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# Moisture Absorption of Coated Composite Materials

by Melissa A. Klusewitz, Christopher P. R. Hoppel,  
Steven H. McKnight, and James F. Newill

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## Moisture Absorption of Coated Composite Materials

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Steven H. McKnight, and James F. Newill  
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## Abstract

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Moisture diffusion measurements were made for coated and uncoated composite materials. Two substrates were used: T650/1914-4 (a graphite fiber reinforced thermoset epoxy) and AS4/Ultem (a graphite fiber reinforced thermoplastic polymer). The specimens were tested uncoated and coated with three polymer coatings—MIL-P-53030 Primer, Humiseal 2A53, and 2031 Siloxirane. The coatings generally absorbed more moisture than the specimens and increased the total moisture absorption for the coated parts. For the AS4/Ultem bars, anisotropic diffusion constants were measured, and diffusion occurs in the fiber direction three times faster than transverse to the fibers.

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## 1. Introduction

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This experimental study investigates the moisture absorption of composite material specimens with and without polymeric coatings. The results obtained here will be used to establish material diffusion constants and validate moisture diffusion models being developed for coated and uncoated composite materials [1].

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## 2. Materials

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Two types of composite materials were used for this study – T650 graphite fiber reinforced 1914-4 epoxy (a thermoset composite system) and AS4 graphite fiber reinforced Ultem (a thermoplastic composite system). These materials have been investigated in several previous studies on moisture absorption [2, 3]. The T650/1914-4 samples were cut from a thin, autoclave-cured four-ply panel. The panels were 1.5 in × 2.75 in × 0.02 in; the fibers were lying in the 1.5 in × 2.75 in plane so that moisture diffusion would occur primarily in the direction transverse to the carbon fibers. The AS4/Ultem samples were manufactured by two methods: bars cut from a thick compression-molded panel, and disks cut from a composite preform manufactured by compression molding. The bars were made with two orientations so that anisotropic diffusion coefficients could be measured. They were made in the fiber direction and transverse to the fibers. The disks were cut from a preform transverse to the fiber direction so that diffusion would be predominately in the fiber direction.

Three coating materials were investigated – Humiseal 2A53 coating, Siloxirane, and an Army CARC MIL-P-53030 primer. The coating materials were sprayed onto the test specimens. The coating thicknesses were measured after the moisture tests were concluded. The samples were sectioned using a Struers, Inc. accutom saw. The freshly cut edge was examined under a Leitz Wild stereomicroscope, model 420, at a nominal 320× power. The samples were mounted in the staging area of the microscope using either a putty type material, or they were held in place by two plastic blocks such that measurements were made perpendicular to the cut surface. The samples were photographed at intervals of 0.2 in using a Hitachi HVC20 digital camera in conjunction with an Oculus-TCI Demonstration Software Program, Version 3.01 by Conco, Inc. These photographs were downloaded to a PC with an Ultra Plus II video capturing board and stored on the hard drive as a tiff file. Thickness measurements were then made using the software, Scion Image, Beta 4.02 by

Scion Corp. To correctly analyze these images, the scaling was set 420 pixels to equal 1 mm. The distance from the inner edge to the outer edge of the coating was measured using a cursor drop function (Figures 1 and 2). In regions where the saw pulled composite material into the coating, no measurements were taken (Figure 3). A full description of all of the test specimens is given in Table 1.

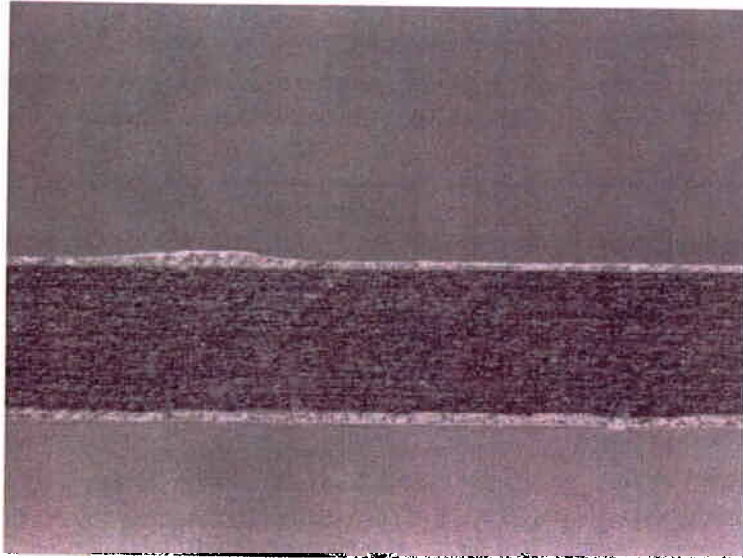


Figure 1. Photograph of the T650/1914-4 plate showing the CARC MIL-P-53030 coating on both sides of the thin four-ply plate.

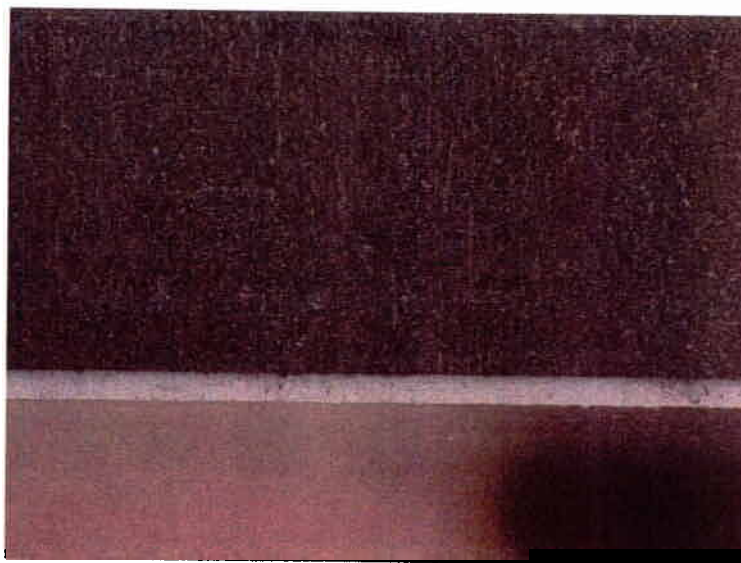


Figure 2. Photograph of the AS4/Ultem bar with the Siloxirane coating on one side.

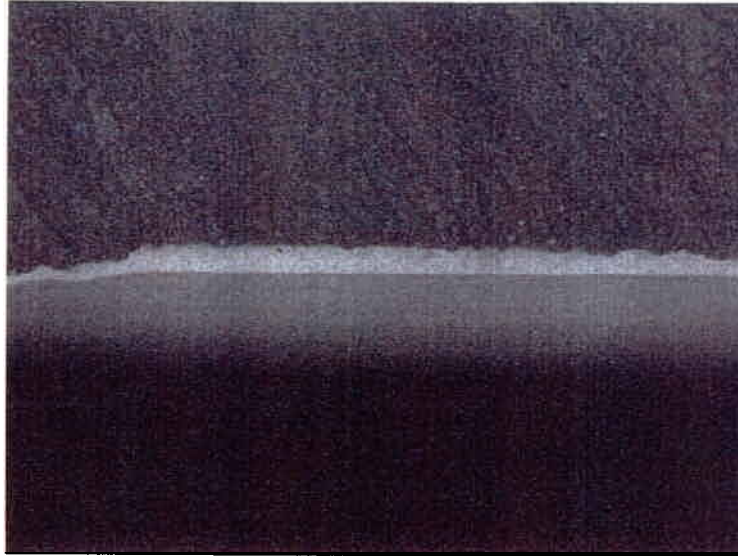


Figure 3. AS4/Ultem bar with the Siloxirane coating on one side showing a region where the cutting process pulled the composite material over the coating region. This occurred after the moisture test, when specimens were being prepared for coating measurements; no measurements were made in this region.

Table 1. Specimens used in the present study.

Substrate	Diffusion Direction	Coating	Coating Thickness (in)	Standard Deviation
T650/1914-4 Plates	Transverse to fibers	Primer	0.00098	0.0007
T650/1914-4 Plates	Transverse to fibers	None	0	–
AS4/Ultem Disks	Fiber Direction	None	0	–
AS4/Ultem Disks	Fiber Direction	Primer	0.00125	0.0004
AS4/Ultem Bars	Fiber Direction	None	0	–
AS4/Ultem Bars	Fiber Direction	Humiseal	0.00104	0.0003
AS4/Ultem Bars	Fiber Direction	Siloxirane	0.00232	0.0007
AS4/Ultem Bars	Transverse to fibers	None	0	–
AS4/Ultem Bars	Transverse to fibers	Humiseal	0.00104	0.0003
AS4/Ultem Bars	Transverse to fibers	Siloxirane	0.00232	0.0007

### 3. Experimental Technique

All of the specimens were thoroughly dried in a vacuum oven prior to testing. For testing, all samples were placed in a constant temperature-humidity chamber. The samples were evenly distributed in a steel mesh tray to ensure

uniform airflow around all specimens. Two test conditions were used: (1) 35 °C and 98% relative humidity (RH) and (2) 50 °C and 98% RH. Prior to testing, the specimens were weighed daily for the first 10 days of the test and every other day until the conclusion of the test.

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## 4. Results

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The moisture weight gain vs. time for the test specimens are shown graphically in Figures 4–8. Diffusion coefficients were calculated for each specimen from equation 1, as specified by ASTM Standard D 5229/D5229M-92 [4],

$$D = \pi \left( \frac{h}{4M_m} \right)^2 \left( \frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}} \right)^2, \quad (1)$$

where  $h$  is the specimen thickness,  $M_m$  is the moisture saturation level, and the quantity in the second set of parentheses is the slope of the initial linear region on a plot of moisture weight gain vs. the square root of time. This initial linear region is normally taken to be 60% of the time required for saturation. Diffusion coefficients were calculated for all of the samples, even though they are not really appropriate for the coated samples (higher order analysis is necessary for the two-part systems and will be described in a subsequent study [5]). For the present study, these diffusion constants were calculated simply for comparison purposes. The reduced experimental data is listed in Table 2.

From the results it is important to note the anisotropic nature of diffusion in the composite materials. For the uncoated bar specimens, the diffusion rates are on the order of three times higher in the fiber direction as opposed to transverse to the fibers. This agrees with results found in the general literature [6]. This means that moisture will absorb much more quickly in composite structures with exposed fiber ends (normal to the surface) than in structures with fibers parallel to the surfaces.

The results reported here should be viewed as laboratory data. The coating thickness is relatively thick as compared to the small specimens. The experimental results will be used to validate theoretical models in a separate study. However, it is interesting to note that the coatings generally increased the maximum moisture contents and the diffusion coefficients as compared to the uncoated substrates.

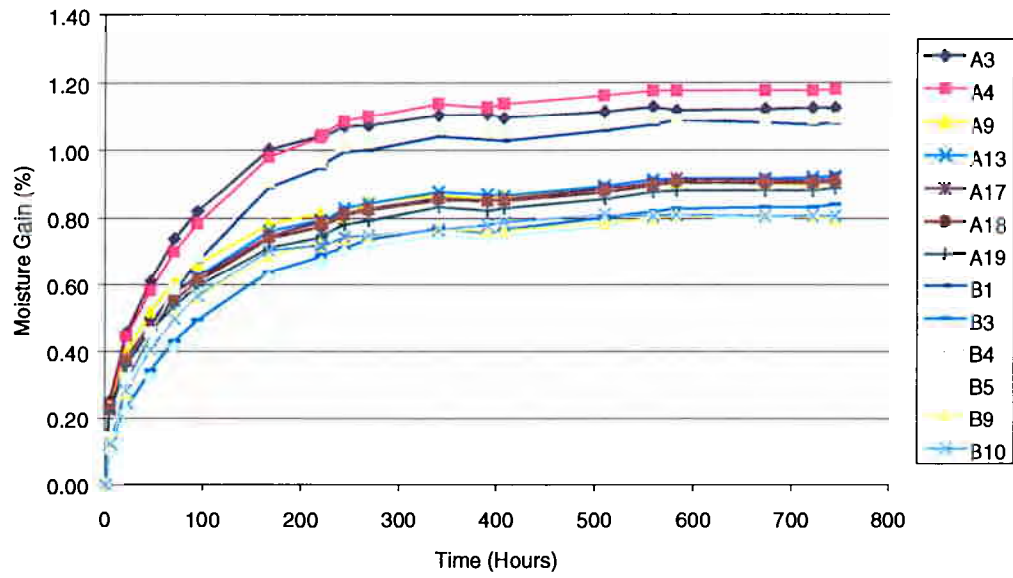


Figure 4. Moisture absorption in T650/1914-4 plates at 35 °C and 98% RH. Group A had the primer coating, and group B had no coating.

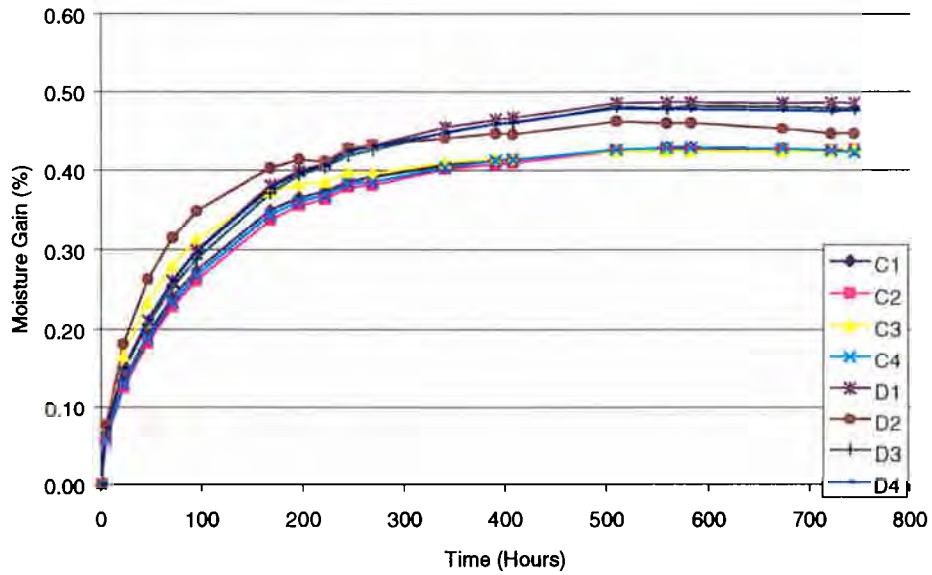


Figure 5. Moisture absorption in AS4/Ultem disks at 35 °C and 98% RH. Group C had no coating, and group D had the primer coating.

Moisture Absorption After 18 Days- Plates at 50 °C

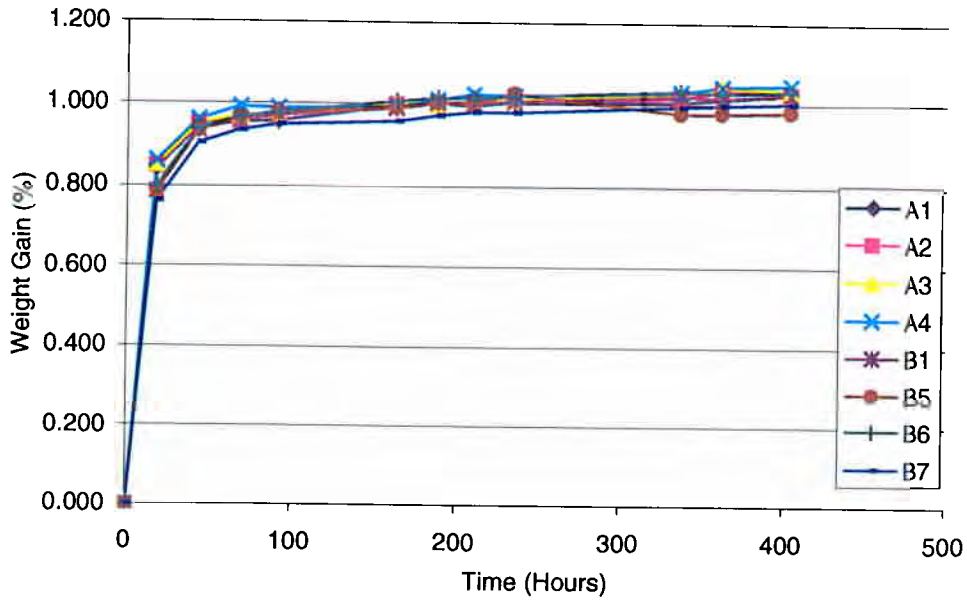


Figure 6. Moisture absorption in T650/1914-4 plates at 50 °C and 98% RH. Group A had the primer coating, and group B had no coating.

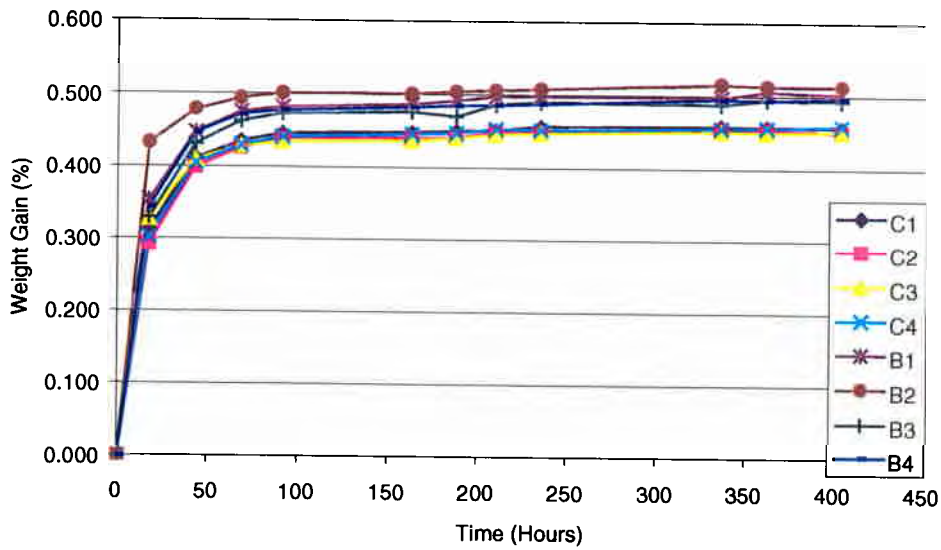


Figure 7. Moisture absorption in AS4/Ultem disks at 50 °C and 98% RH. Group C had no coating, and group B had the primer coating.

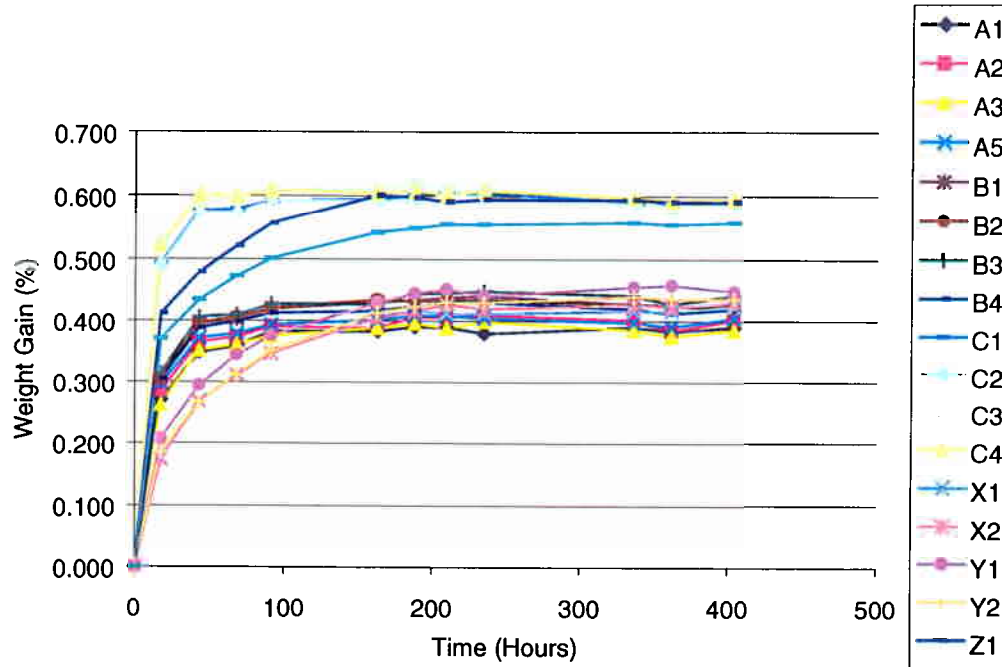


Figure 8. Moisture absorption in AS4/Ultem compression molded bars at 50 °C and 98% RH. None of the specimens in groups A, B, and C had diffusion in the fiber direction. Group A had no coating, group B had Humiseal 2A53 coating, and group C had Siloxirane coating. Groups X, Y, and Z experienced diffusion transverse to the fiber direction. Group X had no coating, group Y had Humiseal 2A53 coating, and group Z had Siloxirane coating.

## 5. Conclusions

Moisture diffusion measurements have been made for coated and uncoated composite materials. Two substrates were used: T650/1914-4 (a graphite fiber reinforced thermoset epoxy) and AS4/Ultem (a graphite fiber reinforced thermoplastic polymer). The specimens were tested uncoated and coated with three polymer coatings (MIL-P-53030 Primer, Humiseal 2A53 and 2031 Siloxirane). The coatings generally increased the total moisture absorption for the specimens. For the AS4/Ultem bars, anisotropic diffusion constants were measured and diffusion occurs in the fiber direction three times faster than transverse to the fibers.

Table 2. Testing results.

Substrate	Coating	Test Temperature (°F / °C)	No. of Specimens	Specimen Thickness (in)	Standard Deviation	Maximum Moisture Content (%)	Standard Deviation	Effective Diffusion Coefficient (in <sup>2</sup> /hr)	Standard Deviation
T650/1914-4 Plates Transverse to Fibers	Primer	95 °F 35 °C	7	2.786E-02	8.522E-04	0.979	0.119	1.682E-06	3.097E-07
T650/1914-4 Plates Transverse to Fibers	None	95 °F 35 °C	6	2.300E-02	1.304E-03	0.906	0.141	3.764E-07	1.269E-07
AS4/Ultem Disks Fiber Direction	None	95 °F 35 °C	4	1.005E-01	1.826E-03	0.425	0.002	7.822E-06	2.341E-06
AS4/Ultem Disks Fiber Direction	Primer	95 °F 35 °C	4	1.083E-01	2.887E-04	0.472	0.017	9.972E-06	1.895E-06
T650/1914-4 Plates Transverse to Fibers	Primer	122 °F 50 °C	4	2.650E-02	1.000E-03	1.072	0.004	4.984E-06	4.731E-07
T650/1914-4 Plates Transverse to Fibers	None	122 °F 50 °C	4	2.150E-02	5.774E-04	1.042	0.022	2.948E-06	2.914E-07
AS4/Ultem Disks Fiber Direction	Primer	122 °F 50 °C	4	1.003E-01	1.708E-03	0.470	0.005	4.860E-05	3.522E-06
AS4/Ultem Disks Fiber Direction	None	122 °F 50 °C	4	1.075E-01	2.380E-03	0.516	0.011	6.121E-05	1.550E-05
AS4/Ultem Bars Fiber Direction	None	122 °F 50 °C	4	9.725E-02	4.193E-03	0.403	0.005	5.074E-05	2.908E-06
AS4/Ultem Bars Fiber Direction	Humiseal	122 °F 50 °C	4	9.875E-02	9.574E-04	0.435	0.004	5.503E-05	2.020E-06
AS4/Ultem Bars Fiber Direction	Siloxirane	122 °F 50 °C	4	1.043E-01	2.630E-03	0.582	0.006	9.280E-05	9.061E-06
AS4/Ultem Bars Transverse to Fibers	None	122 °F 50 °C	2	9.650E-02	2.121E-03	0.436	0.007	1.686E-05	2.565E-07
AS4/Ultem Bars Transverse to Fibers	Humiseal	122 °F 50 °C	2	1.035E-01	6.364E-03	0.457	0.007	2.195E-05	8.319E-08
AS4/Ultem Bars Transverse to Fibers	Siloxirane	122 °F 50 °C	2	1.070E-01	2.828E-03	0.566	0.021	6.138E-05	1.087E-06

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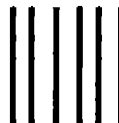
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