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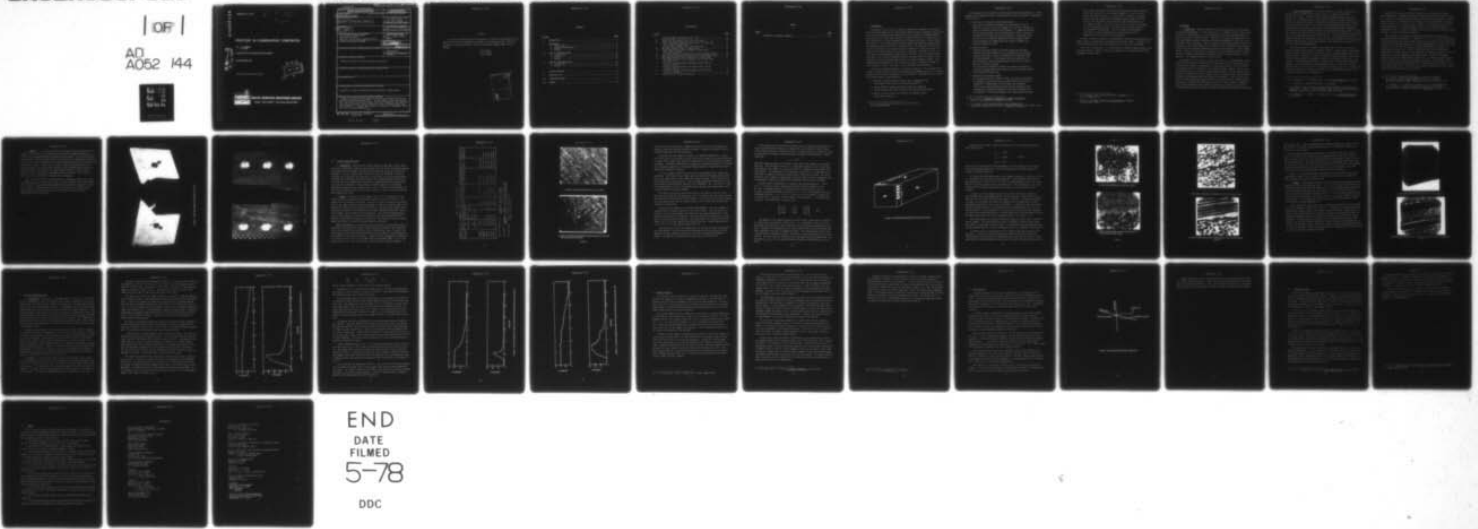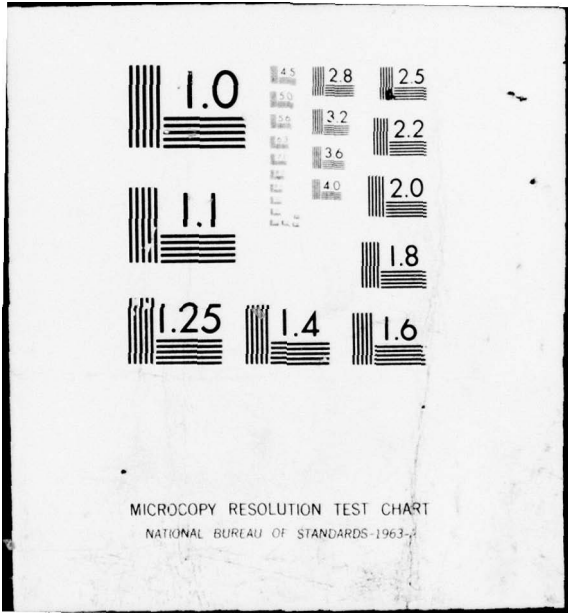UNCLASSIFIED　　　NSWC/WOL/TR-77-179　　　　　　　　　　　　　　NL

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FRACTURE IN CARBON-EPOXY COMPOSITES

BY   J. R. LOWNEY
J. F. GOFF

RESEARCH AND TECHNOLOGY DEPARTMENT

30 SEPTEMBER 1977

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Samples of graphite/epoxy composite from NRL and DRNSRDC/Carderock were analyzed optically and electrically. The NRL samples, which were notched, were fractured by tension while their electrical conductivity was monitored. The results showed that initial fracture was matrix cracking rather than fiber breakage. A fracture model based on a hierarchy of elementary failure mechanisms is being formulated.

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SUMMARY

The research reported herein was carried out in the Solid State Branch of the Materials Division and supported by the Naval Surface Weapons Center, White Oak Independent Research Fund and by the Naval Sea Systems Command under Contract SF54591.

J. R. DIXON
By direction

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ILLUSTRATIONS

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I. INTRODUCTION

The purpose of this task is to develop a quantitative approach to the failure modes of the continuous fiber, carbon-epoxy composite materials which are being used to construct the Composite Box Beam and Foil Flap structures at DTNSRDC/Carderock. These materials are based upon a mixture of Thornel 300 and GY 70 carbon fibers in a 5208 epoxy matrix. We have interacted with a group at NRL which is also studying this problem. The Box Beam material differs from the NRL fracture coupons, which are based upon Thornel 300 alone in the same matrix. We shall show in Section III in which we present an optical and electrical comparison of these two sources of materials that these materials also differ in structure. Therefore, we take as a more specific and meaningful statement of the task that we are to develop a model based upon a set of easily obtainable material parameters that will enable one to calculate the failure-fracture data of these or similar composite systems. The advantage of this approach is that it should allow one to estimate the effect of small changes in matrix, fiber type, and lay-up quickly and inexpensively.

The field of composite materials is rapidly developing. This development has tended to proceed from the previous understanding of homogenous metals. As W. Rosen points out[1] this metallic experience can be misleading. Therefore, we have continually asked ourselves three questions:

- How does a composite material differ from a homogenous metal with regard to both elastic and fracture properties?

- How do different samples of material which are supposedly similar compare and in particular the NRL and Box Beam materials?

- What is the basic process of fracture in continuous fiber, carbon-epoxy materials?

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1. B. W. Rosen, Materials Science Inc., Blue Bell Penna., private communication.

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This report has been organized to answer these questions sequentially. A brief summary of the approach to each of these questions and the conclusions, respectively are as follows:

- Review of the literature and personal contact.
  This review, the personal contacts and conversations, and in particular the work of Pellini[2] supports the view that fracture is always a structural failure of the material. In composite materials the structural elements are of the microscale; that is, they have dimensions on the order of tens of micrometers. Therefore, there will be a set of structural weaknesses of this dimension whose failure sequence will depend upon such things as geometry and type and direction of load.

- Optical studies.
  Polished and etched samples of the NRL coupons and Box Beam materials have been studied by Zeiss metallurgical and scanning electron microscopes in order to determine the structural form of these materials. It was revealed that these materials are quite different from the idealized models that have been used in the past to predict fracture.
  The two sources of materials differ structurally with regard to the matrix spacing between the lamina tow, the interlamina spacing, and the specified fiber type.

- Electrical and Fracture studies.
  We initiated electrical studies of these two sources of materials to capitalize on the fact that the carbon fibers are electrical conductors[3]. Measurements of the in-plane and transverse resistivities without applied stresses gave an indication that the degree of interlamina touching of the fibers differed in the two materials. Failure measurements of the NRL material in which

2. W. S. Pellini, Principles of Structural Integrity Technology, Office of Naval Research, Arlington, VA., 1976.

3. C. N. Owston, "Eddy Current Methods for the Examination of Carbon Fiber Reinforced Epoxy Resins," Materials Evaluation, Nov. 1976, p. 237.

the in-plane resistivity was measured simultaneously with the applied stress gave indication of the fracture sequence. At first only the matrix fractured with only a few percent fiber breakage. Subsequent behavior was more complicated and depended upon the composite lay-up. In some cases the results were consistent with the observations of Dr. Vaughan of NRL[4], in which the initial matrix fracture was followed by fiber disbonding and shear. In other cases the initial fracture was followed by delamination. Some very cursory AC measurements were made to assess the possibility of utilizing the reactive components of the resistivity for NDE purposes.

Finally, we began to explore a new mathematics[5] called Catastrophe/Bifurcation Theory, which is essentially a new approach to stability theory. It appears that this mathematics may have some limited application to this type of problem.

This task is principally a 6.2 effort which was funded by NAVSEA Materials and Structures (03522). However, there was a significant 6.1 contribution from NSWC Independent Research funds.

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4. W. H. Vaughan, Naval Research Laboratory, Washington, D. C., private communication.

5. René Thom, Structural Stability and Morphogenesis, Benjamin, Reading, Mass., 1975.

II. BACKGROUND

A. INTRODUCTION. Composite materials are entering a period of proliferating use and rapid development. In such cases there is a tendency to try to utilize past experience in other materials. In the present case the past experience being implicitly utilized is metallic theory. Since metals are generally homogenous on the microscale whereas composite materials are always inhomogenous on this scale, this experience can be misleading[1]. One can surmise that it might be better to base ones experience on the fracture behavior of a material like reinforced concrete because in that case the structural characteristic of the fracture process is self-evident. However, in the case of concrete the interaction of the reinforcing rods is minimal whereas in composite materials the analogous interaction between fibers is quite important. The composite problem is more involved.

The purpose of this section is to show that there is a contemporaneous, active viewpoint that supports our contention that the fracture of the carbon-epoxy composite materials should be considered as a structural failure. Of course Pellini[2] has pointed out that all fracture is a structural failure. However, in this case we mean that there is a series of sharply defined elements that are inherent to the formation of the material such as matrix, fiber, fiber-matrix bond, and interlamina bond whose failure precipitates the failure of the material. One can hope to formulate a fracture model based upon the failure criteria for each of these elements rather than a criterion for the material as a whole. Until recently attempts of this sort have been based upon unrealistic models of the material and have used averaging techniques that precluded a complete mathematical statement of the problem.

B. <u>GENERAL CONSIDERATIONS</u>. In the past there was an effort to apply the formalisms that were common to the theory of metals to composite materials[6,7]. However, the striking difference between such an inhomogenous and anisotropic substance as a composite material and a nearly homogenous, isotropic substance as a metal led to great difficulties. Customary stress-strain relations that are consistent with the microscopic view are not easily defined in the macroscopic sense. The moduli of these materials vary greatly in different directions. Supposedly similar materials differ markedly because of their structural nature; that is, there can be resin rich and resin poor regions, the fiber tow can be compacted in different ways, or the interlamina bond can be thick or thin. As a result the response of a composite material to a particular stress can be quite complicated, have a statistical variance between samples of the same material, and in general be quite sensitive to quality control during manufacture.

The recent work by I. M. Daniel[8] seems important, for he has carried out an extensive study of the fracture of composite samples. He has actually measured the strains in the vicinity of the crack tip and has described the damage zone at that point just prior to fracture. Both he and Mandell[9] found the concept of the stress intensity factor useful even though its meaning with respect to composites is uncertain. It would appear that there is in some sense an averaging of constituent properties so that the Griffith-Irwin dependence applies; that is, the critical fracture stress varies inversely as the square root of the crack size. However, they find a large amount of scatter in the values for a composite material due to the statistical nature of the inhomogeneities.

6. H. J. Konish,Jr., J. L. Swedlow, and T. A. Cruse, <u>AIAA Journal</u>, <u>11</u> 40 (1973).

7. D. C. Phillips, J. Composite Materials, <u>8</u> 130 (1974).

8. I. M. Daniel, "Biaxial Testing of Graphite/Epoxy Composites Containing Stress Concentration - Part I," Technical Report AFML-TR-76-244, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio, Dec. 1976.

9. J. F. Mandell, F. J. McGarry, S. S. Wang, and J. Im, <u>J. Composite Materials,</u> <u>8</u>, 106 (1974).

Yokota[10] has outlined an electrical method of monitoring a carbon-epoxy during manufacture. Although the process is propietary and details are not given, it is a means for checking the epoxy during fabrication of the composite. He has discovered that it is important that the epoxy be heated sufficiently to outgas it before pressurizing it in the autoclave. However, it is important that the epoxy not be overheated to the gelation point so that it will flow under pressure. Consequently there is a range of temperature within which pressure should be applied in order to obtain a high quality product.

The hierarchy of micromechanical mechanisms which relate to macroscopic fracture have been discussed by J. E. Masters et. al.[11]. In a private conversation he stressed the need for fracture models based upon structural failures rather than linear-elastic fracture mechanics that is based upon homogeneity. There were presentations by E. M. Wu and I M. Daniel at the ARPA/AFML Review of Progress in Quantitative NDE (June 14-17, 1977) at Cornell University which stressed the shortcomings of linear-elastic fracture mechanics as applied to composites. Dr. Wu showed that a judicious mapping of the applied stress and material strength contours gave the loci where fracture would occur. Dr. Daniel discussed the geometrical complexities associated with composite failure. He showed the complex strain patterns which occur in composites by using photoelastic coatings. It is clear that general failure criteria in these materials depend upon a multiplicity of threshold values rather than on a single one.

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10. M. J. Yokota, "Exploratory Development of Controlled Composite Processing," Technical Report AFML-TR-77-76, Pt. I, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio, May 1977.

11. J. E. Masters, Y. T. Yeow, M. R. Louthan, Jr., K. L. Reifsnider, and H. F. Brinson, "A Iniatitive Fault Tree Analysis for the Tensich Failure of Fibrous Laminated Composites," Composites, April 1977, p. 111.

C. <u>SUMMARY</u>. It is becoming accepted that composite materials should be considered as structures whose elements are statistically arranged and whose basic characteristics depend markedly on manufacturing procedures. Our own measurements give an example of the structural nature of the fracture of carbon-epoxy composites. In Figure 1 is shown the fracture of a sample whose cross-ply angle is greater than some critical value. In this case two cracks emanated from the notch-tip in a direction parallel to the two fiber directions with accompanying delamination between the cracks. The behavior for angles smaller than this critical angle is shown in Figure 2; only one crack emanated from the notch-tip. This crack was parallel to one set of fibers. The other set was sheared.

Thus it is clear that the fracture problem in composites differs from that of simpler materials in that one will have a series of failure modes whose sequential failure will depend upon design geometry and the pattern of applied stresses. It is necessary to determine how the fracture characteristic of each of these modes depends upon the properties of the constituents and the constraints imposed upon them by the lay-up.
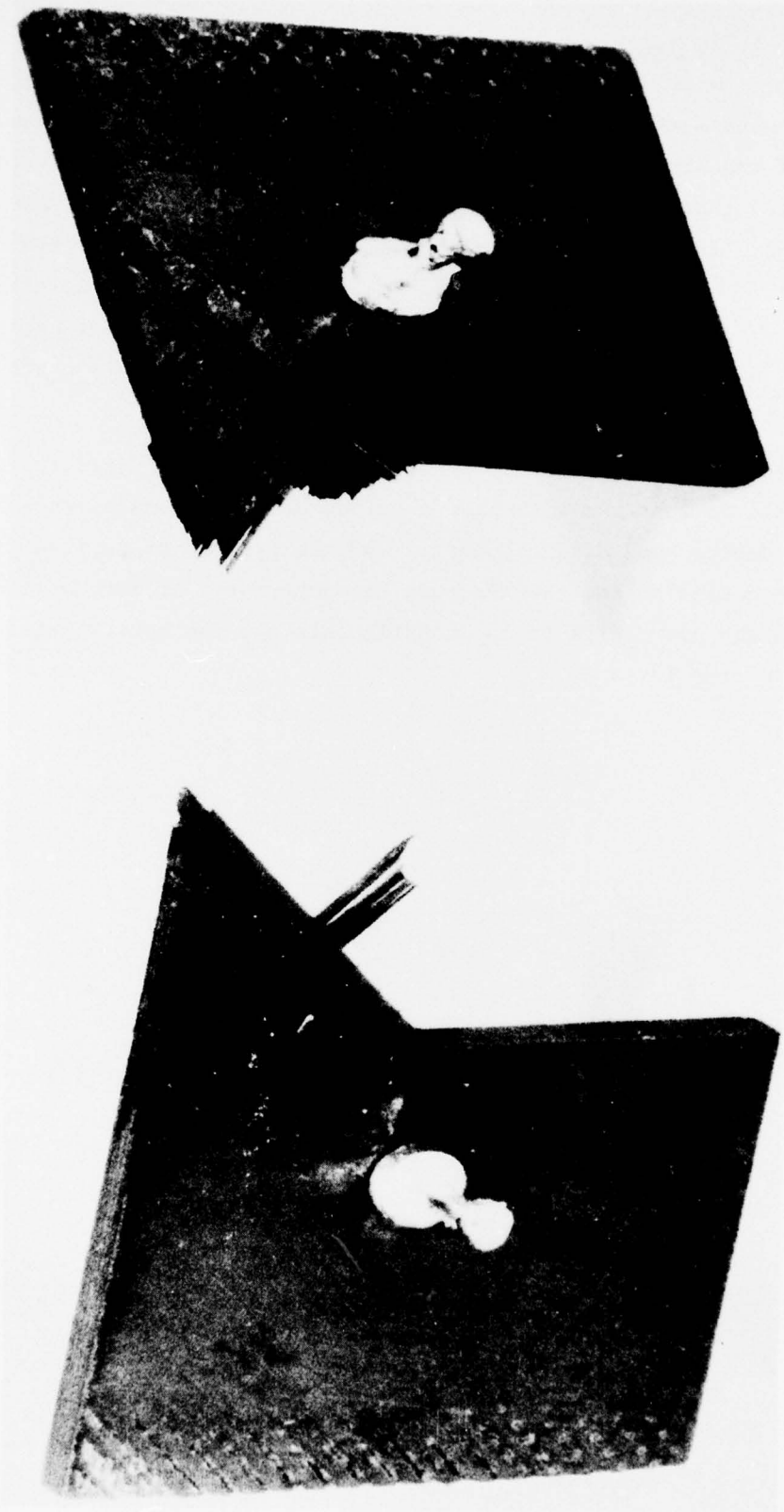
FIGURE 1 FRACTURED SAMPLE WITH CROSS-PLY OF ± 45°

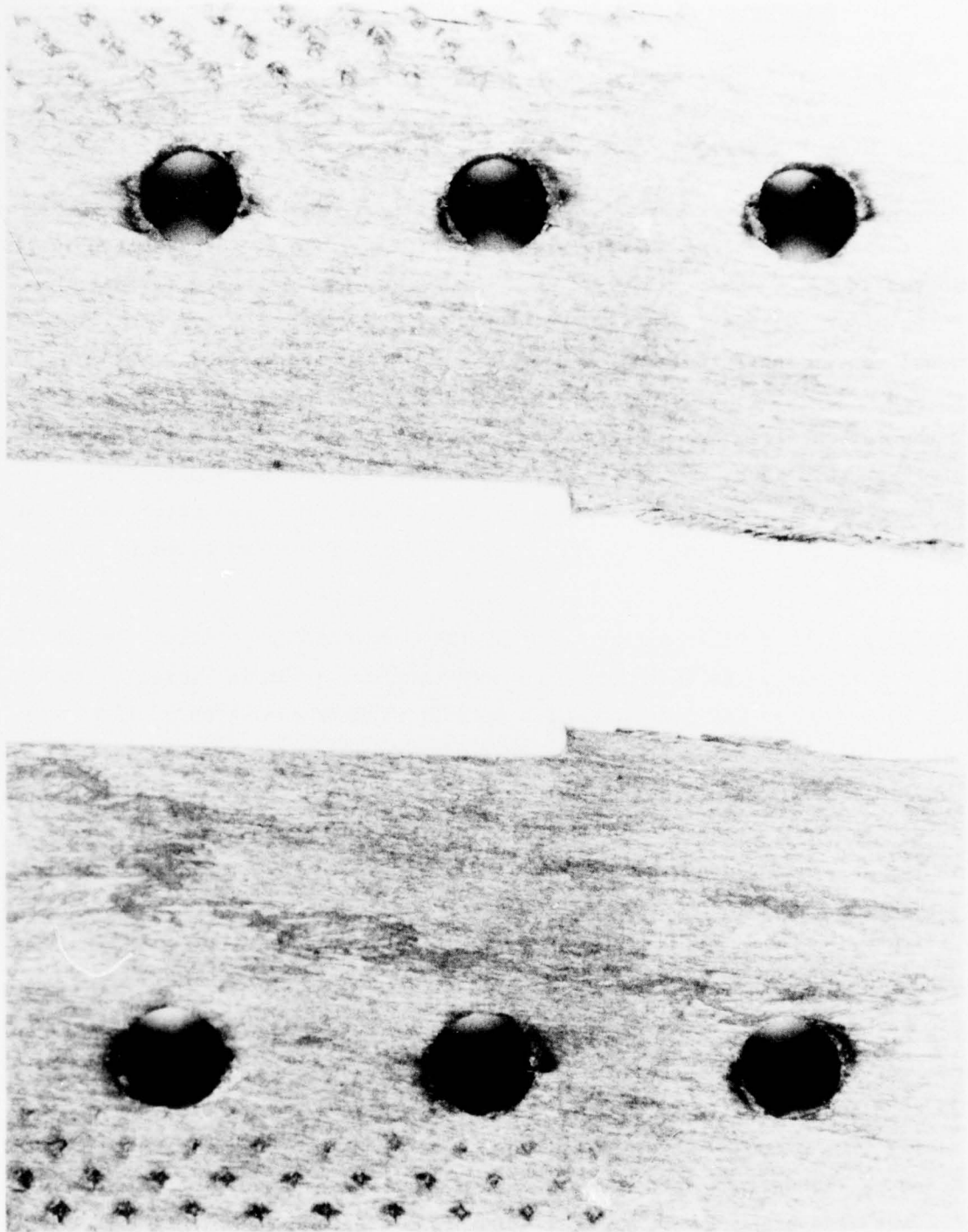FIGURE 2 FRACTURED SAMPLE WITH CROSS-PLY OF $\pm 6\frac{1}{2}^{\circ}$.

III. INITIAL CHARACTERIZATION

A. INTRODUCTION. There were two initial questions: What does a real, continuous fiber, carbon-epoxy composite material look like and how do the research samples being measured at NRL compare in detail with the materials being used to test the Box Beam? In order to answer these questions we used two techniques. The first technique was conventional and consisted of studies made with a Zeiss metallurgical microscope and a scanning electron microscope. The second technique utilized the fact that the carbon filaments are electrically conducting[3] while the epoxy matrix is an electrical insulator. These electrical studies enabled us to determine qualitative and quantitative characteristics such as interfiber and interlamina contact and the relative fracture of the fiber and matrix. These two types of measurements support each other.

B. RESULTS. The details of the samples, their source, composition, lay-up, and the various measurements that were made upon them are given in Table 1. The purpose of these studies was two-fold. The optical studies consisted of investigations of the sample surfaces and were undertaken to develop a realistic model of the material for later use in the analysis of fracture. The electrical studies, which were made in conjunction with the optical ones, gave information about the interior of the samples, which could not be seen optically. This interior information consisted of such things as fiber contacts (both in-plane and interlamina) and the behavior of the fiber with respect to the matrix. Since these two types of studies support each other, they will be reported together in context.

Sample preparation for the optical studies consisted of the following series of operations: cut, polish with Linde A (final), gold plate, optical study, repolish, etch in an RF plasma of O_2, gold plate, and a final optical study. In order to be certain that a true picture was being obtained several samples of the NRL materials were examined by the optical techniques. Figure 3a shows a photograph of sample NRL (577) which was made using the Zeiss Metallurgical Microscope (ZMM). It is a x200 magnification of the sample plane taken to show the fibers running lengthwise. It

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TABLE 1. PARAMETERS OF COMPOSITE SAMPLES

Sample	Structure			Critical Stress		First Relaxed Stress	Fracture	
	Lay-up	Fiber type	Volume	Kg/cm²	Newtons/meter²	Newtons/meter²	Type	Sequence
Box Beam	0°	Thornel 300	63.8%					
	$(0°_3, \pm45°)_8$	Th 300 & GY70	56.9%					
	$0°_3$	Th 300						
	$(\pm45°, 0°_3)_8$	Th 300 & GY70						
	0°	Th 300						
NRL(1 un-labeled)	$(\pm6½°)_{8s}$			72.4	7.10×10^6	4.47×10^6	single crack	-
NRL(2 un-labeled)	$(\pm11°)_{8s}$			233.2	2.29	1.25×10^7	single crack	simple
NRL(577)	$(\pm45°)_{8s}$	Th 300	70%	535.7	5.25×10^7	2.43×10^7	dual crack	simple
NRL(578)	$(\pm45°)_{8s}$	Th 300	70%	566.2	4.57×10^7	2.13×10^7	dual crack	simple
NRL(580)	$(\pm45°)_{8s}$	Th 300	70%	322.4	3.16×10^7	2.13×10^7	dual crack	complex
NRL(607)	$(\pm37½°)_{3s}$	Th 300	70%	297.6	2.92×10^7	2.28×10^7	dual crack	complex

Sample cross-sectional areas:
    unlabeled(1) 0.0781 in² = 0.504 cm²
    unlabeled(2) 0.0625 in² = 0.403 cm²
    labeled   0.0625 in² = 0.403 cm²

Notch lengths:
    unlabeled(1) 0.469 in = 1.19 cm = 60% of sample width
    unlabeled(2) 0.313 in = 0.794 cm = 31.3% of sample width
    labeled   0.563 in = 1.43 cm = 56.3% of sample width

Conversion factors: 1 Kg/cm² = 9.8 × 10⁴ Newtons/meter²

                    1 Newton/m² = 1.4507 × 10⁻⁷ Ksi.

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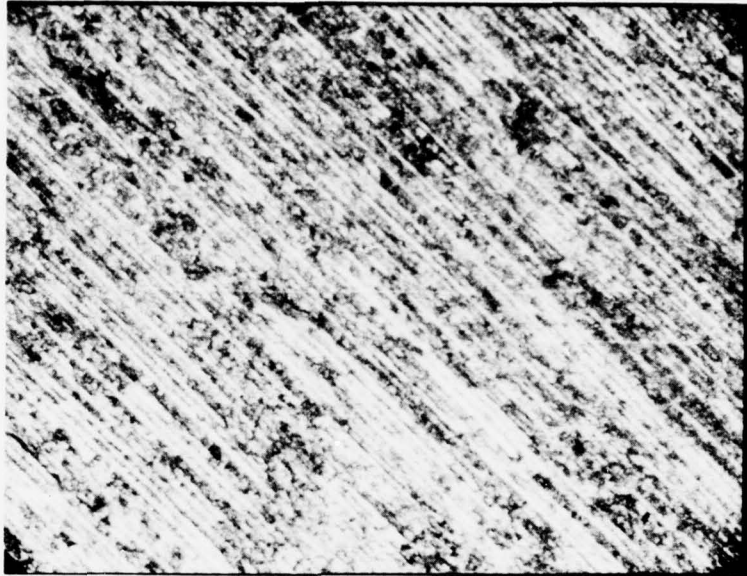
(a) SAMPLE FROM NRL SHOWING FIBERS IN PLANE (X200)

(b) SAMPLE FROM NSRDC-CARDEROCK SHOWING FIBERS IN PLANE (TOP)
AND ON EDGE (BOTTOM) (X200)

FIGURE 3

can be seen that the fibers are irregularly spaced but still do not resemble the tortuous arrays that Owston[3] postulated as models to explain his studies of the AC electrical properties of similar material.

The model of an ideal composite, however, is completely unrealistic. In this case there would be an infinite electrical resistivity except in the fiber directions. In reality these samples show electrical conduction in all directions and only minor anisotropy. Therefore, there must be in-plane and interlamina touching of the fibers. The interlamina touching is an important weakness because it means that the interlamina shear strength is reduced.

In a real composite there must be at least four parameters that describe the conduction. There would be an average length L_c between interlamina contact points made by the fibers and a similar length for contact in-plane. There would also be two average fiber contact areas A_c. The resulting current path through such a material would be quite different from that for a homogenous one. The results of some of the measurements that will be described below indicate that equipotential lines in these material are quite irregular.

Two types of electrical contacts were made to the samples. The first type which was tried consisted of small plugs (1/16" in diameter and 1/16" long), pressed in holes that had been drilled into the samples. The second type consisted of pressing clean copper plate onto the sample surfaces after they had been polished and coated with silver paste.

Two resistivity samples were cut from sample NRL(580). The first sample was oriented with its long axis parallel to one set of fibers. This set would be expected to carry all the current; since the other set, being perpendicular to the current path, would not be expected to carry any current unless there were a great deal of in-plane interfiber touching. The second sample was oriented with the two fiber sets at 45° to the long axis and current direction. Sample dimensions were 3/4" x 1/4" x 1/16" with a plug separation of 1/2".

The resistivity of the first sample which was derived from these measurements was 0.018 ohm-cm. The second sample was measured to give some indication of the magnitude of fiber contacts since there is no direct current path between the probes. Nevertheless, the measured resistivity was only 0.014 ohm-cm.

This decrease in the resistivity ρ between the first and second orientations indicates that interlamina contact is allowing both sets of fibers to carry current. If one presumes that this interlamina contact is allowing current to follow in a zigzag path then an elementary calculation which disregards three dimentional effects shows that

$$\rho_{45} = \rho_0/\sqrt{2}$$

where the subscripts refer to the angles of the fibers with respect to the current direction. The calculated value is $\rho_{45} = 0.013$ ohm-cm, which is only 7% less than the measured value. Thus it is clear that there is a substantial interlamina fiber-fiber contact. A measurement of the through-ply resistivity, i.e. perperdicular to the fibers, by means of the plate contacts gave a value of 3.57 ohm-cm. The interpretation of this number is complicated by the fact that the fibers themselves have an anisotropic conductivity and that the interfiber contact area is not known.

We made similar measurements on a sample of the Box Beam material. A 1/2" x 1/2" x 1" bar was cut from a representative piece of material given to us by Mr. Couch of DTNSRDC/Carderock. Plug contacts were made as shown in Fig. 4. Currents were passed between axial pairs (i = 1, 2, or 3) and voltages were measured between the pairs (j = 1, 2, or 3). In this way it was possible to define the resistances R_{ij} as the ratio of the voltages j to the currents i. The resulting R_{ij} are given - for convenience - in matrix form

$$R = \begin{bmatrix} 0.128 & -0.022 & 0.060 \\ -0.022 & 0.118 & 0.062 \\ -0.060 & 0.062 & 3.06 \end{bmatrix} \text{ ohms}$$

The geometry of the sample and the use of point-like contacts make it difficult to convert these resistances to resitivities. It is important to notice that the off-diagonal resistances are much larger than could be expected from probe misalignment. Therefore, the equipotentials for axial current flow are quite different from those expected on the basis of homogeneous metals. The current paths must be indirect and involve interlamina crossing. Therefore, a probe contact must be used with caution. For this reason we introduced the second type contact described above. The fact that $R_{ij} = R_{ji}$ is to be expected from the Reciprocity Theorem of Passive Circuits.
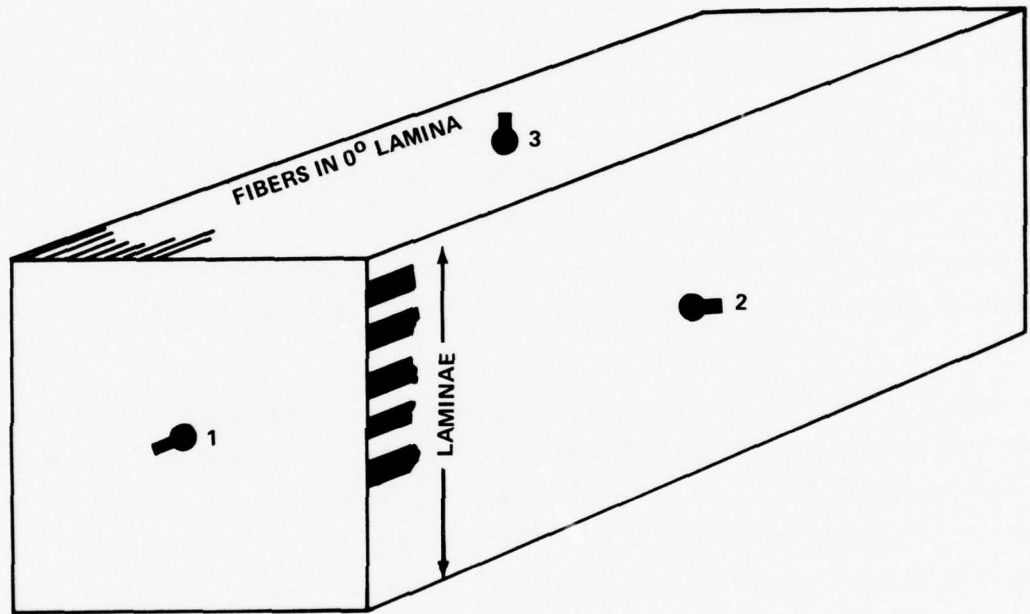
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FIGURE 4 BOX BEAM SAMPLE (NSRDC) WITH ELECTRICAL PLUGS

The values of the axial resistivities measured on the Box Beam material with plate contacts are:

$$\rho_{11} = 0.0051$$

$$\rho_{22} = 0.0094 \qquad \text{ohm-cm}$$

$$\rho_{33} = 6.85$$

Plate resistivity measurements made subsequently on the NRL samples were in agreement with the plug contact ones. This was expected since the plugs penetrated through the laminae completely.

We studied the interlamina region of our samples optically to establish if there is a correlation with the electrical measurements. The interlamina region of the Box Beam material is shown in Fig. 3b, which is a ZMM photograph, x200. The carbon fibers shown running length wise in the upper part of the photograph are not exactly coplaner with the surface and therefore, are seen as extreme elliptical cross-sections.

In order to see the internal structure of the two samples more clearly, we used the SCM with a x1000 magnification. The NRL material is shown in Fig. 5a as an edge view of ± 45° plies, and so the ends appear elliptical. The Box Beam material is shown in Fig. 5b where the fibers run length wise in the upper portion of the photograph and on-edge at 45° in the lower portion. It appears in these photographs that the interlamina layer is much thicker for the Box Beam sample than it is for the NRL material. This observation is consistant with the fact that the transverse resistivity of the Box Beam material is greater than that of the NRL material because one would expect there to be less interlamina contact. Their ratio is 1.9.

These photographs also show the difference between the fibers used to construct the two samples. The Thornel 300 fibers have a distorted, circular cross-section while the GY 70 ones have a dog-bone shape. The different shapes and types of fiber and the different amounts of fiber touching must affect the strength of the material.

A curious result is the difference of the effect of the same etch on the two materials. In the case of the NRL material the carbon fibers were etched away leaving ridges of matrix material interconnected as can be seen in Fig. 6a. On the other hand, the Box Beam material shown in Fig. 6b shows the matrix etched away with

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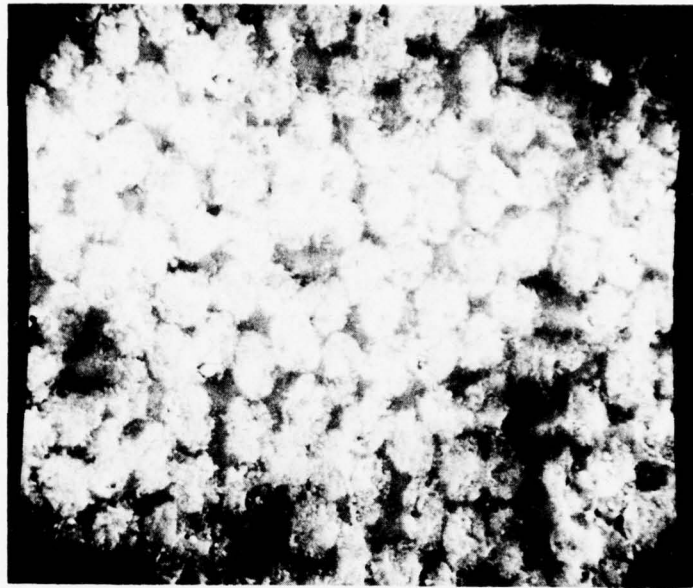
(a) NRL SAMPLE SHOWING FIBERS ON EDGE (X1000)
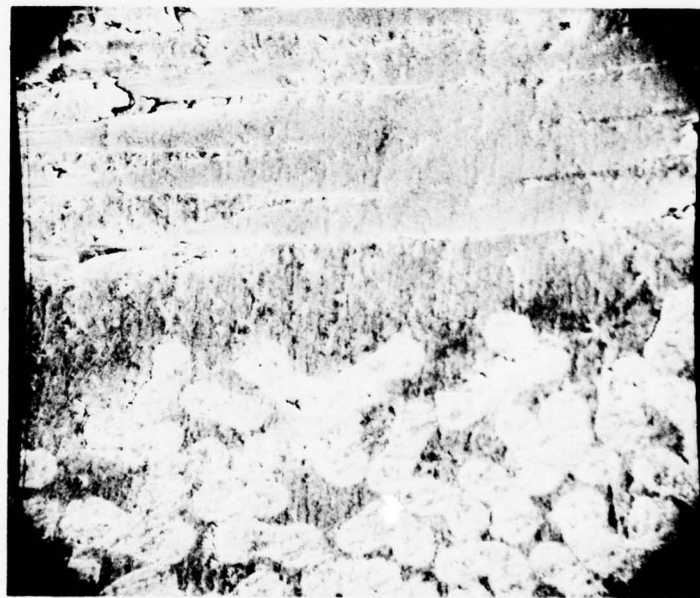
(b) NSRDC SAMPLE SHOWING FIBERS LENGTHWISE (TOP)
AND ON EDGE (BOTTOM) (X1000)

FIGURE 5

(a) NRL SAMPLE SHOWING INTERLAMINA REGION AFTER ETCHING (X1000).

(b) NSRDC SAMPLE SHOWING INTERLAMINA REGION AFTER ETCHING (X1000).
FIGURE 6

fiber spikes left. We do not understand this different behavior but suggest that it may indicate a difference in the manufacturing process of the matrix and/or fiber materials in the two cases.

Another important difference between the two materials can be seen in Fig. 7, which is the same as Fig. 6 except that the magnification is only x50. The NRL material shown in Fig. 7a is more uniform than the Box Beam material shown in Fig. 7b. The Box Beam material shows the fibers collected in large groups called tows, that are separated by matrix-rich layers in much the same way as the lamina are separated. On the other hand the tows in the NRL material are nearly invisible. Again the shear behavior of the materials must be affected by this difference.

Finally, it should be noted that a delamination that is visible in the lower, longitudinal lamina in Fig. 7b would constitute a weak area in the composite and would initiate fracture at subcritical loads.

C. SUMMARY. Representative samples of the NRL and Box Beam materials have been compared by optical and electrical means. The electrical tests are consistent with the optical ones and have the added advantage of giving information about the interior of the material that can not be seen optically. We are trying to develop an electrical means of obtaining information about the interior structure and processes of the carbon-epoxy materials that other groups try to obtain by constructing glass-luicte models. It is possible that glass-lucite materials differ from the carbon-epoxy materials in some fundamental way.

These comparisons indicate that there are differences between the two materials that can be expected to affect their behavior. In addition to the known differences between their lay-ups, there are differences within the lay-ups. Specifically the lamina of the Box Beam material are composed of rather well-defined tows which are connected together by matrix-rich regions in much the same way as are the lamina themselves. The NRL materials show much better interspacing of the tows in this respect. On the other hand the NRL materials have a very thin interlamina bond. As a result the electrical contact between the lamina is greater in that material than it is for the Box Beam. Every such contact point can be expected to be a defect that will lessen the shear strength of the bond.

(a) NRL SAMPLE SHOWING FIBERS ON EDGE AFTER ETCHING (X50)

(b) NSRDC SAMPLE SHOWING FIBER LENGTHWISE AND ON EDGE AFTER ETCHING (X50).

FIGURE 7

IV. FRACTURE CHARACTERIZATION

A. INTRODUCTION. Our view of a composite material is that it is a structure which is composed of a series of elements: fiber, matrix, fiber-matrix bond, interlamina bond. The optical studies indicate that there may be an additional element, the intertow bond. These optical studies - in combination with the electrical ones - indicate that there are variations in these elements that suggest weaknesses in the material design. There seem to be at least two that can be identified at this time. The random position of the fibers results in variations in the constraints imposed upon the matrix by the fibers and is possibly a shear weakness. This randomness in the fiber position also leads to interlamina contact between fibers. These contacts must result in shear weakness in the interlamina bond because these touching fibers can not support shear.

Ultimately one would like to know the mechanical strengths of these elements; these data do not exist as a complete set at this time to our knowledge. At present it would be of great help to know the relative strengths of these elements in forming the fracture model. Some qualitative data have been obtained by fracturing a number of NRL sample coupons with an Instron Universal Testing Instrument. The results corroborate a verbal communication by Dr. Vaughan of NRL but also give additional information; that is, there is a critical lay-up angle for which the failure mode changes. An electrical technique in which the conductance of the samples is monitored during strain and failure has been developed to determine the fracture behavior of the fibers during the fracture process.

B. RESULTS. A series of NRL sample coupons were fractured. Some of these were labeled by NRL and some were not. The dimensions of all labeled coupons were 1 1/2" x 1" x 1/16" with a notch 1/32" wide and 9/16" long cut in the center of the long dimension. The dimensions of the unlabeled samples varied and are given in context. All samples were pulled perpendicular to their notches at a constant rate of 0.05 cm/min. The resulting supported tension was recorded on a strip chart.

The first sample which was measured was unlabeled. It had a lay-up of $(\pm 6\frac{1}{2}°)_{8s}$ and dimensions of 13/16" x 25/32" with a 1/32" wide notch 15/32" long cut in the center of a 13/16" edge. The sample was 0.10" thick. The result of straining this sample to fracture is shown in Fig. 2. The three holes that can be seen in the sample were placed there by NRL and did not affect our results.

The complete failure of this sample occurred in a sequence. When the supported tension reached 36.5Kg, a crack appeared which ran from the notch tip to the sample edge. A check of the sample for electrical continuity after it had been removed from the Testing Instrument showed that a large number of the fibers were still intact; in fact the sample still held together. However, under subsequent strain the sample would support only 2.3 Kg and gradually completely pulled apart. Subsequent metallurgical micrograph studies showed that the crack ran from the notch-tip parallel to one of the sets of fibers. After this initial crack, the other set of fibers pulled and then sheared.

In order to distinguish between the cracking of the matrix and the breaking of a fiber, we monitored the electrical resistance while fracturing sample NRL(577). Leads were attached as shown in Fig. 1 to be close to the fracture without interference. In retrospect these contacts could have been farther away.

A current from a DC power supply was passed through the sample. Its value was obtained by measuring the voltage drop across a 0.1 ohm standard resistor, and its trace recorded. The behavior of this trace is shown in Fig. 8. Some changes in the sample that occur prior to fracture produce small changes in the sample current. These changes which seem to indicate a decrease in the sample resistance may indicate that individual filaments are being compacted more tightly in the fibers. Thus the fiber resistivity may decrease although the filament resistivity would be expected to increase. The initial sample resistance was 0.19 ohms.

The trace of the tension that arose from the constant strain rate is shown in Fig. 8 as well. Fracture is clearly indicated by the sudden drop of this tension from 216 Kg. The non-linearity prior to fracture is not understood at this time. At fracture the supported tension dropped within about $\frac{1}{2}$ sec from 216 Kg to 100 Kg. Thereafter, it gradually decreased as the sample disintegrated and separated. On the contrary the initial drop in the sample current was only 2%. The corresponding change in the sample resistance R_s was:
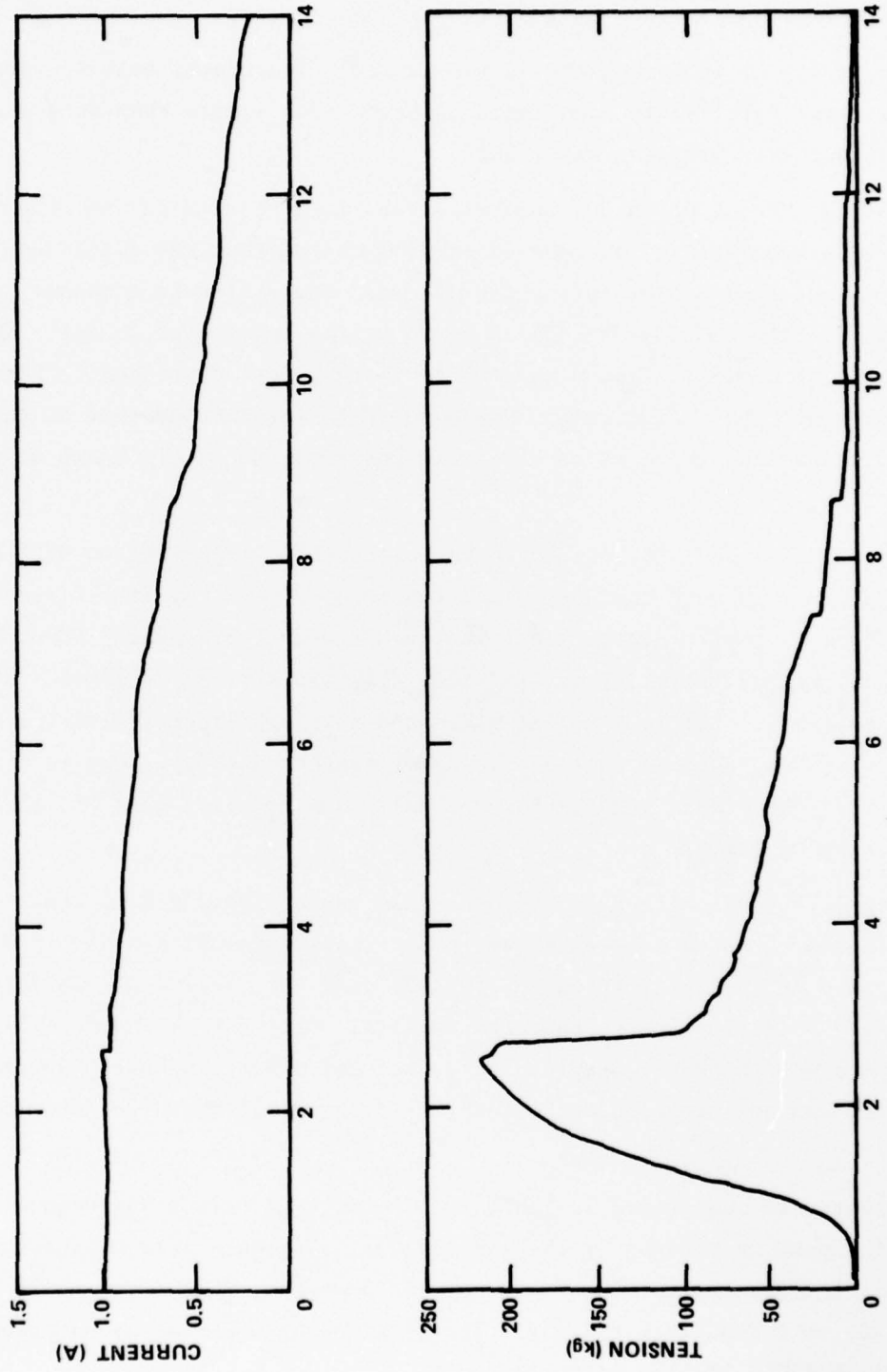
FIGURE 8   VARIATION IN SAMPLE CURRENT AND APPLIED STRESS DURING FRACTURE (NRL 577)

$$\frac{\Delta R_s}{R_s} = \frac{\Delta I_s}{I_s} \cdot \frac{(0.1 + R_s)}{R_s} = 3.1\%$$

Clearly, fiber breakage was a minor part of the failure process.

Examination of Fig. 1 shows how the sample failed. Two cracks emanated from the crack tip; one was parallel to each set of fibers. The sample then delaminated and separated without ever breaking any fibers.

A second sample, NRL(2) which was unlabeled, was investigated in the same way. Its dimensions were identical to the labeled samples except that the notch was only 5/16" long. The sample had a cross-ply angle of $\pm 11°$ and failed in a manner similar to that shown in Fig. 2 but at a tension of 96 Kg (as opposed to 36 Kg). The stress/electrical test shown in Fig. 9 is similar to that just described in Fig.8 although the fracture type is different. However, it is interesting that at the moment of stress relaxation there is no change of current. No fibers broke at that point.

Two samples NRL(578) and NRL(580) which were supposedly identical to NRL(577) were studied. They both showed the dual crack fracture pattern but stress/electrical patterns differed. Their initial electrical resistances were nearly identical: 0.15 and 0.14 ohms, respectively. Their critical fracture stresses differed: 188 and 130 Kg, respectively. Sample NRL(578) exhibited a stress/electrical trace similar to that shown in Fig. 8 while that obtained for sample NRL(580) shown in Fig. 10 is quite different. The stress and the current fall in steps that must indicate a progressive breaking of lamina.

Another sample NRL(607) with a $\pm 37\frac{1}{2}°$ cross-ply angle showed a dual crack fracture like that in Fig. 1 and a complex stress/electrical pattern like that in Fig. 10. This sample had an initial resistance of 0.090 ohms and a critical fracture tension of 120 Kg. A final point is that the critical angle for which the failure mode changes from simple matrix fracture with subsequent fiber disbonding and shear to matrix fracture and delamination without any fiber fracture must lie between $11°$ and $37\frac{1}{2}°$.

All our results are summarized in Table I. The critial stress has been computed by dividing the maximum tension by the sample cross-sectional area measured at the clamps. This ignores implicitly the effect of the notch, which must be included eventually in some more sophisticated way. The first relaxed stress is computed from the first measurable tension after fracture.
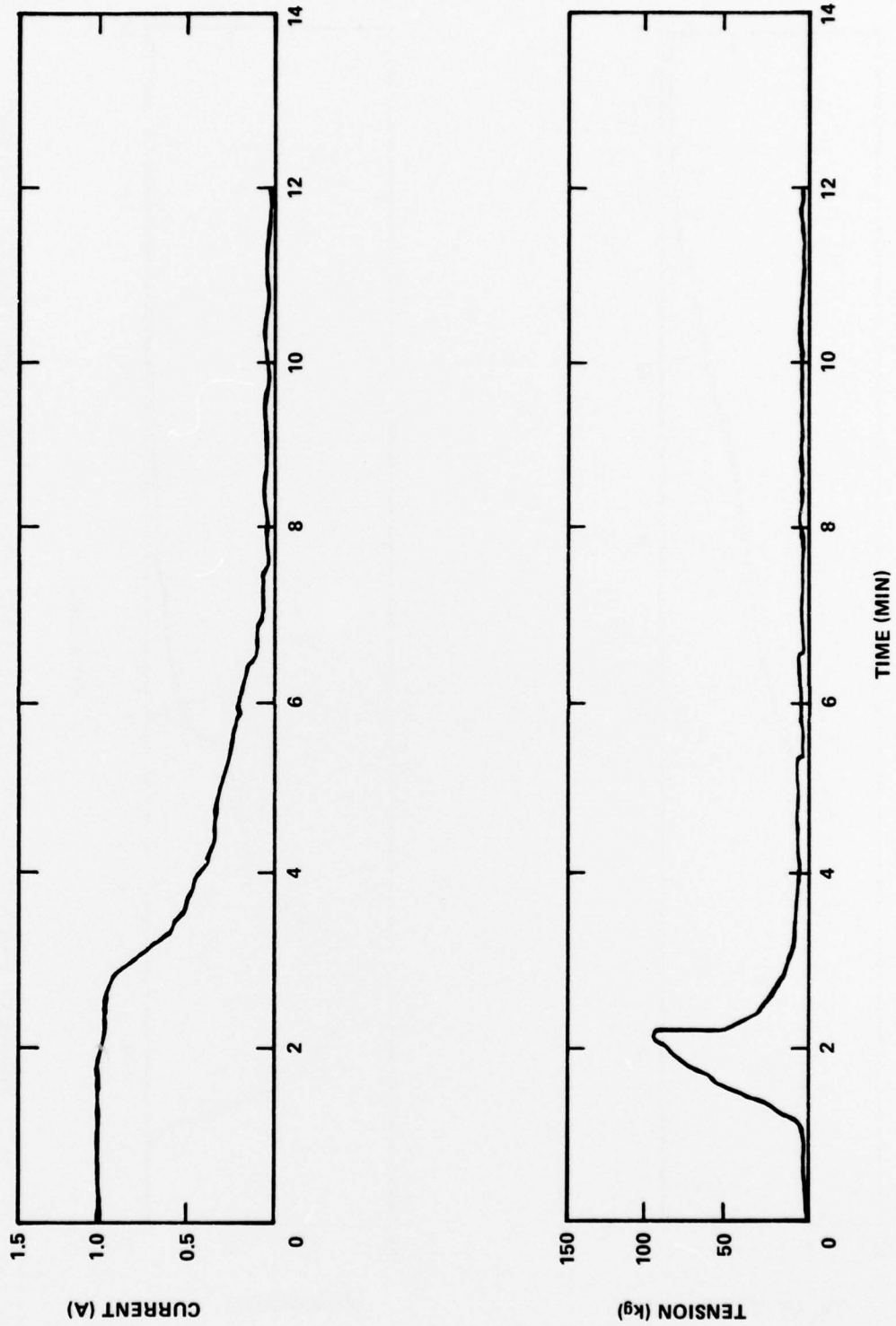
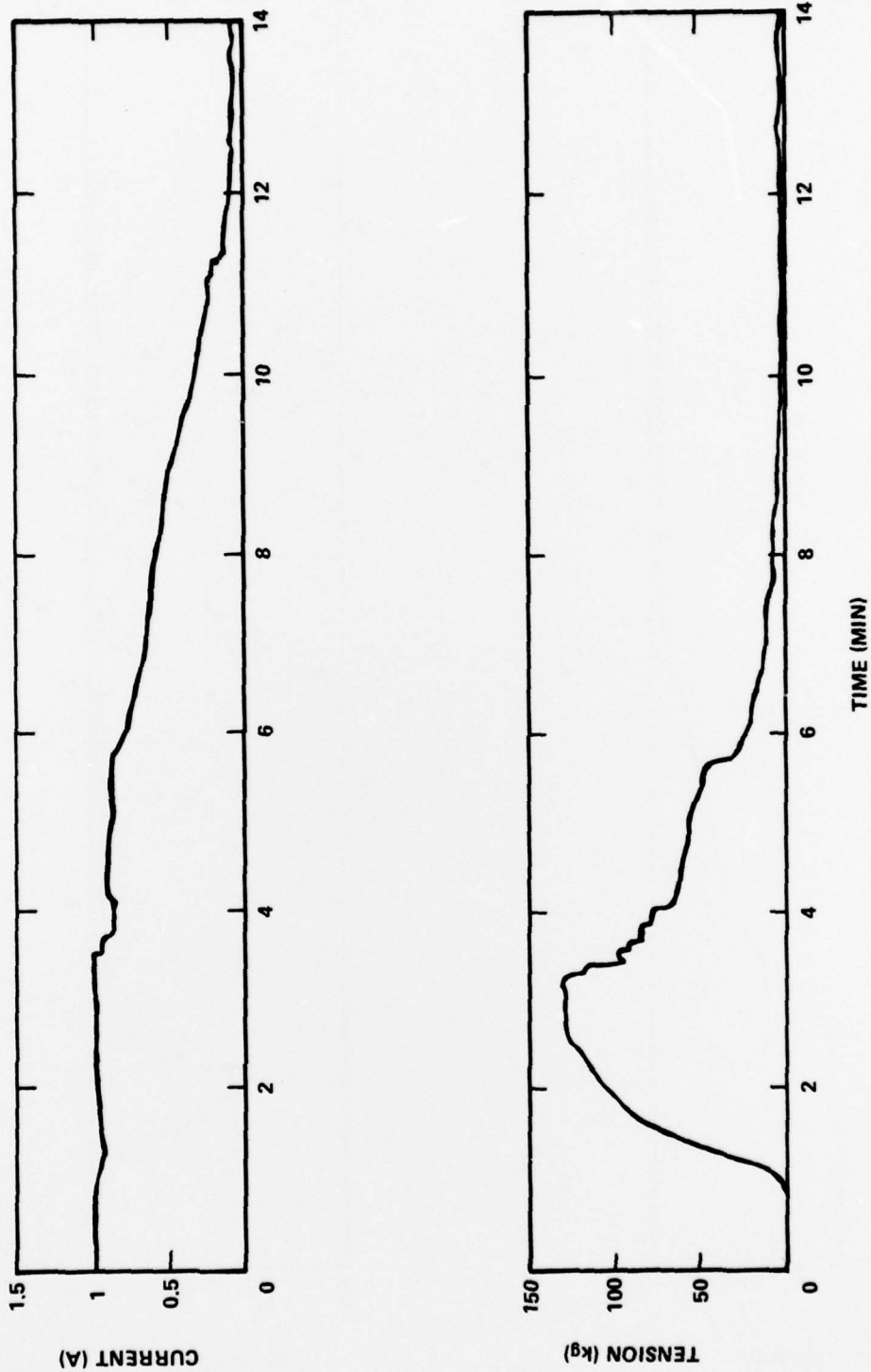FIGURE 9 VARIATION IN SAMPLE CURRENT AND APPLIED STRESS DURING FRACTURE (NRL 2)

FIGURE 10 VARIATION IN SAMPLE CURRENT AND APPLIED STRESS DURING FRACTURE (NRL 580)

V. FRACTURE MODELING

The characterization studies that we have made show that the continuous fiber, carbon-epoxy composites are complex, inhomogenous structures. The two kinds of materials that have been investigated differ in lay-up and composition. One antici-pates that their failure patterns will also differ, but this point has not yet been investigated. We have observed already that the failure patterns of just the NRL samples depend upon their structural geometry.

All the NRL samples which we studied have a notch which is located symmetrical-ly with respect to the lay-up angle θ; that is, the lay-up was given as $\pm \theta$. The asymmetrical case, for instance, where the lay-up is given with respect to the notch as $0°$, + or - 2θ has not been studied. It is also important to note that the stress applied to the NRL samples was not symmetrical with respect to the notch since it was cut into the samples from one edge. A more conventional method is to cut a notch in the middle of the specimen[12].

Fracture of these samples was observed electrically to occur in two stages; an initial stage in which there is no fiber breakage and a subsequent stage in which either the fibers shear or the material delaminates. During this initial stage, which lasts approximately $\frac{1}{2}$ second, the supported stress decreases by nearly 50%. A more complex failure pattern has also been observed in which the supported stress decreases in steps, probably because the matrix does not crack all at once. This failure pattern may be a sign of a defect because both observations are associated with a maximum supported stress that is substantially lower than that associated with the simpler fracture sequence.

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12. R. B. Pipes, Univ. of Del., Newark, Del., private communication.

Two principal fracture modes were observed: for θ less than some critical angle θ_c a single matrix crack appeared parallel to one of the fiber directions with subsequent shearing of the other fiber set; for angles greater than this angle two cracks appeared which ran parallel to both fiber sets with subsequent delamination of the plies. Thus it appears the initial failure is a matrix failure with a subsequent sequence of events that involve delamination, fiber disbonding, and fiber breakage. We plan to study the hierarchy of the subsequent failure events and how their strengths affect the initial fracture.

The fundamental reason why the samples with small cross-ply angles fractured with one crack and sheared fibers while those with greater angles fractured with two and delaminated is that the tensile strength of the fibers is much greater than their shear strength. As the component of stress parallel to the fibers increases, their ultimate strength increases; and after a certain angle is reached, the material will delaminate rather than shear a fiber. The internal stresses are very complicated in such a geometry[13] and inherently three dimensional. However, the fracture behavior is understandable in terms of the hierarchy of elementary mechanisms.

The failure of carbon-epoxy composites is complex from a basic standpoint. Added to this basic complexity there is a variation in material fabrication that leads to a statistical variation in behavior. We would like to be able to assess the critical importance of particular variations. Also we would like to be able to assess the effect of changes in fiber and matrix type so that design experience would not be lost with every material change.

Linear elastic fracture machanics calculations of this problem do not seem to be adequate because they are directed toward elastic design behavior and so average the behavior in a laminate. We feel that one needs a data set which consists of quantitative values of critical material parameters that could be easily obtained for a change of fiber or matrix. Certain elements of this set are obvious: fiber shear and tensile strength, matrix shear and tensile strength, fiber-matrix bond strength, interlamina bond strength, and probably the intertow strength. The problem would be then to calculate the local behavior of some combination of these elements under a given stress field in that neighborhood.

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13. R. B. Pipes and N. J. Pagano, J. Composite Materials, 4, 538 (1970).

We question whether the present materials will have optimum performance from the standpoint of fracture. Pellini[2] points out that structural integrity of design requires ductility of the material. It would seem that ductility of the interlamina bond might be efficacious for impact performance and that some ductility of the matrix itself might allow the composite to adjust to its load. Research has begun into the behavior of hybrid composites[14] which incorporate energy absorbing layers. One wonders whether it might not be possible to incorporate some structural element in the matrix to stop the propagation of cracks. We believe that it is necessary to be able to answer these questions in a reasonable inexpensive manner. To that end we have begun to define the necessary data set.

14. J. P. Favre, _J. Mat. Sci._, _12_, 43 (1977).

VI. ELECTRICAL NDE

Since these materials are electrical conductors it is interesting to ask whether their electrical characteristics might be used for Non-Destructive Evaluations. The problem of practical NDE is more complicated than is implied by the measurements that we have described. One would expect that evaluation of materials that have been damaged in the field could be done from only one side. Furthermore, all surfaces may be covered by a glass-mat skrim cloth that is an electrical insulator.

For these reasons AC techniques are interesting because they can be applied from one surface and can couple to the material through an insulating sheet. Owston[3] has done some intial studies of an eddy current technique. An eddy current is a circulating current in a material that is excited by a mutual inductance coupling with an AC circuit. Since these materials - as we described in Section III - do offer circular current paths as a result of fiber-fiber contact, they are susceptible to eddy currents at practical frequencies.

We did a quick check of the feasibility of the technique in these materials by two tests. First, we measured the change in the real and imaginary parts of the impedance of a ferrite core probe caused by the proximity of a sample of graphite/epoxy composite. At 50 MHz we found that changes in the real and imaginary components of the impedance were, respectively $\Delta R = 5\%$ and $\Delta X = -1.7\%$. These values are in quantitative agreement with those found by Owston.

The second test was a check with a commercial eddy current system at our Dahlgren laboratory by Mr. Clifford W. Anderson. The trace shown in Fig. 11 was done at 1.07 MHz but a similar one was taken at 0.501 MHz. The near vertical axis is the imaginary part of the probe impedance; the near horizontal axis is the real part. These axes are not mutually perpendicular.

The trajectory is produced by crossing a 1/32" crack while traversing the sample. A smaller probe could detect a smaller crack. However, Mr. Anderson said that the signal was strong in comparison to those found for metals.
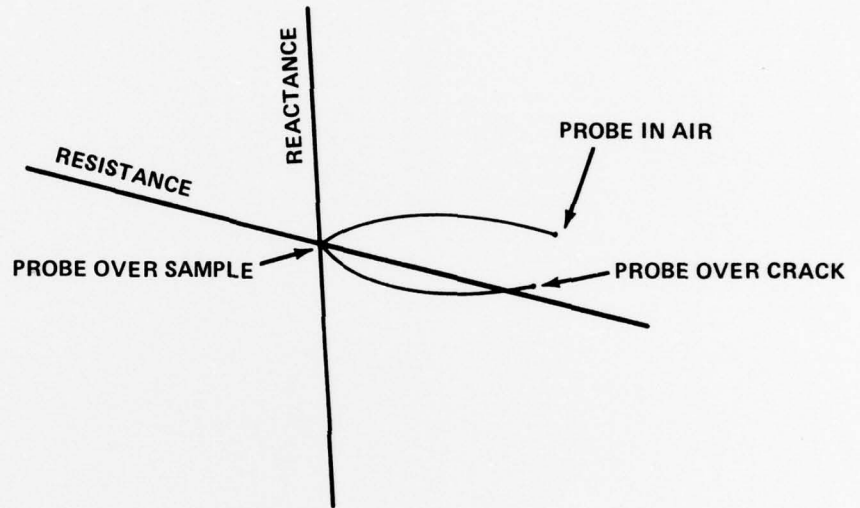
FIGURE 11 EDDY CURRENT TEST SIGNAL TRAJECTORY

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Higher frequencies lead to better resolution and stronger signals while lower ones lead to better penetration. Thus there trade-offs that depend upon the thickness of the structural material. Probe and system optimization studies are needed. Our conclusion is that this method is worthy of study as a possible NDE technique.

VII. CATASTROPHE THEORY

Recently a new branch of topography has appeared. Its founder is René Thom[5], and it has been called catastrophe theory. It deals with the mathematical description of overdamped systems which undergo discontinuous jumps from one stable state to another. The power of catastrophe theory is that for a system of no more than four control variables p_i and any number of system observables x_i there can be only seven topologically distinct catastrophe surfaces.

If $V(x_i, p_i)$ is the potential energy of the system, then the catastrophe or stability surface is the loci of points for which $\partial V(x_i, p_i)/\partial x_i = 0$. These points are stable if $\partial^2 V/\partial x_i^2 > 0$ and unstable if it is less than zero. The points for which it is exactly equal to zero are the loci which separate different stability regions between which the system can jump (that is, undergo a catastrophe). The projection of these loci onto the plane of the control variables is called the bifurcation set.

Since it is a common observation that there are sudden changes of state of biological as well as physical systems, this mathematical theory has been applied to living systems[5]. The result has been much controversy and confusion. It is our opinion that these controversies and complaints are not germane because the systems being discussed in those cases are not even definable in a mathematical sense. The arguments obscure the value of the theory.

Bifurcation theory, which is related to Catastrophe theory, is a much older branch of mathematics. It is not restricted to stability but is the study of equations that branch into multiple solutions at critical values of their variables. Thompson[15] recently showed the mathematical equivalence between the formalism of catastrophe and bifurcation theories. His work leads to the analytic solution of the bifurcation set for various potentials.

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15. J. M. T. Thompson and G. W. Hunt, J. Appl. Math. and Phys., 26, 581 (1975).

Composite materials are suited to such an analysis because they are optimally designed structures. According to Thompson[16] the more optimally designed a structure, the more susceptible to imperfections and off-axis loading it becomes. The fracture of a composite will be susceptible to imperfections and stress distributions in a manner similar to the structures Thompson discusses.

Although catastrophe theory is limited by the restriction to only four control variables, it is valuable. It seems that this value has not been fully appreciated. However, before it can be used it is necessary to understand and formulate a system mathematically.

16. J. M. T. Thompson and G. W. Hunt, A General Theory of Elastic Stability, Wiley, New York, 1973.

VIII. SUMMARY

This first annual report has been devoted to a discussion of our efforts in establishing certain fundamental aspects of composite fracture. To this end we have analyzed samples of graphite/epoxy composite material optically and electrically before and during fracture. The results are:

1. The material supplied to us by NRL is not the same as the Box Beam material from NSRDC-Carderock either in lay-up or in consistency.

2. The fiber array in both materials is non-uniform; this condition may lead to variations in fracture strength throughout a lamina.

3. The interlamina region in the NRL material is thinner than in the Box Beam material; it also varies in thickness in both materials; these variations may lead to concomitant variations in the interlamina bond strength.

4. The electrical conductivity of the samples shows that there is considerable fiber/fiber contact; this fact may lead to points of low shear strength.

5. The very small ($\sim$ 3%) change in electrical resistance of the samples at the initiation of fracture shows that fibers do not break; rather initial fracture is matrix cracking.

6. Two distinct fracture modes were observed; for small cross-ply angles one crack emanated from the notch tip parallel to one set of fibers with subsequent fiber disbonding and shear of the other set; for large cross-ply angles two cracks emanated from the notch tip parallel to each set of fibers, with subsequent delamination but without any fiber shear.

7. The behavior of the stress relief after initial fracture was either simple (one abrupt step and then a very gradual relaxation) or complex (several steps and gradual regions).

8. Eddy current techniques may be useful in graphite/epoxy nondestructive evaluation.

9. Catastrophe/Bifurcation theory may eventually be useful in the analysis of composite fracture since composites are optimally designed structures.

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