containing conductive fiber, composites containing conductive particles with an interconnected percolation structure were found to possess the qualities necessary for detection in situ of fracture or damage in the composites. In particular, CPGFRP enabled the detection of micro-crack formation and propagation in concrete-based materials.

2. After unloading, the CPGFRP showed a residual resistance, which increased with maximum previous strain. The CMC materials also retained a residual resistance.

3. The fracture detection ability of these composites was easily demonstrated by simple monitoring of resistance. It is expected that conductive particles with a percolation structure can be introduced to other composite systems and detectability of fracture or damage can be extended.

Acknowledgment

The authors gratefully thank M. Takada of Nippon Tokushu Goukin Co., Y. Okuhara, H. Yanagida of the Japan Fine Ceramics Center, for their assistance in resistance measurements and helpful discussions.

References

1. N. Muto, H. Yanagida, T. Nakatsuji, M. Sugita and Y. Ohtsuka, "Preventing Fatal Fractures in Carbon-Fiber-Glass-Fiber-Reinforced-Plastic Composites by Monitoring Change in Electrical Resistance," *J. Am. Ceram. Soc.*, **76**(4), 875-79 (1993).
2. H. Matsubara, M. Takada and H. Yanagida, "Application to Structural Materials of Intelligent Concept," *Chemistry and Chemical Industry*, **49**(1), 40-42 (1996).
3. Y. Arai, S.-G. Shin, M. Takada, M. Tsujii, H. Matsubara and H. Yanagida, "Self-Diagnosis of Fiber Reinforced Composites using Electrical Conductivity," *Ceram. Mater. Syst. Compos. Struct.*, **99**, 511-516 (1998).
4. M. Takada, S.-G. Shin, H. Matsubara and H. Yanagida, "Fracture Detection Function of the Composite of Glass Fiber, Plastics and Carbon Powder," *J. Jpn. Soc. Compos. Mater.*, **25**, 225-230 (1999).
5. H. Yanagida and H. Matsubara, "Design and Application of Self-Diagnosis Materials," *J. Jpn. Soc. Mech. Eng.*, **102**, 68-71 (1999).
6. S.-G. Shin, H. Matsubara, Y. Okuhara, H. Yanagida and N. Takeda, "Self-Monitoring of FRP using Electrical Conductivity of Carbon Phase," *Proc. 6th Jpn. Inter. SAMPE Symp.*, **2**, 995-998 (1999).
7. S.-G. Shin, H. Matsubara and M. Takada, "Change of Electrical Resistance in Electrically Conductive FRP Composites under Bending Load," *J. Ceram. Soc. Jpn.*, to be published.