JOURNAL OF MATERIALS SCIENCE **39** (2004) 2573 – 2575

Electrical resistance of Al_2O_3 fiber reinforced RuO_2/glass hybrid composites during tensile loading

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The early detection of fracture or damage in concrete structures is very important factor for their safe use and maintenance. Damage sensing is conventionally performed using attached or embedded damage sensors, such as optical fibers, acoustic sensors and strain gages. Recently, studies of self-diagnosis composites that detect damage *in situ* have been reported [1–4]. These typically utilize polymers containing carbon fibers or carbon particles as conductive composites for self-diagnosis of damage by measuring the change of electrical resistivity. However, electrical resistance change of CFRP (carbon fiber reinforced plastics) composites or carbon fiber dispersed composites does not provide high enough sensitivity because the conductive carbon fibers undergo large elongation and the matrix is a non-brittle epoxy resin. In general, the electrical resistance of self-diagnosis composites depends largely on the nature of the electrically conductive material. Self-diagnosis composites consisting of a brittle matrix and electrically conducting additives are known to show a large variation in electrical resistance during applied loading [5, 6]. We note that RuO_2 reveals metal-like electrical conduction with a room temperature resistivity, ρ_m, of 3.4×10^{-7} Ωm [7] and has a superior electrical conduction to carbon fiber and carbon powder [1, 8]. In addition, formation of microcracks or fracture in the glass matrix occur be easily enough even at low stresses because the glass matrix is very brittle. Consequently, we have designed conductive oxide dispersed hybrid composites consisting of Al_2O_3 fibers as reinforcement, RuO_2 as a conductive material and glass as a brittle matrix. Here we report the development of these self-diagnosis composites and evaluate their damage sensitivity by measuring their electrical resistance changes during tensile test.

In this work, glasses in the Pb-Si-B-Al-O system with low softening temperatures are used to prevent degradation of the reinforced Al_2O_3 fiber during sintering. Conductive pastes made from RuO_2 particles and glass powders co-mixed in an organic solvent were used. The chemical composition of the conductive paste is given in Table I. Al_2O_3 fibers with 300 mm in length were dipped into these conductive pastes. Cylindrical green bodies containing 10 vol% Al_2O_3 fibers embedded in a RuO_2/glass matrix were thus formed. After drying at 130 °C for 2 h, the conductive green bodies were sintered at 850 °C for 30 min. Electrical copper wires were connected at both ends of the sintered conductive bodies and silver paste used to ensure electric contact between both ends of the sintered bodies for good electrical coupling. To provide insulation and protect against any handling damage, glass fiber reinforced epoxy resins were coated on surface of the sintered specimens, and dried at 130 °C. Afterward, ring-type steel tabs with an outer diameter of 12 mm, inner diameter of 10 mm and a length of 50 mm were applied to both ends of the specimen for clamping during tensile tests. The ring tabs were filled with expansive cement to bond the tabs and specimen together solidly. The glass fiber/epoxy coated conductive hybrid specimens ultimately had a diameter of 2.2 mm and a length of 200 mm. Electrical resistance was measured by a two-terminal method using an electrical resistance analyzer. Tensile tests were performed using an MTS 631 tensile test machine with a constant crosshead speed of 1 mm/min. Two types of applied load were used during tensile testing: (1) a linear increase to a maximum load, and (2) cyclic loading at 500 N for up to 1000 cycles. The strain and electrical resistance of the composites were measured simultaneously with increasing tensile load.

Fig. 1 shows a typical microstructure of the cross-sectional surface of a RuO_2 dispersed glass conductive composite. The dark contrast circles are the embedded Al_2O_3 fibers, whereas the surrounding gray area is the RuO_2 containing glass matrix. We could not detect any reaction between the Al_2O_3 fibers and glass matrix, and there is good bonding between the two phases. The RuO_2 particles dispersed into glass matrix provide electrical conduction via points of contact between them.

Fig. 2 shows the relationship between stress, strain and electrical resistance change obtained during linear tensile loading up to failure. The electrical resistance change is defined as $\Delta R/Ro$, where ΔR and Ro are the electrical resistance increase and initial electrical resistance, respectively. An increase in electrical resistivity during applied loading is a clear indicator of fracture or damage to the specimen with increasing strain. The stress curve is linear at strains of <0.1% and thus in the elastic region. The stress behavior shows a non-linear stepwise increase between 0.1–0.7% strain due to onset of fracture of the glass matrix. The change in electrical resistance is remarkable even in the low strain range (<0.5% strain). The large electrical resistance change in the low strain region is attributed to microbreakage or deformation of conduction paths between RuO_2 particles due to the brittle fracture of the glass matrix. The electrical resistance increased approximately linearly with increasing strain up to 1.3% strain. After about

TABLE I Chemical composition of RuO_2 dispersed glass composites

Chemical composition (wt%)				
PbO	SiO_2	B_2O_3	Al_2O_3	RuO_2
38	26	7	2	27

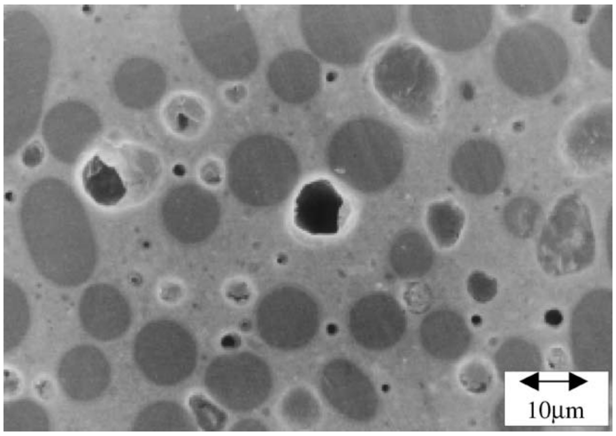

Figure 1 SEM micrograph of a RuO_2 dispersed glass matrix composite.

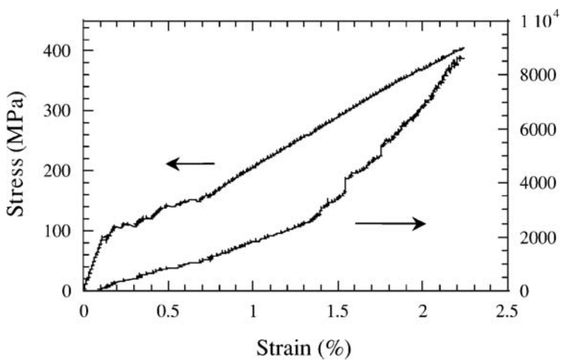

Figure 2 Electrical resistance change and stress of a composite as a function of applied strain during tensile test.

1.3% strain the electrical resistivity no longer varied linearly, which can be related to fracture of the fibers, with the electrical resistance change reaching a value of over a thousand percent. In these specimens, the remarkable increase in electrical resistance is caused by the increase in fracturing of the brittle glass and the consequent decrease in the number of conductive paths between adjacent RuO_2 particles.

Fig. 3 shows the tensile load, total electrical resistance and residual electrical resistance obtained in real time during cyclic tensile loading at 500 N, which corresponds to about 30% of the breaking load. The load was relaxed to zero from the maximum load for each cycle as shown in Fig. 3a.

As seen in Fig. 3b, the electrical resistance increased upon loading and decreased upon unloading in a repetitive wave pattern for each cycle. The maximum electrical resistance of each cycle increased gradually with increasing cycle number. This is attributed to the decrease in number of electrically conductive paths. Beyond about 300 loading cycles, the electrical resistance rises in a stepwise manner. This is related to glass matrix fracture as well as fiber fracture.

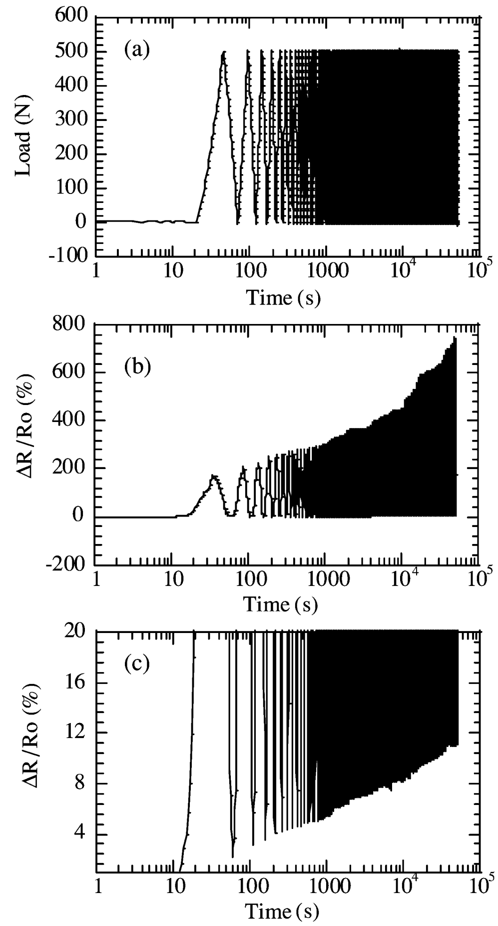

Figure 3 Residual electrical resistance change vs. time for 1000 load cycles at 500 N: (a) cyclic load, (b) total resistance change, and (c) residual resistance change.

Fig. 3c shows a magnified view of the unloading stage of the electrical resistance change of Fig. 3a. The minimum electrical resistance of each cycle continuously increased upon unloading, i.e., the electrical resistance increased irreversibly each cycle after unloading and did not return to zero (starting point of the first cycle). In other words, a residual electrical resistance remained after unloading. The irreversible increase in electrical resistance after the first loading cycle is due to the irreversible increase in the degree of separation between RuO_2 particles forming the electrically conductive paths. If applied to health monitoring of civil structures, this irreversible electrical resistance portion can be used as an indication of the amount of damage in the structure. Consequently, our results show that RuO_2 containing glass hybrid composites provide sensitive self-diagnosis ability for damage detection that utilizes electrical resistance changes during tensile loading.

Acknowledgment
The authors would like to acknowledge the financial support of the New Energy and Industrial Technology Development Organization (NEDO) of the Japanese government for this work.

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Received 2 October
and accepted 21 October 2003