DEVELOPMENT AND EVALUATION OF COATING FOR AN UNFURLABLE RE-ENTRY VEHICLE

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Introduction

The advance of missile and astronautic technology to the present state-of-the-art has introduced new and unique fields for the engineer. To assure the most efficient use of the limited volume and payload capabilities of the present generation booster in establishing orbital flight for large vehicles, a flexible or expandable structure which can be unfurled when orbital trajectory is attained offers the greatest present potential. This type of structure can be completely assembled on the ground, packaged to a minimum symmetrical size compatible with the booster last stage diameter, easily transported to the launch site, installed on the missile, and automatically deployed into the space environment upon achieving the desired orbital position. This flexible structure approach developed by GAC entails the use of either internal pressure or mechanically stabilized fabric.

Once orbital flight has been accomplished the next step requires that instrumentation and personnel must be returned from active space vehicles. Using the expandable structure approach a lightweight vehicle can be adapted to this purpose also and the temperature encountered by this re-entering vehicle can be limited to the order of 1500°F.

In general, the basic structure of the re-entry vehicle would be a woven material. Since this material is porous to some degree, a coating is required which will act as a gas barrier when the structure is pressure stabilized, or as an airflow barrier when the skin is mechanically stabilized. This coating must withstand aerodynamic shear forces, maintain its integrity at 1500°F, and be extremely flexible prior to exposure to maximum temperature. In addition the coating must be compatible with the basic material of the cloth and the cloth construction.

Presented in this paper is a discussion of the approach to the development of such a coating, a description of the tests and test procedure used to evaluate the coating, and some of the test results.

COATING DEVELOPMENT APPROACH

Statement of the Problem

The heat generated by aerodynamic friction on a vehicle as it re-enters the denser atmosphere of the earth has imposed a severe restriction on space flight. Two general approaches have been investigated to cope with this problem.

The first approach is applicable to a high-density structure and requires that excess material be carried by the re-entry vehicle. This excess material is consumed by one process and/or another or functions as a heat sink, thus reducing the temperature to which the basic structural components are subjected.

The second approach is applicable to a low-density structure. That is, it requires that a structural concept be developed which allows the vehicle to have as small a $\frac{W}{C_D A}$ as possible, still maintaining an aerodynamic configuration for flight within the earth's atmosphere. Control of the re-entry path can be such that it will decelerate at a relatively high altitude resulting in a minimum increase in temperature due to aerodynamic heating.

One of the advantages of the second approach is the reduction or elimination of the weight penalty imposed by carrying a thermal shield, since the vehicle does not reach extremely high temperatures and can be cooled by radiation.

Another advantage is that with temperatures restricted to 1500°F levels, currently available materials can be employed in the structure.

An analysis of the temperatures encountered during re-entry with such a radiating structure, assuming an emissivity of 0.8, was conducted. The data generated during the analysis is shown in Figure 1 in which lines of constant temperature are shown as a function of altitude and vehicle velocity. It can be seen that if the velocity of a re-entry vehicle can be reduced at a high enough altitude the maximum temperature it will experience will not exceed 1500°F.

By conducting a trajectory analysis of either a ballistic or glide re-entry vehicle it can be seen that a low-density structure is imperative if the maximum deceleration portion of the re-entry flight is to occur at very high altitudes where, as stated previously, extremely high temperatures can be avoided.

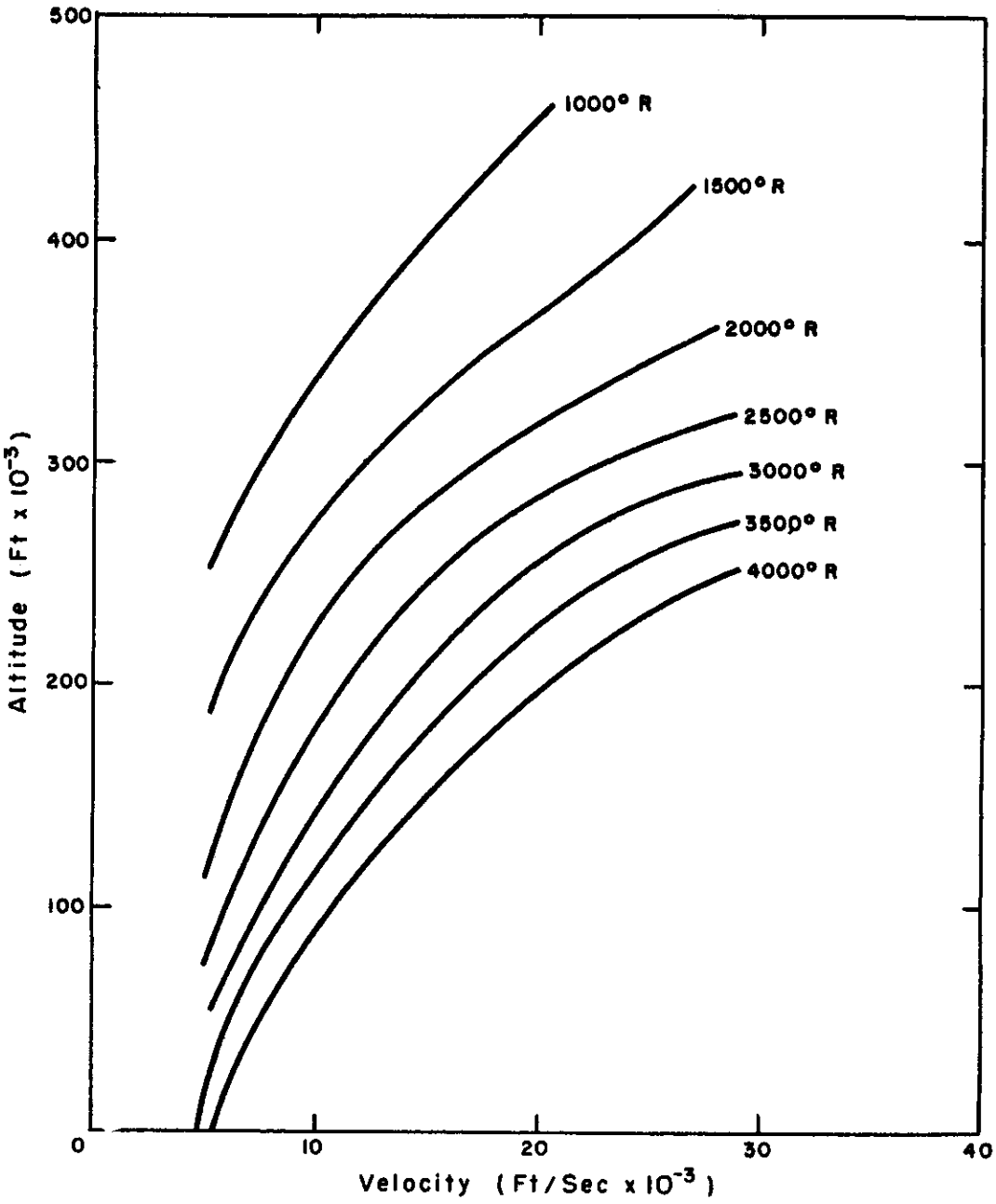

Figure 1 - Re-Entry Temperatures

To further emphasize the effect of structural density on the maximum temperature of the re-entry vehicle consider a glider constructed of structural material cooled by radiation only. Utilizing the equations for wing loading and temperature at the same environmental density, it is possible to show that maximum temperature will be encountered at a velocity of 21,400 fps if orbital velocity is taken at 25,800 fps. The stagnation temperature will vary from 1650°R to 3900°R for a change in wing loading from 0.2 to 200 psf or a temperature change of 700°F for an order of magnitude changing in wing load from 2 to 20 psf. In order to attain a minimum weight vehicle of sufficient size to return a reasonable payload from orbit and contain the maximum temperature encountered within the capability of presently available materials, 1500°F, and be packageable in an ascent vehicle, it is necessary to develop an extremely lightweight, heat radiating, flexible structure. The above requirements imply the use of a woven or cloth-like structure fabricated from a super alloy such as René 41 which can retain good physical properties up to 1500°F. In one case no rigid members would be employed and the structure would have to be pressure stabilized in order to sustain the loads. In the case of a mechanical stabilized structure, it must be able to withstand the aerodynamic pressures developed. The gas or dynamic pressure that may be experienced by the vehicle may vary from 0.3 to 5 psi. In either case, it can be seen that since a fabric is porous a coating material must be employed in conjunction with the basic cloth to satisfy its functional requirements.

In the development of such a high-temperature fabric, the selection of the coating material is as important as the selection of the basic material. In general, the coating material protects the basic material from detrimental effects such as oxidation for the spectrum of operational temperatures and conditions, in addition to acting as an inflation and/or gas barrier. It is conceivable that a coating material could function well for one material-and-cloth combination and have a detrimental effect on another. In addition to having good thermal characteristics, the coating must have good adhesive properties to the basic material, under static and dynamic conditions, and good flexibility so that the resulting fabric is packageable.

Coating Material Selection

As a result of the coating requirements it was determined that an elastomeric coating material would be required. A program was initiated to develop a high-temperature elastomer for a metal cloth. Candidate coatings were screened by casting specimen buttons which were heated to 1500°F in near complete absence of air. After heating they were examined to determine if they retained their original physical properties. One-hundred-sixteen (116) elastomers were examined. Of these materials, in essence, none were completely satisfactory. The program was extended to an investigation of solution-type materials.

Due to the nature of these materials, it is impossible to cast and cure thick samples; therefore, it was necessary to apply coatings of the test materials on wire cloth for test specimens. Effort was concentrated on the silicone elastomer S2077 to improve its high-temperature properties by the combination with additives, such as metal powders, ceramic enamels, and other high-temperature resistant metals.

Approximately 130 formulations were examined. In addition to the additives presented above, such materials as powdered aluminum and antimony, carbon black, and iron oxide were added to the base elastomers.

The test specimens were fabricated as follows: the silicone elastomer was hand-spread on wire cloth. A small sample was mounted in the frame and a thinned-out layer of coating was applied by brush. The sample was then dried in an oven at nominally 250°F until a large percentage of the solvents were driven off. Heavier coating was then brushed on and the sample dried again.

Depending on the particular coating, several more of these heavier coatings were applied to the test specimen, with the drying operation performed between each coating until the desired dry weight was obtained. After the final coating was applied the specimen was pre-cured for 20 minutes at 275°F under pressure to prevent porosity in the coating. It was then cured for 24 hours in an oven at 480°F.

The test specimens were subjected to temperatures of 1500°F and checked for flexibility, strength, and presence of voids. They were examined microscopically to determine the adhesion of the coating to the wire cloth. Porosity was determined by applying a drop of low surface-tension solvent to one side of the specimen.

As a result of these tests, 24 coatings were selected for permeability tests at high temperature. The helium leak-rate data obtained from the permeability tests as a function of time for a specific heat cycle and a quarter-psi helium pressure are presented in Figure 2. (The heat cycle is also presented in this figure.) As a result of these tests, coatings CS105 and CS107 were selected as the most promising. These coatings consist of a mixture of the silicone elastomer S2077 and a glass enamel.

In order to determine the temperature effect only on the state of the coating material, a series of samples were fabricated and heated in a controlled atmosphere oven. As a result of these tests it was determined that a change in the coating occurs at nominally 1100°F. It is believed that at this point the silicone elastomers break down and the additives begin to function as the gas barrier. A second change in the coating occurs at nominally 1400°F at which point it appears that the additives fuse. The specimens were heated to a maximum of 1800°F. After this

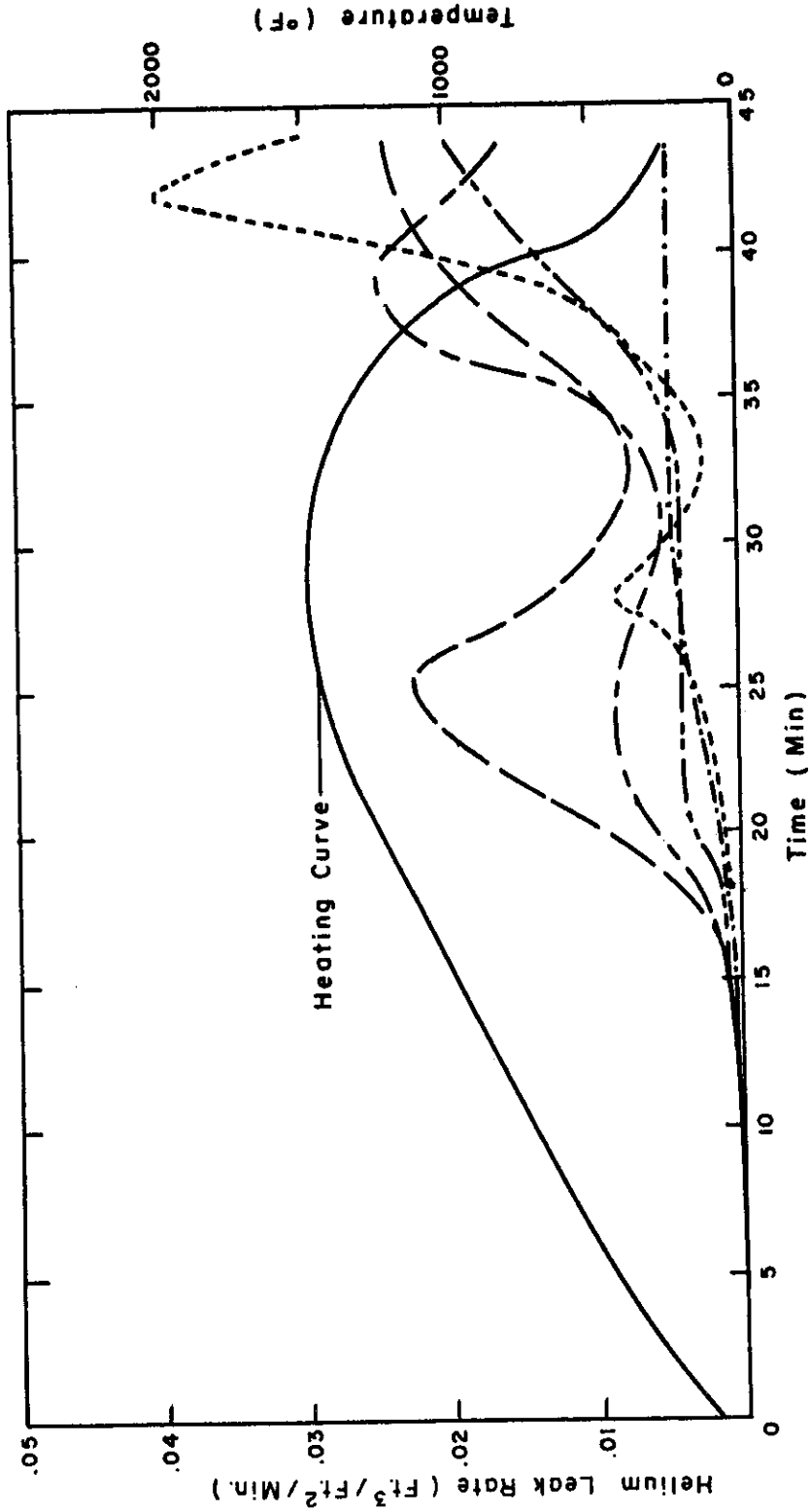

Figure 2 - Helium Leak Rates for Various Coatings

heat exposure and subsequent cooling to room temperature, they retained a flexibility comparable to a sheet metal of the same thickness.

Several specimens were cooled to -320°F in liquid nitrogen to obtain a qualitative indication as to the effect of low temperatures on the coating. This test indicated that the coating retained a large degree of its flexibility even at this temperature extreme.

It was concluded from these screening tests that CS105 offered the greatest promise and should be investigated more thoroughly using René 41 cloth and functional tests.

Functional Tests of CS105

Although it is not possible to simulate the complete environment to which the coating will be exposed during re-entry, it is possible to simulate various aspects of the environment. The functional tests consist of evaluation of permeability, resistance to hot gas flow, and resistance to exposure to high vacuum and ultraviolet radiation. In addition to these experiments tests were conducted to determine the emissivity of the coating material and basic cloth combination as a function of temperature.

The purpose of the permeability test is to determine the effect of temperature on the rate of flow of a gas through the fabric as a function of gas pressure. A specially designed apparatus (Figures 3 and 4) which measures rate of gas diffusion through fabrics heated in a vacuum was used.

Essentially the tests consisted of mounting a coated fabric specimen between two enclosed compartments, one containing helium, the other containing air at an extremely low pressure. The pressure in the helium compartment was controlled. The pressurized portion of the specimen was exposed to radiant heat generated by a bank of quartz lamps, and the temperature was controlled to simulate the desired temperature-time cycle. The temperature was measured using a thermocouple pressed against the heated side of the specimen. The thermocouple was coated with the same type coating as the specimen to that it would have the same reflectivity as the sample.

The pressure difference from one side of the specimen to the other was established by means of a flow regulator and maintained with a vacuum pump. Permeability of the specimen to helium was measured during the test by means of a helium detector, and the rate of leakage was determined. The helium detector is actually a special-purpose mass spectrometer, an electronic analytical instrument designed to analyze gas samples. Gases in the chamber were drawn into the machine and ionized with an electron beam, and the helium concentration was determined by means of electro-

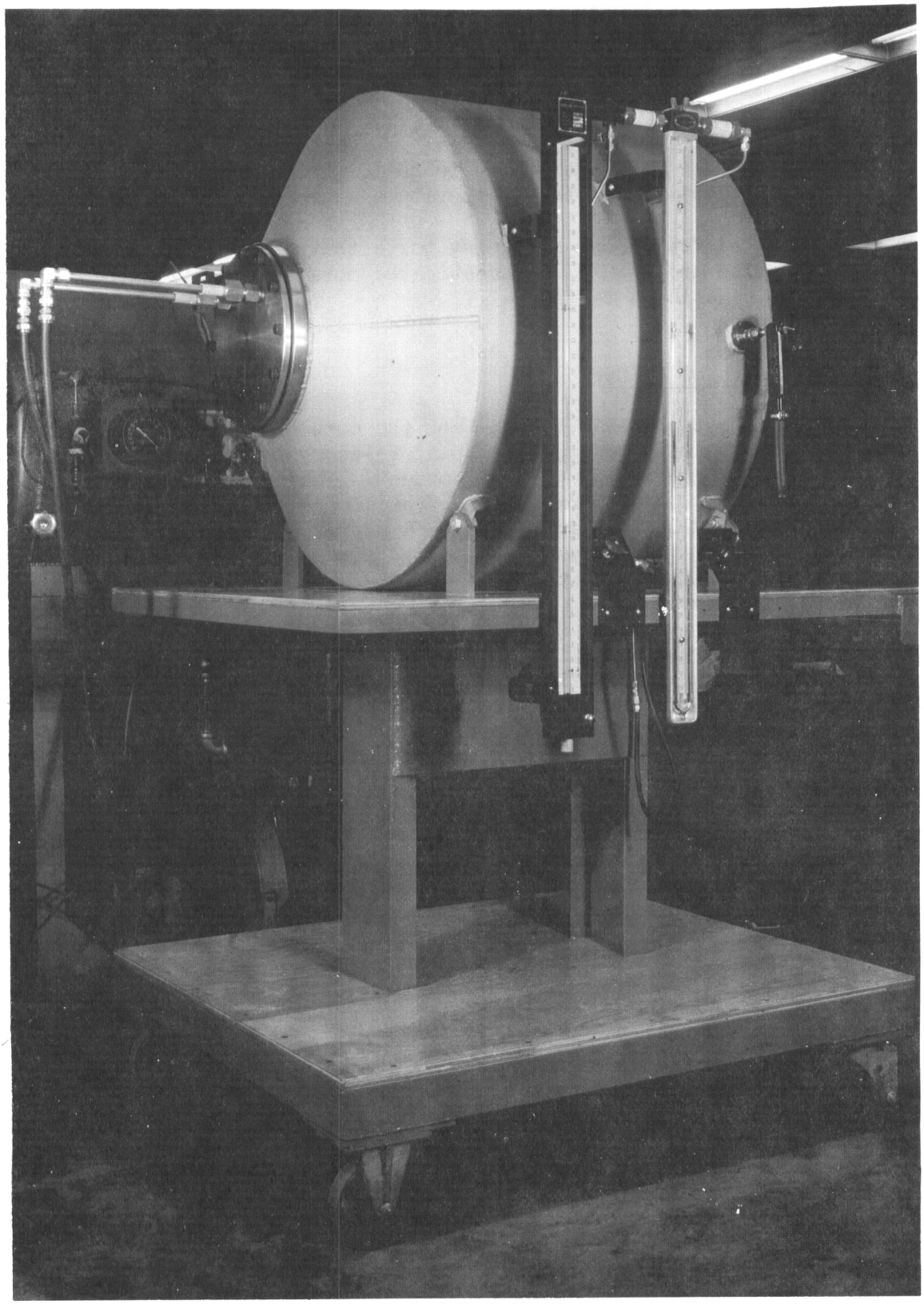

Fig. 3 Fabric permeability apparatus.

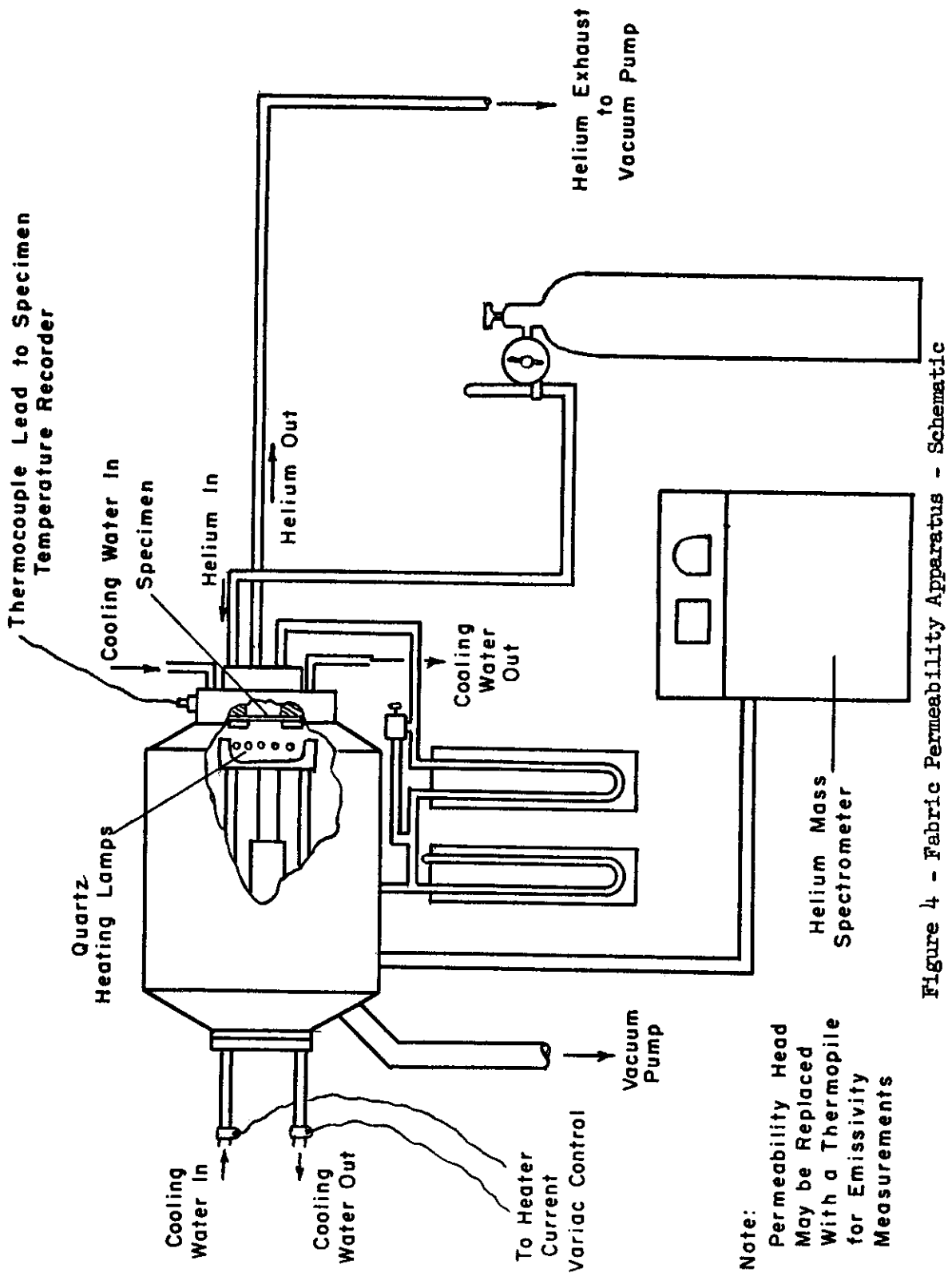

Figure 4 - Fabric Permeability Apparatus - Schematic

magnetic separation. The rate of change of helium concentration in the chamber was then determined graphically from the curve of helium concentration versus time.

Several specimens were tested using an exposed area of about one inch in diameter, but it was found that by using a larger area, two inches in diameter, more accurate and reproducible data was obtained. René 41 plain weave cloth having 200 x 200 filaments per inch 0.0016 inches in diameter with ten different weights of coating were tested with the larger exposed areas.

Table I presents the number of specimens tested together with their coating weights and the differential helium pressures at which each specimen was tested. The maximum permeability as well as the total leakage for the duration of the test are given for each specimen.

Figure 5 shows the permeability curves for several specimens superimposed on the curve of the temperature cycle to which the specimens were exposed.

All of the specimens showed practically no helium leakage during the first 3.0 to 3.5 minutes. Then, just as maximum temperature was being reached the rate started increasing. As the temperature started dropping, the permeability in most cases leveled off.

Since most of the leakage usually took place during the last 1.5 to 2.0 minutes of the test, the maximum permeability was roughly equal to one-half to two-thirds of the total leakage at 5.5 minutes.

Differential pressures of 1.0 psi produced, as would be expected, permeability values about twice as great as those obtained with pressures of 0.5 psi.

Two specimens were tested with a 10-per cent helium-air mixture instead of the usual pure helium to determine the effect of an oxidizing atmosphere on the material. There was a slight discoloring, but the permeability was much lower than expected, even considering that with the total differential pressure of 0.5 psi the helium partial pressure was only about 0.05 psi.

Similarly, tests were conducted using twill and basket weave René 41 cloths. A comparison of the results shows that for an 8-ounce coating the twill weave has a maximum permeability rate approximately $2\frac{1}{2}$ times greater than when a plain weave cloth is used but these values are still extremely low.

The 200 x 200 basket weave resulted in a rate of flow approximately three times greater than for an equivalent coating weight on a plain weave.

TABLE I

HELIUM PERMEABILITY TEST DATA FOR PLAIN WEAVE CLOTH

Specimen No.	Coating Weight (oz/yd^2)	Differential Pressure (psi)	Max. Permeability (ft^3/ft^2) (min)	Total Leakage for 5.5 Min. (ft^3/ft^2)
7A	7.57	0.5*	.0001	.0002
7B		0.5*	.0002	.0003
7C		0.5	.0046	.0065
8A	9.70	0.5	.002	.003
8B		1.0	.006	.011
8C		0.5	.001	.002
13A	4.42	0.5	.0138	.024
13B		1.0	.0216	.045
13C		0.5	.0121	.022
15A	7.50	0.5	.0127	.019
15B		1.0	.0262	.039
15C		0.5	.0193	.032
15D		0.5	.0092	.017
16A	5.22	0.5	.0113	.017
16B		1.0	.0244	.038
16C		0.5	.0115	.017
19A	8.50	0.5	.0136	.027
19B		0.5	.0187	.036
19C		0.5	.0018	.003
19D		0.5	.0093	.015
21A	11.42	0.5	.0016	.033
21B		0.5	.0018	.004
21C		0.5	.0029	.005
38A(vac)	15.70	0.5	.0030	.006
38B(vac)			.0036	.007
38a1(vac&u.v.)			.0042	.008
38a2(vac&u.v.)			.0060	.011
38b1(control)			.0067	.014
38b2(control)			.0105	.018
41A	7.29	0.5	.0149	.030
41B			.0054	.016
41C			.0110	.020
41D			.0087	.017
43A(vac)	14.00	0.5	.0093	.018
43B(vac)			.0128	.023
43a1(vac&u.v.)			.0172	.029
43a2(vac&u.v.)			.0136	.025
43b1(control)			.0097	.020
43b2(control)			.0118	.024

*7A and 7B were tested with a 10% helium-air mixture so that the actual helium partial pressure was therefore more nearly 0.05 psi. All other tests used pure helium.

Material specimens 38 and 43 were exposed to the environmental conditions shown in the above table for 15 days prior to the permeability tests.

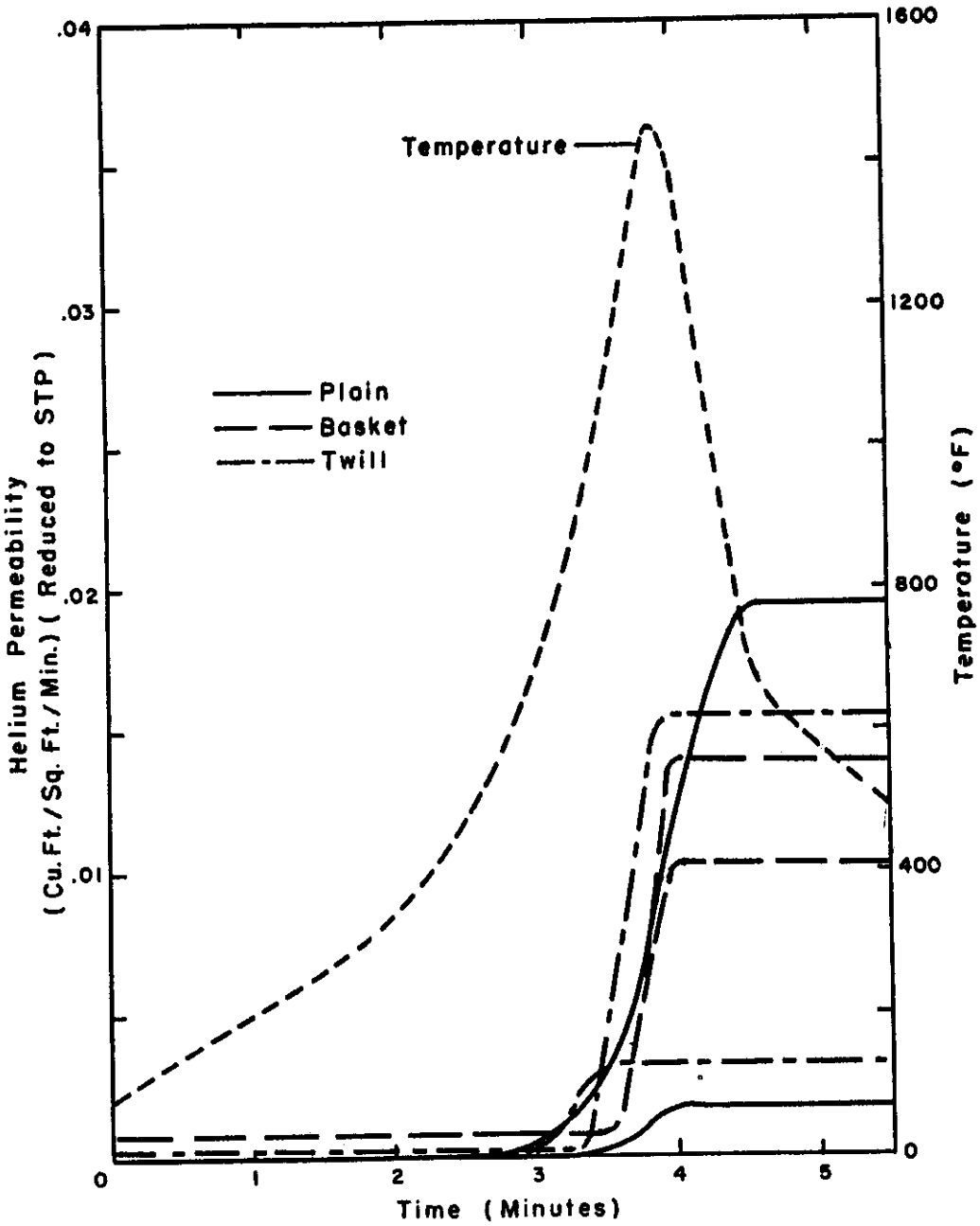

Figure 5 - Maximum and Minimum Permeabilities for Various Weaves

However, the basket weave has approximately 80% larger interstices than those of the 200 x 200 plain weave woven with the same wire diameter of 1.6 mils.

High Vacuum and UV Tests

The objective of this testing program was to determine the effects of combined high vacuum, ultra-violet radiation, and elevated temperature on the weight and performance characteristics of the selected CS105 elastomer coating. This test simulates some of the environmental conditions the coating will be exposed to when in orbit.

A specimen of René 41 wire cloth coated with CS105 coating was conditioned in an air circulating oven at 150°F for two hours and then removed and placed in a desiccator to cool. After the specimen was cooled it was weighed and then installed in the vacuum chamber. Thermocouples were mounted on the specimen by embedding the junction of the wires just below the outer surface of the material. The pressure in the vacuum chamber was reduced to approximately 5×10^{-6} mm Hg and the ultra-violet lamp was then turned on. Specimen temperature was controlled by adjusting the flow of cooling water in the heat exchanger coils. Thermocouples monitored specimen temperatures. A specimen temperature of 300°F was maintained by this method.

Length of exposure time was five days (120 hours), and after this elapsed time period the specimen was quickly reweighed. Samples were stored in a desiccator at all times. Test results are presented in Table II. A weight loss of 0.1% and a slight discoloration of the coating, due to the ultra-violet exposure, were the only deterioration effects. Figure 6 shows the test apparatus.

A second test was run using specimens with different coating weights and exposed for a period of 15 days and the results are presented in Table III. The test samples were 200 x 200 René 41 plain-woven wire cloth coated with CS105 elastomer. The material weight decreased 0.2% when exposed to vacuum and ultra-violet simultaneously.

The exposed samples were also tested for permeability. These test results are presented in the "Permeability Test" section of this paper. There was no significant change in the material performance due to ultra-violet and vacuum exposure.

TABLE II

ENVIRONMENTAL TEST DATA - 5-DAY EXPOSURE

Specimen	Weight Before (gm)	Weight After (gm)	Temperature (F)	Condition	Maximum Vacuum (mm Hg)
1 C8105-coated wire cloth	20.8892	20.8652	168°	Vacuum	5.0×10^{-6}
2 C8105-coated wire cloth	19.4748	19.4521	305°	Vacuum-UV	5.0×10^{-6}

TABLE III

ENVIRONMENTAL TEST DATA - 15-DAY EXPOSURE

Specimen	Weight Before (gm)	Weight After (gm)	Temperature (F)	Condition	Maximum Vacuum (mm Hg)
38 (coating weight 15.7 oz/yd^2)	15.6640	15.6735	165°	Vacuum	5.6×10^{-6}
43 (coating weight 14.0 oz/yd^2)	13.4733	13.4813	175°	Vacuum	5.6×10^{-6}
38a (coating weight 15.0 oz/yd^2)	14.9706	14.9367	300°	Vacuum-UV	5.6×10^{-6}
43a (coating weight 14.0 oz/yd^2)	12.5803	12.5528	300°	Vacuum-UV	5.6×10^{-6}

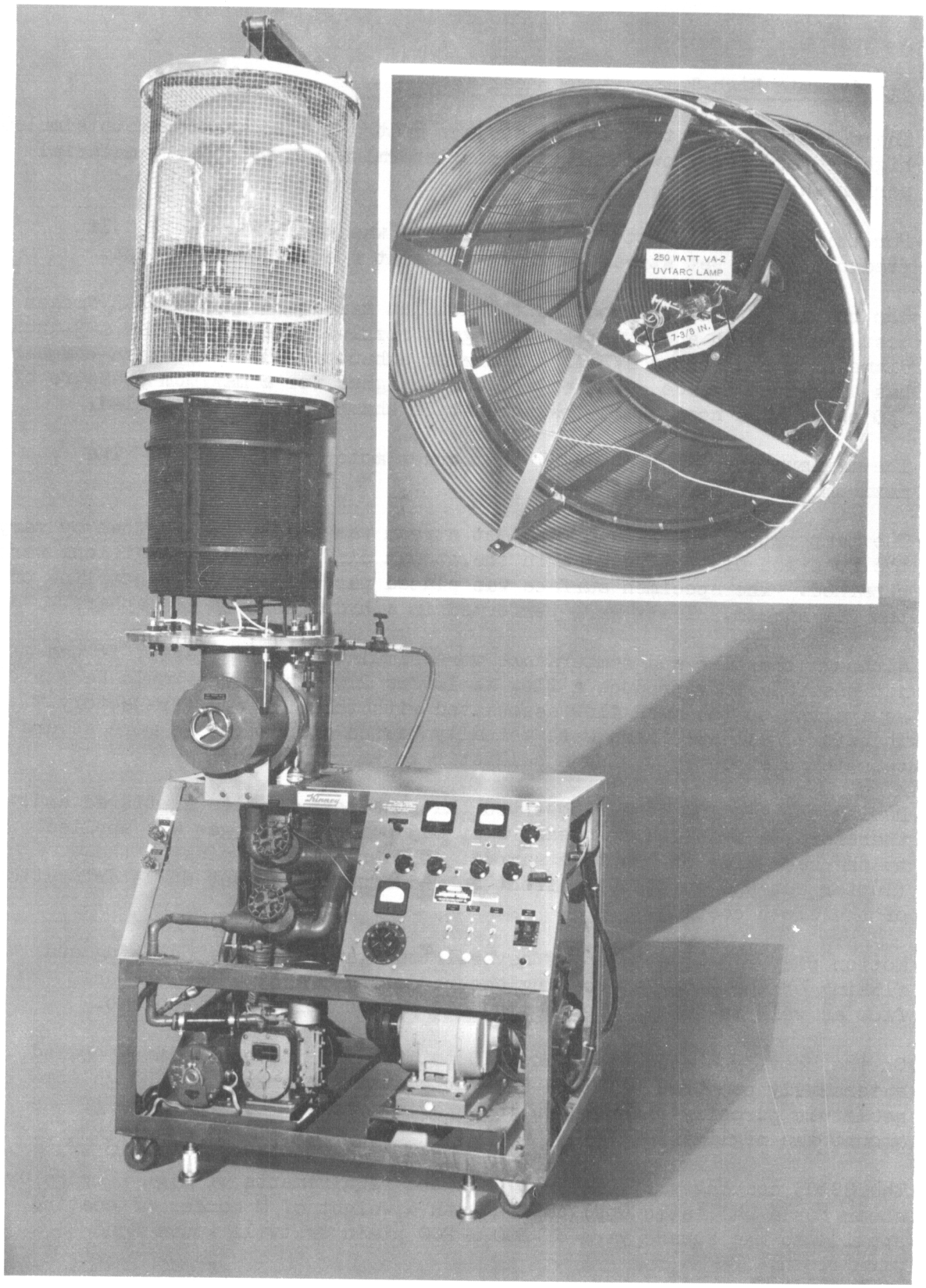

Fig. 6 Environmental test facility.

Aerodynamic High-Temperature Shear Test

The object of this test was to subject coated fabric specimens to simulated high-temperature flow conditions for determination of coating material performance.

For this test GAC's Hot-Roc test facility was used (Figure 7). It produces a hot jet stream to which the test specimen was exposed.

The rocket motor that produces the hot jet stream is an oxygen-hydrogen test rocket with a convergent-divergent nozzle. The nozzle has a 3-inch diameter throat and a 1/2-inch diameter exhaust. The combustion chamber has a $1\frac{1}{2}$-inch inside diameter and was designed for a maximum pressure of 150 psig. The nozzle and the combustion chamber are water cooled.

The maximum thrust produced by the rocket motor is 15 pounds. The maximum temperature is 5000°F.

The temperature and flow of the jet stream was calibrated so that by varying the position of the specimen in the stream different test conditions were obtained. The specimen surface was placed parallel to the centerline of the jet stream. Tests were performed in a hydrogen rich atmosphere.

Although the time and temperature were simulated in the tests, it was not possible to reproduce a flow as low as 200 fps, which would be equivalent to the mass flow associated with this re-entry trajectory. Therefore, all specimens were actually tested at conditions more severe than those expected during a ballistic re-entry trajectory.

During the testing it was observed that the test samples fluttered and, therefore, a stabilizing pressure of 0.5 psi of Argon gas was applied to the back side of one test sample. The pressure eliminated the flutter but had no visible effect on reducing the amount of deterioration of the cloth coating.

Motion pictures were taken of several tests and show that crazing and flaking of the material coating occurs during the high-temperature flow as well as during the cooling-off period following the test.

On the heavily coated specimens, 10 to 16 oz/sq yd, the coating crazed extensively compared with the lighter coated specimens. Although some geometric porosity occurred in the crevices of the crazed coating, the amount was negligible.

The CS105 coating satisfactorily met the requirements of high-temperature shear for a ballistic application with a weight of 8 ounces of coating applied to one square yard of 200 x 200 plain or twill weave René 41 wire cloth.

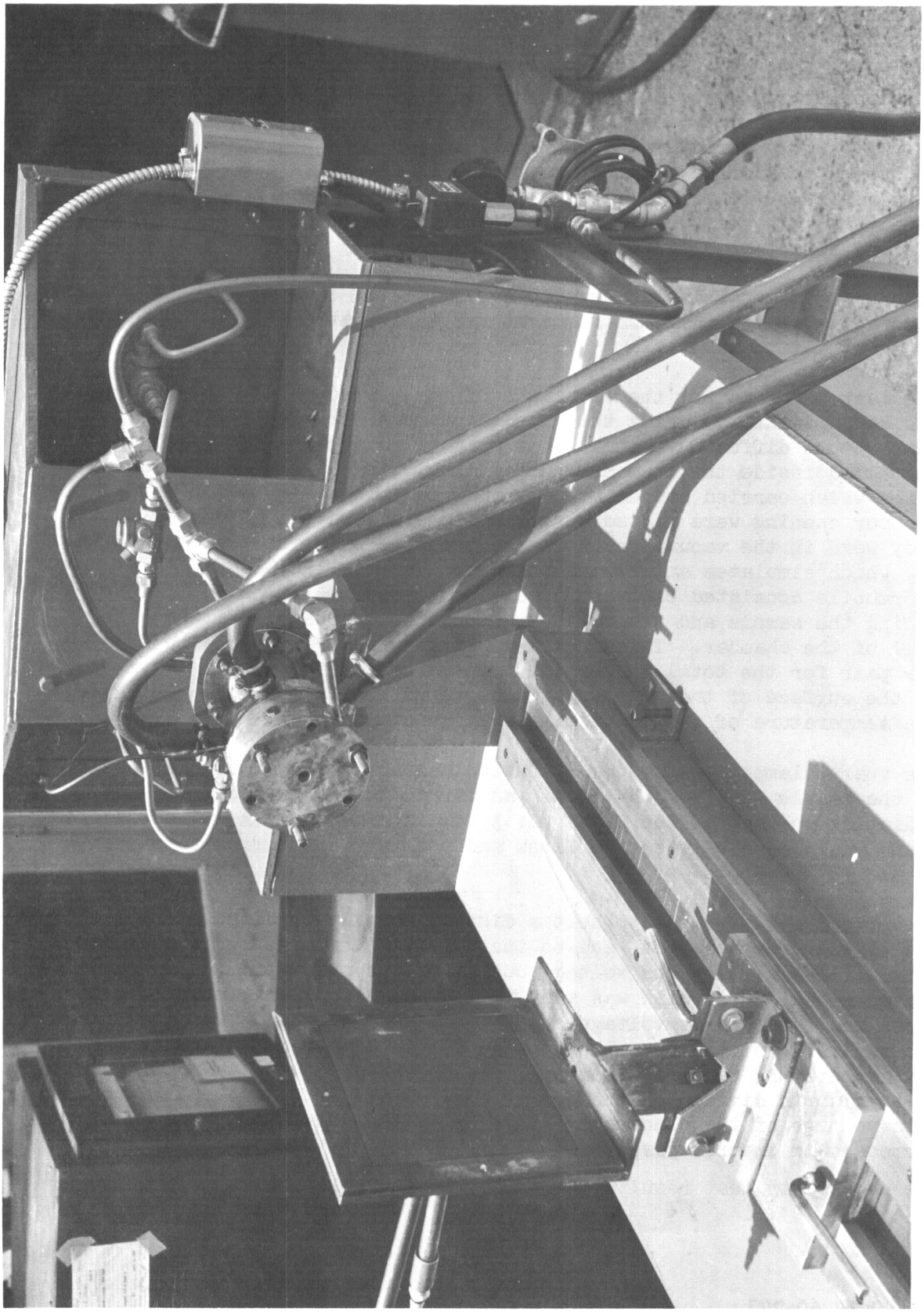

Fig. 7 Hot-roc test facility.

Emissivity Test

The emissivity of the surfaces of objects re-entering the earth's atmosphere from space is one of the most important factors determining the temperature attained by the objects. A high emittance is desirable to radiate the aerodynamic frictional energy off into space and thus to lower the surface temperatures. The maximum possible emittance at any particular temperature is that of the ideal black body and it is usual in making measurements to compare the emittance of a specimen with that of an actual black body or with a gray body of known emittance.

The objective of this test was to determine the emissivity of the coating material over the range of temperatures anticipated and at the simulated re-entry altitudes.

The task of measuring the emittance of the coated metal-cloth fabric was accomplished by using the same vacuum chamber used for the high-temperature diffusion tests. This chamber had quartz heating lamps installed inside the chamber and had an opening at one end for detachable discs which carried the emissivity samples. To a disc which fitted the chamber opening were fitted a thermopile and a sample holder so that they were in the vacuum chamber at a pressure of approximately 0.1 in. Hg, which simulates an altitude of approximately 120,000 ft. The thermopile consisted of twenty thermocouples with the hot junctions facing the sample and the cold junctions inside the vacuum near the wall of the chamber. Three pairs of wires were led out of the chamber; one pair for the total thermopile voltage, another from the thermocouple on the surface of the sample, and the third from a thermocouple giving the temperature of the thermopile cold junctions.

The quartz lamps, as for the diffusion tests, were used to heat one side of the sample and the other side was turned to the thermopile. The radiating surface of the discs was 3 in. in diameter. A stainless-steel plate was placed between the lamps and the fabric specimen to prevent localized hot spots.

The procedure was first to heat a disc of heavily oxidized Inconel, coated with camphor soot, and to increase its temperature at a constant rate while recording the voltage output of the thermopile. Next a coated metal-fabric disc was substituted and a similar temperature-time curve was followed as voltage output from the thermopile was again recorded. The total normal emittance of the high-temperature fabric at any temperature was then the time rate of change of the thermopile voltage of the fabric divided by the corresponding rate of change of the thermopile voltage of the black body (this being the value for the gray body increased in inverse proportion to its known emittance).

The emissivity test results are presented in Table IV.

TABLE IV

EMISSIVITY TEST DATA

Temperature (F)	Emissivity Coated Surface	Emissivity *Uncoated Surface
500°	.63	.72
800°	.82	--
1000°	.92	.95
1200°	.92	.95

The "black-body" used had an emissivity of .95 as estimated from the Handbook of Chemistry and Physics for oxidized metal with a camphor-soot coating.

*The uncoated surface is the reverse side of the same samples.

Conclusions

This program has demonstrated that a coating with an elastomeric base and high temperature additives can be formulated and applied to flexible woven structures, and that such a composite can perform a re-entry mission with the use of only materials which are in active production today.