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MTL TR 85-36

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CAUSES OF DELAMINATION FAILURES OF KEVLAR/EPOXY LAMINATES USED IN HARDENED ARMY TACTICAL SHELTERS

WILLIAM W. HOUGHTON and MARGARET E. ROYLANCE
COMPOSITES DEVELOPMENT DIVISION

December 1985

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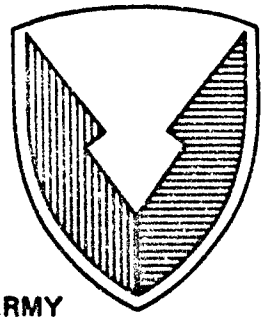

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ABSTRACT

A series of chemical, mechanical, and physical tests were performed on Kevlar/epoxy laminates to determine the cause of failure in a structure which had undergone extensive delamination during a simulated solar load test. The results of these tests indicated that improper fabrication procedures caused microcracking at the fiber matrix interface which made the material particularly susceptible to invasion by environmental moisture and subsequent delamination at elevated temperatures. The material was also found to have been undercured by approximately 30%. Acting on recommendations resulting from this study, the fabricator was able to eliminate the microcracking and significantly increase the degree of cure in the laminates.

Preliminary measurements of the effect of moisture on the mechanical properties of the laminate suggested that design properties of the material should be determined at elevated temperature and humidity.

CONTENTS

INTRODUCTION

In May, 1983, at the request of the U.S. Army Natick Research and Development Center (NRDC), Organic Materials Laboratory (OML) personnel were requested to attend technical review meetings to discuss materials-related problems arising from the use of Kevlar/epoxy panels on the Hardened Army Tactical Shelter (HATS). Shelter A2 had developed extensive delaminations and ply separations during a simulated solar load test.

After examination of shelter A2 and chemical, mechanical, and physical testing of materials removed from this and other shelters, a series of recommendations were made to the HATS technical working group concerning fabrication of the Kevlar/epoxy panels. These recommendations included:

1. Thorough drying of the Kevlar fabric at a temperature above 100°C before impregnation with the epoxy resin.

2. Careful quality control to avoid batch-to-batch variation in the resin.

3. Not diluting the resin with organic solvents such as acetone during impregnation.

4. Curing the panels following the complete recommended cure cycle, with particular attention to the postcure at 200°C.

5. Determination of the design properties of the Kevlar/epoxy at a moisture content and temperature appropriate to the expected environmental exposure of the HATS shelter.

Detailed results of the testing program and reasons for these recommendations will be discussed in this report.

EPOXY RESIN CHEMISTRY

The first test performed on material from A2 was an infrared spectrum (IR) analysis of gas withdrawn from the blisters in the delaminated regions of the shelter roof. These tests were run by the Physical Test Branch of the Measurements and Analysis Division of Aberdeen Proving Ground (APG), at the request of OML. The results of the IR analysis indicated the presence of acetone in the blisters. It was not known if the acetone was originally present in the epoxy resin, since the composition of the resin used by the panel manufacturer MIKI (Erba, Italy) was not specified.

Two batches of the resin (Eposir SP.502) manufactured by S.I.R. Conzorzio Industriale S.P.A. (S.I.R.) of Milan, Italy, were obtained from Harry Diamond Laboratories (HDL) for chemical characterization. A number of techniques were used for chemical characterization of the resin. These included high performance liquid chromatography (HPLC), gas chromatography/mass spectrometry (GC-MS), differential scanning calorimetry (DSC), and fourier transform infrared spectroscopy (FTIR). HPLC, GC-MS, and FTIR were used to determine chemical composition, and DSC was used primarily to measure the extent of reaction in the cured resin and in the laminates.

Two HPLC techniques were used in the identification of the resin: reverse phase high performance liquid chromatography (RPHPLC) on a Perkin-Elmer chromatograph, and size exclusion chromatography (SEC) on a Waters Associates instrument.

The hardener (Eposir ID01801) was analyzed using RPHPLC, and found to be nadic methyl anhydride (NMA), although the dark color of the sample suggests that there is another component present.

The RPHPLC results for the two Eposir resin batches are shown in Figure 1. They indicate significant variations in the intensities of one of the peaks. Comparison of the HPLC fingerprint of the Eposir with that of DEN 431, a Dow epoxy resin containing polyglycidyl ether of phenol-formaldehyde novolac (Figure 2) shows that the Eposir is primarily a novolac resin, but additional components corresponding to the two peaks marked with arrows in Figure 2a have been added. GC-MS was used to identify these two components.

Samples of the two unidentified Eposir fractions were run in a GC-MS apparatus consisting of a Perkin-Elmer model 3920 gas chromatograph coupled with a Finnigan model 3300 mass spectrometer. The results are shown in Figures 3 and 4. The first peak was tentatively identified as diglycidyl ether of resorcinal, commonly used as a reactive diluent in these materials to decrease the viscosity of the resin during processing. The second component was identified by both GC-MS and FTIR as diglycidyl ether of bisphenol-A (DGEBA). This is a widely used difunctional epoxy, and was apparently added by the manufacturer in amounts which varied from 1% to 3% from batch to batch. Apparently, the Eposir resin was still considered experimental by the manufacturer during the time that these two batches were obtained by HDL. The resin system has since been fully commercialized, and presumably no further variations in resin chemistry will be made by the manufacturer unless purchasers are so informed. Since such variations do sometimes occur in practice, the use of some method of incoming QC to monitor resin composition was recommended. HPLC "fingerprinting" is an excellent incoming QC method, and therefore a detailed description of both HPLC techniques used in this study are included for reference in the Appendix.

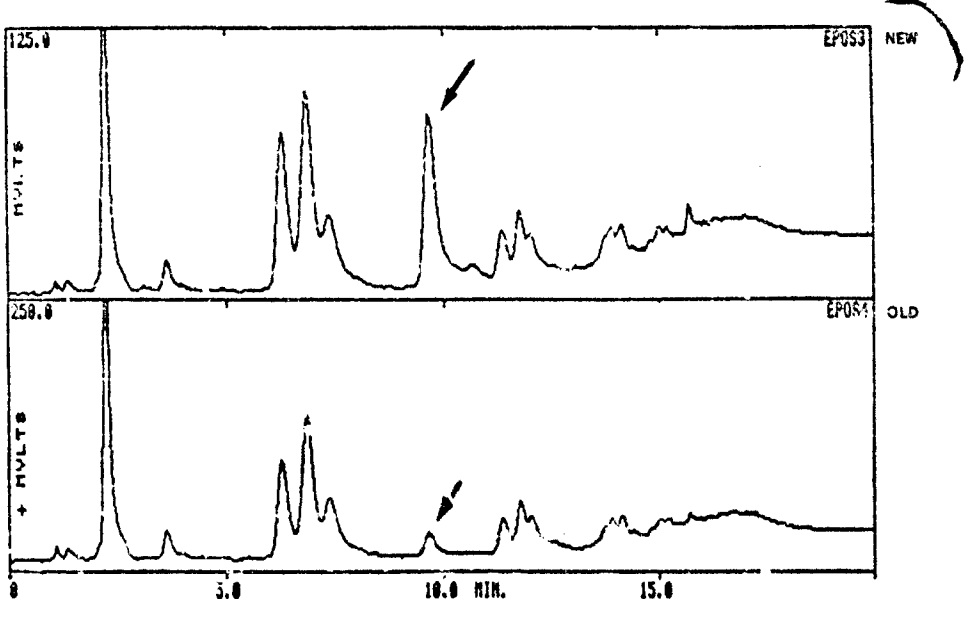


Figure 1. HPLC data for two batches of Eposir resin. Variable peak is marked by arrow.

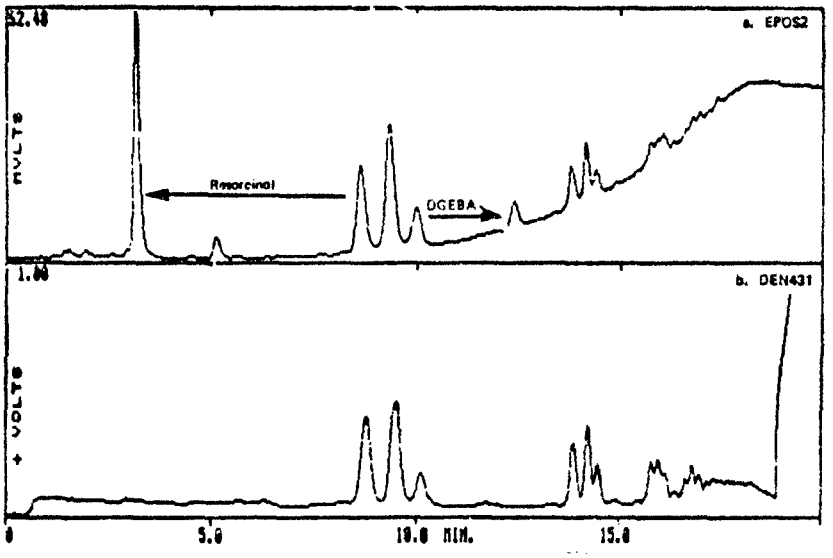
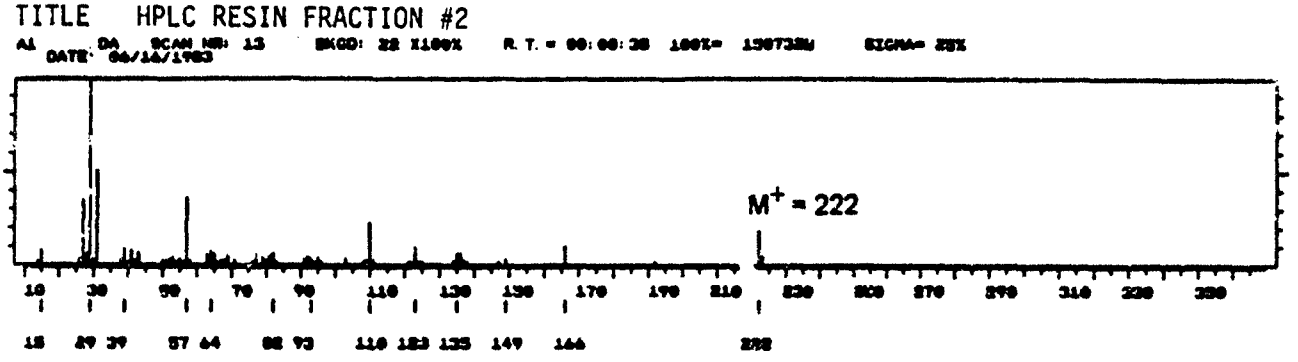


Figure 2. HPLC data for comparison between Eposir resin (a) and DEN 431, a novolac resin (b). Peaks corresponding to resorcinol and DGEBA are marked on Eposir curve.



Tentative: $H_2C - HC - H_2C - O-C_6H_4-O - CH_2 - CH - CH_2$ (epoxide rings)

MW = 222

Functionalities:

m/c 27 C_2H_3

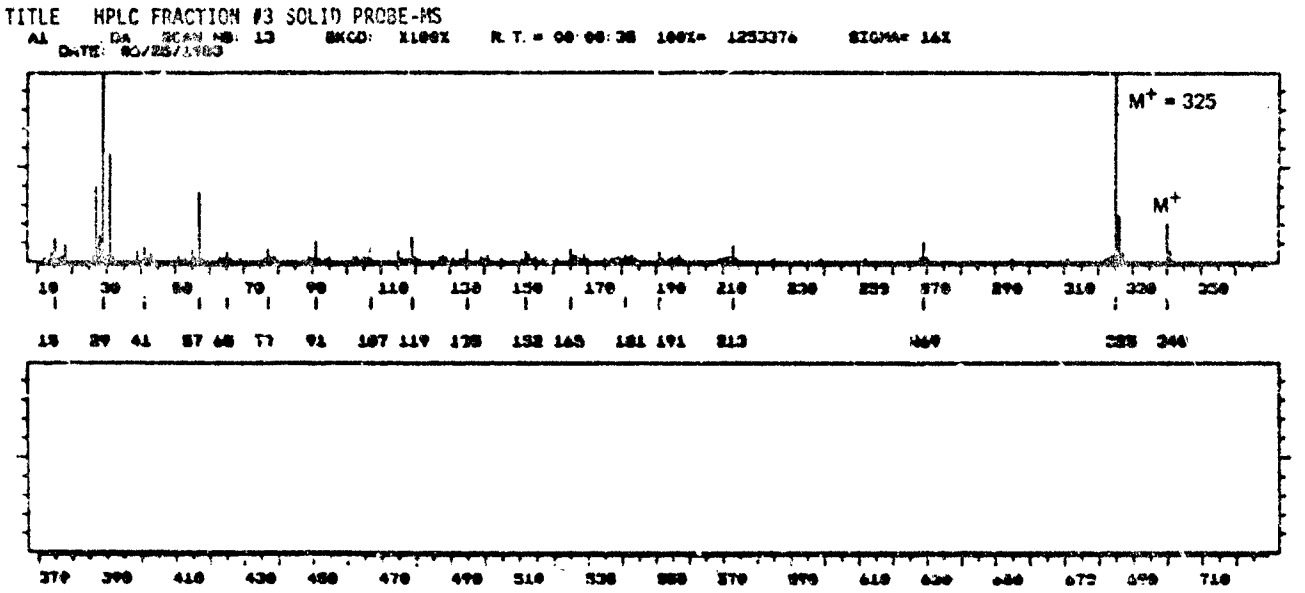
m/c 29 C_2H_5

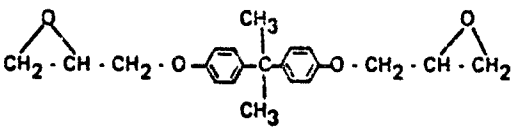
m/c 31 CH_3O

m/c 57 $C_2H_5O - CH_2 - CH(O) - CH_2$

m/c 110 $HO-C_6H_4-OH$

Figure 3. GC-MS data which identify second unknown HPLC fraction as diglycidyl ether of resorcinol.





MW = 340

[DGEBA]

Figure 4. GC-MS data which identify third unknown HPLC fraction as diglycidyl ether of bisphenol-A (DGEBA).

None of the chemical techniques used to determine resin composition indicated the presence of acetone, but during the HPLC analysis, the continual loss of low molecular weight resin components by evaporation was noted. Since a gradual increase in viscosity accompanies this selective evaporation, a fabricator might be tempted to add a solvent such as acetone during a lengthy processing operation. It was finally learned that MIKI had diluted the resin with acetone during the impregnation process. OML recommended that this practice be stopped. Since the acetone is still present in the cured laminate, it is clearly trapped in the material during cure, and could contribute to environmental instability in the panels.

ANALYSIS OF SPECIMENS FROM SHELTER A2

On June 9, 1983, OML personnel visited APG and removed several specimens of the roof and sides of shelter A2 for chemical and physical analysis. Figure 5 shows the number of delaminations which occurred in one specimen location on the roof in the center of the bubble. The specimens were removed with a 3-inch-diameter hole saw. Figures 6 through 12 are color photographs and photomicrographs of some of these specimens. Two significant observations were made concerning these specimens: 1) the generally pale color of the internal delaminated surfaces (see Figures 6 and 7) suggests that the resin inside the laminate is incompletely cured, and 2) while some delaminated surfaces appear to be resin poor, examination of matching

Figure 5. Section of the bubble in the roof of shelter A2. In this region there were two separate ply separations plus delamination from the honeycomb substrate.

fracture surfaces frequently shows them to be resin rich (see Figures 6 versus 7, and 8 versus 9). It appears that the environmentally induced fracture which caused these delaminations occurred at the fiber-matrix interface along the edge of a fabric ply. This is also shown in Figures 10, 11, and 12 which show a partially delaminated specimen. The close view in Figure 12 shows a resin-rich fracture surface on the bottom of the crack and bare fibers exposed on the top surface.

Both the extent of cure of the matrix and the nature of the environmentally induced fracture were determined by analysis of the test specimens.

Extent of Cure

A DuPont 1090 DSC was employed to measure the extent of cure of the resin matrix in the specimens from A2. Extent of cure is determined from DSC measurements by measuring the residual exotherm which occurs in the cured material as the specimen temperature is scanned through the cure temperature, and comparing this value to the original exotherm which occurs during curing of the uncured resin. In order to make this comparison, the volume fraction of resin which is present in the composite must be accurately determined. This is difficult with Kevlar/epoxy composites since most conventional methods for selective removal of the resin from the composite, such as burnout, also remove the organic Kevlar fibers.

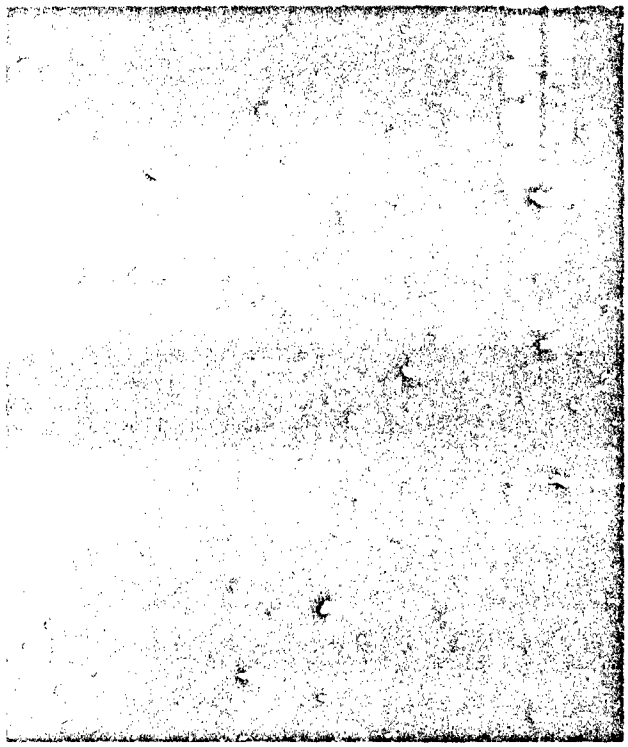


Figure 6. Internal delaminated surface from the section shown in Figure 5. Surface is pale in color and resin starved.

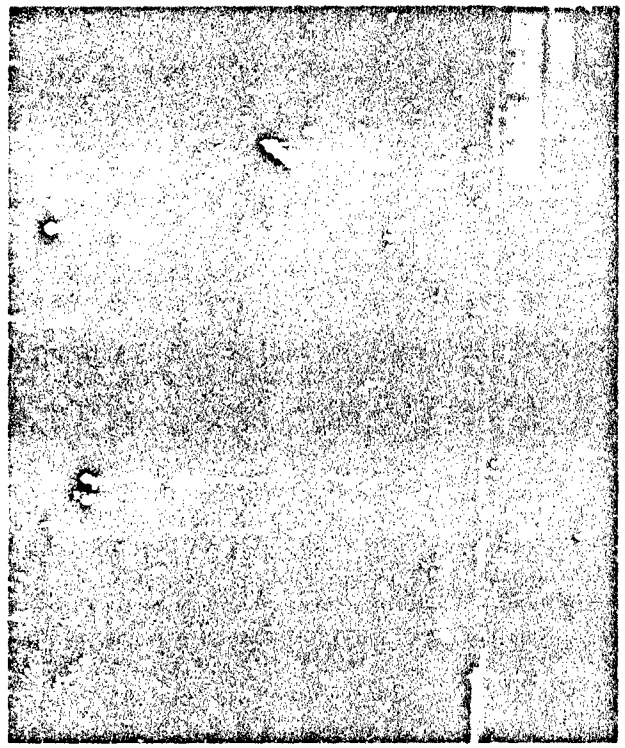


Figure 7. Other side of delamination shown in Figure 6. This is much more resin rich than the matching surface, indicating failure occured at a ply boundary.

The technique which was used in this case was quantitative optical microscopy (QOM). This technique requires considerable skill both in the preparation of the specimen and operation of the microscope, but it is by far the most effective technique for Kevlar/epoxy. QOM measurements showed that the volume fraction of Kevlar fiber in the A2 specimens varied from 48% to 50%. Sample results of QOM measurements are shown in Figure 13. Figure 14 shows the curing exotherm of the unreacted epoxy. With a scanning rate of 5°C/min, the epoxy exhibits a curing exotherm of 263 joule/gram with a center temperature of 137.7°C. The results of a similar measurement on a composite specimen from shelter A2 are shown in Figure 15. This specimen exhibits a curing exotherm of 38.7 joule/gram and, since only roughly half of the material in this specimen is resin, this would correspond to a residual curing exotherm of almost 80 joule/gram in the A2 resin. These data indicate that the resin in the A2 shelter is only 70% cured. This extent of undercure could significantly degrade the environmental stability of the cured composite. The manufacturer's recommended cure cycle for this resin includes curing the panels at 180°C for one hour, and postcuring for 19 hours at 200°C. OML personnel obtained a recorded cure cycle from MIKI which indicated that, while the original cure was performed at 180°C, the 19-hour postcure was carried out at a temperature significantly (5°C to 10°C) below 200°C. OML recommended that, since the postcure contributes strongly to the final extent of cure, the full 19-hour postcure should be carried out at 200°C. It was also recommended that if more than one panel is postcured at a time, adequate space be allowed between them for air circulation.

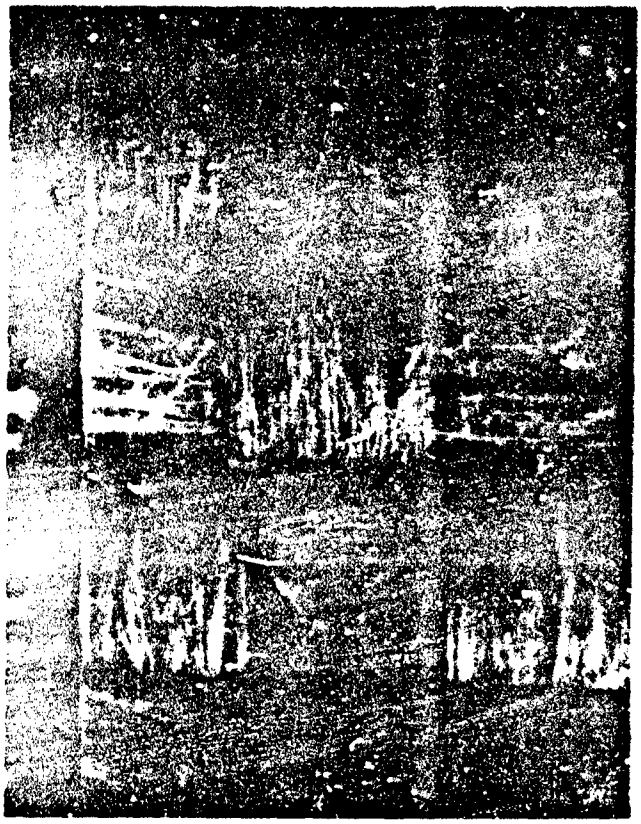


Figure 8. Magnified view of the fracture surface shown in Figure 6.

Figure 9. Magnified view of the fracture surface shown in Figure 7.

These recommendations were followed during the fabrication of shelter A5, and DSC measurements of test specimens from shelter A5 (Figure 16) indicate a residual resin exotherm of about 30 joule/gram compared to a resin exotherm of 80 joule/gram in A2. This represents a considerable improvement in the extent of cure. Elimination of acetone from the resin, and thorough drying of the Kevlar fabric, as discussed below, may also have contributed to a higher degree of cure in shelter A5.

Optical Microscopy

Optical microscope specimens were prepared from several of the delaminated regions of shelter A2. Material removed from the shelter was cut, mounted in epoxy, and polished with a metallurgical polishing wheel. These specimens were used for QOM fiber volume measurements reported in the last section and also for analysis of the internal fracture surfaces generated during the simulated solar load test. Microscope specimens were also prepared from material from shelter B3, which had not been exposed to blast or simulated solar load, for comparison.

Figure 17 shows a micrograph of an A2 specimen along the environmentally delaminated edge from the region shown in Figures 11 and 12. Figure 18 shows micrographs at two different magnifications of specimens from shelter A2 (labeled "bad") and micrographs at the same magnifications of specimens from the B shelter (labeled "good"). Figures 17 and 18 show that, in addition to the actual delamination, the

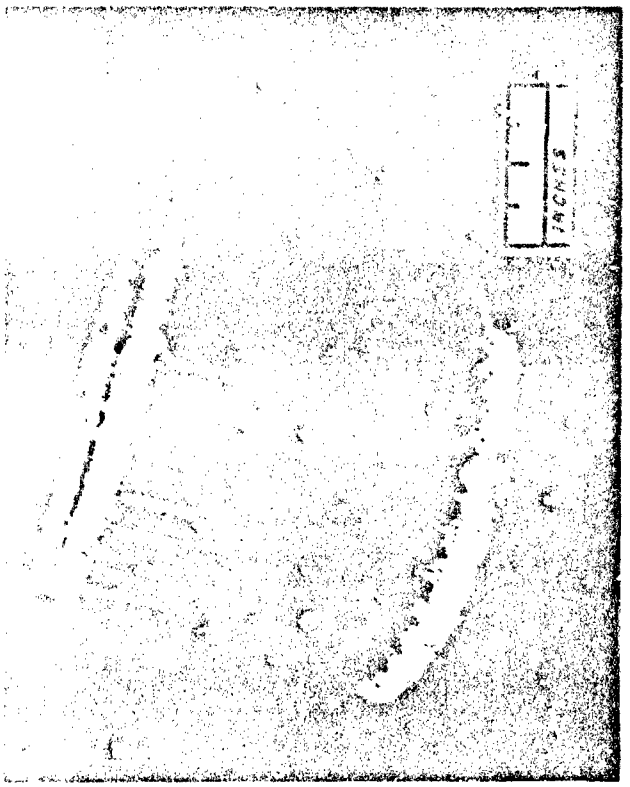


Figure 10. Specimen removed from another section of the roof of A2. The skin was partially delaminated in this region.

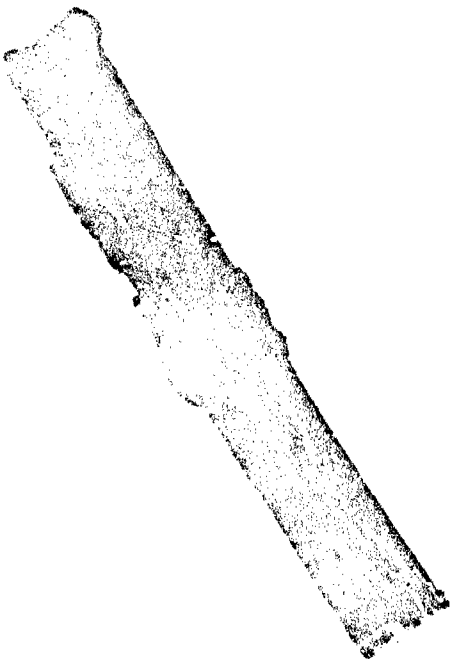


Figure 11. Polished specimen from the section shown in Figure 10.

A2 material contains many small cracks which run along the fiber-matrix interface. The delaminations themselves appear to be the result of coalescence of these cracks at ply surfaces.

As Figure 18 shows, these cracks are present not only in the A2 material which has been exposed to blast and simulated solar load, but also in the material from shelter B3, which had not been exposed to either. This suggests that some difficulty arising during fabrication caused these cracks. The micrographs in Figure 19 indicate that such cracks are not present in all similar Kevlar/epoxy materials. These micrographs show magnified views (400X) of Kevlar/epoxy specimens; two from different locations in shelter A2 - one from the B shelter, and one from a material prepared as part of round-robin testing for Mil Handbook 17, a composite materials properties data base. This material was fabricated from the same resin system used by MIKI, although the material was obtained in the form of prepreg, and the same cure cycle was employed. It does not contain extensive fiber-matrix cracking.

Care was taken during the processing of the Mil Handbook 17 material to ensure that the Kevlar prepreg was dry before laminate fabrication. Manufacturers generally dry the Kevlar fabric adequately before prepregging. The fabric should be dried at temperatures above 100°C for several hours. This procedure was not followed by MIKI during fabrication of the earlier shelters. If environmental moisture is not removed from the Kevlar fabric prior to cure, the elevated temperature will drive

Figure 12. Magnified view of specimen in Figure 11.

it from the fibers into the fiber-matrix interface. This would be expecced to cause the type of damage which was observed in the shelter materials. The correct drying procedure was incorporated into the fabrication of shelter A5, and a microscopic examination of the A5 material showed no such damage.

A likely scenario for the catastrophic delamination which occurred during simulated solar loading of shelter A2 is as follows. Environmental moisture was absorbed into the shelter during storage, pooling in the cracks which were present due to improper drying of the Kevlar fabric. Some residual acetone may also have mixed with the liquid water in the cracks. Since the thermal conductivity of the Kevlar/epoxy composite is considerably lower than that of the metal in the standard shelter, the procedure followed during simulated solar load resulted in measured temperatures at the surface of the laminate directly under the heat source of 260°F to 280°F. These temperatures would be sufficient to induce local boiling within the cracks, causing coalescence and subsequent delaminations both between plies and from the honeycomb substrate. Although the test was much too severe to actually simulate solar loading, it was fortuitous that the delaminations occurred because it brought to light a number of indequacies in the fabrication process which, if uncorrected, were likely to have caused problems with the long-term stability of the shelter.

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ROUTINE
(WRITTEN  22-AUG-83 )   SPECIMEN IDENT =6009      22-AUG-83
                                        PAGE  1 OF   1    UNITS: MICRONS

FIELD 100

INST 3

      DIFFERENTIAL HISTOGRAM)  UNITS : MICRONS

      MIN. VALUE = 26.4503                  MAX. VALUE = 66.9406

      MEAN = 48.6204                        STD. DEV. = 10.1732

      NO OF SAMPLES = 100                   MEDIAN = 49.0769

% KEVLAR FIBER
    0
10
    0
14
    0
18
    0
22
    0
26
    4
30
    6
34
    7
38
    11
42
    12
46
    13
50
    14
54
    12
58
    13
62
    6
66
    2
70
    0
74
    0
78
    0
82
    0
86
    0
90
    0
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Figure 13. Sample QOM data on fiber content of Kevlar/epoxy laminates from shelter A2.

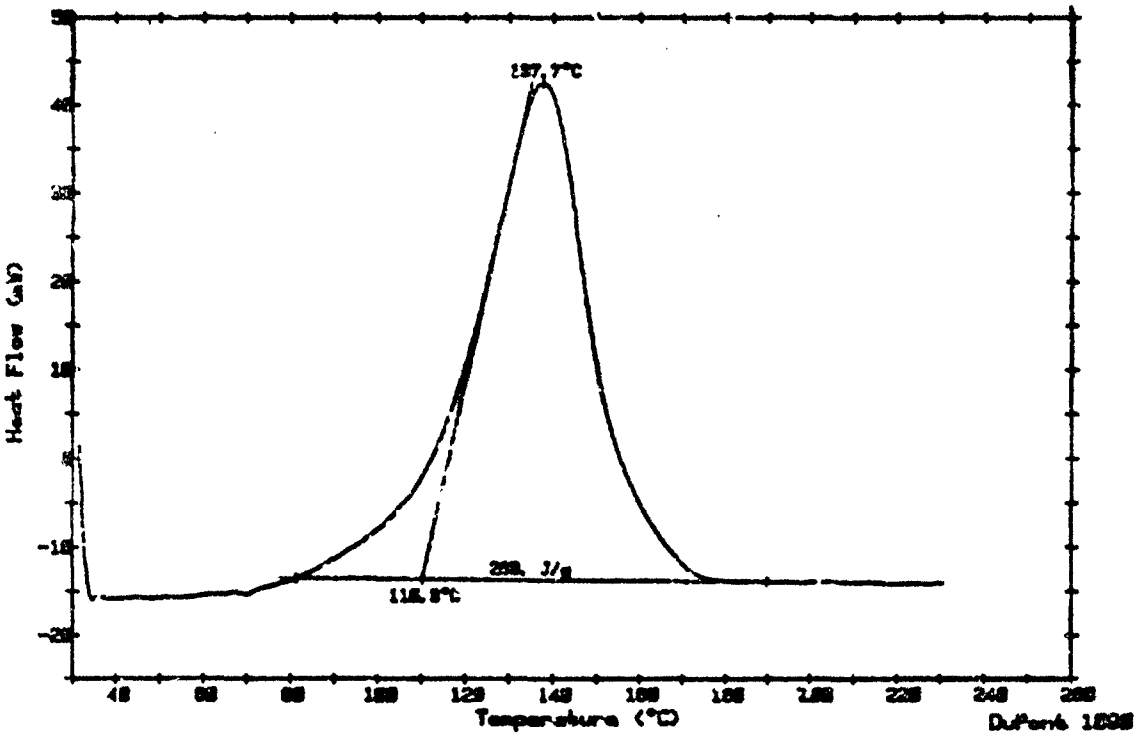

Figure 14. DSC measurement of the curing exotherm in the uncured Eposir HATS epoxy resin.

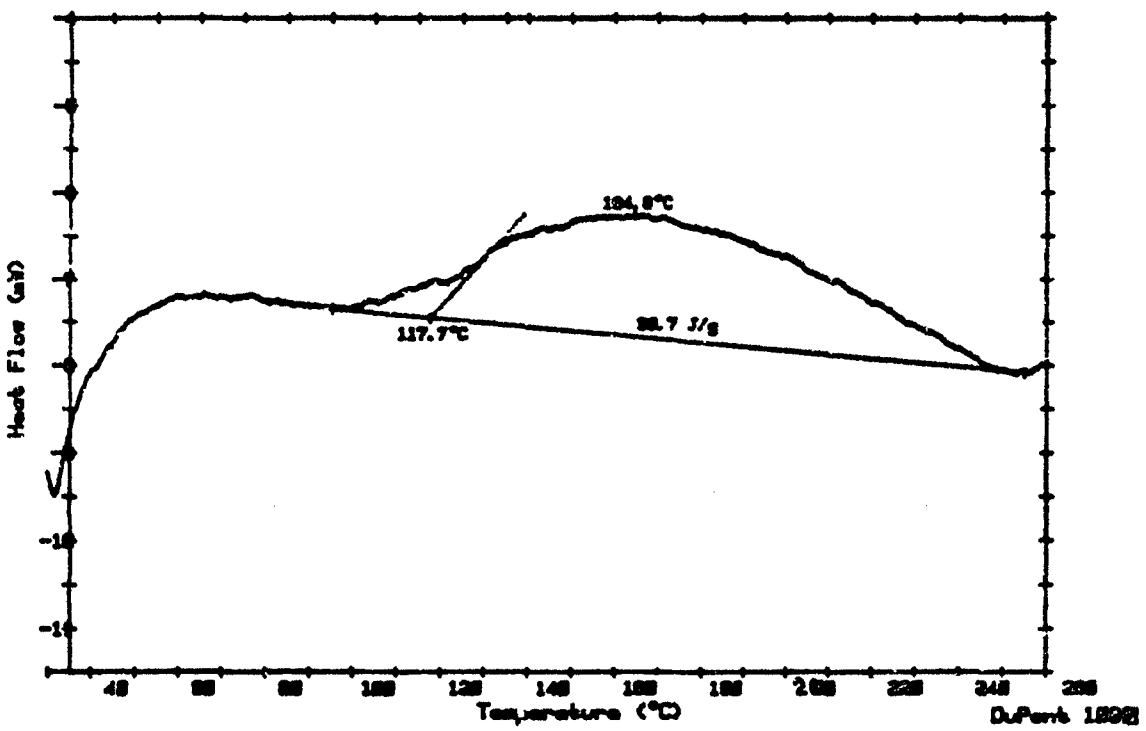

Figure 15. DSC measurement of the residual exotherm in cured Kevlar/epoxy material from shelter A2.

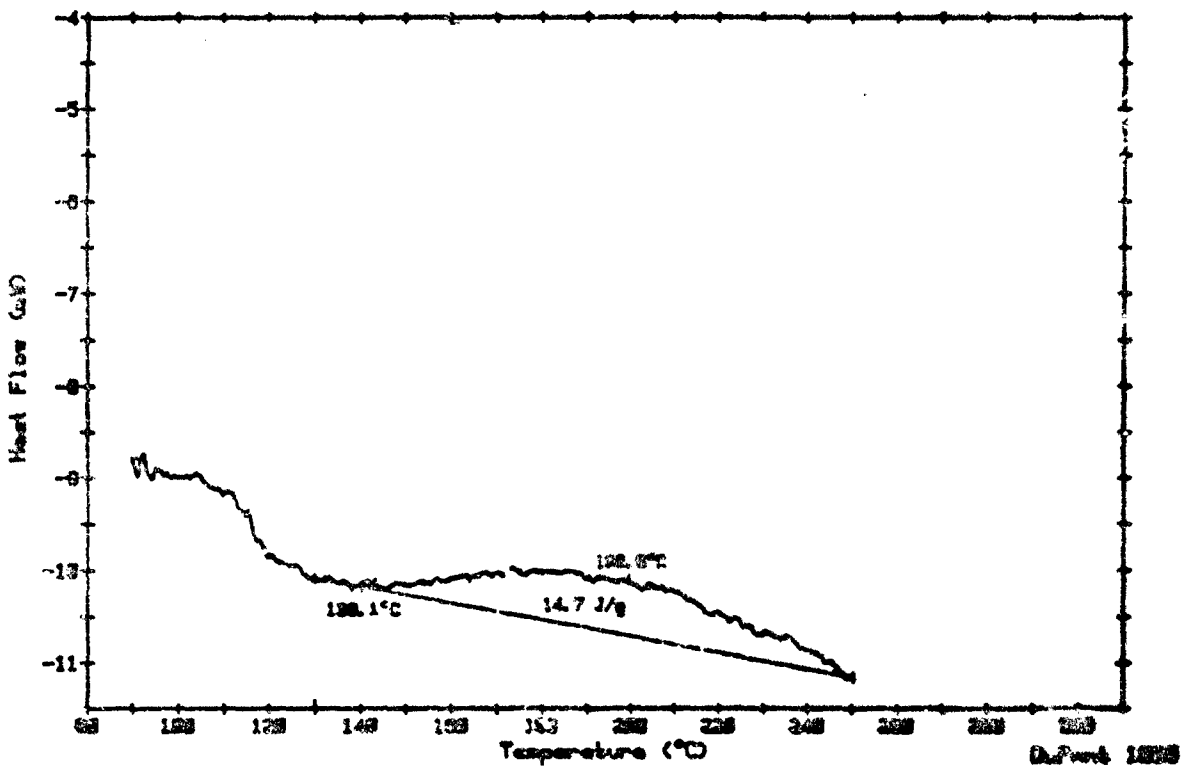

Figure 16. DSC measurement of the residual exotherm in cured Kevlar/epoxy material from shelter A5.

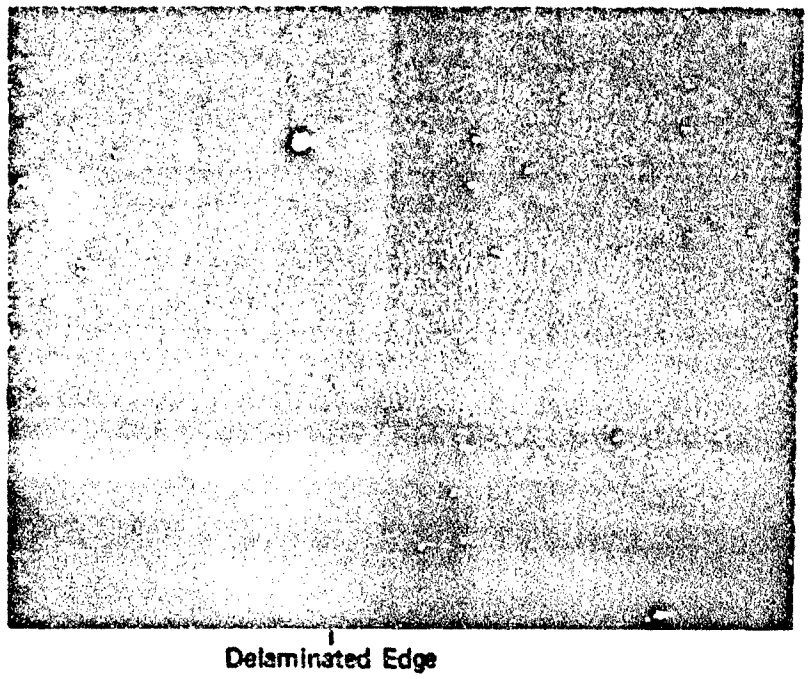

Figure 17. Photomicrograph at 80X of the edge of an environmentally induced delamination from shelter A2. Delamination is also shown in Figures 10 through 12. Figure shows fiber-matrix cracks coalescing at next ply boundary.

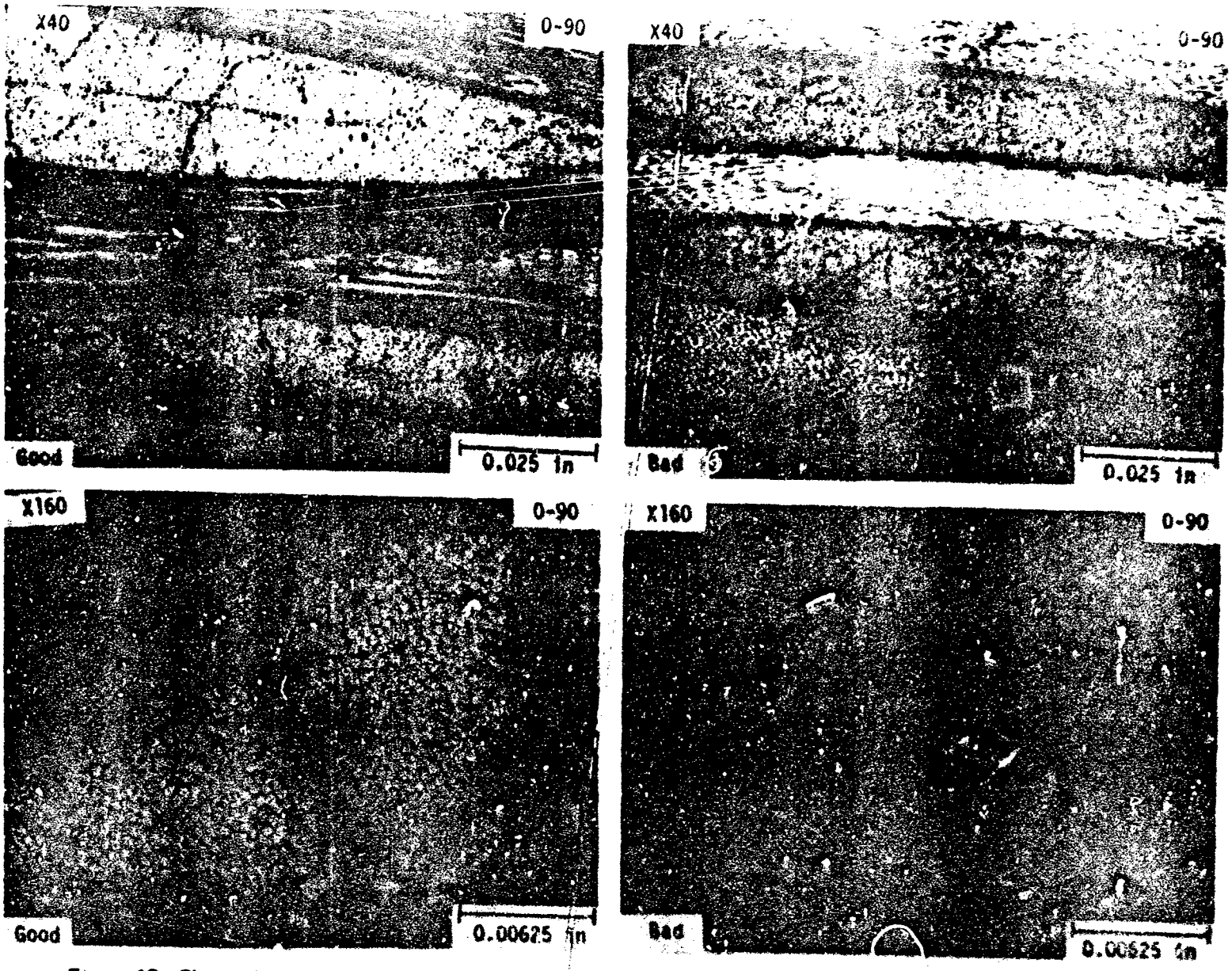

Figure 18. Photomicrographs at various magnifications of specimens from shelters A2 ("good") and R3 ("bad"). Micrographs show extensive fiber-matrix cracking in both materials.

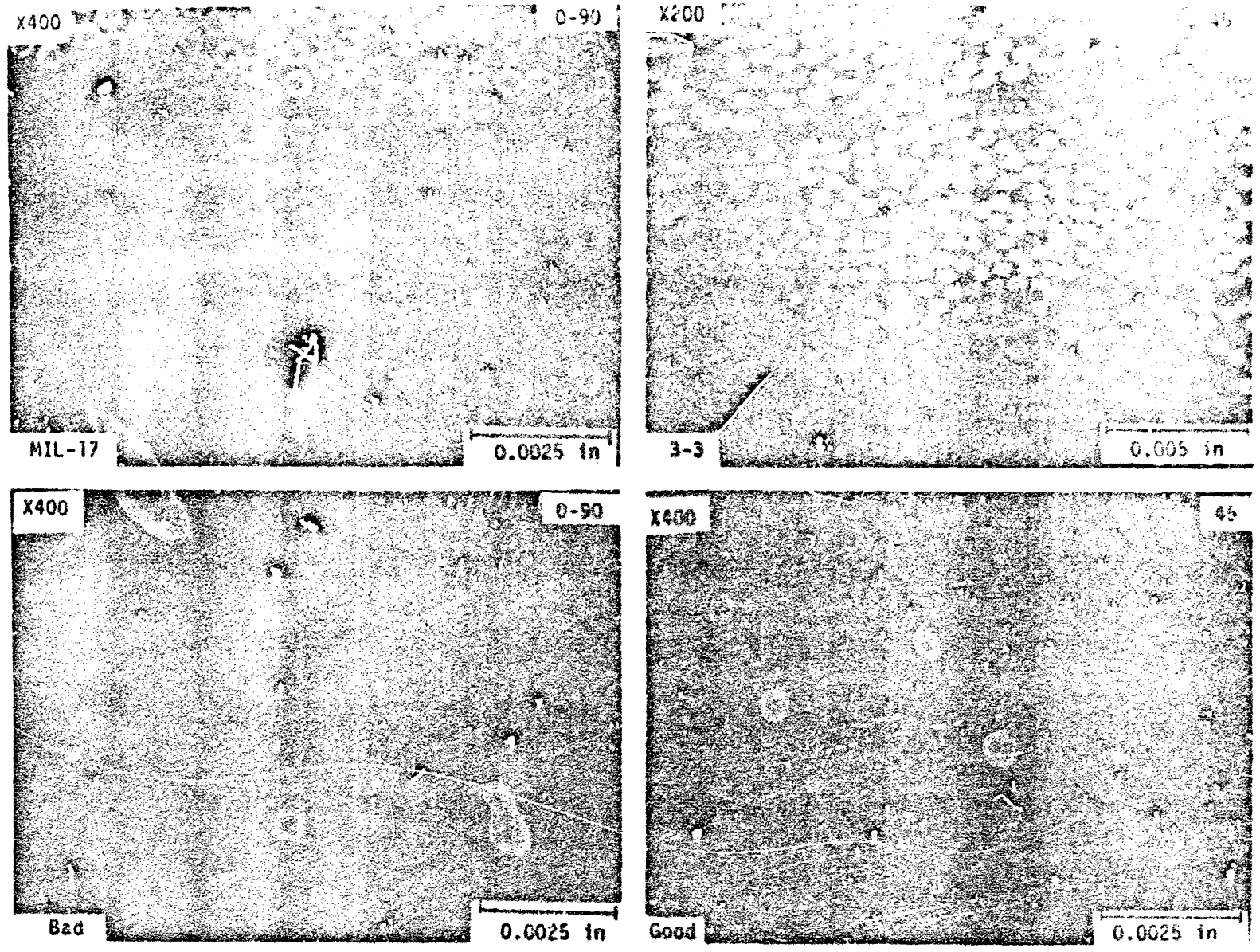

Figure 19. Photomicrographs at 400X of specimens from shelters A2 and B3 compared with a specimen from a Mil Handbook 17 Kevlar/epoxy laminate. Mil Handbook 17 material does not exhibit fiber-matrix cracking.

ENVIRONMENTAL STABILITY OF KEVLAR/EPOXY HATS LAMINATES

To ascertain the stability of the HATS laminates in the presence of environmental moisture, material was obtained from shelter B3 and machined into tension, short beam shear, and torsional pendulum test specimens. These specimens were all machined at 0/90° to the direction of the Kevlar fiber reinforcement. After machining, the specimens were dried in vacuum at slightly above 100°C until their weights stabilized. Half of these specimens were then tested to determine dry mechanical and dynamic mechanical properties. The remaining specimens were immersed in distilled water at 80°C until their weight gains had leveled off at 6% to 7%, and then tested. Immersion time required to achieve saturation was about one and a half months. Results of the tensile and short beam shear tests are shown in Table 1, and the dynamic mechanical data are shown in Figure 20.

The dynamic mechanical data indicate that the HATS material is strongly plasticized by absorbed moisture. Figure 20a is a plot of the square of the resonant frequency of the wet and dry torsional pendulum specimens as a function of temperature. The shear modulus of the specimen is directly proportional to this quantity. Figure 20b is a plot of the percent damping in the wet and dry materials as a function of temperature. The damping is a measure of the energy which is dissipated by the material during each cycle of loading. The organic matrix in these materials undergoes a glass transition between 100°C and 200°C, during which the damping reaches a maximum value, and the shear modulus drops by an order of magnitude.

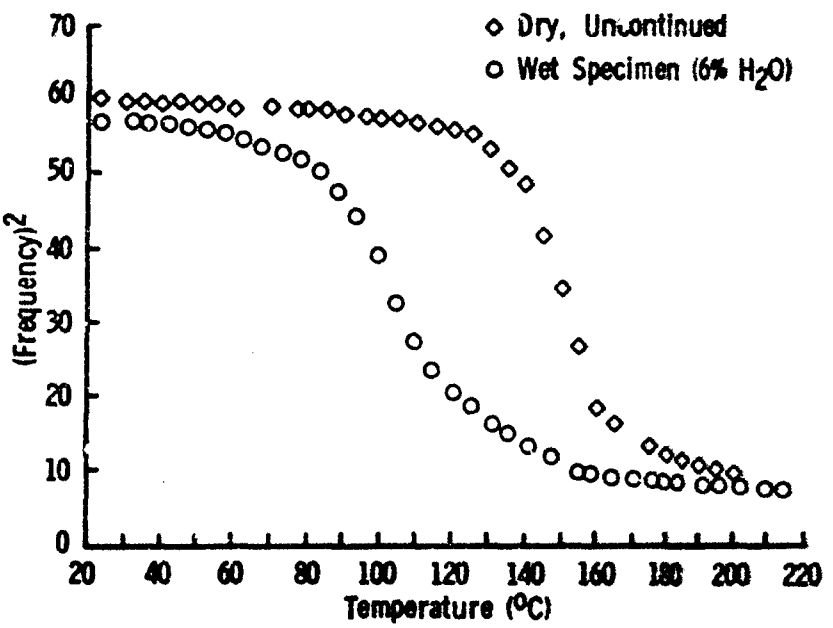

Figure 20a. Torsional pendulum data on high temperature stability of Kevlar/epoxy HATS materials at 0% and 6% absorbed moisture. Natural frequency squared is directly proportional to shear modulus, G1.

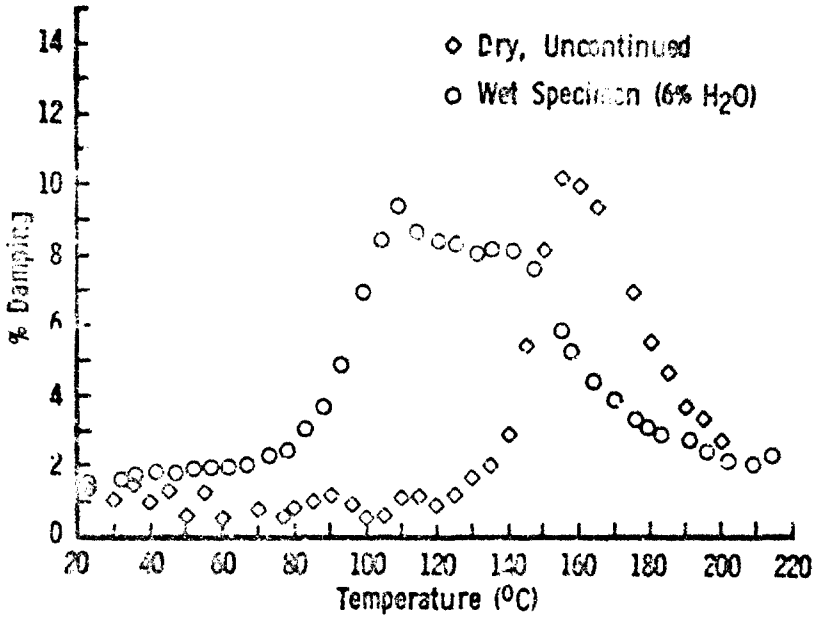

Figure 20b. Torsional pendulum data showing the effect of absorbed moisture on the % damping of Kevlar/epoxy HATS material as a function of temperature. Data indicate approximately 50°C depression of T_g.

Figure 20 shows that the onset of the glass transition occurs at a significantly lower temperature (about 50°C) in the wet material. This lowering of the glass transition temperature could seriously degrade the performance of the material at the upper use temperature, and suggests that design properties for the laminates should be determined at these temperatures after appropriate environmental conditioning. Saturation by moisture is unlikely to occur in use, but several percent moisture uptake could take place in high humidity environments, and even a few percent absorbed moisture could lead to significant depression of the glass transition temperature in highly cross-linked materials such as this epoxy.

The results of the mechanical testing shown in Table 1 also indicate that design properties should be measured after environmental conditioning. The short beam

Table 1. EFFECT OF MOISTURE ABSORPTION ON THE MECHANICAL PROPERTIES OF KEVLAR/EPOXY HATS LAMINATES

Properties	Wet	Dry
Tensile Strength (ksi)	36.7	52.9
Tensile Modulus (msi)	3.44	3.9
Shear Strength (psi)	1085	3360

shear strength is a matrix-dominated property, but a decrease in mean interlaminar shear strength of almost 70% at saturation suggests that an unacceptable loss of strength may occur at lower moisture contents and elevated temperatures. A drop of 30% in mean 0/90 tensile strength also suggests that the effect of absorbed moisture at elevated temperatures on tensile strength should be assessed.

Since the degradation of mechanical properties which may accompany environmental moisture absorption occurs only gradually, problems may not appear for several years. A shelter which passed a blast test as fabricated might not sustain the same blast loads when tested after several years of environmental exposure. The potential for environmental degradation due to moisture absorption is common to all orgranic matrix composites. To minimize deterioration, every effort should be made to assure that composite materials are properly cured to slow down moisture absorption. A properly applied protective surface coating can also inhibit moisture absorption. In addition, structures utilizing composites should be designed using allowables which take enviromental deterioration into account.

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APPENDIX. HPLC EXPERIMENTAL CONDITIONS

RPHPLC Instrument:	Perkin-Elmer - Series 4, ISS 100 Auto Injector, 3600 Data Station
Detector:	Perkin Elmer LC75 variable UV set at UV 214 nm
Column:	Waters Associates uBondapak c18
Mobile Phase:	Grddient 10% THF/35%CH3CN/55%H2O *45/30/25 15 min curve 3
Flow Rate:	2.0 ml/min
SEC Instrument:	Waters Associates model 244, data module, with auto inject
Detector:	Waters 440 UV set at 254 nm
Columns:	IBM SEC "A,C" 5u packing material
Mobile Phase:	THF
Flow Rate:	1.0 ml/min

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William W. Houghton and Margaret E. Roylance

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