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DP-1206-I

AEC RESEARCH AND DEVELOPMENT REPORT

# SAVANNAH RIVER LABORATORY ISOTOPIC POWER AND HEAT SOURCES

## QUARTERLY PROGRESS REPORT

APRIL-JUNE 1969

## PART I - COBALT-60



DU PONT
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## PART I - COBALT-60

Compiled by

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August 1969

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SAVANNAH RIVER LABORATORY
AIKEN, S. C. 29801

*CONTRACT AT(07-2)-1 WITH THE*
*UNITED STATES ATOMIC ENERGY COMMISSION*

## PREFACE

This report is one in a series on the applied aspects of isotopes that are under study at the Savannah River Laboratory (SRL), and that are of interest as isotopic heat source materials. Principal emphasis is on isotopes that are produced by neutron addition, since these are the materials for which the production capabilities of the Savannah River Plant (SRP) reactors and other facilities can be used effectively. Data for other materials will be included if pertinent -- such as the isotopic or chemical composition of fission products that can be recovered from Savannah River process wastes.

These reports are intended to present data that are useful to system designers and also to potential or active user agencies. The reports thus deal with the following subject areas of SRL programs:

1. Properties and reactions of isotopes useful or potentially useful as heat sources.

2. Information on the irradiation and postirradiation processing of these materials, when the information is relevant to their use as heat sources and is not in a sensitive area of production technology.

3. Development of design data directed toward the use of and manufacturing capability for isotopic heat sources.

The report is issued in two parts: Part I includes only information on cobalt; Part II includes information on the other isotopic heat source materials. Both parts contain principally data from work performed during the report period. Previous reports are listed in the Publications section.

SUMMARY

Techniques were established for fabricating wafers of CoO, (Co,Mg)O, and $CoAl_2O_4$ at >85% of theoretical density, and for fabricating assemblies for irradiation of these wafers to high specific activity. (p 1)

Thermal expansion coefficients and oxidation properties of CoO and (Co,Mg)O were measured. (p 7)

Composition and properties of 44 superalloy encapsulating materials were evaluated. (p 11)

Destructive examination confirmed the satisfactory performance of an inactive "Hastelloy" X capsule at 1000°C for 10,000 hr. (1.14 yr). (p 14)

Radioactive capsules of "Haynes" 25, "Inconel" 600, and "Hastelloy" X reached their goal exposure of 5000 hr at 1000°C. (p 14)

Ten tungsten capsules were purchased for weld development and long-term compatibility tests with cobalt metal at ~1200°C. (p 15)

"Inconel" 702 was recommended as a possible core material for the WANL 30 kw(t) demonstration heat source because of its oxidation resistance and relatively high thermal conductivity compared to other superalloys. (p 16)

## CONTENTS

## PROGRAM

The purpose of the Savannah River Laboratory (SRL) program on $^{60}Co$ is to provide data that will be required for designing, fabricating, and operating $^{60}Co$ heat sources. Primary emphasis is on selecting materials for encapsulating cobalt fuel forms and establishing temperature limits for long-term operation of capsules. Development of specific heat source concepts is not at present included in the scope of the SRL program.

## MATERIALS TECHNOLOGY AND DEVELOPMENT

### HIGH-TEMPERATURE FUEL CANDIDATES

Alloys and compounds of cobalt that have higher melting temperatures than cobalt metal are being investigated for use in $^{60}Co$ heat sources, to provide increased safety and possible fuel forms for thermionic converters. Literature data indicate that cobalt oxide (CoO), cobalt oxide-magnesium oxide solid solutions ((Co,Mg)O), cobalt aluminate ($CoAl_2O_4$), and cobalt-rhenium alloys (Co-Re) would have high melting temperatures and, except for cobalt aluminate, sufficiently high power densities for thermionic converters.[1] The ceramic compounds are the preferred candidates because they are resistant to oxidation and could be fabricated before irradiation. The Co-Re alloys would have to be fabricated after irradiation because of the high cross section of rhenium and its conversion to osmium, which forms a lower-melting alloy with cobalt.

#### Fabrication of Oxide Wafers

A cold-pressing and sintering technique has been established as the reference process to make wafers of CoO, (Co,Mg)O, and $CoAl_2O_4$ for irradiation in forthcoming high-flux reactor charges and subsequent development of refractory, oxidation-resistant heat sources.[1,2] Recent adjustment of the temperatures at which the feed materials are calcined reduced porosity in wafers of $CoAl_2O_4$ and (Co,Mg)O and eliminated $Co_3O_4$ as a contaminant in CoO wafers. Densities of sintered wafers of $CoAl_2O_4$ and (Co,Mg)O were increased to 90% and to 85% of theoretical density, respectively, by changing the calcining temperature of the precipitated hydroxide feed material to 900°C and 750°C, respectively.

The lowest temperature at which $CoAl_2O_4$ is formed must be used to calcine the feed material in order to obtain an active powder with fine particle size that will sinter to high density. High-temperature X-ray diffraction analysis of mixed cobalt and aluminum hydroxides showed that complete reaction to form $CoAl_2O_4$ occurred rapidly above 900°C. Reducing the calcining temperature to 900°C for 15 hours, followed by sintering at 1650°C for 4 hours in air, yielded wafers of 90% of theoretical density. Only 70% dense wafers were obtained previously using feed material calcined at 1100°C for 2 hours.[2]

Wafers of $(Co_{0.48}Mg_{0.52})O$ solid solution have been sintered to 85% of theoretical density using powders calcined at 750°C for 4 hours. Sintering of material calcined at 850°C for 4 hours had produced wafers of 80% density.[2]

$Co_3O_4$ has been eliminated as a contaminant in the powder used to produce wafers of CoO. Thermogravimetric analysis showed that decomposition of $Co_3O_4$ to CoO begins at ~920°C in air at a heating rate of 5°C per minute and is nearly complete at 975°C. Consequently, a minimum calcining temperature of 950°C, rather than 900°C as used previously, has been selected to ensure rapid and complete decomposition of precipitated cobalt hydroxides to CoO.

The surfaces of sintered wafers of cobalt oxide oxidized to $Co_3O_4$ during cooling in air from the sintering temperature. The $Co_3O_4$ was converted to CoO by subsequent heating for 16 hours at 1000°C in argon.

## Slug Fabrication Tests

Present fabrication techniques, in which the cladding is applied by die sizing or swaging, can be used to encapsulate wafers of cobalt oxide into 6063 aluminum slugs for irradiation. Fabrication tests, using stand-in glass wafers, demonstrated that tolerances must be carefully controlled and hardened slug components must be used to prevent breaking the wafers.

Four slugs were fabricated using wafers of borosilicate glass, selected because its tensile strength is lower than that of cobalt oxide, Figure 1. Dimensions were selected to provide a maximum clearance of 0.003 inch on each side of a wafer and 0.012 inch on the diameter. Radiographic and metallographic examinations showed that wafers broke in only one of the slugs; in the other three slugs the wafers were intact and clearances were maintained.

Five of the six wafers in one slug were broken during fabrication, Figure 2. One of the D-bars was deformed and was pressing the wafers into the other D-bar. Hardness tests on the two D-bars showed that the deformed bar, although analyzed to be 6063 alloy aluminum, had a Rockwell hardness of 7 (F scale) compared to 70 for the undeformed D-bar. The D-bars used in the other three slugs also had hardnesses of 60 to 70 (Rockwell F scale).

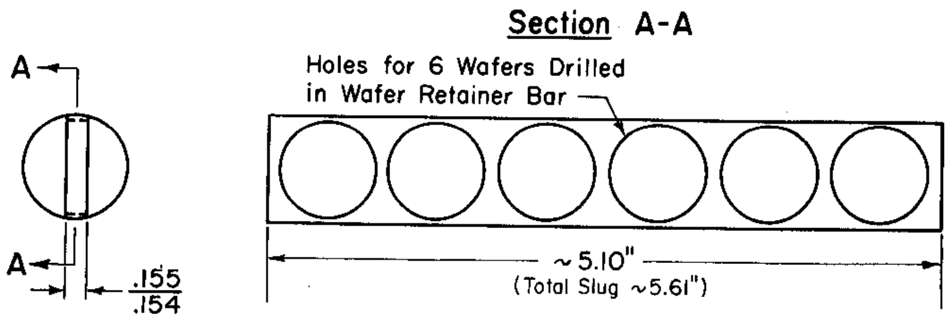



FIG. 1a COBALT OXIDE SLUG - 3-PIECE "D-BAR" ARRANGEMENT
(Before Cladding)

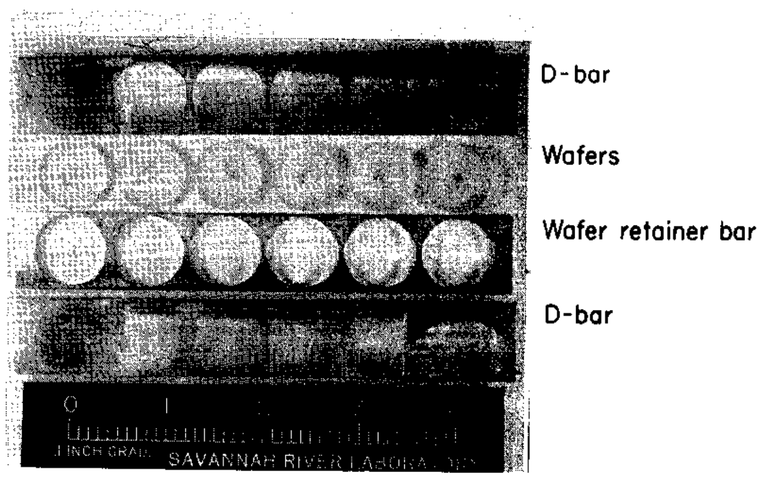



FIG. 1b SLUG ASSEMBLY COMPONENTS

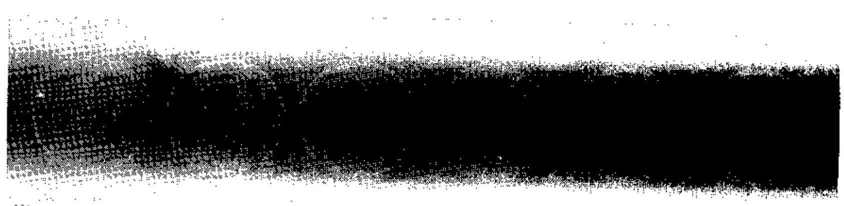


FIG. 2 RADIOGRAPH OF SLUG SHOWING BROKEN WAFERS
(White Lines Are Cracks)

Similar assembly tests will be performed on cobalt oxide wafers. Hardness tests will be made on all slug components, D-bars and wafer retainers, to select only hardened parts for the irradiation tests.

### Compatibility of Cobalt Oxides With Aluminum

Wafers of CoO, (Co,Mg)O, and $CoAl_2O_4$ are not expected to react with the aluminum slug components during irradiation. No reactions were observed between 6063 aluminum wafers and either a CoO wafer at temperatures up to 588°C or a (Co,Mg)O wafer at up to 558°C. Although a $CoAl_2O_4$ wafer showed some reaction at 558°C, no reaction occurred at 467°C. Maximum temperatures expected at interfaces between aluminum and cobalt oxides are below 300°C.

Wafers of CoO, (Co,Mg)O, and $CoAl_2O_4$ were pressed against 6063 aluminum while being heated to between 400 and 600°C to determine the extent of possible reactions during irradiation, Figure 3. Metallographic examination showed no reaction between the aluminum and either (Co,Mg)O wafers after 480 hr at 373, 467, and 558°C, Figure 4a, or CoO wafers after 260 hr at 393, 488, and 588°C, Figure 4b. No reaction was observed between $CoAl_2O_4$ wafers and aluminum at 373 and 467°C after 481 hr, but a slight reaction was observed to a depth of 0.004 inch into the oxide at 558°C, Figure 4c. Compatibility tests will be continued for 3 months at 400, 500, and 600°C.

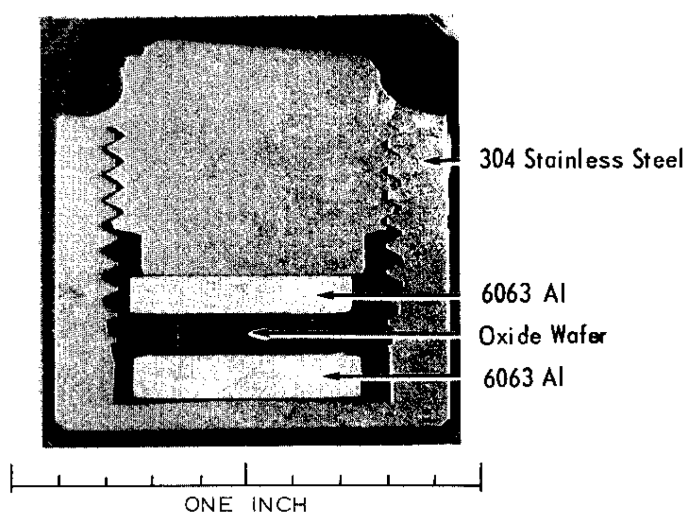
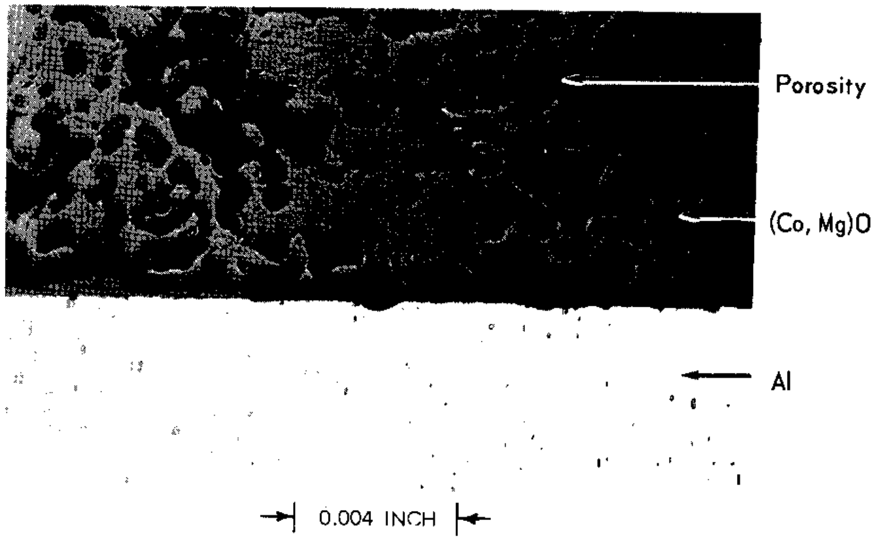
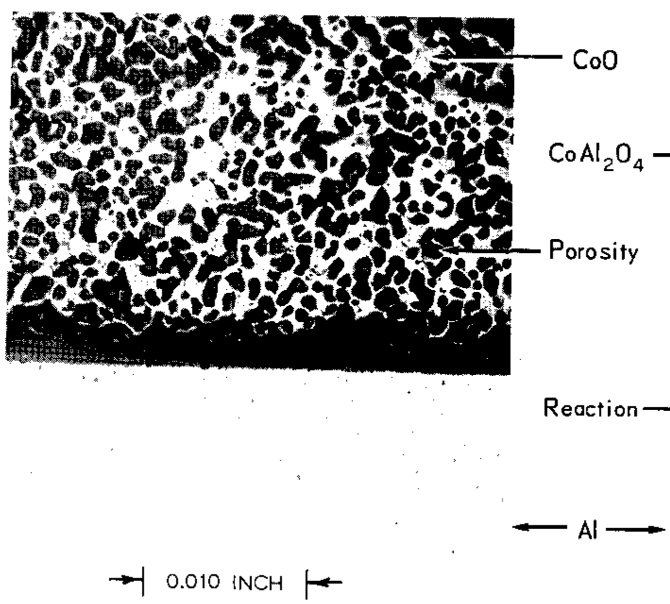



FIG. 3 TYPICAL COMPATIBILITY CAPSULE




a. (Co, Mg)O after 481 hr at 558°C

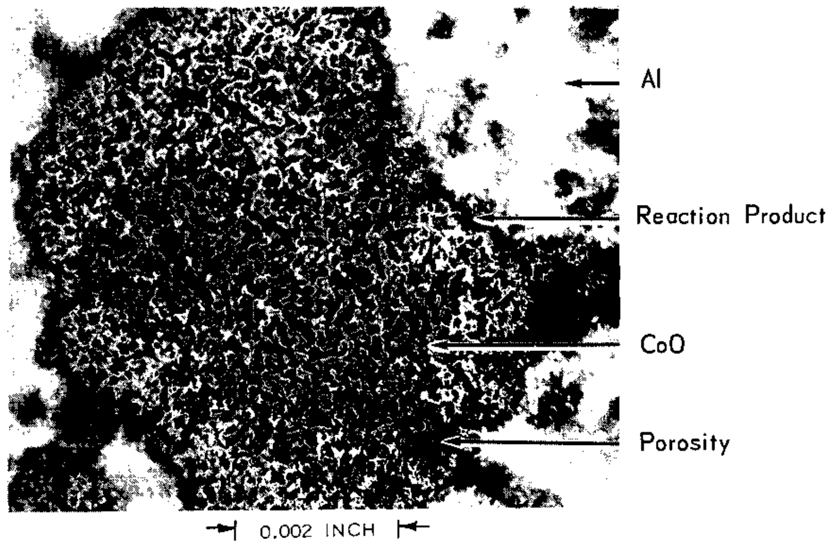



b. CoO after 262 hr at 588°C

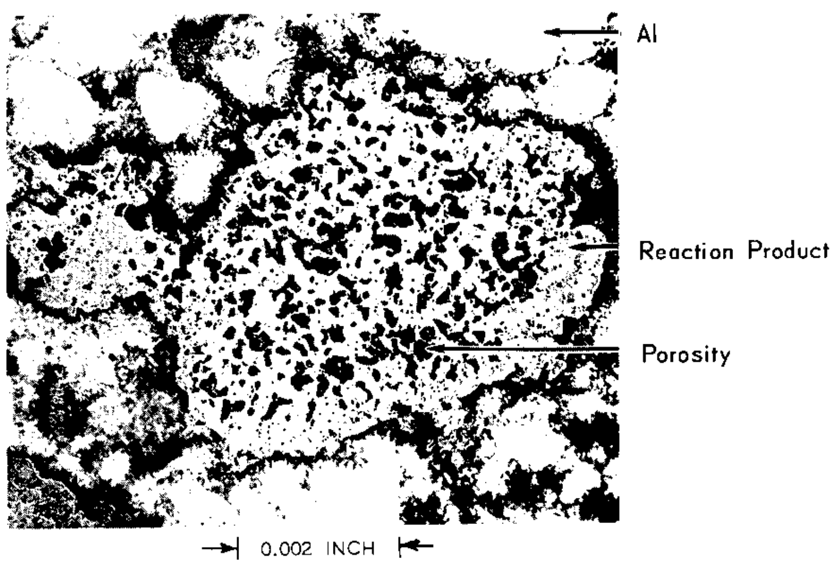

c. $CoAl_2O_4$ after 481 hr at 558°C

FIG. 4 COMPATIBILITY BETWEEN COBALT OXIDES AND 6063 ALUMINUM WAFERS

As a conservative test of interaction, compatibility was also measured with mixed aluminum and cobalt oxide powders that were compacted to shear the protective aluminum oxide coating from the aluminum. Metallographic examination of (Co,Mg)O-Al and $CoAl_2O_4$-Al compacts showed no reaction at 405, 514, or 610°C after 168 hr. CoO-Al did not react at 398°C after 5.5 hr, but showed 5% reaction at 498°C and 50% reaction at 606°C. After 168 hr, CoO-Al reacted 10% at 405°C and 100% at 514 and 610°C, Figure 5.




a. About 5% Reaction at 405°C




b. Complete Reaction at 514°C

FIG. 5 INTERACTION BETWEEN COMPACTED CoO AND ALUMINUM AFTER 168 HR

## Stability and Properties of Cobalt Oxides

General refractory characteristics and thermal expansion coefficients of CoO, (Co,Mg)O, and $Co_3O_4$ (a potential contaminant oxide) have been measured as part of a continuing effort to determine properties of cobalt-60 oxides that affect their production and application in heat sources.[1]

Formation of a solid solution of MgO with CoO reduces the susceptibility to oxidation to $Co_3O_4$ by decreasing the stability range of $Co_3O_4$. The oxidation of CoO-50% MgO powder (calcined in air at 1200°C to remove any $Co_3O_4$ present initially) was characterized by thermogravimetric analysis. Absorbed water was lost below 350°C. Oxidation to $Co_3O_4$ began between 504 and 579°C, increased to maximum rate at 750°C, and stopped after 20 hr at 750°C. The overall weight gain was 0.15%, compared to a possible gain of 7.5% if all the CoO were oxidized. The $Co_3O_4$ formed began to decompose between 750 and 760°C and was completely removed at 906°C. In contrast, similar analyses showed that pure $Co_3O_4$ (as might be formed from CoO) began to decompose at 850 to 935°C; the rate was slow at 935°C.

This value of 850 to 935°C for the decomposition temperature of $Co_3O_4$ agrees with that reported in the literature,[3] but is lower than that observed during fabrication of CoO wafers (see p 2 ). The sources of this discrepancy are being evaluated.

High-temperature X-ray diffraction analyses showed that $Co_3O_4$ has a spinel structure up to 800°C with nonlinear thermal expansion, Figure 6.[4] Above ~800°C, a tetragonal (a = 8.163 ±0.001 Å, b = 8.137 ±0.001 Å at 850°C) distortion of the spinel structure occurs which may be due to slight reduction of $Co_3O_4$. The thermal expansion of CoO at 935°C, 1.22%, is essentially the same as that of MgO. The expansions of MgO and (Co,Mg)O are essentially linear with temperature.

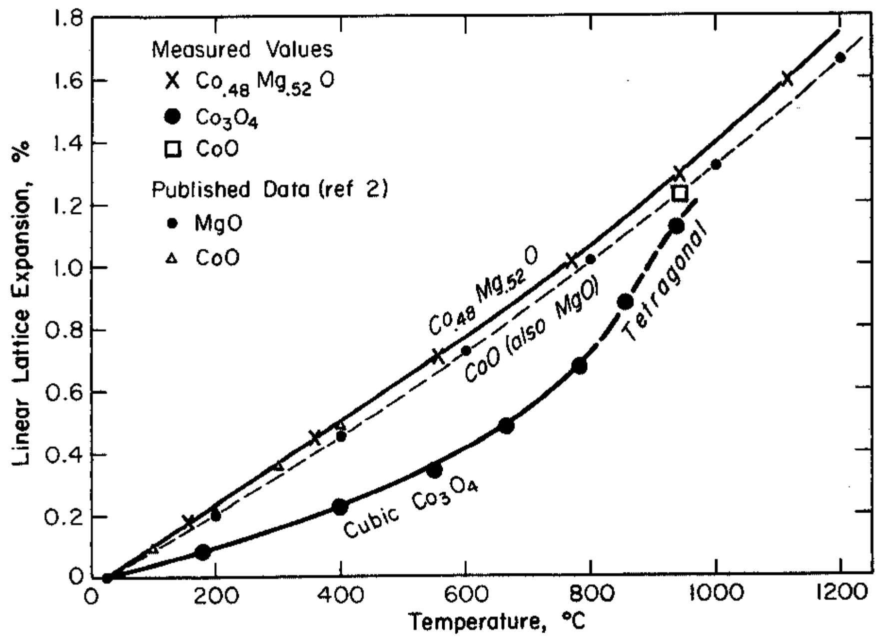



FIG. 6 THERMAL EXPANSION OF CoO, $Co_3O_4$,(Co, Mg)O, and MgO

EVALUATION OF ENCAPSULATION MATERIALS
FOR RADIOACTIVE COBALT METAL

The materials evaluation program is designed to select the most promising alloys for encapsulating $^{60}Co$, to define the limiting operating conditions of these alloys, and to demonstrate capsule integrity at conditions typical of heat source operation. Selection of the most promising alloys is based on extrapolation to the expected service life (1 to 5 years) of short (<500 hr) laboratory data on the properties that affect capsule performance. Limiting operating conditions are defined by the time and temperature dependency of each of the reactions. This selection program and the long-term capsule heating tests are applied to superalloys, serviceable up to 1000°C, and to refractory-metal alloys that are serviceable up to the melting point of cobalt metal (~1500°C).

The predicted behavior of the materials is being verified by 1000-, 5000-, 10,000-, and 50,000-hr (5.7 yr) heating tests of experimental capsules. Current status of tests with superalloy capsules is shown in Tables I and II; similar tests with refractory metal capsules will begin in FY-1970. Tests of companion capsules containing unirradiated or irradiated cobalt measure any effects of the radiation field and the increased nickel content (from radioactive decay of the cobalt) on the performance of the capsule materials.

## TABLE I

## SUMMARY OF $^{59}$Co CAPSULE HEATING TESTS

| Capsule Material | Heating Time, hr | Heating Temp, °C | Wall, mils | No. of Capsules | Approx. Starting Date | Approx. Completion Date | Remarks |
|---|---|---|---|---|---|---|---|
| "Inconel" 600 | 1,000 | 850 | 50 | 1 | 12-66 | 2-67 | Capsule intact |
| | 5,000 | 850 | 50 | 1 | 12-66 | 7-67 | Capsule intact |
| | 10,000 | 850 | 50 | 1 | 12-66 | 1-68 | Capsule intact |
| | 10,000 | 850 | 95 | 1 | 7-67 | 9-68 | Capsule intact |
| | 50,000 | 850 | 95 | 1 | 7-67 | 3-73 | |
| | 1,000 | 900 | 95 | 1 | 11-68 | 12-68 | Capsule intact |
| | → 5,000 | 900 | 95 | 1 | 11-68 | 6-69 | Capsule intact |
| | 5,000(e) | 900 | 95 | 1 | 3-69 | 10-69 | |
| | 10,000 | 900 | 95 | 1 | 11-68 | 1-70 | |
| | 10,000 | 900 | 95 | 1 | 11-68 | 1-70 | |
| | 10,000(e) | 900 | 95 | 1 | 3-69 | 5-70 | |
| | 50,000 | 900 | 95 | 1 | 11-68 | 7-74 | |
| | 1,000 | 1,000 | 50 | 4 | 8-66 | 10-66 | 3 capsules intact; 1 capsule oxidized(b) |
| | 5,000 | 1,000 | 50 | 1 | 4-67 | 11-67 | Capsule intact |
| | 1,000(a) | 1,000 | 95 | 2 | 7-67 | 9-67 | No severe oxidation of Co |
| | 1,000(d) | 1,000 | 95 | 1 | 2-68 | 4-68 | No oxidation of Co or capsule |
| | 5,000(d) | 1,000 | 95 | 1 | 2-68 | 9-68 | No oxidation of Co or capsule |
| | 5,000 | 1,000 | 95 | 1 | 8-67 | 2-68 | Capsule intact |
| | 10,000 | 1,000 | 95 | 1 | 8-67 | 10-68 | Capsule intact |
| | 50,000 | 1,000 | 95 | 1 | 10-67 | 6-73 | |
| | 10,000(d) | 1,000 | 95 | 1 | 11-68 | 1-70 | |
| "Hastelloy" C | 1,000 | 1,000 | 50 | 4 | 8-66 | 10-66 | 3 capsules intact; 1 capsule oxidized(b) |
| | 5,000 | 1,000 | 95 | 1 | 10-67 | 5-68 | Capsule intact |
| | 10,000 | 1,000 | 95 | 1 | 10-67 | 12-68 | Capsule intact |
| | 50,000 | 1,000 | 95 | 1 | 10-67 | 6-73 | |
| | 10,000+ | 1,000 | 95 | 1 | 5-68 | 7-69+ | |
| TD Nickel | 1,000 | 850(c) | 95 | 1 | 10-67 | 12-67 | Capsule intact |
| | 5,000 | 850 | 95 | 1 | 10-67 | 5-68 | Capsule intact |
| | 10,000 | 850 | 95 | 1 | 10-67 | 12-68 | Capsule intact |
| | 50,000 | 850 | 95 | 1 | 10-67 | 6-73 | |
| | 1,000 | 1,000 | 50 | 1 | 12-66 | 2-67 | Capsule intact |
| | 1,000(a) | 1,000 | 95 | 2 | 10-67 | 12-67 | No severe oxidation of Co |
| TD Nickel Chromium | 1,000(a) | 1,000 | 95 | 2 | 10-67 | 12-67 | Co near pinhole oxidized |
| | 1,000 | 1,000 | 95 | 1 | 10-67 | 12-67 | Capsule intact |
| | 5,000 | 1,000 | 95 | 1 | 10-67 | 5-68 | Capsule intact |
| | 10,000 | 1,000 | 95 | 1 | 10-67 | 12-68 | Capsule intact |
| | 50,000 | 1,000 | 95 | 1 | 10-67 | 6-73 | |
| "Haynes" 25 | 10,000 | 850 | 95 | 1 | 11-68 | 1-70 | |
| | 1,000 | 1,000 | 95 | 1 | 10-67 | 12-67 | Capsule intact |
| | 5,000 | 1,000 | 95 | 1 | 10-67 | 5-68 | Capsule intact |
| | 5,000 | 1,000 | 95 | 1 | 5-68 | 12-68 | Capsule intact |
| | 10,000 | 1,000 | 95 | 1 | 10-67 | 12-68 | Capsule intact |
| | 50,000 | 1,000 | 95 | 1 | 10-67 | 6-73 | |
| | 10,000+ | 1,000 | 95 | 1 | 5-68 | 7-69+ | |
| "Hastelloy" X | 1,000 | 1,000 | 50 | 1 | 4-67 | 6-67 | Capsule intact |
| | 5,000 | 1,000 | 50 | 1 | 4-67 | 11-67 | Capsule intact |
| | 5,000 | 1,000 | 95 | 2 | 2-68 | 9-68 | Capsules intact |
| | → 10,000 | 1,000 | 95 | 1 | 2-68 | 4-69 | Capsule intact |
| | – 50,000 | 1,000 | 95 | 1 | 2-68 | 10-73 | |
| | 10,000+ | 1,000 | 95 | 2 | 5-68 | 7-69+ | |

(a) Two capsules, one not welded and one with drilled hole in wall, to test effects of capsule defects.
(b) Capsules reacted with fire-brick. See DP-1094, "SRL Isotopic Power and Heat Sources - Quarterly Progress Report," October-December 1966.
(c) Tests of TD Nickel at 850°C in flowing argon.
(d) Internal atmosphere air instead of helium.
(e) Caustic residue on wafers.
→ New information reported.

## TABLE II

**SUMMARY OF $^{60}Co$ CAPSULE HEATING TESTS**

| Capsule Material | Heating Time, hr | Heating Temp, °C | Wall, mils | No. of Capsules | Activity Spec. Ci/g | Activity Total, Ci | Approx. Starting Date | Approx. Completion Date | Remarks |
|---|---|---|---|---|---|---|---|---|---|
| "Inconel" 600 | 130 | 850 (a) | 50 | 1 | 120 | 16,000 | 2-67 | 2-67 | Swelled due to overheating |
| | 1,000 | ~900 | 50 | 1 | 100 | 5,000 | 4-67 | 6-67 | Capsule intact |
| | 5,000 | ~900 | 50 | 1 | 150 (b) | 15,000 | 4-67 | 10-67 | Capsule intact |
| | 10,000 | ~900 | 50 | 1 | 150 (b) | 15,000 | 4-67 | 6-68 | Increased Co/capsule reaction |
| | 10,000 | ~900 | 50 | 1 | 150 (b) | 9,000 | 5-67 | 10-68 | Increased Co/capsule reaction |
| | 10,000 | 900 | 95 | 1 | 255 (c) | 36,500 | 2-68 | 8-69 | |
| | 10,000+ | 900 | 95 | 1 | 288 (c) | 13,700 | 7-68 | 8-69+ | |
| | 50,000 | 900 | 95 | 1 | 282 (c) | 13,400 | 7-68 | 3-74 | |
| | → 5,000 | 1,000 | 95 | 1 | 295 (c) | 14,000 | 9-68 | 4-69 | **Examination in progress** |
| | 10,000 | 1,000 | 95 | 1 | 288 (c) | 13,700 | 9-68 | 11-69 | |
| | 10,000+ | 1,000 | 95 | 1 | 263 (c) | 12,500 | 9-68 | 11-69+ | |
| | 50,000 | 1,000 | 95 | 1 | 255 (c) | 12,100 | 9-68 | 5-74 | |
| | 10,000 | 850 | 95 | 1 | (d) | - | 9-68 | 11-69 | |
| "Hastelloy" C | 100 | 850 | 50 | 1 | 120 | 9,000 | 1-67 | 1-67 | Capsule intact |
| | 10,000 | 900 | 95 | 1 | 276 (c) | 13,100 | 7-68 | 8-69 | |
| | 10,000 | 1,000 | 95 | 1 | 282 (c) | 13,400 | 9-68 | 11-69 | |
| | 50,000 | 1,000 | 95 | 1 | 270 (c) | 12,800 | 9-68 | 5-74 | |
| "Haynes" 25 | → 5,000 | 1,000 | 95 | 1 | 263 (c) | 12,500 | 9-68 | 4-69 | **Examination in progress** |
| | 10,000 | 1,000 | 95 | 1 | 288 (c) | 13,700 | 9-68 | 11-69 | |
| | 10,000+ | 1,000 | 95 | 1 | 282 (c) | 13,400 | 9-68 | 11-69+ | |
| | 50,000 | 1,000 | 95 | 1 | 295 (c) | 14,000 | 9-68 | 5-74 | |
| | 10,000 | 850 | 95 | 1 | (d) | - | 9-68 | 11-69 | |
| "Hastelloy" X | → 5,000 | 1,000 | 95 | 1 | 250 (c) | 11,900 | 9-68 | 4-69 | **Examination in progress** |
| | 10,000 | 1,000 | 95 | 1 | 263 (c) | 12,500 | 9-68 | 11-69 | |
| | 10,000+ | 1,000 | 95 | 1 | 263 (c) | 12,500 | 9-68 | 11-69+ | |
| | 50,000 | 1,000 | 95 | 1 | 301 (c) | 14,300 | 9-68 | 5-74 | |

(a) Excursion to >1100°C for 3-6 hr.
(b) Activity as of 2-67.
(c) Activity as of 6-68.
(d) Capsule contains $^{59}Co$ but is being heated along with $^{60}Co$ capsules.

→ New information reported.

Additional tests, at higher than normal operating temperatures, are performed with unirradiated cobalt encapsulated in the most promising materials to provide data for safety analyses of $^{60}Co$ heat sources. These tests are designed to evaluate the response of cobalt and capsule materials to environments common to several potential accidents and are restricted to those conditions that are not critically affected by capsule geometry.

## Selection of Superalloys

"Haynes"* 25, "Inconel"** 600, "Hastelloy"* X, and "Hastelloy"* C have been selected as the best capsule alloys for most applications of $^{60}Co$ metal at temperatures up to 1000°C.[2] Selection of the best alloy for any particular application will be based on the final data from the continuing tests of $^{60}Co$ capsules, Table II. These four alloys were chosen on the basis of screening tests of 44 commercially available superalloys. Table III lists the alloys tested along with their compositions and melting points and summarizes the results of the screening tests.

Detailed descriptions of the screening test results are presented in previous reports of this series. Compatibility with cobalt was evaluated by metallographic examination of multilayer diffusion couples heated one week at 800, 1000, and 1200°C.[5,6] Resistance to oxidation was measured by exposing coupon samples to still air for 500 hr at temperatures between 850 and 1150°C and confirmed by tests for 1000 to 10,000 hr at 1000°C and 3000 hr at 1150°C.[7-9] The diffusivity of $^{60}Co$ in alloys with good compatibility and oxidation resistance was measured by conventional radiotracer techniques.[8-11] The mechanical properties of the alloys, particularly the creep and stress rupture strengths, were evaluated by extrapolating available literature data.[1,2,5] Several alloys, such as "Hastelloy" C-276 and WF-11, were not tested because their compositions are almost identical to those that were tested. Two other alloys, "Inconel" 702 and Alloy 713C, that were not tested may also be suitable encapsulating materials because literature data indicate that their compositions and resistance to oxidation are similar to "Inconel" 600.[12-15]

* Trademark of Union Carbide Corp.
** Trademark of International Nickel Co.

TABLE III

PROPERTIES OF SUPERALLOYS AS POTENTIAL ENCAPSULATING MATERIALS FOR COBALT METAL

| Alloy | Nominal Composition, wt % | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Ni | Co | Cr | Fe | Mo | W | Ti | Al | Mn | Si | C | Other |
| **Ni-Based Alloys** | | | | | | | | | | | | |
| Alloy 713C | 74.2 | - | 12.5 | - | 4.2 | - | 0.8 | 6.1 | - | - | 0.1 | 2.0 Cb |
| Astroloy | Same composition as Udimet 700 | | | | | | | | | | | |
| Hastelloy C | 54.0 | 2.5 | 15.5 | 5.0 | 16.0 | 4.0 | - | - | 1.0 | 1.0 | 0.08 | - |
| Hastelloy C-276 | Low Si, low C form of Hastelloy C | | | | | | | | | | | |
| Hastelloy F | 43.0 | 1.0 | 22.0 | 22.5 | 6.5 | - | - | - | 2.0 | 1.0 | 0.05 | 2.0 Cb+Ta |
| Hastelloy G | 45.0 | 1.0 | 22.0 | 19.5 | 6.5 | 0.6 | - | - | 1.3 | 0.35 | 0.03 | 2.0 Cb+Ta<br>2.0 Cu |
| Hastelloy N | 69.0 | - | 7.0 | 5.0 | 16.5 | 0.5 | 0.5 | - | 1.0 | 1.0 | 0.06 | - |
| Hastelloy X | 47.3 | 1.5 | 22.0 | 18.5 | 9.0 | 0.6 | - | - | 0.5 | 0.5 | 0.1 | - |
| Hastelloy X-280 | 49.0 | 0.3 | 22.5 | 18.0 | 8.5 | 0.5 | - | - | 0.5 | 0.5 | 0.1 | - |
| Haynes R-41 | Same composition as Rene 41 | | | | | | | | | | | |
| Incoloy 825 | 42.0 | - | 21.5 | 30.0 | 3.0 | - | 0.9 | 0.2 | 0.6 | 0.5 | 0.03 | 1.8 Cu |
| Inconel 600 | 76.6 | - | 15.8 | 7.2 | - | - | - | - | 0.2 | 0.2 | 0.04 | - |
| Inconel 625 | 61.1 | - | 22.0 | 3.0 | 9.0 | - | 0.2 | 0.2 | 0.15 | 0.3 | 0.05 | 4.0 Cb |
| Inconel 702 | 79.8 | - | 15.6 | 0.4 | - | - | 0.7 | 3.4 | 0.05 | 0.2 | 0.04 | - |
| Nickel 200 | 99.5 | - | - | - | - | - | - | - | - | - | - | - |
| Nickel 270 | 99.98 | - | - | - | - | - | - | - | - | - | - | - |
| RA 333 | 45.0 | 3.0 | 25.0 | 18.0 | 3.0 | 3.0 | - | - | 1.5 | - | 0.08 | - |
| Rene 41 | 55.3 | 11.0 | 19.0 | - | 10.0 | - | 3.1 | 1.5 | - | - | 0.09 | - |
| TD Nickel | 98.0 | - | - | - | - | - | - | - | - | - | - | 2.0 $ThO_2$ |
| TD Nickel Chromium | 78.0 | - | 20.0 | - | - | - | - | - | - | - | - | 2.0 $ThO_2$ |
| Tophet A | 80.0 | - | 20.0 | - | - | - | - | - | - | - | - | - |
| Tophet C | 61.0 | - | 15.0 | 24.0 | - | - | - | - | - | - | - | - |
| Tophet 30 | 70.0 | - | 30.0 | - | - | - | - | - | - | - | - | - |
| Udimet 500 | 53.6 | 18.5 | 18.0 | - | 4.0 | - | 2.9 | 2.9 | - | - | 0.08 | - |
| Udimet 700 | 53.4 | 18.5 | 15.0 | - | 5.2 | - | 3.5 | 4.3 | - | - | 0.08 | - |
| Unitemp HX | Same composition as Hastelloy X | | | | | | | | | | | |
| Waspalloy | 58.3 | 13.5 | 19.5 | - | 4.3 | - | 3.0 | 1.3 | - | - | 0.08 | - |
| 50 Ni - 50 Cr | 50.0 | - | 50.0 | - | - | - | - | - | - | - | - | - |
| **Co-Based Alloys** | | | | | | | | | | | | |
| Haynes 25 | 10.0 | 52.9 | 20.0 | - | - | 15.0 | - | - | 1.5 | 0.5 | 0.1 | - |
| Haynes 150 | 1.0 | 49.0 | 29.0 | 19.0 | 0.2 | 0.5 | - | - | 0.6 | - | - | - |
| Haynes 152 | - | 63.0 | 21.0 | 2.0 | - | 11.0 | - | - | 0.25 | 0.25 | 0.45 | 2.0 Cb |
| Haynes 188 | Same composition as Haynes 8188 | | | | | | | | | | | |
| Haynes 8188 | 20.8 | 40.0 | 23.2 | 1.5 | - | 13.5 | - | - | 0.5 | 0.3 | 0.1 | 0.03 La |
| L-605 | Same composition as Haynes 25 | | | | | | | | | | | |
| Stellite 21 | 2.5 | 60.0 | 27.5 | 2.0 | 5.5 | - | - | - | 1.0 | 1.0 | 0.25 | - |
| Stellite 31 | 10.5 | 52.5 | 25.5 | 2.0 | - | 7.5 | - | - | 0.8 | 0.7 | 0.5 | - |
| WF-11 | Same composition as Haynes 25 | | | | | | | | | | | |
| WI-52 | Same composition as Haynes 152 | | | | | | | | | | | |
| **Fe-Based Alloys** | | | | | | | | | | | | |
| Duraloy HH | 12.5 | - | 25.0 | 59.5 | - | - | - | - | 1.5 | 1.5 | 0.3 | - |
| Incoloy | 32.0 | - | 20.5 | 46.0 | - | - | 0.3 | 0.3 | 0.75 | 0.35 | 0.04 | 0.3 Cu |
| Kanthal A | - | 1.0 | 22.0 | 72.0 | - | - | - | 5.0 | - | - | 0.1 | - |
| N-155 | 20.0 | 20.0 | 21.0 | 31.3 | 3.0 | 2.5 | - | - | 1.5 | 0.5 | 0.15 | - |
| GE 1541 | - | - | 15.0 | 80.0 | - | - | - | 4.0 | - | - | - | 1.0 La |
| GE 2541 | - | - | 25.0 | 70.0 | - | - | - | 4.0 | - | - | - | 1.0 La |

a Solidus temperature.

b Acceptable (A) or unacceptable (U) at expected heat source conditions, up to 5 yr at 1000°C.

c Literature data indicate oxidation resistance equivalent to Inconel 600; other properties also expected to be similar because compositions are similar.

d Low-cobalt form of Hastelloy X; properties expected to be similar to Hastelloy X.

e Strength decreases markedly with temperature in the range 850 to 1000°C.

f Eliminated from consideration because of intergranular embrittlement after 5000 hr at 1000°C.

g Available only in cast forms.

| Melting Point (°C)[a] | Evaluated at SRL[b] Compatibility | Oxidation | Diffusion | Strength | Source | Alloy |
|---|---|---|---|---|---|---|
| | | | | | | **Ni-Based Alloys** |
| 1260 | - | (A)[c] | - | - | Int'l. Nickel | Alloy 713C |
| | | | | | Westinghouse | Astroloy |
| 1270 | A | A | A | A | Union Carbide | Hastelloy C |
| | | | | | " " | Hastelloy C-276 |
| - | - | U | - | - | " " | Hastelloy F |
| - | - | U | - | - | " " | Hastelloy G |
| 1325 | - | - | - | U | " " | Hastelloy N |
| 1260 | A | A | A | A | " " | Hastelloy X |
| - | d | d | d | d | " " | Hastelloy X-280 |
| | | | | | " " | Haynes R-41 |
| 1360 | U | U | - | - | Int'l. Nickel | Incoloy 825 |
| 1370 | A | A | A | A | " " | Inconel 600 |
| - | - | U | - | - | " " | Inconel 625 |
| - | - | (A)[c] | - | - | " " | Inconel 702 |
| 1435 | - | U | - | - | " " | Nickel 200 |
| 1455 | - | U | - | - | " " | Nickel 270 |
| 1300 | U | U | - | - | Rolled Alloys | RA 333 |
| 1315 | - | - | - | U[e] | Allvac Metals | Rene 41 |
| 1450 | A | U | U | A | Fansteel | TD Nickel |
| 1430 | A | A | U | A | " | TD Nickel Chromium |
| 1400 | U | U | - | - | W. B. Driver | Tophet A |
| 1350 | - | A[f] | - | - | " " | Tophet C |
| 1375 | - | U | - | - | " " | Tophet 30 |
| 1300 | - | - | - | U[e] | Special Metals | Udimet 500 |
| 1205 | - | - | - | U[e] | " " | Udimet 700 |
| | | | | | Universal Cyclops | Unitemp HX |
| 1330 | - | - | - | U[e] | Carpenter Steel | Waspalloy |
| - | U | U | - | - | Int'l. Nickel | 50 Ni - 50 Cr |
| | | | | | | **Co-Based Alloys** |
| 1330 | A | A | A | A | Union Carbide | Haynes 25 |
| - | U | U | - | - | " " | Haynes 150 |
| 1315 | - | - | - | A[g] | " " | Haynes 152 |
| | | | | | " " | Haynes 188 |
| - | A | U | - | - | " " | Haynes 8188 |
| | | | | | Universal Cyclops | L-605 |
| 1350 | - | - | - | A[g] | Union Carbide | Stellite 21 |
| 1340 | - | - | - | A[g] | " " | Stellite 31 |
| | | | | | Crucible Steel | WF-11 |
| | | | | | Waimet Alloys | WI-52 |
| | | | | | | **Fe-Based Alloys** |
| - | - | - | - | U | Duraloy | Duraloy HH |
| 1385 | - | U | - | U | Int'l. Nickel | Incoloy 800 |
| 1505 | - | - | - | U | Kanthal | Kanthal A |
| 1275 | - | U | - | U | Carpenter Steel | N-155 |
| - | U | A | - | - | General Electric | GE 1541 |
| - | U | A | - | - | " " | GE 2541 |

## CAPSULE FABRICATION AND TESTING

### Heating Tests of Capsules Containing Unirradiated Cobalt Metal

The amount of oxidation and reaction with the cobalt in an "Inconel" 600 capsule heated 5000 hr at 900°C was in agreement with the reactions observed in a similar capsule that contained $^{60}Co$ and were commensurate with the reactions observed in other $^{59}Co$ capsules heated at 850 and 1000°C. This test is part of the continuing program to demonstrate structural integrity of cobalt heat sources for >10,000 hr at typical temperatures of 850 to 1000°C, Table I.

Two "Hastelloy" X capsules attained their goal exposures of 10,000 hr at 1000°C. Both capsules were intact and dimensional changes were <0.002 inch. Destructive examination of one of the capsules showed that the widths of the zones affected by cobalt-capsule reactions and by oxidation of the exterior capsule surface were in agreement with a parabolic extrapolation of data from previous, shorter term tests. Since capsule performance was satisfactory, heating of the other capsule was resumed toward a goal exposure of 50,000 hr.

### Heating Tests of Capsules Containing Irradiated Cobalt Metal

#### Long-Term Tests

Seventeen superalloy capsules, 15 containing radioactive and 2 containing nonradioactive cobalt metal, are being heated in air at 900 to 1000°C in the High Level Caves (HLC) to demonstrate capsule performance at typical heat source conditions, Table II. Three capsules, one each of "Inconel" 600, "Hastelloy" X, and "Haynes" 25, have attained a goal exposure of 5000 hr at 1000°C. Destructive examination of these capsules has been deferred until January 1970 because of commitment of the High Level Caves to other isotope programs and a scheduled shutdown to install new filters.

#### Test Facilities

FY-1969 capital funds were authorized for construction of a shielded cell in the Isotope Process Development Laboratory (IPDL) to be used for long-term heating tests of capsules containing $^{60}Co$. Final design of the cell is in progress; construction is to begin in September.

Safety Tests

Cobalt Release

Heating of an intentionally defected "Inconel" 600 capsule in flowing air at 1000°C is continuing to measure the release of $^{60}Co$.[2] The capsule has a 0.008-inch diameter hole drilled through the wall and contains a solid rod of unirradiated cobalt that was plated with $^{60}Co$ before encapsulation. Low levels of radioactivity (~10 counts/min above background) are deposited each day on a filter located in the effluent air stream. Total test time to date is 68 days (~1600 hr).

Compatibility of Molten Cobalt and Superalloys with Tungsten and Rhenium

Tests of the compatibility of molten cobalt and superalloys with tungsten and rhenium are continuing to evaluate the consequences of temperature excursions above the melting points of the capsules (~1500°C) that might occur under abnormal conditions, such as loss of coolant, re-entry from space, or burial in the earth. Preliminary tests are described in Reference 2. Interfaces between the molten cobalt and the capsule wall showed that no reaction was occurring in the pure tungsten capsules. In contrast, penetration along grain boundaries occurred in W-2% (Cu + Ni) capsules. Penetrations >0.100 inch occurred opposite the "vapor" space in some of the capsules in 4 hours at 1550°C. In other capsules, where some of the liquid cobalt was lost through faulty welds, up to 0.055 inch of the capsule wall opposite the vapor space was dissolved.

Five additional tungsten capsules were fabricated. These capsules will be used to evaluate the effects of a void space and superalloys inside the tungsten capsule and the effectiveness of a rhenium barrier between the cobalt and the tungsten on the dissolution of tungsten in the molten cobalt.

Refractory Metal Capsules

Development of Welding Techniques

Materials and equipment are being procured for encapsulation of cobalt metal for applications above 1000°C. Capsules of tungsten, tungsten-25% rhenium, and rhenium are being purchased for welding development and long-term compatibility tests at ~1200°C with $^{59}Co$. Ten tungsten capsules have been delivered by the vendor.

Capsules are being fabricated from arc-cast and extruded tungsten and tungsten-25% rhenium and from powder-metallurgy rhenium (arc-cast rhenium is not available). Weld porosity is minimized by use of arc-cast material.(16) Caps and spacers will be machined from sheet instead of bar (except for the tungsten-25% rhenium caps) to provide a more diffusion resistant grain structure.

A facility is being designed for remote TIG welding of refractory metal capsules containing $^{60}Co$. The welding will be done in a vacuum chamber capable of maintaining less than 10 ppm of $O_2$, $N_2$, and water vapor as impurities in a helium atmosphere.

## HEAT SOURCE DEMONSTRATION TESTS

### WANL 30 kw(t) DEMONSTRATION UNIT

The Westinghouse Astronuclear Laboratory (WANL) has been awarded a contract by the Division of Isotopes Development (DID) to design, build, and operate a $^{60}Co$-fueled, 30 kw(t) demonstration unit. In one concept the $^{60}Co$ capsules are embedded in the central region of a cylindrical core that is penetrated by holes for flow of air or inert gas coolant. The coolant enters the core at the bottom, is preheated as it flows upward through the outer region of the core, and finally reaches the desired operating temperature as it flows downward through the central region.

#### Selection of Core Materials

Nickel was initially selected as the reference material for the core of the demonstration unit because of its high thermal conductivity. However, unclad nickel may have inadequate resistance to oxidation at the expected operating conditions of the unit, 1 to 5 years at ~900°C. A review of published data indicates that the best superalloy for the core is "Inconel" 702 because its thermal conductivity and resistance to oxidation are among the highest of the superalloys. Alloy 713C and "Udimet" 700 have adequate oxidation resistances, but their conductivities vary widely over the temperature range of interest.

Of the superalloys, Alloy 713C, "Udimet" 700, Waimet-52, and "Inconel" 702 have the highest thermal conductivities at high temperature. Several alloys are compared to pure nickel in

Figure 7. The conductivities of Alloy 713C, "Udimet" 700, and Waimet-52 increase sharply above ~900°C, possibly because of the dissolution of the aluminum- and titanium-rich precipitates that are responsible for the high strengths of these alloys. "Inconel" 702 has a conductivity that increases nearly linearly with temperature up to 650°C, the limit of available data; extrapolation of the data indicates a conductivity as high or higher than the other superalloys above 750°C.

Of the four alloys with high thermal conductivities, only Waimet-52 has an inadequate resistance to oxidation under the expected conditions. Extrapolation of short-term (<100 hr) tests at 1050°C with "Haynes" 152, which has the same composition as Waimet-52, Table III, indicates that oxide scales 0.030 to 0.050 inch thick would be expected after one year.[14] In contrast, direct comparison of "Inconel" 702 and "Inconel" 600 in 10,000-hr tests at 950°C indicates slightly less spalling of the scale on "Inconel" 702 than on "Inconel" 600 (weight losses of 3.4 $mg/cm^2$ compared to 6.5 $mg/cm^2$) although intergranular penetrations under the scales were the same (0.006 inch).[12,13] Alloy 713C and "Udimet" 700 have oxidation resistances similar to "Hastelloy" X as indicated by 100-hr tests at up to 1050°C.[14,15,17,18] Tests at SRL for up to 10,000 hr at 1000°C have shown that "Hastelloy" X has an acceptable resistance to oxidation.[7-9]

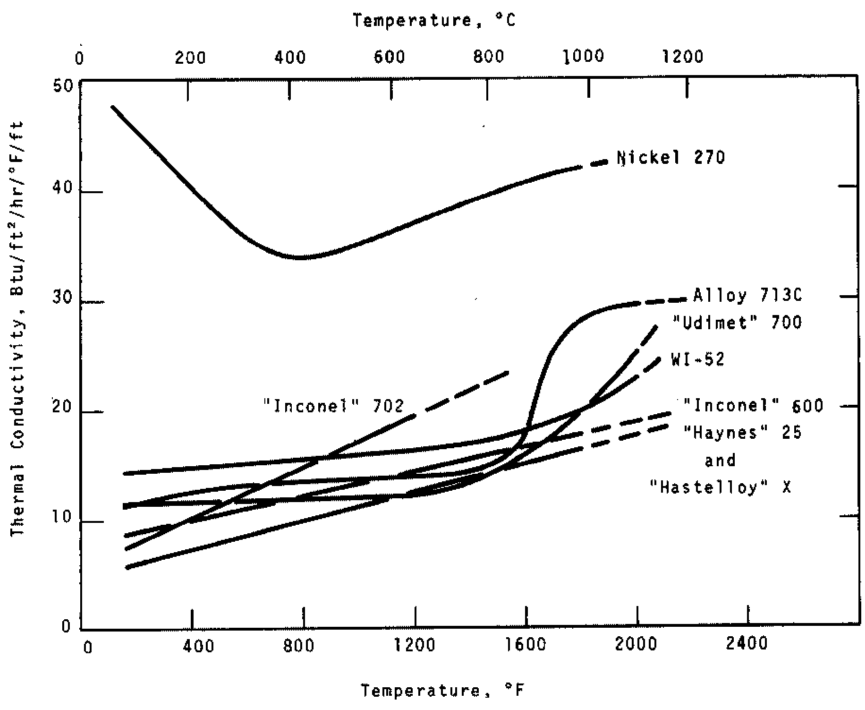



FIG. 7 THERMAL CONDUCTIVITIES OF SELECTED SUPERALLOYS

## HIGH-ACTIVITY $^{60}Co$ FOR HEAT SOURCE DEVELOPMENT

High-activity cobalt that is being stored at Savannah River for experimental programs and heat source development is listed in Table IV.

TABLE IV

$^{60}Co$ for Heat Sources[a]

| Cobalt Shape | Average Activity, Ci/g | No. of Pieces | Total Activity, $10^6$ Ci | Total Power, kw |
|---|---|---|---|---|
| 0.745-inch Wafers | | | | |
| Fuel form - cobalt metal (wrought) | 310 | 2090 | 1.60 | 25.0 |
| Fuel shape - wafers 0.745 ±0.001-inch diameter | 270 | 912 | 0.62 | 9.6 |
| 0.040 ±0.003-inch thick | 250 | 1368 | 0.87 | 13.6 |
| including 0.0005 to 0.001-inch Ni plate | 230 | 1368 | 0.79 | 12.3 |
| Cobalt density - 8.80 ±0.05 g/cm³ | 240 | 152 | 0.09 | 1.4[b] |
| | | 5890 | 3.97 | 61.9 |
| 0.800-inch Wafers | | | | |
| Fuel form - cobalt metal (sintered) | 300 | 255 | 0.22 | 3.4 |
| Fuel shape - wafers 0.800 ±0.001-inch diameter | 320 | 136 | 0.13 | 2.0[c] |
| 0.040 ±0.003-inch thick | 270 | 136 | 0.10 | 1.6[d] |
| including 0.0005 to 0.001-inch Ni plate | | 527 | 0.45 | 7.0 |
| Cobalt density - 8.60 ±0.10 g/cm³ | | | | |
| Nickel-Plated Slabs | | | | |
| Fuel form - cobalt metal (wrought) | 300 | 45 | 0.23 | 3.6 |
| Fuel shape - slabs 3.00 ±0.03-inch long | 280 | 48 | 0.22 | 3.4 |
| 0.640 ±0.002-inch wide | | 93 | 0.45 | 7.0 |
| 0.060 ±0.001-inch thick | | | | |
| including 0.0005 to 0.001-inch Ni plate | | | | |
| Cobalt density - 8.80 ±0.05 g/cm³ | | | | |
| Stainless Steel-Canned Slabs | | | | |
| Fuel form - cobalt metal (wrought) | 300 | 45 | 0.18 | 2.8 |
| Fuel shape - slabs 2.96 ±0.03-inch long | 250 | 48 | 0.17 | 2.7 |
| 0.735 - 0.740-inch wide | | 93 | 0.35 | 5.5 |
| 0.092 ±0.001-inch thick | | | | |
| sheath thickness 0.015-inch min | | | | |
| cobalt dimensions same as nickel-plated slabs above except 2.44-inch long | | | | |
| Cobalt density - 8.80 ±0.05 g/cm³ | | | | |
| Stainless Steel-Coextruded Slabs | | | | |
| Fuel form - cobalt metal (wrought) | 300 | 60 | 0.21 | 3.3 |
| Fuel shape - slabs 3.00 ±0.03-inch long | 280 | 64 | 0.21 | 3.3 |
| 0.740 ±0.002-inch wide | | 124 | 0.42 | 6.6 |
| 0.072 ±0.002-inch thick | | | | |
| SST thickness 0.015-inch min | | | | |
| Cobalt dimensions: 2.75-inch long | | | | |
| 0.71-inch wide | | | | |
| 0.042-inch thick | | | | |
| Cobalt density - 8.80 ±0.05 g/cm³ | | | | |

Grand Total 5.64 MCi 88.0 kw
Average Activity 280 Ci/g

a Activity and power as of June 30, 1969.
b Wafers have central hole of 0.070-inch diameter.
c 110 wafers have experimental compositions, to be used at SRL.
d 68 wafers have central hole of 0.070-inch diameter.

## SAVANNAH RIVER LABORATORY $^{60}Co$ PUBLICATIONS

### Quarterly Progress Reports

"Savannah River Laboratory Isotopic Power and Heat Sources Quarterly Progress Report," compiled by H. S. Hilborn

| | |
|---|---|
| DP-1088 | July - September 1966 |
| DP-1094 | October - December 1966 |
| DP-1105-I | January - March 1967, Part I - Cobalt |
| DP-1120-I | April - June 1967, Part I - Cobalt |
| DP-1129-I | July - September 1967, Part I - Cobalt |
| DP-1143-I | October - December 1967, Part I - Cobalt |
| DP-1155-I | January - March 1968, Part I - Cobalt |
| DP-1169-I | April - June 1968, Part I - Cobalt |
| DP-1177-I | July - September 1968, Part I - Cobalt |
| DP-1192-I | October - December 1968, Part I - Cobalt |
| DP-1196-I | January - March 1969, Part I - Cobalt |

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| | |
|---|---|
| DP-974 | "$^{60}Co$ Heat Sources for 10-60 kw(e) Generators" by A. H. Dexter, July 1965. |
| DP-1012 | "Radioactive Cobalt for Heat Sources" by J. W. Joseph, H. F. Allen, C. L. Angerman, and A. H. Dexter, October 1965. |
| DP-1051 (Rev. 2) | "Properties of $^{60}Co$ and Cobalt Metal Fuel Forms", June 1968. |
| DP-1096 | "Development of $^{60}Co$ Capsules for Heat Sources" by C. P. Ross, C. L. Angerman, and F. D. R. King, June 1967. |
| DP-1145 | "Experimental $^{60}Co$ Heat Source Capsules" by J. P. Faraci, May 1968 |

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A. H. Dexter, W. R. Cornman, and E. J. Hennelly. "The Advantages of $^{60}Co$ for Heat and Radiation Sources", Nucl. Appl. 2(2), 99-101 (1966).

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