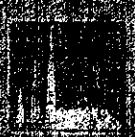

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Isotopic SNAP Program
(Special Distribution)

PACIFIC NORTHWEST LABORATORY MONTHLY ACTIVITIES REPORT FOR MAY 1966

on

Division

of

Isotope Development Programs

by

Members of the Staff of the Chemistry,
Materials, and Applied Physics and
Electronics Departments

Edited by

E. E. Voiland

June, 1966

NOTICE: PRELIMINARY REPORT

This report contains information of a preliminary nature prepared in the course of work under Atomic Energy Commission Contract AT(45-1)-1830. This information is subject to correction or modification upon the collection and evaluation of additional data.

PACIFIC NORTHWEST LABORATORY
MONTHLY ACTIVITIES REPORT
FOR MAY 1966
DIVISION OF ISOTOPE DEVELOPMENT
SUMMARY OF ACTIVITIES

ISOTOPE SOURCE PRODUCTION TECHNOLOGY

Two additional runs (Numbers 4 and 5) were completed in the A-Cell promethium purification equipment. Process and operating innovations were successful in reducing the previous high promethium losses to negligible levels. Each run produced approximately 100 kCi of highly purified promethium.

The first compatibility data were obtained for capsules loaded with actual promethium sesquioxide (Pm_2O_3). Results for capsules held at 1100 °C for 840 hr were in good agreement with earlier (and continuing) work with samarium oxide standin and lend confidence that data obtained with samarium is valid for promethium.

A large number of compatibility experiments are continuing with samarium oxide and various candidate cladding metals and alloys. Rhenium appears to be completely inert to all types of samarium oxide (at least at temperatures to 1100 °C) regardless of oxide impurity content or mode of preparation. A variety of other metals and alloys (some quit reactive with crude Sm_2O_3) are adequately inert to "treated" samarium oxide. Long-term tests are continuing.

Formation of a brittle reaction zone between compatibility couples of stainless steel and several refractory metals heated at 1100 °C was observed. Additional experiments are in progress to determine whether this is a general phenomenon between refractory metals and super alloys and the implications for isotopic heat source design.

Fabrication development effort during the month was concentrated on experimental determination of the parameters involved in the formation, by pneumatic impaction, of capsules of the type required for the Goddard microthruster. A molybdenum clad high-density Sm_2O_3 capsule was successfully fabricated and enclosed (by a second pneumatic impaction) in a Hastelloy X container with dome-shaped ends and support skirt (as required for the revised Goddard microthruster design). In other experiments, it was found feasible to cold press Sm_2O_3 to a density of 70 to 75% of theoretical.

Development of remotized nondestructive testing (NDT) equipment suitable for verifying the integrity of capsules of a variety of isotopic fuels continued. Capsules fabricated by the 3M Compary were examined during the month.

The shielded gloved box facility (for converting purified promethium solutions to fuel-grade oxide) is nearing completion, and cold testing is expected to begin in June. Installation of electrical service to the high-voltage electron beam welder is approximately 80% complete.

A flaw was discovered during the month in the largest (14-in. diam) column of the A-Cell promethium purification complex. This defect will limit purification capacity to ca. 100 to 150 kCi per run pending replacement of the column.

ISOTOPE SOURCE PRODUCTION TECHNOLOGY

PROMETHIUM ISOTOPIC FUELS DEVELOPMENT

A-Cell Promethium Purification (Inventory Account Funded)
(Fission Product Chemistry and Laboratory Facilities)

A 150,000 Ci-scale promethium separation run (Run 4) was initiated on May 11 and completed on May 18. After only two days for turnaround, a second 150,000 Ci run (Run 5) was started on May 20. Run 5 is currently in progress but will be completed by the end of the month. Each run should produce somewhat over 100,000 Ci of purified product (the remaining is recycled).

A major objective in these runs was the reduction of the amount of promethium left behind by prematurely removing columns from the system. Preliminary indications are that this was successful and that the product-plus-recycle yields for both runs will be over 95%. Definitive results will be given next month when all analyses have been completed.

Compatibility Studies - Pm_2O_3 Loaded Capsules
(Fission Product Chemistry)

The three promethium-loaded capsules (described in previous monthly reports) have been sectioned and inspected. When the Inconel cans were opened on a lathe, no contamination was detected. The capsules were then sectioned, polished, and photographed by Radiometallurgy.

Microscopic examination of the etched capsules showed only a slight interaction between Haynes 25 and the oxide, and no interaction between the oxide and Re or Mo–50% Re. A definite interaction was observed, however, between the 304L SS capsules and the Re and Mo–50% Re liners. These results are very similar to those obtained with Sm_2O_3 (described below). Thus, the data obtained with Sm_2O_3 capsules should be applicable to Pm_2O_3 compatibility predictions.

The promethium oxide, after pneumatic impaction and holding at 1100 °C for 840 hr, appeared to be about as hard as Al_2O_3. The Pm_2O_3 was a dark violet color in the massive form, but powder samples obtained with a diamond drill were light violet in color. The sectioned capsules did not appear to pick up moisture from the air and, unlike neodymium oxide, no "flowering" of the oxide was observed in the 2 weeks following sectioning.

Compatibility Studies - Sm_2O_3 Loaded Capsules
(Fission Product Chemistry)

Work is continuing on the compatibility studies with Sm_2O_3 at 1100 °C. Three types of oxide ("process," outgassed, H_2 treated) are being evaluated with five clad materials (304L, Re, Mo–50% Re, W–25% Re, and Ta) for test periods up to 5040 hr. All of the capsules were prepared by step pressing and pneumatic impaction to insure intimate contact between the core and cladding material.

A number of test capsules were sectioned during the month. Little or no interaction was observed between various cladding materials and Sm_2O_3 which had been outgassed at 2400 °F prior to pneumatic impaction. Re, Mo–50% Re, Ta, Ir, and 304L SS all showed excellent resistance to attack when exposed to the oxide for 840 hr. As expected, a capsule lined with Re, and loaded with Sm_2O_3 which was H_2 treated but not outgassed at 2400 °F, showed no interaction between the Re and the oxide after 840 hr at 1100 °C.

In all of the capsules, however, interaction was observed between the 304L SS outer capsule and the compatibility liners. Indications are that the components of the stainless steel diffuse into the liner to form a brittle intermetallic phase. The diffusion is most pronounced with Re and Mo–50% Re, and less noticeable with Ta and Ir. The diffusion layer appears to be about 1 mil thick with Re after 840 hr at 1100 °C. Similar attack was observed when pieces of the compatibility liner were loosely placed in individual 304L capsules (containing no rare earth oxide) and held at 1100 °C for 840 hr. Tests are now underway to see if such interaction occurs between Hastelloy X and the liner materials.

A formal interim progress report on the compatibility investigations to date has been written and will be issued shortly.

Physical Properties Measurement
(Ceramics Research)

There is no progress to report on this activity for the month of May.

Facilities Status and Plans
(Fission Product Chemistry and Laboratory Facilities)

Installation of the shielded glove boxes is nearing completion. The lead glass windows are installed, and the 30 ton press is installed and operating. Cold testing of the completed facility is expected to begin next month.

The previously unused high-capacity 14 in. A-Cell ion-exchange column was filled with resin during the month and found to have suffered a weld (or other) failure between the column and the water jacket. The column had been carefully inspected and tested prior to installation, so it must be assumed that the failure occurred after the cell was "buttoned up" - possibly from accidental application of excessive pressure or vacuum to either the column or the jacket. In any event, the column cannot be operated in its present condition, nor is remote repair practical. As a consequence, operation will be limited to use of the 8 in. (and smaller) columns until current promethium commitments are met. When an adequate stock pile has been established, the cell will be decontaminated sufficiently for entry and installation of a new column. Purification capacity will accordingly be limited to about 100 g (ca. 100 kCi) of purified promethium product per run.

ADVANCED FABRICATION TECHNOLOGY DEVELOPMENT

Isotope Encapsulation - Fabrication Parameter Study
(Materials Process Development)

Experiments to provide encapsulation and densification with precise dimensional control in a single pneumatic impaction were continued, with emphasis on producing a capsule with the dimensions and geometry required for the Goddard Microthruster.

A molybdenum clad capsule was impacted and subsequently machined to the desired Goddard capsule dimension of 1.120 ± 0.001 in. OD. Following machining, the wall thickness was measured by ultrasonic techniques. The wall thickness varied from 0.046 to 0.084 in. This amount of variation is undesirable and techniques for reducing the variation are being investigated.

The molybdenum clad capsule above was loaded into a specially shaped Hastelloy X cladding. The Hastelloy cladding was machined with a cylindrical wall and domed ends. This assembly was inserted into a steel die in an impaction can and the entire assembly was impacted. Dimensional analysis of this piece has not yet been completed; however, the external appearance was excellent. The results indicate that assembly of a complex multiclad configuration can be achieved satisfactorily.

Cold pressing experiments have indicated the feasibility of pressing Sm_2O_3 to a density of 70 to 75%. This was proven in tests simulating a 10 W source. Work is continuing to extend this capability to larger sizes.

Nineteen compatibility test capsules with a variety of materials were prepared (as a service to Fission Product Chemistry).

Advanced Fabrication Development
(Engineering Analysis)

A final formal report on the development work within the Chemical Development Section on pneumatic impaction as a means of encapsulating isotopic heat sources is in preparation and will be authored by V. L. Hammond, C. J. Berglund and B. M. Johnson.

Electron Beam Welder - 324 Building
(Materials Process Development)

Installation of the electron beam welder in the 324 Building is approximately 80% complete.

NONDESTRUCTIVE TESTING DEVELOPMENT

Ultrasonic Testing of Isotopic Heat Source Capsules
(NDT Systems Engineering)

Extensive evaluation of ultrasonic tests for the certification of the weld integrity on 3M Company fabricated isotopic heat sources is in progress. Data from this evaluation, when correlated with destructive data, will provide the required information for the development of a production

type ultrasonic tester. Test parameters under investigation are calibration, reliability, and measurement error for both the depth of weld penetration measurement test, and the weld root crack detection test.

ENVIRONMENTAL TESTING OF ISOTOPIC FUELS
(Process Systems Development)

There is no progress to report on this activity for the month of May.

GODDARD MICROTHRUSTER FUELING
(Materials Department and Chemistry Department)

Robert Viventi and William Young of General Electric's Missile and Space Division, Cincinnati, Ohio (prime contractor for NASA for the Goddard Microthruster Experiment) visited Pacific Northwest Laboratory early in the report period to confer on their changed design requirements for the Goddard promethium capsule. A formal design coordination meeting was held later in the month (May 25-26) at the NASA-Goddard Space Flight Center to finalize design of the thruster. This meeting was attended by representatives of Pacific Northwest Laboratory and the Richland Operations Office. Effect of the design changes is to (1) substitute a rather sophisticated capsule with domed ends (for reentry impact resistance) for the simple cylindrical capsule originally specified, (2) impose more stringent dimensional requirements, and (3) require that Pacific Northwest Laboratory conduct the remote fabrication and assembly of the thruster (since the isotope capsule is now an integral part of the thruster rather than a separately insertable fuel element). It was agreed that Pacific Northwest Laboratory can provide the required service and meet the required delivery schedule - assuming adequate funding availability of rhenium metal (or alloys) in appropriate shapes and wall thickness, and early and satisfactory operability of the high-voltage E.B. welder.