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DEVELOPMENT OF A HIGH-TEMPERATURE VENTED RADIOISOTOPE CAPSULE

FINAL SUMMARY REPORT

JULY 1972

VOLUME 2

DEVELOPMENT AND TESTING OF NONSELECTIVE VENTING SYSTEMS

CONTRACT NO. AT(29-2)-2551

For

UNITED STATES ATOMIC ENERGY COMMISSION

TRW
SYSTEMS GROUP

ONE SPACE PARK • REDONDO BEACH • CALIFORNIA

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CONTENTS

	Page
1. INTRODUCTION AND SUMMARY	1
2. DISCUSSION OF RESULTS	6
2.1 VENT DESIGN AND ANALYSIS	6
2.1.1. Laboratory Evaluation of Alternate Vent Fabrication and Assembly Techniques	7
2.1.1.1 Piece-Part Fabrication	7
2.1.1.2 Alternate Vent Assembly and Bonding Techniques	9
2.1.1.3 Nondestructive Evaluation of Vent Assembly Techniques	11
2.1.1.4 Metallographic and SEM Evaluation of Assembled Vents and Vent Components	17
2.1.1.5 Selection of the Most Promising Alternate Fabrication and Assembly Techniques	32
2.1.2 Detailed Design of Final Phase I Test Vents . . .	34
2.1.2.1 Final Drawings	34
2.1.2.2 Fabrication Specifications	38
2.1.2.3 Material Specifications	44
2.1.2.4 Thermal Analyses of a Coaxial Vent	59
2.1.3 Development of a Helium Permeation Model	61
2.1.3.1 Analysis of Vent Permeability Characteristics	61
2.1.3.2 Physical Properties of the Ceramic . .	70
2.1.3.3 Comparison Between Permeability Theory and Experimental Flow Rates	71

CONTENTS (Continued)

	Page
2.1.3.4 Final Permeability Model	80
2.2 ELEVATED TEMPERATURE HELIUM PERMEATION EXPERIMENTS	80
2.2.1 Nondestructive Evaluation of Final Test Vents	80
2.2.2.1 Elevated Temperature Helium Permeation Test Apparatus	82
2.2.2.2 Standard Short Term Test Matrix.	86
2.2.3 Short Term Elevated Temperature Helium Permeation Testing-Results of Standard Matrix Experiments	87
2.2.4 Long Term, 2000°F Helium Permeation Testing of Vents	87
2.2.5 Metallographic Evaluation of As-Fabricated Vents (H-11, H-17, H-21, C-16, C-17, and C-22)	104
2.2.5.1 H-11 (helical, old thread design, FS-102 alumina)	104
2.2.5.2 H-17 (helical new thread design, FS-102 alumina)	104
2.2.5.3 H-21 (new 90° thread design, gold-plated TRW-100M platinized alumina)	108
2.2.5.4 H-22 (helical, Yttria)	108
2.2.5.5 C-16 (coaxial, FS-102 alumina)	108
2.2.5.6 C-17 (coaxial, TRW-100 alumina)	108
2.2.5.7 C-22 (coaxial, TRW-100M platinized alumina)	108
2.2.6 Metallographic Evaluation of Elevated Temperature Tested Vents	108
2.2.6.1 Vent H-16 (Helica, New Thread Design, FS102 Alumina, 1500 Hours of 2000°F Testing) . . .	108
2.2.6.2 Vent H-10 (Helical, Old Thread Design, FS102 Alumina, 25,020 Hours of 2000°F Testing, 3000°F Simulated Reentry Pulse)	112

CONTENTS (Continued)

	<u>Page</u>
2.2.6.3 Vent H-19 (Helical, New Thread Design, 100 Alumina, 23,920 Hours of 2000°F Testing)	112
2.2.6.4 Vent C-18 (Coaxial, TRW-100 Alumina, 23,920 Hours of 2000°F Testing, 3000°F Simulated Reentry Pulse)	115
2.2.6.5 Vent C-15 (Coaxial, FS102 Alumina, 23,920 Hours of 2000°F Testing).	115
2.2.6.6 Vent C-21 (Coaxial, TRW 100M Alumina, 23,920 Hours of 2000°F Testing).	115
2.2.6.7 C-23 (Coaxial, Yttria, 22,600 Hours of 2000°F Testing)	118
2.2.7 Comparison of Permeability Model with Data for the Final Phase I Test Vents	118
2.2.7.1 Comparison of Measured and Calculated Room Temperature Results	118
2.2.7.2 Comparison of Predicted and Experimental Helium Permeation Rates at Elevated Temperatures	120
2.3 HIGH TEMPERATURE VACUUM STABILITY OF CANDIDATE COATING-SUBSTRATE SYSTEMS	124
2.3.1 Test Specimens and Testing Conditions.	124
2.3.2.1 X-ray Diffraction Analyses	125
2.3.2.2 Metallographic and SEM Analysis.	126
2.3.2.3 Dimensional and Weight Changes	137
2.3.2.4 Surface Area, Pore Volume, and Porosity Measurements	137
2.4 STABILITY OF VENT SYSTEMS UNDER REENTRY HEATING	152
2.4.1 Stability Testing of Candidate Vent Materials Under Reentry Heating Conditions	152
2.4.2 Stability Testing of Vents C-18 and H-10 Under Reentry Heating Conditions	158
3. REFERENCES.	161

ILLUSTRATIONS

	Page
1. Nonselective Vent Configuration	3
2. Baseline Helical Vent Design	8
3. Baseline Coaxial Vent Design	8
4. Cores of helical and Coaxial Configurations	10
5. Dimensioned Sketches of Second Series of Vents	12
6. Partially Completed Coaxial (top) and Helical (bottom) Cores	13
7. Helium Permeation Test Setup for Room Temperature Measurements	15
8. Coaxial Vents	19
9. Helical Vent	20
10. Scanning Electron Microscope Photo of Plasma Arc Sprayed Alumina on Pt-10Rh	21
11. Scanning Electron Microscope Photos of Plasma Arc Sprayed Alumina Showing Both Crystallographic Forms of Alumina	22
12. SEM Photographs of As-Plasma-Arc-Sprayed Alumina: Fansteel-102 (top), TRW-100 (bottom)	23
13. SEM Photographs of Plasma-Arc-Sprayed Alumina after Heating for Two Hours at 2000°F in Air	24
14. SEM Photographs of Plasma-Arc-Sprayed Alumina after Heating for Four Hours at 2800°F in Air	25
15. SEM Photographs of Plasma-Arc-Sprayed Alumina after Heating for Four Hours at 2800°F in Vacuum	26
16. Scanning Electron Microscope Picture of Plasma Arc Sprayed Alumina from a Vent Tested Approximately Three Weeks at 2000°F	28
17. SEM Photographs of the Alumina Annulus in a Tested Coaxial Vent	29

ILLUSTRATIONS (Continued)

	Page
18. SRT-1 after Gas Pressure Bonding at 1800°F and 10,000 psi for 4-1/2 Hours	30
19. A Helical Vent Fabricated by Gas Pressure Bonding at 1800°F and 10,000 psi for 4-1/2 Hours	31
20. Eight Thread Helical Design	35
21. Four Thread Helical Design	36
22. Coaxial Design	37
23. Piece-Part Fabrication Flow Sheet	39
24. Piece-Part Fabrication Flow Sheet	40
25. Piece-Part Fabrication Flow Sheet	41
26. Vent Assembly Flow Sheet	42
27. Helical Vent with Plugs Welded in Ends of Vent Tube	45
28. Assembled Test Vents	46
29. Noble Metal Raw Stock Fabrication Flow Diagram	48
30. Spectrographic Analysis of Platinum Sponge	49
31. Spectrographic Analysis of Rhodium Sponge	50
32. Chemical Analysis of Pt-10Rh Alloy for Rhodium	51
33. Spectrographic Analysis of 0.110-Inch Diameter Rod	52
34. Spectrographic Analysis of 0.110-Inch Diameter Rod	53
35. Spectrographic Analysis of Platinum Sponge	54
36. Spectrographic Analysis of Rhodium Sponge	55
37. Chemical Analysis of Pt-10Rh Alloy for Rhodium	56
38. Spectrographic Analysis of 0.150-Inch O.D. x 0.25-Inch Wall Tubing	57

ILLUSTRATIONS (Continued)

	Page
39. Spectrographic Analysis of 0.150-Inch O.D. x 0.020-Inch Wall Tubing	58
40. Temperature Distribution for a Coaxial Vent	60
41. Pore Size Midpoints of Reheated, Plasma-Sprayed 12 μ Alumina	71
42. Helium Permeation Rate as a Function of Pressure Differential for a Variety of Vents	73
43. Flow Characteristics of an Annular Gap	75
44. Flow Characteristics of a Long Narrow Gap	76
45. Comparison of Predicted and Measured Helium Permeation Rates for Two Coaxial Vents	77
46. Correlation between Permeability Model and Experimental Permeability	79
47. Variation in Helium Flow Rate with Ceramic Area and Porosity	81
48. High Temperature Furnace for Helium Permeation Testing	84
49. Helium Permeation Test Setup for Elevated Temperature Measurements	85
50. Long Term Helium Permeation Test History for Vent H-10	97
51. Long Term Helium Permeation Test History for Vent H-19	98
52. Long Term Helium Permeation Test History for Vent C-15	99
53. Long Term Helium Permeation Test History for Vent C-18	100
54. Long Term Helium Permeation Test History for Vent C-21	101
55. Long Term Helium Permeation Test History for Vent C-23	102

ILLUSTRATIONS (Continued)

	<u>Page</u>
56. Long Term Helium Permeation Test History for Vent H-16, 10 ⁻² Atm ΔP.	103
57. Photomacrographs of H-11 Core and a Photomicrograph of the Assembled Helical Vent H-11 (old thread)	105
58. H-16 Vent Showing New Thread Design (Note: no void regions at the metal land/alumina interfaces)	106
59. Photomicrographs of Cross-Sectioned Vents	107
60. Photomicrographs of Helical Yttria Vent H-22	109
61. Photomicrographs of Cross-Sectioned Vents	110
62. Photomacrographs of Vent H-16 for Assembly and Photomicrographs of the Sectioned Vent after 1500 Hours of Testing	111
63. Noble Metal Vent H-10 (Helical, Old Thread Design, FS-102 Alumina) After 25,000 Hours of Permeation Testing at 2000°F Plus Exposure to a Simulated Reentry Pulse to 3000°F Maximum Temperature.	113
64. Noble Metal Vent H-19 (Helical, New Thread Design, TRW 100 Alumina) After 24,000 Hours of Permeation Testing at 2000°F	114
65. Noble Metal Vent C-18 (Coaxial, TRW-100 Alumina) After 24,000 Hours of Permeation Testing at 2000°F Plus Exposure to a Simulated Reentry Pulse to 3000°F Maximum Temperature	116
66. Noble Metal Vent C-15 (Coaxial, FS-102 Alumina) After 24,000 Hours of Permeation Testing at 2000°F	117
67. Effect of Vent Temperature on Permeation Rate	122
68. Comparison of 2000°F Measured Permeation Rates with Rates Predicted by the Permeation Model for Three Coaxial Vents . .	123
69. Cross Section of FS-102 Al ₂ O ₃ Coated Pt-10Rh After 30-Day 2600°F Vacuum Exposure	127
70. Photograph of Surface of TRW-100 Alumina in "As-Sprayed" Condition	129
71. SEM Photograph of Surface of TRW-100 Alumina After Vacuum Exposure to 2600°F for Three Days.	130

ILLUSTRATIONS (Continued)

	<u>Page</u>
72 SEM Photograph of Surface of TRW-100 Alumina after Vacuum Exposure at 2600°F for Thirty Days	131
73 SEM Photograph of Surface of TRW-100 Alumina after Vacuum Exposure to 2200°F for 18 Days	132
74 SEM Photograph of Surface of FS-102 Alumina in "As-Sprayed" Condition	133
75 SEM Photograph of Surface of FS-102 Alumina after Vacuum Exposure to 2600°F for Three Days	134
76 SEM Photograph of Surface of FS-102 Alumina after Vacuum Exposure at 2600°F for Thirty Days	135
77 SEM Photograph of Surface of FS-102 Alumina after Vacuum Exposure at 2200°F for Eighteen Days	136
78 SEM Photograph of Platinized Alumina in "As-Sprayed" Condition	138
79 SEM Photograph of Platinized Alumina after Vacuum Exposure at 2600°F for Three Days	139
80 SEM Photograph of Platinized Alumina after Vacuum Exposure at 2600°F for Thirty Days	140
81 SEM Photograph of Platinized Alumina after Vacuum Exposure at 2200°F for Eighteen Days	141
82 SEM Photograph of Yttria in "As-Sprayed" Condition	142
83 SEM Photograph of Yttria after Vacuum Exposure at 2600°F for Three Days	143
84 SEM Photograph of Yttria after Vacuum Exposure at 2600°F for Thirty Days	144
85 SEM Photograph of Yttria after Vacuum Exposure at 2200°F for Eighteen Days.	145
86 Ribbon Furnace	153
87 Reentry Temperature Profile	154

ILLUSTRATIONS (Continued)

	<u>Page</u>
88 Untested Yttria Coated Pt-10Rh Specimen	156
89 Reentry Heated Yttria Coated Pt-10Rh Specimen	156
90 Cross-Section of TRW-100 Alumina Coated Specimen after Reentry Heating	157
91 Cross-Section of Yttria Coated Specimen after Reentry Heating	157
92 Reentry Temperature Profile for Testing Vents C-18 and H-10	159

TABLES

	Page
1. Helium Permeation Data and Thermal Testing History for Preliminary Evaluation Vents	16
2. Vents Fabricated for Final Phase I Testing	33
3. Values of K_v for Viscous Flow in Various Sectional Shapes	64
4. Values of K_k Calculated from Clausing's Tables	65
5. Constants Used in Flow Model	70
6. Assumed Properties of Alumina PAS Ceramics	72
7. Permeabilities of Epoxy and Cold Pressed Vents	78
8. Room Temperature Helium Permeation Measurements on Final Phase I Test Vents	83
9. Summary of Initial Elevated Temperature Helium Permeation Measurements on Vents H-10, H-16, H-19, C-15, C-18, and C-21	88
10. Helium Permeation Test History - Vent H-10 (Drawing No. X405345)	90
11. Long Term Helium Permeation Testing of Vent H-19	92
12. Long Term Helium Permeation Testing of Vent C-15.	93
13. Long Term Helium Permeation Testing of Vent C-18.	94
14. Long Term Helium Permeation Testing of Vent C-21.	95
15. Long Term Helium Permeation Testing of Vent C-23.	96
16. Comparison of Measured and Predicted Specific Permeabilities for Coaxial Vents	119
17. Comparison of Measured and Predicted Permeabilities for Helical Vents	121
18. Effect of Temperature on Helium Permeation Rate	120
19. X-ray Diffraction of Vacuum Stability Specimens	125

TABLES (Continued)

	<u>Page</u>
20. Weight and Dimensional Changes from Exposure to 2600°F for Three Days.	146
21. Weight and Dimensional Changes from Exposure to 2600°F for Thirty Days	147
22. Weight and Dimensional Changes from Exposure to 2200°F in Vacuum for Eighteen Days	148
23. Summary of Surface Area Measurements on Coated Tubes.	150
24. Summary of Pore Volume, Mean Pore Size and Porosity Measurements.	151
25. Summary of X-ray Diffraction Results on Reentry Heated Specimens	158
26. Helium Permeation Rate of Vent C-18 Before and After Reentry Heating Test.	160
27. Helium Permeation Rate of Vent H-10 Before and After Reentry Heating Test.	160

1. INTRODUCTION AND SUMMARY

Volume II of this final technical summary report describes the work performed by TRW for the Atomic Energy Commission (November 1969 to June 1972) on the design, development and long term testing of nonselective noble metal/ceramic helium venting systems for high temperature radioisotope heat source capsules. This effort, performed as part of Contract AT(29-2)-2551,* was originally aimed at the development of a high temperature gas management device for use with a radioisotope capsule system consisting of a noble metal liner, refractory metal strength member and noble metal cladding. Although the concept of a high temperature radioisotope heat source with a noble metal liner has, to date, not been actively pursued for space application, the venting systems developed under this contract were the technological precursors to the refractory metal/ceramic pressure relief devices (PRD's) currently being used on the Transit and Pioneer RTG's.

Some eighteen months prior to the start of this contract (1968), TRW initiated extensive investigations under company funding of a wide variety of concepts for venting helium from high temperature radioisotope capsules. Both selective and nonselective devices were evaluated and it rather quickly became apparent that the nonselective venting approach had a number of major advantages over selective venting concepts as was described in detail in the Interim Technical Summary Report.**

In view of the advantages of the nonselective venting approach, the bulk of TRW's company sponsored research and preliminary development efforts were aimed at evaluation and feasibility testing of nonselective venting

* Volume I of this final report describes the work performed under Contract AT(29-2)-2551 on the development of noble metal claddings and high emittance coatings for the SNAP-29 system. Volume III presents a summary of the high temperature compatibility studies conducted in conjunction with the vent development work.

** TRW(A)-11069-040, 1 December 1969.

devices. After an initial screening study, two vent configurations were chosen for more detailed evaluation and were tested extensively. These configurations which TRW designates as the coaxial and helical designs are shown schematically in Figure 1. Both systems consist of thin, porous, ceramic oxide helium permeation paths which are totally supported in a ductile noble metal matrix. In the coaxial design, the helium permeates through a cylindrical ceramic annulus, while in the helical design, the helium follows a spiral ceramic path. TRW evaluation of these two designs during the company sponsored program included successful completion of the following key feasibility demonstration tasks.

- Impact tested a prototype cylindrical vented capsule (end-on) against granite at a velocity of 240 ft/sec and temperature of 1400°F, demonstrating complete integrity of the coaxial vent on the impacted end and the helical vent on the nonimpacted end.
- Demonstrated the ability of the baseline vent configuration to completely retain 0.1 micron radioisotope particles while passing helium at a rate 650 times faster than would be required for the baseline capsule design.
- Conducted helium permeation rate experiments with the two candidate vent configurations and demonstrated the stability of the vents at 2000°F for test periods of up to 3750 hours.
- Conducted krypton and air permeation experiments and demonstrated the stability of the vent system in air (2000°F) for test periods of 48 hours.
- Demonstrated that thermal cycling of the candidate vent configurations between 2000°F and room temperature did not affect the helium permeation rate.
- Demonstrated that thermal shocking the baseline configurations by heating to 2000°F and plunging into water did not affect vent integrity.
- Conducted sea water permeation experiments with a helical vent assembly at a differential pressure of 260 psig and detected no transport of sea water through the vent to a simulated capsule cavity in a time period of 285 hours.

Figure 1. Nonselective Vent Configurations

Based on the successful feasibility demonstration experiments, the helical and coaxial designs were selected as baseline vent configurations for this program. The primary objectives of this vent development phase of the program were threefold, as follows:

- 1) To complete the characterization of the two alternate vent designs.
- 2) To establish a reproducible sequence of fabrication and assembly techniques for each design.
- 3) To verify the stability of the venting systems under very long term, steady state, (2000°F) operating conditions.

The great majority of the vent development and testing effort was successfully completed during the period from April to October 1969. Long term vent stability testing under steady state conditions (2000°F, 10^{-2} atm ΔP) was continued, however, until June 1972. The major results of the vent development and testing program are briefly summarized below:

- Detailed design and manufacturing specifications for both coaxial and helical vent configurations were prepared and the basic reproducibility of the manufacturing sequences were demonstrated (Section 2.1).
- A vent permeation model was developed which correlated reasonably well with the measured helium permeation rates of the final manufactured devices (Sections 2.1.3 and 2.2.7).
- Short term stability of the helium permeation devices over the temperature range from RT-2200°F was demonstrated. The venting systems survived repeated thermal cycles from RT to 2200°F without any change in their measured helium permeation rates (Section 2.2.3).
- Six vents representing both the coaxial and helical designs and several different types of ceramic permeation materials were completely stable in long term helium permeation tests at 2000°F. No change was noted in the helium permeation rates of any of the six vents after approximately 25,000 hours of testing at 2000°F. During this nearly three year testing sequence the six vents were periodically cycled from RT to 2200°F (Section 2.2.4).

- After the 25,000 hour stability tests, two of the vent systems were subjected to a simulated reentry environment (peak temperature 3000°F) and their helium permeation rates (at RT and 2000°F) retested. No change was observed in the helium permeation rates of the vents after the reentry testing (Section 2.4.2).
- Metallographic examination of several of the vents after ~25,000 hours of steady state testing did not show any degradation of the materials (Section 2.2.6).

Based on the results obtained in this vent development work it is concluded that both the helical and coaxial configurations using noble metal materials and porous alumina helium permeation paths are extremely stable under conditions of operation up to at least 2000°F. Details of the development effort and testing program are presented in the body of this volume of the final summary report.

2. DISCUSSION OF RESULTS

This section of the final report details the major accomplishments in the development and testing of nonselective venting systems from program initiation in April 1969 through completion of the contractual effort in July 1972.

2.1 VENT DESIGN AND ANALYSIS

The goal of this task was to design, fabricate, and analyze the performance of helium venting radioisotope filters with the following characteristics:

- Nonselective in that gases other than helium can readily permeate the filtering medium.
- Permeable to helium at a rate of 1.25×10^{-5} atm. cm^3/sec at 10^{-1} to 10^{-3} atmosphere differential pressure at 2000°F.
- Capable of totally retaining all fuel particulate matter during the period of capsule operation in space, reentry, impact, and post impact, a period of five years or more.
- Good thermal shock and impact resistance.
- Capable of withstanding the vibration and acceleration incurred during rocket launch.
- Capable of withstanding with high probability a launch pad abort.
- Chemically and mechanically stable at 2000°F in vacuum for at least five years.
- Chemically and mechanically stable for several minutes at 2800°F in air.
- Extremely good oxidation and corrosion resistance in air-soil-water environment.

The design concept on which each vent configuration was based was to support a thin, porous oxide ceramic filtering medium in a ductile noble metal matrix.

2.1.1 Laboratory Evaluation of Alternate Vent Fabrication and Assembly Techniques

Two nonselective, helium venting configurations (coaxial and helical) were initially evaluated under company sponsorship and form the basis for this program (see Section 1). Both configurations utilize a porous ceramic, helium permeation path such as Al_2O_3 which is plasma arc sprayed (PAS) onto a ductile central metallic core. After finish machining, the core is inserted into a tube and the two components pressed together and bonded by one of several alternate techniques.

2.1.1.1 Piece-Part Fabrication

Figure 2 illustrates the structure of the initial baseline helical vent design evaluated during this program. The starting materials were a Pt-10Rh rod, Pt-10Rh tube, and $\alpha\text{-Al}_2\text{O}_3$ powder. The rod was machined straight and round and its surface finish maintained to insure dimensional tolerances. Extruded seamless tubing was machined and lapped to minimize any inside wall surface imperfections. The baseline sequential component fabrication procedure was as follows:

- Machined helix, straight grooves, and end center in the Pt-10Rh rod.
- Grit blasted the rod with alumina.
- Plasma arc sprayed high purity alumina over the entire cylindrical surface of the rod keeping the end centers clean. The helical and straight grooves were completely filled.
- Ground the PAS rod to the inside diameter of the finished machined tube. This was accomplished using an O.D. grinder with a silicon carbide wheel.
- Finish machined the inside surface of the mating tube utilizing a reaming and lapping operation.

Figure 2. Baseline Helical Design

Figure 3. Baseline Coaxial Design

Figure 3 illustrates the structure of the initial coaxial baseline vent design investigated during this program. The baseline piece-part fabrication procedures for this configuration were as follows:

- Machined rod to insure dimensional tolerances. Machined centers in ends of rod.
- Grit blasted with high purity alumina.
- Plasma arc sprayed high purity alumina over the entire cylindrical surface of the rod keeping the centers clean. The coating thickness was 0.010-inch to allow for final grinding.
- Ground the plasma-arc-sprayed rod to a diameter which was about 0.0002-inch less than the inside diameter of the mating tube. This was accomplished using an O.D. grinder with a silicon carbide grinding wheel.
- Finish machined the inside surface of the mating tube utilizing a reaming and lapping operation.

2.1.1.2 Alternate Vent Assembly and Bonding Techniques

Two methods of assembling and bonding the coaxial and helical core configurations into the vent housings or tubes were evaluated during the first month of the program. One assembly method utilized a cold drawing operation to effect a good mechanical bond between the cores and tubes. This process lent itself to the processing of long lengths of vent cores which in turn were cut up into short sections. The long cores of both the coaxial and helical configuration are shown in Figure 4.

The second assembly and bonding process evaluated utilized a cold pressing operation. In this process, the completed-core and tube were assembled and placed within a split, steel die. A pneumatic press was then used to press the vent into the slightly smaller die cavity in order to effect a bond between the tube and filter core. The stress was varied to obtain optimum bonding results. The full-scale, relatively short length, core subassemblies are shown in Figure 4 after plasma arc spraying the rods with $\alpha\text{-Al}_2\text{O}_3$.

Alternate vent assembly and bonding techniques evaluated in the second and third month of the program were isostatic pressing at room temperature and gas pressure bonding or elevated temperature isostatic pressing. Isostatic pressing utilized hydrostatic or gas pressure for the direct joining

Figure 4. Cores of Helical and Coaxial Configurations

of the components. The components to be bonded were fabricated to final size, cleaned, and assembled. The cores were sealed in the close-fitting housings or tubes by welding in vacuum and placed inside an autoclave and subjected to high external pressure (hydrostatic at room temperature and gas at elevated temperature). Sufficient pressure was generated to deform the components plastically and bring them into intimate contact.

The four above mentioned vent assembly and bonding* techniques were evaluated for both the helical and coaxial configurations. The cold drawing and cold pressing assembly techniques resulted in good PAS alumina-to-rod and PAS alumina-to-tube bonds. However, the cold drawing and cold pressing techniques did not affect as good a metal-to-metal bond (core lands-to-tube) in the case of the helical configuration as was desired. On the other hand, the isostatic pressing techniques resulted in vents with superior metal-to-metal and metal-to-ceramic bonds to those obtained by cold pressing or cold drawing techniques, and the elevated temperature technique (gas pressure bonding) resulted in an adequate diffusion bond between the metal-to-metal portions of the helical assembly. All four candidate assembly techniques, however, effected a much better metal-to-ceramic bond than the shrink fitting technique previously evaluated in TRW's IR & D program.

A total of seventeen preliminary evaluation vents (8 coaxial and 9 helical) were assembled during the early phase of the program and were extensively evaluated. These vent assemblies were fabricated to the baseline design (Figures 2 and 3) and were assembled utilizing the cold pressing, cold drawing, and room temperature isostatic pressing techniques.

A second series of preliminary evaluation vents (6 coaxial and 6 helical) were fabricated to evaluate the elevated temperature isostatic pressing technique for assembling and bonding the vent cores to the housings. Figure 5 shows dimensional sketches of this series of vents. Figure 6 shows partially completed coaxial and helical cores.

2.1.1.3 Nondestructive Evaluation of Vent Assembly Techniques

In order to provide an initial screening test for nondestructively evaluating candidate vent fabrication techniques, measurements of helium

* Cold drawing, cold pressing, isostatic pressing at room temperature, and isostatic pressing at elevated temperature.

(a) HELICAL CERAMIC DESIGN

(b) COAXIAL, ANNULAR CERAMIC DESIGN

NOTE:
ALL DIMENSIONS
IN INCHES

(c) Pt-10 Rh TUBE USED IN BOTH HELICAL AND COAXIAL DESIGNS

Figure 5. Dimensioned Sketches of Second Series of Vents

Figure 6. Partially Completed Coaxial (Top)
and Helical (Bottom) Cores

permeation rates as a function of helium pressure differential were conducted at room temperature. Figure 7 shows a photo of the helium permeation rate test setup for room temperature measurements. The vent being tested was connected between a mass spectrometer type leak detector and a helium pressure manifold. The helium pressure differentials across the vents were varied by evacuating the helium pressure side of the system with a mechanical pump and back-filling with helium to the desired pressure range as indicated by utilizing a MKS Baratron electronic pressure meter which compares pressures to 0.05% accuracy. The rates of helium permeation were measured with a Consolidated Electrodynamics Corporation Type 24-120B leak detector with a sensitivity of 1×10^{-10} std cm³/sec/division. The leak detector was capable of measuring leaks in the 1×10^{-10} to 1×10^{-2} std cm³/sec range. A standard helium leak with a permeation rate of 2.8×10^{-8} std cm³/sec was used to calibrate the leak detector.

Table 1 shows the helium permeation rate data generated for 22 vents fabricated during the period of initial evaluation of alternate vent assembly techniques. The C and H dash numbers refer to coaxial and helical vent configurations, respectively. The SRT-1 identification refers to a coaxial vent fabricated without a ceramic annulus, i.e., the device consists of a Solid Rod and Tube. The room temperature permeation data shown in Table 1 are tabulated as a function of vent history. For example, the following information regarding H-2 (a helical vent) is shown in Table 1:

- The component parts were not heat treated prior to cold drawing.
- After component assembly, the vent was cold drawn.

After cold drawing the following operations were performed sequentially:

- Heat treated for two hours at 2000°F in air.
- Measured helium permeation rate.
- Heat treated for four hours at 2800°F in air.
- Measured helium permeation rate.
- Heat treated again for four hours at 2800°F in air.
- Measured helium permeation rate.

Figure 7. Helium Permeation Test Setup for Room Temperature Measurements

TABLE 1. HELIUM PERMEATION DATA AND THERMAL TESTING HISTORY FOR PRELIMINARY EVALUATION OF VENTS

I. D. NUMBER	2000° F, 2 HRS PRIOR TO FABRICATION	INITIAL FABRICATION TECHNIQUE				PERMEATION TEST AT 10 ⁻² ATM ΔP	2000° F, 2 HRS AFTER FABRICATION	PERMEATION TEST AT 10 ⁻² ATM ΔP	HOURS OF ADDITIONAL TREATMENT			PERMEATION TEST AT 10 ⁻² ATM ΔP	HOURS OF ADDITIONAL TREATMENT			PERMEATION TEST AT 10 ⁻² ATM ΔP	ISOSTATIC PRESS **	PERMEATION TEST AT 10 ⁻² ATM ΔP	HOURS OF ADDITIONAL TREATMENT			ISOSTATIC PRESS **	PERMEATION TEST AT 10 ⁻² ATM ΔP	HOURS OF ADDITIONAL TREATMENT			ISOSTATIC PRESS **	PERMEATION TEST AT 10 ⁻² ATM ΔP																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																									
		A	B	C	D				2000° F	2800° F	3100° F		2000° F	2800° F	3100° F				2000° F	2800° F	3100° F			2000° F	2800° F	3100° F			2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F	2000° F	2800° F	3100° F

A= COLD PRESSED

B= COLD DRAWN

C= ISOSTATICALLY PRESSED AT ROOM TEMPERATURE

D= ISOSTATICALLY PRESSED AT ROOM TEMPERATURE FOLLOWED BY AN ISOSTATIC PRESS AT 1800°F

*Heat treatments were done in air unless otherwise noted.

**The room temperature isostatic pressing done at this point was done after initial fabrication.

***Temperature control was lost and the vent melted at approximately 3375°F.

*****Thermal shock consisted of heating to 2000°F and plunging in ambient water.

- Isostatically pressed at room temperature for six minutes at 60,000 psi.
- Measured helium permeation rate.
- Heat treated again for two hours at 2000°F in air.
- Measured helium permeation rate.
- Heat treated for 187 hours at 2000°F in air.
- Measured helium permeation rate.

The following observations were made from an analysis of the permeation data in Table 1:

- The room temperature helium permeation rate of each vent increased as a result of heat treatment at 2800°F in air regardless of the initial fabrication technique.
- Isostatic pressing at room temperature decreased the room temperature helium permeation rate of a vent regardless of previous history.
- Sequential heat treatments of a vent after an initial heat treatment without intervening pressing had little or no effect on its room temperature helium permeation rate. Permeation rates changed little even after soaks in air at 2000°F for 187 hours.
- Room temperature isostatic pressing followed by high temperature isostatic pressing of assembled vent components resulted in vents with the lowest rates of room temperature helium permeation.

2.1.1.4 Metallographic and SEM Evaluation of Assembled Vents and Vent Components

Metallographic examination of fabricated vents was used to evaluate the effect of several candidate vent assembly and bonding techniques on the ceramic-to-metal and metal-to-metal interfaces of the two vent configurations. Good contact between the plasma-arc-sprayed (PAS) alumina ceramic and the adjacent Pt-10Rh metal parts is required for both the coaxial and helical configurations. Metal-to-metal diffusion bonding between the metal lands of the helical core and inside diameter of the tubing is considered necessary in the case of the helical configuration. Scanning electron microscope (SEM) examinations were used to investigate the microstructure of the PAS alumina filtering media. The primary advantage of the SEM over the optical metallograph was its great depth of focus at magnifications

of up to several thousand times. The topographical details and internal structure of the PAS alumina were analyzed in an as-fabricated geometry.

Figure 8 shows polished cross sections of coaxial vents C-1 and C-2 fabricated by cold drawing and cold pressing, respectively. In both cases, the PAS alumina-to-rod and PAS alumina-to-tube bonds look excellent. Figure 9 shows the polished cross section of the helical vent H-1. The cold drawing technique whereby assembled vent components were drawn through steel die blocks effected a much better bond between the metal lands on the helical core and the tubing wall than a shrink fitting technique (tube heated to fit over cool core) evaluated earlier under TRW funding. Only a very thin line approximately 1-micron wide could be seen between the core lands and tube of H-1 (Figure 9).

Figure 10 is a typical photo of PAS alumina on Pt-10Rh taken with a SEM. A good semi-quantitative evaluation of the size, shape and orientation of the grains as well as pore size and distribution is obtained from SEM photos such as this.

Additional SEM photos are shown in Figure 11. The photos are of PAS alumina on Pt-10Rh after grinding to almost a glazed finish. The following observations were made from examinations of these SEM photos:

- Gamma alumina, the crystal form in the as-PAS condition, was indistinguishable from alpha alumina, the crystal form of alumina obtained on heating gamma alumina to temperatures above 1600°F for several hours.
- The internal pore distribution appeared quite uniform.
- The internal pore size was no larger than a few microns.
- The relatively large smooth surfaces seen at 1000X were probably formed by particle packing or crushing during contact with the grinding wheel.
- Gas holes ranging in size from 0.2 to 1.0 micron were formed in individual alumina platelets during the PAS process.

Figures 12 through 15 show SEM photos of two types of alumina, Fansteel Metallurgical's FS-102 and TRW-100, evaluated for use as vent filter media. Microstructures of the two aluminas were compared in the as-PAS condition, after two hours at 2000°F in air, after four hours at 2800°F in air, and

C-1 As Cold Drawn 100X Original

C-1 As Cold Drawn 400X Original

C-2 As Cold Pressed 100X Original

C-2 As Cold Pressed 400X Original

Figure 8. Coaxial Vents

H-1

As Cold Drawn

100X Original

H-1

As Cold Drawn

100X Original

Figure 9. Helical Vent

Figure 10. Scanning Electron Microscope Photo of Plasma Arc Sprayed Alumina on Pt-10Rh

Area 1 Gamma Alumina 1000X Original

Area 1A Alpha Alumina 1000X Original

Area 2 Gamma Alumina 3000X Original

Area 2A Alpha Alumina 4000X Original

Figure 11. Scanning Electron Microscope Photos of Plasma Arc Sprayed Alumina Showing Both Crystallographic Forms of Alumina

R-1 FS-102 1000X Original

R-1 FS-102 3000X Original

R-6 TRW-100 1000X Original

R-6 TRW-100 3000X Original

Figure 12. SEM Photographs of As-Plasma-Arc-Sprayed Alumina: Fansteel-102 (top), TRW-100 (bottom)

Figure 13. SEM Photographs of Plasma-Arc-Sprayed Alumina after Heating for Two Hours at 2000°F in Air

Figure 14. SEM Photographs of Plasma-Arc-Sprayed Alumina after Heating for Four Hours at 2800°F in Air

R-4 FS-102 1000X Original

R-4 FS-102 3000X Original

R-9 TRW-100 1000X Original

R-9 TRW-100 3000X Original

Figure 15. SEM Photographs of Plasma-Arc-Sprayed Alumina after Heating for Four Hours at 2800°F in Vacuum

after four hours at 2800°F in vacuum (10^{-5} torr range). The following observations were made from examinations of these SEM photos:

- In the as-PAS conditions, the FS-102 structure consisted of lamellae stacked parallel to the surface of the substrate. The TRW-100 structure consisted of an agglomeration of angular particles.
- After two hours at 2000°F in air, no change was detected in the FS-102 structure. The TRW-100 microstructure showed a rounding of particles perhaps due to surface diffusion.
- After four hours at 2800°F in air, the FS-102 particles had rounded and a substructure formed. The TRW-100 particles were rounded and many small particles appeared to be loose.
- After four hours at 2800°F in vacuum, the FS-102 appeared granular. The TRW-100 particles were rounded and appeared sintered into agglomerations.

Figure 16 illustrates the fact that SEM evaluation of an unpolished ceramic is absolutely necessary for a clear understanding of its structure. The top SEM photo shows the polished surface of alumina in a helical vent fabricated during an independent TRW program; it is very similar to those which were taken on a metallograph. The two bottom photos in Figure 16 show a view of the underlying structure through a purposely fractured portion of the alumina. As can be seen readily, the polished surface bears little relationship to the true structure seen below. Figure 17 shows a SEM photo of the PAS alumina portion of the tested coaxial vent C-6 (see Table 1 in Section 2.1.1.3). The structure (rounded particles) is very similar to other PAS FS-102 alumina specimens that have seen 2800°F for a few hours. In addition to the rounded particles, ground surfaces and a few loose particles could also be seen.

Figures 18 and 19 show photomicrographs of SRT-1 (coaxial solid rod and tube) and the helical vent H-13. Both vents were fabricated by the high temperature isostatic pressing technique (1800°F, 10,000 psi, 4-1/2 hours). In both cases, good metallurgical (atomically bonded) bonds were formed between the metal components of each device, i.e., between the solid rod and tube and between the metal lands of the H-13 core and the inner diameter of the H-13 tube. Although some voids were detected along the metal-to-metal interfaces, sufficient diffusion bonding occurred to block helium flow along the metal-to-metal interfaces; this resulted in a no-flow

Area 2 S-7 Vent 1000X Original

Area 1 S-7 Vent 1000X Original

Area 1 S-7 Vent 3000X Original

Figure 16. Scanning Electron Microscope Picture of Plasma Arc Sprayed Alumina from a Vent Tested Approximately 3 Weeks at 2000°F

C-6

1000X Original

C-6

3000X Original

Figure 17. SEM Photographs of the Alumina Annulus in a Tested Coaxial Vent

SRT-1

Etched

100X

Figure 18. SRT-1 After Gas Pressure Bonding at 1800°F and 10,000 psi for 4-1/2 Hours

H-13

Bright Field

50X

H-13

Bright Field

400X

Figure 19. A Helical Vent Fabricated by Gas Pressure Bonding
at 1800°F and 10,000 psi for 4-1/2 Hours

condition for SRT-1 and the desired low helium permeation rate through H-13 (Table 1, Section 2.1.1.3).

2.1.1.5 Selection of the Most Promising Alternate Fabrication and Assembly Techniques

Table 2 shows the alternate vent systems which were fabricated for the final Phase I high temperature helium permeation testing. Two yttria-containing vents are also shown in Table 2. In the case of the helical configuration, three different fabrication techniques were used to build nine vents (three of each type) one for metallographic evaluation, one for high temperature helium permeation testing, and one to be held in reserve. The three techniques included vents fabricated with two different thread designs and vents with gold plated cores. The new thread (90° included angle with a bottom radius) was designed to improve the ceramic-to-metal bond by making it possible to orient the plasma arc spray nozzle almost 90° to the core. A set of three vent cores were coated with a thin (a few microns thick) layer of vapor deposited gold in an effort to enhance the diffusion rate between the core metal lands and the inside diameter of the vent tube. Gold was chosen because of its relatively low melting point (1945°F), and because it forms a continuous series of solid solutions with platinum. All nine helical vents fabricated were isostatically pressed first at room temperature then at high temperature.

Three different fabrication techniques were also used to fabricate nine coaxial vents for the final Phase I high temperature helium permeation testing. The three techniques included vents with three different ceramic materials; Fansteel FS-102 alumina, TRW-100 alumina, and TRW-100M platinized alumina. The goal of the elevated temperature tests was to determine the stability of each metal-ceramic system as measured by its helium permeation rate as a function of time. All nine coaxial vents fabricated had 0.005-inch thick ceramic annuli, and each was isostatically pressed first at room temperature then at high temperature. The reasons for selecting the three types of ceramic materials are summarized below.

FS-102 Alumina

- Most of the plasma arc spray experience to date at TRW has been with this alumina.

TABLE 2. VENTS FABRICATED FOR FINAL PHASE I TESTING

DESIGN PARAMETERS	VENT NUMBER																	
	H-10	H-11	H-12	H-16	H-17	H-18	H-19	H-20	H-21	H-22	C-14	C-15	C-16	C-17	C-18	C-19	C-20	C-21
HELICAL																		
Old Thread Design (full 0.015-Inch Radius)																		
New Thread Design (90° included angle with a bottom radius)																		
Gold Plated Core																		
Yttria																		
Platinized Alumina (TRW-100M)																		
Fansteel Alumina (FS-102)																		
Isostatically Cold Pressed																		
High Temperature Gas Pressure Bonded																		
COAXIAL																		
Fansteel Alumina (FS-102)																		
TRW Alumina (TRW-100)																		
Platinized Alumina (TRW-100M)																		
Yttria																		
0.005-Inch Thick Annulus																		
Isostatically Cold Pressed																		
High Temperature Gas Pressure Bonded																		

It is readily prepared by blending two commercially available alumina powders.

TRW-100 Alumina

- In the as-plasma-arc-sprayed condition, it is $\sim 2/3$ alpha alumina and $\sim 1/3$ gamma alumina as opposed to FS-102 which is mostly in the gamma form as sprayed. Since $\sim 2/3$ of this alumina does not transform on heating to 1600°F or higher, it is more crystallographically stable than FS-102.

TRW-100M Platinized Alumina

- Each particle is coated with platinum and should therefore form a stronger bond to Pt-10Rh than uncoated alumina particles.
- Platinized alumina should be more resistant to thermal shock than pure alumina because it has a higher thermal conductivity than pure alumina, and crack propagation is probably inhibited somewhat by the metal coating on each particle.

The two yttria-containing vents, H-22 and C-23, were fabricated as backups for the alumina-containing vents. Yttria (Y_2O_3) was chosen on account of its high melting point (4730°F), thermodynamic stability ($\Delta F_{298} = -143$ Kcal/g atomic oxygen versus -126 Kcal/g atomic oxygen for Al_2O_3), and crystallographic stability (no crystallographic phase changes due to PAS process or other heat treatment). Yttria can be plasma arc sprayed and ground to a smooth finish just as well as alumina.

2.1.2 Detailed Design of Final Phase I Test Vents

The basic configuration and alternate fabrication techniques of both the helical and coaxial vent assemblies were established after evaluating various processes to assure an adequate seal between the vent core and the surrounding noble metal structure (see Section 2.1.1.5). A detailed design of Phase I test vents was then initiated resulting in the fabrication, assembly, and testing of twenty final Phase I vents.

2.1.2.1 Final Drawings

Figures 20 through 22 present engineering drawings of the three separate vent configurations (two helical and one coaxial) which comprise the twenty vent matrix discussed previously in Section 2.1.1.5 and Table 2. Vents H-10 through H-12 are of the eight thread helical design shown in Figure 20. Vents H-16 through H-22 were fabricated according to the four

Figure 20. Eight Thread Helical Design

Figure 21. Four Thread Helical Design

Figure 22. Coaxial Design

thread helical design shown in Figure 21. Vents C-14 to C-23 are of the coaxial configuration shown in Figure 22.

2.1.2.2 Fabrication Specifications

Flow diagrams showing the step-by-step operations performed in the fabrication of all but three (H-10, H-11, and H-12) of the final Phase I test vents are shown in Figures 23 through 26. Photomacrographs of piece-parts, not all belonging to the same vent assembly, are included on one flow diagram for clarity. Test vents H-10, H-11, and H-12 were fabricated as follows (reference engineering drawing X405345, Figure 20):

Cores

- Machined a helix in 1-inch long, 0.110-inch diameter Pt-10Rh rods.
- Ultrasonically cleaned the machined rods in distilled water and degreasing agents.
- Annealed the rods in air for fifteen minutes at 2000°F.
- Plasma arc sprayed the rods after grit blasting in order to fill the helical grooves with FS-102 alumina.
- Ground the diameters of the rods with a SiC wheel to obtain clean metal lands and alumina-filled helical grooves.
- Ultrasonically cleaned the ground rods in distilled water and degreasing agents.
- Outgassed the cleaned rods in vacuum for thirty minutes at 300°F and one hour at 1000°F.

Tubing

- Drilled the Pt-10Rh tubes (0.150-inch diameter x 0.025-inch wall) to approximately 0.100-inch I.D. and honed the I.D. with a copper rod in an alumina-kerosene suspension.
- Ultrasonically cleaned the honed tubes in distilled water and degreasing agents.
- Soaked the tubes in boiling 20% HNO₃ for fifteen minutes and ultrasonically cleaned the machined tubes in distilled water and degreasing agents.

Figure 23. Piece-Part Fabrication Flow Sheet

TUBING PREPARATION

Figure 24. Piece-Part Fabrication Flow Sheet

END PLUG PREPARATION

Figure 25. Piece-Part Fabrication Flow Sheet

ASSEMBLY

Figure 26. Vent Assembly Flow Sheet

TRW(A)-11069-0067

- Outgassed the tubes in vacuum for ten minutes at 150°C.
- Annealed the tubes for fifteen minutes at 2000°F.

Assembly

- Assembled the rods into the tubes and isostatically pressed the assembly at room temperature for six minutes at 60,000 psi.
- Isostatically pressed the assembly at 1800°F for 4-1/2 hours at 10,000 psi.

Test vents H-16 through H-22 and C-14 through C-22 were fabricated as follows (reference engineering drawings X405347 and X405346, Figures 21 and 23).

Cores

- Machined a helix in 3/4-inch long, 0.110-inch diameter Pt-10Rh rods for the helical vents.
- Ultrasonically cleaned the machined helical rods and coaxial rods in distilled water and degreasing agents.
- Heated the cleaned rods in 800°F air for fifteen minutes.
- Grit blasted the rods with alumina and ultrasonically cleaned the machined helical rods and coaxial rods in distilled water and degreasing agents.
- Plasma arc sprayed the rods to fill the helical grooves and coated the coaxial rods with 0.010-inch thick alumina.
- Ground the diameter of the helical rods until the bare metal lands show (see photo in Figure 23). Ground the ceramic on the coaxial rod to 0.005-inch thickness.
- Cut off extensions, ground to length, and ultrasonically cleaned the machined helical rods and coaxial rods in distilled water and degreasing agents.
- Heated in 800°F vacuum for one hour.

Tubing

- Cut the Pt-10Rh tubes to length.
- Ultrasonically cleaned the tube lengths in distilled water and degreasing agents.
- Drilled and lapped the tubes to dimension.

- Chemically cleaned the lapped tubes in nitric acid.
- Heated the cleaned tubes in 800°F vacuum for one hour.

End Plugs

- Machined the plugs from Pt-10Rh rod.
- Ultrasonically cleaned the plugs in distilled water and degreasing agents.

Assembly

- Assembled cores into tubes.
- Welded end plugs into the tubes (Figure 27).
- Heated the welded assemblies in 2000°F air for one hour.
- Isostatically pressed the welded assemblies at room temperature.
- Isostatically pressed the assemblies at 1850°F.
- Drilled out the end plugs.
- Heated the assemblies in 2600°F air for four hours.

Figure 28 shows photos of seventeen vents after welding, a close-up of a typical electron beam welded end plug, and a vent after high temperature isostatic pressing. A close examination of the pressed vent (H-23) revealed slight indentions in the tube close to the end plugs where the tube has been pressed into intimate contact with the core. The indentions are at the end plug-to-core interfaces.

2.1.2.3 Material Specifications

Platinum-10 Weight % Rhodium Alloy

All Pt-10Rh used in the fabrication of test vents was ordered on TRW purchase order No. BA280 R to the following specification:

- 1) The rhodium content of the alloy shall be 9.75 to 10.25 weight percent.
- 2) The total impurity content of the alloy shall not exceed 2000 ppm (99.8% pure). No single impurity with the exceptions of Pd, Ir, Au, and Ag shall exceed 150 ppm.
- 3) The fabricated shapes shall be in the wrought condition.

Figure 27. Helical Vent with Plugs Welded in Ends of Vent Tube

Seventeen Welded Vents

End Plug EB Welded to Vent

Helical Vent after High Temperature Isostatic Pressing

Figure 28. Assembled Test Vents

- 4) The surface finish of the fabricated shapes shall be 32 micro-inches RMS or better (inside and outside diameters of tubing).
- 5) Straightness shall be 0.010-inch/foot on a best effort basis.
- 6) The fabricated shapes shall be free of pits, seams, laps, dents, or other defects not consistent with good commercial practice.

Figure 29 shows flow diagrams which reference the chemical analyses (Figures 30 through 39) of the platinum and rhodium at each of the principal fabrication stages, sponge to alloy bar to final form (rod or tubing^{*}).

FS-102 Alumina

FS-102 alumina is a product of San Fernando Laboratories, Fansteel Metallurgical Corporation (Pacoima, California). Technical specifications are as follows:

Chemical Formula - Al_2O_3
 Purity - 99.9%
 Crystal Form - alpha (trigonal)
 Particle Size - 1 to 20 microns

TRW-100 Alumina

TRW-100 alumina is a product of TRW Systems (Redondo Beach, California). Technical specifications are as follows:

Chemical Formula - Al_2O_3
 Purity {
 91.5% Al_2O_3
 0.5% Na_2O_3
 0.1% SiO_2
 0.02% Fe_2O_3 (processing at TRW reduced the Fe content to less than 0.005%)
 0.002% TiO_2
 7.0% combined H_2O

* Sponge refers to the purified elemental starting material generally characterized by a porous condition which is the result of the decomposition or reduction of a compound without fusion.

Figure 29. Noble Metal Raw Stock Fabrication Flow Diagram

INSTRUMENTAL ANALYSIS LABORATORY
13 ASTOR STREET
NEWARK, NEW JERSEY 07114
(201) 242-2700

CERTIFICATE OF SPECTROGRAPHIC ANALYSIS No. 5132

Sample Identification: Platinum Sponge R-820 - Lot No. 265
For Par # 7999²

Requested by: TRW Systems Incorporated
S.O. #01478 and #01479
P.O. #BA280 R

Report No: A 37369

Analysis: Quantitative

<u>p.p.m.</u>		<u>p.p.m.</u>	
Rh	3	Ca	<1
Pd	<5	Al	<1
Ir	<5	Ni	<1
Ru	ND	Cr	ND
Os	ND	Mn	ND
Au	19	Sb	ND
Ag	2	B	ND
Pb	ND	Co	ND
Sn	2	As	ND
Zn	ND	Bi	ND
Fe	2	Cd	ND
Cu	<1	Mo	ND
Si	3	Te	ND
Mg	<1	Na	2

*Pt 99.995+%

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INDUSTRIES
A DIVISION OF ENGELHARD MINERALS & CHEMICALS CORPORATION

By: *[Signature]*
A. J. Lincoln
Head, Instrumental Analysis Laboratory

AJL:as

Date: April 24, 1969

* BY DIFFERENCE LESS THAN > - GREATER THAN ND - NOT DETECTED
ALL VALUES REPORTED IN PARTS PER MILLION UNLESS OTHERWISE NOTED

Figure 30. Spectrographic Analysis of Platinum Sponge

INSTRUMENTAL ANALYSIS LABORATORY
113 ASTOR STREET
NEWARK, NEW JERSEY 07114
(201) 242-2700

CERTIFICATE OF SPECTROGRAPHIC ANALYSIS No. 5133

Sample Identification: Rhodium Sponge - Lot No. 221A
For Par #79998

Requested by: TRW Systems Incorporated
S.O. #01478 and #01479
P.C. #BA280 R

Report No: A 37651

Analysis: Quantitative

<u>p.p.m.</u>		<u>p.p.m.</u>	
Pt	<10	Mg	<20
Pd	2	Ca	<10
Ir	<10	Al	<10
Ru	ND	Ni	<1
Au	ND	Cr	ND
Ag	1	Mn	0.9
Pb	ND	Sb	ND
Sn	ND	B	ND
Zn	ND	Co	ND
Fe	10	As	ND
Cu	8	Bi	ND
Si	ND	Cd	ND

*Rh 99.99+%

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By: *[Signature]*
A. J. Lincoln
Head, Instrumental Analysis Laboratory

AJL:as

Date: April 24, 1969

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FORM EM-5531

Figure 31. Spectrographic Analysis of Rhodium Sponge

ASSAY LABORATORY

113 ASTOR STREET
NEWARK, N.J. 07114
(201) 242-2700

CERTIFICATE OF CHEMICAL ANALYSIS

Assay N 2 9 4 6 5
No.

TO: TRW Systems Inc.
P.O. # 1473 R
P.O. # 01473-01479

MATERIAL: Alloy # 7542 Bar # 79998

MARKED:

Rhodium 9.80 %

REMARKS:

ANALYSIS
10/2/69

DATE: May 22, 1969

H. Robinson
HEAD, ASSAY LABORATORY

INSTRUMENTAL ANALYSIS LABORATORY
113 ASTOR STREET
NEWARK, NEW JERSEY 07114
201 242 2700

CERTIFICATE OF SPECTROGRAPHIC ANALYSIS No. 5150

Sample Identification: Alloy No. 7542 (Pt-10%Rh) 0.100" dia. rod
Part #79993

Requested by: TRW Systems Incorporated
S.C. #01478
P.C. #BA280 R

Report No: A 38248

Analysis: Quantitative

p.p.m.		p.p.m.	
Pd	<10	Ca	8
Ir	<20	Al	<1
Ru	ND	Ni	2
Os	ND	Cr	<1
Au	22	Mn	<1
Ag	4	Sb	ND
Pb	ND	B	38
Sn	ND	Co	ND
Zn	ND	As	ND
Fe	13	Bi	ND
Cu	3	Cd	ND
Si	<5	Mo	ND
Mg	<1	Na	<10

*Pt+Rh 99.986+%

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INDUSTRIES
A DIVISION OF ENGELHARD MINERALS & CHEMICALS CORPORATION

By: *A. J. Lincoln*
Head, Instrumental Analysis Laboratory

AJL:as

Date: May 1, 1969

* - BY DIFFERENCE < - LESS THAN > - GREATER THAN ND - NOT DETECTED
ALL VALUES REPORTED IN PARTS PER MILLION UNLESS OTHERWISE NOTED

FORM EM-5531

Figure 33. Spectrographic Analysis of 0.110-Inch Diameter Rod

INSTRUMENTAL ANALYSIS LABORATORY

 113 ASTOR STREET
 NEWARK, NEW JERSEY 07114
 201 242 2700

CERTIFICATE OF SPECTROGRAPHIC ANALYSIS No. 5151

Sample Identification: Alloy No. 7542 (Fe-1C%Rh) 0.110" dia. rod

Sur # 75990r

 Requested by: IRW Systems Incorporated
 S.O. #01479
 P.O. #BA280 R

Report No: A 38249

Analysis: Quantitative

<u>p.p.m.</u>		<u>p.p.m.</u>	
Pd	<10	Ca	6
Ir	<20	Al	<1
Ru	ND	Ni	4
Os	ND	Cr	<1
Au	20	Mn	<1
Ag	4	Sb	ND
Pb	ND	B	38
Sn	ND	Co	ND
Zn	ND	As	ND
Fe	14	Bi	ND
Cu	3	Cd	ND
Si	<5	Mo	ND
Mg	<1	Na	<10

*Pt+Rh 99.986+%

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 A DIVISION OF ENGELHARD MINERALS & CHEMICALS CORPORATION

By:

A. J. Lincoln

Head, Instrumental Analysis Laboratory

AJL:as

Date: May 1, 1969

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 ALL VALUES REPORTED IN PARTS PER MILLION UNLESS OTHERWISE NOTED

INSTRUMENTAL ANALYSIS LABORATORY
113 ASTOR STREET
NEWARK, NEW JERSEY 07114
201 242 2700

CERTIFICATE OF SPECTROGRAPHIC ANALYSIS No. 5155

Sample Identification: Platinum Sponge R-820 - Lot No. 292
for E-2 + 0002

Requested by: TRW Systems Incorporated
S.O. #01477
P.O. #BA280 R

Report No: A 37981

Analysis: Quantitative

p.p.m.

Rh 74
Pd 5
Ir <5
Ru ND
Os ND
Au 15
Ag 2
Pb ND
Sn ND
Zn ND
Fe 2
Cu <1
Si 7
Mg 2

p.p.m.

Ca 2
Al <1
Ni <1
Cr ND
Mn ND
Sb ND
B ND
Co ND
As ND
Bi ND
Cd ND
Mo ND
Te ND
Na 13

*Pt 99.987+%

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By: A. J. Lincoln
Head, Instrumental Analysis Laboratory

AJL:as

Date: May 2, 1969

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ALL VALUES REPORTED IN PARTS PER MILLION UNLESS OTHERWISE NOTED

INSTRUMENTAL ANALYSIS LABORATORY
113 ASTOR STREET
NEWARK, NEW JERSEY 07114
201 242 2700

CERTIFICATE OF SPECTROGRAPHIC ANALYSIS No. 5154

Sample Identification: Rhodium Sponge No. 7464
 Requested by: TRW Systems Incorporated
 S.C. #01477
 P.O. #BA280 R
 Report No: A 38008
 Analysis: Quantitative

<u>p.p.m.</u>		<u>p.p.m.</u>	
Pt	ND	Ca	24
Pd	2	Al	<10
Ir	400	Ni	<1
Au	ND	Cr	ND
Ag	43	Mn	2
Pb	ND	Sb	ND
Sn	ND	B	ND
Zn	ND	Co	ND
Fe	9	As	ND
Cu	2	Bi	ND
Si	ND	Cd	ND
Mg	<20		

*Rh 99.949+%

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A DIVISION OF ENGELHARD MINERALS & CHEMICALS CORPORATION

By: A. J. Lincoln
Head, Instrumental Analysis Laboratory

AJL:as

Date: May 1, 1969

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ALL VALUES REPORTED IN PARTS PER MILLION UNLESS OTHERWISE NOTED

ASSAY LABORATORY

113 ASTOR STREET
NEWARK N.J. 07114
(201) 242-2700

CERTIFICATE OF CHEMICAL ANALYSIS

TO: TRW Systems Inc.

1 . . . 1
: . . 1

Assay

N 3 0 2 2 4

No.

MATERIAL: Alloy # 7542 Bar # 80062

MARKED:

Rhodium 10.02 %

REMARKS:

DATE: May 22, 1969

A handwritten signature in cursive script, appearing to read "H. Robinson".

H. Robinson
HEAD, ASSAY LABORATORY

OKM EM 5923 2

Figure 37. Chemical Analysis of Pt-10Rh Alloy for Rhodium

INSTRUMENTAL ANALYSIS LABORATORY

113 ASTOR STREET
NEWARK, NEW JERSEY 07114
(201) 242-2700

CERTIFICATE OF SPECTROGRAPHIC ANALYSIS No. 5174

Sample Identification: Alloy No. 7542 (Platinum/10% Rhodium)
.150" O.D. x .025" Wall
Car #3002

Requested by: TRW Systems Incorporated
S.C. #01477
P.C. #BA280 R

Report No: A 38440

Analysis: Quantitative

p.p.m.		p.p.m.	
Pd	45	Ca	2
Ir	39	Al	<1
Ru	<10	Ni	1
Os	ND	Cr	2
Au	17	Mn	1
Ag	8	Sb	<20
Pb	ND	B	20
Sn	<1	Co	2
Zn	<10	As	ND
Fe	38	Bi	ND
Cu	1	Cd	ND
Si	<5	Mo	ND
Mg	<1	Na	<10

*Pt+Rh 99.977+%

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By:

A. J. Lincoln
Head, Instrumental Analysis Laboratory

AJI:as

Date: May 13, 1969

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INSTRUMENTAL ANALYSIS LABORATORY
113 A. TOR STREET
NEWARK, NEW JERSEY 07114
(201) 242-2700

CERTIFICATE OF SPECTROGRAPHIC ANALYSIS No. 5173

Sample Identification: Alloy No. 7542 (Platinum/10% Rhodium)
.150" O.D. X .020" Wall
Lot # 80062

Requested by: TRW Systems Incorporated
S.O. #01477
P.O. #BA280 R

Report No: A 38439

Analysis: Quantitative

p.p.m.		p.p.m.	
Pd	37	Ca	2
Ir	32	Al	<1
Ru	<10	Ni	1
Os	ND	Cr	2
Au	15	Mn	1
Ag	8	Sb	ND
Pb	ND	B	11
Sn	ND	Co	1
Zn	ND	As	ND
Fe	28	Bi	ND
Cu	1	Cd	ND
Si	<5	Mo	ND
Mg	<1	Na	ND

*Pt+Rh 99.984+%

ENGELHARD
INDUSTRIES
A DIVISION OF ENGELHARD MINERALS & CHEMICALS CORPORATION

By:
A. J. Lincoln
Head, Instrumental Analysis Laboratory

AJL:as

Date: May 13, 1969

* - BY DIFFERENCE < - LESS THAN > - GREATER THAN ND - NOT DETECTED
ALL VALUES REPORTED IN PARTS PER MILLION UNLESS OTHERWISE NOTED

FORM EM-5531

Figure 39. Spectrographic Analysis of 0.150-Inch O.D. x 0.020-Inch Wall Tubing

Crystal Form - Orthorhombic
Particle Size - 5 to 74 microns

TRW-100M Alumina

TRW-100M alumina is a product of TRW Systems (Redondo Beach, California). Technical specifications are as follows:

Chemical Formula - Al_2O_3 -5 weight % Pt
Purity - $95 \pm 2\%$ TRW-100 alumina, remainder Pt
Crystal Form - Orthorhombic alumina, face centered cubic platinum
Particle Size - 5 to 149 microns

Yttria

The yttrium oxide used in this program was supplied by the Michigan Chemical Corporation (Saint Louis, Michigan). Technical specifications are as follows:

Chemical Formula - Y_2O_3
Purity - 99.99% (based on yttrium content)
Crystal Form - body-centered cubic
Particle Size - 44 to 140 microns

2.1.2.4 Thermal Analyses of a Coaxial Vent

A brief analysis was performed to determine the magnitude of temperature gradients that might be experienced across the components of a typical vent. The analysis was limited to steady-state vacuum operation as the transient behavior would depend largely upon the characteristics of the protective heat shield which were not available in this phase of the program.

The calculated temperature distribution is shown in Figure 40. To obtain the temperatures, a number of assumptions were made:

- The heat shield was designed to maintain the clad temperature at 2000°F.
- The heat flux was uniformly distributed at the clad surface.
- The gradient on the impact member was negligible.

Figure 40. Temperature Distribution for a Coaxial Vent

- The gaps between the platinum and the strength member are filled with helium.

The temperature was calculated by applying the circular fin conduction equation to the clad and liner.

The results indicate that during operation temperature gradients throughout the vent are quite small and will not present a problem.

2.1.3 Development of a Helium Permeation Model

In order to adequately correlate and evaluate the experimental data acquired during this program, a theoretical model for helium permeation through a porous medium was developed. Using only the basic physical properties of the ceramic (porosity and surface area), the model is intended to predict the permeation performance of typical vents over a wide range of pressures, temperatures, and ceramic properties.

In this section of the report, the analytical model is discussed. The first subsection will discuss the vent permeability characteristics. Next, the effect of various heat treatments on the pertinent physical properties of the ceramic are presented. The model is then compared with the preliminary experimental data obtained during the first four months of this program, and finally, the selected model and the computer program using this model are discussed.

2.1.3.1 Analysis of Vent Permeability Characteristics

In order to formulate a model for the helium permeation through the candidate vent assemblies, a survey was made of the available literature on the permeation of gases through porous media. As a result of this survey, an analytical model of the vent was formulated and is presented below. Correlation of the analytical model with some measured vent characteristics is presented in Section 2.1.3.3.

Analysis of the permeation of a gas through a porous medium at relatively low pressures is complicated by the fact that the pore diameters are often less than or comparable with the mean free path of the gas molecules. Under such conditions, the permeation rate in the pores will not follow that predicted by Poiseuille's Law which applies to viscous flow but will be somewhat higher. This is attributed to slippage along the

boundary layer, and hence, is called "slip flow" (References 1, 2, 3, and 4). Under this type of flow, viscosity has a diminished effect. If the pressure is further reduced until the mean free path is much greater than the pore size, viscosity plays no part in the flow since the molecules collide only with the walls and the transport mechanism is one of free molecular effusion, or Knudsen flow (References 3, 4, and 5).

For the permeation rates corresponding to venting during steady-state operation of the capsule, the flow is always in either the slip or free molecular regions. The equations of permeation conditions will first be derived for circular capillaries and then applied to a porous medium typical of the proposed vents.

Flow In Capillaries

For the viscous flow of gases in a circular passage, Poiseuille's Law takes the form of:

$$\frac{u_1 P_1}{\Delta P} = \frac{D^2 P_M}{32 \mu L} \quad \text{Equation (1)}$$

where: u_1 - inlet velocity
 P_1 = inlet pressure
 ΔP = pressure drop
 D = effective pore diameter
 P_M = mean pressure in tube
 μ = absolute viscosity
 L = flow tube length

To account for the increased flow due to slip and/or molecular effusion, an extra term must be added (Reference 1 and 4):

$$\frac{u_1 P_1}{P} = \frac{D^2 P_M}{32 \mu L} + \frac{\delta}{3} \frac{D}{L} V_m \quad \text{Equation (2)}$$

where: V_m = mean molecular velocity = $\sqrt{\frac{8 RT}{\pi M}}$

$$\left\{ \begin{array}{l} \delta = \frac{2-f}{f} \quad D \ll \lambda \text{ (Knudsen flow)} \\ \delta = \frac{3\pi}{16} \left(\frac{2-f}{f} \right) \quad D > \lambda \text{ (slip flow)} \end{array} \right.$$

f = fraction of molecules undergoing diffusion reflection at the wall

R = gas constant

T = absolute temperature

M = molecular weight

ρ = gas density

$$\lambda = \frac{\mu}{\rho} \sqrt{\frac{\pi RT}{2M}} = \text{mean free path in gas}$$

The parameter, δ , varies depending upon the flow regime and the surface conditions and is difficult to evaluate. Fortunately, it has been shown to have a fairly constant value in porous media varying from 0.9 to 1.0 from the slip to the Knudsen flow regime, respectively (Reference 6).

In the case of noncircular capillaries, the effective diameter, D , is replaced by four times the hydraulic radius and a shape factor, K (Reference 4). Equation 2, thus becomes:

$$\frac{u_1 P_1}{\Delta P} = \frac{m^2 P_M}{\mu L K_v} + \frac{4\delta}{3K_k} \frac{m}{L} V_m \quad \text{Equation (3)}$$

where: m = hydraulic radius = $\frac{\text{cross sectional area}}{\text{wetted perimeter}}$

K = shape factor

subscript v refers to viscous flow

subscript K refers to slip and Knudsen flow

Values of K for a variety of different shapes for both viscous and slip flows have been evaluated by Clausing (Reference 7) and have been shown to be a function of both the shape and the length-to-diameter ratio of the tube. These values are presented in Tables 3 and 4.

TABLE 3. VALUES OF K_V FOR VISCOUS FLOW IN VARIOUS SECTIONAL SHAPES

Shape	K_V
1. Circle	2.00
2. Ellipse, semi-axes a, b :	
(i) $a = 2b$	2.13
(ii) $a = 10b$	2.45
3. Square	1.78
4. Rectangles, sides a, b :	
(i) $a = 2b$	1.94
(ii) $a = 10b$	2.65
5. Parallel slit	3.00
6. Equilateral triangle	1.67
7. Annuli in pipes with concentric cores	2.0-3.0
8. Annuli in pipes with eccentric cores, radii a_1 and a_2 :	
(i) $e < 0.7$	1.7-3.0
(ii) $e > 0.7$	1.2-2.0
$e = \text{eccentricity} = b/(a_1 - a_2).$ $b = \text{distance between centers.}$	

TABLE 4. VALUES OF K_k CALCULATED FROM CLAUSING'S TABLES

Shape	K_k
1. Circle, radius r , $m = r/2$, $L/m = \infty$ $L/m = 20$ $L/m = 16$ $L/m = 12$ $L/m = 8$ $L/m = 4$	1.00 1.35 1.44 1.58 1.86 2.60
2. Square, $L/m = \infty$	0.90
3. Rectangles, $b \times h$, $L \gg b$, $L \gg h$, $b = 2h$ $b = 5h$ $b = 10h$	0.87 0.78 0.68
4. Parallel slits, $b \times h$, $b \gg L$, $m = h/2$, $L \gg h$ $L/m = 20$ $L/m = 12$ $L/m = 8$ $L/m = 4$	$2/8 \ln (L/h)$ 1.09 1.36 1.67 2.46

The flow in a narrow annulus, which is of interest in the analysis of helium vent designs, can be obtained from equation 3. This flow is equivalent to the flow in a narrow gap between parallel plates where the gap width is equal to the circumference of the annulus. This has been treated by Carman (Reference 8). The values of the constants K_v and K_k have been evaluated by Clausing for this case (Reference 7) who found that:

$$K_v = 3.0$$

$$K_k = \frac{8}{3 \ln \frac{L}{h}} \text{ when } b \gg L \gg h$$

where: b = width of gap = πD
 h = height of gap = 2 x hydraulic radius
 D = mean diameter of the annulus

Even though for the annular gap b is not greater than L , the equation for K_k is probably valid as the end conditions can be ignored because of the continuity of the annulus. Substituting in equation 3 and applying continuity one obtains:

$$\frac{\dot{v}_1 P_1}{\Delta P} L = \pi D \left[\frac{h^3 P_M}{12 \mu} + v_m \frac{S_k h^2}{4} \ln \frac{L}{h} \right] \quad \text{Equation (4)}$$

where: $\dot{v}_1$ = volume flow rate at the inlet of annulus

Porous Media

The previous derivation was for single capillaries. In a porous medium, the flow path is more torturous and the true flow path length may be appreciably longer than the specimen length. This introduces a new factor, the effective path length, L_{eff} , and a tortuosity function, q , (References 8 and 9) defined as

$$\frac{L_{eff}}{L} = q$$

If porosity, ϵ , is defined as void volume divided by the total volume, then

$$\epsilon = \frac{\text{void volume}}{\text{total volume}} = \frac{L_{eff} A_{eff}}{LA} \quad \therefore \left(\frac{A}{L} \right)_{eff} = \frac{\epsilon}{q^2} \frac{A}{L}$$

where: A = cross section of porous medium
 A_{eff} = effective cross section area of pores
 ϵ = porosity fraction

Substituting in equation 3 and applying continuity, one obtains

$$\frac{\dot{v}_1 P_1 L}{A \Delta P} = \frac{\epsilon m^2 P_M}{K_V \mu q^2} + \frac{4 \epsilon m \delta}{3 q^2 K_k} V_m \quad \text{Equation (5)}$$

A second feature of a porous medium is the difficulty in defining its hydraulic radius. To correct this, Kozeny (Reference 10) defined the hydraulic radius, m , as follows:

$$m = \frac{\text{volume filled with fluid}}{\text{wetted surface}} = \frac{\text{cross-sectional area}}{\text{wetted perimeter}} \\ = \frac{\epsilon}{S'} = \frac{\epsilon}{S(1 - \epsilon)} \quad \text{Equation (6)}$$

where: S' = internal surface area/unit volume of pores
 S = internal surface/unit volume of solid

Substituting these relationships in equation 5, one obtains:

$$\frac{\dot{v}_1 P_1}{A \Delta P} = \frac{\epsilon^3 P_M}{S^2 (1 - \epsilon)^2 \mu L q^2 K_V} + \frac{4}{3} \frac{\epsilon^2 \delta V_m}{S(1 - \epsilon) K_k L q^2} \quad \text{Equation (5a)}$$

To report the flow rate under standard conditions of temperature and pressure, the perfect gas law can be applied:

$$\frac{\dot{v}_1}{\dot{v}_0} = \frac{T_1}{T_0} \frac{P_0}{P_1}$$

where: subscript 0 = applies to standard conditions.

Substituting in equation 5a, one obtains:

$$\dot{v}_0 = \frac{A \Delta P T_0}{L P_0 T_1 q^2} \left[\frac{\epsilon^3 P_M}{S^2 (1 - \epsilon)^2 \mu K_V} + \frac{4 \epsilon^2 \delta V_m}{3 S (1 - \epsilon) K_k} \right] \quad \text{Equation (5b)}$$

Or, since the hydraulic diameter $D_m = 4 \times$ hydraulic radius m

$$\dot{v}_o = \frac{\epsilon A \Delta P T_o}{L P_o T_1 q^2} \left[\frac{D_m^2 P_m}{16 K_v \mu} + \frac{\delta V_m D_m}{3 K_k} \right] \quad \text{Equation (5c)}$$

$$\text{In addition } V_m = \sqrt{\frac{8 R T_1}{\pi M}}$$

then

$$\dot{v}_o = \frac{\epsilon A \Delta P T_o}{L P_o T_1 q^2} \left[\frac{D_m^2 P_m}{16 K_v \mu} + \left(\frac{\delta D_m}{3 K_k} \right) \sqrt{\frac{8 R T_1}{\pi M}} \right] \quad \text{Equation (5d)}$$

From equation 5d, it may be seen that the viscous flow varies inversely with the temperature whereas the slip or Knudsen flow varies inversely with the square root of temperature.

The preceding equations assume that the mean free path of the gas molecules is either equal to or greater than the mean pore size of the ceramic. A more general case derived from the work of Hiby and Pahl (Reference 9) applies a correction factor to the slip flow term of the preceding equations to account for collisions between gas molecules when the mean path is less than the mean pore size. This is shown in equation 7

$$\dot{v}_o = \frac{A \Delta P T_o \epsilon}{L P_o T_1 q^2} \left[\frac{D_m^2 P_m}{16 K_v \mu} + e^{-\frac{k_2 D_m}{\lambda} \left(\frac{D_m V_m \delta}{3 K_k} \right)} \right] \quad \text{Equation (7)}$$

where:

$$K_2 = \text{constant which is a function of } D_m$$

For the case of capillaries where the path length is appreciably longer than the mean pore size, the constant K_2 is approximately equal to 0.2. For helium at room temperature, the mean free path is given by:

$$\lambda (\text{cm}) = \frac{2\mu}{P_m V_m} = 1.86 \times 10^{-5} P_m (\text{atmosphere})$$

Substituting in equation 7, one obtains

$$\dot{v}_o = \frac{A \Delta P T_o \epsilon}{L P_o T_1 q^2} \left[\frac{D_m^2 P_m}{16 K_v \mu} + \frac{D_m V_m \delta}{3 K_k} e^{-\left(1.07 \times 10^4 \frac{D_m}{P_m}\right)} \right] \text{Equation (7a)}$$

In equation 7a, the second term represents the flow resulting from free molecular diffusion through the porous medium while the first term represents the contribution from viscous type flow. This equation then represents the flow characteristics of a porous medium when flow is not turbulent.

At high enough pressure drops, however, the flow will be turbulent. Carman (Reference 9) notes that the onset of turbulent flow is much more gradual in a porous medium than in a pipe, with some slight effect at a Reynolds number of five and fully developed turbulent flow occurring at $Re \geq 500$. For the typical ceramic vent, this corresponds to the flow rate range from 50 to 5,000 cc/sec. In the fully turbulent range, the flow rate is described by equation 8 below.

$$\dot{v}_o = 1.86 \left(\frac{A^2 \epsilon^2}{L q^2} \frac{\Delta P_o}{P_o} \frac{T_o}{T_1} \frac{P_m D_m}{\rho_o} \right)^{1/2} \text{Equation (8)}$$

where:

$$\rho_o = \text{gas density at } T_o \text{ and } P_o$$

For the purpose of the current model, it was assumed that the transition from viscous to turbulent flow occurred sharply at the point where the two flow rates calculated by equations 7 and 8 were equal. This is equivalent to assuming that transition occurs at a Reynolds number of approximately 50. Actually, the transition will be more gradual.

These equations (7 and 8) were programmed for the Tymeshare Computer and were used to predict the flow characteristics of the porous ceramic vents.

The preceding equations require several empirical constants in order to be used to predict the flow rate through a porous Al_2O_3 plug. These factors can be obtained either experimentally or by the use of the recommended values presented in Reference 9. The most difficult parameter to assess is the tortuosity factor, q , since appreciably different values are obtainable depending upon the degree of consolidation of the ceramic. Values are presented below (Table 5) for both the unconsolidated Al_2O_3 powder and for

consolidated and sintered Al_2O_3 . The PAS Al_2O_3 would be expected to lie close to the value for consolidated Al_2O_3 .

TABLE 5. CONSTANTS USED IN FLOW MODEL

	Viscous	Slip	Source
K_v (shape factor)	2.5	-	Reference 7, 9
δ/K_k (shape and collision factor)	-	0.82	Reference 9
q (tortuosity)			
a. Unconsolidated	1.4	1.4	Reference 6
b. Consolidated	3.0	3.0	Reference 8

2.1.3.2 Physical Properties of the Ceramic

Plasma sprayed aluminum oxide undergoes several changes as it is heated to elevated temperatures. The "as-sprayed" coating is largely in gamma crystal configuration, rather than the more common alpha form. Since the gamma form has a lower density (3.60 g/cc) than the alpha form (3.97 g/cc), the alpha sample typically shows an increase in porosity.

Heating of the aluminum oxide above 2000°F also results in appreciable decrease in the internal surface area, corresponding to an increase in the mean pore size. While the majority of the surface area decrease occurs during the phase transformation, in the case of some of the candidate ceramics a further decrease occurs after additional heating at higher temperatures (e.g., 2800°F). After prolonged heating at a given temperature (above 2000°F), the surface area appears to level off and does not change unless the ceramic is subsequently heated to a still higher temperature.

The described behavior has been noted both in the literature (Reference 11) and in the present study. In Reference 11, the variation of the mean pore size of a plasma sprayed alumina as a function of temperature was determined and is shown in Figure 42. These variations in the ceramic properties from the "as-sprayed" to the "heated" condition, make it important that the ceramic vents be heated and stabilized above their expected operating

Figure 41. Pore Size Midpoints of Reheated, Plasma-Sprayed 12 μ Alumina temperature prior to use. In addition, in order to predict their performance, the properties of the ceramic after heating are required. These were determined during this study and are reported on in Section 2.4.2.4. A brief summary of the average properties is presented in Table 6.

The tortuosity of the ceramic is more difficult to ascertain and is primarily a function of the degree of consolidation. As no independent means of evaluating the tortuosity was employed in this study (Section 2.4.2.4), the tortuosity was determined from the actual permeability data.

2.1.3.3 Comparison Between Permeability Theory and Experimental Flow Rates

At the low pressures and flow rates characteristic of the normal vent operation*, only the Knudsen term in equation 5c need be considered; the permeability is described by

$$\dot{V}_0 = \frac{A \Delta P T_0}{L P_0 T_1} \left[\frac{\epsilon D_m \delta V_m}{3 K_k q^2} \right] \quad \text{Equation (9)}$$

* Vents were designed to permeate all of the helium generated from 300 watts of PuO₂ with a capsule backpressure ranging from 10⁻¹ to 10⁻³ atmospheres. 300 watts of fuel generates 1.25 x 10⁻⁵ std cc/sec of helium.

TABLE 6. ASSUMED PROPERTIES OF ALUMINA PAS CERAMICS

Material	Treatment	Average Porosity	Mean Pore Size μ
FS-102 Alumina	As-Fabricated	0.15	0.4
	18 Days 2200°F	0.18	0.9
	3 Days 2600°F	0.18	1.1
TRW-100 Alumina	As-Fabricated	0.16	0.1
	18 Days 2200°F	0.19	0.8
	3 Days 2600°F	0.20	1.50
TRW-100M (platinized) Alumina	As-Fabricated	0.31	1.0
	18 Days 2200°F	0.34	1.5
	3 Days 2600°F	0.34	1.8

Figure 42 shows the variation of flow rate with differential pressure for a variety of different preliminary vents tested in the program. In all cases, the flow is proportional to the applied pressure differential and the vent performance can thus be characterized by defining the permeability function :

$$\phi = \frac{\dot{V}_o L P_o}{A \Delta P} \left(\frac{T_1}{T_o} \right)^{\frac{1}{2}} = \frac{\epsilon D_m \delta V_{m_o}}{3 K_k q^2} \quad \text{Equation (10)}$$

where:

V_{m_o} = mean molecular velocity at T_o

The permeability function of a vent is affected only by the ceramic physical properties (ϵ , D_m and q) and thus, represents a good quantitative measure of the ceramic performance. The specific permeability is defined below.

$$\psi = \frac{\phi}{\epsilon} = \frac{\dot{V}_o L P_o}{A \Delta P} \left(\frac{T_1}{T_o} \right)^{\frac{1}{2}} = \frac{D_m \delta V_{m_o}}{3 K_k q^2} \quad \text{Equation (11)}$$

Figure 42. Helium Permeation Rate as a Function of Pressure Differential for a Variety of Vents

The specific permeability may be determined experimentally by measuring the flow at room temperature with a known pressure differential, and measuring the dimensions of the vent and the value of the porosity.

The first attempt to correlate the experimental permeability data with the theory utilized the data from the cold pressed noble metal vents prepared early in this program and described previously. For this series of vents, the permeation characteristics varied to some extent depending upon the previous heat treatment or pressing operation. It is believed that much of this variation was primarily due to micron or submicron size partial gaps at the wall interfaces and the resulting reduced flow resistance through these gaps. While isostatic pressing at room temperature greatly reduced these gaps, subsequent heat treatment appeared to reopen them with a resulting increase in the permeation rate. As shown in Figures 43 and 44, a gap on the order of 1 micron will result in a flow increase of 2.5×10^{-5} std cm³/sec at 10^{-2} atmospheres. This is of the same magnitude as the flow which passes through the ceramic. Therefore, for the purpose of comparing calculated and experimental results, the flow rates immediately after the isostatic pressing operation should most closely resemble the analytical condition (flow through the ceramic only) and these were utilized to test the theory.

In Figure 45, the predicted permeation rates for two values of tortuosity are compared with measured rates for two coaxial vents. The predicted permeation rate is based upon the porosity and the calculated mean pore size for the FS-102 alumina (heated to 2200°F) which is derived from physical measurement of porosity and surface area (Section 2.4.2.4). As may be seen, the best value of q for the coaxial vents is close to 3, which corresponds to the case of fully consolidated ceramic. The vent permeability increased upon further heat treatment, indicating the possibility of increased permeability around the metal-to-ceramic seal.

Analysis of Epoxy-Imbedded Vents - In order to test whether the theory was correctly predicting the permeability of the ceramic, a series of coaxial ceramic vents were fabricated with the ceramic imbedded in an epoxy cement.

A total of five epoxy vents were prepared by sealing free standing ceramic tubes in Torr Seal epoxy cement. Since the Torr Seal bonded directly to the alumina, flow at the epoxy/alumina interface was effectively eliminated

Figure 43. Flow Characteristics of an Annular Gap

Figure 44. Flow Characteristics of a Long Narrow Gap

Figure 45. Comparison of Predicted and Measured Helium Permeation Rates for Two Coaxial Vents

and all of the flow was through the interior of the ceramic. To test the effectiveness of the epoxy/ceramic seal, one vent was fabricated using a non-porous densified alumina. No helium flow could be detected through this vent indicating the effectiveness of the seal. The characteristics of the various vents are summarized in Table 7. This table shows the calculated and experimental specific permeabilities for the epoxy vents and the two Pt-20Rh coaxial vents previously analyzed. The specific permeabilities are compared with the theoretical predictions for a tortuosity of three. The comparison appears good with most of the variability easily explained by uncertainties in the value of the actual mean pore size that should be used.

TABLE 7. PERMEABILITIES OF EPOXY AND COLD PRESSED VENTS

Sample No	Type	Al ₂ O ₃ State	ϕ = Permeability cm ² /sec Measured	Ψ = Specific Permeability cm ² /sec x 10 ⁻³	
				Measured	Predicted*
6-1	Epoxy/FS-102	α	0.34×10^{-3}	2.58	3.4
6-2	Epoxy/FS-102	As-Sprayed	0.06×10^{-3}	0.97	1.47
6-3	Epoxy/TRW-100	α	0.81×10^{-3}	3.47	3.5
6-4	Epoxy/TRW-100	As-Sprayed	0.81×10^{-3}	3.77	3.5
C-3	Pt-20Rh/FS-102	α	0.59×10^{-3}	2.42	3.4
C-4	Pt-20Rh/FS-102	α	0.67×10^{-3}	3.20	3.4

*See Table 6 for assumed ceramic properties

Correlation at High Pressures - A series of tests were performed measuring the room temperature helium permeability of a typical refractory coaxial vent over the pressure drop range from 10^{-4} to 10^{+2} atmospheres. To cover this wide range of flow rates, three different instruments were used. At low pressure drops, a helium leak detector was used to measure the flow with a downstream pressure of 10^{-5} torr. With ΔP 's of 0.6 to four atmospheres and a one atmosphere downstream pressure a bubble flow meter was employed, and at ΔP 's of 12 to 100 atmospheres, a wet test meter was employed.

The experimental results are compared with the theoretical predictions in Figure 46. The predicted performance was obtained by selecting a porosity

Figure 46. Correlation between Permeability Model and Experimental Permeability

and mean pore size typical of the ceramic ($\epsilon = 0.25$ and $D_m = 2.0\mu$) and solving for the value of tortuosity required to give the measured flow rate at 10^{-4} atmospheres differential pressure. Equation 7a and 8 were then used to calculate the flow rates as a function of pressure drop. As may be seen, the correlation is excellent particularly if the transition between turbulent and laminar flow is assumed to occur gradually.

2.1.3.4 Final Permeability Model

The equations derived in Section 2.1.3.1 appear to adequately model the permeation of helium through a porous coaxial ceramic vent and require only the physical properties of the ceramic and the dimensions of the vent. This model of vent permeability therefore was programmed for use on the Tymshare digital computer. The program was designed to allow parametric analysis of the following variables:

- Vent dimensions - coaxial and helical
- Ceramic porosity
- Tortuosity
- Discharge pressure
- Differential pressure - 10^{-4} to 10^{+3} atmosphere
- Temperature - 70°F, 1000°F, and 2000°F

In addition, simple changes can be incorporated to evaluate the flow of gases other than helium. Figure 47 shows the calculated variation of helium flow rate with porosity and surface area for a tortuosity of three.

2.2 ELEVATED TEMPERATURE HELIUM PERMEATION EXPERIMENTS

In this section, the results of elevated temperature helium permeation experiments with the final Phase I test vents are presented. The alternate fabrication techniques and vent designs which were evaluated in these elevated temperature tests were described previously in Sections 2.1.1.5 and 2.1.2. The helium permeation model developed in Section 2.1.3 is compared with the experimental data.

2.2.1 Nondestructive Evaluation of Final Test Vents

All vent assemblies selected for elevated temperature helium permeation measurements during this program were initially tested at room temperature.

Figure 47. Variation in Helium Flow Rate with Ceramic Area and Porosity

Room temperature helium permeation measurements of the final test vents (20 plus 2 spares) were conducted in the test setup shown in Figure 7, Section 2.1.1.3 above. One end of the vent assembly was connected to a helium leak detector which maintained a vacuum between 1×10^{-4} to 1×10^{-5} torr. The other end of this assembly was connected to a regulated helium supply. Helium permeation rates were measured with pressure differentials across the vent of 10^{-3} and 10^{-2} atmospheres.

Table 8 shows the results of room temperature helium permeation rate measurements on the final Phase I test vents. Six of these vents (H-10, H-16, H-19, C-15, C-18, and C-21) subsequently had 10-inch long Pt-10Rh extension tubes attached to each end to facilitate elevated temperature helium permeation testing.

In general, the specimen-to-specimen reproducibility of data on vents of like designs and core material was very good. For example, vents H-10, H-11, and H-12, had room temperature permeation rates of 3.2×10^{-6} , 4.1×10^{-6} , and 3.6×10^{-6} std cm^3/sec , respectively at 10^{-2} atmospheres ΔP .

2.2.2 Experimental Equipment and Standard Short Term Testing Matrix

The room temperature helium permeation tests were conducted as described in Section 2.1.1.3. This test setup was modified to accommodate both room temperature and elevated temperature helium permeation rate measurements as described below.

2.2.2.1 Elevated Temperature Helium Permeation Test Apparatus

The high temperature furnace designed specifically for helium permeation testing is shown in Figure 48. This setup permitted room temperature and elevated temperature helium flow measurements to be performed sequentially as described in Section 2.2.2.2. After high temperature gas bonding, 10-inch long Pt-10Rh extension tubes were welded to both ends of the vent assembly. The test units were then placed between two silicon carbide rod heating elements, and alumina tubes inserted over the vent assemblies to protect against possible contamination by the elements as shown in Figure 49. A helium pressure manifold was connected to both sides of the high temperature furnace allowing six vent assemblies to be tested simultaneously or independently.

TABLE 8. ROOM TEMPERATURE HELIUM PERMEATION MEASUREMENTS ON FINAL PHASE I TEST VENTS

Vent Number	Drawing Number	Description and Core Material	Room Temperature Helium Permeation Rate* std cc/sec at at 10^{-2} atm ΔP
H-10	405345	Helical, Old Thread Design, FS-102 Alumina	3.2×10^{-6}
H-11	"	" " " " " "	4.1×10^{-6}
H-12	"	" " " " " "	3.6×10^{-6}
H-16	405347	Helical, New Thread Design, FS-102 Alumina	3.1×10^{-6}
H-17	"	" " " " " "	14×10^{-6} (defective vent)
H-18**	"	" " " " " "	1.4×10^{-6}
H-23	"	" " " " " "	2.5×10^{-6}
H-19	405347	Helical, New Thread Design, Gold Plate, TRW-100 Alumina	20×10^{-6}
H-20	"	" " " " " " " "	13×10^{-6}
H-21	"	" " " " " " " "	8.6×10^{-6}
H-22	405347	Helical, New Thread Design, Yttria Core	17×10^{-6}
C-14	405346	Coaxial, FS-102 Alumina Core	21×10^{-6}
C-15	"	" " " " "	6.5×10^{-6}
C-16**	"	" " " " "	7.4×10^{-6}
C-24	"	" " " " "	3.6×10^{-6}
C-17	405346	Coaxial, TRW-100 Alumina Core	9.9×10^{-6}
C-18	"	" " " " "	23×10^{-6}
C-19	"	" " " " "	11×10^{-6}
C-20	405346	Coaxial, TRW-100M Alumina Core	65×10^{-6}
C-21	"	" " " " "	81×10^{-6}
C-22	"	" " " " "	110×10^{-6}
C-23	405346	Coaxial, Yttria Core	7.8×10^{-6}

* Vents were tested following a four-hour, 2600°F air heat treatment.

** Extra vent, not in original matrix.

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Figure 48. High Temperature Furnace for Helium Permeation Testing

Test Vent H-10

Figure 49. Helium Permeation Test Setup for Elevated Temperature Measurements

2.2.2.2 Standard Short Term Test Matrix

The final Phase I vent assemblies were exposed to the following short term test matrix:

- Six of the vent assemblies (one of each combination of configuration and sealing technique) were mounted in plastic, sectioned, and metallographically examined to provide "as-fabricated" standards (see Section 2.2.5).
- Six of the vent assemblies were welded into 10-inch Pt-10Rh tubes. The initial helium permeation rates as a function of temperature and pressure differential were established for the following conditions:

<u>Vent Temperature °F</u>	<u>Upstream Pressure (Atm)</u>	<u>Helium Downstream Pressure (Torr)</u>
70		
1000	10^{-1} to 10^{-4}	10^{-5} to 10^{-6}
2000		
2200		

- After measuring the initial helium permeation rates, these six vents were held at 2000°F for a period of one week and the permeation rate as a function of pressure differential was determined on a daily basis.
- The vents were exposed to a single thermal cycle from 2000°F to room temperature and back to 2000°F over a period of ninety minutes; the helium permeation rates as a function of pressure differential were redetermined.
- The vents were thermally cycled three additional times (as described above) and the permeation rates as a function of pressure differential were redetermined.
- Six of the vent assemblies were used as backup test specimens, as required, to supplement the first six assemblies in the testing sequence.
- The two yttria-containing vents (H-22 and C-23) were held in reserve.
- The two vents (H-23 and C-24) were extra vents not in the original matrix.

2.2.3 Short Term Elevated Temperature Helium Permeation Testing-Results of Standard Matrix Experiments

The vents fabricated for final Phase I testing consisted of twenty specimens, ten helical (H numbers), and ten coaxial (C numbers) designs as presented in Table 2, Section 2.1.1.5. Each of these major groups of ten was further divided into three sets of three identically fabricated vents plus one using yttria in place of alumina (see Table 2). The eighteen alumina vents fall into three categories: 1) vents for initial elevated temperature testing (H-10, H-16, H-19, C-15, C-18, and C-21), 2) vents to be held in reserve (H-12, H-18, H-20, C-14, C-19, and C-20), and 3) vents to be metallographically examined in the "as-fabricated" condition (H-11, H-17, H-21, C-16, C-17, and C-22).

Standard short term matrix testing of vents H-10, H-16, H-19, C-15, C-18, and C-21 was successfully completed and is summarized in Table 9. The important conclusions reached by analyses of the permeation rate data are listed below.

- The helium permeation behavior of these vents remained constant for a given helium pressure differential at all steady-state temperatures measured (R.T. to 2200°F).
- Four thermal cycles from 2000°F to room temperature and back to 2000°F had no measurable effect on the helium leak rate of any of the devices.
- The experimental data correlated reasonably well with theoretical predictions of helium permeation through porous filter media as discussed in Section 2.2.6.
- The short term tests did not differentiate between any of the designs. That is to say all vent designs responded in a completely stable fashion.

2.2.4 Long Term, 2000°F Helium Permeation Testing of Vents

At the conclusion of the short term standard matrix testing described above, the vents H-10, H-16, H-19, C-15, C-18, and C-21 were continued on test at a temperature of 2000°F for periods up to 25,000 hours, while maintaining helium flow through the vents ($\sim 10^{-2}$ atm ΔP). After 1500 hours, vent H-16 was removed from testing and replaced by yttria vent C-23 which remained on test for the remainder of the testing period. The vents

TABLE 9. SUMMARY OF INITIAL ELEVATED TEMPERATURE HELIUM PERMEATION MEASUREMENTS ON VENTS H-10, H-16, H-19, C-15, C-18, AND C-21

Vent Number	HELIUM PERMEATION RATE - STD CC/SEC AT 10 ⁻² ATMOSPHERE ΔP														
	Room Temperature (after assembly)	1000°F	2000°F	2200°F	Permeation Test at 2000°F*							2000°F (thermal cycling)**			
					First Day	Second Day	Third Day	Fourth Day	Fifth Day	Sixth Day	Seventh Day	First Cycle	Second Cycle	Third Cycle	Fourth Cycle
H-10	3.2 X 10 ⁻⁶	2.5X10 ⁻⁶	2.3X10 ⁻⁶	2.2X10 ⁻⁶	2.3X10 ⁻⁶	2.3X10 ⁻⁶	2.2X10 ⁻⁶	2.4X10 ⁻⁶	2.4X10 ⁻⁶	2.2X10 ⁻⁶	2.2X10 ⁻⁶	2.2X10 ⁻⁶ before 2.4X10 ⁻⁶ after	2.2X10 ⁻⁶ before 2.2X10 ⁻⁶ after	2.2X10 ⁻⁶ before 2.4X10 ⁻⁶ after	2.4X10 ⁻⁶ before 2.4X10 ⁻⁶ after
H-16	3.1 X 10 ⁻⁶	2.3X10 ⁻⁶	2.2X10 ⁻⁶	2.1X10 ⁻⁶	2.5X10 ⁻⁶	2.5X10 ⁻⁶	2.5X10 ⁻⁶	2.5X10 ⁻⁶	2.0X10 ⁻⁶	2.2X10 ⁻⁶	2.0X10 ⁻⁶	2.0X10 ⁻⁶ 1.8X10 ⁻⁶	1.8X10 ⁻⁶ 1.8X10 ⁻⁶	1.8X10 ⁻⁶ 1.6X10 ⁻⁶	1.6X10 ⁻⁶ 1.8X10 ⁻⁶
H-19	20 X10 ⁻⁶	13X10 ⁻⁶	11X10 ⁻⁶	8.3X10 ⁻⁶	11X10 ⁻⁶	10X10 ⁻⁶	9.0X10 ⁻⁶	7.6X10 ⁻⁷	6.3X10 ⁻⁶	10X10 ⁻⁶	7.2X10 ⁻⁶	7.2X10 ⁻⁶ 7.2X10 ⁻⁶	7.2X10 ⁻⁶ 7.6X10 ⁻⁶	7.6X10 ⁻⁶ 8.6X10 ⁻⁶	8.6X10 ⁻⁶ 8.6X10 ⁻⁶
C-15	6.5 X 10 ⁻⁶	4.3X10 ⁻⁶	3.6X10 ⁻⁶	3.6X10 ⁻⁶	3.6X10 ⁻⁶	4.0X10 ⁻⁶	3.1X10 ⁻⁶	2.7X10 ⁻⁶	2.3X10 ⁻⁶	3.6X10 ⁻⁶	2.5X10 ⁻⁶	2.5X10 ⁻⁶ 2.5X10 ⁻⁶	2.5X10 ⁻⁶ 2.5X10 ⁻⁶	2.5X10 ⁻⁶ 2.9X10 ⁻⁶	2.9X10 ⁻⁶ 2.9X10 ⁻⁶
C-18	23 X 10 ⁻⁶	14X10 ⁻⁶	11X10 ⁻⁶	9.9X10 ⁻⁶	11X10 ⁻⁶	10X10 ⁻⁶	11X10 ⁻⁶	8.8X10 ⁻⁶	7.2X10 ⁻⁶	10X10 ⁻⁶	10X10 ⁻⁶	8.0X10 ⁻⁶ 7.5X10 ⁻⁶	7.9X10 ⁻⁶ 7.7X10 ⁻⁶	7.7X10 ⁻⁶ 7.4X10 ⁻⁶	7.4X10 ⁻⁶ 7.7X10 ⁻⁶
C-21	81 X 10 ⁻⁶	50X10 ⁻⁶	40X10 ⁻⁶	36X10 ⁻⁶	40X10 ⁻⁶	41X10 ⁻⁶	40X10 ⁻⁶	31X10 ⁻⁶	25X10 ⁻⁶	40X10 ⁻⁶	29X10 ⁻⁶	2.9X10 ⁻⁶ 2.7X10 ⁻⁶	2.7X10 ⁻⁶ 2.7X10 ⁻⁶	2.7X10 ⁻⁶ 3.6X10 ⁻⁶	3.6X10 ⁻⁶ 3.4X10 ⁻⁶

* Vents were tested following a four-hour, 2600°F air heat treatment

** See Section 2.2.2.2

were periodically thermally cycled to room temperature and back to 2000°F. Helium permeation rate measurements at 2000°F were taken on each vent on a regular basis during the course of the long term testing. At the end of the testing program helium permeation rate measurements were made at 70°F, 1000°F, 2000°F, and 2200°F with pressure differentials across the vents of 10^{-2} and 10^{-3} atm.

Through 1 June 1972, vents C-15, C-18, C-21, and H-19 were tested continuously at a temperature of 2000°F for 23,920 hours. Vents C-23 and H-10 were tested under similar circumstances for 22,660 hours and 25,020 hours respectively. These six vents have accumulated a total of 143,360 hours of completely stable steady state testing. Table 10, typical of recorded data for all of the above six vents, presents the helium permeation test history of vent H-10. Tables 11-15 list steady state operation helium permeation data for vents H-19, C-15, C-18, C-21 and C-23 in increments of approximately 500 hours of testing time.

Figures 50 through 56 show representative helium permeation data which indicate the stability of the vents during these long term test periods.

It is apparent from the recorded helium permeation data that all of the subject vents have remained, through 1 June 1972, completely stable, not only during steady-state high temperature operation but also after repeated thermal cycling to room temperature and back to operating temperature.

The important conclusions reached by analysis of the helium permeation rate data are listed below:

- The helium permeation behavior of the test vents remained constant for a given helium pressure differential at all steady-state temperatures (70 to 2200°F) measured.
- Repeated thermal cycling from 2000°F to 70°F and back to 2000°F had no significant effect on the helium permeation rate of any of the vent assemblies.

TABLE 10. HELIUM PERMEATION TEST HISTORY OF VENT H-10 (Drawing No. X405356)

Date	Cumulative Hours of Testing at 2000°F or Greater	Test Temperature °F	Helium Permeation Rate std cc/sec		Remarks
			at 10^{-2} atm ΔP	at 10^{-3} atm ΔP	
7/3/69	-	Room Temperature	2.7×10^{-6}	2.7×10^{-7}	After 4 hours heat treatment at 2800°F in air
7/8/69	-	Room Temperature	2.9×10^{-6}	2.9×10^{-7}	After 8 hours total heat treatment at 2800°F in air
7/15/69	-	Room Temperature	3.2×10^{-6}	3.2×10^{-7}	After welding into Pt-10Rh extension tubes
7/16/69	-	1000	2.5×10^{-6}	2.9×10^{-7}	Initial 1000°F measurement
7/16/69	0	2000	2.3×10^{-6}	2.7×10^{-7}	Initial 2000°F measurement
7/17/69	24	2000	2.2×10^{-6}	2.5×10^{-7}	
7/17/69	28	2200	2.2×10^{-6}	2.5×10^{-7}	
7/18/69	48	2000	2.3×10^{-6}	2.7×10^{-7}	
7/19/69	72	2000	2.3×10^{-6}	2.7×10^{-7}	
7/20/69	96	2000	2.2×10^{-6}	2.5×10^{-7}	
7/21/69	120	2000	2.4×10^{-6}	2.8×10^{-7}	
7/23/69	144	2000	2.4×10^{-6}	2.8×10^{-7}	
7/23/69	168	2000	2.2×10^{-6}	2.6×10^{-7}	
7/23/69	172	2000	2.4×10^{-6}	2.8×10^{-7}	After first thermal cycle 2000°F to R.T. to 2000°F
7/24/69	190	2000	2.2×10^{-6}	2.8×10^{-7}	After second thermal cycle 2000°F to R.T. to 2000°F
7/24/69	194	2000	2.2×10^{-6}	2.6×10^{-7}	After third thermal cycle 2000°F to R.T. to 2000°F
7/25/69	216	2000	2.4×10^{-6}	2.8×10^{-7}	After fourth thermal cycle 2000°F to R.T. to 2000°F
7/28/69	288	2000	1.7×10^{-6}	1.9×10^{-7}	Instrument sensitivity change may account for slight change in permeation rate
8/1/69	384	2000	2.1×10^{-6}	2.2×10^{-7}	
8/4/69	456	2000	1.5×10^{-6}	1.7×10^{-7}	Instrument sensitivity change may account for slight change in permeation rate
8/8/69	552	2000	1.5×10^{-6}	1.5×10^{-7}	
8/11/69	624	2000	1.4×10^{-6}	1.4×10^{-7}	
8/13/69	672	2000	1.4×10^{-6}	1.4×10^{-7}	
8/14/69	690	2000	1.6×10^{-6}	1.9×10^{-7}	
8/15/69	714	2000	1.6×10^{-6}	1.7×10^{-7}	
8/18/69	786	2000	1.4×10^{-6}	1.5×10^{-7}	
8/20/69	834	2000	1.6×10^{-6}	1.8×10^{-7}	
8/22/69	882	2000	1.7×10^{-6}	1.8×10^{-7}	
8/25/69	954	2000	1.6×10^{-6}	1.7×10^{-7}	
8/29/69	978	2000	2.0×10^{-6}	2.5×10^{-7}	
9/2/69	1152	2000	2.3×10^{-6}	2.5×10^{-7}	
9/3/69	1176	2000	1.8×10^{-6}	2.2×10^{-7}	
9/4/69	1200	2000	1.6×10^{-6}	1.8×10^{-7}	
9/5/69	1224	2000	1.5×10^{-6}	1.6×10^{-7}	
9/8/69	1296	2000	1.8×10^{-6}	2.2×10^{-7}	
9/9/69	1320	2000	1.7×10^{-6}	1.8×10^{-7}	
9/10/69	1344	2000	1.6×10^{-6}	1.8×10^{-7}	
9/12/69	1392	2000	1.8×10^{-6}	2.3×10^{-7}	
9/15/69	1464	2000	2.0×10^{-6}	2.3×10^{-7}	
9/17/69	1512	2000	1.8×10^{-6}	2.2×10^{-7}	
9/19/69	1560	2000	2.0×10^{-6}	2.3×10^{-7}	
9/23/69	1656	2000	2.0×10^{-6}	2.3×10^{-7}	
9/24/69	1680	2000	1.8×10^{-6}	2.2×10^{-7}	
9/26/69	1728	2000	1.8×10^{-6}	2.2×10^{-7}	
9/29/69	1800	2000	2.0×10^{-6}	2.2×10^{-7}	
10/3/69	1896	2000	1.7×10^{-6}	2.2×10^{-7}	
10/6/69	1968	2000	2.0×10^{-6}	2.3×10^{-7}	
10/8/69	2016	2000	1.8×10^{-6}	2.3×10^{-7}	
10/10/69	2064	2000	1.5×10^{-6}	2.0×10^{-7}	
10/14/69	2160	2000	2.1×10^{-6}	2.6×10^{-7}	
10/21/69	2328	2000	1.8×10^{-6}	2.4×10^{-7}	
10/27/69	2472	2000	1.8×10^{-6}	2.2×10^{-7}	
10/29/69	2520	2000	2.4×10^{-6}	2.4×10^{-7}	
11/3/69	2614	2000	2.1×10^{-6}		
11/10/69	2782	2000	2.2×10^{-6}		
11/17/69	2950	2000	2.3×10^{-6}		
11/19/69	2998	2000	2.1×10^{-6}		
11/20/69	3022	2000	2.1×10^{-6}		
11/21/69	3046	2000	1.8×10^{-6}		
11/24/69	3118	2000	2.2×10^{-6}		
11/25/69	3142	2000	2.1×10^{-6}		
12/1/69	3286	2000	2.0×10^{-6}		
12/8/69	3454	2000	2.2×10^{-6}		
12/15/69	3622	2000	2.4×10^{-6}		
12/16/69	3646	2000	2.3×10^{-6}		
12/17/69	3670	2000	1.9×10^{-6}		
12/18/69	3694	2000	1.9×10^{-6}		
12/22/69	3718	2000	2.1×10^{-6}		After Thermal Cycle
12/23/69	3742	2000	1.9×10^{-6}		After Thermal Cycle
12/24/69	3766	2000	1.9×10^{-6}		After Thermal Cycle
12/30/69	3910	2000	2.6×10^{-6}		After Thermal Cycle
1/5/70	4054	2000	2.4×10^{-6}		
1/12/70	4222	2000	2.4×10^{-6}		
1/20/70	4414	2000	2.1×10^{-6}		
1/28/70	4606	2000	2.6×10^{-6}		
2/2/70	4726	2000	2.2×10^{-6}		After Thermal Cycle
2/4/70	4774	1000	1.8×10^{-6}		After Thermal Cycle
2/5/70	4798	2000	1.8×10^{-6}		After Thermal Cycle
2/5/70	4798	2200	2.9×10^{-6}		After Thermal Cycle
2/6/70	4822	2000	2.9×10^{-6}		
2/9/70	4894	2000	2.2×10^{-6}		
2/10/70	4918	2000	2.3×10^{-6}		
2/11/70	4942	2000	2.3×10^{-6}		
2/16/70	5062	2000	2.0×10^{-6}		
2/23/70	5230	2000	2.3×10^{-6}		After Thermal Cycle
2/23/70	5230	1000	2.0×10^{-6}		After Thermal Cycle
2/24/70	5254	2000	2.0×10^{-6}		After Thermal Cycle
2/24/70	5254	2200	2.0×10^{-6}		After Thermal Cycle
2/25/70	5278	2000	2.0×10^{-6}		
2/26/70	5302	2000	2.0×10^{-6}		
2/27/70	5326	2000	2.0×10^{-6}		
3/3/70	5422	2000	1.8×10^{-6}		
3/6/70	5494	2000	2.2×10^{-6}		
3/10/70	5590	2000	2.2×10^{-6}		
3/17/70	5758	2000	2.2×10^{-6}		
3/20/70	5830	R.T.	2.2×10^{-6}		
3/23/70	5902	2000	2.2×10^{-6}		
3/24/70	5926	2200	2.1×10^{-6}		
3/25/70	5950	R.T.	1.9×10^{-6}		
3/25/70	5950	1000	2.1×10^{-6}		
3/26/70	5974	2000	2.2×10^{-6}		
3/27/70	5998	R.T.	1.8×10^{-6}		
3/30/70	6070	2000	2.1×10^{-6}		
3/31/70	6094	R.T.	2.3×10^{-6}		
3/31/70	6094	2000	1.5×10^{-6}		

TABLE 10. (Continued). HELIUM PERMEATION TEST HISTORY OF VENT H-10 (Drawing No. X405345)

Date	Cumulative Hours of Testing at 2000°F or Greater	Test Temperature °F	Helium Permeation Rate std cc/sec		Remarks
			at 10 ⁻² atm ΔP	at 10 ⁻³ atm ΔP	
4/1/70	6118	70	3.2 x 10 ⁻⁶		
4/2/70	6142	2000	1.7 x 10 ⁻⁶		
4/3/70	6166	70	3.9 x 10 ⁻⁶		
4/3/70	6170	2000	1.6 x 10 ⁻⁶		
4/7/70	6262	2000	1.5 x 10 ⁻⁶		
4/13/70	6406	2000	2.0 x 10 ⁻⁶		
4/24/70	6670	2000	2.0 x 10 ⁻⁶		
4/28/70	6766	2000	1.7 x 10 ⁻⁶		
5/6/70	6958	2200	2.6 x 10 ⁻⁶		
5/8/70	7106	70	4.6 x 10 ⁻⁶		
5/11/70	7178	1000	3.6 x 10 ⁻⁶		
5/15/70	7274	2000	5.9 x 10 ⁻⁶		
5/16/70	7298	70	4.4 x 10 ⁻⁶		
5/16/70	7306	2000	3.1 x 10 ⁻⁶		
5/18/70	7322	70	4.6 x 10 ⁻⁶		
5/18/70	7328	2000	3.7 x 10 ⁻⁶		
5/19/70	7346	70	4.6 x 10 ⁻⁶		
5/19/70	7352	2000	3.3 x 10 ⁻⁶		
5/20/70	7370	70	4.0 x 10 ⁻⁶		
5/20/70	7376	2000	2.6 x 10 ⁻⁶		
5/21/70	7354	2200	3.4 x 10 ⁻⁶		
5/22/70	7418	70	4.8 x 10 ⁻⁶		
5/22/70	7424	1000	4.1 x 10 ⁻⁶		
5/25/70	7490	2000	3.1 x 10 ⁻⁶		
5/26/70	7514	70	4.3 x 10 ⁻⁶		
5/26/70	7520	2000	2.3 x 10 ⁻⁶		
5/27/70	7538	70	4.6 x 10 ⁻⁶		
5/27/70	7544	2000	3.2 x 10 ⁻⁶		
5/28/70	7562	70	4.2 x 10 ⁻⁶		
5/28/70	7568	2000	2.9 x 10 ⁻⁶		
5/29/70	7586	70	4.4 x 10 ⁻⁶		
5/29/70	7592	2000	3.6 x 10 ⁻⁶		
5/30/70	7616	2000	2.9 x 10 ⁻⁶		
7/70	8190	2000	3.8 x 10 ⁻⁶		
8/70	8862	2000	3.0 x 10 ⁻⁶		
9/70	9558	2000	2.7 x 10 ⁻⁶		
10/70	10,134	2000	2.7 x 10 ⁻⁶		
11/70	10,878	2000	2.0 x 10 ⁻⁶		
12/70	11,478	2000	3.1 x 10 ⁻⁶		
1/71	12,126	2000	2.6 x 10 ⁻⁶		
2/71	12,822	2000	2.4 x 10 ⁻⁶		
3/71	13,926	2000	1.6 x 10 ⁻⁶		
4/71	14,674	2000	1.6 x 10 ⁻⁶		
5/71	15,202	2000	1.4 x 10 ⁻⁶		
6/71	16,090	2000	1.2 x 10 ⁻⁶	1.4 x 10 ⁻⁷	
7/71	17,122	2000	1.9 x 10 ⁻⁶	2.1 x 10 ⁻⁷	
8/71	17,290	2000	2.7 x 10 ⁻⁶	3.9 x 10 ⁻⁷	
9/71	18,130	2000	3.1 x 10 ⁻⁶	3.9 x 10 ⁻⁷	
10/71	19,066	2000	1.8 x 10 ⁻⁶	2.3 x 10 ⁻⁷	
11/71	19,450	2000	2.1 x 10 ⁻⁶	2.6 x 10 ⁻⁷	
12/71	20,122	2000	1.6 x 10 ⁻⁶	2.1 x 10 ⁻⁷	
12/71		70	1.8 x 10 ⁻⁶	2.7 x 10 ⁻⁷	
12/71	20,386	2000	1.1 x 10 ⁻⁶	1.7 x 10 ⁻⁷	
12/71		70	2.9 x 10 ⁻⁶	3.1 x 10 ⁻⁷	
12/71	20,458	2000	2.1 x 10 ⁻⁶	3.1 x 10 ⁻⁷	
12/71		70	2.9 x 10 ⁻⁶	3.6 x 10 ⁻⁷	
12/71	20,482	2000	2.1 x 10 ⁻⁶	3.1 x 10 ⁻⁷	
12/71		70	2.9 x 10 ⁻⁶	3.6 x 10 ⁻⁷	
12/71	21,010	2000	2.1 x 10 ⁻⁶	3.1 x 10 ⁻⁷	
12/71	21,058	2200	2.3 x 10 ⁻⁶	3.1 x 10 ⁻⁷	
1/72	21,922	2000	2.7 x 10 ⁻⁶	3.6 x 10 ⁻⁷	
2/72	22,306	2000	2.3 x 10 ⁻⁶	3.2 x 10 ⁻⁷	
3/72	23,242	2000	2.5 x 10 ⁻⁶	3.2 x 10 ⁻⁷	
4/72	23,770	2000	2.7 x 10 ⁻⁶	3.6 x 10 ⁻⁷	
5/72	24,970	2000	2.5 x 10 ⁻⁶	3.4 x 10 ⁻⁷	
6/72	24,994	2000	2.9 x 10 ⁻⁶	3.6 x 10 ⁻⁷	
6/72		70	3.5 x 10 ⁻⁶	4.2 x 10 ⁻⁷	
6/72		1000	3.1 x 10 ⁻⁶	4.0 x 10 ⁻⁷	
6/72	25,020	2200	2.3 x 10 ⁻⁶	3.1 x 10 ⁻⁷	
6/72		70	3.5 x 10 ^{-6*}	4.2 x 10 ^{-7*}	
6/72		70	3.8 x 10 ^{-6**}	4.6 x 10 ^{-7**}	
6/72		2000	2.9 x 10 ^{-6*}	3.6 x 10 ^{-7*}	
6/72		2000	2.9 x 10 ^{-6**}	4.2 x 10 ^{-7**}	

* Before simulated reentry thermal cycle test.
** After simulated reentry thermal cycle test.

TABLE 11. LONG TERM HELIUM PERMEATION TESTING OF VENT H-19

Date	Cumulative Hours of Testing at 2000°F or Greater	Test Temperature °F	Helium Permeation Rate std cc/sec at 10 ⁻² Atm ΔP	Remarks
8/16/69	0	70	8.6 x 10 ⁻⁶	Initial test at R.T. Initial test at 2000°F
8/28/69	0	2000	1.1 x 10 ⁻⁵	
9/19/69	528	2000	9.0 x 10 ⁻⁶	
10/8/69	1008	2000	8.3 x 10 ⁻⁶	
10/27/70	1464	2000	1.1 x 10 ⁻⁵	
11/24/69	2087	2000	1.0 x 10 ⁻⁵	
12/15/69	2591	2000	1.1 x 10 ⁻⁵	
1/5/70	3032	2000	1.0 x 10 ⁻⁵	
1/28/70	3584	2000	9.0 x 10 ⁻⁶	
2/16/70	4040	2000	8.6 x 10 ⁻⁶	
3/6/70	4472	2000	8.8 x 10 ⁻⁶	
3/30/70	5048	2000	7.0 x 10 ⁻⁶	
4/28/70	5579	2000	7.9 x 10 ⁻⁶	
5/30/70	6000	2000	4.0 x 10 ⁻⁶	
7/70	7216	2000	1.2 x 10 ⁻⁵	
8/70	7864	2000	9.5 x 10 ⁻⁶	
9/70	8560	2000	5.8 x 10 ⁻⁶	
10/70	9136	2000	9.7 x 10 ⁻⁶	
11/70	9880	2000	7.0 x 10 ⁻⁶	
12/70	10,480	2000	1.1 x 10 ⁻⁵	
1/71	11,128	2000	8.6 x 10 ⁻⁶	
2/71	11,824	2000	9.0 x 10 ⁻⁶	
3/71	12,928	2000	6.0 x 10 ⁻⁶	
4/71	13,577	2000	6.8 x 10 ⁻⁶	
5/71	14,105	2000	5.8 x 10 ⁻⁶	
6/71	14,993	2000	5.1 x 10 ⁻⁶	
7/71	16,025	2000	7.3 x 10 ⁻⁶	
8/71	16,193	2000	1.1 x 10 ⁻⁵	
9/71	17,033	2000	1.0 x 10 ⁻⁵	
10/71	17,969	2000	6.8 x 10 ⁻⁶	
11/71	18,353	2000	7.4 x 10 ⁻⁶	
12/71	19,025	2000	7.1 x 10 ⁻⁶	
1/72	20,585	2000	8.8 x 10 ⁻⁶	
2/72	20,969	2000	8.0 x 10 ⁻⁶	
3/72	21,905	2000	8.4 x 10 ⁻⁶	
4/72	22,433	2000	9.7 x 10 ⁻⁶	
5/72	23,633	2000	8.4 x 10 ⁻⁶	
6/72	23,657	2000	8.8 x 10 ⁻⁶	
6/72		70	2.5 x 10 ⁻⁵	
6/72		1000	1.0 x 10 ⁻⁵	
6/72	23,920	2000	8.4 x 10 ⁻⁶	

TABLE 12. LONG TERM HELIUM PERMEATION TESTING OF VENT C-15

Date	Cumulative Hours of Testing at 2000°F or Greater	Test Temperature °F	Helium Permeation Rate std cc/sec at 10 ⁻² Atm ΔP	Remarks
8/16/69	0	70	1.6 x 10 ⁻⁶	Initial test at R.T.
8/28/69	0	2000	3.6 x 10 ⁻⁶	Initial test at 2000°F
9/19/69	528	2000	3.1 x 10 ⁻⁶	
10/8/70	1008	2000	2.7 x 10 ⁻⁶	
10/27/70	1464	2000	3.1 x 10 ⁻⁶	
11/21/70	2087	2000	2.8 x 10 ⁻⁶	
12/15/70	2591	2000	3.2 x 10 ⁻⁶	
1/5/70	3032	2000	3.6 x 10 ⁻⁶	
1/28/70	3584	2000	3.2 x 10 ⁻⁶	
2/16/70	4040	2000	3.1 x 10 ⁻⁶	
3/6/70	4472	2000	4.0 x 10 ⁻⁶	
3/30/70	5048	2000	2.6 x 10 ⁻⁶	
4/28/70	5579	2000	2.2 x 10 ⁻⁶	
5/30/70	6000	2000	4.0 x 10 ⁻⁶	
7/70	7216	2000	5.0 x 10 ⁻⁶	
8/70	7864	2000	3.4 x 10 ⁻⁶	
9/70	8560	2000	3.2 x 10 ⁻⁶	
10/70	9136	2000	3.0 x 10 ⁻⁶	
11/70	9880	2000	2.4 x 10 ⁻⁶	
12/70	10,480	2000	3.1 x 10 ⁻⁶	
1/71	11,128	2000	2.9 x 10 ⁻⁶	
2/71	11,824	2000	2.8 x 10 ⁻⁶	
3/71	12,928	2000	2.0 x 10 ⁻⁶	
4/71	13,577	2000	2.3 x 10 ⁻⁶	
5/71	14,105	2000	1.8 x 10 ⁻⁶	
6/71	14,993	2000	1.7 x 10 ⁻⁶	
7/71	16,025	2000	2.4 x 10 ⁻⁶	
8/71	16,193	2000	3.6 x 10 ⁻⁶	
9/71	17,033	2000	3.6 x 10 ⁻⁶	
10/71	17,969	2000	2.6 x 10 ⁻⁶	
11/71	18,353	2000	2.8 x 10 ⁻⁶	
12/71	19,025	2000	2.8 x 10 ⁻⁶	
1/72	20,585	2000	2.9 x 10 ⁻⁶	
2/72	20,969	2000	2.7 x 10 ⁻⁶	
3/72	21,905	2000	2.9 x 10 ⁻⁶	
4/72	22,433	2000	5.9 x 10 ⁻⁶	
5/72	23,633	2000	2.7 x 10 ⁻⁶	
6/72	23,657	2000	3.6 x 10 ⁻⁶	
6/72		70	4.4 x 10 ⁻⁶	
6/72		1000	3.6 x 10 ⁻⁶	
6/72	23,920	2000	2.9 x 10 ⁻⁶	

TABLE 13. LONG TERM HELIUM PERMEATION TESTING OF VENT C-18

Date	Cumulative Hours of Testing at 2000°F or Greater	Test Temperature °F	Helium Permeation Rate std cc/sec at 10 ⁻² Atm ΔP	Remarks
8/16/69	0	70	5.9 x 10 ⁻⁶	Initial test at R.T. Initial test at 2000°F
8/28/69	0	2000	1.1 x 10 ⁻⁶	
9/19/69	528	2000	9.0 x 10 ⁻⁶	
10/8/69	1008	2000	7.9 x 10 ⁻⁵	
10/27/69	1464	2000	1.1 x 10 ⁻⁶	
11/21/69	2015	2000	7.4 x 10 ⁻⁵	
12/15/69	2591	2000	1.2 x 10 ⁻⁵	
1/5/70	3032	2000	1.0 x 10 ⁻⁵	
1/28/70	3584	2000	1.0 x 10 ⁻⁶	
2/16/70	4040	2000	9.4 x 10 ⁻⁶	
3/6/70	4472	2000	8.4 x 10 ⁻⁵	
3/30/70	5048	2000	1.1 x 10 ⁻⁶	
4/28/70	5574	2000	7.9 x 10 ⁻⁵	
5/30/70	6000	2000	1.0 x 10 ⁻⁵	
7/70	7211	2000	1.2 x 10 ⁻⁵	
8/70	7859	2000	9.9 x 10 ⁻⁶	
9/70	8555	2000	9.6 x 10 ⁻⁶	
10/70	9131	2000	8.9 x 10 ⁻⁶	
11/70	8975	2000	6.2 x 10 ⁻⁶	
12/70	10,475	2000	9.7 x 10 ⁻⁶	
1/71	11,123	2000	8.3 x 10 ⁻⁶	
2/71	11,819	2000	8.7 x 10 ⁻⁶	
3/71	12,923	2000	6.0 x 10 ⁻⁶	
4/71	13,577	2000	6.2 x 10 ⁻⁶	
5/71	14,105	2000	4.8 x 10 ⁻⁶	
6/71	14,993	2000	4.1 x 10 ⁻⁶	
7/71	16,025	2000	6.8 x 10 ⁻⁶	
8/71	16,193	2000	9.8 x 10 ⁻⁶	
9/71	17,033	2000	1.1 x 10 ⁻⁶	
10/71	17,969	2000	4.7 x 10 ⁻⁶	
11/71	18,353	2000	6.9 x 10 ⁻⁶	
12/71	19,025	2000	6.9 x 10 ⁻⁶	
1/72	20,585	2000	7.7 x 10 ⁻⁶	
2/72	20,969	2000	6.9 x 10 ⁻⁶	
3/72	21,905	2000	7.3 x 10 ⁻⁶	
4/72	22,433	2000	8.0 x 10 ⁻⁶	
5/72	23,633	2000	7.3 x 10 ⁻⁶	
6/72	23,657	2000	7.8 x 10 ⁻⁶	
6/72		70	1.3 x 10 ⁻⁵	
6/72		1000	1.1 x 10 ⁻⁵	
6/72	23,920	2200	7.1 x 10 ⁻⁶	

TABLE 14. LONG TERM HELIUM PERMEATION TESTING OF VENT C-21

Date	Cumulative Hours of Testing at 2000°F or Greater	Test Temperature °F	Helium Permeation Rate std cc/sec at 10 ⁻² Atm ΔP	Remarks
8/16/69	0	70	4.3 x 10 ⁻⁵	Initial test at R. T. Initial test at 2000°F
8/28/69	0	2000	4.0 x 10 ⁻⁵	
9/19/69	528	2000	3.8 x 10 ⁻⁵	
10/8/69	1008	2000	3.4 x 10 ⁻⁵	
10/27/69	1464	2000	5.3 x 10 ⁻⁵	
11/21/69	2015	2000	3.5 x 10 ⁻⁵	
12/15/69	2591	2000	4.9 x 10 ⁻⁵	
1/5/70	3032	2000	4.2 x 10 ⁻⁵	
1/28/70	3584	2000	3.2 x 10 ⁻⁵	
2/16/70	4040	2000	5.1 x 10 ⁻⁵	
3/6/70	4472	2000	3.1 x 10 ⁻⁵	
3/30/70	5048	2000	3.1 x 10 ⁻⁵	
4/28/70	5576	2000	2.6 x 10 ⁻⁵	
5/30/70	6000	2000	5.0 x 10 ⁻⁵	
7/70	7213	2000	5.8 x 10 ⁻⁵	
8/70	7861	2000	4.0 x 10 ⁻⁵	
9/70	8557	2000	3.8 x 10 ⁻⁵	
10/70	9133	2000	4.3 x 10 ⁻⁵	
11/70	9877	2000	2.9 x 10 ⁻⁵	
12/70	10,477	2000	4.2 x 10 ⁻⁵	
1/71	11,125	2000	3.9 x 10 ⁻⁵	
2/71	11,821	2000	4.3 x 10 ⁻⁵	
3/71	12,925	2000	3.9 x 10 ⁻⁵	
4/71	13,577	2000	2.6 x 10 ⁻⁵	
5/71	14,105	2000	2.5 x 10 ⁻⁵	
6/71	14,993	2000	2.1 x 10 ⁻⁵	
7/71	16,025	2000	3.6 x 10 ⁻⁵	
8/71	16,193	2000	5.3 x 10 ⁻⁵	
9/71	17,033	2000	4.5 x 10 ⁻⁵	
10/71	17,969	2000	2.9 x 10 ⁻⁵	
11/71	18,353	2000	3.2 x 10 ⁻⁵	
12/71	19,025	2000	4.1 x 10 ⁻⁵	
1/72	20,585	2000	4.4 x 10 ⁻⁵	
2/72	20,969	2000	4.0 x 10 ⁻⁵	
3/72	21,905	2000	4.2 x 10 ⁻⁵	
4/72	22,433	2000	4.0 x 10 ⁻⁵	
5/72	23,633	2000	3.6 x 10 ⁻⁵	
6/72	23,657	2000	3.6 x 10 ⁻⁵	
6/72		70	5.7 x 10 ⁻⁵	
6/72		1000	4.2 x 10 ⁻⁵	
6/72	23,920	2200	3.4 x 10 ⁻⁵	

TABLE 15. LONG TERM HELIUM PERMEATION TESTING OF VENT C-23

Date	Cumulative Hours of Testing at 2000°F or Greater	Test Temperature °F	Helium Permeation Rate std cc/sec at 10 ⁻² Atm ΔP	Remarks
10/29/69	0	70	1.1 x 10 ⁻⁵	Initial test at R.T. Initial test at 2000°F
10/31/69	0	2000	6.4 x 10 ⁻⁶	
11/20/69	504	2000	8.1 x 10 ⁻⁵	
12/8/69	936	2000	1.0 x 10 ⁻⁵	
1/5/70	1536	2000	1.0 x 10 ⁻⁵	
1/28/70	2088	2000	1.0 x 10 ⁻⁵	
2/16/70	2544	2000	1.1 x 10 ⁻⁵	
3/10/70	3072	2000	1.1 x 10 ⁻⁵	
3/30/70	3552	2000	1.2 x 10 ⁻⁵	
4/28/70	4078	2000	9.7 x 10 ⁻⁶	
5/25/70	4530	2000	1.5 x 10 ⁻⁵	
5/30/70	4572	2000	1.2 x 10 ⁻⁵	
7/70	5931	2000	1.2 x 10 ⁻⁵	
8/70	6603	2000	9.5 x 10 ⁻⁶	
9/70	7299	2000	9.6 x 10 ⁻⁶	
10/70	7875	2000	9.5 x 10 ⁻⁶	
11/70	8619	2000	6.4 x 10 ⁻⁶	
12/70	9219	2000	1.2 x 10 ⁻⁵	
1/71	9867	2000	8.6 x 10 ⁻⁶	
2/71	10,563	2000	9.3 x 10 ⁻⁶	
3/71	11667	2000	6.5 x 10 ⁻⁶	
4/71	12,315	2000	5.7 x 10 ⁻⁶	
5/71	12,843	2000	5.1 x 10 ⁻⁶	
6/71	13,731	2000	4.8 x 10 ⁻⁶	
7/71	14,763	2000	6.0 x 10 ⁻⁶	
8/71	14,931	2000	9.5 x 10 ⁻⁶	
9/71	15,771	2000	9.5 x 10 ⁻⁶	
10/71	16,707	2000	5.2 x 10 ⁻⁶	
11/71	17,331	2000	5.5 x 10 ⁻⁶	
12/71	17,763	2000	5.1 x 10 ⁻⁶	
1/72	19,323	2000	7.6 x 10 ⁻⁶	
2/72	19,707	2000	6.5 x 10 ⁻⁶	
3/72	20,643	2000	7.6 x 10 ⁻⁶	
4/72	21,171	2000	8.0 x 10 ⁻⁶	
5/72	22,371	2000	7.1 x 10 ⁻⁶	
6/72	22,395	2000	8.2 x 10 ⁻⁶	
6/72		70	1.3 x 10 ⁻⁵	
6/72		1000	1.0 x 10 ⁻⁵	
6/72	22,660	2200	7.3 x 10 ⁻⁶

Figure 50. Long Term Helium Permeation Test History for Vent H-10

Figure 51. Long Term Helium Permeation Test History for Vent H-19

Figure 52. Long Term Helium Permeation Test History for Vent C-15

Figure 54. Long Term Helium Permeation Test History for Vent C-21

Figure 55. Long Term Helium Permeation Test History for Vent C-23

Figure 56. Long Term Helium Permeation Test History for Vent H-16, 10⁻² Atm ΔP

- The long-term helium permeation testing of the vents did not serve to differentiate between the several vent designs, since the operation of each of the vents was completely stable and satisfactory.

2.2.5 Metallographic Evaluation of As-Fabricated Vents (H-11, H-17, H-21, C-16, C-17, and C-22)

2.2.5.1 H-11 (helical, old thread design, FS-102 alumina)

A photomacrograph of the H-11 vent core and photomicrograph of a polished, longitudinal cross section of the assembled H-11 vent are shown in Figure 57. The H-10 vent tested at 2000°F (reference Section 2.2.4) and the H-12 vent being held in reserve were assembled in the same manner as H-11. Figure 57 graphically illustrates why the old thread design required modifications; small voids can be seen at the metal land/alumina-filled helix interface. Inspection of cores with the new thread design under a 30X stereo microscope showed no voids (Figure 58). The photomicrograph in Figure 57 shows that the high temperature gas pressure bonding technique effected a diffusion bond over a considerable area at the metal land/tubing interface. The bond appears to be at least as good as seen between the rod and tube elements of SRT-1 (Figure 18) which was shown to have a helium tight metal-to-metal bond.

2.2.5.2 H-17 (helical new thread design, FS-102 alumina)

Figure 59 shows a photomicrograph of a longitudinal cross section of H-17. A gap, ~0.0006-inch wide, can be seen between the tube and metal lands of the core. It is hypothesized that the gap was caused by either improper grinding of the core or a faulty end plug-to-tube seal which would negate the bonding effect of the high temperature isostatic pressing operation. The existence of the gap is consistent with the helium permeability data which show that H-17 flowed helium at a rate ~10 times greater than the two vents of similar design, H-16 and H-18 (Table 2).

H-11 After Final Grinding and Cleaning

H-11

Longitudinal Cross Section
of Assembled Vent

400X

Figure 57. Photomicrographs of H-11 Core and
a Photomicrograph of the Assembled
Helical Vent H-11(old thread)

Figure 58. H-16 Vent Showing New Thread Design (Note: no void regions at the metal land/alumina interfaces)

H-17

Original 50X
Polarized Light

H-21

Original 50X
Polarized Light

Figure 59. Photomicrographs of Cross-Sectioned Vents

2.2.5.3 H-21 (new 90° thread design, gold-plated TRW-100M platinized alumina)

Figure 59 also shows a photomicrograph of H-21. As was the case with H-17, no breakout of the alumina occurred at the corners of the new thread. The metal land-to-tube interfaces of H-21, in contrast to H-17, were well bonded.

2.2.5.4 H-22 (helical, yttria)

Figure 60 shows photomicrographs of H-22. The void area at the yttria-to-rod interface was due to metallographic pullout; this pullout was also responsible for most of the scratches in the Pt-10Rh components. The metal land-to-tube bond was poor.

2.2.5.5 C-16 (coaxial, FS-102 alumina)

Figure 61 shows a photomicrograph of C-16. An excellent mechanical bond was achieved between the ceramic-coated rod and the tube. The fairly large void areas in the alumina are a result of pullout during metallographic preparation and not a true representation of the void volume of the PAS alumina.

2.2.5.6 C-17 (coaxial, TRW-100 alumina)

Figure 61 also shows a photomicrograph of C-17. The rod and alumina are well interlocked to form an excellent mechanical bond. Although not shown in Figure 61, the tube-to-alumina bond was as good as shown for C-16.

2.2.5.7 C-22 (coaxial, TRW-100M platinized alumina)

Figure 61 also shows a photomicrograph of C-22. Again as in the case of C-16 and C-17, excellent mechanical bonds were achieved between the ceramic annulus and the Pt-10Rh rod and tube.

2.2.6 Visual and Metallographic Evaluation of Vents After Long Term, 2000°F, Testing

2.2.6.1 Vent H-16 (Helica, New Thread Design, FS102 Alumina, 1500 Hours of 2000°F Testing)

Vent H-16 was removed from the high temperature helium permeation test apparatus after ~1500 hours of operation at 2000°F and replaced with a yttria-containing vent (C-23). Vent H-16 is shown in Figure 62 prior to assembling the vent piece-parts. Note that there is no breakout of ceramic at the

H-22

ORIGINAL 100X

H-22

ORIGINAL 100X

Figure 60. Photomicrographs of Helical Yttria Vent H-22

Figure 61. Photomicrographs of Cross-Sectioned Vents

Figure 62. Photomacrographs of Vent H-16 Ready for Assembly and Photomicrographs of the Sectioned Vent after 1500 Hours of Testing

metal-to-ceramic interfaces with this new thread design. Figure 62 also shows a photomicrograph of a portion of the longitudinal cross section after 1500 hours of 2000°F testing. The voids visible in this photo in the ceramic were caused by pullout during metallographic preparation. The photomicrographs in Figure 62 reveal that the high temperature bonding technique effected a diffusion bond over a considerable area at the metal land-to-tubing interface.

2.2.6.2 Vent H-10 (Helical, Old Thread Design, FS102 Alumina, 25,020 Hours of 2000°F Testing, 3000°F Simulated Reentry Pulse)

Visual examination of noble metal vent H-10 revealed the presence of large grains on the tube housing surface, probably due to thermal etching as a result of the long exposure to a temperature of 2000°F (Figure 63).

2.2.6.3 Vent H-19 (Helical, New Thread Design, TRW-100 Alumina, 23,920 Hours of 2000°F Testing)

Visual examination and microstructural analyses were performed on noble metal vent H-19. As in the case of vent H-10, the visual examination showed that grains were partially visible on the tube housing surface, probably due to etching as a result of the thermal exposure (Figure 64). These grains were not as pronounced as those on vent H-10 due to the fact that vent H-10 was exposed to the higher temperature of the reentry pulse. Photomicrographs of the noble metal portions of the vent show that the tube had an ASTM grain size of 1 to 2 whereas the core had an ASTM grain size of 3 to 4. This difference in grain size between the tube and core may be due to different rates of grain growth which in turn may be due to the difference in the stored strain energy resulting from different processing of the tube and the rod.

Photomicrographs in the unetched condition reveal that partial diffusion bonding occurred at the tube/rod interface between the grooves. The unbonded areas appeared to be particles of alumina embedded in the rod which had not been removed during the final grinding operation prior to assembly of the vent. An excellent mechanical interface still exists between the alumina and the metal of the core and tube in the directions parallel to and normal to the vent centerline after the long-time elevated temperature exposure.

Figure 63. Noble Metal Vent H-10 (Helical, Old Thread Design, FS-102 Alumina) After 25,000 Hours of Permeation Testing at 2000°F Plus Exposure to a Simulated Reentry Pulse to 3000°F Maximum Temperature. Note the large grains visible on the tube surface.

Figure 64. Noble Metal Vent H-19 (Helical, New Thread Design, TRW 100 Alumina) After 24,000 Hours of Permeation Testing at 2000°F. Grains are Partially Visible on Tube Surface (Top Photograph).

This condition is equivalent to that observed previously in untested vent H-11 as described in paragraph 2.2.5.1.

2.2.6.4 Vent C-18 (Coaxial, TRW-100 Alumina, 23,920 Hours of 2000°F Testing, 3000°F Simulated Reentry Pulse)

Visual examination of noble metal vent C-18 showed no apparent changes in the vent or extension tubes from the as-fabricated condition (Figure 65). The absence of large grains on this tube housing surface, in comparison with the surface condition of vent H-10 which experienced essentially the same elevated temperature and reentry conditions, is not apparent.

2.2.6.5 Vent C-15 (Coaxial, FS102 Alumina, 23,920 Hours of 2000°F Testing)

Visual examination and microstructural analyses were performed on noble metal vent C-15. The visual examination again revealed the presence of large grains on the tube housing surface (Figure 66). As in the case of vent H-19, photomicrographs of the noble metal portions of the vent showed that the tube had an ASTM grain size of 1 to 2 whereas the ASTM grain size of the core was 3 to 4. These differences apparently did not affect the vent's performance.

Photomicrographs in the unetched condition (Figure 66) show that the excellent mechanical bond between the alumina and the noble metal core and tube housing still exists in both the direction parallel to and normal to the vent centerline after the long time elevated temperature exposure. This condition is equivalent to that observed previously in untested vents C-16, C-17, and C-22 as described in paragraphs 2.2.5.5, 2.2.5.6, and 2.2.5.7 and confirms the utility of isostatic pressing at elevated temperature. There appears to have been some consolidation of the alumina over the 24,000 hours of operation at 2000°F but the overall permeability of the alumina has remained basically unchanged as verified by the flow rate data.

2.2.6.6 Vent C-21 (Coaxial, TRW 100M Alumina, 23,920 Hours of 2000°F Testing)

A visual examination was performed on noble metal vent C-21. The visual examination revealed the presence of large grains on only a portion of the Pt extension tube and vent tube housing adjacent to the TIG weld on the vent upstream side. These grains were of about the same size as those observed on

Figure 65. Noble Metal Vent C-18 (Coaxial, TRW-100 Alumina) After 24,000 Hours of Permeation Testing at 2000°F Plus Exposure to a Simulated Reentry Pulse to 3000°F Maximum Temperature. Note the apparent absence of the large grains on the tube surface, distinctly visible on the tube surface of Vents H-10, H-19, and C-16.

Figure 66. Noble Metal Vent C-15 (Coaxial, FS-102 Alumina) After 24,000 Hours of Permeation Testing at 2000°F. Note the large grains visible on the tube surface (Top Photograph).

the tube housing surface of vent C-15 but were not as large as those observed on the tube housing of vent H-10.

2.2.6.7 C-23 (Coaxial, Yttria, 22,600 Hours of 2000°F Testing)

A visual examination was performed on noble metal vent C-23. Grain structure was not discernible on the tube housing surface for this vent.

2.2.7 Comparison of Permeability Model with Data for the Final Phase I Test Vents

The final series of Pt-20Rh/alumina vents was prepared in such a manner that the metal/ceramic seal was effected by an isostatic press at elevated (1800°F) temperatures. After pressing, all of the vents were heated to 2600°F for four hours to insure complete transformation to the alpha crystal form. Similar samples were prepared for the determination of porosity and surface area.

2.2.7.1 Comparison of Measured and Calculated Room Temperature Results

The room temperature specific permeabilities of each of the Phase I test vents were calculated using the average physical properties for each of the vent ceramics, and the measured permeability data and compared with the permeabilities predicted in equation 10. The coaxial vent results are shown in Table 16. The data for some of the earlier vents are included in the table for comparison.

The results indicate that the permeability of both the FS-102 and TRW-100 alumina is adequately characterized by assuming a tortuosity of three, corresponding to that which would be expected for a fully consolidated sample. While some variability appears (roughly covering a spread of a factor of two), this is probably the result of using average values for the surface area and the porosity rather than measuring them on the specific samples used.

The permeability of the platinized alumina samples (TRW-100M) appears to correlate best with a lower value of tortuosity (namely $q \approx 2.0$). This corresponds to a partially consolidated sample and suggests that the introduction of the platinum reduces the sintering together of the alumina particles.

The yttria samples yield much higher flows than would be expected even for an unconsolidated sample ($q \approx 1.4$). It is believed that this may be the result of micron size cracks developing in the brittle Y_2O_3 samples.

TABLE 16. COMPARISON OF MEASURED AND PREDICTED SPECIFIC PERMEABILITIES FOR COAXIAL VENTS

Sample No.	Sample Type	Sample Treatment	Assumed Porosity	Area ÷ Length cm x 10 ⁺²	Assumed Tortuosity	Assumed Mean Pore Size* Microns	Measured Flow Rate std cm ³ /sec x 10 ⁺⁶ ***	Measured Specific Permeability cm ² /sec x 10 ⁺¹	Calculated Specific Permeability cm ² /sec x 10 ⁺¹
6-2	Epoxy FS-102	As Sprayed	0.065**	6.22	3	0.4	4.0	1.0	1.47
C-3	FS-102	" "	0.15	2.2	3	0.4	6.4	1.45	1.47
C-4	FS-102	" "	0.15	1.64	3	0.4	4.5	1.37	1.47
6-1	Epoxy FS-102	A	0.13**	6.85	3	0.9	23.0	2.58	3.4
C-3	FS-102	A	0.20**	2.2	3	0.9	11.0	2.42	3.4
C-4	FS-102	A	0.20**	2.10	3	0.9	14.0	3.20	3.4
C-15	FS-102	B	0.18	0.836	3	1.1	6.5	4.31	4.15
C-16	FS-102	B	0.18	0.836	3	1.1	7.4	4.94	4.15
C-24	FS-102	B	0.18	0.836	3	1.1	3.6	2.40	4.15
6-3	Epoxy TRW-100	A	0.24**	3.43	3	0.8	29.0	3.57	3.5
C-17	TRW-100	B	0.20	0.836	3	1.5	9.9	5.8	5.6
C-19	TRW-100	B	0.20	0.83	3	1.5	11	6.6	5.6
C-20	TRW-100M	B	0.34	0.836	2	1.8	65	22.8	26.0
C-21	TRW-100M	B	0.34	0.836	2	1.8	81	28.4	26.0
C-22	TRW-100M	B	0.34	0.836	2	1.8	110	38.4	26.0
C-23	Y ₂ O ₃	B	0.06	0.836	3	0.9	7.8	15.5	1.7

A - Heated to 2200°F for four hours

B - Heated to 2600°F for four hours

*Based on surface area and porosity for typical samples

**Actual measured porosity

***At 10⁻² atmosphere

The measured and calculated specific permeability for the helical vents are compared in Table 17. In all cases, the measured specific permeability was considerably greater than that predicted by the permeability model. It is believed that higher effective porosities and pore sizes at the metal/ceramic interface in the area of the threads is the cause of the high permeation rates for the helical vents.

2.2.7.2 Comparison of Predicted and Experimental Helium Permeation Rates at Elevated Temperatures

Vent H-10, H-16, H-19, C-15, C-18, and C-21 were selected for elevated temperature testing. The data obtained are presented in Table 18 along with average ceramic properties for these vents.

As shown in Figure 67, the helium permeation rates through all of the coaxial vents and the helical vent H-19 varied inversely with the square root of the absolute temperature as predicted by the permeation model. The helical vents H-10 and H-16 varied less than predicted. It is suspected that differential expansion between the metal and the ceramic may have resulted in an increase of the flow area at the higher temperature. At the low permeation rates experienced by these vents, a very slight area change would be sufficient to affect the flow rates.

The permeation rates predicted by the model are compared with the measured data in Figure 68 for the coaxial vents. The average ceramic properties were used in the model. The results indicate that the model can satisfactorily predict the actual coaxial vent flow rates within the limit of the variability of the ceramic properties.

TABLE 18. EFFECT OF TEMPERATURE ON HELIUM PERMEATION RATE

Vent No.	Ceramic	Average Porosity	Average Pore Size	Tortuosity	Helium Permeation Rate std cc/sec at 10^{-2} Atm ΔP ($\times 10^{-6}$)			
					70°F	1000°F	2000°F	2200°F
H-10	FS-102	0.18	1.1	3	3.2	2.5	2.3	2.2
H-16	FS-102	0.18	1.1	3	3.1	2.3	2.2	2.1
H-19	TRW-100	0.2	1.5	3	20	13	11	8.3
C-15	FS-102	0.18	1.1	3	6.5	4.3	3.6	2.9
C-18	TRW-100	0.2	1.5	3	23	14	11	9.9
C-21	TRW-100M	0.34	1.8	2	81	50	40	36

TABLE 17. COMPARISON OF MEASURED AND PREDICTED PERMEABILITIES FOR HELICAL VENTS

Sample No.	Type*	Area ÷ Length cm	Measured Flow Rate at 10^{-2} Atm std cm ³ /sec x 10^{-6}	Assumed Ceramic Properties			Measured Specific Permeability cm ² /sec x 10^2	Predicted Specific Permeability cm ² /sec x 10^2
				Porosity	Mean Pore Size Microns	Tortuosity		
H-10	FS-102 (old design)	5.4×10^{-4}	3.2	0.18	1.1	3	3.2	0.41
H-11	FS-102 (old design)	5.4×10^{-4}	4.1	0.18	1.1	3	4.1	0.41
H-12	FS-102 (old design)	5.4×10^{-4}	3.6	0.18	1.1	3	3.6	0.41
H-16	FS-102 (new design)	5.4×10^{-4}	3.1	0.18	1.1	3	3.1	0.41
H-18	FS-102 (new design)	5.4×10^{-4}	1.4	0.18	1.1	3	1.4	0.41
H-23	FS-102 (new design)	5.4×10^{-4}	2.5	0.18	1.1	3	2.5	0.41
H-19	TRW-100 ** (new design)	5.4×10^{-4}	20	0.20	1.5	3	18.5	0.62
H-20	TRW-100 ** (new design)	5.4×10^{-4}	13	0.20	1.5	3	12.0	0.62
H-21	TRW-100 ** (new design)	5.4×10^{-4}	8.6	0.20	1.5	3	7.95	0.62
H-22	Yttria (new design)	5.4×10^{-4}	17	0.06	0.9	3	52.5	0.11

*All samples heated to 2600°F for four hours prior to test

** Gold plated alumina

TRW(A)-11069-0067

Figure 67. Effect of Vent Temperature on Permeation Rate

Figure 68. Comparison of 2000°F Measured Permeation Rates with Rates Predicted by the Permeation Model for Three Coaxial Vents

2.3 HIGH TEMPERATURE VACUUM STABILITY OF CANDIDATE COATING-SUBSTRATE SYSTEMS

The objective of this series of tests was to determine as a function of temperature and time the extent to which candidate ceramic vent core materials were chemically and physically stable in a elevated temperature high vacuum space environment. Four candidate ceramic materials were evaluated as plasma arc sprayed filters on Pt-10Rh substrates. The coated specimens were exposed to a matrix of temperature-time soaks in vacuum and post-test analyses including visual and metallographic examination, X-ray diffraction, surface area and porosity measurements, weight changes, and scanning electron microscopic examination were performed.

2.3.1 Test Specimens and Testing Conditions

Coatings of FS-102 alumina, TRW-100 alumina, platinized alumina, and yttria, nominally 0.005-inch thick were applied to 0.110-inch diameter, one half inch long Pt-10Rh rods and 0.190-inch diameter, 0.020-inch wall, 1-inch long Pt-10Rh tubes for vacuum stability tests. The coated tubes were used for pore volume and surface area measurements. The coated rods which are the same diameter as a fabricated vent were used for determining the mechanical and chemical stability of the coating-substrate system. One rod and one tube specimen coated with each ceramic material was exposed to each of the following conditions:

<u>Temperature</u>	<u>Time</u>	<u>Vacuum</u>
2600°F	3 Days	10^{-6} Torr
2600°F	30 Days	10^{-6} Torr
2200°F	18 Days	10^{-6} Torr

The specimens were contained in a high purity alumina crucible. The following are results of post-test analyses:

2.3.2.1 X-ray Diffraction Analyses

X-ray diffraction analyses were performed on the candidate coating materials in the "as plasma sprayed" condition and after high temperature vacuum exposure. Diffraction was performed using the Debye-Scherrer method with Ni-filtered copper radiation. Results of analyses are summarized in Table 19. In the "as coated" condition, FS-102, TRW Al_2O_3 , and platinized

TABLE 19. X-RAY DIFFRACTION OF VACUUM STABILITY SPECIMENS

Condition	Structure of Coating Materials			
	FS-102	TRW-100 Al_2O_3	TRW-100M Platinized Al_2O_3	Y_2O_3
As Coated	$\alpha + \gamma$	$\alpha + \gamma$	$\alpha + \gamma$	BCC
2600°F 3 Days	$\alpha +$ some retained γ	All α	$\alpha +$ some retained γ	BCC (no change)
2600°F 30 Days	All α	All α	All α	BCC (no change)
2200°F 18 Days	All α	All α	All α	BCC (no change)

Al_2O_3 contained both α and γ Al_2O_3 phases in appreciable quantities whereas yttria showed a single phase BCC Y_2O_3 structure. Vacuum exposure at any of the three temperature-time conditions effected a conversion of γ - Al_2O_3 to α - Al_2O_3 . Some γ phase was retained in the FS-102 and platinized Al_2O_3 specimens after three days at 2600°F but not in the TRW-100 Al_2O_3 specimen under the same exposure condition. Complete conversion to α - Al_2O_3 was achieved after eighteen days at 2200°F and thirty days at 2600°F for all alumina base materials. The yttria material remained unchanged after exposure to any of the three test conditions.

2.3.2.2 Metallographic and SEM Analysis

Visual examination of all coated specimens after thermal exposure in vacuum revealed no major change in appearance attributable to exposure environments. Adherence of coatings to substrates was generally good. A slight pink discoloration which was observed in the FS-102 and TRW-100 alumina specimens was most likely caused by minute impurities from the furnace used for the 2600°F three-day test. No discoloration was shown in the 2600°F thirty-day test specimens. Hairline cracks were found in one FS-102 specimen and a piece of coating was chipped off mechanically from another FS-102 specimen after the 2600°F thirty-day exposure. These defects were not found in the 2600°F three-day test specimens. Therefore, it is believed that a nonuniform heating or cooling cycle was probably the cause.

Although the yttria coated specimens showed excellent adherence in the 2600°F three-day and 2600°F thirty-day tests, cracking and spalling was noticed in the yttria coated tube specimens after the 2200°F eighteen-day test. Minute cracks were also found in the yttria coated rod specimens from the same test, but the coating remained intact on the substrate.

Conventional metallographic examination was performed on cross-sections of the coated specimens after exposure. There was no evidence of coating-substrate interaction in any of the materials evaluated for any of the test conditions used. A typical microstructure of a cross-section is shown in Figure 69 for the FS-102 Al_2O_3 coated Pt-10Rh tube specimen.

(a) 100X

(b) 400X

Figure 69. Cross Section of FS-102 Al₂O₃ Coated Pt-10Rh After 30-Day 2600°F Vacuum Exposure

Attempts to examine the coating-substrate interface by the scanning electron microscopic (SEM) technique were made but the results were less satisfactory than those obtained by conventional metallography using polished specimens. For this reason, the SEM method was not used for this purpose. However, the SEM techniques were used extensively for examining the structures of the ceramic coatings. SEM photographs at 1000X and 3000X of the coated specimens "as sprayed" and after each thermal vacuum exposure were taken and are described below.

Figures 70 through 73 show the surface of TRW-100 alumina coating in the as-sprayed condition and after three different thermal exposures. The structure in the as-sprayed condition, Figure 70, appears to be made up of combinations of particles and platelets of various sizes. Figure 71 shows the material after 2600°F three-day vacuum exposure. Spheroidization has occurred to some extent and the surface of some of the particles has taken on a "cauliflower" appearance. The latter condition was probably a result of crystallographic changes manifested by substructure formation. Some microcracks have developed in the structure, probably due to stresses induced by the volumetric changes resulting from the γ - α phase transformation of the alumina.

After thirty days at 2600°F, the degree of spheroidization seems greater and the substructure appears to have grown in size relative to the three-day exposure (Figure 72).

Figure 73 shows the surface of TRW-100 alumina after 2200°F eighteen-day exposure. Some spheroidization and micro-cracking has also occurred but the "cauliflower" substructure was notably absent.

Figures 74 through 77 present photographs of the surface structure of FS-102 alumina in the "as sprayed" condition and after the three different thermal exposures. Figure 74 shows the "as sprayed" material with particles stacked in about the same manner as the TRW-100 alumina material. The surface substructure which appeared in the TRW-100 material also appeared in the FS-102 material after exposure to 2600°F for three days and thirty days in vacuum (Figures 75 and 76). The "cauliflower" substructure was also evident in the FS-102 coating (Figure 77) after eighteen-day 2200°F exposure. The significance and effect of the substructure formation on the coating-substrate system cannot be readily assessed at this time.

1000X Magnification

3000X Magnification

Figure 70. Photograph of Surface of TRW-100 Alumina in "As-Sprayed" Condition

BLANK

1000X Magnification

3000X Magnification

Figure 72. SEM Photo of Surface of TRW-100 Alumina After Vacuum Exposure at 2600°F for Thirty Days

1000X Magnification

3000X Magnification

Figure 73. SEM Photograph of Surface of TRW-100 Alumina After Exposure to 2200°F for 18 Days

1000X Magnification

3000X Magnification

Figure 74. SEM Photograph of Surface of FS-102 Alumina in "As-Sprayed" Condition

1000X Magnification

3000X Magnification

Figure 75. SEM Photograph of Surface of FS-102 Alumina
After Vacuum Exposure to 2600°F for Three Days

1000X Magnification

3000X Magnification

Figure 76. SEM Photograph of Surface of FS-102 Alumina After Vacuum Exposure at 2600°F for Thirty Days

1000X Magnification

3000X Magnification

Figure 77. SEM Photograph of Surface by FS-102 Alumina After Vacuum Exposure at 2200°F for Eighteen Days

Figures 78 through 81 show the structure of the platinized alumina in the as-sprayed condition (Figure 78) and after thermal exposure (Figures 79, 80, and 81). No major difference in structural features can be found between the platinized alumina coating and the FS-102 alumina coating for the same test conditions. Some micro-voids were produced from the 2600°F thirty-day and 2200°F eighteen-day exposure (Figures 80 and 81). However, void formation was not evident in the 2600°F three-day specimens.

Figures 82 through 85 show SEM photographs of the yttria coated surfaces. Finer particles and more fusion of small particles are shown in the "as-sprayed" yttria coating, Figure 82, than the alumina coatings. Appreciable porosity within individual particles was developed as a result of high temperature exposure, Figures 83 through 85. The large number of pores shown in the 2600°F three-day test specimen, Figure 83, seems to diminish after thirty days at 2600°F. Apparently, longer sintering time reduced the porosity level considerably. Porosity also existed to some extent after the 2200°F eighteen-day test (Figure 85).

2.3.2.3 Dimensional and Weight Changes

All coated tube and rod specimens were weighed and dimensionally inspected before and after each thermal vacuum exposure. Results are tabulated in Tables 20, 21, and 22 for each of the three temperature-time conditions. Weight losses were insignificantly low (less than 1%) in all cases. Dimensional changes were also negligible although there is a slight volumetric change associated with phase transformation in the alumina materials.

2.3.2.4 Surface Area, Pore Volume, and Porosity Measurements

Total surface area measurements were made on specimens consisting of porous coatings of Fansteel FS-102 alumina, TRW-100 alumina, platinized alumina, and yttria plasma-arc-sprayed on 0.190-inch O.D. Pt-10Rh tubes. Measurements were made by the BET gas adsorption technique using krypton gas at liquid nitrogen temperatures. Surface area determinations were obtained on both "as-sprayed" specimens, and on samples which were exposed to thermal environments of one hour at 2000°F, eighteen days at 2200°F, three days at 2600°F, and thirty days at 2600°F in vacuum. All surface

1000X Magnification

3000X Magnification

Figure 78. SEM Photograph of Platinized Alumina
in "As-Sprayed" Condition

1000X Magnification

3000X Magnification

Figure 79. SEM Photograph of Platinized Alumina After Vacuum Exposure at 2600°F for Three Days

1000X Magnification

3000X Magnification

Figure 80. SEM Photograph of Platinized Alumina After Vacuum Exposure at 2600°F for Thirty Days

1000X Magnification

3000X Magnification

Figure 81. SEM Photograph of Platinized Alumina After Vacuum Exposure at 2200°F for Eighteen Days

1000X Magnification

3000X Magnification

Figure 82. SEM Photograph of Yttria in "As-Sprayed" Condition

1000X Magnification

3000X Magnification

Figure 83. SEM Photograph of Yttria After
Vacuum Exposure at 2600°F for Three Days

1000X Magnification

3000X Magnification

Figure 84. SEM Photograph of Yttria After Vacuum Exposure at 2600°F for Thirty Days

1000X Magnification

3000X Magnification

Figure 85. SEM Photograph of Yttria After Vacuum Exposure at 2200°F for Eighteen Days

TABLE 20. WEIGHT AND DIMENSIONAL CHANGES FROM EXPOSURE TO 2600°F FOR THREE DAYS

Specimen No.	Type	Coating	Initial Weight Grams	Final Weight Grams	Weight Change Grams	Initial Diameter Inches	Change in Diameter Inches
40	(tube)	FS-102 Al_2O_3	1.7663	1.7687	+0.0025	0.2070	**
47	(tube)	TRW Al_2O_3	1.6926	1.6947	+0.0021	0.2035	**
54	(tube)	Platinized Al_2O_3	1.7075	1.7090	+0.0015	0.2024	**
61	(tube)	Yttria	1.7432	1.7458	+0.0026	0.1990	**
72	(rod)	FS-102 Al_2O_3	1.5904	1.5914	+0.0010	0.1208	-0.0006
79	(rod)	TRW Al_2O_3	1.6086	1.6092	+0.0010	0.1257	-0.0002
86	(rod)	Platinized Al_2O_3	1.5868	1.5870	+0.0002	0.1216	-0.0008
93	(rod)	Yttria	1.6476	1.6406	-0.0070*	0.1257	-0.0001

* Small piece of coating chipped off

** Final diameter of tube unsymmetrical for measurements

TABLE 21. WEIGHT AND DIMENSIONAL CHANGES FROM EXPOSURE TO 2600°F FOR THIRTY DAYS

Sample No.	Type	Coating	Initial Weight Grams	Weight Change Grams	Initial Diameter Inches	Change in Diameter Inches
62	Tube	Yttria	1.7850	-0.0028	0.2002	0
41 [*]	Tube	FS-102 Al ₂ O ₃	1.8709	-0.0006	0.2093	-0.0012
48	Tube	TRW-Al ₂ O ₃	1.8157	-0.0010	0.2053	-0.0009
55	Tube	Pt-Al ₂ O ₃	1.7207	-0.0006	0.2018	0
94	Rod	Yttria	1.6097	-0.0019	0.1219	-0.0002
73 ^{**}	Rod	FS-102 Al ₂ O ₃	1.5955	+0.0007	0.1219	-0.0002
80	Rod	TRW-Al ₂ O ₃	1.5881	+0.0011	0.1207	-0.0002
87	Rod	Pt-Al ₂ O ₃	1.5883	+0.0005	0.1218	0

* A small piece of coating chipped off

** Coating has a hairline crack.

TABLE 22. WEIGHT AND DIMENSIONAL CHANGES FROM EXPOSURE TO 2200°F IN VACUUM FOR 18 DAYS

Specimen No.	Type	Coating	Initial Weight Grams	Final Weight Grams	Weight Change Grams	Initial Diameter Inches	Change In Diameter Inches
42	Tube	FS-102 Al_2O_3	1.8285	1.8269	-0.0016	0.2090	*
49	Tube	TRW Al_2O_3	1.7695	1.7674	-0.0021	0.2056	*
56	Tube	Platinized Al_2O_3	1.7813	1.7792	-0.0021	0.2033	*
63	Tube	Yttria	1.7835	1.7747	-0.0098	0.2002	*
74	Rod	FS-102 Al_2O_3	1.6149	1.6149	0	0.1265	-0.0005
81	Rod	TRW Al_2O_3	1.6168	1.6165	-0.0003	0.1277	-0.0007
88	Rod	Platinized Al_2O_3	1.5822	1.5816	-0.0006	0.1203	-0.0003
95	Rod	Yttria	1.6510	1.6479	-0.0031	0.1273	-0.0002

* Final diameter of tube unsymmetrical for measurement

area measurements planned for Phase I of the program are now complete and the data are summarized in Table 23.

The specific surface area (cm^2/gram) of all of the specimens decreased upon thermal exposure at 2000°F or above. It appears that the specific surface area of the PAS FS-102 alumina coatings and the platinized alumina coatings essentially stabilized after only one hour of thermal exposure at 2000°F. That is to say, the surface area changed only slightly after experiments in which FS-102 samples and the platinized alumina samples were exposed to thermal environments of one hour at 2000°F, eighteen days at 2200°F, three days at 2600°F, and thirty days at 2600°F.

Pore size distribution and total pore volume measurements were performed with samples of each of the four candidate coatings in the "as-sprayed" condition and after each of the three thermal exposures. The measurements were performed by Sandia Corporation using an Aminco Mercury Intrusion Porisometer with a maximum pressure capability of 5,000 psi. Table 24 summarizes the results. The mercury penetration results on all of the samples showed a two-peak type of pore size distribution. There was an initial peak at very low pressures (<10 psig) corresponding to pores greater than twenty microns and a second peak at much higher pressures which generally corresponded to pores in the 1.0 to 0.1 micron region. From the scanning electron microscope photographs, it is doubtful whether the pores larger than twenty microns are really representative of the interior structure of the coatings. It seems more likely that this is a surface roughness effect or is due to shallow surface porosity and not to pores which penetrate deeply into the coating. In Table 24 total pore volume is presented both on the basis of mercury intrusion measurements for all pores and on the basis of mercury intrusion measurements for pores less than twenty microns diameter. Similarly, porosity was determined both on the basis of all pores and on the basis of pores less than 20μ . In general, (but not in all cases) the measured porosity based on pores less than twenty microns diameter agreed better with porosities calculated from sample geometry and material density than did the measured porosities based on all pores. The last two columns in Table 21 present the mean pore size of the samples as estimated from the mercury porisometer measurements and as calculated from surface area and porosity measurements, respectively. There is order of magnitude agreement between

TABLE 23. SUMMARY OF SURFACE AREA MEASUREMENTS ON COATED TUBES

Specimen No.	Sample Type	Environmental Exposure	Surface Area Per Gram of Coating cm ² /Gram	Surface Area Per Initial Unit Volume of Coating cm ² /cm ³
46	FS-102 Al ₂ O ₃	As-Fabricated	5,070	15,300
46	"	1 Hour at 2000°F	2,530	7,650
42	"	18 Days at 2200°F	2,980	8,200
40	"	3 Days at 2600°F	2,320	6,250
41	"	30 Days at 2600°F	1,323	3,833
53	TRW-100 Al ₂ O ₃	As-Fabricated	21,800	58,000
53	"	1 Hour at 2000°F	11,200	29,200
49	"	18 Days at 2200°F	3,630	9,650
47	"	3 Days at 2600°F	2,310	5,380
48	"	30 Days at 2600°F	1,534	4,323
59	Platinized Al ₂ O ₃	As-Fabricated	8,100	22,700
59	"	1 Hour at 2000°F	3,640	10,200
56	"	18 Days at 2200°F	4,430	12,200
54	"	3 Days at 2600°F	3,880	5,200
55	"	30 Days at 2600°F	4,710	7,056
67	Yttria	As-Fabricated	9,430	35,500
67	"	1 Hour at 2000°F	4,460	16,800
63	"	18 Days at 2200°F	930	3,630
61	"	3 Days at 2600°F	1,960	8,710
62	"	30 Days at 2600°F	854	3,468

TABLE 24. SUMMARY OF PORE VOLUME , MEAN PORE SIZE AND POROSITY MEASUREMENTS

Sample No.	Sample Type	Thermal Exposure	Coating Volume cm ³	Total Pore Volume cm ³		Porosity - %			Mean Pore Size μ	Mean Pore Size μ
				Hg Intrusion All Pores	Pores Less Than 20 μ	Calculated from Sample Geometry	Hg Intrusion All Pores	Hg Intrusion Pores <20 μ	Estimated From Hg Penetration Data for Pores <20 μ	Calculated from Surface Area and Porosity of Sample for Pores <20 μ
75	FS-102 Al ₂ O ₃	"As-Fabricated"	0.0175	0.0040	0.0026	13%	23%	15%	0.15	0.40
74	FS-102 Al ₂ O ₃	18 Days 2200°F	0.0203	0.0089	0.0037	19%	44%	18%	0.45	0.88
72	FS-102 Al ₂ O ₃	3 Days 2600°F	0.0122	0.0071	0.0022	11%	58%	18%	0.30	1.10
73	FS-102 Al ₂ O ₃	30 Days 2600°F	0.0174	0.0052	0.0037	-	30%	21%	0.40	2.20
83	TRW Al ₂ O ₃	"As-Fabricated"	0.0198	0.0038	0.0031	21%	19%	16%	0.25	0.083
81	TRW Al ₂ O ₃	18 Days 2200°F	0.0229	0.0050	0.0044	25%	22%	19%	0.60	0.79
79	TRW Al ₂ O ₃	3 Days 2600°F	0.0198	0.0053	0.0041	19%	27%	20%	0.70	1.50
80	TRW Al ₂ O ₃	30 Days 2600°F	0.0157	0.0066	0.0026	-	42%	17%	0.50	1.50
96	Yttria	"As-Fabricated"	0.0131	0.00285	0.00185	9.2%	22%	14%	0.06	0.59
95	Yttria	18 Days 2200°F	0.0221	0.00300	0.00190	-	13%	8.6%	0.10	0.95
93	Yttria	3 Days 2600°F	0.0198	0.00640	0.001	-	32%	5.1%	-	0.23
94	Yttria	30 Days 2600°F	0.0175	0.00240	0.0008	-	14%	4.6%	0.10	0.53
90	Platinized Al ₂ O ₃	"As-Fabricated"	0.0134	0.0036	0.0028	31%	28%	21%	0.40	0.36
88	Platinized Al ₂ O ₃	18 Days 2200°F	0.0113	0.0038	0.0026	34%	34%	23%	0.50	1.45
86	Platinized Al ₂ O ₃	3 Days 2600°F	0.0123	0.0042	0.0030	-	34%	24%	0.60	1.77
87	Platinized Al ₂ O ₃	30 Days 2600°F	0.0174	-	-	-	-	-	-	-

1 2 4

the two methods. After heating at 2000°F or above, the three alumina ceramics had mean pore sizes of the order of one micron.

2.4 STABILITY OF VENT SYSTEMS UNDER REENTRY HEATING

2.4.1 Stability Testing of Candidate Vent Materials Under Reentry Heating Conditions

Coated Pt-10Rh specimens were subjected to a thermal cycle to determine the ability of these materials to withstand capsule reentry heating. Results of weight loss measurement and visual examination indicated no apparent coating degradation or chemical interaction between the coatings and the substrate.

A total of five specimens (nominally 3-inches x 1/4-inch strips) were tested:

- Uncoated Pt-10Rh strip
- FS-102 alumina coated Pt-10Rh strip
- TRW-100 alumina coated Pt-10Rh strip
- Platinized alumina coated Pt-10Rh strip
- Yttria coated Pt-10Rh strip

Nominal thickness of the coatings was 0.006-inch coated on both sides and the edges of the specimens. A 1/4-inch of the coated area was removed from both ends of the strips for electrical contact. A small spot at the center of the strips was also abraded to clean metal for thermocouple attachment. The coating materials removed from these specimens were retained for X-ray diffraction analysis for comparison with samples taken after the reentry heating tests.

Tests were conducted in a high temperature ribbon furnace (Figure 86). In the chamber, the test specimen was clamped between water cooled electrodes and resistively heated in a one psia air atmosphere. The reentry temperature profile (Figure 87) was manually controlled by rheostat adjustment. Excellent match of the heating profile was achieved. During the test, a small pressure increase of 0.016 psia was noted.

Post-test analyses consisted of weight loss, visual, microscopic examination and electron microprobe analyses. Weight loss of all specimens was less than 0.01%. Visual examination showed no cracking or spalling of the coatings. The uncoated Pt-10Rh, FS-102 coated, and TRW-100 alumina coated

Figure 86. Ribbon Furnace

-154-

Figure 87. Reentry Temperature Profile

TRW(A)-11069-0067

specimens remained essentially unchanged. Platinized alumina lightened in color during the test. The yttria specimen was slightly darkened in the high temperature zone of the specimen (Figures 88 and 89). Microscopic examination of the surface of these specimens as compared with untested specimens, revealed no evidence of structural changes.

Metallographic examination was made on a cross-section from the center of each specimen where the temperature profile was measured. No coating-substrate interaction was observed in any of the coated specimens typically shown in Figures 90 and 91. The rough edges were caused by grit blasting prior to plasma spraying. Even the discolored yttria specimen indicated no reaction.

The conclusion of no interaction was further substantiated by electron microprobe analysis. Beam traverses were made across the coating-substrate interface for depth of penetration of diffusing elements. The concentration gradient curves were so sharp that, for all practical purposes, diffusion was undetectable.

X-ray diffraction analyses were performed on the ceramic coating materials before and after the reentry heating test. The Debye-Scherrer powder camera method was employed using nickel filtered copper radiation. The d-spacing data obtained were compared with the ASTM card file. Results are tabulated in Table 25. All alumina materials, TRW-100, FS-102, and platinized alumina contained both α - Al_2O_3 and γ - Al_2O_3 in the as-coated condition. As the result of reentry heating up to 2750°F (maximum), partial transformation of the γ -phase to α -phase occurred. This was evident by the disappearance of the (111) γ - Al_2O_3 line and weakening of the (222) γ - Al_2O_3 and (400) γ - Al_2O_3 line intensity in the reentry heated specimens. Transformation, however, was incomplete due possibly to insufficient time and nonequilibrium conditions prevailing in the reentry heating-cooling cycle. It was, therefore, concluded that although no chemical interaction took place, reentry heating tends to promote enrichment of α - Al_2O_3 phase from the as-sprayed α - γ duplex structure.

Figure 88. Untested Yttria Coated Pt-10Rh Specimen

Figure 89. Reentry Heated Yttria Coated Pt-10Rh Specimen

Figure 90. Cross Section of TRW-100 Alumina Coated Specimen After Reentry Heating

Figure 91. Cross Section of Yttria Coated Specimen After Reentry Heating

TABLE 25. SUMMARY OF X-RAY DIFFRACTION RESULTS ON REENTRY HEATED SPECIMENS

Coating Material	Crystal Structure*	
	As Coated	After Heating
TRW-100 Alumina	α and γ Phases	Mainly α ; Some Retained γ
FS-102 Alumina	α and γ Phases	Mainly α ; Some Retained γ
Platinized Alumina	α and γ Phases	Mainly α ; Some Retained γ
Yttria	Y_2O_3	Y_2O_3

* α phase refers to $\alpha-Al_2O_3$ matching ASTM 10-173; γ phase refers to $\gamma-Al_2O_3$ matching ASTM 10-425; Y_2O_3 matching ASTM 5-0574

2.4.2 Stability Testing of Vents C-18 and H-10 Under Reentry Heating Conditions

After long-term 2000°F, helium permeation testing, two vents (C-18 and H-10) were subjected to a thermal cycle (3000°F peak temperature) in air to determine the ability of these assemblies to withstand simulated capsule reentry heating.

The tests were conducted utilizing an R.F. Induction (Lepel) system. The two vent assemblies were positioned within the induction coil, and the reentry temperature profile (Figure 92) was manually controlled by rheostat adjustment. After reentry heating, the vent assemblies were replaced in the helium permeation test assembly and tested at the temperatures and pressure differentials noted in Tables 26 and 27. The helium permeation data indicates that the helium flow rate of the vents remained stable even after being subjected to the rather severe reentry environment.

Figure 92. Reentry Temperature Profile for Testing Vents C-18 and H-10

TABLE 26. HELIUM PERMEATION RATE OF VENT C-18 BEFORE AND AFTER REENTRY HEATING TEST

<u>Temperature (°F)</u>	<u>ΔP (Atmos.)</u>	<u>Helium Permeation Rate std cc/sec</u>	<u>Remarks</u>
70	10^{-3}	1.6×10^{-6}	Before Reentry
70	10^{-3}	1.7×10^{-6}	After Reentry
70	10^{-2}	1.3×10^{-5}	Before Reentry
70	10^{-2}	1.3×10^{-5}	After Reentry
2000	10^{-3}	1.1×10^{-6}	Before Reentry
2000	10^{-3}	1.4×10^{-6}	After Reentry
2000	10^{-2}	7.8×10^{-6}	Before Reentry
2000	10^{-2}	9.7×10^{-6}	After Reentry

TABLE 27. HELIUM PERMEATION RATE OF VENT H-10 BEFORE AND AFTER REENTRY HEATING TEST

<u>Temperature (°F)</u>	<u>ΔP (Atmos.)</u>	<u>Helium Permeation Rate std cc/sec</u>	<u>Remarks</u>
70	10^{-3}	4.2×10^{-7}	Before Reentry
70	10^{-3}	4.6×10^{-7}	After Reentry
70	10^{-2}	3.5×10^{-6}	Before Reentry
70	10^{-2}	3.8×10^{-6}	After Reentry
2000	10^{-3}	3.6×10^{-7}	Before Reentry
2000	10^{-3}	4.2×10^{-7}	After Reentry
2000	10^{-2}	2.9×10^{-6}	Before Reentry
2000	10^{-2}	2.9×10^{-6}	After Reentry

3. REFERENCES

1. Adzumi, H., Bull Chem. Soc., Japan 12 (1937).
2. Carman, P. C., and Arnell, J. C., Canadian J. Research, 26A (1948) 128.
3. Knudsen, M., Ann. Phys., Lpz, 28 (1909) 75.
4. von Smoluchowski, Ann, Phys., Lpz, 35 (1911) 983.
5. Millikan, R., Phys. Rev. 21 (1923) 217.
6. Carman, P. C. and Malherbe, P., J. Soc. Chem. Ind., London, (Trans.) 69 (1950) 134.
7. Clausing, P., Physica 9 (1929) 65.
8. Wyllie, M. R. J. and Rose, W. D., Trans. Amer. Inst. Mining Engrs. 189 (1950) 105.
9. Carman, P. C., Flow of Gases Through Porous Media, Butterworth's Scientific Publication, 1956.
10. Kozeny, J., S. B. Akad., Wiss. Wien, Abt IIa 136 (1927) 271.
11. Thompson, V. S. and Whitlemore, O. J., Ceramic Bull., 47 (1968) 637.