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Radiation Effects on Reactor Materials

AEC Research and Development Report

IRRADIATION OF THORIUM SLUGS

by

R. P. Marshall

Pile Materials Division

May 1956

E. I. du Pont de Nemours & Co.
Explosives Department - Atomic Energy Division
Technical Division - Savannah River Laboratory

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RADIATION EFFECTS ON MATERIALS
(M-3679, 20th Ed., Rev. 1)

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ABSTRACT

Ninety-two thorium slugs were irradiated in the Hanford piles to study the dimensional stability of the metal. The burnups that were incurred covered the range from 300 to 2800 ppm of the Th^{232}. Slugs with burnups as high as 800 ppm were examined for visual and dimensional changes. The dimensional stability of these slugs was excellent, and no significant corrosion occurred on the aluminum cans. A cursory examination of slugs with burnups of greater than 800 ppm showed no gross dimensional [redacted]

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IRRADIATION OF THORIUM SLUGS

INTRODUCTION

In 1953 the Savannah River Laboratory initiated a test irradiation to study the dimensional stability of extruded thorium slugs under thermal neutron bombardment. The information that was available on dimensional stability had been obtained indirectly from other programs and indicated that thorium was dimensionally stable[1].

This report describes the preirradiation characteristics of the thorium slugs, and the postirradiation examination on a portion of the irradiated slugs. The work covered the period from early 1953 to early 1955.

SUMMARY

Thorium slugs, 0.8 inch in diameter and 10 inches long, showed excellent dimensional stability when irradiated to a burnup of 800 ppm of the Th^{232}. Average changes in dimensions were within the accuracy of the measurements: ±0.002 inch in diameter, ±0.005 inch in length, and ±0.002 inch in warp.

No significant corrosion of the aluminum cans occurred during the test.

DISCUSSION

HISTORY OF THE SPECIMENS

The thorium metal for this program was produced at Iowa State College, Ames, Iowa, in late 1952. Derbies of thorium-zinc alloy were obtained by the bomb reduction of thorium tetrafluoride by calcium with a booster of zinc chloride. The zinc was removed by distillation, and the resulting thorium sponge was induction melted and cast in cylindrical molds of graphite. A complete description of the Ames process is given in The Reactor Handbook, Vol. III, and in ORNL-1090[2].

Eight thorium ingots, 5.5 inches in diameter and 16 inches long, were extruded in copper jackets at 1450°F to one-inch diameter rods, each 45 to 55 feet long. A soft steel cone was inserted ahead of the die to streamline the metal flow. The extruded rod was usable without heat treatment or straightening. After pickling to remove the copper cladding, the rods were machined to slugs that were 0.808 inch in diameter and 10.14 inches long.

The bare slugs were double canned in aluminum by mechanical sizing and welding. The first canning was done by die sizing and the second by swaging. A flowsheet for the canning process is shown in Figure 1, and a drawing of the double-canned assembly is shown in Figure 2.

PREIRRADIATION EVALUATION

Slugs prepared for irradiation were measured for diameter, warp, and length as bare slugs, as single-canned slugs, and as double-canned slugs. The characteristics of the thorium metal were determined by chemical analysis, metallographic examination, and mechanical testing.

Chemical Analysis

Samples for spectrographic and wet analyses were taken at about twelve feet from the front of each rod. Additional samples for analysis of carbon, iron, beryllium, and silicon were taken from broken tensile specimens.

The rods were uniform in composition. The ranges of impurities found in the eight rods are summarized below:

Element	Amount, ppm	Element	Amount, ppm
Aluminum	10 - 30	Iron	80 - 170
Beryllium	230 - 475	Magnesium	10 - 15
Carbon	230 - 665	Nickel	8 - 70
Calcium	10 - 50	Silicon	0 - 65

The following elements were not detected or were present in amounts below 15 ppm:

Ag	Cu	Hf	Nd	Sc	V
As	Dy	Ho	P	Sm	W
B	Er	In	Pb	Sn	Y
Cb	Eu	La	Pd	Ta	Yb
Cd	Ga	Lu	Pe	Tb	Zn
Ce	Gd	Mo	Pr	Ti	Zr
Co	Ge	Mn	Sb	Tm	

Rare earth oxides were present in the range of 106 to 117 ppm. The range of the HCl insolubles, representing ThO_2, was 1.2 to 1.5 weight per cent. Densities varied between 11.64 and 11.67 gm/cc.

Metallographic Examination

The microstructure, inclusions, and defects of the metal were studied by metallography. A minimum of four samples was taken from each as-extruded rod.

The rod was completely recrystallized and had an average grain size of 0.020 millimeter, as shown in Figure 3. The grain size did not vary across the cross section or along the length of the rod.

Two principal types of inclusions were found in the as-extruded rod. First, a grain boundary constituent occurred in patterns that simulated the original grains of the casting, as shown in Figure 4. The rounded shape of the constituent and the presence of

isolated globules of similar material indicated that the constituent had conglomerated during heating and extrusion. This constituent is believed to be a low-melting intermetallic compound. Second, relatively heavy stringers of macroinclusions were tentatively identified as thorium oxide. Thorium oxide crystals form in the casting and are fragmented during extrusion, appearing mainly as stringers as shown in Figure 5.

Two types of defects were found in the examination, and it is probable that these defects were present in some of the slugs used for irradiation. Small gas holes were randomly distributed through each rod. A few microcracks were found that extended a maximum of 0.15 inch from the surface of the slug toward the center, as shown in Figure 6. These cracks usually contained included material.

Mechanical Testing

Tensile and hardness tests were made on each rod of thorium. Since the carbon content affects the properties of thorium, particular attention was paid to the variation of mechanical properties with carbon content. Test specimens were machined from samples near the front and rear of the as-extruded rods. Additional samples were taken on one rod to determine if the mechanical properties varied along the length of the rod.

The yield strength, ultimate tensile strength, and hardness increased with increasing carbon content, as shown in Table I. Elongation and reduction of area were not affected by the variation in carbon content of these rods. The true stress - strain curve, shown in Figure 7, shows a true yield point. There was no variation of mechanical properties from end to end of the rod.

IRRADIATION DATA

The slugs were divided into three groups and exposed for three different lengths of time at Hanford. During the irradiation, the slugs were loosely contained inside perforated aluminum tubes that were approximately the same diameter as the Hanford fuel slugs. A drawing of the perforated tube is shown in Figure 8.

POSTIRRADIATION EXAMINATION

Sixty slugs were examined for visual and dimensional changes in the High Level Caves[3] at the Savannah River Laboratory. No radiation damage was found. The maximum burnup that occurred in these slugs was 800 ppm of Th^{232}.

The surfaces of the aluminum cans were in excellent condition. There was no evidence of serious corrosion. When received from Hanford, the surfaces were covered by a white, powdery corrosion product, but this was readily removed by washing with alcohol, as shown in Figure 9. A number of slugs that had been marked with a black lead pencil were accidentally irradiated in that condition. No corrosion was found around the pencil marks. The pins and spacers in the perforated tubes had marred the slug surfaces to a depth of 0.005 inch.

Dimensional changes of the canned slugs were insignificant. The dimensional data are given in Table II. The average change in diameter, length, and warp was less than 0.001 inch. The maximum measured changes in diameter were +0.002 and -0.004 inch, each change occurring only once. All other changes in diameter were 0.001 inch or less. The maximum measured changes in length were +0.010 and -0.011 inch. The maximum measured changes in warp were +0.003 and -0.002 inch. Diameter and length were measured with snap gauges fitted with dial indicators; warp was measured with a special profilometer designed and built by the Cleveland Instrument Company. The accuracies of the measurements are estimated to be ±0.002 inch for diameter, ±0.005 inch for length, and ±0.002 inch for warp.

The dimensional changes took place in a random manner. There was no positive correlation with burnup, position in pile, carbon content, or position in the as-extruded rod.

These data are in agreement with the data reported by other investigators[4], who exposed hot-rolled thorium slugs in the Material Testing Reactor.

Thirty-two slugs were discharged with a maximum burnup of 2800 ppm of Th^{232} and were given a cursory examination. There was no evidence of gross dimensional instability. Six of the slugs with the highest burnup have been retained for future reference.

R. P. Marshall

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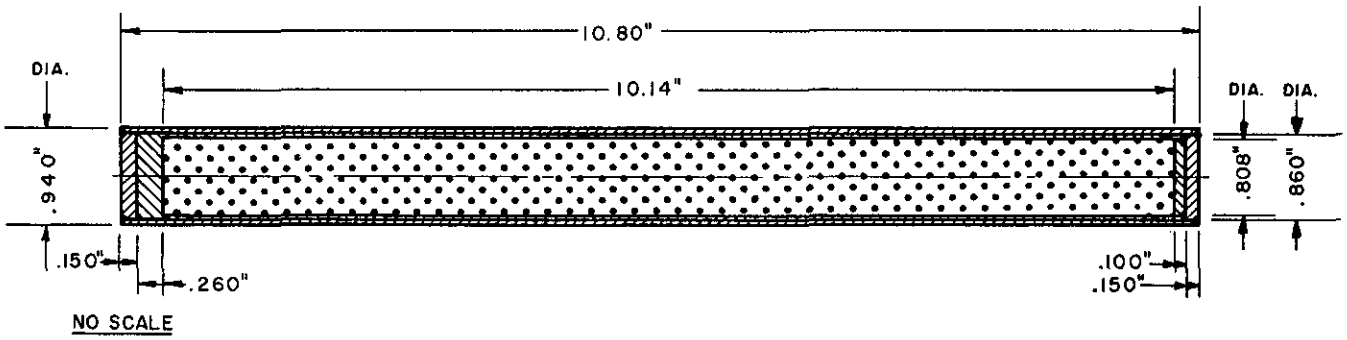
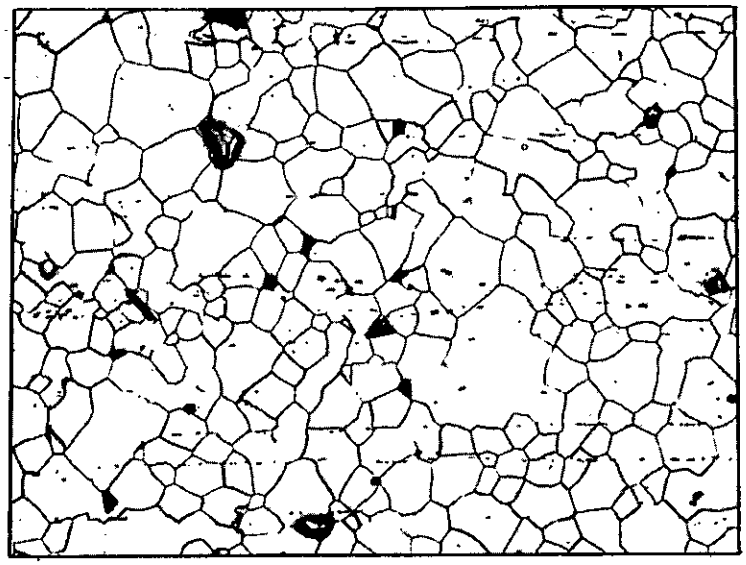
Pile Materials Division

FIGURE 2



DOUBLE-CANNED SLUG ASSEMBLY

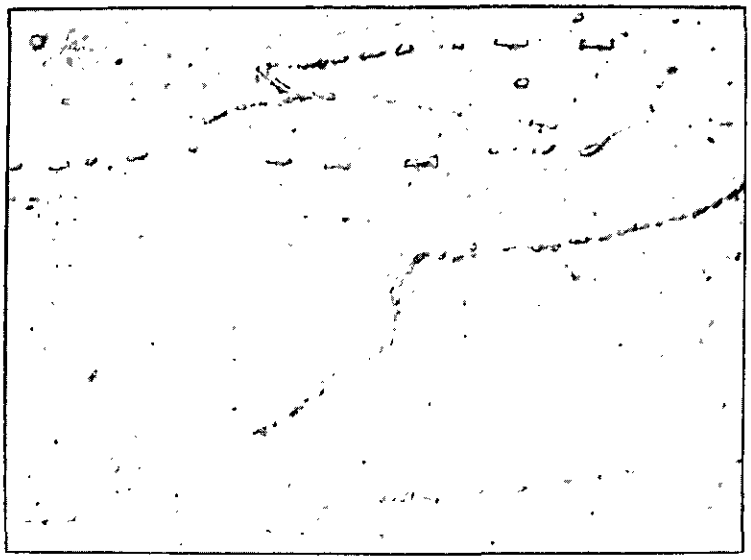
FIGURE 3



LONGITUDINAL SECTION 250X
GRAIN BOUNDARIES ARE SHOWN BY OXIDIZING THE SPECIMEN WITH AN ELECTROLYTIC ETCH, AND REPOLISHING. THE REPOLISH REMOVES THE OXIDE FROM SOME OF THE GRAIN BOUNDARIES.

MICROSTRUCTURE OF AS-EXTRUDED THORIUM ROD

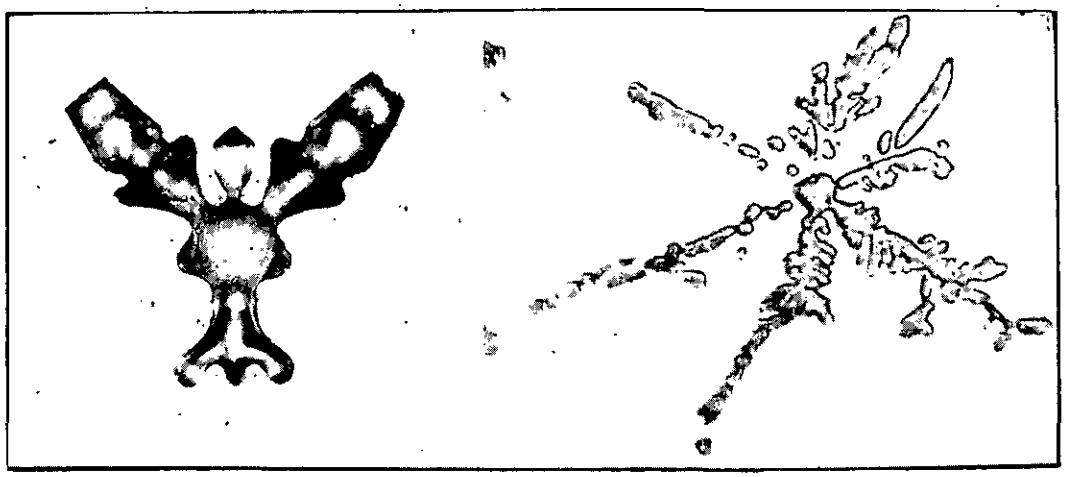
FIGURE 4



TRANSVERSE SECTION, AS-POLISHED. 750X
PATTERN OF CONSTITUENT SIMULATES SIZE AND SHAPE OF CASTING GRAINS. NOTE THAT THE PARTICLES OF CONSTITUENT ARE GLOBULAR, AND THAT SOME ARE RANDOMLY DISTRIBUTED.

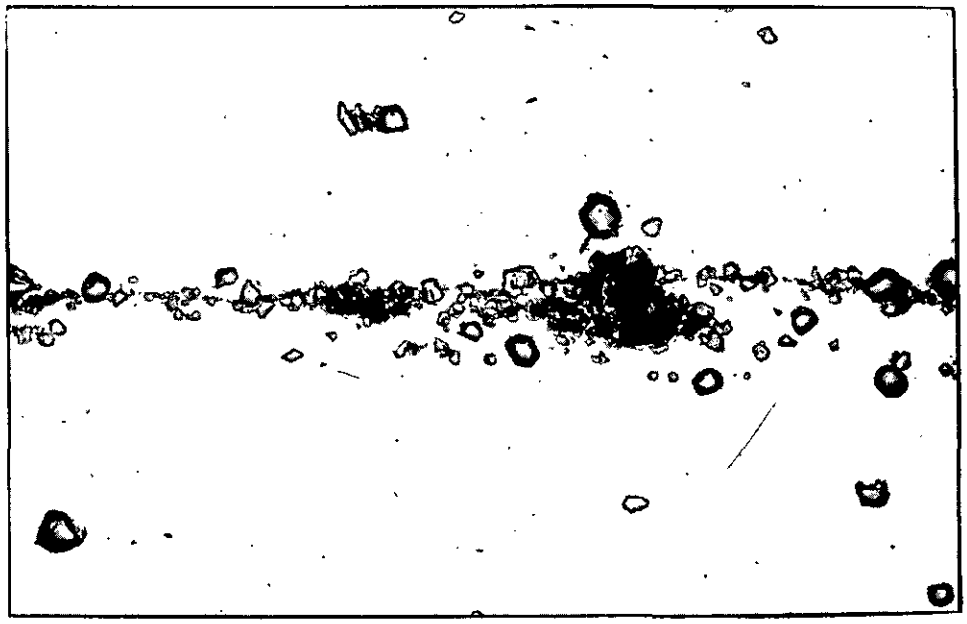
GRAIN BOUNDARY CONSTITUENT

FIGURE 5



1000 X

THORIUM OXIDE CRYSTALS IN THE CAST INGOT, PRIOR TO EXTRUSION. AS-POLISHED

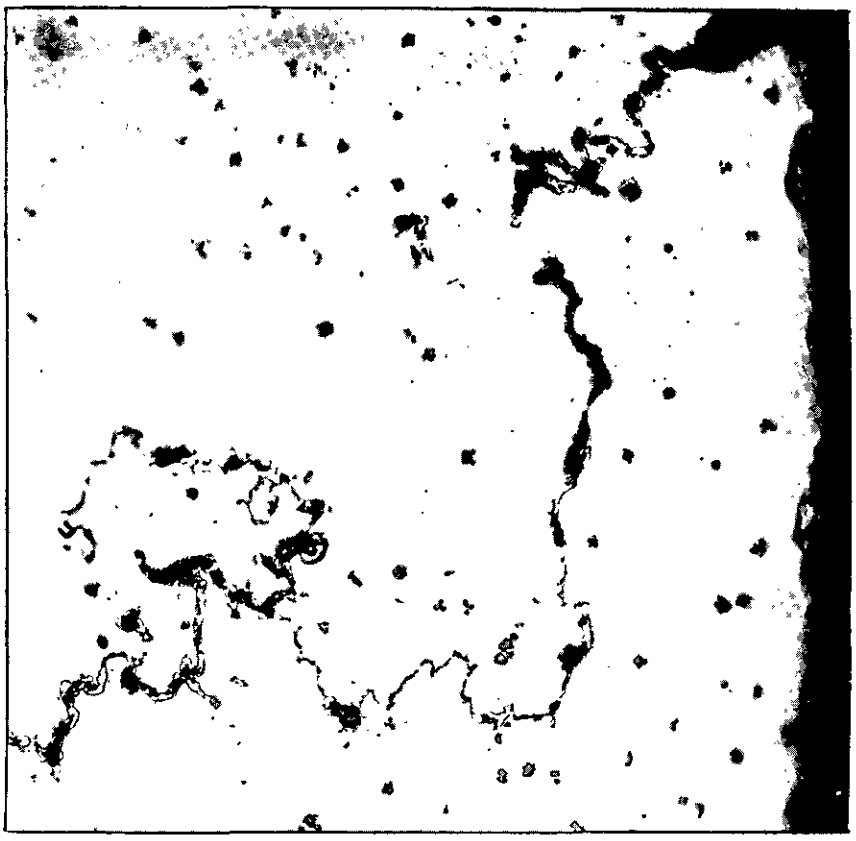


250 X

STRINGER OF THORIUM OXIDE INCLUSIONS IN AN EXTRUDED ROD. AS-POLISHED

THORIUM OXIDE INCLUSIONS

FIGURE 6

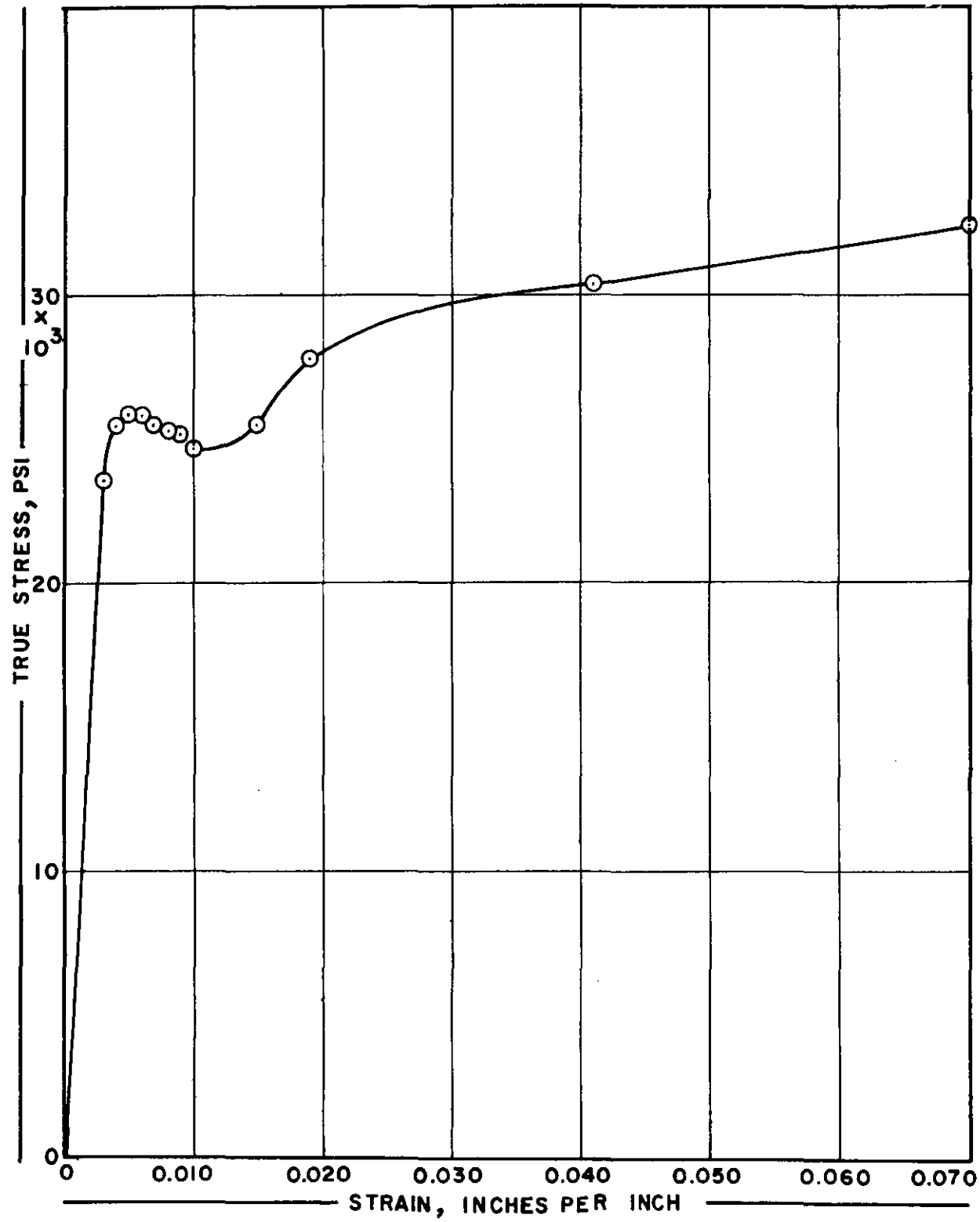


TRANSVERSE SECTION, AS-POLISHED. 100X

CRACK EXTENDED INTO SLUG APPROXIMATELY 0.15 INCH. NOTE INCLUDED MATERIAL.

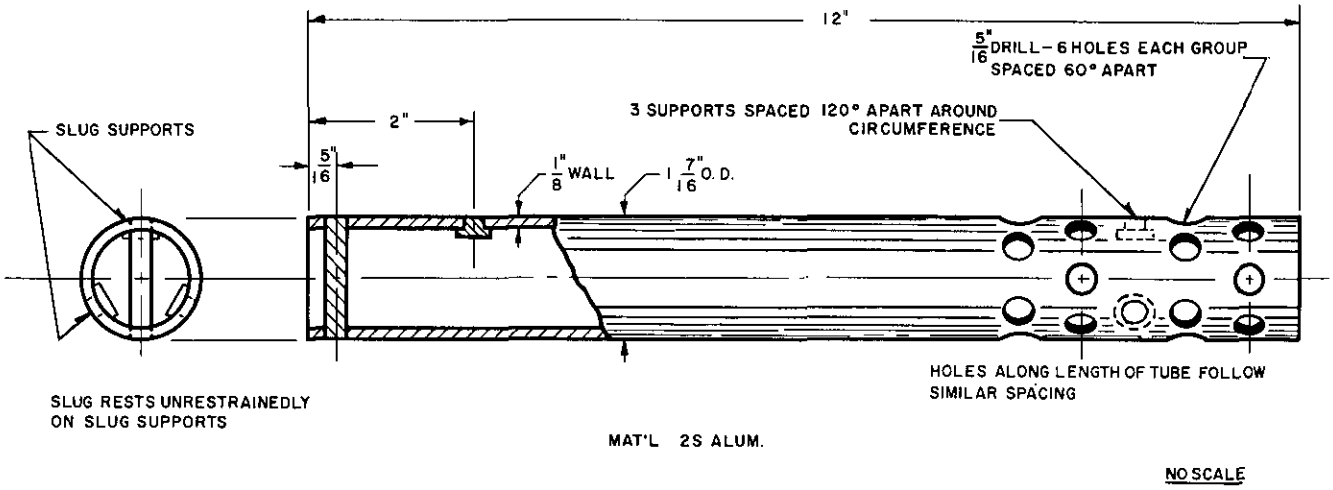
CRACK IN SURFACE OF SLUG

FIGURE 7



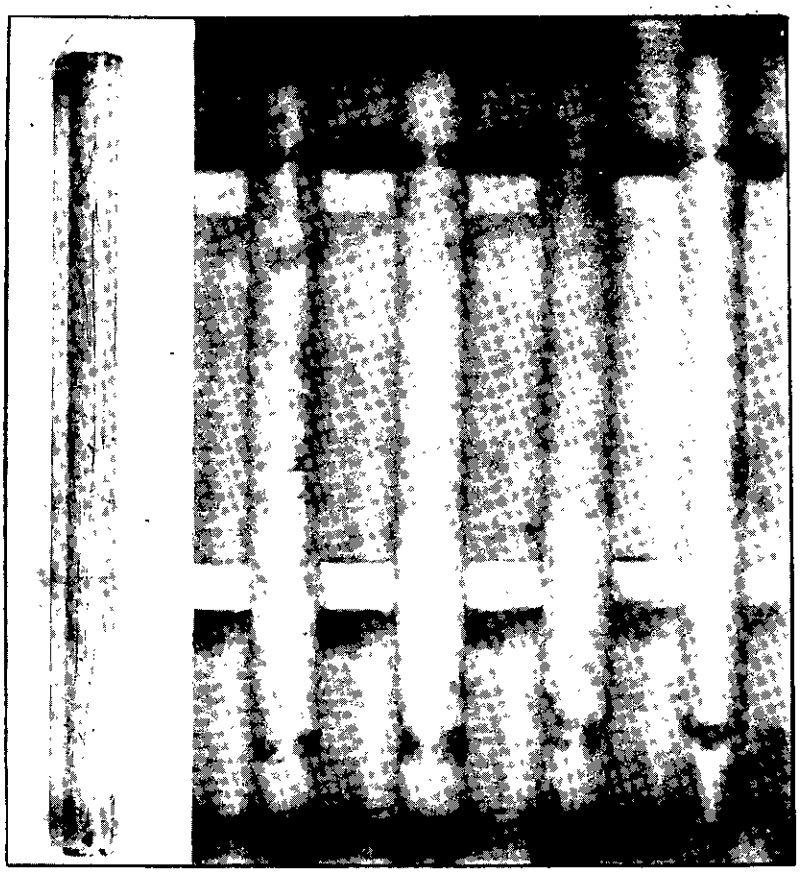
TRUE STRESS - STRAIN CURVE OF AS-EXTRUDED THORIUM ROD

FIGURE 8



PERFORATED TUBE

FIGURE 9



BEFORE IRRADIATION AFTER IRRADIATION 0.4 X

THE THREE SLUGS IN THE MIDDLE OF THE PHOTOGRAPH ARE AS-RECEIVED AT SRL AND ARE COVERED WITH THE WHITE POWDER. THE SLUG ON THE EXTREME RIGHT HAS BEEN WASHED IN ALCOHOL.

CANNED SLUGS, BEFORE AND AFTER IRRADIATION

TABLE I

EFFECT OF CARBON CONTENT ON MECHANICAL PROPERTIES

Billet	Carbon Content, ppm	Yield Strength, psi	Ult. Tensile Strength, psi	Elongation, per cent	Reduction of Area, per cent	Hardness, R_B
A-1102	230	21,400	27,000	46	73	13
A-1091	425	24,600	31,000	47	70	32
A-1095	435	24,600	29,800	45	69	34
A-1093	440	24,400	31,200	46	69	32
A-1096	460	25,600	31,100	45	71	33
A-1099	470	25,200	31,700	45	69	33
A-1105	625	30,400	35,100	45	69	48
A-1109	665	30,900	36,200	44	71	50

TABLE II

DIMENSIONAL CHANGES DURING IRRADIATION

Slug Number	Billet	Burnup, ppm Th^{232}	Δ Length, 0.001 inch	Δ Warp, 0.001 inch
121	A-1102	770	0	+ 1
170	A-1091	600	- 5	+ 1
122	A-1091	620	+ 9	+ 1
159	A-1091	620	- 2	+ 1
128	A-1091	700	+10	0
200	A-1095	450	+ 1	0
182	A-1093	400	- 1	+ 2
183	A-1093	490	- 6	0
193	A-1093	500	+ 2	+ 2
174	A-1093	560	- 8	+ 2
171	A-1093	720	-11	- 1
220	A-1096	330	+ 3	+ 1
209	A-1096	400	- 1	+ 3
211	A-1096	370	- 8	+ 2
217	A-1096	460	+ 1	+ 2
213	A-1096	510	- 1	- 2
215	A-1096	690	+ 4	+ 2
218	A-1096	690	- 5	+ 2
96	A-1099	290	+ 5	- 1
66	A-1099	420	+ 2	0
36	A-1099	550	+ 1	0
54	A-1105	660	-10	+ 1
85	A-1105	770	+ 2	+ 1
232	A-1109	730	- 1	- 1