MAY 20 1968

GEAP-5589
FEBRUARY 1968

JOINT U.S. - EURATOM RESEARCH
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THE FABRICATION AND CHARACTERIZATION OF Zr-Cr ALLOY TUBING

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U.S. ATOMIC ENERGY COMMISSION
CONTRACT AT (04-3)-189
PROJECT AGREEMENT 24

NUCLEAR FUELS DEPARTMENT
GENERAL ELECTRIC
SAN JOSE, CALIFORNIA

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Zr-Cr ALLOY TUBING

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U. S. Atomic Energy Commission
Contract AT (04-3) - 189
Project Agreement 24

Printed in U.S.A. Available from the
Clearing House for Federal Scientific and Technical Information
National Bureau of Standards, U.S. Department of Commerce
Springfield, Virginia
Price: \$3.00 per copy

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FOREWORD

The United States and the European Atomic Energy Community (EURATOM), on May 27, 1958, and June 18, 1958, signed an agreement which provides a basis for cooperation in programs for the advancement of peaceful applications of atomic energy. This agreement, in part, provides for the establishment of a Joint US-EURATOM research and development program which is aimed at reactors to be constructed in Europe under the Joint Program.

The work described in this report represents the Joint US-EURATOM effort which is in keeping with the spirit of cooperation in contributing to the common good by the sharing of scientific and technical information and minimizing the duplication of effort by the limited pool of technical talent available in western Europe and United States.

ABSTRACT

A Zr + 1.5 weight percent Cr + 0.08 weight percent Fe alloy (designated Zr-Cr) has been developed with the potential for improved service reliability as boiling water reactor fuel cladding. The fabrication of significant quantities of fuel cladding tubing and bar stock from a large ingot of Zr-Cr alloy is described. Characterization tests on these wrought products confirmed the excellent high purity water corrosion resistance for this composition at temperatures above 300°C. Mechanical properties were equal to present Zircaloy-2 fuel cladding tubing.

Also described is an irradiation test program where the performance of Zr-Cr alloy clad fuel rods is being compared directly to similar Zircaloy-2 clad fuel rods in a boiling water power reactor environment. Included in this experiment are non-fueled dummy rods to separate the effects of heat transfer during irradiation on the corrosion of the Zr-Cr alloy.

SECTION I

INTRODUCTION

As a result of the original Specific Zirconium Alloy Design Program (PA-24), a Zr + 1.5 wt % Cr + 0.08 wt % Fe alloy, designated Zr-Cr alloy, was developed with the potential for improved service reliability as boiling water reactor fuel cladding.

Of the 31 zirconium alloys tested, including Zircaloy-2, this composition was shown to have far better high temperature, > 300°C, corrosion resistance and, therefore, offers protection against overtemperature conditions which could result in excessive corrosion for the present water reactor cladding alloys, Zircaloy-2 and Zircaloy-4. This superior high temperature water corrosion resistance was demonstrated on laboratory heats to exposures greater than 8000 hours;⁽¹⁾ and warranted further testing of the Zr-Cr alloy under irradiation as actual fuel rod cladding. For this fuel rod irradiation program, the Zr-Cr alloy had to be fabricated into tubing for fuel cladding, and bar stock for end plugs.

The initial large-scale commercial ingot (1200 pounds) was fabricated into tubing along with a commercial ingot of Zircaloy-2.

Except for the ingot melting, both alloys were fabricated into tubing and bar in the same manner by a commercial vendor. A standard Zircaloy-2 process was used which presented no major problems with either alloy. Extensive testing revealed variable corrosion performance for the Zr-Cr alloy tubing fabricated during this initial trial.⁽²⁾ Metallography revealed that the difference between variable corrosion performance by material from the large commercial ingot and the reproducibly excellent performance in the same tests by material from small laboratory melts was caused by the nonuniform distribution of coarse intermetallic compounds in the vendor material. Additional studies resulted in a revised fabrication process to obtain fine, uniformly distributed intermetallics.⁽³⁾ A second large ingot of Zr-Cr alloy was melted and fabricated into tubing by the revised process. The details regarding the fabrication and properties of this second round Zr-Cr alloy tubing are presented in this report.

The fuel rod irradiation program to test the performance of the Zr-Cr alloy relative to Zircaloy-2 as cladding in an operating boiling water reactor is also summarized.

SECTION II

Zr-Cr ALLOY DEVELOPMENT BACKGROUND

Zr-Cr alloy is basically a zirconium-chromium alloy with a minor addition of Fe. From the Zr-Cr phase diagram,⁽⁴⁾ the composition Zr + 1.5 weight percent Cr, should have one high temperature equilibrium phase, beta, which transforms to two lower temperature equilibrium phases, an alpha matrix plus Cr and Fe rich intermetallic compounds. Using dilatometry and metallographic examination, the temperature boundaries for the two-phase, alpha-plus-beta region were found to be 800° and 900°C. These phase boundaries are different from those shown by Hansen⁽⁴⁾ and indicate the alpha-beta transformation is sluggish in both directions.

Precipitation and dissolution behavior in the Zr-Cr alloy was studied in detail.

Kinetic studies show that Zr-Cr₂ precipitate coarsening occurs rapidly in beta-quenched Zr-Cr above 800°C, Figure 2-1, with a resultant adverse effect on corrosion resistance.⁽⁵⁾ It was also established that temperatures above 950°C were required to dissolve these coarse second-phase particles in reasonably short times, ~2 hours, Figure 2-2.⁽⁵⁾ Beta solution temperatures between 955° to 1050°C had no significant effect on corrosion weight gains of quenched and aged samples; solution time is not critical. Optimum corrosion resistance was not achieved with material slow-cooled from the beta solution temperature, indicating quenching rate is an important variable. Beta-quenched material, without subsequent aging, exhibits high corrosion weight gains, but rapid quenching followed by aging can result in optimum corrosion resistance.

The above experiments on quenching and aging led to the tentative conclusion that optimum corrosion performance was associated

with fine, uniformly distributed intermetallics in a strain-free matrix of zirconium containing approximately 0.9 atomic percent Cr (0.52 weight percent Cr). Overaging, particularly in the two-phase, alpha-plus-beta temperature range, leads to matrix regions which are depleted in chromium by the precipitation of coarse particles. The chromium in the intermetallic particles is not as effective in doping the ZrO₂ corrosion film to produce low oxygen diffusion rates as is chromium in solid solution. Quenched samples with high lattice strain (and above optimum chromium content) show poor corrosion performance, perhaps in part because of an adverse effect of the lattice strain on the effective plasticity of the ZrO₂ corrosion film.

Nitrogen contamination was also suspected as a cause for variable corrosion performance of the initial commercial materials. However, a controlled experiment revealed the corrosion performance of Zr-Cr was not specially sensitive to nitrogen, and the commercial specification for nitrogen in the Zircalloys was also adequate for Zr-Cr.⁽⁶⁾

With this background, it was postulated that the nonuniform distribution of intermetallics in the first round tubing was caused by segregation of chromium in the beta-phase during fabrication in the two-phase temperature region. Since all fabrication steps, Figure 2-3, after extrusion were performed at temperatures below 760°C, it is unlikely the precipitate coarsening occurred during or after extrusion. The extrusion billets were solution-treated at 1000°C but the total residence time in the furnace was only 1 hour. The quench cooling rate at 700°C was computed to be at least 28°C/sec, therefore, the large precipitates were not likely to be formed during the quench, see Figure 2-1. The large particles and inhomogeneous structure must

FIGURE 2-1. PRECIPITATE COARSENING IN β -QUENCHED Zr-Cr AS A FUNCTION OF PROCESSING TEMPERATURE AND TIME AT TEMPERATURE

FIGURE 2-2. PRECIPITATE DISSOLUTION IN Zr-Cr AS A FUNCTION OF TIME AT 955°C

(1) FIRST NUMBER IS OUTSIDE DIAMETER OF TUBE, SECOND NUMBER IS WALL THICKNESS.

FIGURE 2-3. FLOW DIAGRAM OF PROCESS USED IN FABRICATION OF FIRST ROUND COMMERCIAL Zr-Cr TUBING

have developed before, and persisted through the solution heat treatment. The material was forged at temperatures between 900° and 1010°C and the billet was slow-cooled at least twice. The billet temperature could well have dropped below 900°C during the forging operation and remained there for a significant time because of slow reheat resulting from its size. Therefore, large intermetallic particles and two-phase segregation could be expected to occur during the forging operation. During the 1 hour solution treatment following the second forging step, the particles were actually growing until the billets reached temperatures above 900°C. The time spent at temperature was too short to allow the coarse intermetallic particles to go into solution and homogenize the microstructure completely. However, some of the particles may have gone into solution

and variations (among billets and along a single billet) in heatup rate, temperature, and time at temperature could well be responsible for the variable performance of this initial commercial Zr-Cr material.

A new process schedule, Figure 2-4, used in fabrication of the second round tubing, was aimed at refining the second phase precipitation without adding any extra costs or processing steps. This was accomplished by increasing the solution heat treating temperature of the extrusion billets to >1000°C for 2 hours at temperature followed by a water quench. This solution treatment was necessary for complete dissolution of all precipitates greater than 1 μ in size. Subsequent processing steps were performed at or below 700°C to avoid coarsening the intermetallic precipitates.

(1) FIRST NUMBER IS OUTSIDE DIAMETER OF TUBE, SECOND NUMBER IS WALL THICKNESS.

(2) REDUCED TUBES WERE FLASH-ETCHED BEFORE ANNEAL.

FIGURE 2-4. FLOW DIAGRAM OF PROCESS USED IN FABRICATION OF SECOND ROUND COMMERCIAL Zr-Cr TUBING

SECTION III

TUBE FABRICATION

As a result of earlier experience, the process used for fabrication of the second round Zr-Cr tubing was carefully controlled. The details of the various steps in the tubing process are presented below.

3.1 MELTING

The second round Zr-Cr ingot was vacuum melted by Wah Chang Corporation using a consumable-electrode arc furnace. Chromium powder and zirconium chunklets were pressed into rectangular bars 4.6 × 6.9 × 69 cm long (2 × 3 × 30 inches long). These bars were then electron-beam welded end-to-end to make up the initial electrode. This electrode was first vacuum-melted to form an 8-inch diameter ingot and then revacuum-melted into a 28-cm (11-inch) diameter ingot. The unconditioned ingot weight was 452 kg (973 pounds). The ingot was conditioned by machining 1.7 cm (3/4 inch) from the outside surfaces resulting in a 24-cm (9.5-inch) diameter by about 122-cm (4-foot) long finished ingot. The chemical analysis of the ingot is given in Table 3-1. Additional chrome was added to the original melt, 1.5 weight percent Cr, to allow for evaporation during melting. Approximately 0.24 weight percent Cr was lost during melting. The as-cast structure, Figure 3-1a, exhibits complete macrohomogeneity. On a finer level, Figure 3-1b, however, microsegregation is clearly evident (different etching characteristics resulting in dark and light areas shown in micrographs).

3.2 HOT WORKING AND SOLUTION HEAT TREATMENT

As mentioned earlier, hot working in the two-phase region, 800° to 900°C, or slow cooling through this region after hot working

should be avoided. The ingot was preheated at 1050°C for 1-1/2 hours, and then hot forged to a 11.4-cm (4-1/2-inch) round. Frequent reheating, every 30 minutes, to 1050°C was required to maintain the surface temperature above 900°C. Without intermediate cooling, the entire forging was solution-treated at 1050°C for 1 hour and then water quenched. All thermal and forging treatments were performed in air. The surface oxide of the final forging was clear and black, Figure 3-2, indicating only minor surface contamination occurred during the hot working and solution operations.

One end of the ingot was forged down to 7.9-cm (3-1/8-inch) diameter by 76-cm (3-feet) long for rod billets. Three rod billets were machined and warm-extruded without any further solution-heat treatments. The tube billets were rough machined from the 11.4-cm (4-1/2-inch) forging to 10.2-cm (4-inch) outside diameter × 1.9-cm (3/4-inch) inside diameter × 25.4 cm (10 inches) long. These rough billets were then given an additional solution heat treatment at 1050°C for 1 hour at temperature followed by a water quench on both inside diameter and outside diameter. The as-quenched microstructure, Figure 3-3, exhibited a fine Widmanstätten structure typical of beta-quenched zirconium alloys with only a minor surface layer, about 0.25-mm (0.01-inch) deep, which etched differently, probably because of oxygen and nitrogen contamination. Prior beta grain boundaries are clearly retained and selectively etched, indicating a composition or structural difference at the boundary. Evidence of these beta boundaries remain even after extrusion and subsequent tube reduction and annealing. No coarse precipitates, >0.5μ, were observed. Final machining of the extrusion billets removed 2.54 mm (0.100 inch) from each surface resulting in

TABLE 3-1
CHEMICAL COMPOSITION OF VENDOR INGOT NO. 2

Element	Specification	VNC Laboratory Analysis (sheet) Alloy Additions (wt. %)	Vendor Analysis (ingot)*
Cr	1.10—1.30	1.16 ± 0.02	1.26 ± 0.02
Fe	0.09—0.11	0.111 ± 0.002	0.106 ± 0.002
	(ppm)	Impurities (ppm)	(ppm)
Ni	<70	23 ± 5	10
N	<60	30 ± 2	44 ± 2
O	900 - 1500	1170 ± 10	955 ± 55
H	<25	11 ± 1	6 ± 1
Al	<75	45 ± 3	49 ± 7
C	<500	135 ± 8	127 ± 3
Si	<120	91 ± 5	<40
Sn	---	<10	55 ± 20
Cu	<50	20 ± 1	<25
Pb	<130	22 ± 2	6 ± 2
Mg	<20	6 ± 1	<10
Mn	<50	12 ± 1	<10
W	<100	<50	25 ± 1
Ti	<50	6 ± 1	<20
Ci	<20	<10	<20
B	<0.5	0.5	0.2
Cd	<0.5	<0.5	<0.3
Co	<20	<2	<5
Hf	<100	<50	80 ± 6

* Vendor also gives Ca <10, Cb <100, Mo <10, Ta <200, U <0.5, V <5, and Zn <50.

contamination free billets. A typical hardness traverse, Figure 3-4, shows a large radial hardness variation across the billets before extrusion. This variation could come from two sources: (1) matrix distortion

strain caused by shear type beta to alpha transformation, and (2) elastic strain caused by chromium in solid solution. Both sources of hardening are increased by increasing quenching rates from beta solution treatment.

(a)

100X

(b)

750X

FIGURE 3-1. AS-CAST COMMERCIAL Zr-Cr INGOT NUMBER 2

FIGURE 3-2. TAPERED SECTION FROM Zr-Cr FORGING AFTER FIRST SOLUTION TREATMENT

BETA
GRAIN BOUNDARY →

(a)

100X

(b)

100X

FIGURE 3-3. MICROSTRUCTURE OF Zr-Cr EXTRUSION BILLETS AFTER HOT FORGING AND DOUBLE SOLUTION TREATMENT. (a) AREA NEAR OUTSIDE SURFACE, (b) AREA AT INSIDE SURFACE. 100X

FIGURE 3-4. HARDNESS TRAVERSE ACROSS THE WALL OF QUENCHED EXTRUSION BILLET

3.3 EXTRUSION

All extrusion billets were jacketed in copper and preheated at 650°C for 2-1/2 hours. Warm extrusion was readily accomplished at 650°C with 25 percent more ram pressure, 612 metric tons of force (675 tons), than normally used for the Zircaloys. The reduction ratio was 9 to 1. One initial extrusion attempt at 635°C and normal pressure was abandoned when the material did not start through the die. Impact starting was subsequently utilized to reduce the possibility of stalling the press. Total time for extrusion was less than 4 seconds. The extruded tube shell size was 4.13-cm (1-5/8-inch) outside diameter by 0.8-cm (5/6-inch) wall by 2 to 2.3 meters (8 to 9 feet) long.

Metallographic examination of end sections from various extrusions, Figure 3-5, showed a generally fine grain alpha structure with a limited amount of grain growth. These large grain areas indicate that the mechanical and thermal conditions were proper in localized regions during extrusion for an exaggerated grain growth phenomenon to start. Hardness measurements indicated a fairly high but relatively constant hardness of 92 R_B across the nose of each extrusion. The tail had similar high hardness on the outside diameter but a lower hardness of 85 R_B near the inside diameter. The lower hardness values were related to an increase in grain size, Figure 3-5.

The initial extrusions were given an alpha anneal at 704°C for 4 hours to reduce the hardness before cold reducing (typical practice for Zircaloys). During this anneal extensive grain growth occurred which resulted in a layer of large grains on both the inside diameter and outside diameter of these extrusions, Figure 3-6. Subsequent cold reduction of these extrusions resulted in extensive cracking at grain boundaries in the large grain layers, Figure 3-7. Additional

processing of these annealed extrusions was abandoned in favor of making new extrusions. The initial alpha anneal was omitted on these final extrusions and cold reduction proceeded without difficulty. Following extrusion the copper jacket was removed by dissolution in nitric acid and the outside diameter surfaces were belt-polished to remove surface irregularities.

3.4 COLD REDUCTION

Cold reduction was accomplished using a 3.8-cm (1-1/2-inch) tube reducer. Four reductions, Figure 2-4, greater than 50 percent reduction in area were completed without major difficulty. A minor surface cracking (crazing) was encountered during each of the four reductions, Figure 3-8. This crazing appeared after rather small amounts of reduction, about 15 percent, and the number of cracks increased with continued reduction to 60 percent. The depth of individual cracks was less than about 0.038 mm (0.0015 inch) and generally appeared rounded at the crack tips. (This condition is not unique to the Zr-Cr alloy but has been observed on titanium alloy and Zircaloy tubing.) In most cases, the crazing was removed by etching the reduced tubes before annealing. Before the final sizing reduction the inside diameter of all the tubes was etched to minimize the inside diameter cracks. After the last reduction belt-polishing was employed to remove about 0.076 mm (0.003 inch) from the outside diameter of the tubing to ensure a crack-free surface before final etching.

All intermediate annealing and final stress relief were performed in vacuum, $<10^{-4}$ torr. All tubes were shiny bright after each anneal. The bright surface turned brown after standing in air at ambient temperature, about 25°C. The brown surface film was readily removed in standard HNO₃-HF etching solutions after each reduction.

(a)

100X

(b)

100X

FIGURE 3-5. TYPICAL MICROSTRUCTURE OF AS EXTRUDED Zr-Cr, (a) OUTSIDE SURFACE OF EXTRUSION WHERE HARDNESS WAS 95 R_B, (b) INSIDE SURFACE OF EXTRUSION WHERE HARDNESS WAS 87 R_B

FIGURE 3-6. SURFACE STRUCTURE OF EXTRUDED Zr-Cr AFTER 704°C (1300°F) ALPHA-ANNEAL

FIGURE 3-7. CRACKED TUBE SHELLS AFTER FIRST TUBE REDUCTION

FIGURE 3-8. SURFACE CRACKING (CRAZING) OBSERVED AFTER TUBE REDUCTION

After etching, the Zr-Cr tubing was coated with a light film or stain of very fine particles. These particles were easily removed by wiping with a sponge or scrubbing with a nylon brush. If these particles were not removed after etching the possibility of fluoride contamination was greatly increased.

Each tube was slightly bowed after the final stress relief and required straightening using a standard three-stand roller straightener. The final tubes were well within the specified straightness tolerance.

SECTION IV

WROUGHT PRODUCTS EVALUATION

The Zr + 1.15 weight percent Cr + 0.08 weight percent Fe alloy (Zr-Cr) which was fabricated into tubing and bar stock for the fuel rod irradiation experiment in Consumers Big Rock Point Reactor (BRP) was carefully characterized to ensure that the potential of the alloy was actually realized on the commercially-produced wrought products. Two tube sizes were fabricated: 0.968-cm (0.380-inch) inside diameter $\times$ 0.081-cm (0.032-inch) wall for use as fuel cladding, and 1.271-cm (0.501-inch) inside diameter $\times$ 0.081-cm (0.032-inch) wall for an out-of-pile heat transfer corrosion test. The latter tubing was necessary to facilitate its use as a heater sheath in existing facilities, and this tubing was used for both heat transfer and isothermal corrosion tests. Both corrosion and mechanical tests were performed on the bar stock. Table 4-1 outlines the test performed on both tubes and bar stock.

4.1 INSPECTION

A 100 percent inspection was performed on the Zr-Cr fuel tubes. All tubes met dimensional tolerances. Inside diameter measurements were within the specified 0.968 ± 0.005 cm (0.379 to 0.383 inch). Wall thickness measurements were within 0.0736 to 0.0864 cm (0.029 to 0.034 inch). Ultrasonic tests revealed no defects greater than 0.0076-cm (0.003-inch) deep. Tube straightness was within the specified 0.0356 cm/43.2 cm (0.014 inch/17 inches) maximum bow. All Zr-Cr fuel tubing was judged suitable for use as fuel cladding.

4.2 CHEMICAL ANALYSIS

The chemical composition of all Zr-Cr wrought products, tubing and bar stock, was checked to verify that no compositional changes had occurred during processing. The results are compared with the ingot analysis and specifications in Table 4-2. No major changes were noted: all wrought products fully met the specifications.

4.3 METALLOGRAPHY

Metallography performed on ten samples picked at random showed the fuel tubing to have a uniform fine grain size, $< \text{ASTM } 10$, Figure 4-1. Individual grains were equiaxed in the transverse direction and slightly elongated in the longitudinal direction, typical for rocked zirconium alloy tubing. No large grains, $> \text{ASTM } 10$, were observed in any of the cross sections examined. Background signals during the ultrasonic testing of this tubing were also representative for homogeneous fine grain material.

Hydride content was determined to be < 20 ppm by metallography which is in agreement with the hydrogen analyses made by the hot extraction method, < 20 ppm hydrogen. The hydride orientation was random, Figure 4-2. Fine alpha grain material with low hydride content was also verified for the bar stock and heat transfer tubes, Figure 4-3.

TABLE 4-1

CHARACTERIZATION TESTS AND EXAMINATIONS FOR Zr-Cr ALLOY TUBING AND BAR STOCK

TESTS	MATERIAL TESTED	RESULTS
1. Incoming Inspection		Tubing within design tolerances.
a. Straightness	Fuel tubes and bar stock	Within 0.0356 cm/43.2 cm maximum bow.
b. Inside Diameter Measurements	Fuel tubes	Within 0.968 ±0.0051 cm.
c. Wall Thickness Measurements	Fuel tubes	Within 0.0736 to 0.0864 cm.
d. Ultrasonic Test for Defects	Fuel tubes and bar stock	No defect greater than 0.0076-cm deep.
2. Corrosion Tests		
a. Isothermal Autoclave Tests at 300°, 400°, and 500°C	Both tubes and bar stock	Weight gains for 2000-hour exposure satisfactory.
b. Heat Transfer Loop Tests (CL-1 loop)	Heat transfer tubes	Will continue for 8000 hours; weight gains after 1868 hours satisfactory.
c. Isothermal Loop Tests (CL-4 loop)	Fuel tubes	Specimens moved to isothermal section of CL-1 loop weight gains after 4,146 hours satisfactory.
3. Mechanical Tests		
a. Tensile Tests 25° to 600°C	Fuel tubes and bar stock	Data reported in Table 4-4.
b. Burst Tests, 25° to 600°C	Fuel tubes only	
c. Stress to Rupture Tests 300° to 600°C	Fuel tubes only	Tests are in progress.
4. Physical Metallurgy Examinations		
a. Chemical Analysis	Both tubes and bar stock	Uniform grain size = ASTM 11-12; ppt size <1/2 micron.
b. Metallography	Both tubes and bar stock	

TABLE 4-2

CHEMICAL ANALYSES OF VENDOR PRODUCTS

ELEMENT	ANALYSIS (weight percent)				
	Specification	Ingot*	Fuel Tubing	Heater Sheath Tubing	Bar Stock
Chromium	1.10 to 1.30	1.26 ± 0.02	1.29 ± 0.05	1.28 ± 0.05	1.30 ± 0.05
Iron	0.09 to 0.11	0.106 ± 0.002	0.105 ± 0.002	0.109 ± 0.005	0.103 ± 0.002
Impurities (ppm)					
Nitrogen	<60	44 ± 2	22 ± 1	24 ± 1	24 ± 2
Oxygen	900 - 1500	955 ± 55	1015 ± 65	1067 ± 80	1068 ± 20
Hydrogen	<25	6 ± 1	17 ± 3	15 ± 2	8 ± 2
Aluminum	<75	49 ± 7	37	37	30
Carbon	<500	127 ± 3	--	--	--
Nickel	<70	10	25	37	24
Silicon	<120	<40	28	28	10
Tin	---	55 ± 20	10	10	10
Copper	<50	<25	18	18	10
Lead	<130	6 ± 2	<5	<5	<5
Magnesium	<20	<10	5	11	7
Manganese	<50	<10	38	38	15
Tungsten	<100	25 ± 1	<50	<50	<50
Titanium	<50	<20	20	20	<5
Chlorine	<20	<20	--	--	--
Boron	<0.5	0.2	<0.2	<0.2	<0.1
Cadmium	<0.5	<0.3	<0.5	<0.5	<0.5
Cobalt	<20	<5	<5	<5	<10
Hafnium	<100	80 ± 6	<50	<50	<50

*Vendor Analysis

a) TRANSVERSE

100X

b) LONGITUDINAL

100X

FIGURE 4-1. TYPICAL MICROSTRUCTURE OF Zr-Cr FUEL TUBING

100X

FIGURE 4-2. TYPICAL CROSS SECTION OF Zr-Cr TUBING SHOWING HYDRIDES

(a)

100X

(b)

100X

FIGURE 4-3. TYPICAL TRANSVERSE MICROSTRUCTURES OF (a) Zr-Cr BAR AND (b) HEAT TRANSFER TUBES

4.4 CORROSION RESISTANCE

Corrosion testing of the Zr-Cr tubing and bar stock has been performed at 300°, 400°, and 500°C in three different corrosion facilities. Weight gains for exposures in 300°, 400°, and 500°C low-flow steam autoclaves (20–23 ppm O₂ + 1 to 3 ppm H₂ added) to 2000 hours are shown in Table 4-3. These data confirm the superior corrosion resistance of all Zr-Cr wrought products as compared to Zircaloy-2 at temperatures above 300°C. The 500°C weight gains are compared to weight gains for sheet made from the best heat of laboratory melts of Zr-Cr alloy in Figure 4-4. Although the corrosion rates for the wrought products are slightly higher than the best laboratory melts, the corrosion rates of the wrought products indicate that the potential of the alloy was realized in all wrought products.

Data at higher exposures, 4146 hours, in the isothermal section of the Cl-1 loop (280°C saturated water containing 0.2 ppm O₂) are also available for fuel tubing, Table 4-3. The weight gains for the Zr-Cr fuel tubing are again lower than the Zircaloy-2 control for this environment.

Heat transfer corrosion tests are also in progress. These tests are being performed in the Cl-1 loop which simulates actual boiling water reactor fuel rod conditions, but without neutron flux. This loop operates with 285°C saturated steam (steam quality 4 percent) similar to boiling water reactor environments (0.2 ppm O₂). Data after exposures of

1868 hours are available. Table 4-4, and indicate normal weight gains for heat transfer specimens operated at 500,000 Btu/h-ft². Isothermal specimens were also included in the same environment. Similar weight gains were experienced for both Zr-Cr and Zircaloy-2. These tests will be extended for 9 months to 1 year (6200 to 8000 hours) for comparison with reactor fuel cladding.

The bar stock corrosion rates were equal to the tubing in all corrosion facilities suggesting that only one solution treatment might be all that is necessary during tube fabrication. Figure 2-4. Elimination of the 1-hour solution treatment after rough machining of extrusion billets would be considered in future Zr-Cr tube fabrication.

4.5 MECHANICAL PROPERTIES

Tensile and burst tests were performed on Zr-Cr fuel tubing and bar stock at ambient temperature and at various other temperatures to 600°C. These tests, Table 4-4, indicate longitudinal tensile properties similar to stress-relieved Zircaloy-2. Adequate ductility was also demonstrated by total tensile elongations of 20 percent. Circumferential fracture elongations from burst tests indicate lower transverse ductility, 10 percent, compared to longitudinal ductility. However, 10 percent circumferential fracture elongation under closed end burst test conditions is considered adequate for the proposed fuel cladding irradiation. Specified properties for stress relieved Zircaloy-2 tubing are also shown in Table 4-4 for comparison.

TABLE 4-3

Zr-Cr TUBING AND BAR CORROSION WEIGHT GAINSLow Flow Steam Autoclaves ⁽¹⁾

Specimen Description and Initial Conditions ⁽²⁾		Average Weight Gain (mg/dm ²)										
500°C Test		24 h	48 h	72 h	144 h	421 h	569 h	966 h	990 h	1579 h	1976 h	2000 h
Zr-Cr Fuel Tube,	1	32.7	--	50.5	--	97.4	--	--	130	--	--	205
Zr-Cr Heat Transfer Tube,	1	32.4	--	49.6	--	93.4	--	--	125	--	--	246
Zr-Cr Bar Stock,	1	--	--	--	--	--	94.5	--	--	167	--	--
Zr-Cr Fuel Tube,	2	--	47.0	--	67.8	--	--	129	--	--	239	--
Zr-Cr Fuel Tube,	3	21.6	--	38.3	--	81.0	--	--	116	--	--	222
Zircaloy-2 Control,	1	74.6	--	105	--	234	--	--	646	--	--	Disintegrated
400°C Test		72 h	96 h	168 h	341 h	413 h	569 h	910 h	982 h	1587 h	1928 h	2000 h
Zr-Cr Fuel Tube,	1	13.2	--	19.9	--	28.7	--	--	41.5	--	--	55.5
Zr-Cr Heat Transfer Tube,	1	15.0	--	21.9	--	29.4	--	--	44.2	--	--	57.7
Zr-Cr Bar Stock,	1	--	--	--	--	--	27.8	--	--	43.3	--	--
Zr-Cr Fuel Tube,	2	--	21.1	--	33.6	--	--	48.9	--	--	60.9	--
Zr-Cr Fuel Tube,	3	7.5	--	14.9	--	23.0	--	--	35.3	--	--	49.4
Zircaloy-2 Control,	1	19.4	32.0	25.2	42.2	33.2	54.8	67.1	53.8	85.5	95.7	84.3
300°C Test		72 h	96 h	168 h	349 h	421 h	918 h	990 h	1928 h	2000 h		
Zr-Cr Fuel Tubes,	1	5.5	--	5.9	--	9.0	--	15.9	--	30.4		
Zr-Cr Heat Transfer Tube,	1	5.1	--	5.4	--	8.1	--	15.6	--	31.8		
Zr-Cr Fuel Tube,	2	--	2.2	--	6.7	--	16.0	--	35.2	--		
Zr-Cr Fuel Tube,	3	1.1	--	1.3	--	2.8	--	8.2	--	20.8		
Zircaloy-2 Control,	1	9.5	9.5	11.7	12.9	14.2	15.7	16.6	19.1	19.7		

(1) 20-23 ppm O₂, 1-3 ppm H₂ added

(2) 1 — Etched and NaOH cleaned.

2 — Etched, NaOH cleaned, and preoxidized at 300°C for 60 hours (2.6 mg/dm²).3 — Etched, NaOH cleaned, and preoxidized at 400°C for 12 hours (5.9 mg/dm²).

TABLE 4-3 (Continued)

C1-1 Loop

Specimen Description and Initial Condition ⁽²⁾	Loop Environment	Average Weight Gain (mg/dm ²)				
		<u>271 h</u>	<u>600 h</u>	<u>871 h</u>	<u>1597 h</u>	<u>1868 h</u>
Zr-Cr Heat Transfer Tube Under Heat Transfer, 1	536 to 546° F at 514,500 $\frac{\text{Btu}}{\text{h-ft}^2}$ ⁽³⁾	--	12.2	--	20.4	--
Zr-Cr Heat Transfer Tube Under Heat Transfer, 3	536 to 546° F at 518,980 $\frac{\text{Btu}}{\text{h-ft}^2}$ ⁽³⁾	5.0	--	9.4	--	15.9
Zr-Cr Heat Transfer Tube, 1	546° F Isothermal ⁽³⁾ Steam-Water	8.7	--	12.4	--	15.4
Zircaloy-2 Control, 1	543° F Isothermal ⁽³⁾ Steam-Water	8.4	--	12.3	--	15.3
		<u>2549 h⁽⁵⁾</u>		<u>3149 h</u>		<u>4146 h</u>
Zr-Cr Fuel Tubing, 1	543° F Saturated ⁽⁴⁾ Water	11.9		14.4		17.2
Zircaloy-2 Control, 1	543° F Saturated ⁽⁴⁾ Water	16.8		16.6		18.4

(3) Heat transfer section of C1-1 loop has 20 ppm O₂ and 2 ppm H₂ in steam phase.

(4) Isothermal section of C1-1 loop with ~0.2 ppm O₂ and ~0.025 ppm H₂

(5) 539° F subcooled water 1.5 ppm O₂ in C1-4 loop, transferred to C1-1 loop.

FIGURE 4-4. STEAM CORROSION DATA - 500°C EXPOSURE

TABLE 4-4

MECHANICAL PROPERTIES OF Zr-Cr TUBING AND BAR

Material	Type of Sample	Test Temperature (°C)	0.2% Offset Yield Stress ⁽¹⁾ (kg/mm ²)	Ultimate or Burst Stress ⁽²⁾ (kg/mm ²)	Elongation		Red in Area (%)
					Uniform (%)	Total (%)	
Bar Stock	Longitudinal Tensile	25	46.8	63.3	9.0	23.4	49
Fuel Tube	Longitudinal Tensile entire tube	25	45.6	63.2	---	19.4	--
Fuel Tube	Longitudinal Tensile cut section	25	48.5	63.5	7.7	14.3	42
Fuel Tube	Circumferential burst	25	----	73.1	---	20	--
Bar Stock	Longitudinal Tensile	100	40.5	55.1	9.1	24.4	54
Fuel Tube	Longitudinal Tensile cut section	100	41.9	55.5	10.4	22.1	45
Bar Stock	Longitudinal Tensile	200	34.0	45.3	7.5	32.5	61
Fuel Tube	Longitudinal Tensile cut section	200	35.4	46.3	7.3	18.0	45
Bar Stock	Longitudinal Tensile	300	28.8	38.0	7.5	27.2	68
Fuel Tube	Longitudinal Tensile cut section	300	32.0	38.6	4.3	16.3	53
Fuel Tube	Circumferential burst	300	----	42.8 ⁽³⁾	---	11.0	--
Fuel Tube	Longitudinal entire tube	343	23.1	30.5	---	22.4	--
Bar Stock	Longitudinal Tensile	400	26.0	31.0	5.7	37.4	79
Fuel Tube	Longitudinal Tensile cut section	400	24.4	30.0	4.2	20.4	65
Fuel Tube	Circumferential burst	400	----	32.5	---	10.0	--
Bar Stock	Longitudinal Tensile	500	18.3	21.6	4.1	51.7	97
Fuel Tube	Longitudinal Tensile cut section	500	18.8	21.1	2.3	28.3	91
Bar Stock	Longitudinal Tensile	600	10.6	12.9	4.8	52.6	~100
Fuel Tube	Longitudinal Tensile cut section	600	7.7	10.0	5.2	57.7	97
Fuel Tube	Circumferential burst	600	----	11.1	---	45.0	--
Zircaloy-2	Tensile ⁽⁴⁾	25	>42	>49		>16	
Zircaloy-2	Tensile	343	>22.5	----		----	

(1) All tensile tests at 0.02 min⁻¹ strain rate.

(2) Hoop stress based on original tube dimensions.

(3) Failure occurred after 7 minutes at pressure of 6.72 kg/mm²

(4) Specified values for stress-relieved Zircaloy-2.

SECTION V

IRRADIATION PROGRAM

The prime objective of this program is to evaluate Zr-Cr clad fuel in Consumers Big Rock Point power plant to a target of from 15,000 to 20,000 MWd/TU average burnup. These rods are positioned in a reload fuel assembly designated B-01 so that they operate as hot as possible to evaluate maximum temperature irradiation conditions. Detailed post-irradiation examinations will be performed to evaluate fuel performance as compared to Zircaloy-2 control fuel irradiated in the same bundle.

5.1 MATERIALS

Two specific materials were used in this experiment:

- a. Commercial Zr-Cr (Vendor Ingot No. 2)
- b. Zircaloy-2 (Heat No. WC-643)

A summary of the Zr-Cr tubing data was presented in Section IV of this report.

At the start of the irradiation Big Rock Point reload fuel assembly B-01 was unirradiated and available at the Big Rock Point site. Twenty-two fuel rods and four nonfueled dummy rods were used in the experiment and replaced 26 existing Zr-2 clad fuel rods in this bundle.

All 22 fuel rods, Figure 5-1, are similar to the reload fuel design for 2.6 percent enriched UO_2 pellet type fuel except that 16 were clad with Zr-Cr and six with annealed Zircaloy-2.

The external geometry of the dummy rods resembles the reload fuel rods and they are readily interchangeable with 2.6 percent enriched fuel rods. Each dummy rod, Figure 5-2, contains seven individual compartments. Three of these compartments are

nonvented and can be separated after irradiation for use as burst or stress rupture type specimens. These compartments also contain six tensile specimens in each, mounted on a tantalum rod. The four remaining compartments are vented by holes in the cladding to allow reactor water to come into contact with specimens inside. These specimens are also mounted on a tantalum rod and provide a means for nonheat transfer corrosion to be measured by weight gain. Autoclave tests in high oxygen steam at 300°C (572°F) (20 ppm O_2) show tantalum exhibits excellent corrosion resistance at reactor temperatures.

The basic design of these dummy rods is as simple as possible to facilitate easy access to tensile and corrosion specimens and provide post-irradiation tube samples for burst testing.

5.2 TEST VARIABLES

Although the prime purpose of this experiment is evaluation of Zr-Cr fuel cladding as a function of exposure, various other variables were incorporated into this experiment to make it more complete. These variables include pre-irradiation condition of cladding and the irradiation environment.

5.2.1 Exposure

The maximum target exposure is 15,000 to 20,000 MWd/T average burnup. However, both fuel and dummy rods will be removed on a scheduled reactor shutdown to obtain data at about 7500 MWd/TU average. Duplicate rods will be inserted to reconstitute the bundle and allow data at 12,500 MWd/TU exposure to be obtained when the target exposure is reached. The irradiation schedule and the various types of fuel and dummy rods for this experiment are shown in Table 5-1.

UNLESS OTHERWISE SPECIFIED USE THE FOLLOWING—				GENERAL ELECTRIC		107C5326	
APPLIED PRACTICES	SURFACES	TOLERANCES UNLESS OTHERWISE SPECIFIED	FRACCTIONS	DECIMALS	ANGLES	TITLE ASM	
145A5481	✓	—	—	—	—	107C5326 FUEL ROD	
FIRST MADE FOR FUEL BUNDLE						107C5326	
REV. NO.						REV. NO.	
PART NO.						PART NO.	
NAME						DRAWING NO., DESCRIPTION, MATERIAL, WEIGHT	
1						END PLUG 129B2141P1	
2						P2	
3						P3	
4						129B2141P4	
5						129B2140P1	
6						P2	
7						P3	
8						END PLUG 129B2140P4	
9						TUBE 129B2139P1	
10						P2	
11						P3	
12						TUBE 129B2139P4	
13						PELLET 107C5040P3	
14						PELLET 107C5040P4	
15						SPRING 159A2384P1 OR 129B3500G4	
16						WAFER .360±.008 DIA. ±.030 THK. B50YP19	
17						WAFER 104B24B2P3

(G1) (G2) (G3) (G4)

NOTES:

1. SCRATCHES, NICKS, ETC. INTRODUCED IN THE TUBING FABRICATION AND HANDLING SHALL NOT EXCEED .003 IN DEPTH.
2. NO MORE THAN 6" TOTAL OF PT. 14 MAY BE USED IN THE UPPER AND/OR LOWER 10% OF FUEL COLUMN OF EACH ROD TO MEET THE SPECIFIED LENGTH.
3. FILL VOIDS WITH GR. A He (99.995% PURE). HELIUM ESCAPE THROUGH TUBES AND END PLUGS OR WELDED REGIONS SHALL NOT EXCEED 10⁻⁸ STD. CM³/SEC. OF HELIUM WITH A VACUUM OF 20 MICRONS OF MERCURY.
4. ALL END PLUGS, CONNECTORS, SPRINGS, WAFERS, AND INTERNAL TUBE SURFACES SHALL BE FREE OF ALL VOLATILE CONTAMINANTS AT ASSEMBLY.
5. OUTSIDE DIAMETER OF END PLUG WELD SHALL NOT EXCEED .468. THICKNESS OF TUBING OR WELD AT WELD ZONE SHALL NOT BE LESS THAN .029.
6. MAXIMUM WATER VAPOR WEIGHT PLUS ALL OTHER GASEOUS PRODUCTS EVOLVED FROM PELLETS WHEN HEATED TO 1550°C SHALL NOT EXCEED 40x10⁻⁶ LITERS PER GRAM OF UO₂ (AT STANDARD CONDITIONS, 0°C AND 760 MM Hg).
7. FUEL WEIGHT (UO₂) PER ROD BETWEEN 1283 GRAMS MINIMUM AND 1320 GRAMS MAXIMUM. EXPECTED AVERAGE WEIGHT PER ROD OF ALL RODS SHALL BE 1297 GRAMS. WEIGHTS OF UO₂ ARE BASED UPON AN AVERAGE "STACKED" DENSITY OF 94.3% OF THEORETICAL, A MINIMUM "STACKED" DENSITY OF 93.3% AND A MAXIMUM "STACKED" DENSITY OF 96.0%.
8. G1 & G2 - P8G-YP90
G3 & G4 - LATER

DESCRIPTION OF GROUPS	REVISIONS	PRINTS TO
G1 - COLD DRAWN ZR-2	1. FEB. 19 67 M.H.T.	
G2 - ROCKED ZR-2	ADD. DWS. NO. ON DT.	
G3 - COLD DRAWN VALLOY	NO. 58	
G4 - ROCKED VALLOY	CHRD MAT'L. CALL OUT 0.2/0	
MADE BY K. Ochoa OCT. 1, 64	APED	107C5326
ISSUED K. S. Graham OCT. 7, 64	SAN JOSE	

FIGURE 5-1. DETAILS OF Zr-Cr AND ZIRCALOY-2 FUEL RODS

JINLESS OTHERWISE SPECIFIED USE THE FOLLOWING—				TOLERANCES ON MACHINED DIMENSIONS		TITLE ASM.	
APPLIED PRACTICES	SURFACES	FRACTIONS	DECIMALS	ANGLES			
45A5481	✓	+	+	+			107C5327
				DUMMY FUEL ROD		FIRST MADE FOR FUEL BUNDLE	
				DRAWING NO., DESCRIPTION, MATERIAL, WEIGHT			
				NAME			
				END PLUG		129B2144 P1	
						129B2144 P2	
						129B2144 P3	
						129B2144 P4	
						129B2138 P1	
						129B2138 P2	
						129B2138 P3	
				END PLUG		129B2138 P4	
				TUBE		129B2139 P1	
						129B2139 P2	
						129B2139 P3	
						129B2139 P4	
				RCD		129B2149 P1	
				TENSILE SPECIMEN		129B2145 P1	
						129B2145 P2	
						129B2145 P3	
				TENSILE SPECIMEN		129B2145 P4	
				WIRE		.020 DIA. WIRE (TYPE 302 STN. STL.) #7A23A

NOTES:

1. SCRATCHES, NICKS, ETC., INTRODUCED IN THE TUBING FABRICATION & HANDLING SHALL NOT EXCEED .003 IN DEPTH.
2. FILL VOIDS WITH GRA-4E (99.995% PURE) HELIUM. ESCAPE THROUGH TUBES & END PLUGS OR WELDED REGIONS SHALL NOT EXCEED 10^{-8} Std. Cm³/SEC. OF HELIUM WITH A VACUUM OF 20 MICRONS OF MERCURY.
3. ALL END PLUGS, TENSILE SPECIMENS, WIRES, AND ROD(P13) SURFACES SHALL BE FREE OF ALL VOLATILE CONTAMINANTS AT ASSEMBLY.
4. OUTSIDE DIAMETER OF END PLUG WELD SHALL NOT EXCEED .468 INCHES. THICKNESS OF TUBING OR WELD AT WELD ZONE SHALL NOT BE LESS THAN .029 INCHES.
5. G1 & G2 - PBG-YF90
G3 & G4 - LATER
6. TENSILE SPECIMENS MAY BE WIRED AT RANDOM.

G1 G2 G3 G4

DESCRIPTION OF GROUPS	REVISIONS	PRINTS TO
G1- COLD DRAWN Zr-2 G2- ROCKED Zr-2 G3- COLD DRAWN Zr-Cr G4- ROCKED Zr-Cr	1. 2. 3. 4. 5. 6. 7. 8. 9. 10. 11. 12. 13. 14. 15. 16. 17. 18. 19. 20. 21. 22. 23. 24. 25. 26. 27. 28. 29. 30. 31. 32. 33. 34. 35. 36. 37. 38. 39. 40. 41. 42. 43. 44. 45. 46. 47. 48. 49. 50. 51. 52. 53. 54. 55. 56. 57. 58. 59. 60. 61. 62. 63. 64. 65. 66. 67. 68. 69. 70. 71. 72. 73. 74. 75. 76. 77. 78. 79. 80. 81. 82. 83. 84. 85. 86. 87. 88. 89. 90. 91. 92. 93. 94. 95. 96. 97. 98. 99. 100. 101. 102. 103. 104. 105. 106. 107. 108. 109. 110. 111. 112. 113. 114. 115. 116. 117. 118. 119. 120. 121. 122. 123. 124. 125. 126. 127. 128. 129. 130. 131. 132. 133. 134. 135. 136. 137. 138. 139. 140. 141. 142. 143. 144. 145. 146. 147. 148. 149. 150. 151. 152. 153. 154. 155. 156. 157. 158. 159. 160. 161. 162. 163. 164. 165. 166. 167. 168. 169. 170. 171. 172. 173. 174. 175. 176. 177. 178. 179. 180. 181. 182. 183. 184. 185. 186. 187. 188. 189. 190. 191. 192. 193. 194. 195. 196. 197. 198. 199. 200. 201. 202. 203. 204. 205. 206. 207. 208. 209. 210. 211. 212. 213. 214. 215. 216. 217. 218. 219. 220. 221. 222. 223. 224. 225. 226. 227. 228. 229. 230. 231. 232. 233. 234. 235. 236. 237. 238. 239. 240. 241. 242. 243. 244. 245. 246. 247. 248. 249. 250. 251. 252. 253. 254. 255. 256. 257. 258. 259. 260. 261. 262. 263. 264. 265. 266. 267. 268. 269. 270. 271. 272. 273. 274. 275. 276. 277. 278. 279. 280. 281. 282. 283. 284. 285. 286. 287. 288. 289. 290. 291. 292. 293. 294. 295. 296. 297. 298. 299. 300. 301. 302. 303. 304. 305. 306. 307. 308. 309. 310. 311. 312. 313. 314. 315. 316. 317. 318. 319. 320. 321. 322. 323. 324. 325. 326. 327. 328. 329. 330. 331. 332. 333. 334. 335. 336. 337. 338. 339. 340. 341. 342. 343. 344. 345. 346. 347. 348. 349. 350. 351. 352. 353. 354. 355. 356. 357. 358. 359. 360. 361. 362. 363. 364. 365. 366. 367. 368. 369. 370. 371. 372. 373. 374. 375. 376. 377. 378. 379. 380. 381. 382. 383. 384. 385. 386. 387. 388. 389. 390. 391. 392. 393. 394. 395. 396. 397. 398. 399. 400. 401. 402. 403. 404. 405. 406. 407. 408. 409. 410. 411. 412. 413. 414. 415. 416. 417. 418. 419. 420. 421. 422. 423. 424. 425. 426. 427. 428. 429. 430. 431. 432. 433. 434. 435. 436. 437. 438. 439. 440. 441. 442. 443. 444. 445. 446. 447. 448. 449. 450. 451. 452. 453. 454. 455. 456. 457. 458. 459. 460. 461. 462. 463. 464. 465. 466. 467. 468. 469. 470. 471. 472. 473. 474. 475. 476. 477. 478. 479. 480. 481. 482. 483. 484. 485. 486. 487. 488. 489. 490. 491. 492. 493. 494. 495. 496. 497. 498. 499. 500. 501. 502. 503. 504. 505. 506. 507. 508. 509. 510. 511. 512. 513. 514. 515. 516. 517. 518. 519. 520. 521. 522. 523. 524. 525. 526. 527. 528. 529. 530. 531. 532. 533. 534. 535. 536. 537. 538. 539. 540. 541. 542. 543. 544. 545. 546. 547. 548. 549. 550. 551. 552. 553. 554. 555. 556. 557. 558. 559. 560. 561. 562. 563. 564. 565. 566. 567. 568. 569. 570. 571. 572. 573. 574. 575. 576. 577. 578. 579. 580. 581. 582. 583. 584. 585. 586. 587. 588. 589. 590. 591. 592. 593. 594. 595. 596. 597. 598. 599. 600. 601. 602. 603. 604. 605. 606. 607. 608. 609. 610. 611. 612. 613. 614. 615. 616. 617. 618. 619. 620. 621. 622. 623. 624. 625. 626. 627. 628. 629. 630. 631. 632. 633. 634. 635. 636. 637. 638. 639. 640. 641. 642. 643. 644. 645. 646. 647. 648. 649. 650. 651. 652. 653. 654. 655. 656. 657. 658. 659. 660. 661. 662. 663. 664. 665. 666. 667. 668. 669. 670. 671. 672. 673. 674. 675. 676. 677. 678. 679. 680. 681. 682. 683. 684. 685. 686. 687. 688. 689. 690. 691. 692. 693. 694. 695. 696. 697. 698. 699. 700. 701. 702. 703. 704. 705. 706. 707. 708. 709. 710. 711. 712. 713. 714. 715. 716. 717. 718. 719. 720. 721. 722. 723. 724. 725. 726. 727. 728. 729. 730. 731. 732. 733. 734. 735. 736. 737. 738. 739. 740. 741. 742. 743. 744. 745. 746. 747. 748. 749. 750. 751. 752. 753. 754. 755. 756. 757. 758. 759. 760. 761. 762. 763. 764. 765. 766. 767. 768. 769. 770. 771. 772. 773. 774. 775. 776. 777. 778. 779. 780. 781. 782. 783. 784. 785. 786. 787. 788. 789. 790. 791. 792. 793. 794. 795. 796. 797. 798. 799. 800. 801. 802. 803. 804. 805. 806. 807. 808. 809. 810. 811. 812. 813. 814. 815. 816. 817. 818. 819. 820. 821. 822. 823. 824. 825. 826. 827. 828. 829. 830. 831. 832. 833. 834. 835. 836. 837. 838. 839. 840. 841. 842. 843. 844. 845. 846. 847. 848. 849. 850. 851. 852. 853. 854. 855. 856. 857. 858. 859. 860. 861. 862. 863. 864. 865. 866. 867. 868. 869. 870. 871. 872. 873. 874. 875. 876. 877. 878. 879. 880. 881. 882. 883. 884. 885. 886. 887. 888. 889. 890. 891. 892. 893. 894. 895. 896. 897. 898. 899. 900. 901. 902. 903. 904. 905. 906. 907. 908. 909. 910. 911. 912. 913. 914. 915. 916. 917. 918. 919. 920. 921. 922. 923. 924. 925. 926. 927. 928. 929. 930. 931. 932. 933. 934. 935. 936. 937. 938. 939. 940. 941. 942. 943. 944. 945. 946. 947. 948. 949. 950. 951. 952. 953. 954. 955. 956. 957. 958. 959. 960. 961. 962. 963. 964. 965. 966. 967. 968. 969. 970. 971. 972. 973. 974. 975. 976. 977. 978. 979. 980. 981. 982. 983. 984. 985. 986. 987. 988. 989. 990. 991. 992. 993. 994. 995. 996. 997. 998. 999. 1000.	
MADE BY R. C. Carter OCT. 2, 1964	APPROVED C. J. Bowers OCT. 7, 1964	APPROVED A. P. E. D. SAN JOSE

FIGURE 5-2. DETAILS OF Zr-Cr AND ZIRCALOY-2 DUMMY RODS

TABLE 5-1

Zr-Cr FUEL IRRADIATION EXPERIMENT

Rod Description

Item	Cladding Alloy	Design Drawing	Pre-irradiation ^(a) Cladding Condition	Rod Type
1	Zr-Cr	107 C 5326-G4	1	Fuel Rod, 2.6% UO ₂ Pellet
2	Zr-Cr	107 C 5326-G4	2	Fuel Rod, 2.6% UO ₂ Pellet
3	Zr-Cr	107 C 5326-G4	3	Fuel Rod, 2.6% UO ₂ Pellet
4	Zr-Cr	161 F 263-G2	1, 2, and 3	Unfueled Dummy Rod
5	Zirc-2	107 C 5326-G2	1	Fuel Rod, 2.6% UO ₂ Pellet
6	Zirc-2	107 C 5326-G2	2	Fuel Rod, 2.6% UO ₂ Pellet
7	Zirc-2	107 C 5326-G2	3	Fuel Rod, 2.6% UO ₂ Pellet
8	Zirc-2	161 F 263-G1	1, 2, and 3	Unfueled Dummy Rod

Initial Loading

Item	Number of Rods	At 5,000 to 7,500 MWd/TU		At 10,000 to 12,500 MWd/TU		At 15,000 to 20,000 MWd/TU	
		Removed ^(b)	Replaced	Removed	Replaced	Removed	Replaced ^(c)
1	4	1 or 2	1 or 2	1 or 2	0	3 or 2	0
2	6	1 or 2	1 or 2	1 or 2	0	5 or 4	0
3	6	1 or 2	1 or 2	1 or 2	0	5 or 4	0
4	2	1	1	1	0	1	0
5	2	1	1	1	0	1	0
6	2	1	1	1	0	1	0
7	2	1	1	1	0	1	0
8	2	1	1	1	0	1	0

Fabrication Schedule

Item	Number of Rods to be Fabricated	Identification of 26 Initial Rods
1	4 plus 1 or 2 spares	FR4-1, FR4-2, FR4-3, and FR4-4
2	6 plus 1 or 2 spares	FR4-5, through FR4-10
3	6 plus 1 or 2 spares	FR-11, through FR4-16
4	2 plus 1 spare	DR1-1 and DR1-2
5	2 plus 1 spare	FR2-4 and FR2-5
6	2 plus 1 spare	FR2-6 and FR2-7
7	2 plus 1 spare	FR2-8 and FR2-9
8	2 plus 1 spare	DR2-3 and DR2-4

NOTES:

(a.) Both Zr-Cr and Zircaloy-2 tubing used in this experiment was rocked to size and annealed. The Zircaloy-2 was annealed, rocked tubing. Number designation refers to:

1. Cladding etched and cleaned.
2. Cladding etched, cleaned, and autoclaved at 300°C for 70 hours.
3. Cladding etched, cleaned, and autoclaved at 400°C for 12 hours.

(b.) Decision whether 1 or 2 rod removal will be made at 5000 to 7500 MWd/TU interim examination.

(c.) Final replacement rods will be made of whatever cladding condition proves best from interim examination.

5.2.2 Pre-irradiation Cladding Condition

Three pre-irradiation cladding conditions are being tested:

- a. As-etched.
- b. Low temperature autoclaved at 300°C for 70 hours.
- c. Standard temperature autoclaved at 400°C for 12 hours.

The testing of these initial cladding conditions was prompted by recent BNWL data⁽⁷⁾ where it was observed that in-reactor corrosion rates for Zircaloy-2 and Zr-Cr varied considerable with pre-irradiation conditioning. Since this variable has not been systematically tested in actual fuel rod experiments, it is considered equally important in this experiment.

5.2.3 Irradiation Environment

Three different environments will exist during irradiation testing:

- a. Heat transfer in reactor water (fuel cladding).
- b. Nonheat transfer in reactor water (corrosion samples in vented compartments in dummy rods).
- c. Nonheat transfer in helium (tensile samples in sealed compartments in dummy rods).

These environments will allow separation of effects, if any, caused by heat transfer

during irradiation on corrosion rates and mechanical properties.

The actual rod positions for the initial 26 rods are shown in Table 5-2 with relative power factors for each fuel rod. The Zr-Cr fuel rods were placed in the highest power positions available in the bundle to obtain the highest cladding temperature possible. In this manner, the Zr-Cr fuel cladding will be appropriately tested to evaluate corrosion resistance in-pile for comparison with that observed for this alloy in out-of-pile tests.

5.3 POST-IRRADIATION TESTS

The post-irradiation examinations will be performed on specific rods both in Big Rock Point fuel storage pool and in hot cells at the Vallecitos Laboratory. Interim nondestructive tests will include leak checking, visual examinations, and profilometry in Big Rock Point spent fuel pool. Selected rods will be sent back to Vallecitos for extensive destructive examination. These include weight gain measurements on pre-cut corrosion specimens, metallography for corrosion film measurement and determination of structural changes in cladding, and burst and tensile tests for measurements of irradiated cladding mechanical properties. Out-of-pile mechanical property and corrosion data are available from characterization studies for direct comparison with post-irradiation data.

TABLE 5-2

ROD LOCATIONS FOR Zr-Cr ALLOY EXPERIMENT
IN B-01 BUNDLE

Rod No.	Bundle Location	Relative Rod Power	Cladding Alloy	Cladding ⁽¹⁾ Condition
<u>Fuel Rods</u>				
FR2-4	a-4	1.14	Zircaloy-2	1
FR2-5	h-1	1.14	Zircaloy-2	1
FR2-6	e-1	1.10	Zircaloy-2	2
FR2-7	k-8	1.14	Zircaloy-2	2
FR2-8	g-11	1.10	Zircaloy-2	3
FR2-9	d-11	1.14	Zircaloy-2	3
FR4-1	c-1	1.22	Zr-Cr	1
FR4-2	k-3	1.22	Zr-Cr	1
FR4-3	i-11	1.22	Zr-Cr	1
FR4-4	a-9	1.22	Zr-Cr	1
FR4-5	d-1	1.14	Zr-Cr	2
FR4-6	k-4	1.14	Zr-Cr	2
FR4-7	h-11	1.14	Zr-Cr	2
FR4-8	a-8	1.14	Zr-Cr	2
FR4-9	b-2	1.16	Zr-Cr	2
FR4-10	j-10	1.16	Zr-Cr	2
FR4-11	i-1	1.22	Zr-Cr	3
FR4-12	k-9	1.22	Zr-Cr	3
FR4-13	c-11	1.22	Zr-Cr	3
FR4-14	a-3	1.22	Zr-Cr	3
FR4-15	j-2	1.16	Zr-Cr	3
FR4-16	b-10	1.16	Zr-Cr	3
<u>Dummy Rods</u>				
DR2-3	f-1	0	Zircaloy-2	1, 2, and 3
DR2-4	f-11	0	Zircaloy-2	1, 2, and 3
DR4-1	a-6	0	Zr-Cr	1, 2, and 3
DR4-2	k-6	0	Zr-Cr	1, 2, and 3

(1) See note a. in Table 5-1.

SECTION VI

DISCUSSION

The superior corrosion resistance of the Zr-Cr alloy after large-scale fabrication of tubing and bar stock has been demonstrated. The process used for tube fabrication is quite similar to that for the Zircalloys and thus negligible increases in cost are expected for production of Zr-Cr tubing. There is, however, some specific process control required to ensure that some Cr is in solid solution before the cold reductions and annealing steps; that is, the complete homogenization of forged material must be accomplished before extrusion to tube shells. The practice of post-extrusion annealing before cold reduction is not necessary nor desirable for the Zr-Cr alloy since exaggerated grain growth occurs during this anneal. Two experiments were performed to determine the temperature range required to obtain exaggerated grain growth and to determine the degree of cold deformation necessary to eliminate it.

The first experiment determined the temperature range at which the excessive grain growth occurs in as-extruded vendor material. Samples from one extrusion were vacuum-annealed at 500°, 600°, 700°, and 750°C for 4 hours. Hardness measurements taken before and after annealing indicated that very little, if any, structural changes occurred during annealing at temperatures up to 600°C. Above 600°C, a drop in hardness occurred which was related to an increase in grain size.

This experiment indicated that exaggerated grain growth will occur during annealing of vendor extrusions at temperatures above 700°C (~1300°F) and, therefore, this initial alpha-anneal was deleted from the vendor tubing process.

The second experiment was aimed at determining the effectiveness of cold reduction in reducing the grain growth that occurred. A sample with varying degrees of cold reduction was obtained in a tapered section of one extrusion which was stopped part way through the first reduction pass. A longitudinal section cut from the tapered tube was alpha-annealed at 704°C (1300°F) for 4 hours. Metallography of the tapered section revealed that greater than 15 percent reduction was necessary to start reducing the grain growth in the extrusions, and 40 percent was necessary to refine the annealed grain size to that typical for this alloy in the alpha-annealed condition. Therefore, all tube reductions including the first pass were maintained at greater than 50 percent reduction in area.

Subsequent tapered sections from each tube reduction were alpha-annealed and examined metallographically. No grain growth resulted in any of these tapered sections. The cracking problems arising from cold reduction of the initial extrusions were a result of the grain growth that occurred during the alpha-anneal prior to the first tube reduction. The breaking down of this large grain structure is very difficult to achieve with further tube reduction, as shown by extensive surface cracking during the later reductions.

The irradiation program described earlier has been initiated with the insertion of 26 experimental fuel and dummy rods into Consumers Big Rock Point reactor. The irradiation began on June 13, 1967. The experimental fuel rods accumulate approximately 800 MWd/TU burnup per month (720 hours) of full power operation. These fuel rods will reach an exposure of between 5000 to 7500 MWd/TU during the first quarter of 1968.

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ACKNOWLEDGMENTS

The authors gratefully acknowledge the technical support given this program by the many contributors. Particularly appreciated were the discussions and data given by Al Reisen of Wah Chang Corporation regarding

the melting and hot working operations, Jack Huber of Wolverine Tube on the warm extrusion operations, and Ed Barack and Don Hanens of Wah Chang Corporation for straightening the finished tubes.

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