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PARAMETRIC STUDIES
ON THE
SHORT-TERM TENSILE MECHANICAL PROPERTIES
OF
Zr - 10 U ALLOY HYDRIDES
(Title Unclassified)

AEC Research and Development Report

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By

L.B. LUNDBERG

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ABSTRACT

Test methods were developed, and short-term tensile tests performed, on the Systems for Nuclear Auxiliary Power (SNAP) fuel — zirconium - 10 wt % uranium alloy hydride. The effects on mechanical behavior, resulting from variations in hydrogen content in the range of 1.45 to 1.94 H/Zr* and carbon content in the range of 0.03 to 0.47 wt %, were studied from room temperature to 1450°F. Hydrogen content was found to have its greatest effect on the Young's modulus of this material, causing a discontinuous drop from about 17×10^6 to 4×10^6 psi across the δ - ϵ hydride two-phase field at room temperature. Varying the carbon content caused the ultimate tensile strength to reach a maximum value of about 10,000 psi at the 0.15 wt % C level, for the hydrogen content and temperature range studied. At higher carbon contents, the tensile strength began to decrease toward the level shown by the noncarbon-modified alloy hydrides — about 4,000 psi. Temperature generally had a normal effect on the tensile properties of this brittle material, causing the strength to rise slightly with temperature. The Young's modulus dropped approximately linearly, from about 7×10^6 psi at room temperature to 3×10^6 psi at 1350°F. Cleavage of the ϵ grains was observed to propagate along a preferred crystallographic plane, which appeared to be the basal (100) plane of the tetragonal structure.

*H/Zr, in this report, indicates an "effective" hydrogen-to-zirconium atomic ratio, based on the assumption that all the zirconium added to the fuel as ZrC does not hydride.

I. INTRODUCTION

The fuel material for the Systems for Nuclear Auxiliary Power (SNAP) reactors (SNAP 2, 8, and 10A) is a hydrided zirconium - 10 wt % uranium (Zr - 10 U) alloy, with hydrogen/zirconium ratios $(H/Zr)^*$ ranging from 1.6 to 1.9. One important design consideration is the mechanical behavior of this nuclear fuel material under operating conditions and during launch and reentry from an earth orbit. Other investigators^{1,2} have reported on the mechanical behavior of Zr-U alloy hydrides, but their work was limited to temperatures below 1200°F, and did not include carbon-modified material (carbon, in the form of zirconium carbide, is now added to the alloy melt to improve the yield of uncracked, massively hydrided fuel rods³).

In the present investigation, the short-term tensile properties of the Zr - 10 U alloy hydride were related to temperature, carbon content, and hydrogen content. These parameters were investigated as follows:

- a) At room temperature, the effects of hydrogen and carbon content were investigated.
- b) Carbon content was held constant (0.15 wt %) at 1350°F, as the hydrogen compositions were varied.
- c) At 1450°F, the hydrogen level was held constant (1.74 H/Zr) as the carbon content of the alloy was changed.
- d) The temperature influence on the tensile properties was studied from 1250 to 1450°F on a single alloy composition (1.74 H/Zr, 0.15 wt % C).

In the tests, the main objective was to obtain accurate stress-strain curves. These curves provided the information necessary for determining the yield strength, ultimate tensile strength, and Young's modulus of the materials tested. These data were then correlated with the test parameters (carbon content, hydrogen content, and temperature). As will be shown later, the ultimate tensile strengths determined in this study are not necessarily the true tensile strengths of this material, due to eccentricities in loading. However, they are mutually comparable, and useful in studying the effects of the variables. The values determined for yield strength and Young's modulus are representative of the true behavior of this material.

*H/Zr, in this report, indicates an "effective" hydrogen-to-zirconium atomic ratio, based on the assumption that all the zirconium added to the fuel as ZrC does not hydride.

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a. Design Used for Room-Temperature Tests Only

b. Design Used for Both Room- and Elevated-Temperature Tests

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Figure 1. Tensile Specimen Designs
(Figure Unclassified)

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II. EXPERIMENTAL

Tensile tests were performed on samples of Zr - 10 U alloy with hydrogen contents ranging from 1.45 to 1.94 H/Zr, and carbon contents from 0.03 to 0.47 wt %. The test temperatures ranged from room ambient to 1450°F.

The Zr - 10 U alloy hydrides are basically brittle materials, and consequently pose a problem with regard to applying a tensile load and attaching a strain measuring device. The load application problem was partially solved by using self-aligning grips, which held the specimens by their test section radii. The strain measuring problem, which, in this study, existed only for the elevated-temperature tests, was overcome by attaching gage strips to the specimen and measuring the strain optically.

Since the method of tensile testing differed substantially between room temperature and elevated temperature, due to dissociation pressure of the hydride at the elevated temperatures, the experiments performed in these two temperature regions will be discussed separately.

A comprehensive post-test analysis program was carried out on the samples used. The experimental techniques utilized in the post-test analysis included metallography, fractography, chemical analysis, and x-ray diffraction.

A. SAMPLE PREPARATION

The tensile samples were machined on a standard lathe, from rods of Zr - 10 U alloy hydride which had been prepared by massive hydriding methods.⁴ The Zr - 10 U alloy starting material had been previously prepared by consumable arc melting in an inert atmosphere (carbon added in the form of -325 mesh ZrC powder), and fabricated into round bars by a warm extrusion process. A typical impurity analysis for the hydrided alloy is given in Table 1.

Two different specimen designs, as shown in Figure 1, were used in the tensile tests. The design of Figure 1a was used only in the room-temperature tests, while the sample design in Figure 1b was used in both low- and high-temperature regions.

B. ROOM-TEMPERATURE TENSILE TESTS

Room-temperature tensile tests were performed on both a Baldwin-Lima-Hamilton Universal Tensile Tester, at a strain rate of 0.001 in./in.-min, and a

7573-5180A

Figure 2. Elevated-Temperature Tensile-Test Equipment
(Figure Unclassified)

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Figure 3. Elevated-Temperature Tensile-Test Specimen
With Platinum Gage Strip Attached
(Figure Unclassified)

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TABLE 1
IMPURITY ANALYSIS FOR THE Zr - 10 U ALLOY HYDRIDE
TENSILE SPECIMENS*

Al	<25	Cu	75	O	1550
B	3	Fe	300	Pb	< 5
Ba	< 5	Li	< 2	Si	< 25
Be	< 1	Mg	< 5	Sn	< 2
Bi	< 5	Mn	10	Ti	5
Ca	<25	Mo	< 10	V	< 5
Cd	< 0.5	N	500	Zn	< 50
Co	< 5	Na	< 50	Hf	< 100
Cr	40	Ni	< 10		

*All values are in parts per million

Model TTCL Instron Tensile Tester, at effectively the same strain rate. Both machines were equipped to automatically plot load-strain curves. In all cases, the sample strain was measured continuously with a Baldwin-Lima-Hamilton microformer-type extensometer. The samples of the design shown in Figure 1a were held either with self-aligning grips which closely mated the radii at each end of the test section or with standard Templin grips. The samples of Figure 1b design were always tested in the self-aligning, test-section radius grips.

C. ELEVATED-TEMPERATURE TENSILE TESTS

Elevated-temperature tensile tests were performed in a special vacuum-pressure retort furnace, mounted on a Model TTCL Instron Tensile Tester. A photograph of the experimental setup is given in Figure 2. The retort furnace was fitted with sealed optical ports, through which telescoping platinum gage strips could be viewed. The platinum gage strips were tied to the specimen with molybdenum wire, as seen in Figure 3. An optical extensometer, consisting of a 20X microscope fitted with a Filar eyepiece, measured the displacement of the two halves of the gage strips during the tests.

The sample pull rods were passed through water cooled, frictionless, packing-gland type seals, located at each end of the retort, and the samples were connected to these pull rods with special test-section radius grips. A unique opposed U-cup design of the pull rod seals, which can be seen in the schematic of the retort furnace (Figure 4), provided a positive, frictionless sliding seal for

Figure 4.
Schematic of
Retort Furnace
(Figure Unclassified)

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Figure 5. Schematic of
Furnace-Temperature-Profile
Control Circuit
(Figure Unclassified)

7568-1548@

both vacuum and pressure testing. The sample grips were designed to be self aligning; but, as will be shown later, the aligning characteristics of these grips were not completely adequate for true uniaxial loading of the test specimens.

The furnace was wound with Nichrome wire, and heated by electrical resistance. Temperature was controlled with a standard saturable-core-reactor control system. Unsheathed Chromel-Alumel thermocouples, attached directly to the sample, were used for temperature measurement. One couple was used for control, a second for over-temperature protection, and two more, each located at opposite ends of the test section, were used for accurate temperature measurements, using a manually operated Rubicon millivolt potentiometer. The temperature of the test section of the specimen was held uniform within $\pm 2^\circ\text{F}$, with the aid of a four-zone, bucking type, temperature-profile control system used in the test furnace. A schematic of the circuit of this system is given in Figure 5. The hydrogen retort pressures used in the tests were measured with a 0 to 50 psia strain gage type transducer, accurate to ± 0.05 psia. Pressures were controlled with a dual stage, diaphragm-type pressure regulator.

The elevated-temperature tensile tests on the Zr - 10 U alloy hydrides were performed at 1250, 1350, and 1450°F. Samples containing a nominal 0.05, 0.15, and 0.30 wt % carbon were tested at 1450°F, under a hydrogen pressure of 45 psia, to determine the effects of carbon content on the tensile properties of fuel with 1.74 H/Zr.* Samples containing a nominal 0.15 wt % carbon were tensile tested at 1350°F, under three different pressures of hydrogen (5.9, 16.2, and 45.6 psi), to examine the effects of hydrogen content. These pressure-temperature conditions provided hydrogen contents of 1.67, 1.74, and 1.80 H/Zr, respectively. At the lowest temperature, 1250°F, tests were performed on 0.15 wt % C material, at 5.1 psia (1.74 H/Zr), to complete the temperature-effect study on the tensile properties of this material. In summary:

- 1) The effects of temperature were determined for 0.15 wt % C, 1.74 H/Zr fuel material
- 2) The effects of zirconium carbide additions in 1.74 H/Zr material were determined at 1450°F

*This is the equilibrium hydrogen-zirconium atomic ratio for this temperature and hydrogen pressure, according to Johnson.⁵

- 3) The effects of hydrogen content in the 0.15 wt % C fuel material were examined at 1350°F.

As a consequence of the brittle behavior of the Zr - 10 U alloy hydrides, particular care was taken in heating each specimen to the desired test temperature, so as not to crack it. The specimens were heated as rapidly as possible to 1000°F, where the dissociation of the hydride became appreciable. At this temperature, the heating rate was reduced to about 200°F/hr. To prevent dehydriding of the specimen, the hydrogen pressure in the retort was increased with temperature, so as to follow the isochore dissociation pressure curve for the desired hydrogen content. After attaining the proper temperature-pressure conditions, relative to the desired H/Zr, the specimen was allowed to soak for at least 1 hr, to attain equilibrium conditions. Some adjustment of hydrogen content was required for all the samples, since their pretest hydrogen contents were not exactly that desired during the tensile tests.

Each of the tensile tests was performed at a constant crosshead speed of 0.002 in./min. The load applied to the specimen was automatically measured and recorded by the testing machine, but the resultant sample elongation was plotted manually, from readings taken with the optical extensometer at incremental loads. Each specimen was loaded to failure, and a stress-strain diagram was constructed. After the samples were tested to failure, some were cooled with intermittent removal of hydrogen pressure from the retort, in order to follow, as close as possible, the dissociation pressure isochore for the particular sample. Chemical analysis was then performed, to determine whether these samples had achieved the desired hydrogen content prior to the tensile test.

D. POST-TEST ANALYSIS

Extensive analyses were performed on the tested samples. Vacuum extraction techniques were used to determine the hydrogen concentration in all the room-temperature samples, and in a select few of the elevated-temperature samples. The carbon contents of all the samples were determined, using the combustion method. In a few cases, oxygen and nitrogen contents were determined, using the vacuum fusion and Kjeldahl methods, respectively (see Table 1). X-ray diffraction patterns were taken on each of the samples chemically analyzed for hydrogen. This was done to determine semiquantitatively the phases present, and to serve as a check on the hydrogen analysis.

Metallographic samples were prepared from each of the tested tensile specimens. Longitudinal sections, which included one of the fracture surfaces in cross section, were mounted, polished, and etched according to the procedures described in the appendix. The remaining fracture surface of the tensile specimen was left intact, and was examined under a low-power microscope.

III. RESULTS

Typical stress-strain diagrams, obtained from room-temperature tensile tests on several different compositions of the fuel, are presented in Figure 6a. It will be observed that Zr - 10 U alloy hydrides do not deform plastically at room temperature. That is, they are completely brittle. This figure also shows the effect of carbon content on the ultimate tensile strength of this material. The ultimate tensile strength, which is indicated by the cross at the terminus of each curve, increases with carbon content from 5000 psi at 0.03 wt % to almost

Figure 6. Typical Stress-Strain Diagrams for Zr - 10 U Alloy Hydrides at: (a) Room Temperature, (b) 1250°F, (c) 1350°F, and (d) 1450°F

10,000 psi at 0.41 wt %, followed by a decrease to about 7000 psi at 0.47 wt %. This effect, plus the effect of hydrogen content on the mechanical properties of the fuel, will be discussed later.

Ultimate tensile strength and Young's modulus values, derived from these and other stress-strain diagrams obtained in room-temperature tests, are given in Table 2. The hydrogen and carbon content of each sample is also listed in this table. The x-ray diffraction results for these samples indicated that all but one were 100% ϵ (bct) phase.* The exception, a 1.45 H/Zr composition, was found to be mostly δ (fcc) phase.

Typical elevated-temperature stress-strain diagrams for Zr - 10 U alloy hydrides are presented in Figures 6b, c, and d. In these figures, the fuel exhibits some ductility at 1250°F and above. The proportional limit, ultimate tensile strength, and Young's modulus values, derived from these and other stress-strain diagrams obtained in this study, are presented in Table 3. In addition to mechanical property data, this table includes post-test analysis results for hydrogen content, carbon content, and x-ray diffraction. The post-test hydrogen analysis results represent data for samples which were isochore cooled after tensile testing. It is important to note that these results agree quite well with the hydrogen concentrations predicted from the hydrogen pressure and temperature conditions of the test.†

A comparison was made between room-temperature ultimate tensile strengths of Zr - 10 U alloy hydrides containing less than 0.05 wt % C and room-temperature transverse rupture strengths reported previously.^{1,8} This was done to deduce whether the load had been applied to the samples in a truly uniaxial manner, and a true ultimate tensile strength subsequently obtained. It has been shown experimentally that transverse rupture strength and ultimate tensile strength of brittle materials are equivalent when the load in the tensile test is applied without eccentricities.⁹ This is in direct contradiction to the Weibull volume theory, which predicts a higher transverse rupture strength from the fact that less volume is under tensile stress at failure in a transverse band sample than in a tensile sample. The room-temperature transverse rupture strength ranged from 4 to 11

*All hydride phase designations in this report are referred to the zirconium-hydrogen binary system according to Beck.⁶

†Attainment of the desired hydrogen contents during pretest equilibration had been predicted from results of calculations involving recent data on the diffusion of hydrogen in δ -phase Zr - 10 U alloy hydride.⁷

TABLE 2
ROOM-TEMPERATURE TENSILE TEST DATA FOR
SOME Zr - 10 U ALLOY HYDRIDES

Hydrogen Content (H/ Zr)	Carbon Content (wt %)	Ultimate Tensile Strength (psi)	Young's Modulus $E \times 10^{-6}$ (psi)
1.91	0.03	4,940	7.5
1.84	0.05	1,820	7.1
1.83	0.06	3,894	7.2
1.89	0.08	5,520	7.6
1.91	0.10	6,160	7.2
1.92	0.14	8,240	6.1
1.82	0.15	6,760	6.9
1.85	0.15	8,400	6.2
1.91	0.15	10,120	6.1
1.91	0.15	9,370	5.9
1.83	0.17	9,030	8.4
1.82	0.19	9,240	6.6
1.84	0.19	8,920	7.8
~ 1.72*	0.38	7,970	9.2
1.71	0.38	3,850	8.3
1.93	0.41	9,570	6.7
1.72	0.45	6,240	9.5
1.73	0.45	6,060	9.9
1.74	0.45	6,770	11.5
1.91	0.45	9,590	6.5
1.45	0.46	6,680	16.8
1.73	0.46	6,680	10.9
1.72	0.47	7,970	9.4
1.94	0.47	7,310	6.2

*Analysis based on x-ray diffraction data

TABLE 3

ELEVATED-TEMPERATURE TENSILE TEST DATA FOR
SOME Zr - 10 U ALLOY HYDRIDES

Test Conditions			Tensile Test Results			Post-Test Analysis		
Temperature (°F)	Hydrogen Pressure (psia)	Equivalent Hydrogen Content (H/ Zr)	Proportional Limit (psi)	Ultimate Tensile Strength (psi)	Young's Modulus $E \times 10^{-6}$ (psi)	Hydrogen Content (H/ Zr)	Carbon Content (wt %)	Hydride Phases Present* (x-ray diffraction analysis)
1250	5.1	1.74	8,400	10,530	3.9	1.71	0.13	ε
1250	5.1	1.74	7,500	9,630	5.5	1.73	0.15	ε
1250	5.1	1.74	7,200	9,440	4.0	1.73	0.13	ε
1250	38.2	1.85	5,130	6,850	2.3	1.81	0.05	ε
1250	38.2	1.85	-	7,050	4.9	1.82	0.05	ε
1350	5.9	1.67	-	6,806	10.0	1.67	0.15	80% δ - 20% ε
1350	5.9	1.67	7,600	10,140	8.9	1.67	0.15	70% δ - 30% ε
1350	5.9	1.67	-	7,750	8.5	1.68	0.16	60% δ - 40% ε
1350	5.9	1.67	6,800	9,620	5.9	1.68	0.14	50% δ - 50% ε
1350	5.9	1.67	7,750	10,470	4.3	1.69	0.16	20% δ - 80% ε
1350	17.6	1.75	6,530	8,490	3.8	1.76	0.16	ε
1350	16.2	1.74	6,460	10,080	3.9	1.77	0.15	ε
1350	16.2	1.74	6,380	7,640	4.2	1.72	0.12	ε
1350	45.6	1.80	5,800	8,490	3.5	1.83	0.15	ε
1350	45.6	1.80	4,600	7,490	2.3	1.85	0.15	ε
1350	45.6	1.80	5,280	7,680	2.9	1.83	0.11	ε
1450	45.0	1.74	-	4,520	4.9	-	0.04	-
1450	45.0	1.74	-	6,810	2.1	-	0.04	-
1450	45.0	1.74	-	3,930	2.0	-	0.03	-
1450	45.0	1.74	-	10,600	7.0	-	0.15	-
1450	45.0	1.74	4,900	9,250	7.1	-	0.15	-
1450	45.0	1.74	-	9,120	-	-	0.16	-
1450	45.0	1.74	5,400	7,720	6.7	-	0.17	-
1450	45.0	1.74	6,000	7,950	4.9	1.77	0.27	ε
1450	45.0	1.74	5,800	7,660	4.0	1.72	0.28	ε
1450	45.0	1.74	6,000	7,930	4.7	1.75	0.23	ε
1450	45.0	1.74	6,000	9,270	4.0	1.79	0.25	ε

*Relative to the zirconium-hydrogen system according to Beck.⁶ The quantitative data are based on an estimate of the relative peak heights for the (311) and (211) planes of the δ(fcc) and ε(bct) phases, respectively.

times higher than the room-temperature ultimate tensile strength. This difference indicates that the true ultimate tensile strength of the fuel is actually much greater than seen in this study. A comparison was not made between the elevated-temperature strengths, because earlier elevated-temperature transverse rupture data are reported to be in serious doubt.⁸

Fracture of the ϵ -phase hydrides was found to be transgranular, with a well defined cleavage plane. As can be seen by careful examination of the photomicrograph in Figure 7, the cracks generally lie at an acute angle to the twin bands. By relating the angle between the crack and the twin bands to the reported¹⁰ plane of the boundaries of the twin bands, it was determined that the cleavage plane of the ϵ (bct) hydride was an (100) type, the basal plane.

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Figure 7. Typical Macrocrack in an ϵ -Hydride Structure
(Figure Unclassified)

IV. DISCUSSION

Tensile tests on Zr - 10 U alloy hydrides at high hydrogen contents show quite clearly that the mechanical properties are significantly affected by temperature, hydrogen content, and carbon content. The effects of these three variables, plus the effect of microstructure on fracture behavior, are discussed in the following sections.

A. EFFECT OF TEMPERATURE

The effects of test temperature on ultimate tensile strength and Young's modulus are shown graphically in Figure 8. The large increase in strength of

Figure 8. Variation of (a) Ultimate Tensile Strength, and (b) Young's Modulus with Temperature for Some Zr - 10 U Alloy Hydrides

low-carbon material (nominal 0.05 wt % C) with temperature (See Figure 8a) is probably due to a reduction in sensitivity to stress concentration and eccentricities in loading at the higher temperature. Probably, for similar reasons, the strength of the high-hydrogen (≥ 1.80 H/Zr), medium-carbon (nominal 0.15 wt % C) material holds nearly constant with temperature.

The Young's modulus of the fuel is reduced by increasing temperature, as seen in Figure 8b. Unusual behavior is seen in the 1.74 H/Zr material between 1350 and 1450°F. The abrupt increase of the Young's modulus, from 4×10^6 to 6×10^6 psi, across this temperature range is suspected to have been caused by a phase change. Justification for this suspicion will be presented in the section which describes the effect of hydrogen content. The trend toward a minimum at 1350°F in the ultimate tensile strength for this same material (See Figure 8a) may also be a manifestation of a phase change.

As will be noted in Table 3, increasing the temperature of the 1.74 H/Zr, nominal 0.15 wt % C material from 1250 to 1450°F causes a decrease in the yield strength (proportional limit) from about 7700 to 5200 psi, which is normal behavior. The ductility of this material, as indicated by the total elongation at failure derived from the stress-strain diagrams in Figure 6, increases only up to 1350°F. At 1250°F, the strain at failure is about 0.3%; while, at 1350°F, it is increased to about 0.4%. At 1450°F, the ductility takes an unexpected tack, and the elongation at failure is reduced to about 0.2%. Also, the shape of the stress-strain diagram for this material at 1450°F is decidedly different from those at 1250 and 1350°F (compare Curve 1 in Figure 6d with Curve 2 in Figures 6b and c). This unusual behavior may be further indication of a phase change between 1350 and 1450°F.

B. EFFECT OF HYDROGEN CONTENT

The effects of hydrogen content on the ultimate tensile strength and Young's modulus of the SNAP fuel are illustrated graphically in Figure 9. In Figure 9a, the ultimate tensile strength will be seen to rise only slightly with increasing hydrogen content at room temperature. A rather different effect is seen at 1350°F, where the strength is seen to decrease above about 1.67 H/Zr.* As will

*The dotted portions of the curves in Figure 9 represent the best estimate of the variation of the mechanical properties of the fuel with hydrogen content, based on other data from this study and that reported elsewhere.^{11,12}

Figure 9. Variation of (a) Ultimate Tensile Strength, and (b) Young's Modulus with Hydrogen Content for Some Zr - 10 U Alloy Hydrides

be noted, there is a difference in the carbon content of the two hydrides, but this is not considered to be a major factor in the difference of behavior. A possible explanation for the different effect of hydrogen content in these two temperature regions is that the fuel appears to be less sensitive to eccentricities in loading, as the hydrogen content is increased in the ϵ phase. As the temperature is increased, this factor becomes less significant; consequently, the true variation of UTS with hydrogen content is probably more closely represented by the 1350°F curve in Figure 9a.

As will be seen in Figure 9b, hydrogen content has a significant effect on the Young's modulus of the Zr - 10 U alloy hydrides. The discontinuity in the Young's modulus in the 1.65 to 1.75 H/Zr region suggests that the phase relationships in the zirconium-hydrogen system is the most important factor. Direct

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Figure 10. Variation of Ultimate Tensile Strength of Some Zr - 10 U Alloy Hydrides With Carbon Content: (a) Room Temperature, 1.82 to 1.85 H/Zr, (b) Room Temperature, 1.89 to 1.94 H/Zr, (c) 1450°F, 1.74 H/Zr

evidence of this fact is seen in Table 3, in the 1350°F data on fuel equilibrated at 1.67 H/Zr. These samples had been isochore cooled to check the hydrogen content during the test. The x-ray diffraction data indicate that these samples contained varying amounts of the two hydride phases, δ (fcc) and ϵ (bct), and it will be seen that the Young's modulus decreased directly with the amount of ϵ phase present.

The relatively large drop in Young's modulus across the δ - ϵ transformation region is to be expected. The transformation from δ to ϵ is accompanied by severe twinning of the structure, and it is this phenomenon which affects the Young's modulus. Twinned structures generally exhibit anelastic behavior,¹³ and Chang¹⁰ has observed this type behavior in ϵ -phase zirconium hydride. The twin boundaries in the ϵ structure move under an applied stress, as a result of a slight shear displacement of the atoms. This results in a relatively large macroscopic deformation which is entirely recoverable (elastic). In the tensile tests, the strain rates were slow enough to allow full relaxation to occur; whereas, in the dynamic determinations of elastic modulus,² the "strain rates" were too rapid, at the frequencies of vibration used, to allow twin boundary motion to affect the elastic behavior. This explains the lack of sensitivity of the dynamic modulus to hydrogen content, as seen in Figure 9b.

The effects of hydrogen content on Young's modulus can be used to further support the argument that the anomalous behavior with temperature of the 1.74 H/Zr, nominal 0.15 wt % C material is due to a phase change. When the temperature of this material was increased from 1350 to 1450°F, its Young's modulus increased, as a result of the appearance of δ -phase hydride in the structure. The similarity in the shape of Curve 1 of both Figures 6c and 6d further supports this hypothesis. The curve in Figure 6c is representative of material containing some δ hydride, and its similarity to the curve in Figure 6d is indication that this material also contains δ phase.

As can be seen in Table 3 and Figure 6c, the yield strength is reduced slightly by increasing hydrogen content. The proportional limit reduces from about 7400 to 5300 psi at 1350°F when hydrogen content is increased from 1.67 to 1.80 H/Zr.

C. EFFECT OF CARBON CONTENT

Carbon content has a considerable influence on the ultimate tensile strength of the Zr - 10 U alloy hydrides, as shown in Figure 10. Here, it will be noted

a. Fracture Surface

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b. Longitudinal Section

Figure 11. Zr - 10 U Alloy Hydride Sample with 1.83 H/Zr and 0.06 wt% C, Tensile Tensile at Room Temperature

that the strength rises quite rapidly with carbon content, to about 0.15 wt %, and then tends to decrease. This appears to be the case for both room temperature and 1450°F.

The strengthening of the fuel with carbon content is, at least partially, due to grain refinement caused by the addition of zirconium carbide. The amount of grain refinement can be seen by comparing the photomicrographs in Figures 11, 12, and 13. A correlation was made between the ultimate tensile strength and the grain size for some room temperature samples with up to 0.2 wt % carbon, and it was found that the strength increased inversely with grain diameter. This amount of grain size effect is somewhat higher than predicted from the Griffith theory for brittle failure,¹⁴ which states that the fracture stress in tension is proportional to the inverse of the square root of the grain diameter.

Grain refinement cannot explain the decreasing strength at carbon contents above about 0.15 wt %. A possible explanation for this type of behavior can be based on the increase in size of the carbide particles with carbon content, as can be seen by comparing Figures 11b, 12b, and 13b. When a critical size is reached, the particles of zirconium carbide phase could act as zones or planes of weakness.

Another mechanism which could explain both the strengthening and weakening of the fuel is the dispersion strengthening mechanism. Strengthening by this mechanism is caused by a submicron, dispersed phase; but, if the size of the particles is increased beyond a certain point, or if they agglomerate, the matrix is weakened. There is some evidence, based on electron microscope studies, to show that a submicron dispersed phase, associated with the carbide phase, exists in the fuel¹⁵. At this point, there is not enough experimental data to indicate which of the two mechanisms most adequately explains the effect of carbon content on the ultimate tensile strength of the fuel. Further electron microscopic study of this material would be in order.

The decreasing influence of carbon upon strength at high carbon contents can be verified by examining the literature.^{1,8} The room-temperature transverse rupture strength reported for Zr - 10 U alloy hydride (1.6 H/Zr) containing 0.4 wt % C⁸ is only 100 psi higher than that reported for a similar fuel containing only residual amounts of carbon (about 0.02 wt %).¹ As will be noted in Figure 10b, this is the same sort of behavior seen in the tensile tests performed under the current program.

a. Fracture Surface

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b. Longitudinal Section

Figure 12. Zr - 10 U Alloy Hydride Sample with 1.83 H/Zr and 0.17 wt% C, Tensile Tested at Room Temperature

a. Fracture Surface

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b. Longitudinal Section

Figure 13. Zr - 10 U Alloy Sample with 1.72 H/Zr and 0.47 wt% C, Tensile Tested at Room Temperature

Some idea of the effect of carbon content on the yield behavior of the fuel at 1450°F can be obtained by examining Figure 6. Curves 2 and 3 in the Figure 6c clearly show that, as the carbon content is increased, the strain at failure is markedly increased. The samples containing 0.05 wt % C (Figure 6b) failed without any plastic deformation, while the 0.30 wt % samples (Figure 6d) plastically deformed about 0.5% (Curve 1 of Figure 6d is thought not to be comparable with the other two curves; as indicated previously, it appears to be representative of δ or $\delta + \epsilon$ hydride behavior, rather than pure ϵ). Sufficient data are not available from this study to define completely the effect of carbon content on the proportional limit.

Figure 14 shows the effect of carbon content on Young's modulus of the fuel at room ambient and elevated temperatures. As will be noted in Figure 14a, carbon content has a relatively insignificant effect at room temperature. This does not appear to be the case at 1450°F, as shown in Figure 14b. As this temperature, the modulus of the 1.74 H/Zr fuel is seen to rise to a maximum, slightly above 0.15 wt % C. The dotted curve* indicates that the maximum in Young's modulus is unique to the 1450°F temperature level. As mentioned previously in this report, the unusually high Young's modulus, measured for the 1.74 H/Zr, 0.15 wt % C fuel at 1450°F, is postulated to be caused by the appearance of δ -phase hydride in the matrix under this set of conditions. If this is indeed the case, it appears that carbon content affects the elastic behavior indirectly, through its effect on the zirconium-hydrogen phase diagram. Additional data are needed to verify this postulate, however.

D. EFFECT OF STRUCTURE ON FRACTURE

As previously mentioned, the fracture mode of the ϵ hydrides was found to be transgranular, along a well-defined cleavage plane. The appearance of the fracture surfaces of these materials reflect the mode of failure plus the grain size and orientation. Figures 11, 12, and 13 illustrate the relationship between the fracture surface and the grain size, and Figures 15, 16, and 17 illustrate the additional effect of grain orientation.

The fracture process in the elevated-temperature tensile samples can be deduced from Figures 15, 16, and 17. These samples were made up primarily of

*The data point in Figure 14b at 0.05 wt % C on the 1350°F curve is estimated from 1250°F on 1.85 H/Zr, listed in Table 3.

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Figure 14. Variation of Young's Modulus of Some Zr - 10 U Alloy Hydrides with Carbon Content:

(a) Room Temperature, 1.89 to 1.94 H/Zr,

(b) Elevated Temperature, 1.74 H/Zr

grains elongated in the radial direction, and the orientation of these grains was such that cleavage would proceed most easily in a radial or tangential direction. This can best be seen by comparing the orientation of the radial direction of the columnar grains relative to the twin bands of the ϵ phase, seen in Figure 18, with the orientation of the cracks relative to the twin bands, seen in Figure 7. Tensile failure of these samples was initiated at the surface, and the cracks propagated inward along the columnar grains, leaving a radially faceted fracture surface.

a. Fracture Surface

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b. Longitudinal Section

Figure 15. Elevated-Temperature Tensile Sample Containing 0.05 wt % C
(Photographed at 20X, Reduced to 13X)

NAA-SR-9875

a. Fracture Surface

7568-1555@

b. Longitudinal Section

Figure 16. Elevated-Temperature Tensile Sample Containing 0.15 wt% C
(Photographed at 20X, Reduced to 13X)
NAA-SR-9875

a. Fracture Surface

7568-1556@

b. Longitudinal Section

Figure 17. Elevated-Temperature Tensile Sample Containing 0.30 wt% C
(Photographed at 20X, Reduced to 13X)
NAA-SR-9875

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Figure 18. Typical Columnar Grain in ϵ -Phase Zr - 10 U Alloy Hydride (0.05 wt % C)(Figure Unclassified)

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Figure 19. Fracture Surface of Zr - 10 U Alloy Hydride Sample with 1.74 H/Zr and 0.03 wt % C, Ruptured at Room Temperature in Transverse Bend (Sample previously had been tensile tested at 1450°F) (Photographed at 20X, Reduced to 13X)

The fracture surfaces of these materials also reflect the stress pattern at failure. This is illustrated very graphically in Figure 19. This sample was failed in transverse bend at room temperature. The fiber stresses in the side of the piece opposite the center load (indicated in the figure) were tensile. The piece failed in tension progressively across its cross section; and, due to the highly orientated grain structure, a "V" pattern was left on the fracture surface in the region which failed last. Cleavage steps, which are commonly observed on fracture surfaces of brittle materials, can also be seen in Figure 19. Eccentricities in loading in the tensile tests are also reflected in the appearance of the fracture surface. A good example of this point is seen in Figure 13a, where striations are observed across the fracture surface. This indicates that failure was initiated on one side of the sample and progressed across it, as a result of moments imposed by eccentricities in loading.

V. CONCLUSIONS AND RECOMMENDATIONS

This study has shown that variations in temperature, hydrogen content, and carbon content significantly affect the short-term tensile mechanical behavior of the Zr - 10 U alloy hydrides. Generally, temperature had a normal effect on the mechanical behavior of this brittle material. Increasing the temperature caused a slight increase in the ultimate tensile strength, probably as a result of reduction in sensitivity to eccentricities in loading. Also, increasing the temperature caused a near-linear reduction in the Young's modulus. A discontinuity was observed in the temperature-Young's modulus relationship, which was postulated to be due to a phase transformation. Hydrogen content had its greatest effect on the Young's modulus of this material. The most significant change in this property came from the δ - ϵ hydride phase transformation. Carbon content had its greatest effect on the ultimate tensile strength of the Zr - 10 U alloy hydrides, over the entire temperature and hydrogen content range studied. The addition of zirconium carbide to this material caused a maximum in the ultimate tensile strength at the 0.15 wt % C level. Carbon content did not significantly affect the Young's modulus, except in the case where it appeared to alter the zirconium-hydrogen phase diagram.

The transgranular cleavage of the ϵ hydride caused the grain structure to significantly affect fracture of a polycrystalline body of this material. Stress patterns which caused failure in a member could easily be deduced from examination of the fracture surfaces, making failure analysis in this material relatively simple.

More data are needed on the mechanical properties of the fuel as a function of other combinations of the variables examined in this study. Due to the limitations of the test method, in providing uniaxial loading to the specimen, it is recommended that further tensile tests on the fuel be performed in a device which would eliminate this problem, such as a self-aligning load system utilizing gas bearings.

The results of this study suggest the need for basic investigations of the mechanical behavior of the SNAP fuels. Mechanical studies on single crystals of this material could prove to be quite instructive. Also, this study points out a need for better definition of the phase relationships of the Zr - 10 U alloy hydrides at elevated temperatures. Further, the effect of small additions of carbon (ZrC) on the phase relationships should be established.

REFERENCES

1. J. W. Raymond and S. M. Toy, "Mechanical Strength of Hydrides of Zirconium and Zirconium-Base Alloys (Title Unclassified)," NAA-SR-6732 (AEC classified report, April 15, 1962)
2. J. D. Watrous, "Dynamic Modulus of Elasticity for Zirconium Alloys and Zirconium Alloy Hydrides," NAA-SR-TDR-5755 (October 5, 1960)
3. J. W. Raymond, "Development of a High Yield Zr - 10 U Alloy Massive Hydride (Title Unclassified)," NAA-SR-7305 (AEC classified report, January 1, 1963)
4. D. C. Campbell et al. "SNAP 8 Experimental Reactor Fabrication and Engineering (Title Unclassified)," NAA-SR-8430 (AEC classified report, March 1, 1964)
5. H. E. Johnson, "Hydrogen Dissociation Pressures of Modified SNAP Fuel (Title Unclassified)," NAA-SR-9295 (AEC classified report, March 15, 1964)
6. R. L. Beck, "Zirconium-Hydrogen Phase System," Trans. ASM 55, 3 (September 1962) pp 542-555
7. S. D. Harkness, unpublished data, Atomics International, Canoga Park, California (October 1963)
8. L. B. Lundberg, "Transverse Rupture Tests on Modified SNAP Fuels (Title Unclassified)," NAA-SR-MEMO-9410 (AEC classified report, January 10, 1964)
9. Southern Research Institute, "The True Tensile Stress-Strain Properties of Brittle Materials to Very High Temperatures," TID-17887 (October 27, 1962)
10. R. Chang, "Phase Transformation, Twinning, and Anelastic Behavior Associated with Zirconium Hydride," NAA-SR-5537 (September 15, 1960)
11. L. B. Lundberg, "Tensile Properties of SNAP 4 Fuel Material at Elevated Temperatures (Title Unclassified)," NAA-SR-MEMO-6957 (AEC classified report, January 3, 1962)
12. D. S. Parker and C. L. Huffine, "Properties of Hydrided Zirconium," APEX-561 (December 1959)
13. C. Zener, Elasticity and Anelasticity of Metals (University of Chicago Press, Chicago, Ill., 1948)
14. George E. Dieter, Jr., Mechanical Metallurgy (McGraw-Hill Book Co., Inc., New York, 1961) p 204

REFERENCES

15. "SNAP 8 Quarterly Progress Report, October 1963-January 1964 (Title Unclassified)," NAA-SR-9392 (AEC classified report, March 16, 1964)
16. D. T. Shoop, Unpublished data, Atomics International, Canoga Park, Calif. (July 1963)

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APPENDIX

METALLOGRAPHIC PREPARATION METHODS

Two different metallographic preparation procedures were used on the tensile samples, each giving slightly different results. The method applied to the elevated-temperature samples seemed to provide the best ϵ -phase hydride structure, particularly in the high-carbon fuels, but the carbides were attacked slightly. Almost the reverse resulted from the method applied to the room-temperature samples, where the carbides stood out clearly and the ϵ bands were hardly visible in the high-carbon samples. Either method was entirely suitable for the low-carbon fuel. The details of these two methods are described in the following sections.

A. PROCEDURES FOR ELEVATED-TEMPERATURE TENSILE SAMPLES

A short piece of material, taken from the test section and including a fracture surface of the tensile tested sample, was mounted in Bakelite and sectioned longitudinally with a silicon carbide wheel, using water as the coolant. The rough face was ground flat on a horizontal grinding wheel, and then the surface was polished with the aid of a Buelher Auto-Met automatic polisher on 180, 240, 400, and 600 grit silicon carbide papers, using kerosene as the coolant. Next, the samples were lapped with an Auto-Met on micro cloth charged with a slurry of diamond paste in an oil-like lapping compound. Three different diamond particle sizes were used (14, 9, and 6 μ).

The polishing and lapping procedures tend to smear the surface of the zirconium-uranium hydrides, to the extent that a true structure cannot be obtained, using normal etching procedures. Because of this, it was necessary to employ a chemical polish technique to prepare the samples for etching. This involved the following procedure. The sample was hand polished on a lapping wheel charged with a slurry of Linde "B" in a 10% oxalic acid-water solution. During the polishing, one or two drops of the chemical polish solution (30 parts conc. HNO_3 , 30 parts conc. H_2O_2 , and 1 part conc. HF) were placed on the wheel. Then the lapping was continued from 1 to 2 min, after which the sample was thoroughly rinsed with distilled water. The chemical polishing time was found to be quite sensitive to carbon content, and shorter times were required to prepare the higher-carbon content materials for etching. Excessive chemical polishing would tend to remove the precipitate particles from the matrix.

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The samples were etched by immersion for about 20 sec in a solution containing 1 part lactic acid and 1 part conc. HNO_3 , with conc. HF added drop by drop until the sample started to bubble. To allow easier examination of the sample under the microscope, using white light and bright field, the samples were stained with an electrolytic etch. The staining process involved placing the sample in a stainless steel cup (cathode) containing 10% oxalic acid and applying a 12-v dc charge to the sample surface with a copper probe (anode). Two quick bursts of current were sufficient to highlight the etched structure. The current density used ranged from 5 to 10 ma/in.² An example of the structure obtained by this technique is shown in Figure 7.

B. PROCEDURES FOR ROOM-TEMPERATURE TENSILE SAMPLES

These samples were prepared exactly as the elevated-temperature samples, up through the 600-mesh SiC papers. This was followed by lapping with 30- μ diamond paste on a Metcloth covered wheel. The samples were then taken to a fairly dry micro cloth charged with Linde "B" and a few drops of 10% oxalic acid solution. At this point, lapping was continued until a brilliant finish was obtained on the sample. The lapping wheel was then wetted slightly with water and a water solution containing approximately 10% HNO_3 and 4 drops of HF. This chemical polish was used to remove the disturbed metal from the sample surface. Here, the reaction rate (which increased with increasing carbon content) was adjusted, not by varying the time of polishing, but by adjusting the concentration of the attacking solution. This procedure was alternated with a 10% oxalic acid electrolytic etch until the desired structure was obtained. Examples of the microstructures obtained by this method are given in Figures 11 thru 13.