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Deformation and Failure Mechanisms of Graphite/Epoxy Composites Under Static Loading

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FOREWORD

This report comprehensively summarizes the results of a three-year study of the deformation and failure mechanisms of graphite/epoxy under static loading. The research investigation was conducted under NASA-Ames Contract NAS2-9989; Dr. Linda L. Clements was the principal investigator.

Comprehensive surveys of the research results are given in the first and second annual program report (dated July 26, 1979 and August 28, 1980, respectively) and in two publications which are attached as Appendices A and B. Consequently, this report will mainly summarize those findings and highlight the most significant results and conclusions, referencing the appropriate report for further detail.

1. INTRODUCTION

Non-metallic-matrix, fiber-reinforced composite materials have enormous potential for use in a wide variety of structural applications of utmost importance to NASA, including the fixed-wing and rotary aircraft. Composite materials are very attractive because of their high strength-to-weight and stiffness-to-weight ratios. Full use of these materials in the aircraft for primary structural applications, however, is hindered by the lack of long-term performance data, due to their short time in actual use and by the absence of a convincingly-reliable predictive techniques for projecting long-term durability from short-term tests.

The basis of any such predictive technique must be an understanding of the basic mechanisms producing the behavioral response of the material. To successfully predict durability of composite materials through accelerated testing, then, one must first understand the mechanisms of damage accumulation which lead to the eventual failure of the material. This requires an understanding of the mechanisms of deformation, degradation, and failure in the material. It is a step toward developing this understanding that has been the goal of this research program.

This research program has been directed to clarifying the mechanisms of deformation and failure of graphite/epoxy composites under static loading. The influence of moisture and temperature upon these mechanisms has also been investigated. Because the longitudinal tensile properties are the most critical to the performance of the composite, these properties were investigated in detail. Both ultimate and elastic mechanical properties were investigated, but the study of mechanisms emphasized those leading to failure of the composite. The graphite/epoxy composite selected for study was the system being used in several current NASA-sponsored flight test programs.

This program has outlined the use and limitations on the use of the accelerating parameters of moisture and moderate temperature for simulating by short-term laboratory tests the long-term service of composites statically loaded in longitudinal tension. In the future, these results will be combined with those of other NASA-Ames programs to develop accelerated testing methodologies for predicting long-term durability of composite parts in actual service.

2. BACKGROUND STUDIES

The material chosen for this study was "T300/5208" graphite/epoxy composite. This composite is fabricated from prepreg tape manufactured by NARMCO Corporation from Union Carbide's Thornel 300 graphite fiber and NARMCO's 5208 epoxy resin. Details of the material, its properties, and fabrication details are given in the first annual program report (Ref. 1).

Early work on the project involved arranging for specimen fabrication, procuring appropriate equipment, and instrumenting specimens. Fabrication of specimens for NASA-Ames projects had for some time been performed by an outside vendor, but numerous problems were encountered with the specimens so fabricated. Thus, it became necessary to formulate an extensive inspection and rework procedure for the specimens received. This procedure is described in detail in the second annual program report (Ref. 2).

In addition to this specimen problem, it was necessary to devise a technique for the instrumentation of the specimens. Contamination of the as received specimen surfaces caused bonded strain gauges, which were originally selected for specimen instrumentation, to debond with time or with exposure to the test environments. Fortunately, we were able to substitute "clip on" extensometers with the aid of a new extensometer that could measure bending strain. This extensometer revealed some alignment problems with the testing system, but these problems were, in turn, solved by the design of special grips and a special ball-bearing "die-set" alignment fixture. It was also necessary to define the requirements and order an environmental chamber which would permit mechanical testing in the desired environments

As will be described in Section 3, significant batch-to-batch variability was encountered in the prepreg material ordered for this and other NASA-Ames graphite-epoxy composite programs. Under the program reported here, these batch-to-batch problems were defined and "solved" for the future by ordering a single large batch of carefully specified material for use in all NASA-Ames programs.

During the period before the equipment and specimens for the graphite/epoxy research were available, earlier NASA-Ames work on deformation mechanisms of another plastic material, high-density polyethylene, was extended and summarized as NASA TM 78544, "Strain-Rate/Temperature Behavior of High-Density Polyethylene in Compression" (November 1978). In this case, the mechanistic approach was successful in creating a "master plot" capable of predicting the creep behavior of polyethylene between 23° and 126°C over a broad range of strain rates and stresses. Thus, the validity of temperature as an accelerating parameter for this plastic material was successfully demonstrated.

The mechanistic study on graphite/epoxy composites began with a scanning electron microscope examination of the failure surfaces of environmentally-conditioned 0°, 90°, and $\pm$45° specimens which had been mechanically tested to failure at temperatures ranging from 25°C to 96°C and in dry, moderately moist, and wet environments. This work is described in Refs. 1 and 2. One conclusion from the SEM work was that unknowns, related to the condition of the specimens, the testing, and their later handling, had to be eliminated by a carefully-controlled, well-documented experimental program. Furthermore, as a part of this program, the effect of specimen edge preparation, cure condition, and batch-to-batch prepreg variation needed to be clarified. The program designed to address these concerns, and the findings of this program are described in Section 3.

3. MECHANICAL PROPERTY STUDIES

The goal of the mechanical property studies described in this section was to determine the influence of various quality control variables upon the mechanical properties of 0° graphite/epoxy specimens as a function of moisture and temperature. This study and its results are described in detail in NASA TM 81246 (Appendix A). Consequently, this section will only summarize the study and its major findings.

3.1 INFLUENCE OF SPECIMEN PREPARATION TECHNIQUE

As was mentioned in Section 2, this and other NASA-Ames programs had encountered problems with specimens having numerous cut-edge flaws as received from the vendor. However, because the vendor was unable to provide specimens with high-quality cut edges, the early NASA-Ames programs proceeded: using these specimens, although in some cases screening and light rework were used to improve the average specimen quality. As was described in Section 2, an extensive screening and rework procedure for the specimens was devised. However, these reworked specimens then appeared to given substantially different mechanical properties than those from earlier NASA-Ames studies. Consequently, the question of the influence of edge preparation technique upon the mechanical properties was addressed in detail in the study reported in Appendix A.

The most significant finding of this study was that the strength of reworked (having "polished" edges) specimens was 15 to 25% higher than that of specimens with unreworked edges. Furthermore, the unreworked specimens did not always display the same environmental responses as the reworked specimens, and the behavior of specimens which had been screened and/or lightly reworked was statistically no different than that of extremely poor "reject" specimens. Thus, the need for complete rework of damaged specimens and, preferably, much more careful original specimen preparation was established.

3.2 INFLUENCE OF PREPREG BATCH

Significant property variations attributable to batch-to-batch prepreg variations were encountered in this and other NASA-Ames programs. As part of this study, one particularly severe variation in properties was studied and was found to be due to "defective" prepreg, i.e. prepreg in which the epoxy around individual fiber bundles was altered and degraded. The details of this study are given in Appendix 1. As a result of this discovery, the influence of this "defective" prepreg batch was found to have no statistically significant effect upon 0° strength for dry specimens tested at 25°C, although the elastic modulus of these specimens may have been somewhat reduced. As temperature or moisture content were increased, however, the strength of the specimens made with the defective prepreg was degraded considerably compared to that of "normal" specimens. Later work, to be discussed in Section 4, confirmed that this strength degradation was particularly severe when temperature and moisture content were simultaneously increased. Thus, the results of this work have established that materials acceptance criteria must include tests at elevated temperature and moisture content, as well as in more moderate environments.

3.3 INFLUENCE OF CURE CONDITION

Although NASA-Ames had specified that the graphite/epoxy specimens were to be made from postcured material, it was learned that the later specimens received from the vendor (and used in several NASA-Ames programs) had not been postcured. Thus, this program also undertook to study the effect of cure condition upon mechanical properties of 0° specimens. Fortunately, in this case, no significant effect upon mechanical properties was found.

3.4 INFLUENCE OF TEMPERATURE AND MOISTURE CONTENT

Once the influence of the quality control variables upon mechanical properties had been established, it was possible to define the influence of temperature and moisture content upon 0° mechanical properties. Temperature was found to produce a significant increase in both strength and modulus. However, the study, as reported in Appendix 1, could not confirm such an increase for increased moisture content, but the later work on failure surface examination led to a data revision which revealed an increase in 0° strength with increased moisture content, as well. However, the combination of a simultaneous increase in both temperature and moisture content may produce a strength decrease.

4. STUDY OF FAILURE MORPHOLOGIES

The goal of the failure morphology study described in this section was to identify the mechanisms which led to the mechanical property effects reported in Section 3. More details are given in NASA TM 81318, provided as Appendix B.

4.1 INFLUENCE OF "FLAWED" SPECIMENS

One of the most significant findings in this study was the discovery that the failure morphologies of "flawed" specimens are quite distinctive and can be easily used to eliminate specimens with undetected flaws from the data prior to statistical analysis. Flawed specimens were found to give a characteristic "low-energy" failure surface morphology, as opposed to the "high-energy" failure morphology of "normal" specimens, and the strengths of such specimens were also found to be significantly reduced. Furthermore, it was found that "flaws" could be such obvious defects as a bent or cracked specimen, or such less apparent defects as degraded matrix material. Thus, both specimens with unreworked edges (see Influence of Specimens Preparation Technique in Section 3) and specimens made from defective prepreg (see Influence of Prepreg Batch in Section 3) were found to yield low-energy failure morphologies, and, thus, lowered strengths.

4.2 INFLUENCE OF TEMPERATURE AND MOISTURE CONTENT

The elimination of "flawed" specimens from the statistics led to the confirmation of the increase in 0° strength with moisture content, as well as with temperature which was described in Section 3. Failure surface examination indicated that decreased flaw sensitivity was probably the reason for both of the increases in strength. Moisture, however, was also found to produce an apparent weakening of the interfacial bond. While this apparently has little or no effect upon strength at room temperature, when both temperature and moisture content are increased, the interfacial

bond is considerably weakened. In addition, the epoxy becomes more brittle, breaking up and falling away from the filaments. Thus, although end-tab problems cast doubt upon the data at "elevated temperature wet," the failure morphology study suggests that strength drops when temperature and moisture content are increased simultaneously.

5. CONCLUSIONS

As is outlined in the previous sections, this program has solved a number of problems and has yielded results which will be useful to current and future graphite/epoxy test programs. Notably, the mechanisms of the influence of environments and quality control variables upon the mechanical properties of 0° graphite/epoxy composites have been clarified. In addition, these results have the following implications for the establishment of accelerated testing rationale:

- Moisture and temperature taken individually have similar effects upon 0° strength, but the microscopic mechanisms are only partly similar. This may well indicate a limit of the use of these variables as accelerating parameters for simulating long-term exposure.

- Moisture and temperature taken together have a very different effect upon 0° strength and failure morphology than when applied separately.* Thus, it seems unlikely that it will be possible to combine their use as accelerating parameters.

- The influence of flaws upon 0° strength properties is considerable. This influence will have to be part of any accelerated testing plan.

*It should be noted that such a synergistic effect of moisture and temperature has been noted for other laminates and other test modes in other NASA-Ames programs, as well. However, such an effect upon 0° strength is far more unexpected.

6. REFERENCES

1. "Lifetime and Strength Retention of Graphite/Epoxy Composites Under Accelerated Static Loading," Annual Program Report, June 26, 1978 to June 25, 1979, NASA-Ames Contract NAS2-9989; ARACOR Report TR-22-1 (July 26, 1979).

2. "Deformation and Failure Mechanisms of Graphite/Epoxy Composites Under Static Loading," Annual Program Report, June 26, 1979 to June 25, 1980, NASA-Ames Contract NAS2-9989; ARACOR Report TR-22-2 (August 28, 1980).

APPENDIX A

NASA Technical Memorandum 81246

Influence of Quality Control Variables on Failure of Graphite/Epoxy Under Extreme Moisture Conditions

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Influence of Quality Control Variables on Failure of Graphite/Epoxy Under Extreme Moisture Conditions*

ABSTRACT: Tension tests on $(0°)_8$ T300/5208 graphite/epoxy composites were performed to determine the influence of various quality control variables on failure strength as a function of moisture and moderate temperatures. The extremely high- and low-moisture contents investigated were found to have less effect upon properties than did temperature or the quality control variables of specimen flaws and prepreg batch-to-batch variations. In particular, specimen flaws were found to drastically reduce the predicted strength of the composite, whereas specimens from different batches of prepreg displayed differences in strength as a function of temperature and extreme moisture exposure. The findings illustrate the need for careful specimen preparation, studies of flaw sensitivity, and careful quality control in any study of composite materials.

KEY WORDS: Composite materials, graphite/epoxy composites, tensile strength, environmental tests, moisture, quality control

The use of composite materials in commercial aircraft primary structures is hindered by the absence of convincingly reliable techniques for predicting composite durability under actual service conditions. Development of such techniques is complicated by the fact that significant changes in composite durability can occur not only at extreme temperatures, but also at moderate temperatures due to extreme moisture contents. This problem is being addressed at NASA-Ames Research Center in a program investigating the

mechanisms of deformation, strength degradation, and failure of graphite/epoxy composites. The portion of that work to be reported here involves an assessment of the influence of various quality-control variables — specifically prepreg batch, cure conditions, and specimen quality — on the effect of moisture and moderate temperature on the tensile properties of $(0°)_8$ Thornel 300/NARMCO 5208 graphite/epoxy composites.

Experimental Procedure

Materials

The "T300/5208" graphite/epoxy composite was fabricated from prepreg tape manufactured by NARMCO Materials, Inc., from Union Carbide Corporation's Thornal 300 graphite fiber, and NARMCO's 5208 epoxy resin. Table 1 gives the physical and mechanical properties of the WYP-30-1/0 (zero twist) grade of Thornel 300 fiber used in the prepreg. The NARMCO 5208 epoxy resin is one of several commercial epoxies based on the TGDDM-DDS system, that is, the main constituents are tetraglycidyl 4,4'-diaminodiphenyl methane epoxy (such as Ciba Geigy MY-720) cured with 4,4'-diaminodiphenyl sulfone (such as Ciba Geigy Eporal). The 5208 system contains about 90 parts-per-hundred (pph) by weight of TGDDM, about 24 pph DDS, and about 10 pph of glycidyl ether of a bisphenol-A novolac epoxy (Celanese SU-8) [*1*].

Specimen Fabrication

The specimens used in this study were fabricated for NASA-Ames by an outside vendor. Large (approximately 1 m^2) panels of the suitable lamination sequence were prepared from one of two different batches of 0.3-m-wide prepreg tape. These panels were cured in an autoclave held for 1/2 h at 135°C and then 2 h at 180°C, under 700 kPa pressure. The average volume percent fiber from these panels was determined to be 64.6%, with a range of 64.3 to 64.8%.

Next, vendor-fabricated tabs made from 0°/90° fiberglass fabric and epoxy resin were bonded to the panels. The tab adhesive used for the panels made from prepreg batch A was FM-143 adhesive by 3M cured for 1 h at 125°C and 50 psig. For the panels made from prepreg batch B, an unknown but reportedly comparable adhesive was used. Specimens were then cut from the panels using a dry carborundum cut-off wheel. The nominal configuration of these specimens was 12.7 mm wide, 1.2 mm thick, with a gage length of 127 mm, and 60-mm-long fiberglass tabs.

The as-received specimens were found to suffer from numerous fabrication defects. Two problems were particularly troubling: (1) extensive torn fibers stuck out from the cut sides, and numerous edge delaminations extended into the cut sides of specimens; and (2) the composite itself, or the composite plus the tabs, tended to be bowed. Because of our concern over these defects and because of reproducibility problems in our preliminary results, most of the specimens used in this study were subjected to extensive screening and rework. This procedure involved screening the specimens and rejecting any specimens that exhibited obvious bow or other irreparable defects. The specimens were then reworked by wet-polishing sufficient material off both cut sides to remove all detectable (at 30×) torn or delaminated material. This screening and rework procedure was done to bring all specimens up to the quality outlined in the ASTM Test Method for Tensile Properties of Oriented Fiber Composites (D 3039-76). The resulting width of these polished-edge specimens ranged from 10 to 12 mm. The properties of unpolished-edge specimens were also investigated. These specimens were screened for bow and for edge defects. Only specimens without visually obvious edge delaminations were retained.

In order to answer questions about the possible influence of degree of cure upon 0° properties, some of the specimens were given a postcure of 2 h at 200°C, followed by a slow oven cool.

Environmental Conditioning

As-received specimens contained 0.15 to 0.45% moisture. Specimens destined for mechanical testing were first dried in a vacuum desiccator at 100°C for 7 days, then held for at least 2 days under vacuum at room temperature. Weight gain/loss studies (on "dummy" specimens, without tabs, yet taken from the same panels) confirmed that this was sufficient time to ensure complete moisture removal from the specimens. Specimens to be tested in the "dry" condition were left in a room temperature vacuum desiccator until being tested at the appropriate temperature and <5% relative humidity (r.h.). All specimens to be tested in the "wet" condition were placed, after drying, in an environmental chamber at 60°C and approximately 100% r.h. for at least 60 days. This process produced essentially complete moisture saturation [*2*]. Work by Adamson, however, has shown that the so-called "reverse thermal effect" can produce an even higher moisture content than "normal" saturation in similar graphite/epoxy composites [*3*]. This reverse thermal effect occurs when specimens are first saturated, or nearly saturated, with water at a given temperature and then are placed in water (or high humidity) at a *lower* temperature. Thus, in order to increase moisture content to a true extreme, wet specimens were finally held at room temperature and at essentially 100% humidity for at least 45 days before testing. Weight gain from the dummy specimens confirmed such an additional increase in moisture content: specimens conditioned for at least 60 days at 60°C and 100% r.h. contained 1.57 ±0.23% of water (by weight), whereas specimens further conditioned for 45 days at room temperature and 100% r.h. contained 2.02 ±0.13%

water.[3] All wet specimens tested in the study were subjected to this additional moisture exposure. In addition, all specimens were held at room temperature and ~100% r.h. until being tested at the appropriate temperature and ~100% r.h.

Mechanical Testing

Tension tests to failure were performed inside an environmental chamber using a 10,000-kg-capacity servo-hydraulic mechanical testing machine. The tensile grips were mounted to an alignment device consisting of a three-post ball-bearing die set. This alignment device was, in turn, mounted to the hydraulic actuator and, through a universal joint, to the load cell and load frame. Longitudinal strain was measured using an axial strain-gage extensometer; for many of the tests, transverse strain was also measured using a diametral extensometer.

All tests were done at a constant elongation rate which resulted in an actual strain rate of 3×10^{-5} s^{-1}. Time to failure (at about 1% strain) was approximately 5-1/2 min.

Experimental Matrix

The experimental conditions studied were as follows:

I.	Temperature:	25° and 96°C
II.	Moisture content:	Dry ≃ 0% (tested at <5% r.h. at 25°C, <2% r.h. at 96°C)
		Wet ≃ 2% (tested at ~100% r.h.)
III.	Prepreg batch:	Batches A and B
IV.	Cure condition:	Not postcured, cured as received
		Postcured 2 h at 200°C

V. Specimen quality: Polished edges = specimens screened for bow and with cut sides wet polished to remove damage

Unpolished edges = specimens screened for bow and for visually obvious damage to cut sides

Nearly every permutation of conditions was studied. However, in the case of unpolished-edge specimens, only specimens from batch A, mostly not-postcured, were considered.

Results and Discussion

Our concern with the effect of prepreg batch upon properties resulted from some early findings in this study. Anomalous results from some early tests were traced to specimens prepared from prepreg batch B. A microscopic investigation of tested and untested batch B specimens and of the prepreg itself was performed. From optical microscopy, scanning electron microscopy (SEM), and consultation with NARMCO and Union Carbide, we concluded that there were indeed some differences between batch B and other prepreg such as batch A. Some of these differences are illustrated in Fig. 1. A photomicrograph of a laminate made from "normal" prepreg (such as batch A) is shown in Fig. 1*a*. The cut and polished filament ends reflect light very effectively and thus appear light-colored. A photomicrograph of a laminate made from prepreg batch B is shown in Fig. 1*b*. In this laminate, there are light and dark areas, with the transition between such areas occurring both between layers and within a single layer. In the dark areas, as can be seen from the magnified view of Fig. 1*c*, the individual filaments have been damaged. Figure 1*c* also illustrates that the dark areas are frequently connected with

individual fiber bundles (3000 filaments to a bundle). The obvious conclusion that the filaments are somehow degraded in these areas was refuted by careful polishing and SEM work, which showed that the epoxy matrix in these areas is somehow altered and weakened. Unless extreme care is exercised the apparent filament degradation actually occurs during metallographic preparation. The altered epoxy matrix allows the filaments to move around and thus be damaged during polishing. Since the altered epoxy occurs within and around individual fiber bundles, the epoxy probably has reacted to some surface effect or contaminant on *some* of the fibers used to make up the prepreg. We suspect that this effect is related to another problem encountered on a few occasions by other T300/5208 users and labeled "zebra tow" by them.[4] In this case, some of the surface tows (fiber bundles) of the composite panels tended to pull loose from the panel when the peel ply was removed. We have noted some such surface features on specimens made from our batch B prepreg.

Figure 2 is a plot of axial elastic modulus, E_{11}, as a function of temperature and moisture content at two strain levels. As can be seen from this figure, E_{11} (as determined from the slope of the stress-strain curve) is statistically significantly higher at an axial strain, ε_{11}, of 0.5% than at ε_{11} = 0.1%. Other researchers have also observed this increase in modulus with strain [*2,4*]. Figure 2 also illustrates that the increase holds true for all combinations of moisture and temperature. One possible explanation for the phenomenon is that curved filaments in the composite straighten with increased strain so that more filaments carry the applied load [*2*]. Another possible explanation is that there may be a strain-induced improvement in orientation of the covalently bonded carbon platelets within the individual filaments.[5]

Other findings on the behavior of E_{11} were as follows:

- There is no statistically significant effect of the cure condition or specimen quality on E_{11}.

- There may be an influence of prepreg batch upon E_{11}. The mean for batch B is systematically lower than that for batch A. For example:

		E_{11} (0.1% ε_{11})	E_{11} (0.5% ε_{11})	
25°C, wet	Batch A	129.5 ±1.9 GPa	139.5 ±2.5	(N = 12)[6]
	Batch B	127.3 ±1.6	137.1 ±1.9	(N = 9)

In this case, the mean of the batch A specimens is greater than that of batch B at the 95% confidence level.

- There is a statistically significant temperature-induced increase in E_{11} for dry specimens only. (See Fig. 2.) Since the axial thermal-expansion coefficient of the fiber is negative, that is, the fiber *contracts* axially with increased temperature, the increase in E_{11} is not surprising. However, it is not clear why no such effect is seen for wet specimens.

- There may be an effect of moisture upon E_{11}, but such an effect is not systematic. At 25°C and 0.1% strain, the mean E_{11} for the wet specimens is *higher* than that for the dry specimens to better than 95% confidence. However, at 96°C and a strain of 0.5%, the mean E_{11} for the wet specimens is *lower* than that for the dry specimens to better than 99% confidence.

Determination of the major Poisson's ratio, ν_{12}, was difficult since the diametral extensometer slipped under many conditions. It was not possible to get any "good" data on wet specimens, but the data obtained indicate no influence of any of the other variables upon ν_{12}. In particular, there appears to be no influence of strain upon ν_{12}. The mean value of ν_{12} was 0.33 ±0.01 (N = 14).

A very important finding of this study was the magnitude of the influence of specimen quality upon 0° strength. Figure 3 compares 0° strength as a function of temperature for polished-edge and unpolished-edge batch A specimens. As can be seen, the strength is significantly increased by the improved specimen quality resulting from polished edges. It is also interesting to compare these results with strengths of specimens previously rejected as having irreparable defects:

		0° Strength	
Batch A, 25°C, dry	Polished edge	1542 ±89 MPa	(N = 9)
	Unpolished edge	1333 ±79	(N = 8)
	Rejects	1313 ±71	(N = 12)

The 0° strength of the "good" unpolished edge is no better than that of the "reject" specimens. This finding confirms that the specimen preparation procedure outlined in ASTM Method D 3039-76 is not overly conservative.

From Fig. 3, it is also seen that edge polishing produced a change in strength behavior as a function of temperature. The 0° tensile strength of dry polished-edge specimens increased significantly with an increase in temperature, whereas the small increase in mean strength for dry unpolished-edge specimens is not statistically significant. Moisture content, on the other hand, had no statistically significant effect upon strength for batch A specimens whether they were edge polished or not. Unfortunately, we were unable to get reliable strength data at 96°C wet because of end-tab failures. This was because the additional moisture content we produced using the "reverse thermal effect," when combined with elevated temperature, caused tab failure to occur before composite failure. (For batch A specimens, the tabs themselves failed prior to composite failure, but for batch B specimens,

the tab adhesive failed at very low loads.) Thus, our observations on the effect of moisture content on strength are valid only at 25°C.

These findings are somewhat in disagreement with those of Lifshitz [2]. Lifshitz studied unpolished-edge specimens and reported an effect of moisture content on strength at both 25° and 96°C. Lifshitz's specimens, however, were conditioned at 60°C and ~100% r.h. only. We suspect that the additional moisture content we induced using the reverse thermal effect produced a degradation that cancelled any strength increase resulting from nominally wet conditions alone. Lifshitz also reported a statistically signficant increase in strength with temperature for his unpolished-edge specimens. However, his data only compared "room" and "wet" (without reverse thermal effect) moisture conditions, whereas ours compares dry specimens only.

Figure 4 illustrates the effect of prepreg batch upon 0° strength. The mean strength at 25°C for wet batch B specimens is less than that for dry batch B specimens at the 90% confidence level (but not at the 95% confidence level). Far more clearly significant is the difference in strength between batch A and batch B specimens at 96°C. As can be seen from Fig. 4, the batch B specimens are much weaker at this temperature. As we have explained, we were unfortunately unable to fail the composite itself at 96°C wet. Nevertheless, we suspect that the combination of temperature and moisture — each of which seems to affect the batch B specimens adversely — may have produced significant degradation in the strength of batch B specimens.

Scanning Electron Microscopy

In an attempt to understand the mechanisms that have produced the property changes described above, we have initiated scanning electron microscopy studies on the failure surfaces of the composite specimens. Figure 5 shows typical failure surfaces for dry, polished-edge, not-postcured specimens.

One specimen from each batch A and batch B was tested at 25° and 96°C. As can be seen from this figure, the appearance of the individual broken filament is the same regardless of the temperature or batch. In fact, this holds true for typical failures at all conditions. Furthermore, there are few single filaments "pulled out" of any of the failure surfaces, and the amount of epoxy remaining on the surface of the filaments indicates that the interfacial bond in all cases is reasonably strong.

It is difficult, however, to pick out differences in the specimens which might explain the observed differences in properties. Obtaining such an explanation from the failure surfaces is complicated by a number of factors. First, the elastic recoil at failure induces significant secondary damage, which complicates failure-surface analysis. Second, there is a great deal of specimen-to-specimen variability at any one condition. Third, even for a given specimen, the differences in appearance between different areas are great. Thus, no single set of micrographs such as is shown in Fig. 5 can represent the specimens.

One difference, however, appears again and again regardless of area or specimen. The matrix in the specimens tested at 96°C shows less of the smooth, cupped, and glass-like signs of conchoidal failure than does the matrix of specimens tested at 25°C. Unfortunately, this is equally true for batches A and B specimens, so it does not explain the observed strength differences between specimens of the two batches. We hope that further microscopy will clarify these observations.

Conclusions

Our results demonstrate that the tensile properties of T300/5208 graphite/epoxy composites are affected by various quality-control variables. Perhaps

the most important finding is the large effect of specimen quality (preexisting torn fibers and edge delaminations even if they are not easily detectable) on strength. Another important finding is that strength and its variation with temperature and moisture content can be influenced by prepreg batch-to-batch variations. These findings lead us to make three points about studies of mechanical properties of graphite/epoxy. First of all, the importance of careful specimen preparation technique cannot be overemphasized. The procedure outlined in ASTM Method D 3039-76 is often compromised in practice, but it is clear from our work that such a rigorous procedure is not overly stringent. Second, the influence of preexisting flaws upon properties should be an integral part of any study of composite properties. And third, the influence of such quality-control variables must either be understood or the variable itself carefully controlled in any composite destined for actual service exposure.

Footnotes

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[3]All limits given in this paper are 95% confidence limits, based on the "t" test.

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[6]N = number of specimens entering into statistics.

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TABLE 1 — Physical and mechanical properties specified for WYP-30-1/0 (zero twist) grade of Thornel 300 graphite fiber[a]

Physical properties	
Filaments/fiber bundle	3000
Twist	None
Filament density[b]	1.73 Mg/m^3
Filament equivalent diameter[b]	6.9 μm
Mechanical properties	
Minimum tensile strength	2660 MPa (385 ksi)
Average tensile modulus	200-240 GPa (32-35 msi)
Minimum average strain to failure	1.1%

[a]Dale Black, NARMCO Materials, Inc., Costa Mesa, Calif., June 1979.

[b]Not part of specification. Taken from Union Carbide Corporation product literature.

Figure Captions

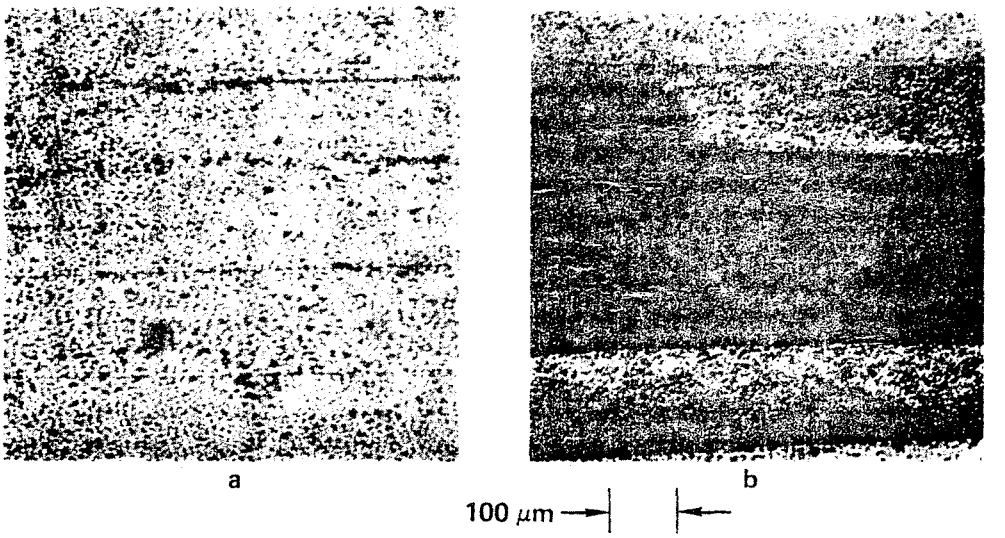
FIG. 1 — *Photomicrographs of T300/5208 laminates from "normal" (batch A) and "anomalous" (batch B) prepreg. (a) batch A, (b) batch B, and (c) batch B: anomalous fiber bundle among normal bundles.*

FIG. 2 — *Axial elastic modulus, E_{11} as a function of temperature at two different axial strains, ε_{11}, levels and moisture contents for batch A specimens. (Error bar shows 95% confidence limits. Numbers in parentheses are numbers of specimens.)*

FIG. 3 — *0° tensile strength as a function of temperature for polished-edge and unpolished-edge batch A specimens at two moisture contents.*

FIG. 4 — *0° tensile strength as a function of temperature for polished-edge batch A and batch B specimens at two moisture contents.*

FIG. 5 — *Scanning electron micrographs of failure surfaces of dry, not-postcured, polished-edge specimens. (a) batch A, tested at 250°C, (b) batch B, tested at 25°C, (c) batch A, tested at 96°C, and (d) batch B, tested at 96°C.*



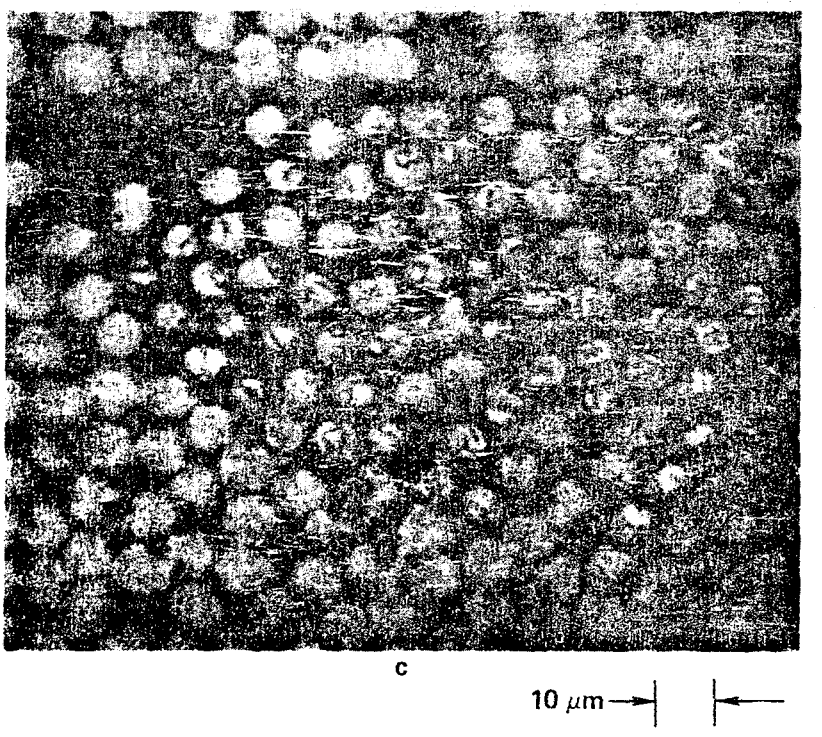


Fig. 1

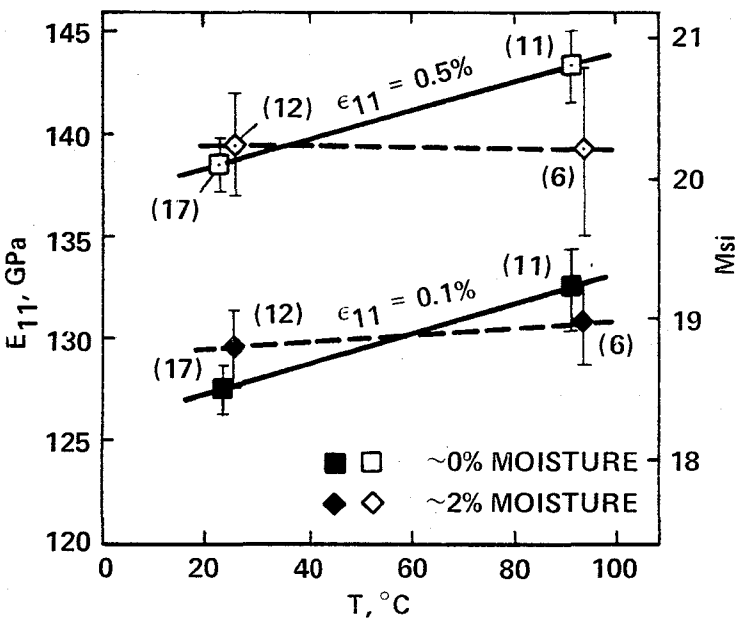
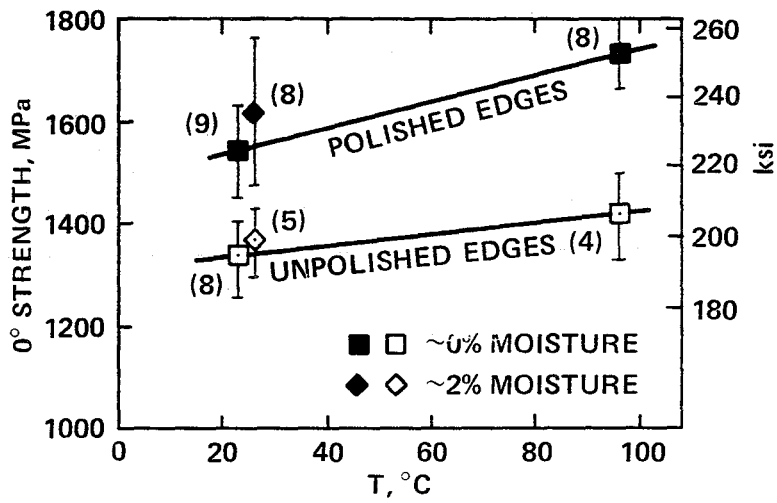

145
140
135
130
125
120
21
20
19
18
Msi
E_{11}, GPa
0
20
40
60
80
100
T, °C
(11)
(12)
ϵ_{11} = 0.5%
(17)
(6)
(11)
(12)
ϵ_{11} = 0.1%
(17)
(6)
~0% MOISTURE
~2% MOISTURE

Fig. 2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1600
1400
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0° STRENGTH, MPa
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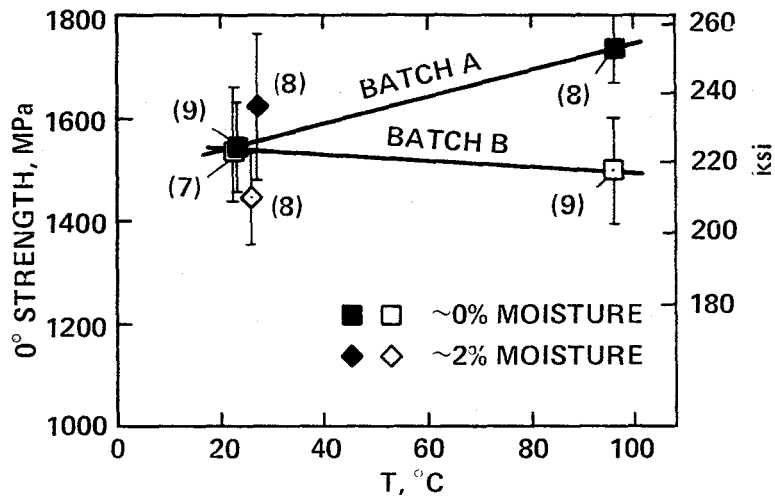
220
200
180
ksi
0
20
40
60
80
100
T, °C
(8)
(9)
(8)
POLISHED EDGES
(5)
(8)
UNPOLISHED EDGES (4)
~0% MOISTURE
~2% MOISTURE

Fig. 3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0° STRENGTH, MPa
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100
T, °C
BATCH A
BATCH B
(8)
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~0% MOISTURE
~2% MOISTURE

Fig. 4

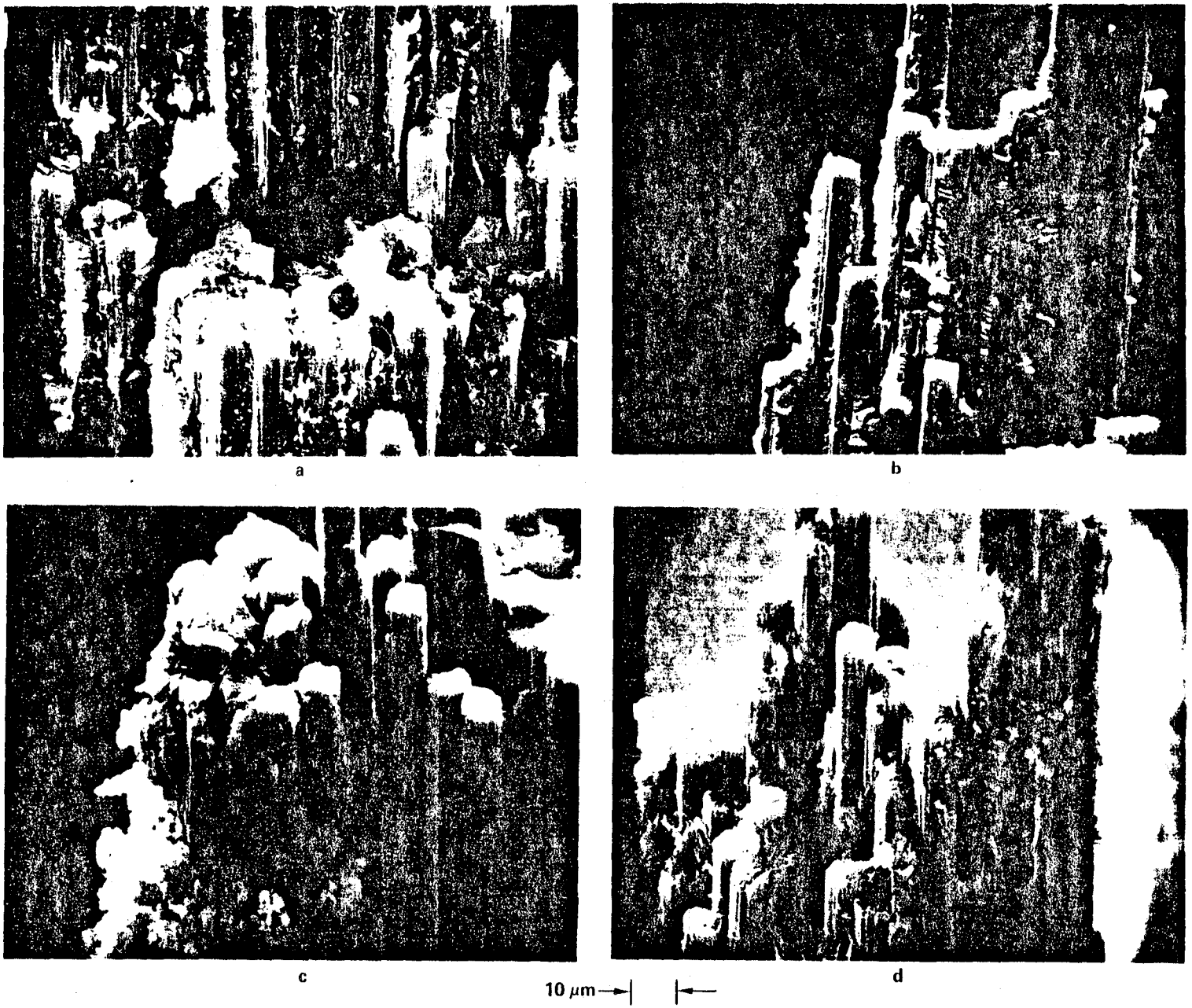

Fig. 5

APPENDIX B

NASA Technical Memorandum 81318

Failure of Morphology of $(0°)_8$ Graphite/Epoxy as Influenced by Environments and Processing

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Failure of Morphology of $(0°)_8$ Graphite/Epoxy as Influenced by Environments and Processing*

ABSTRACT: Optical and scanning electron microscopy were used to investigate the failure morphology of $(0°)_8$ T300/5208 graphite/epoxy specimens which had been tested until tensile failure. Failure morphology was studied as a function of the quality control variables of specimen preparation technique, prepreg batch, and cure condition, and also as a function of the environmental parameters of temperature and moisture content. Defective specimens were found to exhibit a low-energy failure morphology. Poor specimen edge preparation and one batch of prepreg — when tested at elevated temperature or moisture content — also exhibited this low-energy failure morphology. Postcuring had no effect on strength but did slightly alter failure morphology. Temperature or moisture appeared to decrease flaw sensitivity and thus increase strength; however, moisture also appeared to increase interfacial debonding between filament and matrix. When combined moisture and temperature increased interfacial debonding and made the epoxy matrix more prone to fracture.

KEY WORDS: Composite materials, graphite/epoxy composites, failure morphology, scanning electron microscopy, environments, moisture

While high performance composite materials have enormous potential for use in advanced aircraft structures, such utilization is hindered by the current inability to ensure structural reliability comparable to that of existing metal components. Because of the unique properties of composites, new predictive methodologies based on an understanding of the underlying mechanisms that

control properties must be developed. This approach will ensure that the results of short-term accelerated testing will yield predictions applicable to long-term behavior. The work reported here is a portion of a NASA-Ames Research Center study investigating the effects of potential accelerating parameters on the mechanisms of failure of state-of-the-art graphite/epoxy composites. The study is a follow-up to the earlier work of Clements and Lee [*1*], who investigated the influence of several "quality control" and environmental variables on the tensile properties of $(0°)_8$ Thornel 300/5208 graphite/epoxy composites. In the current work, optical and scanning electron microscopy were used to investigate the failure surfaces of the graphite/epoxy composite specimens tested by Clements and Lee, with the aim of correlating the failure morphologies with the observed tensile strengths.

Experimental Procedure

Materials and Specimen Fabrication

The "T300/5208" graphite/epoxy composite was fabricated from prepreg tape manufactured by NARMCO Materials, Inc., which was composed of Union Carbide Corporation's WYP-30-1/0 (3000 filament, zero twist) grade of Thornel 300 graphite fiber and NARMCO's 5208 epoxy resin. The properties of these materials are described in Ref. 1. The specimens used for the mechanical study were fabricated by an outside vendor; however, numerous specimen defects required extensive screening and rework of the specimens. Details of the specimen fabrication, screening, and rework are also given in Ref. 1. Briefly, during screening, specimens that showed obvious bow or other irreparable damage were rejected. The specimens were then reworked by wet-polishing the cut edges to remove all detectable (at 30X) torn or delaminated material. In addition, some of the specimens were screened for bow and for visually obvious edge defects but were not reworked by edge polishing. The results from these

specimens were then compared with those from polished-edge specimens. The nominal configuration was 12.7 mm wide for specimens with unpolished edges, 10 to 12 mm wide for specimens with polished edges, by 1.2 mm thick, 127 mm gage length, and 60-mm-long fiberglass tabs.

Specimens from two prepreg batches were examined. Batch A specimens gave "normal" mechanical behavior, but specimens from prepreg batch B showed anomalous mechanical behavior in early tests. The differences between the two batches will be described more fully under Results and Discussion.

The effects of cure condition were also considered. Some of the specimens were tested as received (cured 1/2 h at 135°C and 2 h at 180°C), while others were postcured for 2 h at 200°C, followed by a slow oven cool.

Environmental Conditioning

Environmental conditioning is described in detail in Ref. 1. All specimens were dried in a vacuum desiccator (at 100°C for 7 days) before moisture conditioning. Specimens to be tested "dry" were then held in a room temperature vacuum dessicator until they were tested. "Wet" specimens were placed, after drying, in an environmental chamber at 60°C and approximately 100% relative humidity (r.h.) for at least 60 days and then were held at room temperature (~25°C) and approximately 100% r.h. for at least 45 days before testing. The resulting water content of the "wet" specimens was 2.02 ±0.13% (by weight).[3] The moisture condition of the specimens will be discussed further under Results and Discussion.

Mechanical Testing

Specimens were tested until failure at a tensile strain rate of 3×10^{-5} s^{-1}. Tests were performed inside an environmental chamber held at the desired temperature and at <5% r.h. for the dry specimens or ~100% for the wet specimens. The testing details and results of the mechanical study are given in Ref. 1.

Microscopy

After failure, the specimens were "reassembled" as much as possible and photographed to record the relative locations of failed regions. The failure surfaces were also examined visually and with a low-power optical microscope, and general observations of failure morphology were recorded. The failure regions were then mounted and sputter-coated with gold for scanning electron microscopy (SEM) examination. In addition, for some of the specimens, sections were taken from the tab regions or from unfailed regions and were mounted in epoxy, polished, and examined with an optical metallograph.

Experimental Matrix

The "quality-control" variables considered were:

Specimen preparation technique:	Polished edges (per ASTM 3039-76)
	Unpolished edges
Prepreg batch:	A — normal mechanical behavior
	B — anomalous mechanical behavior
Cure condition:	Not postcured (as received)
	Postcured 2 h at 200°C

The environmental variables studied were:

Temperature:	25° and 96°C
Moisture content:	Dry ≅ 0% (tested at <5% r.h.)
	Wet ≅ 2% (tested at ~100% r.h.)

In the previous study, Clements and Lee [*1*] mechanically tested four to eight specimens at nearly every combination of variables from this experimental matrix. However, in the case of specimens with unpolished edges, only specimens from batch A, mostly unpostcured, were tested. Table 1 summarizes the tensile strengths reported by Clements and Lee. For the current work, the

results of the mechanical testing led us to select unpostcured batch A specimens with polished edges as our "standard" specimens. Nevertheless, we studied in some detail most of the conditions from the mechanical test matrix. First, we visually examined all of the mechanically tested specimens without magnification and with a low-power microscope. Then we proceeded to an SEM examination of selected specimens. Our initial SEM work revealed that there was considerable specimen-to-specimen and area-to-area variability in failure morphology. Thus, two or three unpostcured and one or two postcured specimens were examined for batches A and B specimens, with polished edges, at each environmental condition. Generally, five or more areas were examined in each specimen. Fewer numbers of specimens with unpolished edges and with known flaws were examined.

Results and Discussion

General Observations

SEM failure-surface examination revealed some general types of failure morphologies resulting from different types of failure propagation modes. Figure 1 illustrates the two most distinctive morphologies. The failure surface in Fig. 1a shows a varied topography with filaments and filament bundles at many different heights. The fracture path in this specimen was quite circuitous, and in fact was probably due not to a single crack but rather to a coalescence of cumulative damage. By analogy with metals, we assume that this morphology resulted from relatively "high energy" failure propagation. On the other hand, the failure surface in Fig. 1b is relatively smooth. As the higher magnification fiew in Fig. 1c shows, this smoother surface even displays the "river patterns" (striations perpendicular to the moving crack front — see arrow) — due to stopping and starting of a propagating crack — which are typical of more homogenous materials. This topography indicates that the failure crack

went through the material easily, with little secondary damage (such as intersecting secondary cracks) or other hindrance to its direct progress. We assume that this morphology results from relatively "low-energy" failure propagation.

Many specimens clearly failed by one or the other of these modes, but some specimens appeared to fail by a mixture of the two modes. Again, by analogy with metals, we assume that these specimens failed by "mixed-mode" failure propagation. Other specimens did not fail by these modes; however, in almost all of these specimens the failure mode was somehow suspect. These specimens included those that failed in or at the tabs, some that failed by splitting and delamination (which frequently was suspected to have originated in the tabs), and a number of batch B specimens which failed in an unusual manner which will be described in a later section.

A distinct relationship was found between the failure mode and the strength of the specimens. For example, all but one of the batch A specimens with polished edges that failed by low-energy failure propagation had strengths well below the mean value. Furthermore, low-energy failure propagation was the typical failure mode for "reject" specimens with known defects such as severe notches or bow; often, the origin of the failure path could be traced back directly to the defect. These observations led us to believe that in "good" specimens, low-energy failure propagation resulted from an undetected defect. Low-energy failure propagation, as an indicator of a defective specimen, becomes important in our analysis of the influence of quality control and environmental variables on failure, to be described in the following sections.

Influence of Specimen Preparation Technique

As was reported in Ref. 1, the strength of specimens having polished edges was found to be 15 to 25% higher than that of specimens with unpolished

edges. Figure 2 compares a typical scanning electron micrograph of a polished edge (Fig. 2a) to one of an unpolished edge (Fig. 2b). While the polished edge has individual damaged filaments, the unpolished edge shows numerous areas where groups of filaments are damaged. An examination of specimen failure mode revealed the apparent effect of these groups of damaged filaments on the failure of unpolished-edge specimens. More than 60% of the specimens with unpolished edges failed by low-energy failure propagation, and the balance failed at the tabs or by splitting and delamination. On the other hand, more than 70% of the specimens with polished edges failed by high energy or mixed modes; few failed by low-energy failure propagation. We conclude that the damaged edges of the unpolished-edge specimens constituted defects which produced the low strengths observed.

Influence of Prepreg Batch

As was reported in Ref. 1, optical microscopy, SEM, and consultation with NARMCO and Union Carbide led us to conclude that the epoxy within and around some of the individual fiber bundles in the batch B prepreg had been altered and degraded. Presumably, this occurred because the epoxy reacted to a surface contaminant on some of the fiber tows (fiber bundles) used to make up the prepreg. Whatever the cause of the properties of batch B material, mechanical testing did show a distinct difference in the behavior of batch B versus the "normal" batch A material. At 25°C dry, the strength of the batch B material was statistically the same as that of batch A, but at 25°C wet and 96°C dry, batch B strengths were significantly lower than batch A. (As will be described later, we were unable to test to failure at 96°C wet.) This strength difference was paralleled by a difference in failure modes. At 25°C dry most of the batch B failures were mixed mode, but with a predominance of low-energy failure regions. At the same condition, most of the batch A specimens also failed by

mixed mode, but high-energy regions predominated. At 25°C wet and 96°C dry, however, most of the batch B specimens failed by the low-energy mode, with the exception of some that failed at the tabs. Metallographic examination of these specimens revealed that almost all were split (in the 0° direction) underneath the end tabs. Also, Clements and Lee were unable to test the batch B specimens at 96°C wet because the specimens split and crushed underneath the tabs when the tensile grips were tightened before testing. In Ref. 1 this problem was attributed to bad end-tab adhesive, but after examination of the less severe splitting which occurred at 25°C wet and 96°C dry, we concluded that the splitting at 96°C wet was at least partially due to deterioration of the composite itself.

SEM examination of the failure surfaces of batch B specimens detected differences in the fiber bundles in the specimens. Figure 3 illustrates this difference in a low-energy failure region. Even in such a relatively smooth area individual fiber bundles stand out. In other specimens, the borders between fiber bundles were traced as the location of a 0° split in the composite, and in others a delamination seemed to originate at the border betwen such bundles. We conclude from these observations that the regions of altered epoxy associated with some of the batch B fiber bundles led to a different failure mode for batch B specimens and to lower strengths at elevated temperature or moisture content. At 25°C dry the differences in failure modes in batches A and B are only slight, thus the strength effect is minimal. (The batch B mean strength is lower than batch A, but the difference is not statistically significant.) The altered epoxy has a more important influence at elevated temperature or moisture content, however, and apparently produces severe degradation when temperature and moisture are combined.

Influence of Cure Condition

The strength data of Clements and Lee [*1*] showed no effect of cure condition. Nevertheless, an analysis of failure morphology revealed some slight differences, on the average, between specimens which were not postcured and those which were. All conclusions stated above for quality control variables and later for environmental parameters hold for postcured specimens as well as those not postcured. However, failure surfaces of the postcured specimens on the average seem to have somewhat longer and cleaner filaments. In addition, the epoxy appears to be somewhat more brittle than in specimens which were not postcured. These differences, however, are slight, so the absence of a statistical influence on strength is as expected.

Influences of Temperature and Moisture Content

Clements and Lee reported that the longitudinal tensile strength of batch A specimens with polished edges increased significantly as temperature increased from 25° to 96°C. They also reported that an increase in moisture content from dry to wet produced no significant change in strength. However, our conclusion regarding low-energy failure propagation as indicative of a defective specimen has led us to reconsider these data. If we eliminate from statistics all specimens that failed by low-energy failure propagation, the batch A failure data are as shown in Table 2. Now we find a significant increase in longitudinal tensine strength with both increasing temperature and increasing moisture content. We believe that this latter conclusion reflects more accurately the actual material behavior of "normal" (batch A or equivalent) T300/5208 graphite/epoxy. (The reader should note, however, that such behavior as a function of temperature and moisture content is representative only of 0° laminates and should not be generalized to any other configuration.)

Figure 4 shows representative failure morphologies for unpostcured batch A specimens with polished edges at the four environmental conditions considered. It should be noted that while a representative micrograph is given for 96°C wet, early end-tab failures on many of the specimens cast doubt upon all of the strength data at this condition. Thus, Clements and Lee did not report 96°C wet strength in Ref. 1. (The data we show in Tables 1 and 2 are taken from their raw data.)

In examining Fig. 4, it is also important to remember that there was considerable specimen-to-specimen and area-to-area variation in morphology for all conditions. Thus, these micrographs in no way represent the diversity of morphologies encountered at any environmental condition, but rather are typical of the most common or average morphology at that condition.

The influence of temperature on failure can be explored by comparing the behavior at 25°C dry and 96°C dry. We found that the differences in failure morphology for these two conditions were again related to failure mode. At 25°C dry there were no failures which were uniquely high-energy mode — most were mixed mode. That is, even though the high-energy mode might predominate in a specimen, there were occasional low-energy regions. At 96°C dry, on the other hand, there were no mixed-mode failures. Most specimens failed by the high-energy mode, although there were a few (presumably defective) specimens which failed by the low-energy mode. Furthermore, at 96°C dry the failure surfaces were macroscopically more irregular. That is, they had a more varied topography, possibly resulting from more secondary damage and thus a higher energy failure at 96°C than at 25°C. We hypothesize that these differences may be due to a decrease in flaw sensitivity with increased temperature.

A comparison of failure morphologies at 25°C dry and 25°C wet illustrates the influence of moisture on failure. At 25°C wet a few specimens showed

mixed-mode failures, but most clearly failed by either the high- or the low-energy modes. The failure surfaces at 25°C wet are macroscopically more irregular than at 25°C dry. We again hypothesize that there may be a decrease in flaw sensitivity, but now with increased moisture content rather than temperature. In the epoxy matrix an increase in either temperature or moisture content acts to decrease hydrogen bonding and thus facilitate molecular rearrangement. Furthermore, moisture is known to lower the glass transition temperature of the epoxy [2]. Thus the epoxy's ductility increases and its flaw sensitivity decreases. In addition, residual stresses in the epoxy matrix are reduced. If these effects are of sufficient magnitude, it is reasonable to expect a corresponding decrease in overall composite flaw sensitivity.

There is another difference, on the average, between the morphologies at the two conditions, however. At 25°C wet the filaments protruding from the failure surface tend to be cleaner and longer than at 25°C dry. The micrographs of Figs. 4a and 4c illustrate this difference. This observation is consistent with increased interfacial debonding between filament and matrix. Since the longitudinal tensile strength nonetheless increases, either the increased interfacial debonding does not weaken the overall composite, or any weakening is offset by decreased flaw sensitivity.

The influences of temperature and moisture are combined at 96°C wet. As is shown in Fig. 4d, at this condition the failure morphology contains many bare filaments – filaments that are longer and considerably cleaner than those at 25°C wet. Such long clean filaments are often considered to be filament "pull-outs," but, as is seen in Fig. 5a, there are few corresponding pull-out holes. Figure 5b demonstrates the reason for this discrepancy. The epoxy between filaments has not only debonded but has also broken up and fallen away. We thus conclude that the combined influence of temperature and moisture

is both to increase interfacial debonding and to make the epoxy more prone to fracture.

Since we have previously assumed that epoxy ductility increases with increased temperature or moisture content, the apparent embrittlement at 96°C wet is contrary to expectations. This result can be explained, however, by a comparison of 25°C wet and 96°C wet testing conditions.

The 96°C wet specimens tested by Clements and Lee actually differed from the 25°C wet specimens in a respect other than temperature. All of their specimens were held at 25°C until shortly before mechanical testing. Adamson [3] has shown that, for temperatures below the conditioning temperature at which moisture was introduced (60°C in this case), the saturation moisture content of graphite/epoxy specimens is inversely proportional to temperature. Thus, as was confirmed by weight-gain studies of Clements and Lee's specimens, their specimens saturated at 60°C picked up yet more water at 25°C. Furthermore, the resulting saturation (or near saturation) moisture content achieved at 25°C is greater than the saturation moisture content at 96°C. Thus, when the temperature of wet specimens held at 25°C was increased, a condition of supersaturation was introduced. Thus, in spite of the essentially 100% humidity of the mechanical test at 96°C wet, desorption would have occurred (and would have continued for several days). During this period of supersaturation, particularly with the aid of the applied tensile stress, covalent bonds may have been broken. Thus, first microcracking and then the type of epoxy cracking shown in Fig. 5b may have resulted.

Three other factors may also have influenced the results at 96°C wet. The glass transition temperature (T_g) of saturated epoxy is altered such that 96°C may have been in or very near the glass transition region of the wet epoxy. This alone, if it resulted in a sufficient loss of epoxy strength, might account

for the matrix cracking observed at 96°C wet. In addition, a reduction in T_g might lead to noticeable physical aging of the epoxy even in the brief time (1 to 2 h) Clements and Lee's specimens were held at 96°C. Such physical aging would then produce epoxy embrittlement [*4*]. Finally, it is also possible that further crosslinking may have occurred in the wet epoxy held at 96°C, again leading to epoxy embrittlement. The importance of these three factors can be neither proven nor disproven without further experiment.

It is unfortunate that the strength data at 96°C wet were unreliable. We would expect the two deleterious effects observed in the failure morphologies to results in a decrease in strength for this condition versus either 25°C wet or 96°C dry. Although the limited strength data reported in Tables 1 and 2 show a decrease in strength, the known end-tab problems render these data questionable. Again further experimentation — with improved end tabs — would be required to define the strength at 96°C wet.

Conclusions

Our conclusions from this study can be summarized as follows:

- Low-energy failure propagation in our specimens probably resulted from undetected specimen defects.
- The damaged edges of our unpolished-edge specimens constituted defects which produced low strengths.
- Regions of altered epoxy in the batch B composite led to lowered strength at elevated temperature or moisture content.
- The failure morphology of postcured specimens showed a tendency toward longer, cleaner filaments and a slightly more brittle matrix than in specimens not postcured. (However, there was no statistical effect of postcuring on strength.)

- An increase in longitudinal tensile strength with increased temperature may be due to decreased flaw sensitivity.
- Moisture apparently increases strength, perhaps again due to decreased flaw sensitivity, but it also produces more interfacial debonding.
- Combined temperature and moisture produce more interfacial debonding and also apparently allow the epoxy to fracture more easily.

Finally, we would like to emphasize one point. The work of Clements and Lee and this follow-up to that work have shown the considerable effect both quality control and environmental variables can have upon the fiber-dominated property of 0° tensile failure. When several variables are combined — such as "defective" prepreg batch, temperature, and moisture — the degradation in properties may be very severe. Because of these findings, we wish to emphasize the importance of full quality control and environmental characterization of composites prior to use.

Footnotes

*Work performed at NASA-Ames Research Center, Materials Science and Applications Office, Moffett Field, CA, under Contract NAS2-9989.

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[3]All limits given in this paper are 95% confidence limits, based on the "t" test.

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[1] Clements, Linda L., and Lee, Pauline R., "Influence of Quality Control Variables on Failure of Graphite/Epoxy Under Extreme Moisture Conditions, ASTM Symposium Composites for Extreme Environments, Bal Harbor, Fla., Nov. 11, 1980.

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TABLES 1 -- Summary of 0° tensile strength data of Clements and Lee [1]

Edges	Batch	Tensile strength, MPa			
		25°C		96°C	
		Dry	Wet	Dry	Wet
Unpolished	A	1333 ± 79[a]	1366 ± 71	1418 ± 84	---
		(9)	(5)	(4)	
Polished	A	1542 ± 89	1620 ± 144	1735 ± 67	1578 ± 148[b]
		(9)	(8)	(8)	(4)
Polished	B	1540 ± 122	1443 ± 93	1500 ± 107	---
		(7)	(8)	(9)	

[a]Limits are 95% confidence limits, based on the "t" test. Numbers in parentheses are numbers of specimens used in statistics.

[b]These data are questionable. See test for discussion.

TABLE 2 -- Longitudinal tensile strengths of batch A specimens with polished edges after low-energy failure data is eliminated

Edges	Batch	Tensile strength, MPa			
		25°C		96°C	
		Dry	Wet	Dry	Wet
Polished	A	1601 ± 69[a]	1765 ± 105	1730 ± 86	1593 ± 276[b]
		(6)	(4)	(5)	(3)

[a]Limits are 95% confidence limits, based on the "t" test. Numbers in parentheses are numbers of specimens used in statistics.

[b]These data are questionable. See text for discussion.

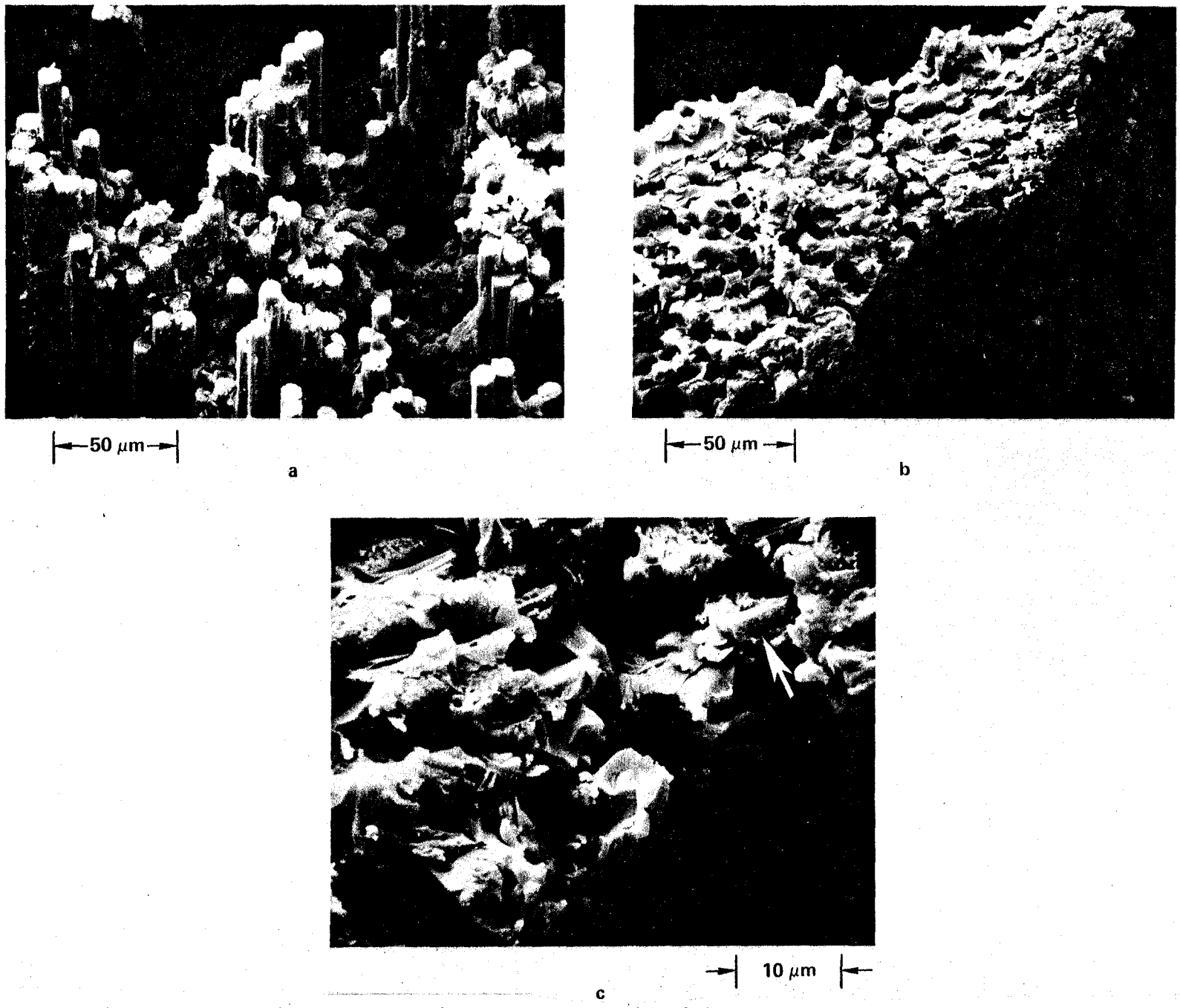

Figure 1.- Scanning electron micrographs comparing failure morphologies resulting from (a) high, and (b) low-energy failure propagation. (c) shows low-energy failure region and "river pattern" (arrow) at higher magnification.

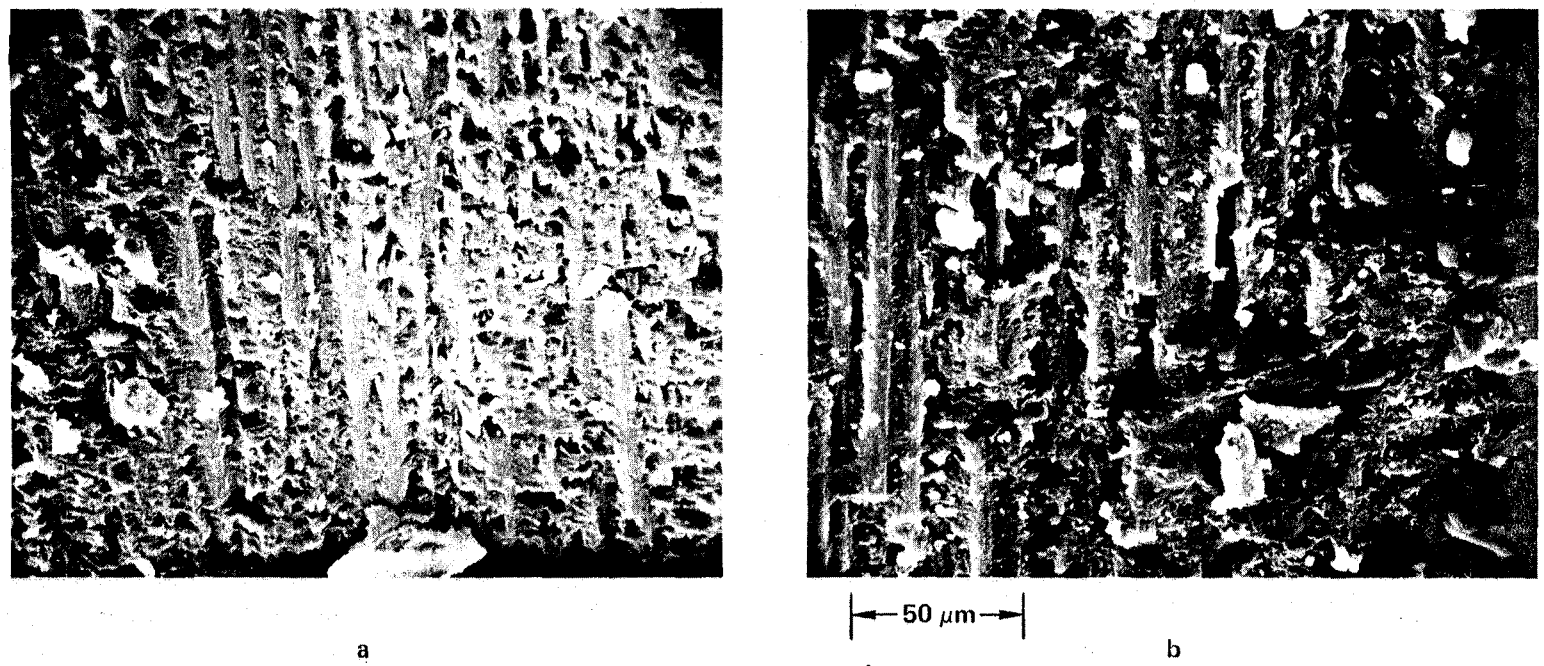

Figure 2.- Scanning electron micrographs comparing appearance of (a) unpolished to (b) polished specimen edges.

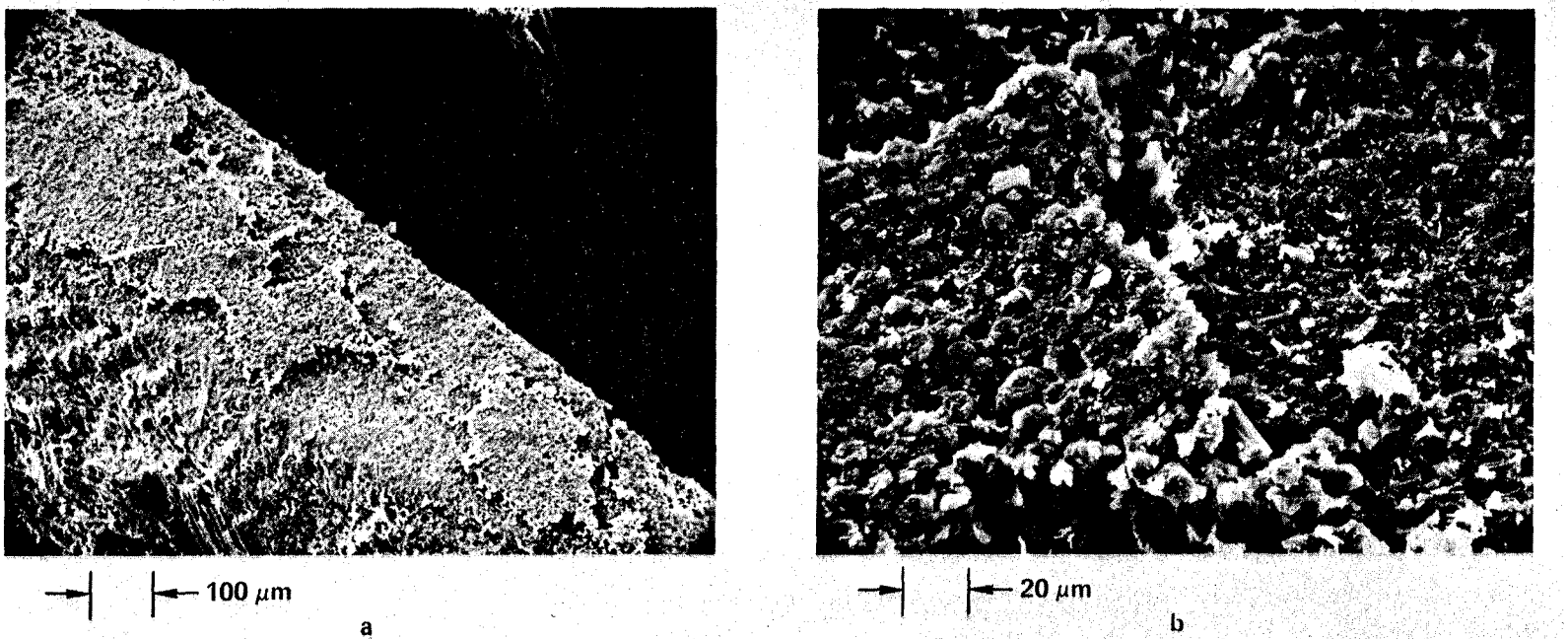

Figure 3.- Scanning electron micrographs showing at two magnifications a low-energy failure region with obvious differences between different fiber bundles in a batch B speciment (tested at 25°C wet).

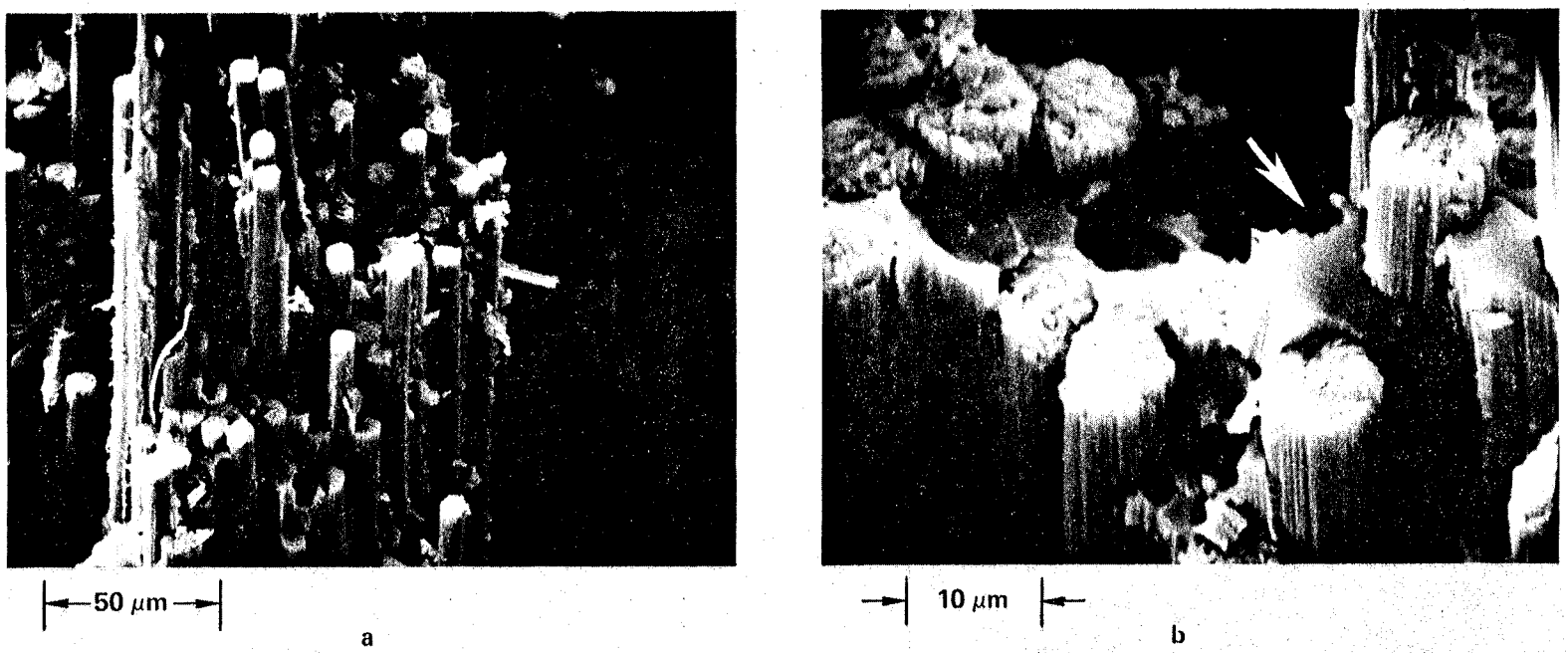

Figure 5.- Scanning electron micrographs of failure surfaces of unpostcured batch A specimens with polished edges tested at 96°C wet. (a) shows long clean filaments but few "pull-out" holes, and (b) shows epoxy that has broken up and is ready to fall away (arrow).

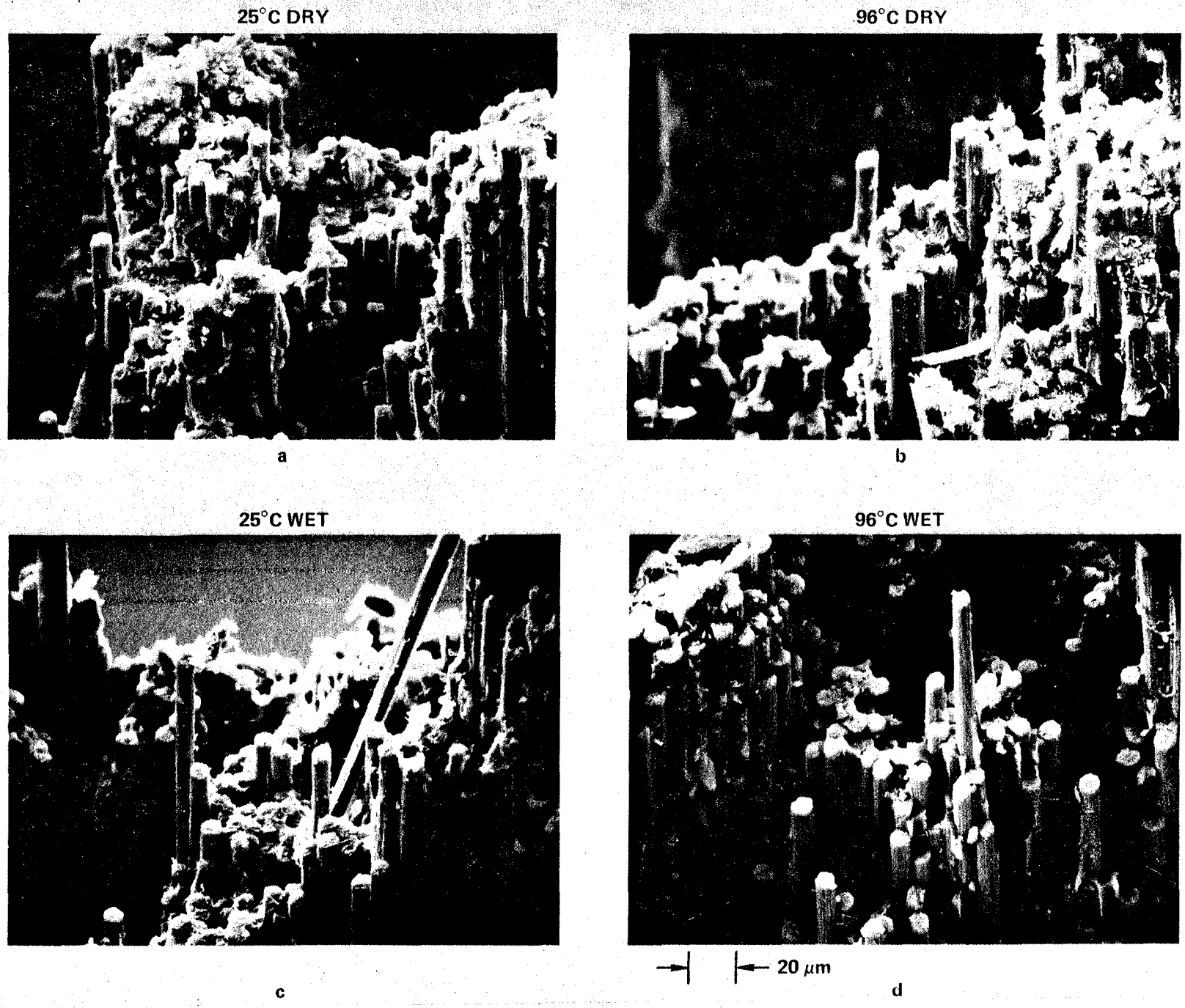

Figure 4.- Representative failure morphologies of unpostcured batch A specimens with polished edges tested at four environmental conditions: (a) 25°C dry, (b) 96° dry, (c) 25°C wet, and (d) 96°C wet.

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15. Supplementary Notes

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Final Report

16. Abstract

Tensile tests on $(0^{\circ})_8$ T 300/5208 graphite/epoxy composite laminates were performed and their failure morphology examined in detail using the scanning electron microscope. Of particular interest was the influence of various quality control variables as a function of moisture content at moderate temperatures. The goal of this work was to provide a rationale for accelerated testing by clarifying the mechanisms of deformation and failure. It was found that moisture and temperature individually have similar effects on 0° strength, but their influences on failure morphology are only partially similar. Together they were found to produce a significant effect. The influence of flaws on 0° failure was also considerable, and a characteristic failure morphology of a flawed specimen was identified. These results suggest that there may be limitation on the use of moisture and temperature as accelerated testing parameters. Additionally, the influence of flaws must be considered in any accelerated testing methodology.

17. Key Words (Suggested by Author(s))	18. Distribution Statement
Failure morphology, scanning electron microscopy, moisture, temperature, accelerated testing.	Unlimited - unclassified STAR Category - 24

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