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EXAMINATION OF IRRADIATED RaLa SOURCE FUEL ROD (PROTOTYPE NO. 2) FOR LOS ALAMOS SCIENTIFIC LABORATORY

by

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Metallurgy Division

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ABSTRACT

A LASL fuel-bearing assembly was irradiated in the Materials Testing Reactor and subsequently examined at ANL. The design was found to be adequate for containing specimens irradiated to high burnup levels if certain defects introduced by the fabrication procedure are corrected.

I. INTRODUCTION

In August, 1953, the Irradiation Group of the Metallurgy Division at Argonne National Laboratory was asked to disassemble and examine a specially fabricated reactor rod, containing a small amount of enriched uranium, which had been irradiated to a high burnup level in the Materials Testing Reactor. The rod, known as the "RaLa Source Fuel Rod," had been fabricated at Los Alamos Scientific Laboratory as part of a special capsule development program.[1] Up to that time fission products desired for use in LASL studies had been obtained through the laborious processing of tons of uranium at Oak Ridge.[2] It was conceived that large amounts of fission products might be concentrated in a small amount of material, thus greatly reducing the labor of extraction, if uranium-235 were irradiated instead of natural uranium. Burnup levels of 10% or more of the fissile atoms were envisioned.

The ANL group was interested in looking at fuel metals having high specific exposures and was particularly glad of the opportunity to examine a specimen in which the estimated burnup of total atoms averaged 7%, a figure considerably higher than had been reached in their own work at that time. The task was accepted, and the work was performed late in 1953.[3] Results were reported by correspondence as they became available. After the major portion of the projected study had been completed, the specimen was found to have less than 20% of the anticipated burnup. Interest at LASL was transferred to advanced designs which benefited from the ANL recommendations.[1,2] Some time later the project was suspended when it became certain that an abundance of fission products would be available through processing of spent MTR fuel elements.[2]

In view of the continued importance of stainless steel in the technology of fabrication of uranium fuel pins some of the ANL findings during the investigation may have general interest. Therefore, the work performed is described in this report, and the results are summarized after a brief initial description of the specimen capsule and its irradiation.

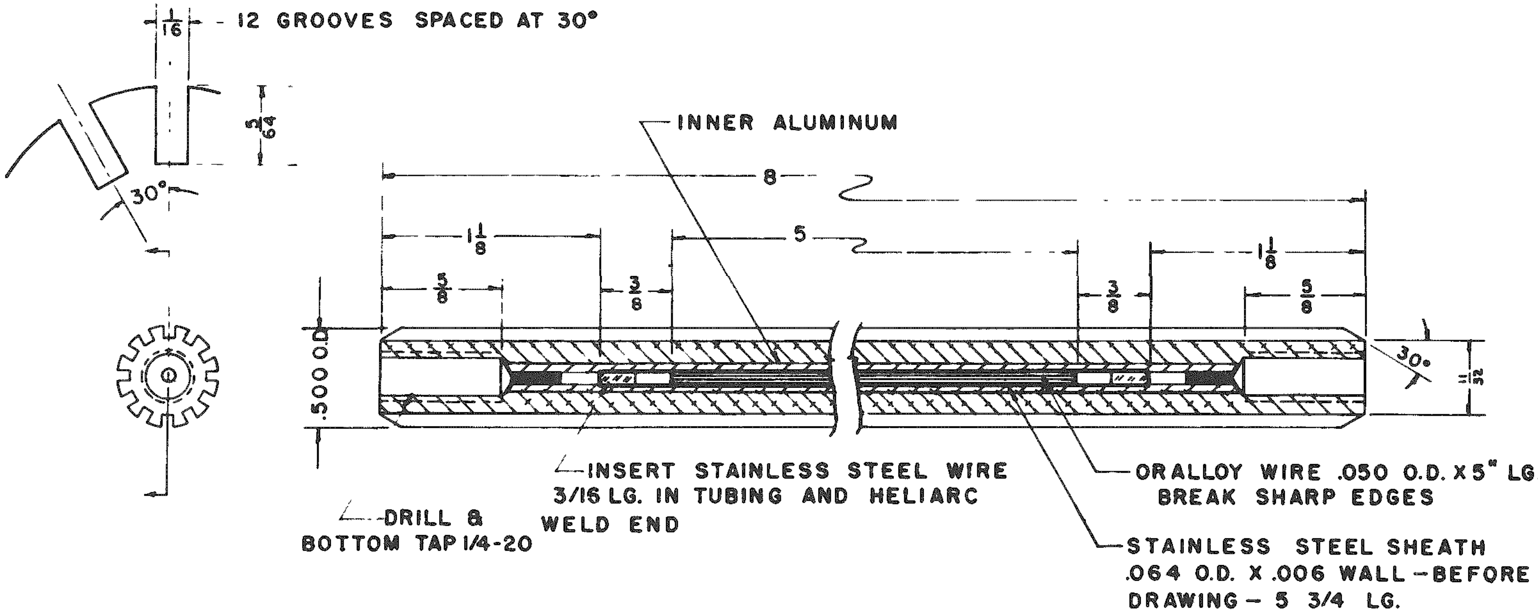
II. DESCRIPTION OF SPECIMEN IRRADIATION

The rod received at ANL was known as Prototype No. 2 in the series of models developed at LASL. Its design and dimensions are indicated in Figure 1. Pertinent metallurgical details of its fabrication have been obtained from LASL sources[1-3] and will be found in Appendix A. It was essentially a 1.27-mm (0.050-in.)-diameter wire of highly cold-worked uranium metal surrounded successively by a 0.152-mm (0.006-in.)-wall capsule of Type 347 stainless steel and two retaining jackets of 2S aluminum, which brought the total diameter to 1.27 cm (0.500 in.). The three enclosures had been sized over the core for good thermal contact by drawing through appropriate dies, and the outer jacket had been fluted longitudinally to increase its cooling surface. All ends had been closed by insertion of plugs of the proper materials, followed by Heliarc welding. Voids had been left between weldments and ends of the core material for accommodation of fission product gases. Figure 2 is a radiograph of the finished assembly, reproduced from a print furnished by LASL.

The assembly was attached to an adapter and inserted in MTR reflector position A-36;[3] the average unperturbed thermal flux is reported to be 1.5×10^{14} nv. Irradiation extended from August 13 to August 26, 1953, a total of 292 hr, and the reactor output was 336 MWD. Total exposure received by the fuel pin was estimated by MTR personnel to be 3.2×10^{19} nvt, after allowing for flux depression and self-shielding. From this number an instantaneous flux of 3.05×10^{13} nv was computed. Temperature of the uranium-stainless steel interface had been estimated at 400°C. The rod was stored in the canal for one week after removal from the reactor, and then was shipped to ANL.

III. EXAMINATION PROCEDURE

The appearance of the specimen rod as received at ANL is shown stereographically in Figure 3. The photograph was made with an L-shaped monocular camera system which could be rotated in the wall of the cave through the proper stereo angle. The upper end of the rod had been cut in such a way that a section of the support and pull rod was still attached. The lower adapter appeared to have been cut flush with the end of the fluted section. The assembly was free from evidence of corrosion except for the bottom end, on which can be seen an accumulation of white powder.



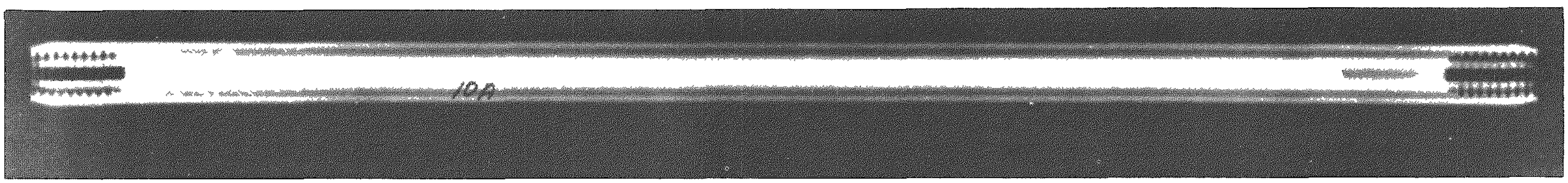
NOTE:

ASS'Y TO BE FABRICATED BY DRAWING STAINLESS ON TO ORALLOY WIRE. THEN DRAW INNER ALUMINUM (.250" O.D. X .035") WALL ON TO STAINLESS AND DRAW TO O.D. OF 0.160". THEN SEAL END OF INNER ALUMINUM: INSERT ALUMINUM WIRE 1/4 LG. & HELIARC WELD. THEN DRAW 0.817" O.D. X 0.438" I.D. OUTER ALUMINUM ON TO INNER ALUMINUM, AND DRAW TO O.D. OF 0.500". THEN MACHINE OUTER ALUMINUM AS REQ'D.

ALUMINUM TO BE 3S OR 2S
STAINLESS TO BE TYPE 347

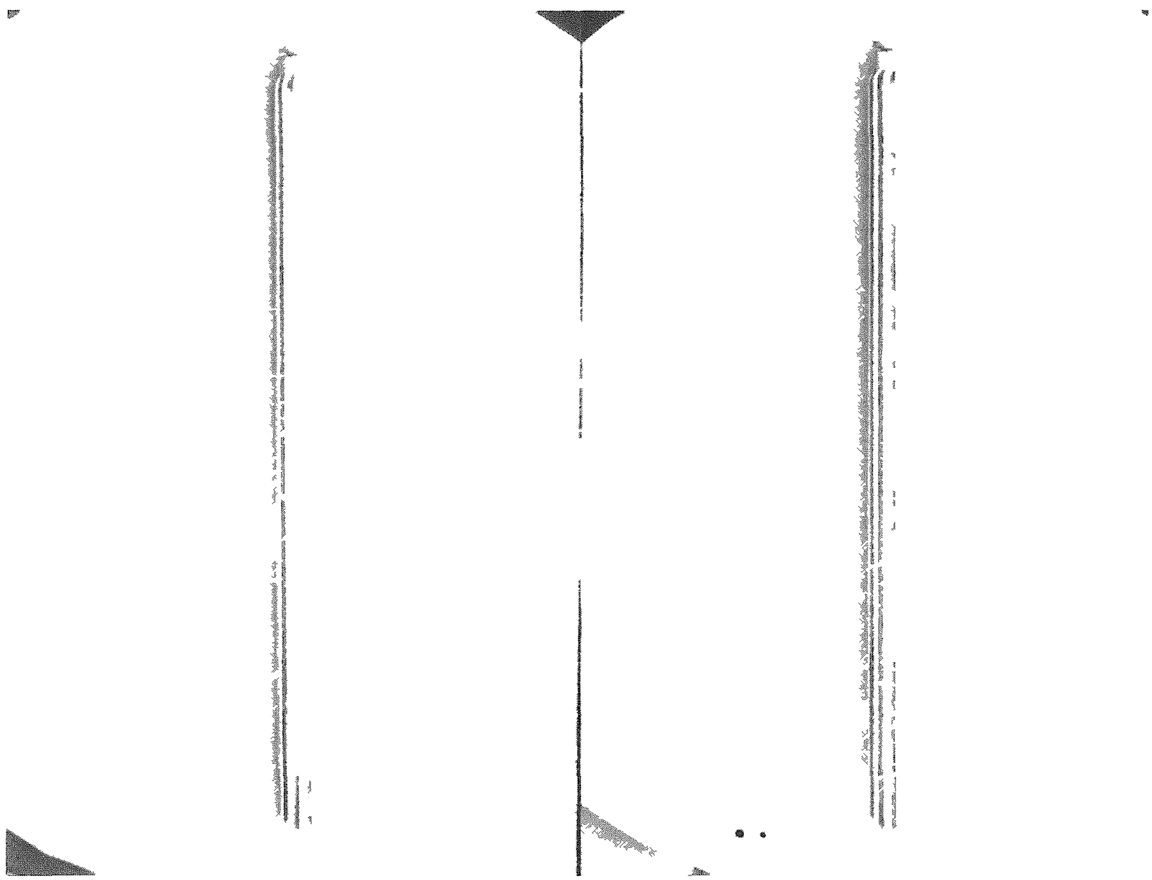
106-7339

Figure 1. Design of RaLa Source Fuel Rod Prototype No. 2 (Reproduced from LASL-006065)



106-7340

Figure 2. Radiograph of RaLa Prototype No. 2 before Irradiation (Reproduced from LASL-006064)



14924 1/2X

Figure 3. Stereophotograph of RaLa Rod as Received at ANL

Before the aluminum jacket was removed, heat generation of the assembly was checked by placing a thermocouple junction in contact with it in the middle of one of the fluted channels. The readings obtained were 4 to 6°C higher than ambient. However, when the assembly was placed in a beaker containing 700 ml of water, the change in temperature was only one degree for an immersion of half an hour. It was, therefore, evident that the stainless steel capsule would not need special cooling after stripping of the aluminum.

All of the aluminum that had been in contact with reactor coolant water was removed in a lathe, so that the diameter was reduced to 6.4 mm (0.25 in.) and the length to slightly greater than that of the inner capsule. In the machining operation the tip of the stainless steel plug on the bottom end of the capsule was accidentally contacted and scarred slightly by the lathe tool.

Next, sodium hydroxide solution was used to completely expose about 6.4 mm ($\frac{1}{4}$ in.) of each end of the stainless steel pin, the length of which was then measured carefully while still embedded in the remainder of its outer jacket. At this point a circumferential crack was observed approximately 3.2 mm ($\frac{1}{8}$ in.) from the end of the pin which had been touched by the lathe tool. This end had been at the bottom of the assembly as it is shown in Figure 3. After the length measurements had been made, the remainder of the aluminum jacket was dissolved in sodium hydroxide solution and the length of the capsule was again measured. No change was detected. The length data are given in Table I.

Table I

LENGTH MEASUREMENTS[a] OF STAINLESS STEEL CAPSULE AND URANIUM CORE

	Length	
	cm	in.
Capsule		
Length as fabricated	14.61	5.75
Length as jacketed with Al[b]	14.828	5.838
Length in jacket after irradiation	14.83	5.84
Length as stripped	14.83	5.84
Core		
Length of unencapsulated wire	12.7	5.0
Length as encapsulated in assembly[b]	12.802	5.040
Length after irradiation	Unknown	Unknown

(a) Measurements made in inches and converted to centimeters.

(b) Deduced from measurements of radiographic print reproduced in Figure 2. Measurements were normalized to 20.32-cm (8.000-in.) overall length of outer jacket.

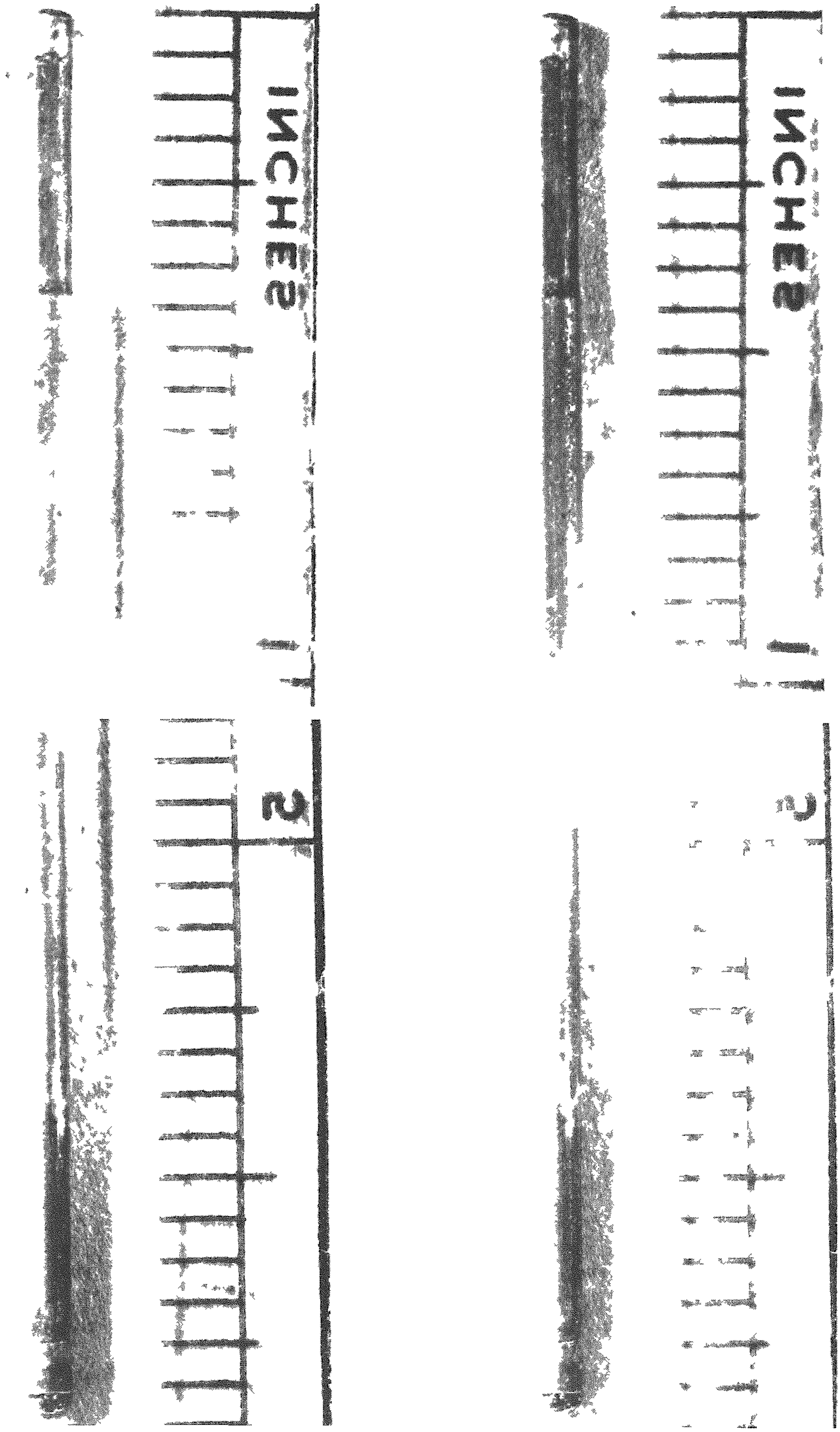
Figure 4 shows the full length of the fuel pin after the stripping operation. The diameter is visibly smaller than average near the ends, and these regions are shown at higher magnification in Figure 5. Their

14916 1X

Figure 4. Stainless Steel Fuel Capsule after Removal of Aluminum Jackets

a)

b)

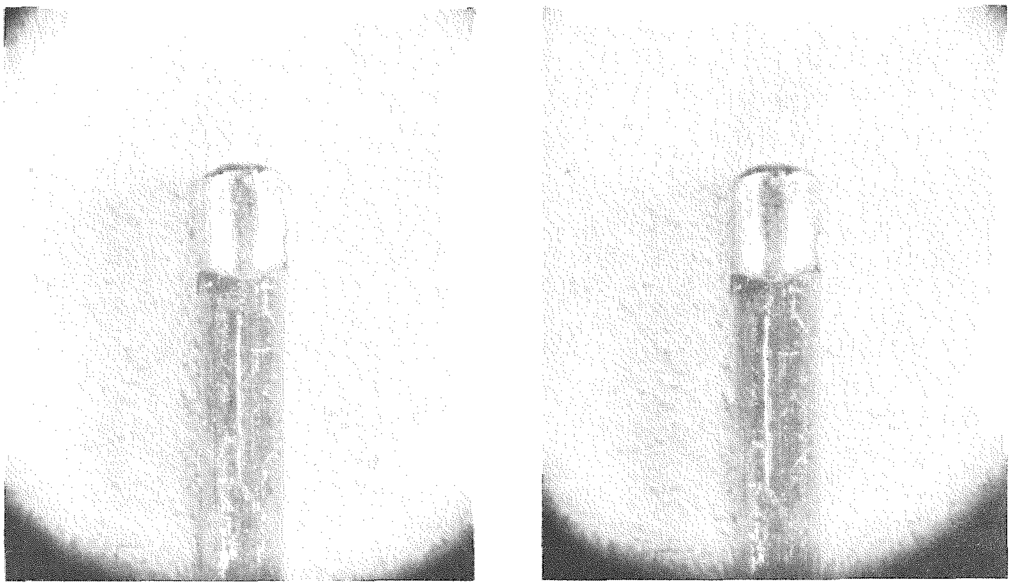


14915,14912

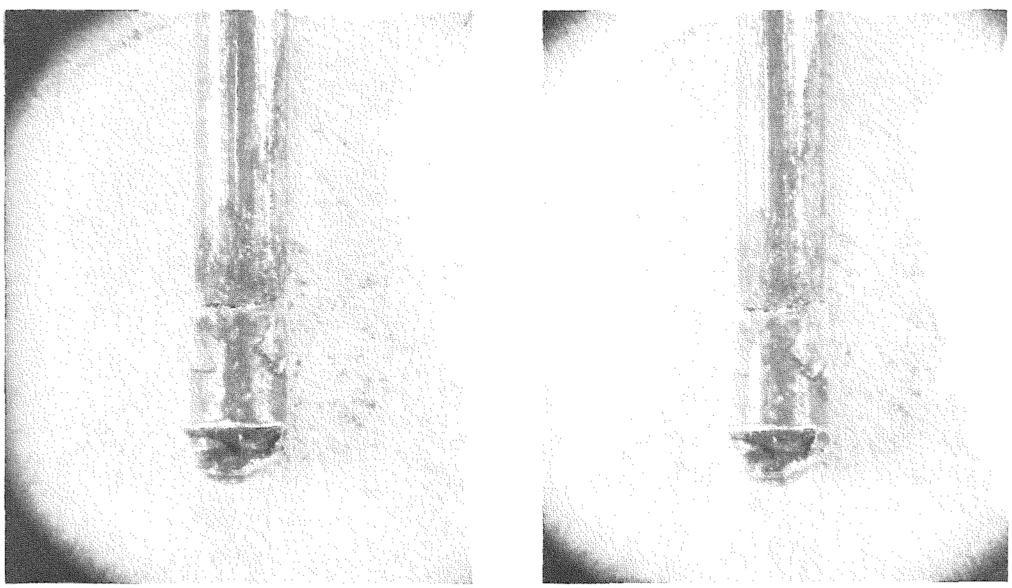
4X

Figure 5 Enlarged View of Ends, Showing Regions of Reduced Diameter a) Upper, b) Lower lengths are about equal and correspond exactly with the portions of the pin which originally contained none of the core material. They correlate also with radiographic measurements of core length which the LASL workers observed prior to irradiation. The necked-down portions are discernible in the print reproduced as Figure 2. The end welds are shown in good detail in Figure 6.

a)



b)



14917, 14918 8X

Figure 6. Enlarged View of End Welds, Showing Circumferential Crack in Cladding. Upper weld (a) was sound, although at first examination a crack was suspected.

Careful measurements of diameter at various points along the length of the pin were made with a 1-in. micrometer and also a Riehle gauge having a knife-edge anvil. The latter measurements show the reduction in diameter near the ends better than the former. All values are tabulated in Table II.

Table II

DIAMETER MEASUREMENTS OF STAINLESS STEEL CAPSULE

Diameter from Top		Diameter Measurements[a]					
		Before Irradiation[b]		After Irradiation			
				By 1-in. Micrometer		By Riehle Gauge	
cm	in.	mm	in.	mm	in.	mm	in.
At Top		1.524	0.060	1.524	0.060	1.524	0.060
0.32	1/8	1.397	0.055	1.516	0.0597	1.397	0.055
0.64	1/4	1.372	0.054	-	-	1.397	0.055
2.54	1	1.524	0.060	1.499	0.0590	1.524	0.060
5.08	2	1.524	0.060	1.524	0.0600	1.524	0.060
7.62	3	1.524	0.060	1.534	0.0604	1.524	0.060
10.16	4	1.524	0.060	1.526	0.0601	1.549	0.061
12.70	5	1.524	0.060	1.526	0.0601	1.524	0.060
14.48	5.7	1.473	0.058	1.499	0.0590	1.448	0.057
At Bottom		1.575	0.062	1.671	0.0658	1.651	0.065

(a)Measurements made in inches and converted to millimeters.

(b)Values obtained by scaling from Figure 2 and from radiographs in Reference (1), normalizing measurements to 1.524-mm (0.061-in.) diameter in middle of capsule.

The surface of the stainless steel cladding was smooth and metallic in appearance, with varying color from light straw to deeper tones. An idea of the extent of this variation may be gained from Figure 4. Deeper discoloration at the ends of the pin is doubtless due to the welding operation.

The maximum central temperature in the uranium had been computed as 450°C, which would place the surface temperature in the region of 400°C. Since an auxiliary diffusion test between well-cleaned 2S aluminum and Type 347 stainless steel had been run at ANL with negative results at 400°C, it was not surprising to find no areas on the pin which suggested anything more than good mechanical contact with the outer jacket. The same test had shown that solid reaction between the two metals would be appreciable at 550°C *

The bond between stainless steel shell and uranium core was examined metallographically in longitudinal and cross sections taken near the middle of the pin. The photomicrographs in Figure 7 are characteristic of the uniform contact between clad and core in this region. The uranium matrix structure proved to be completely insensitive to differentiation under polarized light. The uranium is at the bottom in each photograph.

All sectioning of the specimen for initial evaluation had to be done by a shearing operation. It was observed that cleavage of the uranium had a brittle character, without any sign of ductile flow. Because of the limitation in cutting method, it was decided not to try to explore the ends of the pin during the initial work, but to retain one piece to study when a better method of sectioning had been found. Accordingly, all pieces except the top end and a small cross section for burnup analysis were shipped to Los Alamos for recovery of fission products and for an independent check of burnup. In Table III are given the results of the two burnup analyses, together with a value computed from nominal flux. It is apparent that the level of burnup fell far short of the anticipated 7%.

Final examination of the end piece was done after an Elox spark cutting machine had been adapted for strain-free cutoff work in the ANL Metallography cave. A transverse cut was made through the void between core and end plug. A longitudinal metallographic section of the latter was made, which showed that the end weld was still in excellent condition and that it had suffered no tensile strain. The piece containing the core was mounted for cross-sectional polishing on the freshly cut face. In the mounting process the specimen was inclined approximately 20 to 25° from the vertical, and, therefore, as polishing progressed an oblique section resulted. It is shown in Figure 8 as it appeared just before the last chip of Bakelite in the end void was ground away. The wavy boundary between uranium core and Bakelite indicates that only moderate roughening in the end of the core had occurred. Overpolishing of the specimen and diamond indentations show the uranium to be harder than the stainless steel cladding. Quantitative hardness data were not obtained.

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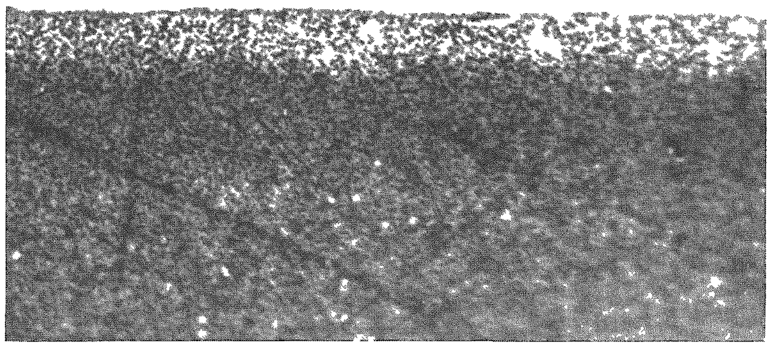
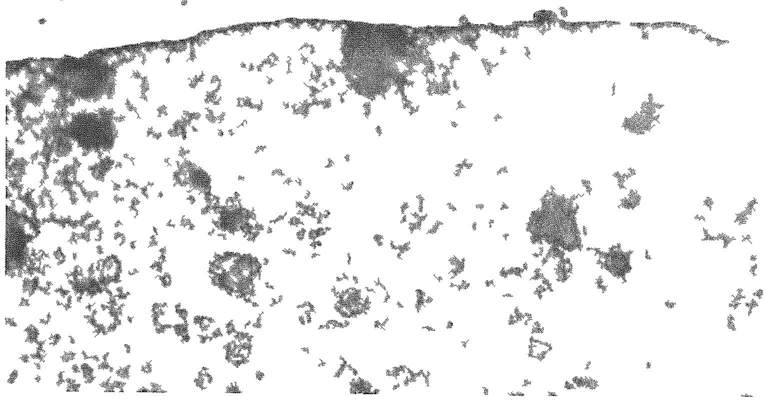
* 90 hr at 400°C - no reaction.
92 hr at 550°C - 0.051-0.102 mm (0.002-0.004 in.) solid reaction.
266 hr at 550°C - 1.524-1.575 mm (0.060-0.062 in.) solid reaction.

(a)



14806 Heat Tinted 250X

(b)



14805 H_3PO_4 Electrolytic Etch 1000X

Figure 7 Photomicrographs of Bond between Core and Cladding
a) Longitudinal Section, b) Cross Section

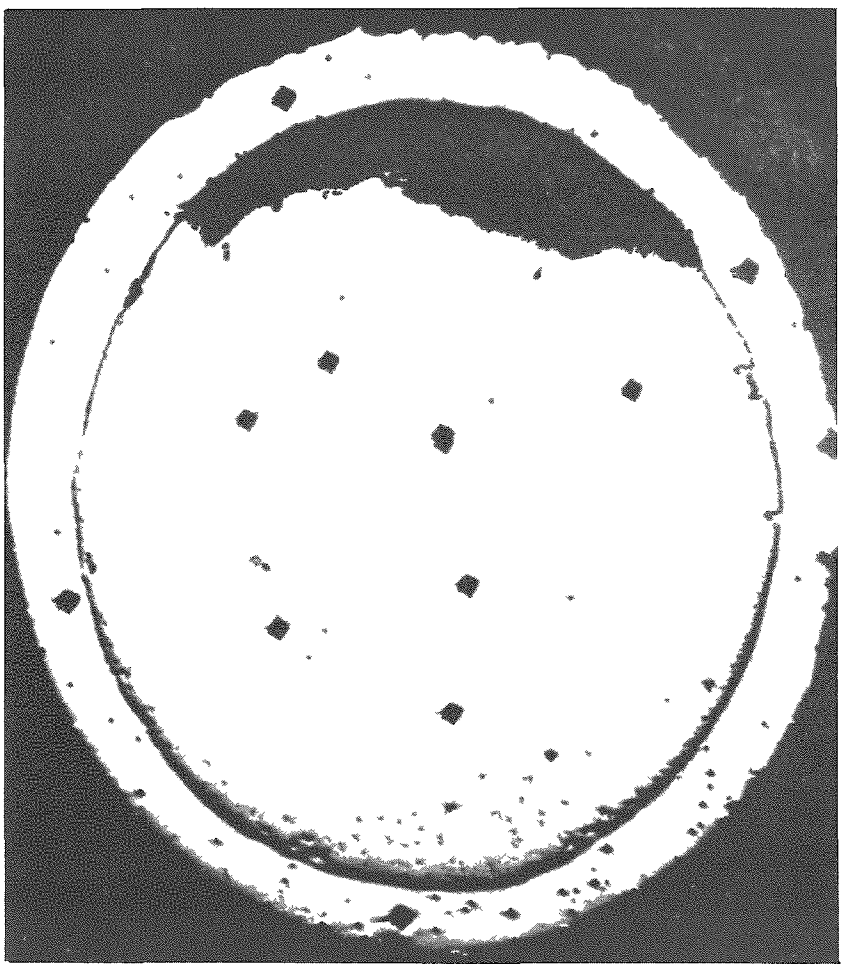
Table III

BURNUP ANALYSIS

Laboratory	Assay Method	Percent Total Atom Burnup	
		Average Away from Ends	Surface[a] Away from Ends
ANL	Cs^{137}	1.2	2.4
LASL	Ce^{144}	1.0 ± 0.05	2.0
ANL	Computed from flux[b]	1.5	3.0

[a] Computed from average burnup by methods described in Reference (4).

[b] Assuming 3.2×10^{19} nvt effective, 484 b.



25999 As Polished 75X

Figure 8 Oblique Section of RaLa Pin at End of Core. Overpolishing shows the core material to be harder than the cladding.

IV. DISCUSSION OF RESULTS

The original request for this work had expressed an interest in discovering:

1. whether or not the level of gamma heating attained by the freshly irradiated uranium pin when freed from its aluminum sleeves would be dangerously high;
2. whether or not rupture of the stainless steel sheath had occurred, and whether or not leakage of fission products was pronounced;
3. condition of bonds between uranium, stainless steel and aluminum;
4. what dimensional or other physical changes might have occurred in the uranium wire and its cladding;
5. how much of the uranium was actually burned by the irradiation.

Answers to most of these questions were found during the course of the initial examination. The gamma heating was not objectionable. Rupture and leakage had occurred, but the latter presented no special problem. Heat transfer conditions had remained optimum. Physical changes in the uranium had occurred, but the metal was yet metal and had been retained with reasonable success by its capsule. A burnup of between 1.0 and 1.5% had been achieved.

During the course of the initial examination of the RaLa pin, it was thought probable that the highly cold-worked core material had elongated and filled up the voids in the ends of the specimen. The overall length of the assembly was evidently greater than the nominal length of the stainless clad pin; moreover, failure of the jacket at the end weld was associated with a necking down of the adjacent portion. ANL work had shown[5] that cold-worked uranium specimens could be expected to double in length for burnups of 0.08-0.10 a/o when irradiated at moderate temperature, but without undue restraint.

Considerable interest was created, therefore, by the realization after further study that elongation of the RaLa pin core had been negligible. Counter to what had been anticipated, there was no detectable invasion of the end voids nor pulling of the core away from the cladding. As for the overall length changes and pinching down at the ends, they were found to correlate with LASL experience in fabrication. Also, the clad failure was in all respects similar to fabrication-induced breakage which had been observed in another prototype specimen,[1] except that X-ray examination before irradiation had not shown a crack in Prototype No. 2.

A rough tensile test was performed to simulate the restraint provided by the outer aluminum jacket. It was found that a mechanical assembly of aluminum swaged onto a smooth steel wire would provide a shear gripping force of approximately 28.1 kg/cm^2 (400 psi) of surface under optimum conditions. Reductions in area of the assembly of about 60% were required. Relaxation through springback of the aluminum sheath was found to occur at only slightly higher values. To overcome this restraint, tensile or compressive stresses of more than 7030.7 kg/cm^2 (100,000 psi) would have to be generated in a specimen having the RaLa pin diameter.

These results explain why the stainless steel capsule could not have elongated during irradiation. However, failure of the free ends of the uranium wire to grow into the end cavities is probably not due solely to this cause. At these points frictional stresses could readily be relaxed by a slight reduction in diameter of the uranium core. Here the significant locking mechanism may well be fission recoil bonding between core and clad before any appreciable growth stresses were developed.(6)

The absence of intermetallic compounds from the clad-core interface limits the irradiation temperature to less than 375°C.(7) In view of the fact that the total burnup was only 20% of that anticipated, it is likely that the interface temperature remained far below this limit during the entire course of the MTR exposure.

V CONCLUSIONS

The findings may be summarized as follows:

1. The RaLa assembly proved to be entirely adequate as an enclosure for fissile materials during irradiation The restraints imposed upon the inner pin completely suppressed a strong tendency of the fuel material to change its shape. Fission recoil bonding between pin clad and core and frictional contact between clad and outer jacket were postulated as the major factors in effecting this stability.

2. Defects, such as the cracked end weld on the inner pin and necking down of the cladding in the end region, have been attributed to the fabrication process. Elimination of voids between core and end plugs would make assembly procedure less critical. Excellent thermal contact between core, clad, and outer sheath was produced by the assembly technique. Added safety in the design could be obtained by beta heat treating the core wire after reduction to final size.

3. The uranium properties and structure, except for complete absence of radiation growth usually associated with heavily cold-worked material, were characteristic of metal irradiated to one or two percent total atom burnup in the low alpha-temperature range. The core material sheared without plastic deformation, was somewhat harder than the stainless steel cladding enclosing it, and was metallographically insensitive to polarized light.

ACKNOWLEDGMENTS

The authors are indebted to A. C Klank, ANL Metallurgy Division, for technical assistance in performing the examinations. Burnup analyses were made by E. H. Turk, ANL Chemical Engineering Division, and by M. A. Melnick and J. D. Knight, LASL.

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APPENDIX A

Fabrication Procedure

The following information was furnished by LASL.(1,3)

I. U^{235} wire

A. The U^{235} was vacuum cast into a 1.03-cm (0.406-in.)-diameter by 15.24-cm (6-in)-long bar. The bar was plated with 51μ (2 mils) of silver.

B. The plated bar was swaged to 3.175-mm (0 125-in)-diameter wire (90% reduction in area).

C The wire was warm (100°C) drawn to 1.27-mm (0.050-in.) diameter (84% additional reduction, total 98%).

D. A 12.7-cm (5-in.)-long section was cut from the wire The silver plating was removed. The 12.7-cm (5-in.) piece was electropolished and inserted into the stainless steel tubing

II Stainless Steel

A. A 20 3-cm (8-in.) length was cut of 1.626-mm (0.064-in.)-diameter by 0 152-mm (0.006-in)-wall Type 347 stainless steel tubing

B The tube section was swaged to a point diameter of 1.473 mm (0.058 in) for a distance of 5.08 mm (2 in.) at one end

C. Acetone was forced through the tube with a hypodermic syringe to degrease it.

D. The tube was etched and passivated by forcing the following liquids through it in the order listed: concentrated HCl, water, concentrated HNO_3, water, and ethyl alcohol. It was then dried in an oven for 5 min at 150°C.

E The freshly electropolished U^{235} wire was inserted in tube (I-D above).

F. The assembly was drawn to a 1.524-mm (0.060-in) outside diameter

G. The assembly was cut to a length of 14.6 cm (5.75 in.) and sealed as follows

1. A 4.76-mm (0.188-in.) length of stainless steel wire was inserted in each end of the tube.
2. The ends were welded by Heliarc.
3. The welds were swaged to 1.524-mm (0.060-in.) diameter and ground to give a length of 14.6 cm (5.75 in.).

III. Inner Aluminum

A. A 30.48-cm (12-in.) length of 6.3-mm (0.250-in.)-OD by 0.889-mm (0.035-in.)-wall 2S-1/4 H aluminum alloy tube was cut.

B. The inside of the aluminum tube was etched by means of an eye dropper with liquids as follows: with warm (75°C) 10% NaOH, rinsed with water, 50% of concentrated HNO_3 in water, water, alcohol. It was then oven dried.

C. A 1.111-cm (0.4375-in.) length of 3.175-mm (0.125-in.)-diameter 2S aluminum wire was driven into one end. That end was swaged in 5.55-mm (0.2187-in.) swaging dies for 7.62 cm (3 in.) to form a point. It was then drawn through a 5.56-mm (0.219-in.)-diameter die.

D. The plugged end was pointed for 10 cm (4 in.) to a 4.75-mm (0.187-in.) diameter. Then the front 5.08 cm (2 in.) was pointed to a 3.96-mm (0.156-in.) diameter.

E. The stainless capsule was inserted all the way into the 2S aluminum tube and then withdrawn 1.27 cm (0.5 in.). The assembly was drawn through 5.56, 4.75, 4.32, and 4.06-mm (0.219, 0.187, 0.170, and 0.160-in.) dies.

F. The stainless steel capsule was sealed in the aluminum tube as follows:

1. The point was cut off the aluminum tube and the ends of the stainless steel capsule were located. The tube was then cut to 17.145-cm (6.75-in.) length.
2. Then 6.35-mm (0.25-in.)-long plugs of 1.52-mm (0.060-in.)-diameter 2S aluminum wire were driven into the ends of the tube. The ends were welded by Heliarc, swaged to 4.06-mm (0.160-in.) diameter and ground to 17.145-cm (6.75-in.) length

G. The outside was then etched and cleaned as described in III-B.

IV. Outer Aluminum

A. A 15.875-cm (6.25-in.) length of 2.54-cm (1-in)-diameter 2S-0 bar stock was cut. A 1.111-cm (0.4375-in.) hole was drilled through the long axis. With the ends of the holes as centers, the outside was turned to a 2.075-cm (0.817-in.) diameter.

B. One end of the machined tube was pointed to a 1.667-cm (0.6562-in.) diameter.

C The inside of the tube was etched and cleaned as described in III-B.

D. The tube was drawn to 1.746 cm (0.6875-in.) and then to a 1.588-cm (0.6250-in.) outside diameter (41% reduction). It was then pointed.

E. The inner aluminum assembly was fully inserted and then with-drawn 1.27 cm (0.500 in.).

F. The assembly was drawn to 1.429 cm (0.5625 in.) and then to a 1.27-cm 0.500-in.) outside diameter (36% additional reduction, total 63%)

G. The assembly was machined and cut to length, as shown in Figure 1.

H. After machining flutes and tapping the ends, the finished rod was degreased. Then it was immersed in 16 N HNO_3 at room temperature and 25.4 cm (10 in.) Hg vacuum applied over the surface There was no gas evolution from the assembly.