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AEC RESEARCH AND DEVELOPMENT REPORT

SAVANNAH RIVER LABORATORY ISOTOPIC POWER AND HEAT SOURCES

QUARTERLY PROGRESS REPORT

JANUARY - MARCH 1967

PART I - COBALT-60

DU PONT
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Savannah River Laboratory

Aiken, South Carolina

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H. S. Hilborn, Compiler

May 1967

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SAVANNAH RIVER LABORATORY
AIKEN, S. C. 29801

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PREFACE

This report is the third 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 present 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 in January, February, and March 1967. Previous reports were issued as single volumes. The first report, DP-1088, presented some background data and reported the data from work in July, August, and September 1966; the second report, DP-1094, reported the data from work in October, November, and December 1966.

Isotopic fuel data sheets on cobalt are included as appendixes to this report, and to DP-1094, and serve to update DP-1051, Revision 1, "Properties of ^{60}Co and Cobalt Metal Fuel Forms" by W. C. Windley, Jr.

SUMMARY

About 9 million curies of ^{60}Co (140 kilowatts) at an average of 400 curies per gram of metal (55 watts/cm^3) will be discharged from the reactor during the next quarter.

5000- and 10,000-hour heating tests of capsules of radioactive cobalt metal contained in "Inconel" 600 were begun at 850°C in air.

An "Inconel" 600 capsule with a 0.050-inch-thick wall that reached a temperature of 1100°C for 3 to 6 hours swelled but did not leak.

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DISCUSSION

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. Specific objectives are to establish allowable operating limits for:

- Capsules of radioactive cobalt metal contained in an oxidation resistant alloy by the end of FY-1968, and
- Capsules of a high temperature fuel form of ^{60}Co contained in a refractory alloy by the end of FY-1970.

The program is a limited one to meet needs of general applicability to heat source designers and users. Development of specific concepts is not at present included in the scope of the SRL program.

PRODUCTION OF ^{60}Co FOR HEAT SOURCE DEVELOPMENT

Production of high-activity cobalt for experimental programs and possible heat source demonstrations was continued. It is estimated that about 9 million curies of ^{60}Co (140 kilowatts), averaging 400 curies per gram of metal (55 watts/cm^3), will be discharged from the reactors during the next quarter. This will include nearly 7,000,000 curies as 0.745-inch-diameter by 0.040-inch-thick wafers (plated with 0.0005 to 0.001 inch of nickel) and the remainder as 0.80-inch-diameter wafers and 3-inch-long slabs of various types (nickel-plated, stainless-steel-clad, stainless-steel-bonded).

MATERIALS TECHNOLOGY AND DEVELOPMENT

High-Temperature Fuel Candidates

Alloys and compounds of cobalt that have higher melting temperatures than cobalt metal are being explored for use in high-temperature ^{60}Co heat sources.(1) Cobalt-rhenium alloys have sufficiently high melting temperatures but would have to be fabricated after irradiation to prevent conversion of the rhenium to osmium and the concurrent formation of a lower-melting alloy.(2) Methods for fabricating Co-Re alloys are being developed that would be suitable for use with irradiated cobalt.

Evaluation of Encapsulating Materials for Cobalt Metal

Selection of suitable encapsulating materials and definition of their limiting operating conditions requires first the assessment of their compatibility with cobalt. Additional data that are required include:

- Rates of diffusion of ^{60}Co through capsule walls
- Rates of oxidation of the capsule alloys, and
- Effects of these reactions on the mechanical properties of the materials.

Direct measurements of these characteristics are supplemented by tests in which capsules fabricated from the most promising materials and containing unirradiated cobalt are annealed for up to 10,000 hr. Effects of the radiation field are being measured in a smaller number of companion tests on identical capsules containing ^{60}Co.

Compatibility

Previous evaluations of compatibility emphasized oxidation resistant nickel- and cobalt-based alloys.[1,2] However, in some heat sources, materials that are less oxidation resistant, such as pure nickel and the refractory metals, could be used to encapsulate cobalt. Measurements of the compatibility of these materials with cobalt metal have been initiated. An "Inconel"* 600 capsule containing alternate layers of radioactive cobalt (100-Ci/g wafers, totaling about 5000 Ci) and wafers of pure

* International Nickel Co., Inc., New York, N. Y.

nickel, tungsten, and rhenium was fabricated for annealing at 850°C for 1000 hr. Wafers of TD Nickel* and TD Nickel Chromium* were also introduced to obtain data on the effects of the radiation field on compatibility. Earlier compatibility tests between pure nickel and unirradiated cobalt showed that a solid solution zone that was metallographically indistinguishable from the nickel was formed at 800, 1000, and 1200°C; no voids were associated with this zone as there were with the nickel- and cobalt-based alloys. With tungsten two intermetallic compounds were formed at these temperatures. Tests with rhenium showed a solid solution zone similar to that observed with nickel, as predicted by the phase diagram. Tantalum was not included because it is incompatible with cobalt above about 1200°C because of the formation of a eutectic.

Diffusion of ^{60}Co

The rate of diffusion of ^{60}Co through various cladding materials is being measured by standard radioactive tracer techniques.[1,2] Initial results showed that the rate of volume diffusion was slowest in "Inconel" 600. In contrast, the rate of grain boundary diffusion was faster in this alloy than in most others; the rate increased with increasing nickel content of the alloys.

For the diffusion tests a layer of cobalt containing a small amount of ^{60}Co was electrodeposited on one surface of various cladding alloys and the specimens were heated in hydrogen for 40 hr at 800, 1000, and 1200°C. The concentration of the ^{60}Co was measured by counting the residual activity in the specimens as successive layers were dissolved electrochemically from the surface. Diffusion coefficients were calculated from the slopes of the penetration curves.[3] Results are shown in Figure 1.

The coefficients of volume diffusion varied little among alloys; between 800 and 1000°C, the coefficient for "Inconel" 600 was slightly lower than for the other alloys, in accord with the diffusion zone measurements in multilayer couples, but higher at 1200°C.

The coefficient of grain boundary diffusion decreased as the nickel content of the alloy decreased. This coefficient was larger (diffusion faster) than the volume coefficient, as in most metals.

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* E. I. du Pont de Nemours and Co., Wilmington, Del.

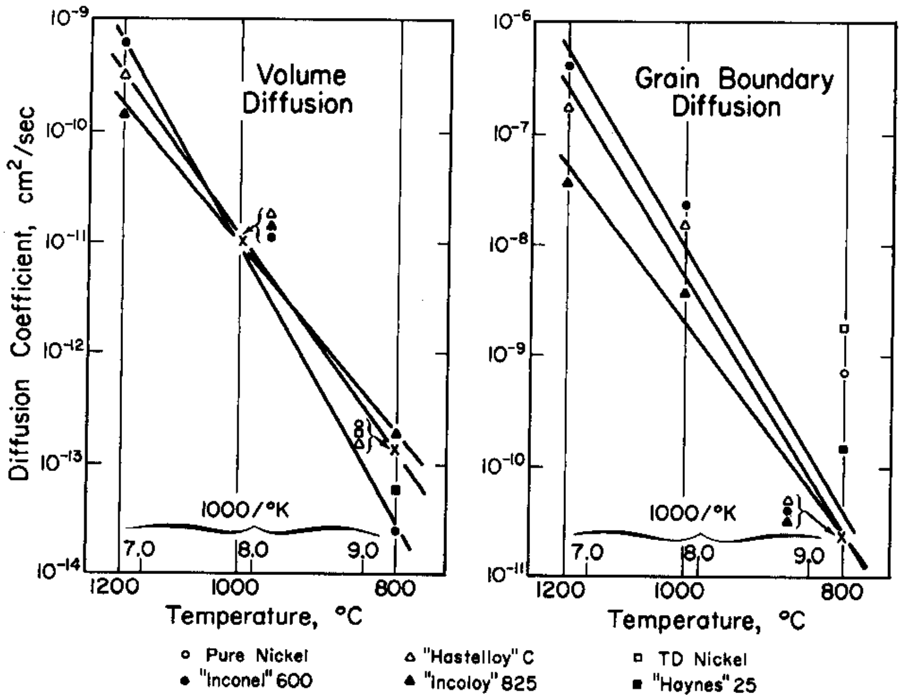


FIG. 1 TEMPERATURE DEPENDENCE OF ^{60}Co DIFFUSION COEFFICIENTS
Based on 40-hr Anneals

This result suggests that over short time periods the high nickel alloys, such as "Inconel" 600 and TD Nickel, might contain the ^{60}Co less effectively than the lower nickel alloys, such as "Hastelloy"* C. However, over long times, such as those expected in heat sources, the rates of grain boundary and volume diffusion would become effectively equal; penetration along grain boundaries in advance of that in the volume of the grains would be negligible.

These diffusion measurements are being continued to provide measurements of the coefficients of the six alloys shown in Figure 1, as well as TD Nickel Chromium, for 4, 40, and 100 hr at 800, 1000, and 1200°C. To provide data for times longer than 100 hr, cobalt-plated wafers of the various alloys are being included in the capsules containing unirradiated cobalt that will be heated for times up to 10,000 hr to test capsule reliability.

Rate of Oxidation

The oxidation resistance of numerous nickel- and cobalt-based heat-resistant alloys is being measured by exposing test coupons to still air at 1000°C for times up to 10,000 hr.[1] Examination of one group of alloys after 1000 hr showed that TD Nickel Chromium had outstanding resistance to oxidation and that "Inconel" 600, "Hastelloy" C, and "Hastelloy" X had satisfactory resistance.[2] Examination of a second group after 1000 hr showed that none of these alloys equaled the resistance of TD Nickel Chromium, but two alloys, "Haynes"* Experimental Alloy 8188, and "Tophet"** A were equivalent to the two "Hastelloys". Data for all alloys are shown in Table I. Companion specimens of the first group of alloys have been exposed for 5000 hr and measurements of oxide penetration are in progress. Exposure of other specimens of both alloy groups to 5000 and 10,000 hr is continuing.

TABLE I

Oxidation of Possible Capsule Materials

(1000 hr in air at 1000°C)

Material	Nominal Composition, wt %							Oxide Thickness, mils	Intergranular Penetration, mils	Total
	Co	Cr	Fe	Ni	W	Mo	Other			
Group I										
TD Nickel Chromium[a]		20		78			2 ThO_2	<0.1	None	<0.1
"Inconel"[b] 600		16	7	76				1.1	0.8	1.9
"Hastelloy"[c] C	2.5	15	5	bal	4	16		0.4	1.9	2.3
"Hastelloy"[c] X	1.5	22	18	bal	0.6	9		0.6	1.7	2.3
"Incoloy"[b] 825		22	30	42		3	0.9 Ti; 1.8 Cu	1.1	1.9	3.0
TD Nickel[a]				98			2 ThO_2	10.8	None	10.8
99.97% Nickel				100				11.1	Complete	Complete
Group II										
"Haynes"[c] 8188	bal	23	2	21	13		0.5 Mn	0.3	1.9	2.2
"Tophet"[d] A		20		80				0.4	1.8	2.2
GE 1541[e]		15	bal				4 Al; 1 La	0.1	2.3	2.4
"Hastelloy"[c] F	1	23	19	46		6.5	2 Mn; 1.6 Nb	1.0	1.9	2.9
"Hastelloy"[c] G	1	22	15	44	0.6	6.5	2 Mn; 2 Cu; 2.2(Nb+Ta)	1.4	1.6	3.0
GE 2541[e]		25	bal				4 Al; 1 La	0.1	2.9	3.0
"Tophet"[d] C		15	24	61				0.4	2.7	3.1
"Tophet"[d] 30		30		70				0.4	2.7	3.1
50 Ni-50 Cr		50		50				0.9	2.6	3.5
"Haynes"[c] "Detaclad"[a]								0.3	3.2	3.5
"Haynes"[c] 25	bal	20	3	10	15	0.5	1.5 Mn	0.7	3.6	4.3
N-155[f]	20	21	bal	20	2.5	3	1.5 Mn; 1.0 Nb	0.8	4.4	5.2
RA-333[g]	3	25	18	45	3	3	1.5 Mn	1.3	5.0	6.3
"Haynes"[c] 150	49	29	19	1	0.5	0.2	0.6 Mn	1.4	6.0	7.4

(a) E. I. du Pont de Nemours and Co.
(b) International Nickel Co.
(c) Union Carbide Corp.
(d) Wilbur B. Driver Co.
(e) General Electric Co.
(f) Carpenter Steel Co.
(g) Rolled Alloys, Inc.

* Union Carbide Corp., New York, N. Y.
** Wilbur B. Driver Co., Newark, N. J.

Capsule Fabrication and Testing

Heating Tests of Inactive Capsules

Reliability and durability of experimental capsules are being evaluated by heating for up to 10,000 hr at 850 and 1000°C in still air.[1] The compatibility and oxidation resistance of "Inconel" 600 was demonstrated by a 1000-hr test at 850°C; heating of companion capsules for 5000 and 10,000 hr is in progress. Both "Inconel" 600 and "Hastelloy" C were also compatible with unirradiated cobalt and had adequate resistance to oxidation during 1000-hr tests at 1000°C.[2]

An additional 1000-hr test at 1000°C has been completed using three capsules -- one each of "Inconel" 600, "Hastelloy" C, and TD Nickel. "Inconel" 600 and "Hastelloy" C again demonstrated adequate compatibility and oxidation resistance. The TD Nickel was compatible with cobalt, but oxidized so severely that it should not be considered for applications in heat sources that employ an oxidizing atmosphere above about 800°C. This test indicated that TIG welding may be an acceptable method for sealing capsules of TD Nickel.

The three capsules were heated in still air for 1000 hr at 1000°C. Each capsule had 0.050-inch-thick walls and was filled with nickel-plated wafers of unirradiated cobalt; spacers of the encapsulating material were used at the top and bottom. A void space of 0.250 inch was provided at the top of each capsule; the internal atmosphere was helium.

Oxidation was minor (about 0.001-inch penetration) at the external surfaces of the "Inconel" 600 and "Hastelloy" C capsules, but severe (about 0.015-inch penetration) on the TD Nickel capsule, Figure 2. These results agree with oxidation rates measured on test coupons, Table I. The oxide penetrations were comparable to those observed in the first test of "Inconel" 600 and "Hastelloy" C capsules.[1]

All three capsule materials were compatible with cobalt. The widths of the diffusion zones between the cobalt and the capsule walls, and the spacers, Figure 2, were 0.002 to 0.006 inch as compared to 0.002 to 0.008 inch expected from compatibility experiments using multi-layer diffusion couples.[2]

No evidence was seen of degradation of the weld in any of the three capsules other than segregation of the thoria in the TD Nickel which occurred during TIG welding. This segregation lowers the strength of the TD Nickel, but may be acceptable because requirements for capsule strength are not stringent.

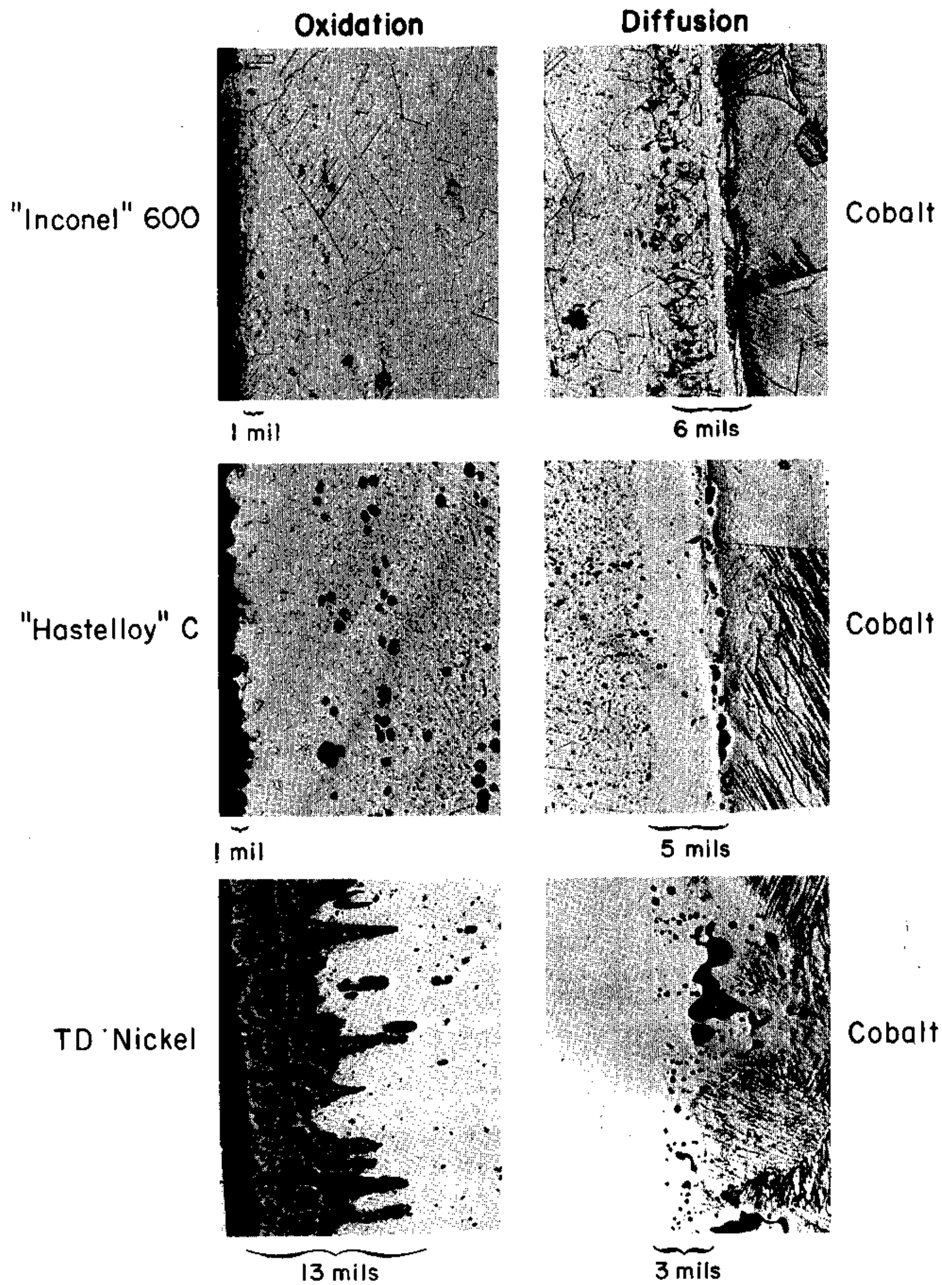


FIG. 2 OXIDATION AND COMPATIBILITY OF ^{59}Co CAPSULES
Heated 1000 hr at 1000°C, 80X

Similar capsules are being fabricated for additional testing, as follows:

- "Inconel" 600 and "Hastelloy" C - 5000 and 10,000 hr at 1000°C
- TD Nickel - 1000, 5000, and 10,000 hr at 850°C
- TD Nickel-Chromium, "Haynes" 25, and "Hastelloy" X - 1000, 5000, and 10,000 hr at 1000°C

<u>Heating of ^{60}Co Capsules</u>

Experimental capsules containing irradiated cobalt are being heated in air between 850 and 1000°C for up to 10,000 hr to evaluate encapsulation procedures and to measure the effects of radiation on oxidation and compatibility.(2) Two capsules with 0.050-inch-thick walls have been tested. A "Hastelloy" C capsule containing 9000 Ci was heated for 100 hr at 850°C. An "Inconel" 600 capsule containing 16,000 Ci was heated under the same nominal conditions, but experienced an inadvertent excursion to above 1100°C. This excursion occurred ~28 hr after the test started and lasted 3 to 6 hr. Since visual examination indicated that the capsule was not damaged, the test was continued at 800°C for an overall time of 130 hr.

The "Hastelloy" C capsule showed no unexpected changes in dimensions or microstructure, which indicated that the radiation field had little, if any, effect on oxidation of the capsule or its compatibility with cobalt. Diameter and length changes were less than 0.001 inch, the accuracy of the measurements. Metallographic examinations showed that the oxide film on the outer surface averaged 0.0002 to 0.0005 inch with occasional pockets up to 0.001 inch deep. The diffusion zone was about 0.004-inch wide.

The "Inconel" 600 capsule showed that the radiation field had no apparent effect on the compatibility or the oxidation of the external surfaces of the capsule, but the capsule length and diameter both increased 0.010 to 0.020 inch to produce a slight barrel shape. The absence of any appreciable weight change indicated that the dimensional changes were not due to severe oxidation.

When the capsule was sectioned the cobalt fell out as a solid rod. The grain structure of the capsule was typical of "Inconel" 600 that had been heated to above 1000°C. A reaction layer, about 0.002-inch thick was observed on the internal surfaces of capsule and the surfaces of the spacers, Figure 3.

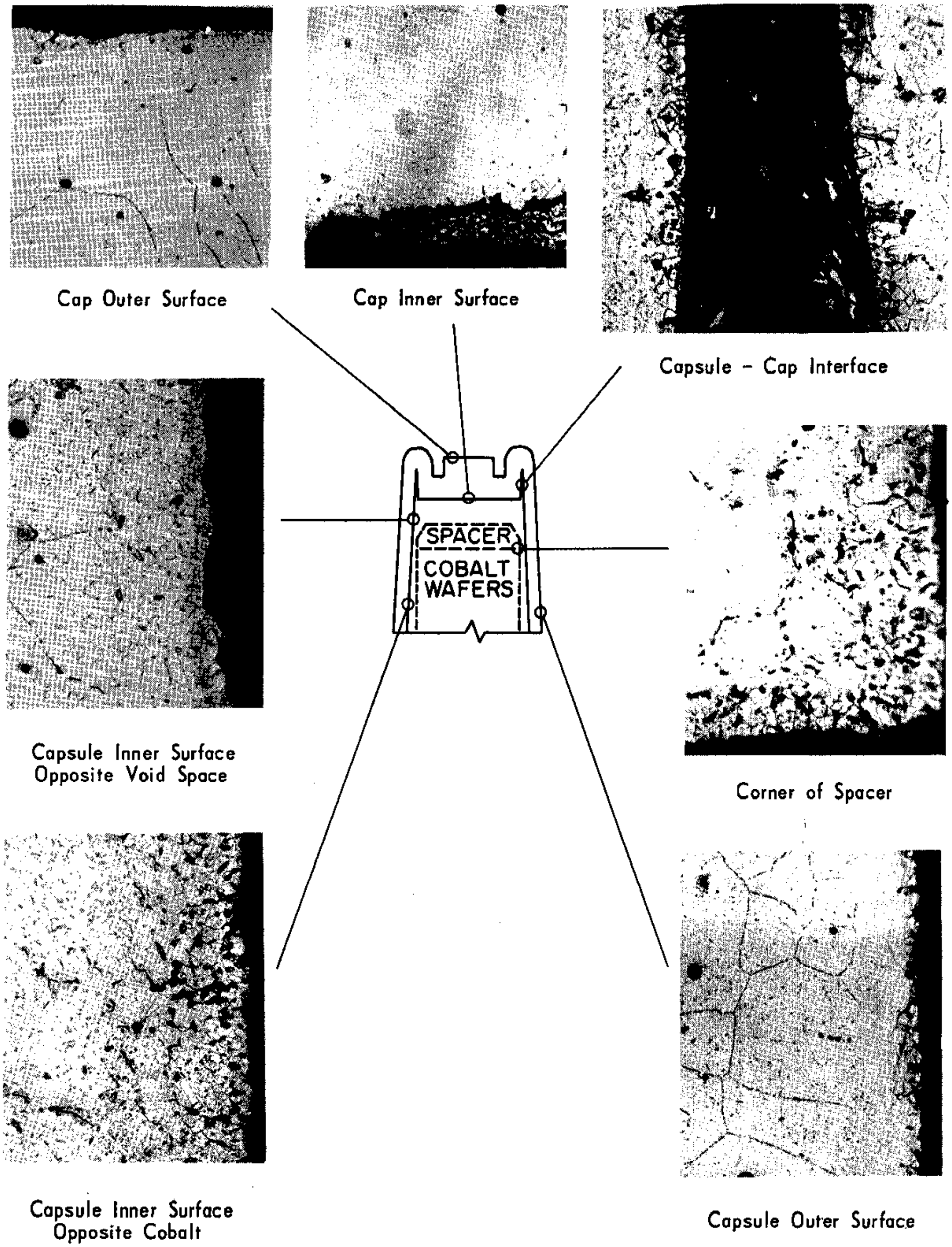
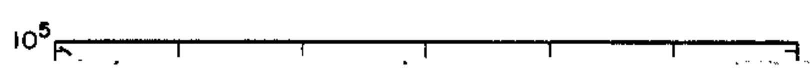


FIG. 3 MICROSTRUCTURES OF SWOLLEN "INCONEL" 600 CAPSULE (200X)

Oxidation of the external surface was not affected by the radiation. The oxide layer was about 0.0005-inch thick, as expected from the annealing conditions. For comparison, a layer 0.0005- to 0.001-inch thick was formed on an "Inconel" 600 capsule that contained unirradiated cobalt and was annealed 1000 hr at 850°C; layers about 0.001-inch thick have been observed on "Inconel" 600 test coupons and similar capsules annealed 1000 hr at 1000°C, as reported above.

Comparison of calculated stresses in the "Inconel" 600 capsule with the strength of "Inconel" 600 at 1100°C showed that the dimensional increase was predictable at the excursion temperature and is consistent with present evaluations of capsule internal pressure and wall strength. The maximum internal pressure at 1100°C for a capsule loaded at 25°C with helium at atmospheric pressure was calculated to be 75 to 110 psig, which causes a maximum hoop stress of 670 to 990 psi.[4] This wall stress, and the yield and creep strengths, of "Inconel" 600 at various temperatures are shown in Figure 4. Above 1060-1080°C the maximum capsule stress exceeds the metal yield strength. Because the capsule initially contained a relatively large void volume (13 to 17% of the cobalt volume), the deformation reduced the capsule internal pressure by only about 30%.



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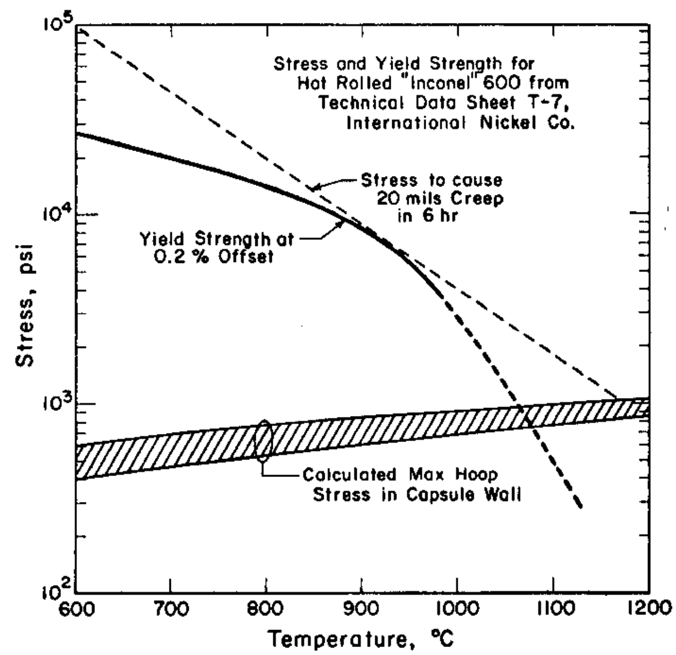

FIG. 4 CALCULATED WALL STRESS IN SWOLLEN "INCONEL" 600 CAPSULE

The deformation of the "Inconel" 600 capsule associated with the temperature excursion illustrates that there is a temperature limit beyond which capsules without external restraint cannot operate, even for short times. This short-term temperature limit can be approximated by the temperature at which the stress in the capsule wall due to internal pressure equals the yield strength of the capsule wall. These temperature limits were calculated for some of the potential capsule materials from strength data in the literature and are shown in Table II.

TABLE II

Limiting Capsule Temperatures to Prevent Deformation

Container Material	Approximate Short-term Temperature Limit, °C	Melting Temperature, °C
"Inconel" 600	1100	1370 to 1425
"Hastelloy" C	1220	1265 to 1340
"Haynes" 25	1280	1330 to 1410
TD Nickel	1350	1450

Two additional capsules of "Inconel" 600 with 0.050-inch-thick walls were fabricated and heating tests were begun at 850°C in still air for 5000 and 10,000 hr. Both capsules were 2.2-inches long and contained about 15,000 Ci (160 Ci/g) of irradiated wafers. Helium leak tests before and after heating for 4 hr at 850°C showed that the capsules had no leaks.

Fabrication has begun on additional "Inconel" 600 capsules for tests at 1000°C and on TD Nickel capsules for tests at 850°C.

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2. H. S. Hilborn (compiler). Savannah River Laboratory Isotopic Power and Heat Sources Quarterly Progress Report, October-December, 1966. USAEC Report DP-1094, E. I. du Pont de Nemours & Co., Savannah River Laboratory, Aiken, S. C. (1967).

3. A. D. Le Claire, "The Analysis of Grain Boundary Diffusion Measurements." Brit. J. Appl. Phys., 14, 351 (1963).

4. C. P. Ross, C. L. Angerman, and F. D. R. King, Development of Capsules for ^{60}Co Heat Sources, USAEC Report DP-1096, E. I. du Pont de Nemours & Co., Savannah River Laboratory, Aiken, S. C. (To be issued.)

LIST OF REPORTS AND PAPERS

The following is a list of reports and papers containing information on cobalt that have been prepared by the Savannah River Laboratory and are available upon request.

H. S. Hilborn (compiler). Savannah River Laboratory Isotopic Power and Heat Sources Quarterly Progress Report, October-December 1966. USAEC Report DP-1094, E. I. du Pont de Nemours & Co., Savannah River Laboratory, Aiken, S. C. (1966).

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* summary

APPENDIX A

ISOTOPIC FUEL DATA SHEET FOR COBALT

This supersedes section I.A.1.a (page 4) of DP-1051, Revision 1, "Properties of ^{60}Co and Cobalt Metal Fuel Forms" by W. C. Windley, Jr.

1. Composition

   a. Recommended composition of cobalt raw material[(a)]

Element	Recommended Content
Co + Ni	99.9 wt % min
Ni	1500 ppm (wt) max
Fe	1000
Cu	100
O	100
Si	100
Th	100
U	100
Al	50
Cd	50
Mn	50
Pb	50
S	50
Cr	20
Mo	20
V	20
W	20
P	10
B	5
Gd	5
Li	5

(a) This composition can be obtained commerically and is suitable to assure satisfactory performance in the reactor and in ^{60}Co heat sources.