

## NONDESTRUCTIVE EVALUATION OF BRITTLE-MATRIX COMPOSITES DURING LOADING BY ELECTRICAL RESISTANCE MEASUREMENT

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### ABSTRACT

Brittle-matrix composites with matrices that were not totally electrically insulating were found to change electrical resistance upon loading such that reversible strain caused the resistance to change reversibly and damage caused the resistance to change irreversibly. This electromechanical behavior allows dynamic strain and fatigue damage to be monitored simultaneously without the use of embedded or attached sensors, as the composites are themselves sensors. This new technique has been demonstrated for carbon-matrix and cement-matrix composites. The carbon-matrix composite with continuous carbon fibers was particularly effective for damage sensing; even damage in the first cycle of tensile loading at a stress amplitude of just 20% of the fracture stress was detected. The cement-matrix composite with short carbon fibers (0.2-0.5 vol.%) was particularly effective for strain sensing; the gage factor (fractional change in resistance per unit strain) was as high as 500, compared to 2 for resistive strain gages.

### INTRODUCTION

Although crack deflection by fibers in a brittle-matrix composite makes the composite less brittle than the matrix itself, the composite remains brittle from the point of view of a design engineer, who demands durability and reliability. One way to alleviate this problem is to continuously or periodically monitor (i.e., nondestructively evaluate) the structural health of the composite structure while the structure is in use. In addition to monitoring the damage, the dynamic strain can be monitored in real time for the purpose of structural control, which is important for smart structures. The monitoring of both damage and dynamic strain has the additional function of providing information on which part of which load cycle (not necessarily periodic) damage occurs.

The monitoring mentioned above is conventionally achieved by embedding into the composite or attaching on the composite one or more

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sensors, such as piezoelectric sensors, magnetostrictive sensors, piezoresistive sensors, optical fiber sensors, etc., or by acoustic emission detection. However, the sensors suffer from high cost, poor durability, limited sensing volume and, in the case of embedded sensors, degradation of the mechanical properties of the composite. Acoustic emission is not sensitive to slight damage or dynamic strain. Instead of using embedded or attached sensors, this work uses the composite itself as the sensor. In other words, the composite is both the structural material and the sensor. Such self-monitoring structural materials do not suffer from the problems described above for the embedded or attached sensors.

This paper addresses two kinds of self-monitoring brittle-matrix composites. They are a carbon-matrix composite with continuous carbon fibers and concrete with short carbon fibers. In both cases, the matrix is not totally electrically insulating and the fibers are electrically conducting. As a result, the electrical resistance of the composite changes upon loading. The scientific origin of the electromechanical effect differs between the two kinds of composite.

## CARBON-CARBON COMPOSITE

Carbon-carbon composites with continuous carbon fibers and a carbon matrix are used for high-temperature aerospace structures and biomedical implants, due to the high-temperature resistance and biocompatibility of carbon. The carbon matrix, though much more high-temperature resistant than a polymer matrix, is much more brittle than a polymer matrix. This brittleness makes carbon-carbon composites prone to matrix cracking. As a result, there is a need for monitoring the condition or health of a carbon-carbon composite structure while the structure is in use, in order to minimise hazards. This monitoring is conventionally conducted by acoustic emission, which is capable of detecting substantial cracking, but not slight cracking. Exceptional sensitivity to even slight damage can be obtained by using the carbon-carbon composite itself as the damage sensor to monitor the composite's own damage (i.e., to self-monitor the damage), using the increase in electrical resistivity as a measure of the irreversible composite damage [1]. The high sensitivity to damage is due to the high conductivity of the carbon matrix, compared to the cement and polymer matrices, and the importance of matrix cracking in the mechanism for damage in a carbon-carbon composite.

Figure 1 shows the stress (curve (a)) and the fractional DC resistance increase ( $\Delta R/R_0$ ) (curve (b)) obtained during static tension up to failure for a carbon-carbon composite having two-dimensionally ( $90^\circ$ ) woven fibers and a heat treatment temperature of  $2000^\circ\text{C}$ , with the resistance and stress in the direction of one of the two perpendicular sets of fibers.  $\Delta R/R_0$  increased monotonically with strain, such that the increase was gradual (only slightly above the increase in  $\Delta R/R_0$  due to the changes in dimensions, curve (c) in Fig. 1) at low strains and abrupt at high strains.

Figure 2 shows  $\Delta R/R_0$  obtained during cyclic tension to a stress

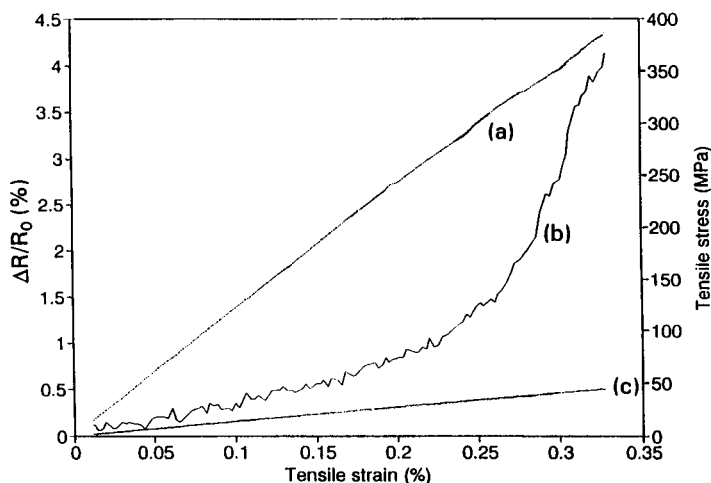


Fig. 1 Plots of (a) tensile stress vs. strain, and (b)  $\Delta R/R_0$  vs. strain, obtained simultaneously during static tension up to failure for a carbon-carbon composite. Curve (c) is the calculated  $\Delta R/R_0$  based on dimensional changes.

amplitude (360 MPa) equal to 94% of the breaking stress. The tensile strain was almost totally reversible. The irreversible strain was 0.040% at the end of the first cycle, and increased very slightly with increasing cycle number.  $\Delta R/R_0$  increased upon loading in every cycle, such that it irreversibly increased slightly after every cycle and the irreversible increase in  $\Delta R/R_0$  was particularly large for the first cycle, as shown in Fig. 2. At fatigue failure,  $\Delta R/R_0$  abruptly increased, such that  $\Delta R/R_0$  did not more rapidly increase irreversibly near the end of fatigue life. Fig. 3 shows the peak  $\Delta R/R_0$  values in a cycle as a function of cycle number throughout the fatigue life up to failure. The peak  $\Delta R/R_0$  increased with cycle number significantly during the first 500 cycles and gradually during all subsequent cycles up to failure. The small step increases in the peak  $\Delta R/R_0$ , for example at  $\sim 1350$  cycles, are not experimental artifacts but are attributed to damage occurring at those cycle numbers, similar to the step increases observed for a continuous carbon fiber polymer-matrix composite under similar cyclic loading [2].

The reversible part of  $\Delta R/R_0$  is mainly due to reversible dimensional changes and correlates with reversible strain. The irreversible part of  $\Delta R/R_0$  is due to damage. Although the increase in irreversible strain and decrease in Young's modulus also indicate damage, the changes in these parameters are very small compared to the change in the irreversible part of  $\Delta R/R_0$ . The great sensitivity of the irreversible part of  $\Delta R/R_0$  to damage is also shown by the significant non-zero value of the irreversible part of  $\Delta R/R_0$  after merely the first cycle, even at a stress amplitude of just 20% of the fracture stress (Fig. 4).

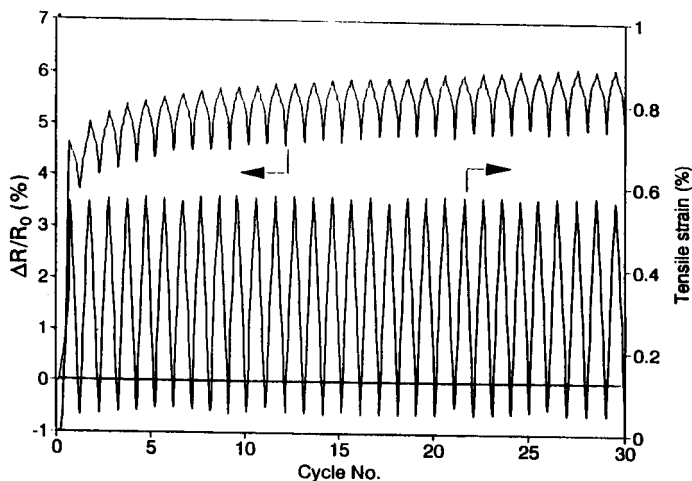


Fig. 2 Plots of  $\Delta R/R_0$  vs. cycle No. and of tensile strain vs. cycle No., obtained simultaneously during first cycle tension at a stress amplitude of 94% of the fracture stress for a carbon-carbon composite.

However, the incremental rise in irreversible  $\Delta R/R_0$  beyond  $\sim 500$  cycles was small. The composite damage probably involved fiber-matrix interface weakening, matrix cracking and fiber breakage; these origins of damage could not be distinguished through the experimental technique used. Nevertheless, the increase of the irreversible part of  $\Delta R/R_0$  as cycling progressed provided a continuous indication of the extent of damage. That the reversible part of  $\Delta R/R_0$  also increased with cycling and that an abrupt increase of the irreversible part of  $\Delta R/R_0$  is associated with an abrupt increase in the reversible part of  $\Delta R/R_0$  suggest that the reversible part of  $\Delta R/R_0$  is partly associated with a phenomenon which intensifies as damage increases, although it is mostly associated with dimensional changes. This phenomenon may be reversible crack opening during tension, as cracks are expected to increase in size and/or density as cycling progresses. This interpretation is consistent with the observation that an abrupt increase in the reversible part of  $\Delta R/R_0$  is associated with an abrupt increase in reversible strain and that the abrupt increase in reversible strain occurs at stress amplitudes beyond the range in which the reversible strain is linear in relation to the stress amplitude. As a result of this phenomenon, the gage factor (reversible  $\Delta R/R_0$  per unit reversible strain) increases slightly with cycle number. The dependence of the gage factor on the cycle number complicates the practical use of the carbon-carbon composite as a strain sensor.

## CONCRETE

Carbon fiber reinforced concrete is attractive for civil structures due to

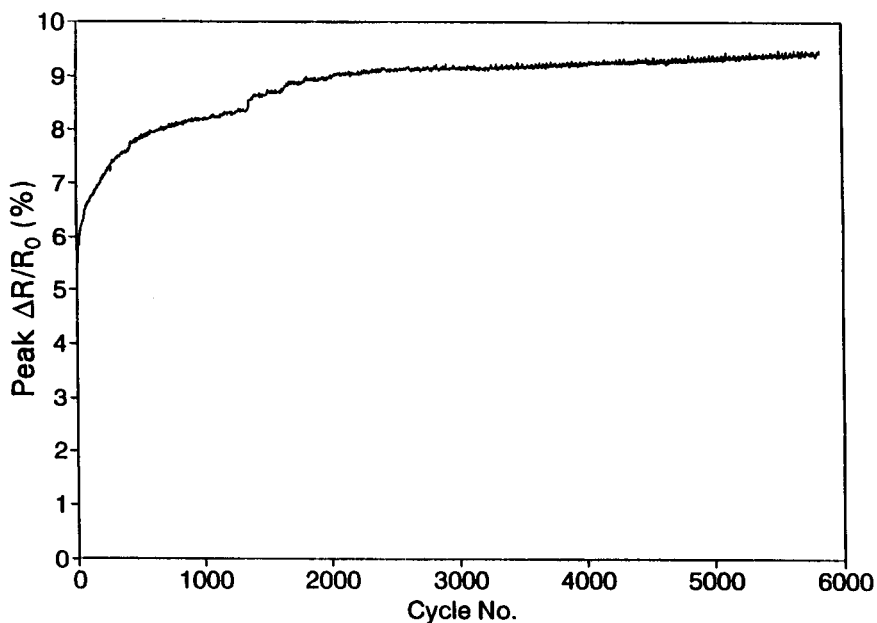


Fig. 3 Variation of the peak  $\Delta R/R_0$  with cycle No. throughout the entire fatigue life at a stress amplitude of 94% of the fracture stress for a carbon-carbon composite.

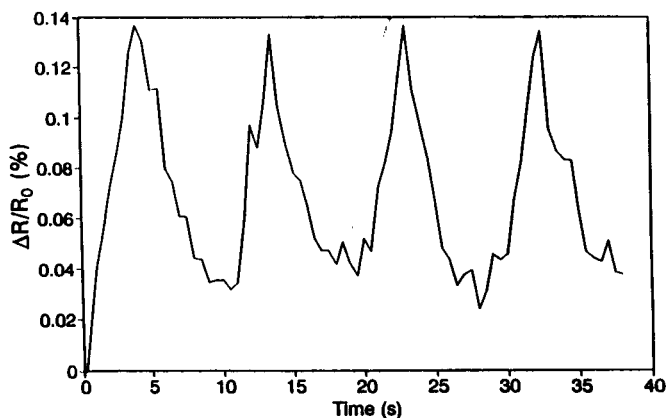


Fig. 4 Plot of  $\Delta R/R_0$  vs. time during the first four cycles at a stress amplitude of 20% of the fracture stress for a carbon-carbon composite.

its high tensile and flexural strengths, high tensile ductility, high flexural toughness, low drying shrinkage [3-13], strain sensing ability [14-22] and low cost. This section focuses on its strain sensing ability. Damage sensing ability exists in this material, but it is not as powerful as the strain sensing ability.

Self-monitoring in concrete was achieved with concrete containing short, electrically conducting microfibers, preferably carbon fibers, in an amount as low as 0.2 vol.%. The DC volume electrical resistivity of the concrete changes reversibly upon reversible strain, such that the fractional change in electrical resistance per unit strain (i.e., the strain sensitivity or the gage factor) reaches 500 (extraordinarily high compared to conventional strain gages). The effect has been observed in cement paste, mortar as well as concrete, under tension, compression and flexure, though the effect is more pronounced in cement paste and mortar than in concrete. The reversible electromechanical effect stems from the reversible increase in the contact electrical resistivity between fiber and matrix upon slight opening ( $< 1 \mu\text{m}$ ) of the crack which the fiber bridges (i.e., slight fiber pull-out), and the consequent reversible increase in the volume electrical resistivity of the cement-matrix composite [14-21].

The evidence in support of the above-mentioned origin of the self-monitoring ability of intrinsically smart concrete is summarized below.

1. The sensing ability was present when the fibers were conducting (i.e., carbon or steel) and absent when the fibers were non-conducting (i.e., polyethylene) [18].
2. The sensing ability was absent when fibers were absent [14,15,18,20].
3. The sensing ability occurred even at low carbon fiber volume fractions, which were associated with little effect of the fiber addition on the concrete's volume electrical resistivity [18,23,24].
4. There was no maximum volume electrical resistivity required in order for the sensing ability to be present [18].
5. The sensing ability was present when the carbon fiber volume fraction was as low as 0.2% (below the percolation threshold) [18,20].
6. Fracture surface examination showed that the fibers were separate from one another [18].
7. The fractional increase in electrical resistance ( $\Delta R/R_0$ ) upon straining essentially did not increase with increasing carbon fiber volume fraction, even though the increase in fiber volume fraction caused large decrease (by orders of magnitude) in the volume electrical resistivity [18].
8. The electrical resistance increased upon tension (fiber pull-out) and decreased upon compression (fiber push-in), except for the first compressive strain cycle at 7 days of curing. The resistance change during the first strain cycle is consistent with the need to weaken the fiber-matrix interface prior to fiber pull-out in case the interface was relatively strong to start with [18,19,21].
9. The presence of carbon fibers caused the crack height to decrease from

~ 100  $\mu\text{m}$ , as observed after deformation to 70% of the compressive strength [20].

10. The presence of carbon fibers caused the flexural toughness and tensile ductility of the composite to greatly increase [24,25].
11. The stress required for fiber pull-out in the short fiber composite was consistent with the shear bond strength between carbon fiber and cement paste, as obtained by single fiber pull-out testing [17].
12. The contact electrical resistivity between carbon fiber and cement paste increased during debonding [17].

Due to the small volume fraction of carbon fibers, the decrease of the concrete's volume electrical resistivity due to the fiber addition is small [18,23]. In order to help the fibers disperse, admixtures such as silica fume, methylcellulose and latex are used along with the fibers [23,24]. The fibers are the most inexpensive type of carbon fibers, namely those based on isotropic pitch.

Due to the high electrical contact resistivity between steel rebar and concrete [26] and the low ability of an electrical current applied on one surface of a concrete structure to penetrate the concrete to depths more than 1 mm, the presence of steel rebars in concrete does not interfere with the performance of the self-sensing concrete around it unless the electrical contacts are around the whole cross-section of the concrete (rather than on one side) and unless the concrete is inside a cage made of the rebars.

Figure 5 gives the fractional DC resistance increase ( $\Delta R/R_0$ ) during first cyclic compressive loading of mortar with 0.24 vol.% carbon fibers at a stress amplitude of 16 MPa, or a strain amplitude of  $8 \times 10^{-4}$ , which was within the elastic regime at 28 days of curing. (The compressive strength was 45 MPa). The resistance was in the stress direction, as measured by the four-probe method (i.e., the outer two probes for passing current and the inner two probes for measuring the voltage), with silver paint for the electrical contacts. Both stress and strain returned to zero at the end of each cycle. The  $\Delta R/R_0$  decreased during compressive loading in each cycle and increased during unloading in each cycle. This is due to fiber push-in during loading and fiber pull-out during unloading. At the end of the first cycle,  $\Delta R/R_0$  was positive rather than zero. This resistance increase is attributed to damage of the fiber-cement interface due to the fiber push-in and pull-out. As cycling progressed, both the maximum  $\Delta R/R_0$  and minimum  $\Delta R/R_0$  in a cycle decreased. This is attributed to damage of the cement matrix separating adjacent fibers at their junction; this damage increased the chance for adjacent fibers to touch each other, thereby decreasing the resistivity. This decrease from cycle to cycle persisted for the first ~ 150 cycles, after which the maximum and minimum  $\Delta R/R_0$  did not change with cycling.

At 28 days of curing (Fig. 5), since the amplitude of resistance variation in a cycle is 0.4 and the strain amplitude is  $8 \times 10^{-4}$ , the gage factor is 0.4

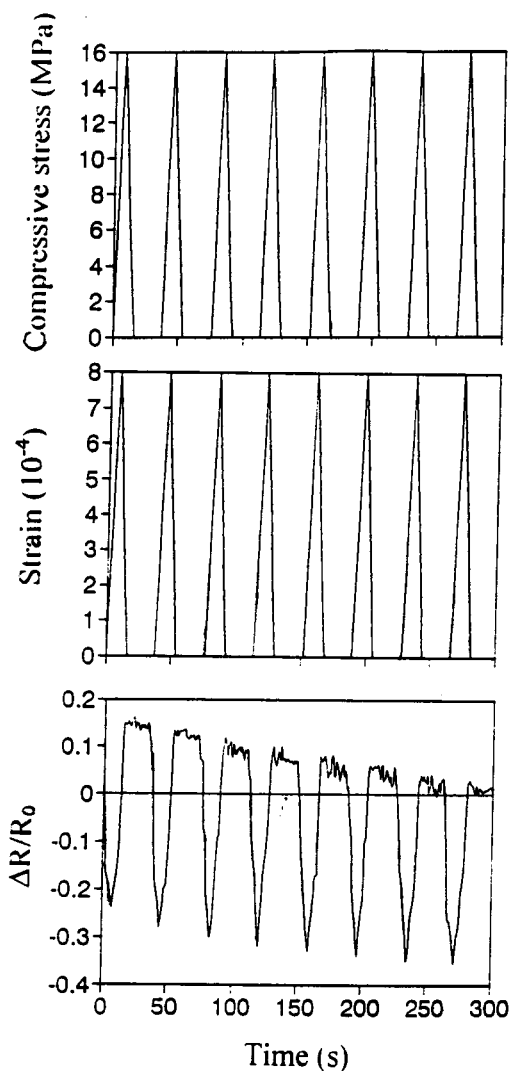


Fig. 5  $\Delta R/R_0$ , strain and stress during first cyclic compressive loading of mortar at 28 days of curing.

divided by  $8 \times 10^{-4}$ , or 500. At 7 days of curing (Fig. 6), the gage factor is 375. These exceptionally large values of the gage factor means that the mortar is an extremely sensitive strain gage. The gage factor of a conventional resistive strain gage is around 2.

Figure 7 shows  $\Delta R/R_0$  versus strain and stress versus strain during first



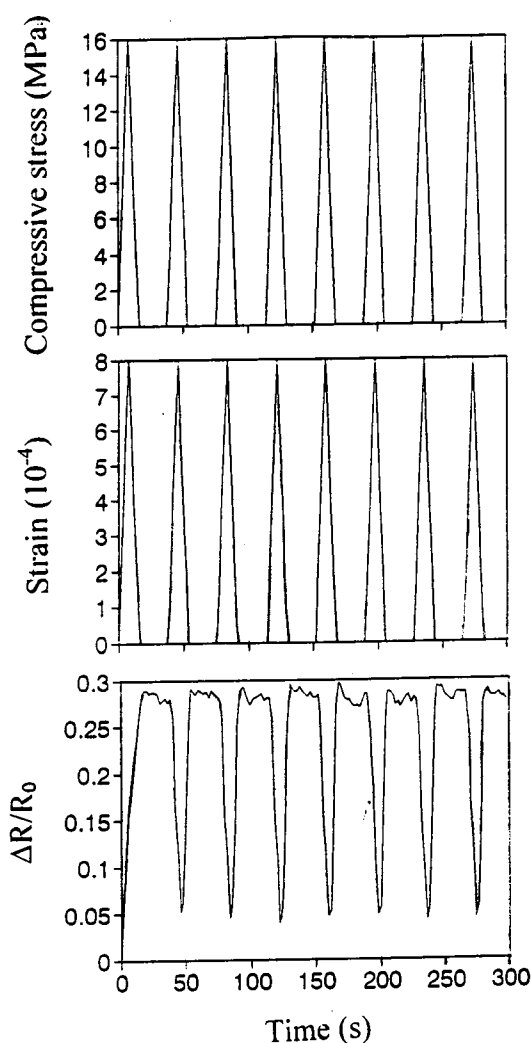


Fig. 6  $\Delta R/R_0$ , strain and stress during first cyclic compressive loading of mortar at 7 days of curing.

static compression up to failure of mortar with 0.24 vol.% carbon fibers at 28 days of curing. The resistance decreased monotonically upon loading up to failure.

Results similar to Figs. 5 and 7 were obtained at 14 days of curing, except that the decrease in maximum  $\Delta R/R_0$  and minimum  $\Delta R/R_0$  from cycle to cycle was less pronounced. However, at 7 days of curing, the resistance

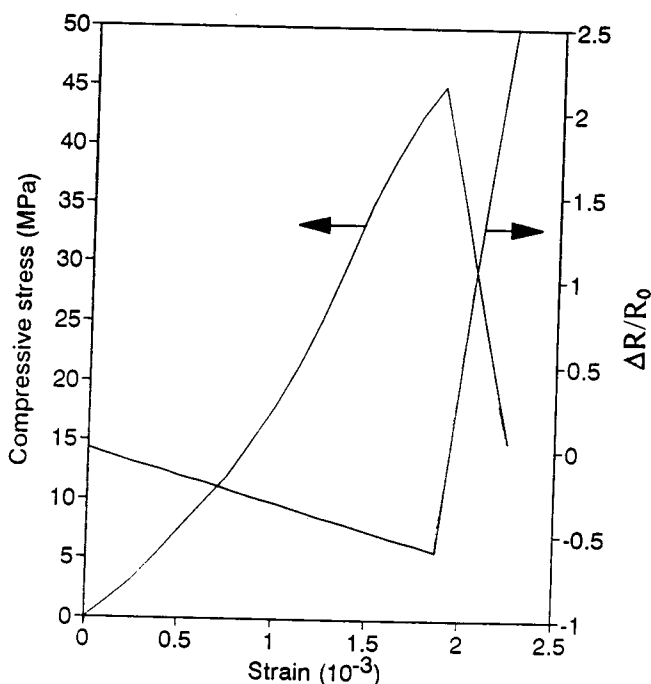


Fig. 7  $\Delta R/R_0$  versus strain and stress versus strain during first static compression up to failure of mortar at 28 days of curing.

increased monotonically upon first static compression up to failure (Fig. 8). Consistent with the static compression result of Fig. 8 is the cyclic compression result of Fig. 6. During the first cycle, the resistance increased upon loading, increased further upon subsequent unloading (due to fiber pull-out), decreased upon loading in the second cycle (due to fiber push-in), and increased upon unloading in the second cycle (due to fiber pull-out); the behavior was similar in second and subsequent cycles, but was different in the first cycle. The behavior at 7 days of curing is attributed to the relatively strong fiber-matrix bonding at 7 days and the consequent need to weaken the bond prior to fiber pull-out. Bond weakening is accompanied by irreversible increase in the contact electrical resistivity [17], which results in the irreversible resistance increase observed in the first cycle [18]. This irreversible resistance increase was larger in magnitude when the stress amplitude increased [18]. The fiber-cement bond strength decreased with increasing curing time from 7 to 14 days, while the contact resistivity increased, as shown for stainless steel fiber [19]. At 14 or 28 days of curing, the bond strength was weak to start with, so bond weakening was not necessary prior to fiber pull-out. The monotonic resistance increase up to failure at 7 days of curing is due to the bond weakening at least

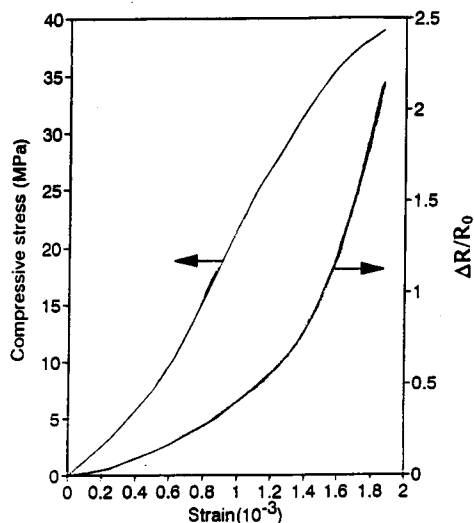


Fig. 8  $\Delta R/R_0$  versus strain and stress versus strain during first static compression up to failure of mortar at 7 days of curing.

in the low stress regime; in the high stress regime, it is probably due to damage. Note from Fig. 6 that the maximum  $\Delta R/R_0$  and minimum  $\Delta R/R_0$  did not change from cycle to cycle, in contrast to the decrease in these quantities at 14 and 28 days of curing. This effect of the curing age is attributed to the decrease in ductility with increasing curing age and the resulting increased tendency for repeated fiber pull-out and push-in during cyclic loading to cause damage to the cement matrix separating adjacent fibers. The effect of the curing age is not due to the effect of the moisture content, as the humidity during curing had essentially no effect on the electromechanical behavior [18] and the volume electrical resistivity of the mortar increased negligibly with curing age [27].

During the few cycles before fatigue failure at 28 days of curing, the resistance did not increase irreversibly and significantly, indicating that the mortar is poor in its ability to monitor damage. This poor damage sensitivity is also reflected by the absence of a resistance increase prior to static failure at 28 days (Fig. 7).

## CONCLUSION

Nondestructive evaluation of carbon-matrix and cement-matrix composites during loading was achieved in real time in terms of both fatigue damage and dynamic strain by measurement of the electrical resistance. The carbon-matrix composite was highly effective for damage sensing (i.e., sensitivity even to the damage after the first cycle of tensile loading within the

elastic regime), because the matrix was conducting and its fracture caused the resistivity to increase irreversibly. The carbon-carbon composite was also a strain sensor, due to the dimensional changes during tension and the resulting reversible increase in the resistance. Concrete containing short carbon fibers was highly effective for strain sensing (gage factor as high as 500), due to slight fiber pull-out during strain and the resulting reversible increase in resistivity. The concrete was also a damage sensor, due to fiber and matrix fracture causing the resistivity to increase irreversibly.

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