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IRRADIATION STABILITY
OF
SCB-1 COATED HASTELLOY N SNAP 8 FUEL CLADDING
EXPERIMENTS NAA 77-2 AND NAA 77-3
(Title Unclassified)

AEC Research and Development Report

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IRRADIATION STABILITY
OF
SCB-1 COATED HASTELLOY N SNAP 8 FUEL CLADDING
EXPERIMENTS NAA 77-2 AND NAA 77-3
(Title Unclassified)

By

C. J. ENGBERG
K. J. MILLER
R. C. COURTRIGHT

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By their very nature, in-pile irradiation experiments are extremely complicated, and therefore require the talents of a number of specially trained and competent individuals. This experiment was no exception. The authors are indebted to a large number of people who worked on this program. Space does not permit the acknowledgment of each individual's part in the experiment, but the contribution of four individuals must be mentioned. These people are:

M. E. Nathan	Initial concept and computer codes for data analysis
A. C. Crawford	Capsule design, assembly, and ETR installation
B. J. Thomas	Capsule irradiation and in-pile permeation measurements
G. Ervin III	Data reduction and analysis

Without the assistance of these people, this irradiation program would not have succeeded.

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ABSTRACT

NAA 77-2 and NAA-77-3 were the second and third irradiation experiments designed to determine the radiation stability of SCB-1 hydrogen barrier material. Each capsule contained a single SCB-1 coated SNAP 8 fuel cladding assembly, designed to determine both neutron and fission fragment bombardment irradiation effects. The capsules were irradiated in the Engineering Test Reactor from March 1964 to February 1965, during which time the capsules received a calculated fast neutron dosage of 1.5×10^{21} nvt ($E_n > 1$ Mev), a thermal dosage of 2.5×10^{21} nvt, and a fission fragment bombardment of 1.3×10^{18} fission fragments/element inch. The in-pile hydrogen permeation measurements revealed that the ionizing radiation field in the reactor did not significantly alter the hydrogen permeation process in SNAP 8 fuel cladding assemblies. Radiation does appear to accelerate the devitrification process in the hydrogen barrier material. The permeation rates during the final day of irradiation were 2.8×10^{-3} cc/hr-cm² for NAA 77-2, and 2.7×10^{-3} cc/hr-cm² for NAA 77-3, at 1500°F and 1 atm hydrogen pressure. These rates may be compared with pre-irradiation permeation rates of 1.0×10^{-3} and 0.88×10^{-3} cc/hr-cm² at 1500°F and 1 atm hydrogen pressure for 77-2 and -3, respectively.

The capsules operated for 4638 hr at temperatures of 1300 and 1500°F for 77-2 and -3, respectively. The internal hydrogen pressure was maintained at 3 atm absolute, which resulted in a hoop stress in the cladding of ~ 1200 psi. Measurements showed the diameters of the Hastelloy N cladding tubes had increased 0.08 and 0.37% for 77-2 and -3, respectively. An additional 0.07% increase was measured at the thermocouple strapping bands on each tube. The higher temperature under the thermocouple bands must have caused this higher creep rate. The average creep rates for 77-2 and 3 were determined, from pre-irradiation and postirradiation measurements, to be 3.5×10^{-7} and 1.0×10^{-6} in./in.-hr, respectively.

The Hastelloy N cladding material experienced grain growth, similar to that for unirradiated samples annealed under similar conditions. On thermal annealing of both the irradiated and unirradiated material, the Hastelloy N grain boundaries became decorated with precipitated particles. The weld zone showed precipitates within the grains, on both irradiated and unirradiated samples.

The NAA 77 series of experiments showed that the SCB-1 ceramic material would meet or exceed the specifications required for the operation of the SNAP 8 hydrogen barrier under normal reactor operating conditions.

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I. INTRODUCTION

The NAA 77 Irradiation Experiment was conceived as the second phase of a study to evaluate the effect of reactor irradiation on the hydrogen barrier materials used in the SNAP reactors. The first phase, Irradiation Experiment NAA 68, undertook to determine the irradiation stability of the hydrogen barrier material, AI8763D, by comparing pre- and post-irradiation hydrogen permeation rates.⁽¹⁾ The second phase of the study was designed to determine the irradiation stability of an advanced hydrogen barrier material, SCB-1, by measuring its hydrogen permeation rate during reactor operation. The advantages of the in-pile permeation rate study were:

- 1) The effect of the ionizing radiation field on the permeation process could be determined while under irradiation.
- 2) The rate of radiation damage to the hydrogen barrier material, if any, could be measured.

The study included four separate capsule irradiations. This report covers the irradiation and examination of Experiments NAA 77-2 and NAA 77-3.

The objectives of Experiment NAA 77-1 were to determine the feasibility of performing in-pile hydrogen permeation rate measurements, and to evaluate the neutron irradiation behavior of the hydrogen barrier material. Experiments NAA 77-2, -3, and -4 had the design objectives of determining the combined effects of fission fragment and neutron radiation damage on the hydrogen barrier materials at temperatures of 1300, 1500, and 1700°F, respectively.

The NAA 77-1 experiment was irradiated for 4343 hr in the Engineering Test Reactor (ETR), from August 1963 to June 1964, during which time it received a fast neutron dosage of 1.54×10^{21} nvt ($E_n > 1$ Mev).⁽²⁾ The NAA 77-4 Experiment failed prematurely, after ~70 hr irradiation; postirradiation examination⁽³⁾ disclosed that the thermal expansion joint seized during operation (irradiation-accelerated diffusion), resulting in transverse rupture of the membrane during a reactor scram.

This report describes the results obtained with the NAA 77-2 and -3 Experiments, which were exposed to fission-product bombardment and neutron irradiation for a period in excess of 4600 hr at elevated temperatures.

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Figure 1. Design of NAA 77-2 and -3 Irradiation Capsules
(Figure Unclassified)

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II. DESCRIPTION OF EXPERIMENT

The design of the NAA 77 irradiation experiments was based upon Darcy's law for gas flow through membranes. Darcy's law, stated mathematically, is:

$$F = \phi P \frac{A}{d}, \quad \dots(1)$$

where

F = total gas flow [cc(STP)/hr]

ϕ = permeation constant [cc(STP)-cm/hr-cm²-atm]

P = pressure differential across membrane (atm)

d = thickness of membrane (cm)

A = conducting area (cm²)

In order to obtain the permeation constant, we must measure the gas flow, the pressure across the membrane, the gas conducting area of the membrane, and the membrane thickness. In this experiment, the gas flow was measured by a mass spectrometer, the pressure by a strain gage transducer, the conducting area was determined from the membrane dimensions and temperature profile, and the thickness was measured during fabrication of the membrane. The use of a mass spectrometer to measure hydrogen flows requires the membrane to be in a vacuum chamber.

The NAA 77-2 and -3 capsules each contained one SCB-1 coated Hastelloy N fuel cladding membrane, an intermediate Hastelloy N containment vessel which served as a gamma heater, and a stainless steel (Type 304) outer cladding, as shown in Figure 1. In addition, each membrane contained an internal fission fragment source, in the form of an alumina tube coated with a glass containing 4.6 wt % fully enriched UO₂. The purpose of this tube was to simulate the presence of a fuel rod within the membrane, by providing a source of fission fragments. During irradiation, the inner walls of the membrane were bombarded by these fission fragments, thereby simulating SNAP 8 reactor conditions.

The space between the membrane and gamma heater was evacuated by means of an external vacuum pumping system. Connected to the vacuum system was

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Figure 2. Axial Temperature Distributions in NAA 77-2 and -3 Irradiation Capsules (Figure Unclassified)

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Figure 3. Temperature Control Range for NAA 77-2 and -3 Capsules From Two-Dimensional Heat Transfer Analysis (Figure Unclassified)

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the permeation measuring equipment, which monitored the hydrogen gas escaping through the membrane wall. The permeation measurement system consisted of two mass spectrometers, each serving as a check on the other. The spectrometers were operated in conjunction with a set of calibrated glass leaks, which served as the leak rate standards on which capsule permeation rate calculations were based.

Temperature control of the cladding membranes was based on the attenuation of gamma rays in the Hastelloy N gamma heater surrounding the membrane, the size of the variable conductivity temperature control gas annulus surrounding the gamma heater, and the composition of the gas passing through the annulus. The gas annulus between the gamma heater and the outer cladding was filled with a controlled variable mixture of helium and nitrogen. The gamma heating rate was dependent upon existing conditions of reactor flux. When the gamma heating rate was high, a greater proportion of helium was introduced into the gas annulus, providing increased heat conduction away from the gamma heater and enclosed membrane because of the relatively high thermal conductivity of helium as opposed to nitrogen. Conversely, when the gamma heating rate was lower, a nitrogen-rich mixture was introduced into the annulus, in order to reduce heat loss and maintain the temperature of the test membrane. Figure 2 shows the axial temperature distribution for both NAA 77-2 and -3. Figure 3 shows the temperature control range for both capsules as a function of γ -heat generation. The membrane temperatures of each capsule were measured by five Chromel-Alumel stainless steel sheathed thermocouples, which were fastened to the outer surface of each membrane with stainless steel straps. Figure 4 shows the complete control console for these experiments.

Reactor flux measurements were made by three EGG* dosimeters strapped to each membrane, and by five dosimeter wires which were procured from Phillips Petroleum Company, placed in stainless steel tubes, and strapped to the exterior of each capsule.

Both capsule membranes were pressurized from a bottled hydrogen supply, located in the external controlling consoles. Figure 5 shows the spatial relationship of the capsules to the reactor and the controlling equipment. The instrumentation and piping diagram, Figure 6, shows the piping and valves necessary for three simultaneous irradiations.

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Figure 4. Control Console,
NAA 77 Experiments
(Figure Unclassified)

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Figure 5. Engineering Test Reactor, Showing Installed
NAA 77 Experiments and Instrumentation
(Figure Unclassified)

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Figure 6. Instrumentation Flow Diagram, NAA 77 Irradiation Experiments (Figure Unclassified)

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III. CAPSULE FABRICATION AND IRRADIATION

A. MEMBRANE FABRICATION

The membranes used in NAA 77-2 and -3 were standard production SNAP 8 cladding tubes, No. RD-35 and RD-36. The cladding assemblies were chromized twice, in order to achieve the chromized layer thickness specified for production material. The tubes were part of Hastelloy N Heat No. Y-8437, which had the following chemical analysis:

	<u>wt %</u>		<u>wt %</u>
Manganese	0.28	Nickel	71.62
Silicon	0.21	Molybdenum	16.04
Iron	4.49	Hydrogen	0.0017
Tantalum	0.024	Oxygen	0.0094
Chromium	7.33	Nitrogen	0.0028
Carbon	0.04	Boron	13 ppm

The membranes had three coats of SCB-1,⁽⁴⁾ with the second and third coats containing about 4% Sm_2O_3 , as noted in Table 1. The chemical composition of SCB-1-25 frit is noted in Table 2, and the composition of SCB-1-25 slip is given in Table 3. The outside surface of each tube was coated with Turco Pretreat, to prevent oxidation during the 2100°F air firing of the coatings. Each coat of SCB-1 was dried at 400°F for 1-1/2 hr, prior to firing. The membranes were fired in the vertical position to minimize bowing. Pitting of the SCB-1 coating was evident, throughout the length of the membranes, after the application of the second layer. The coating covering the 4-in. length near the blind end was very rough and blistered, prior to the third coat. After the third coat, these areas were visible as brown spots, and the rough spots were eliminated. Pitting was also reduced after the third coating.

The pre-irradiation permeation test results are given in Table 4. The conducting area of the membranes during permeation testing at 1500°F was 130 cm², the pre-irradiation permeation rates were 1.0×10^{-3} cc(STP)/hr-cm² for NAA 77-2 and 8.8×10^{-4} cc(STP)/hr-cm² for NAA 77-3, respectively.

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TABLE 1
SCB-1 COATING DATA FOR NAA 77-2 AND -3
TEST MEMBRANES

Coating Layer	Slip Number	Coating Weight (Dry) (g)		Firing Temperature (°F)	Firing Time (min)
		77-2	77-3		
1	SCB-1-25H	1.08	0.97	2100 ± 25	15
2	SCB-1-25G (3.7 wt % Sm_2O_3)	0.92	0.87	2100 ± 25	15
3	SCB-1-25G (3.7 wt % Sm_2O_3)	0.72	0.78	2100 ± 25	15

TABLE 2
COMPOSITION OF
SCB-1-25 FRIT

Oxide	wt %
SiO	46.7
BaO	32.7
TiO ₂	13.1
Al ₂ O ₃	7.5

TABLE 3
COMPOSITION OF SCB-1-25
COATING SLIP

	Parts
SCB-1-25 Frit	100
Black Label Clay*	3
Bentonite	1/4
Cr ₂ O ₃	1
Distilled Water	75
Sm ₂ O ₃ [†]	4

*Ferro Corporation Trademark

[†]Sm₂O₃ was only included on the outer two layers of coating

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TABLE 4
PRE-IRRADIATION PERMEATION
MEASUREMENTS

	NAA 77-2	NAA 77-3
Total Hydrogen Permeation Rate at 1500°F and 1 atm H ₂ [cc(STP)/hr/cm ²]	1×10^{-3}	8.8×10^{-4}
Pressure Dependence (n)	1.00	1.00
Activation Energy in Temperature Range (cal/mole)		
1500 - 1400°F	32,500	28,800
1500 - 1300°F	25,450	25,500
1400 - 1300°F	19,000	23,000

TABLE 5
NAA 77 SERIES IRRADIATION HISTORY

Capsule	Irradiation		Membrane Temperature (°F)	Total Time (hr)	Neutron Dosage		Fission Fragment Dosage/Element Inch
	Started	Ended			Estimated	Dosimetry	
NAA 77-1	8-63	6-64	1500	4343	2.56×10^{21}	1.54×10^{21}	-
NAA 77-2	3-64	2-65	1300	4638	1.47×10^{21}	1.71×10^{21}	1.29×10^{18}
NAA 77-3	3-64	2-65	1500	4638	1.47×10^{21}	1.44×10^{21}	1.31×10^{18}
NAA 77-4	6-64	6-64	1700	100	6×10^{19} to 13×10^{19}	-	7×10^{16} (Membrane) (ruptured)
SNAP 8 Design Parameters (600-kwt core)			~1400	10,000	1.4×10^{21}		7.6×10^{17}

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B. IRRADIATION HISTORY AND DOSIMETER ANALYSIS

NAA 77-2 and -3 capsules were irradiated in ETR Positions N-14 NW and N-14 SE from March 1964 to February 1965, for a total time at temperature of 4638 hr. This history is summarized in Table 5. While the capsules were being designed, the ETR irradiation facilities were measured to have a fast neutron flux of 8.8×10^{13} n/cm²-sec ($E_n > 1$ Mev). Using this value, the estimated neutron dosage achieved in 4638 hr of irradiation was 1.47×10^{21} ($E_n > 1$ Mev).

Two sets of dosimeters were placed on the capsules to monitor the actual neutron flux received by the capsules. One set consisted of five dosimeter wires, extending along the length of the capsules on the outside of the outer capsule can. These wires were removed after the capsules were discharged from the reactor. The five wires included one titanium, one 0.1% Co-Al, two chrome-plated iron, and one nickel wire. Only the titanium and Co-Al wires were analyzed. Problems were encountered with the iron and nickel wires, due to corrosion by the ETR coolant water. However, one value was obtained for each iron wire, as reported in Table 6. The titanium and Co-Al wires were analyzed every 2 in. along their length, with the results reported in Table 6. The peak average fast neutron flux received by the outside of the capsules, based on titanium wire analysis, were 4.94 and 4.87×10^{13} n/cm²-sec for NAA 77-2 and -3, respectively. The corresponding total fast neutron dosages were 8.25 and 8.13×10^{20} nvt ($E_n > 1$) for NAA 77-2 and -3, respectively, or approximately one-half of the calculated dosages.

Additional dosimeters were located at the top, center, and bottom of each membrane. These dosimeters were removed from the membranes, placed in sample bottles, and sent to Edgerton, Germeshausen, and Grier, Inc. for analysis. These dosimeters consisted of small nickel, cobalt, and iron foils, encapsulated in stainless steel tubes. Two of the foils were analyzed after irradiation for Co^{60} and Mn^{54} from the cobalt and iron foils, respectively. The nickel foils were not analyzed, as they became saturated from the extended time in the reactor. The results are reported in Table 7.

The analysis of the data reported in Table 7 was based on 4638 hr of operation, and Co^{60} and Mn^{54} cross sections of 36.3 b and 65 mb, respectively.

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TABLE 6
NAA 77-2 AND -3 EXTERNAL CAPSULE DOSIMETER
WIRE ANALYSIS*

	Position (Inches from End of Wire)	Thermal Neutron Flux (2200 m/sec) ($\times 10^{-14}$)	Total Thermal Neutron Dosage (nvt)($\times 10^{-21}$)	Fast Neutron Flux ($E_n > 1$ Mev) (n/cm ² -sec)($\times 10^{-13}$)			Total Fast Neutron Dosage (nvt)($\times 10^{-20}$)
				Titanium	Iron No. 1	Iron No. 2	
NAA 77-2	1	0.62	1.03	3.62			6.04
	3	1.58	2.63	4.18			6.98
	5	1.51	2.52	4.82	7.56	5.66	8.04
	7	1.60	2.67	4.65			7.76
	9	1.61	2.68	4.94			8.24
	11	1.65	2.75	4.84			8.08
	13	1.61	2.68	4.57			7.63
	15	1.55	2.58	4.41			7.36
	17	1.40	2.33	3.89			6.49
	19	-	-	3.93			6.56
	Average	1.45	2.43	4.39			7.32
NAA 77-3	1	1.62	2.70	3.96			6.61
	3	1.64	2.73	4.26			7.11
	5	1.64	2.73	4.70	7.44	5.61	7.84
	7	1.69	2.82	4.76			7.94
	9	1.63	2.72	4.87			8.13
	11			4.72			7.88
	13			4.71			7.86
	15			4.26			7.11
	17			4.01			6.69
	19			1.62			2.70
	Average	1.64	2.74	4.19			6.99

*The dosimeter wires were irradiated at the ETR during Cycles 62 through 69, for a total of 34,261 Mwd, ending February 14, 1965. A neutron resonance correction was made on the thermal flux values.

In addition to the dosimeters, three samples of the membrane material were analyzed to obtain neutron dosage data. These data, reported in Table 8, were calculated, based on an iron concentration in the alloy of 4.49%, 4638 hr of operation, and a cobalt cross section of 36.3b.

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TABLE 7
EGG DOSIMETER ANALYSIS FOR NAA 77-2 AND -3*

	Position on Membrane	Isotope	Thermal		Fast	
			Neutron Flux (n/cm ² -sec) (x 10 ⁻¹⁴)	Total Neutron Dosage (nvt) (x 10 ⁻²¹)	Neutron Flux (n/cm ² -sec) (x 10 ⁻¹⁴)	Total Neutron Dosage (nvt) (x 10 ⁻²¹)
NAA 77-2	Top	Co ⁶⁰	2.50	4.17	-	-
		Mn ⁵⁴			1.87	3.12
	Middle	Co ⁶⁰	1.28	2.13		
		Mn ⁵⁴			2.31	3.85
	Bottom	Co ⁶⁰	1.35	2.25		
		Mn ⁵⁴			-	-
	Average		1.71	2.85	2.09	3.48
NAA 77-3	Top	Co ⁶⁰	1.14	1.90		
		Mn ⁵⁴			1.86	3.10
	Middle	Co ⁶⁰	1.42	2.37		
		Mn ⁵⁴			0.73	1.21
	Bottom	Co ⁶⁰ †	-	-		
		Mn ⁵⁴			1.55	2.58
	Average		1.28	2.13	1.38	2.29

*Accuracy of Co⁶⁰ measurements are ±10%

Accuracy of Mn⁵⁴ measurements are ±20%

E_n > 1 Mev for all values

†Damaged dosimeter wire.

TABLE 8
RADIOCHEMICAL ANALYSIS OF IRON AND MANGANESE
IN NAA 77-2 AND -3 CLADDING MEMBRANE MATERIAL

	Position	Neutron Flux* (n/cm ² -sec)(x 10 ⁻¹³)	Total Fast* Neutron Dosage (E _n > 1) (nvt)(x 10 ⁻²¹)
NAA 77-2	Top	5.06	0.85
	Center	5.52	0.92
	Bottom	5.98	1.00
	Average	5.52	0.92
NAA 77-3	Top	8.07	1.35
	Center	7.43	1.24
	Bottom	8.53	1.42
	Average	8.01	1.33

*Accuracy of analysis is ±15%

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A summation of the average total neutron dosages calculated from the three different sets of dosimeter data are listed as follows:

Dosimetry	Average Total Neutron Dosage ($E_n > 1$) (nvt)			
	NAA 77-2		NAA 77-3	
	Fast	Thermal	Fast	Thermal
Cladding analysis	9.20×10^{20}		1.33×10^{21}	
EGG	3.48×10^{21}	2.85×10^{21}	2.29×10^{21}	2.13×10^{21}
PPCo*	7.32×10^{20}	2.43×10^{21}	6.99×10^{20}	2.74×10^{21}
Average	1.71×10^{21}	2.64×10^{21}	1.44×10^{21}	2.43×10^{21}

*Phillips Petroleum Company

The differences between dosimeters may be due to the orientation and location of the wires, and the accuracy to which the neutron cross sections of the cobalt, iron, and titanium are known.

All of the dosimeter analyses were corrected for decay after removal from the reactor, but were not corrected for intermittent reactor operation.

From these analyses, it was concluded that the membranes received average fast neutron dosages of 1.71×10^{21} and 1.44×10^{21} nvt ($E_n > 1$) for the NAA 77-2 and -3, respectively, rather than the design estimated dosage of 1.4×10^{21} nvt. Thus, the dosage achieved exceeded that expected in a SNAP 8 reactor core after 10,000 hr operation at 600 kw, by some 20%.

C. IN-PILE HYDROGEN PERMEATION MEASUREMENTS

The performance of the earlier NAA 77-1 irradiation capsule had demonstrated the feasibility of making reliable in-pile permeation tests. However, the permeation rates for NAA 77-1 had to be obtained by use of a mathematical partition function, to separate the hydrogen flow coming from a hot spot in the hydrogen supply line from the flow from the test membrane. The hot spot was eliminated in NAA 77-2 and -3 capsule design by cooling the hydrogen supply line with reactor coolant water, as shown in Figure 1. Hydrogen permeation rates and radiation dose data are presented in Figure 7 for the NAA 77-2 and -3 capsules. The gradual increase in the permeation rate, which was observed in both NAA 77-2 and -3, has been attributed to the devitrification of the hydrogen barrier material. From comparison of these data with data obtained in

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Figure 7. Permeation Rates and Radiation Dose Data for NAA 77-2 and -3 Irradiation Capsules (Figure Unclassified)

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Figure 8. Comparison of Permeation Rate Data for Irradiated and Unirradiated Membranes as a Function of Annealing Time (Figure Unclassified)

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long-time aging tests, it appears that the devitrification process may be enhanced in an irradiation field. Long-time permeation tests have shown that devitrification of the SCB-1 barrier material does not occur in 5000 hr aging at 1500°F, but does occur in 14,000 hr at 1500°F. This behavior may be contrasted with that obtained with the NAA 77-2 capsule, which was operated for a total of <400 hr at 1500°F; in this case, it was found that changes in the permeation rate corresponded to those expected when devitrification occurs in the absence of radiation. The gradual increase in permeation rate which has been associated with devitrification of the barrier material is also illustrated in Figure 8, which compares the permeation rates for irradiated and nonirradiated membranes. The results for NAA 77-1 are inconsistent with the given conclusion. The data for the NAA 77-1 capsule were obtained, however, using a mathematical partition function which was designed to separate the extraneous hydrogen flow through the capsule parts from the hydrogen flow through the membrane, and it is possible that this function may also have tended to mask the gradual increase in permeation rate caused by devitrification.

Analysis of the permeation rate data obtained from the NAA 77-2 and -3 capsules yielded the kinetics constants in Table 9. From the data, it may be seen that the pressure dependence of the permeation process was not affected by the irradiation, while the activation energy for permeation appears to have changed by approximately a factor of two.

TABLE 9
HYDROGEN PERMEATION KINETICS DATA FOR
NAA 77-2 AND -3 CAPSULES

	NAA 77-2		NAA 77-3	
	Pre-Irradiation	During Irradiation 4638 hr 1.47×10^{21} nvt ($E_n > 1$ Mev)	Pre-Irradiation	During Irradiation 4638 hr 1.47×10^{21} nvt ($E_n > 1$ Mev)
Rate at 1 atm and 1500°F [cc(STP)/hr-cm ²]	1×10^{-3}	2.8×10^{-3}	0.88×10^{-3}	2.7×10^{-3}
Pressure dependence	1.0	1.0	1.0	1.0
Activation energy 1500 to 1300°F (cal/mole)	24,500	16,000	25,500	11,000

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Figure 9. Hydrogen Permeation Rate as a Function of Reciprocal Temperature for SCB-1 Coated Tube RD-40

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Figure 9 illustrates the effect of crystallization^(4,5) on an unirradiated SCB-1 coated membrane. The tube, RD-40, was processed in the same batch of tubes as were the test membranes for NAA 77-2 and -3. From this study, crystallization of SCB-1 was found to begin at 1700°F, as measured by hydrogen permeation techniques. This can be seen in Figure 9, by noting the change in hydrogen flow at 1700°F. Approximately 50 hr were required before complete stabilization was reached at 1700°F, and no further recrystallization was detected, even after testing at 1900°F.

Upon crystallization at 1700°F for 50 hr, the activation energy for diffusion changed from -26,000 to -14,750 cal/mole. Data taken at 1500, 1400, 1300, and 1200°F (Figure 9) showed that the change in activation energy also affected the lower temperature permeation rates. Crystallization also stabilized the activation energy (-14,750 cal/mole) from temperatures of 1900 to 1300°F. At about 1300°F, the activation energy again underwent a change, for some unidentified reason. The higher permeation rate for crystallized SCB-1 is consistent with data obtained on about 50 earlier permeation tests, where the average permeation rate for crystallized membranes was 2.5×10^{-3} cc(STP)/hr-cm², as compared to 1.6×10^{-3} cc(STP)/hr-cm² for uncrystallized membranes tested at 1500°F and 1 atm hydrogen pressure. Figure 9 likewise shows an increase in the permeation rate for Tube RD-40 [i.e., a change from 0.70×10^{-3} to 2.3×10^{-3} cc(STP)/hr-cm² at 1500°F and 1 atm H₂ pressure]. The NAA 77-2 and -3 irradiation experiments yielded a similar increased hydrogen permeation rate and a corresponding decrease in the activation energy. This indicates that the hydrogen barrier material had devitrified, although the temperature was below that required for crystallization of nonirradiated Tube RD-40.

General conclusions that may be drawn from the in-pile hydrogen permeation measurements for the NAA 77 irradiation capsule experiments are:

- 1) The ionizing radiation field in a reactor does not significantly alter the hydrogen permeation process in SNAP 8 fuel cladding assemblies.
- 2) Radiation does appear to accelerate the devitrification process in the permeation barrier material, presumably by enhancement of self-diffusion.

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In addition, three more specific conclusions were reached:

- 1) In-pile aging of SCB-1 coated membranes has an effect on hydrogen permeation rates, similar to that observed in out-of-pile aging.
- 2) In-pile aging of SCB-1 coated membranes causes the activation energy for the permeation process to change in a manner similar to that observed in out-of-pile aging. The activation energy changes from $25,000 \pm 2,000$ cal/mole to about $14,000 \pm 2,000$ cal/mole.
- 3) In-pile aging of SCB-1 coated membranes does not alter the pressure dependence for the permeation. The pressure dependence remains a direct proportionality after either in-pile or out-of-pile aging, within the limits of the pressure range studied.

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IV. POSTIRRADIATION EXAMINATION

A. VISUAL EXAMINATION OF CAPSULES

When the capsules were received at the Atomics International Hot Laboratory (AIHL), the outside dosimeter wires had been removed for analysis by PPCo. at the National Reactor Testing Station (NRTS). Otherwise, they appeared unaffected by irradiation or handling (Figure 10).

B. CAPSULE DISASSEMBLY

An Elox machine (Electrical Discharge Machine) was used to make the first disassembly cut, which involved cutting through the capsules just below the upper end cap of the membrane. Figure 11 is a view of the top sections of the capsules after completing the cross-section cuts. On NAA 77-2, the entire top end cap was not completely removed, as shown in Figure 11.² The capsules were then turned upside down, to remove small amounts of water which had entered the capsules during hot knifing operations at the ETR.

Next, a circumferential cut was made, $\sim 3/4$ in. lower on the capsule, to remove the rest of the chill block and pull the membrane from the gamma heater. The NAA 77-2 membrane was easily removed from the gamma heater. The NAA 77-3 membrane was more difficult to remove. The bottom five thermocouple bands were broken, on both membranes, at the point where the bands were attached to the thermocouple wire. Figure 12 is a view of the membranes after removal from the gamma heaters. The thermocouple bands and dosimeter wires were still connected to the membranes. The lower thermocouple bands on both membranes were broken. Figures 12a and 12b both show portions of the outer jacket around the chill blocks, which were stuck on the tops of the membranes. Figure 13 is a view of the membranes after removal of the thermocouple bands, dosimeter wires, and chill block. Note the marks on the membranes under the thermocouple band positions. These marks may have been caused by temperature effects under the bands.

The membranes were examined under the stereomicroscope, to determine the condition of the Hastelloy N cladding. The surface color of the NAA 77-3 membrane was lighter than that of the NAA 77-2 membrane. No surface defects were observed. Figure 14 is a view of the bottom end of the NAA 77-3 membrane,

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a. NAA 77-2

NAA 77-2-1

NAA 77-2-2

a. NAA 77-2 (Capsule was cut through top end cap of membrane)

b. NAA 77-3

NAA 77-3-1

NAA 77-3-2

b. NAA 77-3 (Capsule was cut just below top end cap of membrane)

Figure 10. Capsules After Irradiation
(Figures Unclassified)

Figure 11. Capsule Cross Sections
After Cutting Through Chill Block
(Figures Unclassified)

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a. NAA 77-2

b. NAA 77-3

a. NAA 77-2

b. NAA 77-3

Figure 14. Scratch Marks on NAA 77-3 Membrane (Figure Unclassified)

Figure 12. Membranes After Removal From Gamma Heaters (Figures Unclassified)

Figure 13. Membranes After Complete Removal From Capsules (Figures Unclassified)

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where it fitted into the thermal expansion joint at the bottom of the gamma heater. Some particles of ceramic had adhered to the surface. The scratches were probably caused during removal of the membrane from the expansion joint. The membrane appeared to be tightly lodged in the expansion joint, based on the difficulty encountered in removing the membrane from the gamma heater during disassembly.

C. CREEP MEASUREMENTS

The membranes were placed in the profilometer measuring device for diameter measurements at 0, 60, and 120° orientations. This device consists of a modified lathe bed, with two end stocks to support the object being measured, and two highly sensitive linear displacement transducers which travel axially along the membrane. These transducers transmit a signal to a dual pen recorder, which prints a continuous profile of the object outside the hot cell. For this examination, the equipment was set at maximum sensitivity, to detect any localized swelling or depression of the membrane. Each profile was measured twice, to verify reproducibility. From the profile, measurements were taken every half inch along the length of the membrane. Tables 10 and 11 show the diameter measurements of the membranes.

Table 12 presents a summary of the diameter measurements and a statistical analysis of the data. The statistical "t test" was used to determine whether the difference between the post- and pre-irradiation mean diameter values was significantly different, and the degree of confidence which can be placed on the diameter growth, as detected with the profilometer. These confidence values are reported in Table 12. It can be seen that the NAA 77-3 membrane expanded, diametrically, four times as much as the NAA 77-2 membrane; this is not an expected result, in view of the operating temperatures.

Some bowing of the membranes was also observed, and was calculated to be a maximum of 0.005 and 0.008 in. for the NAA 77-2 and -3 membranes, respectively. This was substantially less than the 0.030 in. bow observed in the NAA 77-1 membrane.⁽²⁾

Localized diameter increases were observed at various locations along the length of the membranes, similar to that noted on NAA 77-1. These areas corresponded to the locations of the thermocouple bands. Tables 13 and 14 list

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TABLE 10
POSTIRRADIATION DIAMETER MEASUREMENTS
OF NAA 77-2 MEMBRANE

Distance From Top of Membrane (in.)	Diameter Measurement (in.)		
	0°	60°	120°
0.5	0.5615	0.5601	0.5615
1.0	0.5610	0.5608	0.5610
1.5	0.5611	0.5615	0.5611
2.0	0.5612	0.5618	0.5612
2.5	0.5615	0.5622	0.5615
3.0	0.5622	0.5619	0.5622
3.5	0.5625	0.5624	0.5625
4.0	0.5628	0.5636	0.5628
4.5	0.5629	0.5625	0.5629
5.0	0.5629	0.5629	0.5629
5.5	0.5629*	0.5622	0.5629
6.0	0.5622	0.5622	0.5622
6.5	0.5622	0.5620	0.5622
7.0	0.5629	0.5622	0.5629
7.5	0.5629	0.5622	0.5629
8.0	0.5636	0.5625	0.5636
8.5	0.5639*	0.5633	0.5639*
9.0	0.5629	0.5629	0.5629
9.5	0.5628	0.5629	0.5628
10.0	0.5634*	0.5636*	0.5634*
10.5	0.5629	0.5629	0.5629
11.0	0.5629	0.5629	0.5629
11.5	0.5629	0.5632	0.5629
12.0	0.5622	0.5629	0.5622
12.5	0.5622	0.5629	0.5622
13.0	0.5622	0.5629	0.5622
13.5	0.5622	0.5631	0.5622
14.0	0.5617	0.5625	0.5617
14.5	0.5615	0.5625	0.5615
15.0	0.5608	0.5625	0.5608
Average Diameter	0.5622	0.5624	0.5622
Standard Deviation	± 0.0023	± 0.0019	± 0.0023
Pre Average Diameter	0.5616	0.5622	0.5616
Standard Deviation	± 0.0027	± 0.0030	± 0.0021

*These values were not included in averages, as the values were located under thermocouple bands.

TABLE 11
POSTIRRADIATION DIAMETER MEASUREMENTS
OF NAA 77-3 MEMBRANE

Distance From Top of Membrane (in.)	Diameter Measurement (in.)		
	0°	60°	120°
0.5	0.5608	0.5609	0.5622
1.0	0.5611	0.5601	0.5636
1.5	0.5622	0.5600	0.5664*
2.0	0.5625	0.5604	0.5662
2.5	0.5629	0.5615	0.5657
3.0	0.5629	0.5618	0.5650
3.5	0.5636	0.5622	0.5650
4.0	0.5636	0.5622	0.5650
4.5	0.5638	0.5622	0.5650
5.0	0.5643	0.5629	0.5657*
5.5	0.5641	0.5622	0.5650
6.0	0.5643	0.5624	0.5657
6.5	0.5643	0.5625	0.5660
7.0	0.5643	0.5625	0.5657
7.5	0.5657*	0.5629	0.5678*
8.0	0.5643	0.5629	0.5668
8.5	0.5638	0.5632	0.5664
9.0	0.5636	0.5632	0.5671
9.5	0.5645	0.5629	0.5671
10.0	0.5657*	0.5636*	0.5678*
10.5	0.5653	0.5633	0.5671
11.0	0.5657	0.5636	0.5678*
11.5	0.5657	0.5639	0.5671
12.0	0.5650	0.5639	0.5670
12.5	0.5643	0.5639	0.5664
13.0	0.5650	0.5636	0.5657
13.5	0.5650	0.5629	0.5664*
14.0	0.5639	0.5622	0.5650
14.5	0.5629	0.5615	0.5636
15.0	0.5622	0.5613	0.5629
Average Diameter	0.5637	0.5623	0.5655
Standard Deviation	± 0.0039	± 0.0035	± 0.0043
Pre Average Diameter	0.5616	0.5619	0.5615
Standard Deviation	± 0.0024	± 0.0021	± 0.0023

*Values not included in averages, as they were taken at swells.

TABLE 12
STATISTICAL SIGNIFICANCE OF DIAMETER
MEASUREMENTS OF NAA 77-2 AND -3

		Average Diameter (in.)		
		77-2	77-3	
	Postirradiation	0.5623	0.5638	
	Pre-irradiation	0.5619	0.5617	
	Diameter Change	0.0005	0.0021	
	Confidence Limit on Difference Between Post- and Pre-irradiation Diameter Measurements (%)			
	Orientation Scan			
	0°	60°	120°	
	NAA 77-2	60	55	68
	NAA 77-3	98	32	99

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TABLE 13
DIAMETER MEASUREMENTS OF NAA 77-2 MEMBRANE
AT THERMOCOUPLE BANDS

Thermocouple Band, Numbered From Top End of Membrane	Distance From Top (in.)	Diameter (in.)			Diameter Increase at Height of Swell (in.)		
		0°	60°	120°	0°	60°	120°
1	0.5	0.5615	0.5601	0.5629	0	0	0
2	1.75	0.5611	0.5615	0.5622	0	0	0
3	2.50	0.5612	0.5622	0.5618	0	0	0.0003
4	3.50	0.5622	0.5624	0.5622	0.0001	0.0010	0
5	4.50	0.5629	0.5632	0.5622	0.0002	0.0003	0
6	5.25	0.5636	0.5629	0.5629	0.0007	0.0001	0.0003
7	6.50	0.5629	0.5624	0.5631	0.0007	0.0002	0
8	7.50	0.5629	0.5624	0.5639	0.0003	0	0.0005
9	8.00	0.5633	0.5633	0.5639	0.0003	0.0007	0.0003
10	9.00	0.5645	0.5636	0.5636	0.0014	0.0006	0
11	10.50	0.5636	0.5636	0.5645	0.0007	0.0007	0.0005
12	12.50	0.5630	0.5633	0.5636	0.0003	0.0003	0.0003
13	14.00	0.5625	0.5631	0.5629	0.0002	0.0002	0
Average Diameter =		0.5627	0.5626	0.5630			
Maximum Bow =		0.003	0.005	0.002			

TABLE 14
DIAMETER MEASUREMENTS OF NAA 77-3 MEMBRANE
AT THERMOCOUPLE BANDS

Thermocouple Band, Numbered From Top End of Membrane	Distance From Top (in.)	Diameter (in.)			Diameter Increase at Height of Swell (in.)		
		0°	60°	120°	0°	60°	120°
1	0.50	0.5608	0.5610	0.5622	0	0.0002	0
2	1.75	0.5629	0.5601	0.5664	0.0008	0	0.0006
3	2.50	0.5629	0.5615	0.5660	0.0003	0	0.0003
4	3.50	0.5636	0.5622	0.5657	0.0002	0	0.0007
5	5.00	0.5648	0.5629	0.5657	0.0011	0.0002	0.0007
6	6.00	0.5646	0.5624	0.5664	0.0003	0	0.0005
7	7.50	0.5657	0.5629	0.5682	0.0016	0	0.0017
8	8.50	0.5640	0.5636	0.5671	0.0002	0.0007	0.0003
9	9.50	0.5657	0.5636	0.5678	0.0007	0.0004	0.0007
10	10.50	0.5664	0.5638	0.5678	0.0007	0.0002	0.0007
11	11.25	0.5661	0.5643	0.5678	0.0008	0.0004	0.0009
12	12.75	0.5659	0.5636	0.5664	0.0004	0.0002	0.0007
13	14.50	0.5622	0.5608	0.5625	0	0	0
Average Diameter =		0.5642	0.5625	0.5661			
Maximum Bow =		0.008	0.005	0.006			

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the diameter measurements at the thermocouple bands. Although the average diameter under the thermocouple bands, as reported in Tables 13 and 14, were larger than the average postirradiation diameter, many measurements as reported do not indicate a diameter increase. Therefore, the swelling characteristics of the membranes can best be judged by comparing the diameter at the swell to the diameter on both sides adjacent to the swell. These data are also tabulated in Tables 13 and 14. The maximum diameter increases occurred in the center and lower portions of the membranes, where the temperatures were highest during irradiation. These effects are believed to be the consequence of creep in the membranes, due to constant internal hydrogen pressure and high temperature for a period of 4638 hr. Sufficiently higher membrane temperatures occurred under the bands to cause a greater increase in the diameter of the Hastelloy N at these locations.

Figure 15 compares the creep rate data for unirradiated samples and for the NAA 77-1, -2, and -3 membranes. It shows that low stress samples, both irradiated and unirradiated, have experienced accelerated creep, as compared to the extrapolated rates of Figure 15. The membranes from the NAA 77-1 and -3 capsules experienced an identical creep rate of $10^{-4}\%$ /hr. Both capsules were operated at the same reactor environment conditions. This result supports the hypothesis that the deformation occurring in low-stress, long-time creep tests is significantly greater than would be predicted from linear extrapolation of data from high-stress creep tests (shorter times). The membrane in the NAA 77-2 capsule exhibited an average creep rate of $3.5 \times 10^{-5}\%$ /hr. It is surprising, at first glance, that the creep rate of this membrane was so high. In fact, however, NAA 77-2 was operated at 1500°F for about 10% of the time, at pressures of 1 and 3 atm. This probably accounts for the high average creep rate.

Also included in Figure 15 are data from two unirradiated tubes, numbered E-808 and -822. These tubes were tested for 14,000 hr at 1500°F and 1 atm internal hydrogen pressure. This internal pressure caused a calculated hoop stress in the membranes of ~400 psi. This stress caused an average creep rate of 3.3×10^{-7} in./in.-hr, or about one-third of that observed for the NAA 77-1 and -3 capsule membranes. This ratio corresponds to the ratio of the applied stresses.

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Figure 15. Low-Stress Creep Studies of Irradiated and Unirradiated SNAP 8 Cladding Assemblies (Figure Unclassified)

TABLE 15
NAA 77 MEMBRANE CREEP RATE DATA

Membrane	Experimental Creep Rate (%/hr)	Extrapolated Creep Rate* (%/hr)
E-808 and -822 unirradiated tubes	3.3×10^{-5}	5.0×10^{-8}
NAA 77-1	10^{-4}	5.0×10^{-6}
NAA 77-2	3.5×10^{-5}	2.5×10^{-8}
NAA 77-3	10^{-4}	5.0×10^{-6}

*Extrapolated from ORNL-2780

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Figure 16. Sampling Diagram of NAA 77-2 and -3 Membranes for Metallography, Adherence, and Dosimeter Samples
(Figure Unclassified)

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Creep data from the NAA 77 irradiation experiments, and from the long-time, low-stress aging tests, are presented in Table 15. Also included are creep rate values that were obtained by extrapolating data on INOR-8 to lower stresses. This comparison is presented to further emphasize the risk associated with such extrapolations. It is obvious that the extrapolated data can result in highly misleading estimates of total deformation (on the low side).

D. PREPARATION OF METALLOGRAPHIC, ADHERENCE, AND DOSIMETER SAMPLES

Figure 16 shows the sampling diagram for metallographic, adherence test, and cladding dosimeter analysis samples. A total of 3 of each type of sample was cut from each membrane, utilizing a diamond-edged cutoff wheel. The samples consisted of 1/2-in. long rings, cut from the membrane. The adherence test samples were slit along one side, to allow the cladding material to be bent open with the hot cell manipulators. A metallographic sample was also obtained from the bottom end cap of each membrane, to observe the grain structure of the bar stock parts, as compared to the tubing. After all the samples were cut, the remaining cladding was slit open, to observe the surface condition of the ceramic coating.

E. ADHERENCE TESTING

A qualitative test, to determine the adherence of the ceramic (SCB-1) to metal, was also performed in the hot laboratory. Each sample was spread open and bent back and forth over a 1/2-in. dowel pin, to test the flaking and spalling characteristics of the ceramic coating. The samples were bent a maximum of 11 times with the manipulators. Figure 17 shows the three samples from each membrane, after testing.

The samples from the top of both membranes had poor ceramic adherence. The coating broke into splinters and spalled off the Hastelloy N. This portion of both membranes was at an irradiation temperature of 500°F. In contrast, the samples taken 7 in. from the top of each membrane had excellent adherence. These samples were irradiated at temperatures of 1300 and 1500°F for NAA 77-2 and -3, respectively. The two samples taken 1 in. from the bottom of the membranes also had excellent adherence. Both of these samples were irradiated at 1000°F.

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NAA 77-2-23

a. NAA 77-2

- SAMPLE 1: Located at top end of membrane. Poor adherence of ceramic to the Hastelloy N. Irradiated at 500°F.
- SAMPLE 2: Located 7 in. from top end of membrane. Excellent adherence of ceramic to the Hastelloy N. Irradiated at 1300°F.
- SAMPLE 3: Located 1 in. from bottom of membrane. Excellent adherence of ceramic to the Hastelloy N. Irradiated at 1000°F.

- SAMPLE 4: Located at top end of membrane. Poor adherence of ceramic to the Hastelloy N. Irradiated at 500°F.
- SAMPLE 5: Located 7 in. from top end of membrane. Excellent adherence of ceramic to the Hastelloy N. Irradiated at 1500°F.
- SAMPLE 6: Located 1 in. from bottom of membrane. Excellent adherence of ceramic to the Hastelloy N. Irradiated at 1000°F.

NAA 77-3-20

b. NAA 77-3

Figure 17. Adherence Test Samples After Testing
(Figures Unclassified)

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The remaining sections of cladding which were slit open for visual examination were also tested for adherence quality. One half-round longitudinal section from the top and bottom portion of each membrane was tested. The sections were flattened in the jaws of a General Mills manipulator. The sections were then bent back and forth in the manipulator jaws, to test the adherence properties.

The top 4 in. from both membranes had poor adherence, with the ceramic chipping off when the tube halves were flattened. However, at this point (4 in. from the top of the membrane), there was a sharp transition zone to excellent adherence. No chipping of the ceramic occurred throughout this higher irradiation temperature zone of the membranes ($>1000^{\circ}\text{F}$).

F. VISUAL EXAMINATION OF MEMBRANE

After the membrane was slit open with the Elox machine and the Elox oil was washed off, the ceramic coating was visually examined with the Kollmorgen periscope and stereomicroscope. Figures 18 and 19 show the slit sections of cladding. Generally, the ceramic appeared to be in good condition. Some small blisters in the ceramic were observed, about 8 in. from the bottom of the NAA 77-2 membrane. This area of ceramic is shown in Figure 20. This was the only location on either membrane where this effect was observed. The same type of blistering was observed in the NAA 77-1 ceramic.⁽²⁾ Also, some of the UO_2 -doped ceramic from the fission tube was fused to the membrane ceramic at various points. This effect was expected, if the two surfaces came in contact during operation, since the ceramic coating on the fission tube and on the membrane were of the same nominal composition (see Figure 21). Parts of the fission tubes are shown in Figures 18 and 19. The tubes were broken and cracked during disassembly of the membranes.

G. METALLOGRAPHIC EXAMINATION

All of the samples were mounted in epoxy resin and ground on 240, 320, and 600 grit paper. The samples were then rough polished with 6μ diamond dust, and final polished with 1μ diamond dust. The samples were etched with a solution consisting of 3 parts concentrated HCl , 2 parts H_2O , and 1 part 10% chromic acid. However, three of the samples would not etch with this solution, and had to be etched with aqua regia to show the grain structure. After etching, the samples were washed with water and dried.

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Figure 18. Ceramic Coating on Inside of NAA 77-2 Membrane
(Figure Unclassified)

Figure 19. Ceramic Coating on Inside of NAA 77-3 Membrane
(Figure Unclassified)

Figure 20. Blisters in the Ceramic, 8 in. From
Bottom End of NAA 77-2
(Figure Unclassified)

a. NAA 77-2

b. NAA 77-3

Figure 21. Inside Surface of Ceramic, Where Fission Tube Contacted Membrane
(Figures Unclassified)

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After the samples were ground and polished, they were examined under the metallograph, in the unetched condition, to observe the SCB-1 structure. The condition of the ceramic is shown in Figures 22 and 23. The samples are identified by their Met. numbers; Figure 16 shows the positions on the membranes from which they are taken. The usual structure, with randomly scattered non-spherical bubbles, was observed. However, there was more porosity in the ceramic near the bottom of the membranes than near the top. A typical "pit defect" is shown in Figure 22. This defect is similar to those observed in unirradiated SCB-1 coatings, which are illustrated in Figure 24. In addition, random thickness measurements of the ceramic were taken, to determine variations of ceramic thickness as a function of axial position in the membranes. These measurements are presented in Table 16. Variations in SCB-1 thickness around the circumference of the membranes were noted, and the ceramic was more than twice as thick at the top of the membranes as at the bottom. This variation in enamel thickness is a normal feature of coatings applied to blind end tubes by the slip washing process.

TABLE 16
SCB-1 THICKNESS MEASUREMENTS FOR NAA 77-2 AND -3
AFTER IRRADIATION
(All measurements in mils)

	NAA 77-2			NAA 77-3		
	Top	Middle	Bottom	Top	Middle	Bottom
	3.1	1.7	0.6	2.2	2.2	0.9
	3.0	2.2	0.9	2.5		1.3
	3.0	2.0	1.6	2.3	Ceramic missing from this sample	1.0
	3.0	2.8	1.4	2.2		1.2
	2.7	1.7		2.2		1.7
	2.3	1.9				1.1
	2.4	1.8				1.0
	2.4	1.6				1.4
	2.1	1.8				0.8
	2.0	1.7				0.9
Average	2.6	1.9	1.1	2.3	2.2	1.1

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a. Sample Met. 1
(Typical view 500°F irradiation temperature. Small randomly scattered bubbles prevalent in ceramic)

b. Sample Met. 2
(View of largest bubble observed in ceramic. 1300°F temperature)

c. Sample Met. 2
(Typical view of structure. 1300°F irradiation temperature)

d. Sample Met. 3
(Typical view. 1000°F irradiation temperature)

Figure 22. SCB-1 Ceramic Structure After Irradiation, NAA 77-2
(Figures Unclassified)

a. Sample Met. 6
(Typical view. 500°F irradiation temperature)

b. Sample Met. 7
(Typical view. 1000°F irradiation temperature)

c. Sample Met. 8
(Typical view. 1500°F irradiation temperature)

Figure 23. SCB-1 Ceramic Structure After Irradiation, NAA 77-3
(Figures Unclassified)

a. 3 Coatings, 10-min Firing

b. 3 Coatings, 10-min Firing

c. 3 Coatings, 45-min Firing
(Corner of end cap)

Figure 24. Unirradiated SCB-1 Ceramic Coating After Firing at 2100°F, Showing Large Void Areas in Ceramic, Similar to Those Shown in Figures 22 and 23
(Figures Unclassified)

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Each cladding sample was examined on the metallograph after etching. ASTM grain size measurements of the Hastelloy N were taken, by comparing the observed grain size to standard grain size charts. Generally, the ASTM grain sizes varied from 1 to 3 across the center portions of the cladding, and from 3 to 7 along the edges. This same phenomenon occurs in unirradiated aged cladding tubes.⁽²⁾

Figure 25a shows the typical Hastelloy N grain structure from the top end of the NAA 77-2 membrane. Figure 25b shows the grain structure about 6 in. from the top (in the 1300°F irradiation temperature region), where numerous precipitates were forming in the grain boundaries. Figures 25c and 25d show the grain structure of the bottom end cap and of the weld zone between the end cap and the tubing, respectively. The samples are identified by their Met. numbers; Figure 16 shows the positions on the membrane from which they were taken. Many precipitates were present throughout the grain structure in the weld area. Very small grains were present along the inside and outside surfaces of the Hastelloy N tubing (ASTM Grain Sizes 6 to 8), with much larger grains (ASTM Grain Sizes 2 to 6) through the bulk of the cladding. The small grains along the surfaces of the cladding tube were present in all photomicrographs.

Figures 26a and 26b show the typical cladding grain structure at the top and ~6 in. from the top of the NAA 77-3 membrane, while Figures 26c and 26d show the grain structure above the bottom end cap weld zone and across the bottom end cap. The sample shown in Figure 26b was from the 1500°F irradiation temperature region, and shows how precipitates were forming in the grain boundaries. Again, the samples are identified by their Met. numbers, shown on Figure 16. Figure 26d shows numerous precipitates in the grain structure, and porous ceramic adhering to the outside surface of the cladding. Figure 26e shows the weld grain structure on the 77-3 membrane between the bottom end cap and tubing. Again, numerous precipitates were present across the grains. These precipitates are formed during welding operations. Figure 27 shows an unirradiated Hastelloy N weld zone, with the same type of precipitates distributed among the grains. The normally elongated grain structure present in the weld zone was not visible here, but very large grains (ASTM Grain Size 0 to 1) were prevalent over 98% of the weld zone surface.

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21-1-2

a. Sample Met. 1 (ASTM Grain Size 1 to 4)

21-2-3

b. Sample Met. 2 (ASTM Grain Size 4 to 6)

21-3-3

c. Sample Met. 3, Across Bar Stock Bottom End Cap (ASTM Grain Size 2 to 6)

Figure 25. Grain Structure of Hastelloy N, NAA 77-2 Membrane (Figures Unclassified)

21-3-4, -5, -6

d. Sample Met. 3, Across Weld Area Where Bottom End Cap Was Welded to Tube Stock (ASTM Grain Size 2 to 6)

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21-6-2

a. Sample Met. 6 (ASTM Grain Size 2 to 5)

21-7-2

b. Sample Met. 7 (ASTM Grain Size 3 to 6)

21-8-4

c. Sample Met. 8, Above Weld Area (ASTM Grain Size 2 to 5)

21-8-6

d. Sample Met. 8, Across Bar Stock Bottom End Cap (ASTM Grain Size 1 to 5)

5945-1-2

Figure 27. Weld Zone in Unirradiated, SNAP 8 Cladding Tube After Chromizing, Showing Numerous Precipitates in Grains (Figure Unclassified)

21-8-8, -9, -10

e. Sample Met. 8, Across Weld Area Where Bottom End Cap Was Welded to Tube Stock (ASTM Grain Size 0 to 4)

Figure 26. Grain Structure of Hastelloy N, NAA 77-3 Membrane (Figures Unclassified)

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Out-of-pile, 14,000 hr, permeation tests at 1500°F have also been conducted on five SNAP 8 cladding membranes. The metallographic examination of these membranes⁽²⁾ has revealed many precipitates in the grain boundaries, similar to that observed in the NAA 77-2 and -3 cladding metallography. The large grain structure observed throughout the NAA 77-2 and -3 metallography samples were also seen in the out-of-pile tests, indicating that grain growth was largely due to thermal conditions, and not to a radiation effect.

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V. CONCLUSIONS

The general conclusions that may be drawn from the NAA 77 series irradiation capsule experiments are:

1) The ionizing radiation field in a reactor does not measurably alter the hydrogen permeation process in SNAP 8 fuel cladding assemblies. Radiation does appear to accelerate the devitrification process in the permeation barrier material, presumably by enhancement of self diffusion.

2) In-pile aging of SCB-1 coated membranes causes an increase in hydrogen permeation rates, similar to that observed in out-of-pile aging.

3) In-pile aging of SCB-1 coated membranes caused the activation energy for the permeation process to change in a manner similar to that observed in out-of-pile aging. The activation energy changed from $25,000 \pm 2,000$ cal/mole to about $14,000 \pm 2,000$ cal/mole.

4) In-pile aging of SCB-1 coated membranes does not alter the pressure dependence for the permeation. The pressure dependence remains a direct proportionality, after either in-pile or out-of-pile aging within the limits of the pressure range studied.

5) The Hastelloy N cladding material experienced grain growth, but not to a greater extent than has been observed in unirradiated samples aged under similar time and temperature conditions. The Hastelloy N grain boundaries of both irradiated and unirradiated material became decorated with precipitated particles, as a result of thermal aging.

6) The creep rates of the NAA 77-2 and -3 membranes were higher than would be predicted from published data for unirradiated Hastelloy N. This observation is in agreement with the creep rate experienced by the NAA 77-1 membrane.

7) The NAA 77 series of experiments showed that the SCB-1 ceramic material would meet or exceed the specification required for operation of the SNAP 8 hydrogen barrier under normal reactor operating conditions.

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