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ROOM-TEMPERATURE COMPRESSIVE PROPERTIES
OF
Zr - 10 U ALLOY HYDRIDE
AT
HIGH HYDROGEN CONTENTS
(Title Unclassified)

AEC Research and Development Report

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ROOM-TEMPERATURE COMPRESSIVE PROPERTIES
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By

L. B. LUNDBERG
J. T. BERLING

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ABSTRACT

Room-temperature, short-term compressive tests and direct determinations of Poisson's ratio were performed on Zr - 10 U alloy hydrides. The phenomenon of hydrogen loss on compression was also observed, and studied in some detail.

Room-temperature compression tests on the fuel, prepared by both standard production and precipitation hydriding techniques, have indicated that carbon content has a significant effect on the ultimate strength and ductility. The Zr - 10 U alloy hydrides with 0.02 wt % C exhibited 35,000 psi ultimate compressive strength and 0.023 in./in. total strain at failure. When the carbon content was increased to 0.29 wt %, the ultimate compressive strength increased to 125,800 psi and the total strain at failure increased to 0.172 in./in. In some cases, the hydrogen content was varied from 0.50 to 1.90 H/Zr. The 0.5 H/Zr material exhibited properties superior to the higher H/Zr fuel alloy compositions. Material near the δ -phase region (1.50 H/Zr) exhibited an increase in Young's modulus and yield strength, by a factor of 2 to 3, and the total strain to failure was a factor of 8 lower than ϵ fuel material. Microdefects in the fuel, possibly resulting from the hydriding process, were seen to have an effect on the Young's modulus and the directly measured Poisson's ratio. Microcracks found in the precipitation-hydrided material caused higher moduli and Poisson's ratios. Microcracks created during plastic deformation also caused an increase in the Young's modulus of the fuel. This phenomenon was attributed to twin-boundary pinning.

An unexpected discovery of this study was the release of hydrogen, as a result of room-temperature compressive testing. The amount of hydrogen released seems to be a function of strain rate, hydride phase, and grain structure. The highest release observed in this preliminary study was the order of 0.02 to 0.03 wt % hydrogen. The mechanism of hydrogen release in the elastic region

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for ϵ -hydride fuel is postulated as follows: since ϵ -hydride material strains by twin interface motion under applied stress, and since this interface is composed of edge and screw type dislocations, it is conceivable that uncombined hydrogen in these twinning dislocations will emerge from dislocations which cut the surface of the sample. The mechanism for hydrogen release in the inelastic region is postulated to be a short-circuiting mechanism, in which hydrogen gas is dragged along with dislocations and released at numerous micro- and macrocracks formed during inelastic straining.

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I. INTRODUCTION

Zirconium - 10 wt % uranium alloy hydride, the basic fuel material for the SNAP reactors under development at Atomics International, is being studied to determine its mechanical stability under service conditions. During launch, startup, and re-entry from earth orbit, the short-term mechanical properties of this material are important factors to be considered. Since the hydride is quite brittle, the tensile strength is of primary interest, with respect to mechanical failure. Tensile properties of both carbon-modified and nonmodified Zr - 10 U alloy hydride in the high hydrogen content regions have been determined for a fairly wide range of temperatures.⁽¹⁾

To enable the designer to achieve greater reliability in predicting mechanical stability, the compressive properties of the material were also required. Some data on the compressive behavior of Zr - 10 U alloy hydride at room temperature are available in the literature.⁽²⁾ The study described in this report represents an expansion of the previous work, with more detailed studies on the effects of hydrogen content and a previously unstudied variable, carbon content. Specifically, this report covers the determinations of the room-temperature compressive properties of Zr - 10 U alloy hydride from 0.5 to 1.91 H/Zr* and carbon content from 0.02 to 0.29 wt %.

The effects of two different hydriding methods on the compressive properties were also examined. Compressive strength, total deformation at failure, and Young's modulus were measured; and, in addition, the Poisson's ratio of a few of the samples was determined, using a direct measurement method.

During the course of the study, several rather interesting phenomena were observed. Among the most unusual were:

- 1) The release of hydrogen from the fuel upon compression
- 2) An increase in the Young's modulus of the fuel due to inelastic deformation.

The loss-of-hydrogen phenomenon was verified; and, during the course of studies to determine the mechanism of loss, the change of modulus phenomenon was observed. Basic causes for both these phenomena have been postulated, and will be discussed.

*In this report, H/Zr is defined as the hydrogen-to-zirconium atomic ratio which assumes all the carbon present is combined with zirconium in the form ZrC, and therefore does not occlude any hydrogen.

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Figure 1. Zirconium-Hydrogen Phase Diagram
(Figure Unclassified)

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II. MATERIALS

All the samples used in this study were prepared from zirconium - 10 wt % uranium alloy hydride. The initial zirconium-uranium alloy ingots were prepared at Atomics International, using consumable electrode arc-melting methods. The ingots were reduced to cylindrical shaped rods by a warm extrusion process. Carbon content of the alloy was adjusted by additions of zirconium carbide powder (-325 mesh) to the raw materials, prior to melting. The preparation procedures to this point were identical for all samples, and are described in detail elsewhere.⁽³⁾

Two different procedures were followed in hydriding the fuel material used in the compression test samples. A majority of the samples were taken from material hydrided by standard production techniques.⁽³⁾ The remainder of the samples were taken from pilot-run fuel rods, hydrided by a thermal precipitation technique.⁽⁴⁾ The main difference in these two hydriding techniques lies in the procedure followed in forming the δ -phase hydride (see Figure 1).^{*} In the former technique, the δ structure is formed isothermally, by providing sufficient hydrogen to the surface of the fuel rod. This results in a high degree of preferred orientation in low-carbon material, due to initiation of crystal growth at the rod surface and subsequent growth inward. The resultant columnar structure of the fuel rods prepared by this method is very pronounced in low-carbon material, as shown in Figure 2a. This structure can be avoided by increasing the carbon content. The carbide precipitates form nucleation sites for δ hydride, and reduce the columnar structure, as illustrated in Figures 3a and 4a. In the precipitation technique, the δ structure is precipitated uniformly throughout the rod at constant composition by cooling through the β - δ boundary.[†] This results in an equiaxed grain structure, as will be noted in Figure 5a. Microcracks, such as seen in Figure 5b, were commonly found in the precipitation hydrided materials. This is in contrast to the samples prepared by carbide nucleation methods. These samples were essentially free from microdefects (see Figures 2b, 3b, and 4b).

^{*}In this report, all hydride phases are as referred to in the zirconium-hydrogen phase diagram according to Beck.⁽⁵⁾

[†]This hydriding method will be referred to, in this report, as the "precipitation" method.

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a. Pretest Macrostructure

4391-4-2

b. Pretest Microstructure

HF Chemical Polish
+10% Oxalic Acid
Electrolytic Etch

4391-5-3

Figure 2. Production Hydrided Sample Containing Nominal 0.05 wt % C
(H/Zr $\sim$ 1.80)
(Figures Unclassified)

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a. Pretest Macrostructure

4391-3-2-1

b. Pretest Microstructure

HF Chemical Polish
+10% Oxalic Acid
Electrolytic Etch

4391-3-2

Figure 3. Production Hydrided Sample Containing Nominal 0.15 wt % C
(H/Zr $\sim$ 1.83)
(Figures Unclassified)

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a. Pretest Macrostructure

4391-8-2

b. Pretest Microstructure

HF Chemical Polish
+10% Oxalic Acid
Electrolytic Etch

4391-9-3

Figure 4. Production Hydrided Sample Containing Nominal 0.30 wt % C
(H/Zr ~ 1.85)
(Figures Unclassified)

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a. Pretest Macrostructure

4571-3-3

b. Pretest Microstructure

HF Chemical Polish
+10% Oxalic Acid
Electrolytic Etch

4571-3-2

Figure 5. Precipitation Hydrided Sample Containing Nominal 0.02 wt % C
(H/Zr ~1.82)
(Figures Unclassified)

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The hydrogen and carbon contents of each sample were determined. Vacuum extraction and weight gain methods were used for hydrogen content determinations, while carbon content was determined by the combustion method. Several samples were analyzed for impurities, and a typical analysis is presented in Table 1.

TABLE 1
IMPURITY ANALYSIS FOR Zr - 10 U ALLOY
HYDRIDE COMPRESSION SPECIMENS

Element	ppm	Element	ppm	Element	ppm
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		

The compression specimens were machined from hydrided rods on a lathe. The specimens were right circular cylinders, with a length-to-diameter ratio of 2, measuring 1 in. long by 1/2 in. in diameter. One sample, used for the Poisson's ratio determination, was 2 in. long by 1 in. in diameter. To minimize eccentricities in loading, the ends of each sample were held parallel and perpendicular to the centerline, within ± 0.0002 in./in.

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III. COMPRESSION TESTS

A. EXPERIMENTAL PROCEDURES

Compression tests were performed on samples of Zr - 10 U alloy hydride in the apparatus shown in Figure 6. The basic test unit was a Baldwin-Lima-Hamilton Universal Testing Machine with a 60,000-lb load capacity. The sample was placed between two blocks of Inconel "X" in a subpress assembly shown in Figure 6b. The Inconel "X" blocks were used to prevent localized plastic deformation of the steel bearing faces of the subpress. A deflectometer, which gauged the change in the distance between the upper face of the subpress and the support table, was used to measure strain in the sample during the test. Since the measurements recorded by the deflectometer included the deformation of the Inconel "X" bearing plates and the subpress plus the deformation of the sample, it was necessary to determine a correction factor which would eliminate the deformation of everything but the sample. This factor was determined by measuring the deformation in a few samples with an optical system and noting the deviation at several stresses from the readings recorded by the deflectometer.

The samples were compression tested to failure at a constant strain rate of 0.005 in./in./min. Strain rate was maintained constant with the aid of a strain pacer shown in Figure 6a. During the entire test, the strain and load were plotted simultaneously on an "XY" recorder mounted on the testing machine. Sample failure occurred with such violence that it was necessary to contain the sample fragments with an explosion shield (see Figure 6a).

B. RESULTS AND DISCUSSION

The main purpose of these tests was to determine the compressive stress-strain behavior of the Zr - 10 U alloy hydrides at room temperature. Typical compressive stress-strain diagrams from this study are presented in Figure 7, and the data derived from these and other curves are summarized in Table 2.

The stress-strain diagrams in Figure 7 show the general effects of carbon content and hydrogen content on the strength and ductility (total deformation at failure) of the Zr - 10 U alloy hydrides. As will be noted, the yield strength increased slightly with carbon content, while the ultimate strength and ductility

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a. Overall View
(Figure Unclassified)

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b. Closeup of Sample in Subpress

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Figure 6. Compression Test Equipment

TABLE 2
AVERAGE ROOM-TEMPERATURE COMPRESSION TEST DATA FOR
Zr - 10 U ALLOY HYDRIDES

Carbon Content (wt %)	Hydrogen Content (H/Zr)*	Hydriding Method	Proportional Limit (psi)§	0.2% Offset Yield Strength (psi)§	Ultimate Compressive Strength (psi)§	Young's Modulus ($\epsilon \times 10^6$ psi)§	Total Strain at Failure (in. /in.)**
0.02	1.79	Precipitation	24,400	27,000	35,000	7.40	0.023
0.02	1.82	Precipitation	27,300 ± 300	28,100 ± 400	35,700 ± 500	8.22 ± 1.96	0.023
0.02	1.88	Precipitation	26,400 ± 100	28,000 ± 400	37,000 ± 3,000	6.98 ± 0.42	0.023
0.04	1.72	Production	29,500 ± 1,000	31,400 ± 200	68,500 ± 2,600	6.67 ± 0.11	0.065
0.04	1.82	Production	27,100 ± 1,600	30,900 ± 300	64,900 ± 1,800	6.26 ± 0.23	0.059
0.05	1.80	Production	29,200 ± 500	33,900 ± 100	72,900 ± 600	5.09 ± 0.01	0.094
0.11	1.68	Production	36,200 ± 600	41,400 ± 700	104,500 ± 3,000	6.26 ± 0.36	0.121
0.09-0.15	1.72-1.75†	Production	32,400 ± 900	38,300 ± 400	109,000 ± 2,200	7.00 ± 0.65	0.148
0.13	1.83	Production	28,500 ± 800	37,300 ± 2,700	112,500 ± 3,500	7.20 ± 0.94	0.152
0.21	1.81	Production	37,900 ± 2,900	41,100 ± 800	120,900 ± 3,000	5.67 ± 0.87	0.194
0.26	1.68	Production	40,700	47,600 ± 800	109,700 ± 600	7.09 ± 0.31	0.132
0.29	1.85	Production	37,300 ± 1,900	43,400 ± 400	125,800 ± 500	7.00 ± 0.72	0.172
0.15	1.50	Production	81,632	97,810	98,000	13.06	0.016
0.15	0.50	Production	97,810	131,386	204,000††	14.60	0.190††

*Determined by vacuum extraction analysis except where indicated

†Determined by weight gain analysis

§All (±) values represent the standard deviation of the mean, based on the standard deviation of an individual observation

** $\frac{\Delta L}{L}$

where L = original length (in.)

ΔL = reduction in length by deflectometer (in.)

††Sample taken to 19% reduction in length

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Figure 7. Room-Temperature Compressive Stress-Strain Curves

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Figure 8. Effect of Carbon Content on Ultimate Compressive Strength

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increased substantially in materials where the hydrogen concentration was basically in the ϵ -hydride phase region. These effects are further illustrated in Table 2, where it will be noted that the 0.2% yield strength is seen to increase from 27,000 to 43,400 psi when carbon content is increased from 0.02 to 0.29 wt %. For the same increase in carbon content, the ultimate compressive strength increases from 35,000 to 125,000 psi, as shown in Figure 8, and the deformation at failure increases from 0.023 to 0.172 in./in. The strength values for the low-carbon materials compare closely with those previously reported.⁽²⁾ The data presented in Table 2 indicate that the Young's modulus of the fuel is not significantly affected by carbon content. The degree of fragmenting of the samples at failure was strongly affected by carbon content, as illustrated by a comparison of Figures 9a and 9b. It will be noted that the material which broke from the regions (exclusive of the cone-shaped end fragments) of the high-carbon sample consisted of a relatively fine aggregate.

It should be noted that the average room-temperature compressive Young's modulus of Zr - 10 U alloy hydride is about 15% lower than the average tensile values reported previously⁽¹⁾ for comparable hydrogen contents. Similar differences in tensile and compressive moduli have been observed for other brittle materials, such as graphite.⁽⁶⁾ As yet, no explanation for the difference has been offered.

It is of interest to examine the effects of hydrogen content on the mechanical behavior of the fuel. As shown in Figure 10, varying the hydrogen content within the ϵ region did not significantly affect the room-temperature compressive properties of the fuel. However, a change in hydrogen content, from ϵ (>1.67 H/Zr) and $\epsilon + \delta$ (>1.60 H/Zr) to $\alpha + \delta$ (1.50 H/Zr), results in an increase in the Young's modulus (by a factor of 2 to 3) and a decrease in the rupture strain (by a factor of 8), for equivalent carbon-modified material. This effect has previously been observed in tension.⁽¹⁾ The strength properties obtained for the 0.5 H/Zr composition, however, are superior to those obtained for both the δ and ϵ phase compositions. Although the correlations indicated by the broken lines in Figure 10 are not exact, the general effect of large variations in hydrogen content is shown.

The method of hydriding could be directly related to variations in the mechanical properties of the fuel. Young's modulus appears to be affected, but this

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a. Unmodified Sample
(≤ 0.02 wt % C, H/Zr ~ 1.80)

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b. Carbon-Modified Sample
(≥ 0.09 wt % C, H/Zr ~ 1.80)

Figure 9. Appearance of Samples, Before and After
Room-Temperature Compression Testing

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a. Young's Modulus

b. 0.2% Offset Compressive Yield Strength

c. Fracture Strain

Figure 10. Room-Temperature Compression Properties

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effect can be explained on the basis of microdefects. As will be noted in Table 2, the Young's moduli of the precipitation hydrided fuel samples containing 0.02 wt % C tended to be higher than production hydrided samples with comparable hydrogen and carbon contents. As will be recalled from Figure 5b, 0.02 wt % C precipitation hydrided samples contained many microcracks. Since the ϵ hydrides deform in the elastic region, primarily by twin boundary motion,⁽¹⁾ microcracks would form discontinuities at twin interfaces, and thereby impede the movement of these interfaces. This resulted in an increase in the Young's modulus. No significant difference was observed in the proportional limit and the 0.2% offset yield strength values obtained for comparable precipitation and production hydrided fuel. However, the values for ultimate compressive strength and total strain to failure, obtained for the precipitation hydrided material, were ~50% lower than those exhibited by the production hydrided material. This difference is also attributed to the presence of microcracks in the fuel that presumably occur during precipitation hydriding.

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IV. DETERMINATION OF POISSON'S RATIO

A. EXPERIMENTAL PROCEDURES

Several of the samples used in the compression tests were also utilized in the direct determination of the Poisson's ratio in compression. As previously mentioned, one of the samples was twice as large as the standard compression specimen.

The test apparatus for this study included a floor model Instron testing machine and the same longitudinal strain-measuring system that was used in the compression tests. The large sample was tested on the Baldwin machine, using opposing dial indicators to measure the lateral strain at various loads below the elastic limit. These readings were compared with the corrected longitudinal strain, and Poisson's ratio was calculated in the usual manner. A similar procedure was followed for the smaller samples. In the case of the smaller samples, the lateral strain was measured with the device shown in Figure 11. This device consists of a hardened steel strip, fitted with balancing foil strain gages, situated so as to measure the change in the opening between the knife edges of the opposing ends of the strip. This strain-measuring device had been previously calibrated with an extensometer calibration fixture. Read-out was accomplished with a Baldwin SR-4 Strain Gage Indicator, Type "N."

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Figure 11. Lateral Strain-Measuring Device
(Figure Unclassified)

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B. RESULTS AND DISCUSSION

The results of the direct compressive Poisson's ratio determinations for the Zr - 10 U alloy hydride are summarized in Table 3. Shear moduli were calculated from the Poisson's ratio and the Young's modulus, using the expression for isotropic solids:

$$G = \frac{E}{2(1 + \nu)}$$

where:

G = Shear modulus (psi)

E = Young's modulus (psi)

ν = Poisson's ratio.

These data are also included in Table 3.

TABLE 3
COMPRESSIVE ELASTIC PROPERTIES OF Zr - 10 U ALLOY HYDRIDES

Sample Number	Carbon Content (wt %)	Hydrogen Content (H/Zr) _{eff}	Hydriding Method	Poisson's Ratio	Young's Modulus ($\epsilon \times 10^6$ psi)	Calculated Shear Modulus ($G \times 10^6$ psi)
1	0.02	1.79	Precipitation	0.50	5.43	1.81
2	0.02	1.82	Precipitation	0.55	6.26	2.02
3	0.04*	1.76	Precipitation	0.52	8.80	2.89
4	0.05	1.82	Production	0.45	6.26	2.15
5	0.11	1.68	Production	0.40	6.26	2.23
6	0.29	1.85	Production	0.38	7.00	2.53
7	0.15	1.50	Production	0.27	13.6	5.35

*Large sample

The Poisson's ratio values for Samples 2 and 3, listed in Table 3, are higher than is theoretically possible (rubber, which maintains constant volume during uniaxial straining, has a Poisson's ratio of 0.50). These two values are thought to be excessive because of "barreling" of the sample. It is suspected that a Poisson's ratio of about 0.5 is characteristic for the equiaxed structure, precipitation hydrided fuel.

Apparently, variations in Young's modulus have no significant effect on Poisson's ratio, since the Young's modulus for Sample 3 (Table 3) was substantially higher than for the rest of the precipitation hydrided samples. This particular sample contained many microcracks (see Figure 12), which explains the high modulus.

HF Chemical Polish
+10% Oxalic Acid
Electrolytic Etch

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Figure 12. Precipitation Hydrided Sample
(Large Sample), Prior to Determination
of Poisson's Ratio in Compression

The production hydrided samples tended to have a lower Poisson's ratio than those prepared by the precipitation hydriding method. This might be caused by the difference in preferred orientation observed between these two groups of samples and/or the large difference in population density of microdefects. Within the production hydrided samples, it appears that Poisson's ratio varies inversely with carbon content, but the data are too meager to provide much confidence in this conclusion. No significant change in Poisson's ratio was observed by varying the hydrogen content in the ϵ region. Changing the hydrogen content, however, to the $\alpha + \delta$ phase region had a significant effect on Poisson's ratio and, consequently, the calculated shear modulus. This is understandable,

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since the composition studied (1.50 H/Zr) consisted primarily of δ -phase hydride that does not possess the severely twinned and easily moved twin boundaries of ϵ hydride.

Far more data are needed to establish reliable Poisson's ratio values for the Zr - 10 U alloy hydride in the high-hydrogen regions, but the data recorded here point up the need for considering the effects of hydrogen content, carbon content, and microstructure. To obtain better values for the Poisson's ratio of this material in compression, it might be well to consider more precise measuring techniques and samples with length-to-diameter ratios sufficient to eliminate errors due to barreling. Values should also be obtained in tension, to check the possibility of a difference due to the mode of stress, such as that seen for Young's modulus.

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V. HYDROGEN LOSS PHENOMENON

As previously noted, the hydrogen content of the fuel was slightly reduced by room-temperature compression testing. This phenomenon was originally observed by chemically analyzing samples for hydrogen before testing (using vacuum extraction techniques) and comparing the results with those obtained by the same method from fragments of the actual sample remaining after the test. Results of this comparison indicated a 10% reduction in H/Zr for samples with an initial H/Zr of 1.85. Fuel material with an original hydrogen content below 1.85 H/Zr exhibited less reduction in H/Zr upon fracture. Essentially no loss was observed for samples with an original H/Zr of 1.69. To support these findings, x-ray diffraction analyses were taken of samples in the as-prepared and as-tested conditions. Data from these analyses did not verify the results obtained from chemical analysis. These conflicting results indicated a better method of verifying the hydrogen loss phenomenon was necessary.

Therefore, a more refined experiment was performed, to determine whether hydrogen is actually evolved from the fuel on compression testing at room temperature, and at what stage of testing it occurs. In this experiment, a sample (1 in. long by 1/2 in. in diameter) was compressed to successively higher stresses, removed from the test fixture, and weighed on an analytical balance. A sample was chosen which was large enough, and had an original hydrogen content high enough (1.80 H/Zr), to allow easy detection of hydrogen losses of the magnitude indicated by chemical analysis in samples previously taken to failure. The stress cycles imposed on the sample are shown graphically in Figure 13. The arrowheads which point upward on the stress-strain diagram denote the upper limits of the stress cycle. As will be noted, the sample was stressed first to slightly below the proportional limit, then to the 0.4% offset yield strength, then to a stress roughly equivalent to two-thirds of the ultimate, and finally to rupture. No measurable hydrogen loss was observed after any of the stress cycles (the accuracy of the balance and the size of the sample allowed detection of losses in weight of the order of 1 part in 250,000). Fragments of the ruptured sample were then chemically analyzed. Comparison of the before-test and after-test analyses indicated a slight reduction in H/Zr due to compression testing. Obviously, these data do not verify the 10% reduction in H/Zr

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Figure 13. Compressive Stress-Strain for
Sample (0.04 wt % C, H/Zr = 1.82),
Hydrogen Evolution Determination

observed previously. Consequently, these variations indicated chemical analysis techniques or the method of obtaining sample material for analysis was a limiting factor for accurate hydrogen loss determinations.

To eliminate these effects, it was necessary to measure the hydrogen at the instant of release during compression. This was accomplished by enclosing the test sample with a fixture which allowed a dynamic vacuum to be maintained on the test sample during normal room-temperature compression testing. A photograph of the assembled test fixture and a longitudinal cross-sectional sketch is presented in Figure 14. A hydrogen mass spectrometer, peaked to detect only hydrogen, was attached to this fixture, thereby allowing the hydrogen released during the test to be monitored continuously.

At this point, a more detailed study was initiated, in order to better reveal any hydrogen loss phenomenon. To determine the effect of grain size and grain orientation on the hydrogen release phenomenon, the carbon content of the

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a. Photograph

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b. Schematic

Figure 14. Test Fixture for Hydrogen Loss Determination During Room-Temperature Compression Testing (Figures Unclassified)

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high-H/Zr (1.91) material was varied from 0.05 to 0.30 wt % C. Typical room-temperature compression stress-strain plots, with associated hydrogen release plots at a constant strain rate of 0.010 in./min, for various carbon compositions in the ϵ -hydride region, are presented in Figures 15a, 15b, and 15c. From these plots, it is evident that three distinct regions of hydrogen release actually do exist. These are the elastic region, inelastic region, and postrupture region. These figures also show that the hydrogen release rate per unit strain is higher for the highly oriented, columnar grain material represented in Figure 15a (equivalent macrostructure, Figure 2a), as compared to the more equiaxed grain material represented in Figures 15b and 15c (equivalent macrostructures, Figures 3a and 4a). The same effect is also evident in a comparison of 0.15 wt % C material, Figure 15b, with the finer more equiaxed 0.30 wt % C material in Figure 15c, but the difference is not as significant. From these observations, it can generally be concluded that the effect of an increase in grain size and preferred orientation in these materials is to increase the hydrogen release rate per unit strain during room-temperature compression testing. The total amount of hydrogen released from these materials, as a result of room-temperature compression testing, ranged from 0.02 to 0.03 wt %. Also, a typical hydrogen release rate of 0.07 cc(STP)/hr was exhibited.

In an attempt to understand the mechanism of hydrogen release during room-temperature compression testing, a correlation of this effect with observations made by other investigators on similar material was considered. Chang⁽⁷⁾ has suggested that zirconium hydride in the ϵ phase strains under an applied stress by twin interface motion. Chang⁽⁸⁾ also suggests this interface must be composed of arrays of edge or screw type twinning dislocations, and that the paths taken by atoms during twin interface motion will be defined by the geometry of the dislocations.

It is reasonable to assume the large, highly ordered, grain structure observed in the 0.05 wt % C fuel would also contain a large number of dislocations with a preferred geometry. Since ϵ -hydride fuel strains, as a result of an applied stress, by twin interface motion, more dislocations would cut the surface per unit strain than in the case of the smaller, less oriented, grain structures found in 0.15 and 0.30 wt % C material.

a. 0.05 wt % C, H/Zr = 1.91

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b. 0.15 wt % C, H/Zr = 1.91

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c. 0.30 wt % C, H/Zr = 1.91

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Figure 15. Compressive Stress-Strain for Samples at 0.010 in./min Strain Rate, With Associated Hydrogen Release

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Another interesting point, as shown in these figures, are the short bursts of hydrogen observed near the rupture points of these materials. This type release is presumably a result of numerous microcracks, formed during inelastic straining, which interconnect due to the excessive deformation attained, and form macrocracks which apparently propagate to the surface of the sample. The high hydrogen releases associated with this type of crack formation is attributed to an instantaneous release of hydrogen which was previously deposited in microcracks by dislocations cutting the surface of the crack.

The rapid buildup and gradual decay of hydrogen observed in the postrupture region for these materials are attributed to anelastic and stress relaxation effects. The anelastic effect⁽⁷⁾ (elastic strain recovery with time) is probably of minor consideration, since large amounts of plastic deformation tend to minimize this effect, as will be discussed later. The primary mechanism for hydrogen release in this region is presumably a stress relaxation effect. This effect is enhanced by the large amount of sample fragmentation on failure. This increase in surface area also shortens the mean path to a surface for microcracks remaining in the bulk material at failure. The stress relaxation phenomenon tends to propagate these cracks much the same as it affects the delayed fracture phenomenon observed in high-strength steels. Zener⁽⁹⁾ has observed this, and has suggested that a gradual relaxation of shear stresses across previously formed slip bands results in a high accumulation of stress concentration at the edges of these bands. This relaxation results in such high stress concentrations that microcracks are formed, which, in turn, are further opened by residual stresses.

The time required for a ruptured sample to reach a point of zero hydrogen release is dependent upon the original microstructure. The hydride phase and strain rate, however, are also controlling factors, as will be shown later.

A test was conducted to observe the effect of strain rate on hydrogen release during room-temperature compression testing. A typical stress-strain plot, with associated hydrogen release plot, is presented in Figure 16. The strain rate chosen was 0.0025 in./min, and the material tested was identical to material used in Figure 15c. A comparison of these figures indicates the total hydrogen released in the elastic region for the slower strain rate (0.0025 in./min) is approximately four times greater than that released by the faster strain rate

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Figure 16. Compressive Stress-Strain for Sample (0.30 wt % C, H/Zr = 1.91) at 0.0025 in./min Strain Rate, With Associated Hydrogen Release

(0.010 in./min). From this observation, it can be concluded that the total hydrogen released in the elastic region is inversely proportional to the strain rate at a constant strain. This relationship is presented graphically in Figure 17a.

Although only two strain rates were evaluated, it is felt that the relationship exhibited in Figure 17a is valid. It is important, however, to note that this is strictly an empirical relationship; and a more quantitative study should be conducted, in order to place statistical reliability on the data.

Another interesting point observed in the elastic region for both strain rates is the relationship of hydrogen release rate to strain. This relationship is presented graphically in Figure 17b. As indicated in the figure, the hydrogen release rate seems to be directly proportional to strain, and is quite independent of strain rate at a constant strain.

These interrelationships were not generally observed in the inelastic region, for the total hydrogen released in the inelastic region was approximately the same for both strain rates.

The lower hydrogen release rate observed in the postrupture region for the slower strain rate, as compared to the hydrogen release observed for the faster strain rate, is presumably due to a reduction in the strain hardening effect normally associated with high strain rates. This effect essentially lowers the internal stresses at failure, and minimizes the stress relaxation effect.

Another test was conducted to determine the effect of constant load on the hydrogen release phenomenon. The material and strain rate chosen was comparable to that tested in Figure 15c. The stressing program, along with the associated hydrogen release plot, is presented in Figure 18. As indicated in the figure, the load was held constant near the 0.2% offset yield point, and the hydrogen release rate dropped off considerably. Continued loading reinitiated the hydrogen release. The load was again held constant near one-half the ultimate strength of the material, and a similar behavior occurred. This behavior supports earlier findings that the hydrogen release phenomenon is strain dependent. Upon releasing the load, the hydrogen release rate increased, and then decayed with time. The increase in hydrogen release upon unloading indicates that strain can be a negative displacement, such as elastic recovery, or

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a. Relationship of Total Hydrogen Released to Strain Rate

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b. Relationship of Hydrogen Release Rate to Strain

Figure 17. Relationships Observed in Determining Effect of Strain Rate on Hydrogen Release in the Elastic Region for ϵ -Hydride Material

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Figure 18. Compressive Stress-Strain for Sample (0.30 wt % C, H/Zr = 1.91) at 0.010 in./min Strain Rate, With Associated Hydrogen Release, Showing Effect of Constant Load on Hydrogen Release

a positive displacement, such as elastic strain or inelastic strain, and still release hydrogen. In the case of the rapid increase on unloading, it is presumably an elastic recovery effect. The decay with time effect is attributed to the anelasticity normally associated with ϵ hydride.⁽⁷⁾ As mentioned earlier, for samples taken to failure, this type release was primarily due to a stress relaxation effect, and the anelastic effect was assumed to be negligible. This is true, for the anelastic effect is significantly diminished by gross inelastic straining in these materials, as will be verified later. Since the total load was less than one-half the ultimate load, the anelasticity in this material is not seriously affected, and is presumed to be the controlling factor for hydrogen release in this case.

Another test was conducted to study the effect of original hydrogen content on the hydrogen release phenomenon. Hydrogen concentrations studied were 0.5 H/Zr ($\alpha + \delta$ phase), 1.50 H/Zr (also $\alpha + \delta$ phase, with much higher δ phase present), and the previously tested 1.91 H/Zr (ϵ) (Figure 15b). To eliminate extraneous effects, the carbon concentration of these materials was maintained at 0.15 wt % and the strain rate was held constant at 0.010 in./min. A typical stress-strain plot, with associated hydrogen release plot, for the 1.50 H/Zr material is presented in Figure 19. As indicated in the figure, no hydrogen release was observed in the elastic region. The fact that this hydride composition does not form the highly twinned structure indicative of ϵ hydride, and that this composition does not release hydrogen in the elastic region, supports earlier assumptions that hydrogen release in the elastic region for ϵ hydride is a result of twin interface motion. Figure 19 also shows the mode of hydrogen release in the inelastic region differs from that observed in the ϵ -hydride material, Figure 15b. The type of release observed in this region is indicative of a crack formation release, such as seen near fracture in the ϵ -hydride material. The hydrogen release observed in the postrupture region was much greater than that observed in the ϵ material. This difference is attributed to the greater internal stresses retained in this hydride composition on rupture, as compared to ϵ hydride. Zener⁽¹⁰⁾ has suggested that twin interfaces will continually adjust their positions, in such a manner as to minimize the shear stress acting across them. Presumably, this effect is continually taking place in ϵ hydride, thereby lowering the internal stresses at failure. Since the 1.50 H/Zr hydride composition is not a twinned structure, and the internal stresses are quite high at

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Figure 19. Compressive Stress-Strain for Sample (0.15 wt % C, H/Zr = 1.50) at 0.010 in./min Strain Rate, With Associated Hydrogen Release

failure; consequently, the stress relaxation effect, similar to that mentioned for hydrogen release in the postrupture region for ϵ -hydride material, would be more significant.

The 0.5 H/Zr sample was loaded to two-thirds its ultimate compressive strength, or $\sim 20\%$ reduction in length. Hydrogen release was not observed in the elastic or plastic regions. The high ductility of this material prohibited sample fracture.

Although the number of samples used for this study does not allow a valid statistical evaluation to be made, the fact that the hydrogen release phenomenon was observed on samples of different hydriding and compositional variations supports the existence of the phenomenon.

From these experiments, it is possible to postulate mechanisms of hydrogen release for various materials tested and for various regions of release observed during these tests.

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The mechanism of hydrogen release for ϵ hydride in the elastic region is postulated to be as follows. Since ϵ -hydride material strains, as a result of an applied stress, by twin interface motion, and since this interface is composed of edge and screw type dislocations, it is conceivable that uncombined hydrogen in these twinning dislocations will evolve from dislocations as they cut the surface of the sample. An increase in grain size and grain orientation in these materials would be expected to enhance hydrogen release by this mechanism, whereby more dislocations would cut the surface per unit strain, due to the resulting preferred orientation of the dislocations. A comparison of Figures 15a, 15b, and 15c tends to substantiate this theory.

The mechanism of hydrogen release for ϵ and the $\alpha + \delta$ (1.50 H/Zr) hydride material in the inelastic region is postulated to be as follows. Since bulk diffusion is extremely slow in these hydrides at room temperature, the transport of hydrogen through the lattice must rely on a short-circuiting process. In iron, for example, the evolution of hydrogen is accelerated by plastic deformation, and this is postulated to be caused by a short-circuiting mechanism, in which hydrogen "atmospheres" are dragged along with dislocations and released when dislocations emerge a free surface, such as cracks.⁽¹¹⁾ It is believed that a similar transport and evolution process is involved in the inelastic region for these materials.

The mechanism of hydrogen release in the postrupture region for all materials tested is postulated to be similar to that suggested for iron by Frank,⁽¹¹⁾ along with a stress relaxation mechanism mentioned earlier. The prior mechanism places hydrogen in cracks, whereas the stress relaxation effect propagates these cracks to a surface, thereby releasing the trapped hydrogen.

Another interesting point observed during the hydrogen loss study was the change in the Young's modulus of ϵ -hydride material due to plastic straining. As will be noted in Figure 13, the Young's modulus obtained after the sample had been stressed to approximately two-thirds of its ultimate strength was substantially higher than the original value (9.9×10^6 psi, as opposed to an original value of 5.8×10^6 psi). Based on previous observations,⁽¹⁾ a phase change was suspected, but this was ruled out by x-ray diffraction. As mentioned previously, the diffraction pattern of the sample at this point was identical with that obtained prior to the test. An examination of the microstructure after the test gave a clue to the cause for this

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unusual behavior. Figure 20 shows a large number of microcracks across the twin boundaries of the ϵ grains. Apparently, these microcracks served to pin the motion of the twin boundaries and essentially eliminated the anelasticity normally associated with ϵ hydride.⁽⁷⁾ Another interesting aspect of the photomicrograph shown in Figure 20 is the bowed appearance of the twin bands. It is felt that this is direct evidence of twin boundary motion and subsequent pinning. The bowing is caused by the twin boundaries "dragging" on the grain boundaries during sample deformation.

HF Chemical-Polish
+10% Oxalic Acid
Electrolytic Etch

4694-1-2

Figure 20. Sample That Was Compression
Tested to Successively Higher Stress
Levels to Determine Effect of
Stress on Hydrogen
Evolution

In addition to providing some insight into the elastic behavior of ϵ Zr - 10 U alloy hydride, Figure 20 gives some clues to the fundamental process of apparent plastic deformation in this material. The large number of microcracks seen in the microstructure of this sample clearly indicate that plasticity of this material is basically caused by the formation of microcracks which propagate through a minimum number of grain boundaries before being arrested. Using this deformation mechanism, it is very easy to understand the apparent higher ductility

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and greater fragmenting-on-failure observed for the high-carbon samples. As carbon content is increased, the grains become smaller; and consequently, since grain boundaries resist crack propagation, many more microcracks will form in the high-carbon samples before catastrophic failure occurs. This results in large amounts of inelastic macrodeformation and powdering of the sample at failure.

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VI. CONCLUSIONS

The major conclusions to be drawn from this study of the room-temperature compressive behavior of Zr - 10 U alloy hydride can be summarized as follows:

- 1) Compressive strength increases markedly with carbon content.
- 2) Compressive inelastic deformation of this material proceeds by massive microcracking, rather than a flow process. The fine grained, high-carbon material consequently required the most mechanical work to cause ultimate failure.
- 3) Hydriding method has no direct effect on the compressive properties, but it can affect them indirectly by affecting the microstructure.
- 4) The Young's modulus and yield point is increased by a factor of 2 to 3, and the total strain to failure is decreased by a factor of 8, by changing the hydrogen content from ϵ to the $\alpha + \delta$ (1.50 H/Zr) phase.
- 5) The Young's modulus is increased by microcracks, formed during either hydriding or compression testing.
- 6) The total amount of hydrogen released from these materials as a result of room-temperature compression testing was in the vicinity of 0.02 to 0.03 wt %.
- 7) Three distinct regions of hydrogen release were observed during room-temperature compression testing of these materials. They are the elastic, inelastic, and postrupture regions.
- 8) Grain size and grain orientation in ϵ -hydride material increases the hydrogen release in all three regions of release.
- 9) The effect of strain rate on hydrogen release is as follows:
 - a) The total amount of hydrogen released in the elastic region for ϵ -hydride material is inversely proportional to the strain rate, for a constant strain.
 - b) The hydrogen release rate in the elastic region for ϵ -hydride material is directly proportional to the strain.

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10) The effect of constant load on the hydrogen release phenomenon resulted in a verification of earlier assumptions that the hydrogen release phenomenon is strain dependent.

11) The effect of H/Zr on the hydrogen release phenomenon resulted in no release of hydrogen in the elastic region for the $\alpha + \delta$ hydride (1.50 H/Zr) material; also no release of hydrogen in $\alpha + \delta$ (0.5 H/Zr) material.

12) Mechanisms of hydrogen release due to room-temperature compression testing were postulated for the three regions of observed release.

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REFERENCES

1. L. B. Lundberg, "Parametric Studies on the Short-Term Tensile Mechanical Properties of Zr - 10 U Alloy Hydrides," NAA-SR-9875 (July 15, 1964)
2. J. W. Raymond and S. M. Toy, "Mechanical Strength of Hydrides of Zirconium and Zirconium-Base Alloys," NAA-SR-6732 (April 15, 1962)
3. D. C. Campbell, H. D. Decker, T. S. Kirsch, C. A. Martin, and F. C. Schrag, "SNAP 8 Experimental Reactor Fabrication and Engineering," NAA-SR-8430 (March 1, 1964)
4. H. Taketani, "Techniques for Massive Hydriding," NAA-SR-9705 (April 20, 1965)
5. R. L. Beck, "Zirconium-Hydrogen Phase System," Trans. ASM, 55, No. 3 (September 1962) pp 542-555
6. F. J. Digesu and C. D. Pears, "Some Parameters of the Mechanical Behavior of Graphite," A paper presented at the American Ceramic Society Meeting, Los Angeles (October 1963)
7. R. Chang, "Phase Transformation, Twinning and Anelastic Phenomenon Associated with Zirconium Hydride," NAA-SR-5537 (September 15, 1960)
8. R. Chang, "Phase Transformation, Twinning and Anelastic Phenomenon Associated with Zirconium Di-Hydride," J. Nuc. Mat. 2, No. 4 (1960) pp 335-340
9. C. Zener, Elasticity and Anelasticity of Metals (The University of Chicago Press, Chicago, Ill., 1948) p 146
10. C. Zener, Elasticity and Anelasticity of Metals (The University of Chicago Press, Chicago, Ill., 1948) p 160
11. R. C. Frank, Proc. Symp. Int. Stresses Fatigue Met, Elsevier, Amsterdam (1959) p 411