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TRITIUM STORAGE DEVELOPMENT

PROGRESS REPORT NO. 13
JULY-SEPTEMBER 1977

Prepared for the
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NUCLEAR WASTE MANAGEMENT RESEARCH GROUP
DEPARTMENT OF NUCLEAR ENERGY

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P. COLOMBO AND R. JOHNSON

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SUMMARY

Engineering scale operation was started on hydriding of zirconium for application to tritium storage systems. Eight runs were made with commercial-grade zirconium sponge at 338° to 499°C and 0 to 770 torr, hydrided to hydrogen-to-zirconium atom ratios up to 2. Reaction rates were determined for hydrogen flow rates of 2.72 to 9.24 cm^3 at STP/g Zr-min. For each run, hydrogen pressure was plotted on a semilog scale against time and H/Zr atom ratio to elucidate the reaction mechanism in terms of reaction kinetics, diffusion, and equilibrium hydrogen vapor pressure. Lack of consistency in some of the runs is probably due to differences in the extent of the diffusion barrier formed on the metal because of reaction with oxygen and other contaminants. Static leach testing of zirconium sponge and rod in distilled water was continued; leach rates ranged from 8.9×10^{-7} to 1.7×10^{-6} cm/day for the 166 to 208 day leach period.

I. TRITIUM STORAGE IN METAL HYDRIDE

A program has been initiated to demonstrate a safe and economical process for the fixation of tritium as a tritide in a metal hydride. For tritium absorption and retention purposes, zirconium appears to be most desirable although alternative such as Ti, Hf, V, Nb and certain alloys of these can also be used. The choice of zirconium as the leading candidate metal for this study is based on the known chemical and physical properties of the metal and its hydride. The process can be reversed, if desired, by heating the hydride above its decomposition temperature (defined as the temperature at which the hydride dissociation pressure is above 1 atm) and collecting the evolved gas. While the fixation process will be developed for long term storage or disposal by burial, retrievability of the tritium will be considered for future needs such as for use in controlled thermonuclear reactors.

A. Engineering Scale Equipment

In the engineering scale flowsheet (see Figure 1), a feed stream of H_2 or HT is regulated by a flow rate controller provided with a flow integrator. The pressure is monitored by gauges and pressure transducers (maximum design pressure: 100 psig). A vacuum system consisting of a cold trap, diffusion pump, and mechanical pump capable of producing a vacuum of 10^{-4} to 10^{-5} torr is provided for outgassing the reaction metal and removal of trace quantities of oxygen, nitrogen, etc., which have an inhibiting effect on the tritide-hydride reaction. The reaction vessel (see Figure 2) is approximately 3 inches in diameter and 19 inches high and is provided with a porous metal filter to prevent discharge of reaction product fines. An electric heater clamped externally to the vessel is used to provide an operating temperature up to 600 °C. Temperatures in the reactor are monitored externally and also internally with a centerline thermowell. The reaction metal is placed in a wire mesh basket inside the reactor to permit optimum positioning for temperature control and measurement, and also to avoid expansion problems

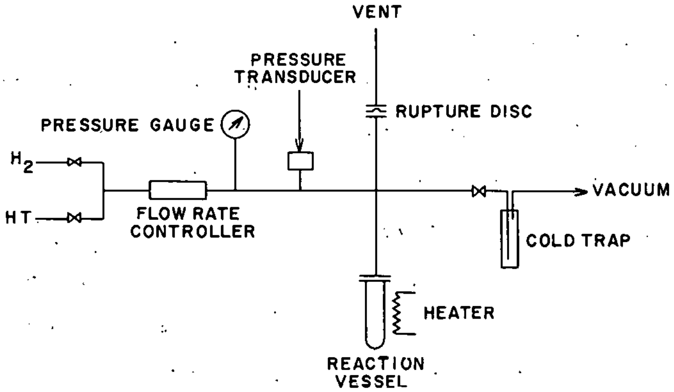

Figure 1. Tritium storage in metal hydride - engineering scale flowsheet.

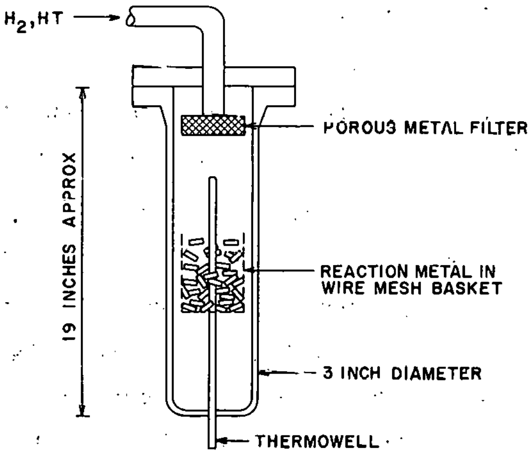

Figure 2. Metal hydride reaction vessel.

that can result from hydride formation. The assembled equipment and instrumentation is shown in Figures 3 and 4.

B. Hydriding Procedure and Results

A total of eight experiments were conducted in the engineering scale equipment with commercial grade zirconium sponge. The zirconium for these experiments were from the same lot.* A void volume of 13.7% was determined on one piece of sponge, which is typically irregular in shape and porous, by machining it to a cube and comparing its bulk density to the density of solid zirconium. Up to three pieces of sponge were used in each run with total weights of 17 to 40 g. The sponge samples were washed in water several times to remove $MgCl_2$ present from the magnesium reduction processing step, degreased with acetone, and then placed in the reaction vessel basket for outgassing prior to hydriding. The samples were outgassed from one to three days at room temperature (or up to 100 °C), and outgassing was continued during heating to operating temperature until a final vacuum reading of approximately $1x10^{-4}$ torr was reached.

For each hydriding run, commercial-grade, 99.95% minimum purity hydrogen was fed into the reaction system at a constant rate to determine the reaction rate during flow under essentially isothermal conditions. Hydrogen feed rates of 2.72 to 9.24 cm^3 at STP/g Zr-min and temperatures from 338°to 499°C were investigated. The hydrogen pressure in the reaction system was measured and recorded during each run and then plotted against time (Figures 5-12). Values were taken from these curves and used in calculating the reaction rate and average H/Zr atom ratio for various reaction times (Tables 1-8). The pressure buildup rate in the reaction system was taken into

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* Supplied by Amax Specialty Metals, Inc., Akron, N.Y., and having the following analysis: 0.09% chromium + Iron, 0.01% hafnium, <0.05% carbon, < 0.01% nitrogen, > 99.2% zirconium and hafnium.

account in these calculations and was subtracted from the hydrogen gas feed rate to determine the reaction rate and also the average H/Zr atom ratio. The "apparent" volume of the system was determined at several temperatures to establish a linear plot and was used in the PVT relationship for calculating the hydrogen pressure buildup.

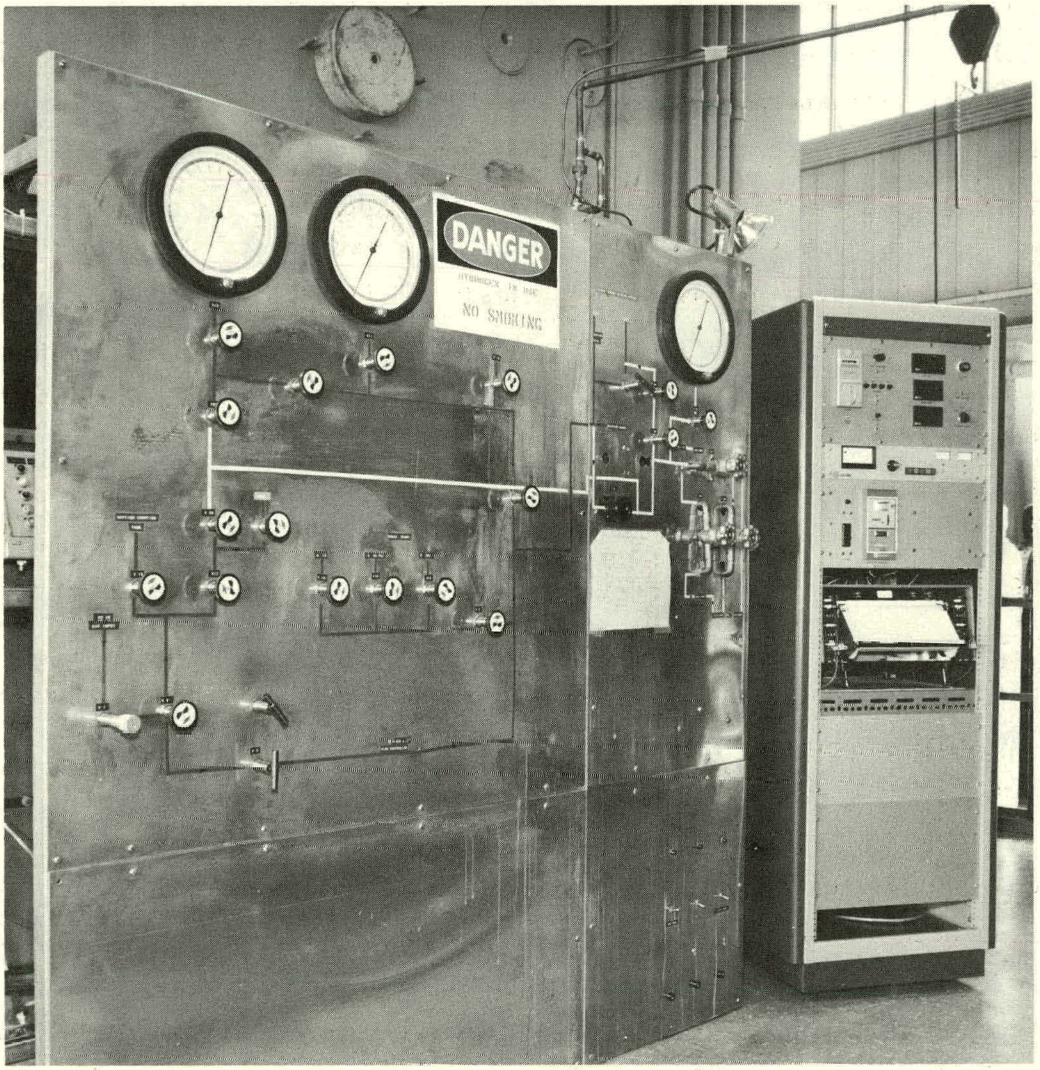

Figure 3. Engineering scale metal hydride system
- front view.

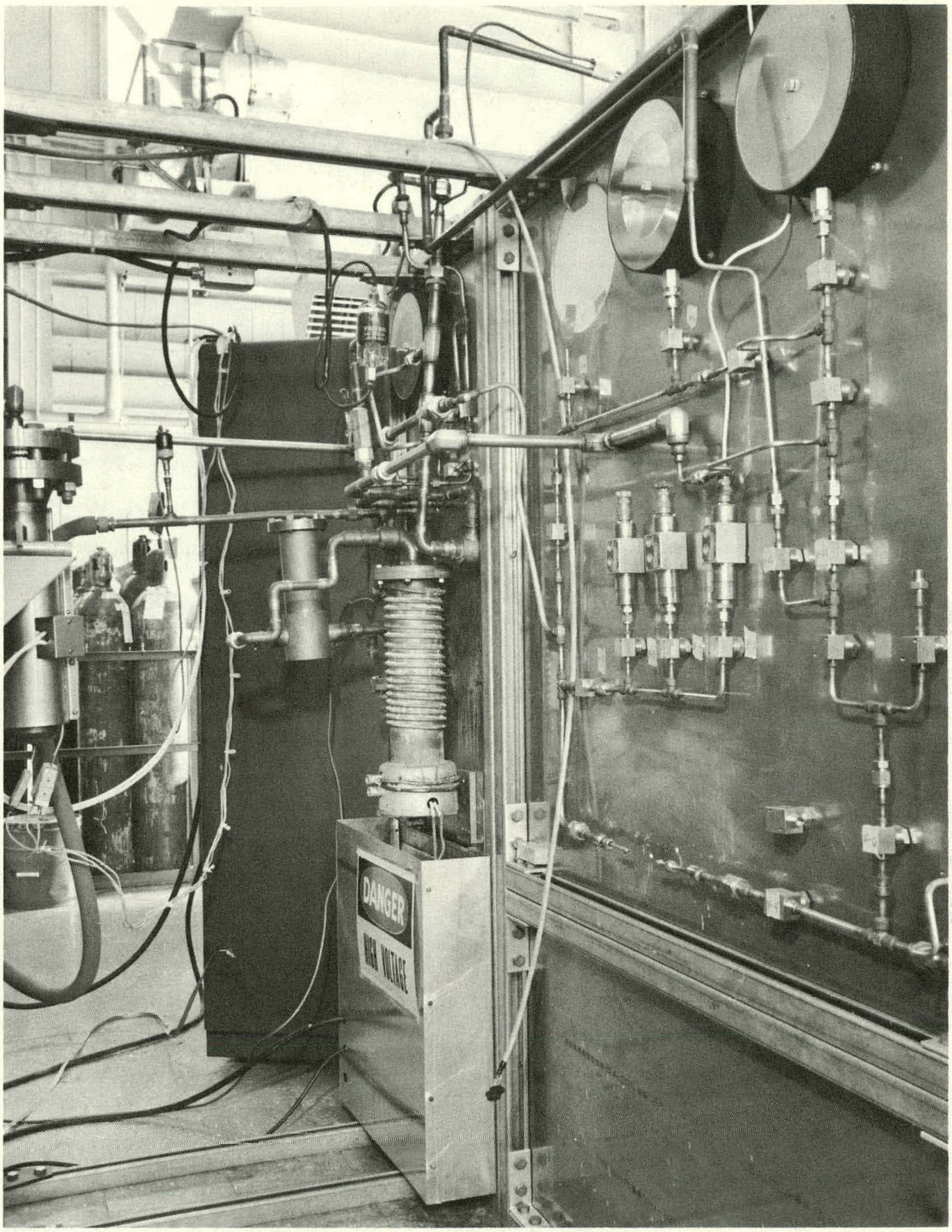

Figure 4. Engineering scale metal hydride system - rear view.

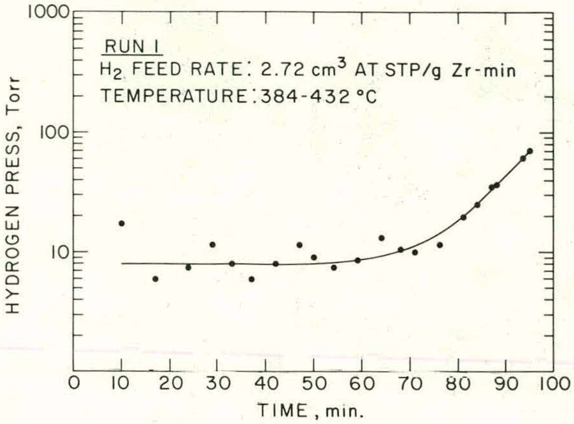

Figure 5. Hydrogen pressure vs. reaction time - run 1.

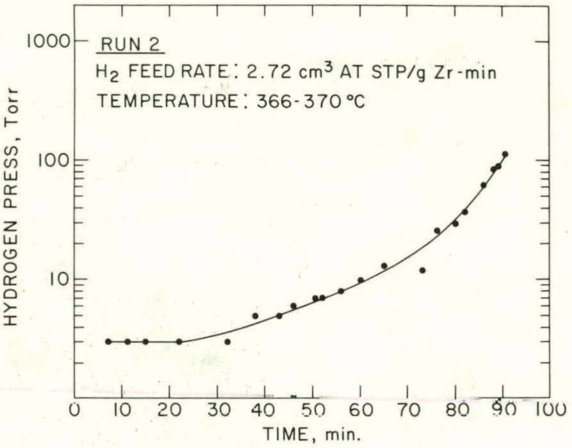

Figure 6. Hydrogen pressure vs. reaction time - run 2.

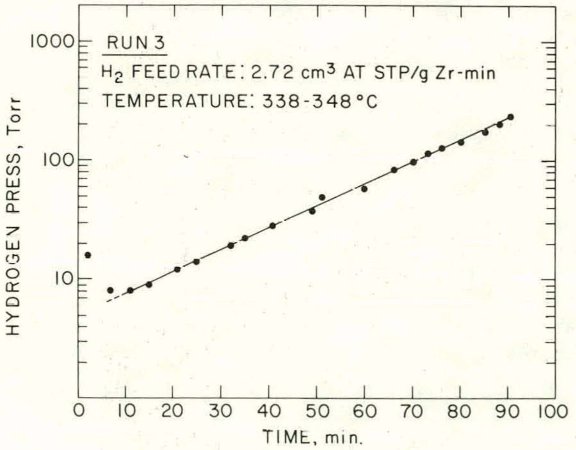

Figure 7. Hydrogen pressure vs. reaction time - run 3.

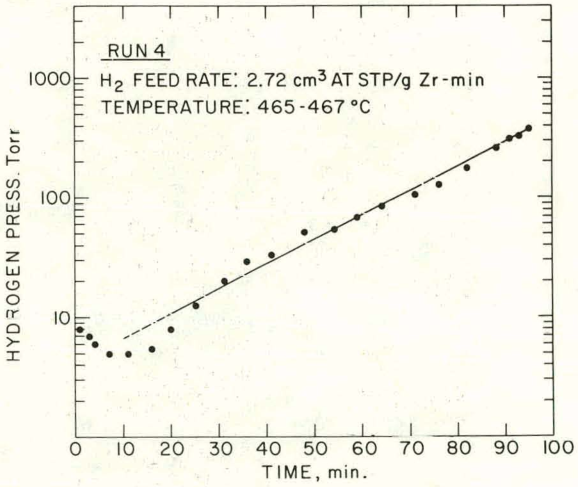

Figure 8. Hydrogen pressure vs. reaction time - run 4.

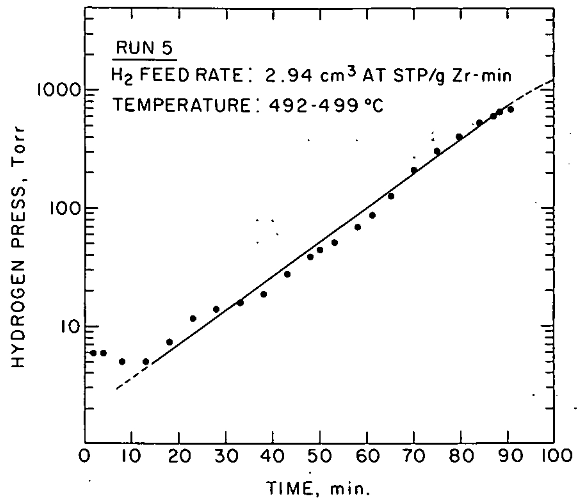

Figure 9. Hydrogen pressure vs. reaction time - run 5.

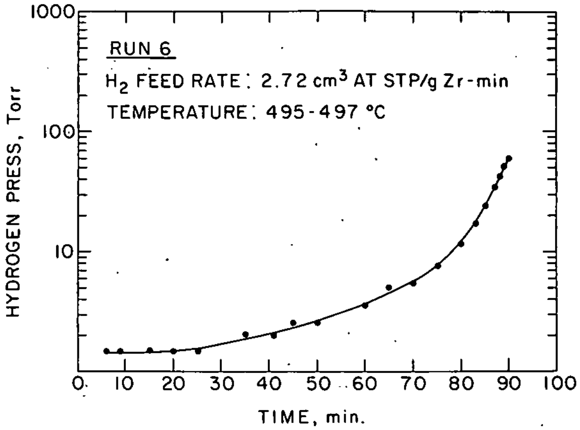

Figure 10. Hydrogen pressure vs. reaction time - run 6.

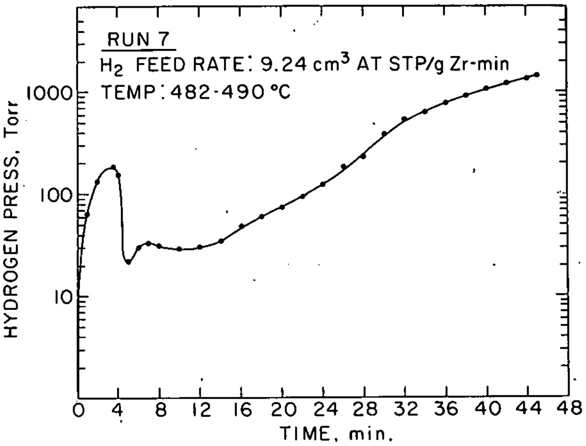

Figure 11. Hydrogen pressure vs. reaction time - run 7.

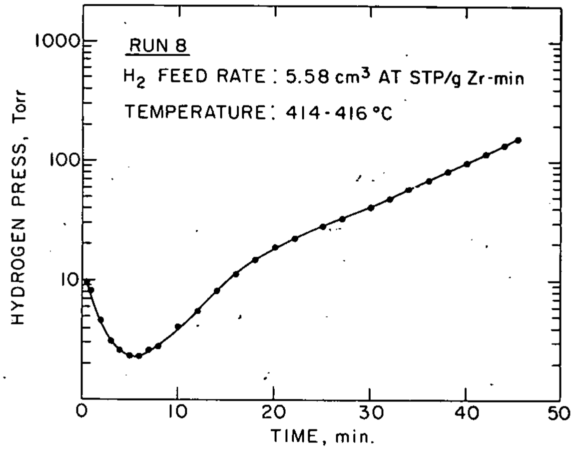

Figure 12. Hydrogen pressure vs. reaction time - run 8.

TABLE 1

Hydrogen Reaction Rate with Zirconium Sponge - Run 1

Time (min)	Pressure (torr)	H/Zr atom ratio	Reaction rate (cm^3 at STP/g Zr-min)
10-50	8.0	0.11-1.00	2.72
55	8.3	1.10	2.72
60	8.8	1.21	2.71
65	9.5	1.32	2.71
70	11.0	1.43	2.70
75	13.2	1.54	2.68
80	18.2	1.65	2.65
85	27.5	1.76	2.57
90	47	1.86	2.47
95	70	1.96	2.24

Zirconium Weight: 36.7463g total (three pieces)

Hydrogen feed rate: 1.36 cm^3 at STP/g Zr-min (0-10 min)
2.72 cm^3 at STP/g Zr-min (10-95 min)

Temperature: 384-432°C (408°C average)

Apparent system volume: 4.325 liters

TABLE 2

Hydrogen Reaction Rate with Zirconium Sponge - Run 2

Time (min)	Pressure (torr)	H/Zr atom ratio	Reaction rate (cm^3 at STP/g Zr-min)
10-20	3.0	0.22-0.44	2.72
25	3.1	0.55	2.72
30	3.4	0.66	2.72
35	3.9	0.77	2.72
40	4.5	0.88	2.72
45	5.4	0.99	2.71
50	6.4	1.10	2.71
55	7.6	1.21	2.70
60	9.4	1.32	2.70
65	11.5	1.43	2.68
70	15.2	1.54	2.66
75	21.5	1.65	2.62
80	32.5	1.75	2.56
85	53	1.85	2.46
90	100	1.93	2.14
90.5	112	1.95	2.09

Zirconium weight: 40.5743g total (three pieces)

Hydrogen feed rate: 2.72 cm^3 at STP/g Zr-min

Temperature: 366-370 °C, 350 °C at start

Apparent system volume: 4.167 liters

TABLE 3

Hydrogen Reaction Rate with Zirconium Sponge - Run 3

Time (min)	Pressure (torr)	H/Zr atom ratio	Reaction rate (cm^3 at STP/g Zr-min)
10	7.6	0.22	2.70
15	9.5	0.33	2.70
20	11.5	0.44	2.69
25	14.5	0.55	2.69
30	18.0	0.65	2.68
35	22.5	0.76	2.67
40	27.5	0.87	2.65
45	34	0.98	2.64
50	42	1.08	2.62
55	52	1.19	2.59
60	64	1.30	2.56
65	79	1.40	2.53
70	98	1.50	2.48
75	120	1.60	2.42
80	150	1.70	2.35
85	185	1.79	2.26
90.5	235	1.89	2.14

Zirconium weight: 40.8885g total (three pieces)

Hydrogen feed rate: 2.72 cm^3 at STP/g Zr-min

Temperature: 338-348 °C, 328 °C at start

Apparent system volume: 4.069 liters

TABLE 4

Hydrogen Reaction Rate with Zirconium Sponge - Run 4

Time (min)	Pressure (torr)	H/Zr atom ratio	Reaction rate (cm^3 at STP/g Zr-min)
10	6.7	0.22	2.70
15	8.5	0.33	2.70
20	10.6	0.44	2.69
25	14.5	0.55	2.68
30	17.5	0.66	2.67
35	22.0	0.77	2.66
40	28.0	0.87	2.64
45	35	0.98	2.62
50	44	1.09	2.59
55	55	1.19	2.56
60	71	1.30	2.51
65	90	1.40	2.45
70	114	1.50	2.38
75	144	1.59	2.29
80	182	1.68	2.17
85	230	1.77	2.03
90	290	1.85	1.85
95	370	1.92	1.61

Zirconium weight: 34.4185g total (three pieces)

Hydrogen feed rate: 2.72 cm^3 at STP/g Zr-min

Temperature: 465-467 °C, 450 °C at start

Apparent system volume: 4.553 liters

TABLE 5

Hydrogen Reaction Rate with Zirconium Sponge - Run 5

Time (min)	Pressure (torr)	H/Zr atom ratio	Reaction rate (cm^3 at STP/g Zr-min)
10	5.0	0.24	2.92
15	5.4	0.36	2.92
20	7.1	0.48	2.92
25	9.9	0.59	2.90
30	13.5	0.71	2.89
35	19.0	0.84	2.86
40	26.5	0.95	2.84
45	37	1.06	2.80
50	51	1.17	2.74
55	72	1.28	2.66
60	100	1.39	2.54
65	140	1.49	2.39
70	195	1.58	2.17
75	275	1.66	1.86
80	380	1.73	1.44
85	530	1.78	0.85
90	740	1.80	0.00
90.3	770	1.80	0.00

Zirconium weight: 36.6760g total (three pieces)

Hydrogen feed rate: 2.94 cm^3 at STP/g Zr-min)

Temperature: 492-499 °C, 475 °C at start

Apparent system volume: 4.671 liters

TABLE 6

Hydrogen Reaction Rate with Zirconium Sponge - Run 6

Time (min)	Pressure (torr)	H/Zr atom ratio	Reaction rate (cm^3 at STP/g Zr-min)
10	1.4	0.22	2.72
15	1.5	0.33	2.72
20	1.5	0.44	2.72
25	1.6	0.55	2.72
30	1.7	0.66	2.72
35	1.9	0.77	2.72
40	2.1	0.89	2.72
45	2.4	1.00	2.72
50	2.7	1.11	2.72
55	3.2	1.22	2.72
60	3.7	1.33	2.71
65	4.6	1.44	2.71
70	5.8	1.55	2.70
75	7.8	1.66	2.68
80	12.0	1.77	2.63
85	24.0	1.88	2.44
90	60	1.96	1.76
90.3	66	1.97	1.76

Zirconium weight: 32.3147 g total (two pieces)

Hydrogen feed rate: 2.72 cm^3 at STP/g Zr-min

Temperature: 495-497 °C, 476 °C at start

Apparent system volume: 4.671 liters

TABLE 7

Hydrogen Reaction Rate with Zirconium Sponge - Run 7

Time (min)	Pressure (torr)	H/Zr atom ratio	Reaction rate (cm^3 at STP/g Zr-min)
2	132	0.01	0.76
3.5	184	0.07	9.24
4	154	0.14	30.9
4.3	70	0.25	< 50
6	30	0.42	8.14
7	33	0.49	9.24
8	31	0.57	9.46
10	29	0.72	9.24
12	30	0.87	9.07
14	35	1.02	8.78
16	48	1.15	8.76
18	60	1.29	8.31
20	74	1.43	8.12
22	94	1.56	7.86
24	125	1.68	6.90
26	184	1.76	5.39
28	230	1.86	2.83
30	383	1.86	0.59
32	504	1.88	0.76
34	639	1.89	0.59
36-45	776-1427	1.90	0.00

Zirconium weight: 17.0977g total (two pieces)

Hydrogen feed rate: 9.24 cm^3 at STP/g Zr-min

Temperature: 482-490°C, 476°C at start

Apparent system volume: 4.631 liters

TABLE 8

Hydrogen Reaction Rate with Zirconium Sponge - Run 8

Time (min)	Pressure (torr)	H/Zr atom ratio	Reaction rate (cm^3 at STP/g Zr-min)
2	4.6	0.09	5.74
4	2.6	0.18	5.61
6	2.3	0.27	5.57
8	2.9	0.36	5.54
10	3.9	0.45	5.51
12	5.6	0.54	5.45
14	8.2	0.63	5.45
16	11.5	0.72	5.42
18	14.9	0.80	5.39
20	18.5	0.89	5.37
22	22.5	0.98	5.36
24	26.4	1.07	5.35
26	30.5	1.15	5.34
28	36.0	1.24	5.27
30	41.5	1.33	5.20
32	49.5	1.41	5.11
34	58	1.49	5.03
36	69	1.57	4.93
38	82	1.65	4.83
40	96	1.73	4.68
42	114	1.81	4.52
44	138	1.88	4.34
45.4	155	1.93	4.20

Zirconium weight: 21.4053g total (one piece)
Hydrogen feed rate: 5.58 cm^3 at STP/g Zr-min
Temperature: 414-416°C, 386°C at start
Apparent system volume: 4.352 liters

A sufficient amount of hydrogen was fed into the reaction system in each run to react the zirconium to its theoretical limit of ZrH_2. In the runs where this theoretical limit was not exceeded, the pressure was down to zero by the following day, with the system at room temperature.

The hydrogen pressure for the eight runs (Figures 5-12) indicates the relationship with time to be either linear or monlinear when plotted on a semilog scale. This relationship can probably be best examined on the basis of the following three steps involved in the overall hydrogen-metal reaction:

(1) hydrogen-metal reaction kinetics,
(2) diffusion of hydrogen into the metal, and
(3) equilibrium hydrogen vapor pressure of the metal hydride.

The rate of reaction of hydrogen with metal, step (1) above, is proportional to the activity of the reactive species, which may be either diatomic or monatomic hydrogen. Gulbransen and Andrew (1) have determined that the reaction of hydrogen with zirconium at 300°C follows the square root of pressure law for initial reaction rates, which indicates that the reactive species is monatomic hydrogen; diatomic hydrogen is too large a molecule, apparently, to penetrate the zirconium lattice. The reaction, therefore, is of order ½ and the reaction rate is approximately proportional to the square root of the system hydrogen pressure when the diffusion of monatomic hydrogen is not a limiting step and the equilibrium hydrogen vapor pressure is not significant. However, regardless of the order of the hydrogen-metal reaction, when step (1) is controlling, the hydrogen pressure should remain constant when the hydrogen feed rate (or reaction rate) is also constant. In Figure 5, the hydrogen pressure is constant during the initial reaction period. However, as the reaction progresses, there is a sharp rise in the hydrogen pressure, which indicates an increase in the diffusion effect. As the diffusion step becomes controlling because of the greater hydrogen penetration depth into the metal, an increase must occur in the hydrogen concentration gradient (or H/Zr atom ratio) with a concomitant higher hydrogen equilibrium vapor pressure. The system hydrogen pressure is therefore the sum of steps (1) through (3).

Runs 2 and 6 (Figures 6 and 10) show a similarity to run 1, but with a much shorter constant or nearly constant reaction period. By comparison, the initial hydrogen pressure in run 1 is higher, possibly because of a diffusion barrier at the surface of the metal. Otherwise, all three runs show a similar increase in pressure with time, which indicates that a similar diffusion mechanism is involved as the reaction progresses.

Runs 3-5 as plotted in Figures 7-9 show very good linearity on a semilog scale, except for the initial monisothermal period. The much higher pressures, however, indicate that the effect of reaction kinetics is insignificant in these runs. Most likely the higher pressures are the result of an appreciable diffusion barrier that extends to a considerable depth into the metal.

Since all of the reaction times were not the same, semilog plots were made of the hydrogen pressure vs. the calculated average H/Z atom ratio so as to permit a more valid comparison. Runs 1 and 8 (Figure 13) were made at approximately the same temperature range (384 - 432°C and 414 - 416°C, respectively) so that the effect of approximately doubling the hydrogen feed rate in run 8 (5.58 vs 2.72 cm^3 at STP/g Zr-min) can be seen in the higher pressure that resulted in run 8, except for the initial reaction period. This same increased pressure effect can be seen when comparing runs 6 and 7 (Figure 14), which were made at higher temperature ranges (495 - 497°C and 482 - 490°C, respectively) with the flow rate in run 7 approximately 3½ times faster than in run 6 (9.24 vs. 2.72 cm^3 at STP/g Zr-min). The increased flow rate in run 7 probably caused the delayed initial reaction of the two sponge specimens as shown by the two pressure peaks on the initial part of the curve. The similarity of runs 6 and 7 can be seen when the ratio of the hydrogen pressure of run 6 to run 7 is compared for any H/Zr atom ratio between 0.8 and 1.7.

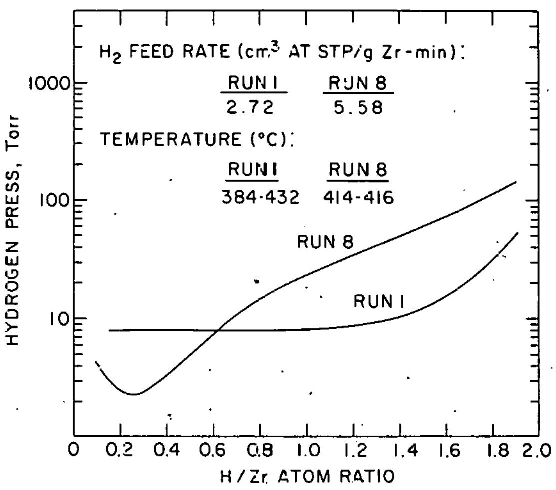

Figure 13. Hydrogen pressure vs. H/Zr atom ratio - runs 1 and 8.

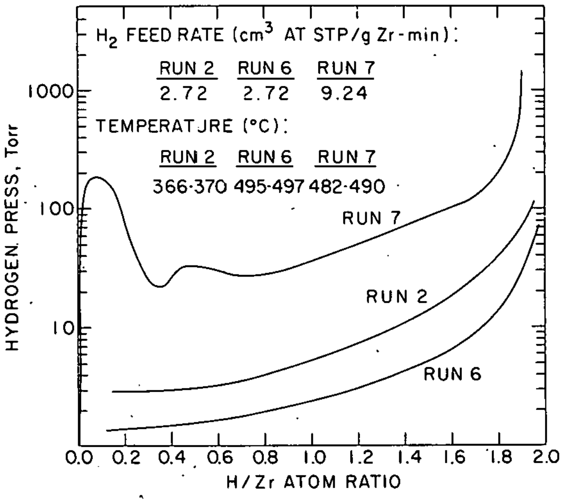

Figure 14. Hydrogen pressure vs. H/Zr atom ratio - runs 2, 6, and 7.

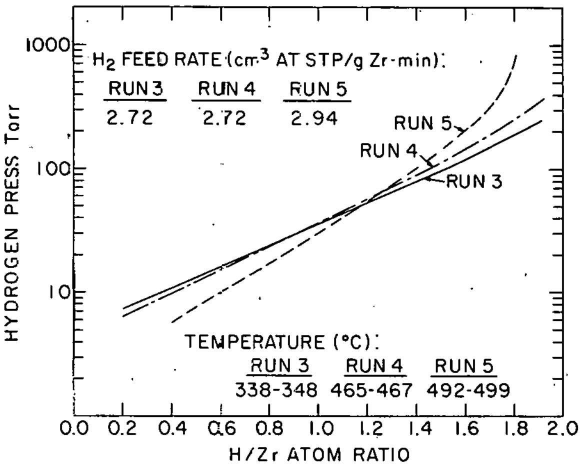

Figure 15. Hydrogen pressure vs. H/Zr atom ratio - runs 3-5.

This value is nearly constant, ranging between 14.5 and 16.7. Runs 2 and 6 were made at the same hydrogen feed rate (2.72 cm^3 at STP/Zr-min) but at different temperature ranges (366 - 370°C vs. 495 - 497°C). The hydrogen pressure ratio of run 2 to run 6 in this case is less constant, however, and varies between 1.9 and 2.8 for H/Zr atom ratios between 0.2 and 1.8.

Runs 3-5 show less linearity when plotted against the H/Zr atom ratio (Figure 15) than when plotted against time, especially at the higher H/Zr atom ratios. Runs 3 and 4 were made at the same hydrogen feed rate (2.72 cm^3 at STP/g Zr-min) but at different temperature ranges (338 - 348°C and 465 - 467°C, respectively), yet the two plots are nearly coincident, thus indicating little temperature effect. The pressure in run 5 shows a steeper increase than in the other two runs, perhaps because of the slightly higher feed rate (2.94 vs. 2.72 cm^3 at STP/g Zr-min) and also because of a greater hydrogen equilibrium pressure effect, especially at the higher H/Zr atom ratios.

The hydrided specimens are shown in Figures 16 and 17 and can be compared with nonhydrided sponge labeled Zr in the same figures. The specimens were reacted to an average H/Zr ratio of at least 1.8 during the hydrogen feed period, and the reactions did continue to or near completion after shut down. In general, the highest degree of physical integrity was observed in those runs where the hydrogen pressure remained low and the semilog plots were nonlinear. The specimens from run 1 and 6 remained essentially intact. By comparison, the specimens from runs 3-5, where the hydrogen pressure was high and the semilog plots were linear, were severly broken. No effect of temperature, which ranged from 338 - 348°C for run 3 to 492 - 499°C for run 5 was observed. The zirconium sponge as received from the supplier normally contains some cracks. Hydriding, even under the best conditions, does tend to increase the number of cracks and enlarge existing cracks. All hydrided specimens were friable and care was required during handling to prevent further breakage.

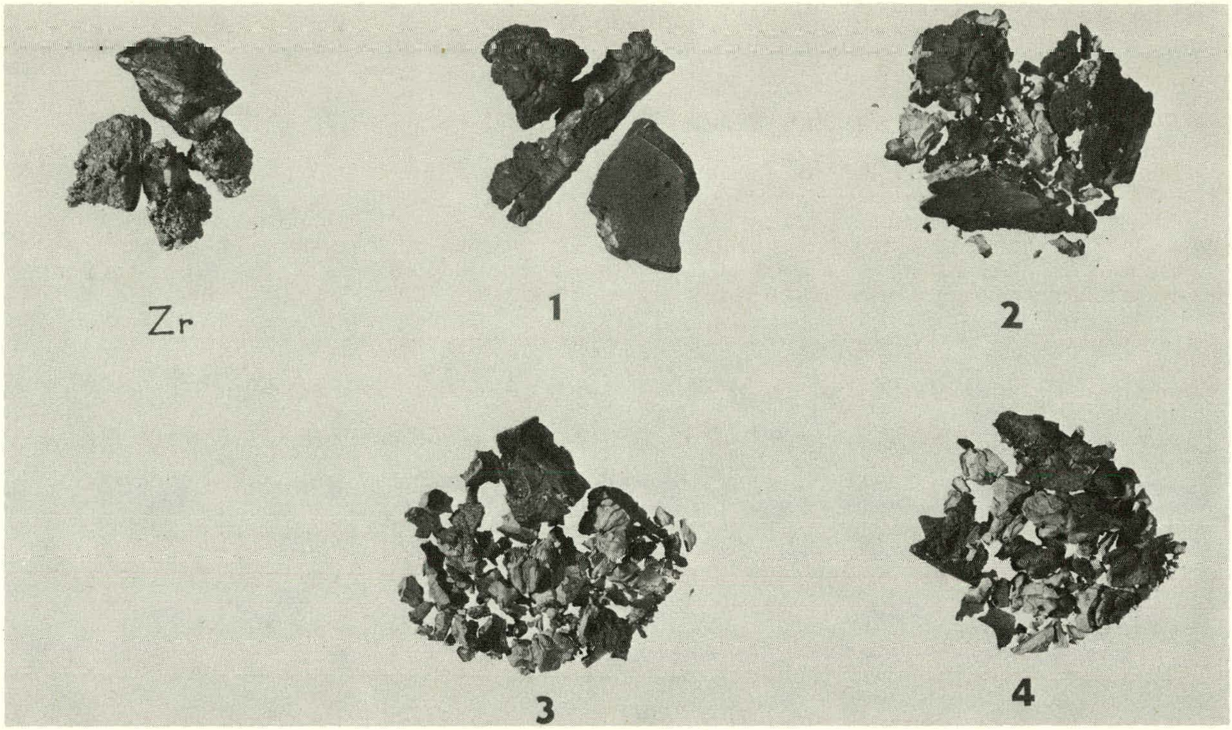

Figure 16. Hydrided specimens from runs 1-4 and nonhydrided zirconium sponge.

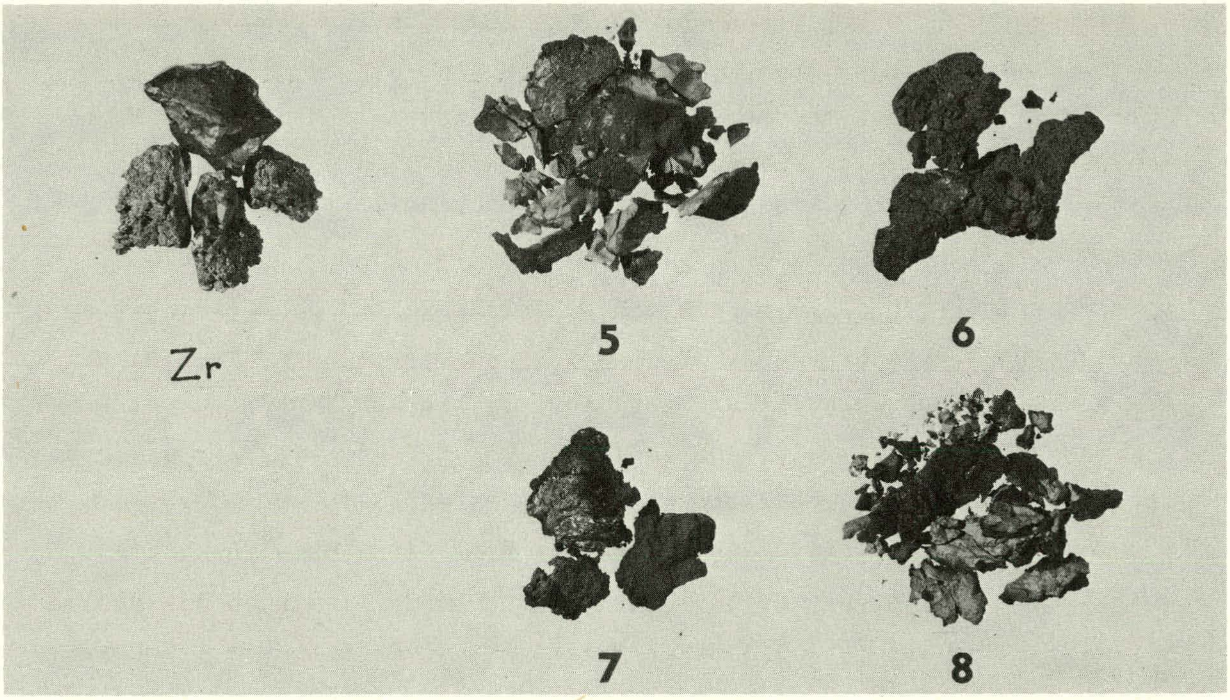

Figure 17. Hydrided specimens from runs 5-8 and nonhydrided zirconium sponge.

Hydriding to a lower hydrogen-to-metal ratio should reduce the friability and improve the physical integrity.

C. Discussion of Hydriding Results

The lack of consistency in the data makes a more quantitative comparison difficult. This lack of consistency can probably be attributed to differences in the rate of diffusion of hydrogen from specimen to specimen and is most likely the effect of oxides, nitrides, etc., forming diffusion barriers on the surface, as well as perhaps deeper into the bulk of the metal. This inhibition effect on the hydriding of zirconium has been observed by other workers (2) and can in fact result in extremely low or negligible reaction rates. The surface preparation procedure and, perhaps even more so, the outgassing procedure and vacuum system are important. Because of the porosity and irregularity of the sponge surface which precludes the use of mechanical treatment, the surface preparation is limited to washing and chemical treatment. Better control of the outgassing procedure with respect to time, temperature, and vacuum attainment should improve the consistency of the results from run to run. Of course the surface condition and impurity content are subject to variation from one source of supply to another and also from batch to batch. In addition, the purity of the hydrogen gas must be taken into account, particularly with regard to reactive impurities such as oxygen, nitrogen, water, and carbon dioxide.

The reaction rates obtained in these runs are more than adequate in terms of tritium retention requirements for the future. If a maximum tritium production rate of 2×10^7 Ci/yr from power-spent reactor fuels for the year 2000 is assumed based on estimates made by Kullen, Trevorrow, and Steindler (3) and an average tritium activity of 5000 Ci/l in the final waste stream prior to electrolysis is also assumed, a reaction rate of 2.5 cm^3 of HT at STP/g Zr-min would require an inventory of only about 3.8 kg of zirconium per batch for a 24 hr/day continuous operation. This would correspond to a 68 min reaction time for an H/Zr atom ratio of 1.5. However, because of the high heat of reaction (approximately 39 kcal/g mole of hydrogen reacted) the limiting factor in the design of the reaction system would probably be heat transfer rather than reaction rate.

D. Static Leach Testing of Zirconium Hydride

Static leach testing of zirconium sponge and rod specimens prepared previously (4), (5) was continued. The tritiated specimens, were immersed in 25 ml of distilled water which was not changed during the 166 to 208 day leach period. Two milliliter aliquots were removed, filtered, and then counted in a liquid scintillation counter.

The leach rates expressed as (cummulative fraction tritium release per day) x (V/S) ranged from 8.9×10^{-7} to 1.7×10^{-6} cm/day (see Table 9). The factor V/S, where V is the specimen volume and S is its geometric surface area, is included so that the tritium release as expressed is independent of sample geometry and size. Zirconium sponge is irregularly shaped and quite porous and its V/S was conservatively estimated to be equivalent to a geometric cylinder of equal weight with a diameter of one half its length. These leach rates are approximately one half the values reported previously (5) for the same specimens that were leached for a shorter time period.

TABLE 9

Static Leach Testing of Zirconium in Distilled Water(a)

Sample No.	Form	H/Zr atom ratio	Cummulative fraction tritium release rate, day^{-1}	V/S, cm(b)	(Cummulative fraction tritium release rate) x (V/S), cm/day
20b	Sponge	1.99	$1.5x10^{-5}$	0.095	$1.4x10^{-6}$
20e	Sponge	1.98	$1.5x10^{-5}$	0.095	$1.4x10^{-6}$
20m	Sponge	1.65	$1.7x10^{-5}$	0.099	$1.7x10^{-6}$
20n	Sponge	1.99	$1.4x10^{-5}$	0.113	$1.6x10^{-6}$
28e	Sponge	1.54	$6.5x10^{-6}$	0.132	$9.0x10^{-7}$
24c	¼" Rod	0.81	$8.0x10^{-6}$	0.127	$1.0x10^{-6}$
24e	½" Rod	0.84	$8.6x10^{-6}$	0.104	$8.9x10^{-7}$

(a) Leach time of 166 to 208 days

(b) V/S for sponge estimated to be equivalent to a geometric cylinder of equal weight and with a diameter of one half its length.

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