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Fractography of unidirectional graphite–epoxy as a function of moisture, temperature and specimen quality

LINDA L. CLEMENTS
Materials Engineering Department, San Jose State University, San Jose, California 95192, USA

The tensile failure surfaces of $(0°)_8$ T300/5208 graphite–epoxy specimens were examined using both optical and scanning electron microscopy. Fractography was used to determine how moisture content and temperature as well as specimen preparation technique, prepreg batch and cure condition affected the failure mode. A distinctive "low-energy" failure morphology was found in defective specimens and also in those whose edges were poorly prepared. This morphology was predominant in failures at elevated temperature or moisture content for specimens which had been made from one suspect batch of prepreg. This finding combined with unusual end-tab failures from such specimens indicated that this batch was indeed "defective", but that such defective batches could in the future be identified by tests under hot, wet conditions. For specimens made from "good" prepreg, temperature or moisture appeared to decrease flaw sensitivity and thus increase strength, even though moisture also seemed to increase interfacial debonding between filament and matrix. When combined, moisture and temperature appeared to degrade performance by increasing interfacial debonding and making the epoxy matrix more prone to fracture.

1. Introduction

Composite materials offer unique structural options, but these options cannot be completely realized without an understanding of the mechanisms that control performance. The work reported here is a portion of an investigation of the mechanisms of degradation and failure of state-of-the art graphite–epoxy composites. This paper reports the fractography results for specimens tested by Clements and Lee [1], who investigated the influence of several environmental and "quality control" (processing) variables on the tensile properties of $(0°)_8$ "T300/5208" 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. The fractography results have also been used to reinterpret the data of Clements and Lee.

In recent years the fractography of fibrous composites has received considerable attention, and numerous authors have examined the failure morphologies of graphite–epoxy composites and delineated the features which arise under various conditions [2–7]. Some authors have specifically investigated 0° tensile failure morphology [8–13]. Of these, many have had only general interests, while others have found that since longitudinal tensile specimens fail with tremendous energy and the resulting failure surfaces display considerable variability, they could draw only very general or tentative conclusions. Even when very specific failure mode conclusions were given [14, 15], few have considered environmental effects. Miller and Wingert [16], however, reported a detailed study including the effects of temperature and moisture. They showed numerous excellent fractographs and gave a detailed analysis of the fracture behaviour. However, their tensile strengths for "System A" (which was apparently T300/5208) were very low, and well below those of the reject specimens tested by Clements and Lee [1]). And, contrary to the results of Clements and Lee, they also found no effect of temperature (or moisture) on strength for System A. However, their high temperature was much higher than that of Clements and Lee – 134 compared with 96° C – and their "wet" specimens were much drier, being conditioned for two weeks rather than more than two months. Thus even the findings of Miller and Wingert [16] could not be generalized to explain Clements and Lee's mechanical results.

This detailed fractographic study was also undertaken for another reason. Because of the large specimen-to-specimen and area-to-area variability in 0° failure surfaces, 0° fractographic conclusions must be based upon statistically determined average behaviour or else they may reflect random specimen selection rather than actual response. A large number of specimens must be examined, each in several areas. Unfortunately, most authors have not addressed this problem. (For example, Miller and Wingert [16], in their otherwise excellent paper, did not report how many specimens were tested nor whether multiple

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areas were examined. As a result, we cannot be certain that their findings are general.) As will be described, all of the fractographic conclusions in this paper are based upon numerous specimens and areas, plus large numbers of SEM fractographs taken for later comparison.

2. Experimental procedure

2.1. Materials and specimen fabrication

The T300/5208 graphite–epoxy composite tested by Clements and Lee [1] and investigated in this study was fabricated from prepreg tape manufactured by Narmco Materials, Inc. (Anaheim, California). The specifications for this material are given by Clements and Lee [1]. In the current study we considered carefully reworked specimens having polished edges, specimens free of visually obvious flaws but having saw-cut edges, and "defective" specimens. Details of the specimen fabrication, screening, and rework procedures are also given by Clements and Lee [1]. The nominal width of the specimens was 12.7 mm for those with unpolished edges and 10 to 12 mm for those with polished edges. Nominal thickness was 1.2 mm, and gauge length was 127 mm. The specimens had fibreglass tabs of length 60 mm.

Specimens from two prepreg batches were examined. Batch A was a prepreg batch whose specimens showed "normal" mechanical behaviour in preliminary tests, while those from prepreg Batch B showed anomalous mechanical behaviour. The effects of cure condition were also considered. Some of the specimens were left as received (cured 0.5 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.

2.2. Environmental conditioning

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. Specimens to be tested "wet" were placed, after drying, in an environmental chamber at 60° C and approximately 100% relative humidity (r.h.) for at least 60 days.* A study of several composite batches found the long-term moisture results from this chamber to be statistically identical to those attained by a 60° C water soak. The specimens were then held at room temperature (approximately 25° C) and approximately 100% r.h. for at least 45 days before testing. This latter treatment produced highly saturated "wet" specimens whose resulting water content was 2.02 ± 0.13 wt %†.

2.3. Mechanical testing

Specimens were tested until failure at a tensile strain rate of $3 \times 10^{-5}\,\text{sec}^{-1}$ according to ASTM Standard D3039–76. The absence of off-axis loading was assured by using grips fixed to a "die-set" alignment device, and the alignment was periodically confirmed by use of back-to-back extensometers. Tests were performed inside an environmental chamber held at the desired temperature and at less than 5% r.h. for the dry specimens, and at approximately 100% for the wet specimens.

2.4. 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 to a nominal thickness of 12 nm for scanning electron microscope (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.

Scanning electron micrographs discussed and shown in this paper were taken using a Cambridge Mark IV Stereoscan microscope using 20 kV accelerating voltage, no image enhancement, and a typical specimen tilt of 30°.

2.5. Experimental matrix

In the current study, the effects upon fractography of both environmental and processing variables were examined. The environmental variables studied were

(a) Temperature: 25 and 96° C.

(b) Moisture content: dry ≈ 0% (tested at < 5% r.h.) or wet ≈ 2% (tested at ≈ 100% r.h.).

The processing variables considered were

(c) Specimen preparation technique: polished edges (according to ASTM 3039–76) or unpolished edges.

(d) Prepreg: Batch A, normal mechanical behaviour or Batch B, anomalous mechanical behaviour.

(e) Cure condition: not postcured (as received) or postcured 2 h at 200° C.

In the previous study, Clements and Lee [1] mechanically tested over 100 specimens. They tested four to eight specimens under most combinations of conditions, except that for specimens with unpolished edges, only specimens from Batch A (mostly not postcured) were tested. Table I summarizes the tensile strengths reported. For the current work, the results of the mechanical testing led us to select un-postcured 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. Two or three un-postcured and one or two postcured specimens were examined under each environmental condition for Batch A and B specimens having polished edges. Usually five or more areas were examined in each specimen.

Because of the large area-to-area and specimen-to-specimen variability, this study involved 660 SEM micrographs and 80 optical micrographs taken from 160 general areas in 85 specimens, 34 of which were

*The actual relative humidity was 99 ± 1%.

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

TABLE I Summary of 0° tensile strength data of Clements and Lee [1]

Edges	Batch	Tensile strength (MPa) with 95% confidence limits*			
		25° C		96° C	
		Dry	Wet	Dry	Wet
Unpolished	A	1333 ± 79 (9)	1366 ± 71 (5)	1418 ± 84 (4)	–
Polished	A	1542 ± 89 (9)	1620 ± 144 (8)	1735 ± 67 (8)	1578 ± 148† (4)
Polished	B	1540 ± 122 (7)	1443 ± 93 (8)	1500 ± 107 (9)	–

*Numbers of specimens included in statistics are given in parentheses.
†96° C wet data considered unreliable due to high number of failures at the tabs.

examined in the SEM study. All conclusions were then based upon the average behaviour for a given condition.

3. Results and discussion

3.1. General observations

It should be noted that each individual batch of prepreg – and perhaps even each individual autoclave cycle – produces a composite which has a different moisture uptake and equilibrium moisture content to any other, and fairly minor changes in moisture conditioning can also vary the resulting moisture uptake. Thus, the results given here for the effects of moisture should be viewed as somewhat qualitative. Presumably, however, the trends reported here should be valid for other comparably conditioned batches of T300/5208.

It should further be noted that because of the statistical nature of many of the findings of this study, based as they are upon a large number of micrographs and a large test matrix, it is for the most part impossible to demonstrate the findings with a single or even a few micrographs. The micrographs shown here can only represent to a poor approximation the overall behaviour observed, and thus must only be considered in conjunction with the descriptive comments and conclusions of this paper.

As is described in detail elsewhere [17], however, two distinctive and distinctly different types of macroscopic failure morphology were found in the study, and these can easily be portrayed in representative micrographs. Those of Fig. 1 show a morphology having varied topography, with filaments and filament bundles at many different heights. By analogy with ductile failure in metals, this morphology was labelled "high-energy". Later association of stress–strain behaviour, prior defects, and the resulting morphologies showed that this morphology resulted from "good", generally high-strength failures in specimens without defects. The other morphology, a typical example of which is shown in Fig. 2, was represented by a relatively smooth failure surface, sometimes appearing almost saw-cut and frequently displaying striations, which represented either "cleavage steps" or "microbuckling" caused by local flexural loading. In most cases we were able to determine that such surfaces had not resulted from macroscopic off-axis loading or from impact with other specimen components after failure. Again, by analogy with the metals where a smooth surface is produced during brittle failure, this type of macroscopic morphology was labelled "low-energy". It became evident that whatever the microscopic mechanisms, this "low-energy" morphology resulted from undetected specimen defects, for example an internal or edge flaw or a bent specimen. The "low-energy" morphology could often be distinguished with the unaided eye or at low magnification since it produced a distinctively flat failure surface.

In some specimens both "high-" and "low-energy"

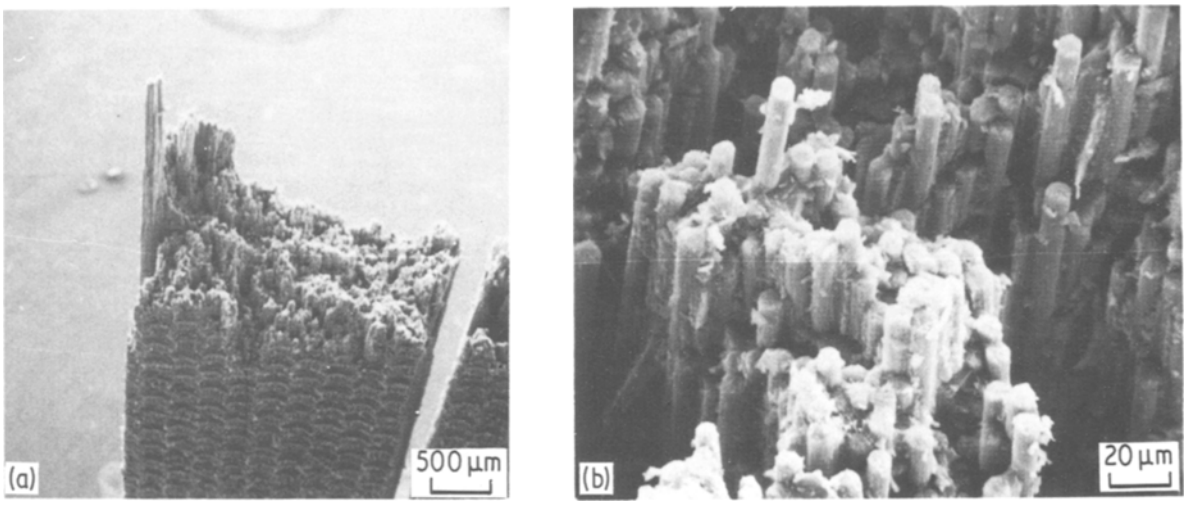

Figure 1 (a, b) Scanning electron fractographs showing typical "high-energy" failure region. Batch A specimen tested at 96° C dry.

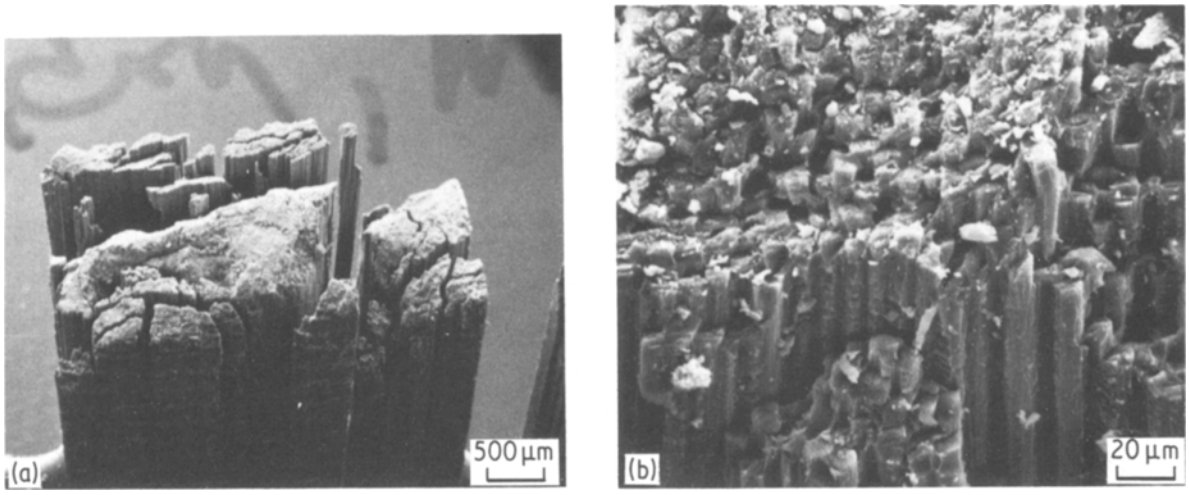

Figure 2 (a, b) Scanning electron fractographs showing one type of typical "low-energy" failure region. Note striations which pass through both fibre and matrix regions. Batch B specimen tested at 96° C dry.

regions were found. By analogy with metals, these were termed "mixed-mode" failures. In all such cases, however, either "high-" or low-energy" regions predominated. For the most part, these morphologies could also be distinguished at low magnification.

Other types of failure which could be observed unaided or with low magnification were also noted in specimens in the study. These included failures at, under, or near the tabs. This, of course, is a type of failure which is traditionally viewed as indicative of a defective specimen or test, and thus the specimen involved is normally eliminated from statistics. (Clements and Lee [1] generally eliminated these from their statistical results, although the tab failures of a few specimens were not recognized until the current study.) Some Batch B as well as some Batch A specimens having unpolished edges failed by in-plane "splitting" in the 0° direction within the gauge section of the specimen.

Thus, altogether six different types of failure morphology representing six different "macroscopic failure modes" were observed. These were "high-energy", "low-energy", "mixed-mode where high-energy regions predominate", "mixed-mode where low-energy regions predominate", "tab", and "splitting". As will be described in later sections, analysis of the conditions under which these different failure modes occurred was extremely useful in understanding the mechanical strength results of Clements and Lee [1].

3.2. Influence of specimen preparation technique

Analysis of the macroscopic failure mode led to an understanding of the dramatic influence of specimen preparation technique upon longitudinal strength. As shown in Table I, Clements and Lee [1] found that the strengths of specimens having polished edges were 15 to 25% higher than those of specimens having unpolished edges. As described elsewhere [17], SEM fractography combined with a macroscopic failure mode gave convincing evidence that extensive edge damage in the specimens with unpolished edges produced the low strengths observed. As a result, specimens with unpolished edges were eliminated from subsequent fractographic conclusions.

3.3. Influence of prepreg batch

Analysis of the macroscopic failure mode also became important in understanding the influence of prepreg batch on strength and morphology. In early tests, Clements and Lee [1] traced anomalous results to specimens made from Batch B prepreg. This was surprising since Batch B had passed all the quality assurance tests. As shown in Table I, in subsequent testing Clements and Lee found that at elevated temperature or moisture content Batch B specimens gave significantly lower strengths than "normal" Batch A specimens, although the decreased strength was still within material specifications. They also expressed concern about the apparently poor properties of Batch B specimens at 96° C wet. Thus, it became important to diagnose what was "wrong" with Batch B in order to devise a means of detecting such a batch in the future.

Clements and Lee [1] performed optical and scanning electron microscopy as well as differential scanning calorimetry on Batches A and B, and also consulted with Narmco and Union Carbide. As a result, they speculated that the epoxy within and around some of the individual fibre bundles in the Batch B prepreg had been altered and degraded. This presumably was due to some foreign substance on the surface of some of the fibre tows prior to prepreg manufacture. This explanation was given credence when the current study revealed that the observed strength differences were paralleled by a difference in failure mode. The failure modes of Batch A and Batch B specimens are summarized in Table II. Among polished specimens at 25° C dry (where the strengths from the two batches were not statistically different), 75% of the Batch B failures were "mixed mode", with a predominance of "low-energy" failure regions, while 60% of the Batch A failures were also "mixed mode", but with a predominance of "high-energy" regions. However, at 25° C wet and 96° C dry (where Batch B strengths were

TABLE II Macroscopic failure mode as a function of environmental condition (data for specimens having polished edges only)

Batch	Environmental condition*	Failure modes					
		"High energy"	"mixed mode"		"Low energy"	Tab failure	Splitting
			HE†	LE‡			
A	250 C dry (10)	0%	60%	0%	20%	20%	–
	25° C wet (8)	50%	0%	12%	38%	0%	–
	96° C dry (9)	78%	0%	0%	22%	0%	–
	96° C wet (9)	11%	11%	0%	11%	67%	–
B	25° C dry (8)	0%	0%	75%	0%	0%	25%
	25° C wet (9)	0%	0%	0%	44%	44%	11%
	96° C dry (12)	0%	0%	0%	75%	25%	0%
	96° C wet	Crushed during gripping prior to testing					

*Total numbers of specimens successfully tested at each condition are given in parentheses.
†"Mixed mode" failures showing predominately "high-energy" regions.
‡"Mixed mode" failures showing predominately "low-energy" regions.

significantly lower than those of Batch A), 62% of the Batch B compared with 29% of the Batch A specimens failed by the "low-energy" mode.

It was also observed that 5% of the Batch B specimens at 25° C wet and 96° C dry failed by splitting within the gauge section. The remaining 33% of the Batch B specimens under these conditions failed at the tabs, but sections and micrographic examination revealed all of these to show splitting in the 0° direction underneath the tabs. One possible explanation for such splitting was greater fibre misalignment in Batch B as opposed to Batch A specimens, but sectioning and micrographic examination of specimens from the two batches revealed no such misalignment. But the "hot wet" results offered another explanation. At 96° C wet, Clements and Lee [1] had been unable to test Batch B specimens because they crushed and split under the tabs as the tensile grips were tightened. While they attributed this at least in part to bad end-tab adhesive, micrographic examination of the specimens revealed that the material had been severely weakened at some of the interfaces between fibre bundles. It thus appeared that this splitting, as well as the less severe splitting at 25° C wet and 96° C dry, was primarily due to deterioration of the composite itself.

The problem with Batch B was further clarified by an SEM examination of the failure surfaces of Batch B specimens which did not fail at the tabs. As Fig. 3 illustrates, even in "low-energy" failure regions, differences were found in the fibre bundles in the specimens. Although these micrographs resemble those produced by "microbuckling" in 0° compression as described by other authors [7, 15], the morphologies here resulted from the distinct difference in properties between different fibre bundles. Even in relatively smooth areas, individual fibre bundles stand out. In some such specimens, the borders between fibre bundles were determined to be the location of an intra-ply 0° split in the composition, and in others an inter-ply delamination seemed to originate at the border between bundles. Interestingly enough, such differences between bundles even showed up in saw-cut surfaces.

All of these observations are consistent with the speculation [1] that there were regions of altered epoxy associated with some of the Batch B fibre bundles. At elevated temperature or moisture content the altered epoxy was quite adversely affected, leading to a very different macroscopic failure mode to that of Batch A specimens, and thus reduced strength. And when elevated temperature and moisture were combined, the altered epoxy regions appeared to be severely degraded, producing total loss of specimen integrity under very moderate (gripping) stress. Thus, elevated

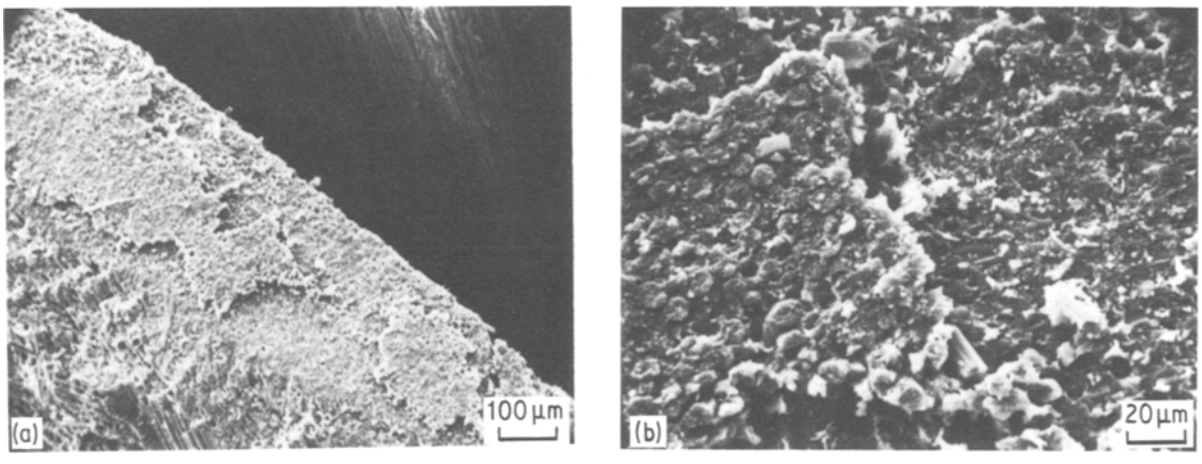

Figure 3 (a, b) Scanning electron fractographs illustrating a "low-energy" failure region in a Batch B specimen in which individual fibre bundles stand out. Batch B specimen tested at 25° C wet.

temperature and moisture should easily be able to detect or confirm the existence of any such "defective" batch in the future. This is important, since standard quality assurance did not detect any problem with this batch prior to the mechanical study. A flexural study to be reported in another paper (in preparation) confirms this conclusion and indicates that flexural testing at elevated temperature and moisture could also detect such a defective batch.

3.4. Influence of cure condition

The strength data of Clements and Lee [1] showed no effect of cure condition. Thus, it is not suprising that an analysis of failure morphology revealed very few differences between specimens which were not postcured and those which were. However, it did appear that postcured specimens *on the average* under the same conditions had protruding filaments which were slightly longer (approximately 10%) and "cleaner" (having less epoxy adhering to their surfaces) than those in un-postcured specimens. Furthermore, the failure morphology in matrix-rich regions was consistent with a slight increase in brittleness of the epoxy over that in specimens which were not postcured. These differences, however, were slight, so the absence of any statistical influence on strength is not unexpected. Thus, all conclusions stated above for quality control variables and later for environmental parameters hold for postcured specimens as well as for those not postcured.

3.5. Influences of temperature and moisture content

Clements and Lee [1] reported that the longitudinal tensile strength of dry 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 at 25° C produced no significant change in strength. (The mean strength increased, but the difference in means was not different to 95% confidence.) However, the recognition of "low-energy" failure propagation as indicative of a defective specimen has led to a reconsideration of these data. After all specimens that failed by "low-energy" failure propagation were eliminated from statistics, the Batch A failure data were as shown in Table III. These "corrected" data show a significant increase in longitudinal tensile strength either at elevated temperature (dry) or at high moisture content at room temperature. We believe that this latter conclusion reflects more accurately the actual *material* behaviour of "normal" (Batch A or equivalent) T300/5208 graphite–epoxy.

For the most part, the effects of temperature and moisture on the mechanical properties were paralleled by changes in the failure morphology. Unfortunately, however, these changes could only be observed by comparing and averaging the results of a large number of fractographs, and thus it is difficult or impossible to

TABLE III Revised 0° tensile strengths of Batch A specimens with polished edges (data eliminated from specimens showing low-energy failure)

Tensile strength (MPa) with 95% confidence limits*			
25° C		96° C	
Dry	Wet	Dry	Wet
1601 ± 69 (6)	1765 ± 105 (4)	1730 ± 86 (5)	1593 ± 276† (3)

*Numbers of specimens included in statistics are given in parentheses.

†96° C wet data include one probable tab failure and are considered unreliable.

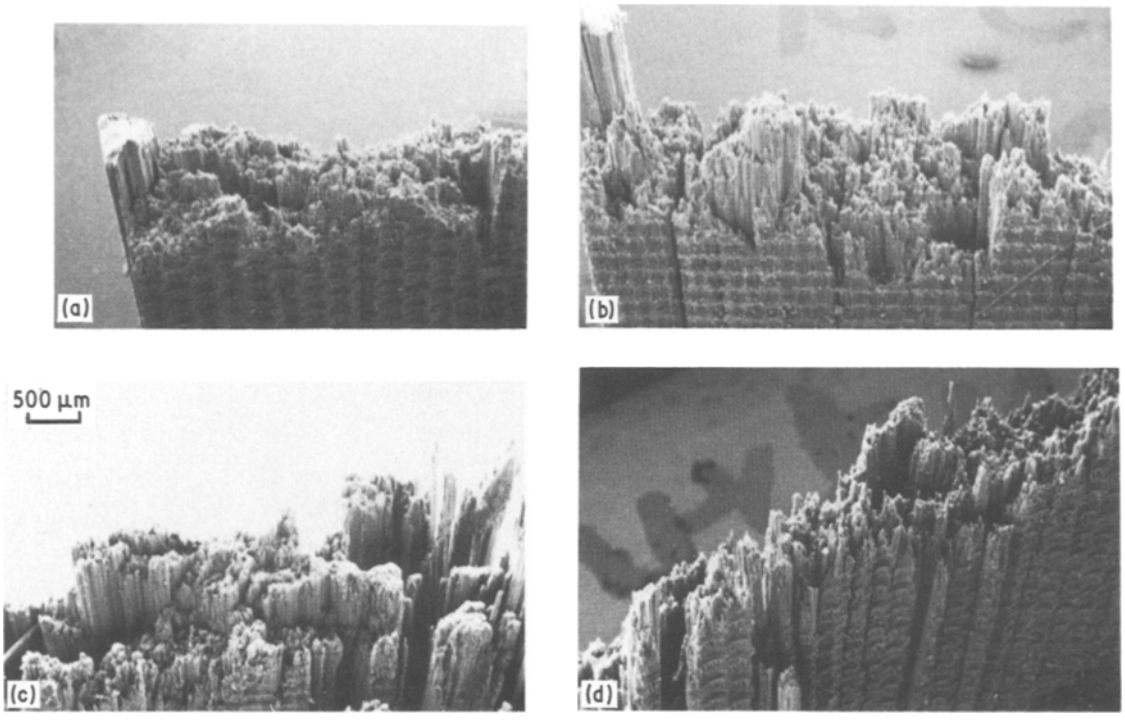

Figure 4 "Representative" low-magnification failure morphologies of un-postcured Batch A specimens with polished edges tested under different environmental conditions: (a) 25° C dry, (b) 96° C dry, (c) 25° C wet, (d) 96° C wet. Note that these are single fractographs and can only crudely represent the diversity of morphologies and the average behaviour observed under the various conditions.

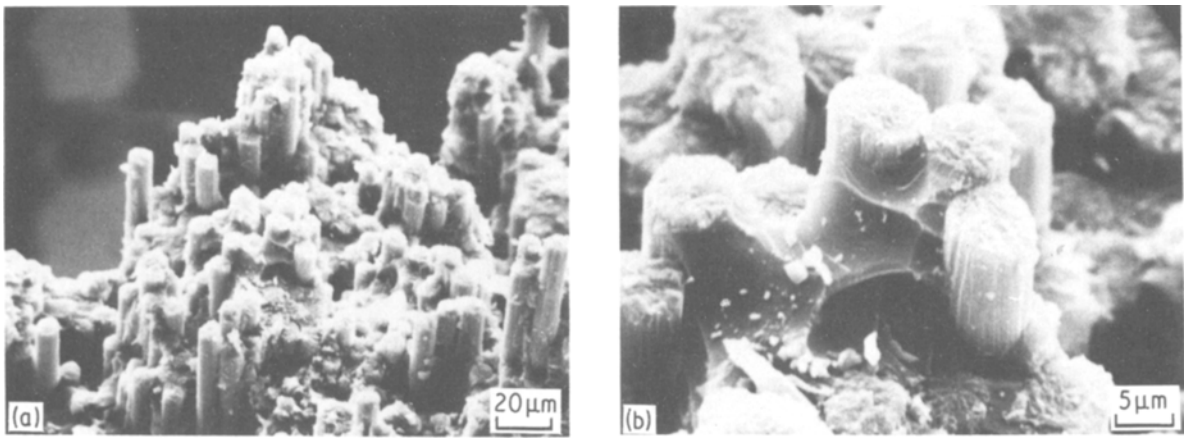

Figure 5 (a, b) Representative failure morphologies for Batch A specimens tested at 25° C dry. Note concoidal failure of epoxy in resin-rich area.

represent the different morphologies with only a few fractographs. None the less, Fig. 4 compares low magnification views of specimens tested under the four conditions, and Figs. 5 to 8 show "representative" failure morphologies (all for un-postcured Batch A specimens having polished edges) for the four environmental conditions considered.

Our "baseline" condition was 25° C dry. As can be seen from Figs. 4a and 5, under these conditions we found – in "high-energy" regions – a tiered microstructure resulting from cooperative fibre failure, as was found in similar graphite–epoxy system by Miller and Wingert [16]. Few long and/or bare filaments or pull-out holes were observed. (Purslow [15] referred to such a microstructure as "hill and valley". It is common in state-of-the-art systems, but is considerably more irregular than that observed by Sinclair and Chamis [9] in the older Modmor I/ERLA 4617–MDPA system.) The epoxy in matrix-rich regions was found to fail by concoidal fracture.

We next examined the fractography at 96° C dry to determine whether the increased strength at elevated temperature (for "normal" Batch A specimens) was reflected in the failure morphology. As described in Table II, we found differences in macroscopic failure modes for 25°C dry and 96° C dry. At 25° C dry there were no failures which were uniquely "high-energy" mode; 60% were "mixed mode" (where "high-energy" regions predominated), and the balance were tab or "low-energy" failures. Thus, 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, and 78% of the specimens failed by the "high-energy" mode. The remainder (presumably defective) specimens failed – all at low strengths – by the "low-energy" mode. Furthermore, at low magnification the failure surfaces were on the average more irregular at 96° C dry than at 25° C dry. Fig. 4b attempts to illustrate that the average surface had a more varied topography, possibly resulting from more secondary damage at 96° C dry than at 25° C dry. All of these differences are consistent with a decrease in flaw sensitivity with increased temperature.

In some of the 96° C dry specimens, considerable delamination (failure between layers) was seen. However, this was not a consistent finding, and on the average there was not significantly more delamination at 96° C dry than at 25° C dry.

On the average, in comparing 250 micrographs taken from 57 general areas in 12 specimens, we found no statistically significant difference in numbers or lengths of protruding filaments, nor in the amount of adhering epoxy, for 96° C dry against 25° C dry specimens. This similarity between the two conditions can be seen by comparing Figs. 5 and 6. (Note, however,

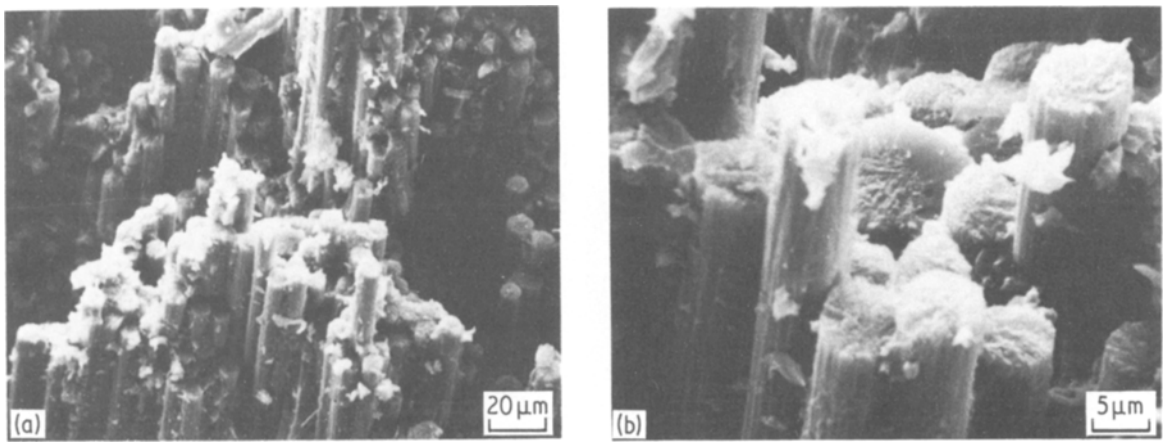

Figure 6 (a, b) Representative failure morphologies for Batch A specimens tested at 96° C dry.

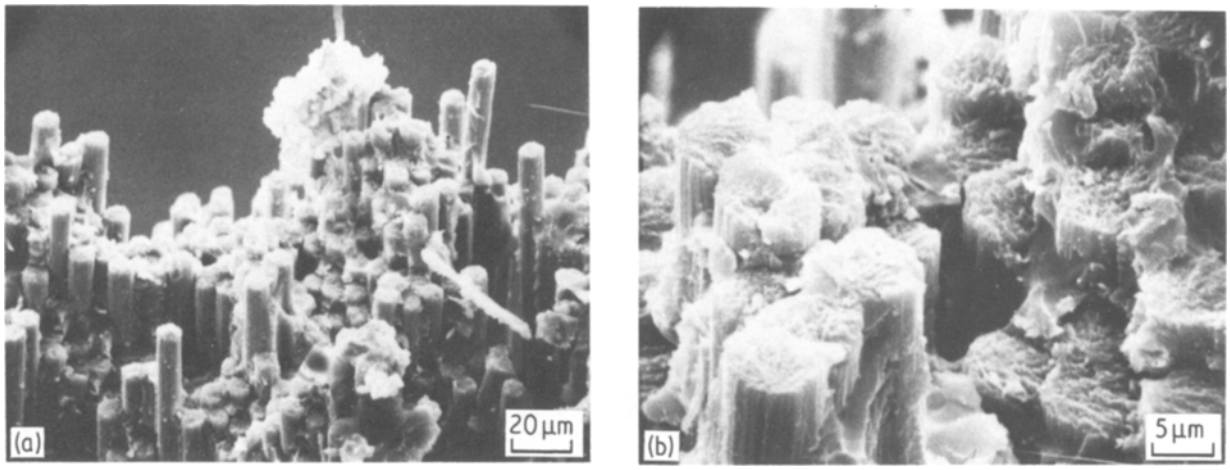

Figure 7 (a, b) Representative failure morphologies for Batch A specimens tested at 25° C wet.

that some individual pairs of specimens and/or areas under the two conditions did display different protruding filament lengths and amounts of adherent epoxy. This did, however, average out to no difference when all specimens and areas were compared.) Miller and Wingert [16], on the other hand, found longer and cleaner filaments following high-temperature failure. However, this may or may not indicate an actual difference from our findings since their "high" temperature of 134° C was well above ours of 96° C. We did, however, find that matrix-rich regions at 96° C dry showed more evidence of shear failure than of brittle concoidal failure. Again, this is consistent with increased epoxy ductility and thus decreased flaw sensitivity at 96° C dry.

An examination of the fractographs at 25° C wet was used to determine whether the apparent increase of strength with increased moisture content (once "low-energy" failures were eliminated) was similarly reflected in the failure morphologies. A comparison of macroscopic failure modes at 25° C dry and 25° C wet (see Table II) revealed that at 25° C dry there were 60% "mixed-mode" failures while at 25° C wet there were 50% "high-energy" and only 12% "mixed-mode" failures. Furthermore, the failure surfaces at 25° C wet were more irregular at low magnification than those at 25° C dry (compare Figs. 4a and c). This is once again consistent with a decrease in flaw sensitivity, but now with increased moisture content rather than increased temperature.

These effects due to increased temperature or moisture content can easily be rationalized since 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 [18], and thus increase ductility while decreasing flaw sensitivity. Moisture or temperature also reduces residual stresses in the epoxy matrix. Although matrix effects on 0° strength are frequently discounted, if these effects are of sufficient magnitude it is reasonable to expect a corresponding decrease in overall 0° composite flaw sensitivity.

Loose, broken-up epoxy (as in the area shown in Fig. 7) was found in some specimens or areas at both 25° C dry and 25° C wet. On the average, however, there seemed to be no greater occurrence under one condition than the other. The matrix appeared to fail primarily by concoidal fracture in epoxy-rich areas under both conditions, but some indication of shear character in the failure was found at 25° C wet. On the average, at 25° C wet the filaments protruding from the failure surface were about 20% longer and somewhat cleaner than at 25° C dry. (This is consistent with

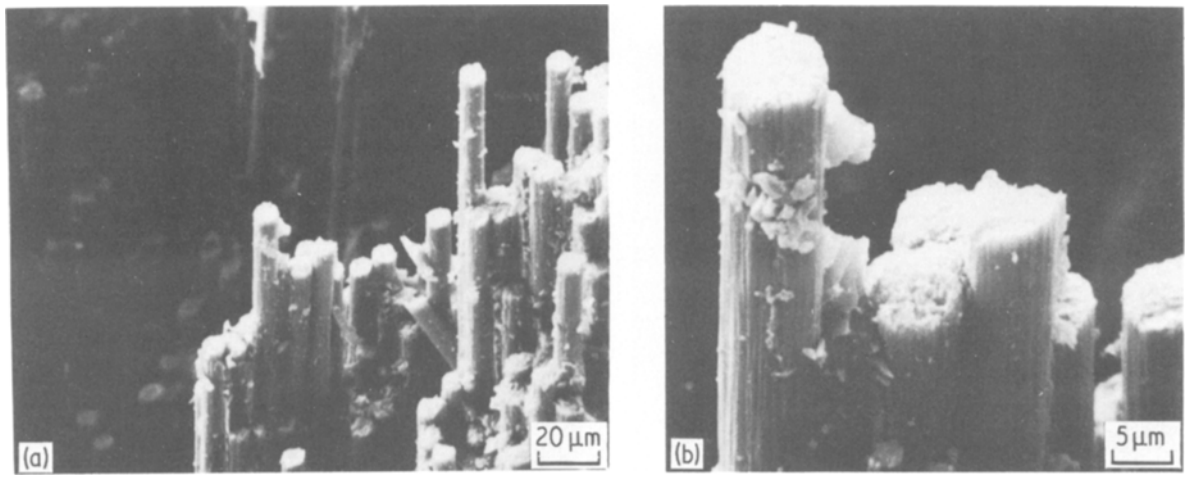

Figure 8 (a, b) Representative failure morphologies for Batch A specimens tested at 96° C wet.

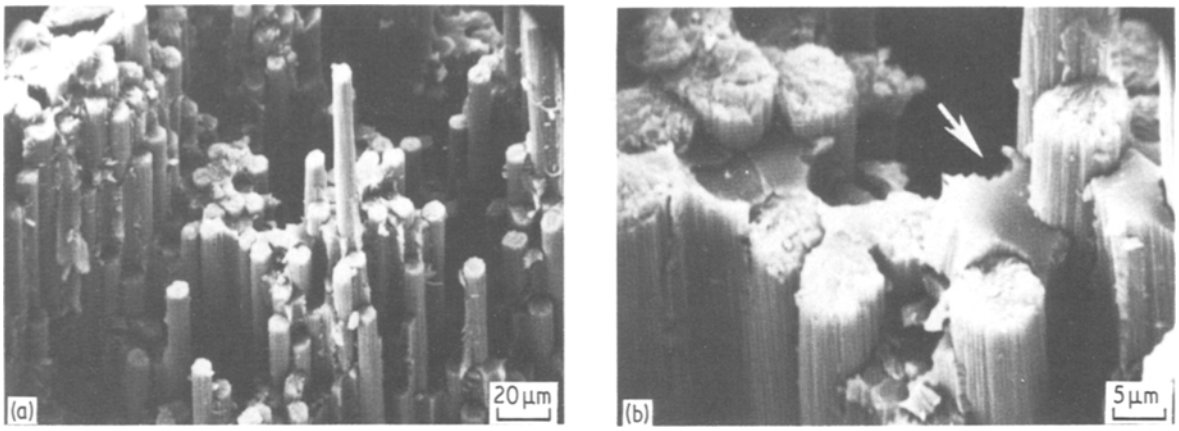

Figure 9 Scanning electron fractograph of Batch A specimen tested at 96° C wet: (a) shows long clean filaments but few pull-out holes, and (b) shows epoxy that has broken up and appears ready to be lifted away (arrow).

the findings of Miller and Wingert [16] on much drier "wet" specimens.) This difference unfortunately is not particularly apparent from comparison of Figs. 5 and 7. As is illustrated by Figs. 4a and c, there were consistently more delaminations in the failure surfaces at 25° C wet than at 25° C dry.

These observations are consistent with somewhat increased interfacial debonding between filament and matrix. As stated previously, when "low-energy" failures are discounted, the longitudinal tensile strength increases from 25° C dry to 25° C wet. This would indicate that the increased interfacial debonding did not weaken the overall composite, or that any weakening was offset by decreased flaw sensitivity. However, it is possible that the fairly large number (38%) of "low-energy" failures at 25° C wet (plus 12% "mixed-mode" failures where "low-energy" regions predominated) may imply a deleterious effect of moisture content rather than simply a random concentration of defective specimens. More study would be required to clarify this.

The influences of temperature and moisture were combined at 96° C wet. At low magnification the failure surfaces under these conditions showed on the average both more delamination and more intralaminar splitting than for any other conditions. This difference is only partially illustrated in the fractographs of Fig. 4d. Figs. 8 and 9 attempt to show that the failure surfaces on the average contained more bare filaments than those at 25° C wet or at 96° C dry. The filaments were about 30% longer and generally 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 Figs. 8 and 9, there were few corresponding pull-out holes. Examination of many opposing failure surfaces gave the same results: (relatively) long, clean filaments, but few or no pull-out holes. But such an examination also revealed an explantation, as demonstrated in Fig. 9. In this figure a small piece of inter-fibre epoxy (see arrow) has broken loose, and seems to be ready to be lifted out. Such broken epoxy pieces were observed again and again in failure surfaces at 96° C wet. Apparently not only did the epoxy–filament interface fail adhesively but the epoxy itself also broke up and fell away. It thus appears that the combined influence of temperature and moisture was both to increase interfacial debonding and to make the epoxy more prone to fracture. Thus although the strength data at 96° C wet were unreliable, we would expect the two deleterious effects observed in the failure morphologies to result in a decrease in strength for this condition compared with either 25° C wet or 96° C dry. Once again, this conclusion seems to be supported by the results of the flexural study to be reported in another paper (in preparation).

4. Conclusions

The conclusions of this study can be summarized as follows:

1. A distinctive type of failure morphology shown by our specimens, which we have labeled "low-energy", probably resulted from undetected specimen defects. The "low-energy" failure morphology could generally be used as an indicator of a defective specimen or test.
2. The damaged edges of our unpolished-edge specimens constituted defects which produced low strength and a "low-energy" failure morphology.
3. Regions of altered epoxy in the Batch B specimens led to a lowered strength at elevated temperature or moisture content. Such a defective batch could be easily in the future detected by testing at elevated temperature and moisture content.
4. Postcuring had no statistical influence on strength, but may have produced slightly longer, cleaner protruding filaments and a slightly more brittle matrix.
5. Elimination of defective specimens and tests led to the conclusion that an increase in moisture content from dry to wet at 25° C produced a significant increase in strength. This may have resulted from decreased flaw sensitivity with increasing moisture content.
6. The increase in longitudinal tensile strength of dry specimens with increased temperature may have been due to decreased flaw sensitivity with increasing temperature.
7. Combined temperature and moisture produced more interfacial debonding and also apparently allowed the epoxy to fracture more easily. However,

unreliable strength data could not confirm the anticipated strength degradation.

Finally, we would like to emphasize one point. The work of Clements and Lee [1] and this follow-up to that work have shown the considerable effect that both quality control and environmental variables can have on the fibre-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, particularly under "worst state" conditions such as "hot wet".

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