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EFFECT OF 25 CYCLES OF LAUNCH PAD EXPOSURE  
AND SIMULATED MISSION HEATING ON SPACE SHUTTLE  
REUSABLE SURFACE INSULATION COATED WITH  
REACTION CURED GLASS

**FOR REFERENCE**

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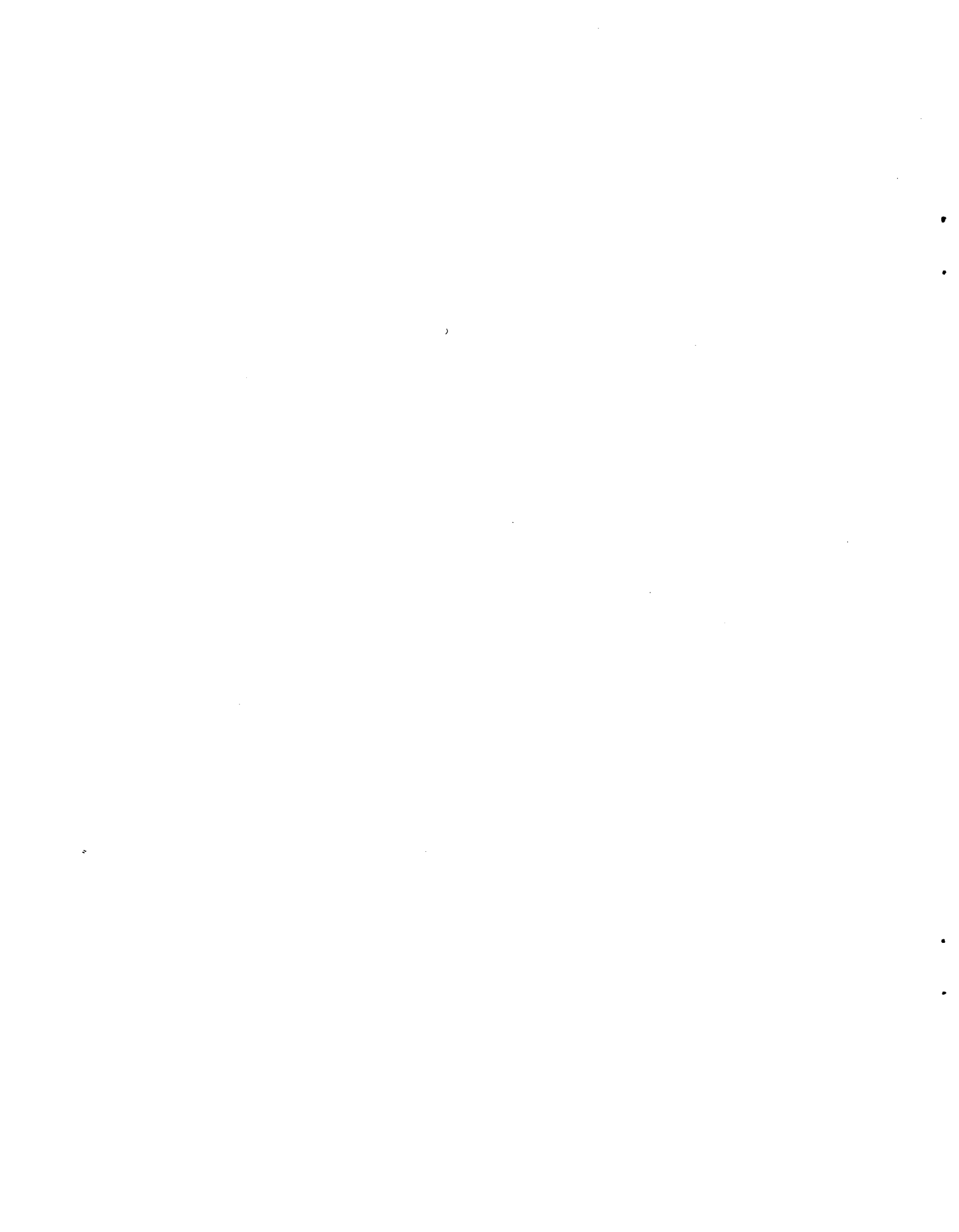
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SUMMARY

Four sample production tiles of the space shuttle reusable surface insulation (RSI) coated with reaction-cured glass have been alternately subjected to 25 cycles of launch pad exposure and simulated mission heating. The coatings cracked as a result of the combined exposures and the tiles absorbed up to 150 weight percent water in one cycle during the launch pad exposures. The cracked coatings remained adherent to the insulation but a cristobalite phase was observed in the insulation fibers at the coating/insulation interfaces of the tiles. Growth of the cristobalite phase in the interface may eventually result in separation of the coating from the RSI.

INTRODUCTION

About 500 square meters of the surface of the space shuttle orbiter will be protected from reentry heating with high-temperature reusable surface

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insulation (HRSI). This thermal protection system is composed of glass coated reusable surface insulation (RSI) tiles and a Nomex strain isolation system with silicone rubber adhesives. The RSI is a low-density fibrous silica material and requires an impervious glassy coating to provide a water-resistant aerodynamic surface with a high emittance. Development of a coating which will withstand up to 100 shuttle missions and prevent water absorption by the porous, hygroscopic silica tiles has been a major technical problem. Although the RSI fibers are treated with a waterproofing material during manufacture, the waterproofing will burn out to a depth of about 20 mm during the first reentry. If tiles have cracked coatings, they are vulnerable to moisture absorption during subsequent launch pad exposures.

Previous coatings developed for the HRSI were found to degrade seriously as a result of devitrification when exposed to the launch pad and reentry heating environments (refs. 1 and 2). The current baseline coating, designated as reaction cured glass (RCG), was developed to alleviate this problem. This paper reports results of tests on HRSI tiles (coated with RCG) which were subjected to 25 cycles of 2-week launch pad exposure plus simulated mission heating and includes any results concerning these tiles which were reported in reference 3 (ref. 3 covers results of the first 12 launch pad exposures and heating cycles including destructive evaluation of other tiles at the end of that period).

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

## SPECIMENS

The test specimens (figure 1) which were supplied to NASA by Lockheed Missiles and Space Company, were used in the test program described in reference 3. These specimens were assigned numbers 3, 4, 5 and 6 in reference 3 and will be so identified in this report to avoid confusion. Details concerning specimen fabrication and preparation for testing can also be found in reference 3.

## TEST PROCEDURES

### Pre-Test Inspection

Each specimen was examined visually with the aid of a low power stereo microscope to identify coating cracks before testing. Specimen surfaces were wiped with alcohol to enhance crack visibility during examination. Each specimen received at least two inspections by different persons before testing.

### Testing

The specimens were subjected to 25 test cycles. Each cycle consisted of a two-week exposure to the launch pad environment at Kennedy Space Center (KSC) followed by simulated mission heating. The specimens were weighed and examined after each launch pad exposure period. Simulated mission heating was accomplished using the vacuum radiant heating apparatus described in reference 3. The temperature and pressure profiles followed are shown in figure 2. The specimens were examined immediately after each simulated

mission heating exposure and were weighed to insure that all moisture was removed before starting the next launch pad exposure.

### Evaluation

Samples of coating and RSI were removed from specimens 5 and 6 for X-ray and laboratory examination after 25 exposure cycles (specimens 3 and 4 will not be destructively evaluated until 50 cycles are completed). Solid samples of coating were examined using an X-ray diffractometer to identify cristobalite and other crystalline phases which may form as a result of the reentry heating. Nickel-filtered  $\text{Cu}/\text{K}\alpha$  radiation was used in conjunction with a graphite crystal monochromator. Both the top face of the coating and the coating/RSI interface were examined. Each surface was alternately etched with hydrofluoric acid and scanned in the X-ray diffractometer to determine the extent of cristobalite penetration into the coating. Powder samples of RSI scrapings from the coating/RSI interface were also examined. Surface deposits were removed from the coatings with cellophane tape and were also examined for indications of cristobalite formation. Cross sections of coatings were examined in a scanning electron microscope (SEM).

### RESULTS AND DISCUSSION

Four HRSI tiles have been subjected to 25 cycles of launch pad exposure and simulated mission heating as a part of the test program described in reference 3. Two of the tiles (specimens 5 and 6, ref. 3) have been destructively evaluated after completing 25 exposure cycles.

The coatings on all four tiles cracked during the exposure tests. Specimens 3 and 4 cracked after 8 cycles while specimens 5 and 6 cracked after 3 and 11 cycles, respectively. Photographs taken of specimens 5 and 6 after 25 exposure cycles are shown in figure 3. Visibility of the cracks has been enhanced by the accumulation of surface deposits. The photographs show that the top face of specimen 6 has a much greater number of cracks and a greater total crack length than specimen 5. All cracks on the sides of the specimens are associated directly with top-face cracks and none of the side-face cracks run to the coating termination line near the bottom of the specimens.

#### Moisture Absorption

Moisture absorption and rainfall data for the launch pad exposures of the specimens are presented in table 1. Moisture absorption was related to amounts of rainfall after cracking had proceeded significantly. Weight gains for specimens 3 and 4 were comparatively insignificant before cycle 21. Maximum weight gain of about 30% for both specimens occurred during cycle 23. Large weight gains were recorded for specimen 5 during cycles 4, 5, 9, and 17 through 25. Large weight gains were recorded for specimen 6 during cycles 12, and 16 through 25. Maximum weight gains of 84 percent for specimen 5 and 153 percent for specimen 6 occurred during cycle 20. After the coating on specimens 5 and 6 had cracked, specimen 6 absorbed more water than specimen 5, probably because of greater crack length (see fig. 3). The differences in amount of water absorbed had an effect on the test results. This will be discussed in the following section.

## Post Test Evaluation

Evaluation of specimens 5 and 6 after 25 cycles indicated that the cracked coatings (see fig. 3) remained adherent to the RSI after the repeated exposures to water containing dissolved atmospheric contaminants (sea salts). Adherence appeared to be as good as that observed on 25-cycle specimens (with cracked coatings) that were exposed only to deionized water and simulated mission heating (specimens 9 and 15, ref. 3). SEM analyses revealed no significant degradation of the coating/RSI interface (fig. 4), however, X-ray diffraction studies showed that traces of cristobalite were present at the interface. The diffraction patterns in figure 5 were taken from the interface side of coating samples of both specimens. Reflections from the (101) plane of cristobalite are readily identified (the (101) reflection from a source with a high level of cristobalite is also shown for comparison). These patterns indicate that the amount of cristobalite on the coating was small but also reveal that the interface exposed to the most water (containing contaminants) had the greater amount of cristobalite. Even though no loss of adherence was apparent after 25 cycles, the presence of cristobalite suggests potential future problems. The amount of cristobalite phase may be expected to increase with further heating and may eventually cause disintegration of RSI fibers at the interface as a result of cristobalite's abrupt changes in volume when undergoing polymorphic transformations under cyclic heating conditions. Re-waterproofing of the tiles after each heating cycle might alleviate this problem by precluding contact of interface material with water containing dissolved atmospheric contaminants.



Photographs of a clean coating surface and a coating surface which has been exposed to 25 launch pad and heating cycles are shown in figure 6. The white deposit on the launch pad specimen resulted from the accumulation of fine sand on the surface during launch pad exposures and subsequent sintering of the sand to the coating during heating. Chemical analysis of fine sand samples from the launch pad area showed that the sand was composed mostly of quartz and calcium carbonate with traces of chlorides and titanium dioxide. Over the duration of 25 test cycles, the accumulated sand was converted mostly to glass but also contained a trace of cristobalite as indicated by the X-ray diffraction pattern (fig. 7) obtained from deposit samples.

Analyses showed that cristobalite was present on both sides of coating samples taken from specimens 5 and 6. The cristobalite was removed by a hydrofluoric acid etchant. Upon further examination, the interior of the coating was found to be free of cristobalite and other crystalline phases. During the etching procedure (alternate etching and X-ray scans), the complete disappearance of cristobalite from the interface side of the coating coincided with removal of the last traces of embedded RSI fibers. This would indicate that the cristobalite was concentrated in the vicinity of embedded fibers and probably originated at the fiber sites, but does not exclude the possibility that some cristobalite growth may have occurred in a very thin layer of coating at the interface. The RCG coating may have a layer up to 0.05 mm thick on both faces which is deficient of boron silicide. This would make these surface layers slightly less resistant to devitrification than the main body of the coating. A similar analysis of the outer surface of the coating indicated that cristobalite on the surface was primarily associated

with deposit material. This is possible because the deposit material has contaminated the coating surface with ions of sodium, calcium, magnesium and potassium derived from sea salt and sea shells. These ions, when introduced into a silica glass, break silicon-oxygen bonds, thus weakening the structure. The softening temperature is reduced, the thermal expansion coefficient is increased, and the activation energy for diffusion is reduced. The net result would be increased chemical activity in the vicinity of the deposited material. Therefore, conditions would be favorable for cristobalite growth to preferentially consume coating material in the vicinity of the deposit whether nucleated from the deposit material or from the boron silicide-deficient layer of the coating.

Microscopic examination of the coating surface showed that the coating was pitted directly beneath islands of deposited material. The SEM photograph in figure 8 illustrates such pitting. The reason for pitting is not clearly understood but may be attributed, to some degree, to the growth of cristobalite into the coating surface as described above. Additionally, pitting could occur because of the localized effect of deposit material on coating properties such as softening point and thermal expansion coefficient. During maximum heating, softened glass under the deposit can readily wet the deposit particles at points of contact. Upon cooling, the differential thermal expansion from coating to particles can cause local fracture, creating a new interface between coating and deposit material. Thus, cyclic heating and cooling can cause this interface to advance into the coating. The presence of cristobalite in the region would further increase the probability of fracture during cyclic heating because of the volume changes that occur in cristobalite crystals as they transform from one polymorphic form to another.

## CONCLUSIONS

High temperature reusable surface insulation (HRSI) tiles for space shuttle were subjected to 25 cycles of exposure to the Kennedy Space Center environment, with each cycle followed by simulated mission heating. The following conclusions resulted from analyses of the specimens:

1. Moisture absorption in HRSI tiles with cracked coatings increased with the number of launch pad exposures and simulated mission heating cycles and was increasingly related to rainfall as cracking proceeded.
2. RCG coating remained adherent to the RSI for 25 cycles of launch pad exposure and simulated mission heating even with cracks in the coatings for up to 23 of the test cycles.
3. Introduction of contaminants from the launch pad environment to the RSI/RCG interface region via water absorption caused nucleation of cristobalite phase in the interface RSI fibers. Growth of this phase may eventually result in separation of the coating from the RSI because of the thermal expansion characteristics of cristobalite.
4. RCG was inherently resistant to nucleation and growth of cristobalite. Formation of cristobalite at the surface of the specimens was related to environmental contaminants which promote nucleation.

## REFERENCES

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

2. Ransone, Philip O.: The Effect of Silicon Carbide on Devitrification of Coatings for the Space Shuttle Reusable Surface Insulation. NASA TMX 78755, 1978.
3. Ransone, Philip O., Morrison, J.D., and Minster, John E.: Environmental Tests on Space Shuttle Reusable Surface Insulation Coated with Reaction Cured Glass, NASA TM 80071, 1979.

TABLE I - SPECIMEN WEIGHT GAIN DURING LAUNCH PAD EXPOSURES

Exposure cycle	Rainfall during each 2-week exposure cycle (mm)	Weight gained each cycle (% of dry specimen weight)			
		Specimen 3	Specimen 4	Specimen 5	Specimen 6
1	0	0	0	0	0
2	21.6	0	0	0	0
3	22.9	0	0	0*	2.4
4	7.6	0	0	11.0	2.4
5	16.5	0	0	11.0	0
6	165.0	0	0	0.6	3.0
7	45.7	0	0	0.3	0
8	7.6	0*	0*	0.85	0.1
9	61.0	0.3	0.3	25.0	1.0
10	17.8	0.4	0.3	1.4	0.05
11	57.2	0.3	0.3	1.9	1.0*
12	63.5	0.4	0.3	1.8	43.0
13	25.4	0.3	0.3	0.2	3.0
14	0	0.2	0.1	0.1	0
15	0	0.2	0.1	0.1	0.2
16	17.5	0.1	0.1	0.1	18.0
17	69.9	0.3	0.3	13.0	58.0
18	not measured	0.2	0.2	19.0	23.0
19	137.2	0.3	0.3	19.0	112.0
20	139.7	0.3	0.3	84.0	153.0
21	38.1	9.4	23.7	13.0	40.0
22	38.1	13.6	16.6	18.0	42.0
23	40.6	30.0	34.6	35.0	42.0
24	0	5.0	9.1	15.7	20.6
25	not measured	5.4	7.1	16.0	21.0

\* coating cracked at cycle indicated

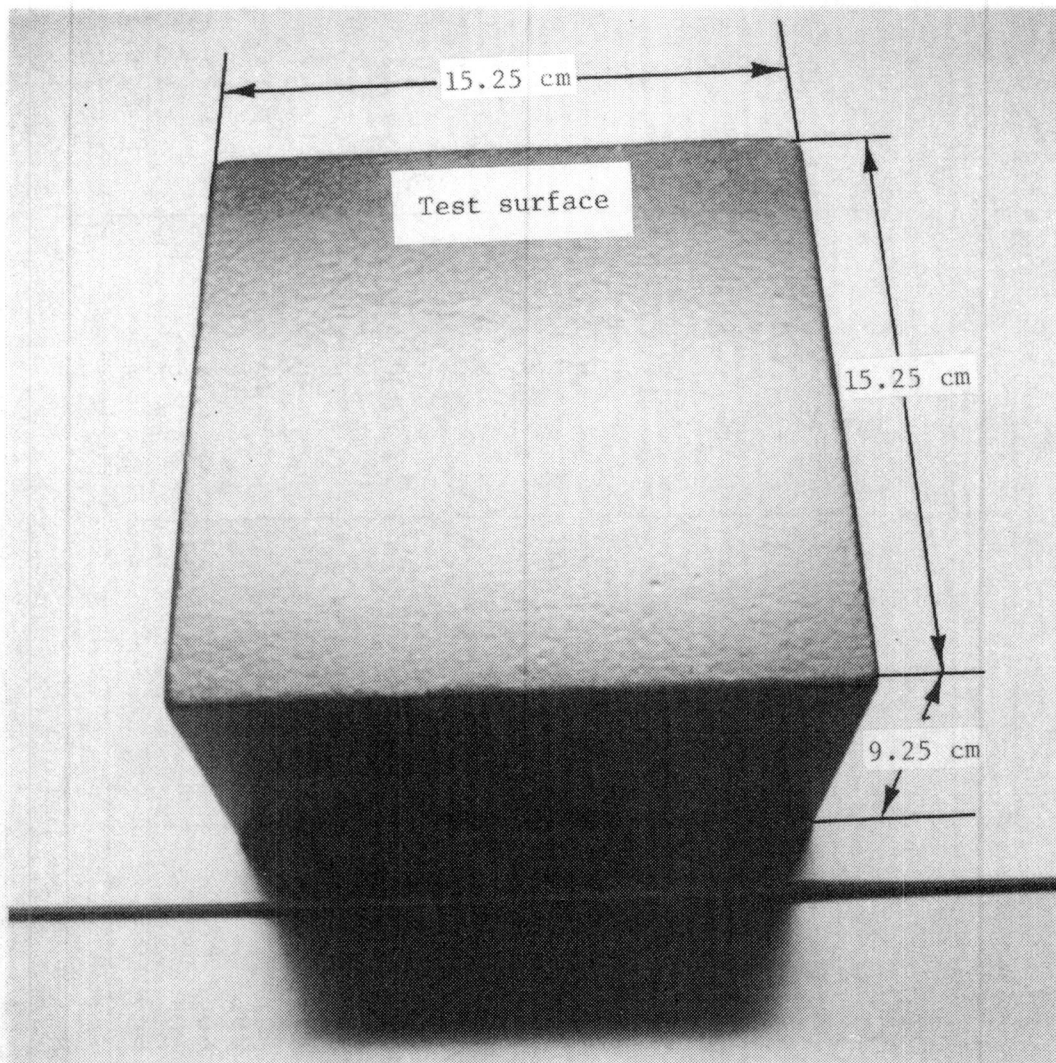


Figure 1. Test specimen - HRSI tile coated with reaction cured glass.

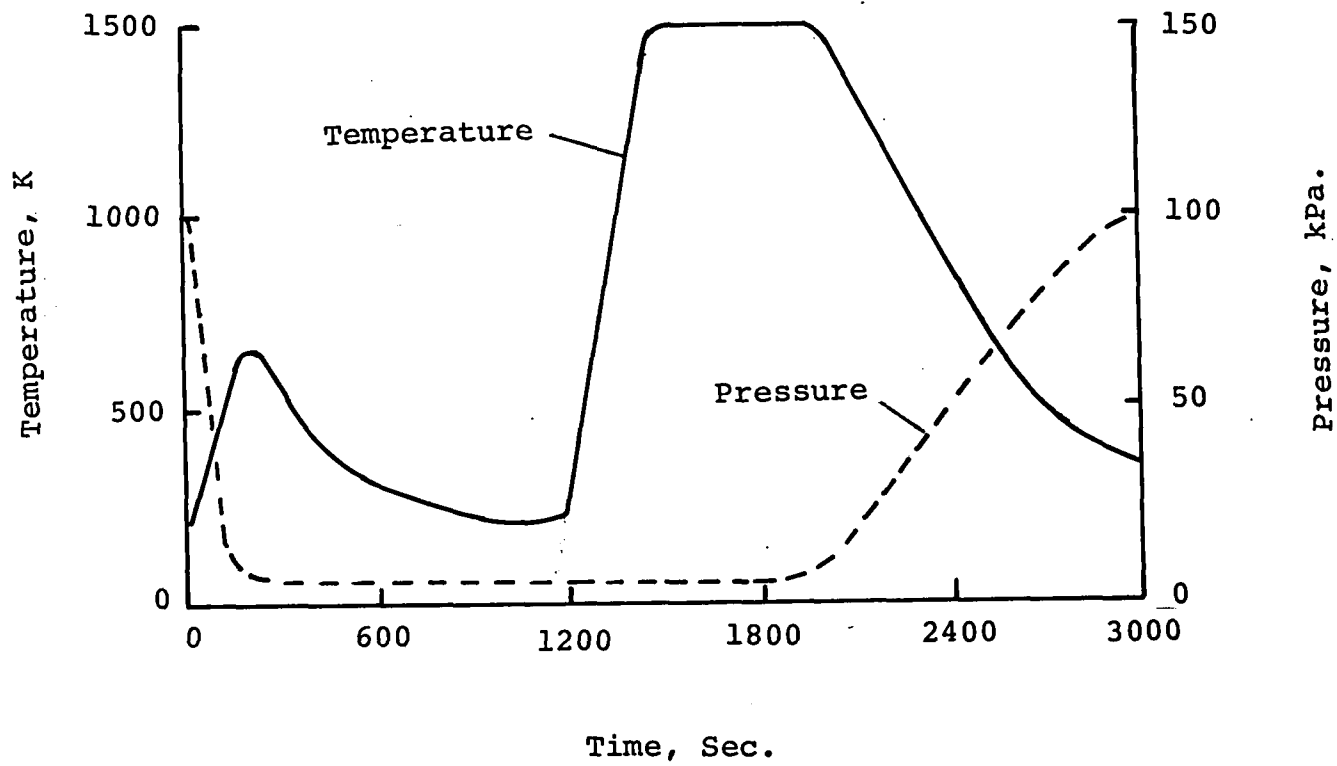
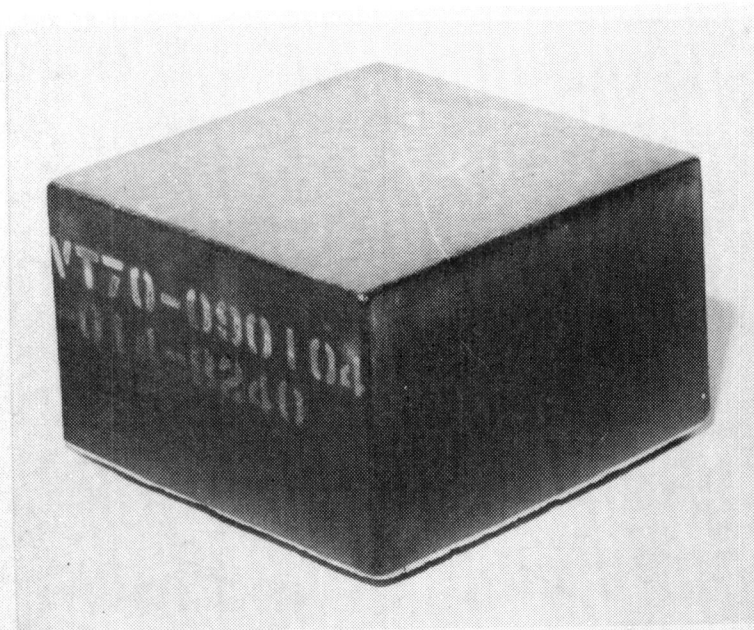
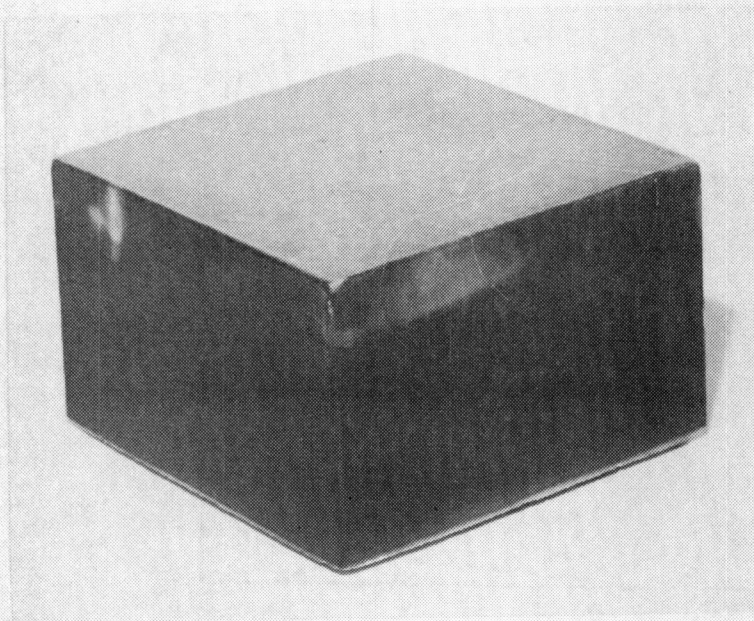


Figure 2. Temperature and pressure profiles for mission simulation.



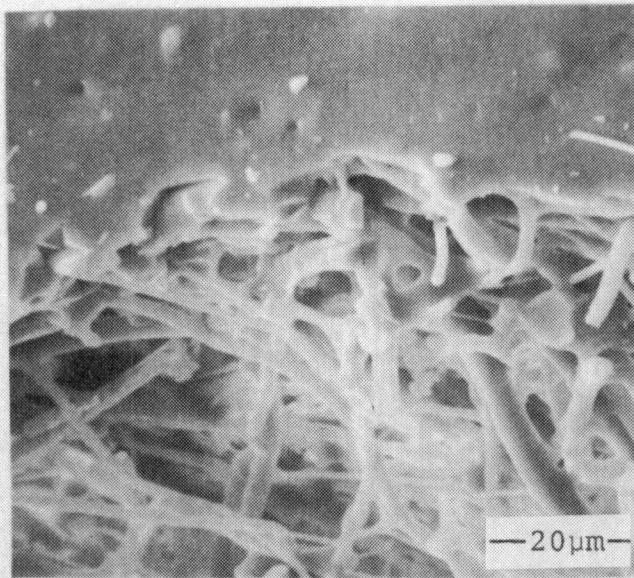
a. Specimen 5



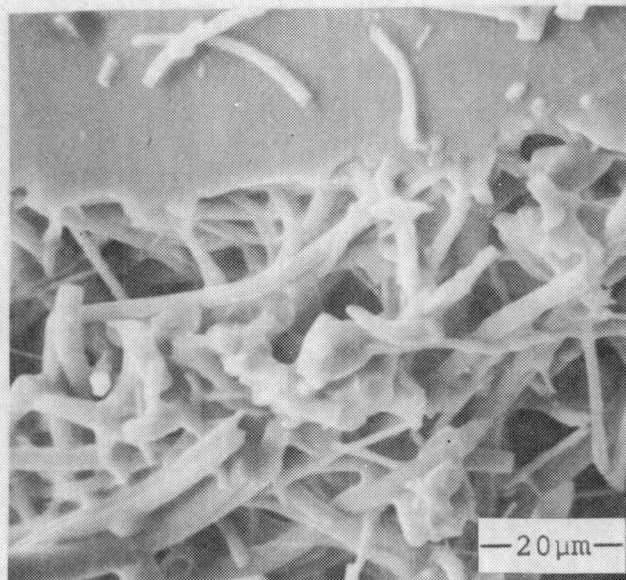
b. Specimen 6

Figure 3. Test specimens after 25 cycles exposure to launch pad environment and simulated mission heating.





a. As received



b. After 25 cycles

Figure 4. Fracture surfaces showing coating/RSI interfaces from as-received sample of HRSI and from specimen 6 after 25 cycles exposure to launch pad environment and simulated mission heating.

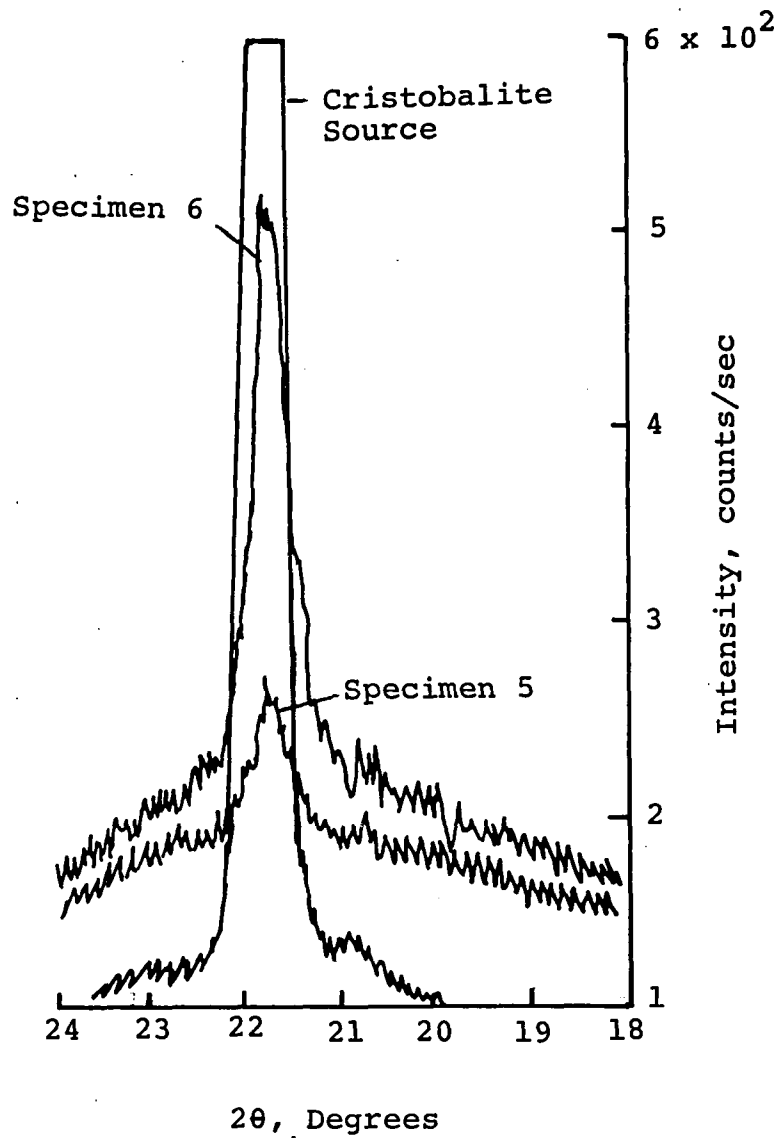
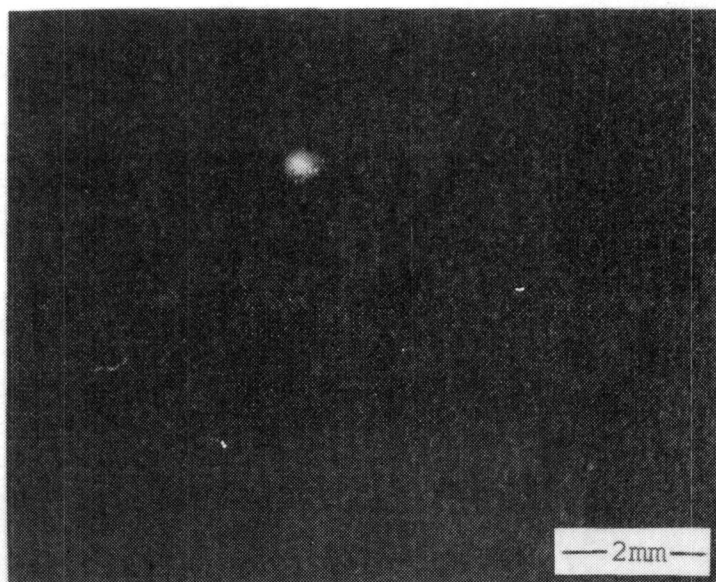
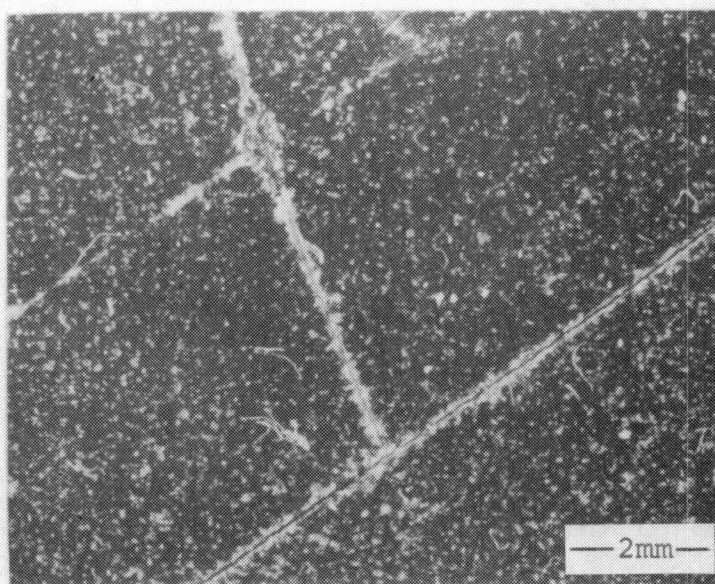


Figure 5. X-ray diffraction patterns from coating/RSI interface of each specimen after 25 cycles exposure to launch pad environment and simulated mission heating. Cristobalite source pattern shown for reference.



a. As received RCG coating



b. RCG coating on specimen 6  
after 25 test cycles

Figure 6. Comparison of clean, as received RCG coating with coating surface after 25 cycles exposure to launch pad environment and simulated mission heating.

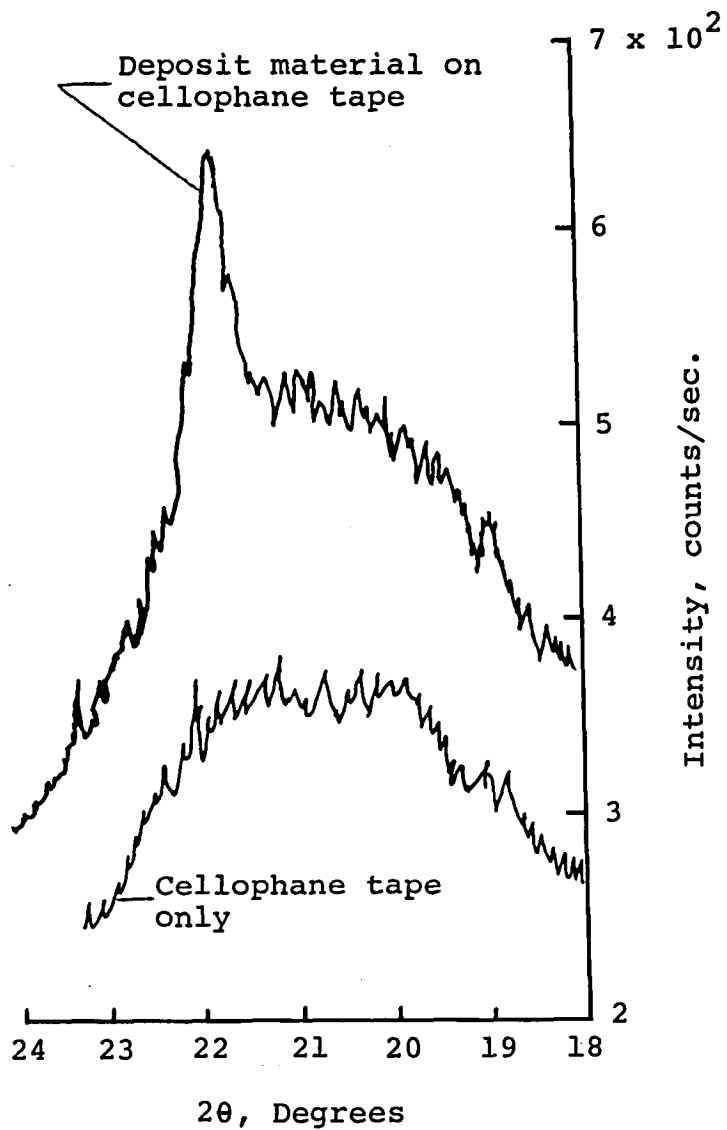


Figure 7. X-ray diffraction pattern from deposit material lifted from surface of Specimen 5 after 25 cycles exposure to launch pad environment and simulated mission heating. Cellophane tape pattern shown for reference.

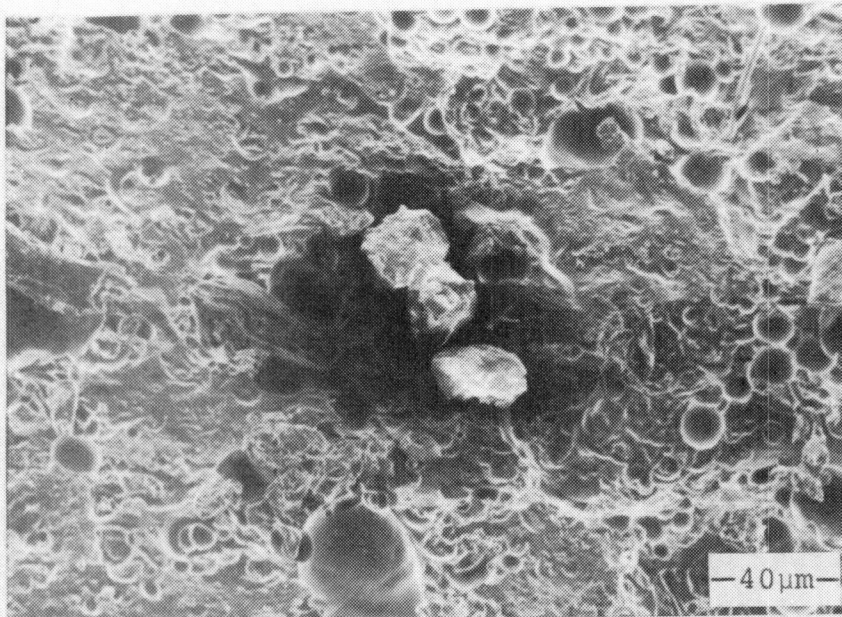


Figure 8. SEM photograph of etched RCG coating surface showing pit under undissolved deposit material.



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16. Abstract  Tiles of space shuttle reusable surface insulation coated with reaction cured glass have been subjected to 25 cycles of launchpad exposure and simulated mission heating. The coating could not withstand the environment without cracking. Water absorption after cracking reached as high as 150 weight percent. Exposure of insulation fibers beneath the coating to contaminants dissolved in absorbed water has initiated fiber degradation.					
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