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QUARTERLY TECHNICAL PROGRESS REPORT
FUELS, MATERIALS, AND COOLANT
CHEMISTRY PROGRAMS
JULY-SEPTEMBER 1971

AEC Research and Development Report

Atomics International
North American Rockwell

P.O. Box 309
Canoga Park, California 91304

R2341

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The preceding Progress Report (Annual) was
AI-AEC-13007

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Program: Sodium Technology			
AEC Task: 1 - FFTF Sodium Component Qualification			
Project Manager: G. W. Meyers			
Reporting Period: July-September 1971		189A: 13625	
General Order: 06507	Subaccount: 39610	Category: 04-40-04-04.1	

Principal Investigators: J. L. Ballif, J. Guon, R. L. Eichelberger,
R. L. McKisson

I. PROJECT OBJECTIVES

The general objective of this work is to provide component testing services and/or ordering information on FFTF component vapor traps, freeze vents, and filters. Specifically, the objectives are:

- 1) Construct and evaluate the performance of three vapor trap designs, at flow rates over the range of 1 to 5 scfm, while maintaining the space restrictions imposed by FFTF applications, and issue evaluation reports
- 2) Revise and issue the RDT Standard for vapor traps
- 3) Construct and evaluate the performance of one freeze vent, and issue an evaluation report
- 4) Participate in the revision of the RDT Standard for the freeze vent
- 5) Prepare an RDT Standard for filters for the FFTF sodium charging system.

II. ACCOMPLISHMENTS DURING FISCAL YEAR 1972

A. SUMMARY

The multiple-baffle vapor trap concept was selected as having the greatest probability of meeting the 5-scfm requirement in the smallest envelope size. The existing trap, which operated in the 0.5- to 0.8-scfm range, has been modified in an attempt to increase its performance, and testing is underway.

TABLE 1
MULTIPLE-BAFFLE VAPOR TRAP - JULY RESULTS
(Packed mesh installed downstream of baffles,
saturator temperature, 1050°F)*

Date (1971)	Argon Flow (scfm)	Analysis of Sodium in Gas (wppm)		Removal Factor†		
		Baffle Out	Packed Mesh Out	By Baffles	By Mesh	Overall
June 29	0.3	72	20	133	3.6	480
30	0.3	76	22	127	3.5	436
July 1	0.3	44	5	218	8.8	1,920
2	0.3	44	12	218	3.7	800
3	0.3	44	17	218	2.6	565
4	0.3	44	17	218	2.6	565
5	0.3	44	17	218	2.6	565
6	0.3	2	1	4,800	2.0	9,600
7	0.3	2	1	4,800	2.0	9,600
8	0.3	0.6	0.2	16,000	3.0	48,000
9	0.3	0.1	0.1	96,000	1.0	96,000
10	0.3	0.1	0.1	96,000	1.0	96,000
11	0.3	0.1	0.1	96,000	1.0	96,000
12	1.0	12.0	10.0	800	1.2	960
13	1.0	26.0	22	369	1.2	436
14	1.0	2.0	1.8	4,800	1.1	5,330
15	1.0	1.0	0.6	9,600	1.7	16,000
16	0.9	12.0	1.0	800	12.0	9,600
17	0.9	12.0	1.0	800	12.0	9,600
18	0.9	12.0	1.0	800	12.0	9,600
19	0.9	2.4	1.8	4,000	1.3	5,330
20	1.0	48.0	37.0	200	1.3	260
21	1.0	5.6	4.0	1,710	1.4	2,400
22	1.0	2.8	2.4	3,430	1.2	4,000
23	1.0	0.6	0.5	16,000	1.2	19,200
24	1.0	0.6	0.5	16,000	1.2	19,200
25	1.0	0.6	0.5	16,000	1.2	19,200

*Argon stream leaving saturator contains 9300 wppm sodium vapor.

†(Sodium concentration entering)/(Sodium concentration leaving)

Aerosol filter loading tests have been completed on 5-, 20-, and 35- μ pore size filters. The data have been included in the appendix of the vapor trap standard.

A slightly modified controlled-temperature-profile design has been selected for the prototype 1.0-scfm vapor trap, which is being designed to meet the requirements of the FFTF facilities. Design layouts and procurement of materials have been completed.

The testing program conducted on the freeze vent is nearing completion. The test data have generally been close to prediction.

The appendix for the design of controlled-temperature-profile vapor trap condensers, for flow rates up to 1.0 scfm, has been added to the draft of RDT Standard E4-14.

The appendix for the design of the vapor trap filter assembly has been prepared in draft form.

B. VAPOR TRAPS

1. Multiple-Baffle Vapor Trap (MBVT)

The testing of this trap, with the packed mesh added to the outlet section, was started early this quarter. Data were obtained over the range of 0.3 to 1.0 scfm, with the argon saturated with sodium at 1050°F (9300 wppm sodium in the argon). Results of the continuous test run are shown in Table 1. Data were obtained at the entrance and exit of the packed mesh, to obtain a measure of the mesh performance.

The data support the hypothesis that a conditioning period is required before the vapor trap can operate at its highest efficiency. In the test series at low flow rates, a 7-day period was required before the trap reached peak efficiency. The data on the packed mesh suggest that "York Mesh" is moderately effective in reducing the concentration of sodium in the exit of the condenser section.

During a program review in July, AI was asked to select the best trap design, of those under test, to be scaled up for use at 5 scfm. Review of all data suggests that the multiple-baffle vapor trap has the best chance of reaching

Figure 1. 1-in. Diameter Type 304 Stainless Steel Pipe, Partially Plugged With Condensed Sodium Vapor

that design goal in a modest-sized envelope. Analysis suggests re-entrainment of the sodium droplets occurs as reflux sodium drips down the baffle cones. The principle of operation of the multiple baffle is thought to be centrifugal force, which is developed when the flow path is sharply reversed between baffles. Two ways to upgrade performance of the present trap are under consideration. The first is to change the gas velocity by varying the baffle spacing, and the second is to eliminate the uncontrolled dripping of the reflux sodium by the use of wire collectors and wicks to transport the sodium downward and out of the baffle region.

Testing was stopped on the existing multiple-baffle vapor trap (MBVT) on August 2, 1971, according to schedule. The modifications to the MBVT discussed in the previous paragraph were completed in August. Several interesting observations were made during the modification work. The first was the presence of heavy deposits in the 1-in. diameter pipe, downstream of the condenser unit. Cross sections of the pipe and the deposits can be seen in Figure 1. This figure indicates that plugging of small lines is quite possible, even when small quantities of sodium vapor are passing through the line. This suggests the advisability of going to the 2-in. diameter line size to minimize the tendency to plug.

When the mesh section was examined, the mesh bed was found to be displaced. It is therefore concluded that the mesh material requires a holddown device to keep it in place. The old design was modified by installing a perforated plate to act as a mesh retainer. Figure 2 shows a view of the MBVT with the mesh extension section, as built and installed. Testing of the modified trap was started in the first week of September 1971.

2. Controlled-Temperature-Profile Vapor Trap (CTPVT)

In the AI-HEDL program review, AI was also asked to select the best vapor trap under test for the ≤ 1.0 -scfm vapor trap application. The current CTPVT operated well at 0.8 scfm, but has had difficulty in operating at 1.0 scfm. The AI recommendation was to use the CTPVT, with dimensional and internal modifications, to assure meeting the 1.0-scfm FFTF facility requirement. These modifications are discussed in Section II-B-4.

Testing of the CTPVT at 0.8 scfm, with the sodium saturator at 1050°F, has been carried out from June 30 to July 21, 1971. Then, the sodium saturator

Figure 2. Multiple-Baffle Vapor Trap

temperature was reduced to 800°F for a 4-day run. Following that, the CTPVT system was used in a series of tests of filter loadings, as is described in Section II-B-3.

Data from the 0.8-scfm tests performed during this quarter are seen in Table 2. These data suggest that the sodium output levels in the argon gas have been quite low, both at 1050 and 800°F. These results reflect the trap performance at 0.8 scfm. At 1.0 scfm, previous data from this unit have shown an unacceptably high level of sodium content.

There have been occasions when the gas flow line between the exit of the trap and the final filter has become partially plugged. When this occurs, the system is pressurized and vented (blown down) to remove sodium clinging to the pipe wall. The plugs generally occur at the inlet to the filter (the sodium does not reach the filter leaves), due to the constriction and because that section may be colder. The filter leaves do not show a significant amount of sodium. The sodium causing line blockage results from the discharge of sodium particles which have accumulated on the piping walls from previous runs. A change in system parameters appears to cause some system upset, which discharges larger than normal quantities of sodium and dislodges particles of sodium sticking to the pipe wall.

3. Filter Studies

The first studies of filter performance were made under "normal" operating conditions (i.e., when the effluent argon gas leaving the vapor trap condenser had a 1- to 2-wppm sodium concentration). It was found that rather small amounts of sodium in such an argon stream would plug a filter. From 1 to 4 gm Na was found to plug a 4 ft², 5μ filter, and as little as 1.35 gm Na could plug a 35μ filter.

Additional operating experience on vapor trap condensers has shown that they have periods of operational "upset," during which times the sodium concentration in the effluent stream is characteristically several hundred wppm. However, it has also been found that the sodium characteristic of upset operating conditions has a lower propensity for plugging the filter. In order to determine the filter loading for upset conditions, and for the three sizes of filters, so that this information could be used in Appendix D of RDT Standard E4-14, the

TABLE 2
CONTROLLED-TEMPERATURE-PROFILE VAPOR TRAP -
JULY RESULTS*

Date (1971)	Flow (scfm)	Saturator Temperature (°F)	Analysis of Sodium in Trap Outlet (wppm)	Removal Factor†
June 30§	0.8	1050	0.7	13,700
July 1	0.8	1050	0.7	13,700
2	0.8	1050	2.0	4,800
3	0.8	1050	2.0	4,800
4	0.8	1050	2.0	4,800
5	0.8	1050	2.0	4,800
6§	0.8	1050	2.0	4,800
7	0.8	1050	2.0	4,800
8	0.8	1050	2.0	4,800
9	0.8	1050	2.0	4,800
10	0.8	1050	4.6	2,085
11	0.8	1050	4.6	2,085
12§	0.8	1050	4.6	2,085
13	0.8	1050	0.4	24,000
14	0.8	1050	0.2	48,000
15	0.8	1050	0.3	32,000
16	0.8	1050	0.3	32,000
17	0.8	1050	2.0	4,800
18	0.8	1050	2.0	4,800
19	0.8	1050	5.8	1,655
20§	0.8	1050	5.8	1,655
21§	0.8	1050	0.3	32,000
22	0.8	800	0.4	1,250
23	0.8	800	0.1	5,000
24	0.8	800	0.1	5,000
25	0.8	800	0.1	5,000

*Argon stream leaving saturator at 1050 has 9300 wppm sodium vapor; at 800, it has 500 wppm sodium vapor.

†(Sodium concentration entering)/(Sodium concentration leaving)

§System blown down

CTPVT system was operated under "upset" conditions. Tests of the 5, 20, and 35 μ filters were made. The upset operating condition chosen for the tests produced a discharge gas having ~ 220 wppm Na at a flow rate of 1.7 scfm, with the sodium saturator operating at 800°F. The criteria for plugging was an increase of >10 in. H₂O pressure drop across the filter body. Table 3 shows a summary of the data from the filter tests. These data have been incorporated in Appendix D of the RDT Standard E4-14 draft, for submission to HEDL and RDT. Gas samples, downstream of the filters, indicate that no detectable sodium is present in the filter effluent gas from any of the filters. Therefore, based on these test results, the 35 μ pore size is preferred. However, it does appear that a test of a larger pore size should be scheduled, because, if the sodium loading can be increased, the filter area required for any given service will decrease.

TABLE 3
SUMMARY OF FILTER DATA RUN IN TEST RIG No. 2 -
CTPVT - UPSET CONDITIONS

Average Pore Diameter for Powdered Metal Filter (μ)	Sodium Loading for 4 ft ² of Filter (gm)*		Sodium Loading Factor (gm/ft ²)
5	5.5, 6.3, 6.3	6.0 average	1.5
20	5.8	5.8 average	1.5
35	6.6, 6.8, 7.7	7.0 average	1.75

Note: The CTPVT was operated at upset conditions during these tests, to accelerate loading.

*Time for loading, on the average, was ~ 3 hr.

4. 1.0-scfm Standard Vapor Trap (SVT)

In accordance with the recommendations made during the July review meeting with HEDL, the 1.0-scfm standard vapor trap was determined to be a version of the controlled-temperature-profile vapor trap. In order to increase the trap's flow capacity, the diameter was increased from 8 to 10 in., and the packing depth was increased from 42 to 48 in. A dry wall device will also be installed, to keep the reflux sodium from being exposed to the hot wall and picked up by the incoming gas, and a sodium inlet line and distribution plate are being added above the Rachig Ring bed. The sodium inlet line will be used to condition the

*NOTE SECTIONS I, II, AND III ARE PART OF THE VAPOR TRAP ASSEMBLY
SECTIONS SHOULD BE AS CLOSE AS POSSIBLE WITH CONSIDERATION OF CLEARANCE

6507-1816

Figure 3. Proposed Standard Vapor Trap Assembly

vapor trap prior to system startup, and for maintenance, should the trap performance become degraded during the test. In order to improve the axial temperature profile of the gas stream, a central plug has been added to form a 3-in. annulus for gas flow adjacent to the controlled temperature wall surface. It is planned to build this as a prototype unit to the RDT Standard and to HEDL Ordering Data, and to test it for a 3-month period.

Figure 3 shows a sketch of the device being designed. As is noted, the vapor trap assembly consists of three sections: the preheater, the condenser, and the aerosol filter. The constraints of clearance may cause the preheater and the filter to be in other than straight-line positions. The design is being made up with three distinct parts, so that a standardized trap will require an assembly drawing, depending upon its application, but the components are standardized in size and capacity.

The current status of the Composite Reactor Components Test Activity (CRCTA) vapor trap requested by HEDL is that the design layouts of both the condenser and the filter have been completed for initial design review. Long-lead-time items have been ordered, and preliminary stress considerations have been examined. More detailed stress analysis is dependent upon the receipt of complete reaction loading and vibration information from HEDL. It is necessary to have this information before a preliminary stress analysis report can be written.

C. FREEZE VENT

During this quarter, an unfinned freeze vent section was fabricated, and installed in the test rig. Figure 4 is a photograph of the test rig with the new section in place. The purpose of testing an unfinned section was to determine whether there were any differences in the performance of this vent, as opposed to the finned vent tested earlier. If the unfinned vent's performance is satisfactory, then it would be the preferred design, because of its simplicity, both in manufacture and in the design stress analyses required to qualify it to code.

The tests performed to investigate the position of the seal as it formed in the vent involved two ambient temperature ranges, 90°F (85 to 95°F) and 140°F (130 to 144°F), three bulk sodium temperatures, 300, 400, and 500°F, and

Figure 4. Unfinned Freeze Vent Section in Place on Test Rig

initial sodium velocities in the vent annulus ranging from 2.5 to 32 ft/sec. Each test was performed by first adjusting the sodium bulk temperature and the ambient temperature. Then, by applying gas pressure to the free surface of sodium in the supply tank, the sodium was forced out of the tank and into the vent. By monitoring the flow rate of the sodium and the flow rate of the gas in the vent as a function of time, the entering flow velocity in the vent could be determined. After the position of the seal in the annulus was measured, using a PVT gas expansion technique, the seal was broken, using the vent heater, and the sodium drained into the supply tank to conclude the test.

The test results of the finned and the unfinned vents are presented in Figure 5a and b, and in Table 4. Examination of the figures shows that the seals form in positions which are generally expected, from a consideration of the controlling variables, sodium temperature, ambient air temperature, and sodium velocity entering the annulus.

The seal positions are higher in the annulus than are predicted from Equation A-5 of E4-13T. This is attributed to the actual heat transfer coefficient's being less than was expected for the transfer of heat from the moving sodium to the stainless steel wall. Because the heat does not escape as fast as was originally assumed, the sodium stays liquid for a longer time, and thus moves farther along the annulus. Typical freezing times are 0.03 to 0.10 sec.

A second test series was carried out with the unfinned vent. This was a test of the way in which a seal, originally made near the entrance to the vent annulus, would move when the temperature of the bottom of the annulus was increased. This is a simulation of the sequence in a reactor system in which the seal would be made as the gas was vented at, say, 400°F, and then the system temperature would be raised to, say, 1100°F. In such a sequence, the temperatures along the vent would increase, perhaps to the extent that the temperature of the position of the original seal would eventually exceed the melting point of sodium.

It was found that, when one made a seal at a low sodium velocity, and at a low ambient external temperature, the seal was made easily, and the sodium penetrated only a few inches into the vent section. Then, if the sodium system temperature is raised to ~700°F (or higher), a distinct breaking of the seal (as opposed to a creeping motion) is seen, with the subsequent abrupt movement of

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a. 90°F Vent Ambient Temperature

6507 4762

b. Nominal 140°F Ambient Temperature

Figure 5. Freeze Vent Test Data

TABLE 4
SUMMARY OF EXPERIMENTAL DATA FOR THE FREEZE VENT DEVICE

Run No.	Sodium Temperature (°F)	Vent Temperature (°F)	Top* of Sodium Column (in.)	Flow† Time (sec)	Sodium Flow Rate (fps)
<u>Finned Vent</u>					
1	300	85	0.7	ND	ND
2	303	85	3.0	ND	ND
3	311	86	6.2	ND	ND
4	303	88	6.9	1.6	35
5	310	90	8.0	1.4	39
6	308	88	8.0	ND	ND
7	310	85	4.1	1.3	ND
8	310	110	8.7	1.2	22
9	512	95	11.3	1.25	22
10	520	95	12.0	1.04	25
11	501	95	13.8	0.81	30
12	490	88	15.1	0.44	33
13	416	82	7.7	1.24	22
14	429	85	13.3	0.98	25
15	414	80	7.5	1.18	24
16	417	82	13.1	0.98	26
17	412	80	10.4	0.52	24
20	414	80	10.5	1.34	22
21	322	132	10.1	4.0	8.6
22	318	130	11.1	2.7	12.4
23	324	132	15.3	2.4	14.6
24	323	130	16.7	1.1	16.5
25	326	136	6.3	8.8	3.7
26	314	135	6.1	8.8	3.5
27	321	135	11.5	3.3	11.7
<u>Unfined Vent</u>					
28	320	90	3.8	2.7	11.5
29	322	90	5.7	>1.7	19.1

(Continued)

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TABLE 4
SUMMARY OF EXPERIMENTAL DATA FOR THE FREEZE VENT DEVICE
(Continued)

Run No.	Sodium Temperature (°F)	Vent Temperature (°F)	Top [†] of Sodium Column (in.)	Flow [§] Time (sec)	Sodium Flow Rate (fps)
Unfinned Vent (Continued)					
30	385	90	8.7	1.8	18.6
31	370	90	7.3	>1.7	19.1
32	430	130	8.0	8.2	3.2
33	425	132	7.8	6.7	4.3
34	425	133	9.6	2.9	11.1
35	420	125	16.2	2.5	12.5
36	410	143	21.2	2.8	12.0
37	430	140	24.3	4.6	7.2
38	413	140	12.9	7.9	3.6
39	428	142	14.1	>4.5	5.1
40	422	142	12.2	6.2	5.1
41	420	142	18.2	5.2	6.5
42	520	142	12.4	-	2.3
43	515	142	12.0	9.6	2.3
45	525	141	20.5	6.9	4.4
46	520	143	18.8	6.9	3.6
47	330	144	9.3	~10	2.9
48	300	141	10.1	6.6	3.7
49	300	140	13.8	4.9	6.8
51	310	89	7.3	2.2	16.3
53	300	90	7.3	4.3	7.5
54	300	90	6.6	3.9	8.5
55	300	90	5.7	4.6	6.5

*Level of sodium in the 0.055-in. annulus; total length of annulus is 27.5 in.

†Assuming flow in the repressure arm (gas lines at upper left in freeze vent system schematic) has stopped.

sodium up the column, until it again reaches a point where it freezes. Since the melting point of sodium is just over 208°F, the sodium seal will melt when the wall temperature of the vent adjacent to the seal reaches that value. This suggests that, when a seal is made on startup in the reactor system, it is desirable to have the seal as far up the vent section as possible. Ideally, the seal should originally be made so high up the vent that it would not melt out during operation. It has also been suggested that a valve in the upper end of the vent section would assure that sodium would not be pushed beyond the vent.

The third sequence of tests in the unfinned vent involved the application of pressure to the seal having a fixed temperature profile and an ambient temperature of 140°F. With an imposed pressure of 300 psi acting for a 24-hr period, no seal movement was detected. After the 300-psi test, the pressure was raised to 500 psi and held for 2 hr, again with no seal movement detected.

This can be explained as follows. When the seal is originally made, the sodium moves a significant distance beyond the point at which the temperature is 208°F. When it does stop flowing and the leading edge has solidified, the solidification progresses downward along the annulus toward the entrance. It stops at the point at which the metal temperature is 208°F. The result is a rather long frozen region, whose shear strength capability at the walls is much larger than the net force that is developed, even by a 500-psi pressure load. Therefore, so long as there is no change in the temperature profile along the seal from that present when the seal was formed, it is unlikely that a seal can be moved by the application of pressure.

The third series of tests was a study of the effect of contaminated sodium on the ability to break a seal, to simulate the problem of opening a vent after it has been in service for a long period, exposed to sodium having a modest oxygen impurity contamination level. The test was run with 0.4 gm of oxygen in the system. The oxygen was caused to diffuse to the seal region in the annulus, by heating the sodium to 1100°F while the freeze seal plug remained solid (<208°F). After 90 hr, the sodium was allowed to cool to 400°F. During this 90-hr period, the amount of oxygen which could have transferred along the annulus by concentration gradient diffusion was only a fraction of a milligram at most. Even with the relatively large concentration gradient used in the test, compared to FFTF conditions, the seal was broken very easily. Because it is

evident that very long time periods will be necessary to permit the diffusion of larger amounts of oxygen into the vent annulus, and because there was no indication in the first test of any impairment of the ability to melt and open the seal, further testing of this phenomenon is not warranted.

A fourth series of tests has begun, in which the seal is contaminated on the gas side. In the first test, a new seal was formed, and the gas space was evacuated and refilled with pure gaseous oxygen. After reacting for ~ 175 hr, ~ 12 mg of oxygen had apparently reacted with the exposed sodium surface in the annulus. This seal was broken easily by heating the vent to a temperature near 250°F . The time needed to break the seal was 7 min, with a current of 7 amp through the vent heater and a pressure difference across the vent of ~ 7 lb/in.². Within the limits of measurement, this result is the same as it is with contaminated seals. Additional tests will be run, in an attempt to provide a higher level of contamination of the freeze seal.

D. STANDARDS AND ORDERING DATA

CURRENT STATUS

Document	Review	Status	Responsible
E4-14 Vapor Trap Standard	HEDL & RDT	3rd draft submitted 8/17/71; Appendix D on filter design complete, but not yet sub- mitted to RDT or HEDL	AI
E4-13T Freeze Vent Standard	RDT	Submitted - Feb. 1971	LMEC
E11-2 Filter	HEDL	Comments received from HEDL - Sept. 1971	AI
HWS- Vapor Trap Ordering Data	HEDL	Received draft 9/20/71; document under review	HEDL
HWS-1118 Freeze Vent Ordering Data	HEDL	Submitted April 1971 to HEDL; HEDL will assume responsibility for final release	HEDL

One change has occurred since the last progress report. HEDL formally accepted responsibility for the completion of the ordering data for the vapor trap and freeze vent. This was originally AI's responsibility. AI still is required to provide the test information to be appended to the ordering data, and for the operating instructions and design drawings with the associated documentation.

An appendix to RDT Standard E4-14 was added to that document. It describes the experimentally derived design information for a continuous operation controlled-temperature-profile vapor trap, up to 1.0 scfm flow. Appendix A provides an empirical relation in the form of a reduced temperature profile curve that delineates the desirable operational temperature gradients along the packed bed. The curve is seen in Figure 6. In the vapor trap, sodium vapor is removed in the packed bed by the molecular diffusion of the sodium vapor to bed surfaces. Superheating of the argon gas in the preheater section of the vapor trap is specified, in order to vaporize any sodium droplets, thus ensuring

Figure 6. Reduced Temperature Profile

their condensation by molecular diffusion. For this reason, it is necessary that the temperature gradient at every point along the length of the trap be less than the critical temperature gradient for sodium fog formation.

An Appendix D has been prepared for addition to the RDT Standard E4-14. The appendix describes the design procedure for sizing the filter components of the vapor trap assembly. In this document, the three sources of sodium which must be removed from the argon stream leaving the vapor trap condenser are identified as follows:

- 1) The steady-state effluent sodium content
- 2) The upset sodium release (where upset means non-steady-state conditions)
- 3) The steady-state oxygen-induced sodium carryover (oxide particulates).

The sodium vapor in the steady-state operation is expected to be the worst offender, since it is probably sodium metal in the form of extremely fine particles. Such material will tend to be very adherent, to pack very well, and to plug filter openings with the least amount of sodium. Due to the different nature of each of these three types of sodium-bearing particles, loading factors are assigned for the purpose of estimating the amount of filter area that should be provided to accommodate their collection without plugging the filter during the maintenance period. These factors are tabulated in Appendix D for reference. Thus, the filter area required for a given service is the sum of the areas required to accommodate the three types of sodium loading.

III. IMPACT ON LMFBR PROGRAMS

The schedule for the 1-scfm vapor trap for the CRCTA requires procurement activities to begin in September 1971. The fabrication and test schedules now proposed appear to meet the schedule.

The delivery requirement for the 5-scfm vapor trap for FTR is scheduled for September 1972. The current schedule is devised to meet that timing and need.

The need for the unfinned freeze vent information for FFTF test facilities was satisfied with the data obtained during this period.

All the standards required for this program were issued on schedule in draft form, and have been submitted for review. There does not appear to be any pacing aspect of this part of the task. Revisions are complete on the Appendix for E4-14, the Vapor Trap to cover the condition of the 1.0-scfm requirement.

IV. NEXT REPORT PERIOD ACTIVITIES

MULTIPLE-BAFFLE VAPOR TRAP

Tests will be completed, and a decision made on the future of that trap.

CONTROLLED-TEMPERATURE-PROFILE VAPOR TRAP

Fabrication of the scaled-up 1-scfm flow rate vapor trap will be completed.

FREEZE VENT

Tests and a writeup will be completed on all the freeze vent work.

Program: Sodium Technology		
AEC Task: 1 - Sodium Cleaning and Requalification		
Project Manager: G. W. Meyers		
Reporting Period: July-September 1971		189A: 13625
General Order: 06507	Subaccount: 36100	Category: 04-40-04-04.1

Principal Investigators: J. L. Ballif, F. W. Poucher

I. PROJECT OBJECTIVES

The general objectives of this program are to develop methods and procedures for cleaning and requalification of systems and components after uses in sodium service and/or after a sodium leak or spill. The more specific tasks are:

- 1) The determination of the effect of sodium leaks or spills on reactor components (Leak Corrosion Rate)
- 2) The evaluation of methods for removing sodium or its reaction products, and the storage of components that have been exposed to a sodium environment (Sodium Cleaning and Storage)
- 3) The development of methods and procedures to requalify sodium components (Requalification Technology)
- 4) The evaluation of methods for sodium leak detection (Sodium Leak Detection).

II. ACCOMPLISHMENTS DURING FISCAL YEAR 1972

A. SUMMARY

The work plan was modified, and submitted to San Francisco Operations office, in accordance with the July 1971 review meeting held at AI.

Experimental apparatus has been assembled, and is being readied for laboratory testing.

A draft of a cleaning procedures guide, to define areas requiring technical backup, has been completed.

A draft of a sodium technology design guide, for cleaning maintenance and repair, has been completed.

Following the HEDL review held in July 1971, a revised work plan was submitted to AEC-SAN and to HEDL for additional comments. In general, HEDL concurred on the plan. Their specific suggestions related to the priority of testing, and to a method suggested for mechanical properties evaluation. AI suggested low-cycle fatigue or instrumented Charpy mechanical properties tests as alternatives to the stress-rupture tests originally proposed.

The assembly of the experimental apparatus for the leak corrosion rate work is nearly complete. Checkout and final welding of the system are underway. Test specimens have been prepared, and have been welded. The calibration of the leak remains to be done before experimental operations can commence.

A draft of a cleaning procedures guide has been completed, in order to help design-oriented people determine areas of need. The design guide helps clarify specific procedures to be considered, and indicates methods which are currently available. A list of questions has been received from the HEDL Engineering Department, after their examination of a preliminary draft of this document. These questions will aid in guiding this program toward the needs of the designer and component builder.

A draft of a sodium design guide has also been prepared. This document, coupled with the cleaning guide, should find use in reminding engineers about the practical considerations necessary in general design and in design for maintenance and repair. The relation to cleaning is that maintenance and repair start with cleaning, hence the ability to clean is of great importance.

III. IMPACT ON LMFBR PROGRAM

The work reported here represents only one segment of the work required to solve cleaning and requalification problems that impact directly on the radioactive engineering and maintenance tasks. The AEC's decision to develop a planned National Program on cleaning and requalification will eventually require a concerted program to meet the needs of the FFTF and subsequent reactors for proven standards and procedures to handle the cleaning and requalification tasks required in an LMFBR.

IV. NEXT REPORT PERIOD ACTIVITIES

The experimental test will be set up, and testing begun.

Initial assessment of leak detection methods accompanying the leak corrosion rate tests will be made.

Program: Sodium Technology

AEC Task: 1 - FFTF Support Work - Sodium Frost

Project Manager: G. W. Meyers

Reporting Period: July-September 1971

189A: 13625

General Order: 06507

Subaccount: 39810

Category: 04-40-04-04.1

Principal Investigators: R. M. Oliva, T. T. Shimazaki

I. PROJECT OBJECTIVES

- 1) Develop an analytical model of sodium oxide deposition on surfaces exposed to simulated FFTF reactor cover gas (argon), based on a typical LMFBR reactor operating cycle. This effort consists of the following parts:
 - a) Develop an analytical expression for the rate of formation and distribution of sodium oxide layers on a surface, due to reaction of a sodium film with free oxygen in the cover gas
 - b) Develop an analytical expression for the rate of removal of the sodium oxide deposit by refluxing sodium during reactor operation
- 2) Verify the conclusions of the analytical model by experimental test
- 3) Relate the verified model to design application of FFTF components susceptible to the sodium frost phenomenon (viz., vapor trap, rotating plug annuli, IVHM, and instrument tree).

II. ACCOMPLISHMENTS DURING FISCAL YEAR 1972

A preliminary experimental loop system checkout run on the rate of formation of sodium oxide on sodium-coated surfaces was made at an oxygen pressure of 245 torr and a temperature of 27°C. The partial pressure of water was 2.25 torr. The results are shown in Figure 7. About 5% of the sodium was reacted in 100 min. The data shown in Figure 7 can be expressed as

$$x = 0.077 + 0.113 \log \theta ,$$

Figure 7. Reaction Rate [Na(s), O₂(g), H₂O(g)]

where:

x = weight increase (mg/cm^2)

θ = time (min).

These results indicate that the oxidation rates can be adequately measured by the weighing technique employed. A uniform coating of sodium on the stainless steel tab was not obtained in this run, so no attempt was made to measure the change in coating thickness. The data obtained in the preliminary checkout run indicated that the experimental test system performed satisfactorily. This puts the program on the threshold of starting the actual test matrix specified for the sodium oxide experiments.

Further testing activity was temporarily halted at the end of July 1971, due to a transfer of funds to higher priority tasks. Therefore, Project Objectives 2 and 3 have not as yet been met. The analytical studies described in Project Objective 1 were completed, and reported in TI-707-350-002, but have yet to be verified by experimental tests.

Concern has recently arisen as to the potential problems in the FTR head region, where uncontrolled formation of sodium and sodium oxide deposits cannot be tolerated. Some of the more important areas are the reflective thermal shielding, energy-absorbing plates, IVHM and instrument tree plugs, and the annuli below the omega seal. AI has developed analytical models to predict the formation of sodium and sodium oxide deposits in the cover gas region. These models will now be applied and/or further developed, as required, to fit the FTR geometry under a "redirected" scope of work under this same subaccount. Negotiations and liaison between AI-HEDL are in progress to establish the specific test objectives and the extent of analytical effort, but it is generally agreed that the scope of effort will concentrate on studying the reflective, thermal shield structure.

III. IMPACT ON LMFBR PROGRAMS

Refined analytical models of sodium oxide formation and dissolution rates on surfaces exposed to LMFBR cover gas will, in general, permit determination of adequate design and/or required operating conditions (purge rate, shutdown temperature, etc.) for control of sodium frost. This program, in

particular, will now concentrate on analytical calculations of the formation of sodium and sodium oxide deposits in the reflective, thermal shield regions of the FTR head, and its effect on the performance of the thermal shield.

The model of sodium oxide formation rate may also be applicable to sodium oxide deposition in sodium vapor traps, and therefore can be used to aid in the interpretation of sodium vapor test data.

IV. NEXT REPORT PERIOD ACTIVITIES

AI will apply previously developed AI models, in combination with sodium oxide models recently developed under the AEC program: (1) to predict the formation of sodium and sodium oxide deposits in the reflective, thermal shield regions of the FTR head, and (2) to indicate its effect on the performance of the thermal shield. Two concepts of the reflective thermal shield will be analyzed [viz., uncanned plates (Combustion Engineering design), and canned plates (AI design)].

In order to expedite the early evaluation of the sodium frost problem in the thermal shield region, simplified equations will be used for preliminary calculations. These simplified equations will exclude the effects of sodium fog formation in the upper lateral leg and sodium fog evaporation in the cold leg of convective cells, the effects of unequal annuli wall temperatures, and will be discontinuous at very low convection rates. They will also exclude the effects of fog formation and evaporation on the gas density, which affects the convection rate. A preliminary report will be issued at the completion of these preliminary calculations. These preliminary calculations will tend to result in information which is more optimistic than the actual case. A more detailed calculation will then be made (but probably will not be started during the next report period) to include the refinements excluded from the analysis in the preliminary calculations.

Program: Sodium Technology		
AEC Task: 1 - FFTF Support Work - Friction Tests		
Project Manager: G. W. Meyers		
Reporting Period: July-September 1971		189A: 13625
General Order: 06507	Subaccount: 21100	Category: 04-40-04-04.1

Principal Investigators: W. J. Kurzeka, R. M. Oliva, F. W. Poucher

I. PROJECT OBJECTIVES

The objective of this program is to conduct screening tests to measure the breakaway friction and sliding friction coefficients of various materials and/or material combinations, when used in an environment of high-temperature, high-purity liquid sodium. These materials, which will be selected by HEDL, are to be evaluated for possible use as FFTF driver duct wear pads.

II. ACCOMPLISHMENTS DURING FISCAL YEAR 1972

Atomics International was authorized, in August, to proceed with a friction test to specifically support material selection for the FFTF fuel duct wear pads. These tests, which will determine the friction coefficients of various material "couples," will be run in a specially designed test rig that provides environmental conditions of sodium purity, flow, and temperature, along with friction surface load, geometry, surface finish, and sliding speed.

A review of the FFTF Fuel Driver Duct Pad Friction Test Program Plan among AI-HEDL-ARD was held on 8/12/71. Written agreement was reached among the three organizations as to: (1) the specific test program to be followed, (2) matrix of test couples, (3) quality control and documentation of the materials, (4) sodium loop and test apparatus operating procedures, (5) specimen design, (6) schedule, and (7) scope of work and responsibilities of each organization.

A Work Plan, based on the agreements noted previously, was written and released. A Test Plan (TP-707-240-002) was released; a detailed test procedure is in preparation.

Drawings of the friction test specimens (riders and plates) were prepared and released. HEDL furnished the 20% cold-worked Type 316 stainless steel material (1/2- and 1-in. diameter rod) required for the specimens; other materials were ordered by AI. Special heat treat procedures for Inconel 718 were also provided by HEDL.

Sufficient test couples, specimen adapters, coating discs, "practice" discs, and special coating holding fixtures were fabricated by AI to complete a full loading (first of three) of the AI 18-station test apparatus. Test articles requiring coating were transported by a HEDL representative to Mech Electron and to Linde (both in Los Angeles), where specimen coating with various materials was directed and supervised by HEDL.

The results of a very detailed dimensional surface finish and weight determination were recorded, prior to coating the specimens. The post-coating inspection and final cleaning has also been completed, and specimen installation into the test apparatus has begun.

Testing is scheduled to start on the first test matrix, the first part of October. Fifteen of the test couples will comply with the matrix agreed upon during the AI-HEDL-ARD meeting on 8/12/71. The materials and/or desired coatings for the remaining three could not be obtained without undue schedule delays; so HEDL specified, and will officially document, specimen substitutions.

The remaining test specimens and parts that could be identified with the second and third matrix have been fabricated.

III. IMPACT ON LMFBR PROGRAMS

The results of this program are needed for the selection of materials for the driver ducts for FFTF and FBR's. The results also affect the method and forces required to clamp a core after refueling.

This program will also support LMFBR technology by providing friction (breakaway and sliding) data for various materials and/or material combinations when used in a high-temperature (400 to 1160° F), high-purity (<5 ppm) sodium environment under surface contact pressures of 300 and 800 psi.

IV. NEXT REPORT PERIOD ACTIVITIES

The first test matrix of 18 friction couples will be installed in the test apparatus, and subjected to the specified test spectrum. As friction data are generated, HEDL will finalize the matrix for the second group of 18 specimens. Fabrication and/or coating, plus a detailed inspection, of these new specimens will proceed concurrently with the first test, so that a complete set of couples is ready and waiting for installation upon conclusion of the first test. Likewise, specimen selection and fabrication for the third set of 18 will be carried out during the second test. Both the first and second test matrices will be tested and evaluated the next quarter; the third and final group will undoubtedly extend into the following report period.

Upon completion of a test series, selected friction couples will be re-examined by inspection to determine dimensional and weight changes. Photomacrographs, light metallographs, and electron micrographs will be made, as required, to assist in the metallurgical examination of the wear surfaces.

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Program: Sodium Technology		
AEC Task: 1 - Cover Gas Analysis by Molecular-Beam Mass Spectrometry		
Project Manager: G. W. Meyers		
Reporting Period: July-September 1971	189A:	13625
General Order: 06507	Subaccount: 35100	Category: 04-40-04-04.1

Principal Investigators: H. Farrar IV, G. Ervin Jr.

I. PROJECT OBJECTIVES

The objectives of this task are to: (1) provide a rapid and accurate method of determining low concentrations of impurities in the cover gas of the FFTF and LMFBR's on a continuous basis, using a commercially available quadrupole mass spectrometer with a modulated molecular-beam source, (2) develop a sampling system that will bring a representative sample of the cover gas at an elevated temperature to the mass spectrometer at a flow rate adequate for high sensitivity and fast response, and (3) evaluate the whole system, with and without the presence of sodium vapor, and prepare plans to integrate it with an appropriate reactor system.

II. ACCOMPLISHMENTS DURING FISCAL YEAR 1972

A. SELECTION OF A MASS SPECTROMETER SYSTEM

Earlier considerations in this program have led to the conclusion that a modulated molecular-beam-source quadrupole mass spectrometer will provide the necessary speed and sensitivity for the analysis of impurities in noble cover gases. Such a system was under development for this purpose at LASL, and now is available commercially as the EMBA-II, manufactured by Extranuclear Laboratories, Pittsburgh. The system is capable of sampling gas mixtures at pressures ranging from <1 torr to >1 atm, using a simple orifice inlet. Furthermore, the system should not suffer from problems experienced by HEDL in obtaining a representative gas sample which includes sodium vapor, which itself should easily be detectable.

One of the advantages of using the molecular-beam mass spectrometer is that the beam makes a clean pass through the ionizer without suffering any

collisions. This means that there is no possibility of thermal degradation. Also, since there is complete thermal decoupling between the gas source and the mass spectrometer, there are almost no restrictions on temperature or pressure of the gas to be sampled. This method permits analysis of not only chemically stable gases, but also gases containing free radicals, internally excited atoms and molecules, and condensable species such as sodium. The EMBA-II also makes use of modulation of the molecular beam; and analysis of the phase permits almost complete separation of background from sample ion beam. It is obvious that these capabilities are ideally suited for direct hot cover-gas analysis, as long as a means can be developed to transport a representative sample of gas to the instrument.

Observation of the EMBA-II in operation, during a visit to Extranuclear Laboratories in Pittsburgh, showed that it is a quality research instrument with wide capabilities and high sensitivity. The present configuration has rather a large number of controls. By proper adjustment, the peaks become smooth, and relatively free from noise. The mass spectrum obtained by analyzing laboratory air was, as expected, quite complex. When the instrument was operated at maximum sensitivity, there were peaks (mostly small) at almost every mass number examined (up to ~ 150 amu). The problem, under these conditions, is that not only was the multitude of gases and hydrocarbons in air being examined, but also these same gases tended to be those occurring as general background in the mass spectrometer. As a result, it was not always easy to be completely sure of the origin of all mass peaks. When the controlled inlet system planned for the preliminary tests is used, on the other hand, the number of gases permitted to enter will be dramatically reduced, and the resulting mass spectrum will be greatly simplified. It will be during these tests that the effective sensitivity of the EMBA-II will be determined.

In order to differentiate between signal and background, the mass spectrometer must be scanned at a rate which allows several modulated cycles to elapse on the top of each peak. Because of this, a scan from masses 1 to 150 will normally require ~ 5 min. This time could be partly reduced by peak switching, or bypassing mass ranges where no peaks generally occur. Such a mode of operation would have to be chosen carefully, so as not to compromise the ability of the system to analyze for all possible impurity gases.

As far as can be told from the tests conducted so far, the EMBA-II appears suitable for the task of analyzing reactor cover gas at temperature. Although it basically meets all the sensitivity requirements, its complexity, and the need to operate near the limits of sensitivity, indicate that a significant amount of development will be needed to make this an easy-to-operate on-line instrument, capable of performing routine cover gas analyses.

B. DESIGN OF A SAMPLE INLET SYSTEM

Some time has been spent examining possible ways of bringing the cover gas to the mass spectrometer on a continuous basis. One of the most important reasons for analyzing the cover gas "at temperature" is to prevent the sodium from condensing, thereby scavenging an undetermined fraction of the impurities of interest. As an alternative to maintaining the sample line at $\sim 850^{\circ}\text{F}$, the possibility of reducing this temperature while lowering the gas pressure within the line has been explored. If the pressure in the gas line were to be reduced from 1 atm to 1 torr, using a small orifice at the reactor end of the tube, the sodium vapor would remain above its dew point as long as the temperature were kept above $\sim 470^{\circ}\text{F}$. This means that the line would need to be maintained at only $\sim 470^{\circ}\text{F}$ to prevent the sodium from condensing. Under these circumstances, the gas line would also serve as a convenient first pump-down stage for the mass spectrometer source. Reducing the pressure to 1 torr would have the added advantage of making it possible to increase the diameter of the orifice of the mass spectrometer source to 0.4 mm, thereby reducing the possibility of blockage while maintaining optimum mass spectrometer molecular-beam size. The amount of gas taken from the reactor by the line, in this case, is governed only by the diameter of the first orifice. Calculations show that a diameter of 0.5 mm would result in a circulation rate of ~ 0.08 scfm. Using a rotary vacuum pump, the pressure within a 100-ft long, 2-in. diameter line would drop from 1 atm to ~ 1.6 torr at the first orifice, and then to 1 torr along the length of the pipe. Transit time for the gas through this pipe would be in the order of a few seconds. These conditions appear to be compatible with the EMBA-II design, and would result in a molecular beam of optimum size.

Theoretical calculations of the limits of detection of the mass spectrometer have been reviewed with the management of Extranuclear Laboratories. The

6507-1817

Figure 8. Apparatus for Sampling Argon Over Hot Sodium

general picture is that sensitivity is quite unaffected over a wide range of source parameters, including beam-forming geometry, pumping speeds, operating pressures, and orifice sizes. The sensitivity of the EMBA-II is ultimately limited by the accidental modulation of background gases in the mass spectrometer, which can therefore not be distinguished from the modulated sample beam. Although most background appears as a dc output which can be electronically eliminated, a certain fraction of the background atoms are "pushed" through the source by the stream of sample atoms at the modulated frequency. To minimize this phenomenon, and thereby optimize sensitivity, a satisfactory vacuum ($\sim 10^{-4}$ torr) must be maintained in the chamber which forms the molecular beam. To accomplish this, a compromise must be made between the size of the vacuum pumps (400 l/sec was used on the demonstration instrument) and the size of the orifice, which should be sufficiently large to preclude blockage by deposits or particulates.

C. CONSTRUCTION AND TESTING OF A PRELIMINARY SAMPLING SYSTEM

One of the principal problems encountered by HEDL in development of a mass spectrometer system for cover gas analysis was their apparent inability to observe sodium in their hot gas sample, even though the sodium vapor concentration should have been significant. To dispel any doubts that this problem might affect the proposed sample inlet system for the molecular-beam mass spectrometer, a simple test has been devised. Figure 8 shows an apparatus that has been designed and built to demonstrate that sodium vapor can be transported at reduced pressure through hot lines and orifices, and on into a vacuum chamber equivalent to the first stage of the EMBA-II molecular-beam mass spectrometer. The system is constructed from Type 304 stainless steel, and has a pot containing sodium and argon at slightly greater than atmospheric pressure. There are two pumping stages, connected by orifices, and the entire line and the orifices are operated at elevated temperature. Although the apparatus is intended to fit directly on the EMBA-II source, the first tests will take place without the use of a mass spectrometer. A molecular beam of argon, containing sodium vapor will be produced at the heated second orifice, and this beam will strike a collector disk which will be subsequently analyzed for the sodium condensed on it. The amount of sodium collected will then be correlated with source pot temperature, line pressure, and time.

After completion of the preceding tests of the sampling apparatus, it will be transported to Extranuclear Laboratories, in Pittsburgh, for tests on their operating EMBA-II mass spectrometer. A decision on whether to procure an EMBA-II will be based on these tests.

III. IMPACT ON LMFBR PROGRAMS

The on-line monitoring of the cover gas should provide early indications of leakage into the sodium of organic materials, and should provide measurements of leakages of shield seals and fuel elements. The ability to do this directly, at elevated temperature, removes the uncertainties of scavenging of impurities of interest by the condensing sodium.

All indications have thus far confirmed the potential value of the molecular-beam mass spectrometer for this application. Observations of such an instrument in operation have pointed up its complexity, and the need for a development period lasting several months to simplify its operation. It is possible that this could affect the timetable for its availability to an on-line system.

IV. NEXT REPORT PERIOD ACTIVITIES

Atomics International has been directed to defer further work on this project.

Program: Irradiation Damage in Cladding and Core Structural Materials			
AEC Task: 2 - Irradiation Damage			
Project Manager: G. W. Meyers			
Reporting Period:	July-September 1971	189A:	13495
General Order:	06506	Subaccount:	29621, 29624
		Category:	04-40-02-01.1

Principal Investigators: K. R. Garr, D. W. Keefer, D. Kramer, A. G. Pard

I. PROJECT OBJECTIVES

- 1) Study the process of void formation and helium embrittlement in fuel cladding and core structural alloys, by means of accelerator irradiations and reactor irradiations
- 2) Determine the effects of fluence, temperature, helium concentration, and microstructure on void formation and helium embrittlement
- 3) Determine the effect of stress on void formation
- 4) Perform preliminary evaluations of modified and new alloys developed by other laboratories.

II. ACCOMPLISHMENTS DURING FISCAL YEAR 1972

A. VOID FORMATION IN TYPE 316 STAINLESS STEEL

The study of swelling as a function of damage proceeded, with the examination of samples irradiated at 400 and 600°C. The results are presented in Table 5, along with previous results at 600°C, and a plot of swelling as a function of damage is shown in Figure 9.

Swelling appears to increase with temperature, from 400 to 600°C, for equivalent amounts of damage. There is a trend for the average void size to increase with temperature for a given damage, and a trend for the void density to decrease with temperature for a given damage rate. Examples are shown in Tables 6 and 7. It should be noted that the data so far obtained at 400 and 600°C show more scatter than at 500°C.

6506-4714

Figure 9. Swelling as a Function of Damage for Type 316 Stainless Steel

TABLE 5
VOID FORMATION AT 400 AND 600° C

Sample No.	Temperature (°C)	Helium (appm)	Damage (d/a)	Damage Rate (d/a-sec)	Void Density (/cm ³)	Average Void Diameter (Å)	Volume Increase (%)
79	400	4.5	2.3	2.9×10^{-4}	9.4×10^{15}	106	0.65
79	400	4.5	9.2	1.2×10^{-3}	7.9×10^{15}	93	0.46
47	400	5.8	18	6.9×10^{-4}	1.9×10^{16}	110	1.9
80	600	4.5	5	1.0×10^{-3}	7.7×10^{14}	375	3.5
38	600	5.8	16	6.8×10^{-4}	1.6×10^{15}	590	20
38	600	5.8	21	8.6×10^{-4}	2.0×10^{15}	590	24

Treatment: 1 hr at 980° C + 8 hr at 760° C

TABLE 6
EFFECT OF TEMPERATURE ON VOID SIZE

Temperature (°C)	Damage (d/a)	Average Void Diameter (Å)
400	18	110
500	15	260
600	16	590

TABLE 7
EFFECT OF TEMPERATURE ON VOID DENSITY

Temperature (°C)	Damage Rate (d/a-sec)	Void Density (/cm ³)
400	6.9×10^{-4}	1.9×10^{16}
500	7.0×10^{-4}	3.0×10^{15}
600	6.8×10^{-4}	1.6×10^{15}

Figure 10. Voids in Type 316 Stainless Steel, Proton Irradiated at 400°C to 18 d/a (Transmission-electron micrograph) 1-188

Figure 10 illustrates the high density of small voids that form at 400° C. Neither sample that was irradiated at 400° C was magnetic, indicating that little or no ferrite was present. Previous irradiations at 500 and 600° C resulted in some transformation of austenite to ferrite.

B. DISPLACEMENT CALCULATIONS

The atom displacement calculations used for proton irradiations are based on the damage-production model of Kinchin and Pease (K-P).^{*} In their formulation, it was assumed that the scattering events involving displaced atoms (knock-ons), and atoms of the lattice, were hard sphere in character. That is, the atom-atom interactions were assumed to be screened, long range, and of low energy. Since the number of secondary and higher-order displacements depends on the type of atom-atom interaction operating, it is necessary to know the nature of the interaction. A simple calculation, in which the closest distance of approach is equated to the screening radius of the old Bohr theory, gives an approximate "border-line" energy for the transition from hard sphere to Rutherford scattering. For iron, this knock-on energy is $\sim 5 \times 10^4$ eV. It is now necessary to calculate the maximum energy, T_m , which can be transferred to an iron atom by an incoming proton of energy E . From the usual expression for T_m , we find that, for this case, $T_m = 6.95 \times 10^{-2} E$ (eV). In the current irradiations, the incident proton energy has been 1.0 Mev or less, in which case, $T_m \lesssim 7 \times 10^4$ eV. Since the average transfer energy is considerably less than this, and since the microscope foils are taken at proton penetrations corresponding to proton energies of ~ 0.6 , 0.45 , and 0.12 MeV, it is clear that the vast majority of primary knock-on atoms possess energies well below 5×10^4 eV. Therefore, the K-P assumption of hard sphere scattering is justified. Inclusions of a "hybrid" scattering law, to take account of "border-line" energy transfer events, would have almost no effect on the calculated number of displacements.

A second important assumption made in the K-P model is the following: If the energy of a knock-on is sufficiently low, so that its velocity is less than that of an orbital electron, all energy loss by that knock-on is by elastic nuclear processes. Since, in fact, inelastic losses to electrons do continue to

^{*}G. H. Kinchin and R. S. Pease, "The Displacement of Atoms in Solids by Radiation," Reports on Progress in Physics, 18 (1955) pp 1-51

occur at low energies, the importance of the inelastic loss process must be determined. Obviously, the K-P assumption might tend to overestimate the number of additional displacements created by knock-ons. Lindhard and co-workers* have made detailed calculations of the stopping cross sections for both electronic and nuclear interactions. Their results are included in the calculations that follow. The correction term for electronic interactions is found to be small.

In the K-P model, it is also assumed that energy is dissipated via random collisions. Therefore, the possibility of channeling and focusing processes is not taken into account. Because of the relatively low energies of primary knock-ons created in the proton irradiations, channeling events can safely be ignored. Focusing probably does occur; and, since such a process would lead to somewhat fewer than the expected number of displacements, it is possible to artificially account for it by raising slightly the threshold energy, E_d , for displacement production. However, an accurate E_d value for the irradiated alloys is not known, and rather arbitrary adjustments from the estimated value of 25 eV seem fruitless. In any case, the effect of focusing is expected to be very small.

Finally, the K-P model allows for no mutual recombination of interstitials and vacancies. Clearly, because of the mobility of both interstitials and vacancies at the irradiation temperatures, recombination may be of considerable importance. However, the solution to this problem must await the application of computer techniques. In the meantime, one can only ignore recombination, and make calculations which are appropriate only for 0°K. (Spontaneous recombination of close pairs probably occurs, even at this temperature, but its extent is not known.)

The total number of displacements per atom, $N_d(E)$, produced by a fluence of ϕt particles of energy E is given by

$$N_d(E) = \phi t \int_{E_d}^{T_m} d\sigma(T) \cdot n(T) \quad \dots (1)$$

*J. Lindhard, M. Scharff, and H. Schiott, "Range Concepts and Heavy Ion Ranges," Danske Vid. Selsk, Mat.-Fys. Medd., 33 No. 14 (1963) pp 1-42

In Equation 1, $d\sigma(T)$ is the differential cross section for transferring energy in the interval T to $T + dT$. For protons in the energy range of interest to us, the Rutherford scattering law holds, and we can write, for protons incident on iron,

$$d\sigma(T) = 4\pi a_o^2 Z_p^2 Z_{Fe}^2 \frac{M_p}{M_{Fe}} \frac{E_R^2}{E} \frac{dT}{T^2} \quad \dots (2)$$

In Equation 2, a_o is the Bohr radius of the hydrogen atom, and Z_p , M_p and Z_{Fe} , M_{Fe} are the charge numbers and masses of protons and iron atoms, respectively. E_R is the Ryberg energy, 13.6 eV. With the appropriate numerical values, Equation 2 becomes

$$d\sigma(T) = 7.88 \times 10^{-3} \frac{1}{E} \frac{dT}{T^2} \quad \dots (3)$$

In Equation 1, $n(T)$ is the total number of displacements per primary recoil atom of energy T . In the interval $E_d \leq T \leq 2E_d$, the primary is incapable of displacing a second atom, so $n(T) = 1$. In the interval $2E_d \leq T \leq T_m$, we let

$$n(T) = f(T) \frac{T}{2E_d} \quad \dots (4)$$

where $f(T)$ is a factor introduced to include the loss of recoil atom energy in inelastic collisions. From Lindhard et al.,* $f(T)$ is the fraction of primary energy which is dissipated in elastic collisions,

$$f(T) = \frac{S_n}{S_n + S_e} \quad \dots (5)$$

where S_n is the cross section for elastic nuclear stopping, and S_e is the cross section for inelastic electronic stopping. S_n is independent of particle energy,

*ibid.

and has a value of $3.55 \times 10^{-25} \text{ cm}^2/\text{atom}$. $S_e = CT^{1/2}$, where $C = 4.15 \times 10^{-28} \text{ cm}^2\text{-eV}^{-1/2}$.

From Equations 1, 3, 4, and 5,

$$N_d(E) = 7.88 \times 10^{-13} \frac{\phi t}{E} \left[\int_{E_d}^{2E_d} \frac{dT}{T^2} + \frac{S_n}{2E_d} \int_{2E_d}^{T_m} \frac{dT}{T(S_n + CT^{1/2})} \right].$$

Integrating and putting in the limits,

$$N_d(E) = 3.94 \times 10^{-13} \frac{\phi t}{E \cdot E_d} \left\{ 1 + \ln \frac{T_m}{2E_d} - 2 \ln \left[\frac{S_n + CT_m^{1/2}}{S_n + C(2E_d)^{1/2}} \right] \right\}, \quad \dots (6)$$

where E_d is assumed to be 25 eV.

Equation 6 is the expression used in calculating the displacements in proton irradiated samples. The third term in the braces in Equation 6 is the correction term for electronic stopping; it amounts to <10% correction, at even our highest proton energy, so one is left with essentially the old K-P result.

The energy of a proton, as a function of its initial energy and depth of penetration into stainless steel, can be obtained from the tabulations of Janni.* The energy at selected penetration distances is then used in Equation 6 to obtain the number of displacements per unit proton fluence.

C. VOID FORMATION IN TYPE 316 STAINLESS STEEL + 0.2 Ti

Samples of Type 316 stainless steel + 0.2 Ti from ORNL were proton irradiated in the annealed and cold-work condition, and the irradiation conditions are shown in Table 8. Pre-irradiation examination of this alloy revealed a high density of fine precipitates (most probably carbide particles) in the grain

*J. F. Janni, "Calculations of Energy Loss, Range, Pathlength, Straggling, Multiple Scattering, and the Probability of Inelastic Nuclear Collisions for 0.10 to 1000 MeV Protons," AFWL-TR-65-150 (September 1966)

TABLE 8
PROTON-IRRADIATION SAMPLES OF TYPE 316 STAINLESS
STEEL + 0.2 Ti

Sample No.	Temperature (°C)	Helium (appm)	Damage (d/a, max.)	Treatment
99 T	600	4.6	10	1 hr at 1050°C + 12 hr at 650°C*
102 T-20	600	4.8	10	1 hr at 1050°C + 20% CR + 12 hr at 650°C*

*The time at 650°C is a result of bonding the sample to copper.

boundaries of the annealed sample, whereas they were absent in the cold-worked sample. Some precipitation was also observed in the matrix of the annealed sample, and possibly there is matrix precipitation in the cold-worked sample as well.

Voids were observed after proton irradiation in both the annealed and cold-worked samples, where the damage was 9.2 d/a; and, in both cases, void formation was quite inhomogeneous. In the annealed sample (99T), void formation varied from regions where there are high densities of small voids to regions where the opposite is the case. Difficulty in identifying many features as voids or artifacts has prevented meaningful void counting to date.

In the cold-worked sample (102T-20), only ~25% of the volume exhibited resolvable voids which occurred in random patches. Figure 11 shows two regions of the sample, one with and one without voids. In the regions with voids, densities ranged from 4×10^{14} to $2 \times 10^{15}/\text{cm}^3$, and the average void size ranged from 270 to 350 Å. The swelling for 9.2 d/a is estimated to be <1%. This is about one-tenth as much swelling as observed for ordinary annealed Type 316 stainless steel (see Figure 9) at 600°C. At this time, it is not known whether the reduced swelling is due to cold work, titanium, or a combination of both.

a. Region Where Voids Are Present 1-187

b. Region Without Voids 18-186

Figure 11. Cold-Worked Type 316 Stainless Steel + 0.2 Ti, Proton Irradiated at 600° C to 9.2 d/a (Transmission-electron micrographs)

AI-AEC-13015

D. EBR-II IRRADIATION EXPERIMENTS

Two capsules from X034, and one from X035, were opened at the AI Hot Laboratory hot cell. The samples were cleaned of sodium, and sorted by alloy. They were all in good condition, and the Type 316 stainless steel samples were removed from the hot cell and sent to the laboratory for examination and testing. Table 9 summarizes the irradiation conditions, obtained from ORNL.

A preliminary observation in the electron microscope of a sample of Type 316 stainless steel from X035, solution annealed for 1 hr at 980°C, showed the presence of voids between 100 and 200Å in size. Samples of Type 316 stainless steel from X035 in each of the four pre-irradiation heat treatments were tested for their attraction to a magnet, as a crude measure of the presence of ferrite. The observations are shown in Table 10, indicating that some ferrite may have been produced as a result of the irradiation.

Preparations have begun for the insertion of two capsules in X100, to replace two capsules scheduled to be removed in the next quarter. Each capsule is to be irradiated at 500°C, one to a fast fluence of 2×10^{22} n/cm², and the other to 3×10^{22} n/cm². The capsules will contain tensile samples of Type 316 stainless steel and Sandvik 12R72, plus a variety of alloy samples for electron microscopy. These alloys are listed in Table 11, and each alloy will be represented by samples with different heat treatments and with cold work.

E. THE EFFECT OF STRESS ON SWELLING OF TYPE 316 STAINLESS STEEL

A new experiment has begun, whose aim is to determine the effect of a tensile stress on swelling during proton irradiation. Two methods are being explored, one which utilizes a rotating tubular sample and another which utilizes a flat sample. Both methods seek to eliminate unknown and uncontrolled stresses that might arise, due to diffusion bonding of the sample to a copper heat sink. To do this, a liquid metal, which will be sodium or NaK for the tubular sample and tin or indium for the flat sample, will be used as a heat transfer medium. Tensile stresses will be applied by deadweight loading.

Three techniques are being investigated for the removal of heat from a tubular sample during proton irradiation. These are: (1) use of a heat pipe, with sodium as the working fluid, (2) use of a closed condenser system, using

TABLE 9
IRRADIATION CONDITIONS

Capsule	Subassembly	Fluence (n/cm ² > 0.1 MeV)	Temperature (°C)	
			Begin	End
1 and 2	X034	4.3 x 10 ²²	690	830
3	X035	2.3 x 10 ²²	450	not available

TABLE 10
ATTRACTION OF TYPE 316 STAINLESS STEEL
SAMPLES FROM X035 TO A MAGNET

Pre-irradiation Treatment	Irradiated Sample	Control Sample
1 hr at 980° C	Very weak	None
1 hr at 980° C + 8 hr at 760° C	Weak	None
50% CR + 100 hr at 760° C	Moderate	Weak
25% CR + 24 hr at 480° C	Weak	-

TABLE 11
ELECTRON MICROSCOPE SAMPLES IN CAPSULES 7
AND 8 FOR X100

Austenitic Alloys	Nickel-Base Alloys	Tantalum Alloys
316 A286	Inconel 718	Ta
321 NSMC Mo + Ti	PE 16	T-111

sodium, and (3) use of circulating NaK. Calculations have shown that all three techniques are capable of maintaining the sample at constant temperature, with acceptable small gradients during irradiation. However, practical difficulties with a heat pipe may eliminate this method from further consideration.

Tests are in progress to determine the compatibility of Type 316 stainless steel with tin and indium. Samples have been bonded to nickel-plated copper by liquid tin, and held at 500°C for several hours. Metallographic examination is in progress to evaluate the bond and to detect any attack of the Type 316 stainless steel by tin.

F. EFFECT OF CARBON ON TENSILE BEHAVIOR OF TYPE 316 STAINLESS STEEL

Samples of a special low-carbon and high-carbon Type 316 stainless steel were obtained from HEDL, and injected with helium. Tensile-test results at 700°C for the low-carbon Type 316 stainless steel are presented in Table 12, along with previous results for ordinary Types 316 and 316L stainless steels. All samples were solution annealed for 1 hr at 980°C, and precipitation annealed for 8 hr at 760°C, prior to helium injection.

TABLE 12
TENSILE-TEST RESULTS FOR TYPE 316 STAINLESS STEEL
AT 700°C

Carbon (wt %)	Helium (appm)	Strength (kg/mm ²)		Elongation (%)	
		Yield	Tensile	Uniform	Total
0.06	0	9.5	22.5	22	45
(316)	40	8.0	22.5	12	15
0.025	0	11.3	25.7	26	50
(316L)	20	10.4	24.9	22	26
0.003	0	11.4	24.6	23	53
(316 HEDL)	30*	12.4	21.3	23	33

*Nominal value

TABLE 13
CREEP RESULTS AT 700°C FOR AN INITIAL STRESS OF 10 kg/mm²

Sample	Helium (appm)	Failure Time (hr)	Creep Strain (%)				Secondary Strain Rate (10 ⁻⁴ /hr)
			Primary	Secondary	Tertiary	Total	
C-27	0	428	1	5	6	12	1.8
C-28	0	469	2	3	6	11	1.2
22-20	30*	299	2	1	3	6	1.3
22-21	30*	203	2	1	3	6	1.5

*Nominal

Treatment: 1 hr at 980°C + 8 hr at 760°C + 22% CR.

Figure 12. Effect of Helium on Total Elongation
of Type 316 Stainless Steel + 0.2 Ti

In the absence of helium, the uniform elongation does not appear to be sensitive to the carbon content, whereas the total elongation increases as the carbon content decreases. Helium markedly reduces the uniform elongation of Type 316 stainless steel, but has no effect on the very-low-carbon Type 316 stainless steel. The total elongations are all affected by helium, but the effect is smallest in the very-low-carbon Type 316 stainless steel.

Although the helium contents may not be the same, past experiments have shown that elongations are sensitive to only decade changes in helium contents. The explanation of these results involves the morphology of carbide precipitates on the grain boundaries. It appears that their absence from the grain boundary, as is probably the case when the carbon content is very low, renders Type 316 stainless steel more resistant to helium embrittlement.

G. DUCTILITY OF TYPE 316 STAINLESS STEEL + 0.2 Ti

Samples of Type 316 stainless steel + 0.2 Ti from ORNL (see Section II-C) were injected with helium, and tensile tested between 500 and 800°C, in vacuum, at a strain rate of 0.02/min. The uniform elongation remained unchanged by the presence of helium, whereas the total elongation was affected above 600°C, as shown in Figure 12.

The addition of titanium has prevented any loss of uniform elongation, and reduced the loss of total elongation caused by helium in ordinary Type 316 stainless steel (see AI-AEC-12670). The total elongation at 700°C for the annealed condition may be compared with ordinary Type 316 stainless steel in Table 12; the uniform elongation of Type 316 stainless steel + 0.2 Ti at 700°C is 27%, with or without helium.

H. CREEP OF TYPE 316 STAINLESS STEEL

Four cold-worked samples, two with helium and two without, were creep tested to failure at 700°C. The data are shown in Table 13, and two creep curves are plotted in Figure 13. The duration of the second and third stages of creep are reduced, resulting in an overall 50% reduction in strain at failure. Cold work decreases the strain at failure and the secondary creep rate, for samples with or without helium, as compared to samples with the same heat treatment (see AI-AEC-13009).

71-S8-43-41

Figure 13. Effect of Helium on Creep of Cold-Worked Type 316 Stainless Steel

I. PUBLICATION

"The Effect of Helium on the Creep of Type 316 Stainless Steel at 700°C,"
K. R. Garr, C. G. Rhodes, and D. Kramer, AI-AEC-13009 (September 1971)

III. IMPACT ON LMFBR PROGRAMS

Swelling of annealed, helium-injected, and proton-irradiated Type 316 stainless steel increases with temperature at the same dose (displacements/atom, d/a), over the range from 400 to 600°C. It is concluded that the temperature of maximum swelling in accelerator-irradiated annealed Type 316 stainless steel is higher than 600°C. Establishing the maximum temperature is of importance, in order to relate the proton-irradiation results to fast reactor irradiation effects.

The favorable effects of titanium addition to Type 316 stainless steel, and of low carbon content (in separate accelerator-irradiated samples), indicate that relatively simple modification of the reference Type 316 stainless steel composition can improve alloy ductility, and perhaps also its resistance to swelling.

The observation that EBR-II - irradiated Type 316 stainless steel is more magnetic than identical unirradiated control samples (annealed, aged, and cold worked) indicates that the austenite-ferrite conversion must be taken into account for bulk properties of Type 316 stainless steel under FBR conditions. This is, as far as we know, the first determination that austenite transforms under fast reactor irradiation. We had previously observed it in accelerator-irradiated samples.

IV. NEXT REPORT PERIOD ACTIVITIES

- 1) Swelling of samples of Type 316 stainless steel, proton irradiated at 400, 600, and 650°C, will be determined.
- 2) Swelling of samples of Type 316 stainless steel from X035 will be determined.
- 3) Two capsules for the X100 subassembly in the EBR-II will be prepared, and sent to ORNL.

- 4) Design and testing of experimental methods, to determine the effect of stress on swelling, will continue.
- 5) Creep testing of Type 316 stainless steel at 700° C will continue.

Program: Irradiation Damage in Cladding and Core Structural Materials			
AEC Task: 2 - Integral (n, α) Cross-Section Measurements			
Project Manager: G. W. Meyers			
Reporting Period: July-September 1971		189A:	13495
General Order: 06506	Subaccount: 29623	Category:	04-40-02-01.1

Principal Investigator: H. Farrar IV

I. PROJECT OBJECTIVES

The objectives of this task are to: (1) determine which elements in commonly used alloys are contributing to the high production of helium in fast neutron irradiations, so that their use might be eliminated or minimized, (2) determine the spectrum-integrated (n, α) cross sections of 50 elements in various fast reactor neutron spectra, (3) make estimates of the differential (n, α) cross sections of the same elements, by correlating the integral cross sections in the different neutron energy spectra by using unfolding techniques, and (4) use this information to predict, with some confidence, what helium production might be expected from any present or new alloy contemplated for fast reactor use.

II. ACCOMPLISHMENTS DURING FISCAL YEAR 1972

A. HIGH-TEMPERATURE FURNACE

In order to determine the (n, α) cross section of nitrogen, which has been irradiated in the form of several high-melting-point-temperature compounds, it became necessary to devise a method to release the helium that was generated in the specimens. After a number of preliminary experiments were conducted, using various techniques, a multiple-sample resistance-heated furnace, shown in Figure 14, was designed and built. The small, replaceable, tantalum carbide crucible is contained within a small stainless steel enclosure. This furnace chamber is separated from the much larger sample-loading assembly by a quartz glass vacuum lock.

The procedure for making a mass spectrometric determination of the helium content of a specimen is very similar, in principle, to the method used during the last several years. The tungsten wire basket crucibles used earlier

6506-4715

Figure 14. High-Temperature Furnace

were sufficiently small that the amount of adsorbed gases released when the tungsten was heated could easily be handled, along with unwanted (non-noble) gases from the sample, by the mass spectrometer getters. The much greater size of the tantalum carbide crucibles, however, makes it necessary to add a step to degas the crucibles under vacuum before the samples are loaded. After degassing, one of the samples, stored under vacuum in small holders in a rack, is lifted by the manipulator, and dropped through the funnel into the hole in the crucible. Following this, the funnel is removed, and the quartz disk vacuum lock, which also serves as a viewing window, is closed. The entire loading operation can be performed in a few minutes, without releasing the vacuum.

The crucible for vaporizing or decomposing samples can be made from a number of high-melting-point-temperature materials. During tests, specimens of titanium nitride (mp 2930°C) have been successfully decomposed in a tantalum carbide (mp $\sim 3880^{\circ}\text{C}$) crucible. Hafnium carbide and HfC-TaC crucibles (mp ~ 3890 and $\sim 3950^{\circ}\text{C}$, respectively) have been ordered. Crucibles with a larger capacity will later be used to melt relatively large specimens (~ 50 mg) with lower melting-point temperature.

Although not shown in the figure, three crucibles will be mounted in the furnace chamber. Each may be heated separately, by applying a high current through the electrodes. Tantalum shields minimize the radiated heat, and the copper electrodes and chamber walls are water cooled. The original insulators used for the high-current (400 amp) feedthroughs proved unsatisfactory, and two have been temporarily replaced by a different variety, while others are being procured. Testing has so far been conducted without attaching the furnace to the mass spectrometer system. The apparatus operates satisfactorily, although a number of simple improvements are being made. One difficulty is that TiN samples sometimes decompose too rapidly, and are forced out of the crucible, presumably by the pressure of the released nitrogen. A slower heatup rate appears to be one solution to this problem.

B. MASS SPECTROMETER VACUUM SYSTEM

Installation of a greatly improved vacuum system for the mass spectrometer sampling system and for the new high-temperature furnace has been completed. Three completely independent roughing and intermediate vacuum systems, and

an additional ion pump, have been installed on a new extension to the bench. The four existing multiple-sample ovens have been re-installed behind lead bricks, and flexible bellows and flanges have been welded to sections of the vacuum line leading to the mass spectrometer. Some of the changes were necessary to allow the high-temperature furnace to be placed as close as possible to the mass spectrometer. The final system is far more versatile, and will greatly reduce the between-sample evacuation time, permitting analysis of one sample to take place while background gases from previous analyses are being pumped away.

In the spiking system, the calibrated volumes containing known small quantities of pure ^3He , and a mixture of ^3He and ^4He , have always consisted of a combination of aluminosilicate glass and stainless steel, rather than Pyrex, which is relatively pervious to helium. The calibrated volume used to dispense the ^4He , however, was originally intended to contain other gases, such as argon or xenon, and was therefore made of Pyrex. To reduce the small but measurable amount of in-leakage of ^4He from the atmosphere, this latter system was replaced. All the volumes of the stainless steel and aluminosilicate glass components were calibrated.

C. PURE-ELEMENT SAMPLES FROM X034 AND X035 SUBASSEMBLIES

Six small subcapsules, containing a number of pure elements and compounds, irradiated for several years in EBR-II Subassemblies X034 and X035, have been retrieved from three larger capsules containing irradiated tensile specimens. The subcapsules have been opened in the AI Hot Laboratory hot cell. Shipment to the mass spectrometer laboratory will be made after completion of a hazards report, now in preparation.

D. INTERLABORATORY LMFBR REACTION RATE PROGRAM

A considerable amount of planning has been made, in collaboration with other principal investigators, in the Interlaboratory LMFBR Reaction Rate (ILRR) Program. This program is expected to help improve the absolute accuracy of reaction rates and of flux-fluence measurements, through the examination of several "standard" cross sections at different laboratories by various techniques. The objective of Atomics International's contribution is to determine

integral (n, α) cross sections of several isotopes in a variety of well-established and permanent neutron fields, including several that cover the neutron energy range of interest for FTR and LMFBR's. The determination, by mass spectrometry, of the amount of helium generated in milligram-size specimens will provide an additional and independent absolute base for normalization of available energy-dependent (n, α) cross-section data.

The initial effort will be to study the integrated (n, α) cross sections of ^6Li and ^{10}B in the Coupled Fast Reactivity Measurement Facility (CFRMF) neutron spectrum. The neutron flux in CFRMF, apart from being quite similar to that anticipated in a typical LMFBR core, is one of the more intense of those under consideration for irradiations in the program, and is therefore rather suitable for the first round of experiments.

Two "standard" (n, α) cross sections to be measured are for the ^6Li (n, α) ^3H and ^{10}B (n, α) ^7Li reactions. Gram quantities of separated ^6Li and ^{10}B will be procured, with the highest isotope purity available. To be sure that all the helium generated in these isotopes is retained for the mass spectrometric measurements, the milligram-size specimens will be sealed under vacuum in thin capsules. The material to be used for the capsules has not been selected, but it must be compatible with the contents, and must have a relatively low (n, α) cross section, since present plans call for the entire capsules and their contents to be included in the mass spectrometric analysis. Identical sealed capsules, without ^{10}B or ^6Li , will be included for blank measurements.

Lithium, because of its low melting point (179°C), is difficult to be welded inside very small capsules. Hydride and deuteride (LiH , LiD) compounds would release large amounts of hydrogen gas upon analysis, which would interfere with the mass spectrometric measurement of helium. The compounds LiF and Li_2CO_3 have the disadvantages of having other elements whose (n, α) cross sections are significant. The fluorine (n, α) cross section could be measured if additional capsules containing, for example, Fe and FeF_2 , were included, but this would require a considerable number of additional measurements. Nevertheless, a simple procedure for converting lithium metal to clear crystals of LiF has been worked out. Boron, and lithium compounds, in crystalline form, are preferable to powders, because the crystals will also tend to contain the generated helium.

In the ILRR Program, some of the experiments will be performed simultaneously, while others will require separate reactor runs. The integral experiments of AI and HEDL both require the maximum feasible exposure (8 or more hours), and will be combined, if flux-depression (self-shielding) calculations at HEDL show that this can be accomplished without significant interference. To minimize the differences in the neutron spectrum experienced by the various experiments as a result of neutron scattering and absorption, it was agreed at the first planning session, held at Idaho Falls on June 25, that each experimental assembly should be as similar as possible. The NBS aluminum fission chamber was the most bulky and least changeable, and was therefore chosen. Materials and plans needed to mock up the fission chamber have been received from NBS, although they may not be needed, if the Atomics International capsules are included in HEDL's assembly.

The possibility of using CFRMF, at some later date, for the determination of other (n, α) cross sections is under consideration. Of particular interest is the $^{27}\text{Al}(n, \alpha)^{24}\text{Na}$ reaction, because its high threshold energy will result in aluminum being a useful detector in an energy range (>6 MeV) where little is known about the ^6Li and $^{10}\text{B}(n, \alpha)$ cross sections. Comparison of this integral cross section obtained by mass spectrometry with the same entity obtained by ^{24}Na radiochemistry at HEDL will also be of considerable interest.

The (n, α) cross section of aluminum in the CFRMF neutron spectrum is, however, four orders of magnitude less than those of ^6Li and ^{10}B . To obtain an accuracy of 2 to 3% in a helium concentration measurement by mass spectrometer, however, a sample should contain at least $\sim 10^{11}$ atoms of ^4He . Preliminary calculations indicate that 5-mg samples of ^6Li and ^{10}B will produce this quantity, after a CFRMF irradiation lasting 8 hr; but 100 gm of aluminum would be needed to generate this amount of helium. Even using the new high-temperature furnace, which should be able to accept samples weighing as much as 100 mg, the low amount of helium would result in a considerable loss of accuracy in the measurement. If the power of CFRMF is raised, as anticipated, to 10 kw, the resulting number of ^4He atoms would still be only $\sim 10^9$, which is barely enough to give an accuracy of 10%. In later experiments, where longer irradiations are made, and after the mass spectrometer sensitivity has been increased by installation of a new getter system, this accuracy should be improved, especially if several duplicate specimens are analyzed.

There are also several elements and isotopes of interest in LMFBR alloys which have spectrum-averaged (n, α) cross sections equivalent to, or higher than that of aluminum (e. g., ^{14}N , ^{16}O , ^{28}Si , ^{32}S , and Ni). Measurement of these, along with the other samples, should prove very interesting, because the (n, α) cross sections that will result will be integrated over a neutron spectrum (CFRMF) similar to that anticipated in a typical LMFBR core. Comparison of these measured values with values obtained indirectly from other neutron spectra using unfolding techniques would be very useful, and would also help in estimating the uncertainties of other cross sections too small to be measured in CFRMF by this technique.

III. IMPACT ON LMFBR PROGRAMS

Helium production rates and (n, α) cross sections of several of the most important elements in LMFBR cladding alloys have been measured, after irradiation at various locations in EBR-II. Among the next most important fast (n, α) cross sections that should be measured are those of boron, carbon, nitrogen, and oxygen. These elements were irradiated in the same EBR-II locations, but in the form of high-temperature-melting-point compounds. The extraction of the helium for mass spectrometric measurement, however, required the development of the high-temperature furnace, and the modification of the mass spectrometer vacuum system.

The Interlaboratory LMFBR Reaction Rate (ILRR) Program seems to be a realistic way of relating certain integral and differential cross section data. After preliminary test runs, and after some minor modifications to improve sensitivity, the absolute accuracy of the helium measurements by mass spectrometry is expected to meet the requirements for the program, especially for irradiations in the higher-flux test reactors. When the problems of encapsulation have been resolved, the short-term goal of obtaining (n, α) cross sections of ^{10}B and ^6Li , integrated over the CFRMF neutron spectrum, should present no difficulties. The measured, spectrum-integrated (n, α) cross sections will be subsequently compared with the same integral cross sections obtained in the ILRR Program by using radiochemical techniques, and both will then be reconciled with available energy-dependent (n, α) cross-section data. This will provide the latter with a completely independent and absolute normalization. The

outcome of the ILRR Program should go far in helping resolve some present inconsistencies in dosimetry.

IV. NEXT REPORT PERIOD ACTIVITIES

- 1) A new high-temperature furnace chamber, with three pairs of copper feedthroughs, will be assembled.
- 2) The entire high-temperature furnace will be attached to the mass spectrometer system, for tests with samples containing helium.
- 3) Vacuum gauge and ion pump power supplies will be installed in an equipment rack, and the vacuum system will be labelled for convenience and simplicity of operation.
- 4) The spiking volumes will be refilled, and the mass spectrometer system will be put back into operation.