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MASTER

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ERDA Task:	1- Sodium Removal and Decontamination of Components		
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I. PROJECT OBJECTIVES

This project has several tasks, each with its own specific objectives. One of these tasks is the Sodium Removal Process Development. The first objective of this task was to develop and evaluate techniques for sodium (Na) removal from components by the reaction of sodium with alcohol. The culmination of this program was the incorporation of the Sodium Removal Process in the ERDA Standard F5-9, and a Topical Report presenting the pertinent sodium-alcohol reaction rate data. The second objective of this task is to determine the process parameters for the removal of Na from test specimens and components by the use of evaporation techniques. Work in this area had been deferred since Fiscal Year 1975, except for a feasibility study on the removal of Na from fuel elements by an evaporative technique, conducted during the interim quarter (July-September 1976).

A second task of this project is the Decontamination Process Development. The objective of this effort is to develop a well-characterized chemical decontamination process for application to the LMFBR primary system components that subsequently permits contact maintenance and allows requalification of the components for reuse in reactors.

A new task, decontamination by sputtering, was initiated last quarter. The objective of this task is to determine the feasibility of using argon ion sputtering as a method for removing diffused radioisotopes from LMFBR components.

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II. MAJOR ACCOMPLISHMENTS DURING FISCAL YEAR 1977

The major accomplishments during the Fiscal Year 1977 are summarized as follows:

A. SODIUM REMOVAL STUDIES

- 1) The high temperature reaction rate tests on sodium and denatured ethanol in a pressurized system were completed. The reaction was found to be smooth, and slower at temperatures up to 121C (250F) than at temperatures below the 78C (173F) -boiling point of the alcohol.
- 2) A Topical Report, "Sodium Technology: The Reaction Rates of Sodium with Alcohols", AI-ERDA-13192, was released.
- 3) A paper entitled "Reaction Rates of Sodium with Ethanol and other Alcohols" was presented at the ANS Summer meeting.
- 4) A report "Sodium Removal Process - A Review," (N707TI830016) was issued. The report included a comprehensive review of the water vapor-nitrogen process, the alcohol process, and the evaporative removal process, plus a general survey of numerous other methods of sodium removal.
- 5) The Sodium Removal Process section of the Nuclear Systems Materials Handbook was prepared.
- 6) The final draft of the RDT Standard F5-9 was reviewed for final approval.
- 7) The large vacuum chamber of the Sodium Evaporation tests was refurbished, and coupled with a new Quadrapole Mass Spectrometer which enabled the quantity and mass of released gases to be monitored.
- 8) The parametric tests on the removal rates of Na from surfaces and crevices was completed, and a report (N707TR830011, "The Evaporative Removal of Sodium - Parametric Tests"), was issued.
- 9) A preliminary outline specification (N707TI830020, "Evaporative Removal of Sodium from Components - Preliminary Outline Specification") was issued describing the basic steps of the Na removal process for general components.

- 10) A group of stressed, sodium-wetted tensile specimens was cleaned of sodium by the evaporative process. The post-test mechanical properties of the specimens indicated no deleterious effects of the sodium removal operation. Even though some of the specimens were exposed to severe out-of-control post-test conditions, their mechanical properties appeared normal.
- 11) The Na-wetted IHX Tube Bundle Mockup was successfully cleaned of Na by the evaporative process.
- 12) A report, N707TI830015, "Removal of Residual Sodium from CRBR Spent Fuel Assemblies Containing Failed Fuel Pins" was issued which showed the feasibility of a cyclic application of convection evaporation and vacuum evaporation.
- 13) A Turbomolecular vacuum pump and its associated equipment was procured, and assembly of a new vacuum system was begun. The new system will be used to conduct tests on the removal of Na from mockup fuel assemblies.
- 14) A preliminary outline specification, N707TI830019, "Evaporative Removal of Sodium from Fuel Elements - Preliminary Outline Specification" was issued describing the basic steps of the Na removal process to be used for the fuel assembly cleaning.

B. DECONTAMINATION STUDIES

A sodium deposition loop, for producing well characterized metal specimens with typical hot and cold leg radioactive deposits, was started up and successfully operated. The specimens produced to date are the most useful yet available for decontamination studies.

The program to quantify the metal removal capabilities of the candidate decontaminating agents (selected in the FY 76 screening program) continued in FY 77. The major part of the program resulted in selecting a reference decontaminating process. A specification, N707TI830017, was issued for a process for decontaminating hot leg LMFBR components, April 15, 1977. Revision A was issued June 21, 1977.

A comprehensive technical report, N707TI830018 was prepared, summarizing the technical studies carried out on the acid decontamination process during FY 77.

A feasibility study of sputtering as an alternative method of decontaminating LMFBF components was initiated. The expected advantage of this process is a minimum amount of damage to the component while removing deposited and diffused radioactive isotopes. A progress report, N707TI830021, was issued.

Decontamination of cold leg specimens was initiated, but work was discontinued in order to concentrate on hot leg decontamination. Both FFTF and CRBRP personnel put a higher priority on the hot leg work.

During the course of the cold leg work, both mechanical and chemical methods for assisting acid reagents were developed. Preliminary indications were that a cold leg process can be developed which does not substantially differ from the hot leg process and which can be carried out in the same equipment.

Information that pump designers are considering the use of both 316 and 304 austenitic steel in the sodium had a major impact on the program. Prior information was that only 304 would be used. Immediate action was taken to acquire 316 in the form of both sheet stock and castings. An evaluation program and new laboratory techniques appropriate to 316 are under development.

III. PROGRESS DURING REPORT PERIOD

A. ALCOHOL PROCESS DEVELOPMENT

1. ERDA Standard F5-9

Final approval and release of the ERDA Standard, "Sodium Removal Processes" is still in progress.

B. SODIUM EVAPORATION PROCESS DEVELOPMENT

1. General - Evaporation Rates

The results of the completed series of tests in the three sizes of crevice specimens were plotted as % completion vs time in hours, as illustrated in Figure 1. From these data and the crevice length, the evaporation rates can be calculated to be 30 mm/hr for the smallest crevice, decreasing to 1.27 and

1.15 mm/hr for the larger crevices. These rates are opposite to rates calculated from orifice diffusion theory, that is, the smaller crevice should take the longer time.

This discrepancy is explained by the observed expulsion of molten sodium from the crevice. This expulsion phenomena had been observed in the earlier work.*

The Time vs % Completion curves of Figure 1 also show the magnitude of the expulsion, that is, about 10% of the sodium is expelled from the 0.33 mm (13 mil) crevice, as compared to about 65% from the 0.15 mm (6 mil) and nearly 100% from the 0.025 mm (1 mil) crevice.

If the removal rates are calculated for those portions of the curves after the expulsion has occurred, markedly different rates are found, as noted in Table I. These rates are found to be in the proper order, that is, the larger the crevice the faster the evaporation. The value for the 0.025 mm (1 mil) was estimated based on the observation that the rate of diffusion from an orifice is roughly proportional to the square root of the orifice size.

TABLE I

Evaporation Rates of Sodium from Crevices - Results of Tests at 316°C (600 F) and 1.3×10^{-3} to 1.3×10^{-4} Pa (10^{-5} to 10^{-6} torr).

<u>Size</u>		<u>Overall Rate</u>	<u>Rate after Expulsion</u>
<u>mm</u>	<u>mil</u>	<u>mm per hr</u>	<u>mm per hr</u>
0.025	1	30	0.2*
0.15	6	1.27	0.44
0.33	13	1.15	0.93

*Estimated

Comparison of the two sets of rate values accentuates the great assistance the expulsion phenomena gives in the removal of sodium from small crevices.

*AI-ERDA-13154, "Annual Technical Progress Report-Sodium Technology and Cover Gas Seal Development Programs-Government FY 1975", August 15, 1976.

Figure 1. Vacuum Evaporation Of Sodium From Crevices, 316° C (600° F) And 1.3×10^{-3} to 1.3×10^{-4} Pa (10^{-5} to 10^{-6} torr)

The est data from the earlier work, (op. cit.) on bolt specimens and an open cup specimen were plotted on Figure 2, plus other data, including the data generated in this test. The central curve on the Arrhenius plot fairly well defines the slope, that is, the effect of temperature on the rate. The open cup data, where no orifice diffusion is present, is represented by the lower curve.

Surprisingly, the data point for the 0.025 mm (1 mil) crevice lies almost on the central curve. However, this is only a coincidence - the slower diffusion being offset by the expulsion phenomena, resulting in an experimental time nearly equal the calculated value, whereas the actual time would be expected to be much greater than the calculated time.

The data points for the 0.15 mm (6 mil) and 0.33 mm (13 mil) lie well above the central curve, and quite close together. In actuality, these curves should be well separated, but the large amount of sodium expelled from the smaller crevice significantly reduced the time to completely remove all the sodium.

Two other specimens were tested to obtain other data points. These specimens were 7.77 mm (0.306 inch) I.D. stainless steel tubes, welded closed at the bottom, containing a 100 mm (4 inch) column of sodium. The specimens were tested for 25 and 100 hours. After 25 hours, 75% of the sodium was removed, and after 100 hours all of the sodium had been evaporated, providing a rate of 3 mm/hr.

Referring to the rates shown in Figure 2 and Table I, it can be calculated that the real time to remove sodium from the crevices would be at least 700 hours for the 0.025 mm (1 mil) crevice and about 350 and 165 hours, respectively for the 0.15 mm (6 mil) and 0.33 mm (13 mil) crevices. These values show the extreme time reduction gained by the expulsion of the sodium from the crevices. This expulsion is postulated to be produced by the release of gases dissolved in the sodium. One less apparent, but very beneficial effect of the expulsion is that the expelled sodium carries any impurities to the surface, whereas these impurities (oxides, and other materials in the sodium) normally would remain in the crevice as the sodium evaporates. Since the expulsion phenomena is more evident with very small crevices (0.025 mm or 1 mil) or shrink fit clearances where solvents or reactants do not penetrate readily, the benefits are even greater.

Such crevice sodium is not necessarily evaporated after expulsion, but may drop to cooled collector plates for later removal. The nature of the gases which may produce this expulsion, and their potential use as end-point control media are still under investigation.

Again reviewing the data presented on Figure 2, it can be estimated that sodium is evaporated from open pools at 316 degrees C (600 degrees F) at a rate of about 3 mm/hr, based on the open cup data which agrees with the rate of removal of sodium from tubes or deep holes.

Medium size bolts, 12.5 mm (1/2 inch) diameter by 90 mm (3-1/2 inches) long with either tightly pressed ("O" clearances) or 0.025 mm (1 mil) spacings were cleaned of sodium in 8 hours. Different times could be expected for bolts of different types and sizes. These observations are summarized in Table II.

Lowering the evaporation temperature to 260C (500F) will apparently increase the processing time by a factor of 5 which becomes prohibitive for medium-sized crevices. Increasing the temperature to 371C (700F) will apparently reduce the processing time by a factor of 4. There is no reasonable objection to the use of the higher temperature, if the pumping speed of the vacuum system is adequate to handle the increased load of gases.

Using the evaporation rate data at the selected evaporation temperature, and the estimated residual sodium in open pools, crevices, etc., from the component drawings, a reasonable estimate of the total evaporation time can be made. For example, if an open, undrained pool is present with a depth of 75 mm of sodium, about 25 hours would be required at 316C (600F). On the other hand, if the sodium is in a narrow crevice of 0.15 to 0.33 mm (6 to 13 mils) with the same depth, it can be estimated that about 75 hours would be required.

It was concluded from these tests that an ordinary diffusion-pumped vacuum system, capable of achieving pressures of 1.3×10^{-3} to 1.3×10^{-4} Pa (10^{-5} to 10^{-6} torr), is adequate to effectively evaporate sodium from hardware, and the sodium can be evaporated from the surfaces of components at a temperature of 316C (600F) in a few hours, depending on the thickness of the layer.

TABLE II

Evaporation Rates of Sodium from Test Hardware at 316C (600F) and 1.3×10^{-3} to 1.3×10^{-4} Pa (10^{-5} to 10^{-6} torr)

<u>Type</u>	<u>Size</u>		<u>Removal Rate</u>
	<u>mm</u>	<u>mil</u>	<u>mm/hr</u>
Open pools	-----		3
Deep holes	75	300	3
Crevice	0.025	1 (or less)	30
Crevice	0.15-0.33	6-13	1
Bolts	0-0.025	0-1 (gaps)	--- (8 hours)

The results of these tests were incorporated into a Test Report, N707TR830011, "The Evaporative Removal of Sodium - Parametric Tests", Sept. 13, 1977. Also a preliminary outline specification, N707TI830020, "Evaporative Removal of Sodium for Components - Preliminary Outline Specification", was issued on Sept. 27, 1977.

2. General-Sodium Evaporation from Stressed Tensile Specimens

Twelve Type 304 stainless steel tensile specimens, fabricated to AI Drawing N707000018, were placed in the test holders (Figure 3) and wetted with sodium for 48 hours at 565C (1050F). The specimens were transferred to an N₂ atmosphere box, their lengths measured, and the tensile bars stressed to 100% of 0.2% offset yield.

The specimens were subjected to various atmospheres and temperatures, etc., as noted in Table III.

During the various operations, the specimens acquired a thin layer of sodium compounds on the surface which was not removed by the vacuum evaporation at 316C (600F) for 100 hrs at 1.3×10^{-3} Pa (10^{-5} torr). Figure 4 shows two typical specimens - one after inert gas storage for 72 hours, and the other after air storage (50% relative humidity) for 72 hours. The later specimen

CAUSTIC STRESS CORROSION TENSILE SPECIMEN

Figure 3

6507- 40747

50% Relative Humidity Air

6507-40748

Dry Inert Gas

Figure 4. Appearance of Tensile Specimens after 72 Hours Storage

TABLE III
TEST SEQUENCE FOR Na-WETTED TENSILE SPECIMENS

	SPECIMEN NUMBER			
	1, 2, 3, 4, 5, 6	7,8,9,10,11, 12	C1	C2
Argon Store Air Store 8 Hrs.	1-6	7-12		(Referee)
Evaporate Na	1-6	7-12		
Argon Store, 72 Hrs, photograph	1-3	7-9		
Air Store 50% Relative Humidity 72 Hrs, photograph	4-6	10-12		
Disassemble, wash with alcohol, tensile test	1, 4	7, 10		
Bake 204C (400F) in air, 24 Hrs, dis- assemble, wash with alcohol, tensile test	2, 5	8, 11		
Bake 427C (800F) in air, 24 Hrs, dis- assemble, wash with alcohol, tensile test	3, 6	9, 12		

may be seen to have a wet appearance, while the former appears dry.

When the holders were disassembled at the end of the test, no trace of metallic Na was observed. The specimens were cleaned of any Na compounds and tensile tested to determine the effects of the treatment on their mechanical properties; Table IV shows the results of their tensile testing.

TABLE IV
MECHANICAL PROPERTIES OF TYPE 304 TENSILE SPECIMENS
AFTER
EVAPORATIVE REMOVAL OF SODIUM PLUS VARIOUS EXPOSURE
CONDITIONS

Specimen #	Air Store 8 Hrs	Air Store 8 Hrs 50% RH	Evap. Rem. Na 100 Hrs	Air Store 72 Hrs	Air Store 72 Hrs 50% RH	No Treatment	Air Bake 400F 24 Hrs	Air Bake 800F 24 Hrs	0.2% Yield Strength psi	Ultimate Strength psi	Elong. %	Red. in Area %
1	X		X	X		X			39,100	91,100	70	77
2	X		X	X			X		39,700	89,800	68	79
3	X		X	X				X	41,000	90,000	64	78
4	X		X		X	X			39,700	89,800	66	78
5	X		X		X		X		38,100	89,900	66	77
6	X		X		X			X	44,200	89,800	64	77
7		X	X			X			43,400	90,000	65	77
8		X	X				X		39,900	89,200	63	78
9		X	X					X	41,800	89,800	63	76
10		X	X			X			39,500	90,100	64	76
11		X	X				X		39,200	89,900	65	77
12		X	X					X	40,000	89,600	63	76

In general, little difference is noted in the mechanical properties of the twelve specimens. However, Specimens 1, 2 and 3 do show increasing yield strengths and decreasing % elongation as the post treatment varies from "none" to "Airbake 204C (400F) to "Airbake 427C (800F)", respectively. It is risky to make a firm judgement on single specimen tests, but a trend might be present. Metallographic examination of the tensile specimens, including the threaded portion, are underway, and the specimens will be checked for any evidence of caustic stress corrosion.

Based upon the mechanical property data only, it must be concluded that no obvious caustic stress corrosion has occurred as a result of the evaporative removal of sodium or the various pre- and post-treatments.

3. General-Sodium Evaporation from the IHX Tube Bundle Mockup

The IHX Tube Bundle Mockup (Figure 5) which had been used as a cleaning demonstration model in the Alcohol Process was assembled and wetted with sodium at 565C (1050F) for 48 hours. The Mockup was transferred into the Vacuum Chamber which had been equipped with a pair of flat plate heaters to accommodate the large test specimen.

The sodium evaporation was conducted for 100 Hrs at 316C (600F) at 1.3×10^{-3} Pa (10^{-5} torr). No evidence of metallic Na was found upon disassembly of the Mockup, and the vacuum evaporation operation was judged to be excellent in its sodium removal ability to remove Na from the surfaces, the bolt holes, and the crevices built into the Mockup.

4. Fuel Assembly Cleaning

All of the major components of the special vacuum apparatus needed for the evaporative removal of Na from fuel assemblies were received, including the Turbomolecular Vacuum pump, refrigerator unit and power supply.

Currently, the 150 mm (6-inch) diameter piping, flanges, etc., are being welded, and the valving connected to mate the new apparatus to the large Stokes roughing pump outside Bldg. 042, Santa Susana, and the present vacuum

Figure 5. Layout of IHX Tube Bundle Mockup

chamber inside the building. This will enable the new Turbomolecular pump to be used for either the fuel assembly cleaning or the general component cleaning.

It is planned to construct the fuel assembly cleaning chamber next quarter.

In preparation for the coming tests, a preliminary outline specification was issued (N707TI830019) "Evaporative Removal of Sodium from Fuel Elements - Preliminary Outline Specification", Sept. 26, 1977.

C. DECONTAMINATION STUDIES

A major activity during this period was the preparation of a report, N707TI830018, presenting the decontamination, metal removal and metallurgical data leading to the selection of glycolic-citric acids as the reference agent for decontamination 304SS hot leg components. The preliminary specification for this process was issued in April, N707TI830017.

Another development was the confirmation that CRBRP sodium pump components may be fabricated from either 316SS or 304SS. Both may be present in the same pump. This situation introduces a major new variable into the decontamination program.

Other work included the completion of the process parametric studies and the addition of cast structures of 304 and 316 to the metal removal evaluation program.

The sputtering process evaluation as an alternative to acid decontamination was initiated last quarter and some preliminary results were obtained during this period.

1. Report on FY 1977 Decontamination Studies

Since the reference hot leg decontamination process for 304SS was selected during FY 1977, a comprehensive technical report was prepared describing the basis of its selection. The reagent system selected, a glycolic-citric acid solution, was generally superior to other candidate reagents when all the criteria for maximizing decontamination and minimizing metal damage and waste disposal problems were taken into consideration.

Several technical subjects in addition to process development are discussed in the report. New laboratory evaluation techniques developed during the course of the program are described, with emphasis on the importance of sensitization conditions on metal corrosion in acids. A post-decontamination process was developed to protect the component against subsequent corrosion after it is removed from the decontamination vessel. The necessity for oxygen control at a very low level during decontamination was examined. The use of travelers to determine the process end point is another feature described in the report.

2. Fabrication of LMFBR Components from 316SS

Until recently it has been stated that only 304SS was contemplated for use in components which were to be decontaminated for re-use. It now appears that both 304SS and 316SS may be used for fabricating parts of the same component. This introduces some extremely difficult new variables into acid-type decontamination process development.

Since the specified hot leg decontamination process was based on 304SS, its rate of attack on 316SS is likely to be much too low under the specified conditions. If it is made more aggressive it may produce too much metal damage on 304SS in the time required to decontaminate 316SS.

The effect of molybdenum, an alloying element in 306SS, on sensitization rate is also quite significant, so quantitative information on the effect of sensitization on metal removal rates will be needed. It is true that in a component both alloys will be subjected to the same time-temperature cycle, but the effect on metal removal rate for the two alloys may differ considerably.

Decontamination has not been evaluated for 304SS castings, so now it is planned to evaluate the relative rates of metal removal for casting of both alloys and compare them to rates with mechanically worked specimens. During this report period, suitable suppliers for both castings and sheet stock of 316SS were identified and orders were placed for three carbon levels of both. Eighteen casting "keel blocks" were obtained from a potential pump casting vendor and test specimens are now being prepared.

Preliminary tests with 316SS sheet stock were made using the 304SS sensitization procedure of 1 hour at 1250°F (677 C) and the glycolic-citric acid mixture at 70°C. Little metal removal was obtained. A review of the work of Binder, et al.* indicates that for 316SS, 4 hours at 1350°F may be equivalent to 1 hour at 1250°F for 304SS. Such a sensitization cycle will be evaluated by the nitric acid test during the next report period.

*Binder, et al., ASM Trans Quart., 41, 1301 (1949)

3. Completion of Parametric Test on the Glycolic-Citric Acid Process

The reference hot leg process for 304SS based on glycolic-citric acids will subsequently be referred to as the GCA process. As reported in AI-ERDA 13204, April-June 1977, new bench-scale equipment was fabricated to simulate a typical large-scale decontamination plant and a program to measure the effect of significant process parametric variations on metal removal was initiated. This program is complete with the results presented in Table V.

TABLE V
PARAMETRIC TESTS: HOT LEG DECONTAMINATION PROCESS, 304SS
(Specification: 2.5% Glycolic Acid, 2.5% Citric Acid, 70°C — Sparge
Rate 1 ft³/hr per 300 gallons of Solution)

Parameter Varied	Amount	METAL REMOVAL RATE, DAYS TO REMOVE 1 Mi1					
		Vac. Sen. 1 hr at 1250°F				Deposition Loop Sen.	
		Ht 74821	Ht 74966	Ht 8651438	Ht 55183	Ht 74966	Ht 55183
Temp	-3C	27	41	143	30	19	17
	0	27	45	151	33	23	21
	+3C	18	34	110	24	13	12
	+10C	14	26	108	18	10	12
	+20C	9	9	106	35	7	12
Concen- tration of Acid	-10%	24	41	109	28	16	17
	0	17	31	88	19	15	13
	+10%	29	46	231	37	21	21
Sparge Rate	-25%	25	30	139	40	17	22
	0	*	*	*	*	*	*
	+25%	25	46	166	35	18	17

* See duplicate tests under temperature and concentration

The test specimens included four heats of 304 sensitized for one hour at 1250°F (677°C) and two identical heats with radioactive deposits from the deposition loop sensitized for 3000 hours in sodium at 1000°F (538°C). The critical process parameters were varied within the specification limits as shown, and in addition temperature was varied in 10°C steps up to 90°C. Since the specification requires that process control be based on the metal heat with the highest corrosion rate, heat No. 74966 from the deposition loop is the heat which would be used for controlling process time. At 70°C, the process would be run for 17 days to remove 0.75 mil of metal from 74966. A variation of ± 0.25 mil is permitted so the control limits on process time is between 11 to 23 days. On this basis, none of the three parameters tested would cause off-specification results when varied within the specified limits. The simultaneous variation of two or more in the same direction could produce off-specification metal removal.

Actually, the metal removal limits established in the specification are arbitrary since no tolerable amount of metal removal has been established using metallurgical data as a basis. In addition, the diffusion profile for 54 Mn becomes relatively flat after about one-half mil penetration, so that the removal of more than the amount of metal produces a relatively small amount of additional decontamination. Consequently, the accurate prediction of the process time is not a critical requirement.

The opinion has been expressed by some engineers that the typical process time of the GCA process at 70°C is too long. Consequently tests were run at 80°C and 90°C using the same equipment and procedures used in the parametric tests with these metal removal results shown in Table V. Chemical kinetics theory would predict a doubling of metal removal rate for every 10°C increase in temperature and this occurred within the limits of experimental error. At 90°C, the process time would be five days compared to 17 at 70°C.

The most important concern connected with the use of a higher temperature is the possibility of increased metal damage at higher temperature. Metallurgical cross-section photos at a magnification of 250X were prepared for Heats 74966 and 74821, vacuum sensitized, taken from roughly equivalent metal removal tests at 70°C, 80°C and 90°C. Cross-section photos are also shown for Heat 74966, sensitized for 3000 hours in the R/A sodium loop. These photos are shown in Figures 6 to 14.

The differences between the specimens are minor, showing no significant damage in any test. The mechanism of metal removal may differ at 90°C, since a rounding effect is observed in some heats, which may be a desirable effect. Consequently, it is planned to revise the specification to allow a process temperature range of 70 - 90°C, at the user's option.

8931-1

250X

Figure 6. Heat #74966, Vac.Sen., at 70°C, GCA Process

8931-2

250X

Figure 7. Heat #74966, Vac.Sen., at 80°C, GCA Process

8931-3

250X

Figure 8 . Heat #74966, Vac. Sen.,
at 90°C, GCA Process

8931-4

250X

Figure 9. Heat #74821, Vac. Sen., at
70°C, GCA Process

8931-5 250X

Figure 10. Heat #74821, Vac. Sen., at 80°C, GCA Process

8931-6 250X

Figure 11. Heat #74821, Vac. Sen., at 90°C, GCA Process

8932-1 250X

Figure 12 . Heat #74966, Loop Sens., at
70°C, GCA Process

8932-2 250X

Figure 13. Heat #74966, Loop Sens. at
80°C, GCA Process

8932-3

250X

Figure 14 . Heat #74966, Loop Sen., at
90°C, GCA Process

4. Glow Discharge Sputtering

The acid process of decontamination is a chemical process and is inherently affected by chemical variations in the base metal such as alloy type, carbon concentration and grain size. LMFBF components will be fabricated from a large number of different metal heats and compositions so that there is no possibility that all will react with the acid solution at the same rate. Likewise, it may be impossible to protect surfaces which should not be etched, such as bearing surfaces of pumps.

An evaluation of the feasibility of glow discharge sputtering for decontamination has been initiated. One potential advantage of this process is that the chemical variations in austenitic steels should not affect metal removal rates. Another advantage is that it should be relatively easy to shield areas where no metal removal is desired. A disadvantage of the process is the difficulty of achieving a uniform rate of removal from complex shapes.

The most difficult experimental problems are the selection of a suitable power supply and the design of the feed-through and connecting cables.

The vacuum system and power supply design are still in the process of modification so a detailed description of the experimental equipment and procedure will be deferred until a subsequent report period.

Preliminary tests using a stainless steel cathode (where metal is removed) and an aluminum anode (where metal is deposited) have been encouraging. Using planar electrodes about 50 cm² in areas with a 1-inch spacing metal removal rates have been achieved which are higher than rates typically achieved with acids. The cathode surface has a matte finish with tiny pits and no evidence of significant damage.

A Progress Report on work to date, N707TI830021, has been issued. Quantitative results will be reported after the experimental equipment design has been finalized.

IV. IMPACT ON LMFBR PROGRAMS

This program addresses itself specifically to: 1) problems of cleaning surfaces exposed to sodium, 2) investigation and development of the alcohol sodium removal process and the associated process control, 3) preparation of procedures and specifications for the decontamination of sodium-wetted components. This information is directly applicable to the FFTF and CRBRP activities, and the LMFBR Target Plant design program.

V. NEXT REPORT PERIOD ACTIVITIES

Assembly of the mockup fuel assembly apparatus for sodium removal will be completed.

The Test Procedure will be prepared, and initial sodium removal tests begun.

The major activity during the next report period will be the development of information on the behavior of 316 SS using the reference decontamination process. A sensitization procedure will be developed which is comparable to the procedure now used for 304SS. After a suitable procedure is available, metal removal tests with different heats of 316 will be started.

The evaluation of castings will be initiated. Both 304 and 316 castings will be evaluated if a sensitization procedure for 316 is developed in time.

Tests will be run to establish the need for oxygen control in the decontamination process. An evaluation of an industrial instrument which may be sensitive enough to use for the control of oxygen in the decontamination process in the 0-10 parts per billion range will be initiated.

Sputtering feasibility evaluation will continue at a low rate.

The deposition loop operation will be continued and the long term "traveler" investigation using an ion pumped furnace will be initiated.