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DP-1169-I

AEC RESEARCH AND DEVELOPMENT REPORT

SAVANNAH RIVER LABORATORY ISOTOPIC POWER AND HEAT SOURCES

QUARTERLY PROGRESS REPORT

APRIL - JUNE 1968

PART I - COBALT-60

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Savannah River Laboratory

Aiken, South Carolina

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H. S. Hilborn, Compiler

July 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

Diffusion of ^{60}Co in TD Nickel Chromium was significantly slower when perpendicular rather than parallel to the extrusion direction. (p 1)

Oxidation of proposed capsule alloys at 1000°C was proportional to $\sqrt{time}$ in 10,000-hour tests. Several nickel- and cobalt-based alloys are expected to have adequate oxidation resistance up to 5 years in air. (p 6)

"Haynes" 25, "Hastelloy" X, "Hastelloy" C, and "Inconel" 600 were selected as capsule materials for the first long-term tests of ^{60}Co capsules at 1000°C. Some of these capsules will have thermocouples with 0.040-inch diameter sheaths spot-welded to the surface for accurate capsule temperature measurement. (p 9)

An "Inconel" 600 capsule containing radioactive cobalt was in satisfactory condition after 10,000 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 cobalt-capsule compatibility reactions,[1,2] diffusion of ^{60}Co,[2-5] and oxidation,[4,6] are being measured using short-term (<500-hr) laboratory tests. 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. Published data on mechanical properties, such as creep, have been evaluated, but will be confirmed at a later date by tests with pressurized capsules. The predicted behavior of the materials is being verified by 1000-, 5000-, and 10,000-hr heating tests of experimental capsules, Table I.[5] 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.

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 a limiting factor for "Haynes"* 25, Hastelloy"* C, or "Hastelloy"* X during operation for 5 years (one half-life of the ^{60}Co) because the maximum specified concentration of 1 ppm ^{60}Co in the capsule material does not occur beyond 0.080 inch from the surface of the cobalt -- a typical capsule wall is 0.100-inch thick. Use of "Inconel"** 600 for 5 years would be limited by diffusion at 1000°C, but not at

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

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	No oxidation of Co or capsule
	5,000[f]	1000	95	1	-	-	2-68	9-68	
	→ 5,000	1000	95	1	-	-	8-67	2-68	Capsule intact
	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	Capsule intact
	10,000	1000	95	2	-	-	10-67	12-68	
	→10,000+	1000	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	
	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	Capsule intact
	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	Capsule intact
	→ 5,000	1000	95	1	-	-	5-68	12-68	
	10,000	1000	95	1	-	-	10-67	12-68	
	10,000	1000	95	1	-	-	10-67	12-68	
	→10,000+	1000	95	1	-	-	5-68	7-69+	
"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	
	→10,000+	1000	95	2	-	-	5-68	7-69+	
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	Examination in progress
	→ 10,000	~900	50	1	150[e]	9,000[e]	5-67	7-68[h]	
	→ 10,000	~850	95	1	265[g]	38,000[g]	2-68	6-69[h]	
"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.
h. Removed from furnace in April during equipment modifications. Test to resume in July.
→ New information reported since DP-1155-I.

800°C,[5] in situations where the capsule wall is exposed to a flowing coolant.

Although the basic techniques for measuring the ^{60}Co concentration profile after diffusion and the contributions by both volume and grain boundary diffusion are based on the work of other investigators,[7,8] recent experiments verified the actual procedures being used and supported the conclusion that grain boundary diffusion is a contributing factor. Another series of experiments showed that diffusion in TD Nickel Chromium*** was faster parallel to the extrusion direction than transverse to this direction; in TD Nickel*** diffusion was equivalent in both directions.

The ^{60}Co concentration profile is being measured by the residual-activity technique; the radioactivity remaining in the sample, which is proportional to the concentration, is counted after successive layers have been removed. Either this method, the layer-counting, or the surface-decrease methods are most frequently used. In the layer-counting method the activity of the material removed by mechanical or chemical methods is counted. In the surface-decrease method the activity is counted before and after the diffusion anneal without any sectioning. Both the residual-activity and layer-counting methods are capable of measuring the simultaneous contributions of grain boundary and volume diffusion, whereas the surface-decrease method assumes that only one mechanism of diffusion is operative. The residual-activity method avoids the tedious effort to collect and concentrate the material removed in sectioning. Several investigators have shown that there is little difference in the diffusion coefficients measured by the three techniques.[9-11]

Most measurements of the diffusion of cobalt reported in the literature were based on counting one of the characteristic gamma radiations by means of a pulse-height analyzer, but, as pointed out by Gruzin[7] and confirmed by recent experiments at SRL, equivalent results are obtained by counting the beta activity. One sample each of "Inconel" 600 and pure cobalt was plated with ^{60}Co and heated for 100 hr at 1000°C. As each layer was removed the residual activity was counted by two methods; first with a Radiation Instrument Development Laboratory 400-Channel Analyzer to obtain the activity produced by each of the gamma radiations, and second with a beta-gamma counter. The ^{60}Co activity registered by this latter instrument is composed of 90% beta and 10% gamma. The diffusion coefficients calculated from the activity profiles obtained by the two counting methods were in excellent agreement, as shown in Table II.

*** Product of Fansteel Metallurgical Co.

TABLE II

Comparison of ^{60}Co Diffusion Coefficients Obtained by Two Counting Methods

Material	400-Channel Analyzer Diffusion Coefficients, cm^2/sec		Beta-Gamma Counter Diffusion Coefficients, cm^2/sec	
	Volume	Grain Boundary	Volume	Grain Boundary
"Inconel" 600	1.3×10^{-11}	2.3×10^{-6}	1.5×10^{-11}	1.8×10^{-6}
Cobalt	1.4×10^{-11}	1.6×10^{-6}	1.9×10^{-11}	1.4×10^{-6}

The specific treatment of the activity data depends on the type of radiation being counted. The relationship between the measured activity and the diffusion equations is as follows:[7]

$$\mu I_n + \frac{\partial I_n}{\partial x_n} = f(C,t,T,D_V \text{ and } D_B) \qquad (1)$$

where μ is the linear absorption coefficient for the radiation used, I_n is the measured activity for any layer n at a distance x_n from the original surface, C is the initial concentration of radiotracer, t is the time of the diffusion anneal, T is the temperature of the anneal, and D_V and D_B are the volume and grain boundary diffusion coefficients, respectively. The linear absorption coefficients for the beta and gamma radiations are $1 \times 10^5/cm$ and $5 \times 10^{-1}/cm$, respectively. When using gamma radiation, the first term on the left side of equation (1) is small compared to the second term and may be neglected. The reverse is true for beta radiation. For example, typical data from the "Inconel" 600 sample show that for gamma radiation $\partial I_n/\partial x_n$ is about 5000 times larger than μI_n and for beta radiation μI_n is about 250 times larger than $\partial I_n/\partial x_n$.

As predicted by the diffusion models, comparison of two "Inconel" 600 samples - one with a grain size of 25 μ and the other with a grain size of 320 μ - showed that the depth of penetration by the ^{60}Co decreases as the grain size increases. The larger grain size was produced by heating for 96 hr at 1150°C prior to the diffusion anneal, which was at 1000°C for 100 hr. As shown in Figure 1, the initial portions of the concentration (activity) profiles, which result from volume diffusion, were identical for the two samples. In contrast, the remaining portions, which result from grain boundary diffusion, show less

penetration for the sample with the larger grain size. This reduction in diffusion with increased grain size may lead to smaller penetrations in an actual capsule wall since the grains in the capsule material grow at the service conditions.

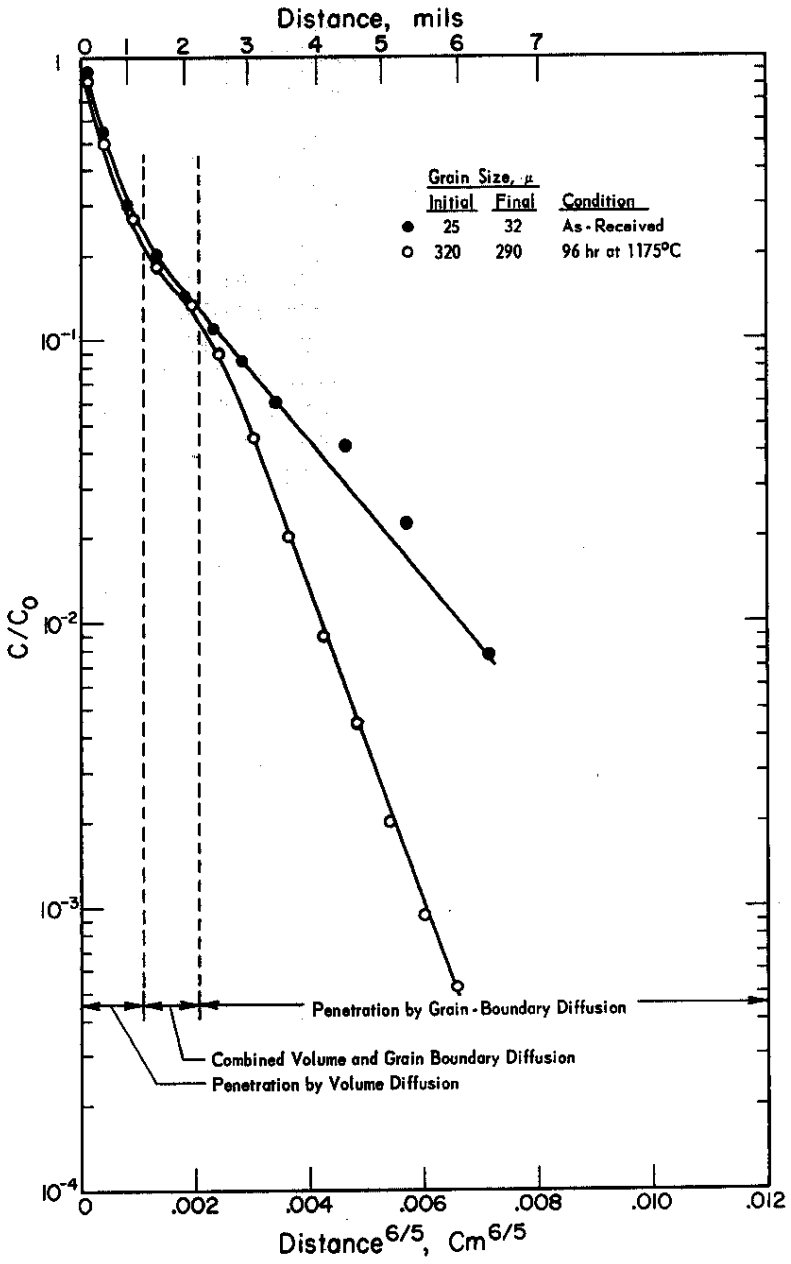

FIG. 1 EFFECT OF GRAIN SIZE ON DIFFUSION IN "INCONEL" 600
(Diffusion Anneal – 100 hours at 1000°C)

Previous results had shown that diffusion was unacceptably fast in TD Nickel and TD Nickel Chromium when measured in a direction parallel to the extrusion direction.[3,4] To determine if the stringers of thoria particles contributed to this fast diffusion, additional measurements were made on samples machined so that the diffusion direction was transverse to the extrusion direction. With TD Nickel equivalent results were obtained in both directions, but with TD Nickel Chromium diffusion was significantly slower in the transverse direction than in the longitudinal direction. Detailed metallographic examinations are in progress to relate the differences in diffusion with differences in grain structure and continuity of the thoria stringers.

Oxidation Resistance

The oxidation characteristics of two groups of encapsulating alloys are being defined at 1000°C for periods up to 10,000 hr.[4,8] Tests of the first group, terminated after 9400 hr, showed that TD Nickel Chromium, "Hastelloy" X, "Hastelloy" C, and "Inconel" 600 had acceptable resistance to oxidation. Extrapolation of these results to 50,000 hr indicated that depths of 0.015 to 0.030 inch would be affected by oxidation.

Heating for 10,000 hr in still air at 1000°C to measure oxidation resistance was completed on the second group of alloys. The thicknesses affected by the formation of a surface scale, by intergranular penetration, and by alloy depletion are shown in Table III, along with similar data from the first group. Of the alloys in this second group, only "Haynes" 25, "Tophet"* C, and GE2541** had acceptable resistances to oxidation. More spalling of the surface scale occurred in the second group of alloys than in the first.

The results of all the tests at 1000°C are summarized in Figure 2. The alloys can be arranged in three categories. First, TD Nickel Chromium is significantly more resistant to oxidation than any of the other alloys. Second, a group of six alloys have acceptable resistance to oxidation; 0.015 to 0.030 inch are expected to be affected during exposure for 50,000 hr. Third, a group of 20 alloys have unacceptable resistances to oxidation; 0.040 to 0.080 inch are expected to be affected by oxidation during exposure for 50,000 hr.

* Trademark of Wilbur B. Driver Co.
** Product of General Electric Co.

TABLE III

Oxidation of Potential Capsule Materials

(Still air at 1000°C)

Material	Zone Thickness, mils			
	Total Affected[a]	Surface Scale[b]	Intergranular Penetration	Alloy Depletion
Group I - 9400 hr				
TD Nickel Chromium	2.4	2.4		
"Hastelloy" X	9	2.0	7	7
"Hastelloy" C	11	3	3	8
"Inconel" 600	11	2	9	
"Incoloy" 825	17	4	13	13
TD Nickel	34	34		
Nickel 270	56	36	20	
Group II - 10,000 hr				
"Haynes" 25	8	2(2)	4	4
GE 2541	9.5	0.5(5)	4	4
"Tophet" C	12	1(7)	4	4
"Haynes" 8188	12.5	1.5(4)	5	7
N-155	13	2(3)	5	8
RA 333	16	6	8	10
"Tophet" 30	16	4(2)	6	10
50 Ni-50 Cr	18	2	7	16
"Hastelloy" F	20	2(3)	6	15
"Tophet" A	20	2(8)	6	10
"Hastelloy" G	21	3(2)	7	16

(a) Sum of visible surface scale, scale that spalled, and larger of intergranular penetration or alloy depletion.

(b) Number in parentheses is estimated thickness of scale that spalled.

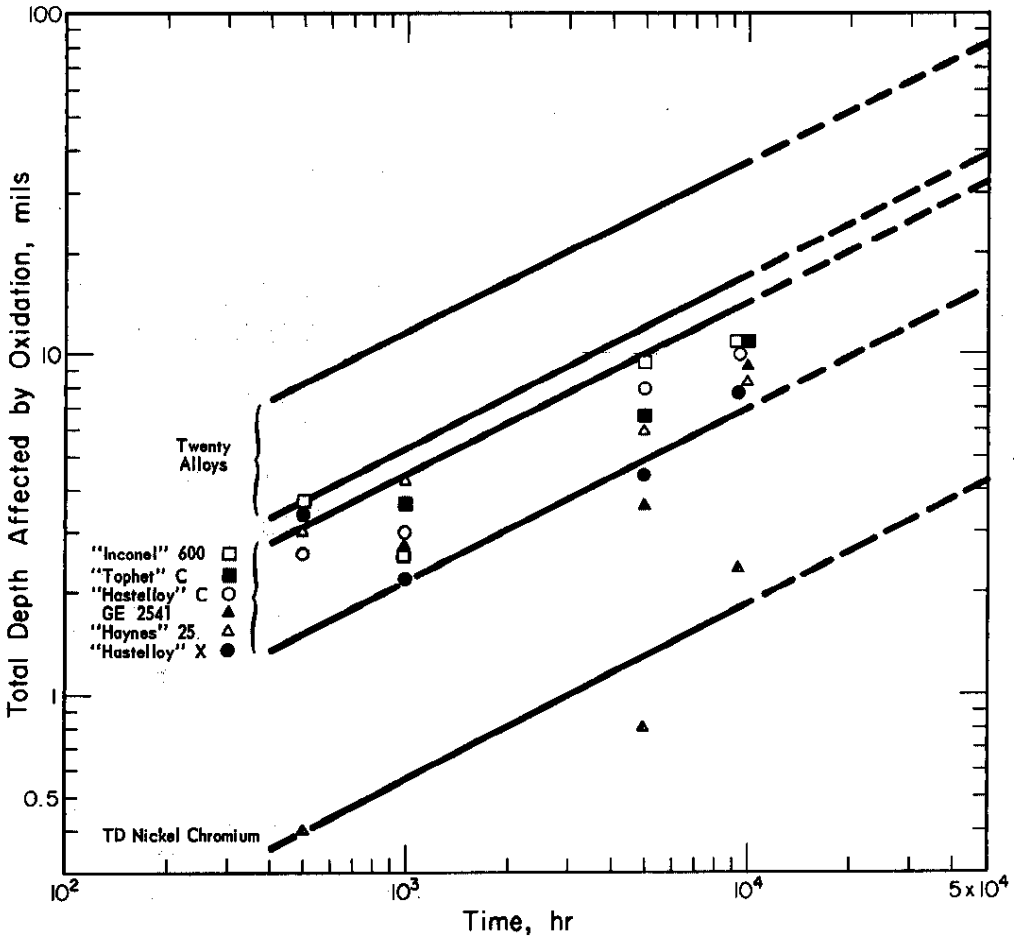

FIG. 2 OXIDATION OF CAPSULE MATERIALS AT 1000°C

Although there was some scatter in the data for the individual alloys, the total depth affected by oxidation was proportional to the square root of exposure time, as expected. Previous results had shown that data from 500-hr tests fit the expected Arrhenius relationship with temperature between 850 and 1150°C.[4] These observed kinetics are being verified by a 3000-hr test at 1150°C. This exposure should produce the same amount of oxidation as predicted for 50,000 hr at 1000°C. X-ray and microprobe analyses of the surface scales and adjacent metal are also in progress.

Capsule Fabrication and Testing

Material Selection

"Haynes" 25, "Hastelloy" X, "Hastelloy" C, and "Inconel" 600 are being used as the capsule materials for tests with irradiated cobalt at 1000°C for 10,000 hr or more. This selection was based on the evaluations of encapsulation materials described in earlier reports of this series. The predicted fractions of the 0.100-inch-thick wall that will be affected by compatibility, oxidation, and diffusion of ^{60}Co after 50,000 hr at 1000°C are shown in Figure 3. Each capsule is approximately 1.3 in. long and contains about 13,000 Ci (280 Ci/g). Tungsten wafers are included in some of the capsules to provide data on its compatibility with cobalt and the respective superalloys. Tungsten is a potential shield material and also a material for encapsulation of cobalt metal for service at 1200°C or higher.

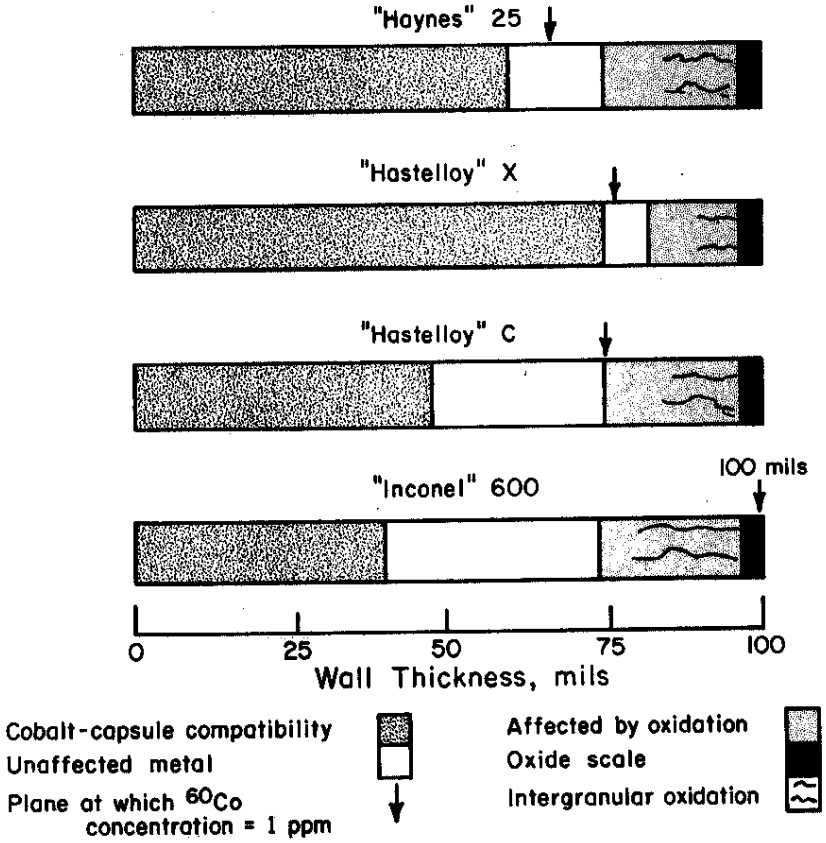

FIG. 3 PREDICTED REACTION ZONES IN CAPSULE WALL
(Expected after 50,000 hours at 1000°C)

All of the 17 capsules required for the heating tests at 850 to 1000°C were fabricated. Thermocouples to measure the surface temperatures will be spot welded to selected capsules, and heating tests will be started early in the next quarter.

Heating Tests of Capsules Containing Unirradiated Cobalt

Tests at 850°C

One capsule of TD Nickel was heated for 5000 hr in flowing argon at 850°C. Integrity was maintained and no dimensional changes occurred. As expected, the thickness of the capsule-cobalt compatibility zone was $\sqrt{5}$ times that in a previous 1000-hr test. The oxide scale was about 0.0015 inch thick or about 1/10th of that expected if the exposure was in air.

Tests at 1000°C

One capsule each of "Inconel" 600, "Hastelloy" C, "Haynes" 25, and TD Nickel Chromium were heated in air at 1000°C for 5000 hr. No detrimental effects were observed. The depths affected by the cobalt-capsule compatibility reaction and by oxidation of the capsule wall were in general agreement with previous short-term screening tests and capsule tests, Figure 4. All the data fit the expected parabolic relationship with time. Heating tests are continuing with 22 capsules that contain unirradiated cobalt in five different capsule materials.

An "Inconel" 600 capsule that had an internal atmosphere of air instead of helium, as used in all other capsules, was heated for 1000 hr at 1000°C. No oxidation of the cobalt wafers or the internal surfaces of the capsule was apparent. The thickness of the compatibility reaction zone was typical of the heating conditions.

Heating Tests of Capsules Containing Irradiated Cobalt

The first demonstration of satisfactory 10,000-hr durability of encapsulated ^{60}Co at ~900°C was completed with an "Inconel" 600 capsule that contained 15,000 Ci of irradiated cobalt. Integrity was maintained and no dimensional changes occurred. Similar results had been obtained in previous 1000- and 5000-hr tests.[2-5] Metallographic measurements of the cobalt-capsule compatibility reaction and the oxidation of the capsule wall will be made for comparison with those from earlier tests of "Inconel" 600 capsules containing unirradiated cobalt.[5]

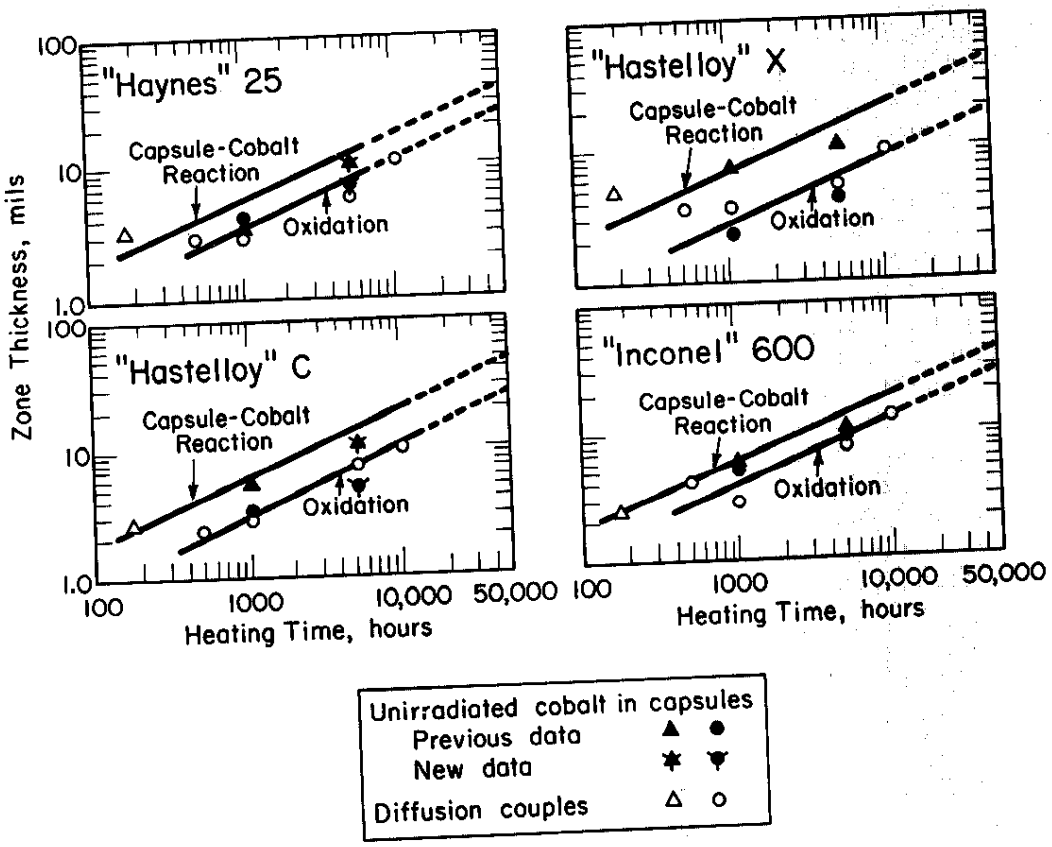

FIG. 4 GROWTH OF COBALT-CAPSULE REACTION AND CAPSULE OXIDATION ZONES (Comparison of Screening and Capsule Heating Tests at 1000°C)

Measurement of Surface Temperature of Electrically Heated Capsule

Comparison was made of the response of several thermocouples attached to an electrically heated capsule, that simulated a ^{60}Co capsule, to allow selection of suitable thermocouples for the live capsules. The "Inconel" 600 test capsule was 0.940-inch OD and 3 inches long. A 3/4-inch-OD electric cartridge heater was placed inside the capsule to simulate heat generation with ^{60}Co. The capsule was tested inside a small muffle furnace of the type that will be used in the High Level Caves (HLC) for heating ^{60}Co capsules.

In the first series of tests, bare chromel and alumel wires were embedded in the outer surface of the capsule such that the thermocouple junction formed was at the surface. This thermocouple was used to compare chromel-alumel thermocouples, which had 0.011-inch and 0.020-inch diameter sheaths, that were

mechanically staked into longitudinal grooves in the outer surface of the capsule, and a chromel-alumel thermocouple, which had a 1/16-inch diameter sheath, that was either clamped or spot-welded to the capsule surface.

The 0.020-inch thermocouple indicated a temperature only 4°C lower than the bare wire thermocouple at a capsule surface temperature of 850°C. For calibration, power input was 200 watts to the capsule, with the furnace heaters off. The 0.011-inch thermocouple indicated 6°C lower than the bare junction and was erratic, probably because of a poor junction; therefore, no further reference will be made to it. The 0.020-inch thermocouple was selected as the reference for comparison with the 1/16-inch thermocouple because of the fragility of the bare wire thermocouple. These tests were conducted at a capsule surface temperature of 850-900°C with power inputs of 175 watts to the capsule and 100 watts to the furnace heaters to simulate the expected heat balance with ^{60}Co.

With the 1/16-inch thermocouple fastened with a hose clamp such that 3/16 inch of the tip was exposed, its temperature indication was 45°C lower than the reference. A very small gap could be seen between the tip and capsule. This method of attachment duplicates that used to measure the surface temperature of a ^{60}Co capsule recently. With the clamp directly over the tip to insure good contact, the indicated temperature was 35°C lower than the reference. The clamp acted as a heat fin causing a large ΔT across the thermocouple sheath. With the tip spot welded to the capsule to provide good contact and eliminate the heat-finning clamp, the indicated temperature was 17°C lower than the reference.

For measurement of ^{60}Co capsule temperatures, a thermocouple sheath diameter of 0.040 inch was selected. This provides adequate durability for remote attachment by spot welding but reduces the area for heat transfer, compared to the 1/16-inch sheath, thereby lowering the ΔT across the sheath. Chromel-alumel thermocouples with 0.040-inch "Inconel" sheaths were obtained and another series of comparison tests was performed.

In addition to the thermocouple with a 0.040-inch diameter sheath, a thermocouple with a 0.020-inch diameter sheath was staked into a groove on the surface of the capsule, and a bare junction thermocouple was formed by chromel and alumel wires individually spot-welded to the surface of the capsule. The indications from all three thermocouples were essentially the same, showing a capsule surface temperature of 848°C. The test was conducted with an electrical power input of 175 watts to the capsule heater and 100 watts to the furnace heater, simulating the heat balance with a ^{60}Co capsule (60,000 Ci), with a surface temperature of about 850°C, inside a similar furnace.

Thermocouples (0.040-inch dia) will be spot-welded to six of the recently completed ^{60}Co capsules. Three of these instrumented capsules will be heated in the large 1000°C furnace and one each in the small 1000°C and the two 850°C furnaces (see below). Temperature measurements by these thermocouples will allow adjustment of the electrical power input to the furnaces to maintain the desired capsule surface temperatures.

Preparation for Encapsulated ^{60}Co Heating Tests

Three furnaces have been modified and installed in the HLC to test encapsulated ^{60}Co with capsule surface temperatures of 850-1000°C in still air. These furnaces are in addition to the large furnace installation reported in DP-1155-I. Two of the furnaces are for testing 60,000 Ci each at 850°C and the other for testing 60,000 Ci at 1000°C. Individual lead shield enclosures were fabricated and installed; structural steel supports were installed in the cell to support the shields. Remote temperature controllers for all four furnaces are being constructed and should be completed during July.

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