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THE MECHANICAL PROPERTIES OF
ZIRCONIUM AND ZIRCALOY 2

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THE MECHANICAL PROPERTIES OF
ZIRCONIUM AND ZIRCALOY 2

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The elevated-temperature tensile properties of zirconium and of Zircaloy 2 were investigated and the creep properties were determined at temperatures up to 500 C. The effect of time at test temperature on the tensile strength of the 25 per cent cold-worked materials was investigated. The increased tensile strengths of cold-worked zirconium and Zircaloy 2 were reduced to that of the annealed material by holding at 500 C for approximately 90 and 200 hr, respectively. Cold-worked Zircaloy 2 tested in creep had superior 1000-hr rupture strengths for all test temperatures, except 500 C. At 500 C, the 1000-hr rupture strength of the cold-worked material reverted to that of annealed Zircaloy 2 at the same temperature. The stress to produce a creep rate of 10^{-5} per cent per hr in the annealed Zircaloy 2 was 25,000 psi at 150 C and 3200 psi at 500 C.

Hydraulic-bursting tests also were made on seamless Zircaloy 2 and on welded zirconium tubing. The bursting pressures were considerably higher in the seamless tubes than in the welded tubing, 6800 psi as compared with 5400 psi at room temperature and 3150 psi versus 2200 psi at 400 C. The welded zirconium tubing failed at the longitudinal-seam weld under hydraulic pressure.

INTRODUCTION

Because of its excellent corrosion resistance and low-thermal-neutron cross section, zirconium and zirconium alloys are being considered for claddings and for tubes to contain pressurized coolants in reactors. The strength of these materials can be materially increased by cold working, therefore the present study was undertaken to determine whether these higher mechanical properties could be utilized in the design of the reactor components.

The tensile properties of both annealed and of 25 per cent cold-worked strip were studied at temperatures up to 500 C and the effect of time at test temperature was determined. The creep and creep-rupture properties in this temperature range were also investigated.

In addition, bursting tests were made on samples of welded zirconium tubing and on seamless Zircaloy 2 tubing. These tests were made at temperatures up to 460 C by capping the ends and applying a hydraulic pressure inside the tubes.

FABRICATION

The zirconium and Zircaloy 2 sheet and rod used in these studies were fabricated at the Bureau of Mines for HAPO. The fabrication schedule as supplied by the Bureau of Mines was as follows: The zirconium and Zircaloy 2 were forged from 500-lb 10-in. - diameter ingots at 850 C to a 2-3/4 by 19-in. slab in several operations. After forging,

the oxide was removed by sand blasting and the surface defects were removed by grinding. In the preparation of sheet, sections of the slab were hot rolled to 0.150-in. -thick sheet in several stages. This hot-rolled sheet was annealed 15 min at 750 C, cleaned, cold rolled to 0.080 in., reannealed, and finally cold rolled to 0.060 in. This cold-rolled sheet was tested as rolled and after a final vacuum anneal.

The rod was prepared from 3-in. sections of the forged slab. The section was heated 1 hr at 750 C and hot rolled through alternate diamond and square grooves with reductions of approximately 5 per cent per pass. When the rod had been rolled approximately to the desired size, it was finished by hot swaging at 750 C. The rods were then cold swaged to obtain the desired degree of cold work.

The analyses of the sheet materials are shown in Table 1.

SHORT-TIME TENSILE PROPERTIES

Test Methods

Room-temperature elastic-strain measurements on longitudinal sheet specimens were obtained with Sr-4 Type-A7 strain gages, while Bakelite strain gages were used at 150 C. A supplementary clip-type extensometer was employed to measure both elastic and plastic strain. Strain measurements with the gages were sensitive to $\pm 1 \mu$ in. and were accurate to $\pm 4 \mu$ in. The strain sensitivity of the extensometer was $\pm 15 \mu$ in., while the accuracy was $\pm 30 \mu$ in. The testing strain rate was 0.006 per min.

The tensile specimens used for the study concerning the effect of time at temperature on the cold-worked properties were heat treated at the desired temperature and then tested at that temperature. At temperatures above 150 C, a flowing atmosphere of argon was used to prevent oxidation.

Annealed Zirconium and Zircaloy 2

The elastic-property determinations (duplicate tests) at 27 C and 150 C on annealed and cold-worked zirconium and Zircaloy 2 are summarized in Table 2. The modulus of elasticity for zirconium has been reported to be 13.8×10^6 psi at room temperature, while the value of Poisson's ratio is reported to vary from 0.32 to 0.35⁽¹⁾. In general, the values of the elastic modulus and Poisson's ratio obtained in this study are greater than the reported values. The comparatively large values obtained for Poisson's ratio suggest that the material tested possessed directional properties from the rolling process which were not eliminated by annealing.

The variation of the mechanical strength of annealed zirconium and Zircaloy 2 with temperature is shown in Figure 1 and the data are tabulated in Table 3. The data in Figure 1 are apt to be misleading with regard to the tensile strengths of zirconium and Zircaloy 2. The mechanical properties of zirconium and its alloys are very sensitive to impurity content, particularly oxygen. Published data for the tensile strength of sponge

(1) References at end of report.

TABLE 1. ANALYSES OF ZIRCONIUM AND ZIRCALOY 2 SHEET

Analysis			
Sponge Zirconium, ppm		Zircaloy 2 ^(a) , w/o	
Aluminum	150	Chromium	0.049
Boron	5	Iron	0.11
Calcium	<10	Nickel	0.045
Copper	<10	Tin	1.61
Chromium	70	Nitrogen	0.003
Hafnium	120	Carbon	0.03
Iron	300	Oxygen	0.00970
Lead	80	Hydrogen	0.00028
Manganese	20		
Magnesium	<10		
Molybdenum	10		
Nickel	10		
Silicon	100		
Tin	60		
Carbon	300		
Oxygen	1250		
Hydrogen	21		
Nitrogen	50		

(a) Zircaloy 2 was produced from a sponge-zirconium base.

TABLE 2. ELASTIC MODULUS AND POISSON'S RATIO OF ZIRCONIUM AND ZIRCALOY 2

Specimen Condition	Test Temperature, C	Elastic Property in Tension	
		Modulus, 10 ⁶ psi	Poisson's Ratio
Annealed zirconium	27	14.2-14.3	0.358
	150	12.7-13.0	0.430-0.442
Cold-worked zirconium	27	13.8-14.0	0.425-0.465
	150	13.8-15.0	0.383-0.390
Annealed Zircaloy 2	27	13.9-14.3	0.368-0.380
	150	13.3-13.8	0.425-0.460
Cold-worked Zircaloy 2	27	14.5	0.382-0.406
	150	13.9-14.2	0.392-0.432

FIGURE 1. VARIATION OF MECHANICAL STRENGTH OF ANNEALED ZIRCONIUM AND ZIRCALOY 2 WITH TEMPERATURE

TABLE 3. MECHANICAL PROPERTIES OF ANNEALED ZIRCONIUM
AND ZIRCALOY 2 AT VARIOUS TEMPERATURES

Properties	27 C	150 C	230 C	345 C	425 C	500 C
<u>Annealed Zirconium</u>						
0.2 Per Cent Offset Yield Strength, psi	31,700-32,700	16,800-17,500	10,700	7,800	7,500	5,200
Tensile Strength, psi	54,500-54,750	38,400-34,800	26,600	18,800	14,500	11,200
Elongation, per cent	42.0-38.0	47.7-55.5	55.3	55.5	59.1	88.5
<u>Annealed Zircaloy 2</u>						
0.2 Per Cent Offset Yield Strength, psi	41,000-43,000	27,200-27,500	19,700	17,500	16,200	13,800
Tensile Strength, psi	54,800-54,600	39,300-38,400	29,700	23,500	20,900	19,500
Elongation, per cent	32.0-28.7	46.5-42.0	46.5	40.4	37.4	42.1

zirconium⁽²⁾ and crystal-bar-base Zircaloy 2⁽³⁾ are similar to values obtained in this investigation for zirconium and Zircaloy 2. As a result, no particular significance should be attached to the fact that on this material the tensile strengths of zirconium and Zircaloy 2 were similar at temperatures up to 230 C.

Effect of Time at Temperature on Strength of Cold-Rolled Zirconium and Zircaloy 2

The effect of time at temperature on the strength properties of the cold-worked materials was studied in an effort to provide information as to the time period during which zirconium and Zircaloy 2 would retain the strength benefits derived from pre-strain. Since appreciable recovery and recrystallization was not expected to occur in reasonable time periods at 345 C, emphasis was placed on obtaining a limited amount of data at 425 C and 500 C. The data obtained are shown graphically in Figures 2 and 3 and are tabulated in Table 4.

Figure 2 shows data plotted after the fashion of typical reaction curves. The fractional change in yield strength was obtained by using the yield strength of the annealed material at the particular temperature as the base value, while the yield strength

at 1/2 hr provided the upper limit, i. e., $f_t = \frac{\text{Y.S. of C.W.} - \text{Y.S. at time (t)}}{\text{Y.S. of cold worked} - \text{Y.S. of annealed}}$.

It readily can be observed that the recrystallization process in Zircaloy 2 is delayed considerably with relation to zirconium having this particular composition. The recrystallization behavior of zirconium with approximately 25 per cent cold reduction as reported in the literature⁽⁴⁾ is slightly faster than that obtained in this study. Again, the observed difference in behavior may be attributed to variation in impurity content. Another factor might be the uncertainty in the amount and distribution of cold work in the rolled sheet. For example, the amount of reduction per pass through the rolls greatly affects the distribution of plastic strain even though the total reduction may be the same.

The same data are shown in Figure 3, but plotted in another manner. The yield strength is plotted against a parameter relating temperature and time at temperature.

Parameters for analyzing properties subject to change with time at temperature often prove to be of value in permitting a limited amount of extrapolation for desirable information. The particular parameter utilized in Figure 3 can be obtained from the Arrhenius-type rate equation:

$$\text{rate (r)} = \frac{1}{t} = A e^{-Q/RT}, \quad (1)$$

where A is a constant, Q the activation energy, R the gas constant, and T the temperature. By taking the logarithm of this equation, the following expression is obtained:

$$-\log t = \log A - Q/RT, \quad (2)$$

$$\text{or} \quad T(c + \log t) = \frac{Q}{R} \quad (\text{a constant}), \quad (3)$$

where $c = \log A$. This expression was employed with considerable success by Larson and Miller⁽⁵⁾ in studying the recrystallization of several metals. Unfortunately, the data were insufficient to adequately define the curve for Zircaloy 2. However, from the

FIGURE 2. ANNEALING BEHAVIOR OF COLD-WORKED ZIRCONIUM AND ZIRCALOY 2 AT 425 C AND 500 C

FIGURE 3. VARIATION OF YIELD STRENGTH WITH TIME AT 425 C AND 500 C

TABLE 4. EFFECT OF TIME AT TEMPERATURE ON THE
TENSILE PROPERTIES OF COLD-WORKED
ZIRCONIUM AND ZIRCALOY 2

Time at Temperature ^(a) , hr	Temperature, C	0.2 Per Cent Offset Yield Strength, psi	Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent
<u>25 Per Cent Cold-Worked Zirconium</u>					
1/2	230	--	--	--	--
1		--	--	--	--
2		--	--	--	--
5		--	--	--	--
10		--	--	--	--
25		--	--	--	--
50		--	--	--	--
100		45,400	49,600	17.2	51.6
500		--	--	--	--
1/2	345	--	--	--	--
1		--	--	--	--
2		--	--	--	--
5		--	--	--	--
10		--	--	--	--
25		--	--	--	--
50		--	--	--	--
100		30,200	36,100	19.2	63.6
500		--	--	--	--
1/2	425	18,900	27,300	25.9	68.5
1		22,600	29,500	21.5	73.5
2		--	--	--	--
5		--	--	--	--
10		20,000	26,650	26.1	64.6
25		--	--	--	--
50		17,200	23,200	33.1	74.0
100		15,700	22,850	33.6	74.1
500		13,900	19,210	37	73.7
1/2	500	12,200	18,400	34.9	78.6
1		12,400	17,000	39.2	80.7
2		12,600	17,100	36.9	80.2
5		10,000	14,600	53.3	82.1
10		8,000	10,800	63.5	76.0
25		6,800	10,800	74.2	77.1
50		--	--	--	--
100		--	--	--	--
500		5,300	10,500	93	84.5

TABLE 4. (Continued)

Time at Temperature(a), hr	Temperature, C	0.2 Per Cent Offset Yield Strength, psi	Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent
<u>25 Per Cent Cold-Worked Zircaloy 2</u>					
1/2	230	--	--	--	--
1		--	--	--	--
2		--	--	--	--
5		--	--	--	--
10		--	--	--	--
25		--	--	--	--
50		--	--	--	--
100		61,000	65,250	13.6	54.8
500		--	--	--	--
1/2	345	--	--	--	--
1		--	--	--	--
2		--	--	--	--
5		--	--	--	--
10		--	--	--	--
25		--	--	--	--
50		--	--	--	--
100		47,500	52,400	15.8	62.4
500		--	--	--	--
1/2	425	35,700	42,200	19.2	68.2
1		39,200	44,500	19.5	67.3
2		--	--	--	--
5		--	--	--	--
10		38,700	43,900	18.2	67.3
25		--	--	--	--
50		37,500	43,100	19.1	67.2
100		37,700	40,600	20.0	66.8
500		34,200	40,900	21	67
1/2	500	22,800	32,900	24.7	74.2
1		20,400	32,800	29.5	74.8
2		20,300	32,800	27.2	75.0
5		19,600	29,000	30.5	76.7
10		20,200	31,000	29.1	71.0
25		18,600	28,500	30.5	72.3
50		--	--	--	--
100		--	--	--	--
500		12,900	18,500	44.5	79.3

(a) Time at temperature before load was applied.

zirconium plot, it may be seen that zirconium should have recrystallized at 500 C in 90 hr, or a time on the order of 30, 000 hr would be needed to achieve annealed properties at 425 C. Similarly, a time of approximately 200 hr at 500 C would be needed to obtain the annealed properties for Zircaloy 2. A word of caution is in order concerning the weight to be given to the calculations. The value of such a plot is in direct proportion to the accuracy of the plot. The times indicated above for complete recrystallization are probably at best only of value as an order-of-magnitude indication.

CREEP AND RUPTURE PROPERTIES

The creep and stress-rupture properties of Zircaloy 2 bar and sheet material were determined over a temperature range of 150 to 500 C (300 to 932 F). Both bar and sheet specimens were evaluated in the cold-worked and annealed conditions. The sheet material was also tested in the transverse and longitudinal directions. A complete series of tests, consisting of about six per temperature, was made on the transverse sheet with only a few longitudinal-sheet and bar specimens tested for check purposes.

It was planned to obtain: (1) 1-, 10-, 100-, and 1000-hr rupture stresses; (2) curves of stress versus creep rate from which stresses to produce a creep rate of 0.00001 per cent per hr can be extrapolated; and (3) design curves showing 0.1, 0.2, 0.5, 1.0, 2.0, 5.0, and 10 per cent total elongation at each test temperature. It was not possible to obtain all these data but effort was made in this direction. Due to the lack of resistance to oxidation of Zircaloy 2 at the higher temperatures, it was decided to conduct the 150 and 230 C (300 and 450 F) tests in air and the 345, 425, and 500 C (650, 800, and 932 F) tests in vacuum.

In order to obtain the aforementioned data, it was decided to conduct three creep-rupture and three creep tests on the Zircaloy 2 sheet material in the annealed and cold-worked conditions in the transverse direction and at each of the five indicated temperatures. Only about three creep and creep-rupture tests were made in the longitudinal directions at each temperature to determine whether marked directional properties existed in the sheet. A few scattered check tests were also made on the bar material to see how the results compared with the sheet data.

Test Method

These creep and rupture tests were made in standard Battelle creep-testing frames with a 9:1 ratio lever-arm arrangement. These units are equipped with either an air or a vacuum furnace which is about 18 in. long and 6 in. in diameter. The heaters are made of Chromel A wire wound on an Alundum tube. The air furnaces contain Sil-O-Cel for insulation while the vacuum furnaces have stainless steel reflector shields. Brass bellows at the top and bottom of the vacuum furnaces allow creep deformation to occur. A small window is provided at both the front and back of the furnace for measuring the deformation of the specimen by optical means.

The vacuum furnaces are evacuated by means of a Welch Duo-Seal mechanical pump and a Distillation Products oil-diffusion pump. Vacuum pressures are, generally, maintained below $2-3 \times 10^{-3}$ mm of mercury within the testing furnace.

The temperature of each test specimen within the furnace is maintained by means of a Foxboro controller to which an anticipating device has been incorporated to improve the temperature control. The rate of heating to the desired temperature varies, depending on the test temperature and on whether the test is being conducted in air or vacuum but is, generally, in the range of about 50 to 150 F per hr. The test temperature is checked and, if necessary, corrected daily. The gage section is usually held within $\pm 2-3$ F and the temperature cycle within $\pm 1-2$ F.

Deformation occurring in the test specimen is measured by means of a platinum-strip extensometer attached to the gage section by clamps. The extensometer consists of two strips, one of which slides within the folded edges of the other. The surfaces of the strips are polished to provide a good reflecting surface and a series of very fine cross marks are scribed on them. Two cross marks, one on each strip, are chosen as reference marks and the distance between them is measured periodically with a filar microscope. The change in length or creep is determined by measuring the change in the distance between the reference marks.

The microscope with which deformation readings are made has an eyepiece fitted with a filar micrometer and is mounted in a graduated screw. The smallest division on the filar eyepiece is 0.00005 in. which, on a 2-in. gage length, would be equal to 0.0025 per cent.

The zero reading (no deformation) is taken when the specimen is at the test temperature with no stress applied. The initial deformation is obtained by applying the entire stress. Succeeding deformation readings are then taken daily or oftener by two observers. Rupture times are recorded on a timer which stops when the specimen fails.

The optical deformation readings are converted to percentages and plotted as time-deformation (creep) curves on rectangular coordinates. The readings of both observers are averaged to produce these curves. Deformations at any time period within limits of the test are then obtainable from these creep curves. Also obtainable from these curves are the minimum creep rate or the creep rate at any desired time period. The most common method of showing all these data is as: (1) curves of stress versus rupture time, (2) design curves of stress versus time for various amounts of total deformation, and (3) curves of stress versus minimum creep rate. All available data have been reproduced in this manner as shown in Figures 4 through 18.

Test Results

The creep and creep-rupture test results obtained on Zircaloy 2 sheet and bar material are contained in Tables 5 through 9. Tables 5 and 6 have the results of tests made on annealed Zircaloy 2 sheet in the transverse and longitudinal directions. Data obtained on cold-worked Zircaloy 2 sheet in the transverse and longitudinal directions are shown in Tables 7 and 8. All data obtained on the bar material are displayed in Table 9.

FIGURE 4. STRESS-RUPTURE CURVES FOR ANNEALED ZIRCALOY 2 SHEET TESTED IN THE TRANSVERSE AND LONGITUDINAL DIRECTIONS AT TEMPERATURES RANGING FROM 150 TO 500 C

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FIGURE 5. STRESS-RUPTURE CURVES FOR COLD-WORKED ZIRCALOY 2 SHEET TESTED IN THE TRANSVERSE AND LONGITUDINAL DIRECTIONS AT TEMPERATURES RANGING FROM 150 TO 500 C

FIGURE 6. STRESS-RUPTURE CURVES FOR ANNEALED AND COLD-WORKED ZIRCALOY 2 BAR MATERIAL TESTED AT TEMPERATURES RANGING FROM 150 TO 500 C

FIGURE 7. DESIGN CURVES FOR ANNEALED ZIRCALOY 2 SHEET TESTED IN THE TRANSVERSE AND LONGITUDINAL DIRECTIONS IN AIR AT 150 C

FIGURE 8. DESIGN CURVES FOR ANNEALED ZIRCALOY 2 SHEET TESTED IN THE TRANSVERSE AND LONGITUDINAL DIRECTIONS IN AIR AT 230 C

FIGURE 9. TIME-STRESS CURVES FOR ANNEALED ZIRCALOY 2 SHEET TESTED IN THE TRANSVERSE AND LONGITUDINAL DIRECTIONS IN VACUUM AT 345 C

FIGURE 10. DESIGN CURVES FOR ANNEALED ZIRCALOY 2 SHEET TESTED IN THE TRANSVERSE AND LONGITUDINAL DIRECTIONS IN VACUUM AT 425 C

FIGURE 11. DESIGN CURVES FOR ANNEALED ZIRCALOY 2 SHEET TESTED IN THE TRANSVERSE AND LONGITUDINAL DIRECTIONS IN VACUUM AT 500 C

FIGURE 12. TIME-STRESS CURVES FOR COLD-WORKED ZIRCALOY 2 SHEET TESTED IN THE TRANSVERSE AND LONGITUDINAL DIRECTIONS IN AIR AT 150 AND 230 C

FIGURE 13. TIME-STRESS CURVES FOR COLD-WORKED ZIRCALLOY 2 SHEET TESTED IN THE TRANSVERSE AND LONGITUDINAL DIRECTIONS IN VACUUM AT 345 C

FIGURE 14. DESIGN CURVES FOR COLD-WORKED ZIRCALOY 2 SHEET TESTED IN THE TRANSVERSE AND LONGITUDINAL DIRECTIONS IN VACUUM AT 425 C

FIGURE 15. DESIGN CURVES FOR COLD-WORKED ZIRCALOY 2 SHEET TESTED IN THE TRANSVERSE AND LONGITUDINAL DIRECTIONS IN VACUUM AT 500 C

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FIGURE 16. STRESS VERSUS CREEP RATE FOR ANNEALED ZIRCALOY 2 SHEET MATERIAL TESTED IN THE TRANSVERSE AND LONGITUDINAL DIRECTIONS AT TEMPERATURES RANGING FROM 150 TO 500 C

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FIGURE 17. STRESS VERSUS CREEP RATE FOR COLD-WORKED ZIRCALOY 2 SHEET MATERIAL TESTED IN THE TRANSVERSE AND LONGITUDINAL DIRECTIONS AT TEMPERATURES RANGING FROM 150 TO 500 C

FIGURE 18. STRESS VERSUS CREEP RATE FOR ZIRCALLOY 2 BAR MATERIAL TESTED AT TEMPERATURES RANGING FROM 150 TO 500 C

TABLE 5. CREEP-RUPTURE DATA ON ANNEALED ZIRCALOY 2 SHEET MATERIAL TESTED IN THE TRANSVERSE DIRECTION AT TEMPERATURES RANGING FROM 150 to 500 C

Specimen	Temperature, C	Stress, psi	Rupture Time, hr	Time to Reach Indicated Percentage Deformation, hr							Final Deformation, per cent	Minimum Creep Rate, per cent per hr
				0.1	0.2	0.5	1.0	2.0	5.0	10		
6-1	150	37,000	0.4	--	--	--	--	--	--	0.14	42.4	45.0
6-3	150	35,000	1.2	--	--	--	--	0.02	0.12	0.44	38.0	12.7
6-24	150	30,000	117.5	--	--	--	--	0.2	4.0	16.5	37.6	0.39
6-5	150	27,500	1110.0(a)	--	--	0.05	0.85	7.0	270	--	9.58	0.0052
6-25	150	25,000	1005.0(a)	--	--	0.11	6.0	300	--	--	2.50	0.0005
6-27	150	23,000	1003.4(a)	--	--	5.0	150	--	--	--	1.15	0.0001
6-8	230	29,000	0.3	--	--	--	--	--	0.03	0.1	54.4	76.4
6-6	230	26,000	16.7	--	--	--	--	0.08	4.6	--	36.8	1.0
6-14	230	24,500	27.3	--	--	--	0.01	0.12	7.6	--	28.0	0.095
6-17	230	23,800	136.8	--	--	--	0.05	0.6	16	88	27.5	0.055
6-10	230	23,000	1000.0(a)	--	--	0.05	1.5	60	--	--	2.14	0.00002
6-2	345	22,500	0.4	--	--	--	--	--	0.03	0.13	34.8	39.0
6-23	345	21,000	1000.0(a)	--	--	--	--	0.05	--	--	4.36	0.0004
6-21	345	17,500	1005.0(a)	--	--	--	--	--	--	--	0.75	0.00007
6-13	345	12,500	1004.6(a)	--	0.3	--	--	--	--	--	0.46	0.00003
6-7	345	9,000	1005.7(a)	--	--	--	--	--	--	--	0.18	<0.00001
6-12	425	20,000	3.8	--	--	--	--	--	0.32	1.66	32.8	2.75
6-20	425	17,500	49.6	--	--	1.0	2.2	6.4	18	32	32.0	0.20
6-4	425	15,000	816.9	--	0.16	3.5	6.4	12	52	220	57.2	0.022
6-16	425	10,000	1005.5(a)	15	163	--	--	--	--	--	0.387	0.00015
6-22	425	9,000	1032.0(a)	--	1.4	--	--	--	--	--	0.387	0.00007
6-18	500	19,000	0.35	--	--	--	--	--	0.03	0.07	42.0	>40
6-11	500	14,500	18.6	--	--	0.05	0.25	0.75	2.5	5.0	50.0	1.6
6-9	500	10,000	225.0	0.1	0.35	3.8	10	24	67	123	50.4	0.072
6-15	500	7,000	1001.2(a)	60	355	--	--	--	--	--	0.375	0.00027
6-19	500	5,800	1000.5(a)	50	182	655	--	--	--	--	0.725	0.00062
6-26	500	4,000	1004.6(a)	215	920	--	--	--	--	--	0.206	0.00010
6-28	500	2,500	1005.8(a)	375	--	--	--	--	--	--	0.155	0.00006

(a) Test discontinued.

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TABLE 6. CREEP-RUPTURE DATA FOR ANNEALED ZIRCALOY 2 SHEET MATERIAL TESTED IN THE LONGITUDINAL DIRECTION AT TEMPERATURES RANGING FROM 150 TO 500 C

Specimen	Temperature, C	Stress, psi	Rupture Time, hr	Time to Reach Indicated Percentage Deformation, hr							Final Deformation, per cent	Minimum Creep Rate, per cent per hr
				0.1	0.2	0.5	1.0	2.0	5.0	10		
3-6	150	30,000	141.6	--	--	--	0.1	0.6	9.0	33	40.0	0.17
3-5	150	27,500	1000.4(a)	--	--	0.06	0.28	4.0	97	490	15.3	0.01
3-7	150	23,000	1013.0(a)	--	--	16	--	--	--	--	0.91	0.00012
3-16	150	20,000	1009.0(a)	--	--	--	--	--	--	--	0.70	0.00002
3-1	230	27,500	2.5	--	--	--	--	0.02	0.25	0.85	48.8	8.0
3-4	230	26,000	6.2	--	--	--	--	--	0.4	1.8	39.6	3.3
3-2	230	24,000	1006.4(a)	--	--	--	--	0.2	38	--	6.95	0.00007
3-15	345	22,000	1004.6(a)	--	--	--	--	--	10	--	5.66	0.00053
3-11	345	20,000	1008.3(a)	--	--	--	--	0.05	--	--	3.16	0.00033
3-10	345	15,000	1052.7(a)	--	--	1.7	--	--	--	--	0.64	0.00005
3-12	425	17,500	80.0	--	0.15	0.95	3.5	8.5	22	43	42.0	0.15
3-13	425	12,500	1029.4(a)	--	--	27	120	365	--	--	4.42	0.0035
3-14	425	9,000	1030.0	0.8	112	--	--	--	--	--	0.36	0.00015
3-3	500	12,500	40(b)	--	--	0.3	0.7	1.6	4.2	9.6	59.2	0.90
3-8	500	8,000	1054.2(a)	2.5	11	42	92	200	405	735	16.6	0.0093
3-9	500	5,000	1006.3(a)	75	265	1100 est.	--	--	--	--	0.465	0.00035

(a) Test discontinued.

(b) Estimated rupture time.

TABLE 7. CREEP-RUPTURE DATA ON COLD-WORKED ZIRCALOY 2 SHEET MATERIAL TESTED IN THE TRANSVERSE DIRECTION AT TEMPERATURES RANGING FROM 150 TO 500 C

Specimen	Temperature, C	Stress, psi	Rupture Time, hr	Time to Reach Indicated Percentage Deformation, hr							Final Deformation, per cent	Minimum Creep Rate, per cent per hr
				0.2	0.5	1.0	2.0	5.0	10			
5-19	150	74,000	0.35	--	--	--	--	0.25	--	--	10.4	2.3
5-17	150	70,000	27.6	--	--	--	0.2	23	--	--	13.2	0.014
5-15	150	67,000	313.2	--	--	--	1.4	--	--	--	7.2	0.0009
5-20	150	64,000	1000.0(a)	--	--	--	575	--	--	--	1.02	0.000045
5-10	150	60,000	522.0(a)	--	--	--	--	--	--	--	1.14	0.00004
5-6	150	54,000	1002.9(a)	--	--	0.05	--	--	--	--	0.63	0.00002
5-18	230	65,000	0.25	--	--	--	0.01	0.21	--	--	13.2	4.5
5-14	230	59,000	46.1	--	--	--	0.5	--	--	--	7.6	0.012
5-11	230	56,000	876.5(a)	--	--	--	170	--	--	--	1.21	0.00023
5-21	230	53,000	1004.1(a)	--	--	--	12	--	--	--	1.25	0.00008
5-24	230	40,000	1006.4(a)	--	--	--	--	--	--	--	0.725	0.00001
5-13	345	45,000	23.2	--	--	--	1.3	13	--	--	14.0	0.083
5-9	345	41,000	448.2	--	--	--	9.5	168	427	--	17.2	0.0054
5-23	345	25,000	1000.6(a)	--	--	30	--	--	--	--	0.765	0.00011
5-26	345	20,000	1007.0(a)	--	0.1	--	--	--	--	--	0.445	0.00005
5-5	425	38,500	1.15	--	--	--	0.07	0.42	1.05	--	16.8	2.73
5-3	425	32,000	34.7	--	--	0.05	1.2	8.0	--	--	22.8	0.15
5-8	425	24,000	867.1	--	--	1.05	37	176	538	832	24.8	0.0071
5-16	425	10,000	1072.5(a)	1.2	40	990	--	--	--	--	0.52	0.00017
5-25	425	5,000	1001.0(a)	15	--	--	--	--	--	--	0.187	0.000075
5-1	500	34,000	0.05	--	--	--	--	--	--	--	19.6	--
5-2	500	30,000	0.45	--	--	--	0.03	0.12	0.31	--	22.4	10.3
5-4	500	22,500	7.9	--	--	0.02	0.4	1.5	4.3	9.0	31.2	0.85
5-7	500	11,000	338.5	--	0.13	3.6	18	51	122	202	42.0	0.030
5-12	500	5,000	1000.5(a)	2.6	17	70	318	840	--	--	2.22	0.0017
5-22	500	2,000	1005.0(a)	165	420	--	--	--	--	--	0.342	0.0002

(a) Test discontinued.

TABLE 8. CREEP-RUPTURE DATA FOR COLD-WORKED ZIRCALOY 2 SHEET MATERIAL TESTED IN THE LONGITUDINAL DIRECTION AT TEMPERATURES RANGING FROM 150 TO 500 C

Specimen	Temperature, C	Stress, psi	Rupture Time, hr	Time to Reach Indicated Percentage of Deformation, hr							Final Deformation, per cent	Minimum Creep Rate, per cent per hr
				0.1	0.2	0.5	1.0	2.0	5.0	10		
7-4	150	68,000	37.3	--	--	--	0.05	26.4	--	--	6.8	0.02
7-15	150	66,000	1054.4(a)	--	--	--	5.5	--	--	--	1.565	0.0002
7-9	150	60,000	1001.6(a)	--	--	--	3.5	--	--	--	1.125	0.00002
7-12	230	59,000	37.6	--	--	--	1.2	--	--	--	12.0	0.3
7-13	230	55,000	1004.0(a)	--	--	--	35	--	--	--	1.35	0.0002
7-8	230	31,000	1002.7(a)	--	--	--	--	--	--	--	0.41	0.000035
7-16	230	30,000	1008.6(a)	--	--	--	--	--	--	--	0.37	0.000025
7-3	345	43,500	105.5	--	--	0.1	5.4	43	--	--	16.0	0.003
7-18	345	35,000	1073.4(a)	--	--	--	23	515	--	--	2.5	0.00096
7-7	345	25,000	1065.2(a)	--	--	--	400	--	--	--	1.7	0.000075
7-5	425	30,000	44.3	--	--	0.05	0.6	4.1	23	34	26.0	0.11
7-10	425	15,000	1132.5(a)	--	1.8	90	650	--	--	--	1.35	0.00071
7-17	425	5,000	1012.2	950	--	--	--	--	--	--	0.11	0.00008
7-1	500	16,000	43.8	--	--	--	1.5	5.8	16.4	28	39.6	0.23
7-2	500	5,000	1005.3(a)	3.7	15	62	210	635 est.	--	--	1.94	0.0017
7-11	500	2,000	1172.0(a)	10	80	1350 est.	--	--	--	--	0.49	0.00010

(a) Test discontinued.

TABLE 9. CREEP-RUPTURE DATA ON ANNEALED AND COLD-WORKED ZIRCALOY 2 BAR MATERIAL
TESTED AT TEMPERATURES RANGING FROM 150 TO 500 C

Specimen	Temperature, C	Stress, psi	Rupture Time, hr	Time to Reach Indicated Percentage Deformation, hr						Final Deformation, per cent	Reduction of Area, per cent	Minimum Creep Rate, per cent per hr
				0.2	0.5	1.0	2.0	5.0	10			
<u>Annealed Condition</u>												
2-5	150	37,500	3.1	--	--	--	--	0.05	0.6	40.8	42.9	8.0
2-11(a)	150	33,500	1002.1(b)	--	--	--	3.5	20	375	13.8	--	0.006
2-7	150	30,000	1008.6(b)	--	--	--	2.0	32	575	12.0	--	0.0046
2-8	230	31,000	0.6	--	--	--	--	--	0.13	35.9	61.9	31.4
2-4	230	27,500	48.3	--	--	--	--	0.5	6.3	34.9	55.1	0.3
2-14(c)	230	26,000	383.0(b)	--	--	--	<0.1	--	--	4.64	--	0.0001
2-9	230	26,000	1002.7(b)	--	--	--	1.0	20	--	5.49	--	0.0004
2-1	345	20,000	1002.2(b)	--	--	--	95	--	--	2.43	--	0.00033
2-10	425	21,000	25.9	--	--	--	--	3.4	10.7	34.6	75.5	0.69
2-2	425	17,500	327.4	--	--	0.3	4.5	37	104	40.9	81.6	0.074
2-3	500	15,000	6.7	--	--	--	0.1	0.63	1.8	58.4	90.0	4.4
2-13(c)	500	10,900	149.2	--	0.6	3.2	9.0	30	59	46.4	81.7	0.13
2-6	500	10,000	286.6	--	1.0	5.0	16.5	49	103	64.0	90.3	0.09
2-12	500	8,000	1008.8(b)	1.0	6.0	25	75	255	550	20.6	--	0.017
<u>Cold-Worked Condition</u>												
4-2	150	80,000	(d)	--	--	--	--	--	--	18.5	36.1	--
4-4	150	72,000	0.2	--	--	--	--	--	0.11	21.2	33.9	58.5
4-1	230	68,000	(d)	--	--	--	--	--	--	17.6	52.3	--
4-3	230	62,000	1004.9(b)	--	--	1.0	92	--	--	3.8	--	0.0016

(a) Test made in vacuum.

(b) Test discontinued.

(c) Test conducted in helium gas.

(d) Failure occurred on loading.

Note: Tests at 150 and 230 C were made in air; 345, 425, and 500 C tests were made in vacuum.

Tables 5 through 9 contain the following test data:

- (1) Rupture times
- (2) Time to attain 0.1, 0.2, 0.5, 1, 2, 5, and 10 per cent total deformation
- (3) Final deformation when test failed or was discontinued
- (4) Minimum creep rate attained during test.

In addition, data on reduction of area for the bar specimens are given in Table 9. The rupture times for the various conditions of Zircaloy 2 were plotted as curves of stress versus rupture time on semilog coordinates as shown in Figures 4, 5, and 6. Where possible, 1-, 10-, 100-, and 1000-hr rupture-strength values for the transverse sheet were then taken from these curves and summarized in Table 10. It was necessary to extrapolate the data and estimate the rupture-strength values in some cases because the rupture times did not extend from 1 to 1000 hr for all test conditions. Moreover, at some temperatures there were only two and in one instance at 345 C (Figure 4) only one rupture-time value was plotted in getting the stress-rupture curves. The 1-, 10-, 100-, and 1000-hr rupture-strength values for the Zircaloy 2 bar material are incomplete because only a few tests were made on bar stock.

Although creep-rupture tests were also conducted on Zircaloy 2 sheet in the longitudinal direction, a similar summarization of 1-, 10-, 100-, and 1000-hr rupture-strength values was not made for two reasons. First, there were only a few creep-rupture tests made on the longitudinal sheet, generally, only one at each temperature, and not enough to produce independent stress-rupture curves as shown in Figures 4, 5, and 6. Secondly, the few tests that were made on the longitudinal sheet indicated its creep-rupture strength to be equivalent to the transverse sheet as shown in Figures 4 and 5. Therefore, the summarizing values given in Table 10 for the transverse Zircaloy 2 sheet should serve to indicate the strength of the longitudinal sheet as well.

Examination of the data in Table 10 shows the 1-hr rupture strength of annealed Zircaloy 2 sheet in the transverse direction to decrease from about 35,200 psi at 150 C to 17,700 psi at 500 C. The 1000-hr rupture strength drops from 27,700 psi at 150 C to about 7,200 psi at 500 C. In the cold-worked condition, this material has a 1-hr rupture strength of 73,000 psi at 150 C and 27,500 psi at 500 C. The 1000-hr rupture strength of this cold-worked sheet is about 65,500 psi at 150 C and only 7,500 psi at 500 C. Thus, Zircaloy 2 sheet has at least twice the rupture strength in the cold-worked as in the annealed condition at temperatures of 150, 230, 345 C. At temperatures in excess of 345 C, the difference in strength of Zircaloy 2 produced by the two conditions decreases with increase of either time or temperature. Although most 1000-hr rupture stresses were estimated, these data indicate that at 500 C the 1000-hr rupture strength of both the annealed and cold-worked materials is very nearly the same.

The test data for the annealed bar stock of Zircaloy 2 indicate that it has, in most instances, a slightly greater rupture strength than the sheet material. The superiority seems to be greater at the lower temperatures. The data on the cold-worked Zircaloy 2 bar material, which are limited to 150 and 230 C, indicate that, in general, this material also has a slightly superior strength in bar form. As with the sheet material, Zircaloy 2 bar appears to have about twice the strength in the cold-worked as in the

TABLE 10. SUMMARY OF STRESS-RUPTURE-STRENGTH
PROPERTIES OF ZIRCALOY 2

Temperature, C	Stress to Produce Rupture in Indicated Time, psi			
	1 Hr	10 Hr	100 Hr	1000 Hr
<u>Annealed Sheet, Transverse Direction</u>				
150	35,200	32,500	30,000	27,700 est.
230	28,000	26,000	24,000	23,000 est.
345	22,200	22,000	21,800 est.	21,600 est.
425	21,300	19,000	16,800	14,900
500	17,700	15,100	11,400	7,200 est.
<u>Cold-Worked Sheet, Transverse Direction</u>				
150	73,000	70,500	68,000	65,500 est.
230	63,000	60,500	58,000	55,500 est.
345	50,000	46,000	43,000	40,000 est.
425	38,000	34,000	29,000	24,000
500	27,500	21,500	14,500	7,500 est.
<u>Annealed Bar, Longitudinal Direction</u>				
150	38,000	37,000	36,000	35,000 est.
230	30,000	29,000	27,500	26,000
345	--	--	23,000 est.	21,000 est.
425	--	22,000	19,000	16,000
500	17,000 est.	14,000	11,500	9,000 est.
<u>Cold-Worked Bar, Longitudinal Direction</u>				
150	71,000	70,500	--	--
230	66,000	65,000	64,000	63,000

annealed condition. Metallographic examination of the structure of the rod and the sheet prior to testing showed that the grain size of the sheet material was approximately twice the grain size of the rod. This difference in grain size is probably one of the contributing factors in the pronounced differences in creep properties at the test temperatures investigated. The difference in creep properties decreased with increasing test temperature. It was also noted that the fracture of the rods was not symmetrical. Necking occurred nonuniformly and the resulting cross section of the rod at fracture was rectangular.

Where sufficient data exist, design curves which relate stress and time for various amounts of total deformation were plotted on semilog coordinates. For this purpose, total deformations of 0.1, 0.2, 0.5, 1.0, 2.0, 5.0, and 10 per cent were selected. The times to reach these deformations during each test were determined, recorded in Tables 5 through 9, and plotted versus appropriate stresses as design curves. Time or stress required to reach other than these deformations can be found by interpolation.

At least partially completed design curves were made for the transverse Zircaloy 2 sheet in both the annealed and cold-worked conditions. Design curves for the annealed material are shown in Figures 7 through 11 and for the cold-worked material in Figures 12 through 15. The limited data obtained on Zircaloy 2 sheet in the longitudinal direction are also shown in Figures 7 through 15. The limited data obtained on Zircaloy 2 sheet in the longitudinal direction are also shown in Figures 7 through 15. The longitudinal tests were made for the purpose of checking the similarity of creep-rupture results in the two directions. Examination of these curves indicates that the creep-rupture strength of Zircaloy 2 sheet is practically identical in the transverse and longitudinal directions. There are some slight variations in strength properties which could be attributed to normal scatter or minor testing differences.

Some of the figures representing design data do not have curves for all the prescribed percentages of deformation. These deformations were either exceeded on loading the test specimen or were never attained during the life of the test. Where design data are almost completely lacking, stress versus time (design) curves were estimated as in Figures 9, 12, and 13.

The design curves (Figures 7 through 15) include the stress-rupture curves that have already been presented in Figure 4, 5, and 6, but are shown again to indicate the ultimate limitations of Zircaloy 2 in design applications. The rupture points on these stress-rupture curves also show the elongation of the specimens at failure. These elongation figures are also presented in Tables 5 through 9, as a final deformation. As expected, the annealed sheet exhibits a relatively large amount of deformation (30 to 60 per cent). The cold-worked sheet is much less ductile, deforming only about 7 to 17 per cent at 150, 230, and 345 C and about 17 to 42 per cent at 425 and 500 C, where annealing of the material probably occurs during test. The annealed Zircaloy 2 bar material appears to have about the same ductility as the sheet; however, cold-worked bar seems to be more ductile than sheet.

The minimum creep rates for all Zircaloy 2 tests, reported as per cent per hr in Tables 5, 6, and 7, were plotted graphically on log-log coordinates as shown in Figure 16 for the annealed sheet, Figure 17 for the cold-worked sheet, and Figure 18 for the bar material. It was hoped that plotting the creep rates versus the stresses on log-log coordinates would produce essentially straight-line curves permitting extrapolation to lower creep rates. Examination of these figures will show that this occurred only to a limited extent. As the creep rates decrease, the slope of the curves of the

stress versus creep rate gradually increase for most of the test temperatures. This change in slope is more pronounced in the cold-work material, probably due to recrystallization taking place.

Figures 19, 20, 21, and 22 show metallographically the effect of time at test temperature. At the 500 C test temperature, the cold-worked Zircaloy 2 recrystallized and grain growth was started. The reason for the change in slope in the curves of stress versus creep rate for the annealed material is not known, however, a possible explanation is that the tests have not been allowed to continue long enough to reach actual minimum creep rates. Because of this continuing change in slope, it becomes very difficult to estimate stresses that will result in a creep rate of 0.00001 per cent per hr.

A summary of the creep strength of Zircaloy 2 is shown in Table 11. These data were taken from the curves of stress versus creep rate in Figures 16, 17, and 18. Stresses that are required to produce creep rates of 0.00001, 0.0001, 0.001, and 0.01 per cent per hr are shown for annealed and cold-worked sheet in the transverse direction. Some data are also shown for the bar material but they are mostly at the higher creep rates of 0.001 and 0.01 per cent per hr. Creep data obtained on longitudinal Zircaloy 2 sheet are also shown in Figures 16 and 17. Based on these results the creep strength of Zircaloy 2 is practically the same in the two rolling directions. The only exception seems to be at 230 C in the cold-worked condition (Figure 17) where two longitudinal specimens stressed at 30,000 and 31,000 psi have what appears to be rather high creep rates. It may be that these tests did not attain their minimum creep rates. Therefore, for the most part, creep as well as the stress-rupture strength of Zircaloy 2 seems to be the same in the longitudinal as in the transverse direction.

The summary of creep-strength values, shown in Table 11, indicates the same order of creep-strength relationship between cold-worked and annealed sheet material as was shown earlier (Table 10) by the stress-rupture results. This also seems to hold true for the bar material as nearly as can be determined by the limited results. This relationship is that the cold-worked materials have, generally, in excess of twice the creep strength of the annealed materials at 150 and 230 C. At 425 and 500 C, the creep-strength ratio of the two conditions of Zircaloy 2 is less than two. The ratio continues to decrease with an increase of both time and temperature. At creep rates of about 0.0002 per cent per hr for 425 C and 0.01 per cent per hr for 500 C, the ratio of cold-worked to annealed Zircaloy 2 creep strength becomes less than one. This means that for these and more severe test conditions the annealed material has the higher creep strength.

A few tests were made under test conditions other than the usual to determine the effect of environment. An annealed Zircaloy 2 bar specimen was tested at 150 C in vacuum rather than in air. Indications from this single test are that the creep strength of the material is slightly greater under these conditions (Figure 18). Two tests on this same annealed Zircaloy 2 bar were made in a helium-gas atmosphere at 230 and 500 C with little, if any, apparent effect on its creep- or rupture-strength properties (Figures 6 and 18). It must be remembered that these are the results of a very limited number of tests and should be considered as indications only.

FIGURE 19. COLD-WORKED ZIRCALOY 2 (25 PER CENT) AFTER
1072 HR AT 1000 PSI AND 425 C

FIGURE 20. ANNEALED ZIRCALOY 2 AFTER 1005 HR AT
10,000 PSI AND 425 C

FIGURE 21. COLD-WORKED ZIRCALOY 2 (25 PER CENT) AFTER 1172 HR AT 2000 PSI AND 500 C

FIGURE 22. ANNEALED ZIRCALOY 2 AFTER 1005 HR AT 4000 PSI AND 500 C

TABLE 11. SUMMARY OF CREEP-STRENGTH PROPERTIES
OF ZIRCALOY 2

Temperature, C	Stress, psi, to Produce Indicated Creep Rate, per cent per hr			
	0.00001	0.0001	0.001	0.01
<u>Annealed Sheet, Transverse Direction</u>				
150	22,000	24,000	25,500	27,000
230	20,500	22,000	23,000	24,000
345	9,000	19,000	20,800	21,000
425	6,000 est.	9,500	11,400	14,000
500	<2,500	4,000	6,000	8,000
<u>Cold-Worked Sheet, Transverse Direction</u>				
150	46,000 est.	63,000	66,000	68,000
230	40,000	54,000	56,000	58,000
345	10,000 est.	24,000	33,000	42,000
425	<5,000	6,400	15,000	24,500
500	<1,500	1,500	4,000	8,000
<u>Annealed Bar, Longitudinal Direction</u>				
150	--	--	29,000 est.	31,000
230	--	25,500	26,000	26,500
345	--	19,000 est.	20,300 est.	21,500 est.
425	--	--	--	14,500 est.
500	--	--	--	7,500
<u>Cold-Worked Bar, Longitudinal Direction</u>				
150	--	--	--	68,000 est.
230	--	--	60,000 est.	--

TUBE-BURST TESTS

Fabrication History

The bursting strengths of welded zirconium and of seamless Zircaloy 2 tubes were determined at 28, 150, 230, 345, and 460 C. The internally ribbed tubes were formed by two different processes. The zirconium tube had been formed by roll forming and longitudinal closure made by inert arc welding. Two zirconium ribs approximately 1.5 in. apart were first welded on 0.068-in. -thick annealed zirconium sheet. The tube was formed in a Yoder-type mill in seven steps with specially designed rolls. An arc-welded closure was made in an inert atmosphere along the length of the tube. The welded tubes were cold swaged in a special die to reduce the wall thickness to approximately 0.065 in. and smooth up the welded seam and the ribs.

The Zircaloy 2 tubes were extruded from a billet into 20-ft lengths and cold worked on Rockrite tube-reducing machines. These tubes were tested in the as-worked condition. The amount of cold work was estimated to be approximately 50 per cent. The wall thickness varied from 0.045 in. between the ribs to 0.065 in. at a point diametrically opposite.

Test Method

Samples to be tested were prepared from 5-in. lengths of tubing. This was the minimum length that could be used without an end effect. Although several types of end closures were tried, the most successful was a combination weld and mechanical seal.

This end-closure method consisted of welding an end cap to the wall of the tubing at each end of the test piece. A hole was drilled and tapped in the center of one end cap to provide a connection for the hydraulic system. In initial tests, it was found that the welds were not sufficiently strong to withstand the end forces on the tube and failure occurred at the weld prior to tube failure. In order to provide additional support at the ends, stainless steel end caps, large enough to slip easily over the welded end of the tube and down approximately 0.375 to 0.50 in., were used. One cap was drilled out to allow access to the hydraulic fitting attached to the tube. The two caps were connected by two 0.50-in. -diameter Zircaloy 2 bolts running the length of the tube on each.

The bolts were tightened only enough to hold the end caps snugly in place, but no compressive load was applied to the tube.

Although there was a heat-affected zone adjacent to the welds at the tube ends, the end caps supported the tube in this area and no failures were experienced in this weakened area.

The bursting strength of the tubes was measured by internally pressurizing with a liquid. Water was used for tests at 28 and 150 C. For tests over 150 C, a chlorinated biphenyl (Aroclor 1248, Monsanto Chemical Company) was used. Aroclor has properties which make it satisfactory as a hydraulic fluid in the intermediate-temperature range up to 400 C. It is liquid at room temperature and can be poured easily. It has a flash point in the range 193-196 C as determined by the Cleveland open-cup method, but no fire point has been found. There was a tendency at 400 C and above for the liquid to

dissociate, forming HCl gas and a carbon precipitate. The liquid serves quite well as a transmitter of pressure for short periods of time above 400 C. For prolonged periods at this temperature or above, some difficulties may be encountered due to the accumulation of HCl gas. The time involved in the tests performed was such that no difficulties from accumulating gas were experienced.

A hydraulic system was chosen for two reasons: (1) to minimize the stored-energy characteristic of a liquid system as compared with a gas system; and, (2) the availability of pressurizing equipment. The major components of the system were the hydraulic pump, the intensifier or accumulator, and the test piece. A schematic drawing of pressure equipment, heating apparatus, and test piece are shown in Figure 23.

The test procedure was essentially as follows: (1) The test piece and line leading from the accumulator were filled with liquid and a connection made to the system. (2) The specimen-shutoff valve was closed and approximately 400 cm³ of liquid was pumped into the accumulator, which had a capacity of 2 liters. (3) Helium gas at tank pressure, 2000 to 2500 psi, was charged into the accumulator above the liquid. By pumping fluid into the accumulator, the pressure in the accumulator was increased to a value greater than that required to burst the test specimen. (4) The pressure was gradually increased in the test piece by slowly opening the specimen-shutoff valve. The pressure to cause fracture was read at the gage in the line with the specimen.

Tests at elevated temperatures were made by pressurizing in the same manner after the test specimen had been raised to the desired temperature. Heating was done by a resistance-wire furnace having a 30-in. hot zone. Temperature was measured and controlled by three 28-gage Chromel-Alumel thermocouples attached to each end in the center of the test section. A Leeds and Northrup two-point-recorder one-point controller and a switching arrangement allowed temperature measurements to be made at each thermocouple. The temperature was controlled by the center thermocouple. There was 25 to 50 C difference in temperature between the ends and the center of the test piece while the sample was coming to temperature. However, as the test temperature was approached and attained, the temperature difference decreased. By holding approximately 20 min at the test temperature prior to testing, the temperature difference from end to center was within ± 2.5 C.

Test Results

Bursting pressures obtained under various test conditions are tabulated in Table 12. Duplicate tests were made at most of the test temperatures, but were limited by the supply of available tubing.

In the welded zirconium tubing, all of the fractures occurred at the junction between the weld nugget and the base metal of the longitudinal closure weld. Figure 24 shows the typical appearance of the welded tubes fractured at various temperatures. A hardness traverse across the weld is shown in Figure 25, and the approximate location of the fracture is indicated. The microstructure at the fracture was coarse grained and martensitic, typical of zirconium quenched from a relatively high temperature.

All of seamless Zircaloy 2 tubes failed between the ribs where the tube wall was thinnest. The wall thickness between the ribs of the single tube available for testing was approximately 0.045 in. The remainder of the tube averaged about 0.065 in.

FIGURE 23. DIAGRAMMATIC LAYOUT OF TESTING EQUIPMENT

A 22269

TABLE 12. BURSTING PRESSURES OF EXTRUDED ZIRCALOY 2 TUBE
AND COLD-FORMED AND WELDED ZIRCONIUM TUBES
AT ELEVATED TEMPERATURES

Temperature, C	Seamless Zircaloy 2		Welded Zirconium	
	Bursting Pressure, psi	Calculated Hoop Stress, psi	Bursting Pressure, psi	Calculated Hoop Stress, psi
28	7,460	136,000	6,250	82,250
28	7,100	129,000	5,400	69,000
28	6,800	127,000	5,650(a)	73,300
28	--	--	6,000(a)	74,750
28	--	--	5,100(b)	64,500
28	--	--	4,200(b)	54,000
150	5,950	111,000	6,250	82,000
150	--	--	4,800	60,000
150	--	--	2,500	31,300
195	5,650	103,000	--	--
230	--	--	3,350	43,400
345	4,005	72,800	2,500	32,800
345	4,005	73,000	2,750	35,700
400	3,150	57,500	2,200	29,400

(a) Shot peened.

(b) Shot peened, annealed 1 hr at 1500 F in vacuum.

44

44

1X

28 C

150 C

225 C

350 C

460 C

N26474

FIGURE 24. WELDED ZIRCONIUM TUBE AFTER TESTS AT VARIOUS TEMPERATURES

45

45

FIGURE 25. HARDNESS OF WELDED SEAM AND ADJACENT AREA OF WELDED ZIRCONIUM TUBE

The calculated ultimate strength based on Barrow's formula for thin-walled tubes

$$\sigma_c = \frac{PD}{2t} \quad (4)$$

where

σ_c = circumferential stress in psi

P = internal pressure

D = inside diameter in inches

t = wall thickness in inches

is shown in Table 12. The ultimate strength of the zirconium tubes was higher than that observed in sheet tensile tests on cold-rolled and annealed zirconium sheet. The difference is believed to be the result of the much higher strain rate in the tube-bursting tests and to inherent differences in various lots of arc-melted sponge zirconium.

The seamless Zircaloy 2 had a consistently higher bursting strength than the welded-zirconium tube, as would be expected. The amount of cold work in the Zircaloy 2 tube was estimated to be approximately 50 per cent and this cold work contributed to the high strengths observed.

The effect of shot peening the welded longitudinal closure weld prior to testing at 28 C was investigated. The joint, 1/4 in. on each side of the weld was shot peened with steel shot in a Pangborn Rotoblast machine. These tubes were tested at 28 C and failed at 5650 and 6000 psi as compared with 5400 and 6250 psi internal pressure for untreated tubes. As in the untreated samples, the fracture occurred at the junction of the weld nugget and the base material of the longitudinal closure weld. Shot peening had no significant effect on the burst strength and did not change the place or mode of fracture.

Two additional samples of the welded tube were annealed for 1 hr at 816 C in vacuum after shot peening. These tubes failed at 4200 and 5100 psi, and the fracture occurred adjacent to one of the ribs in the heat-affected zone, rather than at the closure weld. Metallographic examination of the fracture area showed that the weld joining the rib to the sheet prior to roll forming and final swaging was incomplete. As shown in Figure 26, a small crack was found at the edges of one of the ribs. This resulted in a slight reduction in wall thickness. Apparently, annealing the tube resulted in improved properties in the longitudinal closure weld, so that failure occurred at the reduced section caused by the crack.

100X

Polarized Light

N33830

FIGURE 26. CRACK AT EDGE OF INTERNAL RIB

CONCLUSIONS

The effect of annealing time on the elevated-temperature tensile strength of cold-worked zirconium and Zircaloy 2 was investigated.

It was concluded that at 500 C the rate of softening was rapid and the increased strength resulting from cold working could not be utilized.

At lower temperatures, the cold-worked sheet maintained its strength advantage over annealed material for long periods. At 425 C for example, annealed Zircaloy 2 had a 0.2 per cent offset yield strength of 16,200 psi, but the 25 per cent cold-worked sheet after 500 hr at 325 C still had a yield strength of 34,200 psi.

The creep strength of annealed Zircaloy 2 decreased rapidly with increasing temperature in the range from 150 to 500 C, the maximum temperature investigated. The stress to produce a creep rate of 10^{-5} per cent per hr was 25,000 psi at 150 C and 3200 psi at 500 C.

At low temperatures, the cold-worked material had a higher creep strength than annealed sheet. At 500 C, however, the cold-worked material apparently recrystallized under stress, and the creep strength was lower than that of the annealed sheet.

No significant directional properties were found in the rolled sheet.

Welded zirconium tubing failed at the longitudinal-seam weld under hydraulic loading. The bursting pressure varied from 5400 to 6250 psi at room temperature. At 400 C the bursting pressure in a single test was 2200 psi.

Seamless Zircaloy 2 tubing failed between the ribs because the wall thickness was a minimum at this point. The bursting pressures were considerably higher in the

seamless tubing than in the welded tubing. At room temperature, the bursting pressure varied from 6800 psi to 7460 psi. At 400 C, a pressure of 3150 psi was required.

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