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METALLOGRAPHIC STANDARDS FOR ESTIMATING HYDROGEN CONTENT OF ZIRCALOY-4 TUBING
(LWBR Development Program)

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NOTICE

FOREWARD

The Shippingport Atomic Power Station located in Shippingport, Pennsylvania was the first large-scale, central-station nuclear power plant in the United States and the first plant of such size in the world operated solely to produce electric power. This program was started in 1953 to confirm the practical application of nuclear power for large-scale electric power generation. It has provided much of the technology being used for design and operation of the commercial, central-station nuclear power plants now in use.

Subsequent to development and successful operation of the Pressurized Water Reactor in the Atomic Energy Commission (now Department of Energy, DOE) owned reactor plant at the Shippingport Atomic Power Station, the Atomic Energy Commission in 1965 undertook a research and development program to design and build a Light Water Breeder Reactor core for operation in the Shippingport Station.

The objective of the Light Water Breeder Reactor (LWBR) program has been to develop a technology that would significantly improve the utilization of the nation's nuclear fuel resources employing the well-established water reactor technology. To achieve this objective, work has been directed toward analysis, design, component tests, and fabrication of a water-cooled, thorium oxide-uranium oxide fuel cycle breeder reactor for installation and operation at the Shippingport Station. The LWBR core started operation in the Shippingport Station in the Fall of 1977 and will finish routine power operation on October 1, 1982. After End-of-Life core testing, the core will be removed and the spent fuel shipped to the Naval Reactors Expended Core Facility for detailed examination to verify core performance including an evaluation of breeding characteristics.

In 1976, with fabrication of the Shippingport LWBR core nearing completion, the Energy Research and Development Administration, now DOE, established the Advanced Water Breeder Applications (AWBA) program to develop and disseminate technical information which would assist U. S. industry in evaluating the LWBR concept for commercial-scale applications. The AWBA program, which is concluding in September 1982, has explored some of the problems that would be faced by industry in adopting technology confirmed in the LWBR program. Information already developed includes concepts for commercial-scale prebreeder cores which would produce uranium-233 for light water breeder cores while producing electric power, improvements for breeder cores based on the technology developed to fabricate and operate the Shippingport LWBR core, and other information and technology to aid in evaluating commercial-scale application of the LWBR concept.

All three development programs (Pressurized Water Reactor, Light Water Breeder Reactor, and Advanced Water Breeder Applications) have been conducted under the technical direction of the office of Deputy Assistant Secretary for Naval Reactors of DOE.

Technical information developed under the Shippingport, LWBR, and AWBA pro-grams has been and will continue to be published in technical memoranda, one of which is this present report.

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METALLOGRAPHIC STANDARDS FOR ESTIMATING HYDROGEN CONTENT OF ZIRCALOY-4 TUBING

(LWBR Development Program)

B. Z. Hyatt

I. INTRODUCTION

Photomicrographs of hydrided LWBR Zircaloy-4 (recrystallized annealed) tubing were needed to provide accurate standards of comparison for rapidly estimating the hydrogen content of unirradiated and irradiated Zircaloy-4 fuel rod cladding. Previous photomicrographic hydride standards used in LWBR development were based on Zircaloy-2 bar or sheet material.

In the present study Zircaloy-4 tubing was exposed in LiOH solutions in various autoclave tests to promote corrosion and hydriding. Sections of the tubes were metallographically prepared for microstructural and hardness examinations, and sections adjacent to the metallographic samples were chemically analyzed for hydrogen content to provide calibration of the photographs.

II. EXPERIMENTAL PROCEDURE

Specimens were prepared for the controlled hydriding of Zircaloy-4 tubing in concentrated lithium hydroxide solutions in a series of autocalve exposures based on a previously established experimental technique (Reference 1). (It was assumed that the rate of cooling in the autoclave corrosion test approximated that of cooling in-reactor.)

LWBR recrystallized annealed seed tubing (similar to LWBR core seed rod tubing, P.O. 369044, Lot 8K, Tube 4B3B1) was cut into one inch lengths, vibratool engraved and then subjected to a standard LWBR pickle* removing one to two mils from the tube surfaces. Each tube specimen was then measured and weighed prior to the corrosion test. The corrosion test parameters or the autoclave times and temperatures for a particular LiOH solution were based on curves of weight gain (mg/dm^2) versus time as reported in Reference 1.

Multiple cycles of eight hour heating periods were applied in most of the autoclave exposures, since previous data developed for sheet specimens (Reference 1) had indicated that a uniform distribution of the hydride phase could be achieved throughout the metal substrate by multicycle exposure rather than by single cycle (in which case a nonuniform hydride distribution or a hydride rim would occur).

After the corrosion test, each one-inch specimen was weighed and then sectioned for metallographic examination and hydrogen analysis by hot vacuum extraction. Adjacent metallographic and hydrogen sections were cut from the central portion of each specimen. A final swab etchant composed of 60 volume percent H_2O_2, 40 volume percent HNO_2, and 6 drops of HF was used after the alternate repolishing and re-etching of each metallographic section.

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* Pickle consists of acid etch in a hot (100F) solution of 3.0 volume percent hydrofluoric acid, 39 volume percent nitric acid, balance water.

Transverse tube sections were selected for metallographic examination over longitudinal tube sections because transverse sections show edge views of the hydride platelets, which tend to be oriented parallel to the longitudinal direction (Reference 2). Because longitudinal sections generally show hydrides in clusters and patches, it was judged that they would not provide microstructures that are easily used for comparisons. These considerations are limited to recrystallized annealed tubing with textures such that basal planes and hydride habit planes are parallel to the tube axis. Tubing variations which change the radial to tangential basal plane texture should affect hydride platelet orientation but should not change the visible quantity of hydride platelets.

III. RESULTS AND DISCUSSION

Tables I, II, and III list the test specimens, cycles of autoclave exposure to the LiOH solutions, the weight gains, total and corrected hydrogen*, and Vicker's hardness. Table IV lists similar test results for as received and preconditioned tubing.

Figures 1 to 28 show the resulting metallographic structures of the transverse tube sections for hydrogen contents in the range of 10 to 11,600 ppm**. Each of these figures presents a 100X and either a 250X or a 500X magnification photograph and indicates total hydrogen content and the Vicker's hardness.

Microstructure of as-received (noncorrosion tested) tubing containing 10 ppm of hydrogen, shown in Figure 1, etched as a peppery structure devoid of hydride platelets. However, tubing exposed to normal LWBR preconditioning treatment (3.5 days at 650°F, NH_4OH water at pH 9 to 10.2 at 25°C) had an

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* See Appendix for the hydrogen correction. This correction was made to account for the oxide weight.

** Based on available hydrogen standards the estimated uncertainty in hydrogen determination is ± 10% of the value reported.

increased hydrogen content of 25 ppm, and developed the characteristic irregular black line segments or hydride platelets shown in Figure 2. At progressively greater hydrogen contents, the proportions of hydride in the metal increased and the characteristic irregular segments of platelets of hydride developed into a continuous elongated network (at approximately 560 ppm, Figure 11). At hydrogen levels above 1000 ppm, the hydride network became more continuous, and widths of the platelets become larger. Note that Figures 15-18 show structures that have a greater concentration of hydride at the surface than in the central region of the specimen; this rimming effect appears to be characteristic of specimens only subjected to one or a few cycles of corrosion. With multiple cycles of exposure (Figure 22), the cellular network became still finer, and the hydride platelets resembled a basket weave structure. At hydrogen levels of 6100 ppm to 11,600 ppm (Figures 25 to 28) the hydride structure was characterized by a feathery white network which is likely the highest hydrogen phase (epsilon).

Hydride microstructures (Figures 5 and Figure 10) of unirradiated Zircaloy-4 tubing with hydrogen contents of 100 ppm and 560 ppm respectively were similar to the hydride microstructure (Figure 29) of an irradiated Zircaloy-4 tubing specmen with measured hydrogen content of 64 ppm and with the hydride microstructure (Figure 30 a,b) of an unirradiated Zircaloy-4 sheet specimen with measured hydrogen content of 580 ppm.

The Vicker's hardness (at 2 Kg load) versus the hydrogen content (corrected for oxide) is shown in Figure 31. Several changes in slope are indicated which are most likely related to changes in the distribution of hydride. In the 1000 to 3000 ppm hydrogen range, the microstructures indicate some rimming and no significant increase of hydride in the center portions of the tube microstructures. This may account for the shallow change in slope. However, in the 3500 to 6000 ppm hydrogen range, there is a significant increase in hardness that represents the increase in hydride density and change in hydride morphology. Above 6000 ppm hydrogen the hardness again forms a shallow slope and reflects the hardness of the highest hydrogen phase.

Presented in Figures 32, 33, and 34 are weight gains (mg/dm^2) versus total hours of autoclave exposure for the Zircaloy-4 tubes. Corrosion rates, slopes of the least square fit of the data, and standard deviations of the slopes for the three different corrosion environments are given in Table V. The corrosion rates determined for the annealed Zircaloy-4 tubes were found to be comparable to the rates developed for the annealed Zircaloy-4 sheet coupons as reported in Reference 1.

The appendix illustrates the correction made to the measured total hydrogen content of each heavily corroded Zircaloy-4 tube specimen to account for oxide weight. It was assumed that there was no hydrogen in the oxide. Corrected hydrogen values are given in Table III.

IV. SUMMARY AND CONCLUSION

Metallographic standards for estimating hydrogen content of LWBR Zircaloy-4 tubing were prepared by corrosion of tubing specimens in LiOH solution. Photographs of specimens with total hydrogen contents that vary from 10 ppm to 11,600 ppm are presented.

Microstructures of an irradiated Zircaloy-4 tubing specimen with measured hydrogen content of 60 PPM and an unirradiated Zircaloy-4 sheet specimen with measured hydrogen content of 580 PPM were in good agreement with the microstructures and hydrogen contents of several of the standards.

Vickers hardness data and corrosion weight gain data for the Zircaloy-4 tubing specimens are presented.

ACKNOWLEDEMENTS

The author gratefully acknowledges the review of this report by J. J. Kearns, E. Hillner, P. L. Pfennigwerth, M. M. Hall, and S. D. Harkness. R. R. Schorr prepared and beautifully photographed all of the corrosion specimens. L. A. Waldman and J. Sherman provided the photograph of the irradiated hydrided Zircaloy-4 blanket tubing specimen. B. F. Kammenzind provided the photograph of the unirradiated hydrided Zircaloy-4 sheet specimen.

REFERENCES

1. S. Kass, "Corrosion and Hydrogen Pickup of Zircaloy in Concentrated Lithium Hydroxide Solutions," WAPD-TM-656, October 1967.

2. J. J. Kearns and C. R. Woods, "Effect of Texture, Grain Size and Cold Work on the Precipitation of Oriented Hydride in Zircaloy Tubing and Plate," J. Nucl. Matls. 20 (1966) 241.

APPENDIX

HYDROGEN CORRECTION

The correction made to measured total hydrogen content of each heavily corroded Zircaloy-4 tube specimen to account for oxide weight is illustrated below. It was assumed that there was no hydrogen in the metal oxide; however recent research (Reference A) showed that some hydrogen was detected in oxide films on Zircaloy-2.

$$H_{met} = H_{spec} \left[\frac{wt\ spec}{wt\ met}\right] = H_{spec} \frac{(\rho V)_{oxide} + (\rho V)_{met}}{(\rho V)_{met}}$$

where

H = hydrogen concentration (ppm)

wt = weight (gm)

ρ = density (gm/cc)

V = volume (cc)

$_{oxide}$ = 5.73 gm/cc

$_{metal}$ = 6.54 gm/cc

Because the length of each specimen is the same for both oxide and metal, the ratio of areas (oxide/metal) may be substituted for the volume ratio. Oxide and metal thickness measurements were made on polished metallographic mounts by using a toolmakers microscope (with an accuracy of ± 0.0005 inch).

Reference

A. I. S. Woolsey and J. R. Morris, "A Study of Zircaloy-2 Corrosion in High Temperature Water Using Ion Beam Methods," Corrosion, Volume 37, No. 10, October 1981, p. 241

TABLE I

Corrosion Data for 600°F 0.3N LiOH Solution

		Hours per Cycle / Total Hours	Identification of Specimen Removed From Test	Total Weight Gain / Surface Area (mg/dm^2)	As Reported Hydrogen (Rounded) (ppm)	Vickers Hardness (2 kg load)
Run A		8	2	13.5	47	165
	Total	24	3	15.5	(50)	
Run B		8	4	19.9	76	167
	Total	32	5	20.7	(80)	
Run C		8	6	23.8	101	165
		40	7	25.8	(100)	
Run D		8	8	38.5	153	165
	Total	64	9	42.3	(150)	

TABLE II

Corrosion Data for 600°F 0.7N LiOH Solution

		Hours per Cycle / Total Hours	Identification of Specimen Removed From Test	Total Weight Gain / Surface Area (mg/dm^2)	As Reported Hydrogen (Rounded) (ppm)	Vickers Hardness (2 kg load)
Run A		8	10	44.8	225	169
	Total	24	11	45.9	(220)	
Run E		8	11	146.2	646	177
	Total	72			(650)	
Run B		8	12	61.3	331	168
	Total	32	13	62.1	(330)	
Run F		8	13	147.1	611	179
	Total	72			(610)	
Run C		8	14	80.9	390	169
	Total	40	15	81.3		
		8	15	153.2	710	179
	Total	112				
Run D		8	16	101.5	559	171
	Total	48	17	101.4	(560)	
Run G		8	17	138.7	570	178
	Total	64				

TABLE III

Corrosion Data for 680°F 0.7N LiOH Solution

	Hours per Cycle / Total Hours	Identification of Specimen Removed From Test	Total Weight Gain / Surface Area (mg/dm^2)	As Reported Hydrogen (ppm)	Corrected Hydrogen (ppm)	Vickers Hardness (2 kg load)
Run A	7	18	182.2	982	1000	171
		19	183.7			
Run B	8	20	378.4	2072	2200	183
Total	15	21	377.3			
Run C	8	22	569.1	3031	3400	195
Total	23	23	563.4			
Run D	8	24	760.3	3722	4200	210
Total	31	25	759.8			
Run E	8	26	957.6	4090	5000	225
Total	39	27	958.8			
Run F	8	28	1135.8	4419	5600	243
Total	47	29	1134.7			
Run G	8	30	1293.8	4806		273
Total	55	31	1297.3	4617	6200	259
Run H	8	32	1414.2	4799		
Total	63	33	1414.8	4639	6400	274
Run I	8	34	1567.7	7375	10,600	279
Total	71	35	1566.3			
Run J	8	36	1673.3			
Total	79	37	1668.1	7937	11,600	289
Run K	2					
	2					
	3					
Total	7	38	246.5	1140	1200	181
Run L	4	39	353.5	1779	1900	186
Total	11					
Run M	4	40	455.3	2100	2300	195
Total	15					
Run N	8	41	612.9	2581	2900	209
Total	23					

TABLE IV

Initial Hydrogen and Hardness Data

a) As Received Tubing

1. P.O. 369044, Lot 8K, Tube 4B3B1

Specimen No.	Hydrogen ppm	Vickers Hardness (2 kg load)
1C	10	164

b) Preconditioned Tubing

3.5 days at 650°F NH_4OH water pH @ 25°C, 9-10.2

1. Same as a)

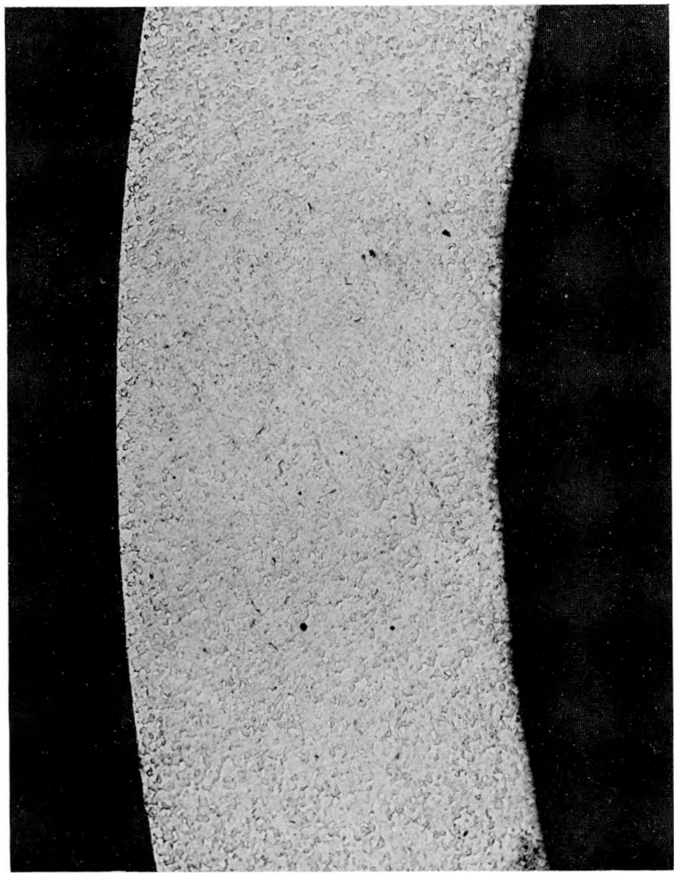
Specimen No.	Total Weight Gain / Surface Area (mg/dm^2)	Hydrogen ppm	Vickers Hardness (2 kg load)
42C	8.9	20	163
43C	7.9	25	163

TABLE V

Summary of Corrosion Data

Test Temp.	Normality of LiOH	Weight Gain Rate ($mg/dm^2/hr$)	Standard Deviation of Rate
600°F	0.3	.6	.03
600°F	0.7	2.2	.02
680°F	0.7	22.0	.16

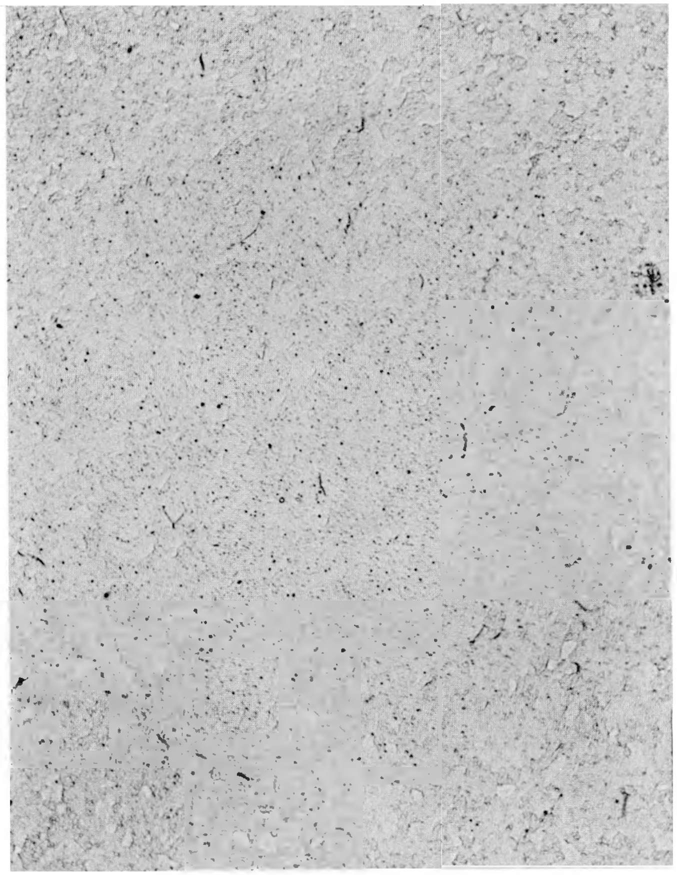
ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION
(AS RECEIVED - NOT CORROSION TESTED)



100X

10 ppm H_2

VICKERS HARDNESS
164
(2 Kg LOAD)



250 X

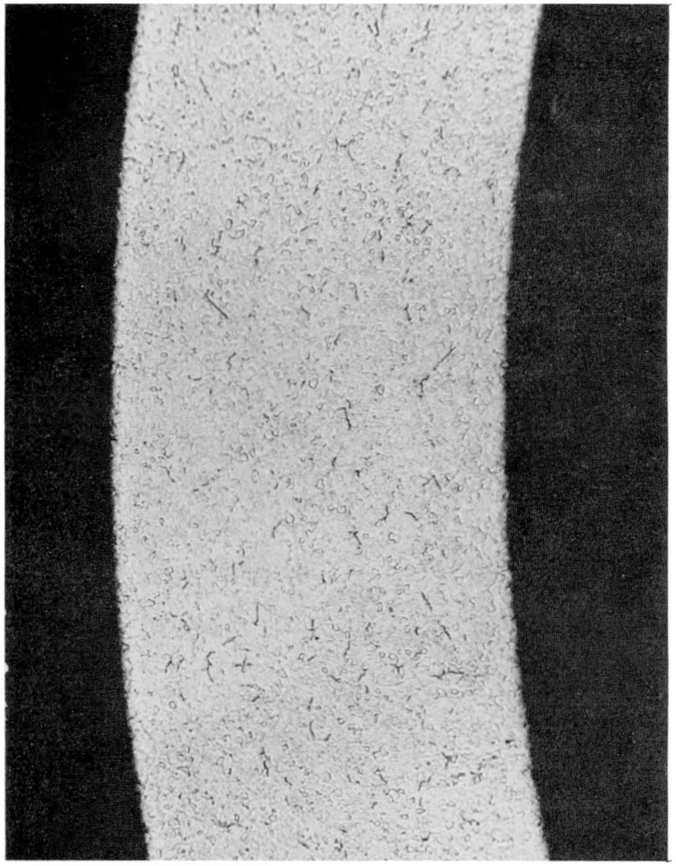
MOUNT NO. 4642 J
SPECIMEN NO. 1C

FIGURE 1

ZIRCALOY 4 CORROSION–HYDRIDE SPECIMEN

LWBR SEED TUBING TRANSVERSE SECTION

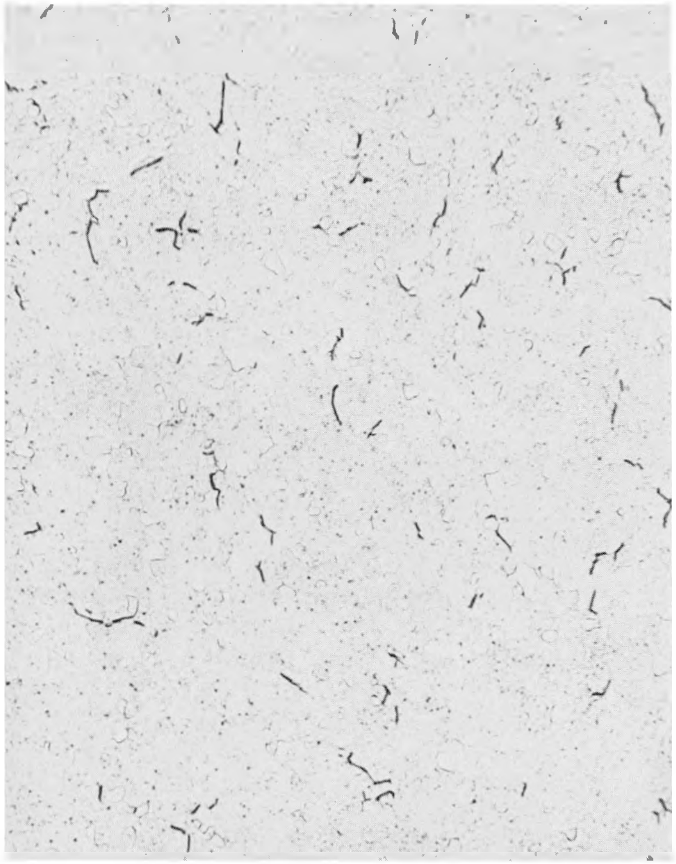
3½ DAYS AT 650°F NH_4OH WATER (pH AT 25°C 9.0-10.1)



100 X

25 ppm H_2

VICKERS HARDNESS
163
(2 Kg LOAD)

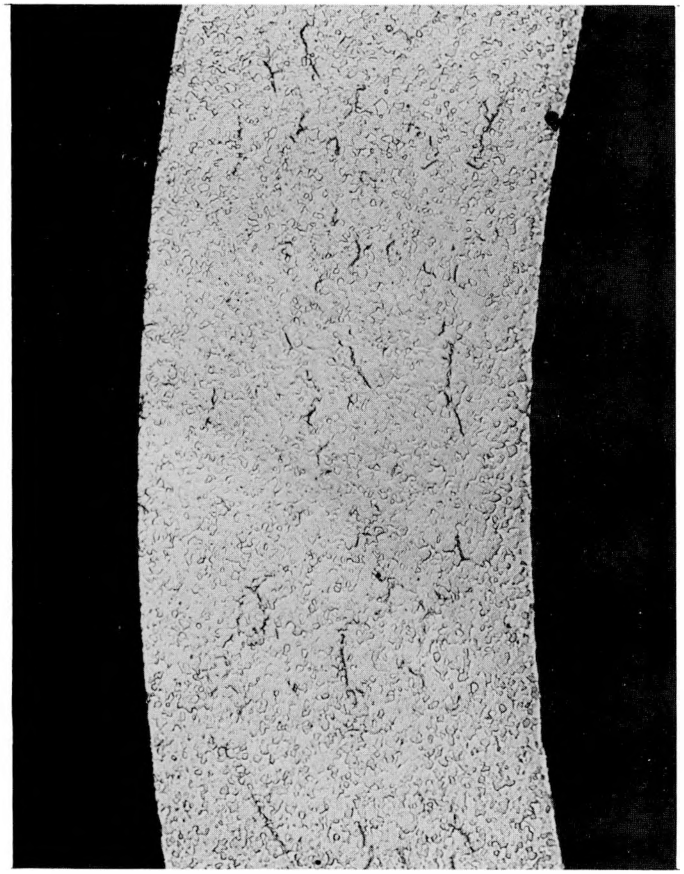


250X

MOUNT NO. 5014 J
SPECIMEN NO. 43C

FIGURE 2

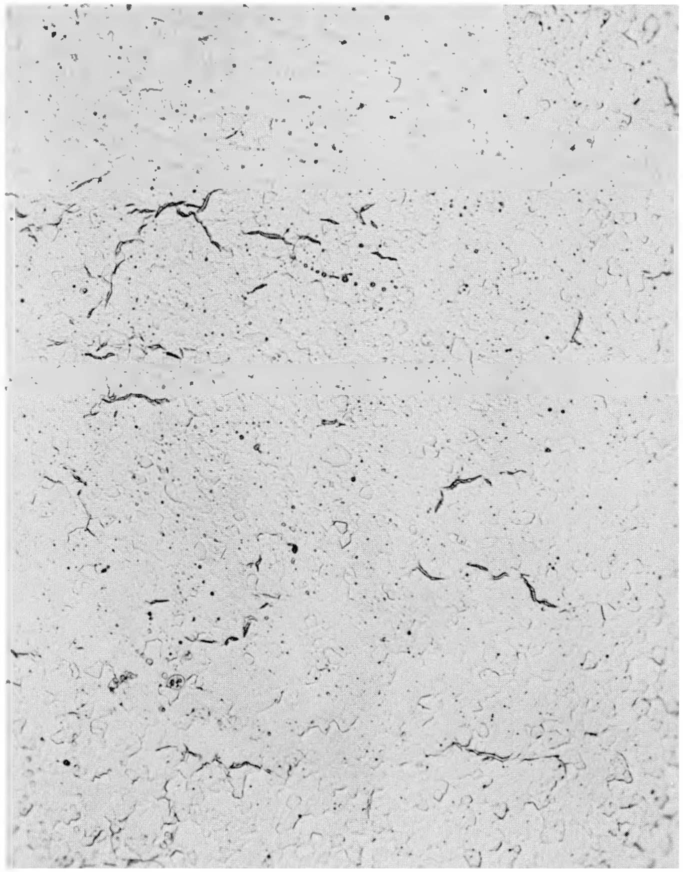
ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100 X

50 ppm H_2

VICKERS HARDNESS
165
(2 Kg LOAD)

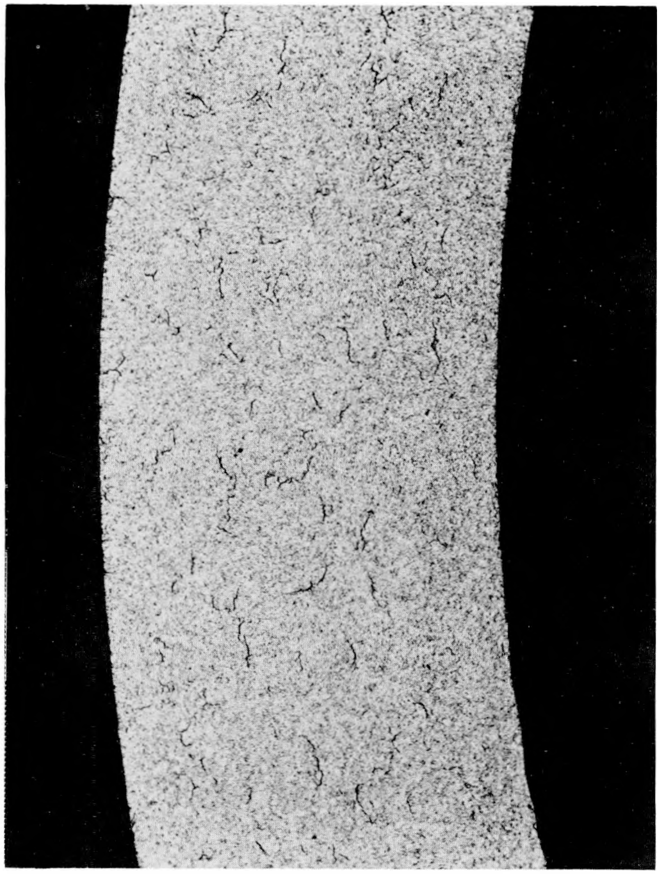


250 X

MOUNT NO. 4641J
SPECIMEN NO. 2C

FIGURE 3

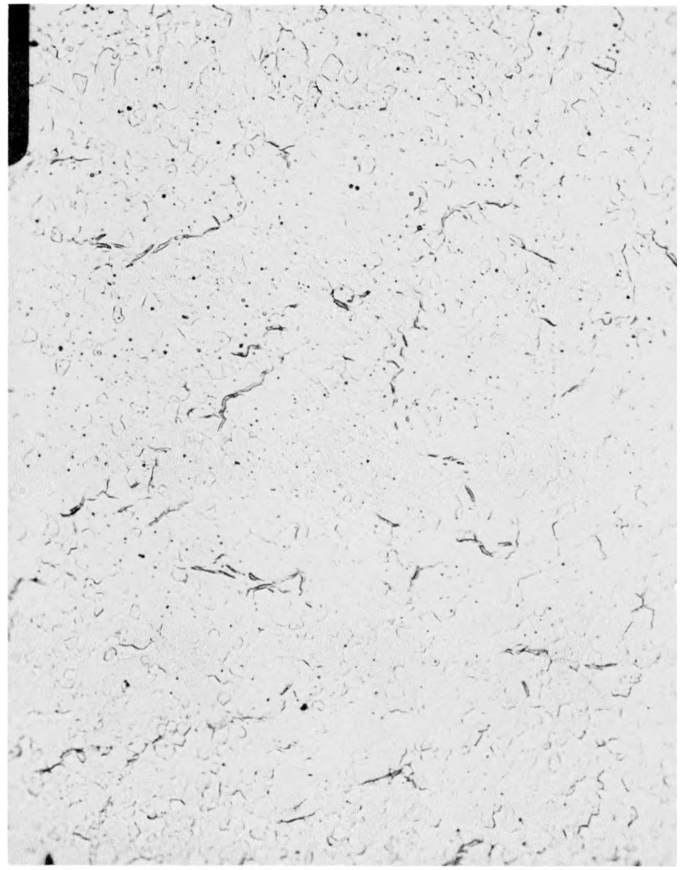
ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100 X

80 ppm H_2

VICKERS HARDNESS
167
(2 Kg LOAD)

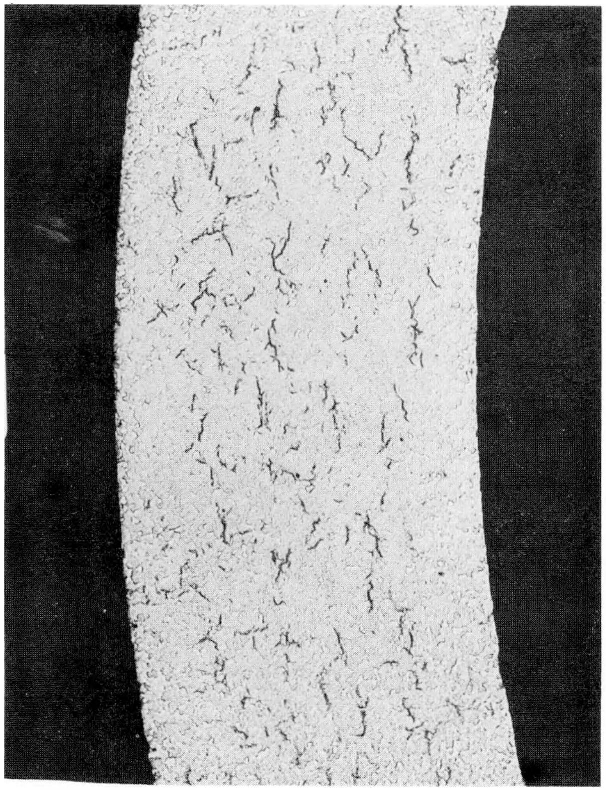


250 X

MOUNT NO. 4640 J
SPECIMEN NO. 4C

FIGURE 4

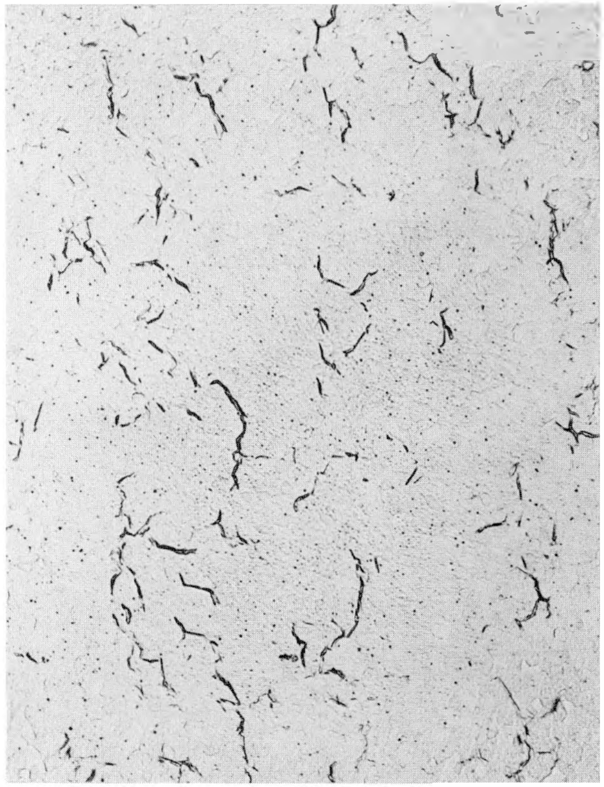
ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100X

100 ppm H_2

VICKERS HARDNESS
165
(2 Kg LOAD)

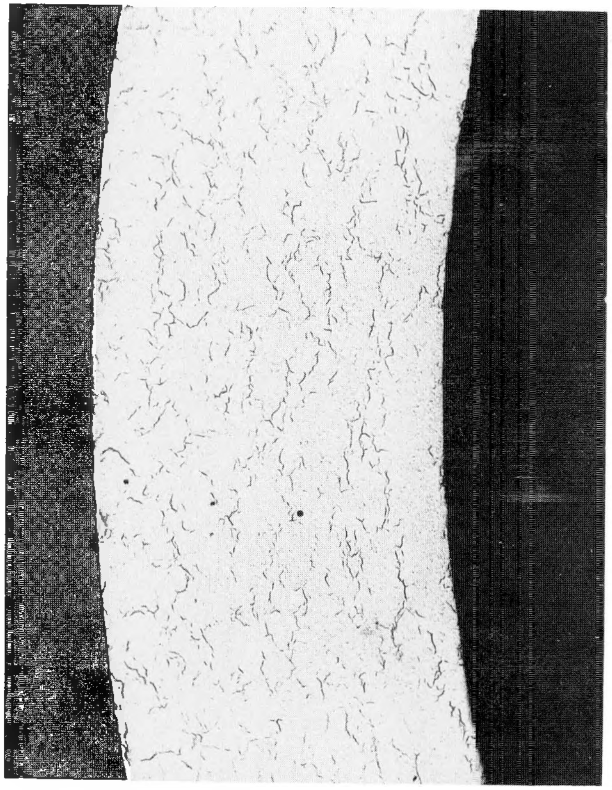


250X

MOUNT NO. 4648 J
SPECIMEN NO. 6C

FIGURE 5

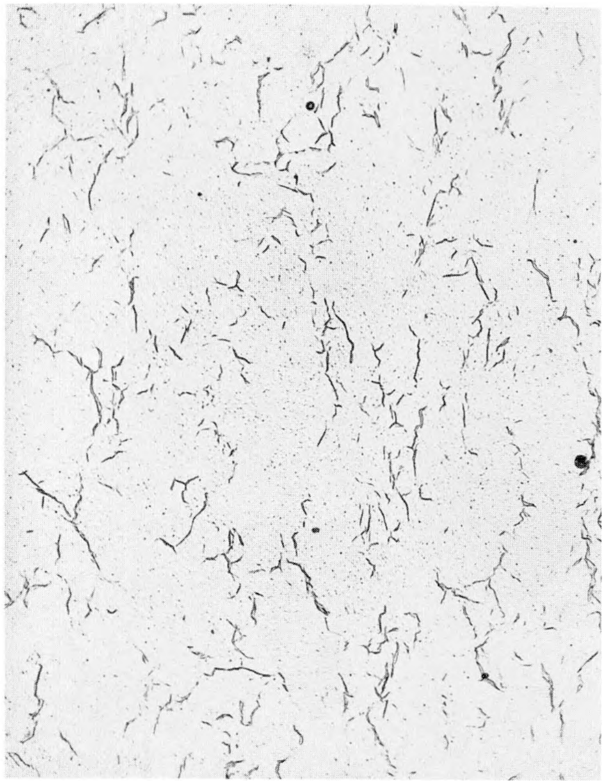
ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100 X

150 ppm H_2

VICKERS HARDNESS
165
(2 Kg LOAD)

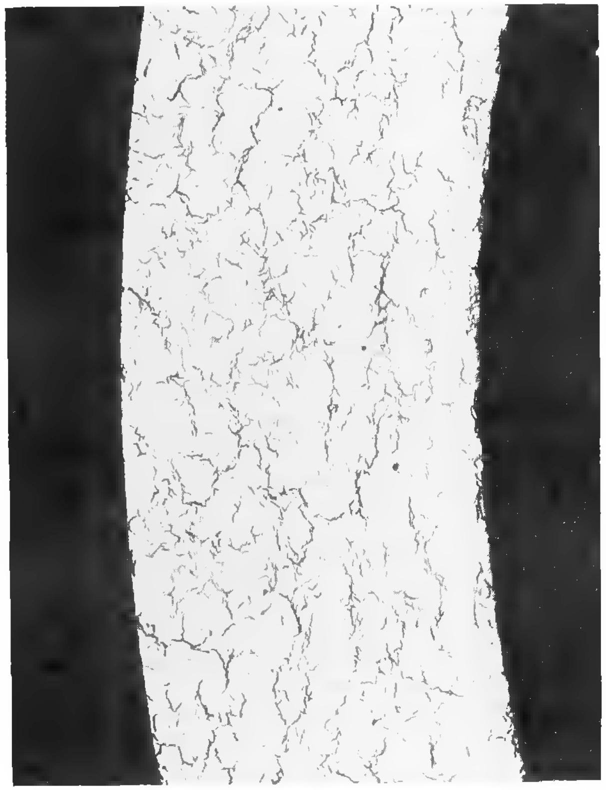


250 X

MOUNT NO. 4649 J
SPECIMEN NO. 8C

FIGURE 6

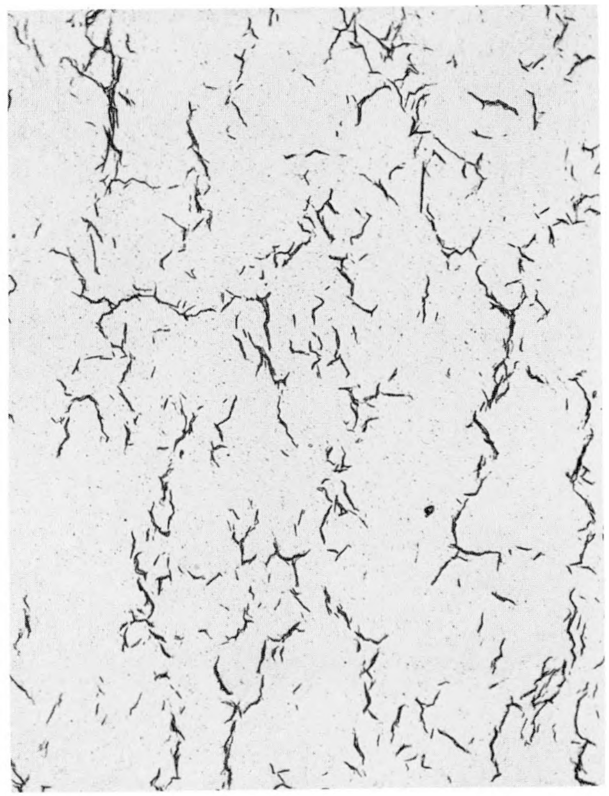
ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100X

220 ppm H_2

VICKERS HARDNESS
169
(2 Kg LOAD)

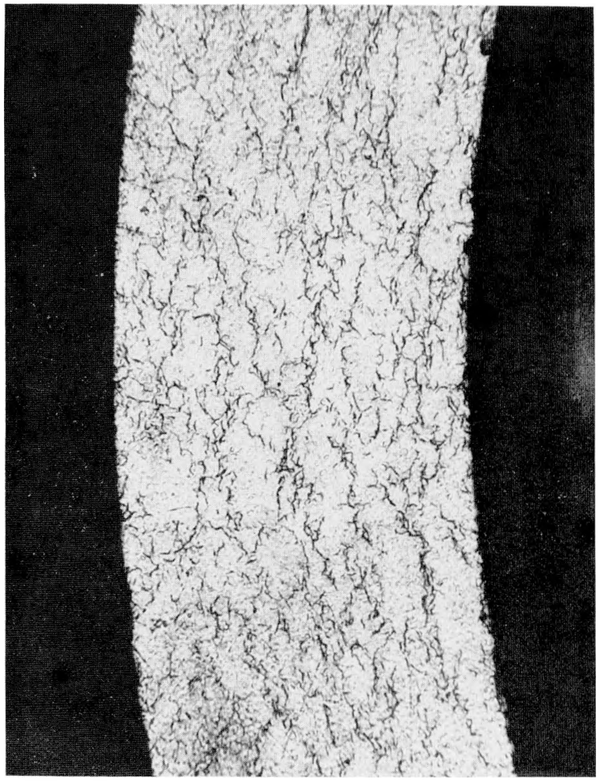


250X

MOUNT NO. 4686 J
SPECIMEN NO. 10C

FIGURE 7

ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100X

330 ppm H_2

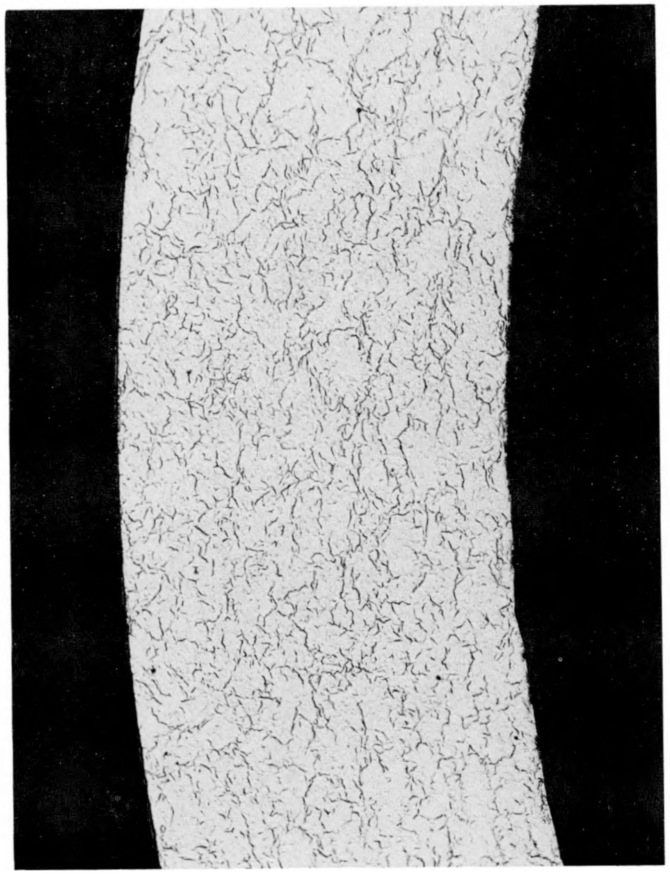
VICKERS HARDNESS
168
(2 Kg LOAD)

250X

MOUNT NO. 4684 J
SPECIMEN NO. 12C

FIGURE 8

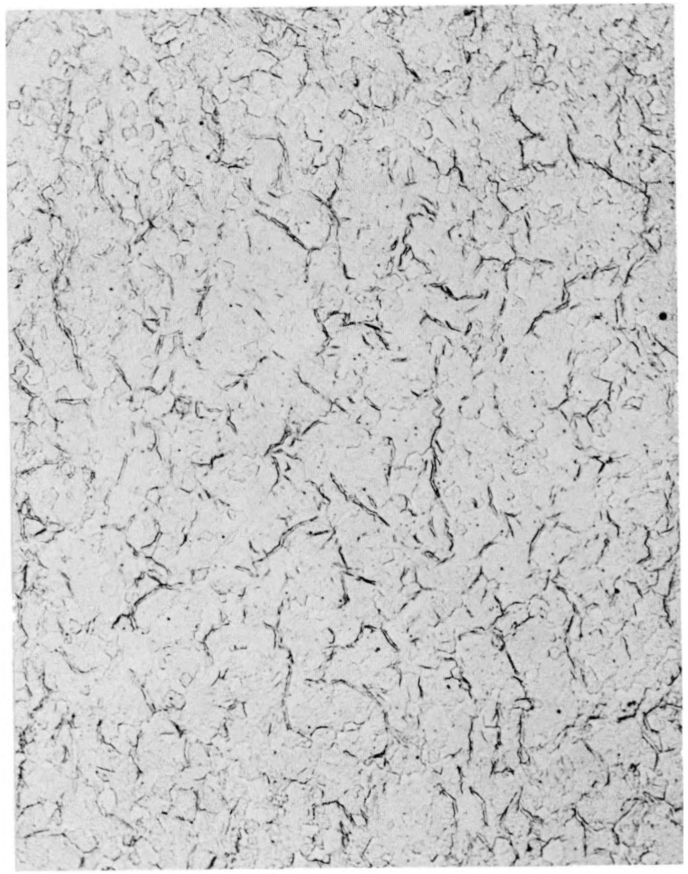
ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100X

400 ppm H_2

VICKERS HARDNESS
169
(2 Kg LOAD)

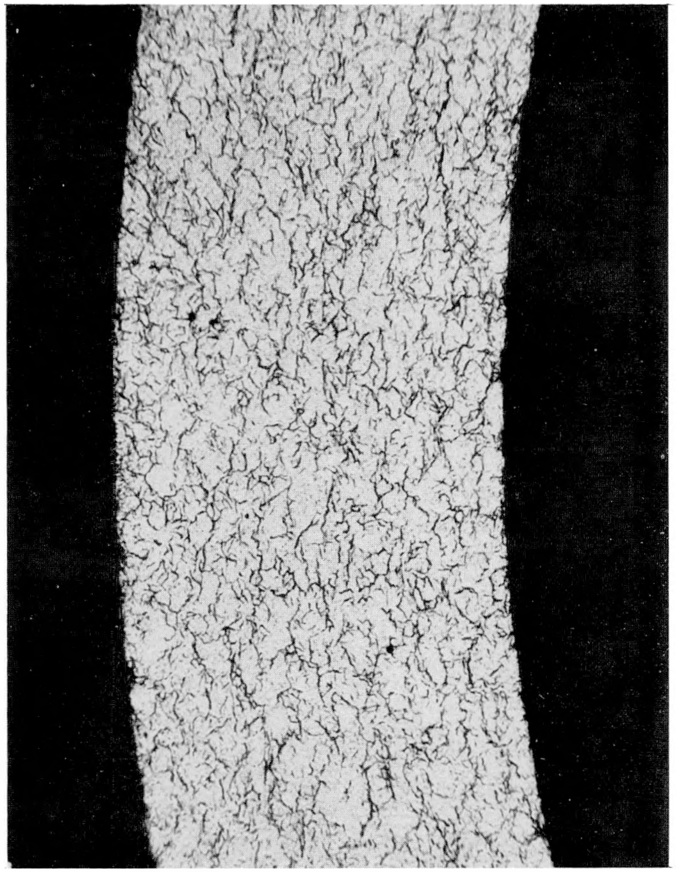


250X

MOUNT NO. 4683 J
SPECIMEN NO. 14C

FIGURE 9

ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100 X

560 ppm H_2

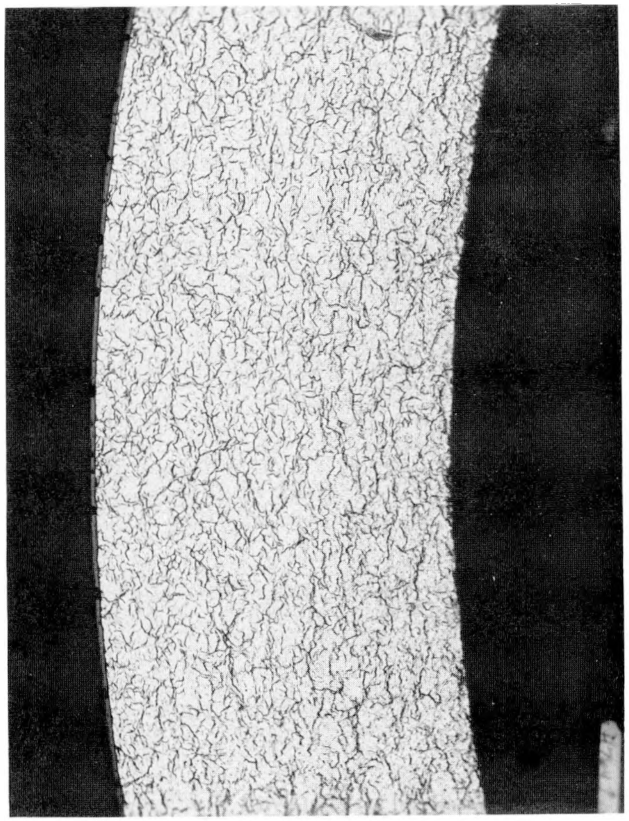
VICKERS HARNESS
171
(2 Kg LOAD)

250 X

MOUNT NO. 4687 J
SPECIMEN NO. 16C

FIGURE 10

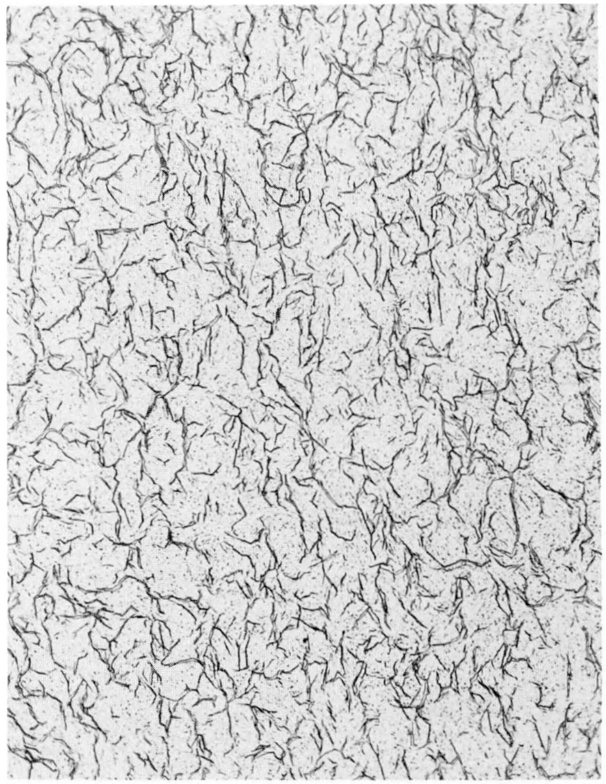
ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100 X

570 ppm H_2

VICKERS HARDNESS
178
(2 Kg LOAD)

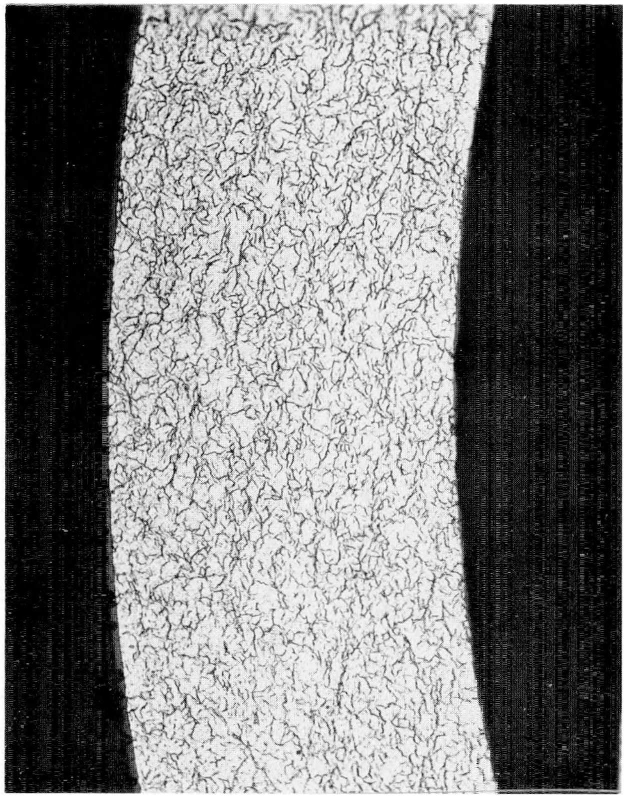


250 X

MOUNT NO. 4764 J
SPECIMEN NO. 17C

FIGURE 11

ZIRCALOY 4 CORROSION - HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100X

600 ppm H_2

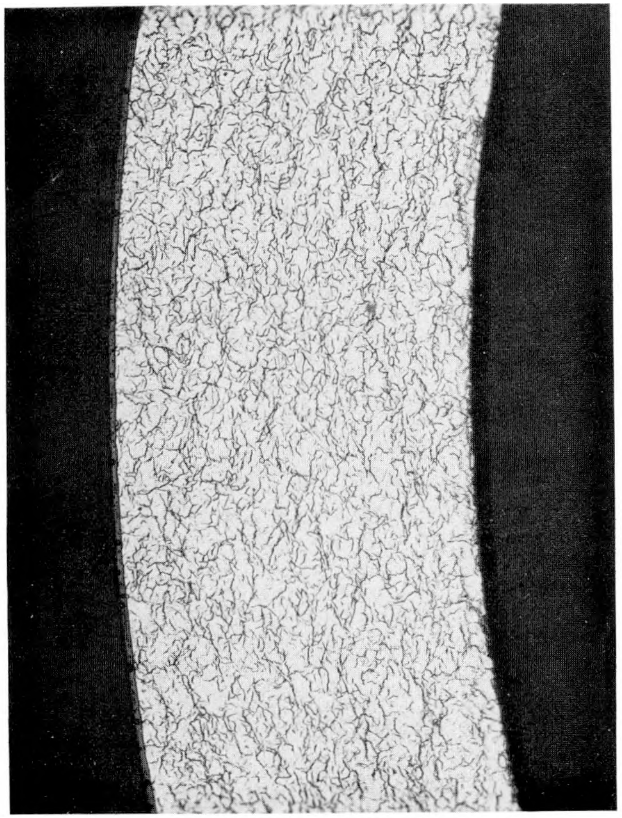
VICKERS HARDNESS
179
(2 Kg LOAD)

250X

MOUNT NO. 4762 J
SPECIMEN NO. 13C

FIGURE 12

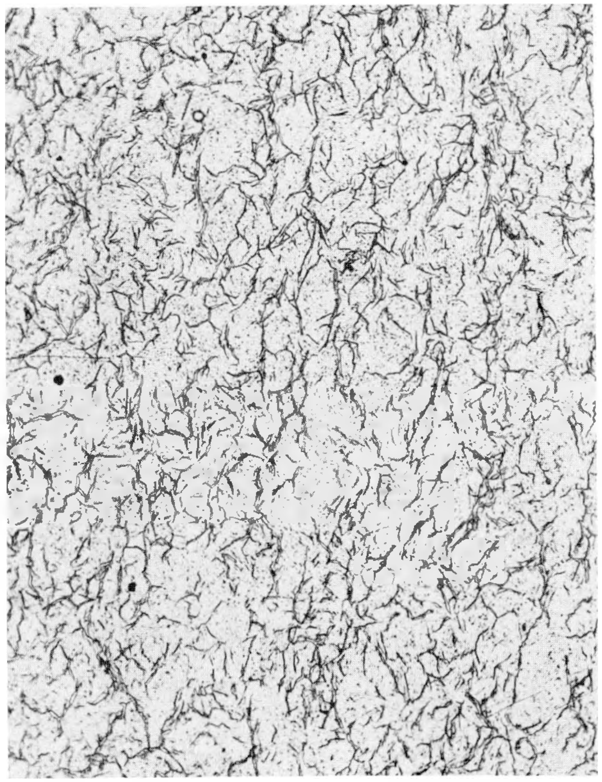
ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100 X

650 ppm H_2

VICKERS HARDNESS
177
(2 Kg LOAD)

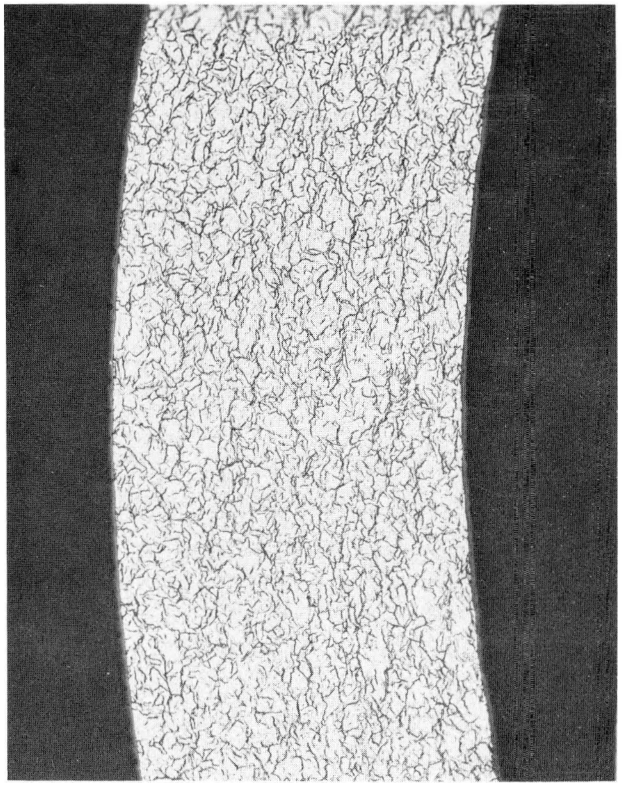


250 X

MOUNT NO. 4761 J
SPECIMEN NO. 11C

FIGURE 13

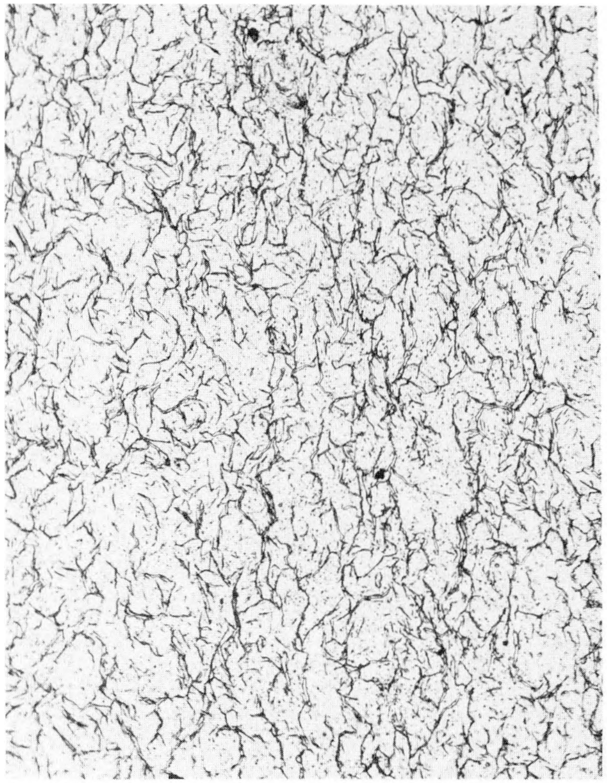
ZIRCALOY 4 CORROSION - HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100X

710 ppm H_2

VICKERS HARDNESS
179
(2 Kg LOAD)

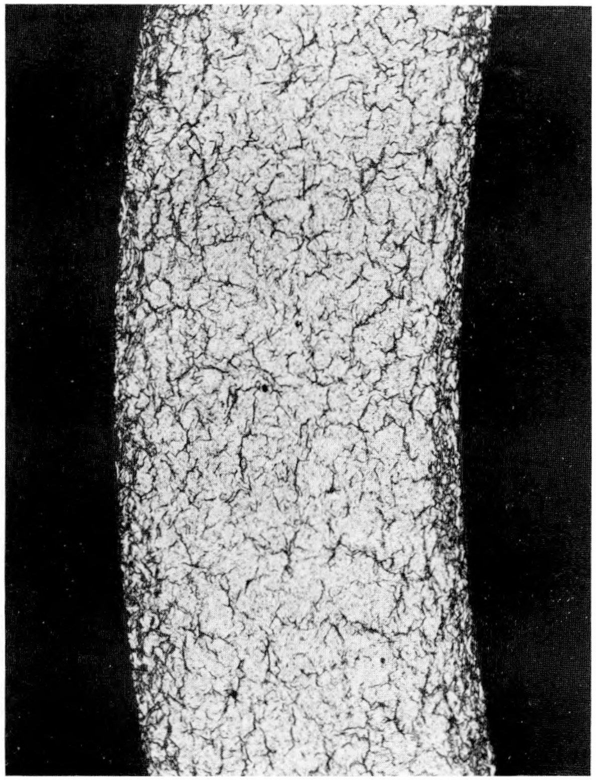


250X

MOUNT NO. 4763J
SPECIMEN NO. 15C

FIGURE 14

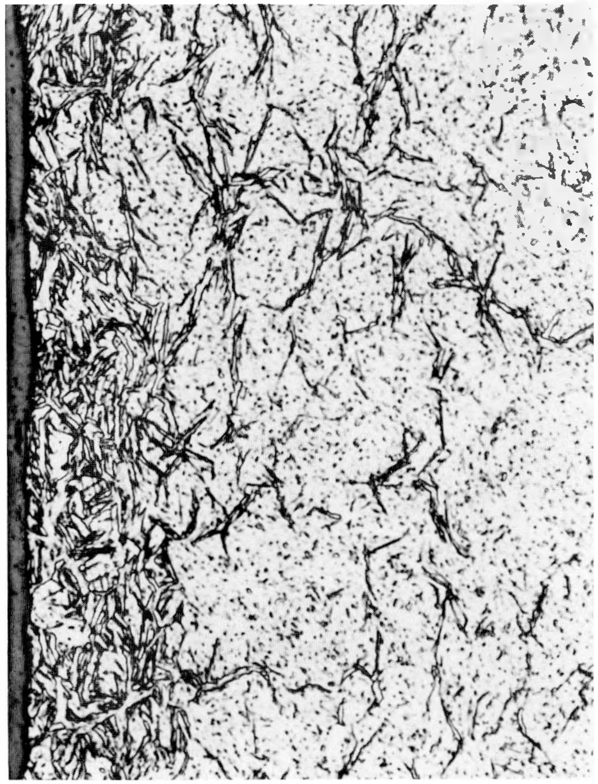
ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100X

1000 ppm H_2

VICKERS HARDNESS
171
(2 Kg LOAD)

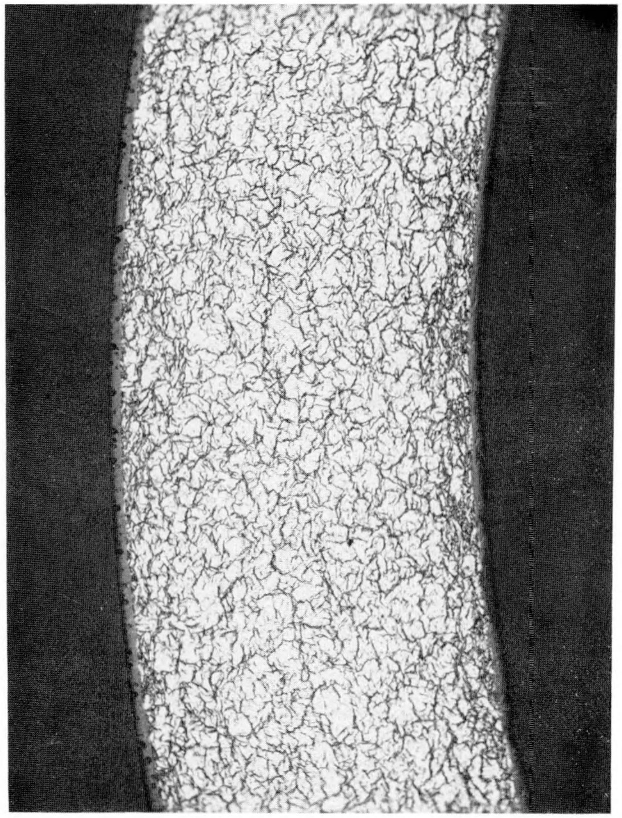


500X

MOUNT NO. 4639J
SPECIMEN NO. 18C

FIGURE 15

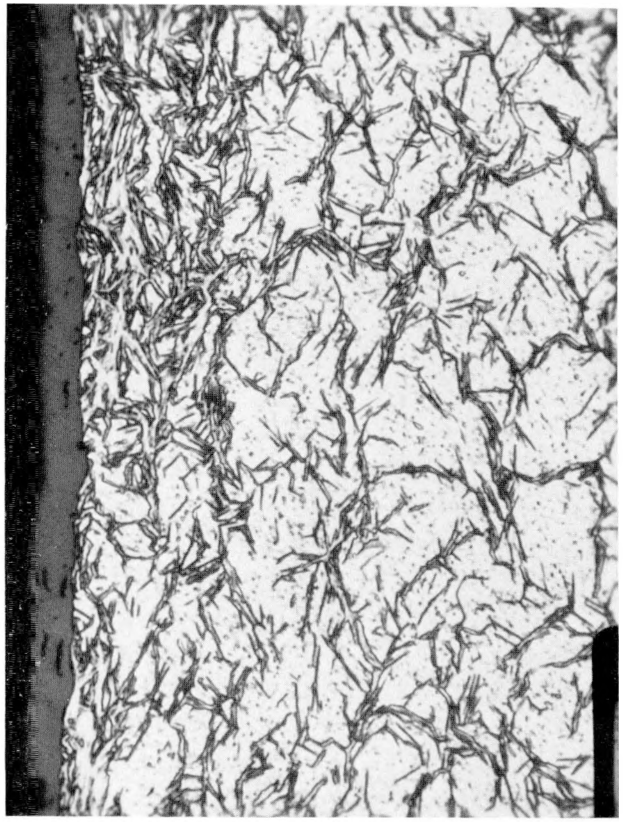
ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100X

1200 ppm H_2

VICKERS HARDNESS
181
(2 Kg LOAD)



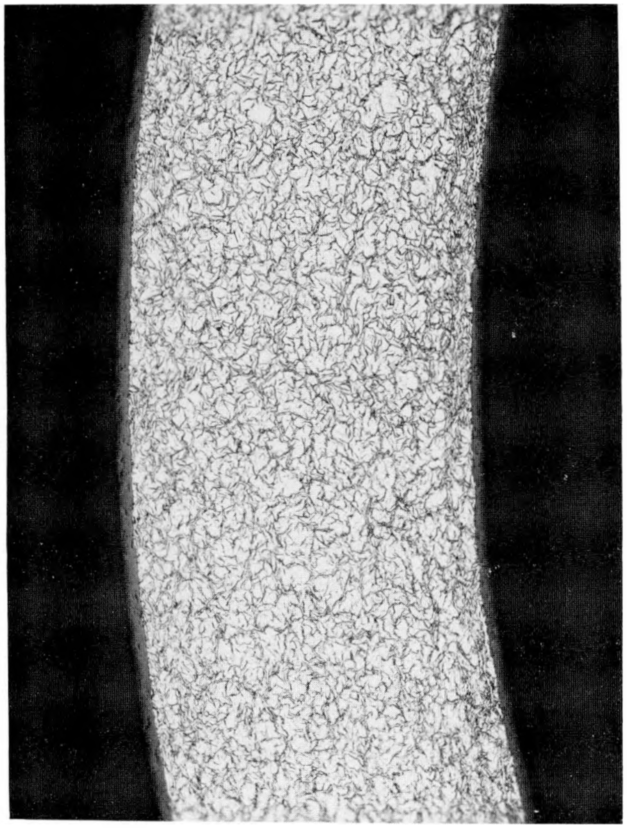
500X

MOUNT NO. 4739 J
SPECIMEN NO. 38C

FIGURE 16

ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN

LWBR SEED TUBING TRANSVERSE SECTION



100X

1900 ppm H_2

VICKERS HARDNESS
186
(2 Kg LOAD)

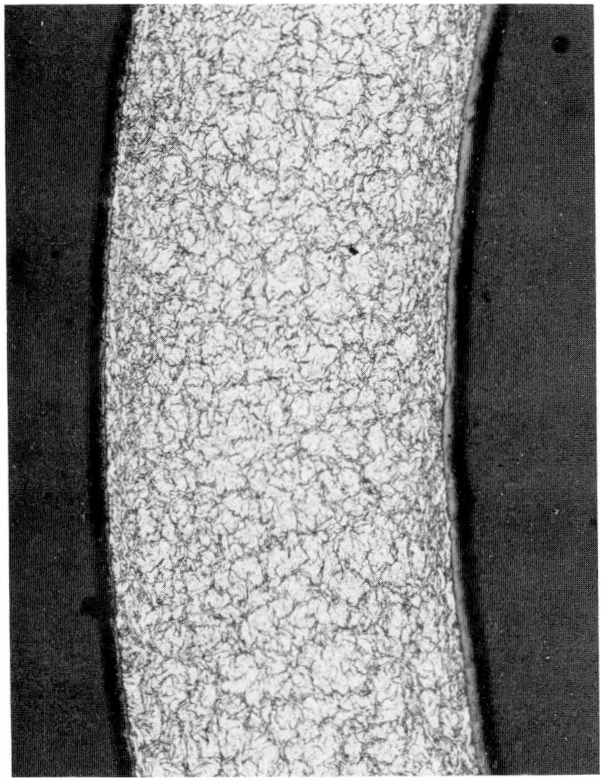
500X

MOUNT NO. 4744 J
SPECIMEN NO. 39C

FIGURE 17

ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN

LWBR SEED TUBING TRANSVERSE SECTION



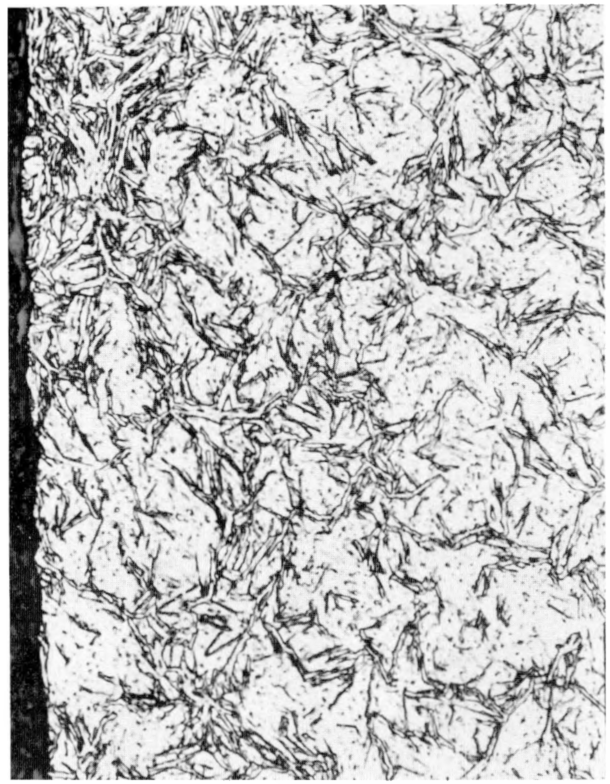
100X

2200 ppm H_2

VICKERS HARDNESS

183

(2 Kg LOAD)



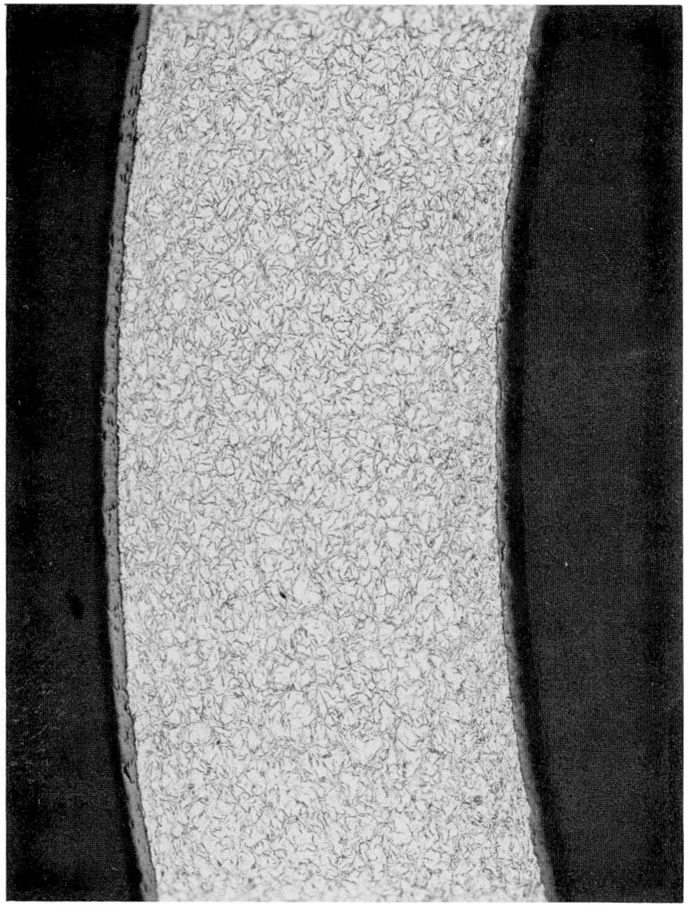
500 X

MOUNT NO. 4645J

SPECIMEN NO. 20C

FIGURE 18

ZIRCALOY 4 CORROSION - HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100X

2300 ppm H_2

VICKERS HARDNESS
195
(2 Kg LOAD)

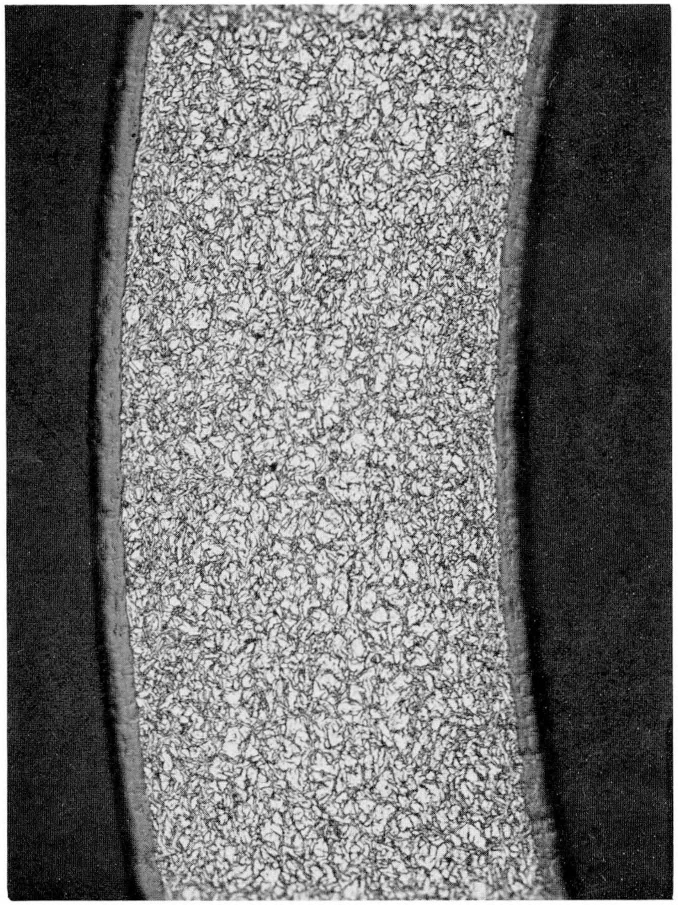
500X

MOUNT NO. 4742J
SPECIMEN NO. 40C

FIGURE 19

ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN

LWBR SEED TUBING TRANSVERSE SECTION



100X

2900 ppm H_2

VICKERS HARDNESS

209

(2 Kg LOAD)

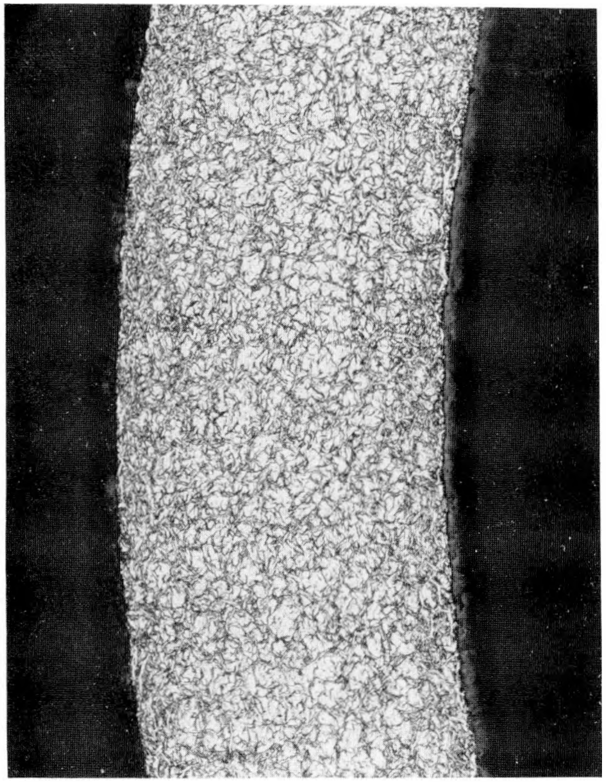
500 X

MOUNT NO. 4644 J

SPECIMEN NO. 22 C

FIGURE 20

ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100X

3400 ppm H_2

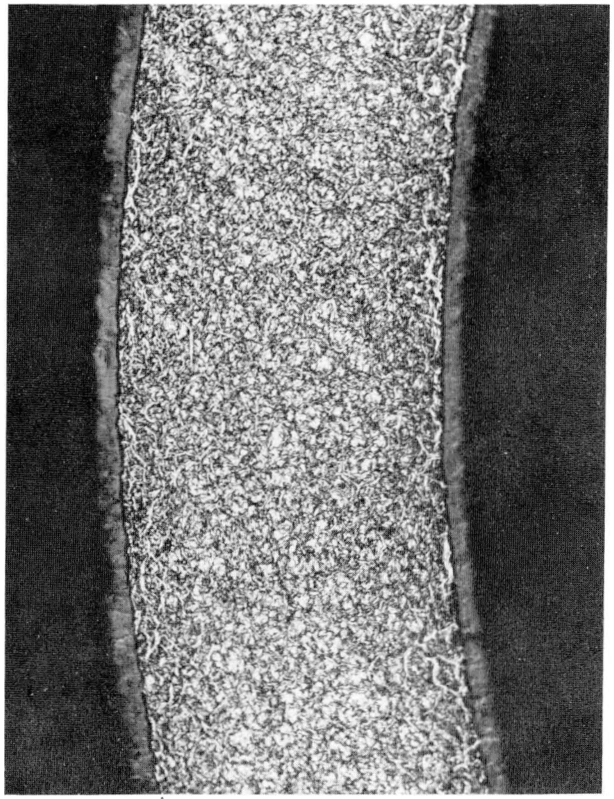
VICKERS HARDNESS
195
(2 Kg LOAD)

500X

MOUNT NO. 4741 J
SPECIMEN NO. 41C

FIGURE 21

ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100X

4200 ppm H_2

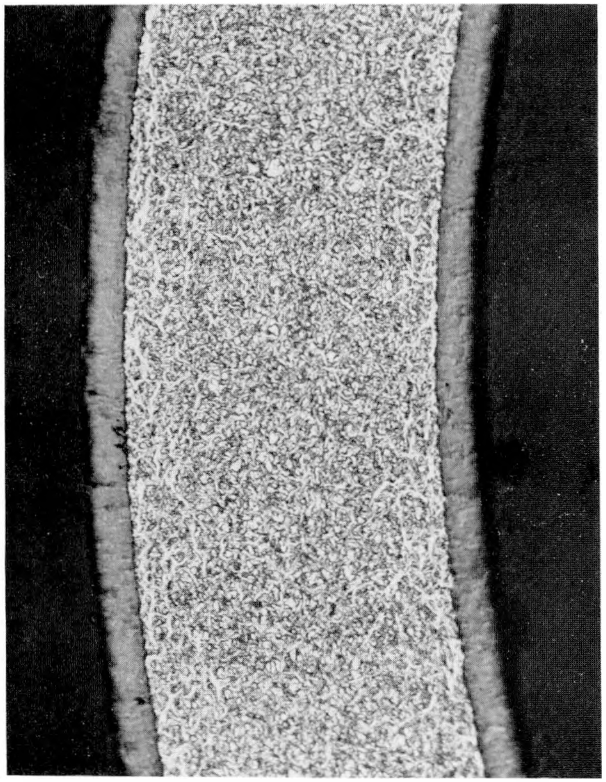
VICKERS HARDNESS
210
(2 Kg LOAD)

500X

MOUNT NO. 4643 J
SPECIMEN NO. 24C

FIGURE 22

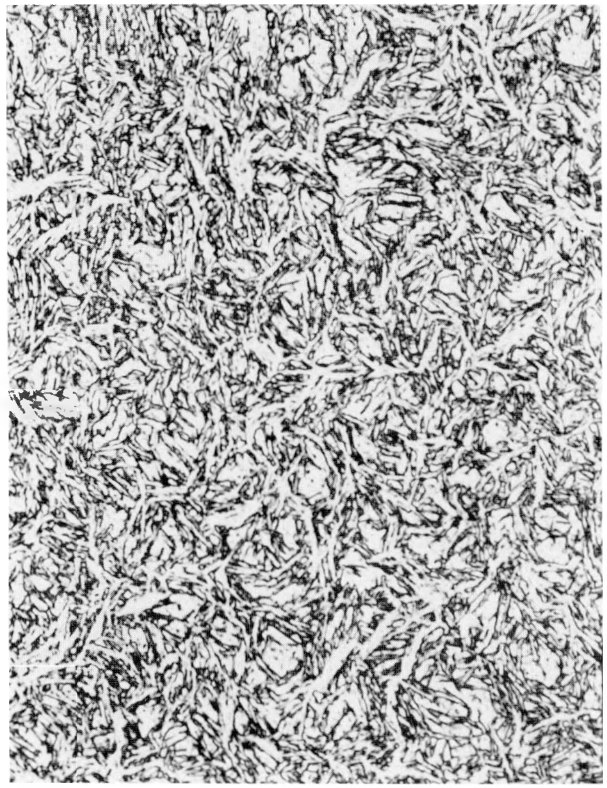
ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100X

5000 ppm H_2

VICKERS HARDNESS
225
(2 Kg LOAD)

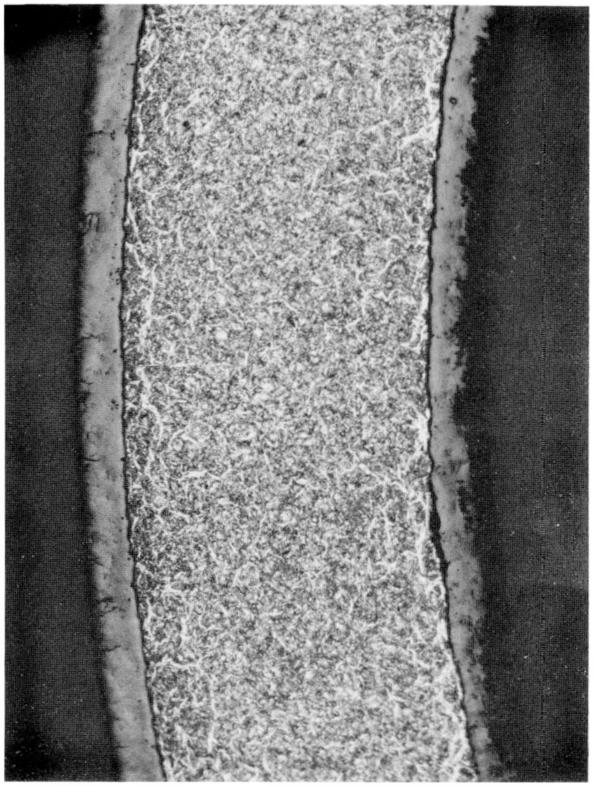


250X

MOUNT NO. 4647 J
SPECIMEN NO. 26C

FIGURE 23

ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100 X

5600 ppm H_2

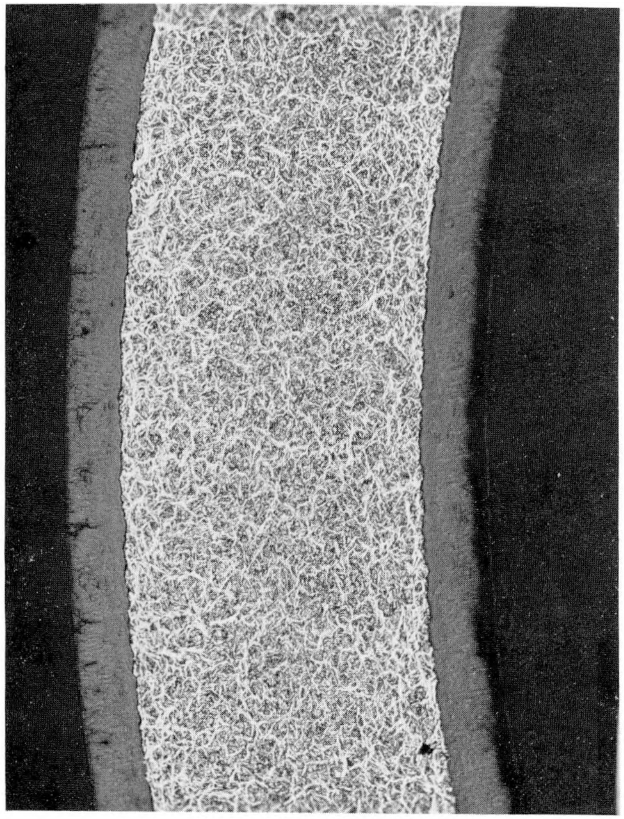
VICKERS HARDNESS
243
(2 Kg LOAD)

500 X

MOUNT NO. 4646 J
SPECIMEN NO. 28C

FIGURE 24

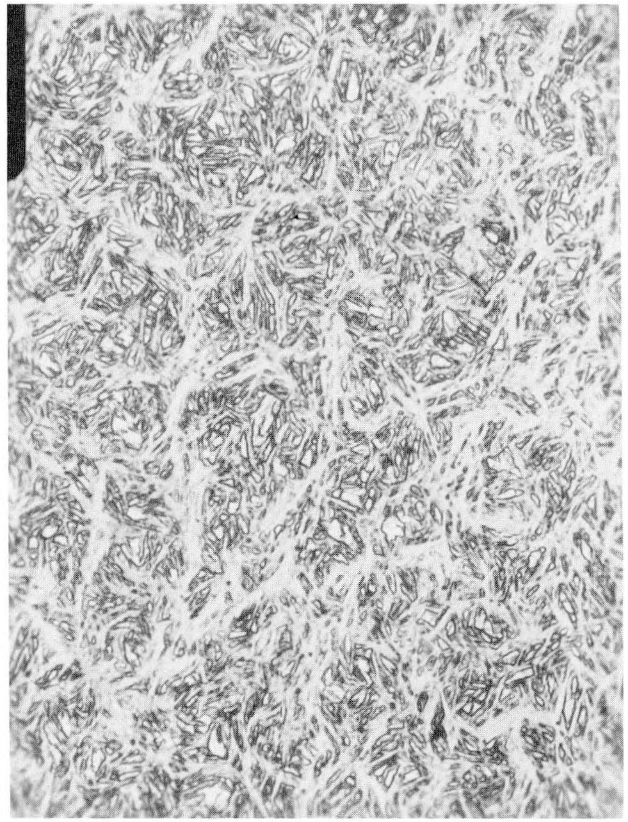
ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100 X

6200 ppm H_2

VICKERS HARDNESS
259
(2 Kg LOAD)

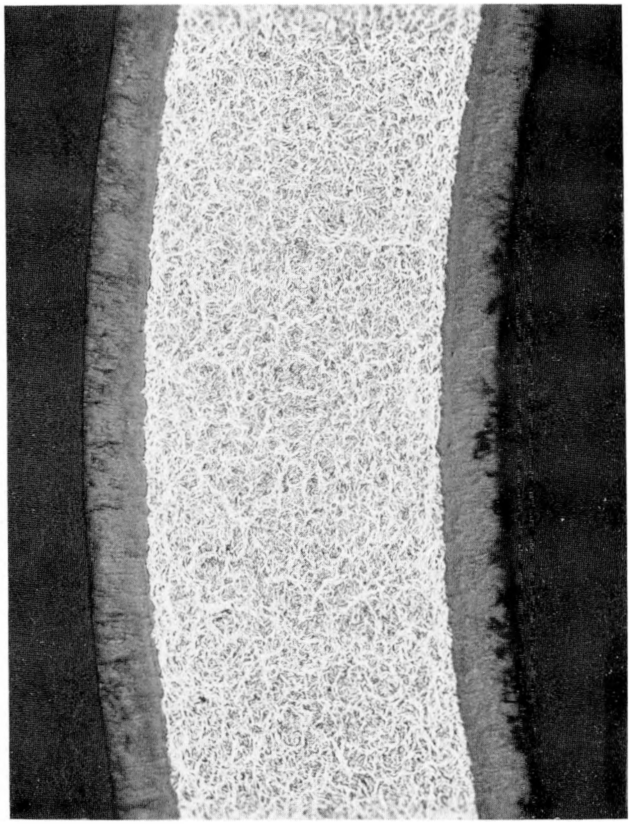


500 X

MOUNT NO. 4740 J
SPECIMEN NO. 31C

FIGURE 25

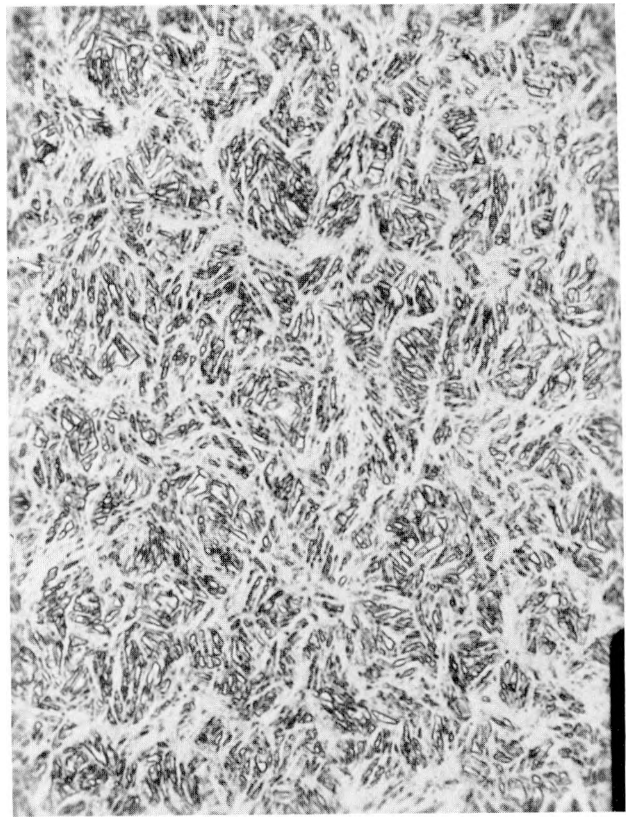
ZIRCALOY 4 CORROSION – HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100X

6400 ppm H_2

VICKERS HARDNESS
274
(2 Kg LOAD)



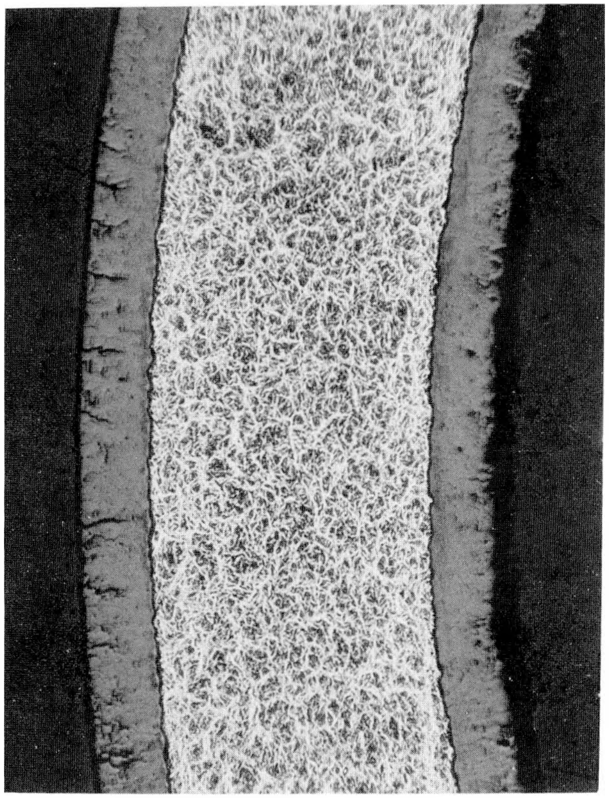
500X

MOUNT NO. 4743J
SPECIMEN NO. 33C

FIGURE 26

ZIRCALOY 4 CORROSION–HYDRIDE SPECIMEN

LWBR SEED TUBING TRANSVERSE SECTION



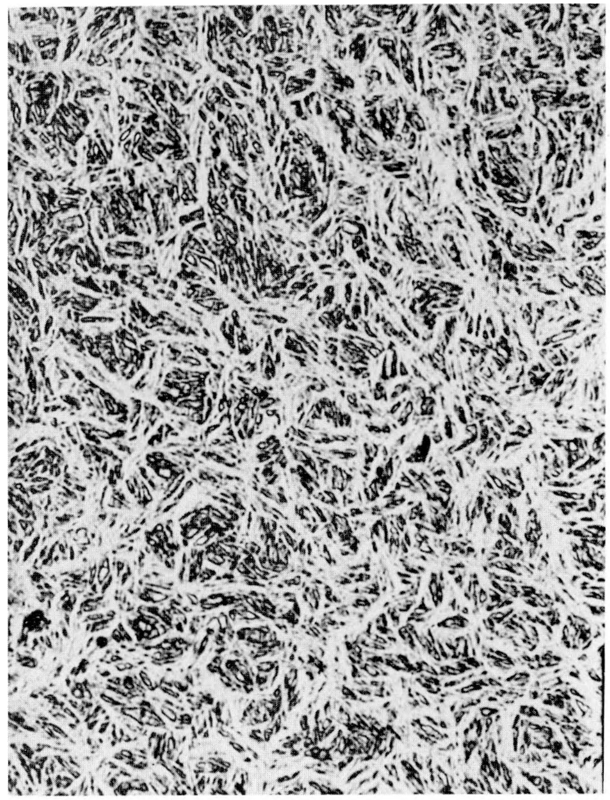
100 X

10,600 ppm H_2

VICKERS HARDNESS

279

(2 Kg LOAD)



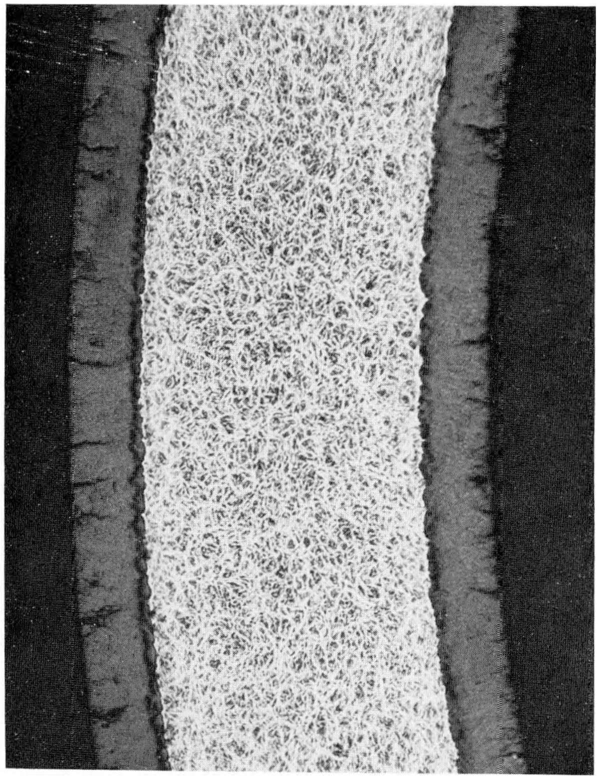
500 X

MOUNT NO. 4682 J

SPECIMEN NO. 34 C

FIGURE 27

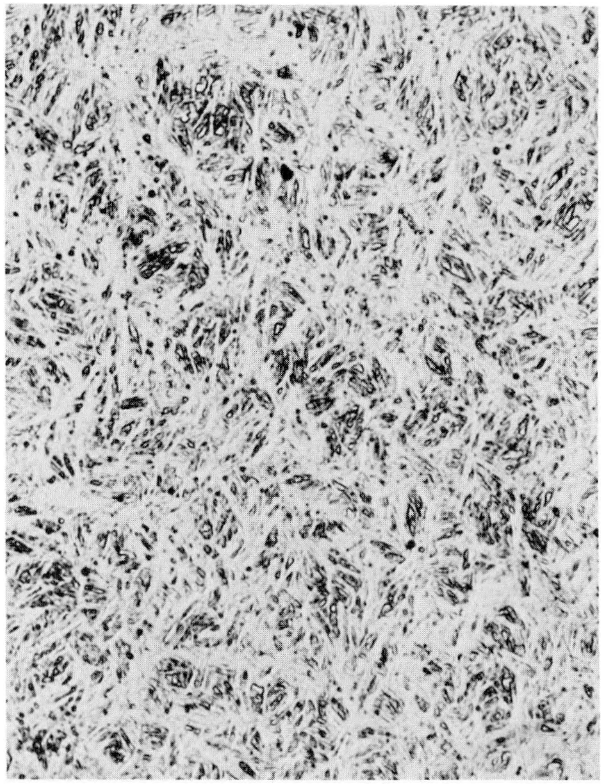
ZIRCALOY 4 CORROSION-HYDRIDE SPECIMEN
LWBR SEED TUBING TRANSVERSE SECTION



100 X

11,600 ppm H_2

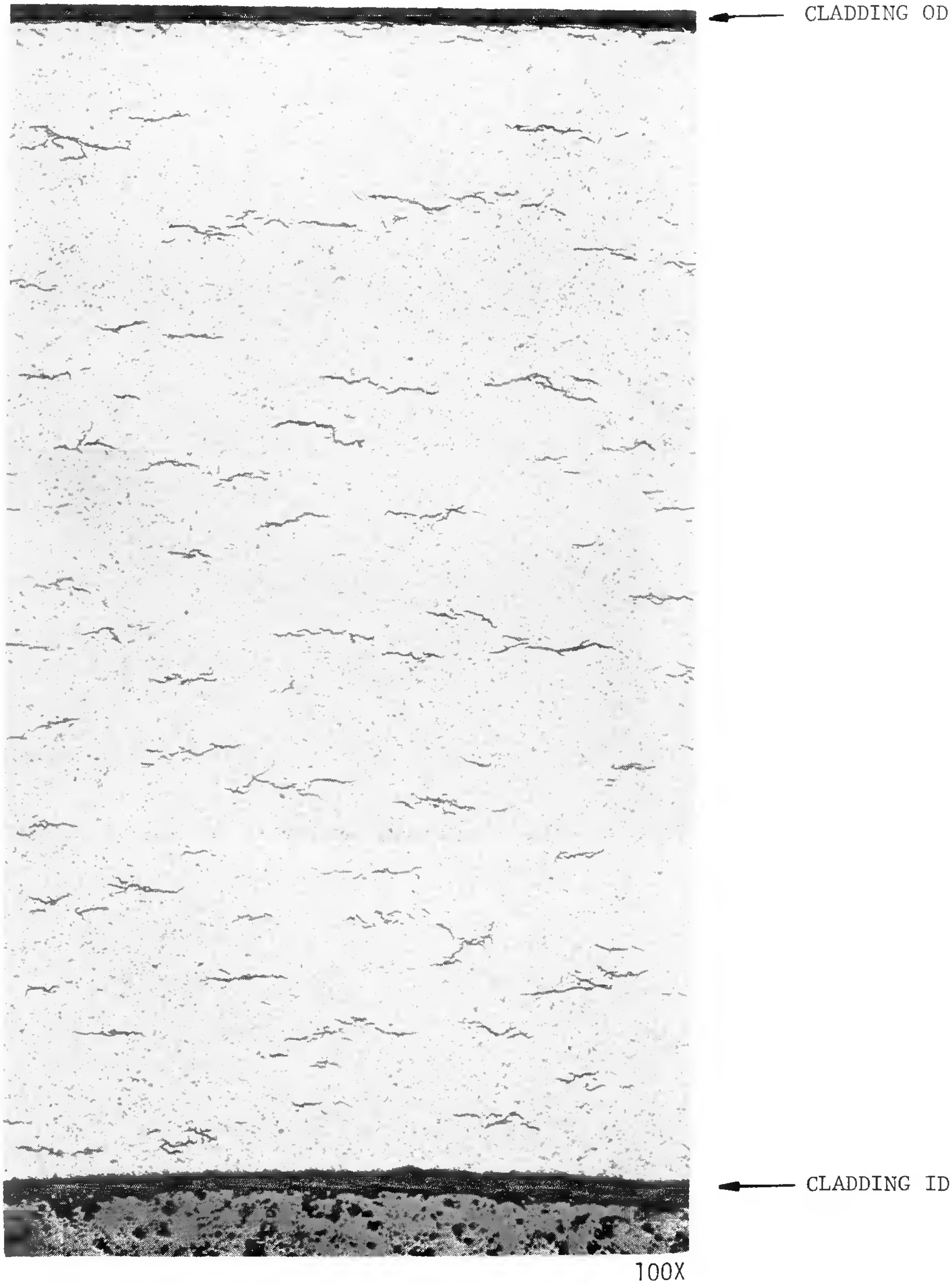
VICKERS HARDNESS
289
(2 Kg LOAD)



500 X

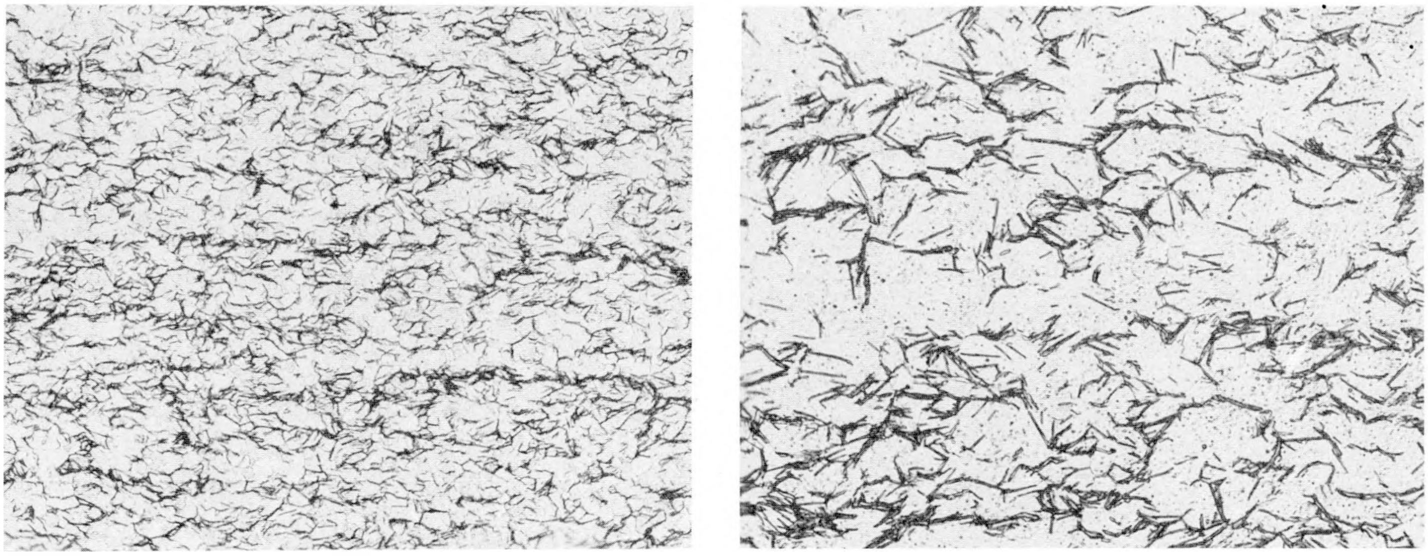
MOUNT NO. 4685 J
SPECIMEN NO. 37C

FIGURE 28



Hydride in Irradiated Corroded Zircaloy-4 Tubing (∿ 30 Mil Wall) Exposed 12,080 Hot Water Hours, For Fluence of ∿ 13×10^{20} n/cm^2, Measured Total Hydrogen, 60 PPM. (Compare to Figure 5)

FIGURE 29



(a) Area 1 100X (b) Area 2 250X

Hydride in Unirradiated Zircaloy-4 Sheet
(Transverse Section, Original Thickness 40 Mils).
Hydride Obtained by Gaseous Equilibration with ZrH_2 at 650°C.
Measured Total Hydrogen, 580 PPM.
(Compare to Figure 10).

FIGURE 30

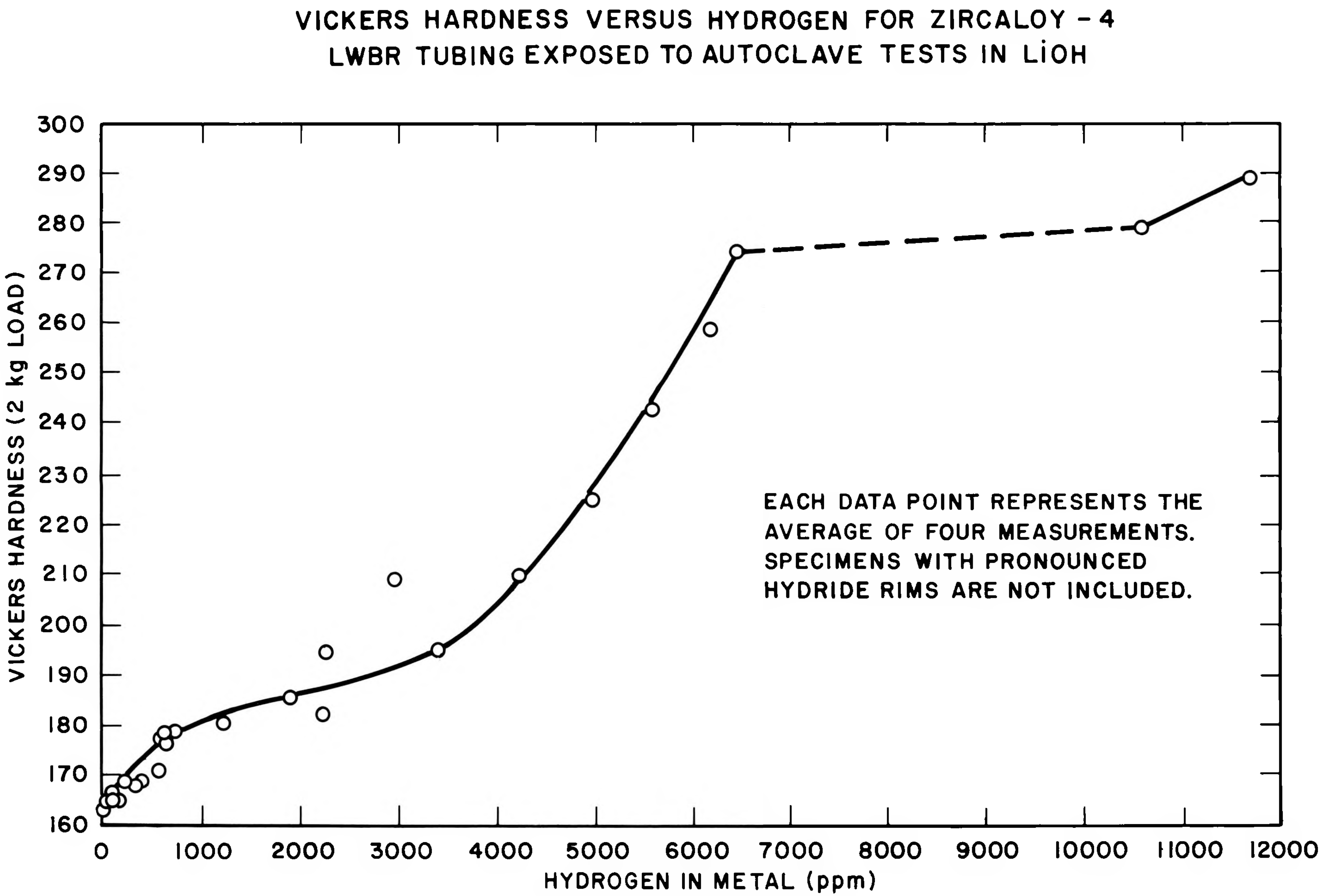

VICKERS HARDNESS VERSUS HYDROGEN FOR ZIRCALOY - 4
LWBR TUBING EXPOSED TO AUTOCLAVE TESTS IN LiOH
VICKERS HARDNESS (2 kg LOAD)
300
290
280
270
260
250
240
230
220
210
200
190
180
170
160
0
1000
2000
3000
4000
5000
6000
7000
8000
9000
10000
11000
12000
HYDROGEN IN METAL (ppm)
EACH DATA POINT REPRESENTS THE AVERAGE OF FOUR MEASUREMENTS. SPECIMENS WITH PRONOUNCED HYDRIDE RIMS ARE NOT INCLUDED.

FIGURE 31

FIGURE 32
WEIGHT GAIN VERSUS EXPOSURE OF ZIRCALOY-4
LWBR TUBING SPECIMENS

(600°F, 0.3 N LiOH)

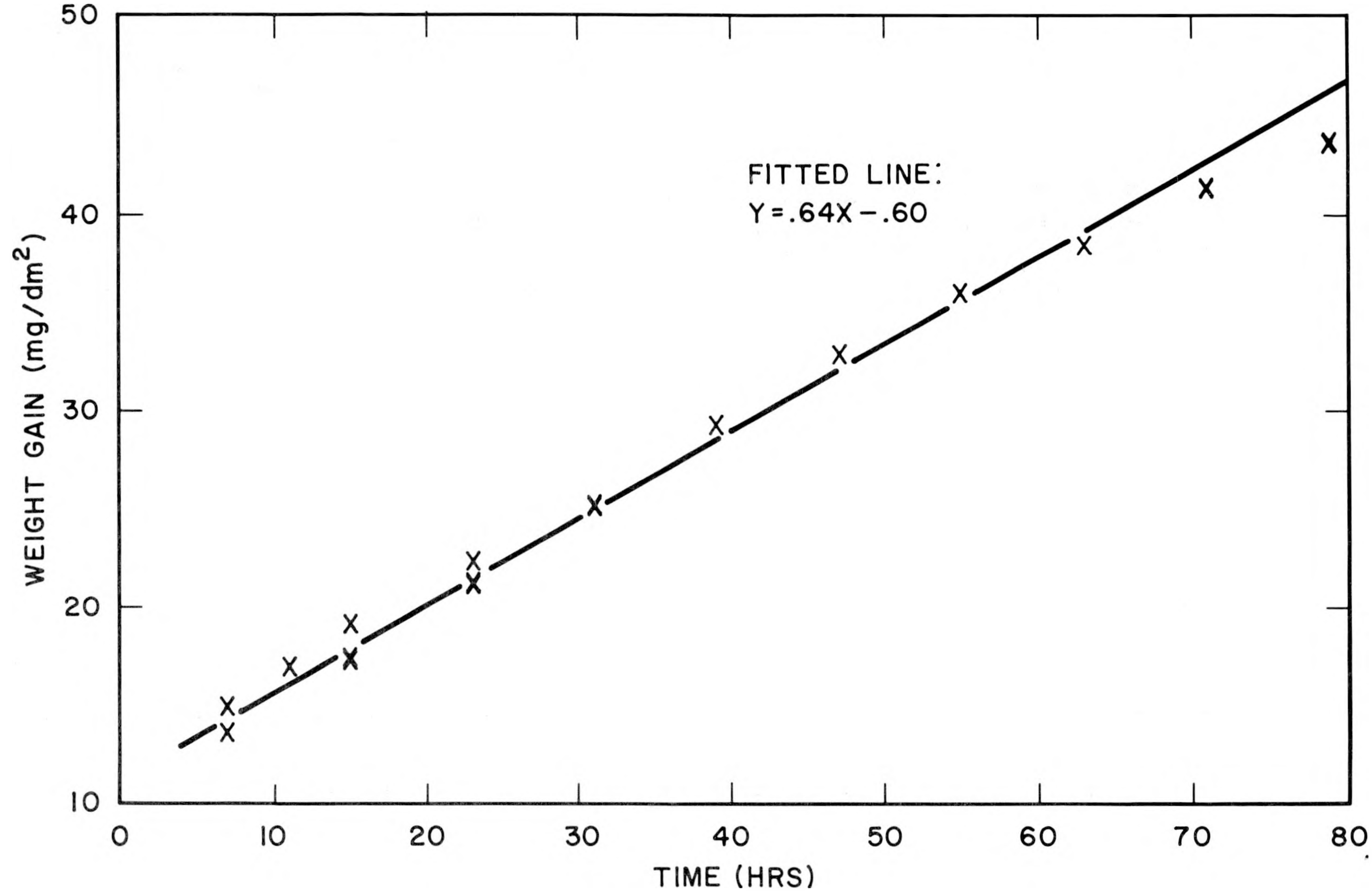


FIGURE 33
WEIGHT GAIN VERSUS AUTOCLAVE EXPOSURE OF ZIRCALOY-4 LWBR TUBING SPECIMENS

(600°F, 0.7 N LiOH)

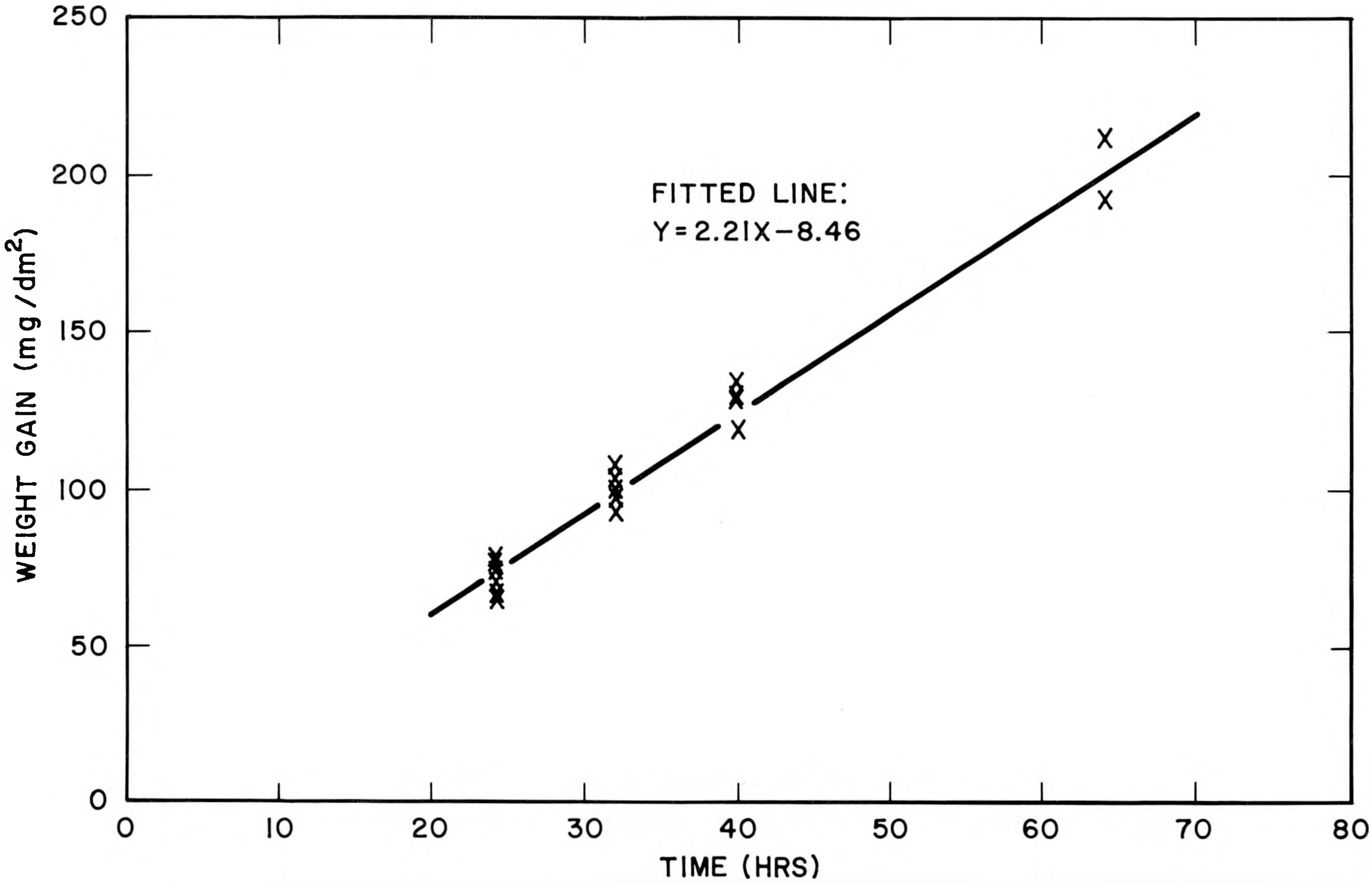


FIGURE 34

WEIGHT GAIN VERSUS AUTOCLAVE EXPOSURE OF ZIRCALOY-4 LWBR TUBING SPECIMENS

(680°F, 0.7 N LiOH)