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**THE EFFECTS OF ENVIRONMENTAL EXPOSURE
ON REUSABLE SURFACE INSULATION
FOR SPACE SHUTTLE**

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SUMMARY

Coated specimens of reusable surface insulation (RSI) were exposed to alternate radiant heating and atmospheric exposure cycles at the John F. Kennedy Space Center (KSC) to study the effects of surface contamination on the RSI coating. Different methods of heating were employed on clean and artificially contaminated specimens to determine the contributions of heating conditions to coating devitrification.

Launch-site exposures at KSC resulted in a high failure rate of specimens (70 percent). Two primary modes of failure occurred. One was characterized by eruptions of coatings and expulsion of coating fragments during the first five temperature cycles. The other was characterized by macrocracking of coatings accompanied by devitrification of coatings and shrinkage of substrate insulation. Eruption failures were attributed to the interaction of water with the coating during temperature cycles. Cracking failures were attributed to devitrification of coating combined with shrinkage of substrate insulation. Devitrification was found to result primarily from contamination with sea salt. The effects of heating atmospheres on the extent of devitrification were small when compared to the effect of sea salt contamination. Thus, radiant heating in air is a valid test method for studying devitrification in RSI coatings.

INTRODUCTION

The reusable surface insulation (RSI) for the space shuttle thermal protection system is coated with a silicate glass which is designed to serve three important functions. It must protect the system from liquid water and other contaminants, it is a carrier for the emittance agent which is required to produce a reradiative surface during convective heating, and it provides the mechanical integrity required for handling. The coating must be capable of withstanding up to 100 mission temperature cycles while maintaining these

characteristics. However, the coating begins to crack after only a few thermal cycles (ref. 1). This cracking is believed to be associated with devitrification of the coating to cristobalite, a crystalline polymorph of silica.

Devitrification in silica bodies is highly influenced by the presence of impurities – particularly, contaminant particles on the surfaces. The particles can act as nucleation sites from which crystals can grow when the temperature is sufficiently high. Contamination of RSI coating surfaces could create such nucleation sites.

Since the space shuttle will be based at John F. Kennedy Space Center (KSC), it will encounter exposures to the KSC atmosphere during prelaunch operations. The atmosphere in the vicinity of these operations frequently has high concentrations of sea salt. Laboratory tests in which an RSI coating was artificially contaminated with sea salt before temperature cycles showed significant increases in devitrification rates (ref. 1). Those findings led to this experimental program in which coated RSI specimens were exposed alternately to the KSC atmosphere and simulated reentry temperature cycles. Temperature simulation was accomplished using radiant heating with specimens exposed to the laboratory atmosphere. In some silica systems, oxygen and water vapor present in the air contribute to devitrification. Therefore, supplemental laboratory tests were conducted using various heating environments to ascertain the contribution of laboratory air and water vapor to devitrification. Results of these tests are also reported.

The data presented in this paper are from specimens tested from February 1973 to May 1974. The scheduled number of cycles for each specimen is shown in table I. Since this is an interim report, some of the conclusions may be modified when the test program is complete. It should be noted that, although the coating studied in this investigation is no longer being used for the shuttle, it is very similar to the current baseline formulation. Therefore, the results should be relevant.

MATERIALS AND SPECIMENS

The material tested in this investigation was silica RSI LI-0942, supplied by Lockheed Missiles and Space Company. LI-0942 consists of LI-0900 silica tiles with a LI-0042 coating. The coating is a silicate glass formulation containing a silicon carbide emittance pigment in the outer layer. The thickness of the coating is nominally 0.4 mm. The pigmented layer has a significant bubble structure, reportedly resulting from evolution of gases from oxidation of the silicon carbide during the initial firing process. A cross section of the coating is shown in figure 1.

Coated surfaces on some of the furnished tiles appeared to be underfired in that they were not well glazed. An example of these surfaces is shown in figure 2 with the surface in figure 2(a) considered normal and the surface in figure 2(b) showing little evidence of

having melted and flowed. The underfired coatings were observed only on the smaller of the two tile sizes supplied.

Two sizes of specimens with coating on five sides (fig. 3) were tested. The nominal dimensions of the specimens were 2.8 by 2.8 by 2.5 cm and 12.7 by 12.7 by 2.5 cm. A silicone adhesive was used to bond the small specimens to aluminum mounting pads and the large specimens to bakelite pads. Two of the small specimens were instrumented with Pt/Pt-0.13Rh thermocouples for use as temperature control samples in the radiant heating tests. The large specimens were not instrumented.

TEST PROCEDURES

Both launch-site tests and laboratory tests were conducted in this investigation. At the launch site (KSC), test specimens were subjected to radiant heating and environmental exposures. Other specimens were subjected to radiant heating without environmental exposure. Laboratory tests were conducted to study the effects of the test conditions on devitrification of the coating. In these tests, specimens were artificially contaminated with sea salt and subjected to three types of heating (radiant, vacuum furnace, and arc jet) which are described subsequently. Also, to study the effect of water vapor on devitrification, an uncontaminated specimen was subjected to radiant heating cycles after being soaked in deionized water.

Launch-Site Tests

Atmospheric exposures.- Twenty small specimens were attached to four aluminum holder plates with an equal number of specimens on each side of each plate. The plates were horizontally affixed to the exposure rack so that half the specimens would be shielded from rain and settling dust. Figure 4 shows the exposure rack with some of the specimens in place. The LI-0942 specimens are in the second row from the left. The other specimens shown are of other materials which were also being evaluated after KSC exposures, but which are of no current interest and are not considered in this report. Launch Complex 39B is seen in the background to the north. Specimens were arranged in 1-, 2-, and 4-week exposure groups on separate plates to facilitate handling. Specimen identification and exposure details for the LI-0942 specimens only are shown in table I.

Radiant heating tests.- After each atmospheric exposure period, the specimens were removed from the rack and placed indoors to dry overnight. Half of the specimens exposed for 2 weeks were rinsed with tap water after exposure to remove accumulated salts. No attempt was made to remove absorbed moisture from specimens prior to thermal exposures. The specimens were then subjected to a radiant heating cycle in the air. Six small specimens were subjected to radiant heat cycles with no atmospheric exposures to obtain baseline data for comparison with results from launch-site exposure specimens.

These specimens are listed in table II with other laboratory test specimens. Figure 5 presents surface temperature and pressure profiles for a space shuttle reentry simulation. The radiant heat cycle used resulted in the surface temperature profile shown by the solid line in figure 5(a). This profile is representative of the temperature history during reentry for the space shuttle lower wing and fuselage surfaces.

The heat source used was a radiant heater consisting of a 25- by 38-cm array of infrared quartz tube lamps (fig. 6). Up to seven specimens, including a control sample, could be heated simultaneously to a uniform surface temperature. The control sample thermocouple was connected to a feedback control system and programmer to regulate power to the lamps. Specimens were positioned under the lamps on a water-cooled aluminum plate. The RTV bonds between the tiles and pads were shielded from radiation by using a mask made from a commercial alumina-silica insulative felt.

Laboratory Tests

Artificial contamination.- One large and four small specimens were contaminated with measured amounts of sea salt by means of exposure to a sea-water fog for 1 hour. The fog apparatus used for the exposures is depicted in figure 7. The sea water was taken from the Atlantic Ocean 50 miles off the Virginia Coast. The weight of salt deposited on each specimen was arbitrarily chosen as 0.124 mg/cm^2 . This was found to be the weight of salt deposited per hour on a 2.54- by 2.54-cm glass slide placed in the apparatus with the selected operating conditions.

Heating tests.- The artificially contaminated specimens and an equal number of clean specimens were exposed to 12 consecutive cycles of the temperature profile shown in figure 5(a) by using the three heating methods described. The small specimens were exposed to radiant and vacuum furnace heating cycles while the large specimens were heated in an arc tunnel. Specimens and exposure details are listed in table II.

The radiant heating procedure was identical to that previously described for heating specimens after launch-site exposures. An identical heating apparatus was used (fig. 6).

For the vacuum furnace test, a vertical muffle-tube furnace depicted by the sketch in figure 8 was used. This apparatus was designed so that the temperature profile of figure 5(a) could be closely approximated by manually moving the specimen into and out of the furnace hot zone at a predetermined rate. At the same time, the pressure could be manually controlled to approximate the reentry pressure profile shown in figure 5(b). This was done by evacuating the muffle tube and then bleeding in air against the vacuum system at a preselected rate.

Convective heating tests were conducted in apparatus D of the Langley entry structures facility. This arc tunnel is described in reference 2. The specimens were mounted on a wedge-type holder (also described in ref. 2) for insertion into the arc-jet stream.

The holder could be positioned in the stream or off to the side by remote control. The tunnel conditions (table III) required to obtain the maximum surface temperature of figure 5(a) were established with the specimen out of the stream. While out of the stream, the specimen was preheated to 1273 K by use of an auxiliary radiant heater which was programmed to follow the temperature rise rate specified by figure 5(a). A calibration specimen placed near the test specimen and instrumented with a thermocouple was used to control the radiant heater. When a surface temperature reached 1273 K, the specimen was moved into the arc-jet stream. While in the stream, the specimen surface temperature was monitored with a radiation pyrometer which senses radiation in the range 2.0 to 2.6 μm . An emittance value of 0.65 was used to compute the temperature. After 800 seconds in the stream, the specimen was removed and allowed to cool according to the temperature profile of figure 5(a) by using the radiant heater to maintain the cooling rate. The deviation from the desired temperature profile for a given cycle is indicated by the dash-line curve in figure 5(a).

Materials Analysis

Launch-site test specimens.- Upon completion of exposure testing, X-ray diffraction scans were made on the top surfaces of specimens over the range of Bragg angles, $19^\circ \leq 2\theta \leq 40^\circ$. The radiation source was nickel filtered $\text{Cu K}\alpha$. Specimens showing strong cristobalite patterns were subjected to quantitative cristobalite determinations as follows. Powder samples were prepared by removing a sample of the coating from the top surfaces of the specimens. Each sample was ground with an alumina mortar and pestle, passed through a 400-mesh sieve, and thoroughly mixed with proportions of an internal standard (5 μm alumina). The samples were then scanned at a 2θ rate of $0.4^\circ/\text{min}$ over the appropriate range of Bragg angles using $\text{Cu K}\alpha$ radiation. Weight fractions of cristobalite were determined from the diffraction data using the internal standard quantitative X-ray diffraction technique (ref. 3).

Coatings were examined before and after testing by using optical and scanning electron microscope techniques. Photomicrographs and photographs were made of significant features.

Laboratory test specimens.- After 12 temperature cycles, coating samples were taken from the top surfaces of each specimen for quantitative cristobalite determinations. Powder X-ray samples were prepared and analyzed as described. The average weight fractions of cristobalite for each group of samples were compared to determine the effects of heating environment on the extent of devitrification.

RESULTS AND DISCUSSION

Launch-Site Tests

Launch-site specimens experienced up to 52 exposure and reentry cycles depending on designated exposure period (1, 2, or 4 weeks). Two primary types of failures were observed in these tests. Some failures were characterized by eruptions of coatings and expulsion of coating fragments which occurred after a few cycles, while other failures were characterized by cracking that occurred after about 15 or more cycles. Significant devitrification was associated with cracked specimens.

Eruption failures. - Eruption failures occurred during the second through fifth temperature cycles and affected at least half of the total number of specimens. About 40 percent of all specimens tested were damaged by eruption to the extent that they had to be retired after five atmospheric exposure and temperature cycles (table I). No significant devitrification was detected in the retired specimens. Figure 9 shows photographs of the 2-week and 1-week specimens after 5 and 10 temperature cycles, respectively. These photographs show that top specimens degraded more than bottom while specimens rinsed with tapwater degraded more than unrinsed specimens. These visual observations and preliminary examination of weight change data suggest that water played an important role in the eruption mechanism.

The exact eruption mechanism is not known; however, it could involve one or more of the following possibilities. The outer layer of the LI-0042 coating is very porous as shown in the coating cross section of figure 1. There could be sudden steam pressure generated from water entrapped in the pores of the coating. The resulting localized pressure could conceivably blow out coating fragments during portions of the temperature cycle when the temperature of the coating is lower than its softening point. The coating was apparently too refractory because in some cases it appeared to be underfired. In fact, coating fragments were expelled from the surfaces with considerable force only during the early or late stages of temperature cycles when temperatures were low. A significant number of eruption failures did not extend through the sublayer of the coating. This is a good indication that the origin of failure was in the coating and cannot be associated with steam pressure originating in the insulation beneath.

Corrosion of the silicon carbide in the coating might also generate localized areas of internal pressure. During a significant portion of each thermal cycle, the coating temperature exceeds the minimum temperature of 1140 K required for oxidation of silicon carbide. References 4, 5, and 6 report that finely divided silicon carbide when unprotected by an oxide layer will oxidize rapidly to SiO_2 , CO, and CO_2 in the presence of water vapor and oxygen at temperatures as low as 1140 K. Since the outer layer of the coating is porous with at least a small percentage of pores open to the test atmosphere, water vapor

may come into contact with vulnerable particles of silicon carbide. The increase in the effective volume of a particle due to oxidation and growth of an SiO_2 layer might induce the pressure necessary to pop out a coating fragment. The gases of oxidation or CO and CO_2 might also increase the pressure in affected pores.

An additional contributing factor might be residual stress associated with variations in coating thickness and geometry at edges and corners. Such effects, even if present, could not be recognized on specimens used in the launch-site tests because of their small size; however, results seen on large tiles used in another investigation (ref. 7) indicated a definite tendency toward eruption failures near edges.

Cracking failures. - Figure 10 shows photographs of three launch-site test specimens after eighteen 2-week exposures and temperature cycles and one specimen after five cycles. Cracks were first visually detected on specimens 9 and 16 after the 15th cycle. Degradation proceeded rapidly during the next three cycles and final results are seen in figure 10. It is assumed that the cracks were not present before the 15th cycle because of the rapid degradation witnessed from the apparent onset of cracking through the 18th cycle. Specimen 10 survived the 18 cycles without cracking and specimen 15 was retired after 5 cycles because of eruption damage.

Figure 11 shows two launch-site specimens after twenty-nine and thirty-eight 1-week exposures and temperature cycles. Comparison of these specimens with the 2-week exposure group of figure 10 shows that cracking commenced after approximately the same total atmospheric exposure time (30 weeks) even though the 1-week exposure group had experienced about twice as many temperature cycles. Therefore, figures 10 and 11 show that cracking depends on time exposed to environment rather than number of cycles. They also show that specimens exposed to water impingement (i.e., specimens on top side) suffered the most degradation by eruption failure and/or macrocracking.

Devitrification. - The specimens shown in figures 10 and 11 were analyzed for devitrification. The spalled areas on the lower right or 4th quadrant surface of specimen 9 resulted from crumbling of the coating, while other damaged areas resulted from eruption failures that did not extend through the sublayer of coating. The coatings of specimens 9 and 16 were found to be very friable when they were removed from specimens for X-ray diffraction analysis. Analysis revealed that these coatings contained about 85 percent by weight cristobalite which would account for the extreme friability of the coating. (Friability is characteristic of devitrified glass bodies.) Specimen 10 did not show an appreciable amount of cristobalite after 18 cycles. As indicated previously, this was the only specimen of its group which did not crack after 18 cycles and was the only one of its group which was not exposed to water impingement. Specimen 5 which was exposed to water impingement, qualitatively indicated a high cristobalite content (probably 80 to 90 percent).

On the other hand, specimen 6 which was not exposed to water impingement, qualitatively indicated much less cristobalite.

Slight sagging of the test faces on all specimens shown in figures 10 and 11 was noted. This sagging was not attributed to devitrification of the coatings but rather to shrinkage of the underlying insulation. Sagging was evident on nearly all specimens, clean or contaminated, after 12 to 15 cycles.

Laboratory Tests

A clean specimen (specimen 7, table II) was exposed to 52 radiant heat cycles with soaks in deionized water before each cycle without producing cracking or significant devitrification. The only damage suffered was eruption damage during the first few cycles and slight sagging of the test face due to insulation shrinkage. Because of this finding, it is speculated that contaminants in the water and in the surface were responsible for devitrification and cracking. Rain and rinse water apparently concentrated, rather than diluted, contaminants by dissolving them and redepositing them in pores of the coatings. In addition, top specimens were probably exposed to settling salt-laden dust. The poor performance of the rinsed bottom specimens might further be explained in terms of the chemistry of the rinse water used. This water taken from a city water supply was found to contain a significant concentration of sodium salts since sodium is added to the water supply to displace water-hardening ions. Rinsing may therefore have contributed to increased contaminant concentration.

Specimens exposed to radiant heating for up to 52 cycles, but without water exposure or other means of contamination, showed no eruption failure, macrocracking, or significant devitrification (table II). The only degradation noted was a slight sagging of some of the test faces, probably as a result of insulation shrinkage. As pointed out previously, all specimens exposed to 12 or more cycles seemed to experience some sagging. It is now believed that the macrocracking of devitrified coatings was not caused by devitrification of the coatings but by a combination of devitrification of the coating and shrinkage of the underlying insulation – a combination which produced stresses in the weakened coating.

In the heating tests where different test atmospheres were used, artificially contaminated specimens showed significantly more cristobalite than uncontaminated specimens after 12 test cycles. (See fig. 12.) In all cases, water was removed from the specimens before the tests were begun. Cracking did not occur, nor would it be expected to occur, since the percentage of cristobalite found was still relatively low and the number of cycles was small. From figure 12 it can be seen that the air pressure in the heating atmosphere had little effect on devitrification and certainly what effect it did have was relatively small when compared to the effect of contamination with sea salt. The contaminated arc-tunnel specimen probably produced less cristobalite than the others as a result of the low test

pressure which may have permitted significant vaporization of the salt contaminants. Also the test for this specimen was in flowing air and the specimen was contaminated only once before receiving the 12 heating cycles. All these factors would result in a more rapid depletion of available contaminant.

CONCLUDING REMARKS

Test specimens of LI-0942, reusable surface insulation (RSI), were exposed to alternating radiant heating and atmospheric exposure cycles at John F. Kennedy Space Center (KSC) to study the effects of surface contamination on RSI coatings. Also, different methods of heating (radiant, vacuum furnace, and arc jet) were employed in the laboratory on clean and artificially contaminated specimens to determine the contributions of air and water in heating atmospheres to coating devitrification.

Launch-site exposures at KSC resulted in a high failure rate of specimens (70 percent). Forty percent of the specimens tested failed during the first five cycles. Failure was characterized by eruptions of the coating and expulsion of coating fragments. These failures were attributed to the interaction of water with the coating and not the insulation during temperature cycles. An additional 30 percent of the specimens failed before completing their designated number of test cycles. Failure was characterized by macro-cracking accompanied by devitrification of coatings and slight shrinkage of the insulation. Even though these specimens had been exposed to water impingement from rain or rinsing while the specimens that survived had not, the cause of failure could not be attributed directly to contact with water. One specimen exposed to deionized water between temperature cycles did not crack or experience significant devitrification after 52 test cycles. It is suspected that rain and rinse water enhanced contamination by dissolving surface contaminants and redepositing them within the pores of the coating. Furthermore, the rinse water used was from a municipal water supply and contained concentrations of the same contaminant substances found in the exposure atmosphere.

Laboratory tests on artificially contaminated specimens showed that the effects of the heating mode and atmospheres on the extent of devitrification in the LI-0042 coating were small when compared to the effects of sea salt contamination. Thus, heating in air is a valid test method for studying devitrification of RSI coating provided that the possibility of eruption failures is precluded by the removal of excess water from the material before temperature exposure. Both launch-site and laboratory tests indicated that the

LI-0042 coating will not remain impervious to water and that environmental exposures may cause early coating damage as a result of the water absorbed.

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REFERENCES

1. Ransone, Philip O.; and Dicus, Dennis L.: Effects of Sea Salts on the Physical Characteristics of Reusable Surface Insulation. Symposium on Reusable Surface Insulation for Space Shuttle, Volume II. NASA TM X-2720, 1972, pp. 765-792.
2. Chapman, Andrew J.: Thermal Performance of 625-kg/m³ Elastomeric Ablative Materials on Spherically Blunted 0.44-Radian Cones. NASA TM X-2612, 1972.
3. Klug, Harold P.; and Alexander, Leroy E.: X-Ray Diffraction Procedures for Polycrystalline and Amorphous Materials. John Wiley & Sons, Inc., c.1954.
4. Lea, A. C.: The Oxidation of Silicon Carbide Refractory Materials. J. Soc. Glass Technol., vol. 33, 1949, pp. 27-50.
5. Miller, Warren J.: High Temperature Oxidation of Silicon Carbide. M. S. Thesis, Air Force Inst. Technol., 1972.
6. Suzuki, Hiroshige: A Study of the Oxidation of Pure Silicon-Carbide Powders. NASA TT F-10,211, 1966.
7. Hunt, L. Roane; and Bohon, Herman L.: Performance of LI-1542 Reusable Surface Insulation System in a Hypersonic Stream. NASA TM X-71955, 1974.

TABLE I.- LAUNCH-SITE TEST SPECIMENS

Specimen	Exposure period	Exposure position	Scheduled no. of cycles (a)	Completed no. of cycles (a)	Remarks
1	1 week	Top	18	18	Eruptions, shrinkage, slight devitrification
2	1 week	Bottom	18	18	Slight shrinkage
3	1 week	Top	36	5	Eruption failure
4	1 week	Bottom	36	36	Slight shrinkage
5	1 week	Top	52	38	Cracked before 29th cycle, devitrified, shrinkage
6	1 week	Bottom	52	52	Cracked on 29th cycle, slight devitrification, ^b shrinkage
7	2 weeks	Top	9	5	Eruption failure
8	2 weeks	Bottom	9	9	No visible damage
9	2 weeks	Top	18	18	Eruptions, cracked, devitrified
10	2 weeks	Bottom	18	18	Shrinkage
11	2 weeks	Top	27	5	Eruption failure
12	2 weeks	Bottom	27	27	Shrinkage
13	2 weeks	Top	^c 9	5	Eruption failure
14	2 weeks	Bottom	^c 9	5	Eruption failure
15	2 weeks	Top	^c 18	5	Eruption failure
16	2 weeks	Bottom	^c 18	18	Cracked, devitrified, shrinkage
17	2 weeks	Top	^c 27	5	Eruption failure
18	2 weeks	Bottom	^c 27	5	Eruption failure
19	4 weeks	Top	12	12	Slight shrinkage, slight devitrification
20	4 weeks	Bottom	12	12	No visible damage
21	-----	-----	---	---	Control specimen
22	-----	-----	---	---	Control specimen

^aCycle consists of an exposure period followed by a heating period.

^bSlight devitrification, ≤ 5 percent cristobalite.

^cSpecimen rinsed before each temperature cycle.

TABLE II.- LABORATORY TEST SPECIMENS

Specimen	Size	Heating	Contaminant	Number of heating cycles	Remarks
1	Small	Radiant	None	9	No visible damage
a2	Small	Radiant	None	12	Slight devitrification ^b
3	Small	Radiant	None	18	Slight devitrification, slight shrinkage
4	Small	Radiant	None	27	Slight devitrification, slight shrinkage
5	Small	Radiant	None	36	Slight devitrification, slight shrinkage
6	Small	Radiant	None	52	Slight devitrification, slight shrinkage
7	Small	Radiant	Deionized water	52	Eruptions, slight devitrification and shrinkage
8	Small	Radiant	Salt fog	12	Devitrified
9	Small	Radiant	Salt fog	12	Devitrified
10	Small	Vacuum furnace	Salt fog	12	Devitrified
11	Small	Vacuum furnace	Salt fog	12	Devitrified
12	Small	Radiant	None	12	Slight devitrification
13	Small	Vacuum furnace	None	12	Slight devitrification
14	Small	Vacuum furnace	None	12	Slight devitrification
15	Large	Arc jet	Salt fog	12	Devitrified
16	Large	Arc jet	None	12	Slight devitrification

^aUsed with specimen 12 for data in figure 12.

^bSlight devitrification, \approx 5 percent cristobalite.

TABLE III.- ARC-JET TEST CONDITIONS

Total enthalpy, MJ/kg	17.39
Cold-wall heat-transfer rate, kPa	280
Stagnation pressure, kPa	6.93
Local surface pressure, ^a kPa	0.537
Maximum surface temperature, K	1430

^aAverage along center line of test specimen parallel to flow direction.

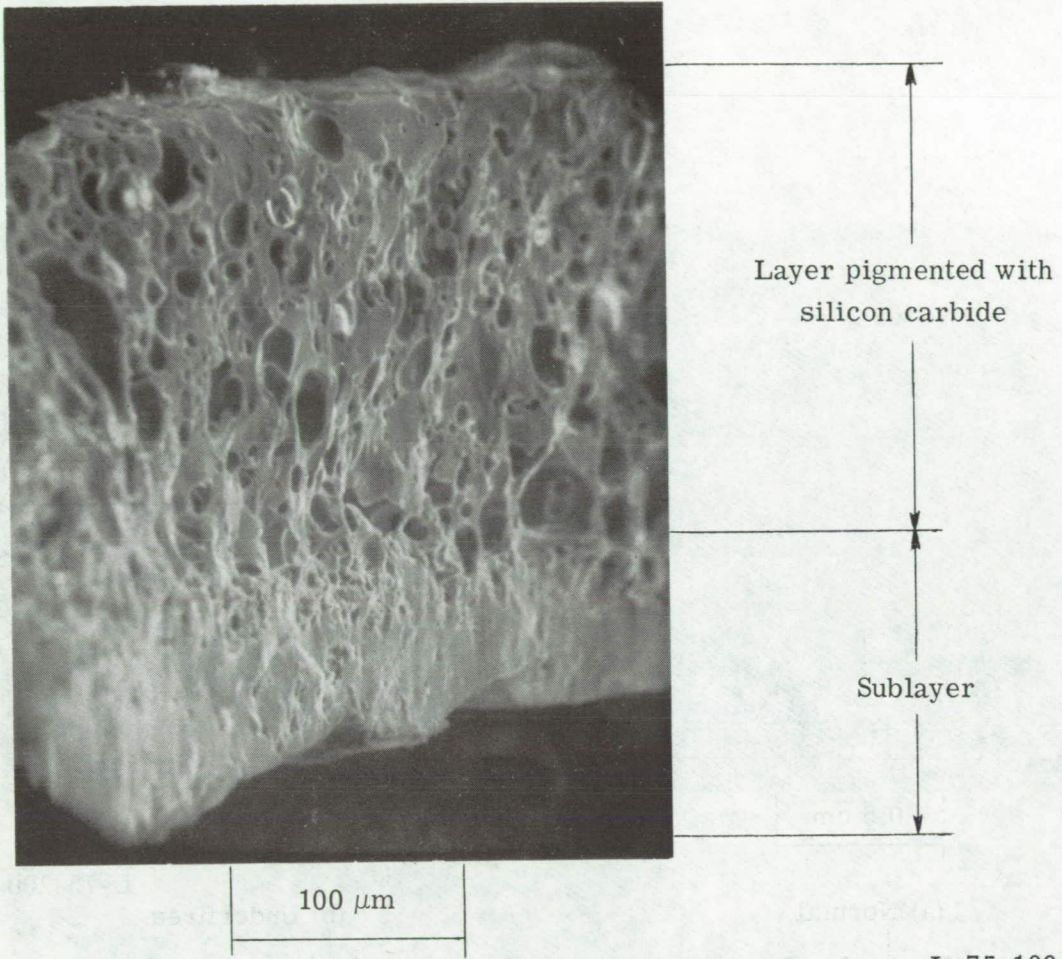
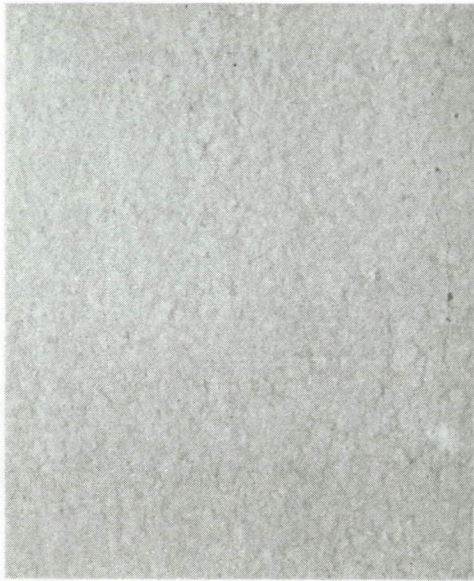


Figure 1.- Fractograph of LI-0042 coating.

L-75-199



0.5 cm

(a) Normal.

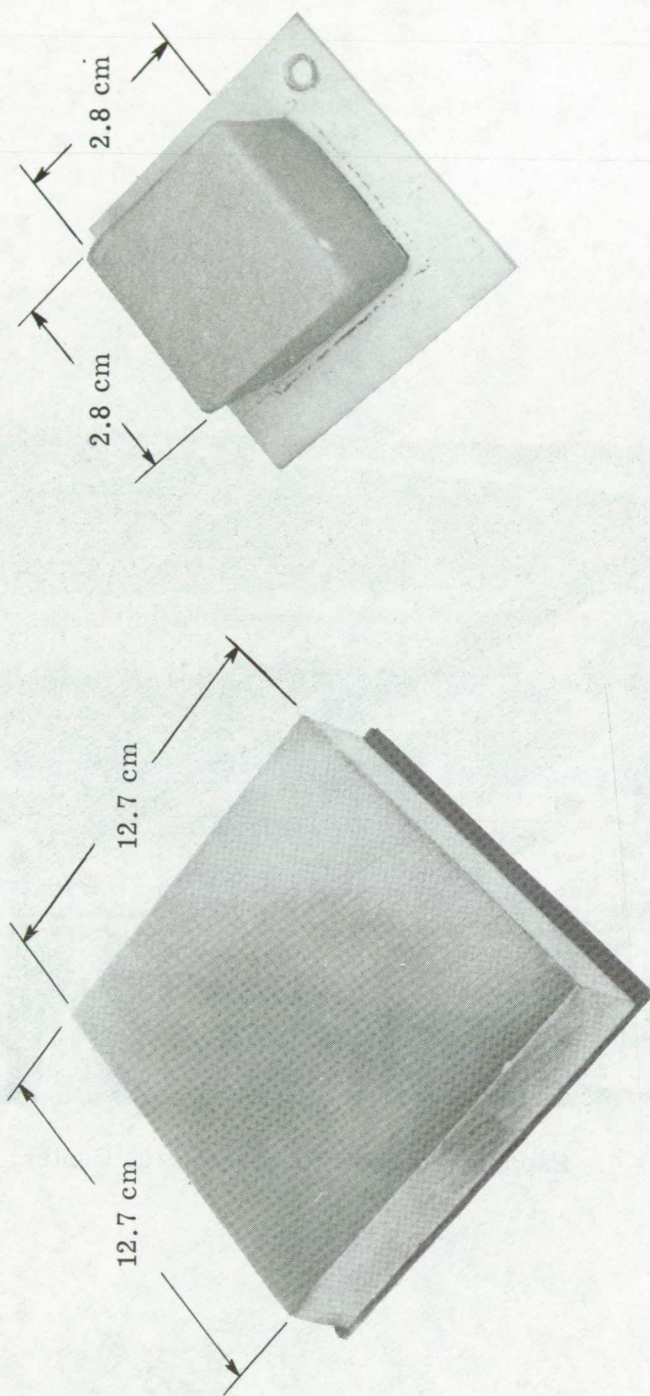


0.5 cm

(b) Underfired.

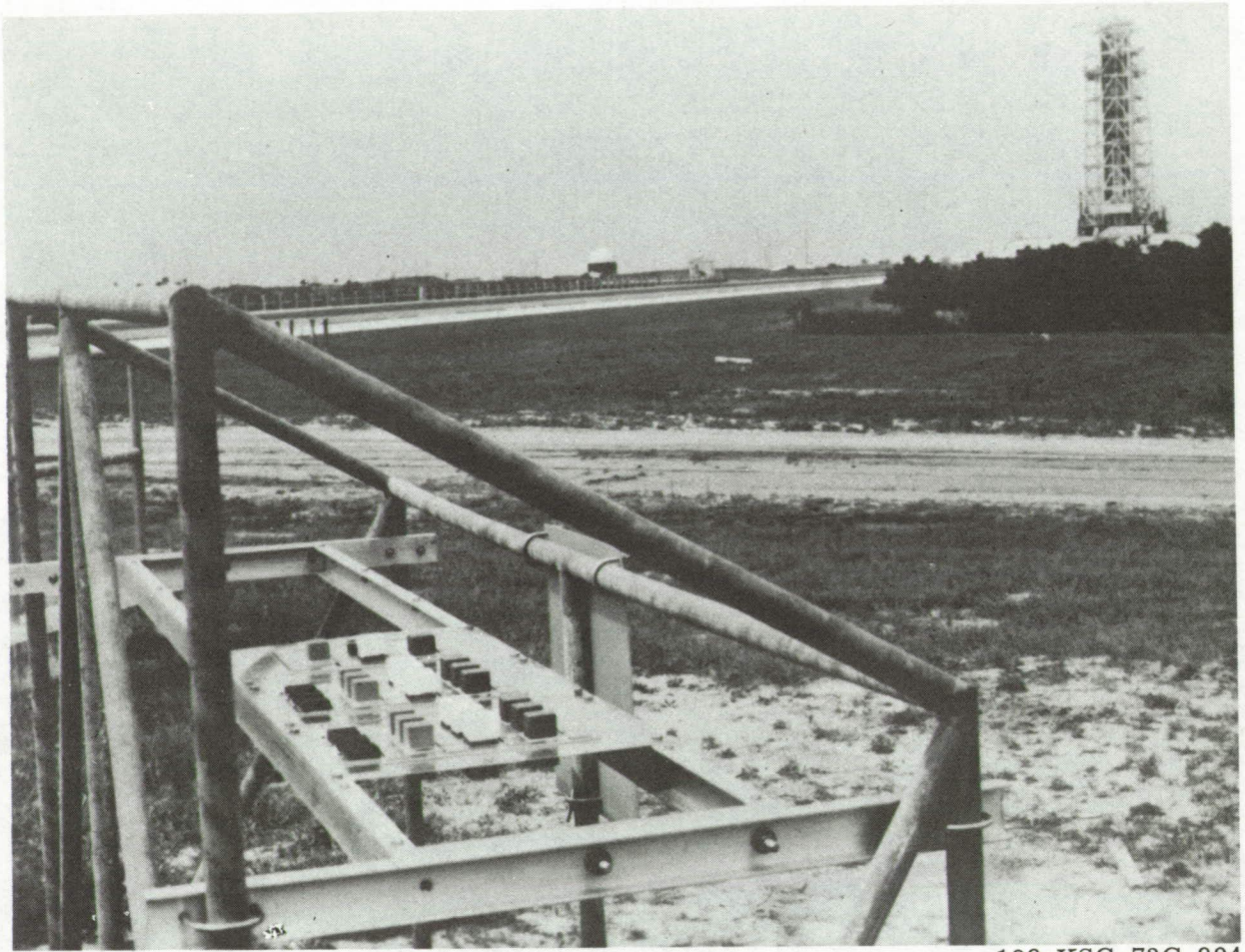
L-75-200

Figure 2.- Comparison of a normal coating surface and an underfired coating surface.



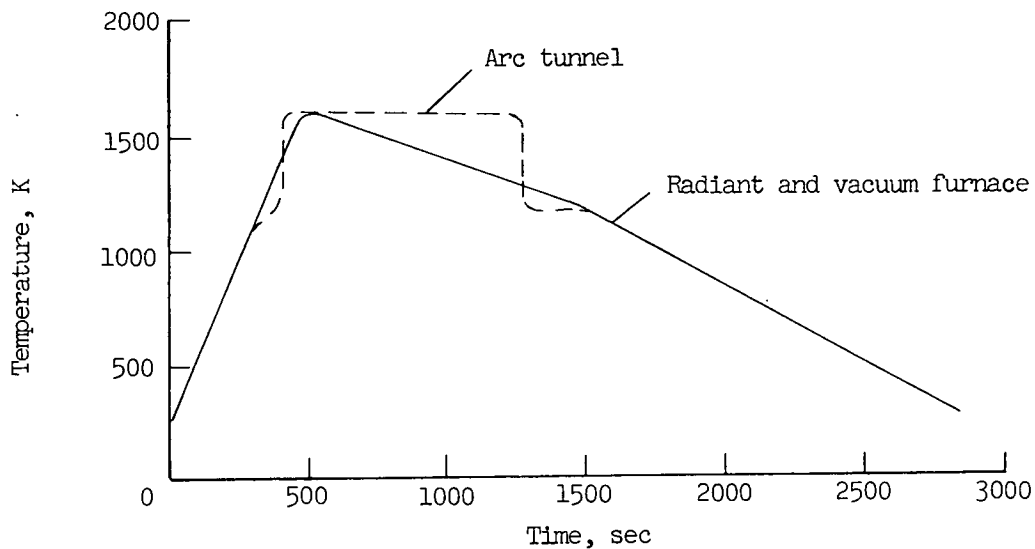
L-75-201

Figure 3.- Specimen configuration.

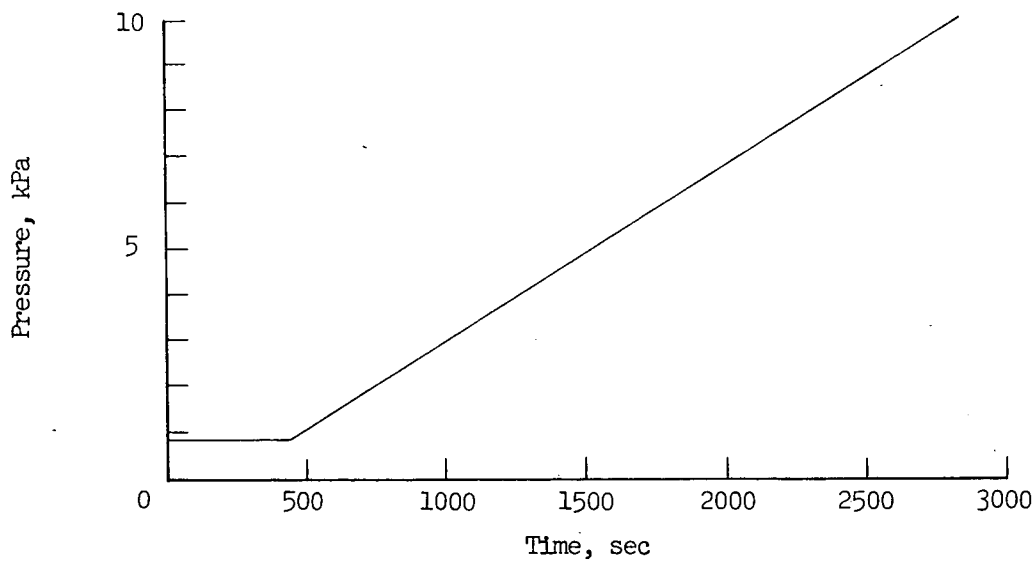


108-KSC-73C-894

Figure 4.- Exposure rack at Kennedy Space Center.

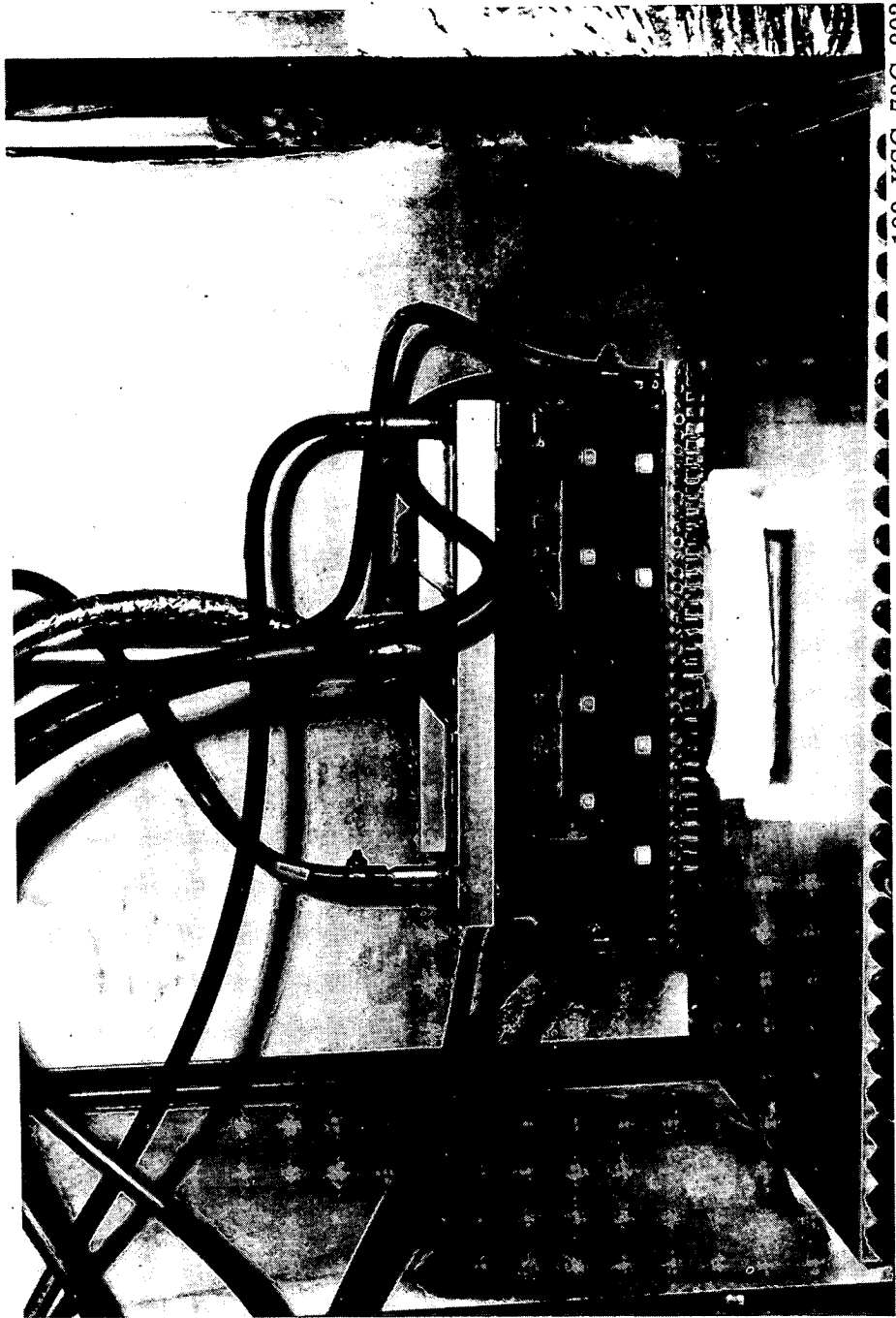


(a) Temperature profile.



(b) Pressure profile (for vacuum furnace test only).

Figure 5.- Surface temperature and pressure profiles for a space shuttle reentry simulation.



108-KSC-73C-892

Figure 6.- Radiant heating apparatus used for heating tests on launch-site and laboratory specimens.

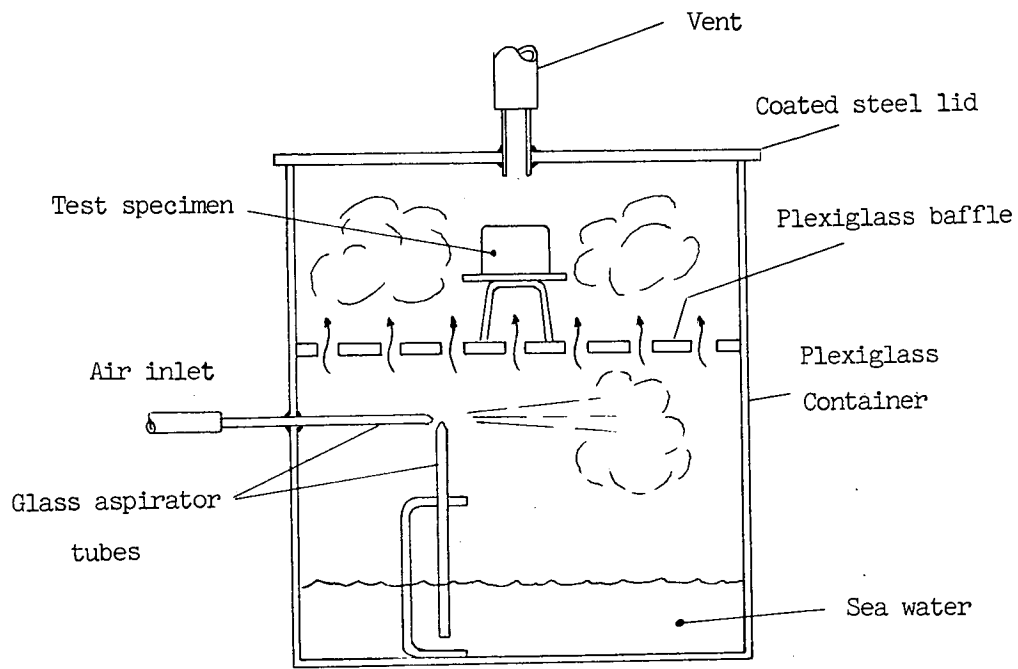


Figure 7.- Salt fog apparatus used to artificially contaminate laboratory specimens.

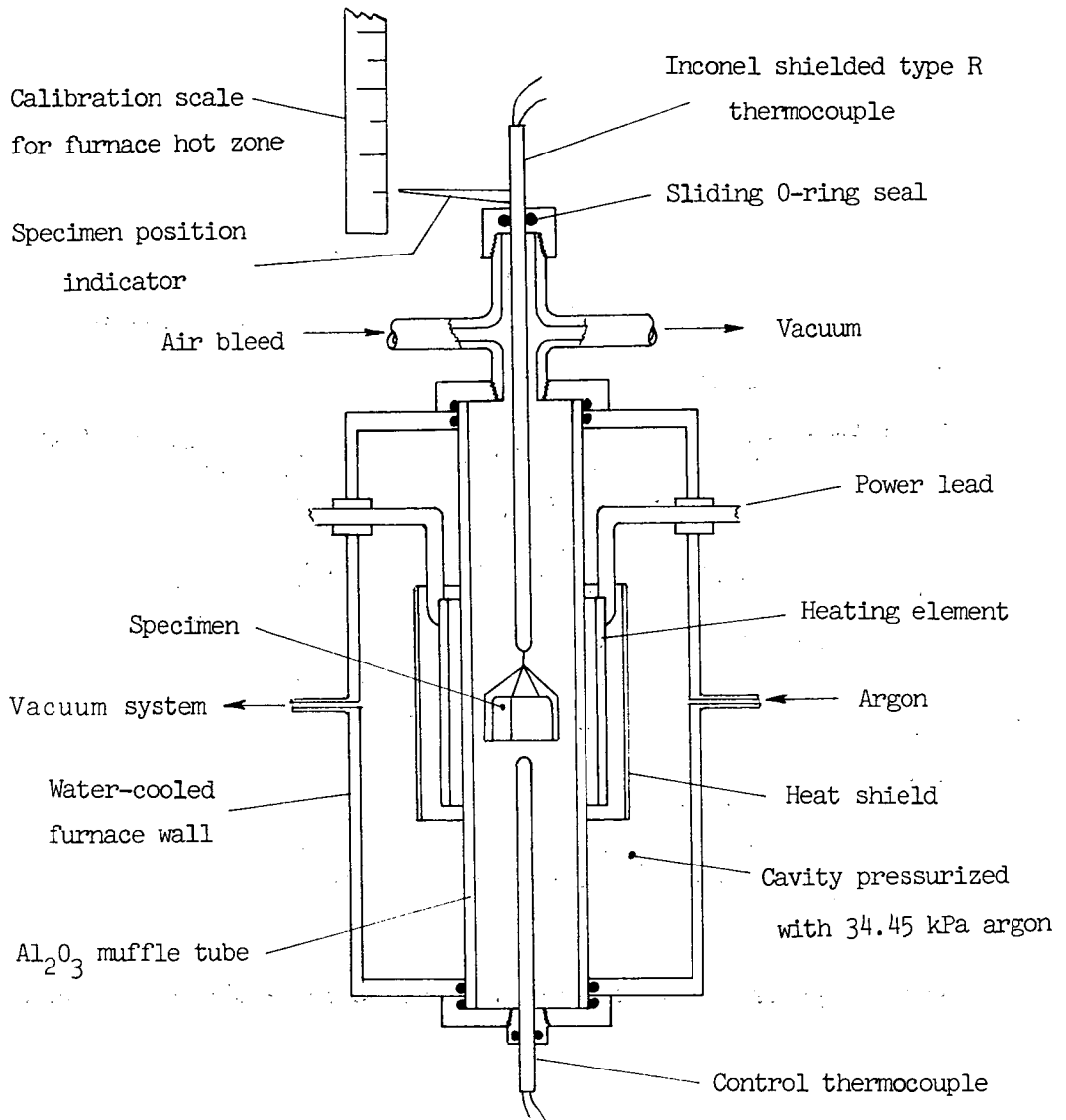
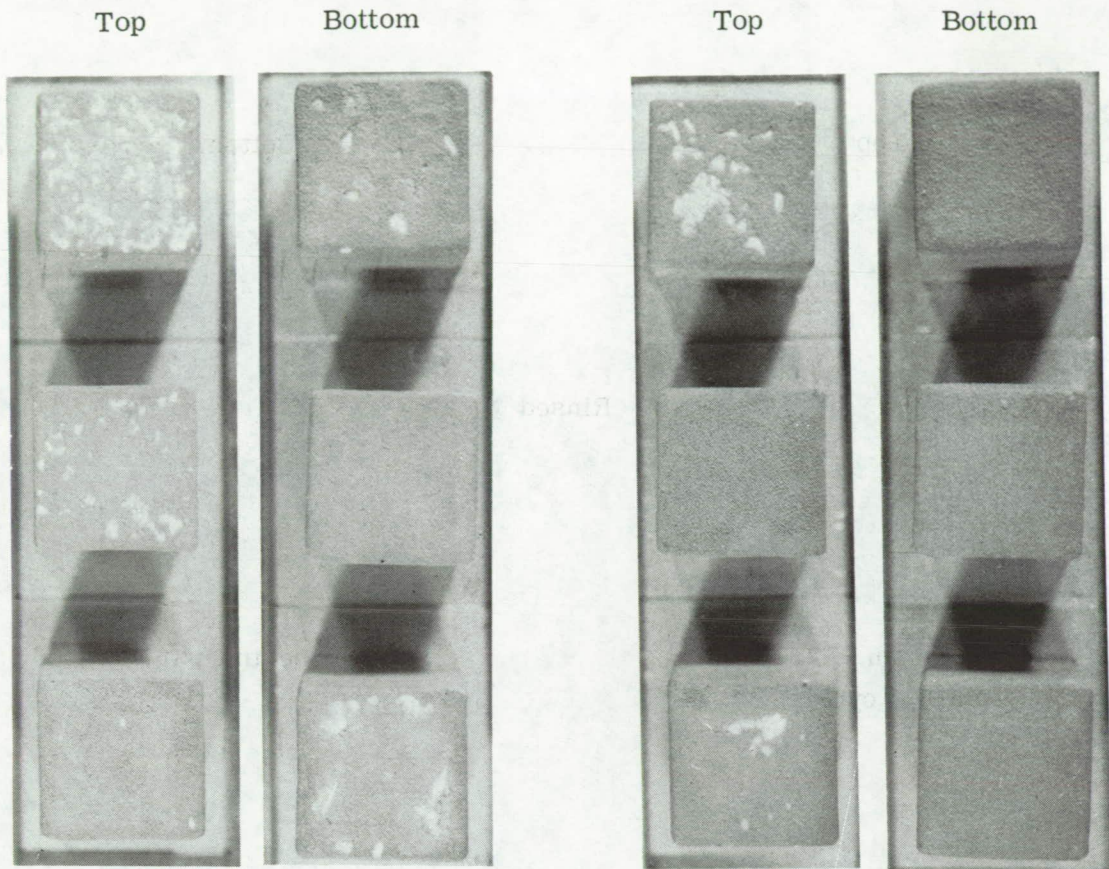
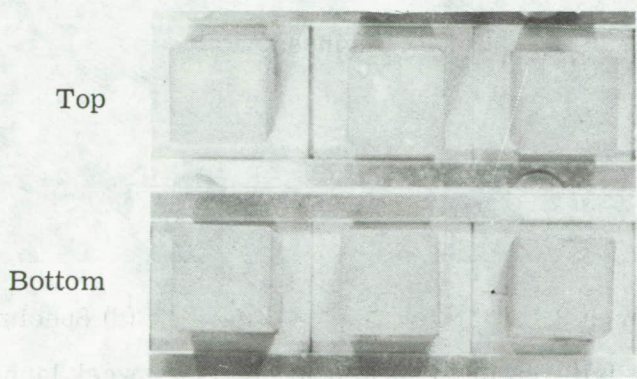


Figure 8.- Vacuum furnace and test setup used for pressure profile heating of laboratory specimens.



(a) 2-week exposure; rinsed;
5 heating cycles.

(b) 2-week exposure; not rinsed;
5 heating cycles.



(c) 1-week exposure; not rinsed;
10 heating cycles.

L-75-204
Figure 9.- Eruption failures in LI-0042 coating.

Top



(a) Specimen 15 (retired after 5 cycles).

Bottom



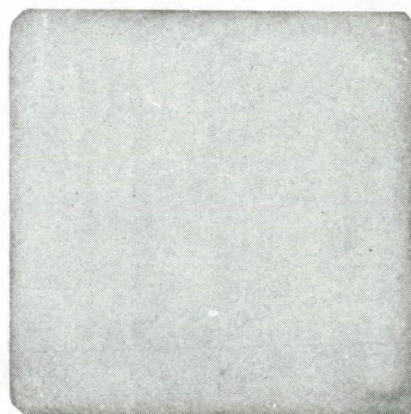
(c) Specimen 16.

Rinsed

Unrinsed



(b) Specimen 9.



(d) Specimen 10.

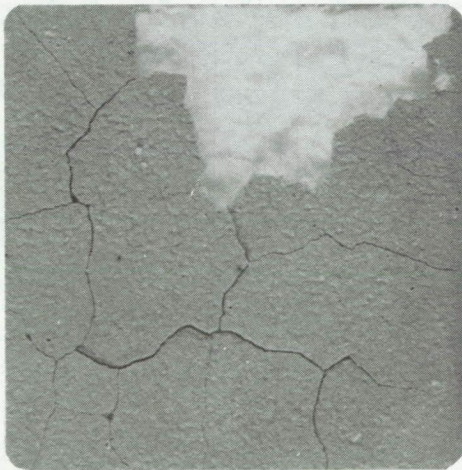
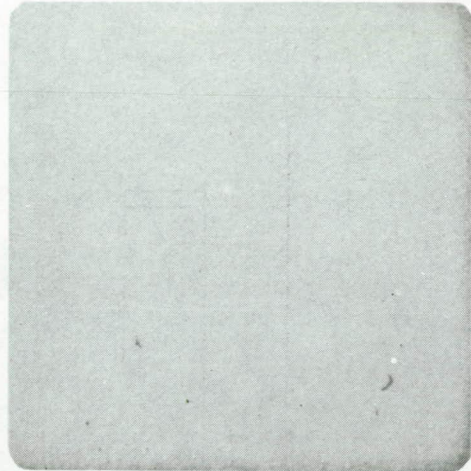
Figure 10.- Cracking failures in LI-0042 coating. Two-week launch-site exposures; 18 heating cycles (table I). L-75-205

Top

Bottom



29 cycles



38 cycles



Specimen 5

Specimen 6

L-75-206

Figure 11.- Cracking failures in LI-0042 coating. One-week launch-site exposures; 38 heating cycles (table I).

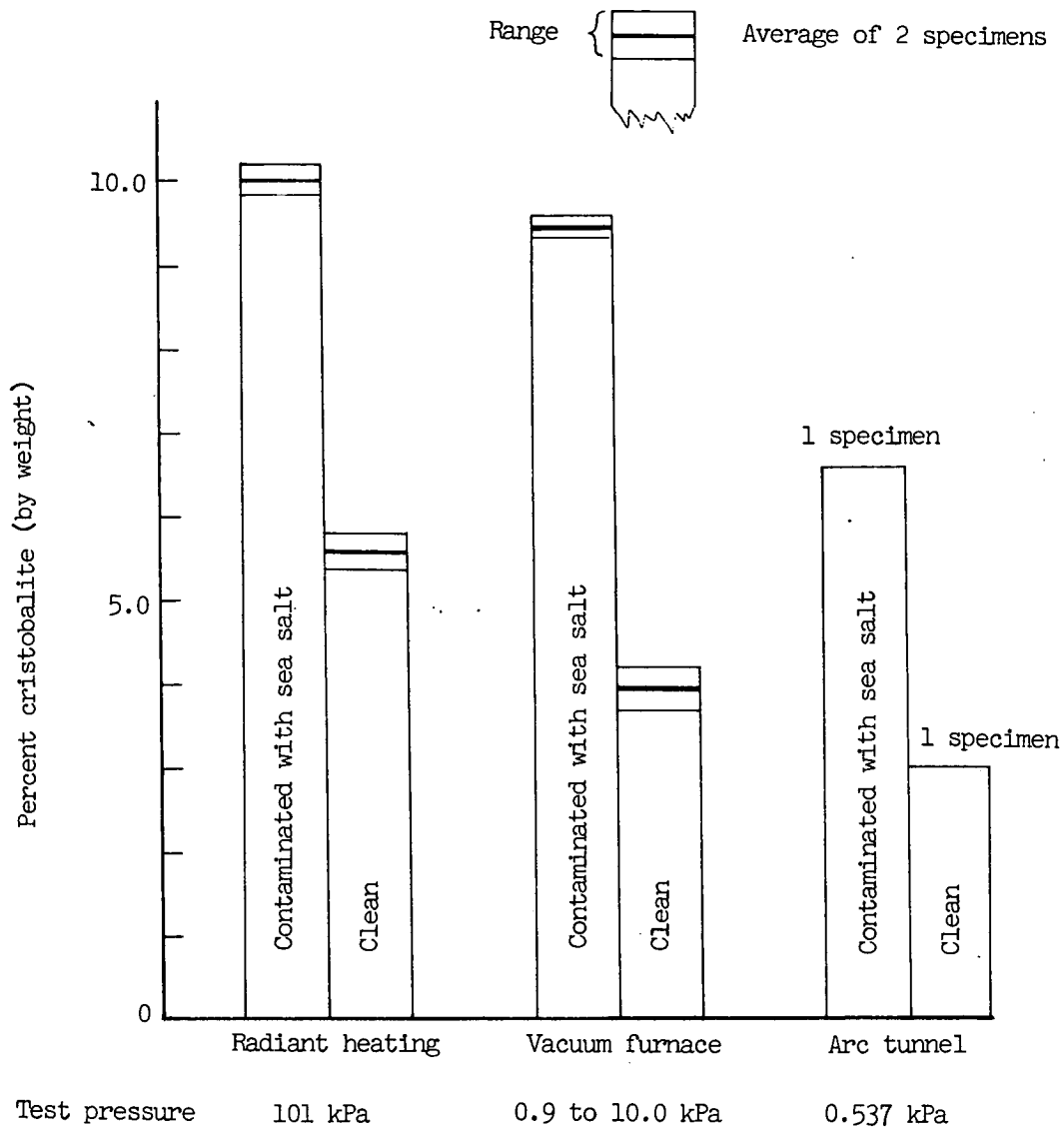


Figure 12.- Percent cristobalite in LI-0042 coating after 12 temperature cycles in three different heating atmospheres (contaminated before first cycle only).



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