MASTER

DOE/SF/00824--T6

Program: Sodium Technology

AEC Task: 1 [Sodium Removal, Storage, and Requalification of Components]

Program Manager: G. W. Meyers

Project Manager: J. G. Asquith

Reporting Period: May 1974

Category: HN 04 01 01.5

General Order: 06507

Subaccount: 36103, 36300

189a Number: 13625

Principal Investigators: R. M. Oliva, F. H. Welch

AM03 76 SF00824

I. PROJECT OBJECTIVES

The objectives of this program are to devise, develop, test, and evaluate techniques for sodium removal and storage of test specimens and components, and to expand and refine, by test and analysis, the sodium removal and storage techniques and procedures for use in processing typical LMFBR components.

II. MAJOR ACCOMPLISHMENTS DURING FISCAL YEAR 1974

The AI program in FY 1974 has concentrated on three major tasks: (1) Sodium Removal and Storage, (2) Bag Handling, and (3) Sodium Removal Process Development. The following are the major accomplishments in FY 1974 for the various tasks.

A. SODIUM REMOVAL-STORAGE

Three load roller mechanisms, prototypical of those found in the IVHM, were subjected to simulated operational tests in sodium, bagged, stored, re-tested in sodium, and cleaned. These tests: (1) demonstrated the feasibility of performing manual operations through a plastic bag in the sodium vapors above a pool of hot (400°F) sodium, (2) provided data to assist in the selection of an acceptable water content in the inert storage atmosphere for sodium-wetted components, and (3) established the suitability of several sodium-removal processes for removal of sodium from complex geometrics such as the load roller assemblies. A presentation of the test data is made in TR-707-820-003.

Sodium-wetted Type 304 and A-286 bolt short-term storage specimens which were stored in an inert gas bag for 25 and 76 days, and then rewetted

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

1

DISCLAIMER

This book was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

and cleaned, showed no deleterious effect as a result of the specified wetting-storage-rewetting-cleaning sequence. The results are presented in TR-707-210-001.

Twelve long-term storage bolt specimens which were wetted, cleaned, and then stored (four in bags with no atmosphere control, and eight in a nitrogen bell jar with 50 ppm moisture plus 50 ppm oxygen) for 90 days showed no sodium weeping, damage, or anomalies.

B. BAG HANDLING

Creep data for Film-O-Rap material were obtained by test. In addition, the mechanical integrity of the material was shown to be maintained, even when a 400°F metal plate was dropped onto the surface. Seam strength of Aclar 22A film was determined.

The usefulness of the plastic bag concept for removal and storage of sodium-cooled components was successfully demonstrated in the Load Roller Test. Atmosphere control for this demonstration was maintained by a special Bag Atmosphere Maintenance (BAM) cart, especially designed and fabricated for this purpose.

Fabrication of a 10-ft diameter by 50-ft long demonstration bag and associated end disks and envelope control loops was completed. Preliminary raising and lowering tests and reefing techniques were evaluated, and no problems were encountered. A final test report, TR 707-830-002, presenting the results of the bag tests and the pertinent cart performance characteristics, was prepared.

C. SODIUM REMOVAL PROCESS DEVELOPMENT

Test bolts exposed to 20 wt % alcoholate at 125°F under argon 7-, 14-, 28-, and 56-day periods gave no indication of caustic cracking.

The test facility for use in the alcohol cleaning process development was completed, checked out, and put into operation. Determination of sodium-alcohol reaction rates is now in progress.

Work was completed on a series of tests to establish the threshold(s) (time and temperature combinations) at which surface discoloration (magnetite

or "rust") occurs on Type 304 stainless steel material from exposure to various concentrations and/or solutions of NaOH. This information will be helpful in establishing cleaning process parameters which will exclude or minimize surface deposits on components which will be subsequently reinserted into sodium. Test results are presented in AI-AEC-13122, and also summarized in AI-AEC-13120.

Sixteen separate tests were performed in a program to establish the degree of gap cleaning required before subsequent water immersion can be safely made. Sodium-filled tubes of various inside diameters were placed in water to determine the reaction rates of the sodium. A tabulation of these data is presented in AI-AEC-13120.

Work is in progress on a program to measure sodium-water vapor reaction rates, and using this information to establish the atmosphere suitable for storage of as-drained nondisassembled components.

Attempts to clean a 3-in. sodium-filled valve, using the HEDL reference moist nitrogen cleaning process (7-1/2 vol % steam, 150°F system temperature), were unsuccessful, and the demonstration was stopped after 50 hr because of the abnormally slow reaction rate (see AI-AEC-13120).

Upon the direction of RRD, AI prepared and submitted a detailed sodium-removal program plan, the performance of which will provide the parameters and specific methodology for the alcohol-glycol process and for the evaporative process. Work on this program began in March 1974, and will continue for the next 18 months.

III. PROGRESS DURING REPORT PERIOD

A. SODIUM REMOVAL-STORAGE

Long-term storage continued on 48 Type 316 stainless steel and A-286 bolt assemblies which were previously assembled, torqued to 100% yield, wetted with sodium at 1050°F, and subsequently removed from the sodium pot and cleaned prior to placing the bolts under appropriate storage conditions per the HEDL-approved test plan. Twelve specimens are planned for examination July 1974 after one year of storage.

B. BAG HANDLING

Mylar/Dacron scrim/Aclar plastic film was received at the end of this period. Ten yards of the film will be used in running weld and burst tests. This final evaluation will complete the planned bag handling/fabrication program.

The testing of the Bag Atmosphere Maintenance (BAM) cart was completed last month, and a final report, TR 707-830-002, presenting the results of all bag tests as well as pertinent cart performance (pumping characteristics, dessicator bed and line pressure drops) was prepared.

C. SODIUM REMOVAL PROCESS DEVELOPMENT

1. Alcohol Process Development

a. Reaction Rate Tests

Sodium-alcohol reaction tests are continuing, in order to determine the parameters of the alcohol-glycol ether-water cleaning process, so that an accurately defined process specification can be prepared. These parameters include:

- 1) The reaction rates of sodium with the cleaning solution as functions of temperature, water content, geometry, etc.
- 2) The component temperatures during cleaning, and the subsequent formulation of thermal models which may be used to predict the temperatures of full-scale components
- 3) The process side reactions (reactant degradation, etc.) which may affect the cleaning process and its efficiency.

Current testing is being performed in small (all glass) reaction vessels which are installed in a special loop having an appropriate cooling apparatus and related temperature control instrumentation. Recent modifications to this system make it possible to study sodium-alcohol reactions at bulk alcohol temperatures as low as about 35°F. Through the utilization of sodium-filled glass tubes, the rate and nature of the reaction can be visually observed.

Previous test results on the recession rates of sodium in the glass reaction tubes showed sufficient non-linearity to require a reexamination of the technique

used to cast the sodium specimens. Sodium specimens with uniform densities, that is specimens essentially free of voids, cracks, fissures, etc. are required in order to obtain uniform rates, since such specimen irregularities introduce unpredictable variation of the sodium surface area exposed to alcohol which in turn causes recession rate variations.

A change in the sodium freezing pattern in the glass specimen tube to permit the sodium to freeze upwards from the bottom to the top has apparently improved the sodium uniformity. With this changed procedure, the presence of the various voids due to sodium shrinkage during freezing appears to be minimized.

Preliminary reaction rate tests on two 0.85-in. ID specimens tend to confirm the improved sodium uniformity. These specimens were reacted in anhydrous ethanol* at 120°F and produced reaction rates of 2.50 and 2.58 in/hr. The peak temperatures, noted in the tests by Cr-Al thermocouples imbedded in the sodium, were 155 and 160°F, respectively. As shown in Figure 1, the sample-to-sample agreement is excellent, but these new rate data are significantly slower than the previously obtained reaction rates of 4.5, 5.3, 5.5, and 6.0 in./hr which were reported for specimens with the less uniform densities. Additional testing will be required to confirm these lower rates before they are factored into the system studies.

A special loading tank has been designed and fabricated to facilitate the filling of the glass tube specimens in the sodium. This new system should significantly reduce the time and effort required to prepare a sodium test sample.

b. Thermal Modeling Studies

The effort on the development of thermal models of sodium in slots and holes reacting with alcohol is continuing.

One inch diameter glass tubes (0.85 in. ID) are being cast with a 0.020-in. diameter, stainless steel sheathed Cr-Al thermocouple imbedded in the sodium. These small diameter thermocouples are being used to provide fast response and to minimize heat conduction effect on the thermocouple junctions.

*denatured, typical denaturants — small percentage of methyl, isopropyl, and methyl isobutyl ketone

Anhydrous Ethanol-Sodium Reaction

Test data, obtained to date, have indicated that the temperature of the alcohol inside of the tube is slightly higher than the bulk alcohol temperature. To provide a method for controlling the reaction temperature to obtain direct reaction-rate temperature data, a simple test fixture is being built (Figure 2) which will control the reaction temperature by forced cooling of the outer surface of the test vessel by flowing alcohol.

A TAP model of an earlier test, performed in November 1973, which utilized a partially filled sodium vessel, 4-in. diameter, being flooded with Dowanol PM, flowing at 0.5 gal/min across the sodium surface and splashing over the edge, indicated that a splash cooling-type heat transfer coefficient about 1500 times the normal convection heat transfer coefficient would limit the sodium surface temperature rise to about 1°F. In the actual test, essentially no temperature rise was noted in the sodium.

Based upon the test data and the findings of the computer study, the test fixture, illustrated in Figure 2 was designed.

c. Safety

A process safety evaluation of the alcohol sodium removal process has been initiated. The hazards involved in the storage, transfer, and use of alcohol are being considered as well as those resulting from the liberation of hydrogen by the sodium-alcohol reaction. The significant hazards with either chemical are fire or explosion, with the magnitude of a hydrogen reaction being far more significant. Toxicity is not a major problem with either material.

The recommendations and requirements published by the National Fire Protection Association and the Occupational Safety and Health Administration (SHA) have been studied as have relevant articles in the technical literature. Handbook data on the chemical and physical properties of the materials have been correlated.

The fundamental guide line being followed is the establishment of system safeguards requiring two coincidental equipment failures in the same system before the occurrence of an incident is possible. Checks and monitoring systems will be incorporated which make even one failure unlikely and guarantee its immediate detection should it occur.

FIG. 2
ISOTHERMAL SODIUM-
ALCOHOL REACTION
TEST FIXTURE
3X-SCALE

The net result of this effort will be an alcohol sodium removal safety report and an alcohol sodium removal safety specification. The specification will present the requirements for operation in the two failure mode, while the report will provide background information giving insight into the rationale involved. Both documents will be updated as the alcohol sodium removal program develops.

2. Hot Gas Lance Tests

The preliminary evaluation of the hot gas lance technique (Seeley, Model 63) for the removal of sodium from test components was completed and a report prepared (TI-707-830-006). Table 1 shows the estimated argon temperatures as a function of heater wattage and argon gas pressure.

TABLE 1
ARGON GAS TEMPERATURES (°F) VS ARGON GAS
PRESSURE AND LANCE WATTAGE

Pressure Wattage	8 lbs	6 lbs	4 lbs
350	450	625	725
500	725	875	1050
650	900	1050	1300

(all temperature at 0.25-in. from round tip)

With helium as the gas, the lance temperatures would be slightly less than one-half the temperature noted for the argon.

Based upon these preliminary tests, a larger device is being devised. The new unit will have a 5000 watt heater and will feature a vacuum pickup to remove the sodium as melted to minimize splatter.

3. As-Drained Storage

The program to establish the atmosphere suitable for storage of as-drained components is continuing. The planned approach is to:

- 1) Determine the sodium reaction rate with selected concentrations of moisture and oxygen

- 2) Perform an analytic calculation, and, using this data, establish the hydroxide formation depth vs moisture content for crevice shapes.

Currently, the reaction rate of sodium, when exposed at room temperature to argon containing 50 ppm water vapor and 50 ppm oxygen, is being measured. Under these conditions, it has been concluded, from data in the literature, that the ultimate sodium reaction product will be anhydrous sodium hydroxide. Not until 200 ppm of water vapor is attained, will the lowest sodium hydroxide hydrate be expected to form. A test plan, TP-707-810-006, "Reaction Rate of Sodium Exposed to Argon Containing 50 ppm Water Vapor and 50 ppm O₂ at Room Temperature," is being used as the test guideline.

The initial tests using sodium samples of 6.1 and 3.7 cm² area produced weight gain rates of about 10 and 18 μg of sodium/cm²-hr (on the basis that the reaction product was Na-OH) which are equivalent to 0.004 and 0.0086 mils of sodium/hr. In an effort to obtain better wetting of stainless steel tab by sodium and to improve the precision of the data, a new specimen preparation technique was tried. Apparently the sodium sample became contaminated during preparation by a film of NaOH hydrate and the sample lost weight when heated as the water of hydration was lost.

Work is still in progress on the method of sample preparation, including the use of nickel foil instead of stainless steel. To date, the degree of wetting of the nickel has been no better than that of the stainless steel.

IV. IMPACT ON LMFBR PROGRAMS

This program addresses itself specifically to: (1) the problems of cleaning surfaces exposed to sodium and the subsequent requalification of components for reuse, (2) the prevention of functional degradation by means of proper storage of cleaned components, (3) the development of practical storage containers, (4) the investigation and development of the alcohol-glycol ether sodium removal process and the associated process control (including reagent regeneration, (5) the development of local sodium removal tools and techniques, (6) the development of sodium removal by evaporation via vacuum chambers, and (7) the preparation of procedures to direct the various sodium cleaning operations.

VI. SCHEDULE

During the third quarter, the AI scope of effort on this program was re-directed by RRD to reflect an expansion of the sodium removal process development. The schedule of activities and milestones, Figure 3, was updated to reflect this redirection.

Figure 3

TASK 1 - Sodium Removal, Schedule and Milestones