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PROCUREMENT AND PROCESSING OF EBR-I CORE IV URANIUM AND HARDWARE

by

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and C. H. Bean

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PROCUREMENT AND PROCESSING OF EBR-I CORE IV URANIUM AND HARDWARE

by

R. A. Beatty, F. D. McCuaig,
and C. H. Bean

ABSTRACT

Jacket assemblies plus other components of zirconium alloys, stainless steel, and uranium were manufactured for the plutonium fuel rods, depleted-uranium blanket rods, and thermocouple rods for the Core IV loading of EBR-I. Considerable effort was expended to produce small diameter (0.760- and 0.203-cm-OD) Zircaloy-2 tubing and to inspect this tubing adequately for defects. Unidentified random imperfections were detected in Zircaloy-2 jacket tubing inspected by a dual-frequency eddy-current test system. A major cause for rejecting assemblies in X-ray inspection was a decrease in wall thickness resulting from spacer-rib welding and lower-end closure welding. It is recommended that future fuel elements be designed to take advantage of spacer-rib welding techniques that were developed to give a small weld-affected zone. Advanced techniques applied to the fabrication of small-diameter, Zircaloy-2 fuel-jacket and thermocouple tubing are described.

I. INTRODUCTION

This report covers the work of the Foundry and Fabrication Group of the Metallurgy Division associated with the procurement, fabrication, and inspection of hardware and uranium components required for Core IV of EBR-I. This loading required 420 fuel rods, 120 blanket rods, 10 fuel thermocouple rods, and five blanket thermocouple rods. Materials used in rod components included zirconium, Zircaloy-2, Zircaloy-3, Type 304 stainless steel, Inconel X, and depleted uranium.

Zirconium and zirconium-alloy hardware required for all four rod types consisted of the fuel or blanket jacket, NaK-level spacer, spacer-rib wires, rod tip, connector, and filler plug. The thermocouple rods required a Zircaloy-2 thermocouple-well and brazing sleeve. Stainless-steel and Inconel X hardware included the extension rods, rod inserts, and compression springs. Uranium components included upper and lower blanket slugs for the fuel rods and upper, center, and lower slugs for the blanket

rods. Slugs for the thermocouple rods had a drilled hole along the longitudinal axis to allow the thermocouple well to be centrally seated to the prescribed depth.

The development and manufacture of plutonium-alloy fuel slugs and final loading and assembly of the various types of rods were conducted in the Fuel Fabrication Facility by the Plutonium Fabrication Group of the Metallurgy Division.¹

II. DESCRIPTION OF HARDWARE AND URANIUM COMPONENTS

A. Fuel- and Blanket-rod Hardware

The EBR-I Core IV fuel and blanket rod is shown in Fig. 1. The center section of the fuel rod has four plutonium-alloy slugs. These are replaced in the blanket rod by two longer, depleted-uranium, blanket slugs. The Zircaloy-2 jacket tube (Item 1) is the same for both the fuel and blanket rod. The jacket tube has a 0.653-cm (0.257 in.) ID and a wall thickness of 0.053 cm (0.021 in.) and is 58.7 cm (23.13 in.) long. Spot-welded to the outside of the jacket tube are three spacer ribs 0.122 cm (0.048 in.) high and 0.140 cm (0.055 in.) wide. These longitudinal ribs are spaced 120° apart. The jacket tube is closed at its lower end by a Zircaloy-2 tip (Item 2), welded to the jacket tube. The jacket tube, with its tip and spacer ribs welded in place, is referred to (throughout this report) as a "jacket assembly." The Type 304 SS extension rod (Item 3) is threaded onto the top end of the fuel or blanket rod. Each fuel and blanket jacket assembly was delivered for loading complete with connector, NaK level spacer, spring, and filler plugs (Items 4, 5, 6, and 7, respectively).

B. Thermocouple-rod Hardware

The thermocouple rod is shown in Fig. 2. It differs from the fuel and blanket rods in having a thermocouple well extending down the rod axis. The internal components (fuel and/or blanket slugs) were drilled with a 0.218-cm (0.086-in.) central hole to allow the passage of the well to the proper depth.

The thermocouple-well subassembly (Item 7 in Fig. 2) consists of a Zircaloy-2 tube with a Zircaloy-2 sleeve (Item 8) brazed in position. The thermocouple tube has 0.203-cm (0.080-in.) OD x 0.127-cm (0.050-in.) ID and is 48.1 cm (18.94 in.) long. Tubular extension rods (Item 3) were provided for the thermocouple rods to accommodate the long thermocouple leads. The Type 304 stainless-steel insert (Item 9) helps guide thermocouples in and out of the well. Each jacket assembly delivered for use in a thermocouple rod included the connector, NaK level spacer, spring, and thermocouple-well subassembly.

Fig. 1. Fuel- and Blanket-rod Assembly

Fig. 2. Thermocouple-rod Assembly

C. Uranium Components

Depleted-uranium components are detailed in Fig. 3. Each fuel rod contains one upper blanket slug and one lower blanket slug (Items 8 and 9 in Fig. 1). The blanket rods contain one upper blanket slug, two center blanket slugs, and one lower blanket slug. The fuel and blanket thermocouple rods have a similar slug arrangement.

35709

Fig. 3. Depleted-uranium Blanket Slugs

III. PROCUREMENT AND INSPECTION OF HARDWARE COMPONENTS

A. Fuel- and Blanket-rod Jacket Tubing

The bulk of the fuel- and blanket-rod jacket tubing was procured on a conversion basis during 1960-1961 from a commercial supplier. A year later a small additional quantity was obtained on a nonconversion basis from another supplier to make up for jacket losses in spacer-rib-wire welding.

At the time of the initial procurement of Zircaloy-2 seamless tubing, the technology and the commercial practice were not advanced to the point where a comprehensive purchase specification could be applied. This was particularly true for tubing less than 0.762 cm (0.300 in.) in diameter. As long as tubing was being fabricated on a conversion basis with ANL-furnished ingot material, close liaison could be maintained with the supplier in an effort to produce an acceptable product. By coordinating the ANL evaluation and nondestructive testing with the production work, we eliminated many difficulties. Tubing material was accepted on the basis of nondestructive testing at ANL. Instructions were furnished with the purchase order for tubing to cover the manufacturing requirements. These instructions are included in Appendix A of this report.

Tubing purchased at a later date on a conversion basis was ordered in accordance with ANL Specification ZST-44201, Specification for Seamless Zirconium-alloy Tubing for Nuclear Reactor Service (Appendix B).

Tubing inspection and processing at ANL are summarized in Table I. Multiple lengths picked at random from each lot of jacket tubing were inspected for conformance to the visual and dimensional requirements. All tubing was within dimensional tolerance, except about ten tubes from the initial lot, which were undersize on the ID.

TABLE I. Inspection and Processing of Jacket Tubing at ANL

-
1. Inspect visually for surface quality.
 2. Inspect visually for straightness.
 3. Perform dimensional inspection.
 4. Hand-straighten to within 0.102 cm (0.040 in.) TIR in 61 cm (2 ft).
 5. Perform eddy-current inspection for cracks in tube wall.
 6. Assign identifying code number to tube.
 7. Perform eddy-current inspection for plate-type defects in tube wall.
 8. Salvage good material from between defective areas in tubes.
 9. Machine tubes to length.
 10. Acid-pickle and rinse.
 11. Weld rod tips to tubes.
 12. Weld spacer-rib wires to tubes.
-

After degreasing and arbor-straightening, all tubing was inspected for wall defects by using a dual-frequency, eddy-current, test system² as well as micro and macro techniques. Only about 1.5% of the second lot of tubing was rejected for structural defects; approximately 25% of the first lot of tubing was rejected.

Two types of defects became apparent in the eddy-current testing and were the cause of most of the rejected material: (1) radial cracks and (2) random unidentified imperfections. (Random imperfections were found parallel to the tube circumference as well as radially and at all angles in between these two extremes.) The two defect types are illustrated in Figs. 4 and 5.

The unidentified defects shown in Fig. 5 do not appear to be open cracks when examined metallographically, although they do produce a signal response equivalent to crack-type defects during eddy-current inspection. Some correlation was suspected between the formation of these defects and the method of fabrication, although we never definitely determined what these defects were. There is even some question as to whether they were deleterious.

The eddy-current equipment used is shown in Fig. 6. (The electronic controls are not shown.) An electronic probe and a pen recorder were mounted on the carriage of a lathe capable of handling tubes up to 200 cm (79 in.) in length. The tube was clamped at each end and rotated at about 187 rpm. The probe traversed the tube at 0.159-cm (0.062-in.) pitch

Micro 30499

BF

100X

Fig. 4. Radial Crack in Zircaloy-2 Jacket Tube

Micro 30500

BF

90X

(a)

Defect in Tube Wall as Detected
by Eddy-current Inspection

Micro 30501

BF

500X

(b)

Enlarged View of Defect Shown
in Fig. 5a

Fig. 5. Unidentified Defect in Zircaloy-2 Tubing

106-5236

Fig. 6. Dual-frequency Eddy-current Test Equipment for Tubing Inspection

in one pass from end to end. Approximately 6.66 cm (2.265 in.) at each end of the tube was not inspected because of the end clamps. These ends were later removed when the tubing was cut to finished lengths.

The selection of a standard helped maintain uniform and consistent results. The standard contained a natural defect and served as a reference for rejecting material having radial cracks deeper than 0.013 cm (0.005 in.).

Eddy-current pen traces provided a permanent record of each defect indication. When the tube was removed from the lathe, an identifying code number was written on the trace and indelibly on the tube to permit later matching of tubes with their respective traces. It was thereby possible to salvage usable jacket lengths from between rejected segments.

Little time was available for even a cursory study of the effects of radial cracks and other defects upon tube integrity. Because of a shortage of material, it was arbitrarily decided that 0.013-cm-deep radial cracks 0.157 cm long would not be cause for rejection. A less generous cut-off point would not have provided sufficient material. The nominal wall thickness was 0.053 cm (0.021 in.), and a 0.013-cm radial crack represents a defect penetrating 25% of the wall.

The unidentified defects shown in Fig. 5 could not be evaluated. Pressure testing and thermal cycling between room temperature and 450 to 550°C indicated no deleterious effect of this defect upon tubing integrity. While it was generally agreed that the best policy would be to use no material having these defects, this policy was not possible because of scheduling requirements. Thus, tubing containing random unidentified defects that produced indications by eddy-current testing that were smaller than 0.0076-cm-deep cracks 0.157 cm long were arbitrarily accepted and designated for use only in the lower-temperature sections of the reactor core.

The first lot of tubing fabricated was divided into three groups after eddy-current inspection. One group contained no defects greater than 0.013 cm deep and none of the unidentified defects, a second group contained no radial cracks greater than 0.013 cm deep along with unidentified random-type defects, and a third group contained unacceptable radial cracks. The first group, classified as fuel quality, was used for jacketing fuel rods and some blanket rods. The second group was used in blanket rods only, and the third group was rejected. None of the unidentified random-type defects was detected in any of the second lot of tubing fabricated.

B. Instrument Tubing

During 1959 and 1960, orders were placed with several commercial tubing suppliers for seamless, small-diameter, Zircaloy-2, instrument tubing. Tubing of 0.203-cm (0.080-in.) OD x 0.038-cm (0.015-in.) wall was needed for thermocouple instrumentation. This tubing was generally referred to as thermocouple tubing.

Only 22 m of thermocouple tubing was received at ANL by late 1960. All of the material received contained severe inner-surface cracks, folds, and wall-thickness irregularities as illustrated in Fig. 7. The remaining material on order was cancelled, and fabrication of the tubing was undertaken at ANL.

Fig. 7. ID Defects in Zircaloy-2 Instrument Tubing

C. Spacer-rib Wire

A total of 1370 m of 0.157-cm (0.062-in.)-diam Zircaloy-2 spacer-rib-wire stock was procured from a commercial source. Metallographic

inspection of the wire at ANL indicated that it contained essentially continuous centerline voids throughout. No matter where the section was taken, a centerline void of 0.013 cm (0.005 in.) or more was found. Eddy-current inspection of 805 m of the wire also revealed no sections having voids smaller than 0.013 cm (0.005 in.). Because of the pressure of production, all the rib wire was accepted even though tests revealed that this wire would be difficult to weld to the Zircaloy-2 jacket tubing without having many defective welds.

D. Springs

The Inconel-X compression springs (Item 6 of Figs. 1 and 2) were commercially procured. Number 1 temper Inconel-X wire 0.177 cm (0.046 in.) in diameter was used to fabricate the springs. Finished springs were precipitation-hardened at ANL (16 hr at 1300°F), followed by a wet abrasive blast and acid pickle to remove heat-treating scale.

E. Extension Rods and Rod Inserts

Type 304 stainless-steel extension rods were purchased as finished. Stainless-steel extension-rod inserts (Item 9 in Fig. 2) were machined at ANL. After receipt and inspection by ANL, 705 fuel and blanket extension rods and 20 thermocouple extension rods, complete with rod inserts, were shipped directly to the EBR-I facility in Idaho.

IV. ANL FABRICATION AND INSPECTION OF HARDWARE COMPONENTS

A. Fuel- and Blanket-rod Jacket Tubing

Because of delays in the fabrication and delivery of the required amounts of acceptable fuel- and blanket-rod jacket tubing from commercial suppliers, some of this material was fabricated at ANL as a backup effort. Some of the fabrication techniques developed for this tubing were later applied to the fabrication of smaller-diameter Zircaloy-2 thermocouple tubing.

Forged Zircaloy-2 ingot stock, 2.86 cm (1.125 in.) in diameter, was machined into extrusion hollows. These were jacketed in 0.08-cm-thick copper cans and extruded on a 100-ton vertical hydraulic press at 650°C at an extrusion ratio of 11.5:1 and a ram speed of 20.3 cm/sec. The extruded tube blanks were 76 to 91 cm long by 1.03-cm OD with a 0.109-cm wall. These blanks were sound with fairly good wall concentricity (thickness variations less than $\pm 10\%$). The material was comparable to the quality of commercially extruded material at this stage. Because of the smaller diameter of the tube blanks, we reasoned that the lower total reduction required to finish tubing to size would result in fewer structural defects.

The copper jacket was removed from the inner surface by selective acid-pickling. A 70-30 brass rod, of approximately 0.800-cm OD, was inserted in the tube blank for ductile-core drawing.³ With the core in place, the blank was drawn to 0.879-cm (0.346-in.) OD. The drawn tubes were annealed 30 min at 600°C in air, after which the ductile core was stretched in tension to reduce its diameter for removal from the tube. A hardened tool-steel mandrel was then inserted, and the tube was finish-sized to 0.766-cm (0.305-in.) OD. After this reduction, the mandrel was removed and the tube was pickled in a 50-50 nitric acid-water solution to remove the copper jacket.

At this point the Zircaloy-2 tube OD and ID were within the final dimensional tolerances; however, the tube exterior finish, while acceptable, was uniformly covered with very shallow striations that originated during the extrusion operation. These striations were less pronounced on the inner surface.

To improve the outer surface of finished tubing, the fabrication technique was altered slightly. The copper was removed from the outer surface after the anneal following the 0.879-cm pass instead of at 0.766-cm diam. The tube was then drawn in two passes over a tool-steel mandrel to the finished size. The diffused-copper layer that remained on the surface after the unreacted copper jacket was removed from the outer surface was found to be an extremely effective lubricant. When this layer, which was identified by X-ray diffraction as a zirconium-copper compound, was not etched away, a mirror-like as-drawn outer surface was obtainable. Flash-pickling in a 35 v/o HNO_3 -5 v/o HF solution removed any remaining diffused copper following the final drawing operation. The conditions were not established to make it possible to reproduce this surface finish in all the jacket tubing fabricated at ANL. However, as evidenced by this experience, the concept of diffusing metallic lubricants into surfaces for a perfect bond between lubricant and work pieces holds considerable promise in solving more difficult metal-forming lubrication problems.

Sufficient defect-free tubing was fabricated at ANL to serve as a backup for 90 fuel-jacket tubes.

B. Instrument Tubing

Since efforts to procure 0.203-cm (0.080-in.)-OD Zircaloy tubing commercially were unsuccessful, the required tubing was fabricated at ANL by ductile-core drawing.³ Starting material was 4.12-cm (1.625-in.)-diam bar, commercially forged from atmosphere-melted Zircaloy-2 ingot stock. Extrusion billets were machined, jacketed in copper, and extruded at 725°C at an 8.2:1 extrusion ratio with available tooling developed for other extrusion work. The resulting tube blanks were approximately

60.9 cm (24 in.) long with a 1.05-cm (0.413-in.) OD and 0.165-cm (0.065-in.) wall (a D/t ratio of 6.4:1). The quality of extruded tube blanks was comparable to that produced for fuel-jacket tubing.

The tube blanks were prepared for drawing with a ductile-brass core of 0.682-cm (0.269-in.) OD, as described for jacket tubing above. Drawing proceeded to 0.381-cm (0.150-in.) diam with intermittent 600°C air anneals. The composite tubing was cut to 38.1-cm (15-in.) lengths and annealed. The brass cores were then stretched and removed. The tubing was drawn to 0.211-cm (0.083-in.) diam over three successive nondeformable mandrels to iron out inner-surface folding and minimize inner-surface radial cracks in the finished tube. At this point, all remaining copper was pickled off the outer surface of the tube, and the tubes received a final 675°C vacuum anneal. The final step was bright-etching to the 0.203-cm (0.080-in.) OD by the procedure outlined in Table II.

TABLE II. Procedure Used to Clean Zirconium and Zirconium Alloys

1. Degrease.
2. Bright-pickle in 40°C 35-5 v/o HNO₃-HF solution.
3. Rinse in tap water.
4. Rinse in hot, distilled water, afterward by ethyl alcohol.
5. Air-dry.

Thermocouple tubing was inspected with encircling-coil eddy-current equipment.² The best 19 thermocouple tubes were approved for reactor use. Transverse sections through 0.203-cm (0.080-in.)-OD instrument tubing, prepared both by die-sinking and by ductile-core drawing, are shown in Fig. 8. A decided improvement can be seen in the quality of tubing inner surfaces as a result of the ductile-core technique. One of the 19 tubes had inner-surface radial cracks approximately 0.013 cm (0.005 in.) deep. Ten of the tubes had radial cracks 0.0076 cm (0.003 in.) deep, and the balance contained cracks 0.005 cm (0.002 in.) deep or less. The walls of the finished tubes were from 0.034 to 0.037 cm thick.

C. Spacer-rib Wire

In addition to the 0.157-cm (0.062-in.)-diam, Zircaloy-2, spacer-rib wire that was fabricated commercially, separate lots of Zircaloy-3 and unalloyed zirconium wire were fabricated from available materials at ANL. This wire fabrication was intended to supply samples for welding development, as a backup effort for commercial wire, and in an effort to overcome the problem of continuous centerline voids.

The Zircaloy-3 wire was produced from 4.12-cm-diam forged bar stock. Bars were heated in air to 850°C and rolled to 0.82-cm (0.323-in.) diam

Macro 33111

20X

Fig. 8. Comparison of Transverse Sections of 0.203-cm (0.080-in.)-OD x 0.127-cm (0.050-in.)-ID Zircaloy-2 Tubing Finished by Conventional Die Sinking (upper photos), with Tubing Finished by Ductile-core Drawing (lower photos)

jacket assemblies needed to replace rejected assemblies. Vacuum arc-melted crystal-bar zirconium ingot and pure crystal-bar zirconium were used for the starting material. The zirconium was cold-rolled from 1.58- to 0.64-cm diam and swaged to 0.475-cm diam.

The 0.475-cm rods were jacketed in 0.079-cm (0.031-in.)-thick copper tubes and drawn a total of 89% reduction in area. Drawing reductions of 10-15% per pass were used and intermediate, 600°C, air anneals were performed at 40% intervals and after the last pass. The copper was removed by nitric acid pickling, and the wire was sized to 0.158-cm (0.063-in.) diam by acid etching, using procedures outlined in Table II.

Finished zirconium wire in lengths of 2 to 3 m was inspected by eddy-current equipment² and found to be 100% free of voids. Less than 1 m was rejected for lap-type cracks. The overall yield from machined bar to finished wire was greater than 40%. Final processing of all wire used in the Core IV loading included straightening, acid-cleaning (Table II), and cutting to lengths suitable for welding.

*Aldraw J-2 Lubricant, manufactured by Hangsterfer Laboratories, Inc., Mantua, New Jersey.

at 6 to 16% reduction in area per pass. Hot-rolled bars were centerless-ground to 0.070-cm (0.275-in.) diam to remove surface laps, seams, and to contaminated metal. This was followed by cold-swaging 54% reduction in area to 0.475 cm (0.187 in.). Annealed wire was drawn to size at 3 to 15% reduction in area per pass. A phosphate chemical coating on the wire surface helped retain the drawing lubricant* and prevented seizing in the die. Wire was given an intermediate vacuum-anneal at 850°C after each 50-60% reduction in area and after the final drawing operation.

Eddy-current inspections performed on 600 m (1968 ft) of Zircaloy-3 wire showed more or less continuous centerline voids in the wire. This wire supplemented the commercially produced Zircaloy-2 wire, and the quality of both were much alike.

A relatively small quantity of unalloyed zirconium wire, 109 m (358 ft), was fabricated for additional jacket assemblies needed to replace rejected assemblies. Vacuum arc-melted crystal-bar zirconium ingot and pure crystal-bar zirconium were used for the starting material. The zirconium was cold-rolled from 1.58- to 0.64-cm diam and swaged to 0.475-cm diam.

The 0.475-cm rods were jacketed in 0.079-cm (0.031-in.)-thick copper tubes and drawn a total of 89% reduction in area. Drawing reductions of 10-15% per pass were used and intermediate, 600°C, air anneals were performed at 40% intervals and after the last pass. The copper was removed by nitric acid pickling, and the wire was sized to 0.158-cm (0.063-in.) diam by acid etching, using procedures outlined in Table II.

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D. NaK Level Spacer

The NaK level spacer (Item 5 in Figs. 1 and 2) separates the compression spring and the upper uranium slug. Length was the only critical dimension, and considerable leeway was allowed in material quality and in the ID and wall dimensions. Zircaloy-2 tubing of 0.952-cm OD by 0.063-cm wall, was swaged to 0.64-cm OD and 0.091-cm wall for most of the required spacer stock. The balance was converted by drawing the 0.952-cm tubing to 0.64-cm OD by 0.071-cm wall.

The reduced tubes were vacuum-annealed for 1 hr at 850°C. Reductions were effected in both drawing and swaging without intermediate anneals. The spacer stock was acid-etched to the 0.63-cm (0.249-in.) specified OD (Table II) and machined.

E. Tips, Connectors, Filler Plugs, and Thermocouple-well Sleeves

Stock for tube-end closure, filler plug, and thermocouple sleeve components (see Figs. 1 and 2) was fabricated from the same Zircaloy-2 ingot used for thermocouple tubing. The forged bar was rolled at 850°C with reductions of 6 to 16% per pass to 0.88-cm (0.347-in.) diam. Rolled rod was annealed at 850°C.

After an 850°C anneal, the 0.88-cm (0.347-in.)-diam rod was straightened and centerless-ground to the final 0.76-cm (0.299-in.) diam required. After grinding, the rods were arbor-straightened in preparation for nondestructive testing and machining.

The 0.76-cm (0.299-in.) rods in 38- to 66-cm (15- to 26-in.) lengths were inspected for center bursts and rolling laps, 0.079 cm (0.031 in.) or more in any direction, with the use of ultrasonic through-transmission equipment.² Of approximately 162 m (370 ft) of rod inspected, only 3% was rejected. Rolling laps and seams not removed in grinding and end bursts were the only defects found.

V. HARDWARE COMPONENT ASSEMBLY AND INSPECTION

A. Jacket Assemblies

The jacket assembly (Item 1 in Figs. 1 and 2) consists of a jacket tube, a rod tip, and three spacer ribs. Jacket assemblies were interchangeable for all four types of EBR-I Core IV rods.

The jacket tubes were positioned in a semiautomatic welder with the rod tip for the bottom end closure in place. The closure weld was made under an argon gas cover by TIG welding with the tube rotated in a

glass lathe. The welding power supply was a 300-Amp d.c. rectifier welder operating at 26 Amp and 12 V. Power remained constant during the 360° weld cycle, after which a crater eliminator cut in. The work was rotated from 5 rpm at the start to 15 rpm at the completion of the weld. Speed variation during the 360° rotation was necessary to compensate for the heat buildup in the end plug. The automatic weld cycle included a 10-sec helium purge at 20 cu ft/hr before and after welding. The only manual operation performed was positioning the electrode, a critical operation in obtaining good-quality welds, as demonstrated in Fig. 9. A typical section through a rod tip-to-jacket tube weld is shown in Fig. 10.

Rib wires were attached to the tubes by a condenser-discharge-type spot-weld developed for rib welding in the EBR-I Core III loading.⁴ A Raytheon capacitor-discharge power supply of 400- μ F capacity was used to weld the three spacer wires to the Zircaloy-2 fuel tube. The welding parameters were 375 μ F with a line voltage of 104 V and 40-psi gun pressure. A helium gas cover of 10 cfh was maintained at all welds. The welding

Macro 30516

BF

3X

a) Rod Tip-to-Jacket Tube Weld Showing Weld Buildup and Sharp Corners Caused by Improper Positioning of Welding Electrode

Macro 30516

BF

3X

b) Smooth-tapered Rod Tip-to-Jacket Tube Weld Made with Properly Positioned Welding Electrode

Fig. 9. Sections through Rod Tip-to-Jacket Tube Welds

Micro 30829

BF

50X

Fig. 10. Section of a Typical Rod
Tip-to-Jacket Tube Weld

cycle was designed for a sequence of three welds, one on each wire. Figure 11 shows the tube and spacer-wire guide and the three electrode guns for sequence welding. Welds were made at 0.32-cm (0.125-in.) intervals along the tube length. Positioning of the work for each weld interval was automatic. The wire was held in contact with the jacket tube with 0.63-cm-diam copper electrodes specially contoured so as to exert a point contact between the wire and tube. A 0.63-cm-diam copper rod was used as a supporting mandrel and also served as a heat sink on the inside of the Zircaloy-2 tube. Two hundred twenty-eight welds were used to attach the three wires to each tube. A typical section through a fuel-jacket tube showing sound welds with zirconium spacer wires is shown in Fig. 12.

106-3510

Fig. 11. Portion of the Condenser-discharge Welding Equipment, Consisting of Jacket-tube and Rib-wire Guides and Three Air-driven Electrodes for Automatic Sequence Welding of Spacer-rib Wires to Fuel-jacket Tubes

Macro 32156

BF

12X

Fig. 12. Section of Typical Fuel-jacket Tube with Zirconium Spacer-rib Wires Welded to the Tube Exterior

The presence of central voids in Zircaloy-2 and Zircaloy-3 rib-wire stock varied the metal volume in the cross-sectional area of the wire. This in turn caused a variation in resistance to the weld current. As a result, it was impossible to obtain uniform quality in the wire-to-tube welds by using present welding parameters. Variations within each wire made it impossible to correct the weld settings to compensate for these changes. In some cases, the temperature of the wire and jacket interface was high enough for the metal to be molten. In many welds, molten metal was ejected from between the rib and the jacket and an elongated cavity was formed. Figure 13 illustrates this type of defect, which was always associated with porosity in the wire and with weld-metal expulsion.

The jacket assembly with welded tip and ribs was straightened to 0.22-cm (0.010-in.) maximum bow, 0.04-cm (0.020-in.) total runout, and the rib wires and rod tip were machined. Of 827 jacket assemblies delivered to the shop for final machining, 5.2% were scrapped for miscellaneous machining defects after postmachining inspection by shop personnel. The final operation before delivery of jacket assemblies for fuel loading was a helium mass-spectrometer leak check.

After the fuel-loading and NaK-filling operation had begun in the fuel assembly area, some weld-metal expulsion was discovered under several of the rib welds adjacent to the rod tip. Two loaded jacket assemblies developed NaK leaks through the tube wall in this weld area during the 440°C NaK bonding operation. As all jacket assemblies delivered for loading had passed a helium leak inspection, it is probable that these leaks occurred at weak points under the spacer rib that failed when the assembly was heated to promote NaK bonding.

Metallographic inspection of the leaking rib welds showed cracks at the base of cavities at the tip end of the jacket tube. These cracks occurred only where the terminal rib weld covered the area of the tube wall, which had been thinned to 0.046 cm by a reaming operation designed to provide a tight fit for the rod tip. Serious cavities at the tip end were eliminated in the jacket assemblies by shortening the ribs enough to eliminate the weld over the reduced tube-wall thickness.

All jacket assemblies were X-ray-inspected for jacket thinning due to weld-metal expulsion. Special high-contrast techniques were used that were capable of detecting depressions as small as 0.008 cm in depth.¹ Forty jacket assemblies were rejected because of weld defects at the rod tip, which had thinned the jacket to less than 0.033 cm.

A total of 64 jacket welds, 7.4%, were rejected after visual, leak, and X-ray inspection.

Production data concerning all jacket assemblies produced are summarized in Table III.

Micro 33122

BF

48X

a) Longitudinal Section

Micro 33130

BF

48X

b) Transverse Section

Fig. 13. Longitudinal and Transverse Sections through a Typical Cavity
Formed by Expulsion of Weld Metal under Spacer-rib Wires
Welded to Zircaloy-2 Jacket Tubing

TABLE III. EBR-I Core IV Jacket Assembly Production Data

Jacket Assembly No. (stamped on tip)	Rod Type	Rib-wire Material	Total Loaded and Shipped to EBR-I
2 through 173	Blanket	Zircaloy-3	72
175 through 371	Fuel	Zircaloy-3	137
375 through 733	Fuel	Zircaloy-2	221
734 through 803	Blanket	Zircaloy-2	48
822 through 903	Fuel	Zirconium	62

B. Thermocouple Rods

The NaK-filling hole in thermocouple rods was closed by welding the thermocouple-well subassembly (Item 7 in Fig. 2) in place. This subassembly consisted of a Zircaloy-2 sleeve brazed to the 0.203-cm (0.080-in.)-OD Zircaloy-2 thermocouple tube.¹ Figure 14 is a cross section of the braze area in a typical, thermocouple-well subassembly.

Macro 34629 BF 4X
Fig. 14. Longitudinal Section through a Tube-to-Sleeve
Brazed Joint in a Zircaloy-2 Thermocouple-
well Subassembly

Nineteen thermocouple-well subassemblies were made. Inspection consisted of vacuum-leak testing the braze and end seal, X-ray inspection of the braze, and metallographic examination of control samples prepared concurrent with production brazing. Thirteen of 19 acceptable subassemblies were delivered for loading.

VI. URANIUM PROCESSING AND INSPECTION

Uranium used for EBR-I Core IV blanket slugs was 0.80- and 1.10-cm-diam rod stock, 23 to 28 cm long. The material was unalloyed, beta-treated, depleted uranium containing 0.20 ± 0.02 w/o U^{235} . This uranium was excess slug stock fabricated for the initial blanket loading of EBR-II.⁵ The rods were inspected by an ultrasonic through-transmission technique capable of determining grain size, heat treatment, and internal defects. No defects greater than 0.079 cm (0.031 in.)

in any direction were specified for finished material, and all material was found to be completely free of structural defects. Photomicrographs showing typical beta-treated structure and inclusion content of the uranium bar stock are presented in Fig. 15.

Micro 25816

PL

75X

a) Typical Structure of Beta-treated Unalloyed Uranium Blanket Material

Micro 25817

BF

100X

b) Typical Inclusion Content of Fabricated Uranium Blanket Material

Fig. 15. Metallographic Structure of Depleted-uranium Rod Used for EBR-I Core IV Blanket Slug Stock

Procedures followed in preparing uranium blanket slugs included rough machining by bar-turning to 0.60-cm (0.236-in.) diam, centerless-grinding to 0.596 cm (0.2345 in.), finishing 0.05 cm (0.020 in.) oversize in length, and upsetting projections for radial spacers at the slug ends. Uranium handling, described in Table IV, was carefully controlled to avoid pitting of machined surfaces during storage periods of 2 weeks or more between machining and upsetting.

TABLE IV. Procedure Used in Handling EBR-I Core IV Blanket Uranium Slugs during Processing

1. Machine slugs to size. Finished slugs were stored between operations in distilled H₂O with an additive* to prevent corrosion. (This solution prevented pitting for a period of 2 to 3 weeks.)
2. Clean for warm upset operation:
 - a. Submerge for 10 sec max in mildly concentrated detergent solution, followed by two tap-water rinses of 10 sec each.
 - b. Submerge for 10 sec in alcohol.
 - c. Remove excess alcohol, and air-dry.
 - d. Handle with clean cotton gloves.
3. Preheat in air furnace to 300°C.
4. Upset slugs in closed die, and air-cool.
5. Handling slugs with gloves, perform dimensional inspection (exposure of dry slugs to freely circulating room air was permitted for periods not exceeding 3 hr). Store inspected slugs in distilled H₂O with additive.
6. Clean prior to NaK loading. See Steps 2a through 2c.

*White and Bagley No. 1500, manufactured by E. C. Mullen Company, Chicago, Ill.

Hardened-steel punches and split dies were used for 300°C upsetting of the blanket uranium slug ends. Both ends of each 0.596-cm (0.2345-in.)-diam slug were upset to form four 0.015-cm (0.006-in.)-high by 0.79-cm (0.031-in.)-long x 0.079-cm (0.031-in.)-wide projections, as shown in Fig. 3. Slugs were clamped in the split die, and two steel punches were forced into the ends simultaneously with a hydraulic arbor press. As a result of this upsetting process, slugs were shortened about 0.05 cm (0.020 in.) and increased 0.0013 cm (0.0005 in.) in diameter. The three sets of dies, one for each slug size, were used to upset 718 upper-blanket slugs, 355 center-blanket slugs, and 739 lower-blanket slugs to satisfy uranium component requirements for both instrumented and noninstrumented rods. Finished slugs were inspected for conformity to dimensional tolerances. Oversize projections raised in the upsetting operation were machined to the specified diameter.

Deep-drilling of the uranium components required for 15 thermocouple rods was accomplished by electrical-discharge machining. With this method, a hole 19.7 cm (7.750 in.) long by 0.24 ± 0.02 cm (0.095 ± 0.008 in.) in diameter could be drilled at the rate of 8.9 cm (3.500 in.)/hr. The drilled hole in some of the slugs accepted for reactor use was larger in diameter and less uniform than originally requested, but the tolerances were relaxed because of the difficulties inherent in drilling deep holes. Slugs with deep-drilled holes were upset in the same manner as solid slugs.

VII. PROCESS ANALYSIS AND RECOMMENDATIONS

In general, the procedures followed in the procurement, fabrication, assembly, and inspection of hardware and uranium components required for Core IV of EBR-I were satisfactory. Equipment was simple and procedures straightforward. Many of the operations were automated, assuring reproducible results. Evaluation steps were planned so as not to depend unduly on the skill of equipment operators. Quality control was reduced to the measurement of specific quantities, eliminating errors in personal judgment.

A. Procurement

A gradual improvement in both the delivery and quality of commercial jacket tubing provided for EBR-I Core IV is an indication of the rate at which the fabrication state-of-the-art for Zircaloy-2 tubing was developing. Little or no reactor-grade tubing was commercially available in 1957,⁶ but at present, tubing of sufficiently high quality is available to permit, without excessive jacketing costs, the specification of maximum permissible defect depths in the range of 5 to 10% of the wall.⁷ In light of the conditions prevailing at the time of jacket-tubing procurement for EBR-I Core IV, it was necessary to accept tubing containing defects extending to a depth of 25% of the tube wall.

B. Fabrication

The yield of usable ANL-fabricated instrument tubing was low, less than 10%, from the extruded tube blanks. This was partly due to the fact that extrusion tooling was not available to extrude tube blanks having a D/t ratio near to, but slightly smaller than, the 5.3:1 ratio desired. Therefore, "controlled sinking" was undertaken. A combination of sinking and ironing passes was carried out with difficult-to-remove, nondeformable mandrels. A 30% loss was incurred as the result of instances in which mandrels could not be withdrawn or in which tubes were damaged during mandrel removal.

A more practical schedule for processing the tube blanks would have been to reduce the D/t ratio to the 5.1:1 to 5.3:1 range before ductile-core drawing so that any required wall thinning would be done while the inside diameter was large enough to facilitate mandrel removal. The final core-drawn tubes could then be sized after core removal by outer-surface etching. The effectiveness of this modification of the "core-drawing" technique has been proven at ANL on other difficult-to-fabricate materials.⁸

Much process-refinement work remains to be done in the area of high-quality, Zircaloy-2, instrument-tubing fabrication. Similar problems exist in fabrication of small-diameter, thin-wall tubing of other materials.

A combined attack on these problems will help to achieve the one objective they have in common--the prevention of wall defects.

Both ANL work and work by Knecht at Hanford⁹ have indicated the adequacy or advisability of annealing Zircaloy-2 at lower temperatures than the 850°C used for ANL rib-wire and rod production for this project.

After EBR-I Core IV production had been completed, samples of the commercial Zircaloy-2 rib wire used in EBR-I Core IV jacket assemblies were corrosion-tested for 14 days in 750°C, 1500-psi steam. The samples contained much loose, white, corrosion product. At present, there is no reported correlation between the steam-corrosion resistance of Zircaloy-2 and its resistance to the impure NaK (contaminated with Na₂O) in EBR-I.

C. Assembly

Future fuel elements having welded spacer ribs should be assembled by a method other than the intermittent-resistance welds used on the EBR-I Core IV rods. Continuous-rib welding methods, as used for PRTR fuel,¹⁰ appear to be a great improvement over the EBR-I Core IV method. Extruding ribbed tubes may even appear feasible for future fuel jackets, provided the quantity of material is sufficient to justify the development costs.

Evaluation and testing have eliminated components that appeared likely to fail in reactor service. The most meaningful evaluation of the hardware, however, is its ability to perform well in service. Periodic inspections should be undertaken to detect the possible occurrence of accelerated NaK corrosion of Zircaloy-2 rib wires.

APPENDIX A

Ordering Instruction to Accompany Purchase Order for Manufacture on a Conversion Basis of Small-diameter Seamless Zirconium-alloy Tubing for Reactor Service

1. STARTING MATERIAL

- a. The following reactor-grade Zircaloy-2 ingot shall be supplied by ANL:

Ingot No. MS-28188

Weight: 2100 lb

Size (approx): 16-in. diam x 51-in. long

BHN hardness range: 149-156

BHN hardness average: 153

Surface: Machine-conditioned

Ultrasonically tested and indicated defects removed.

Note: Additional material will be supplied if required by fabricator.

- b. All scrap ingot material, forging scrap, extrusion butts, and tubing scrap not converted to finished tubing shall be returned to ANL.

2. INGOT BREAKDOWN

- a. Fabricator shall supervise breakdown of ingot to material size suitable for extrusion billets. If this work is performed outside fabricator's plant, the following shall be specified and records kept accordingly:

Preheat temperature

Preheat time

Reductions made on forging

Number of reheats required

Reheat time

Reheat temperature.

- b. The above operations, when followed by suitable cleaning operations to remove not more than 1/32 in. of material from all surfaces, shall be performed so that no contamination will occur that will impair or reduce the ductility, mechanical properties, or high-temperature (650°F) water-corrosion resistance of the Zircaloy-2.
- c. Any defects or irregularities in the working of this material shall be noted.
- d. Customer shall be notified at least 24 hr in advance of working this material so that field representative may be present.

3. TUBING FABRICATION

- a. Extrusion billets shall be designed and extruded in a manner that will result in a finished extruded product having uniform dimensions and mechanical properties throughout its length and be free from contamination on all surfaces.
- b. The size of the extruded tube shall be such that not more than 80 to 90% total reduction in cross-sectional area is needed to work the extruded tube to the finished product.
- c. All reductions (within the limits set by paragraph b above), annealing, method of fabrication, lubrication, and cleaning shall be done at the manufacturer's discretion, provided no contamination that will introduce source-fissionable materials or impair the 650°F water-corrosion resistance of the tubing will result from such operations.
- d. All material shall be fully annealed after fabrication by holding at a temperature of $850 \pm 10^{\circ}\text{C}$ for 15 min in a vacuum or inert atmosphere of sufficient purity to prevent visual contamination of the surface.

4. FINISHED TUBING

- a. All tubing shall be finished to nonferrous-tubing commercial tolerances for OD, wall thickness, and eccentricity. Length tolerance shall be $\pm 1/4$ in.
- b. Straightness tolerance shall be within 50% of standard straightness tolerance for commercial round tubing, that is, one part in 1200.
- c. Surface finish, both inside and outside, shall be clean, smooth, and free from slivers, radial cracks, longitudinal cracks, seams, checks, laminations, and longitudinal striations. Results of eddy-current inspection of 100% of the finished tubing by either the supplier or ANL inspection shall be used to determine freedom from internal cracks.
- d. Ends shall be cut square with the longitudinal axis and deburred.

APPENDIX B

Specification for Seamless Zirconium-alloy Tubing for Nuclear Reactor Service

1.0 SCOPE

- 1.1 This specification covers reactor-grade, seamless, zirconium-alloy tubing for jacketing nuclear fuel for reactor-core loadings at Argonne National Laboratory.

2.0 APPLICABLE SPECIFICATIONS AND STANDARDS

- 2.1 MIL-Z-22124 (SHIPS)
Military Specification, Zirconium Alloy Ingots.
- 2.2 ASTM-E8
Methods of Tension Testing of Metallic Materials.
- 2.3 ASTM-E91
Method for Determining the Average Grain Size of Nonferrous Metals, Other than Copper, and Their Alloys.

3.0 MANUFACTURING PROCESSES

3.1 Material Used for Manufacture of Tubing

- 3.1.1 All material used for fabrication of tubing under this specification shall be from ingots produced by vacuum-melting and inspected for quality.
- 3.1.2 Ingot stock used for fabrication shall be supplied by the vendor in accordance with paragraph 3.1.1.

3.2 Seamless Tube Fabrication

- 3.2.1 Seamless tube may be made by any method that will yield a product meeting the requirements of this specification. This method may consist of forging and extrusion of billets followed by cold or warm working, by drawing, swaging, or tube reducing with intermediate anneals until the required dimensions are reached.
- 3.2.2 All extruded base-tube material shall be inspected for defects and surface-conditioned as necessary prior to any finishing operations.

3.2.3 All annealing operations shall be performed in vacuum with material cleaned prior to annealing. A suitable vacuum shall be maintained during annealing to avoid contamination which might prevent the finished material from meeting chemical analysis or corrosion tests.

3.2.4 For identification, all material of the same size and finished product from the same ingot processed in the same steps and annealed in the same vacuum-annealing charge shall be considered as an individual batch or lot. This lot size shall not exceed 100 lb or 1000 ft, whichever is less, and in no case shall it contain material from more than one ingot.

4.0 REQUIREMENTS

4.1 Chemical Requirements

The chemical composition of the ingots and the chemical composition of the finished material shall conform to the following requirements:

4.1.1 Alloying elements.

	<u>Zircaloy-2</u>
Tin	1.20-1.70 w/o
Iron	0.07-0.20 w/o
Chromium	0.05-0.15 w/o
Nickel	0.03-0.08 w/o
Zirconium	Balance

Total of iron, chromium and nickel shall fall within the range of 0.18-0.38 w/o.

4.1.2 Impurities (maximum permissible).

<u>Element</u>	<u>ppm</u>	<u>Element</u>	<u>ppm</u>
Aluminum	75	Manganese	50
Boron	0.5	Nitrogen	80
Cadmium	0.5	Oxygen*	1500
Carbon	270	Silicon	100
Chlorine*	50	Sodium	20
Cobalt	20	Titanium	50
Copper	50	Tungsten	100
Hafnium	100	Vanadium	50
Hydrogen	25	Uranium total	3.5
Lead	130	U ²³⁵	0.025
Magnesium	20		

*Waived if certified analysis of sponge shows less than 50 ppm.

4.2 Mechanical Properties; Destructive Tests

The mechanical properties of the finished tubing shall conform to the following requirements:

4.2.1 Finished annealed tube samples shall have the following tensile properties:

Tensile strength	67,000-70,000 psi
Yield strength (0.2%)	45,000-55,000 psi
Elongation in 2-in. gage length	20% min

4.2.2 The maximum hardness of finished annealed tubing shall not exceed the following values for the method used:

Hardness	Maximum Individual Reading
Rockwell B	98
Rockwell 15T	92
Rockwell 30T	80
Rockwell 15N	68

4.2.3 Metallographic examinations shall be made on both longitudinal and transverse sections of cut tubes.

4.2.3.1 The grain size shall not exceed ASTM No. 7 on a transverse section, as determined in accordance with the methods specified in ASTM-E91.

4.2.3.2 There shall be no evidence of inclusions of a carbonytride type or other hard inclusions at any stage of the tube manufacturing or in any of the metallographic samples.

4.2.3.3 There shall be no evidence of the presence of any longitudinal or transverse cracks in metallographic specimens.

4.2.3.4 The ID surfaces of 12-in.-long longitudinal tube sections shall be inspected for inclusions and fissures or cracks, using at least 10X stereomicroscope. After this inspection, both halves of the tube shall be flattened and reinspected for defects, noting carefully those areas that may have been suspect on the first examination. The presence of any laps, seams, cracks, or inclusions after flattening shall be cause for rejection of the entire length of tube from which the sample was cut.

4.3 Mechanical Properties; Nondestructive Tests

4.3.1 Tubing shall be free from internal defects, as determined by nondestructive eddy-current inspection.

4.3.2 Eddy-current inspection equipment used for tubing inspection shall have sufficient sensitivity to detect any defects greater than 10% of the wall thickness, 0.0025 in., in any diametral plane, and with a length not to exceed 1/16 in.

4.3.3 Any defects greater than specified in paragraph 4.3.2 shall be cause for rejection of the tube for a minimum of 1 in. on each side of the defect.

4.4 Corrosion Tests

4.4.1 Finished tubing prepared and tested for corrosion resistance, per paragraphs 5.2.3 and 5.3.9 of this specification, shall have demonstrated satisfactory corrosion resistance as follows:

- a. A continuously adherent black corrosion film with no corrosion-film defects (white or brown corrosion products, surface stains, cracks, streaks or blisters).
 - b. A weight gain between 18 and 38 mg/sq decimeter.
- 4.4.2 Should any tubing specimen fail to meet this corrosion test, a second randomly selected tube, fabricated as a portion of the same batch or lot processed with the original test specimen, shall be similarly tested. Rejection of the second sample shall be cause for rejection of all tubing processed in that batch.

4.5 Dimensional Tolerances

4.5.1 Tubing shall be supplied to a nominal ID, wall thickness, and overall length.

4.5.1.1 The tubing shall have a nominal ID of 0.257 + 0.000 - 0.004 in.

4.5.1.2 The tubing shall have a nominal wall thickness of 0.021 in. with a tolerance of ± 0.0025 in.

4.5.1.3 Tube lengths shall be minimum 6 ft or multiples thereof, with a tolerance of + 0.500 - 0.000 in.

4.5.2 All tubing shall be supplied free of bends or kinks and with a maximum uniform bow not to exceed one part in 1200 (0.010 in./ft).

5.0 QUALITY ASSURANCE

5.1 Workmanship

5.1.1.1 All tubing shall have a uniform surface that is free from scabs, cracks, slivers, blisters, seams, laps, pitting, and foreign matter as revealed by visual examination or non-destructive methods of testing and in accordance with standards approved by the purchaser.

5.1.1.2 Tube ends shall be cut square with the longitudinal axis and deburred.

5.2 Sampling for Acceptance Inspection

5.2.1 Sampling for Chemical Analyses

- 5.2.1.1 One sample of tubing shall be selected from each lot for analysis of all elements listed in paragraphs 4.1.1 and 4.1.2, except uranium and U²³⁵.
- 5.2.1.2 One sample shall be selected at random from tubing finished from each zirconium-alloy ingot and delivered to Argonne National Laboratory for the uranium and U²³⁵ analysis specified in paragraph 4.1.2.

5.2.2 Sampling for Mechanical Tests

- 5.2.2.1 One tensile test shall be made on one randomly selected tube from each lot of finished tubing.
- 5.2.2.2 At least one sample of tubing shall be selected for hardness testing from the same tube selected for the tensile-test specimen in paragraph 5.2.2.1.
- 5.2.2.3 One longitudinal section at least 2 in. long and one transverse section shall be taken randomly from each tube lot for metallographic examination. An additional 12-in.-long section shall be cut from the same tube for inspection in accordance with paragraph 4.2.3.4.
- 5.2.2.4 All tubing in all lots shall be subjected to nondestructive eddy-current inspection.

5.2.3 Sampling for Corrosion Tests

- 5.2.3.1 One 3-in.-long piece of tubing, selected from each lot, shall be prepared for corrosion testing as follows:
- a. The tube shall be sectioned longitudinally into two halves.

- b. All cut surfaces shall have 0.010 in. minimum machined off of edges. Finish-machined surfaces shall be 60 rms or better.
 - c. A 1/8-in.-diam drilled hole shall be located 1/4 in. from the end of each specimen.
 - d. All test specimens shall be permanently marked with the batch or lot number of tubing from which the sample was taken.
- 5.2.3.2 All corrosion coupons shall have received the same final heat treatment and surface finishing as the batch of material from which the coupons were selected.
- 5.2.3.3 Duplicate identified specimens for corrosion testing shall be made available to the purchaser upon request.

5.3 Analysis and Testing

- 5.3.1 Unless otherwise specified, the vendor is responsible for testing and analysis prior to submission for purchaser's inspection and acceptance. Unless otherwise specified, the vendor may utilize his own facilities or any commercial laboratory acceptable to the purchaser.
- 5.3.2 Samples selected for chemical analysis may be either chips or solid, in accordance with the analytical techniques to be used. Duplicate samples shall be made available to the purchaser on request. These samples shall be tested for all elements except uranium and U^{235} as stated in paragraphs 5.2.1.1 and 5.2.1.2 to determine compliance with paragraph 4.1.1 or 4.1.2. Analyses shall be made by a method and laboratory approved by the purchaser. If any individual analysis does not conform to paragraph 4.1.1 or 4.1.2, the material represented by that analytical specimen shall be rejected.
- 5.3.3 For uranium analysis, one sample selected at random from tubing finished from each ingot will be wet-chemically analyzed to determine the total uranium content and will be isotopically analyzed to determine the U^{235} and U^{238} ratio. Chemical and isotopical analysis will be performed by Argonne National Laboratory.
- 5.3.4 Analysis of a lot for sodium may be omitted if the zirconium used as starting material or the ingot used in producing that lot was not prepared by the sodium-reduction process.

- 5.3.5 All tensile test specimens shall be prepared and tested in accordance with the methods and procedure of ASTM-E8. The testing speed shall be 0.05 ± 0.02 in./in. of gage length per minute.
- 5.3.6 Samples selected for hardness testing shall be prepared by machining a flat surface sufficient to expose an area suitable for the testing procedure utilized. The ID of the tube shall be adequately supported by a mandrel or matching curved support, and the specimen shall have sufficient remaining wall thickness to avoid penetration of the indenter through the tube wall. The material tested shall be at room temperature. Duplicate samples shall be made available to the purchaser upon request. The type of test used shall be at the option of the vendor. If any sample does not comply with paragraph 4.2.2, the lot represented by that sample shall be rejected.
- 5.3.7 Metallographic examination for grain-size determination shall be performed in accordance with ASTM-E91.
- 5.3.8 All nondestructive-testing standards and methods shall be qualified by the vendor and shall be approved by the purchaser prior to use.
- 5.3.9 All sample selected for corrosion test shall be tested for 14 days in 1750°F, 1500-psi steam.

5.4 Retesting

- 5.4.1 The purchaser may retest duplicate samples of materials selected from any lot and reject any material that is not in accordance with the requirements of this specification.
- 5.4.2 Any lots of tubing rejected for noncompliance with chemical analysis or mechanical testing may be retested in accordance with the procedures outlined in paragraph 5.3 if there is evidence in the opinion of the purchaser that the original results are erroneous. Results of the retest shall be in compliance with Section 4.0, or the lots in question shall be rejected.

5.5 Records and Reports

Inspection records of all fabrication steps, examinations, and tests shall be kept complete and available to the purchaser.

- 5.5.1 Fabrication records shall be maintained to show the following information:
- Reduction by forging to include temperature, heating time, reheat time, reduction between reheat, and total reduction.

- b. Extrusion data to include dimensions of extrusion billet, heating time prior to extrusion, extrusion temperature, and reduction.
- c. Methods of cleaning base tube and preparation for subsequent finishing.
- d. Method of reduction and total reduction obtained between annealing operations.
- e. Temperature, time, and vacuum pressure maintained during annealing.
- f. Final cleaning and rinsing operations to produce the desired surface finish.
- g. Suitable cross-reference data to be kept on original ingot, extrusion billet, and lot number of finished tubing.

5.5.2 Prior to shipment of any lot of tubing, the vendor shall furnish three copies of certified reports giving the following information:

- a. Purchase-order number.
- b. Date of purchase order.
- c. Material-specification number.
- d. Vendor's name.
- e. Original ingot number with extrusion-billet number and tube-fabrication lot number cross-referenced.
- f. All test and inspection results.

5.5.3 The vendor shall maintain a complete record of all information in paragraphs 5.5.1 and 5.5.2 for the ingots and tubing for 5 years after shipment.

5.5.4 While work on the contract for the purchaser is being performed, the inspector representing the purchaser shall have free entrance at all times to all parts of the vendor's plants that are concerned with the manufacturing and inspection of the material being fabricated. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being finished in accordance with the requirements of this specification.

5.5.5 Unless otherwise specified, all tests and inspections shall be witnessed by an Argonne National Laboratory inspector.

6.0 MARKING, IDENTIFICATION, AND PACKAGING

- 6.1 Individual tubes shall be legibly marked with ink showing vendor's name, trademark, ingot number, and lot number.
- 6.2 Finished tubing representing more than one lot number may be packaged in the same container and made part of the same shipment, provided positive means of separating different lots of material and identification of each lot is maintained in each shipment.
- 6.3 All tubes shall be individually separated by clean paper and packed in a container that will prevent damage and/or contamination during shipment and storage.
- 6.4 Each separate package, box, or bundle of finished tube shall be legibly and conspicuously marked with the following information:
 - a. Destination.
 - b. Purchase order or contract number.
 - c. Type of material.
 - d. Size.
 - e. Lot and ingot number.
 - f. Gross, net, and tare weights.
 - g. Vendor's name.

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