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QUARTERLY TECHNICAL PROGRESS REPORT
FUELS, MATERIALS, COOLANT CHEMISTRY,
AND FUEL HANDLING PROGRAMS
OCTOBER-DECEMBER 1971

AEC Research and Development Report

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OCTOBER-DECEMBER 1971

The preceding Quarterly Progress Report was AI-AEC-13015

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Program:	Sodium Technology		
AEC Task:	1 - FFTF Sodium Component Qualification		
Project Manager:	G. W. Meyers		
Reporting Period:	October-December 1971	189A:	13625
General Order:	6507	Subaccount:	39610, 39620, 39630
		Category:	04-40-04-04.1

Principal Investigators: J. Guon, J. E. Bodine, 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 vapor traps, freeze vents, and filters for sodium service. 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, coordinating space and operational restrictions with HEDL
- 2) Revise and issue the RDT Standard for vapor traps
- 3) Construct and evaluate the performance of one unfinned and one finned 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. MAJOR ACCOMPLISHMENTS DURING FISCAL YEAR 1972

- 1) The test results on the original multiple-baffle vapor trap proved encouraging, at 0.5- to 0.8-scfm flow rates; and modifications were made, in an attempt to increase the flow capacity. The test data from the modified trap showed no improvement in performance. With HEDL's concurrence, the test program and other efforts to improve this design have been terminated.

- 2) Aerosol filter loading tests have been completed on 5, 20, and 35 μ pore size stainless steel frit filters. The data have been included in Appendix A of the current draft of RDT E4-14, the Vapor Trap Standard.
- 3) A controlled-temperature-profile (CTP) vapor trap condenser design has been selected for the prototype 1.0-scfm vapor trap assembly which is being designed to meet the requirements of the FFTE and Composite Reactor Components Test Activity (CRCTA) facilities. Design layouts and the material specifications have been completed. Bids for the procurement of most of the parts have been received; however, parts cannot be ordered until AI receives an approved Ordering Data. Documentation required, in conformance to the draft RDT Standard E4-14 and HWS-1895, Ordering Data, is being prepared.
- 4) The controlled-temperature-profile vapor trap condenser design has been selected for the 5.0-scfm test unit. Design, and the specification of materials, are in progress.
- 5) The test program conducted on the freeze vent is complete. The draft of the final report is being reviewed. Based upon the findings of the experimental program, suggested modifications to RDT Standard E4-13T have been drafted, and submitted for consideration.
- 6) The draft of the Vapor Trap Standard RDT E4-14 has been revised to reflect the recent comments from reviewing organizations. The revised draft (October 1971) provides design criteria for three types of vapor traps: (1) continuous-flow CTP units, up to 1.0 scfm, (2) intermittent flow units, and (3) back-diffusion barriers for gas inlet lines. The revised draft was delivered to HEDL, and HEDL has issued a December 1971 draft of the Standard.

III. PROGRESS DURING REPORT PERIOD

A. VAPOR TRAPS

1. Multiple-Baffle Vapor Trap (MBVT)

Testing of the modified MBVT unit began in September, and was completed in October. The modifications made on the trap which originally showed promise for use in the 0.5- to 0.8-scfm range included: (1) doubling the gap between adjacent baffles, (2) adding inter-baffle wicks to aid in draining the condensed sodium from one baffle to another, so as to minimize the probability of re-entrainment of droplets, and (3) the inclusion of a backup plate, to prevent the wire mesh from being displaced. The intent of these modifications was to extend the operating range of this trap design toward the 5.0-scfm goal. Successful operation would have provided scaleup information for the design of a multiple-baffle-type 5.0-scfm unit.

The data from the trap tests are shown in Table 1. Prior to the start of these tests, an attempt was made to condition the trap by heating it to $>900^{\circ}\text{F}$ in several temperature steps, and exposing the trap surfaces to argon containing a saturation concentration of sodium vapor at 900°F at a low flow rate ($1/4$ scfm). The data shown in Table 1 are for a 1.0-scfm argon flow rate, at which rate the argon should have been adequately cleaned up, if the scaleup were successful. The data on the sodium content of the argon at the exit of the baffles is seen to be several hundred times greater than the amount that can be tolerated (1 to 2 wppm Na). The mesh packing is seen to remove a small additional fraction of the sodium. However, the amount of sodium in the filter inlet is too high to meet FFTF requirements, in all cases.

In an attempt to gain insight into the operating principles involved in this type of trap, tests were conducted at increased system pressure. The sodium content of the trap effluent, as monitored by a fog detector, was not the expected function of either the linear or the mass flow rate. The original model for the MBVT predicts that, for constant linear flow rate, condenser performance would be independent of mass flow rate. The original model predicts the condenser performance should be a first- or second-order function of linear flow rate. A suitable model for the performance of a MBVT is therefore not available.

6507-4780

Figure 1. Effect of System Pressure on Flow Rates for Controlled-Temperature-Profile Vapor Trap Condenser

TABLE 1
EFFECT OF MULTIPLE-BAFFLE VAPOR TRAP OPERATION
ON QUALITY OF EFFLUENT ARGON
(1-scfm argon)

Date	Temperature (°F)					Sodium Content (wppm)		
	Saturator	Gas, Inlet to Trap	Baffle	Gas, Inlet to Mesh	Mesh	Baffle Outlet	Mesh Outlet	Filter Inlet
9/28	1050	400	300-450	200	240	1120	570	91
9/29	1050	500	300-450	290	400	770	680	15
9/30	1050	500	300-450	290	400	610	320	42
9/30	1100	680	390-590	310	390	690	480	11
10/1	1100	905	400-600	320	400	231	490	250
Pressurization Tests Conducted								
10/6	1100	875	430-650	345	400	>525*	700	6

*Portion of sample lost during chemical analysis

The data of Table 1 show that the performance of the modified MBVT unit has not been improved. The results of the pressure - flow rate tests show a suitable model for the MBVT does not exist. As a result, with HEDL's concurrence, efforts on the MBVT have been discontinued.

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

Because the CTPVT concept is expected to be used on systems operating at pressures above 1 atm absolute, a series of tests was performed to determine the effect of system pressure on trap performance. The tests were conducted, using a fog detector to indicate the flow rate at which the appearance of a fog was barely discernible. The use of this detector makes it possible to perform tests of short duration, for which chemical sampling is impractical. The fog detector has been calibrated against chemical samples, and has been found to have a limit of detection of $9 \mu\text{g Na/l}$, or 5 wppm at standard conditions. For the tests whose results are shown in Figure 1, the saturator was operated at 800°F, and the condenser was operated with the profile as specified in Appendix A of draft Standard E4-14. The data show that the break-through flow rate

is proportional to the linear flow rate, within experimental accuracy. This is consistent with the premise that the vapor trap condenser removes sodium vapor to a given exit sodium partial pressure, if the linear flow rate capacity of the bed is not exceeded. Thus, doubling the absolute pressure would reduce the sodium mass concentration (expressed as wppm) and the specific volume of the argon to one-half of the original value. Then, by doubling the argon mass flow rate, the linear flow rate of the argon would be identical to that at the original pressure, and it would carry the same partial pressure of sodium vapor (at half of the mass concentration, expressed as wppm). Thus, in accordance with a constant (psia/scfm) ratio, a given CTPVT condenser would have a capacity which is proportional to the absolute system pressure. This pressure effect has been incorporated in the current draft of the RDT Standard E4-14.

The development of an analytical model for the CTPVT is continuing at a relatively low level of effort. The differential equations (developed jointly by AI and HEDL), believed to represent the mass transfer processes occurring within the condenser, have been solved for three cases: (1) the driving force for mass transfer being the difference between a gas supersaturated in sodium vapor and a sodium wetted surface, (2) the driving force for mass transfer being related to the presence of a sodium fog in the gas, and (3) the driving force for mass transfer being the presence of both supersaturated gas and fog. None of the solutions for the preceding cases are completely consistent with AI and HEDL experimental results obtained on CTPVT's. The effect of other system parameters (such as radial temperature gradient through the bed) are being investigated.

3. 1.0-scfm Standard Vapor Trap (1-SVT)

The design of the 1-SVT assembly is continuing, based upon the December 1971 drafts of the RDT Standard E4-14 and HWS-1895, the Ordering Data for the CRCTA unit.

The 1-SVT consists of preheater, condenser, and filter units, as is called out in the draft RDT E4-14. The preheater unit, as presently conceived, is a section of 4-in. Schedule 40S pipe with a capped 2-ft length of 2-1/2-in. Schedule 10S pipe on its axis. The argon flows in the annular region between the pipes, and is heated by a 1-kw heater clamped to the periphery of the 4-in. pipe.

The preheater is insulated with a 4-in. thickness of pipe insulation, to maintain heat economy.

The condenser unit consists of a 10-in. Schedule 40S pipe with a capped internal 4-in. Schedule 10S pipe on its axis. The annular region between the pipes is filled with 1/4-in. Raschig rings, to a depth of 41 in. This is the bed along which a succession of heaters, clamped to the external wall, will impose the controlled temperature profile defined by the draft RDT E4-14. The entrance (lower) end of the condenser is fitted with a dry-wall funnel fixture, and the exit (upper) end is fitted with a sodium flush fixture. The ends of the 10-in. pipe are fitted with 4-in. to 10-in. reducers which connect to the preheater and the filter units.

The filter unit is a length of Schedule 40S 12-in. pipe which contains a 64 ft², 35 μ stainless steel frit filter unit. This filter area is sized, according to the draft of RDT E4-14, to be adequate for the 120-day maintenance cycle called out in the CRCTA Ordering Data.

An interim design review meeting was held on the 1-SVT, and the conclusions are reported in a design review report. Based on this design review, and the decision to proceed with the design as summarized previously, material lists have been prepared, and quotations on price and delivery have been obtained. The filter unit has a 12 to 14 week delivery time, and all other major parts have delivery times in the range of 6 to 10 weeks. The purchase of pressure-boundary material is awaiting the receipt of the approved Ordering Data Package.

The documentation required, in conformance with the December drafts of RDT E4-14 and HWS-1895, is being prepared. The required drawings are ~75% complete. The Quality Assurance Functional Plan (Par. 4.1.2), and some Quality Assurance Acceptance Procedures, have been prepared in draft form, and are being reviewed. The Fabrication Plan (Par. 3.7.2) is being prepared. The Design Report (Par. 3.7.1) is being prepared, but cannot be completed until the fully approved Ordering Data and Standard are received.

4. 5.0-scfm Standard Vapor Trap (5-SVT)

The design of a 5-SVT for the primary gas recirculation circuit of FFTF (VT-12 and VT-44) has begun. The chemical engineering design of the condenser

is based on the design equations in Appendix A of the draft of RDT E4-14, intended for condensers having capacities up to 1 scfm, and what are believed to be sound engineering judgments relative to the scaleup required. Tests to be conducted with this condenser are intended to:

- 1) Define a startup and conditioning cycle that will adequately activate the operating surfaces of the condenser
- 2) Qualify the chemical engineering design
- 3) Provide information for a 5-scfm condenser appendix to RDT E4-14.

In order to assure that the condenser being tested will have the same operating characteristics as a unit built to RDT Standards, care is being exercised to assure that:

- 1) The design details are such as to permit upgrading of a condenser, built at some time in the future to RDT Standards, with little or no significant modification of the design.
- 2) The pressure barrier materials are procured to RDT Standard.
- 3) Nonpressure barrier materials are procured to materials specifications referenced in the appropriate RDT Standards, and to the chemical compositional requirements of the appropriate RDT Standards.
- 4) The fabrication procedures are such as to permit upgrading of a condenser, built some time in the future to RDT Standards.
- 5) Finished, fabricated parts, exposed to the working gas, have cleaning and cleanliness consistent with parts that would be fabricated to RDT Standards.

Close liaison is being maintained between Manufacturing, Quality Assurance, and Materials and Process personnel to assure and provide verification that the preceding requirements are met.

The design effort on the three parts of the vapor trap assembly (preheater, condenser, and filter) continue. The mechanical and thermal design of the preheater is complete. The mechanical design of the condenser is complete. The stress and thermal analyses are each over 90% complete. A commercially available filter will be used; therefore, no design effort on this subassembly is

needed. A layout of the 5-SVT installation in the test rig has been completed. Orders for the purchase of all long-lead material items for the test have been written, and are being processed.

B. FREEZE VENT

The final experiment in the unfinned freeze vent was one in which the freeze plug was formed ~ 8 in. into the vent, and a pressure of 2.5 atm of pure oxygen was introduced to the gas side of the plug. Over a period of 12 days, the total weight of oxygen absorbed by the plug was 35 mg, compared to 12 mg in the previous test. The plug was broken within 7 min after applying a differential pressure of 5 psi across it, while passing a current of 7 amp through the heater. This ease of breaking the plug is within the range of experience gathered with an uncontaminated plug. The conclusion drawn from the experiments with a contaminated plug is that the extent of oxidation achieved did not affect the ease with which the plug was broken and the vent re-opened.

Data analysis of the results of the freeze vent experiments has been completed. The close-out report on the freeze vent studies has been prepared, and is being reviewed.

One of the purposes of these tests was to verify the design approach outlined in RDT Standard E4-13T. In general, RDT E4-13T was found to be adequate. However, the section of RDT E4-13T relating to the time of freezing has been shown to give calculated freezing times which are much shorter than the experimentally observed freezing times. The reason for this discrepancy is thought to be that, in the calculational approach, the heat transfer coefficient between the sodium in the annulus and the annular wall is implied to be infinite, while the actual coefficient, although large, is finite. Consequently, the calculated freezing times are low by an order of magnitude. Because this calculation is used in the design sequence of RDT E4-13T to size the annulus, it is necessary to modify the Standard, to avoid the selection of an unworkable, oversized annular dimension. Therefore, recommended changes to RDT E4-13T have been drafted for consideration. The most significant of these is the restriction of the vent annulus width to the range of 0.015 to 0.060 in.

C. STANDARDS AND ORDERING DATA

The current status of the Standards and Ordering Data is summarized in Table 2. During the report period, the principal activity was the revision of the August 1971 draft of the Vapor Trap Standard, RDT E4-14. Comments and recommendations for changes were received from LMEC, HEDL, and RDT. Conflicts in these recommendations were resolved; and a draft, dated October 1971, was prepared.

TABLE 2
CURRENT STATUS - STANDARDS AND ORDERING DATA

Document	Review	Status	Responsible
E4-14 Vapor Trap Standard	HEDL, RDT	Draft submitted to HEDL 10-16-71. HEDL has edited this draft, and is including it in the Ordering Data package with HWS-1895, for RDT approval.	HEDL
E4-13T	LMEC, HEDL, RDT	Modification recommendation submitted in early November.	LMEC
E11-2	HEDL	Comments received from HEDL; review to begin in December 1971.	AI
HWS-1895	HEDL	Draft being used for 1-SVT design; document being made to conform with latest revision to RDT E4-14. To be submitted to RDT for approval when complete.	HEDL
HWS-1119 Ordering Data FFTF	HEDL	Being held until input design data are available.	HEDL
HWS-1118	HEDL	Submitted April 1971 to HEDL; HEDL will assume responsibility for final release.	HEDL

The most visible change in the October draft involved a reorganization of the appendices, so that the new Appendix A includes the design procedures for the preheater, the condenser, and the filter units of the controlled-temperature-profile vapor trap assembly. Appendix B is a design procedure for one type of intermittent-gas-flow vapor trap, and Appendix C is a design procedure for a back-diffusion barrier for gas addition lines. (This draft was forwarded to HEDL, and HEDL has since issued a December 1971 draft.)

In connection with the revisions to the Standard, an attempt was made to keep in frequent communication with HEDL, in order that the CRCTA Ordering Data (HWS-1895) could be most expeditiously updated to correspond to the most recent draft of the Standard. HWS-1895 is being prepared by HEDL.

IV. IMPACT ON LMFBR PROGRAMS

The material procurement for the 1-SVT vapor trap is being delayed until the Ordering Data Package is approved. Because the delivery time of some parts is 12 to 14 weeks, and because fabrication and installation will require 10 to 12 weeks, the initiation date of the qualification test is not anticipated to occur before the last week of May 1972, assuming an approved Ordering Data Package is received by the first week in January 1972. It is not clear, at this time, that the CRCTA procurement activity (scheduled to start September 1972) is consistent with the preceding schedule and the 120-day test program required to qualify this design.

The delivery requirement for the 5-scfm vapor trap for FFTF is scheduled for September 1972. The current schedule for the presently defined program will meet that timing and need.

The preparation of the final report on the freeze vent study will complete that effort on schedule.

The drafts of the Standards required for the program are complete, and were issued on schedule.

V. NEXT REPORT PERIOD ACTIVITIES

The programs on the Multiple-Baffle Vapor Trap and the Freeze Vent are complete. Therefore, no more activity in these areas is anticipated. Some minor effort may be required in the revision of the Freeze Vent Standard.

The present experimental program on the CTPVT has been completed. The CTPVT test rig will be used to test the sodium-loading capacity of 65 and 165 μ filters.

Fabrication of the 5-SVT should be completed, and the unit shipped to the test facility.

Program:	Sodium Technology		
AEC Task:	1 - Sodium Cleaning and Requalification		
Project Manager:	G. W. Meyers		
Reporting Period:	October-December 1971	189A:	13625
General Order:	6507	Subaccount:	36100
		Category:	04-40-04-04.1

Principal Investigators: R. L. McKisson, K. E. Moore, 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 use in sodium service and/or after a sodium leak or spill. The 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. MAJOR ACCOMPLISHMENTS DURING FISCAL YEAR 1972

- 1) The revised work plan on Sodium Cleaning and Requalification has been completed.
- 2) The sodium leak corrosion rate apparatus design concept was successfully demonstrated.
- 3) A refined version of the sodium leak corrosion rate apparatus was assembled, and its first leak test was performed.
- 4) A literature search of sodium cleaning technology has been completed.

- 5) A draft of a cleaning procedures guide, to define areas requiring technical backup, has been completed.
- 6) A draft of a sodium technology design guide for cleaning, maintenance, and repair has been completed.

III. PROGRESS DURING REPORT PERIOD

A revised work plan, "Sodium Cleaning and Requalification," has been prepared, and will be submitted to RDT and HEDL for review. This document defines the problem areas of cleaning and re-using materials exposed to sodium in liquid, solid, and vapor phases. The main thrust of this program is to conduct straightforward, and yet comprehensive, studies to develop specific procedures for applying the inert gas - moist inert gas - water cleaning process. This process has been selected as the reference process for the HEDL Alkali Metal Cleaning Facility and the LMEC Component Handling and Cleaning Facility. A parallel, but lower-keyed, effort will be conducted on other cleaning methods, such as Dowanol-EB - water and evaporation processes. An investigation of storage methods and requalification procedures will also be a part of the cleaning processes evaluations. The results of these evaluations will become an integral part of the Sodium Cleaning and Requalification Procedures Handbook. The Handbook is intended to be a presentation of sodium cleaning and requalification procedures, directed toward the technologists actually performing maintenance and repair operations.

As a necessary prelude to preparing the Sodium Cleaning and Requalification Procedures Handbook and initiating cleaning processes evaluations, an exhaustive literature search was conducted. This information is now being compiled; it will be issued as a report, early in 1972.

Another phase of this program involves investigating the corrosion of stainless steel resulting from a sodium leak or spill into an oxidizing atmosphere. The two primary areas of concern in this program are leaks into the core vessel blanket gas region (N_2 - 1% O_2), and in the intermediate heat exchanger area (air atmosphere). The severity of the corrosion, under various conditions of time, temperature, and environment, will serve as a basis for

the later definition of cleaning procedures, and as an indication of the time factors involved in the development of pitting and/or grooving corrosion.

The experimental apparatus, and the specimen configuration, for the sodium leak rate corrosion tests were successfully demonstrated in concept. However, design changes which will increase the efficiency of performing the tests, and an alternate method of heating the samples, are being developed. Assembly of a test rig, capable of running three of these tests simultaneously, each with its own atmosphere control and analysis, is nearing completion. One of three test chambers, used in a shakedown experiment, is shown in Figure 2. The leak rate specimen consists of two milled and ground 1/4-in. thick plates of Type 304 stainless steel, stacked and welded on three sides. Sodium is forced through a 1/8-in. OD tube in the top plate (1 by 1 in.) and the unwelded opening, onto the platen or apron formed by the 1 by 2 in. lower plate. The apron leak area is used for evaluation of sodium - sodium reaction product corrosion rate.

Figure 2. One of Three Test Chambers for Sodium Leak Corrosion Testing

The first shakedown run, using the modified apparatus shown in Figure 2, was performed at $\sim 1000^{\circ}\text{F}$. The specimen had a gas leak rate of 0.8 cc/sec at 40 psig. About 20 cc of sodium was forced through this leak in ~ 5 min. The cover gas purge, into which the sodium leak occurred, was supplied from a bottle containing 99% N_2 - 1% O_2 with <10 ppm H_2O . The sodium ignited at the beginning of the test, indicating that the gas in the test chamber initially contained $>4\%$ O_2 ,^{*} probably resulting from a leak in the system and incomplete initial purging. The specimen temperature reached 1080°F during the burning period, which lasted ~ 2 to 3 min. From this point, the specimen temperature was maintained at 975°F for 1 hr in the 99% N_2 - 1% O_2 gas mixture. Post-test examination revealed that the specimen and the heater block were completely wetted with sodium, and were covered with dendrite protrusions of sodium and sodium reaction products. Upon cleaning and preliminary macro-examination, evidence of pitting corrosion could be seen.

IV. IMPACT ON LMFBR PROGRAMS

This program is among the first to address itself in a quantitative way to the problems of sodium-leak-induced corrosion, of cleaning surfaces exposed to sodium, and of requalification of components for re-use. Because of the high costs of fabrication of reactor hardware, proven cleaning and requalification procedures will assume an increasing importance to the economics of reactors in general, and to test reactors in particular.

V. NEXT REPORT PERIOD ACTIVITIES

The majority of the guard vessel annulus blanket gas sodium leak corrosion experimental work will be completed. Process development and evaluation of the nitrogen - moist nitrogen sodium cleaning concept will be initiated. A literature survey report on sodium cleaning technology will be issued.

*T. S. Krolipowski, "Violently Sprayed Sodium Air Reactions in an Enclosed Volume," ANL-7472 (September 1968)

Program:	Sodium Technology		
AEC Task:	1 - FFTF Support Work - Sodium Frost		
Project Manager:	G. W. Meyers		
Reporting Period:	October-December 1971	189A:	13625
General Order:	6507	Subaccount:	39810
		Category:	04-40-04-04.1

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

I. PROJECT OBJECTIVES

The basic objectives of this project are:

- 1) Develop an analytical model of sodium oxide deposition on surfaces exposed to simulated FFTF reactor cover gas (argon), based on a typical LMFBFR 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).

The specific current objective of this project is to perform an analytical study of possible sodium frost problems associated with the following two thermal shield concepts for the FFTF closure head: (1) the spaced, seven-plate design, and (2) the stacked, hollow-plate design.

II. MAJOR ACCOMPLISHMENTS DURING FISCAL YEAR 1972

It was determined, from a preliminary checkout run, that sodium oxidation rates can be adequately measured by the weighing technique employed in the

6507-4781

Figure 3. Critical Gap Between Horizontal Plates for Bridging by Liquid Sodium

sodium oxidation rate test apparatus. This placed 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.

The effort on the current objective (viz., to perform an analytical study of possible sodium frost problems associated with the spaced, seven-plate, and the stacked, hollow-plate, thermal shield concepts) was initiated in early October.

The critical gap between horizontal plates for bridging by liquid sodium has been calculated, and is found to be significantly smaller than the smallest gap in the FFTF head shielding.

A two-dimensional analysis of gas convection through the FFTF head shielding with the seven reflector plates, and with the stacked, hollow plates, has been made; and computer programs for the solution of the respective cases have been written.

III. PROGRESS DURING REPORT PERIOD

The effort on the current objective (viz., to perform an analytical study of possible sodium frost problems associated with the spaced, seven-plate, and stacked, hollow-plate, thermal shield concepts) was initiated in early October.

A literature search was initiated for information on the critical gap between horizontal plates for bridging by liquids. Figure 3 shows a plot of the predicted critical gap between horizontal plates for sodium, based on experimental measurements made with mercury between glass plates.*

The plot shown in Figure 3 is obtained from the expression

$$H_{cr} = K \sqrt{\frac{\gamma}{g(\rho_l - \rho_v)}} \quad \dots (1)$$

where:

g = gravitational acceleration

H_{cr} = critical gap

*R. P. Forslund, "Interface Stability in Rectangular Tubes with Gravity Field," NAA-SR-TDR-8960 (September 4, 1963).

a. With Spaced, Seven Reflector Plates

b. With Stacked, Hollow Plates

Figure 4. Flow Passages in Two-Dimensional Analysis of Convection in FFTF Head Shielding

K = constant

ρ_l = liquid density

ρ_v = vapor (gas) density

γ = surface tension

In the cited reference, the observed value of K was 2.4 for mercury and 1.84 for water. Possible reasons for the difference in the value of K for these two fluids include: (1) experimental inaccuracy, and (2) difference in the degree of wetting (i. e., better wetting, in the case of water). If the latter is the reason, the results shown in Figure 3 should be conservative, for those regions of the head shielding where operating temperatures are high enough to induce wetting. In both head shielding concepts, the smallest gap between horizontal plates is 0.75 in., which is $\sim 70\%$ greater than the maximum critical gap shown in Figure 3.

A survey was initiated on the design and performance of reflective, thermal shields used in other liquid-metal-cooled reactors.

A two-dimensional analysis of convective gas flow through the FFTF head shielding with the spaced, seven reflector plates, and with the stacked, hollow plates, was made.

Computer programs for the solution of the respective cases were written. Figure 4a shows the assumed flow passages in the two-dimensional analysis of convection in the head shielding with the spaced, seven reflector plates. Figure 4b shows the assumed flow passages in the two-dimensional analysis of convection in the head shielding with the stacked, hollow plates. Computed convection rates through these passages are shown, in Figures 5a and b for these respective configurations, for the case in which convection is induced by a cold, vertical channel between the innermost vertical reflector plate and the outer edge of the head shielding, and a hot vertical channel formed within the head shielding by a penetration. The following assumptions were made:

- 1) Width of hot vertical channel, $Z_h = 0.625$ in.
- 2) Width of cold vertical channel, $Z_c = 0.150$ in.

6507-4784

a. With Spaced, Seven Reflector Plates

6507-4785

b. With Stacked, Hollow Plates

Figure 5. Argon Convection in FFTF Head Shielding

- 3) Temperature distributions in hot and cold vertical channels, as determined from data given in Figures 58 and 47, respectively, of Combustion Engineering Calculation No. RT-4
- 4) Argon temperatures assumed to be the same as the channel temperatures.

Negative flow rates shown in Figures 5a and b signify that the flow direction is opposite the direction shown in Figures 4a and b.

It is noted, in Figures 5a and b, that w_0 , the rate of gas convecting into the head shielding from the cover gas space below it, decreases as the distance between the hot and cold vertical channels is decreased. This is apparently due to the fact that, as the distance between the hot and cold vertical channels is decreased, internal circulation (short circuiting) is facilitated.

It is also noted, from Figures 5a and b, that the rate of gas convecting into the head shielding from the cover gas space below it is two to three times greater for the shielding with the spaced, seven reflector plates than for the shielding with stacked, hollow plates.

The effect of cold, vertical-channel width on convection is shown, in Figure 6a, for the head shielding with the spaced, seven reflector plates, and in Figure 6b for the head shielding with the stacked, hollow plates.

IV. IMPACT ON LMFBR PROGRAMS

The current study will establish design guidelines, as well as define the limitations, of the open, reflector-plate type of thermal barrier, when used in the cover gas space under the top head of FBR's.

V. NEXT REPORT PERIOD ACTIVITIES

The convection results will be combined with developed sodium frost models: (1) to predict the formation of sodium frost (sodium and sodium oxide) deposits in the FFTF head shielding, and (2) to indicate its effect on the performance of the thermal shield. Two concepts of the thermal shield will be examined (viz., the spaced, seven-reflector plate design, and the stacked, hollow-plate design).

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a. With Spaced, Seven Reflector Plates

6507-4787

b. With Stacked, Hollow Plates

Figure 6. Effect of Cold, Vertical-Channel Width on Convection in FFTF Head Shielding

In order to expedite the early evaluation of the sodium frost problem in the FFTF head shielding, simplified models and equations will be used for preliminary calculations. A preliminary report will be issued in January, at the completion of these preliminary calculations. A more detailed calculation will then be initiated, to include refinements excluded from the preliminary analysis.

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Program:	Sodium Technology		
AEC Task:	1 - FFTF Support Work - Friction Tests		
Project Manager:	G. W. Meyers		
Reporting Period:	October-December 1971	189A:	13625
General Order:	6507	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. MAJOR 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. Since that time, test specimens and holders for two matrices (18 couples each) were designed, fabricated, and inspected at AI, utilizing materials specified and/or furnished by HEDL. Various surface coatings for the test specimens were and are being specified, directed, and obtained by HEDL from various sources outside of AI.

Testing of the first matrix of 18 friction couples started on October 12, 1971, and was completed during the first week of November. Breakaway, static, and dynamic friction coefficients were determined for all specimens at the prescribed temperature levels. Those specimens utilizing an Inconel 718 base generally experienced the least wear; the least worn specimen pair (No. 6) was the set with Cr_3C_2 - 15 Ni coating on Inconel 718. A detailed presentation of the data appears in this report.

The second matrix friction test couples was specified by HEDL on December 1, 1971. This group was placed in the sodium test apparatus, and testing

TABLE 3
FIRST TEST MATRIX

Specimen Couple	Rider	S/N	Plate	S/N
1	Inconel 718	34-003	Inconel 718	33-003
2	Chrome Plate* (Hard Chrome)	36-002	Chrome Plate* (Hard Chrome)	35-001
3	K165*	36-010	K165*	35-010
4	Cr ₃ C ₂ - 15 Ni* polished	36-004	Cr ₃ C ₂ - 15 Ni* polished	35-003
5	Cr ₃ C ₂ - 15 Ni* (as deposited)	36-018	Cr ₃ C ₂ - 15 Ni* (as deposited)	35-017
6	Cr ₃ C ₂ - 15 Ni (on Inconel 718)	34-001	Cr ₃ C ₂ - 15 Ni (on Inconel 718)	33-001
7	TZM	38-001	TZM	37-001
8	TiC ₂	36-009	TiC ₂ *	35-009
9	Cr ₃ C ₂ - 15 Ni*	36-005	TiC ₂ *	35-016
10	Cr ₃ C ₂ - 15 Ni* (on Inconel 718)	34-002	Inconel 718	33-004
11	Inconel 718	34-004	Cr ₃ C ₂ - 15 Ni (on Inconel 718)	33-002
12	Cr ₃ C ₂ - 15 Ni*	36-006	K165*	35-011
13	K165*	36-017	Cr ₃ C ₂ - 15 Ni*	35-004
14	Chrome Plate* (Hard Chrome)	36-003	Cr ₃ C ₂ - 15 Ni*	35-005
15	TZM	38-002	Inconel 718	33-005
16	TZM	38-003	K165*	35-012
17	Inconel 718	34-005	K165*	35-018
18	Inconel 718	34-006	Chrome Plate* (Hard Chrome)	35-002

*Base material was Type 316 stainless steel, reported to be 20% cold worked
[Ref: Atomics International IL from L. R. Stone to R. T. Thexton
(September 20, 1971)]

was started on December 8, 1971. Testing is scheduled to be completed the first week in January. The program is following the same basic test procedures as did the first matrix. Test results are not yet sufficiently reduced and evaluated to enable presentation of more than a preliminary preview of trends in this report.

III. PROGRESS DURING REPORT PERIOD

The test program on the first test matrix (Table 3), which started on October 12, 1971, was finished during the first week of November. Throughout the program, sodium samples analyzed at specified temperatures according to ANL/ST-6 showed O_2 content <5 ppm, and testing progressed normally until the $1160^\circ F$ and 800 psi contact pressure condition was reached in early November (see Figure 7). At this point in the test, a sodium sample indicated O_2 in excess of 20 ppm (sodium plugging temperature, however, was still in the proper range), so the test was put on hold. A subsequent investigation by the Chemistry Group revealed that their instrumentation had malfunctioned, and had given an erroneous reading; so no friction test anomalies actually occurred. While the Chemistry Group was in the process of recalibrating their instrumentation, and because the final friction readings at $450^\circ F$ and 800 psi contact pressure had already been taken, it was decided to return to $1160^\circ F$ for a repeat of the friction readings, in the event the O_2 level had indeed been as high as reported. While at this temperature, a second set of readings was taken. The 800-psi loading remained on the specimens for ~ 6 hr after the test program, and was then removed, so that each couple had zero load. A measurement was then made to determine the shear load required to separate the couples. These load values are listed in Table 4, to indicate the degree of apparent self-welding or metallurgical bonding that occurred between the various specimen material combinations, under the environmental conditions noted.

All of the data from the first test matrix have been reduced. A typical data record is shown in Figure 8, which indicates how the pertinent friction coefficients were measured. These data have been plotted to show friction coefficients versus temperature for: (1) initial cycle breakaway friction (Figure 9), (2) maximum static friction (Figure 10 — average of peak values for Cycles 3 to 10, 50 to 55, and 94 to 99), and (3) maximum dynamic friction

TEST CONDITIONS

- 1) TEMPERATURE CHANGE OF 50°F/hr (max.)
- 2) STABLE TEMPERATURE $\pm 5^\circ\text{F}$
- 3) OVERNIGHT HOLD PER DATA TEMPERATURE LEVEL
- 4) PLUGGING TEMPERATURE AT EACH TEMPERATURE LEVEL
- 5) OXYGEN CONTENT TO BE 5 ppm OR LESS. IF O₂ LEVEL EXCEEDS 5 ppm, SHUT DOWN, COLD TRAP, AND REPEAT TEST PROGRAM.

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Figure 7. Temperature Test Profile

TABLE 4
FIRST TEST MATRIX BREAKAWAY FORCES

Specimen Couple	Rider	Plate	Breakaway Force (lb)
1	Inconel 718	Inconel 718	<5
2	Chrome plate	Chrome plate	13.7
3	K165	K165	3.8
4	Cr_3C_2^* polished	Cr_3C_2 polished	41
5	Cr_3C_2 as deposited	Cr_3C_2 as deposited	18
6	Cr_3C_2 (on 718 [†])	Cr_3C_2 (on 718)	0
7	TZM	TZM	0
8	TiC_2	TiC_2	9.1
9	Cr_3C_2	TiC_2	32.6
10	Cr_3C_2 (on 718)	Inconel 718	0
11	Inconel 718	Cr_3C_2 (on 718)	0
12	Cr_3C_2	K165	72
13	K165	Cr_3C_2	54
14	Chrome plate	Cr_3C_2	18
15	TZM	Inconel 718	0
16	TZM	K165	48 ± 12
17	Inconel 718	K165	24
18	Inconel 718	Chrome plate	6.7

* Cr_3C_2 - 15 Ni

†Inconel 718

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Figure 8. Typical Visicorder Trace

a. Couples 6, 7, 10, 11, and 15

b. Couples 1, 3, 8, 17, and 18

Figure 9. Breakaway Friction, Initial Cycle
(Sheet 1 of 2)

c. Couples 2, 5, 9, and 14

d. Couples 4, 12, 13, and 16

Figure 9. Breakaway Friction, Initial Cycle
(Sheet 2 of 2)

a. Couples 6, 7, 10, 11, and 15

b. Couples 1, 3, 8, 17, and 18

Figure 10. Maximum Static Friction
(Sheet 1 of 2)

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c. Couples 2, 5, 9, and 14

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d. Couples 4, 12, 13, and 16

Figure 10. Maximum Static Friction
(Sheet 2 of 2)

(Figure 11 — average of peak values for Cycles 3 to 10, 50 to 55, and 94 to 99. Data plots were purposely put in sets of four sheets each, for ease of reading. The grouping of curves on each sheet was quite arbitrary; this presentation shows the sets in groups which are ranked from the minimum self-welding or metallurgical bonding forces demonstrated to the maximum. These data were presented to HEDL on December 1, 1971, at a meeting attended by AI, LMEC, and AEC representatives. HEDL and LMEC expressed satisfaction with the results of the test, and stated that there was excellent correlation with test information from other sources.

Inspection of the specimens showed that all but one or two pairs were significantly worn or galled. The least worn specimen, No. 6, was the set with Cr_3C_2 - 15 Ni coating on Inconel 718, which, incidentally, also displayed the lowest friction coefficients. In general, the least wear occurred on those specimens with an Inconel 718 base material. Self-welding was very evident on couples with the Type 316 stainless steel substrate; little or no self-welding occurred on the couples with Inconel 718 base material. Figure 12 shows photographs of the wear surfaces.

The second matrix of test specimens, which was specified by HEDL at the meeting at AI on December 1, 1971, is shown in Table 5.

To expedite testing of the second matrix, some reductions and/or changes in the scope of the test program were made. None of these changes will compromise the results of the tests. The changes consisted of: (1) decreasing the number of wear cycles at each temperature from 100 to 25, (2) recording friction Cycles 1 to 10 and 20 to 25, (3) eliminating the 700°F temperature step, (4) deleting all testing at 800-psi contact pressure, except for the 1160°F - 800 psi dwell, to determine the extent of metallurgical bonding at 0 load, and (5) reducing the measurements of the oxygen content of sodium to those at 450, 1000, and 1160°F (plugging temperatures are still to be run at all temperatures). One addition was made in the program — before increasing the temperature to 800°F for cold trapping, all specimens were to be subjected to 5 wear cycles at 450°F with a 300-psi load, so that a measure of the friction could be obtained before sodium wetting occurred.

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a. Couples 6, 7, 10, 11, and 15

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b. Couples 1, 3, 8, 17, and 18

Figure 11. Maximum Dynamic Friction
(Sheet 1 of 2)

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c. Couples 2, 5, 9, and 14

6507-47101

d. Couples 4, 12, 13, and 16

Figure 11. Maximum Dynamic Friction
(Sheet 2 of 2)

a. Couples 1 through 4

b. Couples 5 through 8

Figure 12. Wear Surfaces of First Test Matrix Specimen Couples
(Sheet 1 of 3)

c. Couples 9 through 12

d. Couples 13 through 16

Figure 12. Wear Surfaces of First Test Matrix Specimen Couples
(Sheet 2 of 3)

e. Couples 17 and 18

Figure 12. Wear Surfaces of First Test Matrix Specimen Couples
(Sheet 3 of 3)

TABLE 5
SECOND TEST MATRIX

Specimen Couple	Rider	Plate	Base
1	LC-1C*	LC-1C	718 Base - Nondimensional finish
2	LC-1C	LC-1C	316 Base - Nondimensional finish
3	LC-1C	LC-1C	718 Base - $\sqrt{16}$ ground finish
4	LC-1C	LC-1C	316 Base - $\sqrt{16}$ ground finish
5	LC-1C	LC-1C	316 Base - Nondimensional finish
6	Chromized	Chromized	718 Base - As received
7	Chromized	Chromized	316 Base - As received
8	Chromized	Chromized	718 Base - $\sqrt{16}$ ground finish
9	Chromized	Chromized	316 Base - $\sqrt{16}$ ground finish
10	Electrolized	Electrolized	718 Base - As received
11	Electrolized	Electrolized	316 Base - As received
12	Hastelloy C	Hastelloy C	
13	Hastelloy C	718	
14	TiC - 10 Mo	TiC - 10 Mo	316 Base
15	TiC - 10 Nb	TiC - 10 Nb	316 Base
16	K 151 A	K 151 A	316 Base
17	316 [†]	316	
18	718 [§]	LC-1C**	** 316 Base - Nondimensional finish

*Proprietary material - Linde Division, Union Carbide Corp.

[†]Type 316 stainless steel

[§]Inconel 718

AI-AEC-13021

The 18 couples in the second test matrix were installed in the friction test apparatus, and testing was started on December 8, 1971. When the sodium temperature initially reached 450°F, 5 wear cycles were run on each couple with a 300-psi contact pressure. Each specimen was then unloaded, and the temperature was raised to 800°F, with cold trapping in progress. A sodium sample, taken at about 700°F and analyzed according to ANL/ST-6, showed that the O₂ content in the sodium was <5 ppm. On Monday, December 13, 1971, the first friction measurements at 400°F (as noted on Figure 7) were run. Thereafter, the test program followed the modified test sequence, up to the 1160°F temperature level. This temperature was reached on December 23, 1971, and the test was put on hold at that temperature until January 3, 1972. The remainder of the test program will be run the first week in January.

A portion of the data from the second test matrix has been reduced. This information, which shows breakaway coefficients of friction versus temperature, is tabulated in Table 6. Because this test data does not show values at the higher temperatures, and because the static and dynamic friction coefficients are not yet included, it is merely a preliminary preview of the second matrix test results, and should be treated as such.

IV. 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 pressure of 300 and 800 psi.

V. NEXT REPORT PERIOD ACTIVITIES

Testing of the second matrix of friction test couples will be completed. Data, in the form of breakaway, static, and dynamic coefficients of friction, will be reduced, evaluated, and reported. Test specimens will be removed

TABLE 6
SECOND TEST MATRIX BREAKAWAY COEFFICIENTS
OF FRICTION vs TEMPERATURE

Specimen Couple	Pretest Measurements at 450° Before Cold Trapping	Test Temperature				
		(10-cycle sequence) 400° F	Overnite Dwell at 400° F	Overnite Dwell at 450° F	Overnite Dwell at 500° F	Overnite Dwell at 600° F
1	0.13	0.20	0.20	0.21	0.26	0.25
2	0.15	0.16	0.18	0.22	0.19	0.21
3	0.23	0.26	0.17	0.13	0.21	0.21
4	0.32	0.33	0.27	0.19	0.26	0.29
5	0.08	0.13	0.14	0.15	0.19	0.24
6	0.25	0.31	0.25	0.22	0.22	0.25
7	0.08	0.16	0.14	0.22	0.21	0.26
8	0.30	0.48	0.48	0.45	0.41	0.38
9	0.30	0.34	0.42-0.47	0.45	0.53	0.35
10	0.32	0.33	0.34	0.25	0.32	0.28
11	0.29	0.36	0.34	0.39	0.38-0.43	0.32
12	0.21	0.47	0.51	0.39	0.48	0.53
13	0.31	0.34	0.45	0.58	0.44	0.35
14	0.39-0.47	0.43	0.26	0.32	0.27	0.20
15	0.37	0.32	0.39	0.35	0.33	0.38
16	0.30	0.22	0.25	0.32	0.31-0.35	0.30
17	0.25	0.39	0.38	0.35	0.33	0.28
18	0.16	0.23	0.28-0.34	0.23	0.23	0.28

from the test apparatus, inspected, cleaned, and photographed. Several test specimens will be selected (by HEDL) for post-test metallographic examinations.

The need and/or description of a third test matrix has not yet been determined. If a detailed evaluation of the first and second matrix test data shows that additional friction test data are still needed, a third matrix of couples will be defined, fabricated, and tested at AI, under the direction of HEDL and with the approval of RDT.

Program:	Irradiation Effects		
AEC Task:	2 - Irradiation Damage in Cladding and Core Structural Materials		
Project Manager:	G. W. Meyers		
Reporting Period:	October-December 1971	189A:	13495
General Order:	6506	Subaccount:	29621, 29524
		Category:	04-40-02-01.1

Principal Investigators: K. R. Garr, D. W. Keefer, D. Kramer,
M. M. Nakata, 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. MAJOR ACCOMPLISHMENTS DURING FISCAL YEAR 1972

Proton irradiations of Type 316 stainless steel have shown that the average void size increases while the void density decreases, for equal amounts of damage, as the irradiation temperature increases from 400 to 600°C.

The pre-irradiation microstructure of Type 316 stainless steel was demonstrated to have a major influence on the resulting swelling, in samples irradiated in the EBR II as part of X035. Swelling increased with increasing volume fraction of precipitates, which may be related to the loss of carbon from solid solution. Void formation was absent in ferritic Type 405 stainless steel samples irradiated under the same conditions as the Type 316 stainless steel samples.

Ordinary Type 316 stainless steel is more susceptible to helium embrittlement than either high- or low-carbon modifications.

TABLE 7
VOID FORMATION AT 600°C

Helium (appm)	Damage (d/a)	Damage Rate (d/a-sec)	Void Density (/cm ³)	Average Void Size (Å)	Volume Increase (%)	Sample No.
4.5	5	1.0×10^{-3}	7.7×10^{14}	375	3.5	80
5.8	16	6.8×10^{-4}	1.6×10^{15}	390	20	38
5.8	21	8.6×10^{-4}	2.0×10^{15}	590	24	38
~5	25	1.4×10^{-3}	4.7×10^{14}	920	20	82

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

TABLE 8
VOID FORMATION AT A CONSTANT DAMAGE RATE

Damage (d/a)	Void Density (/cm ³)	Average Void Size (Å)	Volume Increase (%)
6.5	4×10^{15}	150	0.78
23	5×10^{15}	280	5.7

TABLE 9
THREE-SECTION SAMPLE IRRADIATED AT 500°C

Section	Helium (appm)	Damage (d/a)	Damage Rate (d/a-sec)	Preliminary Observations
1	0.001	1	1.2×10^{-3}	No voids
2	0.6	5	1.2×10^{-3}	Low void density
3	2.4	20	1.2×10^{-3}	Moderate void density

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

The Seventh Proton Irradiation, which included samples of 20% and 50% cold-worked Type 316 stainless steel, was completed.

III. PROGRESS DURING REPORT PERIOD

A. VOID FORMATION IN PROTON-IRRADIATED TYPE 316 STAINLESS STEEL

Examination of Sample 82, irradiated at 600°C, was completed, where the damage was 25 d/a. The results are presented in Table 7, along with previous data from samples irradiated at 600°C. The swelling of Sample 82 is in reasonable agreement with the older results. The diminished void density and the large increase in void size are probably due to agglomeration of voids. Large voids, formed by the agglomeration of smaller ones, are easily recognized in Figure 13 by their nonsymmetrical shape.

The density of nucleated voids produced by proton irradiation appears to be a function of the damage rate, and not the total damage; void nucleation begins and ends after small amounts of damage. To verify this observation, Sample 56, with 5.9 appm helium, was irradiated at 500°C to two different total amounts of damage. This was done by shielding half of the sample during part of the irradiation. Void counts have been made at 6.5 d/a from one half of the sample, and 23 d/a from the other half of the sample, for identical damage rates of 5.3×10^{-4} d/a-sec. The results are given in Table 8, where it is seen that the additional damage of 16.5 d/a did not increase the void density.

In order to determine the effect of incrementally adding helium during a proton irradiation on void formation, Sample 117 was irradiated in three sections, as shown in Table 9. The sequence was as follows:

- 1) The whole sample received 0.001 appm helium, and was proton irradiated to produce 1 d/a at a damage rate of 1.2×10^{-3} d/a-sec
- 2) Section 1 of the sample was shielded, and the remainder of the sample received:
 - a) Additional helium, raising the concentration to 0.6 appm
 - b) Additional proton irradiation, raising the damage to 5 d/a, at the same damage rate as before

Figure 13. Agglomerated Voids in Type 316 Stainless Steel, Proton Irradiated at 600°C to 25 d/a (Sample No. 82)

5-193

3) Sections 1 and 2 were shielded, and the remainder of the sample received:

- a) Additional helium, raising the concentration to 2.4 appm
- b) Additional proton irradiation, raising the damage to 20 d/a, again at the same damage rate.

The preliminary observations on this sample are given in Table 9. The absence of voids in Section 1 shows that a helium content of 0.001 appm is apparently too low to aid void formation. The low void density found in Section 2 indicates that 0.6 appm helium is only marginally effective in aiding void formation.

Void statistics and swelling to be obtained from Section 3 will be compared with data from other samples in which all the helium was present at the beginning of the irradiation. Although incremental addition of helium is a closer approximation to the neutron case where helium is formed continually during irradiation, the resulting swelling may not be significantly different.

B. VOID FORMATION IN PROTON-IRRADIATED TYPE 316 STAINLESS STEEL + 0.2 Ti

Void counting of the annealed Sample 99T was completed, where the damage was 7.2 d/a. The voids were quite inhomogeneously distributed. Some regions had no voids, whereas other regions had voids which produced a local swelling of 13%. An approximate average value of the swelling is 2%, but this number is subject to considerable uncertainty.

Figure 14 illustrates the inhomogeneous void formation observed. Also of interest is the absence of the small matrix-carbide particles that were present before irradiation. These had precipitated during the bonding of the sample to copper, which is done at 650°C. We may tentatively assign a radiation-induced dissolution mechanism to explain this observation.

It does not appear that titanium imparts significant swelling resistance to Type 316 stainless steel, under present conditions of damage and temperature. This evaluation is based upon a comparison with the swelling results for Type 316 stainless steel shown in Table 7.

a. Region Having High Void Density

17-190

b. Region Having Low Void Density

15-190

Figure 14. Inhomogeneous Void Formation in Annealed Type 316 Stainless Steel + 0.2 Ti, Proton Irradiated at 600°C to 7.2 d/a (Sample 99T)

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C. SEVENTH PROTON IRRADIATION

The Seventh Proton Irradiation was successfully completed, and the samples that were irradiated are listed in Table 10; they are all Type 316 stainless steel.

TABLE 10
SEVENTH PROTON IRRADIATION

Treatment	Temperature (°C)	Helium* (appm)	Damage (d/a, max.)	Sample No.	Purpose
20% CR†	500	5	10	179	Effect of cold work
20% CR	500	5	20	177	Effect of cold work
50% CR	500	5	20	182	Effect of cold work
6	600	5	2	160	Temperature effect
6	400	5	50	152	Temperature effect
6	500	5	20	111	Effect of damage rate
6	500	5	10	133	High carbon, HEDL
6	500	0	10	173	Hydrogen analysis
6	500	5	10	174	Hydrogen analysis

Treatment 6: 1 hr at 980°C + 8 hr at 760°C

*Concentrations are nominal, pending analysis

†Solution annealed and cold rolled

D. EBR-II IRRADIATION EXPERIMENTS

1. Swelling of Type 316 Stainless Steel in X035

Calculations of the swelling of the Type 316 stainless steel samples, based on transmission-electron microscopy, were completed, and the results are shown in Tables 11 and 12. It is noteworthy that variations in the pre-irradiation microstructure could produce a five-fold difference in swelling in annealed samples. The least swelling among the annealed samples was found for the solution annealed condition, with increased swelling for the solution annealed and aged sample. The greatest swelling was observed in the sample recrystallized after cold rolling. Cold work produced the expected suppression of swelling. The average helium concentration in these samples was 1.6 appm, determined by mass spectroscopy.

a. Before Irradiation

6233

b. After Irradiation

10191

Figure 15. Type 316 Stainless Steel, Solution Annealed

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TABLE 11
EFFECT OF MICROSTRUCTURE ON THE SWELLING
OF TYPE 316 STAINLESS STEEL

Treatment	Description	Volume Increase (%)	Figure No.
5: 1 hr at 980C	Solution annealed	0.14	15
6: 1 hr at 980°C + 8 hr at 760°C	Solution annealed and aged	0.26	16
11: 50% CR + 100 hr at 760°C	Cold rolled and recrystallized	0.72	17
12: 25% CR	Cold rolled	very low	18

Preliminary Data: Fluence = 2.3×10^{22} n/cm² (E < 0.1 MeV)
Beginning irradiation temperature = 450°C
Ending irradiation temperature to be determined

TABLE 12
VOID FORMATION IN TYPE 316 STAINLESS STEEL

Treatment	Void Density (/cm ³)	Average Void Size (Å)	Volume Increase (%)
5	1.4×10^{15}	120	0.14
6	1.1×10^{15}	160	0.26
11	2.1×10^{15}	170	0.72

The amount of swelling correlated with the amount of precipitates in the microstructure. The recrystallized sample contained carbide and sigma particles in the matrix, and carbide particles on the grain boundary. Voids were found in the sigma particles themselves. The solution annealed and aged sample had only carbides on the grain boundaries, and the solution annealed sample contained no precipitates. No new precipitates induced by the irradiation were apparent in any of the samples. Figures 15 through 18, as listed in Table 11, show these pre- and postirradiation microstructures.

It is possible that the carbon in solid solution exerts an influence on void formation, and the degree of precipitation is merely an inverse indicator of the carbon remaining in solution. This hypothesis will be tested when the results of proton irradiations on high- and low-carbon Type 316 stainless steel samples are available.

a. Before Irradiation

6518

b. After Irradiation

10343

Figure 16. Type 316 Stainless Steel, Solution Annealed and Aged

a. Before Irradiation

6285

b. After Irradiation

10314

Figure 17. Type 316 Stainless Steel, Cold Rolled and Recrystallized

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a. Before Irradiation

6011

b. After Irradiation

10304

Figure 18. Type 316 Stainless Steel, Cold Rolled

AI-AEC-13021

2. Observations on Type 405 Stainless Steel Irradiated in X035

Table 13 lists the pre-irradiation treatments of the Type 405 stainless steel samples, which were irradiated in the same capsule as the Type 316 stainless steel samples described previously. No voids were observed in any of the samples of this ferritic steel. The irradiation did produce, in all samples, dislocation loops whose nature remains to be determined, and a high density of 25-Å defects which appear as spots. Additional coherent precipitates, 50 to 100 Å in size, were found in the recrystallized sample.

TABLE 13
TYPE 405 STAINLESS STEEL IRRADIATED IN X035

Treatment	Description
5: 1 hr at 980°C	Solution annealed
7: 1 hr at 980°C + 100 hr at 760°C	Solution annealed and aged
10: 32% CR + 24 hr at 760°C	Cold rolled and recrystallized

The average helium content of the samples after irradiation was 1.2 appm, which is unexpectedly high, compared to the Type 316 stainless steel samples, which had 1.6 appm. The composition of this steel is Fe - 15.4 wt % Cr - 0.27 wt % Ni - 0.15 wt % Al - 0.06 wt % C, and the low nickel content has not resulted in a major decline of helium production.

These results are discussed further in the following Quarterly Progress Report, "Integral (n,α) Cross-Section Measurements." The absence of voids in Type 405 stainless steel may be due to the body-centered-cubic structure, or to the early formation of a fine dispersion of precipitates, the black spots observed at a high density. It is not due to a lack of helium.

3. Tensile Tests of Type 316 Stainless Steel Irradiated in X035

A tensile test program has begun for the samples described in Table 11, covering the temperature range of 400 to 700°C. First tests were done at 700°C, and the results are presented in Table 14. The expected decrease in ductility is manifest in all cases, but strength changes present a mixed trend.

TABLE 14
TENSILE TEST RESULTS ON TYPE 316 STAINLESS STEEL
AT 700°C

Treatment*	Condition*	Strength (kg/mm ²)		Elongation (%)	
		Yield	Tensile	Uniform	Total
5	Control	11.7	28.0	26	43
5	Irradiated	12.5	23.8	12	13
6	Control	13.8	27.5	24	49
6	Irradiated	12.6	24.8	19	25
11	Control	10.5	22.1	14	49
11	Irradiated	19.7	26.4	9	16
12	Control	25.9	44.1	4	9
12	Irradiated	32.7	39.6	3	6

*See Table 11

These changes in tensile properties actually arise from a combination of radiation effects, followed by a partial anneal which occurs at a test temperature of 700°C. Results then depend on a pretest time and test duration, making interpretation difficult, and perhaps of little value. Tensile tests within the estimated irradiation temperature range, 450 to 550°C, will give a more faithful accounting of property changes due to the irradiation, since no changes in structure are likely to occur during testing.

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

1. Flat Sample Experiment

The usual technique for proton irradiations employs a flat sample, diffusion bonded to a copper block which serves to remove heat during proton irradiation. However, unknown and uncontrollable stresses may arise in the sample, because its swelling tends to be restrained by the copper. These stresses can be eliminated by employing a liquid-metal bond between the sample and the copper at the irradiation temperature, thus allowing a well-defined stress to be applied. However, it is necessary that the liquid metal: (1) possess adequate thermal

conductivity, (2) wet, and be compatible with, both sample and copper, and (3) have a tolerably low vapor pressure, for use in a vacuum.

Tin appears to be a good candidate, and tests were made for wetting and compatibility with copper and Type 316 stainless steel. Results demonstrated that wetting was satisfactory, but that tin reacted too rapidly with both metals at 500°C. Further compatibility tests were conducted, in which satisfactory protection was afforded to both the copper and Type 316 stainless steel by means of thin layers of nickel and tungsten.

Tests were then performed in the proton beam of the Dynamitron, using samples stressed in uniaxial tension by means of deadweight loading. These tests, which were all of short duration, confirmed that, at full beam power, tin has sufficient thermal conductivity. The wetting of tin on the surfaces of the protected copper and sample was not satisfactory, and some problems in design also became apparent. However, these impediments should be surmounted during the next quarter.

2. Tubular Sample Experiment

An alternative to the use of a flat sample is a rotating tube. This type of sample offers the hope of a closer simulation to a fuel cladding tube, since it is possible to impose a biaxial stress by means of pressurization. However, the difficulties encountered in the conceptual design to date have been somewhat formidable.

It has been decided to remove heat, and maintain constant temperature, by flowing sodium through an annulus within the tube. Other techniques, such as a heat pipe or a condenser, cannot remove heat sufficiently rapidly. The tube will have a wall thickness of <0.001 in., and the pressure necessary to move the sodium at an adequate rate may endanger the integrity of the tube. Availability of a small electromagnetic sodium pump, to operate within the target chamber, is also uncertain.

Heat flow calculations and design work are continuing. No hardware has yet been fabricated. Thin-walled tubing of Type 316 stainless steel has been ordered, and delivery is due in February 1972.

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

Tensile-test results of the 0.13 wt % C "Type 316" stainless steel are presented in Table 15, along with previous results for this material with other carbon contents. All samples were solution annealed and aged, prior to cyclotron helium injection. Helium has the least effect on total elongation in Type 316 stainless steel with the highest and lowest carbon contents; uniform elongations are not affected by helium.

TABLE 15
EFFECT OF CARBON CONTENT ON TENSILE PROPERTIES
OF TYPE 316 STAINLESS STEEL AT 700°C

Carbon (wt %)	Helium (appm)	Strength (kg/mm ²)		Elongation (%)	
		Yield	Tensile	Uniform	Total
0.13	0	14.8	25.3	20	63
0.13	25	14.2	25.5	20	42
0.06	0	13.8	27.5	24	49
0.06	20	9.8	22.3	16	21
0.025	0	11.3	25.7	26	50
0.025	20	10.4	24.9	22	26
0.003	0	11.4	24.6	23	53
0.003	25	12.4	21.3	23	33

Treatment 6: 1 hr at 980°C + 8 hr at 760°C

These results suggest that the distribution of carbides on the grain boundary, their size, and their spacing are responsible for the trends shown in elongation. It is likely that intergranular cracking is reduced in the high- and low-carbon steels by two different mechanisms, which involve the amount of sliding occurring and ease of crack nucleation and growth.

IV. IMPACT ON LMFBR PROGRAMS

The observed influence of pre-irradiation microstructure on the swelling of neutron-irradiated Type 316 stainless steel may be indicative of a carbon effect, since the amount of carbon remaining in solution depends upon the pre-irradiation heat treatment. The most swelling occurred in a sample in which the smallest amount of carbon is likely to have remained in solution. The influence of carbon will be elucidated upon examination of proton-irradiated samples of low- and high-carbon Type 316 stainless steel. Modification of current carbon levels in commercial Type 316 stainless steel may turn out to be desirable.

The absence of voids in the ferritic Type 405 stainless steel is noteworthy. Further irradiation experiments, with fluence goals of 1×10^{23} n/cm² ($E > 0.1$ MeV), are in progress in the X100 Subassembly. Stabilized ferritic stainless steels might have an application in an LMFBR core, if minimal swelling yields a net benefit. Proton irradiations could rapidly determine their swelling resistance at high amount of damage, above 1×10^{23} n/cm² (fast).

V. NEXT REPORT PERIOD ACTIVITIES

- 1) Examination of samples of Type 316 stainless steel, proton irradiated at 600 and 400°C, will continue.
- 2) Examination of proton-irradiated samples of cold-worked Type 316 stainless steel will begin.
- 3) Tensile testing of Type 316 and Type 405 stainless steel samples from X035 will continue.
- 4) Experimentation with the flat-sample configuration for the stress-swelling study will continue.

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Program: Irradiation Effects

AEC Task: 2 - Integral (n,α) Cross-Section Measurements

Project Manager: G. W. Meyers

Reporting Period: October-December 1971

189A: 13495

General Order: 6506

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. MAJOR ACCOMPLISHMENTS DURING FISCAL YEAR 1972

A work plan, describing and identifying the near- and long-term priorities for helium analyses of irradiated pure elements and alloys, has been prepared. Among the next most important fast neutron (n,α) cross sections that are to be measured are those of boron, carbon, nitrogen, and oxygen. These elements were irradiated at several EBR-II locations, but in the form of high-temperature-melting-point compounds. In order to extract the helium from the samples for mass spectrometric measurements, however, a new high-temperature furnace had to be developed and built, and the mass spectrometer vacuum system was enlarged and completely modified. Testing of the high-temperature furnace under mass spectrometer operating conditions has shown that the crucibles, even after prior heating to expel unwanted gases, still evolve more gas than can be adsorbed by the present getter system. Steps are therefore being taken to reduce both the amount of degassing and to improve the gettering capabilities.

In the meantime, the mass spectrometer was put back into operation, and helium determinations were made on numerous alloy specimens that were neutron irradiated for long periods in EBR-II Subassemblies X018, X034, and X035, and on several α -injected foil specimens. Pure elements that were also irradiated in X034 and X035 have been recovered, and each specimen has been positively identified.

Preliminary planning has been made in collaboration with other principal investigators in the Interlaboratory LMFBR Reaction Rate (ILRR) Program. The initial effort will be to study the integrated (n,α) cross sections of ^6Li and ^{10}B in the CFRMF neutron spectrum. These integrated measurements will subsequently be compared with the same integral cross sections obtained in the ILRR Program, but by using radiochemical techniques; and both will then be reconciled with available energy-dependent (n,α) cross-section data. These combined measurements are very important, because they will provide a completely independent and absolute normalization for these two "standard" cross sections.

III. PROGRESS DURING REPORT PERIOD

A. EXAMINATION OF PRIORITIES

A detailed work plan, reviewing the priorities in this task and the reasons behind their selection, was prepared, and submitted to RDT. The plan contained a complete listing of all the samples on hand, and those now in pile or awaiting shipment to Atomics International. Details on the various irradiation histories were also included, along with criteria for future irradiations required for the program. The general order in which specimens will be analyzed for helium content during GFY 1972 and future years was spelled out.

B. MASS SPECTROMETER SYSTEM

The improved vacuum system was tested, and put into operation. Following this, the whole mass spectrometer system underwent a number of calibration runs, which included the refilling of the spiking volumes with accurately known amounts of ^3He and/or ^4He . The modified vacuum system has resulted in a better ultimate vacuum being obtained between sample analyses, and this is accomplished in a shorter period of time.

C. PURE ELEMENT SAMPLES FROM SEVERAL EBR-II IRRADIATION EXPERIMENTS

Eight capsules containing pure elements, compounds, alloys, and separated isotopes, and 3 capsules containing specially prepared ^{10}B specimens, were irradiated in August 1971 during the 62.5-Mw EBR-II Dosimetry Test (Run 50H). These, along with 3 other capsules irradiated in the Thermocouple Experiment (Subassembly X091), have been shipped to Atomics International. Consultations are now in progress with personnel at HEDL, to determine which 4 or 5 of the 8 Run 50H capsules should be opened and analyzed first. The choice will be based on the preliminary results of the dosimetry test, which should identify the combination of capsules which represent maximum differences in neutron spectrum shape. The haste with which the capsules are being prepared is the result of an increased priority being placed by AEC-RDT on this test, in general, and a greater interest in the helium analyses of the specimens irradiated during the test.

Six smaller subcapsules, containing a number of pure elements, compounds, and alloys irradiated for several years in EBR-II Subassemblies X034 and X035, have been opened. All the pure-element specimens that were received have been positively identified, and the alloys are in the process of being analyzed for helium content. Of the 152 specimens included in the capsules, 128 were found. The specimens that were not retrieved in recognizable form included all the samples of magnesium, calcium, sodium chloride, and potassium chloride. These elements and compounds were still retained within their subcapsules, but had become mixed together as a fine powder. Essentially all the specimens of the most important "high priority" elements were recovered, however.

D. HELIUM IN LONG-TERM EBR-II IRRADIATED ALLOYS

Helium analyses were performed on numerous neutron-bombarded and α -injected alloy specimens. Of particular interest are the preliminary results, shown in Table 16, from specimens irradiated in EBR-II Subassemblies X034 and X035, since they show the relative helium production rates of Types 304, 316, 304 + Ti, 405, and Inconel-800 stainless steels. The data shown in the table are measured absolute values of helium concentrations; but they are

TABLE 16
HELIUM CONCENTRATIONS IN EBR-II IRRADIATED ALLOYS

Subassembly Number	Alloy	Location Inside Capsule	Mass of Specimen (mg)	Helium Concentration (appm)
X035	Type 304 SS	Top	3.645	1.32
		Top	2.945	1.36
		Bottom	2.308	1.01
		Bottom	2.340	1.03
	Type 316 SS	Top	2.689	1.63
		Top	1.806	1.66
		Bottom	1.976	1.37
	Type 304 + Ti	Top	2.955	1.15
		Top	2.574	1.17
		Bottom	2.279	1.42
	Inconel-800	Top	2.897	2.77
		Top	2.669	2.91
	Type 405 SS	Top	2.472	1.18
		Bottom	2.092	1.11
X034	Type 316 SS	Top	2.897	8.44
		Top	2.609	8.13
		Bottom	3.034	10.31
	Inconel-800	Top	2.641	14.6
		Top	3.220	13.6
		Bottom	3.753	17.4
X018	V - 20% Ti	Top	1.230	4.32
		Top	1.432	4.50
		Bottom	1.371	5.14
	V - 15% Ti - 8% Cr	Top	1.967	4.40
		Top	1.826	3.91
		Bottom	1.677	4.14

subject to confirmation in the near future, as additional specimens are analyzed. In general, the agreement between duplicate specimens is good, although the difference in helium concentrations occasionally exceeds the estimated 3% (1 σ) standard deviation of a single determination. In the few cases where this happens, the tungsten coil in which the specimen was melted and vaporized will be reheated within a new coil, to release any helium that might have been trapped. When the duplicate specimens have been analyzed and the reheats have been completed, corrections for location of the specimens within the capsules will be made, and helium production rates will be compared.

Also shown in Table 16 are the results of helium analyses of specimens of vanadium-based alloys. Additional analyses have been conducted on some Inconel-744X, also irradiated in EBR-II Subassembly X018, but the results are not ready for reporting.

E. HIGH-TEMPERATURE FURNACE

As a part of the plan to measure the (n, α) cross sections of all elements that might possibly be present in the variety of alloys considered for use in fast reactor core regions, a number of high-temperature-melting-point elements and compounds have been neutron irradiated. To release the generated helium gas from samples, a high-temperature furnace was constructed which is capable of reaching temperatures above 3300°C for the short time necessary for decomposition.

The furnace consists of a small, water-cooled copper chamber, through which 3 pairs of high-current terminals protrude. The terminals support small crucibles, so that they may be resistance heated. The oven chamber is connected, by means of a glass vacuum lock, to an antechamber in which samples are stored under vacuum. A manipulator is provided for moving the samples from a storage rack to the crucibles where they are decomposed.

The procedure for making mass spectrometric determinations of the helium content of a specimen is similar, in principle, to the method used during the last several years. The very small tungsten wire basket crucibles are replaced by crucibles of the higher-melting-point-temperature materials, tantalum carbide and hafnium carbide, when refractory compounds such as tantalum nitride are to be decomposed. For vaporizing relatively large (~ 100 mg) samples of

lower-melting-point-temperature materials, such as stainless steel or vanadium, a larger-capacity, alumina-coated, molybdenum crucible is used. Both types of crucible have been used successfully to melt the types of specimens for which they are intended. Before a mass spectrometer run, both the tantalum carbide and the alumina-coated molybdenum crucibles are separately preheated at maximum temperature until degassing has subsided. When the crucibles are reheated for a short period during a simulated mass spectrometer run, however, the amount of additional degassing is low, but is still too great to be handled in the isolated mass spectrometer system by the present getter arrangement. The released gases have not yet been mass analyzed, but water vapor seems to be one likely candidate, since hydrogen is one of the principal masses in the spectrum. The mass peaks at 3 (HD) and 4 (D_2) interfere with the measurements of helium. Nevertheless, the helium content of an α -injected niobium (mp 2415°C) specimen was measured without excessive error due to the background gases.

Several steps have been, and are now being, taken to reduce the degassing problem. Discs of aluminosilicate glass, which has a much lower gas permeation rate than quartz, have been ordered for replacement of the quartz presently used in the vacuum lock. Thermal shielding of the crucibles will be increased, and all crucibles are being heated for an extended time under vacuum, prior to being loaded (in air) in the furnace, where they are further outgassed before the samples are loaded. Alternative O-ring materials have been obtained for trial in the vacuum lock seal.

If the improvements now being made do not eliminate the persistent degassing, the use of improved getters should eliminate the problem. The zirconium-titanium alloy getters presently used for cleaning up unwanted gases in the mass spectrometer samples have been adequate for most samples analyzed to date. Further improvements need to be made, however, before the performance of the high-temperature furnaces will be completely satisfactory.

At present, the getters are located in an appendage, rather than in the line between the furnace and the mass spectrometer. Locating them in-line should greatly increase their efficiency. Also, more experimentation is needed to find the optimum temperature conditions for their operation. Making these improvements will also decrease the time needed for measuring the helium content of all other samples, because fewer data need be taken and analyzed to correct for background interferences.

IV. IMPACT ON LMFBR PROGRAMS

Now that the mass spectrometer system is back in operation, sample helium analyses are progressing rapidly. Important samples from the long-term irradiations in Subassemblies X034 and X035 are being prepared for analysis, and capsules recently irradiated during the second EBR-II Dosimetry Test (Run 50H) are ready to be opened.

The high-temperature furnace, which is needed for the determination of several very important (n,α) cross sections, will require some more testing before it can be used effectively. Improvements to the mass spectrometer getter system should enable it to handle the increased amounts of unwanted gases that evolve from the furnace.

V. NEXT REPORT PERIOD ACTIVITIES

Four capsules irradiated during the 62.5 Mw EBR-II Dosimetry Test (Run 50H) will be selected and opened, and identification of the pure element samples will commence.

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Program:	Tantalum Control Assembly Development		
AEC Task:	5 - Tantalum Control Assembly Development		
Project Manager:	L. Bernath - G. W. Meyers		
Reporting Period:	October-December 1971	189A:	13645
General Order:	9000	Subaccount:	21310, 21320
		Category:	04-40-02-05.1

Principal Investigators: E. R. Specht, D. Kramer, T. Springer

I. PROJECT OBJECTIVES

The objectives of this task are to evaluate, in detail, the reactor system implications of using tantalum control rods in LMFBR's, and to conduct tests needed to assure the reliable behavior of tantalum control rods in a reactor environment. Mechanical and physical properties, nuclear (decay) behavior, and irradiation damage, especially swelling, are included.

II. MAJOR ACCOMPLISHMENTS DURING FISCAL YEAR 1972

Instrumentation specifications were prepared for the thermal-equilibration and temperature-measuring circuits in the tantalum heating calorimeter. Assembly and checkout of instrumentation was initiated. This equipment will be utilized in the tantalum heating subtask.

III. PROGRESS DURING REPORT PERIOD

A revised Preliminary 189a was completed, and transmitted to RDT.

A preliminary design of a calorimeter for the tantalum heating subtask was developed. Specifications were prepared for the instrumentation required for the thermal-equilibration circuit and for the temperature-measuring circuit.

The thermal-equilibration circuit consists of a copper-constantan thermocouple, with one junction located in good thermal contact with the tantalum sample and the other junction in good thermal contact with the copper heat shield. The emf output of the thermocouple used in the differential mode is amplified by a dc amplifier whose output is fed directly to a 3-mode, current-adjust-type controller. The output of the controller is, in turn, hooked directly to a Teflon-

insulated resistance wire that is wound around the copper heat shield. The temperature difference between the tantalum sample and heat shield is to be maintained to within $\pm 0.005^\circ\text{C}$, and the power available from the controller will be between 0 and 20 mw.

Four methods for measuring the temperature rise in the tantalum slug were investigated, and the efficacy and drawbacks of each were reviewed. The method which will be used initially is the quartz crystal thermometer, which has a standard temperature resolution of 10^{-4}°C , and, for long sampling times, an extended resolution of 10^{-5}°C .

The next most promising technique that was investigated was the thermistor technique, which has the greatest resistivity, but is most subject to radiation damage.

The third most promising method for measuring the temperature change in the sample was the resistance thermometer. Although the use of platinum as the resistance material has been developed to the greatest extent, and is used routinely as a very stable and sensitive temperature-measurement standard, it is an element with a high atomic number, and therefore has a higher probability of interacting with a gamma ray than does tantalum, on a per-atom basis; consequently, it may be useful only for obtaining information on tantalum capture heating.

The fourth measurement technique involved the use of a calorimeter in a cryogenic environment. A germanium thermometer will be employed to measure the temperature of the tantalum. The advantage of making tantalum heating measurements in the cryogenic region lies in the fact that the heat capacity of tantalum increases, by a factor of ~ 100 , compared to its value at room temperature. Thus, a given amount of heat energy deposited in the sample will produce a much larger temperature increase, if the sample is at 10°K , than with the tantalum sample at room temperature.

Work was also initiated on the tantalum cooling, materials, and reactivity subtasks.

Results of the proton-irradiation effort on tantalum are as follows.

A. PROCUREMENT

Three vendors, Wah Chang, Fansteel, and Hamilton Precision, have been queried about their ability to supply tantalum and T-111 in thicknesses <0.0005 in. Hamilton will sell us tantalum at any thickness we need, but they can't answer us yet on T-111. Wah Chang and Fansteel cannot comply with our request. A purchase order to Hamilton, for 50% cold-rolled tantalum with thicknesses of 0.0002 and 0.0005 in., is being processed.

B. SAMPLE PREPARATION

Samples for proton irradiation will be 0.0002 in. thick, so that a 1-Mev proton will pass completely through the sample, producing nearly uniform damage. This will eliminate the time-consuming process of extracting electron-microscope foils from measured depths within the sample. Since these foils are thinner than those used previously, it becomes necessary to determine accurately the extent of copper penetration during the bonding operation.

For this purpose, foils of tantalum, 0.0001 in. thick, were annealed at 2000°C at the Science Center. They will be cut to size, diffusion bonded to copper, and then examined by light microscopy and x-ray fluorescence.

C. FABRICATION OF HIGH-TEMPERATURE SAMPLE HOLDER

Nickel has been chosen as the metal for the new sample holder which will operate at 800°C , and a purchase order for stock is being processed. A supply of a gold-bearing braze, suitable for use at 800°C , has been acquired.

IV. IMPACT ON LMFBR PROGRAMS

The uncertainties in the structural integrity, nuclear worth, and heating and cooling rates to be experienced by the tantalum control rod absorber in the LMFBR Demonstration Plant will be resolved, to permit reliable and economic designs to be developed. This task will provide the engineering information necessary to evaluate the potential of such control rods in LMFBR Demonstration and Target plants.

V. NEXT REPORT PERIOD ACTIVITIES

A. TANTALUM HEATING SUBTASK

A calorimeter will be designed and constructed for measuring the heating of a large (~ 1.50 in. OD) cylinder of tantalum. This calorimeter, and the instrumentation necessary to measure the temperature rise of the tantalum, will be checked out. Experiments will then begin for the calibration of thermoluminescent detectors (TLD's) which will be used for the measurement of tantalum gamma heating in an FBR spectrum.

B. TANTALUM ABSORBER COOLING SUBTASK

Test requirements will be established for an experiment to measure the thermohydraulic characteristics of a tantalum control subassembly for FBR's. The equipment needed for this experiment, and its availability, will also be ascertained.

C. TANTALUM MATERIALS SUBTASK

Tantalum samples will be prepared for proton irradiation. Tantalum materials data, particularly neutron irradiation information, will be reviewed. Definitive recommendations will be transmitted to RDT on the amount and type of materials information that is still needed to design a reliable tantalum control absorber.

D. TANTALUM REACTIVITY SUBTASK

The compilation of all useful tantalum worth measurements will be completed.

Program:	Fuel Transfer Machine Component Test		
AEC Task:	7 - Fuel Transfer Machine Component Test		
Project Manager:	K. W. Foster - G. W. Meyers		
Reporting Period:	October-December 1971	189A:	13638
General Order:	9002	Subaccount:	24100
		Category:	04-01-61-03.5

Principal Investigator: G. E. Berg

I. PROJECT OBJECTIVES

A. SUBTASK 1 - FUEL TRANSFER MACHINE TESTS

Attempt to prove, by environmental test, the ability of a fuel transfer machine, without the use of rotating plugs, to successfully locate, grapple, transport, reorient, and deposit Demonstration Plant fuel in various locations within the reactor vessel, through the full range of expected fuel distortions arising from rated fuel burnup.

B. SUBTASK 2 - FUEL HANDLING MACHINE TESTS

Establish, through environmental test, the operating limits of the fuel handling machine decay heat removal system for normal and failure modes.

C. SUBTASK 3 - ANCILLARY COMPONENT TESTS

Develop, through design and bench testing, equipment necessary to support major fuel handling system component operation, and demonstrate the suitability of this ancillary equipment through environmental test.

II. MAJOR ACCOMPLISHMENTS DURING FISCAL YEAR 1972

None. Task was begun this quarter.

III. PROGRESS DURING REPORT PERIOD

A revised Form 189a was transmitted to RDT. A literature survey of previous articulating arm machine designs was started. In parallel, concept sketches have begun, to define requirements and reactor system problems of

using an articulating arm machine in the FBR Demonstration Plant design with under-the-shield refueling.

IV. IMPACT ON LMFBR PROGRAMS

A. SUBTASK 1 - FUEL TRANSFER MACHINE TESTS

Use of an articulated arm refueling machine for under-the-shield refueling could eliminate the need for rotating top shield plugs, which could result in simplification and cost reduction in the top shield design. However, the articulated arm concept has not been brought to a state of development comparable to that of the refueling machines which are used with rotating shield plugs and which employ simple push-pull vertical motion only. This project will attempt to develop a machine that can be used in a reactor refueling system.

B. SUBTASK 2 - FUEL HANDLING MACHINE TESTS

Verification of the heat rejection capability of the ex-vessel fuel handling machine could eliminate the space required for in-vessel storage of spent fuel, and reduce the fuel inventory, by allowing direct removal of fuel without long periods of in-vessel storage to allow for fuel decay. Uncertainties and potential risks associated with failure of machine cooling system or a fuel assembly stuck in the top shield would also be reduced.

C. SUBTASK 3 - ANCILLARY COMPONENT TESTS

Improved reliability and simplification could result from successful testing of equipment, such as an ultrasonic core sweep, necessary to support major fuel handling system component operation.

V. NEXT REPORT PERIOD ACTIVITIES

A. SUBTASK 1 - FUEL TRANSFER MACHINE TEST

A document, listing the functional requirements of an articulated arm fuel transfer machine with no rotating plugs, will be completed. Sketches of articulated arm machine concepts, and the resulting fuel handling and reactor systems, will be prepared, to allow selection of the most promising concept for further design and development. Conceptual design of the selected system will

be completed, and preliminary design will begin. In the event that sufficient information has not been accumulated to allow final selection of an articulated arm concept, several concepts may be carried into conceptual design, to allow a final selection to be made between them. This could delay the start of preliminary design.

B. SUBTASK 2 - FUEL HANDLING MACHINE TESTS

No activity planned for Fiscal Year 1972.

C. SUBTASK 3 - ANCILLARY COMPONENT TESTS

No activity planned for Fiscal Year 1972.

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Program: Cover Gas Seals

AEC Task: 11 - FFTF Fermi Seal

Project Manager: G. W. Meyers

Reporting Period: October-December 1971

189A: 13646

General Order: 9006

Subaccount: 81100

Category: 04-40-04-04.1

Principal Investigators: K. Foster, W. Kurzeka, R. Oliva, F. Poucher

I. PROJECT OBJECTIVES

The objective of this test program is to determine a suitable seal material and lubricating medium for the dynamic seal of the IVHM rotating shield plug assembly. The program includes: (1) evaluation of three candidate seal materials for permeability and solubility of oxygen and radioactive gases, (2) testing of the recommended seal configuration under static, dynamic, and dwell-startup conditions, and (3) the development of a preliminary process specification for installation and handling of a Fermi-type seal.

II. MAJOR ACCOMPLISHMENTS DURING FISCAL YEAR 1972

In mid-October 1971, AI was directed to proceed with the FFTF IVHM Fermi Seal Program. The scope of this effort, which is described in an enclosure to AI letter 71AT-2548 ("Feature Test - IVHM Fermi Seal Test"), will be modified to comply with new FFTF requirements specified by the program-authorizing document.

Accomplishments during GFY 1972 are also those accomplished during this report period, and are discussed in the following section.

III. PROGRESS DURING REPORT PERIOD

A literature survey, data analysis, and material evaluation of candidate materials for the Fermi-type seal were initiated in late October, and completed in late November. The results of these studies were compiled in a technical information report. This document states the basis for the selection of the three seal materials which will be procured and tested - urethane, EPR (Ethylene-

Propylene), and High-Nitrile Buna N. Prospective suppliers who have the capability of making the elastomers, as well as fabricating the finished seal, have been contacted, and are preparing bids for the materials for the permeation and solubility tests. Urethane material cannot be satisfactorily extruded, and must be molded. If this material is selected for actual seal testing, special molds will be required. The other two materials can be extruded.

Design of the 6-ft diameter stainless steel turntable which will be used with the AI seal test apparatus was completed. A fabrication contract will be established, based on bids expected in January.

A review of the IVHM Fermi seal design and installation was conducted, as part of this program, and it revealed that insufficient clearance existed above the bolts used to clamp the Fermi seal in place. With the existing design, the lips of the seal (under recommended compression) would contact the tops of the bolt heads, and adversely affect the sealing and wear characteristics of the seal. A recommendation to trim the tops of the bolts in question to eliminate the interference was reviewed by FFTF personnel, and approved for incorporation into the final design.

IV. IMPACT ON LMFBR PROGRAMS

This investigation is a feature test, intended to prove the IVHM seal selection or to recognize problems early in the program. Information gained will be of direct benefit to future LMFBR's employing this same seal concept.

V. NEXT REPORT PERIOD ACTIVITIES

An order will be placed for the 6-ft diameter stainless steel turntable assembly. Preparations will be made to revise the existing test apparatus to accept the new assembly.

Permeability and solubility tests will be run on the three most promising seal material candidates. The results of these tests will establish the material to be used in the fabrication of the test seals.

The Fermi-type seal requirements will be established, and four sets of seals of the same material will be ordered. The four sets will allow flexibility in matching pairs, and will provide at least one spare in the event of damage during installation and/or testing. The best matched pair will be selected for use in the CRCTA.

Tests will be started to determine the static and dynamic sealing capability, friction drag, and seal life of the Fermi-type seal. Useful information gained concerning assembly procedures will be documented.

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Program:	Cover Gas Seals		
AEC Task:	11 - FFTF-LMFBR Seal Test Program		
Project Manager:	G. W. Meyers		
Reporting Period:	October-December 1971	189A:	13646
General Order:	9006	Subaccount:	81200
		Category:	04-40-04-04.1

Principal Investigators: K. Foster, W. Kurzeka, R. Oliva, F. Poucher

I. PROJECT OBJECTIVES

The objectives of this program are: (1) to conduct static and dynamic tests to demonstrate or determine the mechanical performance of full-size (cross-section) FFTF fuel transfer machine and reactor vessel head seals intended for use in a sodium vapor - inert gas environment, (2) to develop and demonstrate these FFTF seals, or new seal configurations, to attain seals with acceptable fission product and cover gas retention capabilities at LMFBR Demonstration Plant operating environmental conditions, other than radiation, and (3) to develop improved seals and seal technology for the LMFBR Demonstration Plants to support the national objective to reduce all atmospheric contaminations to low levels.

II. MAJOR ACCOMPLISHMENTS DURING FISCAL YEAR 1972

Atomics International was directed, in mid-October, to proceed with the FFTF-LMFBR Seal Test Program. Progress, since the initiation of effort, is primarily that achieved during November and December, and is discussed in the next section.

III. PROGRESS DURING REPORT PERIOD

A detailed Work Plan, defining the intent, purpose, and scope of work, was written, and then reviewed, on October 28, 1971, with personnel from ARD and HEDL. General agreement was reached, at that time, as to the content and approach outlined in the draft work plan. ARD and HEDL provided specific comments on the test matrix and content during November. These suggestions were incorporated, and the Work Plan was formally submitted to RDT on December 1, 1971.

A survey of all FFTF seal applications was started in October, and is still in progress. A technical information report is being updated, to document the results of this survey. The elastomer seals for the Instrument Tree, Coldwall Chargeout Fixture, Floor Valves, IVHM, and CLEM have been reviewed and tabulated. Seals utilized in the Interim Decay Storage are being studied. A summary of seals in other FFTF components will be incorporated in the report, as received from HEDL and ARD.

The survey of available elastomer materials is continuing. Known materials are being evaluated; catalogs have been requested from the major U. S. seal manufacturers; pertinent government specifications have been ordered; trade magazine articles have been reviewed. The study is currently concentrating on LMEC Information Center reports concerning material characteristics when in contact with sodium vapor and reactor gases. Particular attention is being made, in this material evaluation, to the radiation-resistance characteristics of the various elastomers. Materials which are currently being considered for seal applications are as follows:

1) Urethane	}	Will be investigated for use as the IVHM seal.
2) EPR (Ethylene-Propylene)		
3) High-Nitrile Buna N		
4) Buna S		
5) Natural Rubber		
6) Butadiene		
7) Butyl		
8) Silicone		
9) Adduct Rubber	}	Not commercially available in USA under these names - promising prospects.
10) Methyl Rubber		
11) Halogenated hydrocarbons (presently not authorized for use with sodium - sodium vapor)		

Material samples from the preceding list, for use in material evaluation tests scheduled to start in January 1972, are being ordered. Samples are being

sought from at least four manufacturers of each selected material. Only those manufacturers who both make the material and fabricate the seal will be considered.

IV. IMPACT ON LMFBR PROGRAMS

While the initial phase of this test program is being scheduled to specifically support seal selection for all FFTF applications, this first phase, and the later programs, will directly support the LMFBR industry, advance the state of the art, and lead to the generation of RDT seal standards for use by the industry.

V. NEXT REPORT PERIOD ACTIVITIES

Information from HEDL and ARD regarding FFTF seal applications will be incorporated into the seal census and study. Both the seal survey and the seal material evaluation studies will be continued, as required, in order to remain current with FFTF seal applications and uses.

Permeability and solubility tests will be started on select elastomer materials. These tests will evaluate all of the seal materials contemplated for FFTF application.

Special test fixtures and test apparatuses required for the static and dynamic seal tests will be designed. Fabrication of these devices will be started.