

## Self-Diagnosis of the Composite Containing Electrically Conductive Phase

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The electrically conductive fiber reinforced plastics (FRP) composites and ceramics matrix composites (CMC) have been designed and fabricated in order to introduce the self-diagnosis function in these materials. The electrical conductivity was achieved by adding carbon and TiN as a conductive phase into the FRP and the CMC, respectively. The resistance of the conductive FRP containing carbon particles had linear response to the strain and high sensitivity in the wide strain range. The load-unload cycle induced residual resistance in the FRP with carbon particles at unloading state, which increased with applied maximum stress or strain. The FRP with carbon particles embedded in mortar specimens diagnosed micro-crack formation and propagation. The CMC materials indicated not only fine response of resistance to the applied strain but also resistance increase with increasing count of load-unload cycle during cyclic load test. These results suggest that these composite materials with fracture detection function can be brought to many industrial applications concerning the self-diagnosis of deformation and damage in structural materials.

Key words: self-diagnosis, composite, resistance, FRP, ceramics

### 1. INTRODUCTION

The overall deterioration of structural materials due to aging or disaster such as earthquake has resulted in the expansion of demand for non-destructive evaluation of the health condition. Recently, the health monitoring technique utilizing the structural materials with function to diagnose own condition, so-called self-diagnosis materials or intelligent materials, has been attracting attentions. Some methods of the fracture detection in the fiber reinforced plastics (FRP) which were applied to structural materials as reinforcement have been proposed.<sup>1)-7)</sup> Muto et al. have reported that the changes in electrical resistance in carbon-fiber-glass-fiber-reinforced plastics (CFGFRP) were applied to monitor the fatal fracture.<sup>1)</sup> Although the CFGFRP showed appreciable resistance change in the strain range above 0.7-1.5 % due to fracture in carbon fiber, the detectable strain level is relatively large considering the diagnosis of local damage in structural materials. An acoustic emission method or fiber optics sensor have been researched with the aim of applying to health monitoring, but these systems are expensive. It is therefore necessary to develop the self-diagnosis materials showing higher sensitivity in the small strain region and to achieve simple system.

In the present work, the electrical conductive composites having continuous structure of conductive particles, so-called percolation structure, were designed and fabricated. The structure was formed in

the FRP composites and the ceramics matrix composites (CMC). These self-diagnosis functions were evaluated from the changes in resistance with applied strain.

### 2. EXPERIMENT

Figure 1 is the 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 (pitch-based CF, Toho Rayon Co., Ltd. BESFIGHT UM63) introduced in replacement of a part of glass fiber forms conductive path and enhances strength in the long direction of composite. The carbon particles (graphite, SEC Co., Ltd. SPG5) dispersed in a part of matrix create conductivity due to the percolation structure. The composite containing carbon particles was indicated by carbon-particles-glass-fiber-reinforced plastics (CPGFRP).

Figure 2 is the schematic drawings of structural design for CMC materials. The composites were fabricated by the filament winding method using  $\text{Si}_3\text{N}_4$  particles (Ube Industries Co., Ltd. SN-COA) as the matrix and SiC fiber (Nippon Carbon Co., Ltd. NL-401) as the reinforcement for strengthening or toughening. A portion of the fibers was replaced with W wire (Nippon tungsten Co., Ltd.  $\phi$  30  $\mu\text{m}$ ). Conductive particles of  $\text{Si}_3\text{N}_4$ -40%TiN (Japan New

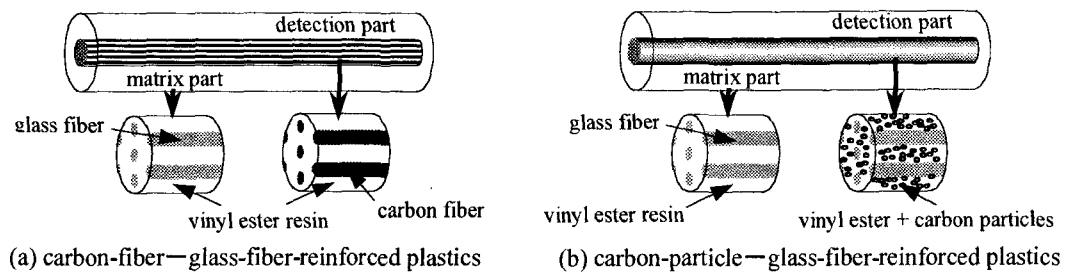


Fig. 1 Schematic drawings of the structural design for CFGFRP (a) and CPGFRP (b).

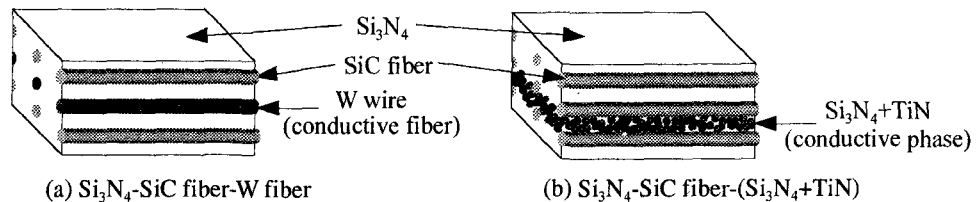


Fig. 2 Schematic drawings of the structural design for CMC containing W wire (a) and TiN particles (b).

Metals Co., Ltd.) were dispersed in a part of the matrix. These composites were hot pressed under 40 MPa at 1773 K in N<sub>2</sub> atmosphere for one hour. Sintered specimens were cut into bars of 3×4×45 mm for bending test pieces, with either conductive wire or particles located 0.5 mm from tensile side.

The self-diagnosis functions of these materials were evaluated by simultaneous measurement of stress and electrical resistance change as a function of applied strain in tensile loading test or in bending test. 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. The types of loading were in two ways; 1) normal tensile or bending test until specimen fracture, 2) cyclic loading-unloading test below the maximum stress level.

### 3. RESULTS AND DISCUSSION

#### 3-1 Self-Diagnosis Function of FRP composite

Figures 3 shows the changes in electrical resistance and applied stress as a function of applied strain in tensile test for CFGFRP and CPGFRP.<sup>7)</sup> The stresses in both specimens increased linearly in proportion to the strains until fracture of carbon fiber or glass fiber. The CFGFRP indicated slight change in resistance below 0.5 % strain and tremendous change around 0.7 % strain; namely, the resistance of CFGFRP exhibited 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 slight electrical contact between carbon particles in percolation structure. It can be seen that the CPGFRP indicated linear increase in resistance from small strain region to fracture in the composite as shown in Fig. 3 (b). The response of the resistance to applied strain appeared around the strain of 0.01 % (100  $\mu$  strain) or

less. Comparing Fig. 3 (a) with (b) has shown that the CPGFRP possesses higher sensitivity in the small strain range and wider detectable strain range than CFGFRP. These results mean that the percolation structure of carbon particles enables more sensitive and pliable monitoring than structure of carbon fiber. The fine characteristics of CPGFRP were attributed to

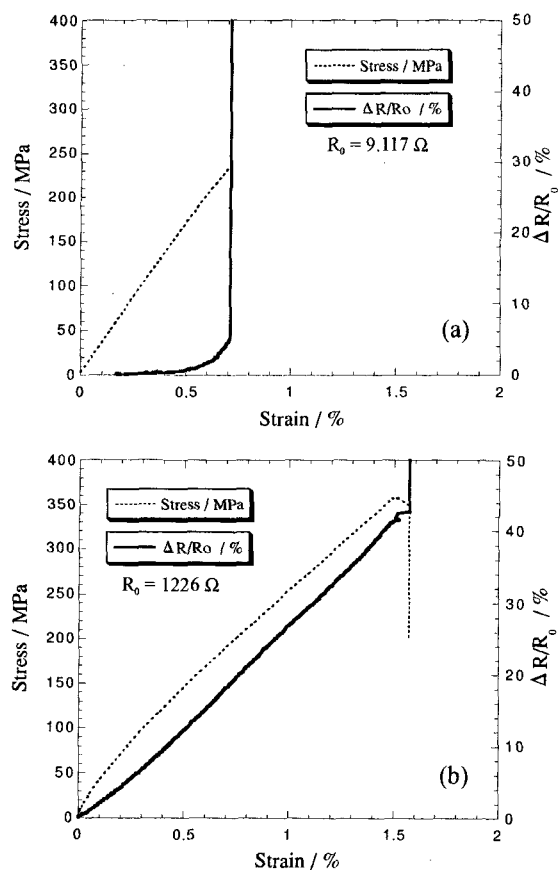


Fig. 3 Changes in electrical resistance (solid line) and applied stress (dashed line) as a function of applied strain for tensile test of CFGFRP (a) and CPGFRP (b).

local break in electrical contact or to the rearrangement of the percolation structure under applied tensile stresses.

Figure 4 shows resistance change and applied strain as a function of time in cyclic loading test for CPGFRP.<sup>7)</sup> Specimens were loaded and unloaded cyclically under a progressive increase in strain. It can be seen that the resistance change corresponded well with change in strain. It is worthy of notice that the resistance change decreased but did not completely return to zero value at unloading state. The residual resistance appeared after the application of above 0.2 % strain, and increased with maximum previous strain applied in the past. Figure 5 shows the maximum resistance change during loading indicated by  $\Delta R_{max}$  and the residual resistance change after unloading denoted by  $\Delta R_{res}$  as a function of maximum strain applied in the past.<sup>7)</sup> The change in residual resistance correlated closely with previous maximum

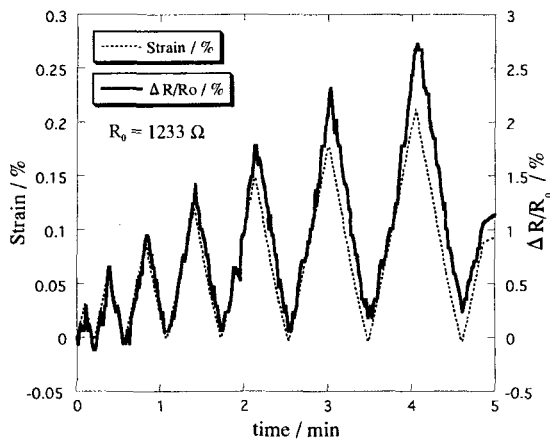


Fig. 4 Change in resistance (solid line) and applied strain (dashed line) as a function of time in cyclic loading test for the CPGFRP.

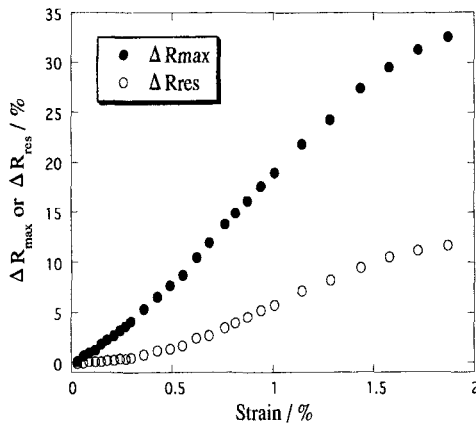


Fig. 5 Maximum resistance change at loading state and residual resistance change at unloading state as a function of applied strain in cyclic loading test for the CPGFRP.

strain, indicating that the CPGFRP has the ability to memorize maximum deformation or damage inflicted in the past. It seems likely that this phenomenon is due in part to the change in percolation structure. Although the elongation of CPGFRP removed elastically after unloading, the percolation structure must have not returned completely to initial state because of the crack formation in conductive path or the rearrangement of percolation structure.

### 3-2 Application of Self-Diagnosis FRP composite

The FRP containing carbon particles was embedded in tensile side of mortar specimens in order to demonstrate the self-diagnosis function. Figure 6 shows the applied load and the resistance change of CPGFRP as a function of displacement in bending test.<sup>3)</sup> The load-displacement curve indicated discontinuous change at the point of ① and ②, which corresponded to crack formation and propagation in the mortar specimen respectively. It can be seen that the resistance of CPGFRP started increasing simultaneously with crack formation and discontinuous change in resistance appeared in response to crack propagation. The residual resistance was observed in the FRP material after unloading at the point of ③. These results indicate that the CPGFRP has the ability to estimate minute crack formation/propagation and loading history in concrete and cement based structural materials.

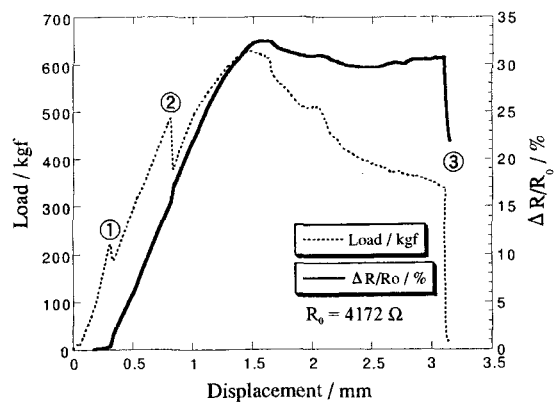


Fig. 6 Changes in resistance (solid line) and applied load (dashed line) in bending test for CPGFRP embedded in mortar specimen.

### 3-3 Self-Diagnosis Function of CMC

The dependence of applied stress and change in resistance on strain for CMC are shown in Fig. 7.<sup>3)</sup> The electrical conductivity was achieved by adding W wire or TiN particles. Both composites indicated non-linear response of resistance change to applied strain and fractured at about 0.2 % strain. The CMC with W wire showed slight change in resistance in the small strain

region and then drastic change with own fracture. The CMC containing TiN particles exhibited distinct change in resistance from small strain to fracture in the composite as shown in Fig. 7 (b). These results suggest that the change in electrical characteristics of percolation structure in a matrix of ceramics is also advantageous to diagnose a damage for CMC material.

Figure 8 presents the variation of resistance for the CMC with TiN particles in cyclic bending test as a function of number of cycles.<sup>3)</sup> The stress applied at lording state 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. It should be noted that the residual resistance proportionally increased with increasing count of loading cycles after 20 cycles, which was distincter for cyclic loading under 70 % of maximum stress. This result suggests that the CMC containing TiN particles have the ability to diagnose a cumulative fatigue for the composite by estimation of the residual resistance.

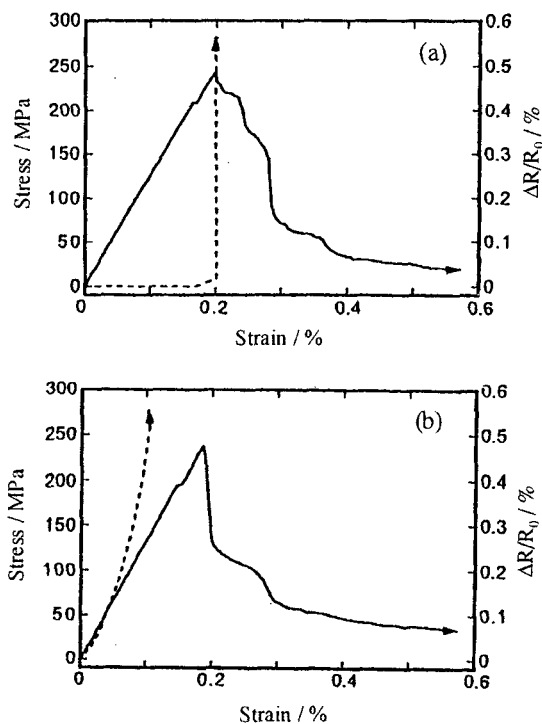


Fig. 7 Changes in electrical resistance (dashed line) and applied stress (solid line) as a function of applied strain in bending test for CMC with W wire (a) and with TiN particles (b).

#### 4. CONCLUSION

The self-diagnosis functions for several types of conductive composites have been investigated. Compared with the composites including conductive fiber or wire, the composites containing percolation

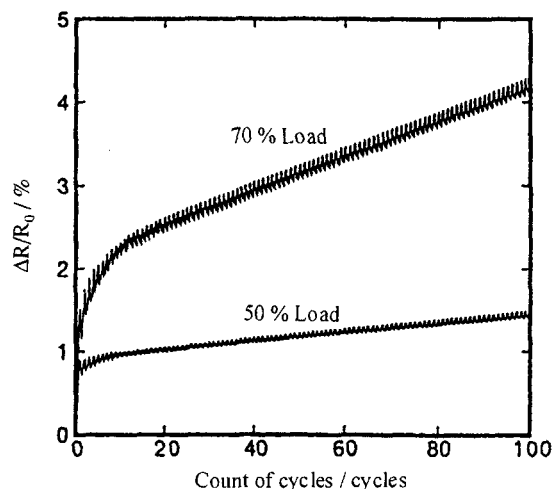


Fig. 8 Change in resistance of CMC containing TiN particles in cyclic bending test.

structure were found to possess the advantages to diagnose deformation or damage in the composites by themselves. The self-diagnosis function of the FRP composites containing carbon particles in particular realized the detection of micro-crack formation and propagation in cement based materials. Moreover, the FRP composites after unloading showed residual resistance, which increased with maximum strain applied in the past. The CMC materials also indicated residual resistance, which increased proportionally with increasing count of loading cycles under constant applied stress. It should be noted that these self-diagnosis functions can be easily obtained by simple monitoring of resistance. It is expected that the percolation structure has the ability to apply in other composite systems and the range of possible applications for self-diagnosis can be extended.

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