Self-monitoring of strain in silicon carbide whisker reinforced silicon nitride

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Abstract. An Si_3N_4-matrix composite with 30 vol.% SiC whisker was found to be able to sense its own tensile strain, including reversible strain, but not compressive strain. The sensing ability is based on the volume electrical resistivity of the composite reversibly decreasing upon tensile loading. The effect is attributed to the reversible decrease in the contact electrical resistivity at the whisker-matrix interface for whiskers that are somewhat parallel to the stress.

1. Introduction

Due to their brittleness, ceramics are not common structural materials, in spite of their high-temperature resistance. By reinforcing the ceramic with fibers or whiskers, the ductility is improved, but the brittleness remains a concern to structural design engineers. One way to alleviate this situation is to monitor the deformation or strain during use of the ceramic, so that remedies or changes in service conditions can be provided before catastrophic failure takes place. This monitoring requires sensors, in particular strain sensors, placed in strategic locations. They can be optical fibers, piezoelectric sensors, piezoresistive sensors, etc. Structures having this sensing ability are commonly known as smart structures. Although the placement of sensors is common in smart structures, it suffers from poor durability (due to the tendency for the attached sensors to come off the structure or to be damaged during the use of the structure), limited sensing volume (due to the fact that each sensor can only sense the strain in its immediate vicinity and the impracticality of having sensors covering the whole structure) and high cost (due to the high cost of the sensors and, in some cases, of the peripheral equipment, such as lasers and electronics, needed for the sensors to function). Self-monitoring refers to the ability for the structural material to monitor its own strain; i.e., the structural material is itself a sensor, so that there is no need to place any sensor on the structure. By using a self-monitoring material, the three disadvantages mentioned above are removed, as the structural material is durable and the whole structure (not just in limited locations) can sense. The self-monitoring of strain (both tensile and compressive) has been previously reported for concrete containing short carbon fibers [1–4].

Strain monitoring must be distinguished from damage monitoring, as strain can be reversible whereas damage is irreversible. Because damage is irreversible, it does not have to be monitored in real time. However, reversible strain must be monitored in real time. Furthermore, the deformation associated with damage tends to be greater than that associated with reversible strain. As a result, the monitoring of reversible strain is much more challenging than that of damage. Although reversible strain tends to cause little or no damage, the monitoring of reversible strain

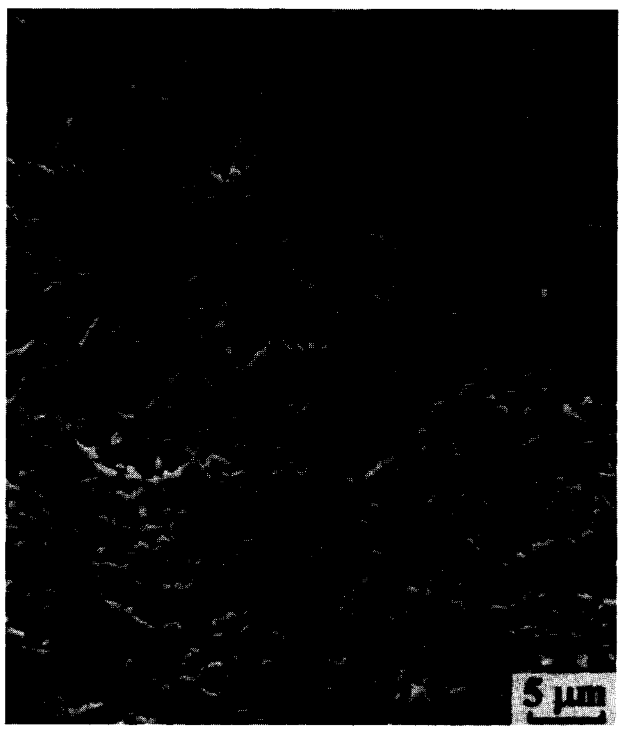

Figure 1. SEM micrograph of the Si_3N_4-matrix SiC-whisker composite after etching.

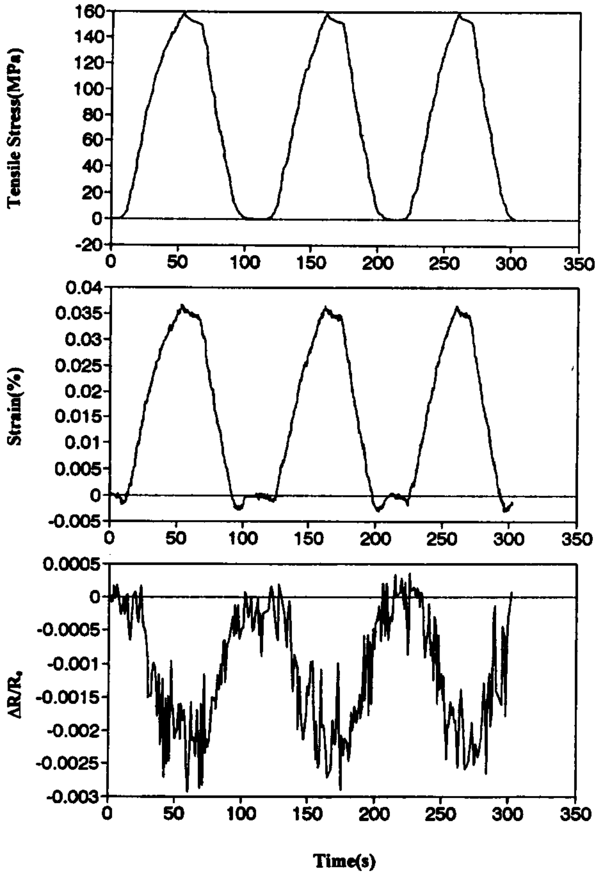

Figure 2. Variation of fractional electrical resistance increase ($\Delta R/R_0$), strain and stress during cyclic tensile loading at a stress amplitude equal to 40% of the fracture stress.

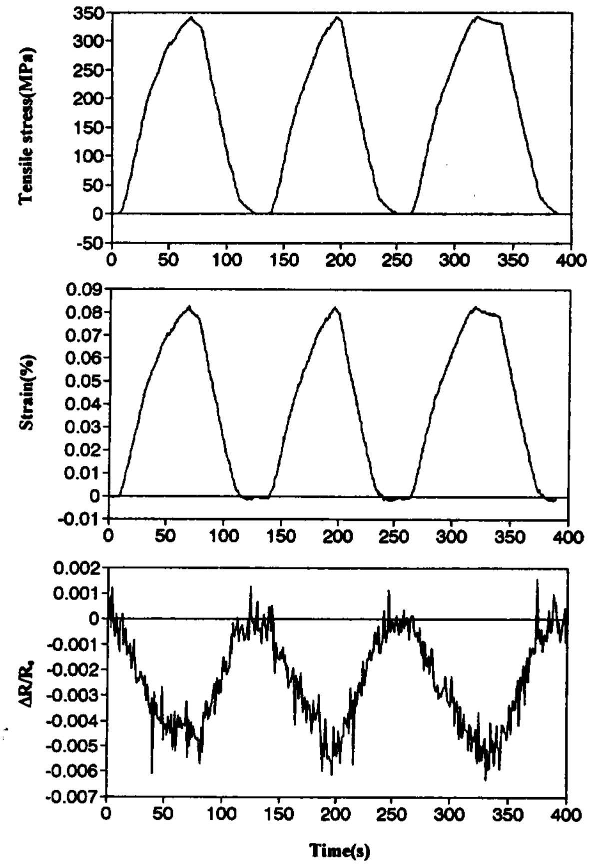

Figure 3. Variation of fractional electrical resistance increase ($\Delta R/R_0$), strain and stress during cyclic tensile loading at a stress amplitude equal to 83% of the fracture stress.

is useful for the purpose of recording the history of strain (or stress) experienced by the structure. This history is valuable for keeping track of the deformations during the usage of the structure and using this information to control the usage in real time or to determine the cause of a subsequent failure of the structure. The combination of sensing and control is the key to the smartness of the structure.

Damage self-monitoring has been previously reported in ceramic-matrix composites, such as a CaF_2-matrix SiC-whisker composite [5], as the volume electrical resistivity increased irreversibly upon damage (probably due to cracks, which are electrically insulating). However, strain (reversible) self-monitoring has not been previously reported in ceramic-matrix composite other than concrete containing short carbon fibers [1–4]. In the case of concrete containing short carbon fibers, the reversible strain sensing ability stems from the reversible increase in the volume electrical resistivity of the concrete when the contact electrical resistivity between fiber and matrix reversibly increases due to the very slight but reversible pull-out of the crack-bridging fibers during slight crack opening, which occurs upon tensile loading or upon compressive unloading [1–4]. In this paper, we report the reversible strain sensing ability of an Si_3N_4-matrix SiC-whisker composite, which is one of the most attractive high-temperature lightweight structural ceramic-matrix composites [6].

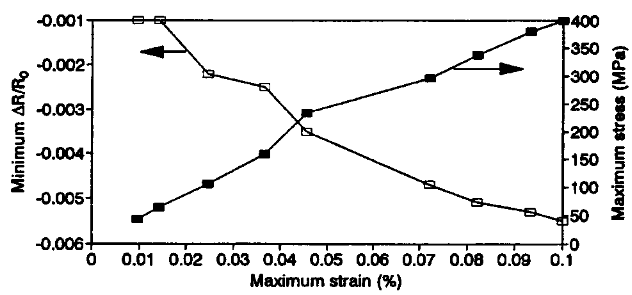

Figure 4. Relationship between minimum $\Delta R/R_0$ ($\Delta R/R_0$ amplitude), maximum stress (stress amplitude) and maximum strain (strain amplitude) for cyclic tensile loading.

2. Experimental methods

The Si_3N_4-matrix SiC-whisker composite kindly provided by NASA Lewis Research Center Cleveland, Ohio) contained 30 vol.% SiC whiskers. The composite's volume electrical resistivity was 1.73 Ωm—many orders of magnitude lower than that of Si_3N_4. This means that the SiC whiskers contributed substantially to the electrical conductivity of the composite.

Simultaneous to mechanical testing, DC electrical resistance measurement was made using a Keithley 2001 multimeter. For compressive testing, specimens were of size $24.4\times8.15\times5.99$ mm and the axis of compression was

Table 1. $\Delta R/R_0$ as a function of tensile stress and strain amplitudes.

Maximum stress (MPa)	Maximum stress / Fracture stress	Maximum strain	Maximum strain / Fracture strain	$\Delta R/R_0$	$\Delta R/R_0$ / $\Delta R/R_0$ at fracture	$\Delta\rho/\rho_0^*$
42.1	10.6%	9.63×10^{-5}	9.63%	−0.001	18%	−0.001
63.4	15.9%	1.42×10^{-4}	14.2%	−0.001	18%	−0.001
105	26.3%	2.44×10^{-4}	24.4%	−0.0022	40%	−0.0026
159	39.8%	3.65×10^{-4}	36.5%	−0.0025	45%	−0.0031
233	58.4%	4.55×10^{-4}	45.5%	−0.0035	64%	−0.0042
296	74.2%	7.20×10^{-4}	72.0%	−0.0047	85%	−0.0059
338	84.7%	8.22×10^{-4}	82.2%	−0.0051	93%	−0.0064
379	95.0%	9.33×10^{-4}	93.3%	−0.0053	96%	−0.0068
399	100%	1.00×10^{-3}	100%	−0.0055	100%	−0.0071

* Calculated from $\Delta R/R_0$ and maximum strain by assuming that the Poisson ratio v is 0.3.

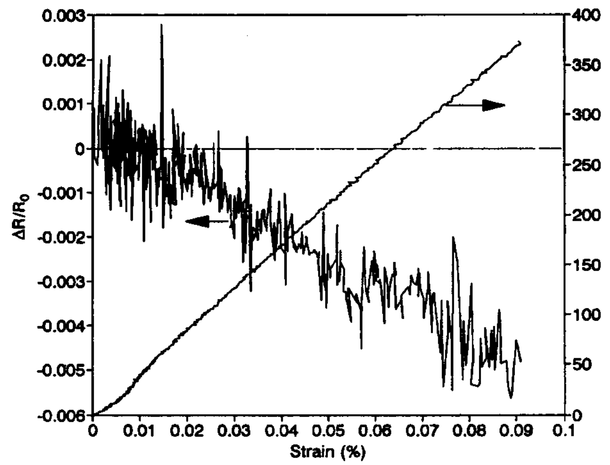

Figure 5. Variation of $\Delta R/R_0$, strain and stress during static tensile loading up to fracture.

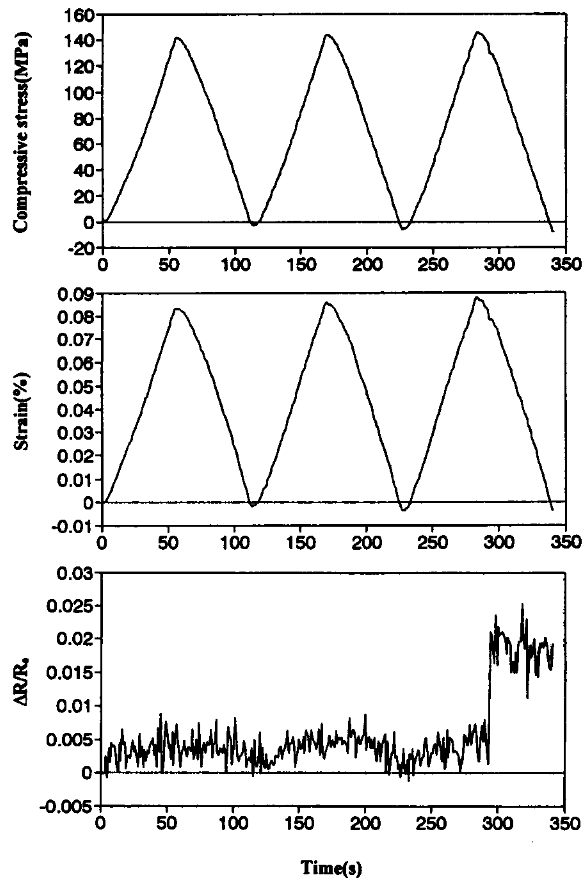

Figure 6. Variation of fractional electrical resistance increase ($\Delta R/R_0$), strain and stress during cyclic compressive loading at a stress amplitude equal to 7.5% of the fracture stress.

along the longest dimension. For tensile testing, specimens were of size 49.5 × 5.11 × 0.826 mm and the axis of tension was along the longest dimension. The strain was measured by a strain gage in both tensile and compressive testing, while the fractional change in electrical resistance along the stress axis was measured using the four-probe method. The electrical contacts were made by silver paint. The separation between the voltage (inner) probes was 15 and 30 mm for compression and tension respectively. Testing was performed under cyclic loading (tensile or compressive) at various stress amplitudes and under static loading up to fracture. For compressive testing, a hydraulic mechanical testing system (MTS Model 810) was used, such that the displacement rate was 0.50 mm/min. For tensile testing, a screw-action mechanical testing system (Sintech 2/D) was used, such that the displacement rate was either 0.20 or 0.30 mm/min.

3. Results

Figure 1 shows a scanning electron micrograph of the Si_3N_4-matrix SiC-whisker composite which had been mechanically polished and then etched in molten NaOH at 425 °C for ~4 min to remove the matrix at the surface. The whiskers were randomly oriented and quite uniformly distributed. The whisker diameter was ~0.5 μm; the whisker length was ~5 μm.

Figure 2 gives the fractional resistance increase ($\Delta R/R_0$), strain and stress simultaneously obtained during cyclic tensile loading (displacement rate = 0.20 mm/min) at a stress amplitude equal to 40% of the fracture stress. The $\Delta R/R_0$ decreased upon loading and increased upon unloading in every cycle, which was within the stress regime in which the strain was reversible.

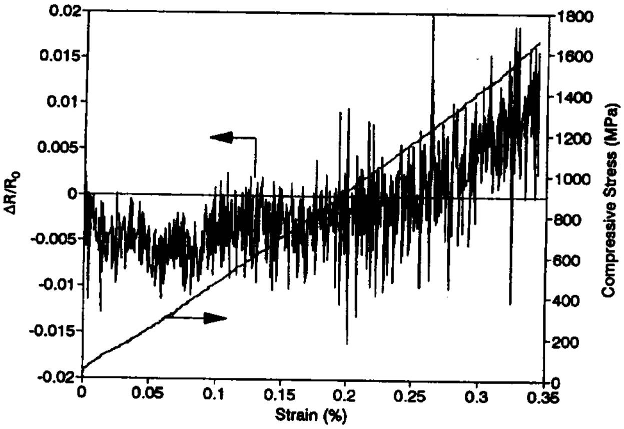

Figure 7. Variation of $\Delta R/R_0$, strain and stress during static compressive loading up to fracture.

Figure 3 gives similar data obtained during cyclic tensile loading (displacement rate = 0.30 mm/min) within the elastic regime at a stress amplitude equal to 83% of the fracture stress. Similar data obtained at various stress amplitudes (table 1 and figure 4) show that the $\Delta R/R_0$ amplitude increased monotonically with increasing stress/strain amplitude (referred to as maximum stress/strain in figure 4), so that $\Delta R/R_0$ provides an indication of the stress/strain amplitude. Table 1 shows that the fractional increase in resistivity ($\Delta\rho/\rho_0$, where $\rho = R\frac{A}{l}$, with A = cross-sectional area and l = length) is more negative than $\Delta R/R_0$ at each stress amplitude. This is because the positive strain (elongation) upon tension contributed positively to $\Delta R/R_0$.

Figure 5 shows data obtained during static tensile loading up to fracture (displacement rate = 0.20 mm/min). The $\Delta R/R_0$ changed negligibly as the stress increased until the stress reached 19% of the fracture stress, at which $\Delta R/R_0$ started to decrease (become more negative) with increasing stress/strain. This dependence of $\Delta R/R_0$ on stress is consistent with that observed during cyclic loading (table 1, figures 2–4). At fracture during static loading, the $\Delta R/R_0$ abruptly increased to a value of +0.005 (not shown in figure 5), obviously due to cracks.

Figure 6 gives $\Delta R/R_0$, strain and stress simultaneously obtained during cyclic compressive loading (displacement rate = 0.50 mm/min) at a stress amplitude equal to 7.5% of the fracture stress. The $\Delta R/R_0$ changed only slightly (with much noise) during cyclic compression. Similar noisiness was observed at higher compressive stress amplitudes. Thus, the strain sensing ability was absent under compression. The irreversible jump in $\Delta R/R_0$ in figure 6 in the third cycle is probably due to damage, which is supported by the emission of a sound at the time of the jump. Although the stress and strain changed only slightly at the time of the damage, $\Delta R/R_0$ jumped. This indicates that $\Delta R/R_0$ is a good indicator of damage. Figure 7 shows $\Delta R/R_0$, strain and stress obtained during static compression up to fracture. In spite of the noise, the $\Delta R/R_0$ changed negligibly until fracture was near. This is consistent with the negligible change in $\Delta R/R_0$ during cyclic compression (figure 6).

Due to the very high resistivity of Si_3N_4, similar tests on Si_3N_4 (without SiC whiskers) failed to give any result; the resistance was too high to be measured with the electronics used.

4. Discussion

Under tension, the Si_3N_4-matrix SiC-whisker composite has its $\Delta R/R_0$ reversibly decreased during loading. This reversible effect is opposite in direction from that in concrete containing short carbon fibers, which has its $\Delta R/R_0$ increased during loading and decreased during unloading [1–4]. This difference stems from the difference in mechanism. In the Si_3N_4-matrix SiC-whisker composite, the decrease in $\Delta R/R_0$ during tensile loading is probably due to the decrease in the contact resistivity at the interface during tensile loading, as this interface is known to be not strong and the whiskers are much more electrically conducting than the Si_3N_4 matrix. The mechanism for the concrete case is described in the introduction and in [1–4]. The mechanism for the Si_3N_4-matrix composite cannot be piezoresistivity, which would make $\Delta R/R_0$ increase upon tensile loading. (Piezoresistivity refers to the change in resistivity due to the change in separation between adjacent conducting units in the composite.) The mechanism also cannot be simply related to the resistance change accompanying dimensional changes (while the resistivity is constant), as elongation along the resistance direction occurs upon tension and this will increase (rather than decrease) the resistance.

The SiC whiskers in the Si_3N_4-matrix composite are randomly oriented, so a tensile stress in a given direction is expected to decrease the contact resistivity of the whisker-matrix interface for whiskers that are somewhat parallel to (say, within 45 °C from) the stress direction but is expected to increase the contact resistivity of the interface for whiskers that are somewhat perpendicular to the stress direction. On the other hand, the volume electrical resistivity of the composite in the stress direction is governed mainly by the whiskers that are somewhat parallel to the stress direction, since the whiskers are much more electrically conducting than the matrix. Therefore, the increase of the contact resistivity of the interface for whiskers that are somewhat perpendicular to the stress direction does not much affect the volume electrical resistivity of the composite in the stress direction. Therefore, the observed decrease in the volume resistivity in the stress direction is attributed to the decrease of the contact resistivity of the interface for whiskers that are somewhat parallel to the stress direction.

Under compression, the Si_3N_4-matrix SiC-whisker composite fails to be a strain sensor. In contrast, concrete containing short carbon fibers functions as a strain sensor both under tension and compression. The absence of an electromechanical effect in the Si_3N_4-matrix composite under compression is consistent with the inherently weak interface between whisker and matrix. Further weakening this interface apparently does not further increase the contact resistivity at the interface.

The gage factor of a strain gage is defined as $\Delta R/R_0$ divided by the strain amplitude. The gage factor of the Si_3N_4-matrix composite ranges from -10 to -6 under tension. In contrast, the gage factor of cement paste containing short carbon fibers is up to 500 under tension [1]. This large difference in the magnitude of the gage factor occurs in spite of the large SiC whisker volume fraction (30%) and the low carbon fiber volume fraction (0.53%). This large difference is consistent with the basic difference in mechanism. Even though the magnitude of the gage factor is much lower for the Si_3N_4-matrix composite than for the cement composite, the magnitude of the gage factor of the Si_3N_4-matrix composite is comparable to those of piezoresistive polymer-matrix composites containing short carbon fibers [7].

5. Conclusion

The Si_3N_4-matrix SiC-whisker composite was found to be a sensor of tensile strain, including reversible strain, so that this composite is a self-monitoring structural material. The $\Delta R/R_0$ reversibly decreased upon tensile loading. This electromechanical effect is attributed to the reversible decrease in the contact electrical resistivity at the whisker-matrix interface for whiskers that are somewhat parallel to the stress. Under compression, this composite failed to be a strain sensor.

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