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DP-1155-I

AEC RESEARCH AND DEVELOPMENT REPORT

SAVANNAH RIVER LABORATORY ISOTOPIC POWER AND HEAT SOURCES

QUARTERLY PROGRESS REPORT

JANUARY - MARCH 1968

PART I - COBALT-60

DU PONT

REG. U. S. PAT. OFF.

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H. S. Hilborn, Compiler

April 1968

E. I. DU PONT DE NEMOURS & COMPANY
SAVANNAH RIVER LABORATORY
AIKEN, S. C. 29801

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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 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 on the last page.

SUMMARY

Operation of capsules at 1000°C for five years will not result in excessive diffusion of ^{60}Co in "Hastelloy" X, "Hastelloy" C, or "Haynes" 25. (p 1)

An "Inconel" 600 capsule containing nonradioactive cobalt was in satisfactory condition after 10,000 hours at 850°C. (p 7)

An "Inconel" 600 capsule containing radioactive cobalt was in satisfactory condition after 5000 hours at ~900°C. (p 10)

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. The initial objective is to establish allowable operating limits for capsules of radioactive cobalt metal contained in oxidation resistant alloys. Development of specific heat source concepts is not at present included in the scope of the SRL program.

MATERIALS TECHNOLOGY AND DEVELOPMENT

Evaluation of Encapsulating 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. The kinetics of compatibility reactions,[1,2] diffusion of ^{60}Co,[2,4] and oxidation,[4,5] are being measured using short-term (<500 hr) laboratory tests. Data on mechanical properties, such as creep, have been obtained from the literature, but will be confirmed at a later date by tests with pressurized capsules. Selection of the most promising alloys is based on extrapolation of these results to the expected service life (1 to 5 years). Limiting operating conditions are defined by the time and temperature dependency of each of the reactions. The predicted behavior of the materials is being verified by 1000-, 5000-, and 10,000-hr heating tests of experimental capsules, Table I.[4] Tests of companion capsules of unirradiated and 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.

Diffusion of ^{60}Co

The diffusion of ^{60}Co through prospective encapsulation materials is being measured to determine whether it will be a limiting factor in the design of heat sources. Earlier calculations showed that diffusion would not be limiting for "Haynes"* 25, "Hastelloy"* X, or "Hastelloy"* C during one year of operation at temperatures up to 1000°C. Diffusion would be limiting for "Inconel"** 600 and TD Nickel*** operating at 1000°C in applications where the capsule wall is exposed to flowing coolant because ^{60}Co concentrations greater than 1 ppm are expected at the outer surface of the capsule.[4]

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* Trademark of Union Carbide Corporation

** Trademark of International Nickel Company

*** Product of Fansteel Metallurgical Corporation

TABLE I. SUMMARY OF COBALT CAPSULE HEATING TESTS

Capsule Materials	Heating Time, hr	Heating Temp, °C	Wall, mils	No. of Capsules	Activity Specific Ci/g	Activity Total Ci	Approx. Starting Date	Approx. Compl. Date	Remarks
A. Inactive Capsules									
"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	2	-	-	7-67	9-68	
	1,000	1000	50	4	-	-	8-66	10-66	3 capsules intact; 1 capsule oxidized[b]
	5,000	1000	50	1	-	-	4-67	11-67	Capsule intact
	1,000[a]	1000	95	2	-	-	7-67	9-67	No severe oxidation of Co
	→ 1,000[f]	1000	95	1	-	-	2-68	4-68	
	→ 5,000[f]	1000	95	1	-	-	2-68	9-68	
	→ 5,000	1000	95	1	-	-	8-67	2-68	Examination in progress
	10,000	1000	95	1	-	-	8-67	10-68	
	10,000	1000	95	1	-	-	10-67	12-68	
"Hastelloy" C	1,000	1000	50	4	-	-	8-66	10-66	3 capsules intact; 1 capsule oxidized[b]
	5,000	1000	95	1	-	-	10-67	5-68	
	10,000	1000	95	2	-	-	10-67	12-68	
TD Nickel	→ 1,000	850[c]	95	1	-	-	10-67	12-67	Capsule intact
	5,000	850	95	1	-	-	10-67	5-68	
	10,000	850	95	1	-	-	10-67	12-68	
	10,000	850	95	1	-	-	10-67	12-68	
	1,000	1000	50	1	-	-	12-66	2-67	Capsule intact
	→ 1,000[a]	1000	95	2	-	-	10-67	12-67	No severe oxidation of Co
TD Nickel-Chromium	→ 1,000[a]	1000	95	2	-	-	10-67	12-67	Co near pinhole oxidized
	→ 1,000	1000	95	1	-	-	10-67	12-67	Capsule intact
	5,000	1000	95	1	-	-	10-67	5-68	
	10,000	1000	95	1	-	-	10-67	12-68	
	10,000	1000	95	1	-	-	10-67	12-68	
"Haynes" 25	→ 1,000	1000	95	1	-	-	10-67	12-67	Capsule intact
	5,000	1000	95	1	-	-	10-67	5-68	
	10,000	1000	95	1	-	-	10-67	12-68	
	10,000	1000	95	1	-	-	10-67	12-68	
"Hastelloy" X	1,000	1000	50	1	-	-	4-67	6-67	Capsule intact
	5,000	1000	50	1	-	-	4-67	11-67	Capsule intact
	→ 5,000	1000	95	2	-	-	2-68	9-68	
	→10,000	1000	95	2	-	-	2-68	4-69	
B. ^{60}Co Capsules									
"Inconel" 600	130	850[d]	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[e]	15,000[e]	4-67	10-67	Capsule intact
	10,000	~900	50	1	150[e]	15,000[e]	4-67	6-68	
	10,000	~900	50	1	150[e]	9,000[e]	5-67	7-68	
	→10,000	850	95	1	265[g]	38,000[g]	2-68	4-69	
"Hastelloy" C	100	850	50	1	120	9,000	1-67	1-67	Capsule intact

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. Excursions to >1100°C for 3-6 hr.
e. Activity as of 2-67.
f. Internal atmosphere air instead of helium.
g. Activity as of 2-68.
→. New information reported since DP-1143-I.

Recent calculations show that operation for five years (one half-life of the ^{60}Co) at up to 1000°C will not be limited by diffusion in "Hastelloy" X, "Haynes" 25, or "Hastelloy" C because the maximum specified concentration of 1 ppm ^{60}Co in the cladding material does not occur beyond 0.080 inch from the surface of the cobalt -- typical cladding is 0.100-inch thick. Use of "Inconel" 600 for five years would be limited by diffusion at 1000°C, but not at 800°C, Figure 1.

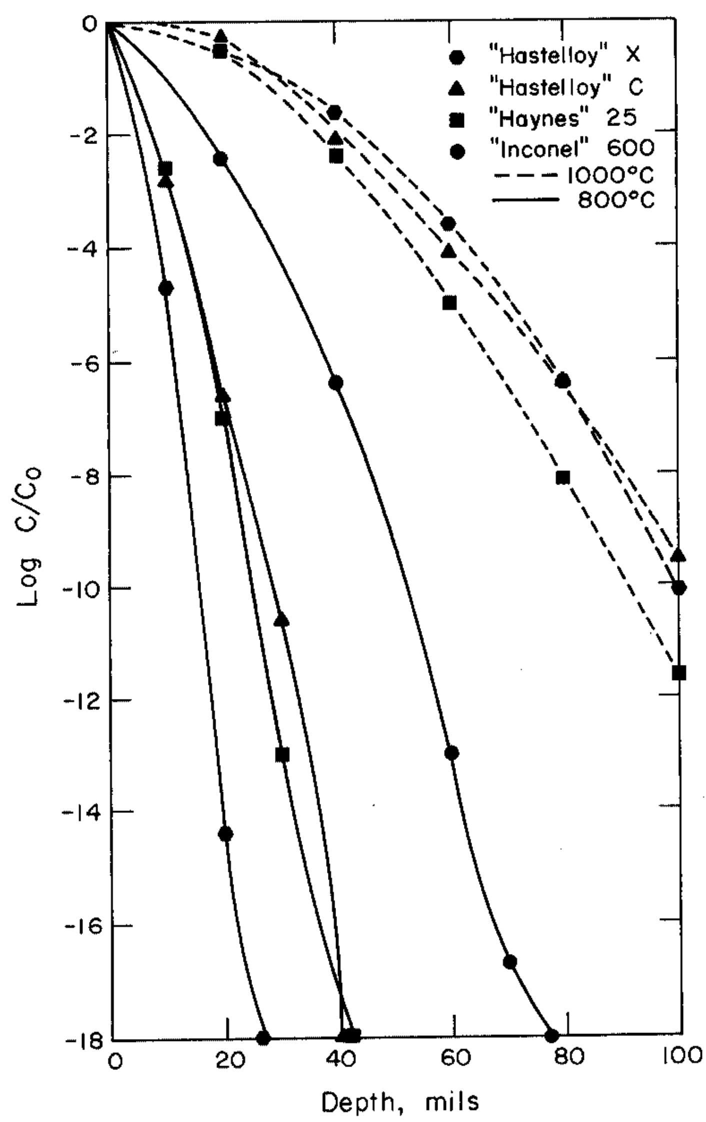


FIG. 1 ^{60}Co CONCENTRATION PROFILES EXPECTED AFTER FIVE YEARS OPERATION AT 800 AND 1000°C

These calculations were based on radiotracer measurements of the coefficients of volume and grain-boundary diffusion during 100-hr tests at 800°C and during 40-hr tests at 1000°C. These tests were analyzed with the instant-source model because this approximates the experimental conditions, and the predicted concentrations were computed with the constant-source model.* The data are compared in Table II with previous data for one year of operation.(4) The deeper penetrations in "Inconel" 600 reflect the larger coefficient of grain-boundary diffusion (a factor of 7 larger than that for "Hastelloy" C). Diffusion is assumed to occur in a wall of infinite rather than 0.100-inch thickness. This assumption leads to ^{60}Co concentrations that are slightly lower than the value for 0.100-inch capsule walls, but are satisfactory for ranking the candidate alloys.

TABLE II

CALCULATED DIFFUSION OF ^{60}Co IN CAPSULE MATERIALS

	Depth at Which ^{60}Co Concentration is ~1 ppm, mils			
	800°C		1000°C	
Material	1 yr	5 yr	1 yr	5 yr
"Hastelloy" X	8	12	35	77
"Haynes" 25	12	18	37	67
"Hastelloy" C	12	18	45	76
"Inconel" 600	20	38	~100	>100

Partial confirmation of the extrapolation of the short-term data to typical operating times was obtained with a wafer of "Hastelloy" X, electroplated with ^{60}Co, and heated in a "Hastelloy" X capsule for 5000 hr at 1000°C. As shown in Figure 2, the measured concentration was less than the concentration predicted by either the instant- or constant-source models for diffusion.

* The instant-source model assumes that a small, finite amount of the diffusing species is present initially and is conserved during diffusion; the constant-source model assumes that an infinite supply of the diffusing species is available at all times. These models are being compared with actual data to determine which model represents more accurately the conditions in ^{60}Co capsules.(6)

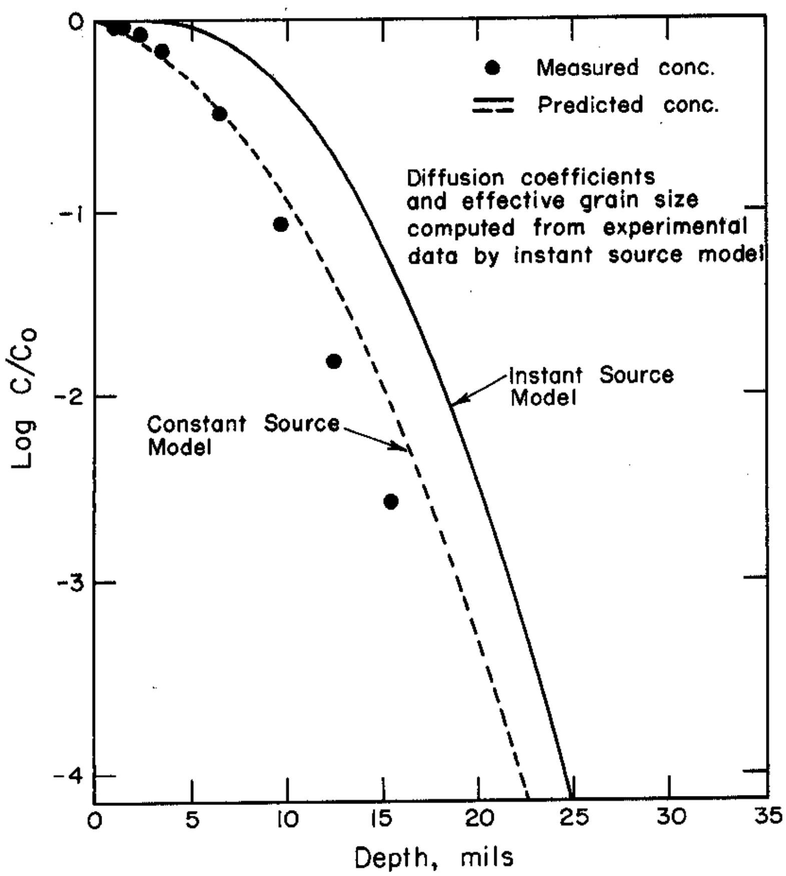


FIG. 2 COMPARISON OF MEASURED AND CALCULATED ^{60}Co CONCENTRATIONS IN "HASTELLOY" X HEATED 5000 HR AT 1000°C

Experiments are in progress to measure the effects of changes in the grain size of "Inconel" 600 that occur during the diffusion anneal, and the effects of the thoria stringers on diffusion in TD Nickel and TD Nickel Chromium* alloys. All of the samples have been annealed 100 hr at 1000°C to produce diffusion; analysis of the resulting distribution of the ^{60}Co is in progress. The samples of "Inconel" 600 were heat treated prior to the diffusion anneal to produce different initial grain sizes; one sample was heated 96 hr at 850°C and the other was heated 96 hr at 1150°C. One sample of each of the two TD-alloys was machined so that the direction of diffusion was transverse to the thoria stringers; all previous measurements of diffusion in these alloys were made parallel to the stringers. These tests showed unacceptably fast diffusion rates, possibly because of enhanced diffusion along the stringers of thoria particles.

* Product of Fansteel Metallurgical Corporation

Oxidation Resistance

The oxidation characteristics of two groups of encapsulating alloys are being defined at 1000°C for periods up to 10,000 hr.[4,5] Tests of the first group, terminated after 9400 hr, showed that TD Nickel Chromium, "Hastelloy" X, "Hastelloy" C, "Inconel" 600, and "Incoloy"** 800 had acceptable resistances to oxidation. Extrapolation of these tests to 50,000 hr indicated that depths of 0.020 to 0.040 inch would be affected by oxidation. Tests of the second group through 5000 hr indicated that GE*** 2541, "Haynes" 25, and 50Ni-50Cr alloys also have acceptable resistances.

Heating for 10,000 hr in still air at 1000°C has been completed on the second group of alloys. Metallographic measurements of scale thickness and the depths of intergranular penetration, internal oxidation, and alloy depletion are in progress.

Three experiments will be performed in the coming quarter to confirm the kinetics established by previous tests of the dependency of oxidation on time and temperature.

- Remeasure the oxidation of TD Nickel Chromium for up to 5000 hr to confirm the anomalously high slope obtained by plotting the depth affected by oxidation against time.

- Identify the oxide scales and compositions of the layers depleted in alloying elements by X-ray diffraction and electron microprobe analyses to confirm the earlier assumption that the mechanism of oxidation does not change with time.

- Oxidize specimens of the acceptable alloys at 1150°C (for approximately 3000 hr) to produce the same total depth affected by oxidation as would be expected after 50,000 hr at 1000°C to confirm that the time dependency holds for all temperatures up to 1150°C.

** Trademark of International Nickel Company

*** Product of General Electric Company

Capsule Fabrication and Testing

Welding of Capsules

Tungsten inert gas (TIG) techniques are used to weld test capsules of prospective materials. Sound, leak-tight welds have been made in "Inconel" 600, "Hastelloy" C, and "Hastelloy" X capsules with the wall thickness reduced at the ends of the capsules from 0.095 to 0.060 inch. Similar TIG welds of TD Nickel and TD Nickel Chromium are acceptable for capsule heating tests although the welds have irregular surfaces and contain random voids and agglomerates of thoria.

Initial attempts to TIG-weld "Haynes" 25 capsules resulted in cracking of the heat-affected zone.[4] Recent changes in the welding procedure have eliminated the cracking, although some liquation (melting) at the grain boundaries of the heat-affected zone is still present, Figure 3. According to the manufacturer's literature, sound welds can be made when a minimum of heat input is used, followed by rapid cooling of the weld deposit. Accordingly, the welding time was decreased, the inert gas flow was increased, the overlap withdrawal time was minimized, and the arc voltage was increased. Welding current was increased to maintain the desired penetration.

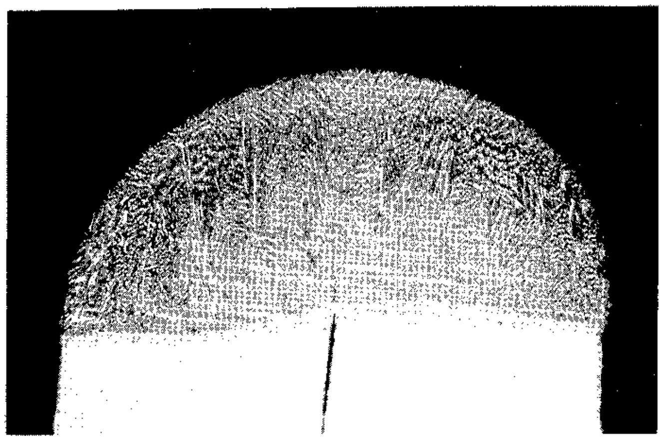
Heating Tests of Capsules Containing Unirradiated Cobalt

Tests at 850°C

Satisfactory performance of encapsulated cobalt for 10,000 hr at 850°C was demonstrated with an "Inconel" 600 capsule that had 0.050-inch thick walls. No changes in dimensions or loss of integrity were detected. Similar results had been obtained in previous 1000- and 5000-hr tests.[2,5]

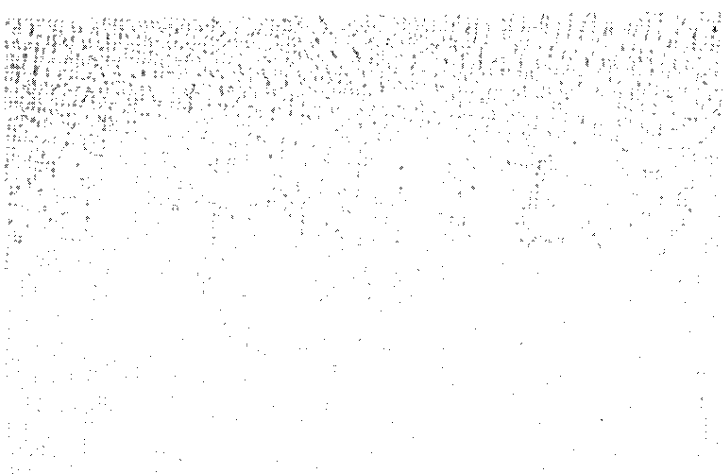
In this 10,000-hr test, the width of the reaction zone at the cobalt-capsule interface averaged 0.0075 inch. This result is in general agreement with previous data that extrapolates to an expected width of 0.016 inch after 50,000 hr, Figure 4. The cause of the anomalously wide reaction zone in the capsule heated 1000 hr is not known. Data for tests through 5000 hr at 1000°C are also shown in Figure 4 for comparison.

The observed total depth affected by oxidation was 0.003 inch, although measurement of the residual wall thickness indicated that about 0.001 inch of oxide scale spalled off during the test. The depth of 0.003 to 0.004 inch was expected from a parabolic extrapolation of data ($\sqrt{\text{time}}$) from previous 1000- and 5000-hr capsule tests.[4]



25X

a. Capsule weld



← Weld

Heat affected zone. Wide grain boundaries are indicative of liquation

250X

b. Enlargement of heat affected zone showing liquation.

FIG. 3 IMPROVED "HAYNES" 25 WELD

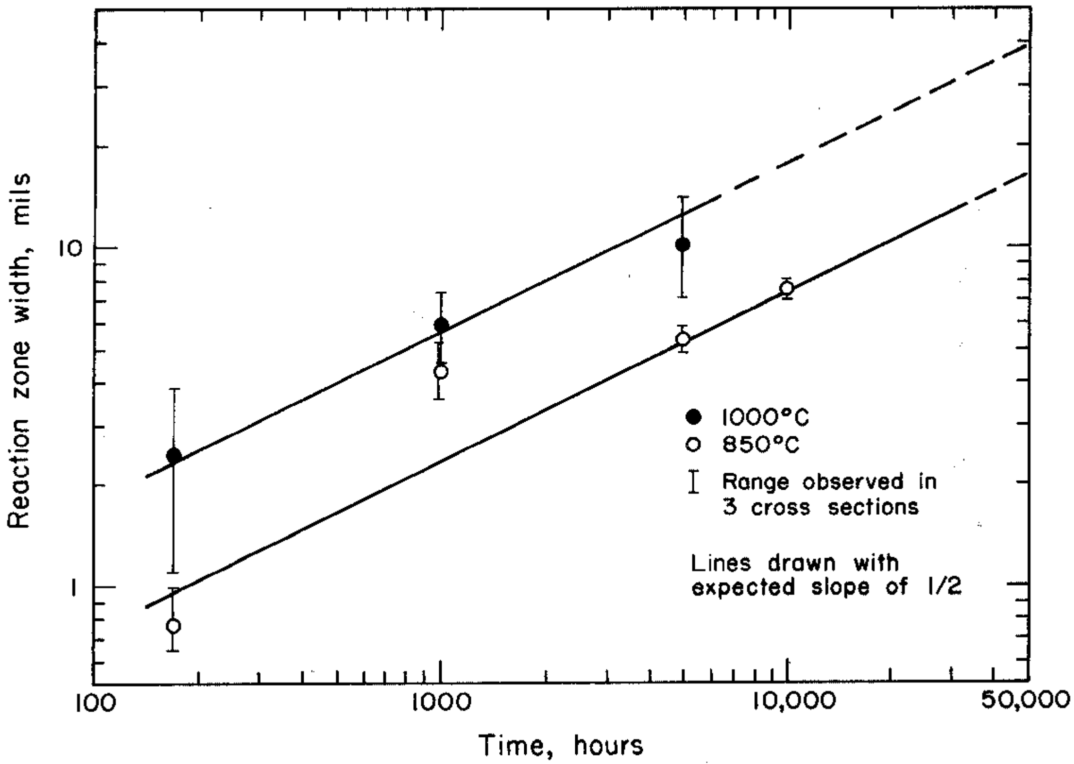


FIG. 4 GROWTH OF REACTION ZONE BETWEEN "INCONEL" 600 AND UNIRRADIATED COBALT

A TD Nickel capsule remained intact during heating for 1000 hr in flowing argon at 850°C. The capsule exhibited acceptable compatibility with the cobalt. The oxide scale on the exterior surface was about 0.002 inch thick, or 1/5 of that formed on capsules that were heated in still air under the same time and temperature conditions.

Tests at 1000°C

Satisfactory performance through 1000 hr at 1000°C was demonstrated with one capsule each of "Haynes" 25 and TD Nickel Chromiun; these tests were the first for these two alloys. Previous tests at 1000°C through 5000 hr had demonstrated the satisfactory performance of "Inconel" 600 and "Hastelloy" X capsules.[4]

No detrimental effects of heating for 1000 hr at 1000°C were observed on the capsules of "Haynes" 25 and TD Nickel Chromium. Each had 0.095 inch-thick walls. The reaction zones at the cobalt-capsule interfaces were about half as wide as expected from previous 168-hr diffusion couple tests.[1,2] These lower values probably reflect the impedence to diffusion caused by the incomplete contact between cobalt and capsule that was apparent during metallographic examination. The total thicknesses affected by oxidation were 0.003 inch and <0.001 inch for the "Haynes" 25 and TD Nickel Chromium capsules, respectively. These values are in accord with data from coupon tests of oxidation resistance that were performed at the same conditions.[5]

No gross oxidation of the cobalt inside intentionally defected capsules of "Inconel" 600, TD Nickel, or TD Nickel Chromium occurred during heating for 1000 hr at 1000°C. One capsule of each material had an 0.008-inch-diameter hole drilled through the capsule wall; the cap of the other capsule was not welded. Negligible oxidation of the cobalt occurred in the pinholed "Inconel" 600 and TD Nickel capsules, apparently because the holes became plugged with the oxide scales from the capsules themselves. In contrast, an 0.008-inch-thick oxide scale formed on the cobalt in the TD Nickel Chromium capsule in the vicinity of the pinhole. Only negligible oxidation of the TD Nickel Chromium occurred and the resulting scale could not plug the hole. There was no evidence of spalling of the cobalt oxide scale. Neither the cobalt nor the interior capsule walls of the unwelded capsules oxidized, indicating that the press fit of the cap formed a reasonably tight seal. Tests are being developed to detect traces of ^{60}Co that may be released from defective capsules.

Heating tests are continuing on 22 capsules that represent the six most promising capsule materials, Table I. Heating of six of these capsules was started in this past quarter.

Heating Tests of Capsules Containing Irradiated Cobalt

Satisfactory performance was demonstrated with an "Inconel" 600 capsule that contained 15,000 Ci and was heated for 5000 hr at ~900°C. Heating was started on another "Inconel" 600 capsule containing 38,000 Ci, the highest activity encapsulated to date. Previous tests with "Inconel" 600 and "Hastelloy" C capsules had shown satisfactory performance for up to 1000 hr.[3,5]

Maxima of 0.004 inch of surface oxidation and 0.013 inch of cobalt-cladding reaction were found in the "Inconel" 600 capsule containing 15,000 Ci of ^{60}Co (150 Ci/g) after 5000 hr of heating in air at a calculated wall temperature of 880-900°C. These values are consistent with previous data for a 5000-Ci capsule (100 Ci/g) heated 1000 hr at the same temperature.[8] The depth of the oxidation-affected zone and that of the compatibility zone are each proportional to the square root of the heating time, in accord with the expected relationship for processes that are controlled by diffusion. Previous data showed that the capsule heated for 5000 hr was dimensionally stable and structurally sound.[4]

The furnace temperature in these tests was controlled at 850°C. Analysis of the effects of self-heating from the ^{60}Co now indicates that the capsules had surface temperatures of 880 to 900°C rather than the nominal temperature of 850°C. Consistent with this analysis, the zones of oxidation and compatibility are slightly thicker than those formed in companion capsules of unirradiated cobalt heated at the same furnace temperature.

A 10,000-hr test was started on an "Inconel" 600 capsule with a surface temperature of 850°C and containing 38,000 Ci of ^{60}Co (265 Ci/g). The ^{60}Co is in the form of 57 stacked wafers each 0.745 inch in diameter and 0.040-inch thick. The capsule has a wall thickness of 0.095 inch, is 2.9 inches long and has a 0.94-inch OD (Figure 4 of Reference 4). The grounded tip of an "Inconel"-sheathed chromel-alumel thermocouple is clamped on the surface of the capsule, midway between the ends, to measure the maximum surface temperature. With the capsule surface temperature of 850°C, the measured temperature of the furnace air is about 775°C. Laboratory tests with cartridge heaters to simulate the ^{60}Co capsules indicated that this furnace, recently installed in the High Level Caves (HLC), can be used to heat capsules containing a total of 50,000 to 60,000 Ci at 850°C with margin for temperature control by the furnace heaters.

Installation in the HLC has been completed on a larger furnace to be used to heat capsules at 1000°C. Tests with cartridge heaters indicate that a total of 150,000 to 170,000 Ci (or about 10 short capsules) can be heated at 1000°C in this furnace with margin for control by the six silicon-carbide globar heaters in the furnace.

Heating is continuing at ~900°C on two "Inconel" 600 capsules containing 9000 and 15,000 Ci, respectively. Exposures of 10,000 hr will be reached in July, 1968.

Measurement of Surface Temperature of Cobalt Capsules

There is no accurate correlation between the air temperature in a test furnace and the surface temperature of ^{60}Co capsules that contribute to heating the air. Heat transfer by radiation, conduction, and convection contribute to this temperature difference. Since the error in calculated ΔT may be more significant for the higher activity cobalt now being tested, and since the actual capsule temperature must be known as accurately as possible for comparison with tests of nonradioactive capsules, means to measure the surface temperature and center temperature of radioactive capsules are being investigated.

Tests of electrically heated capsules, simulating those containing ^{60}Co, will be run to help determine practical methods for embedding thermocouples in the capsule cladding. Such tests will also show the difference between capsule surface temperature and air temperature within the furnace.

Two types of thermocouples have been procured for the tests. The first type, chromel-alumel wires with grounded junction inside an "Inconel" sheath of 0.011- or 0.020-inch diameter, will be staked into longitudinal grooves machined in the capsule surface. The second type, bare chromel and alumel wires separately embedded directly into holes drilled radially in the capsule surface, should give the most accurate temperature measurement but will be the most fragile. In addition, a standard 1/16-inch-diameter sheathed thermocouple will be clamped on the surface in an arrangement similar to that of the radioactive capsule under test in the HLC.

To most accurately simulate the size of ^{60}Co capsules under consideration, electric cartridge heaters of 3/4-inch OD have been procured for the heating tests. An "Inconel" 600 cylinder has been machined to fit over the heater and have the same outside diameter (0.94 inch) and surface finish (emissivity) as for live capsules (capsules containing radioactive cobalt).

During the tests, power input to the electric cartridge heater will be adjusted as calculated for self-heating in a range of representative ^{60}Co capsules. Power input to the furnace heaters will be as required to maintain desired equilibrium temperature on the surface of the capsule. Readings from all the "surface" thermocouples can be compared to each other and to the standard furnace thermocouple. Other methods of temperature measurement, such as optical pyrometry, will also be investigated.

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