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RAIN EROSION MECHANISMS OF FLUOROELASTOMER AND POLYURETHANE COATINGS ON SELECTED COMPOSITE SUBSTRATES

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AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO 45433

NOTICE

This technical report has been reviewed and is approved for publication.

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This report documents a test program which was conducted to evaluate the rain erosion resistance of selected coated composite materials at 30° and 90° impact angles in a 1 inch/hour rainfall at 500 mph. Tests included: weight loss, percent coating removal, and SEM examinations.

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PREFACE

The effort documented in this report was conducted as a joint enterprise by the following persons: Charles J. Hurley, University of Dayton Research Institute, under Contract Nos. F33615-81-C-5030 and F33615-82-C-5039; Dr. Joseph Zahavi, Visiting Scientist, under Contract No. F33615-79-C-5129 with Universal Technology Corporation; and George F. Schmitt, Air Force Wright Aeronautical Laboratories, Materials Laboratory, under Project No. 2422, Task No. 242201. The work described herein was conducted during the period from July 1980 to February 1983.

The authors wish to acknowledge the contributions of Timothy Courney and James Gannon of the University of Dayton Research Institute for performing the rain exposure tests. Sincere appreciation is extended to Robert Brodecki of Systems Research Laboratories for the scanning electron microscopy operations and photography.

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SECTION I
INTRODUCTION

1. BACKGROUND

For many years, rain droplet impingement erosion of polymeric coatings and composite materials used in exterior aircraft structures have been a problem.[1,2,3,4] Aircraft and missile systems operating in adverse weather environments at moderate to high velocities were subject to progressive surface erosion or structural damage. Current fluoroelastomer and polyurethane coatings for leading edges, radomes, antenna covers, and helicopter rotor blades have been relatively successful in protecting reinforced plastic components from erosion by rain. Developments in improved rain erosion resistance elastomeric coatings were characterized or evaluated by rotating arm test apparatus and actual flight tests. These tests provided a relative ranking of materials performance.

2. OBJECTIVE

The object of this program was to investigate the erosion damage mechanisms involved with polymeric coatings on different types of reinforced plastic composites. The polymeric coated composites were to be evaluated for visible erosion damage, weight loss after rainfield exposure and in-depth analysis of erosion mechanisms utilizing electron microscopy techniques. The performance of fluoroelastomer and polyurethane coatings on similar reinforced plastic composites was to be assessed. One military specification fluoroelastomer and polyurethane coating was to be evaluated on glass epoxy, graphite epoxy, and quartz polyimide substrates.

SECTION II
EXPERIMENTAL PROCEDURE

1. MATERIALS

The materials for this investigation were supplied by the Air Force Wright Aeronautical Laboratories. Materials were furnished in flat sheet form and processed to be representative of the coated substrates used in aircraft components. The MIL-C-83445A white polyurethane coating was spray-applied 14 mils thick to 100 mil glass epoxy, 95 mil quartz polyimide, and 85 mil graphite epoxy composite sheets. The AF-C-VBW-15-15 white fluoroelastomer coating was spray-applied 13 mils thick to similar composite sheets.

2. TEST SPECIMEN FABRICATION

All test specimens were fabricated from the parent material sheets. Great care was taken to insure that the coated surfaces were not damaged or adversely affected by fabrication. The test specimen configurations used for rain erosion evaluations are shown in Figures 1 and 2.

3. TEST SPECIMEN PRETEST EXAMINATION

The as-received coated composite test specimens were numbered and logged according to the standard procedure required for the rain erosion test apparatus. All specimens were visually examined for defects or damage. Test specimens were dried overnight in a forced air oven at 125°F, cooled to ambient temperatures in a dessicator and then weighed. Pretest scanning electron micrographs were prepared on selected specimens from each coated substrate group.

4. MACH 1.2 RAIN EROSION TEST APPARATUS

The rotating arm apparatus consists of an eight-foot diameter double arm blade. It is designed to produce high tip

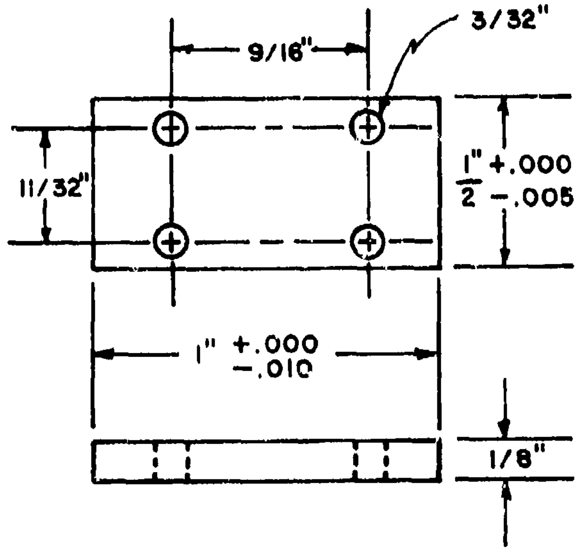


Figure 1. Rain Erosion Test Specimen for 90° Impact Angle.

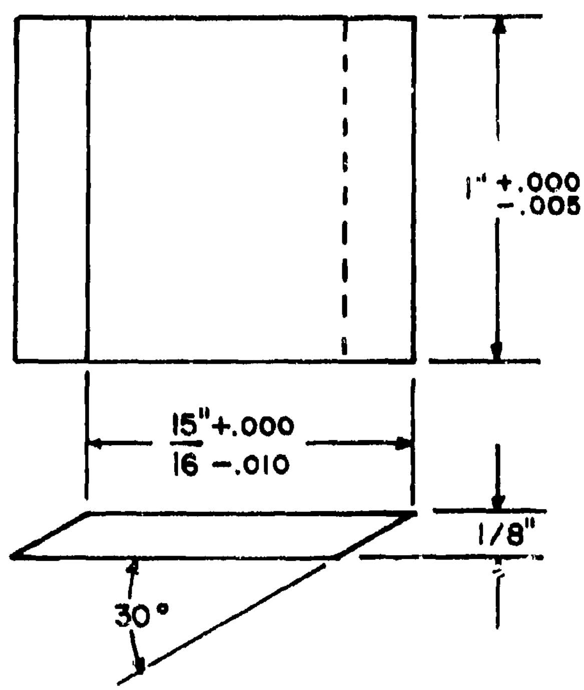


Figure 2. Rain Erosion Test Specimen for 30° Impact Angle.

velocities with negative lift and a low drag coefficient. Mated test specimens are mounted at the leading edge tip sections of the double rotating arm. The test specimens can be subjected to variable speeds of 0 to 900 mph. The double arm is mounted horizontally on a vertical drive shaft as shown in Figure 3. Simulated rainfall is produced by four curved manifold quadrants. Each manifold quadrant has 24 equally-spaced capillaries. Raindrop size and drop rate are controlled by the capillary orifice diameter and the head pressure of the water supply. The manifold quadrants are mounted above the tips of the double rotating arm. Raindrops from the simulation apparatus impact the test specimens throughout their entire annular path. Rain droplets are 2.0 mm diameter and generated at the rate of one inch/hour of simulated rainfall. This apparatus is fully described in AFML-TR-70-240.[5]

For the purposes of this study, matched pairs of specimens were inserted into the specimen holders at a 30° or 90° angle of incidence to the rain droplet impact. All rain erosion testing was conducted at 500 mph. Duration of the rainfield exposure tests was variable depending upon the observed surface conditions after 5 and 10 minute intervals of rainfield exposure.

5. POST-TEST OBSERVATION AND EXAMINATION PROCEDURES

a. Visual Observation

All specimens were examined visually after rainfield exposure with a lighted magnifier and the surface condition was recorded. Recorded comments included scratches, pitting, percentage of coating removal, coating adhesion loss, and composite damage.

b. Target Mass Loss

All test specimens were forced air oven dried at 125°F overnight and cooled to ambient temperature in a dessicator after rainfield exposure. Target mass loss was recorded after each exposure interval. Mass loss was recorded to within 0.001 g.

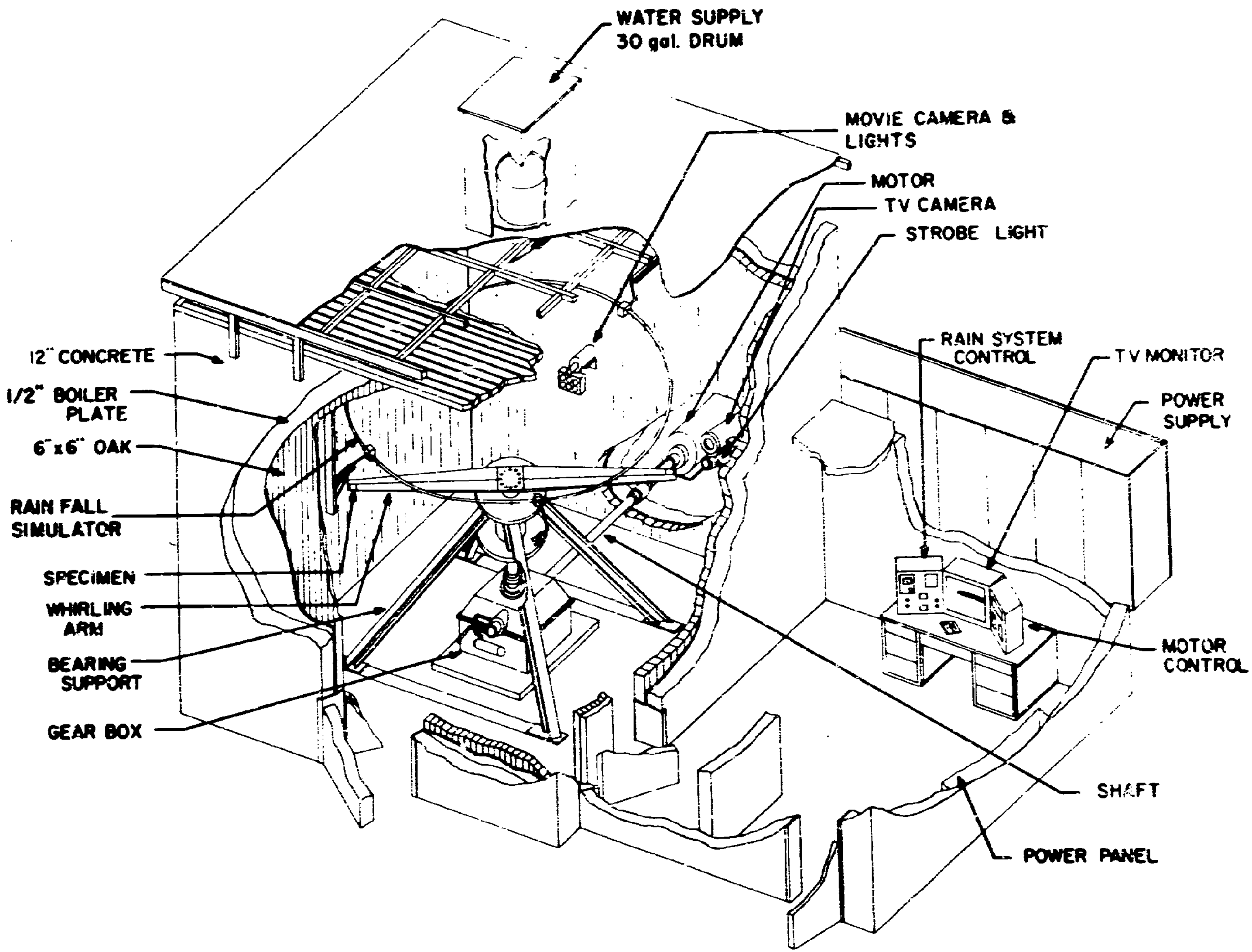


Figure 3. Mach 1.2 Rain Erosion Test Apparatus.

c. Scanning Electron Microscopy Examination

The ETEC Autoscan High Resolution Scanning Electron Microscope is a second generation instrument. It incorporates such features as electron optics, specimen chamber, electron detection and display systems. This instrument bridges the specimen gap between light and transmission electron microscopes. Specimens are vapor shadowed with a heavy metal or carbon to provide contrast. Resolution is of the order of 200Å and useful magnification up to 50,000X.

SECTION III
TEST RESULTS

1. RAIN EROSION TEST RESULTS - VISUAL EXAMINATION

a. Fluoroelastomer Coating - Glass Epoxy Substrate - 90° Impact Angle

Visual examination, under a lighted magnifier, of the fluoroelastomer coated glass epoxy substrates exhibited initiation of pitting on the exposed edges of coated specimens during the first five-minute interval of rainfield exposure. At the ten-minute interval of rainfield exposure, surface pitting and several areas of very localized adhesion loss were observed at the exposed edges of the coated specimens. Continued rainfield exposure resulted in increased pitting with 10% coating removal and increasing adhesion loss at the 15-minute interval. Thirty minutes of rainfield exposure resulted in 50% coating removal with additional adhesion loss and erosion of the glass epoxy substrate. Percent coating loss as a function of rainfield exposure time is shown in Figure 4.

b. Fluoroelastomer Coating - Graphite Epoxy Substrate - 90° Impact Angle

The fluoroelastomer coated graphite epoxy specimens exhibited no visible surface damage after five minutes of rainfield exposure. At the 10-minute interval of rainfield exposure, however, coating losses averaged 5% of the coated surface area and up to 50% adhesion loss of the fluoroelastomer from the graphite epoxy substrate. Percent coating loss versus rainfield exposure time is shown in Figure 4.

c. Fluoroelastomer Coating - Quartz Polyimide Substrate - 90° Impact Angle

Five minutes of rainfield exposure of the fluoroelastomer coated quartz polyimide substrate resulted in the initiation of pitting and 1% coating removal. Ten-minute exposures exhibited increased pitting, approximately 2% coating removal,

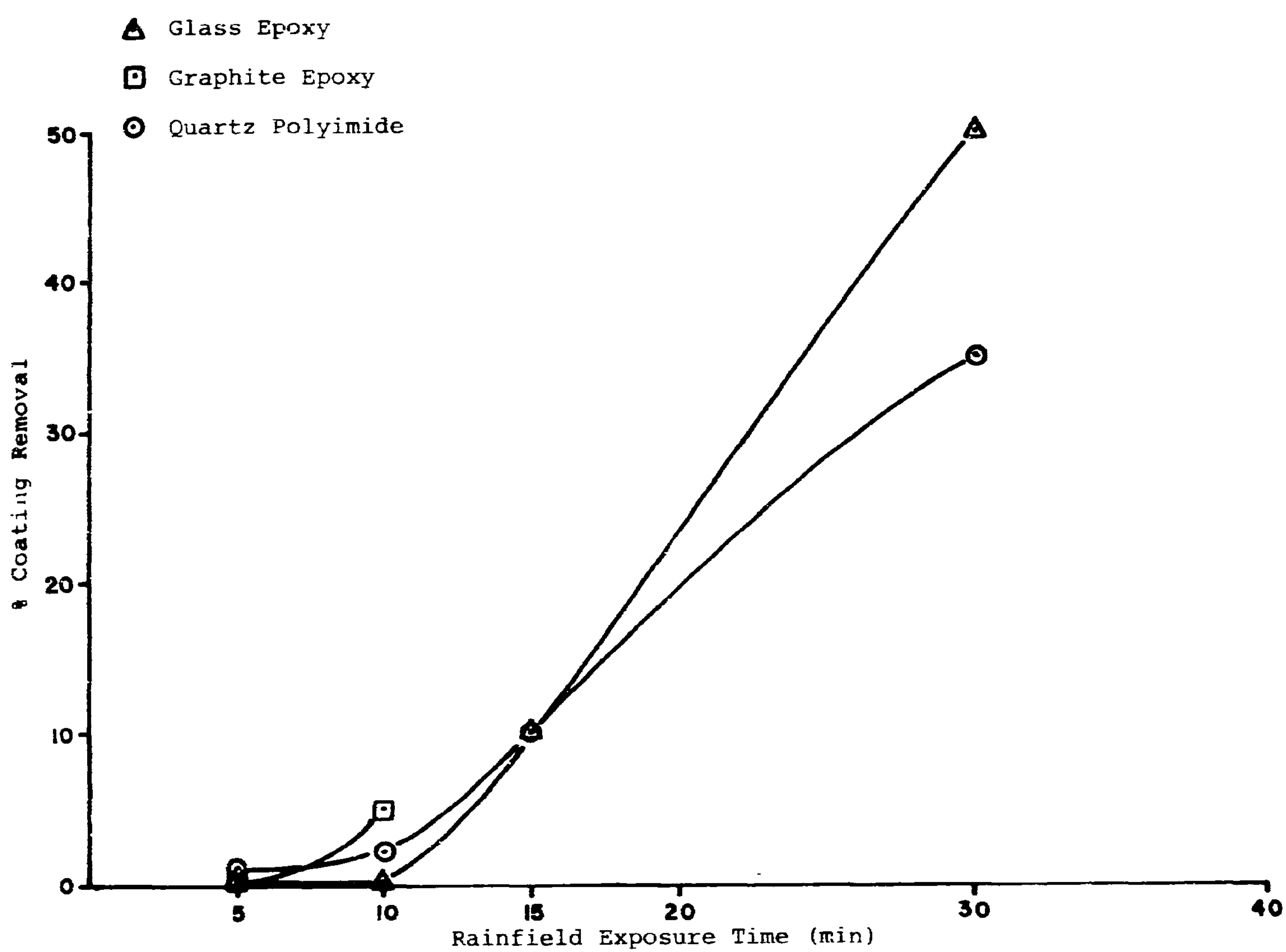


Figure 4. Fluoroelastomer Coating - % Coating Removal vs. Exposure Time, 90°.

and partial adhesion loss of the fluoroelastomer coating to the substrate. Continued pitting, 10% coating loss and partial adhesion loss occurred at the 15-minute interval of rainfield exposure. Thirty-minute exposures exhibited 35% coating loss, partial coating adhesion loss, and severe erosion of the exposed quartz polyimide substrate. Results are shown in Figure 4.

d. Fluoroelastomer Coating - Glass Epoxy Substrate - 30° Impact Angle

Examination of the fluoroelastomer coated glass epoxy substrates exposed to a five-minute interval of rainfield exposure resulted in no visible damage. Thirty-minute rainfield exposures visually indicated pitting initiation and no measurable coating loss. Increased pitting, partial coating adhesion loss and composite substrate damage occurred after 165 minutes of rainfield exposure at 500 mph. Results are shown in Figure 5.

e. Fluoroelastomer Coating - Graphite Epoxy Substrate - 30° Impact Angle

Five-minute rainfield exposure of the fluoroelastomer coated graphite epoxy substrate resulted in no visible damage. Thirty-minute exposure resulted in partial coating adhesion loss and composite damage. Exposure to 180 minutes of rainfield exposure showed partial adhesion loss and increased composite substrate damage. The results are shown in Figure 5.

f. Fluoroelastomer Coating - Quartz Polyimide Substrate - 30° Impact Angle

Visual examination of the fluoroelastomer coated quartz polyimide substrate after five minutes of rainfield exposure indicated no visible damage. Exposure to thirty minutes of the rainfield environment also resulted in no visible damage. Further exposure to 180 minutes of the simulated rainfall produced pitting, partial coating edge delamination, and composite damage. The results are shown in Figure 5.

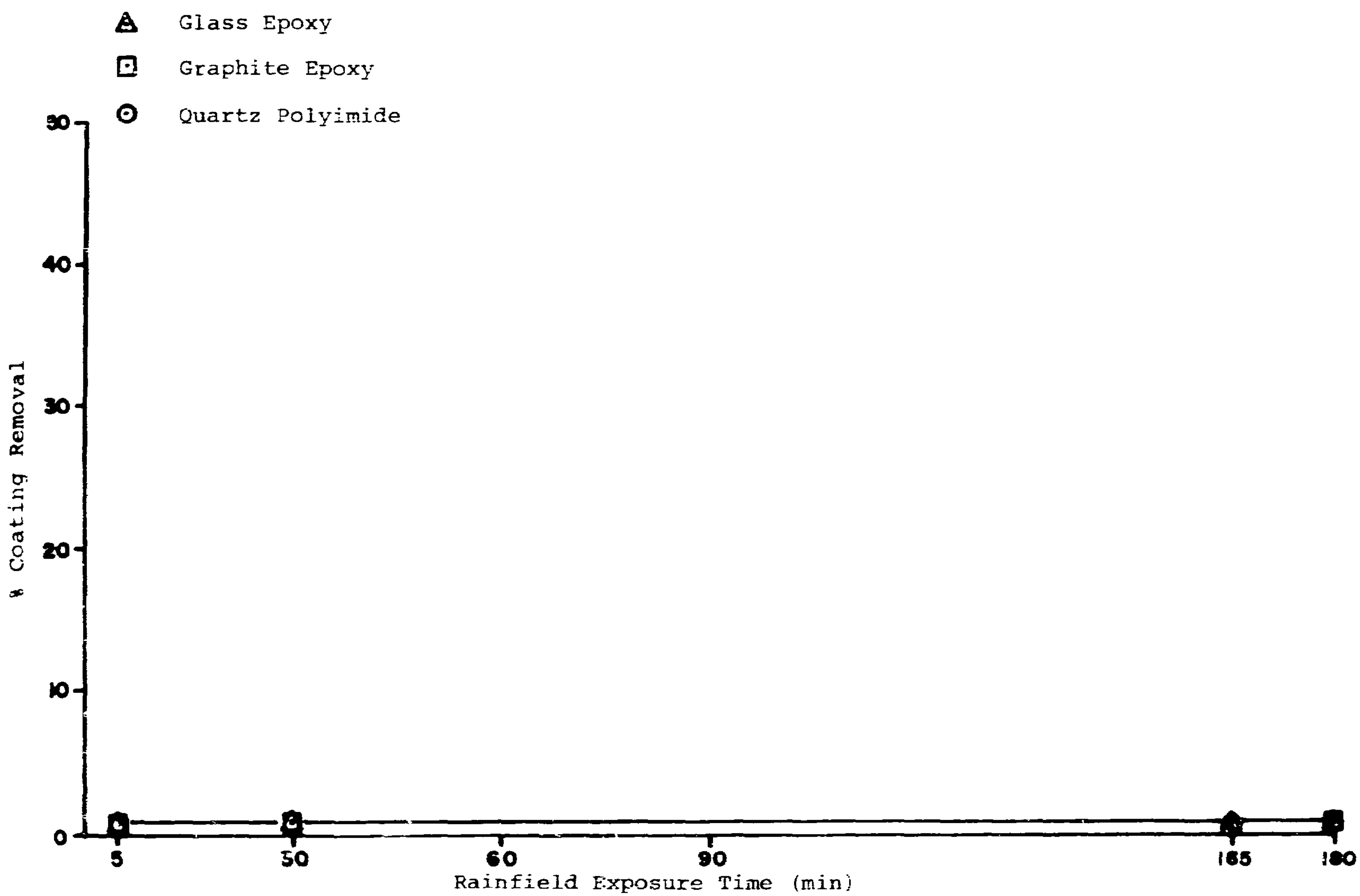


Figure 5. Fluoroelastomer Coating - % Coating Removal vs. Exposure Time, 30°.

g. Polyurethane Coating - Glass Epoxy Substrate - 90° Impact Angle

Visual examination, under a lighted magnifier, of the polyurethane coated glass epoxy substrate revealed no visible damage after five minutes of rainfield exposure. At the 10-minute interval of rainfield exposure, 2% of the coating was removed with partial coating adhesion loss at the edges of the specimens. Continued rainfield exposure resulted in slight pitting, no increase in coating removal, and increased adhesion loss at the coating edges at the 20-minute exposure interval. Forty minutes of rainfield exposure exhibited 10% coating removal and increased coating adhesion loss. Results are shown in Figure 6.

h. Polyurethane Coating - Graphite Epoxy Substrate - 90° Impact Angle

The polyurethane coated graphite epoxy specimens exhibited no visible surface damage after five minutes of rainfield exposure. At the 10-minute interval, coating removal losses averaged 10% of the coated surface area and up to 60% coating adhesion loss of the polyurethane from the graphite epoxy substrates. Percent coating loss versus rainfield exposure time is shown in Figure 6.

i. Polyurethane Coating - Quartz Polyimide Substrate - 90° Impact Angle

Five minutes of rainfield exposure of the polyurethane coated quartz polyimide substrates resulted in partial coating adhesion loss at the specimen edges. Ten-minute exposures exhibited increased coating adhesion loss of the polyurethane from the quartz polyimide specimens and 2% coating removal. Further rainfield exposures totaling 20 minutes produced 10% coating loss and increasing coating adhesion losses. Results are shown in Figure 6.

j. Polyurethane Coating - Glass Epoxy Substrate - 30° Impact Angle

Five-minute and 30-minute rainfield exposures of the polyurethane coated glass epoxy substrates revealed no visible

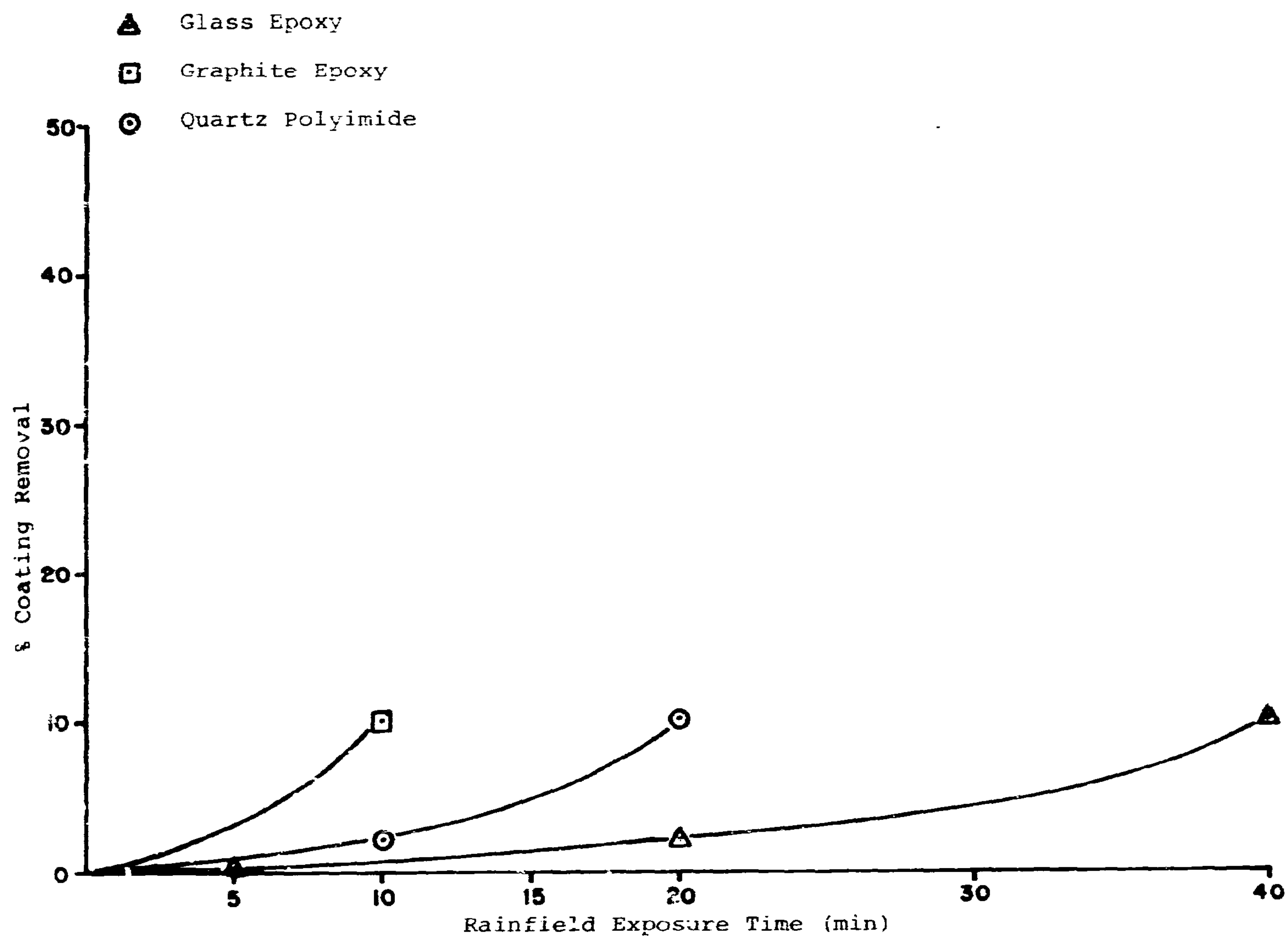


Figure 6. Polyurethane Coating - % Coating Removal vs. Exposure Time, 90°.

damage. Exposures to 180 minutes of the rainfield environment resulted in 3% coating removal and partial coating delamination from the glass epoxy substrates. Percent coating removal versus rainfield exposure time is shown in Figure 7.

k. Polyurethane Coating - Graphite Epoxy Substrate - 30° Impact Angle

Exposures of five minutes and 30 minutes to rainfield exposure conditions resulted in no visible damage of the polyurethane coated graphite epoxy specimens. One hundred and eighty minutes of exposure produced partial coating adhesion losses and substantial damage to the graphite epoxy composites. Results are shown in Figure 7.

l. Polyurethane Coating - Quartz Polyimide Substrate - 30° Impact Angle

No visible damage was observed after five minutes and 30 minutes exposure to rainfield conditions for the polyurethane coated quartz polyimide specimens. One hundred and eighty minutes of exposure produced slight coating adhesion loss and very slight damage to the quartz polyimide substrates. Results are shown in Figure 7.

2. RAIN EROSION TEST RESULTS - TARGET MASS LOSS

a. Fluoroelastomer Coating - Glass Epoxy Substrate - 90° Impact Angle

Fluoroelastomer coated glass epoxy substrates exposed at a 90° impact angle resulted in an average of 0.0035 g weight loss after five minutes of rainfield exposure. Ten minutes of rainfield exposure produced an average of 0.0060 g of specimen weight loss. Fifteen minutes produced a magnitude increase in weight loss of 0.0188 g. Rainfield exposures of 30 minutes duration exhibited another magnitude increase in mass loss of 0.1313 g.

b. Fluoroelastomer Coating - Graphite Epoxy Substrate - 90° Impact Angle

Rainfield exposure of five minute duration with fluoroelastomer coated graphite epoxy specimens resulted in an average

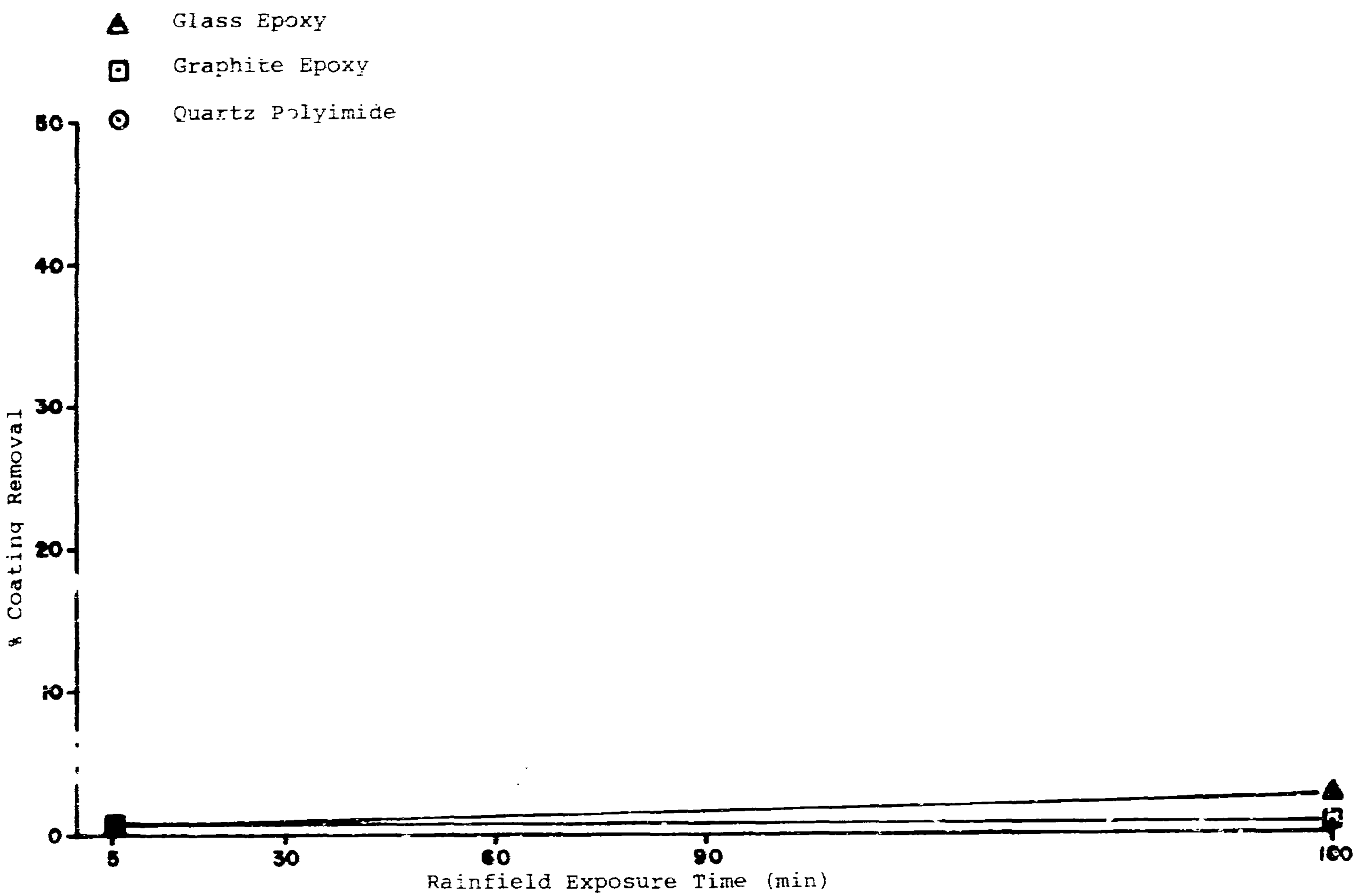


Figure 7. Polyurethane Coating - % Coating Removal vs. Exposure Time, 30°.

mass loss of 0.0076 g. An order of magnitude increase in target mass loss occurred at 10 minutes of rainfield exposure. Mass loss at this interval resulted in a 0.039 g weight loss.

c. Fluoroelastomer Coating - Quartz Polyimide Substrate - 90° Impact Angle

Five-minute rainfield exposures of fluoroelastomer coated quartz polyimide specimens resulted in an average target mass loss of 0.0078 g. An order of magnitude mass loss occurred at the 10-minute interval of rainfield exposure. This resulted in an average weight loss of 0.0117 g. Fifteen minutes of exposure exhibited a mass loss average of 0.0296 g. Rainfield exposures of 30 minutes resulted in another order of magnitude increase in target mass loss. These specimens averaged a 0.2114 g weight loss.

Figure 8 shows a comparison of target mass loss for the fluoroelastomer/composite combinations exposed at a 90° impact angle and a velocity of 500 miles per hour.

d. Fluoroelastomer Coating - Glass Epoxy Substrate - 30° Impact Angle

Fluoroelastomer coated glass epoxy substrates exposed to a 30° impact angle resulted in an average weight loss of 0.0030 g after five minutes of rainfield exposure. Thirty minutes of rainfield exposure produced an average target mass loss of 0.0130 g. This was an order of magnitude increase in mass loss. A minimal weight loss increase occurred after 165 minutes of rainfield exposure. Target mass loss totaled 0.0199 g.

e. Fluoroelastomer Coating - Graphite Epoxy Substrate - 30° Impact Angle

Five-minute rainfield exposure of the fluoroelastomer coated graphite epoxy substrates resulted in an average mass loss of 0.0097 g. An order of magnitude change in target mass loss occurred at the 30-minute exposure interval. The average weight loss was 0.0616 g. A minimal increase in mass loss occurred after 180 minutes of rainfield exposure. Average target mass loss totaled 0.0960 g.

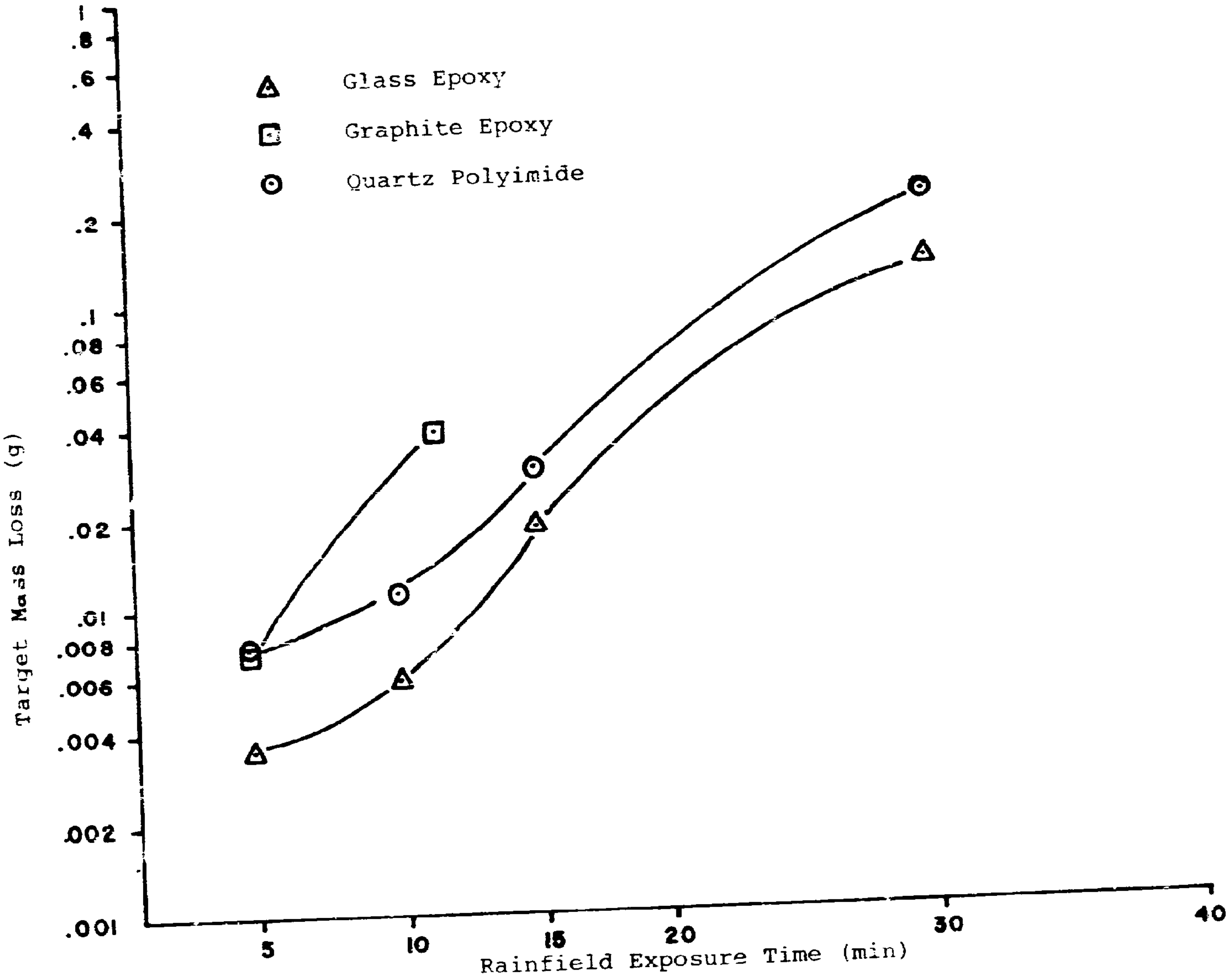


Figure 8. Target Mass Loss of Fluoroelastomer Coated Composites Exposed at a 90° Impact Angle, 500 mph, 1 inch/hr Rainfall.

f. <u>Fluoroelastomer Coating - Quartz Polyimide Substrate - 30° Impact Angle</u>

Five-minute rainfield exposure of the fluoroelastomer coated quartz polyimide substrates resulted in an average weight loss of 0.0055 g. An average mass loss of 0.0218 g occurred after 30 minutes of rainfield exposure. One hundred and eighty minutes of exposure produced an average total mass loss of 0.0454 g.

Figure 9 shows a comparison of target mass loss for the fluoroelastomer/composite combinations at a 30° impact angle and a velocity of 500 miles per hour.

g. <u>Polyurethane Coating - Glass Epoxy Substrate - 90° Impact Angle</u>

Polyurethane coated glass epoxy substrates exposed to a 90° impact angle exhibited an average target mass loss of 0.00265 g after five minutes of rainfield exposure. Ten-minute rainfield exposure produced an average mass loss of 0.0077 g. A mass loss of 0.0075 g occurred at the 20-minute interval. A total average target mass loss of 0.0172 g was measured after 40 minutes of rainfield exposure.

h. <u>Polyurethane Coating - Graphite Epoxy Substrate - 90° Impact Angle</u>

Rainfield exposure of five minute duration with polyurethane coated graphite epoxy specimens resulted in an average mass loss of 0.0080 g. Exposure to 10 minutes of rainfield conditions produced an order of magnitude increase in target mass loss for these materials. Total average mass loss at 10 minutes was 0.0301 g.

i. <u>Polyurethane Coating - Quartz Polyimide Substrate - 90° Impact Angle</u>

Five-minute rainfield exposure of the polyurethane coated quartz polyimide substrates resulted in an average mass loss of 0.0052 g. An average mass loss of 0.0088 g occurred after 10 minutes of exposure for this coating/substrate combination. Twenty-minute rainfield exposure produced an average total target mass loss of 0.0188 g.

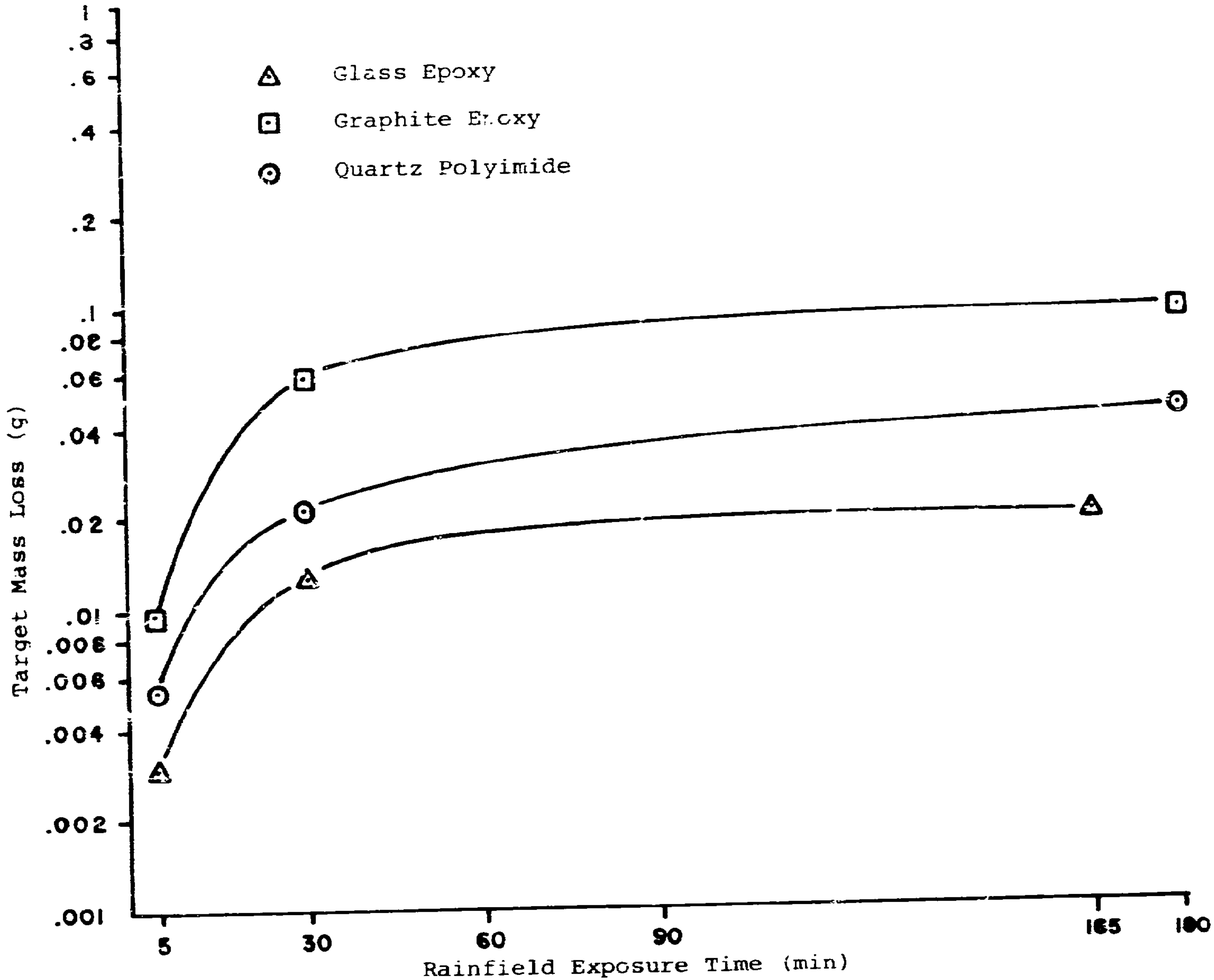


Figure 9. Target Mass Loss of Fluoroelastomer Coated Composites Exposed at a 30° Impact Angle, 500 mph, 1 inch/hr Rainfall.

Figure 10 shows a comparison of target mass loss for the polyurethane/composite combinations exposed at a 90° impact angle and a velocity of 500 miles per hour.

j. Polyurethane Coating - Glass Epoxy Substrate - 30° Impact Angle

Polyurethane coated glass epoxy substrate exposed at a 30° impact angle resulted in an average target mass loss of 0.0070 g after five minutes of rainfield exposure. Thirty minutes of rainfield exposure produced an order of magnitude increase in mass loss averaging 0.0170 g. A minimal weight loss increase occurred after 180 minutes of rainfield exposure. Target mass losses averaged 0.0285 g.

k. Polyurethane Coating - Graphite Epoxy Substrate - 30° Impact Angle

Target mass loss for the polyurethane coated graphite epoxy substrates exhibited an order of magnitude increase for each exposure interval. At five minutes of rainfield exposure, an average mass loss of 0.0077 g was recorded. Thirty minutes exposure resulted in an average target mass loss of 0.0183 g. Weight loss averaging 0.1183 g was recorded after the 180-minute rainfield exposure.

l. Polyurethane Coating - Quartz Polyimide Substrate - 30° Impact Angle

Five-minute rainfield exposure of the polyurethane coated quartz polyimide substrates resulted in an average mass loss of 0.0074 g. An average mass loss of 0.0244 g occurred after 30 minutes of rainfield exposure. One hundred and eighty minutes of exposure produced an average total mass loss of 0.0718 g.

Figure 11 compares the target mass loss of the polyurethane/composite combinations at a 30° impact angle and a velocity of 500 miles per hour.

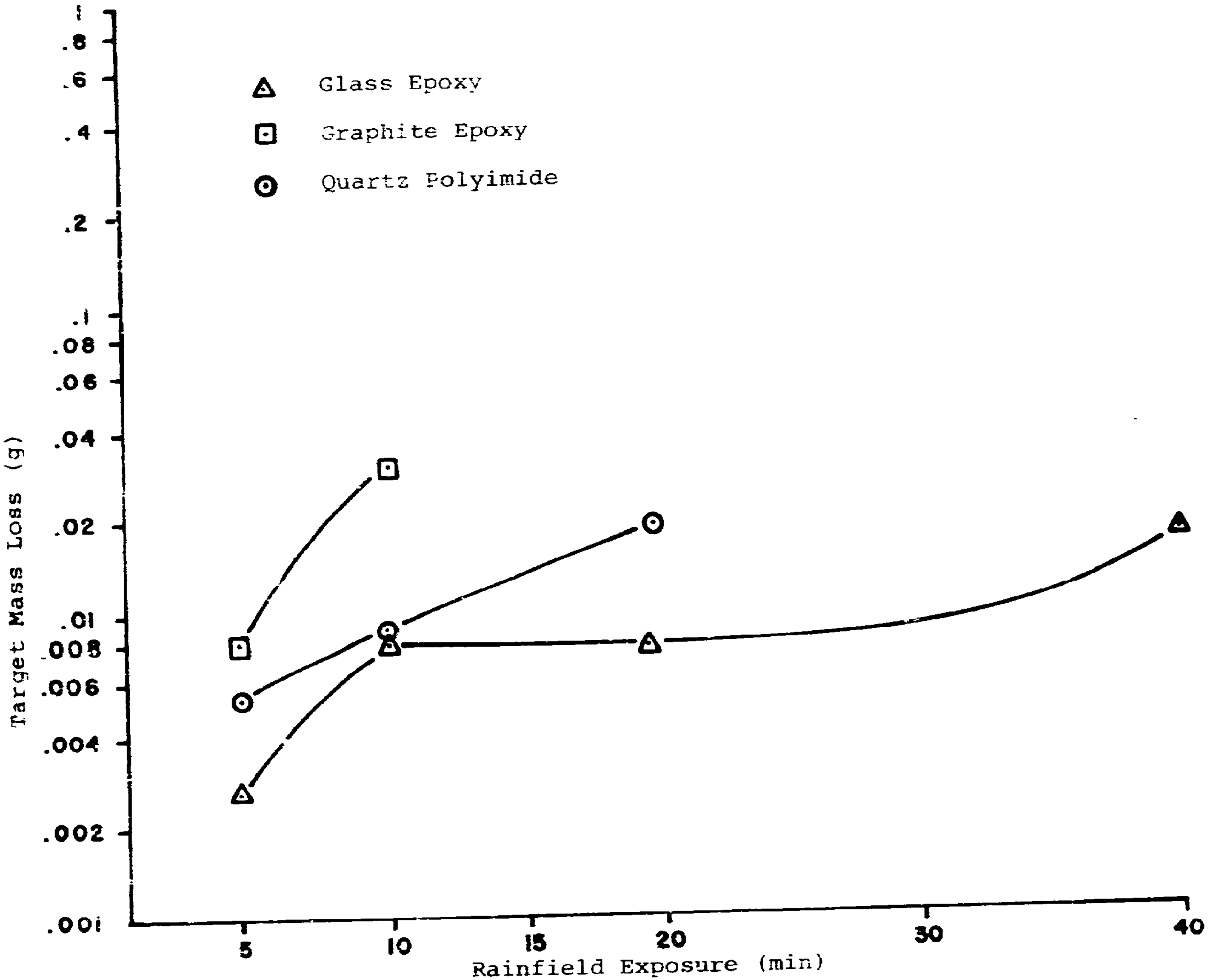


Figure 10. Target Mass Loss of Polyurethane Coated Composites Exposed at a 90° Impact Angle, 500 mph, 1 inch/hr Rainfall.

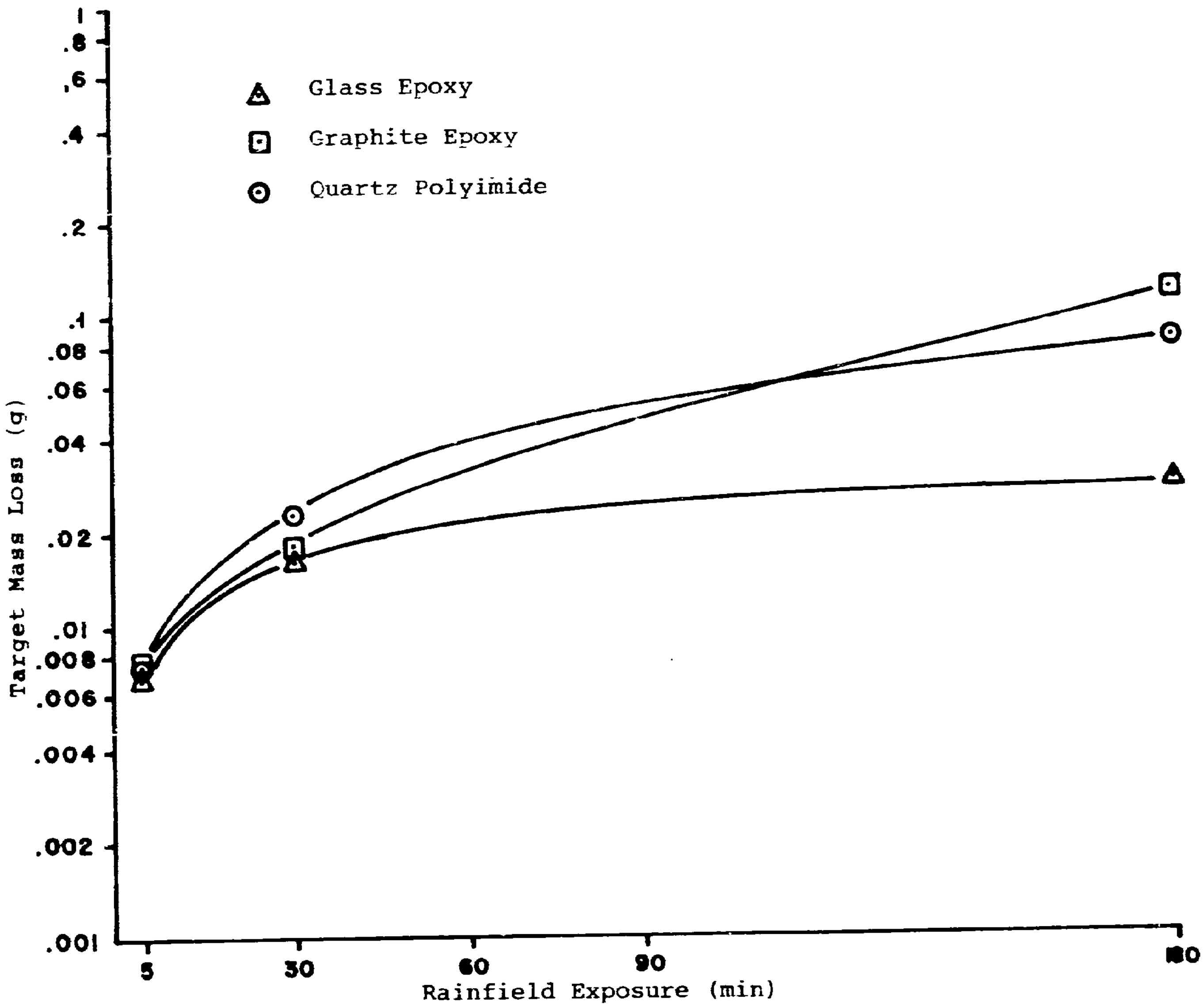


Figure 11. Target Mass Loss of Polyurethane Coated Composites Exposed at a 30° Impact Angle, 500 mph, 1 inch/hr Rainfall.

3. RAIN EROSION TEST RESULTS - SEM OBSERVATIONS

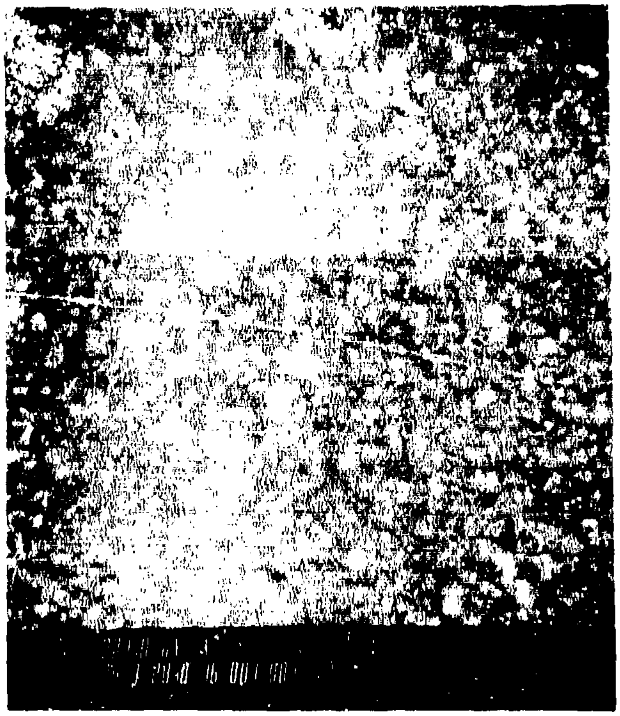
Selected test specimens which were exposed for varying time intervals to a 1 inch/hour simulated rainfall at 500 miles per hour were examined by scanning electron microscopy (SEM) for characterization of the mode of erosion damage and erosive processes. The surface morphology of the glass epoxy, graphite epoxy and quartz polyimide coated with the fluoroelastomer or polyurethane coatings was observed before and after exposure to rainfield conditions. The results obtained for these materials, supplied by the U.S. Air Force, are described herein.

a. Fluoroelastomer Coating - Glass Epoxy Substrate - 90° Impact Angle

The (AF-C-VBW-15-15) white fluoroelastomer coating on a glass epoxy substrate before exposure to rainfield conditions is shown in Figure 12. The surface of the fluoroelastomer coating exhibited a uniform morphology with homogeneous distribution of titanium dioxide pigment as shown in Figure 12A. However, the coating also contained elongated striations of the type shown in Figure 12B.

Exposure of the fluoroelastomer coated glass epoxy composite substrates to a rainfield environment at a 90° impact angle for timed durations up to 30 minutes resulted in localized surface erosion damage as well as crack formation, coating removal, and subsequent erosion damage of the glass epoxy composite as shown in Figures 13, 14, and 15.

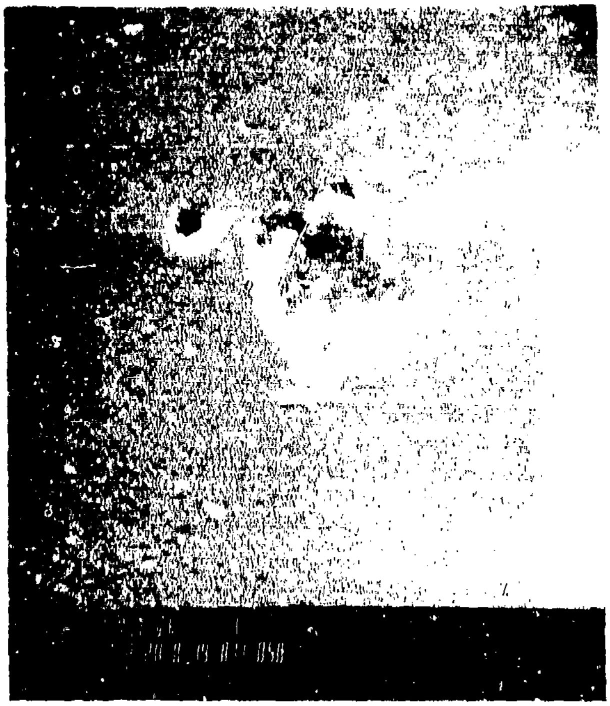
The formation of isolated eroded areas as shown in Figure 13 are clearly observed after rainfield exposure durations of five minutes (Figure 13A), 10 minutes (Figure 13B) and 30 minutes (Figure 13C). These localized erosion zones were in the order of 10 microns in size and tended to combine or agglomerate together as shown in Figure 13B. The erosion damage observed on the exposed surfaces was characterized by localized erosion events (Figure 13) or agglomeration of events (Figure 14C). Other damage mechanisms were also observed, such as coating buckling



A No Rainfield Exposure
4000X

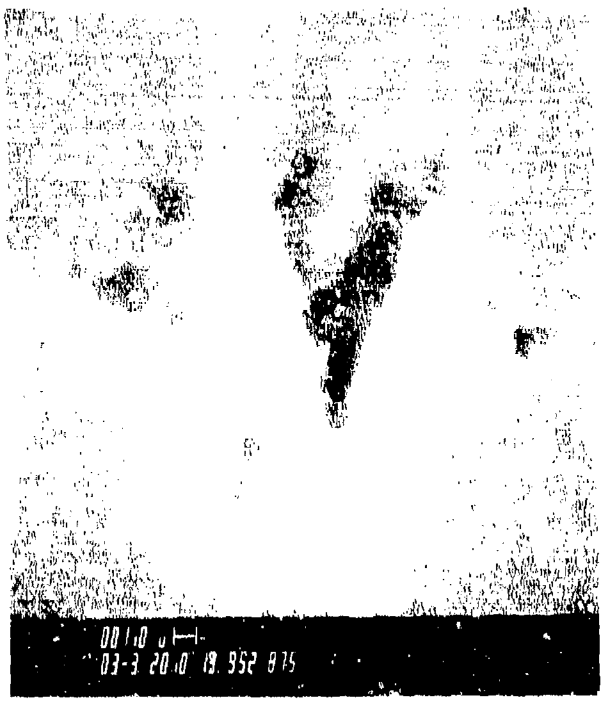
B No Rainfield Exposure
4000X

Figure 12. Fluoroelastomer Coated Glass Epoxy Composite.



A 5.0 min Rainfield Exposure
1000X

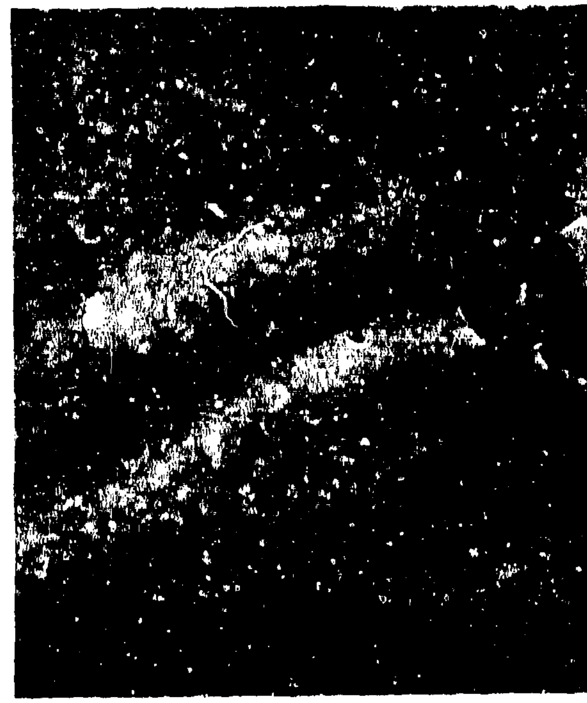
B 10.0 min Rainfield Exposure
800X



C 30.0 min Rainfield Exposure
3000X

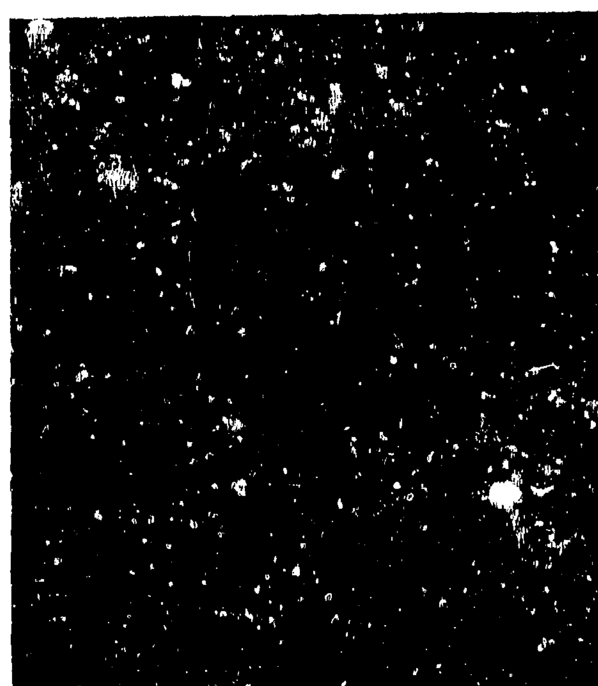
Figure 13. Fluoroelastomer Coated Glass Epoxy Composite, 90° Impact Angle.

A 15.0 min Rainfield Exposure
1700X



B 15.0 min Rainfield Exposure
4000X

C 15.0 min Rainfield Exposure
1000X



D 30.0 min Rainfield Exposure
5000X

Figure 14. Fluoroelastomer Coated Glass Epoxy Composite, 90° Impact Angle.

A 30.0 min Rainfield Exposure
60X

B 30.0 min Rainfield Exposure
900X

Figure 15. Fluoroelastomer Coated Glass Epoxy Composite, 90° Impact Angle.

(Figures 14A and 14B), microcracks (Figure 14D) leading to coating delamination and removal (Figure 15A) and subsequently to substrate erosion damage as shown in Figure 15B. It should be noted that these processes were associated with the presence of elongated striations found in the fluoroelastomer coating before exposure to rainfield conditions as shown in Figure 12B.

b. Fluoroelastomer Coating - Graphite Epoxy Substrate - 90° Impact Angle

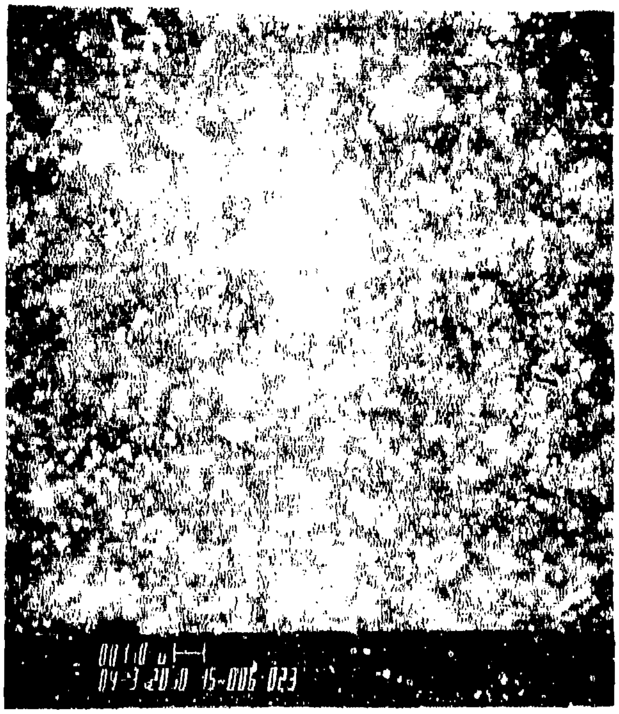
The fluoroelastomer coated graphite epoxy composite substrate exposure to rainfield conditions is shown in Figure 16. The coating contains a uniform surface morphology with homogeneous distribution of titanium dioxide pigment in the size range of 0.5 microns. This description is exhibited in Figure 16A. It also contained elongated striation defects as shown in Figure 16B.

Erosion damage was introduced into the fluoroelastomer coating during exposure to rainfield conditions at an impact angle of 90°. The surface damage after five and 10 minutes of rainfield exposure is shown in Figure 17. The erosion damage was characterized by local removal of coating materials, resulting in single crater formations in the 10 micron size range (Figure 17A) or agglomeration of such craters (Figure 17B). Furthermore, crater formation was associated with microcracks in the coating as revealed in Figure 17C. These microcracks were several microns in length and one to two microns in width.

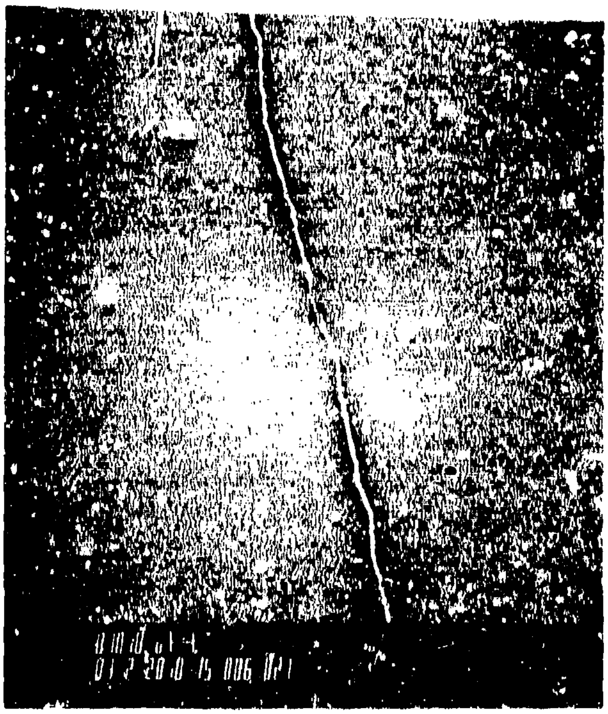
c. Fluoroelastomer Coating - Quartz Polyimide Substrate - 90° Impact Angle

A uniform fluoroelastomer coating was obtained on quartz polyimide substrates as shown in Figure 18A. The bright zones indicated a homogeneous distribution of titanium dioxide pigment. However, the coating contained elongated striations probably resulting from the coating application procedure as shown in Figure 18B. The elongated striations were approximately one micron in width and several dozen microns in length.

Figures 19 and 20 exhibit the erosion damage at a 90° impact angle on the fluoroelastomer coating surface after



A No Rainfield Exposure
4000X



B No Rainfield Exposure
300X

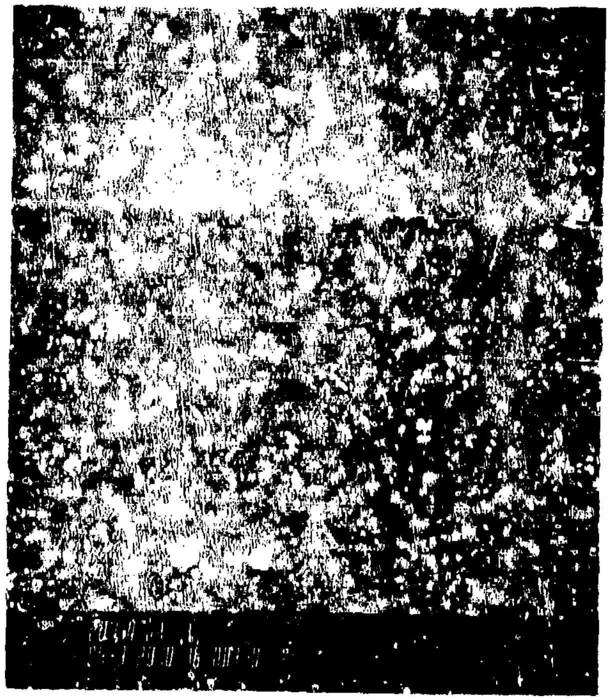
Figure 16. Fluoroelastomer Coated Graphite Epoxy Composite.

A 5.0 min Rainfield Exposure
5000X

B 10.0 min Rainfield Exposure
3000X

C 10.0 min Rainfield Exposure
2000X

Figure 17. Fluoroelastomer Coated Graphite Epoxy Composite, 90° Impact Angle.



A No Rainfield Exposure
4000X

B No Rainfield Exposure
4000X

Figure 18. Fluoroelastomer Coated Quartz Polyimide Composite.

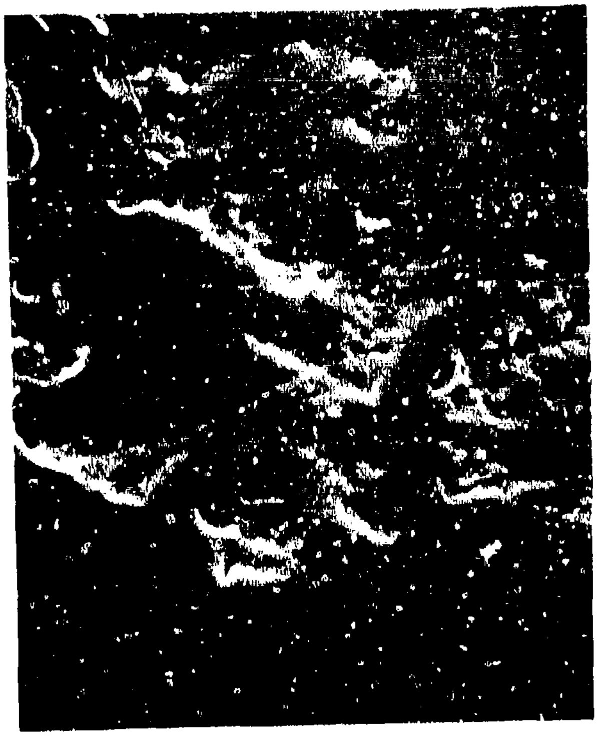
A 5.0 min Rainfield Exposure 3000X

B 5.0 min Rainfield Exposure 5000X

C 10.0 min Rainfield Exposure 7000X

D 10.0 min Rainfield Exposure 6000X

Figure 19. Fluoroelastomer Coated Quartz Polyimide Composite, 90° Impact Angle.



A 15.0 min Rainfield Exposure
500X

B 15.0 min Rainfield Exposure
1000X

C 30.0 min Rainfield Exposure
3000X

D 30.0 min Rainfield Exposure
8000X

Figure 20. Fluoroelastomer Coated Quartz Polyimide Composite, 90° Impact Angle.

exposure to rainfield conditions up to 30 minutes in duration. The damage illustrated was characterized by formation of localized erosion sites in the 10 micron size range after five minutes (Figure 19A and 19B) and 10 minutes (Figure 19C and 19D) of rainfield exposure. Rainfall exposure intervals of 15 minutes to 30 minutes resulted in the formation of clusters of single erosion sites as shown in Figure 20. Furthermore, the erosion damage after 30 minutes of rainfield exposure at a 90° impact angle resulted in coating cracking and delamination (Figure 21A) followed by severe erosion damage incurred in the quartz polyimide substrate (Figure 21B and 21C). Erosion damage in the quartz polyimide substrate material was characterized by broken quartz fibers (Figure 21B) as well as by polyimide resin removal (Figure 21C).

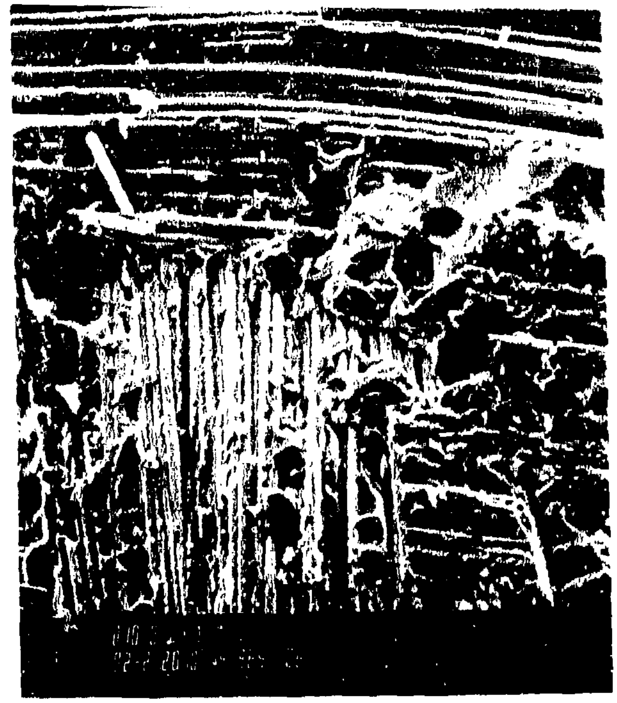
d. Fluoroelastomer Coating - Glass Epoxy Substrate - 30° Impact Angle

The fluoroelastomer coated glass epoxy substrates exposed to rainfield conditions at a 30° impact angle for time intervals up to 165 minutes exhibited a surface erosion damage mode shown in Figure 22. Isolated erosion sites of localized coating removal in the range of 10 microns were characterized and were observed in Figures 22B and 22C. A rainfall exposure interval of 165 minutes resulted in localized erosion sites as well as evidence of microcracks associated with the isolated erosion sites.

e. Fluoroelastomer Coating - Graphite Epoxy Substrate - 30° Impact Angle

The graphite epoxy substrates coated with the fluoroelastomer coating and exposed to rainfield conditions at a 30° impact angle for time intervals of five minutes to 180 minutes duration resulted in surface erosion damage as shown in Figure 23. The mode of damage was characterized by local coating material removal followed by the formation of isolated craters several microns in size.

A 30.0 min Rainfield Exposure
900X



B 30.0 min Rainfield Exposure
200X

C 30.0 min Rainfield Exposure
1100X

Figure 21. Fluoroelastomer Coated Quartz Polyimide Composite, 90° Impact Angle.

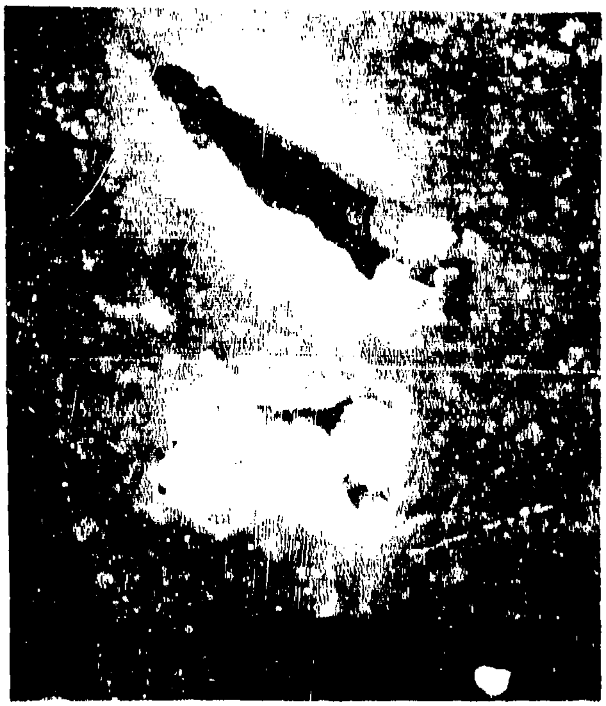
A 5.0 min Rainfield Exposure
1000X

B 30.0 min Rainfield Exposure
1500X

C 30.0 min Rainfield Exposure
1000X

Figure 22. Fluoroelastomer Coated Glass Epoxy Composite, 30° Impact Angle.

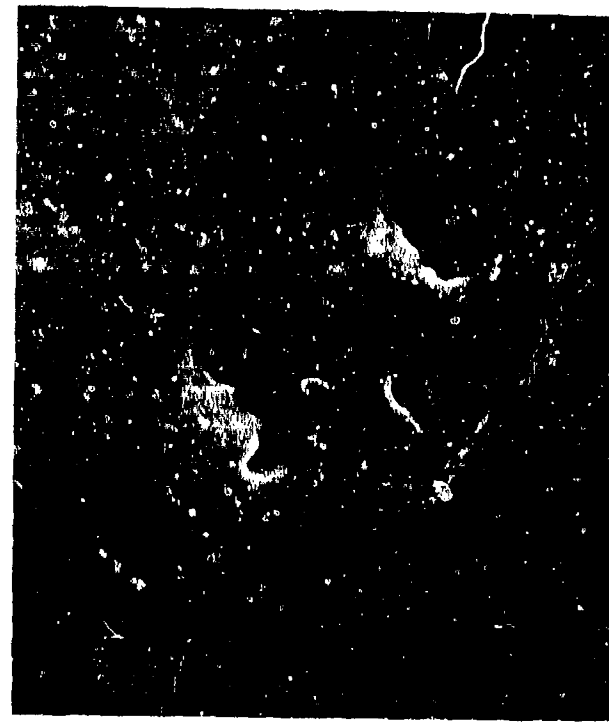
D 165.0 min Rainfield Exposure
1500X



E 165.0 min Rainfield Exposure
5000X

Figure 22 (Cont'd). Fluoroelastomer Coated Glass Epoxy Composite, 30° Impact Angle.

A 5.0 min Rainfield Exposure
2000X



B 30.0 min Rainfield Exposure
5000X

C 180.0 min Rainfield Exposure
4000X

Figure 23. Fluoroelastomer Coated Graphite Epoxy Composite, 30° Impact Angle.

f. Fluoroelastomer Coating - Quartz Polyimide Substrate - 30° Impact Angle

Scanning electron microscopy observations of fluoroelastomer coated quartz polyimide substrate materials exposed at a 30° impact angle to rainfield conditions for five minutes to 180 minutes revealed surface erosion damage as shown in Figure 24. The erosion damage was characterized by localized coating removal at erosion sites resulting in crater formation. These local events up to 10 microns in size were either isolated as shown in Figure 24A or agglomerated as depicted in Figures 24B and 24C.

g. Polyurethane Coating - Glass Epoxy Substrate - 90° Impact Angle

The MIL-C-83445A white polyurethane coating on glass epoxy composite substrate is shown in Figure 25 prior to rainfield exposure. The coating exhibits a uniform homogeneous distribution of titanium dioxide pigment as depicted in Figure 25A up to 1 micron in diameter. Unexpectedly, however, low magnification observations revealed the presence of a layered structure consisting of "grain boundaries" having a hexagonal ring shape. These structures range in size up to 100 microns in diameter and are clearly observed in Figure 25B.

Surface structure and morphology of the polyurethane coated glass epoxy composite substrates after exposure to rainfield conditions at a 90° impact angle for intervals of five minutes to 40 minutes are shown in Figure 26. The erosion damage was associated with localized material removal from the coating surface resulting in single craters or clusters of craters as shown in Figure 26C and 26E. The presence of the grain-type structures as well as the titanium dioxide pigments were not directly associated with local erosion processes as clearly shown in Figures 26A, 26D, and 26B, respectively.

h. Polyurethane Coating - Graphite Epoxy Substrate - 90° Impact Angle

The polyurethane coated graphite epoxy composite substrates exhibited the same uniform homogeneity of pigment

A 5.0 min Rainfield Exposure
8000X

B 30.0 min Rainfield Exposure
8000X

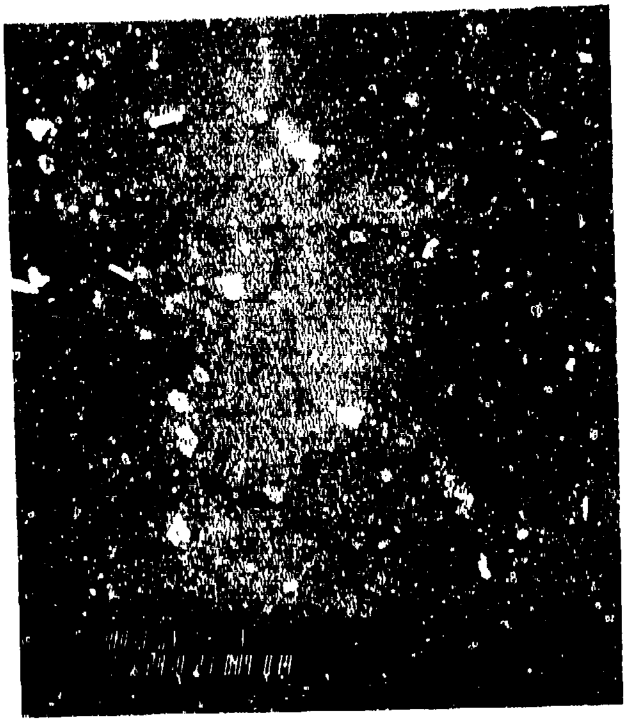
C 180.0 min Rainfield Exposure
4000X

Figure 24. Fluoroelastomer Coated Quartz Polyimide Composite, 30° Impact Angle.

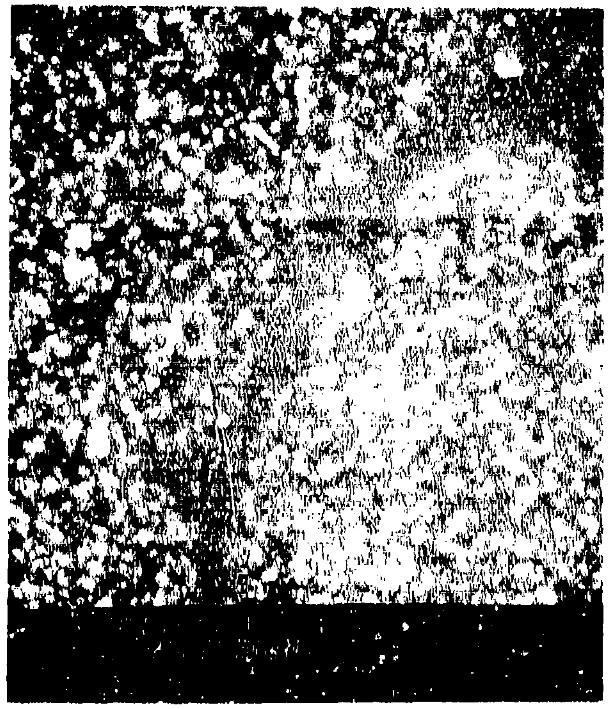
A No Rainfield Exposure
4000X

B No Rainfield Exposure
100X

Figure 25. Polyurethane Coated Glass Epoxy Composite.

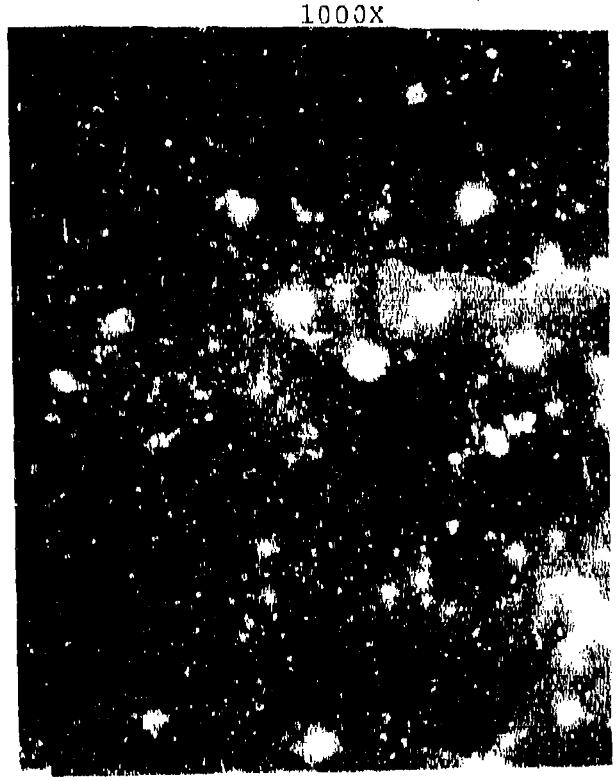


A 5.0 min Rainfield Exposure
100X



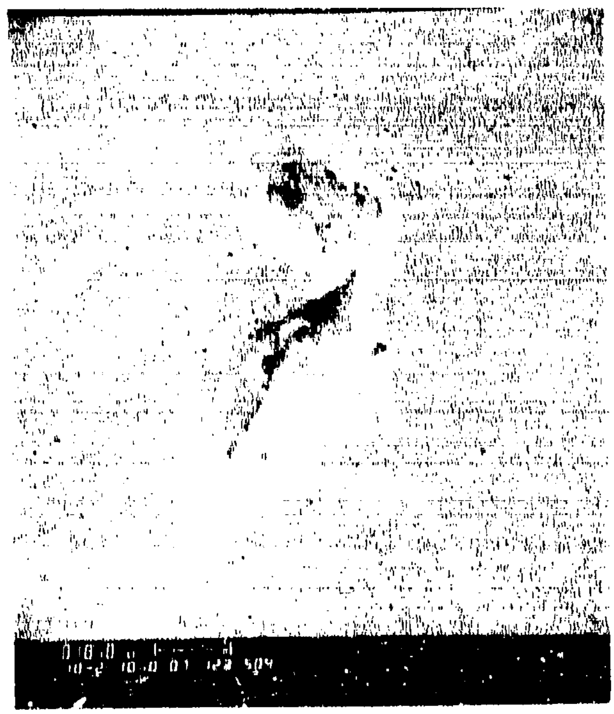
B 5.0 min Rainfield Exposure
1000X

C 20.0 min Rainfield Exposure
700X



D 40.0 min Rainfield Exposure
1000X

Figure 26. Polyurethane Coated Glass Epoxy Composite, 90° Impact Angle.



E 40.0 min Rainfield Exposure
1000X

Figure 26 (Cont'd). Polyurethane Coated Glass Epoxy Composite, 90° Impact Angle.

dispersion as previously described and is again depicted in Figure 27. The same hexagonal grain structure ring shape was observed in Figures 27A and 27B and previously described.

Erosion damage was introduced into the polyurethane coating surface on the graphite epoxy composite materials during rainfield exposure at a 90° impact angle. Five-minute exposures to rainfield conditions resulted in single craters or clusters of craters as illustrated in Figures 28A and 28B. An exposure to rainfield conditions of 10 minutes duration exhibited extensive microcracking in the polyurethane coating surface and is shown in Figure 28C.

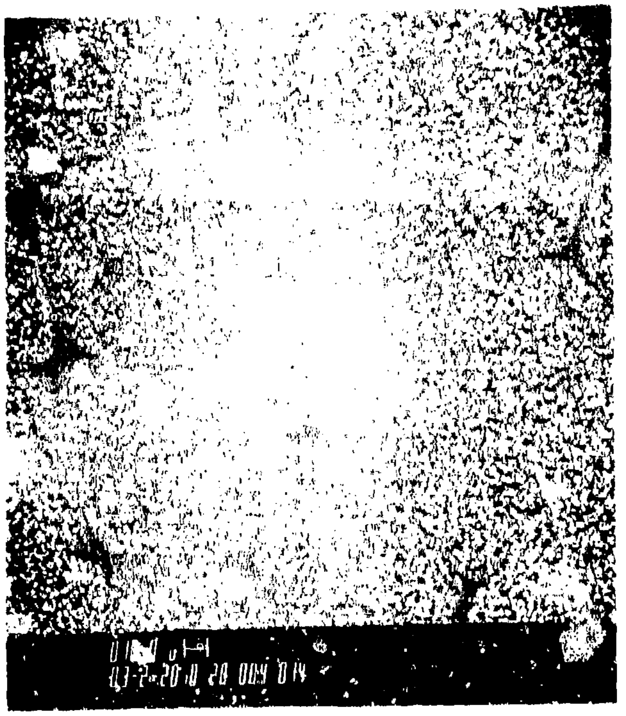
i. Polyurethane Coating - Quartz Polyimide Substrate - 90° Impact Angle

As expected, the polyurethane coated quartz polyimide composite substrates exhibited the familiar uniform pigment dispersion characteristics. Hexagonal ring structures up to 100 microns in diameter again appeared in the coating surface. These observations are illustrated in Figures 29A and 29B.

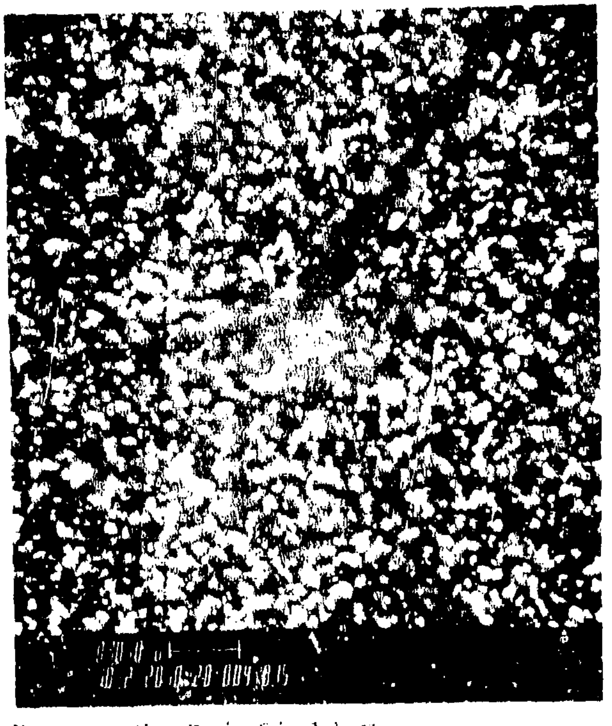
Figures 30 and 31 exhibit the erosion damage at a 90° impact angle on the polyurethane coated quartz polyimide composite substrates after exposure to rainfield conditions up to 20 minutes in duration. Localized surface erosion in the form of craters and microcracking of the polyurethane coating was exhibited after five minutes and 10 minutes of rainfield exposure as shown in Figures 30A, 30B, 30C, and 30D. Some minor coating tearing along the path of the microcracks was noted as in Figure 30B. Surface characterization after 20 minutes duration indicated continued localized crater production and increased microcracking. The microcracks observed did not appear to be associated with crater formation as shown in Figure 31.

j. Polyurethane Coating - Glass Epoxy Substrate - 30° Impact Angle

The polyurethane coated glass epoxy substrates exposed to rainfield conditions at a 30° impact angle for intervals of



A No Rainfield Exposure
300X



B No Rainfield Exposure
1000X

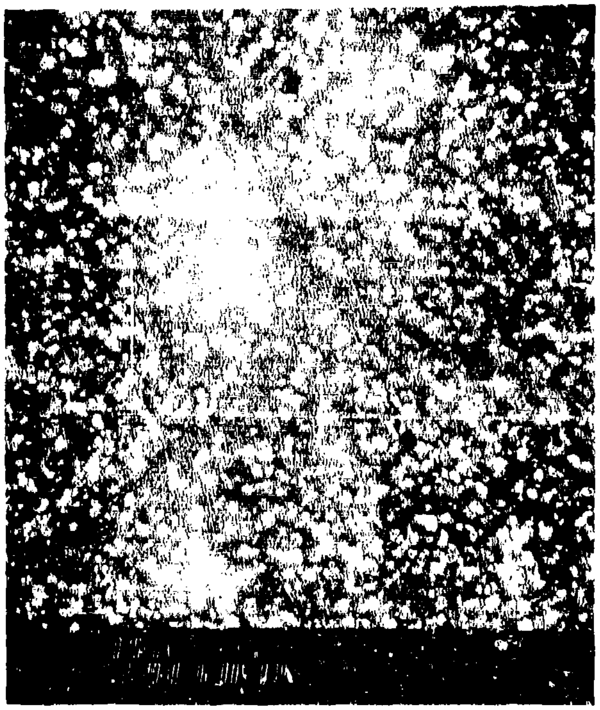
Figure 27. Polyurethane Coated Graphite Epoxy Composite, 90° Impact Angle.

A 5.0 min Rainfield Exposure
1000X

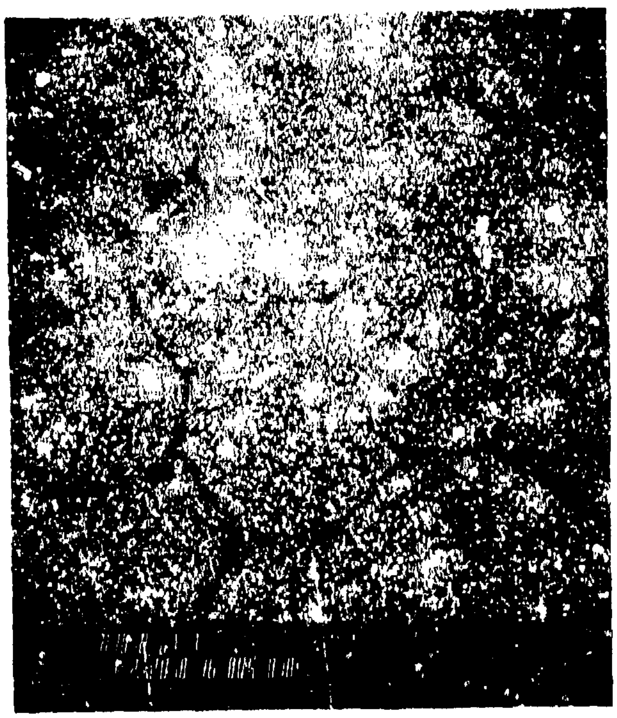
B 5.0 min Rainfield Exposure
3000X

C 10.0 min Rainfield Exposure
2000X

Figure 28. Polyurethane Coated Graphite Epoxy Composite, 90° Impact Angle.



A No Rainfield Exposure
1000X

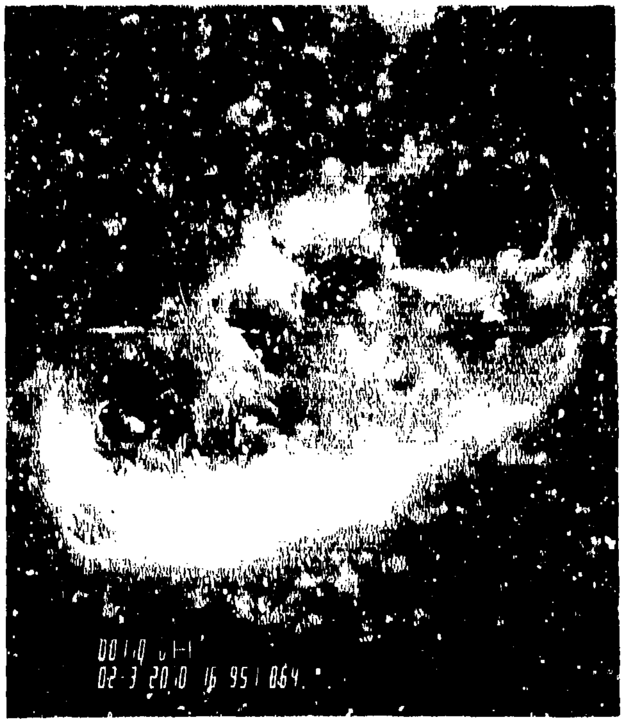


B No Rainfield Exposure
300X

Figure 29. Polyurethane Coated Quartz Polyimide Composite, 90° Impact Angle.

A 5.0 min Rainfield Exposure
2000X

B 5.0 min Rainfield Exposure
2000X

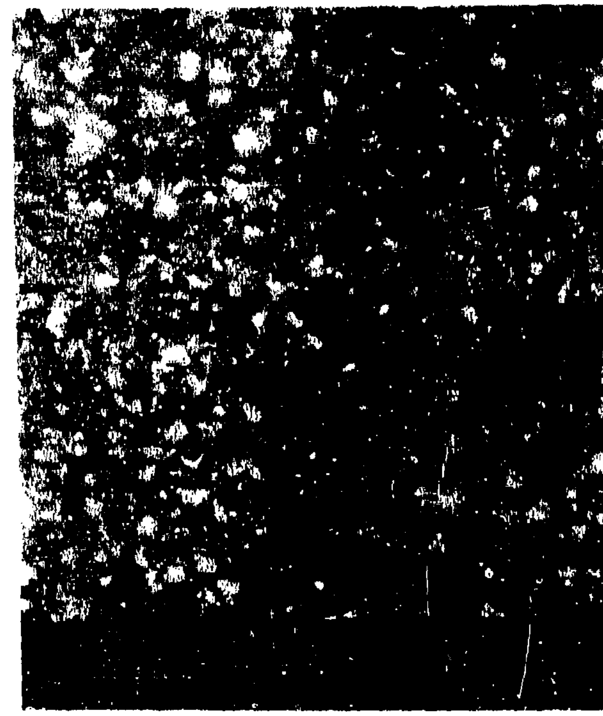


C 10.0 min Rainfield Exposure
2000X

D 10.0 min Rainfield Exposure
4000X

Figure 30. Polyurethane Coated Quartz Polyimide Composite, 90° Impact Angle.

A 20.0 min Rainfield Exposure
2000X



B 20.0 min Rainfield Exposure
2000X

C 20.0 min Rainfield Exposure
4000X

Figure 31. Polyurethane Coated Quartz Polyimide Composite, 90° Impact Angle.

five minutes to 180 minutes resulted in surface erosion as shown in Figure 32. The erosion damage was characterized by the formation of isolated craters in the coating surface after 30 minutes (Figure 32C) and 180 minutes (Figure 32D) of rainfield exposure. Furthermore, microcracking of the polyurethane coating was observed after 30 minutes exposure (Figure 32B) and 180 minutes exposure (Figure 32E), respectively. These microcracks are probably associated with the "hexagonal shaped grain boundary structure" previously observed in the polyurethane coating as shown in Figure 32B. The observed microcracks in the polyurethane coating were a surface phenomena and did not penetrate to the glass epoxy interface (Figure 32E).

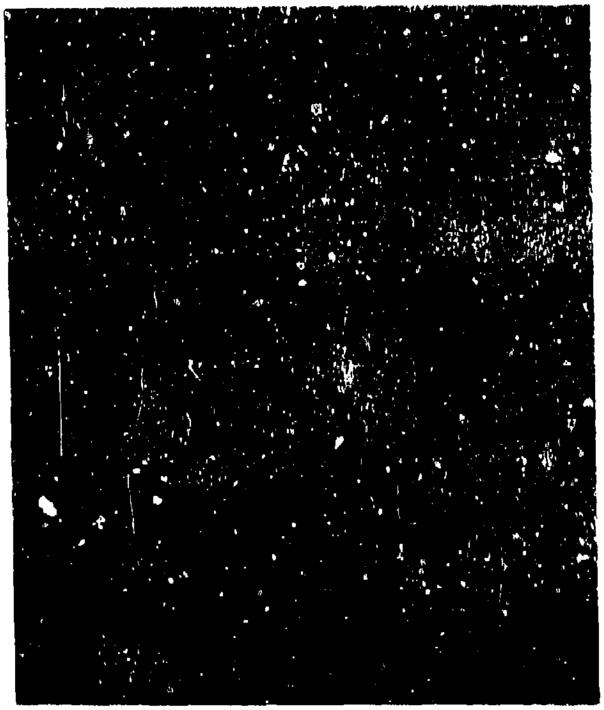
k. Polyurethane Coating - Graphite Epoxy Substrate - 30° Impact Angle

Figures 33 and 34 illustrate the mode of erosion damage introduced into the polyurethane coating on graphite epoxy composite substrates, exposed at an impact angle of 30°, after rainfield exposure conditions for five minutes to 180 minutes. Microcracks developed in the coating; however, they did not penetrate to the coating/substrate interface as is clearly shown in Figures 33B and 33D. In addition to microcrack formation, isolated craters were observed in Figures 33C and 34B. These localized erosion zones did not penetrate to the coating/substrate interface even after 180 minutes of rainfield exposure.

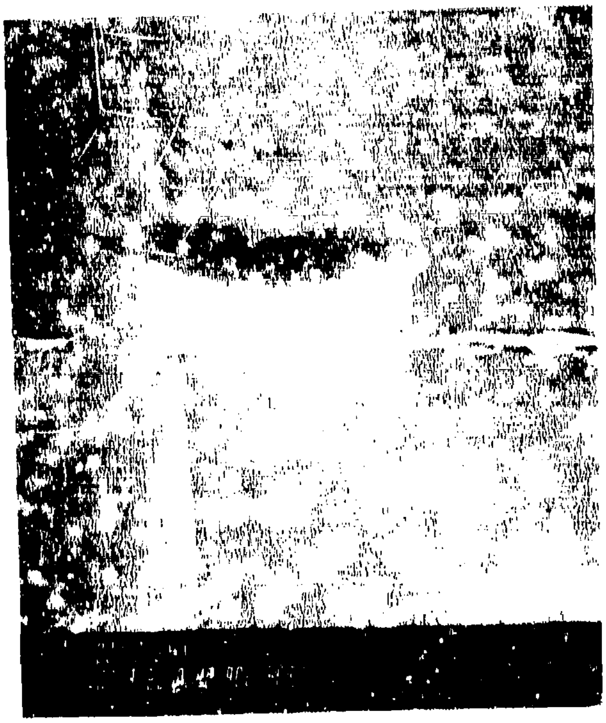
l. Polyurethane Coating - Quartz Polyimide Substrate - 30° Impact Angle

Exposure to a 30° impact angle in the rainfield of the polyurethane coated quartz polyimide substrates for 30 minutes to 180 minutes resulted in localized erosion damage as well as in coating crack formation. These events are shown in Figures 35 and 36. The cracks were several microns in length and 1 micron in width as illustrated in Figures 35C and 36C, respectively. In some cases, the cracks were associated with localized crater formation and are depicted in Figure 36A. The cracks did not penetrate through to the coating substrate interface and, therefore, no coating delamination was observed.

A 5.0 min Rainfield Exposure
2000X



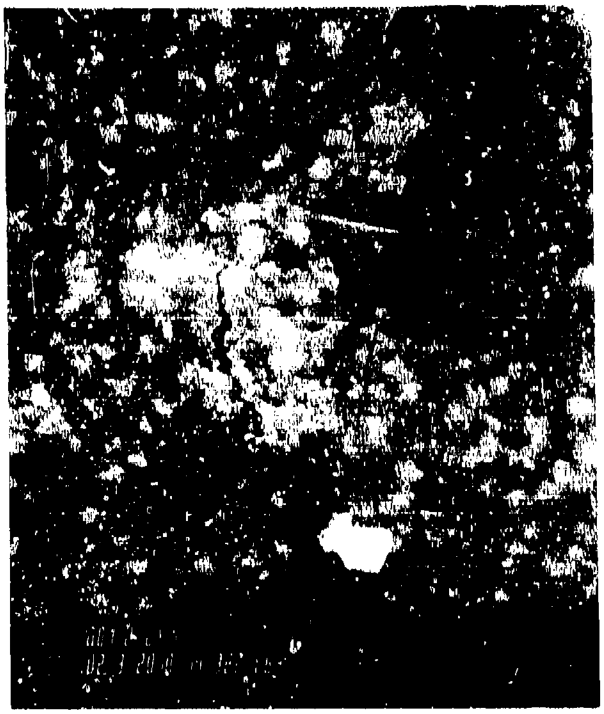
B 30.0 min Rainfield Exposure
200X



C 30.0 min Rainfield Exposure
2000X

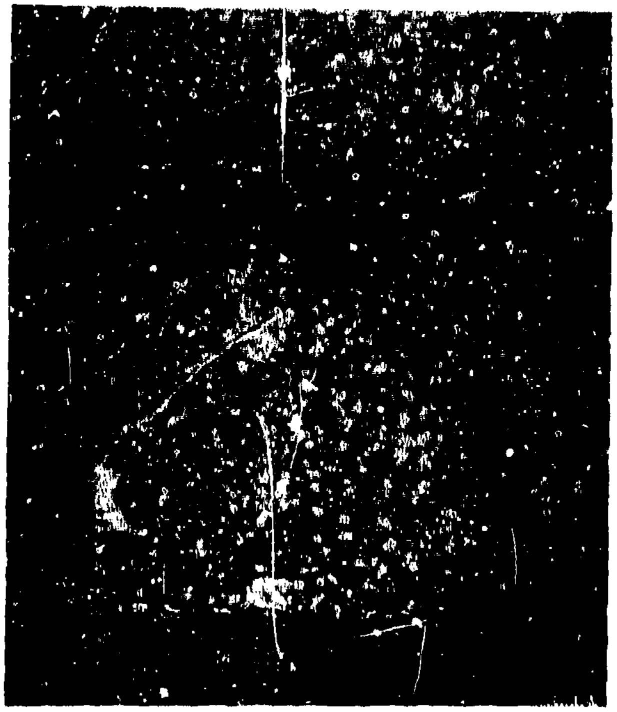
D 180.0 min Rainfield Exposure
1000X

Figure 32. Polyurethane Coated Glass Epoxy Composite, 30° Impact Angle.



E 180.0 min Rainfield Exposure
2000X

Figure 32 (Cont'd). Polyurethane Coated Glass Epoxy Composite, 30° Impact Angle.



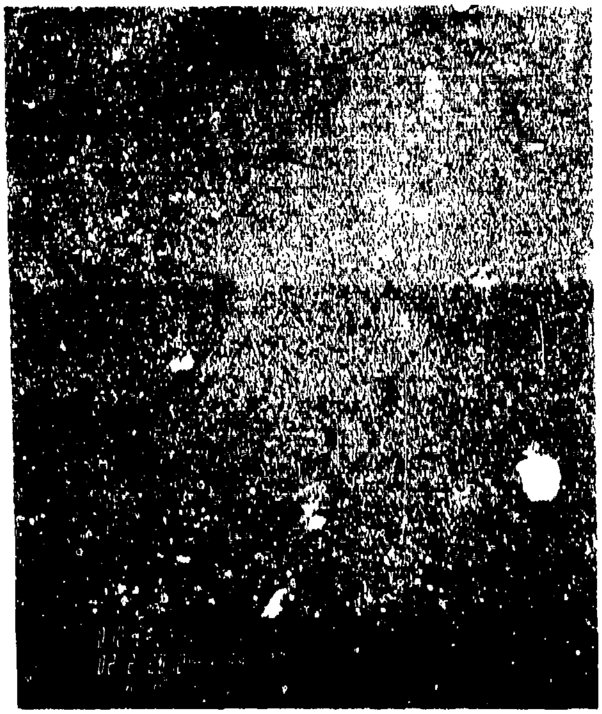
A 5.0 min Rainfield Exposure
900X

B 5.0 min Rainfield Exposure
9000X

C 30.0 min Rainfield Exposure
3000X

D 30.0 min Rainfield Exposure
3000X

Figure 33. Polyurethane Coated Graphite Epoxy Composite, 30° Impact Angle.



A 180.0 min Rainfield Exposure
200X

B 180.0 min Rainfield Exposure
4000X

C 180.0 min Rainfield Exposure
2000X

Figure 34. Polyurethane Coated Graphite Epoxy Composite, 30° Impact Angle.

A 30.0 min Rainfield Exposure
5000X

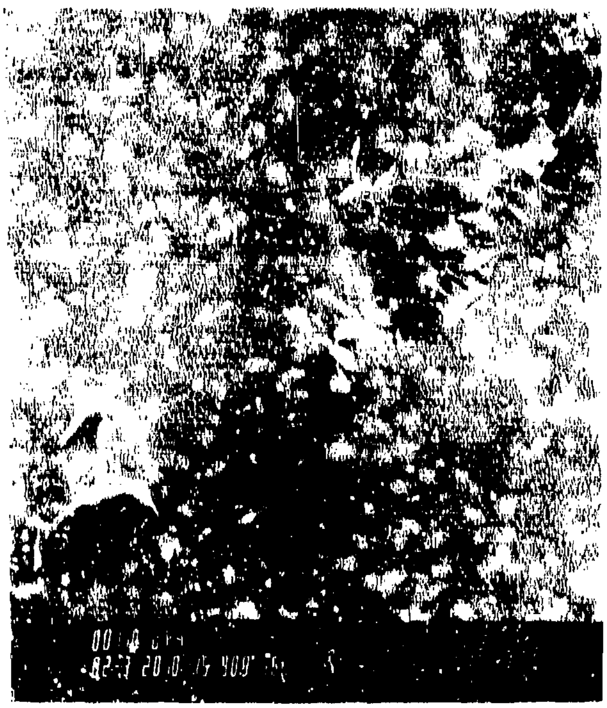
B 30.0 min Rainfield Exposure
4000X

C 30.0 min Rainfield Exposure
2000X

Figure 35. Polyurethane Coated Quartz Polyimide Composite, 30° Impact Angle.

A 180.0 min Rainfield Exposure 1000X

B 180.0 min Rainfield Exposure 1000X



C 180.0 min Rainfield Exposure 2000X

Figure 36. Polyurethane Coated Quartz Polyimide Composite, 30° Impact Angle.

SECTION IV
DISCUSSION

1. RAIN EROSION BEHAVIOR - FLUOROELASTOMER

a. Kinetics

The kinetic behavior of the fluoroelastomer coated glass epoxy, graphite epoxy and quartz polyimide composite materials under rain erosion conditions were evaluated in terms of specimen mass loss and percent coating removal. At the 90° impact angle, coating removal increased gradually with rainfield exposure time up to the ten minute interval as shown in Figure 4. This correlates with the gradual increase in target mass loss shown in Figure 8. Further exposure of the coated glass epoxy and quartz polyimide to rainfield conditions at a 90° impact angle resulted in sharp increases in coating removal and target mass loss. The material losses were associated with excessive coating removal after a rainfield exposure interval of fifteen minutes followed by severe erosion damage of the composite substrate material.

At an impact angle of 30° under rainfield exposure conditions, the amount of visible coating removal was practically nil and independent of exposure time as shown in Figure 5. However, the target mass loss increased gradually with rainfield exposure up to the thirty minute interval and thereafter no further significant mass loss was observed as shown in Figure 9. This behavior indicated material removal from the coating surface without visibly apparent coating removal and substrate erosion.

b. Surface Characterization

Coating surface morphology was characterized by scanning electron microscopy observations. The effects of rainfield exposure conditions such as impact angle, exposure time and the type of composite substrate material were evaluated.

c. Effect of Impact Angle

Exposure of the fluoroelastomer coated composite substrate at a 90° impact angle resulted in the formation of single craters or cratering agglomeration in the coating surface as shown in Figure 13. Furthermore, microcracks were formed resulting in coating delamination as depicted in Figure 14. This led to subsequent damage of the composite substrate material shown in Figure 15.

Rainfield exposure conditions at a 30° impact angle resulted primarily in the formation of isolated craters in the coating surface as shown in Figure 22C. Some microcracking was evident in the coating after an exposure duration of one hundred and sixty five minutes for the fluorocarbon coated glass epoxy composite as shown in Figure 22D. Furthermore at the 30° impact angle, no visibly substantial coating removal was observed. These observations correlate with the kinetic data shown in Figure 5.

d. Effect of Exposure Time

The exposure duration of the various fluoroelastomer coated composites under rain erosion conditions resulted in the formation of craters in the coating surface after five minutes of rainfield exposure at both 90° and 30° impact angles. After five minutes exposure at a 90° impact angle, microcracks were also formed. This resulted in partial coating delamination and separation at the fifteen minute rainfield exposure interval. Subsequently, damage was introduced into the composite substrate material during the fifteen to thirty minute exposure interval. It should be noted that the coating removal and substrate damage incurred during this interval is in correlation with the kinetic curves shown in Figure 4 and 8. High increases in target mass loss were observed after the fifteen and thirty minute exposure intervals.

When the test specimens were exposed to rainfield exposure conditions at a 30° impact angle, crack formation was observed only after one hundred and sixty five minutes as shown in Figure 22D. This correlates well with the amount of coating removal versus rainfield exposure time shown in Figure 5.

e. Effect of Composite Material

The effect of composite construction on the rain erosion performance of the fluoroelastomer coating is discussed herein. The fluoroelastomer coated glass epoxy composite specimens exposed at a 90° impact angle to rainfield conditions exhibited the least amount of overall visible damage and mass loss. Fluoroelastomer coated quartz polyimide followed the same general trend as the coated glass epoxy, but exhibited severe erosion of the composite substrate after coating removal. The graphite epoxy coated with fluoroelastomer exhibited the most significant degree of visible damage and target mass at the 90° impact angle. At the 30° impact angle, the fluoroelastomer coated composites exhibited essentially no visible damage until thirty minutes of rainfield exposure. In terms of target mass loss, the coated composites exhibited an order of magnitude increase in mass loss during the first thirty minutes of rainfield exposure conditions. From thirty to one hundred and sixty five minutes of rain exposure, the coated composites exhibited no substantial increase in mass loss. The fluorocarbon coated graphite epoxy showed the largest increase in material loss during the first thirty minutes of exposure.

2. RAIN EROSION BEHAVIOR-POLYURETHANE

a. Kinetics

The kinetic behavior of the polyurethane coated glass epoxy, graphite epoxy and quartz polyimide composite materials under rainfield exposure conditions were evaluated in terms of specimen mass loss and percent coating removal. At a 90° impact angle, coating removal increased gradually with rainfield exposure time up to the ten minute interval as depicted in Figure 6. This correlates well with the target mass loss curves shown in Figure 10. Further exposure of the coated glass epoxy and quartz polyimide to rainfield exposure conditions at a 90° impact angle resulted in slight increases in coating removal and target mass loss. The material losses were associated with moderate coating removal after a rainfield exposure interval of fifteen minutes.

At the 30° impact angle under rainfield exposure conditions, the amount of coating removal was essentially nil and independent of exposure time as shown in Figure 7. Target mass loss increased gradually with rainfield exposure time up to thirty minutes and increased slightly thereafter, except for the polyurethane coated graphite epoxy specimens. Target mass loss versus rainfield exposure is shown in Figure 11. Their behavior indicated material removal from the coating surface without visibly apparent coating removal and substrate erosion.

b. Surface Characterization

Coating surface morphology was characterized by scanning electron microscopy observations. The effects of rainfield exposure conditions such as impact angle, exposure time and type of composite substrate material were evaluated.

c. Effect of Impact Angle

Exposure of the polyurethane coated composite substrates at a 90° impact angle resulted in the formation of single craters or cratering agglomeration in the coating surface as shown in Figure 26. Furthermore, microcracks were formed resulting in the initiation of coating delamination as depicted in Figure 28.

Rainfield exposure conditions at a 30° impact angle resulted primarily in the formation of isolated craters in the coating surface. Microcracking was evident in the polyurethane coating on all three substrates. The microcracking process occurred between five minutes and one hundred and eighty minutes of rainfield exposure depending upon the type of substrate. In no case did the cracks penetrate to the substrate, therefore, no delamination of the polyurethane coating was observed. These observations correlate with the kinetic data shown in Figure 7.

d. Effect of Exposure Time

The exposure duration of the various polyurethane coated composite specimens evaluated under rainfield exposure conditions resulted in the formation of craters in the coating surface after five minutes for polyurethane coated graphite epoxy and quartz polyimide specimens and twenty minutes for the polyurethane coated glass epoxy at the 90° impact angle. After ten minutes exposure, microcracks were formed on the coated graphite epoxy and quartz polyimide specimens. No microcracking was observed in the polyurethane coated glass epoxy after forty minutes of rainfield exposure at a 90° impact angle. This correlates well with the kinetic curves shown in Figures 6 and 10.

Rainfield exposure at a 30° impact angle resulted in isolated craters at the thirty minute interval for all the polyurethane coated composites. Crack formation was observed after thirty minutes of rainfield exposure in the polyurethane coated graphite epoxy and quartz polyimide composite specimens and after one hundred and eighty minutes as shown in Figure 32 for the polyurethane coated glass epoxy. This correlates well with the amount of coating removal versus rainfield exposure time shown in Figure 7.

e. Effect of Composite Material

The effect of composite construction on the rain erosion performance of the polyurethane coating is discussed herein. The polyurethane coated glass epoxy composite specimens exposed at a 90° impact angle to rainfield conditions exhibited the least amount of overall visible damage and mass loss. Polyurethane coated quartz polyimide exhibited the same amount of coating removal as the coated glass epoxy, but in only half the exposure time as the coated glass epoxy. Mass loss versus exposure duration of the coated quartz polyimide followed the same general curve shape, but slightly higher. The graphite epoxy coated with polyurethane exhibited the most significant degree of visible damage and target mass loss at the 90° impact angle. At the 30° impact angle, the polyurethane coated composites exhibited little visible damage up to the thirty minute interval of rainfield exposure. In terms of target mass loss, the coated composites exhibited an order of magnitude increase in mass loss during the first thirty minutes of rainfield exposure. From thirty to one hundred and eighty minutes of rain exposure, the coated composites (the coated glass epoxy specimens) exhibited no substantial increase in mass loss. The coated graphite epoxy specimens exhibited a continuing increase in mass loss. The coated quartz polyimide specimens exhibited a continuing mass loss with a lessening of the rate of mass loss at the one hundred and eighty minute interval.

SECTION V
CONCLUSIONS

The polyurethane coated composites exhibited greater observable visual damage after exposure to a 90° angle of droplet impingement versus the 30° angle of exposure. The fluoroelastomer coated composites also revealed greater observable visual damage at a 90° droplet impact angle versus a 30° angle of exposure. The polyurethane coatings exhibited a significantly lower rate of mass loss than the fluoroelastomer coatings at 90°, except in the case of the graphite coated composites (see Figures 2,3). Polyurethane and fluoroelastomer coatings on graphite epoxy composites followed the same general trend of mass loss rate at 90° impingement angles. Mass loss rates for both coatings at 30° impingement angles were generally similar (see Figures 4, 5).

Polyurethane coated glass epoxy exhibited the least amount of visible damage and mass loss after exposure at 90°. Polyurethane coated quartz-polyimide closely followed the results of the glass-epoxy materials. Polyurethane coated graphite epoxy exhibited the greatest degree of visible surface damage at the 90° angle of droplet impact. Fluoroelastomer coated composites exposed at 90° followed the same general trend of damage distribution among the composite substrates but exhibited greater degrees of damage in shorter exposure periods. No substantial difference between the polyurethane and fluoroelastomer on the three different composite substrates at 30° angle of rainfield exposure occurred.

Polyurethane coated composites exhibited less visible damage and less mass loss than the fluorocarbon coated composites at a 90° angle of exposure for time periods of up to 40 minutes. Polyurethane and fluorocarbon coated composite structures of glass epoxy, glass polyimide, and graphite epoxy exposed at a 30° angle of droplet impact exhibited essentially minor differences in the amount of target mass loss after 180 minutes of rainfield exposure.

Selected specimens were examined using scanning electron microscopy techniques. Cratering initiation and crack propagation in the polymeric coating was observed. The degree of coating surface damage was greater for the fluoroelastomer coated composites as a function of rainfield exposure time regardless of droplet impact angle. In terms of composite construction, the graphite epoxy substrate was the most significant in its influence on the degree of damage to the coating surfaces. Glass epoxy substrates exhibited the least amount of damage to the surface coatings.

Of the two erosion resistant coatings, the polyurethane coating provided greater protection of the composite substrates than the fluorocarbon coating. The glass-epoxy substrates exhibited increased performance for the polyurethane and fluoroelastomer coatings. Quartz-polyimide substrates closely followed the performance of the coated glass epoxy materials. Polyurethane and fluorocarbon-coated graphite-epoxy substrates exhibited the greatest degree of coating surface damage. Surface erosion mechanisms and characteristics of coated materials can be identified and analyzed by SEM techniques.

The degree of protection which these two classes of coatings provide should be kept in perspective since they are by far the most rain erosion resistant polymeric coatings available. Other polymeric type coatings fail very rapidly under similar rain exposure conditions and only with these elastomeric systems is true rain protection for extended exposure periods possible.

SECTION VI

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