Sensing Delamination in a Carbon Fiber Polymer-Matrix Composite During Fatigue by Electrical Resistance Measurement

XIAOJUN WANG *and* D. D. L. CHUNG

Composite Materials Research Laboratory
State University of New York at Buffalo
Buffalo, New York 14260-4400

Delamination in a crossply [0/90] continuous carbon fiber polymer-matrix composite was sensed in real time during fatigue by measuring the electrical resistance of the composite in the through-thickness direction. Upon 0° tension-tension fatigue at a maximum stress of 57% of the fracture stress, the resistance irreversibly increased both in spurts and continuously, because of delamination, which started at 33% of the fatigue life. The resistance increased upon loading and decreased upon subsequent unloading in every cycle, thereby allowing strain sensing. The minimum resistance at the end of a cycle irreversibly increased during the first 0.1% of the fatigue life. The resistance became noisy starting at 62% of the fatigue life, at which delamination occurred rapidly and the fraction of laminate area delaminated reached 4.3%.

INTRODUCTION

Delamination is one of the main types of flaw in a continuous fiber polymer-matrix composite. It refers to the local separation of the fiber layers in the laminate. It is usually associated with the cracking of the matrix between the fiber layers, although it can also be associated with debonding at the fiber-matrix interface.

As delamination affects the mechanical properties of the laminate, nondestructive sensing of delamination is needed. Microscopy can be used for observing delamination, but it is sensitive to only large delaminations at the edge of the laminate and is not amenable to real-time monitoring and control. Ultrasonic inspection is sensitive to interior delaminations and is amenable to real-time monitoring and control, but it is not sensitive to small degrees of delamination. The embedding of optical fiber sensors in the laminate is yet another method for sensing delamination, but it does not give quantitative information on the extent of delamination and it is an intrusive method. This paper presents the method of electrical resistance measurement in the through-thickness direction of the composite for sensing delamination in carbon fiber polymer-matrix composites nondestructively, nonintrusively, quantitatively, at high sensitivity and in real time during fatigue. Fatigue occurs during dynamic loading, which is commonly encountered by composites, such as those for helicopter rotors, turbine blades, and any moving component.

The method presented in this paper is based on the notion that carbon fibers are electrically conducting and the electrical resistivity of the composite in the through-thickness direction is governed by the extent of contact between fibers in adjacent fiber layers. Although a continuous fiber polymer-matrix composite as described in textbooks appears to have no contact between fibers of different layers, in reality some contact always occurs (as indicated by the fact that the electrical resistivity of the composite in the through-thickness direction is not ∞), owing to the imperfect alignment of the fibers and the flow of the epoxy during composite fabrication. Delamination decreases the extent of contact, thereby causing the resistivity of the composite in the through-thickness direction to increase.

The use of electrical resistance measurement in the fiber direction of a composite to sense fiber breakage has been previously reported (1–8). In contrast, this paper addresses the sensing of delamination through resistance measurement in the through-thickness direction.

The method presented in this paper applies to composites with continuous fibers that are electrically much more conducting than the matrix. It is not limited to carbon fiber composites. However, carbon fiber polymer-matrix composites were chosen for this work because of their dominance among advanced composites.

EXPERIMENTAL METHODS

Composite samples were constructed from individual layers cut from a 12-inch-wide unidirectional carbon fiber prepreg tape manufactured by ICI Fiberite. The product used was Hy-E 1076E, which consisted of a 976 epoxy matrix and 10E carbon fibers. The fiber and matrix properties are shown in *Table 1*.

The composite laminates were laid up in a 4 × 7 inch (102 × 178 mm) platen compression mold with laminate configuration $[0/90]_{3s}$, i.e., a crossply composite. The individual 4 × 7 inch fiber layers (12 per laminate) were cut from the prepreg tape. The layers were stacked in the mold with a mold release film on the top and bottom of the layup. No liquid mold release was necessary. The density of the laminate was 1.50 ± 0.03 g/cm^3. The thickness of the laminate was 1.5 mm. The volume fraction of carbon fibers in the composite was 52%. The laminate was cured using a cycle based on the ICI Fiberite C-5 cure cycle. The curing occurred at 179 ± 6°C (355 ± 10°F) and 0.61 MPa (89 psi) for 120 min. Afterwards, it was cut to pieces of size 180 × 15 mm. Glass fiber reinforced epoxy end tabs were applied to both ends on both sides of each piece, such that each tab was 30 mm long and the inner edges of the end tabs on the same side were 100 mm apart. The tensile strength was 695 ± 18 MPa. The tensile ductility was (1.06 ± 0.18)%. The electrical resistivity in the 0° direction was 8.84×10^{-2} Ω.cm; that in the direction perpendicular to the fiber layers was 45.7 Ω.cm.

The electrical resistance R was measured in the direction perpendicular to the fiber layers using the four-probe method while either static or cyclic tension was applied in the 0° direction. Silver electrically conducting paint was used for all electrical contacts. The four probes consisted of two outer current probes and two inner voltage probes. The resistance R refers to the sample resistance between the inner probes. For measuring R in the direction perpendicular to the fiber layers, the current contacts were centered on the largest opposite faces parallel to the stress axis and in the form of open rectangles of length 75 mm in the stress direction and width 11 mm, while each of the two voltage contacts was in the form of a solid rectangle (of length 20 mm in the stress direction) surrounded by a current contact (open rectangle). Thus, each face had a current contact surrounding a voltage contact. A strain gauge was attached to the center of one of the largest opposite faces parallel to the stress axis for samples for measuring R in the 0° direction as well as samples for measuring R in the direction perpendicular to the fiber layers. A Keithley 2001 multimeter was used for DC electrical measurement. The displacement rate was 1.0 mm/min. A hydraulic mechanical testing system (MTS 810) was used for tension-tension cyclic loading in the 0° direction, with stress ratio (minimum stress to maximum stress in a cycle) 0.05 and with maximum stress 395 MPa (at which strain = 0.57%). Each cycle took 2 s. The total number of cycles before fatigue failure was on the average 217,392. Although the results shown in this paper are for one particular fatigue test, testing of similar samples confirmed that the results presented here are reproducible.

Table 1. Carbon Fiber and Epoxy Matrix Properties (According to ICI Fiberite).

10E—Torayca T-300 (6K) untwisted, UC-309 sized	
Diameter	7 μm
Density	1.76 g/cm^3
Tensile modulus	221 GPa
Tensile strength	3.1 GPa
976 Epoxy	
Process temperature	350°F (177°C)
Maximum service temperature	350°F (177°C) dry 250°F (121°C) wet
Flexural modulus	3.7 GPa
Flexural strength	138 MPa
T_g	232°C
Density	1.28 g/cm^3

RESULTS AND DISCUSSION

Static Loading to Failure

Figure 1 shows the tensile stress, strain (in the 0° direction) and $\Delta R/R_o$ in the through-thickness direction, obtained simultaneously during static loading up to failure. The $\Delta R/R_o$ increased gradually with increasing strain and then increased abruptly from $\Delta R/R_o = 0.02$ as the strain increased beyond 0.8%, such that $\Delta R/R_o$ reached a maximum of 0.10. The abrupt increase in $\Delta R/R_o$ is attributed to the increase in the degree of 0° fiber alignment and the consequent decrease in the chance that adjacent fiber layers touch one another. The effect is smaller than that for a [0] unidirectional composite (8), as the presence of the 90° fibers in the [0/90] composite makes the increase in the degree of 0° fiber alignment only slightly affect the chance that adjacent fiber layers touch one another.

Dynamic Loading

Figure 2 shows the tensile stress, strain (in the 0° direction) and $\Delta R/R_o$ in the through-thickness direc-

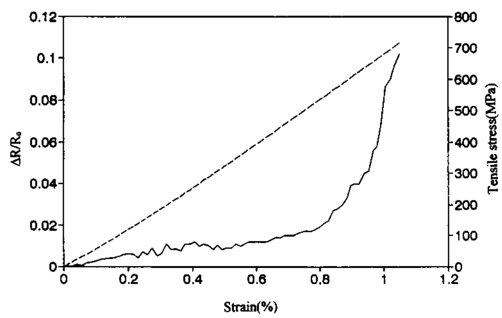

Fig. 1. Tensile stress, strain and $\Delta R/R_o$ in the through-thickness direction of crossply composite obtained simultaneously during static tension up to fracture, which occurs at the highest strain in the curves. Solid curve: $\Delta R/R_o$ vs. strain. Dashed curve: stress vs. strain.

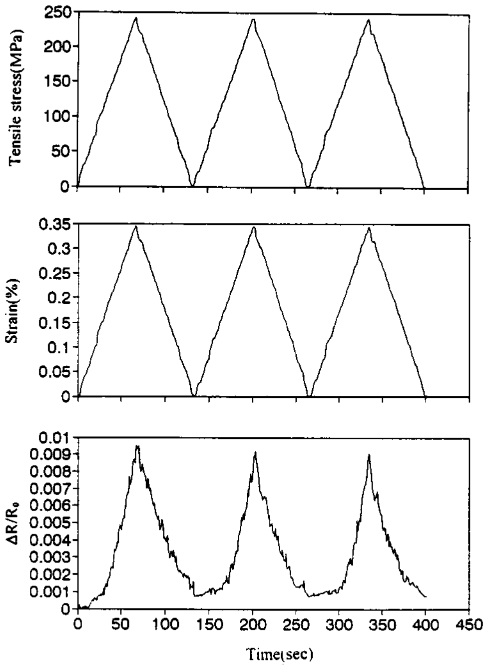

Fig. 2. Variation of $\Delta R/R_o$ in the through-thickness direction, 0° tensile stress and 0° tensile strain with cycle number during cyclic tension to a stress amplitude equal to 35% of the fracture stress for crossply composite.

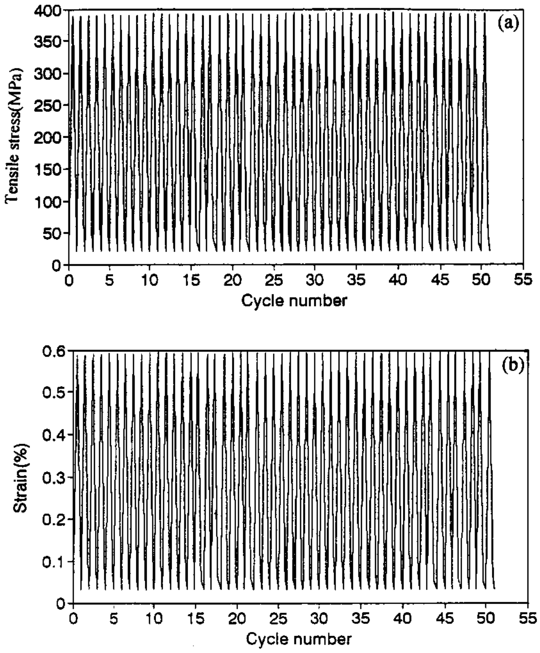

Fig. 3. Variation of tensile stress and strain with cycle number during tension-tension fatigue testing of crossply composite.

tion, obtained simultaneously during cyclic tension to a stress amplitude equal to 35% of the breaking stress. The strain returned to zero at the end of each cycle. The $\Delta R/R_o$ increased upon loading and decreased upon unloading in every cycle, such that R irreversibly increased slightly after the first cycle (i.e., $\Delta R/R_o$ did not return to 0 at the end of the first cycle). The behavior of *Fig. 2* is similar to that of the [0] unidirectional composite of Ref. 8 (*Fig. 3* of Ref. 8), except that $\Delta R/R_o$ is smaller and the irreversible portion of $\Delta R/R_o$ is positive for the crossply composite but negative for the 0° unidirectional composite. The small value of $\Delta R/R_o$ for the crossply composite compared to the unidirectional composite is due to the presence of the 90° fibers in the crossply composite and that these fibers make the increase in the degree of 0° fiber alignment only slightly affect the chance that adjacent fiber layers touch one another. That the irreversible portion of $\Delta R/R_o$ is negative for the [0] unidirectional composite (*Fig. 3* of Ref. 8) is due to the irreversible decrease in the degree of neatness of the fiber arrangement within a fiber bundle and the consequent increase in the chance that adjacent fiber layers touch one another. That the irreversible portion of $\Delta R/R_o$ is positive for the crossply composite (*Fig. 2*) is due to the presence of the 90° fibers, which (i) make the irreversible decrease in the degree of neatness of the fiber arrangement have negligible effect on the chance that the adjacent fiber layers touch one another, and (ii) cause the 0° fibers to be somewhat wavy (with the 0° fibers partly penetrating the gap between the 90° fiber bundles of the adjacent 90° fiber layer) after composite fabrication by compression molding and this waviness enhances the chance for the 0° and 90° fiber layers to touch each other. After a cycle of tensile loading in the 0° direction, the waviness is irreversibly lessened, thereby decreasing the chance for the adjacent fiber layers to touch each other and thus irreversibly increasing R perpendicular to the fiber layers.

Dynamic tensile loading was conducted on the [0/90] crossply composite at different stress amplitudes. The results at a stress amplitude equal to 35% of the fracture stress are shown in *Fig. 2* for $\Delta R/R_o$ in the through-thickness direction. The behavior at different stress amplitudes are similar, though the magnitudes of both reversible and irreversible portions of $\Delta R/R_o$ increase with increasing stress amplitude, as shown in *Table 2*. For the same stress amplitude (expressed as a fraction of the fracture stress), the magnitudes of both reversible and irreversible portions of $\Delta R/R_o$ are higher for the unidirectional composite (8) than the crossply composite (this work).

Fatigue Testing

Fatigue testing was conducted using the dynamic 0° stress and 0° strain variation shown in *Fig. 3*. The

Table 2. Effect of Stress Amplitude on the Reversible and Irreversible Parts of $\Delta R/R_o$.

Maximum Stress/Fracture Stress (%)	Strain Amplitude (%)	$\Delta R/R_o$ in the Through-Thickness Direction (%)	
		Reversible	Irreversible
6.1	0.061	0.20	0
9.2	0.092	0.30	0.04
19.4	0.21	0.53	0.07
35	0.35	0.84	0.11

stress and strain did not return to zero at the end of each cycle.

Figure 4 shows $\Delta R/R_o$ in the through-thickness direction, 0° tensile stress and 0° tensile strain simultaneously obtained during cyclic tension-tension loading of the crossply composite. The resistance R increased upon loading and decreased upon unloading in every cycle, as in *Fig. 2*.

During the first 210 cycles (or 0.099% of fatigue life), the minimum R (at the end of a cycle) as well as the peak R (in the middle of a cycle) increased gradually cycle after cycle (*Fig. 5a*). This is attributed partly to the partially irreversible increase in the degree of 0° fiber alignment and partly to matrix cracks, as both tend to decrease the number of contacts between fibers of different layers at the end of each of the first 210 cycles.

After the first 210 cycles, both minimum R and peak R increased very slowly because of the matrix cracks in 90° plies. These matrix cracks could change the current path perpendicular to the fiber layers and increase the resistance. The first spurt of peak R (in the middle of a cycle) was observed at 69,987 cycles (33% of fatigue life) (*Figs. 5c, 5d*, and *6*). The jump in peak R is probably due to the 0° cracks and 0° fiber fracture, which change the current path when the composite is loaded in the 0° direction. In *Fig. 5c*, the minimum R at the end of a cycle continuously increased while the peak R in the middle of a cycle showed a jump at 69,987 cycles (33% of fatigue life),

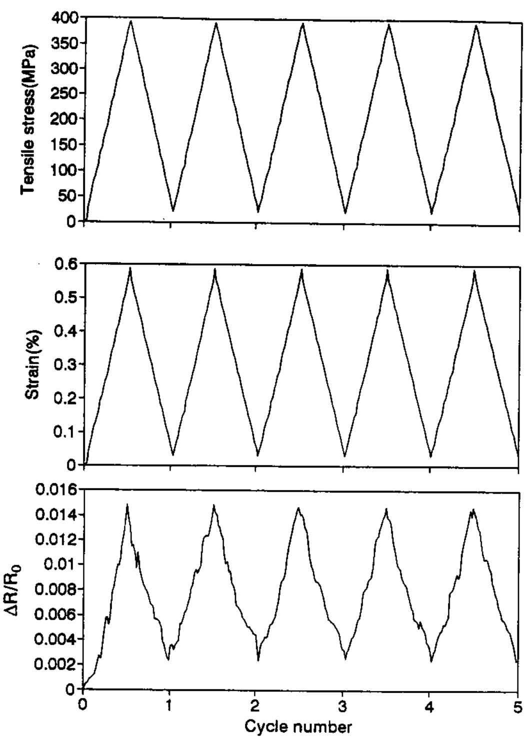

Fig. 4. Variation of $\Delta R/R_o$ in the through-thickness direction, 0° tensile stress and 0° tensile strain with cycle number during the first few cycles of tension-tension fatigue testing of crossply composite.

after which the peak R continuously increased in every cycle within *Fig. 5c*. Subsequently, both minimum R and peak R continued to increase in every cycle (*Fig. 5e*), but much more gradually than in *Fig. 5c*. When the composite is unloaded, it is possible that some of the broken fibers will touch each other. Therefore, the minimum R (at the end of a cycle) did not show a similar jump as the peak R. However, the 0° cracks and fiber fracture decreased the chance of fiber contacts so that the minimum R continuously increased.

As the fatigue testing developed, both minimum R and peak R continued to increase in each cycle, as shown in *Fig. 5e*, but much more gradually than in *Fig. 5c*. These observations in *Fig. 5e* imply that further 0° cracks occurred. *Figures 5f* and *6* show a second resistance spurt at 105,981 cycles (50% of fatigue life). At this spurt, both minimum R and peak R jumped together. This is attributed to the formation of local delaminations. A local delamination may be formed around the intersection of a transverse crack and a 0° crack. Delamination is expected to greatly affect the resistance through the thickness because the delamination decreases the chance that fibers in adjacent layers will touch each other.

As the fatigue loading continued, more severe resistance changes were observed. At about 62% of fatigue life (*Fig. 5g* and *6*), the minimum R (at the end of a cycle) showed a big jump while the peak R (in the middle of a cycle) increased only gradually, so that the amplitude of the resistance change greatly diminished. The minimum R showed a big jump because of the irreversible nature of delamination, which was extensive, because local delaminations grew and coalesced. *Figure 7* shows the percentage of composite area that exhibited delamination, as calculated from the increase in peak $\Delta R/R_o$, assuming that a delaminated area was open-circuited in the through-thickness direction. Delamination started at 33% of fatigue life and occurred both in spurts and continuously. At 62% of fatigue life, delamination occurred rapidly and the percent of area delaminated reached 4.3%.

R was observed to be increasingly noisy as the fatigue testing continued (*Fig. 5h*). Beyond 68% of fatigue life, the noise in R perpendicular to the fiber layers was so severe that the R data collection was stopped. As fatigue testing progressed, more delamination and fiber fracture occurred, which made the fiber contacts between adjacent layers more variable and the resistance change therefore became more noisy.

CONCLUSION

The sensing of delamination in a crossply [0/90] continuous carbon fiber polymer-matrix composite during fatigue was demonstrated in real time by electrical resistance measurement in the through-thickness direction. Upon 0° tension-tension fatigue at a maximum stress of 57% of the fracture stress, this resistance irreversibly increased both in spurts and continuously, owing to delamination, which started at

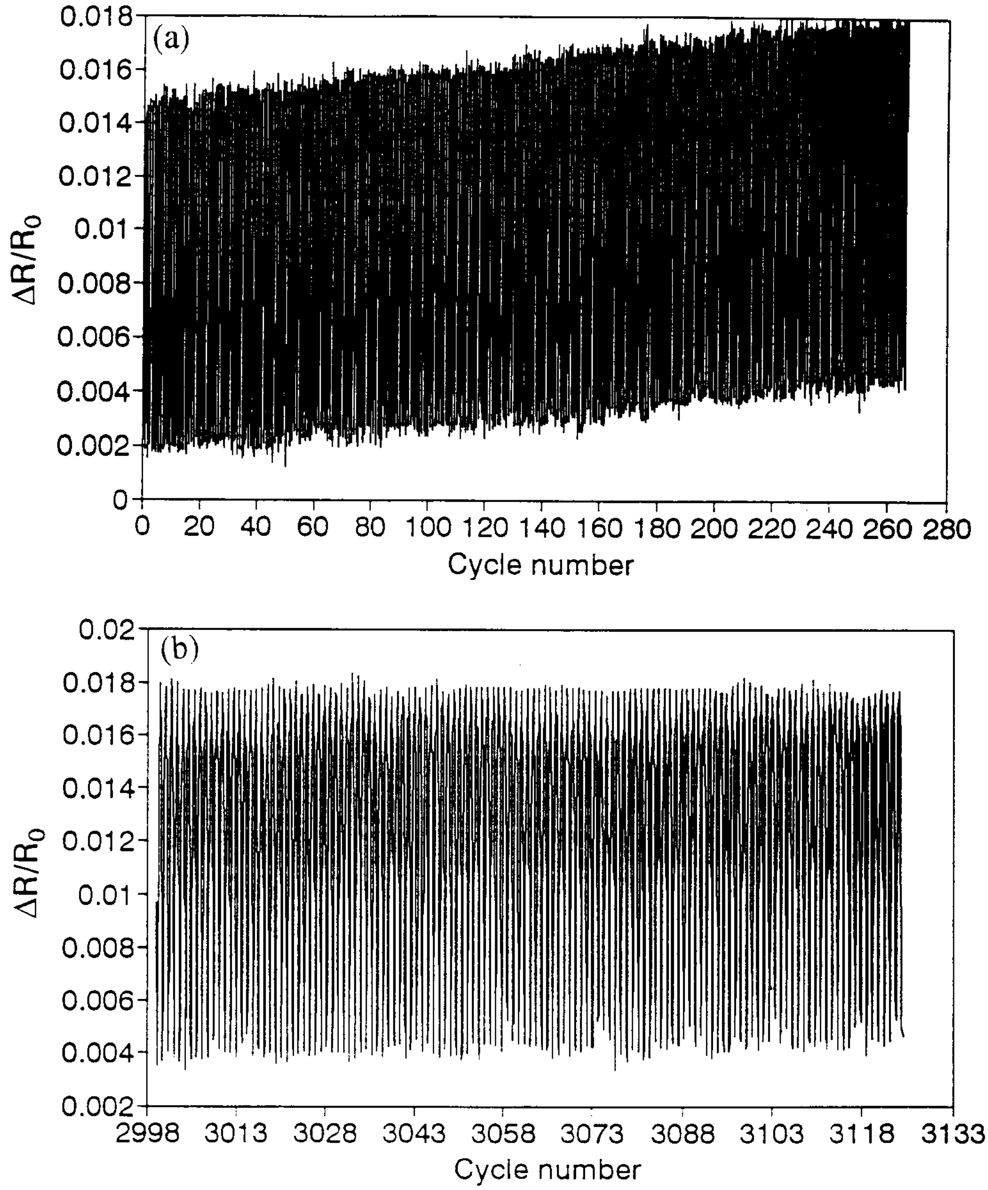

Fig. 5. Variation of $\Delta R/R_o$ in the through-thickness direction with cycle number during tension-tension fatigue testing for crossply composite.

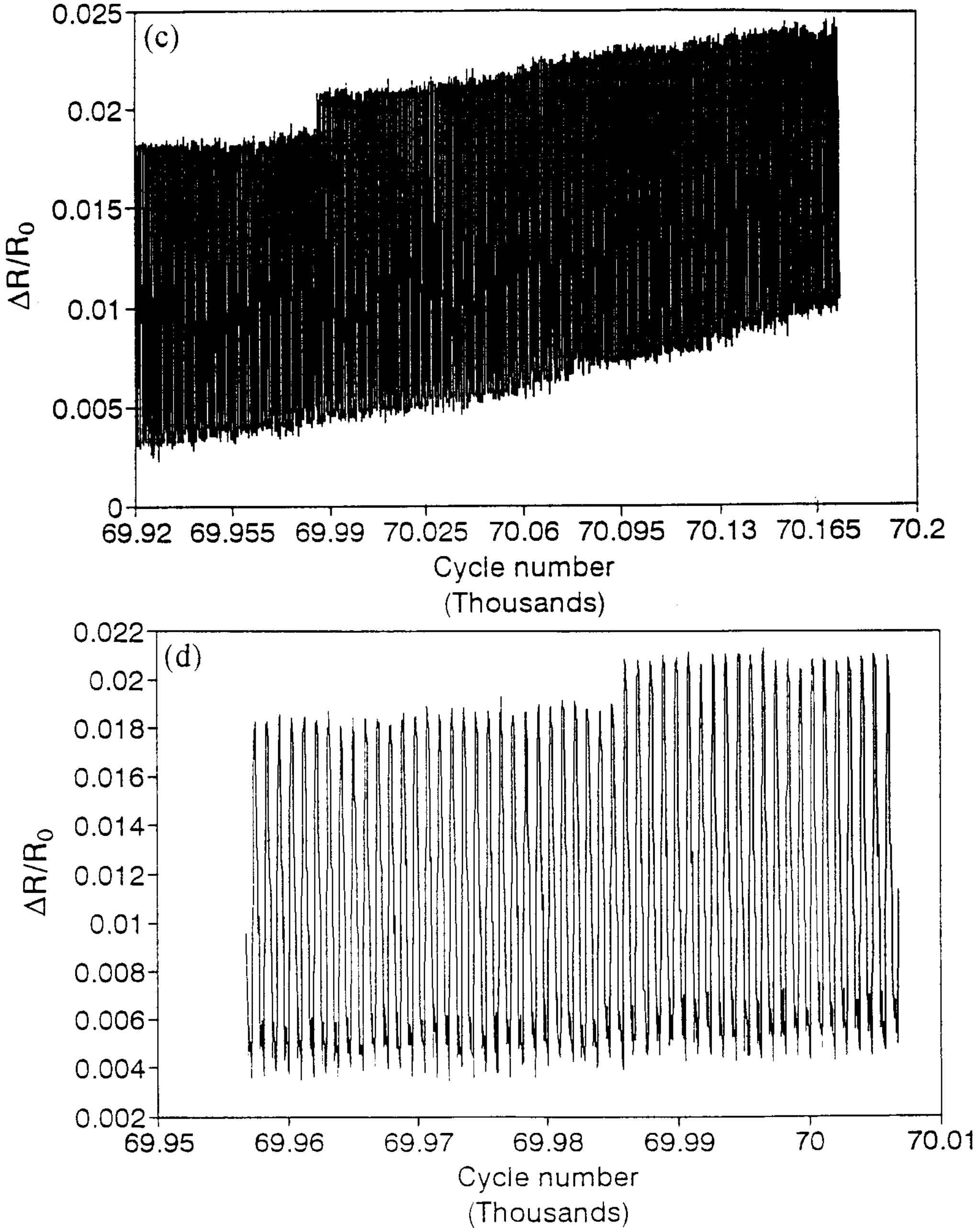

Fig. 5. Continued.

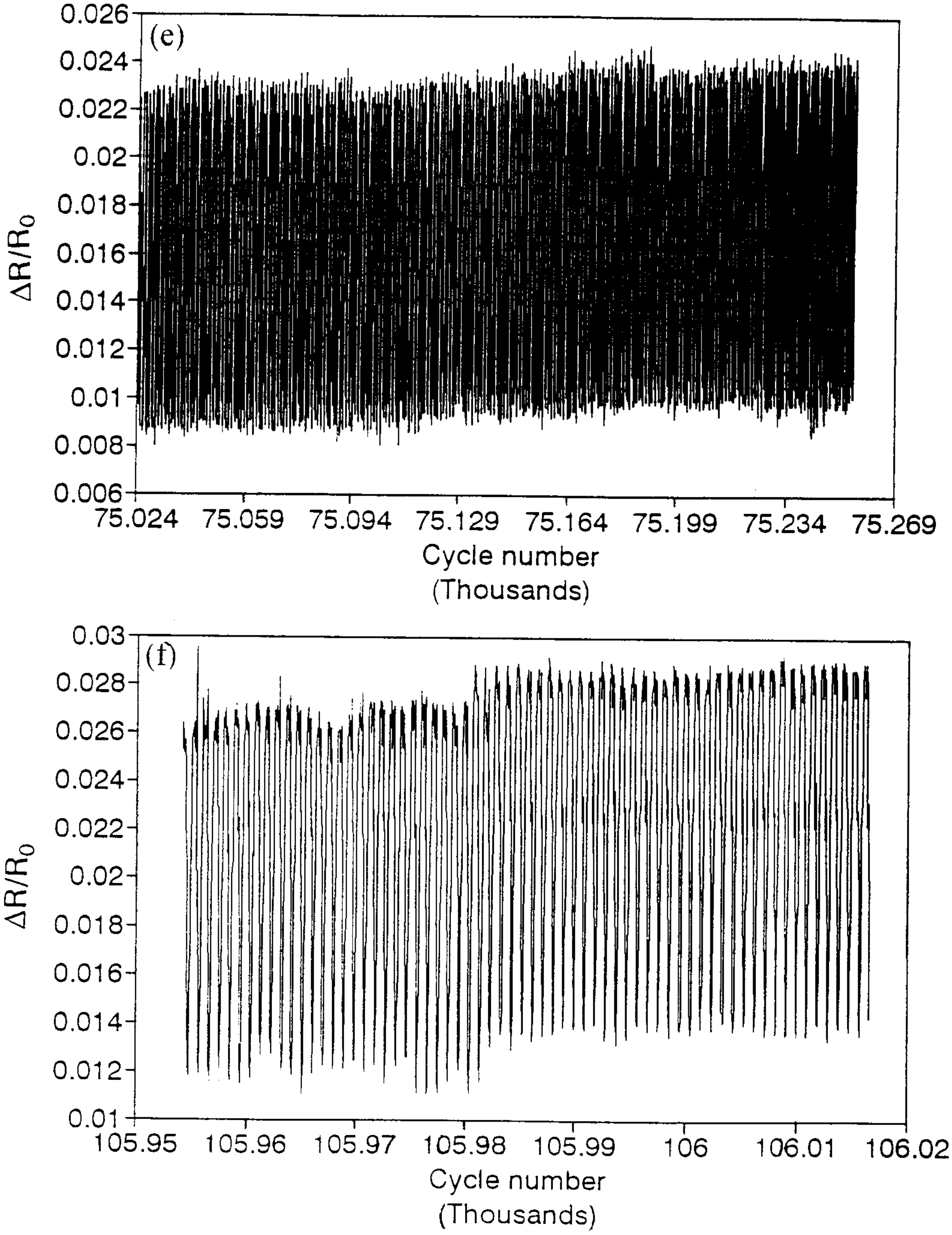

Fig. 5. Continued.

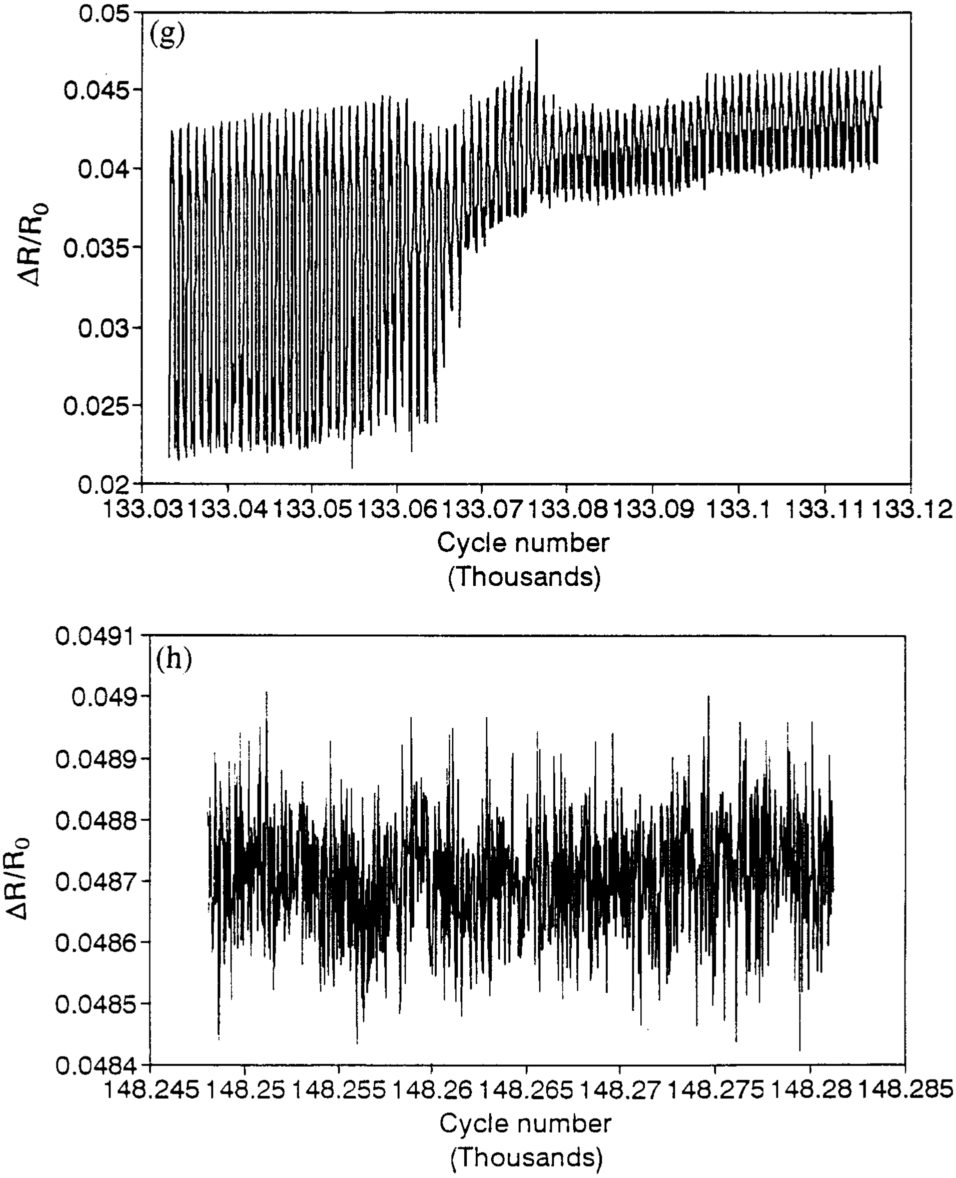

Fig. 5. Continued.

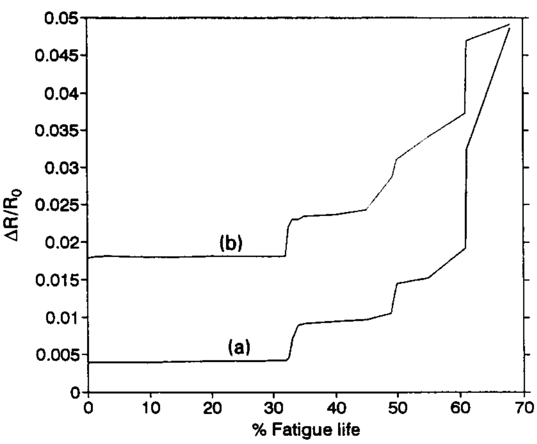

Fig. 6. Variation of $\Delta R/R_o$ in the through-thickness direction with the percentage of fatigue life during tension-tension fatigue for crossply composite. (a) Minimum $\Delta R/R_o$ at the end of a cycle. (b) Peak $\Delta R/R_o$ in the middle of a cycle.

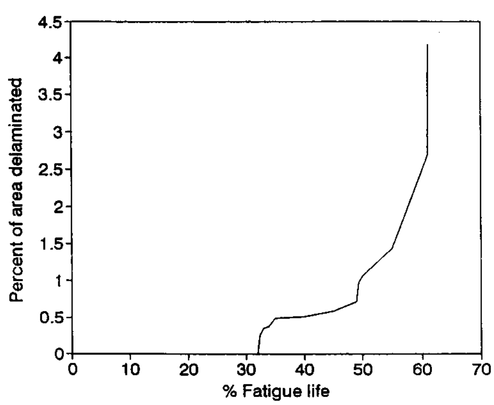

Fig. 7. Variation of the percentage of area delaminated with the percentage of fatigue life during tension-tension fatigue testing for crossply composite.

33% of the fatigue life. The resistance increased upon loading and decreased upon subsequent unloading in every cycle, thereby allowing strain sensing. The minimum resistance at the end of a cycle irreversibly increased during the first 0.1% of the fatigue life. The resistance became noisy starting at 62% of the fatigue life, at which delamination occurred rapidly and the fraction of laminate area delaminated reached 4.3%.

ACKNOWLEDGMENT

This work was supported in part by the Center for Electronic and Electro-Optic Materials of the State University of New York at Buffalo.

REFERENCES

1. C. Fischer and F. J. Arents, *Compos. Sci. Technol.*, **46**, 319 (1993).
2. R. Prabhakaran, *Experimental Techniques*, p. 16–20, Feb. 1990.
3. N. Muto and H. Miyayama, *J. Ceram. Soc. Jpn.*, **18**, 144 (1992).
4. N. Muto, H. Yanagida, M. Miyayama, T. Nakatsuji, M. Sugita and Y. Ohtsuka, *J. Ceram. Soc. Jpn.*, **100**(4), 585 (1992).
5. N. Muto and H. Miyayama, *Adv. Compos. Mater.*, **4**, 297 (1995).
6. M. Sugita, H. Yanagida and N. Muto, *Smart Mater. Struct.*, **4(1A)**, A52 (1995).
7. A. S. Kaddour, F. A. R. Al-Salehi, S. T. S. Al-Hassani and M. J. Hinton, *Compos. Sci. Technol.*, **51**(3), 377 (1994).
8. X. Wang and D. D. L. Chung, *Smart Mater. Struc.*, **5**, 796 (1996).