NASA Technical Memorandum 80071

NASA-TM-80071 19790021161

Environmental Effects on Space Shuttle Reusable Surface Insulation Coated With Reaction Cured Glass

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AUGUST 1979

1 Report No	2 Government Accession No	3 Recipient's Catalog No
NASA TM-80071		

4 Title and Subtitle	5 Report Date
ENVIRONMENTAL EFFECTS ON SPACE SHUTTLE REUSABLE SURFACE INSULATION COATED WITH REACTION CURED GLASS	August 1979
	6 Performing Organization Code
7 Author(s)	8 Performing Organization Report No
Philip O. Ransone, J. D. Morrison, and John E. Minster	L-12962
	10 Work Unit No
9 Performing Organization Name and Address	506-16-43-11
NASA Langley Research Center Hampton, VA 23665	11 Contract or Grant No
	13 Type of Report and Period Covered
12 Sponsoring Agency Name and Address	Technical Memorandum
National Aeronautics and Space Administration Washington, DC 20546	14 Sponsoring Agency Code

15 Supplementary Notes

Philip O. Ransone: Langley Research Center.
J. D. Morrison and John E. Minster: John F. Kennedy Space Center.

16 Abstract

Sample tiles of the Space Shuttle reusable surface insulation coated with reaction-cured glass have been subjected alternately to simulated mission heating and either real or simulated environmental exposure for up to 34 cycles. The coating cracked as a result of exposure to high temperature and moisture conditions, and insulation with cracked coatings absorbed significant quantities of water in the launch-pad environment. Cracking was a complex function of time, temperature, and moisture exposure. Cracked coatings remained adherent to the insulation for up to 24 cycles past initial cracking.

17 Key Words (Suggested by Author(s))	18 Distribution Statement
Insulation Space Shuttle Environmental tests Glass coating	Unclassified - Unlimited Subject Category 27

19 Security Classif (of this report)	20 Security Classif (of this page)	21 No of Pages	22 Price*
Unclassified	Unclassified	20	$4.00

*For sale by the National Technical Information Service Springfield Virginia 22161

NASA-Langley, 1979

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and Space Administration

**Scientific and Technical
Information Branch**

1979

SUMMARY

Sample tiles of the Space Shuttle reusable surface insulation coated with reaction-cured glass have been subjected alternately to simulated mission heating and either real or simulated environmental exposure for up to 34 cycles. The coating cracked as a result of exposure to high temperature and moisture conditions, and insulation with cracked coatings absorbed significant quantities of water in the launch-pad environment. Cracking was a complex function of time, temperature, and moisture exposure. Cracked coatings remained adherent to the insulation for up to 24 cycles past initial cracking.

INTRODUCTION

About 500 m^2 of the Space Shuttle surface will be protected from reentry heating with high-temperature reusable surface insulation (HRSI) tiles. The HRSI system consists of reusable surface insulation (RSI) with an impervious glass coating. The RSI is a low-density rigidized fibrous silica material which requires a glass coating to provide a water-resistant aerodynamic surface with a high emittance. Development of a coating which can withstand up to 100 shuttle missions and prevent water absorption by the porous silica tiles has been a major technical problem. Although the RSI fibers are coated with a waterproofing agent during manufacture, the waterproofing burns out to a depth of about 2 cm during the first reentry. During subsequent launch-pad exposure, the RSI is vulnerable to moisture absorption if the glass coating is cracked.

Coatings developed previously for the HRSI were found to degrade seriously as a result of devitrification when exposed to the launch-pad and reentry environments (refs. 1 and 2). The current baseline coating, known as reaction-cured glass (RCG), was developed to alleviate this problem. This paper reports results of a test program designed to evaluate environmental effects (exposure to moisture, sea salt, sand, and simulated mission heating) on the RCG coating and the effects on the RSI if the coating cracks.

Names of manufacturers used in this report do not constitute an official endorsement of such manufacturers, either expressed or implied, by the National Aeronautics and Space Administration.

SPECIMENS AND MATERIALS

Lockheed Missiles and Space Company supplied specimens (tiles) of HRSI to NASA. These tiles, which were 15.25 cm square and 9.25 cm thick, were representative of Space Shuttle production tiles. A typical tile is shown in figure 1. The top surface and four sides of each tile were coated with RCG to produce a continuous coating over areas of the tile which are normally exposed to aerodynamic heating. The bottom surface, normally used for bonding the tile to the Space Shuttle skin, was uncoated. The coating on the four sides was terminated about 3 mm from the bottom edge to provide venting of the tiles when bonded in place. Each tile was waterproofed during manufacture by immersion in a hydrophobic agent which coated the individual RSI fibers.

Sixteen tiles were prepared for testing by bonding a 12.5 cm by 12.5 cm by 0.13 cm aluminum plate to the uncoated face with a silicone adhesive. The aluminum plates served as attachment points between specimens and exposure fixtures when required. Two tiles which were not bonded to aluminum plates were included for comparison with the bonded tiles in the event that bonding should introduce a thermal expansion problem. These tiles did not require fixtures.

TEST PROCEDURES

Pre-Test Inspection

Prior to testing, each tile was visually and microscopically examined for coating cracks. A stereomicroscope was used for the microscopic inspections. Specimen surfaces were wiped with alcohol to enhance crack visibility in both the visual and microscopic inspections. Each specimen received at least two independent visual and microscopic inspections by different persons before testing. Specimens were also inspected after each exposure to mission heating.

Testing

Table I summarizes test conditions for each specimen. The specimens were divided into three groups for environmental exposures. One group of eight specimens, two of which had cracked coatings at the outset of the program, was exposed to a launch-pad environment. A second group of eight specimens was exposed to a water dip and the laboratory environment. The third group consisting of two specimens was utilized as a control group exposed only to the laboratory environment. All specimens except the two which had cracked coatings from the outset were exposed alternately to the indicated environmental conditions and simulated mission heating. Selected specimens were withdrawn from the program after 12 and 25 cycles for evaluation of the cumulative effects of testing on both the RCG coating and the RSI.

Space Shuttle mission heating was simulated with the vacuum radiation heating apparatus shown in figure 2. The specimen temperature and pressure were controlled during simulation to produce profiles representative of those expected for Space Shuttle boost and reentry (fig. 3).

<u>Specimens exposed to launch-pad environment</u>.- Specimens 1 through 6, which had uncracked coatings at the outset, were subjected to 12 test cycles. Each cycle consisted of 2 weeks of exposure to the launch-pad environment at John F. Kennedy Space Center followed by simulated mission heating. The specimens were weighed and microscopically examined before and after each launch-pad exposure period. Specimens 7 and 8, which had cracked coatings at the outset, received two simulated mission heatings to remove the waterproofing from the

RSI fibers adjacent to the top surface. They were then exposed continuously to the launch-pad environment for 30 days with daily weighings but no further thermal exposure. At the end of the 30 days, these two specimens were microscopically examined.

Specimens exposed to water-dip environment.- Specimens 9 through 16, which had uncracked coatings at the outset, were subjected to as many as 34 test cycles. Each cycle consisted of exposure to simulated mission heating followed by a cooling period and then a 10-second submersion of the test face in deionized water. Specimens 9, 11, 12, 14, 15, and 16 were allowed to cool for at least 1 hour after heating before being water-dipped. They were typically allowed to stand for a week before repeating the cycle. Specimens 10 and 13, which did not have aluminum attachment plates, received a similar treatment except that they were allowed to cool for at least 12 hours after heating to avoid any possibility of thermal shock when water-dipped.

Specimens exposed to laboratory environment.- Specimens 17 and 18 were control tiles. They were exposed to test cycles consisting of simulated mission heating followed by about 1 week of exposure to the "dry" laboratory environment. The specimens were microscopically examined after each cycle. The intent of this test was to maintain the specimens in a relatively moisture-free environment. This was not completely achieved because the air-conditioning system, which dehumidified the air in the laboratory, was turned off during weekends.

Evaluation

Samples of the RCG coating and RSI fibers were removed from a tested tile for X-ray and microscopic examination. Solid samples of the RCG coating were examined with an X-ray diffractometer to observe the amount of cristobalite (a crystalline polymorph of silica) present. Both the top side of the RCG coating and the side next to the RSI were scanned. Powder samples of RSI scraped from the backside of the RCG coating were examined in the same manner. A nickel filtered Cu K_α radiation source was used with a 3° beam slit and a 0.02° detector slit. Cross sections of the coating-RSI interface were examined with a scanning electron microscope.

RESULTS AND DISCUSSION

Cracks in RCG Coating

Table I summarizes the condition of specimen coatings before, during, and after testing. Pre-test visual and microscopic examinations of the RCG coatings indicated that only the coatings on specimens 7 and 8 were cracked before testing. The cracks were extremely difficult to detect visually because of their proximity to corners and edges. The nature and location of the cracks suggested that they resulted from impact. These specimens were used for continuous launch-pad exposure after two thermal exposures to remove the waterproofing. Three specimens (numbers 14, 15, and 16) experienced unusually high weight gains immediately or very soon after the first thermal exposure.

Subsequent microscopic examination revealed impact damage at corners that had escaped detection in pre-test inspections.

Water absorption ultimately proved to be the best indication of the presence of cracks. Of the 16 tiles exposed to water (rain at the launch pad or water dip in the laboratory), 11 did not absorb water until after the first few thermal exposures. When absorption occurred, microscopic examination always verified that the coatings had cracked. This cracking was not the result of impact. Only 2 of the 11 initially uncracked specimens (numbers 9 and 10 from the water-dip group) remained free of cracks at the time of this report. Specimen 9 was withdrawn after 25 cycles for evaluation. Specimen 10, one of the two tiles not having an aluminum attachment plate, is still being tested. Even though pre-test inspection indicated no cracks, specimens 17 and 18 (control specimens) could not be positively qualified as crack free before testing since they had not been exposed to water.

Specimens Exposed at Launch Pad

Specimens exposed to launch-pad environment and simulated mission heating.- Moisture absorption data for specimens 1 through 6, which were exposed to the launch-pad environment and mission heating simulation, are shown in figure 4. Weight gain is plotted in percent of dry specimen weight as a function of time in days. Each symbol corresponds to weight gain measured at the end of an atmospheric exposure period (2 weeks). Zero on the time scale corresponds to the beginning of the first launch-pad exposure. Thus, thermal exposures occurred on the time scale just after each symbol. Weight gains ranged from 0 to 66 percent. The 66-percent weight gain in specimen 1 (fig. 4(a)) resulted from water entering through a 5-mm-diameter hole caused by impact. The damage occurred during the third launch-pad exposure after initial cracking of the RCG coating.

Specimens 1, 2, 3, and 4 behaved almost identically with absorption occurring after about eight cycles (figs. 4(a), (b), (c), and (d)). In each case, microscopic examination verified that cracking had occurred. The coating on specimen 5 cracked during, or immediately after, the third thermal exposure and subsequent water absorption was quite significant (fig. 4(e)). The coating on specimen 6 (fig. 4(f)) cracked during, or immediately after, the 10th thermal exposure as determined by microscopic examination. This specimen subsequently incurred a 43-percent increase in weight due to water absorption. It had intermittently absorbed much lesser amounts of water during the first 10 cycles because of a small open flaw in the top-face coating.

No correlation was found between water absorption in the launch-pad specimens and amounts of daily rainfall. This suggests that absorption was not a direct function of rain rate, but more likely a function of the duration of wetness.

Specimens exposed to launch-pad environment without simulated mission heating.- Figure 5 shows water absorption data for specimens 7 and 8, which were exposed continuously to only the launch-pad environment after two thermal exposures to remove waterproofing. Each symbol represents the accumulated moisture in percent of dry specimen weight versus number of exposure days.

Weight gains of 32 and 42 percent were recorded for these specimens at the first heavy rainfall. Water was absorbed through cracks that were extremely difficult to locate with the unaided eye without prior indications from the microscopic examinations.

Specimens Exposed to Water-Dip Environment

Water absorption data for specimens 9 through 16, which were exposed to simulated mission heating and water-dip cycles, are shown in figure 6. Weight gain, in percent of dry specimen weight, is shown as a function of time where zero on the time scale corresponds to the start of the launch-pad exposures. Each symbol corresponds to a weight gain measured after a water dip. The specimens were exposed to heating before the first water dip. On the average, the cycle was repeated weekly for specimens 9, 11, 12, 14, 15, and 16. Exposure of specimens 10 and 13 was begun late, and therefore these specimens received simulated mission heating cycles more frequently until the number of cycles approximately coincided with the remainder of the group.

Specimens 9 and 10 (figs. 6(a) and (b)) absorbed no water, and no cracks were detected in the coatings during microscopic examinations. Specimens 11, 12, and 13 (figs. 6(c), (d), and (e)) absorbed no water until after 12 to 16 cycles of exposure. After water absorption occurred, cracks were observed during microscopic examinations. These data confirmed the pre-test microscopic examinations which indicated that the coatings of these specimens were not cracked at the outset of the program.

Specimens 14 and 15 (figs. 6(f) and (g)) absorbed water from the outset, and specimen 16 (fig. 6(h)) absorbed water after 1 cycle. These results indicate that coating cracks were present but were not detected in the pre-test microscopic examinations. The weight gains stabilized at 2.5 to 3 percent after 4 to 6 cycles. Specimen 15, which was the only one of these three exposed to more than 12 cycles, experienced a sudden increase in weight gain after 15 cycles. The time and number of cycles at which this increase occurred compare reasonably with the time and number of cycles at which specimens 11, 12, and 13 began to gain weight. A similar comparison between figures 4 and 6 shows that cracking occurred on launch-pad specimens at about the same time as on water-dip specimens even though the water-dip specimens had been exposed to twice as many cycles. This indicates that cracking was not a function of the number of thermal cycles alone but rather a function of temperature exposure, moisture conditions, and total exposure time.

Specimens Exposed to Laboratory Environment

Specimens 17 and 18 were exposed only to the "dry" laboratory atmosphere and simulated mission heating cycles. No weight data were recorded for these specimens because weight changes due to absorption of atmospheric moisture from the air-conditioned laboratory were expected to be negligible. However, these specimens were exposed to some atmospheric moisture because of periods of high relative humidity in the laboratory. The high humidity resulted because the laboratory air-conditioning system was not continuously in operation. Cracks in the coating were visually and/or microscopically detected after 13 cycles for specimen 17 and 8 cycles for specimen 18.

Post-Test Evaluations

No effect was observed of stresses associated with the bonding of aluminum plates to the specimens nor of thermal shock from water-dipping too soon after heating. Water absorption was always a reliable indicator of cracked coatings. All coating cracks detected on the specimens in the test program allowed absorption of measurable amounts of water.

X-ray diffraction scans made on coating and RSI samples taken from specimen 15 after 25 cycles showed no cristobalite. When coating samples were removed from this tile, the coating was still strongly adherent to the RSI, even though it contained numerous cracks and had been cracked during 24 of the 25 cycles. Scanning electron micrographs of cross sections showed no significant degradation at the coating-RSI interfaces (fig. 7).

The fact that the cracked RCG coating remained adherent to the RSI, combined with the fact that cracked specimens do not absorb moisture before the hydrophobic agent has been removed, suggests that the useful life of the HSRI system could be extended by replenishment of the waterproofing agent after each flight.

CONCLUSIONS

Sample tiles of the Space Shuttle high-temperature reusable surface insulation (HRSI) were subjected alternately to simulated mission heating and either real or simulated moisture exposure for up to 34 cycles. The following conclusions resulted from this investigation:

1. The reaction-cured glass (RCG) coating cracked on most of the sample tiles during the testing cycles. In most cases cracking was not a result of impact but rather the result of complex interactions between time, temperature, and exposure to moisture.

2. All specimens with cracked coatings absorbed measurable quantities of water after the waterproofing agent had burned out of the top 2 cm of RSI. In fact, significant quantities of water were absorbed by the RSI through cracks in the coating that escaped detection during microscopic or macroscopic visual inspections. The best indication that a coating was not cracked was the absence of weight gain upon exposure to water after burnout of the waterproofing agent.

3. The cracked RCG coating remained strongly adherent to the RSI for up to 24 cycles of exposure to water and mission heating after initial cracking. Examination of the coating-RSI interface with an X-ray diffractometer and a scanning electron microscope revealed no degradation in coating or RSI morphology after 25 cycles of exposure to water and mission heating.

4. Treatment of the HRSI with a waterproofing material after each flight may be a viable means of extending the life of tiles with cracked coatings.

Langley Research Center
National Aeronautics and Space Administration
Hampton, VA 23665
June 13, 1979

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1. Ransone, Philip O.; and Morrison, J. D.: The Effects of Environmental Exposure on Reusable Surface Insulation for Space Shuttle. NASA TM X-3252, 1975.

2. Ransone, Philip O.: Effect of Silicon Carbide on Devitrification of a Glass Coating for Reusable Surface Insulation. NASA TM-78755, 1978.

TABLE I.- TEST SPECIMENS

Specimen number	Exposure conditions				Number of exposure cycles	Coating condition		Number of cycles to cracking	Vendor's serial number
	Launch pad	Water dip	Control	Heating		Before test	After test		
1	X			X	12	Uncracked	Cracked	[a]8	8188
2	X			X	12	Uncracked	Cracked	8	8207
3	X			X	12	Uncracked	Cracked	8	8239
4	X			X	12	Uncracked	Cracked	8	8242
5	X			X	12	Uncracked	Cracked	3	8240
6	X			X	12	Uncracked	Cracked	11	8211
7	X			(b)	---	Cracked	Cracked	0	50095
8	X			(b)	---	Cracked	Cracked	0	50099
9		X		X	[c]25	Uncracked	Uncracked	---	8262
[d]10		X		X	32	Uncracked	Uncracked	---	50093
11		X		X	34	Uncracked	Cracked	17	8252
12		X		X	34	Uncracked	Cracked	13	50048
[d]13		X		X	32	Uncracked	Cracked	17	8260
14		X		X	[c]12	Uncracked[e]	Cracked	1	8257
15		X		X	[c]25	Uncracked[e]	Cracked	1	8348
16		X		X	[c]12	Uncracked[e]	Cracked	2	50082
17		X	X	X	33	Uncracked[f]	Cracked	13	8243
18		X	X	X	33	Uncracked[f]	Cracked	8	8250

[a]Impact damage during 11th launch-pad exposure.
[b]Two heat cycles to remove waterproofing before 30-day launch-pad exposure.
[c]Withdrawn for evaluation after number of cycles shown.
[d]Specimens not bonded to aluminum attachment plates.
[e]No cracks found in pre-test inspection, but water absorption data indicate possible pre-test cracks.
[f]No cracks found in pre-test inspection; however, specimens may have had pre-test cracks, since they were not exposed to water to verify pre-test inspection.

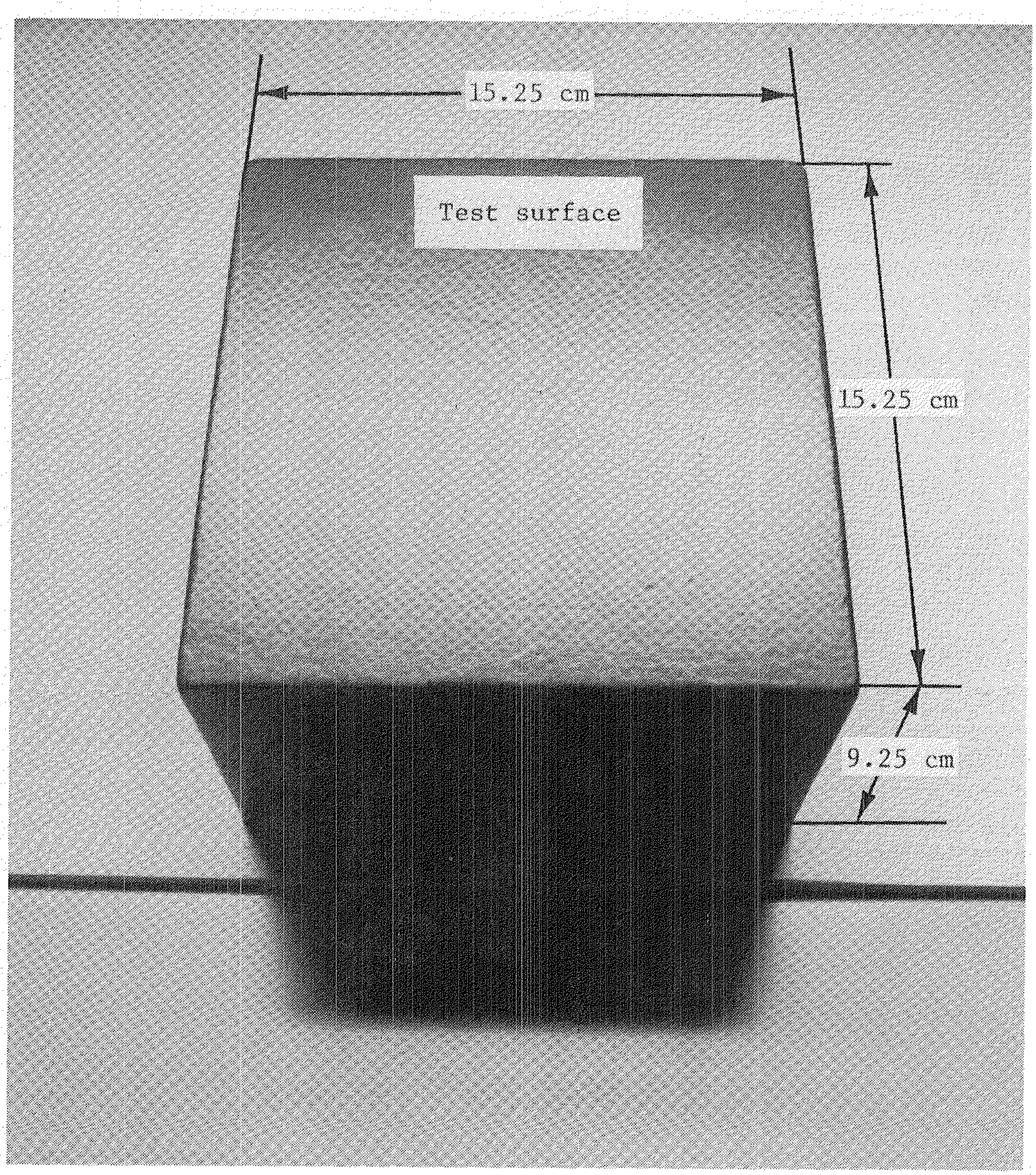

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Figure 1.- Typical test specimen.

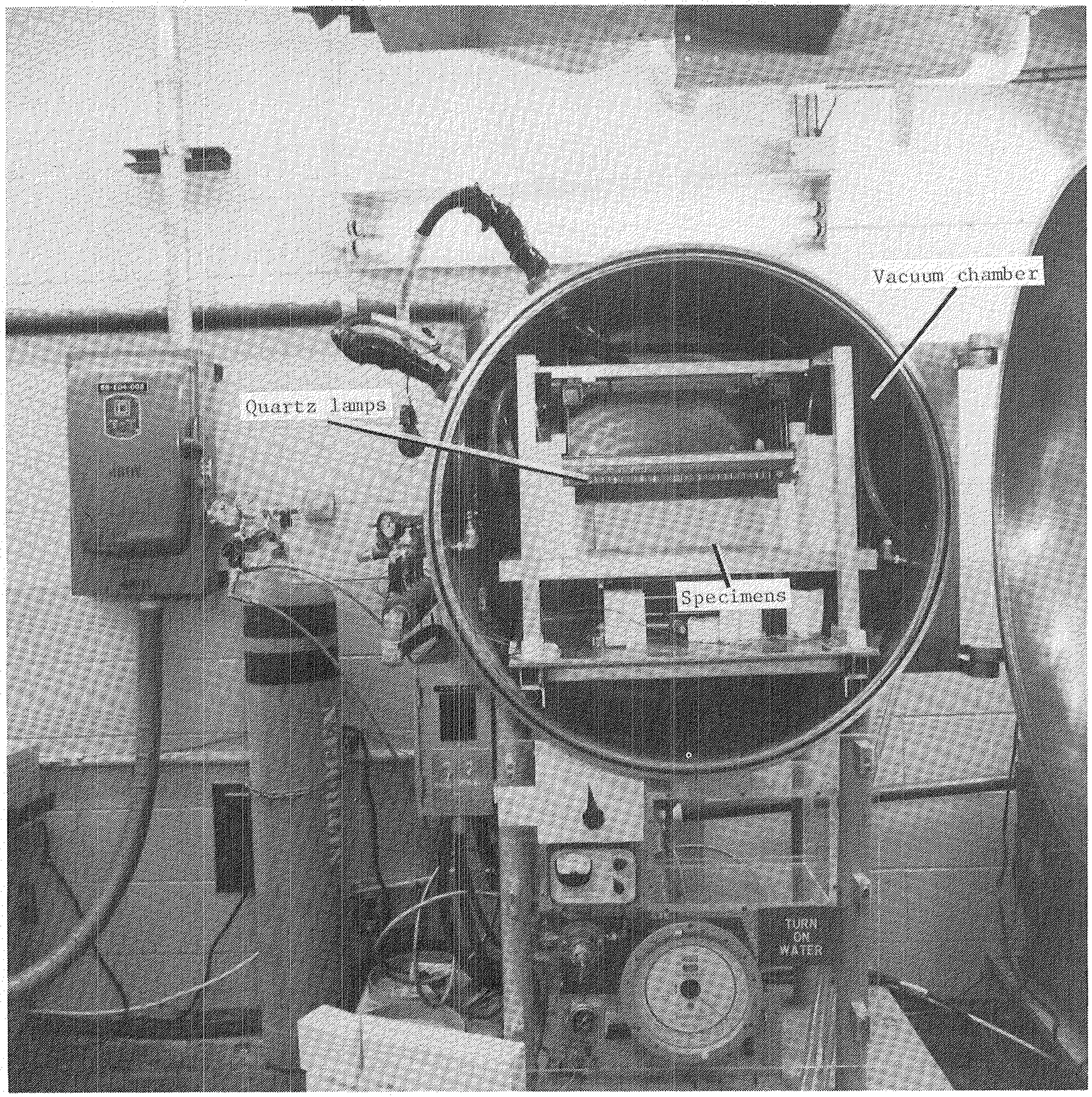

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Figure 2.- Vacuum radiant heating apparatus.

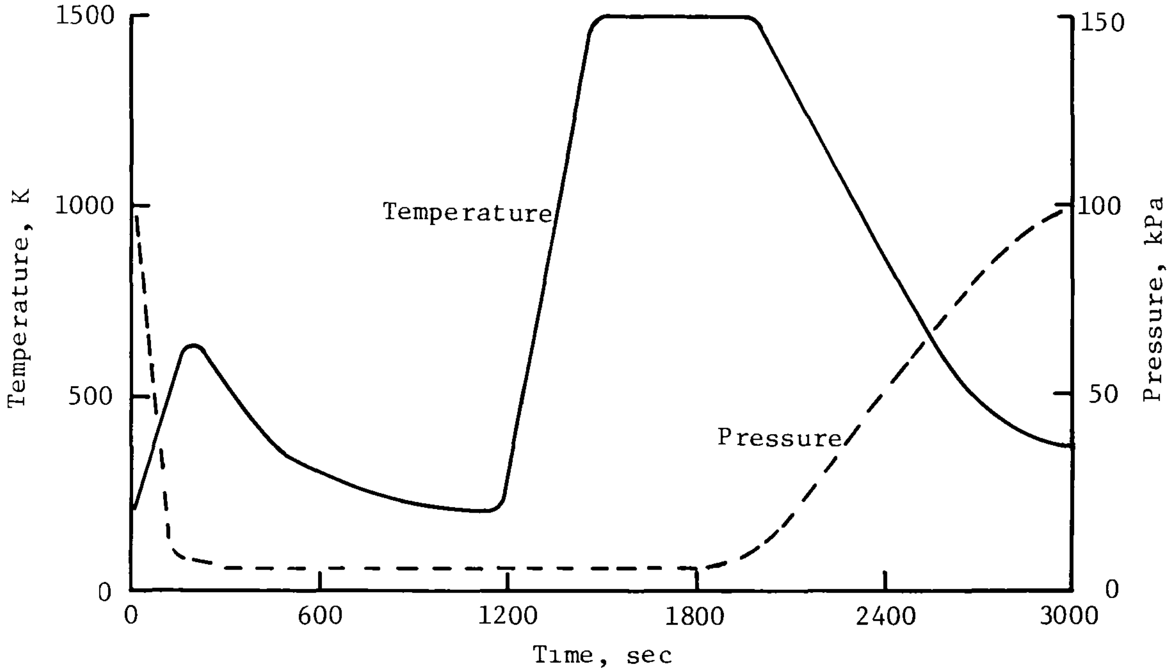
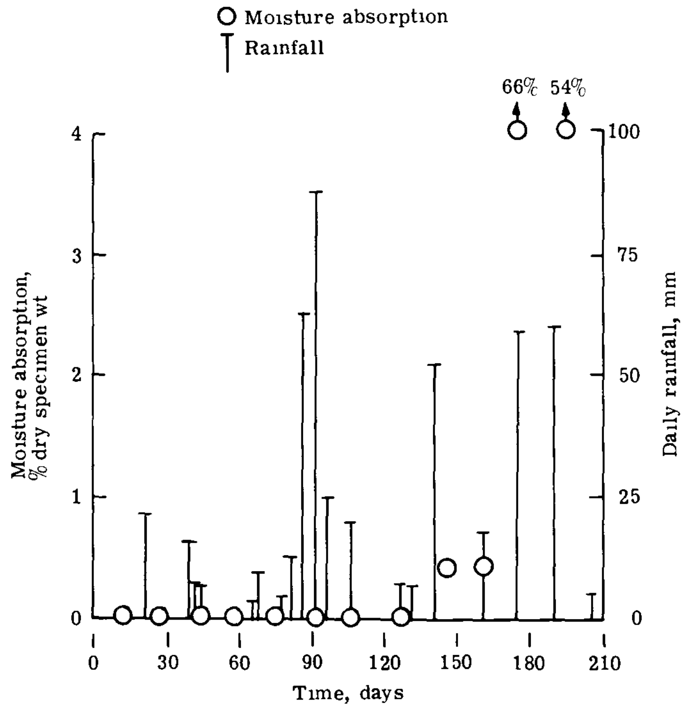
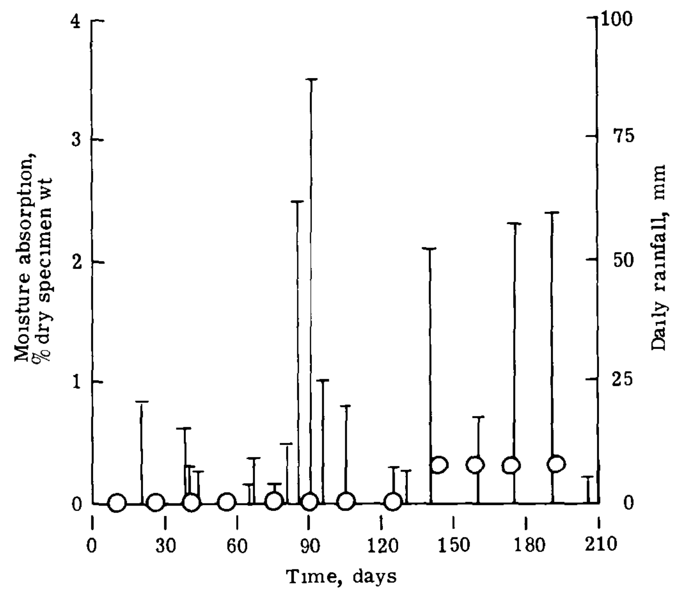


Figure 3.- Temperature and pressure profiles for mission simulation.

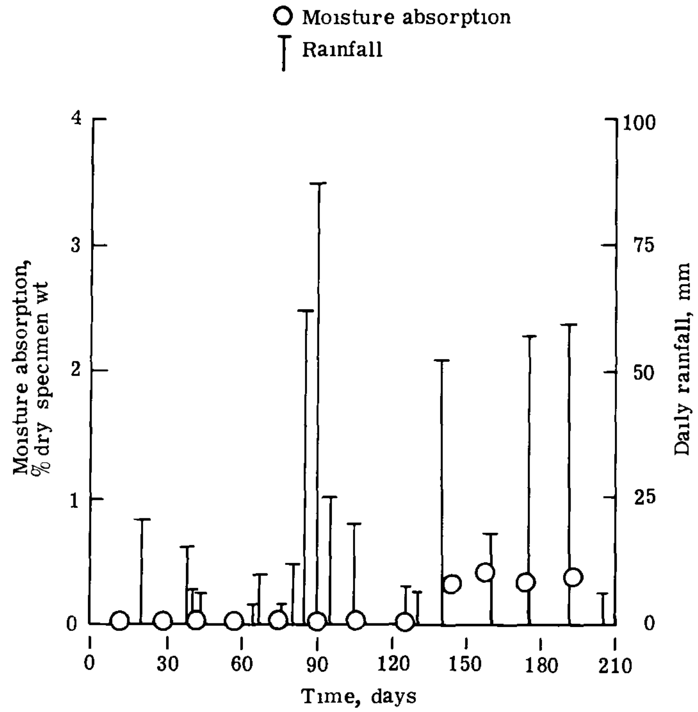


(a) Specimen 1.

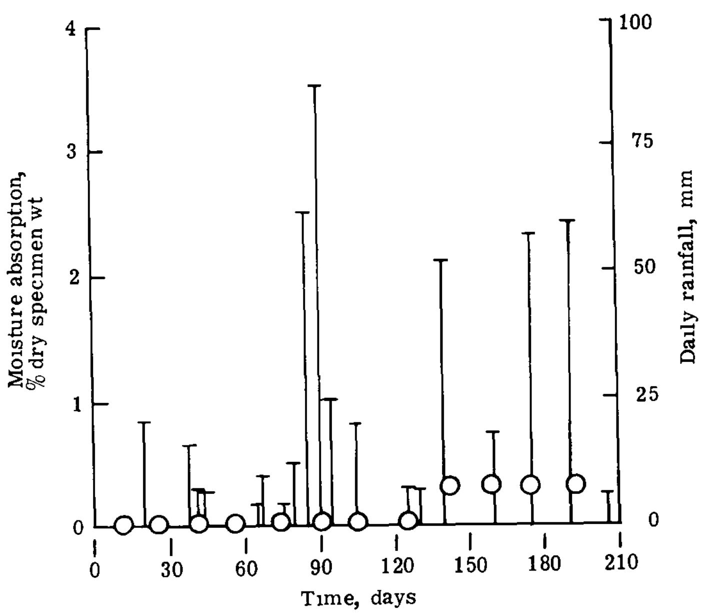


(b) Specimen 2.

Figure 4.- Moisture absorption and rainfall histories for launch-pad specimens.

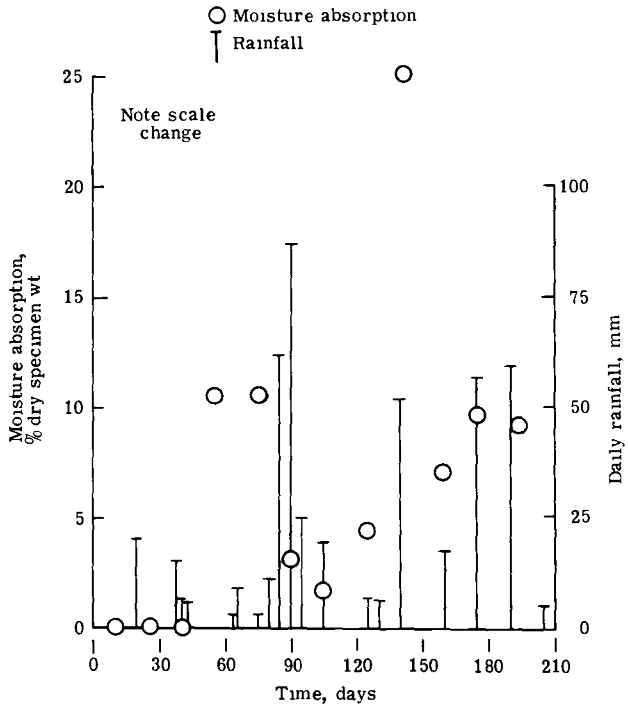


(c) Specimen 3.

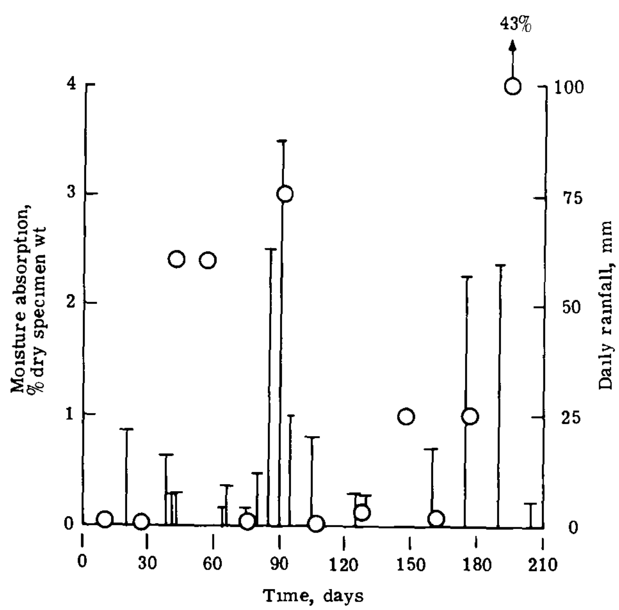


(d) Specimen 4.

Figure 4.- Continued.



(e) Specimen 5.



(f) Specimen 6.

Figure 4.- Concluded.

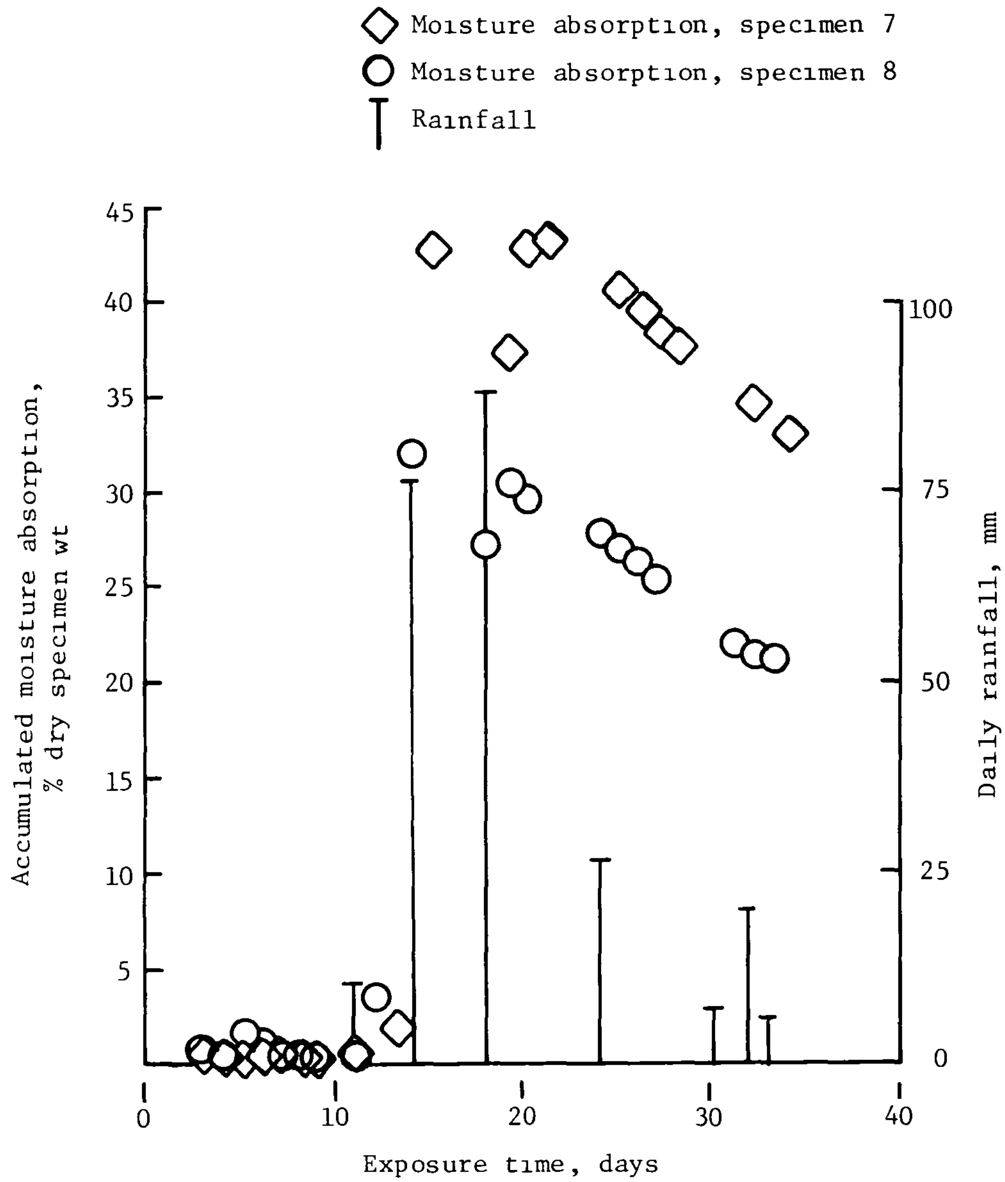
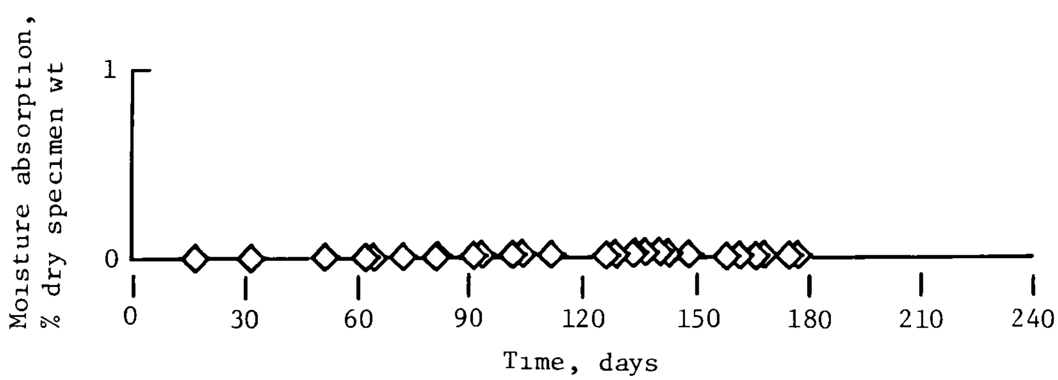
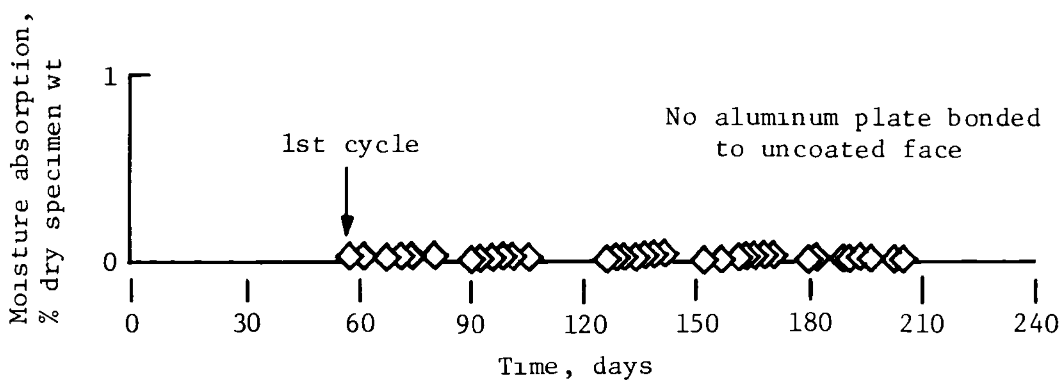


Figure 5.- Moisture absorption accumulated during continuous exposure at the launch pad.

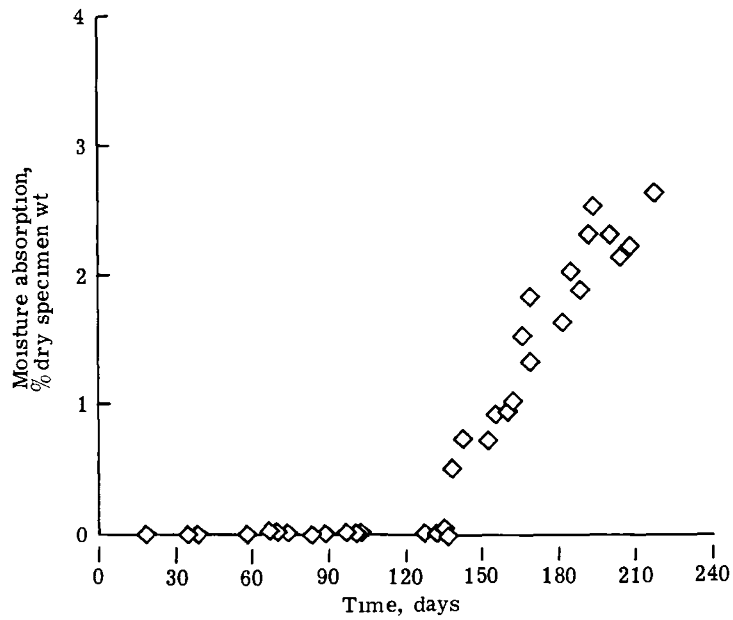


(a) Specimen 9.

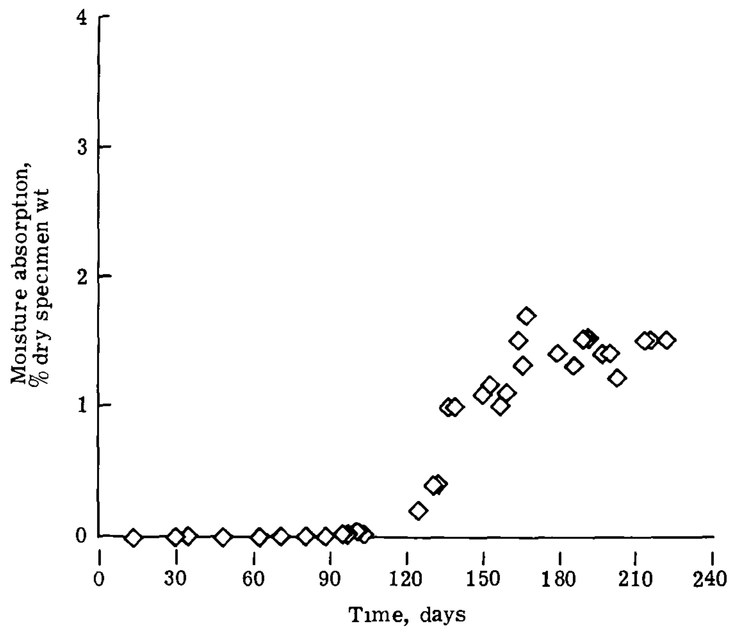


(b) Specimen 10.

Figure 6.- Moisture absorption histories for water-dip specimens.

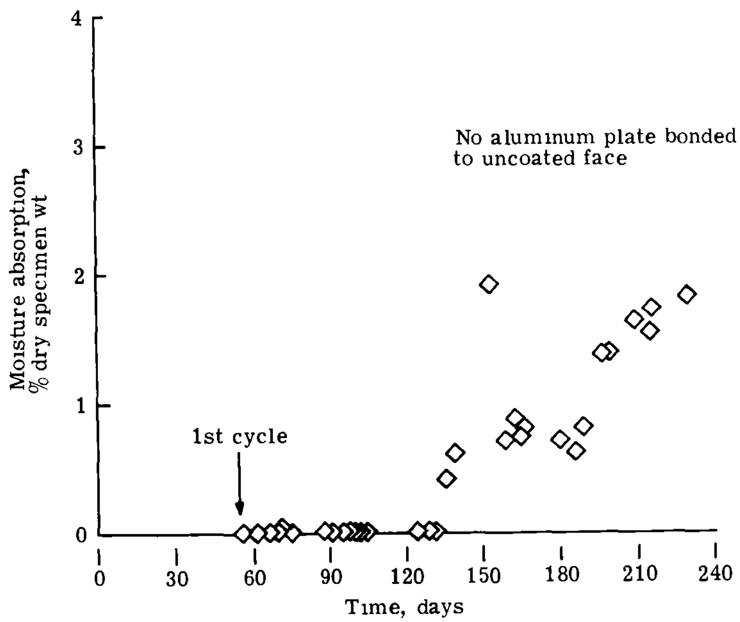


(c) Specimen 11.

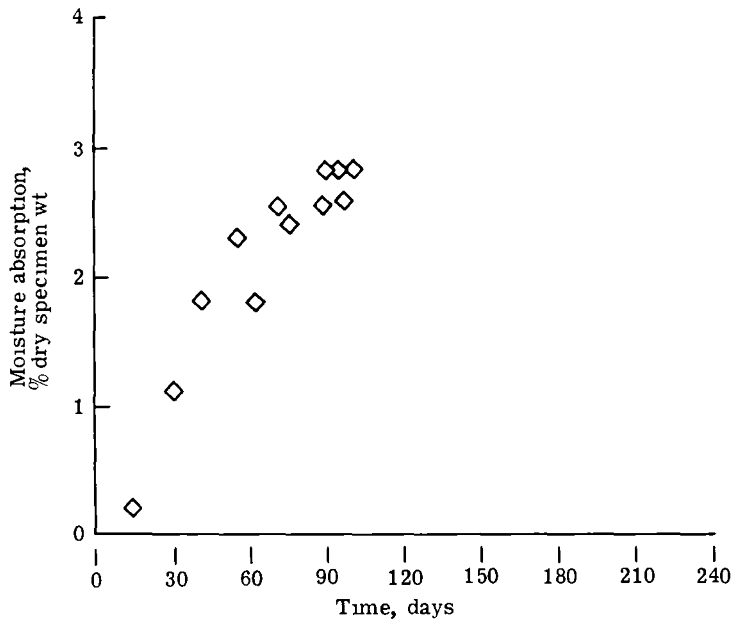


(d) Specimen 12.

Figure 6.- Continued.

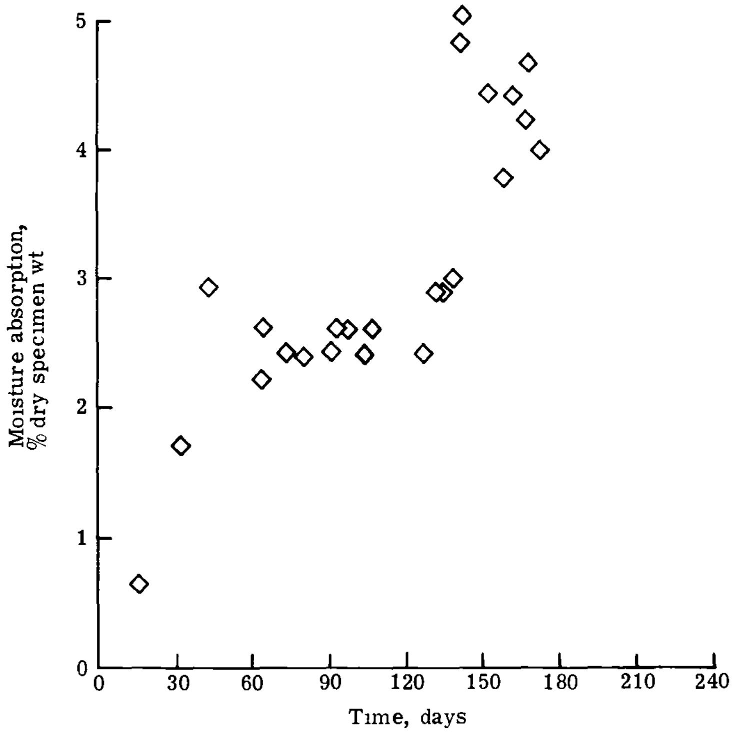


(e) Specimen 13.

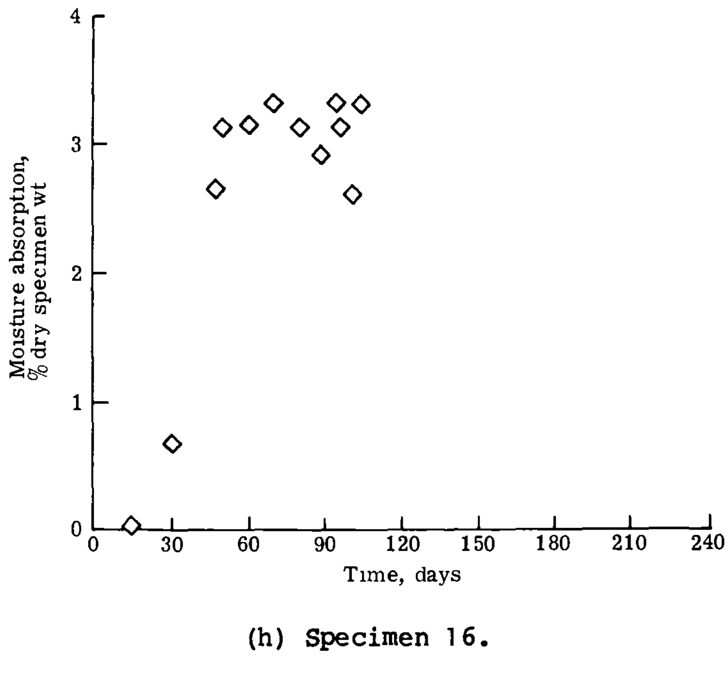


(f) Specimen 14.

Figure 6.- Continued.

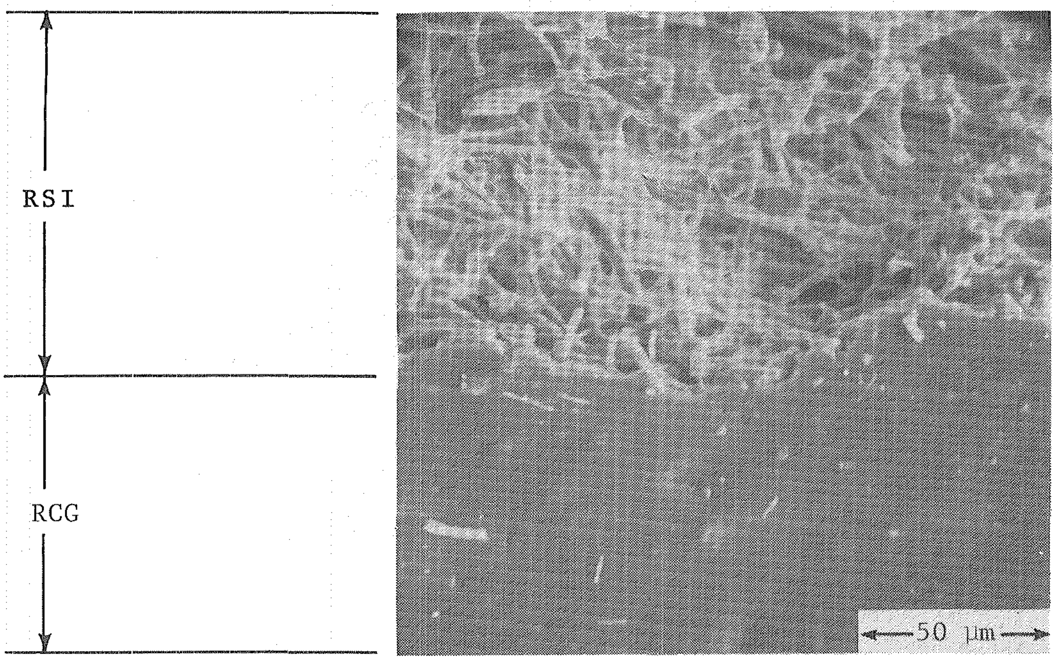


(g) Specimen 15.

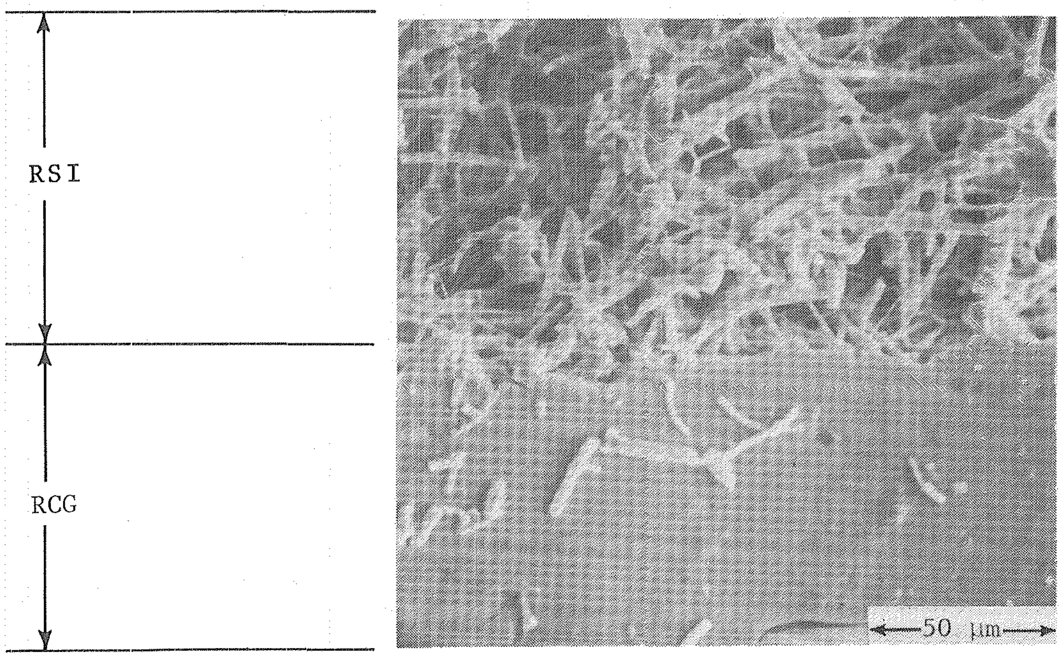


(h) Specimen 16.

Figure 6.- Concluded.



(a) As received.



(b) After 25 cycles.

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Figure 7.- Tile cross sections showing interface between reaction-cured glass (RCG) coating and reusable surface insulation (RSI) before and after test. Sections produced by fracturing coating.

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