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MANUFACTURE OF URANIUM-NIOBIUM FUEL RODS FOR IRRADIATION
IN THE MATERIALS TESTING REACTOR

by

W. B. Haynes

September 16, 1955

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METALLURGY AND CERAMICS

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Chief, Declassification Branch

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Abstract

Zircaloy-2 clad fuel rods whose fuel was U²³⁸-10 w/o Nb-5 w/o U²³⁵ were fabricated for testing in the Materials Testing Reactor. The fuel was duplex melted and given a homogenizing heat treatment prior to assembly into extrusion billets. The rods were prepared by co-extruding the fuel alloy in Zircaloy-2 cups, then drawing the rods to size.

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MANUFACTURE OF URANIUM-NIOBIUM FUEL RODS FOR IRRADIATION
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Introduction

Zircaloy-2 clad rods whose fuel alloy was U²³⁸-10 w/o Nb-5 w/o U²³⁵ were fabricated for testing in the Materials Testing Reactor. Clad specimens are to be tested in process water and specimens from which the cladding has been removed are to be tested in NaK filled capsules. These tests are to provide data on the effects of radiation on U-10 w/o Nb. The 5% enrichment was necessary so that higher burnups could be obtained than with natural material.

The rods were fabricated by co-extruding duplex melted fuel in Zircaloy-2 cups to 0.500-in. diameter. The extruded rods were finished by cold drawing to 0.304-in. diameter.

Induction Melting

One vacuum induction melt was made in a beryllia washed graphite crucible and cast into a 1 1/2-in. diam water cooled copper mold.

The 9464-gram melting charge consisted of 1/8-in. diam niobium rod chopped into 1/2-in. lengths, enriched uranium pellets 1/8-in. diam by 1/8 in. thick, and Fernald uranium Ingot P-1334. The Fernald ingot was prepared by vacuum induction melting uranium derbies in zirconia washed graphite molds and hot rolling into 0.3-in. thick strip.

The melt was superheated to 1700°C and held for 15 min at which time it was cooled to 1675°C so that it would not burn through the copper mold. The melting was accomplished without incident and a good casting was obtained.

Consumable Remelting

The induction cast ingot was drilled and tapped to be used as a consumable electrode. The casting was consumably remelted in a 20-in. atmosphere of 50-50 helium and argon. Melting time was 5 minutes. The maximum voltage reached during melting was 33 volts and the maximum amperage was 1400 amperes.

The top and bottom of this billet were remelted using a tungsten tipped electrode to eliminate any porosity.

Machining and Homogenization

The consumably remelted electrode was machined into billets for homogenization heat treatment. (See Fig. 1, the center portion was excess material). The material was readily machinable with tungsten carbide tipped tool bits.

Figs. 2 and 3 show the hardness values obtained from these billets prior to homogenization. The soft spots in the top and bottom of this ingot are often observed in consumably remelted billets. This is probably due to gigantic columnar grains in the center of the ingot. These soft spots disappear about 1/4 in. below the surface. None of this soft material is used in a fuel rod since most of it is machined off in machining the fuel extrusion component to size. Also approximately 1/4 in. of each end of

the fuel extrusion component is end loss in the extruded rod.

The billets were homogenized in a vacuum by the Metallurgy Sub-division. The homogenizing treatment for each billet is listed in Figs. 2 and 3. A black oxide scale, a few mils thick formed on the air cooled billet. The furnace cooled specimen had a dull gray metallic lustre after homogenization.

These homogenized billets were machined to 1.892-in. diam by 3.250 in. long and hardness readings made once more. It can be seen in Figs. 2 and 3 that there was considerable increase in hardness in both billets indicating that the homogenizing treatment had quite an effect.

Assembly

The fuel billets were washed in alcohol and assembled into Zircaloy-2 cups 1.893-in. inside diameter and 2.410-in. outside diameter. These were covered with Zircaloy-2 end caps 0.125 in. thick. Steel shells 0.062-in. wall thickness were used as jacketing material. Into the steel shells were placed copper nose plugs, the Zircaloy-2 assemblies and copper back-up plugs (Drawing 53-C-1035).

Steel end caps were fitted inside the steel shells. The whole assembly was welded closed in a welding box which had been evacuated then filled with helium.

Extrusion

The extrusion assemblies were heated in a chloride salt bath for 15 min at 1625°F. They were extruded through a 0.512-in. diam chromium plated die and had excellent surface finish. Extrusion data are tabulated

below. It can be seen from the examination of Figs. 7 and 11 that the fuel is quite irregular inside the Zircaloy-2. This indicates that 1625°F is probably too high a temperature for the co-extrusion of this material.

<u>Rod No.</u>	<u>Fuel</u>	<u>Load Start</u>	<u>Load Running</u>	<u>Load Time</u>	<u>Ram Speed</u>	<u>K</u>
Mo3c4-2	034B	360 tons	290 tons	9 sec	60	41,200
Mo3c4-1	034T	370 tons	280 tons	9 sec	30	42,300

Lubricant - "Led-Plate" Ram Speed - in. per min Container - 750°F

Drawing

After extrusion the rods were pickled in dilute HCl to remove the steel jacket which revealed the Zircaloy-2 cladding to be smooth and bright. They were heated for 20 min in air at 1150°F to oxidize the Zircaloy-2 so that the drawing lubricant of molybdenum disulfide in lacquer would not be stripped from the rods by the drawing dies.

Drawing was accomplished in 3 passes. Drawing data are tabulated below:

<u>Die Diameter</u>	<u>Area Reduction</u>	<u>Tonnage Required</u>
0.437 in.	23.6%	6 1/2
0.375 in.	26.7%	6
0.304 in.	34.3%	4 1/2

After drawing the rods were sand blasted to remove the oxide scale and lubricant. Then they were bright etched in a solution of 35 v/o HNO₃-5 v/o HF-60 v/o H₂O. Excellent rod surfaces, free of striations, pits, and imperfections, were obtained.

Evaluation

Chemical analyses and isotopic assays were made on chips obtained from the top, bottom, and center of the consumable remelt. Results are tabulated below:

	<u>U²³⁵</u>	<u>Nb</u>	<u>C</u>
Top	4.89	10.1	0.040
Center	4.95	10.1	0.034
Bottom	5.17	9.6	0.035

Diffusion zone measurements between the Zircaloy-2 cladding and the fuel were made by using a Tukon machine with a 4 mm objective. The results tabulated below represent the averages of four readings taken at 90° to each other.

<u>Rod No.</u>	<u>As-Extruded</u>	<u>As Drawn</u>
Mo3cl4-1	0.000064 in.	0.000037 in.
Mo3cl4-2	0.000063 in.	0.000031 in.

Photomicrographs of this diffusion zone in the as-extruded condition may be seen in Figs. 8 and 12. The diffusion zone after extrusion may be seen in Figs. 10 and 14. All of these photomicrographs are at 1000X. In each case the Zircaloy-2 cladding is on the bottom and the fuel is on the top.

Photomicrographs showing the fuel-cladding configuration at 3 diameters may be seen in Figs. 7 and 11. The fuel cladding configuration after drawing may be seen at 7 diameters in Figs. 9 and 13. Fig. 11 also shows the steel jacket around the Zircaloy-2 cladding after extrusion;

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this does not appear in Fig. 7. Fig. 5 is a graph showing the variation of the fuel diameter along the rod length in Rod Mo3cl-1 and Fig. 6 shows diameter variation in Rod Mo3cl-2. These charts were prepared by radiographing the rods from two angles 90° apart and plotting the rod diameter against the fuel length. Only sections of the rod in which the fuel diameter lay between 0.230 in. to 0.250 in. were used in the preparation of samples.

Sample Preparation

The rods were machined into the various samples as illustrated in Fig. 4. From Rod Mo3cl-1, 42 A specimens, 3 C specimens, and 2 D specimens were obtained. Rod Mo3cl-2 yielded 4 A specimens, 52 B specimens, 3 C specimens, and 1 D specimen.

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SECTIONING OF THE CONSUMABLY REMELTED INGOT FOR
HOMOGENIZATION.

FIG. 1

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FIG. 2

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FIG. 3

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"A" SPECIMEN
CLAD SPECIMEN

"B" SPECIMEN
BARE SPECIMEN

SPECIMEN "C"
CORROSION SPECIMEN

SPECIMEN "D"
METALLOGRAPHIC SPECIMEN

FIGURE 4

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FIG. 5

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FIG. 6

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Fig. 7

Rod Mo3c4-1, as extruded from 2.6-in. diam to 0.5-in. diam
Magnification 3X

Fig. 8

Rod Mo3c4-1, as extruded from 2.6-in. diam to 0.5-in. diam. Zircaloy-2
cladding on bottom, fuel on top. Magnification 1000X, bright field
illumination, cathodic vacuum etched.

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Fig. 9

Rod Mo3c4-1, as drawn from 0.5-in. diam to 0.304-in. diam. Magnification 7X.

Fig. 10

Rod Mo3c4-1, as drawn from 0.5-in. diam to 0.304-in. diam. Zircaloy-2 cladding on bottom, fuel on top. Magnification 1000X, bright field illumination, cathodic vacuum etched.

Fig. 11

Rod Mo3c4-2, as extruded from 2.6-in. diam to 0.5-in. diam. Fig. shows steel jacketing on outside, Zircaloy-2 cladding, fuel. Magnification 3X.

Fig. 12

Rod Mo3c4-2, as extruded from 2.6-in. diam to 0.5-in. diam. Zircaloy-2 cladding on bottom, fuel on top. Magnification 1000X, bright field illumination, cathodic vacuum etched.

Fig. 13

Rod Mo3cl₄-2, as drawn from 0.5-in. diam to 0.304-in. diam. Magnification 7X.

Fig. 14

Rod Mo3cl₄-2, as drawn from 0.5-in. diam to 0.304-in. diam, Zircaloy-2 cladding on bottom, fuel on top. Magnification 1000X, bright field illumination, cathodic vacuum etched.