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CREVICE CORROSION OF URANIUM AND URANIUM ALLOYS

by

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and

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March, 1955

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Chief, Declassification Branch

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CREVICE CORROSION OF URANIUM AND URANIUM ALLOYS

by

J. W. Frank and A. H. Roebuck

ABSTRACT

In order to study crevice corrosion of uranium in high temperature (500°F) water, three types of experiments were undertaken. These were (1) capsule experiments, (2) bonded plate-pin hole experiments, and (3) gaseous hydriding experiments. These experiments are described in detail.

The first two types of experiments showed that the corrosion of enclosed uranium by water at this temperature differed markedly from that of uranium in open contact with water, as had been found to be true at lower temperatures.(1) In order to verify that the mechanism previously proposed(2) held true for this temperature, the third type of experiment was undertaken. These experiments were made at high temperatures and low pressures, and showed a definite relationship between rate of reaction and (1) temperature, (2) gas pressure, and (3) alloy reacting. The corrosion resisting alloys apparently react readily, but require an induction period.(2) After the induction period, they behave similarly to unalloyed uranium.

Capsule Experiments

In order to extend the work of other investigators(1,3,4) to water at 500°F, crevice corrosion was simulated, such as might take place with unbonded fuel elements. Cylinders of uranium and uranium alloys were machined to fit loosely into perforated stainless steel capsules. These "capsule" specimens were exposed to high temperature water until the corrosion process caused swelling and rupture.

Results of these experiments showed or verified that:

1. The swelling and rupture processes are rapid but not explosive.
2. Swelling and rupture result from pressure of the corrosion products.
3. The position of the pin hole is not a major factor in determining the corrosion rate, and the location of rupture is not limited to the pin hole area.

4. Further evidence is given for the postulate(2) that the mechanism of crevice corrosion is a two-step process involving uranium hydride. This is effective only when the uranium is enclosed in such a way as to trap part of the hydrogen evolved. Uranium hydride has been identified as a corrosion product in capsule tests containing unalloyed uranium in water at 500°F, as was previously reported at lower temperatures.(3)

5. The corrosion resistance of either uranium - 3% niobium alloy or uranium - 1-1/2% niobium - 5% zirconium alloy was superior to that of unalloyed uranium.

Sample Description

Each sample consisted of a cylinder of uranium or uranium alloy, 3/16 in. diameter by 3/4 in. long, sealed in an AISI type 304 stainless steel capsule, 1/4 in. diameter and 1-5/8 in. long. A 1/32-in. pinhole was drilled in the side wall of the capsule; the position of the pinhole relative to the sample was varied. When assembled, there was about 1/8 in. void space above the uranium cylinder in the capsule and about 3 mils clearance on the side between the uranium and the inside wall of the capsule.

Equipment

All samples were tested in the thermal syphon leg of a large autoclave. The syphon leg was equipped with a sight glass which permitted visual observation and study by time-lapse motion picture techniques. Several uranium alloys which exhibited high corrosion resistance were removed for further long-time testing in standard small autoclaves.

Experimental Conditions

All tests were carried out in neutral, high purity water.

In the sight glass, the maximum steady-state temperature was 460F. The flow rate past the sample was estimated to be about 10 fps. Analyses showed that the water contained from 4-6 ml dissolved oxygen per liter at STP.

Tests in the standard small autoclaves were run in 500F water containing about 1 ml dissolved oxygen per liter.

Experiments Performed

A number of experiments were performed using the "capsule" type sample described above. Several of the more interesting are discussed below:

Experiment	Conditions
1.	Normal uranium, alpha annealed. Hole near top of sample.
2.	Normal uranium, alpha annealed. Hole near bottom of sample.
3.	Uranium-3% niobium, quenched from 1470F. Hole near center of sample.
4.	Uranium-3% niobium, as-rolled. Hole near center of sample.
5.	Uranium 1-1/2% niobium - 5% zirconium, quenched from 1470F. Hole near center of sample.

Discussion

Experiments 1 and 2

The first two experiments were run on normal uranium in order to obtain reference data for comparison with alloyed specimens. The position of the hole was varied in order to determine whether it was a factor in determining corrosion rates.

The test results are shown below:

Elapsed Time, min.	Experiment 1 (Hole near Top)		Experiment 2 (Hole near Bottom)	
	Temp., F	Comments	Temp., F	Comments
0	70		70	
15			100	
25			200	Bubbles evolving from pinhole slowly.
45			300	Rapid bubbling.
50	125	Bubbles evolving from pinhole slowly.		
75	170	Bubble rate increasing.		
85	280	Rapid bubbling.		
90	320	Bubbling stopped.		
115			350	Rapid bubbling.
145	360	Bubbling stopped.	400	Bubbling stopped.
160			425	Sample started to swell above hole.
165	400		450	

Elapsed Time, min.	Experiment 1 (Hole near Top)		Experiment 2 (Hole near Bottom)	
	Temp., F	Comments	Temp., F	Comments
180	420	Sample started to swell below hole.		
185			450	Sample split above hole; rapid evolution of gas bubbles.
190	440			
195	450	Sample split below hole; rapid evolution of gas bubbles.		
205			450	Split approx. 1/2 in. long and 1/8 in. wide.
215	450	Split approx. 1/2 in. long and 1/8 in. wide.		

Both samples split after approximately the same exposure times (taking into consideration the rates of heating, etc.). Therefore, it was concluded that the position of the hole was not a major factor in determing the corrosion rate.

In the experiments with normal uranium, there were three fairly distinct phases of gas evolution from the capsule. During the first phase, bubbles were evolved rapidly from the pinhole:

$$U + 2H_2O \longrightarrow UO_2 + 4H_2 \qquad (1)$$

During the second phase, the bubbling slowed down or ceased. This phase of the experiment usually lasted about 1/2 to 1 hr, and was followed by a rapid swelling and rupture (see above table). The third phase, immediately following the rupture, involved a rapid evolution of bubbles.

The corrosion of uranium under the conditions of this test apparently involves more than a simple, one-step reaction. A portion of the uranium corroded is first converted by the hydrogen evolved from the reaction shown in Equation (1) into the hydride:(3)

$$U + \frac{3}{2}H_2 \longrightarrow UH_3 \qquad (2)$$

Uranium hydride has been identified as a corrosion product in corroded capsules which contained normal uranium. The hydride reacts readily with water by Equation (3) to form UO_2 and liberate hydrogen:

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$$UH_3 + 2H_2O \rightarrow UO_2 + \frac{7}{2} H_2 \qquad (3)$$

The extent of the conversion shown in Equation (2) and the role of UH_3 in uranium corrosion is apparently a function of the access of water to the uranium. When uranium is enclosed so that water has only limited access, and evolved hydrogen may be trapped and exert an appreciable partial pressure on the uncorroded metal, hydride formation is postulated(2) to be an important factor in the corrosion rate. In open contact with high temperature water, the corrosion of uranium is apparently much less complicated by hydride formation. It is to be noted that with a loose fit, such as in these experiments, hydride swelling did not appear below the boiling point. Further, in the corrosion of unclad uranium in steam at 180°C, more hydride was found than when uranium was corroded in liquid water at the same temperature. Therefore it appears that, to form hydride, more isolation from the water is required the lower the temperature.

Experiments 3, 4, and 5

Experiments 3, 4, and 5 were conducted to determine the effect of alloy addition and heat treatment on the corrosion of uranium.

A uranium alloy containing 3% niobium was tested in the quenched condition (Experiment 3) and in the as-rolled condition (Experiment 4). The alloy was quenched from 1700°F; rolling was done at 1550°F. The pin holes were near the center of both samples.

Test results for the quenched alloy are shown below:

Time of Test, hr.	Temp., °F	Comments
56	460	Tested in sight glass. No apparent corrosion.
97	500	Tested in autoclave. No apparent corrosion.
137	500	No apparent corrosion.
88	500	Slight swelling above hole.
66	500	Jagged split through hole extending about 1/4 in. upward and 1/4 in. downward with a slight spiralling. Approximate width of split: 1/8 in.
444 Total		

The as-rolled sample ruptured in about 5 hr. at 460F. Swelling was noticeable about 20 minutes before the rupture occurred. Thus, it is seen that proper heat treatment is an important factor in achieving maximum corrosion resistance for the uranium-3% niobium alloy.

In Experiment 5, a uranium alloy containing 1-1/2% niobium and 5% zirconium was tested after quenching from 1470F.

The results of the tests were as follows:

Time of Test, hr.	Temp., °F	Comments
28	460	No apparent corrosion.
163	500	Sample split.

This alloy exhibited corrosion resistance superior to normal uranium, but inferior to quenched uranium-3% niobium alloy.

Other experiments showed that capsuled samples of the non-heat treated uranium 1-1/2% niobium - 5% zirconium failed in less than 7 hours.

BONDED PLATE: PIN-HOLE EXPERIMENTS

Sample Description

Several small roll bonded plates which were welded on the sides were corrosion tested after the protective clad had been pierced to produce a known defect. The results of these tests are discussed below.

Equipment

High pressure autoclaves were used for these tests.

Discussion and Experimental

Some of the long-time water corrosion tests of uranium or uranium alloys clad in zirconium or Zircaloy have used the pin-hole technique,(1) where a hole approximately 1/32 in. in diameter is drilled through the bonded clad protective layer, thereby exposing the uranium to the action of water at temperatures of 500F or above.

In general, corrosion effects were not limited to the immediate neighborhood of the drilled hole. Blisters were quite common on the surface of the plates and occasionally a rupture was found in the cladding, almost always along an edge. By the use of the helium mass-spectrometer type leak detector, the presence of passages in the plate between breaks in the zirconium was confirmed.

Figures 1 and 2 show one such specimen with the cladding completely machined from one face. The dimensions of this specimen are about 2 x 1/2 x 1/8 in. It is to be noted that the hole was not greatly enlarged by

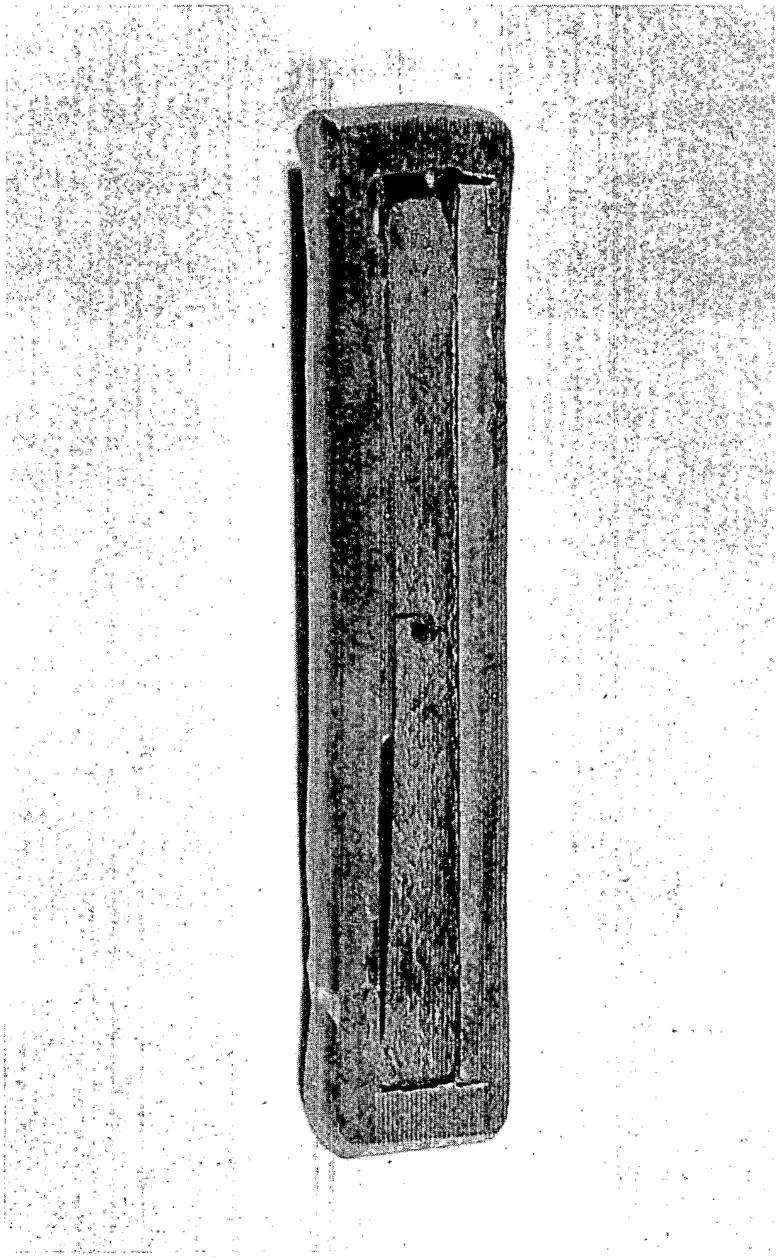


Figure 1. 3% Nb-U Alloy, Zirconium-Clad With Small Hole in Cladding, Corroded in Water, With Top Cladding Removed.

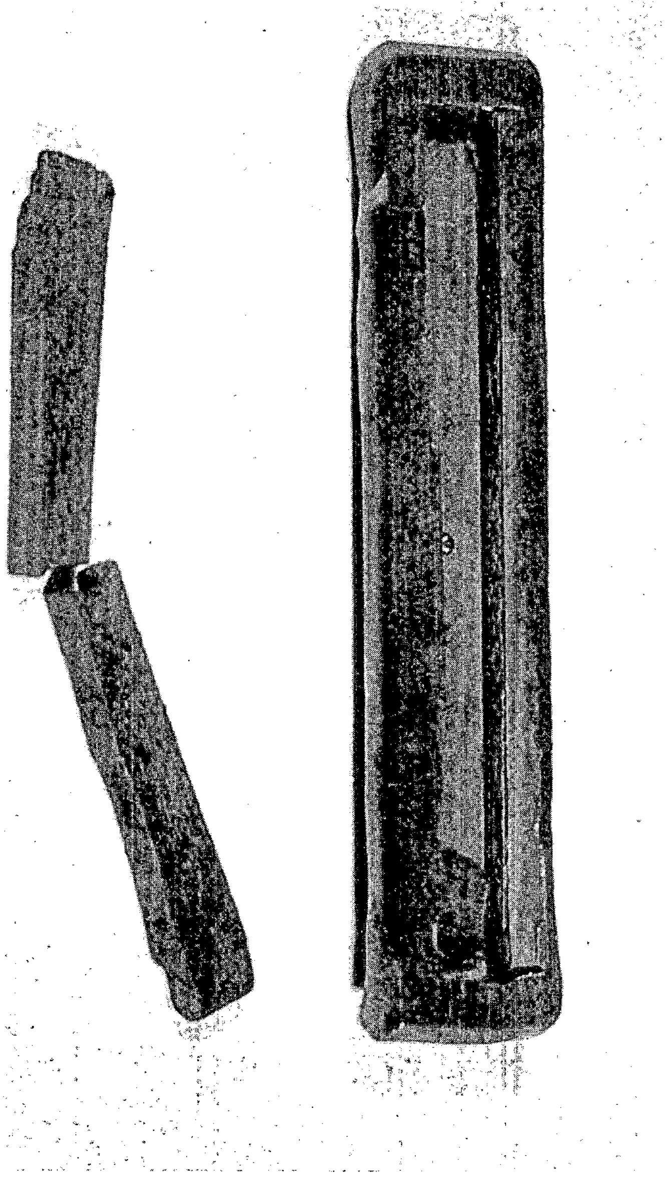


Figure 2. 3% Nb-U Alloy, Zirconium-Clad With Small Hole in Cladding, Corroded in Water, With Top Cladding Removed. (Note that Core is Completely Unbonded)

corrosion and that there was no bond between the clad and base metal at the time of machining. The core alloy of the plate was completely covered by a loose oxide coating. Figure 3 shows a large size plate (approximately 1/8 x 1-3/4 x 7 in.) that had an initial closure defect. After subsequent corrosion test, many blisters and breaks occurred, one of which may be seen near the center of the upper edge. Again all the defects had common communicating passages in the plate. Cladding was removed from one face in two steps. As with the smaller plate, the entire core surface was covered by a powdery oxide and adherence was limited to a few widely scattered points.

This behavior closely parallels that observed by others(1,5,6,7,8) in the corrosion of aluminum-clad uranium specimens.

Artificial Crevice Specimen, Sleeve-Cap:

Water at 260°C, the temperature of then current interest, is capable of dissolving relatively large amounts of hydrogen under moderate pressures.(9) The following experiment was designed to differentiate between the effects of gaseous hydrogen and hydrogen in solution on the water corrosion of uranium. Essentially this is a high temperature extension of the work of Hoxeng and Rebol.(3) A strong cap of zirconium was fitted closely over a uranium cylinder so as to leave about half of the uranium exposed to the water. The cap was designed to be strong so that the expanding corrosion products would be compressed tightly. Since the presence of water is incompatible with hydride formation,(1) a hydrogen reaction would imply that it was under a total pressure of about 600 psi. It would further imply that the water in the immediate neighborhood would contain 200 cc of hydrogen per kg. At this temperature the equilibrium pressure of hydrogen over UH_3 is approximately 0.15 psi.(10)

Uranium specimens and zirconium caps were made according to Figure 4. In test, the zirconium cap was placed over the longer end of the uranium specimen, and the assembly placed into an autoclave of 320 ml capacity, with the capped end up. About 105 ml of distilled water was added on the assumption that 100 ml would remain after corrosion was complete. In some cases other materials were added to the water. A special autoclave cover was made with a 1/4 in. steel tube welded into it. This communicated with a 3000 psi gauge. Both gauge and tube were filled with distilled water prior to assembly, to prevent distillation of liquid from the autoclave during test. The autoclave was put into a heater and heated rapidly to 260°C. This required slightly less than an hour, on the average. Temperature was maintained for a predetermined period, 1/2, 1, 1-1/2 hours or longer; then the sample was taken out of the heater and cooled rapidly (10 minutes or less) to room temperature. Temperatures and pressures were read at frequent intervals, as well as the final pressure after cooling. This final pressure was assumed to be exerted by uncombined hydrogen.

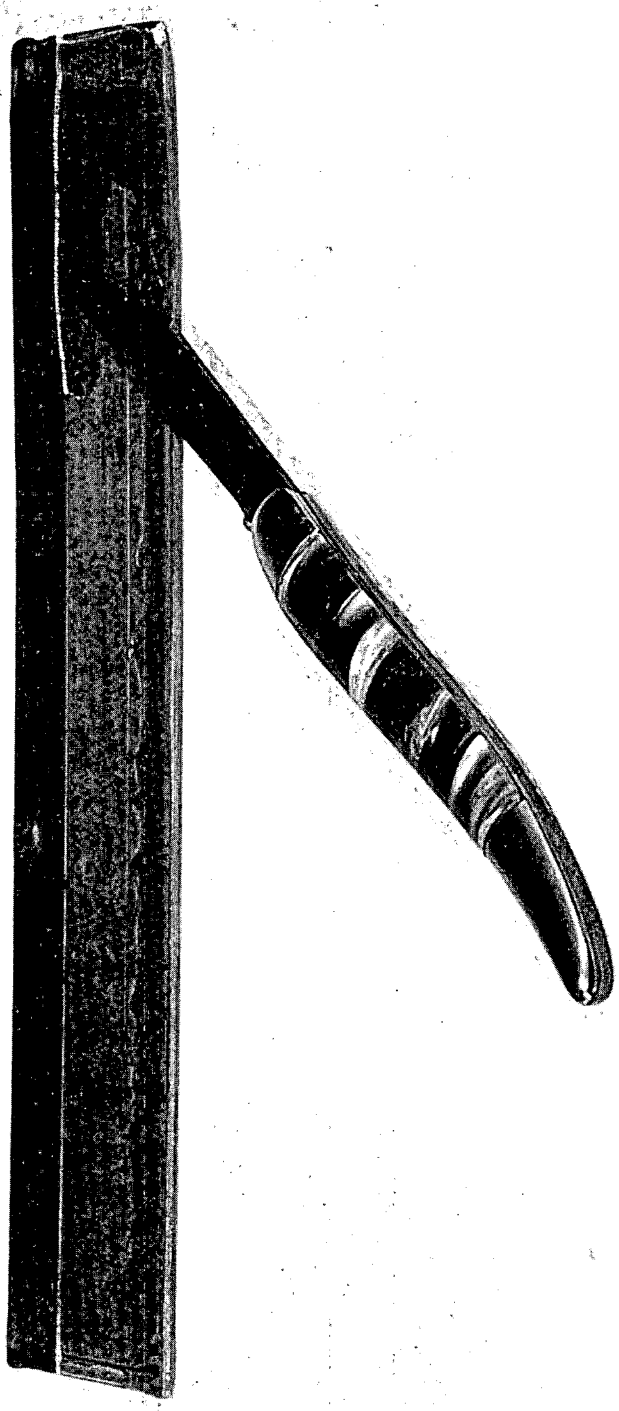
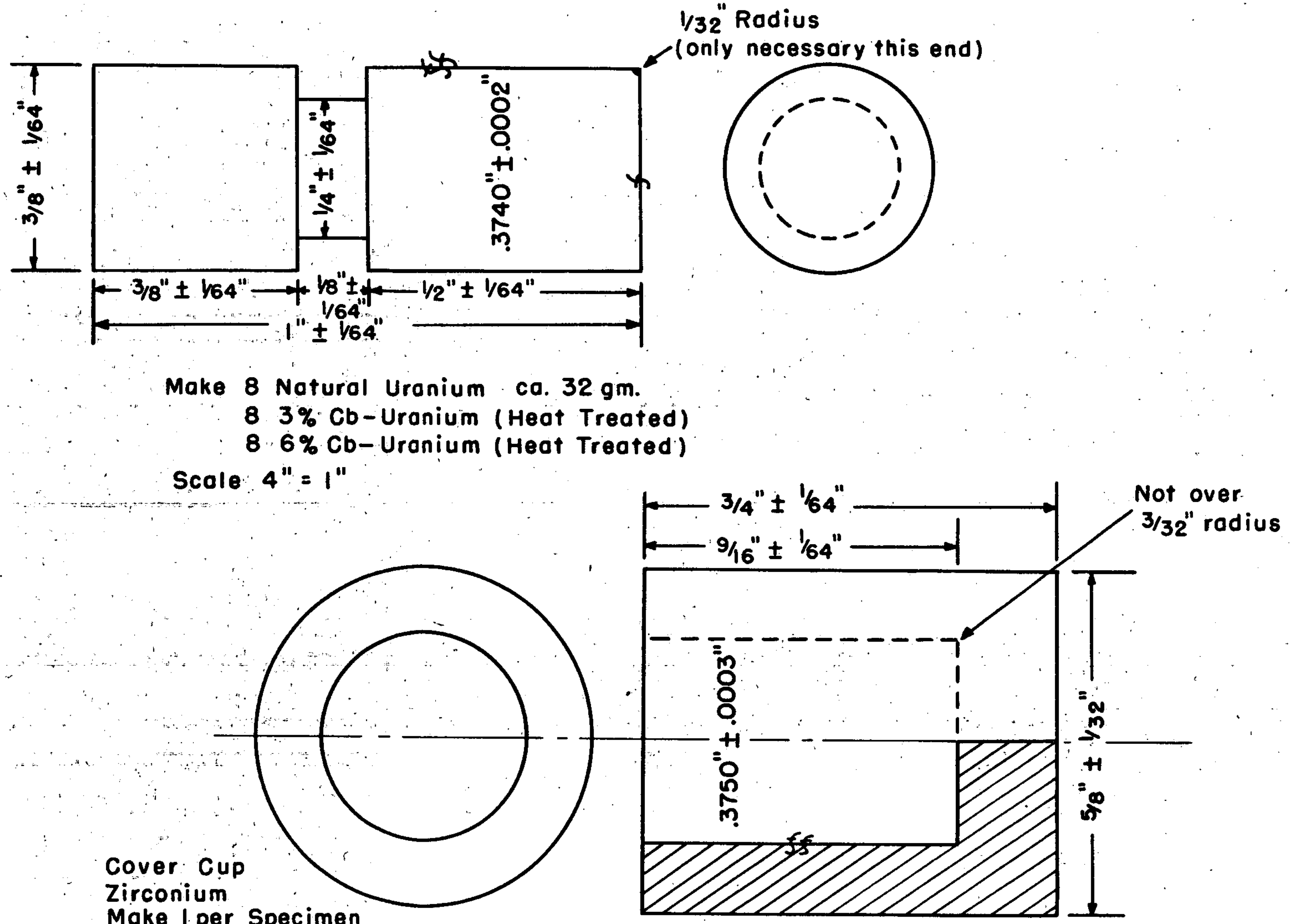


Figure 3. Corroded Fuel Plate, With Top Cladding Removed in Two Steps. (Note Bond Failure)


1/32" Radius
(only necessary this end)
3/8" ± 1/64"
1/4" ± 1/64"
.3740" ± .0002"
3/8" ± 1/64"
1/8" ± 1/64"
1/2" ± 1/64"
1" ± 1/64"
ff
f
3/4" ± 1/64"
9/16" ± 1/64"
Not over 3/32" radius
.3750" ± .0003"
5/8" ± 1/32"
ff

Make 8 Natural Uranium ca. 32 gm.
8 3% Cb-Uranium (Heat Treated)
8 6% Cb-Uranium (Heat Treated)
Scale 4" = 1"

Cover Cup
Zirconium
Make 1 per Specimen
Scale 4" = 1"

FIGURE 4.

URANIUM OR URANIUM ALLOY SPECIMENS.

Figures 5 to 9 show time pressure curves for two 1/2-hour periods, a 1-hour period, a 2-hour and a 3-1/2-hour period at temperature. It is to be noted that pressures are about those to be expected with water at 260°C, until about 1-1/2 hours at temperature have elapsed, when the pressure rises sharply. In the long time test the pressure oscillated at a higher level, due to water temperature changes, after the sharp rise at 1-1/2 hours. It is also to be noted that final pressures were approximately 50 psi for experiments that did not show the sudden pressure increase and greater than 250 psi after such rise occurred.

Figure 10 shows the appearance of two zirconium cups after pressure release (right and left) and a dissembled uranium specimen and cap prior to test (center). Figure 11 shows an assembled specimen before testing, and three specimens where the testing was interrupted prior to pressure release. The bulging of the cap can be seen in all tested specimens. At the right of Figure 10, a bulge can be seen on the bottom of a cap.

Figure 12 shows the uranium specimens after the postulated hydrogen release. It is to be noted that the enclosed end is much more severely corroded than the end directly exposed to water. All pictures show a series of cavities rather deeply etched into the exposed end, characteristic of the corrosion of rolled uranium.

A mechanism of corrosion can be written:

$$4\ H^+ + U \longrightarrow U^{4+} + 4\ H \quad .$$

However, in the absence of a strong acid the oxide UO_2 would be formed as soon as the solubility product was exceeded. This would imply that uranium corrosion might be sensitive to changes in pH at the high end of the range and might be deterred by high alkalinity. At 260°C, the ionic dissociation constant of water is about 2×10^{-11} as compared to 10^{-14} at room temperature.(11) Changes in pH with temperatures are shown in Figure 13, as derived from Dorseys' data.

Under these considerations, a sample was tested in N/100 LiOH which had a pH of 11.8 at room temperature. This should become pH 9.1 at 260°C. The specimen was held at 260°C, within apparatus limitations, for 140 minutes. As shown in Figure 14, pressure was suddenly released after about 100 minutes, and the pressure was 330 psi when cold.

~~Although N/100 would seem to be the greatest practical amount of alkalinity for potential reactor use~~, a series of tests was made using 3 grams of LiOH added as a solid to the usual 105 ml of water used in an experiment. Periods of tests were 1/2 hour, 1 and 2 hours at temperature. Bulging of the can was less than usual in the two shorter periods, and the uranium suffered less than usual amount of attack during the two-hour

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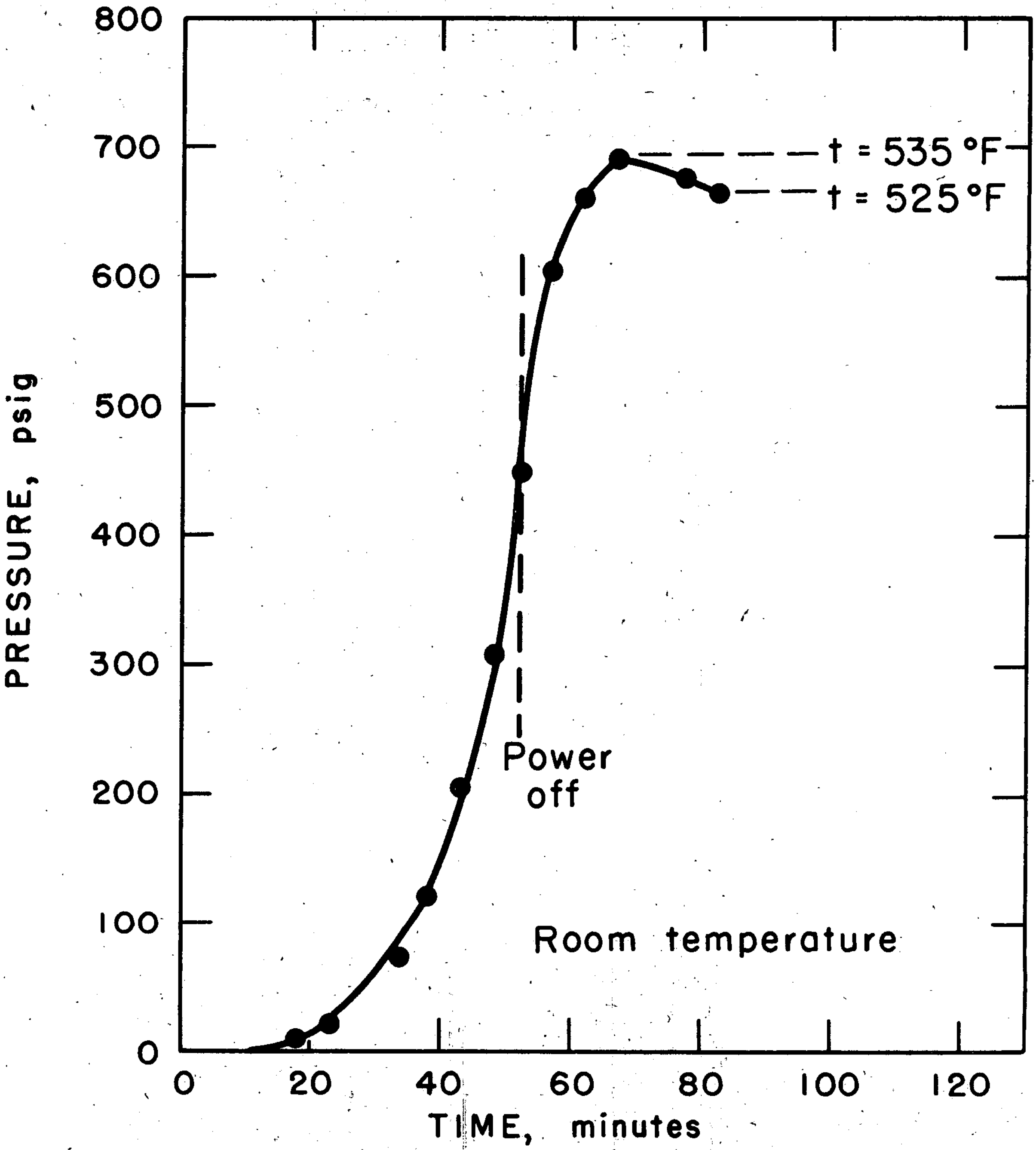

FIGURE 5.

Run 3. NORMAL URANIUM CORROSION.
~ 1/2 HOUR IN 500 °F WATER.

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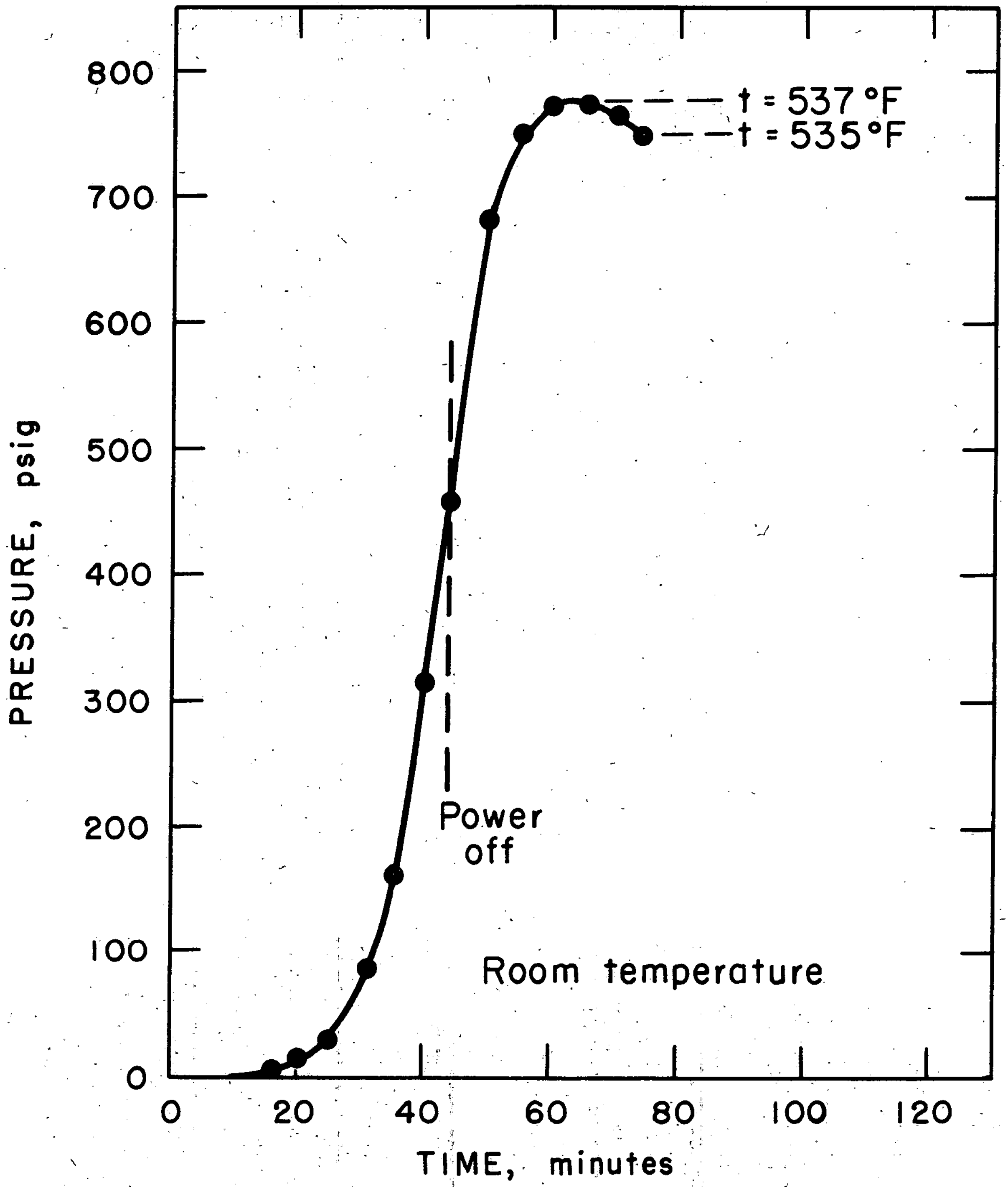

FIGURE 6.

Run 8. NORMAL URANIUM CORROSION.
~ 1/2 HOUR IN 500 °F WATER.

17

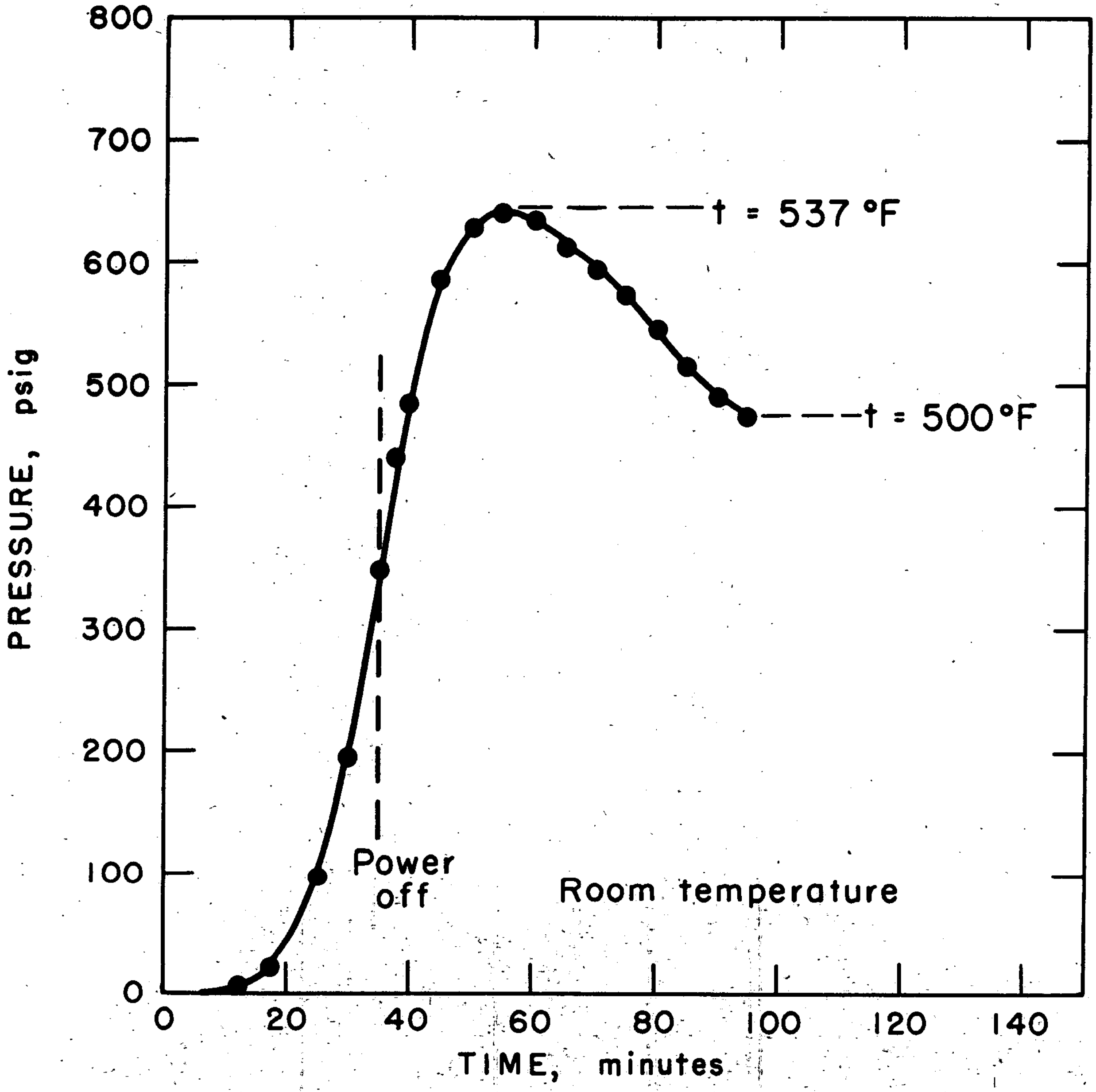

FIGURE 7.

Run 9. NORMAL URANIUM CORROSION.
~1 HOUR IN 500°F WATER.

18

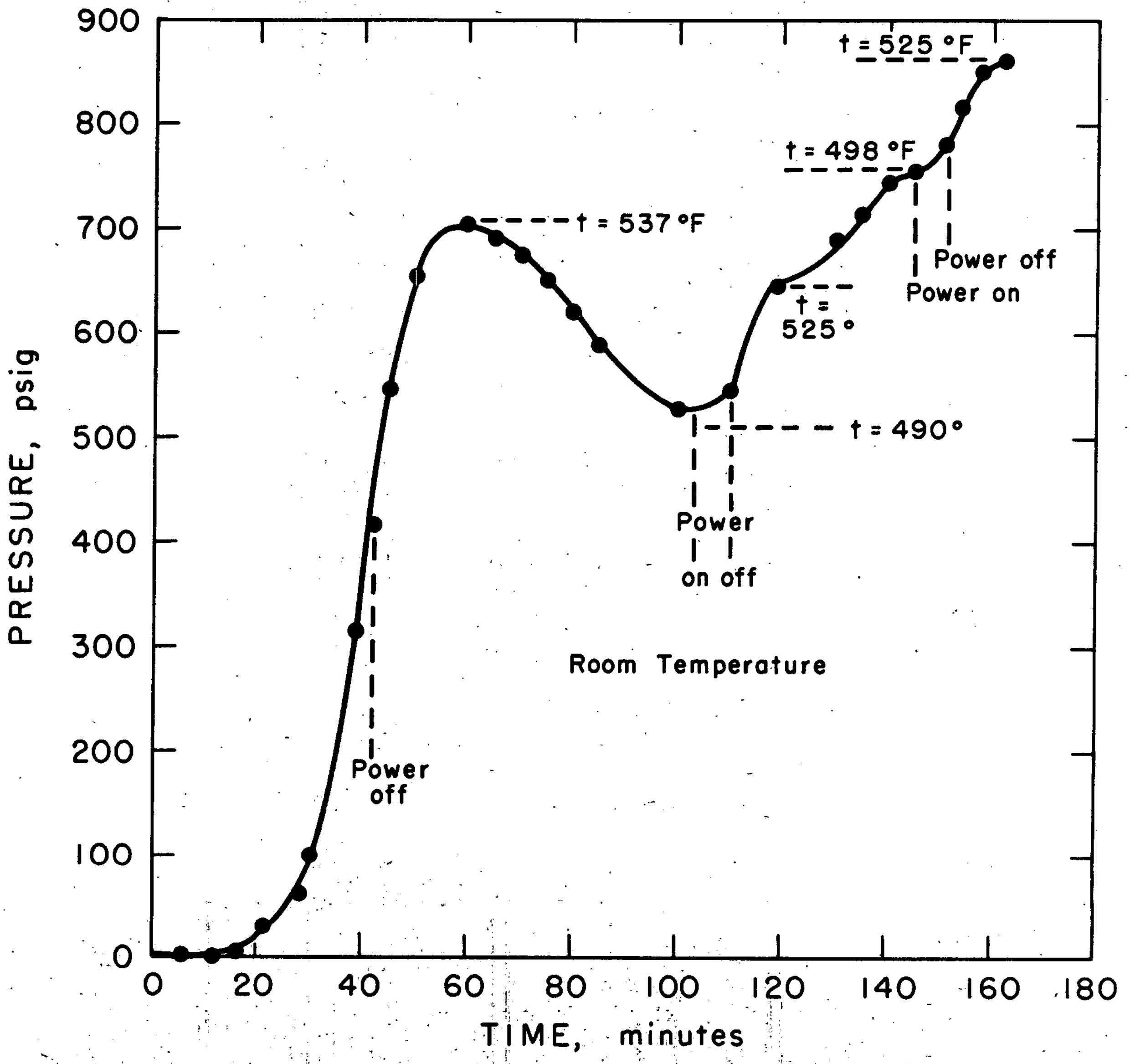

FIGURE 8.

Run 10. NORMAL URANIUM CORROSION.
DISTILLED WATER. ~500 °F.

19

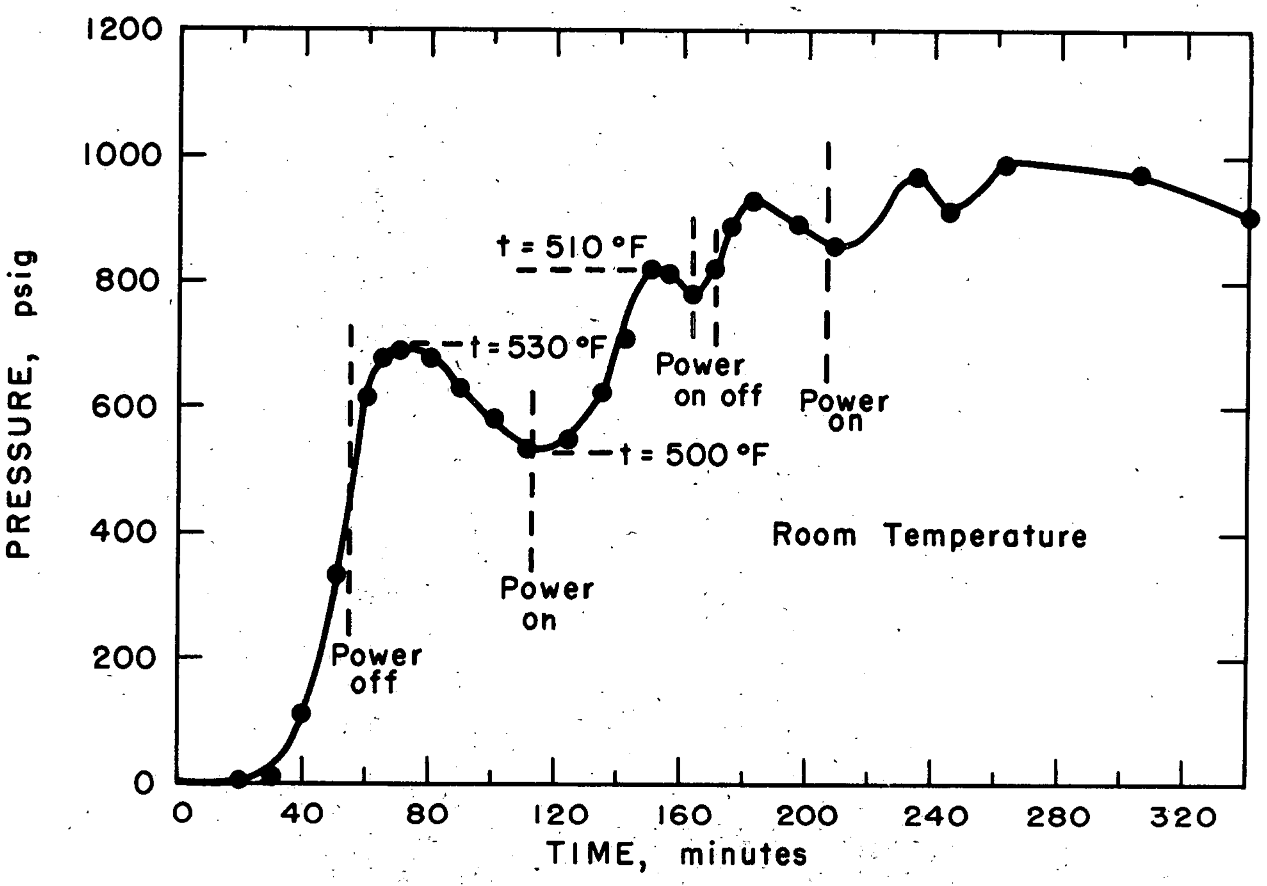

FIGURE 9.

Run I. NORMAL URANIUM CORROSION.
DISTILLED WATER AT 500 °F.

20

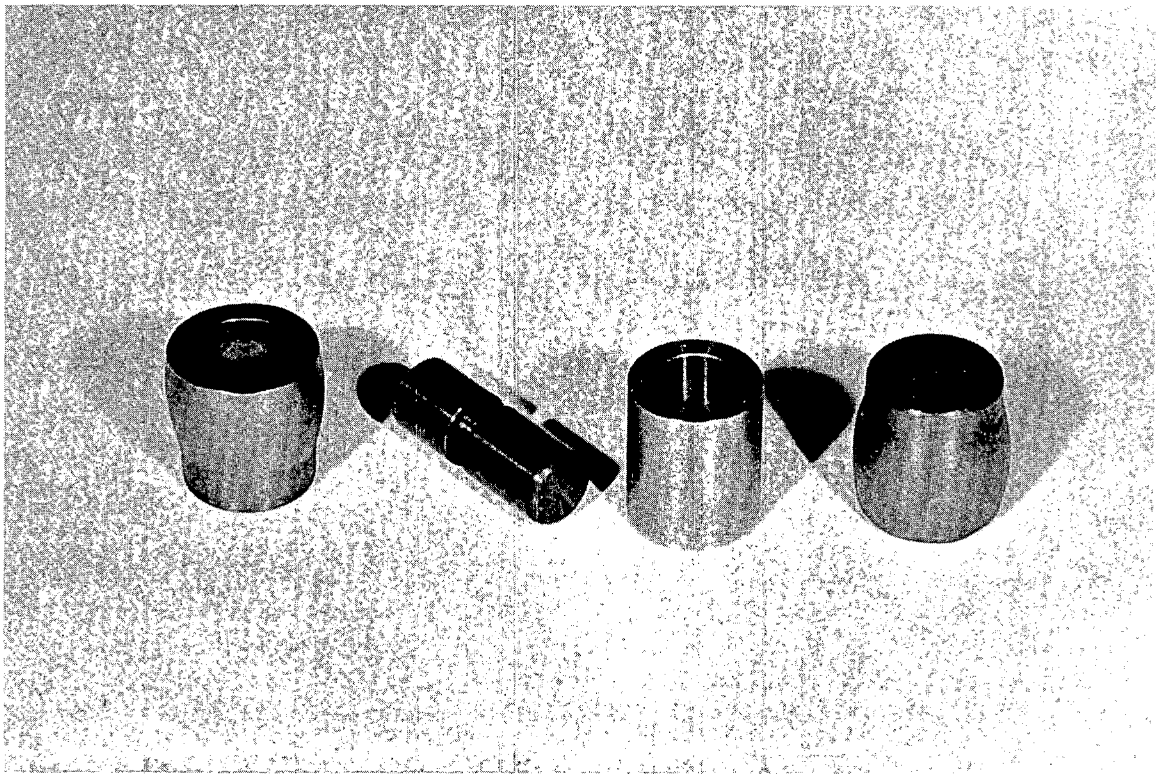

Figure 10. Zirconium Caps Distorted by Uranium Corrosion (Outside) Uranium Corrosion Specimen and Zirconium Cap before Testing.(Center)

21

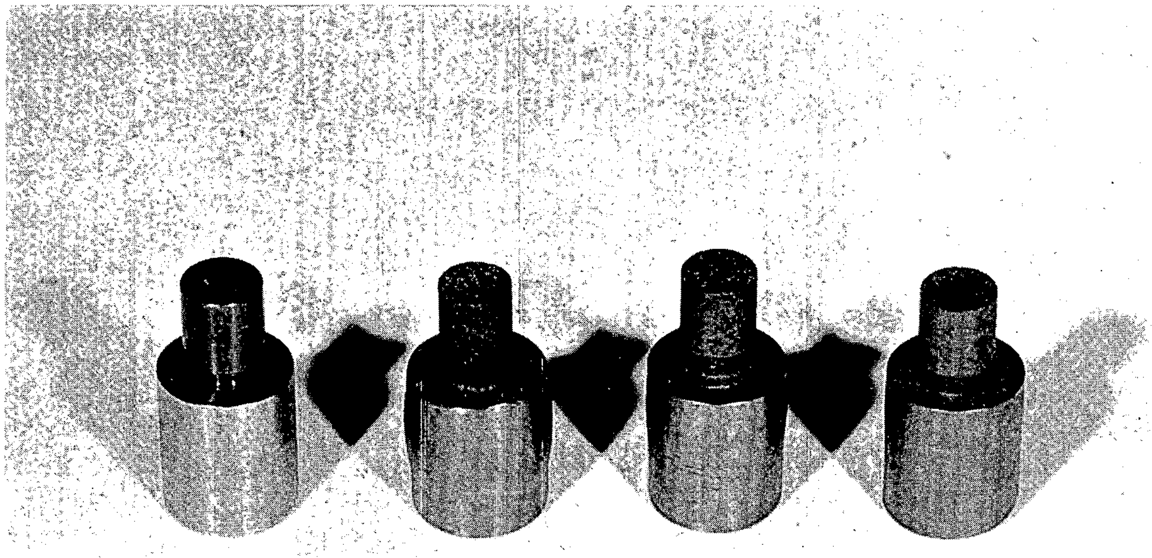

Figure 11. Three Zirconium Caps Distorted by Uranium Corrosion Interrupted Prior to Pressure Release and One Assembled Specimen Prior to Test. (Left)

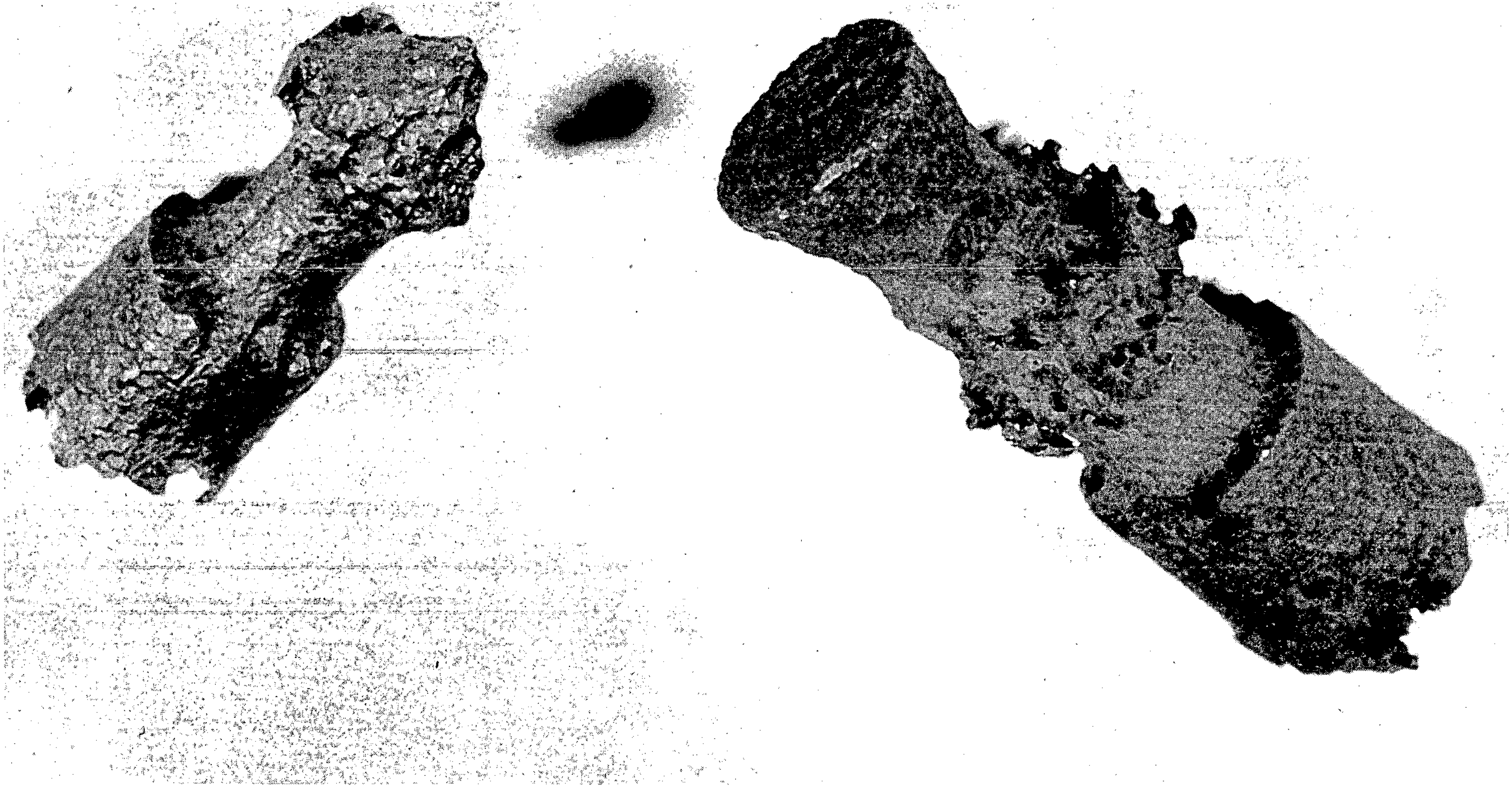

Figure 12. Uranium Specimens After Postulated Hydrogen Release.

23

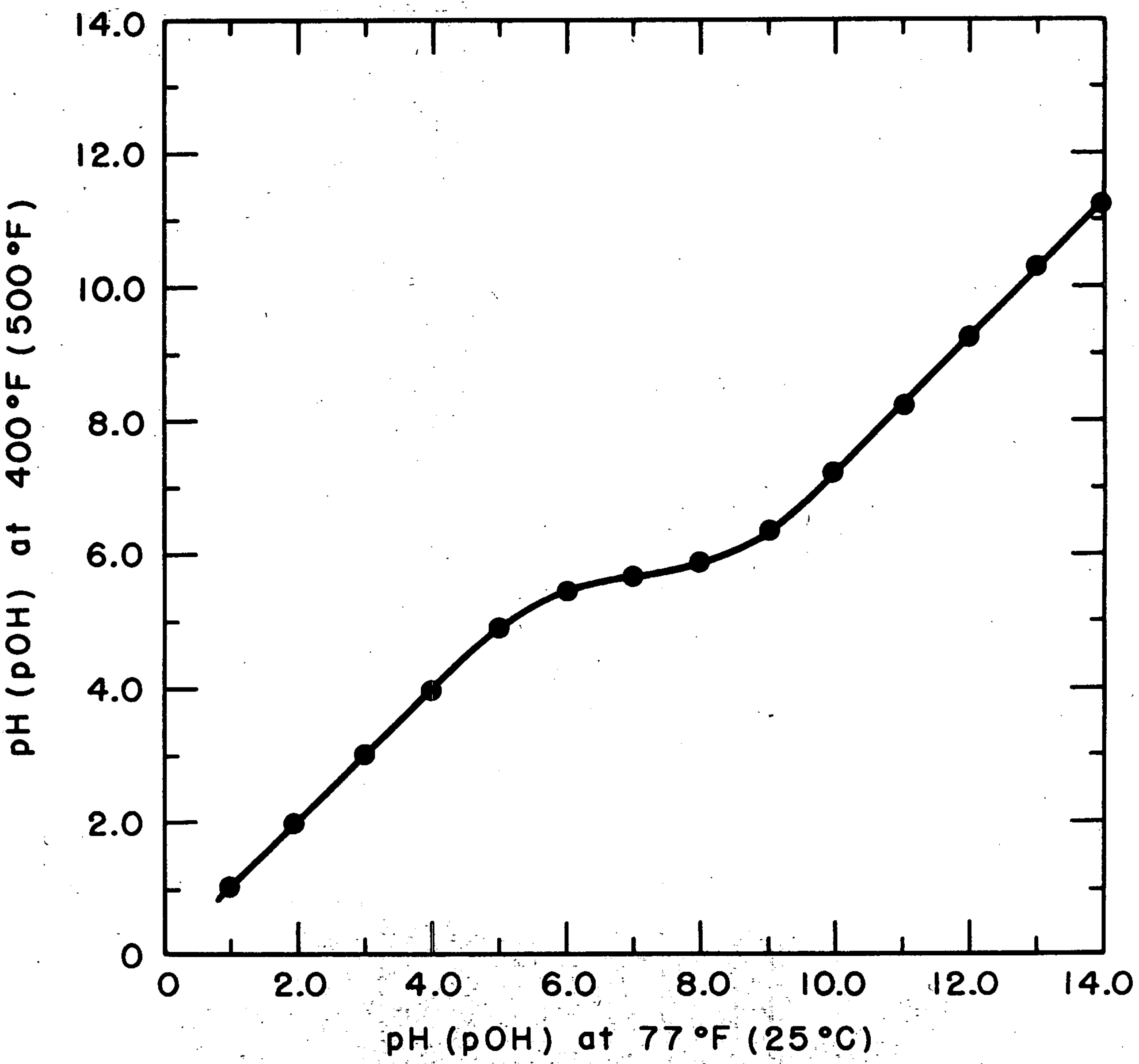

FIGURE 13.

CHANGE IN pH OR pOH FROM ROOM TEMPERATURE TO 260°C. (Also, by coincidence at 205°C)

24

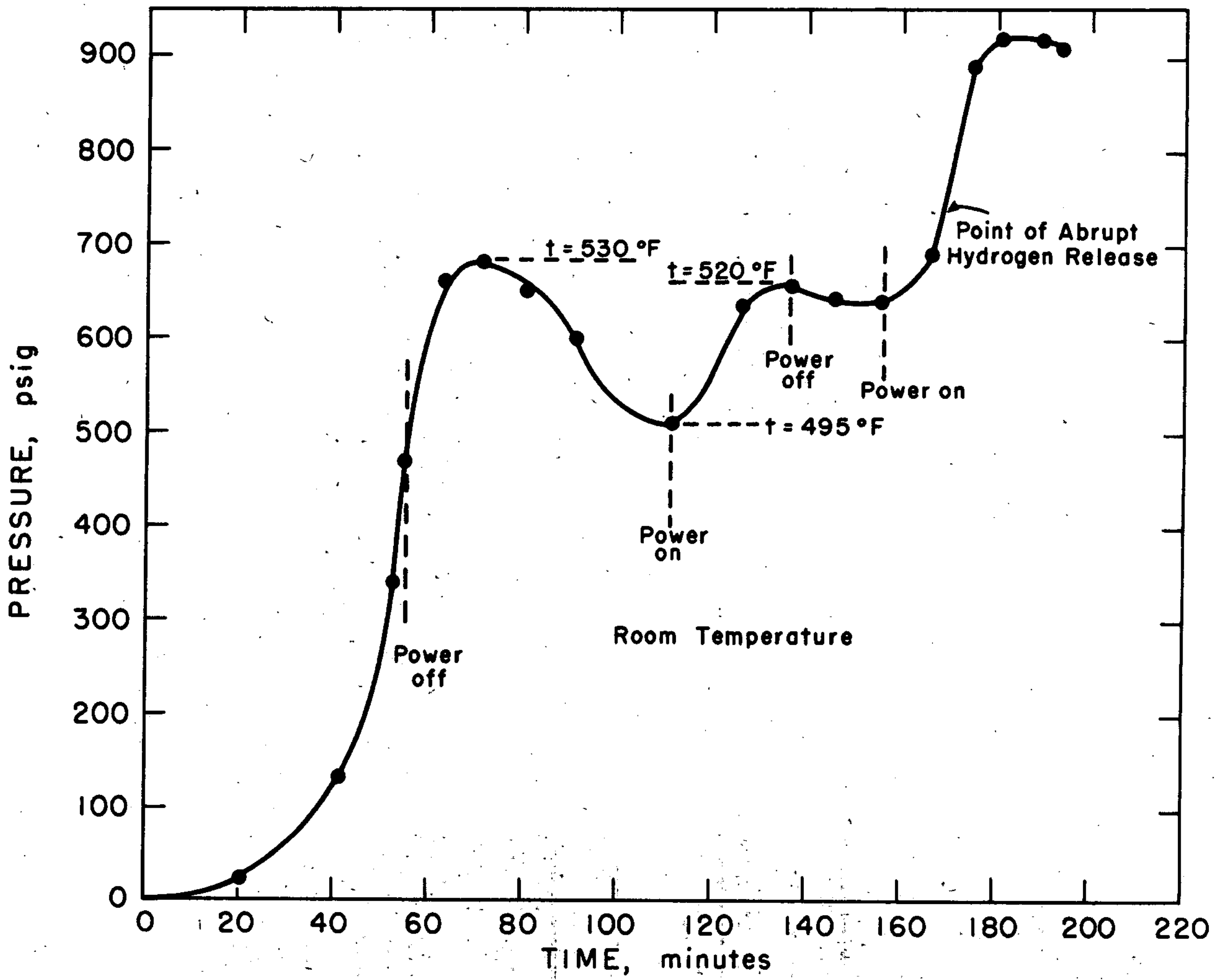

FIGURE 14.

Run 2. NORMAL URANIUM CORROSION.
N/100 LiOH SOLUTION. 500 °F.

25

corrosion test. This is the specimen shown at the right of Figure 11. Pressure-temperatures curves are shown in Figures 15, 16, and 17. Reference to the record of the two-hour test shows a relatively low pressure, which should accompany less corrosion.

A final experiment of this series was made with a stainless steel cup containing 0.65 gm of lithium metal suspended in the vapor phase, and the air imperfectly purged with welding grade argon to determine whether or not hydrogen dissolved in the water would accelerate uranium corrosion at this temperature. Figure 18 shows the results of a 1/2-hour test. The pressure was less than calculated, rather than higher as expected. However, a very small kernel of unreacted lithium was found inside of the dense oxide formed, so exact calculations cannot be made.

The sudden release of pressure can be interpreted as water gaining access to the UH_3 previously formed, as postulated by earlier experimenters.(3) Dissolved hydrogen as such plays a lesser role than gaseous hydrogen. Alkalinity seems to deter uranium corrosion, but only in large amounts is the effect noteworthy.

Gaseous Hydriding Experiments

Primarily, these experiments were made to compare the rates of reaction with hydrogen of the niobium-zirconium-uranium alloys of current interest with that of unalloyed uranium which has been studied earlier.(2,12-17) Further, with crevice corrosion of uranium or its alloys in high temperature water it seems likely that hydrogen at very high pressure can come in contact with the metal, so the effect of pressure on the reaction rate was the subject of one set of experiments.

Specimens were made according to Figure 19. They were suspended near the bottom of a quartz tube attached to a vacuum line by an iron-constantan thermocouple inserted into the hole. A cylindrical heating coil was placed outside of the quartz tube near the specimen. The temperature was raised and held at 260°, 316° and 371°C (500°, 600°, 700°F) at a vacuum of 5×10^{-5} or better. In the meantime, the system storage bulb was filled with hydrogen purified by diffusion through palladium. The storage bulb was of about 1000 ml capacity, and opened into a manifold, which connected directly to a manometer. In these experiments, the storage bulb and its manifold system were filled to a pressure of about 70 cm of mercury. After the system was pumped to a good vacuum, the pump valves were closed and the hydrogen contained in the storage system was released to the specimen. Pressure and temperature were read periodically. The initial pressure with the entire system open to the hydrogen was about 45 cm.

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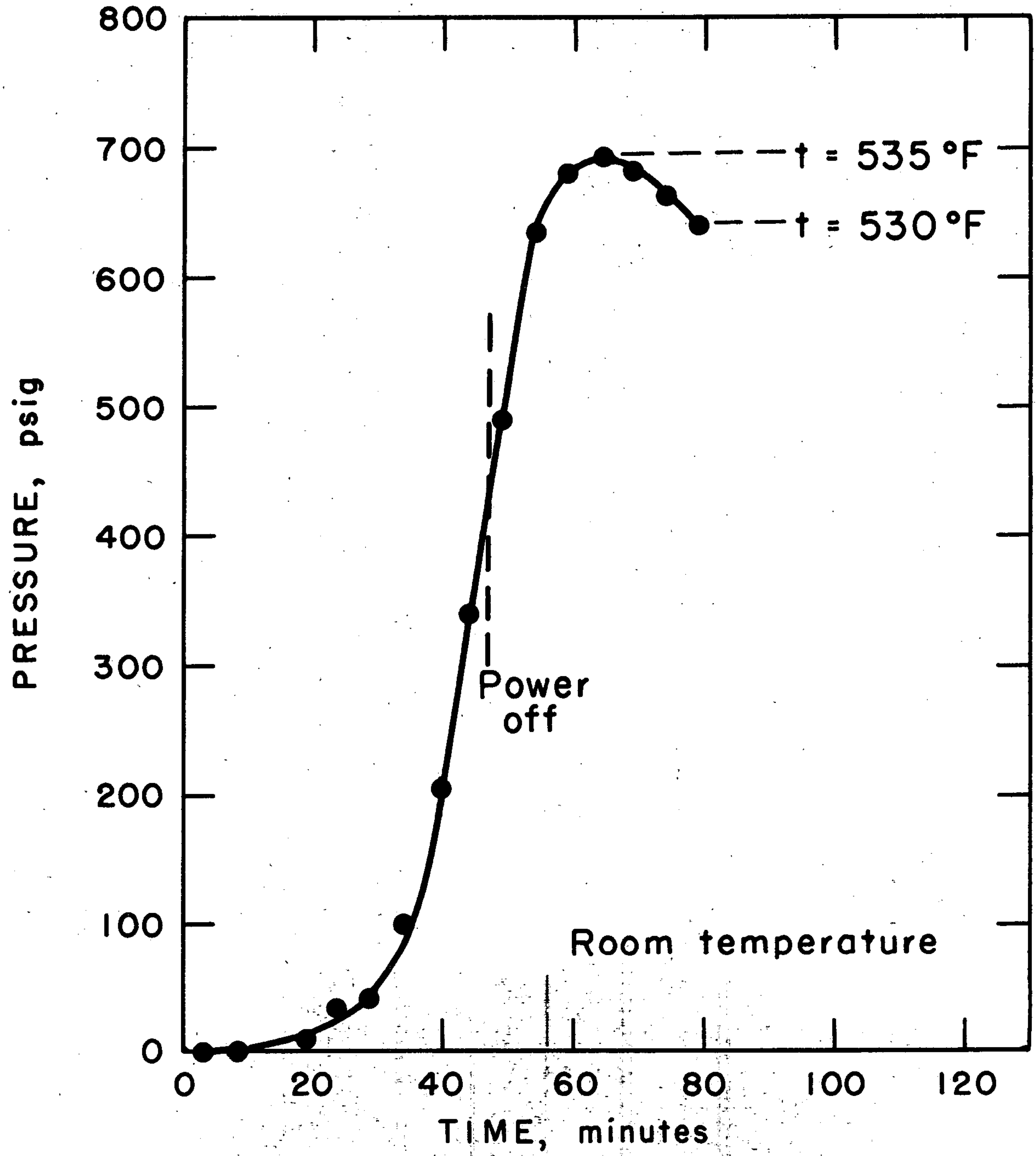


FIGURE 15.

Run 5. NORMAL URANIUM CORROSION.
3 gm LiOH IN 105 ml WATER.
~ 1/2 HOUR AT 500 °F.

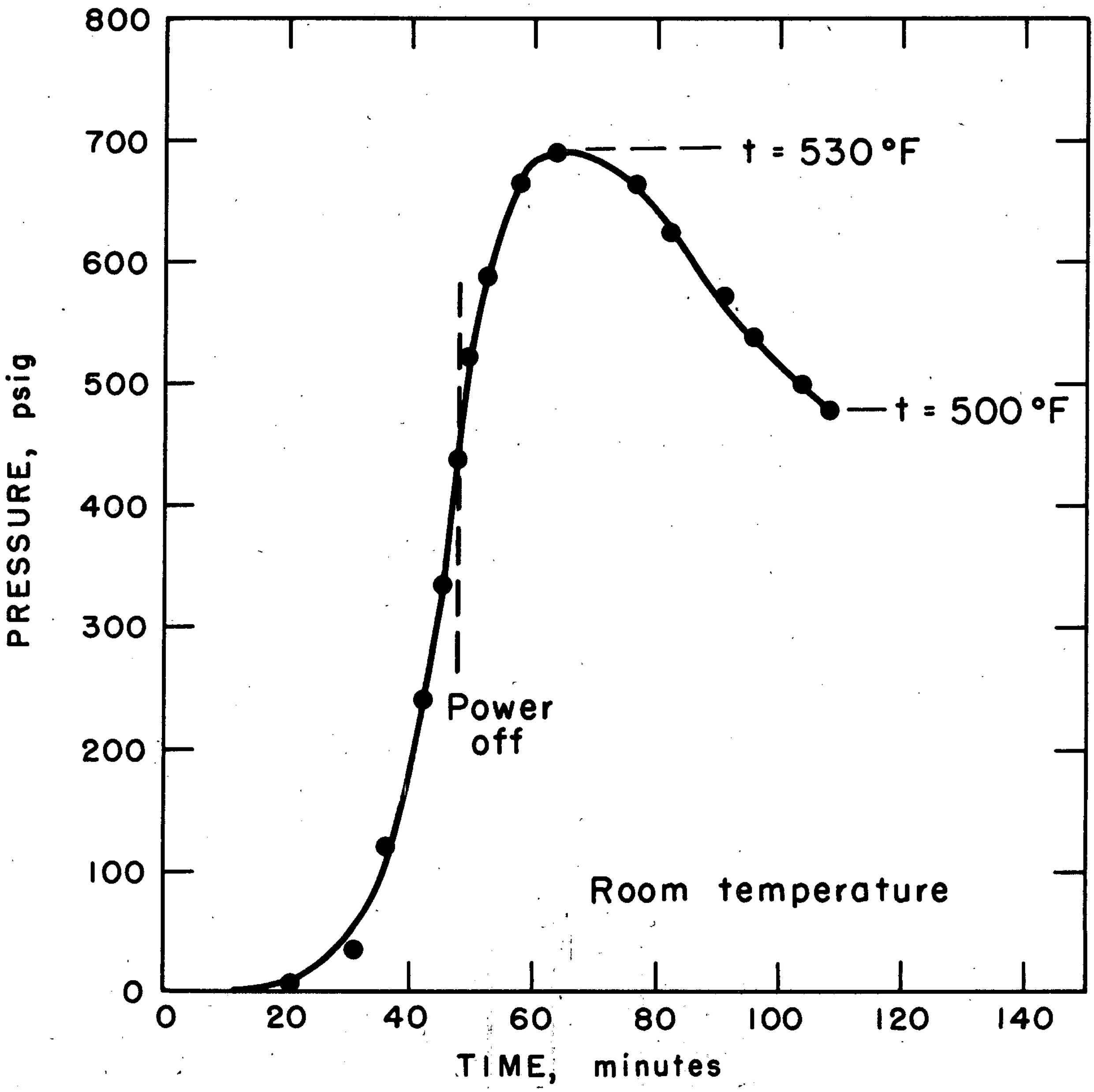


FIGURE 16.

Run 6. NORMAL URANIUM CORROSION.
3 gm LiOH IN 105 ml WATER.
~ 1 HOUR AT 500 °F.

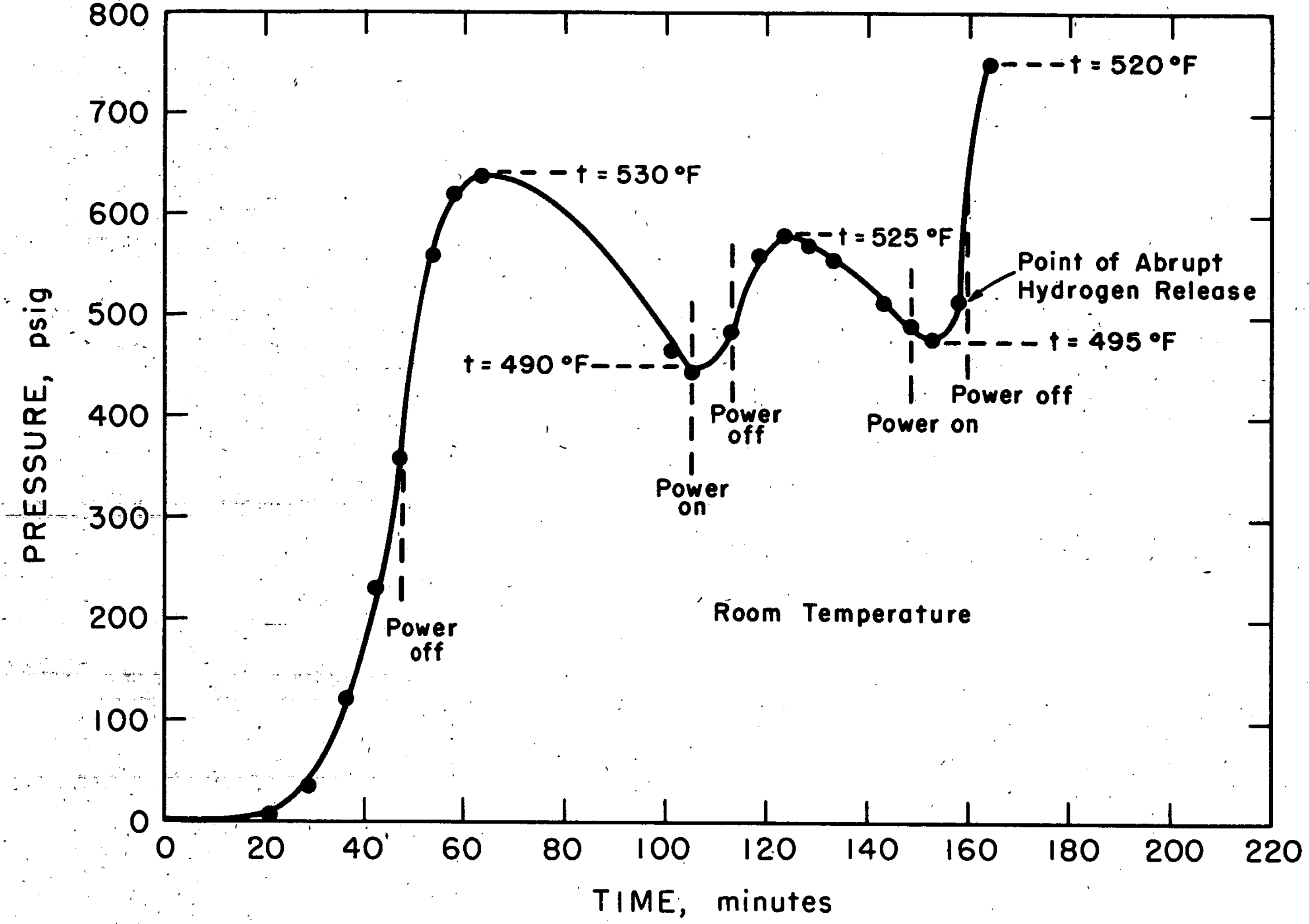


FIGURE 17.

Run 7. NORMAL URANIUM CORROSION.
3 gm LiOH IN 105 mℓ WATER.
~2 hours AT 500 °F.

29

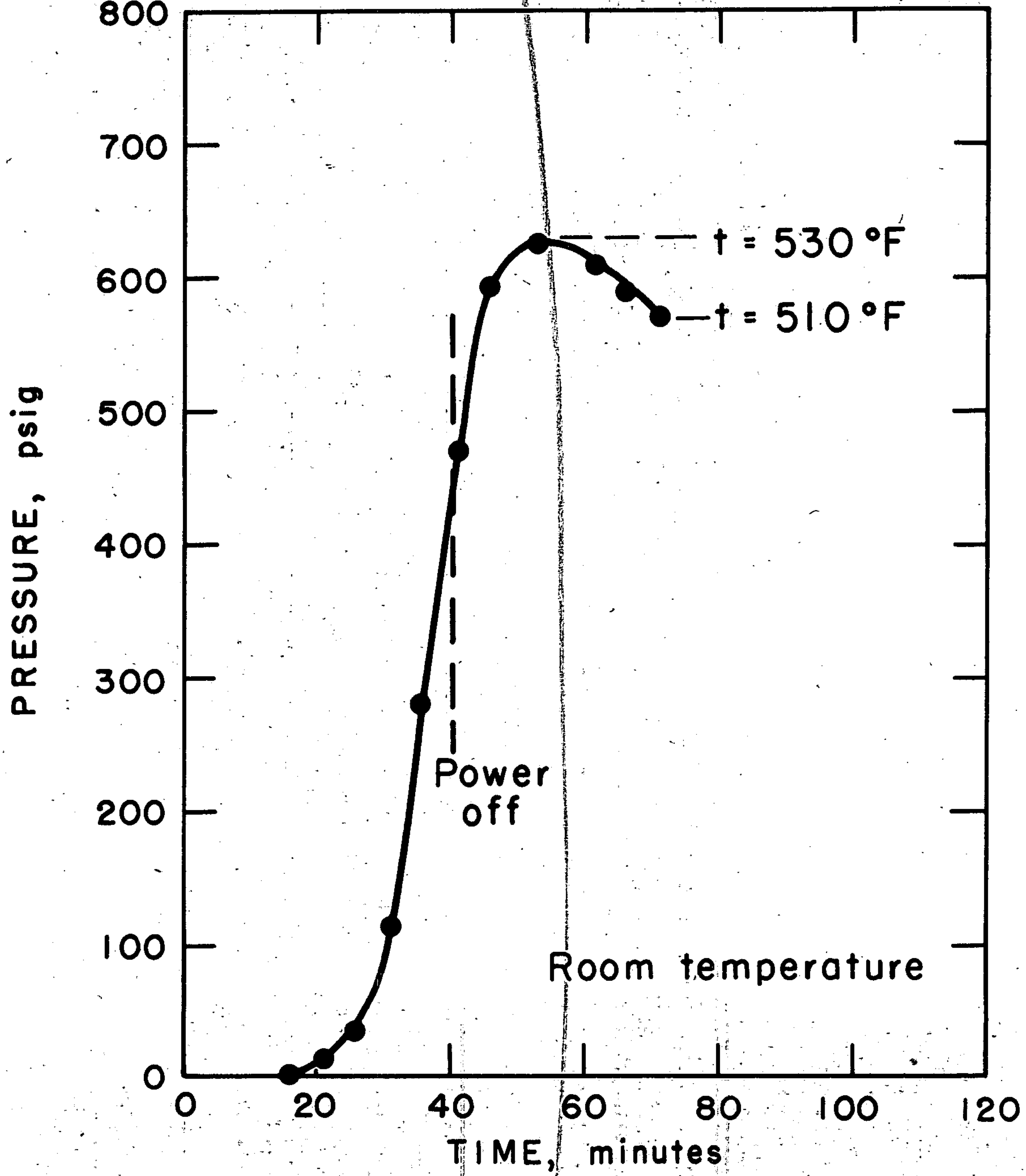
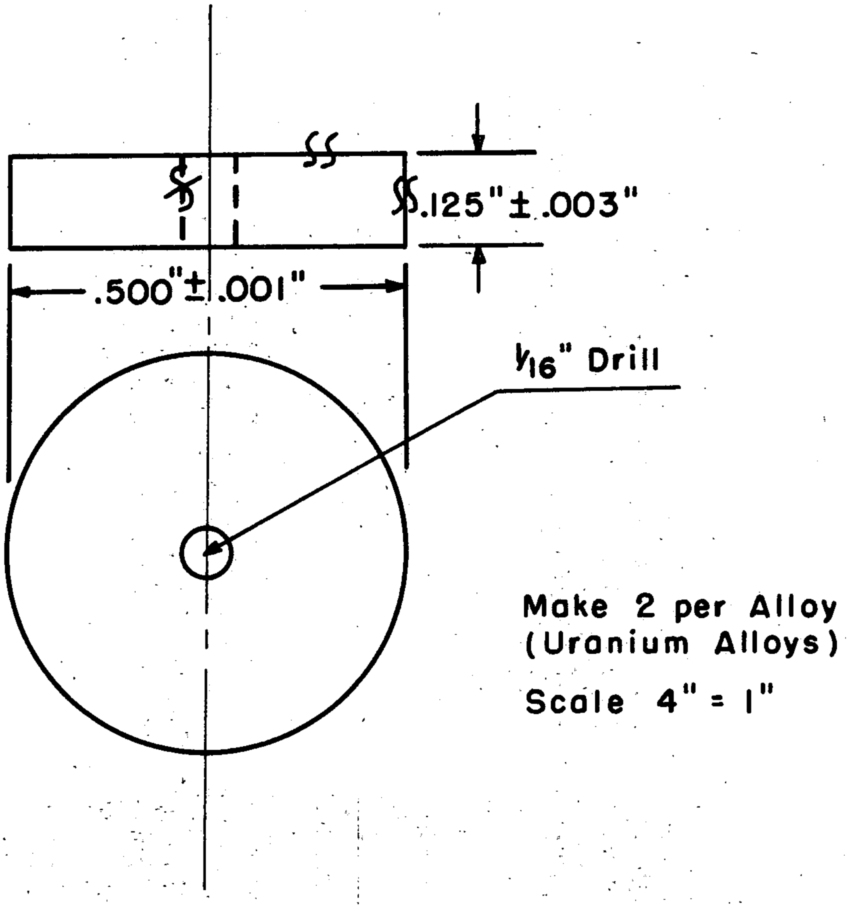


FIGURE 18.

Run 4. NORMAL URANIUM CORROSION. ARGON GAS PHASE OVER WATER. 0.65 gm Li METAL IN CUP IN GAS PHASE (to generate hydrogen).



.125" ± .003"
.500" ± .001"
1/16" Drill
Make 2 per Alloy
(Uranium Alloys)
Scale 4" = 1"

FIGURE 19.

HYDRIDING SPECIMENS.

Results of experiments on unalloyed uranium at the three temperatures are summarized in Figure 20. It is to be noted that rates of reaction are slowest at 371°C and most rapid at 260°C. This is in agreement with the work of Burke and Smith.(12)

Figure 21 shows the influence of pressure. In this set one of the experiments was made at a pressure very nearly half of the usual 45-cm pressure. In this graph, the ordinate is the ratio of pressure to original pressure. The curve indicates that doubling the pressure almost doubles the rate of reaction. This is somewhat at variance with the relation obtained by Johnson and Newton.(16)

$$-\frac{dp}{dt} = K (p - p_0)^{5/2}$$

for the initial rate of the reaction of powdered uranium at 357°C. In this equation p_0 is the decomposition pressure of UH_3 at the temperature of the experiment. In the present series of experiments p_0 would be less than 1 cm. Certainly conditions are markedly different in the amount of surface exposed to the hydrogen in the two cases.

The induction periods in the reaction of hydrogen with the niobium-zirconium-uranium alloys were similar to those found with niobium - uranium specimens.(2) Figure 22 shows the results with a 4% zirconium, 2% niobium alloy, water quenched from 800°C. It is to be noted that after an induction period of about 80 minutes, the reaction rates were very similar to those of unalloyed uranium. Figure 23 is a curve of the reaction with 5% zirconium 1-1/2% niobium - uranium alloy, not heat treated. Here the induction period was about 225 minutes and the shape of the curve differed somewhat from that of unalloyed uranium. Figure 24 shows the results with the same alloy quenched from 800°C on the scale of the other experiments, and Figure 25 with a changed time scale so as to include the induction period. Here the induction period is very long and the hydriding rate has been slowed down.

It is evident that both compositions tested react with hydrogen at 260°C, quenched or unquenched. Figure 26 shows the general appearance of the resultant hydrides. Unalloyed uranium produces a dull black powder; quenched 4% zirconium 2% niobium - uranium alloy has the appearance of flake graphite; the non-heat treated material produces irregular scales, as was the case with the 5% zirconium 1-1/2% niobium alloy, heat treated.

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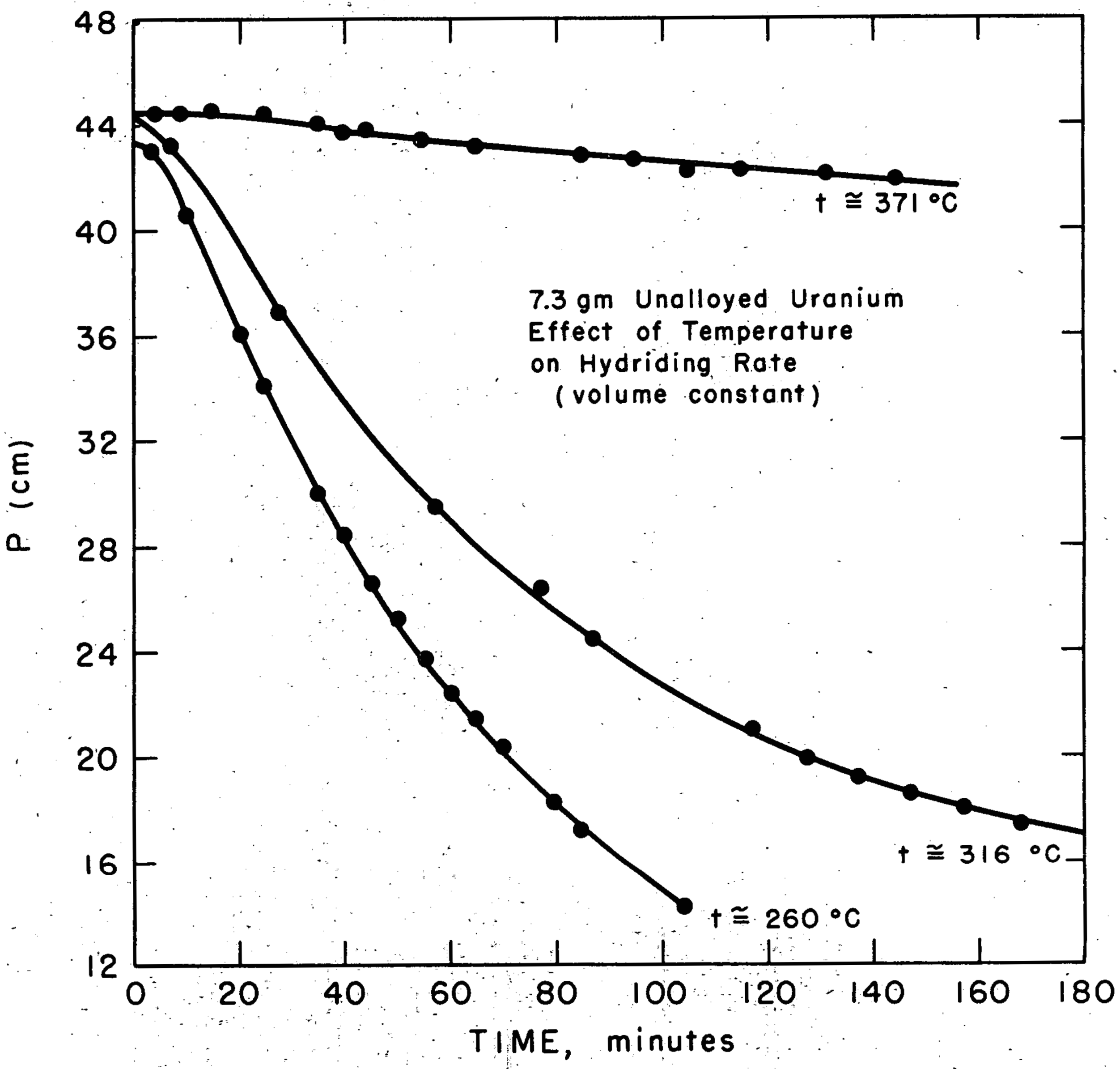

FIGURE 20.

EFFECT OF TEMPERATURE ON REACTION OF HYDROGEN WITH URANIUM.

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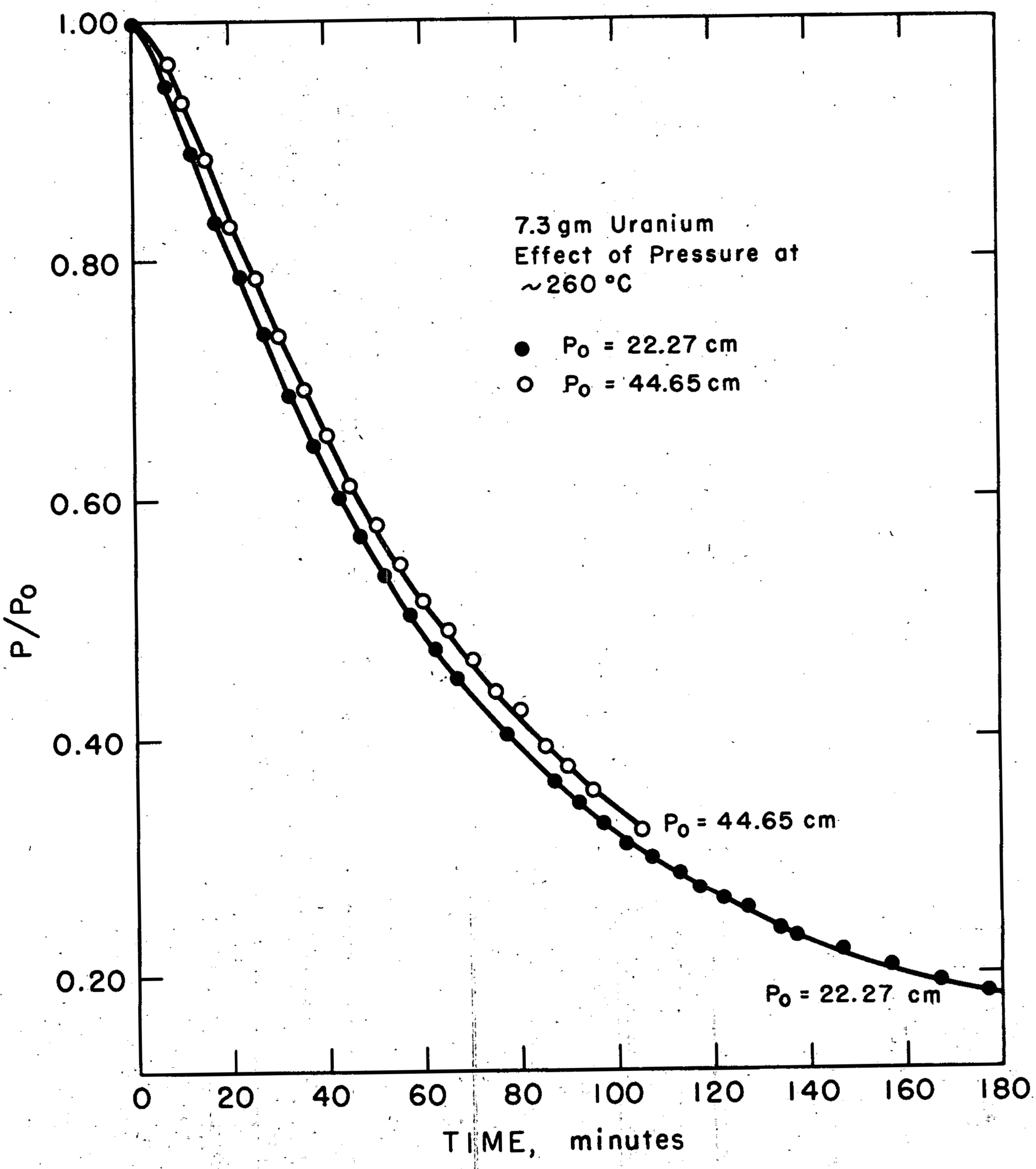

FIGURE 21.

EFFECT OF DOUBLING PRESSURE ON UH_3 FORMATION AT 260 °C.

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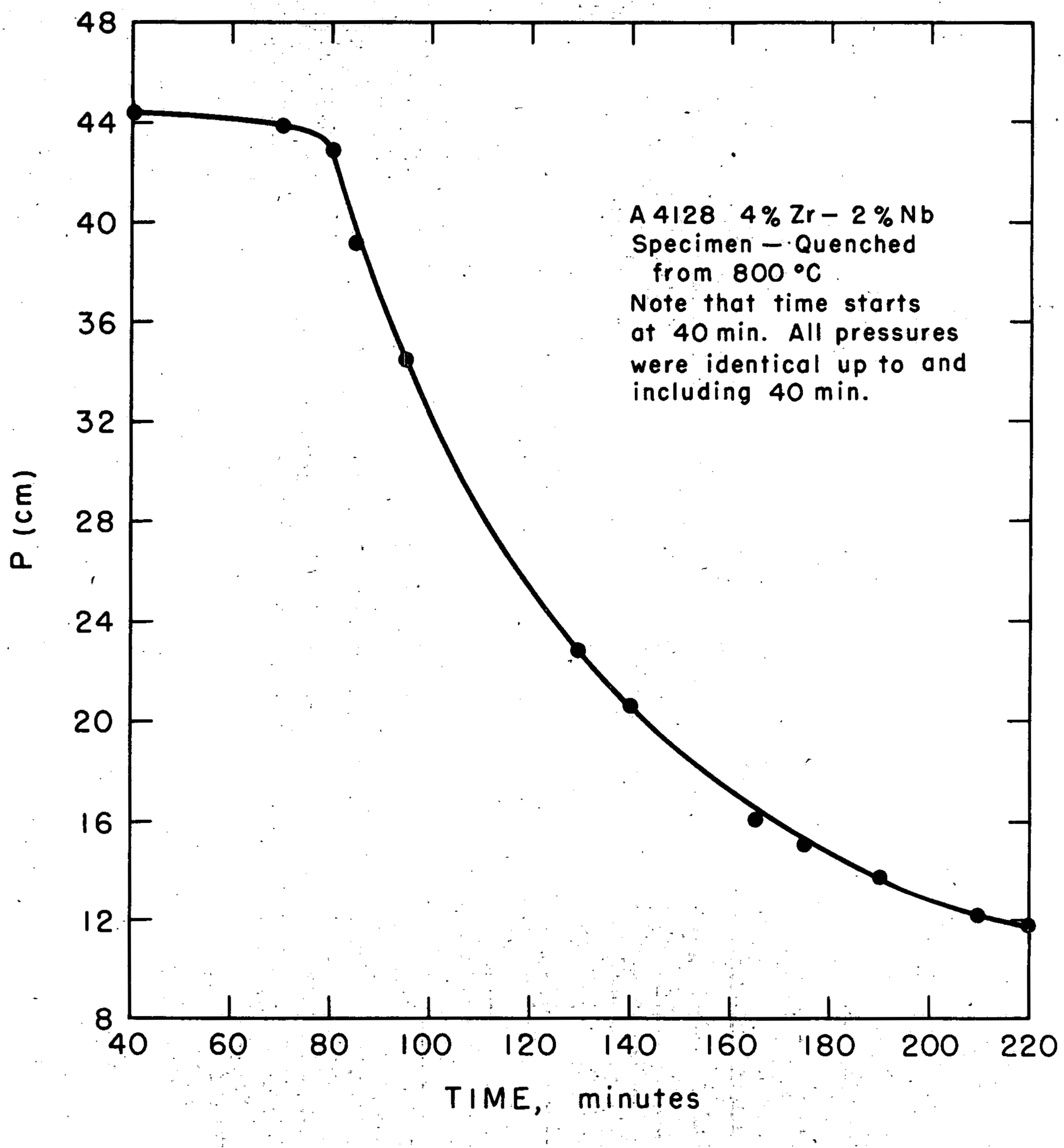

FIGURE 22.

TIME – PRESSURE CURVE FOR HYDROGEN AND QUENCHED 2% Nb – 4% Zr – U ALLOY AT 260 °C.

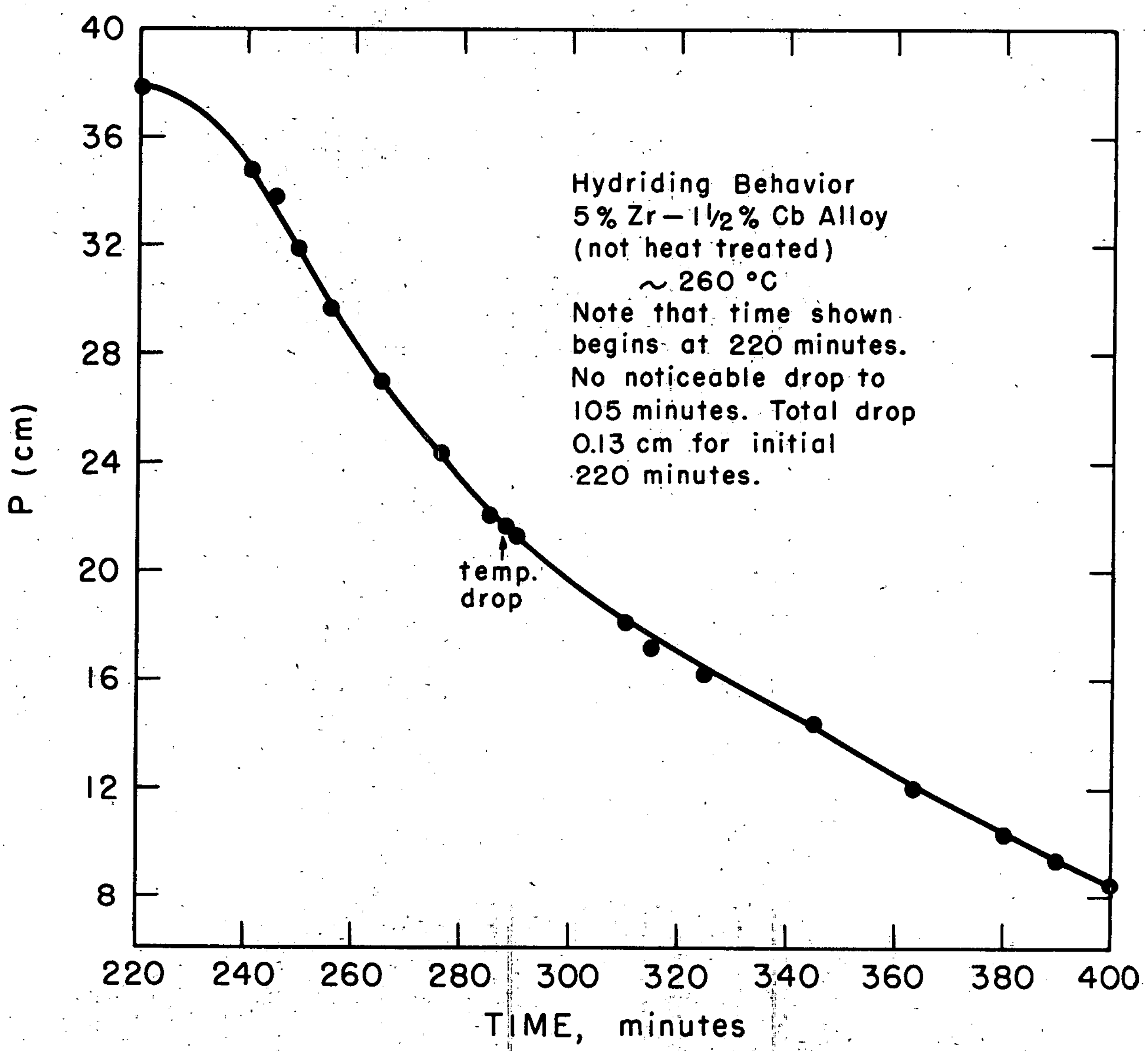

FIGURE 23.

TIME–PRESSURE CURVE FOR HYDROGEN AND UNQUENCHED 1½% Nb–5% Zr–U ALLOY AT 260 °C.

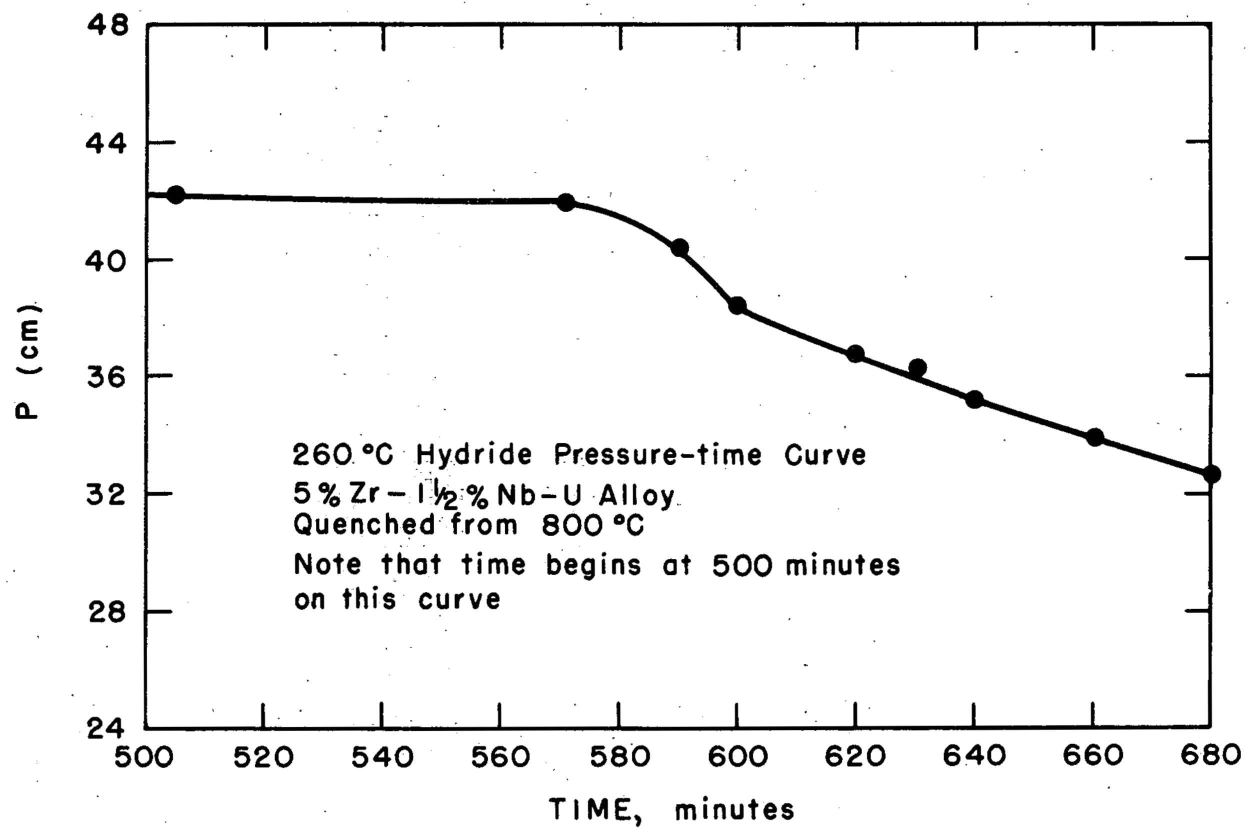

FIGURE 24.

TIME – PRESSURE CURVE FOR HYDROGEN
AND 800 °C QUENCHED 1½ % Nb –
5 % Zr – U ALLOY AT 260 °C
(time scale same as others for this series)

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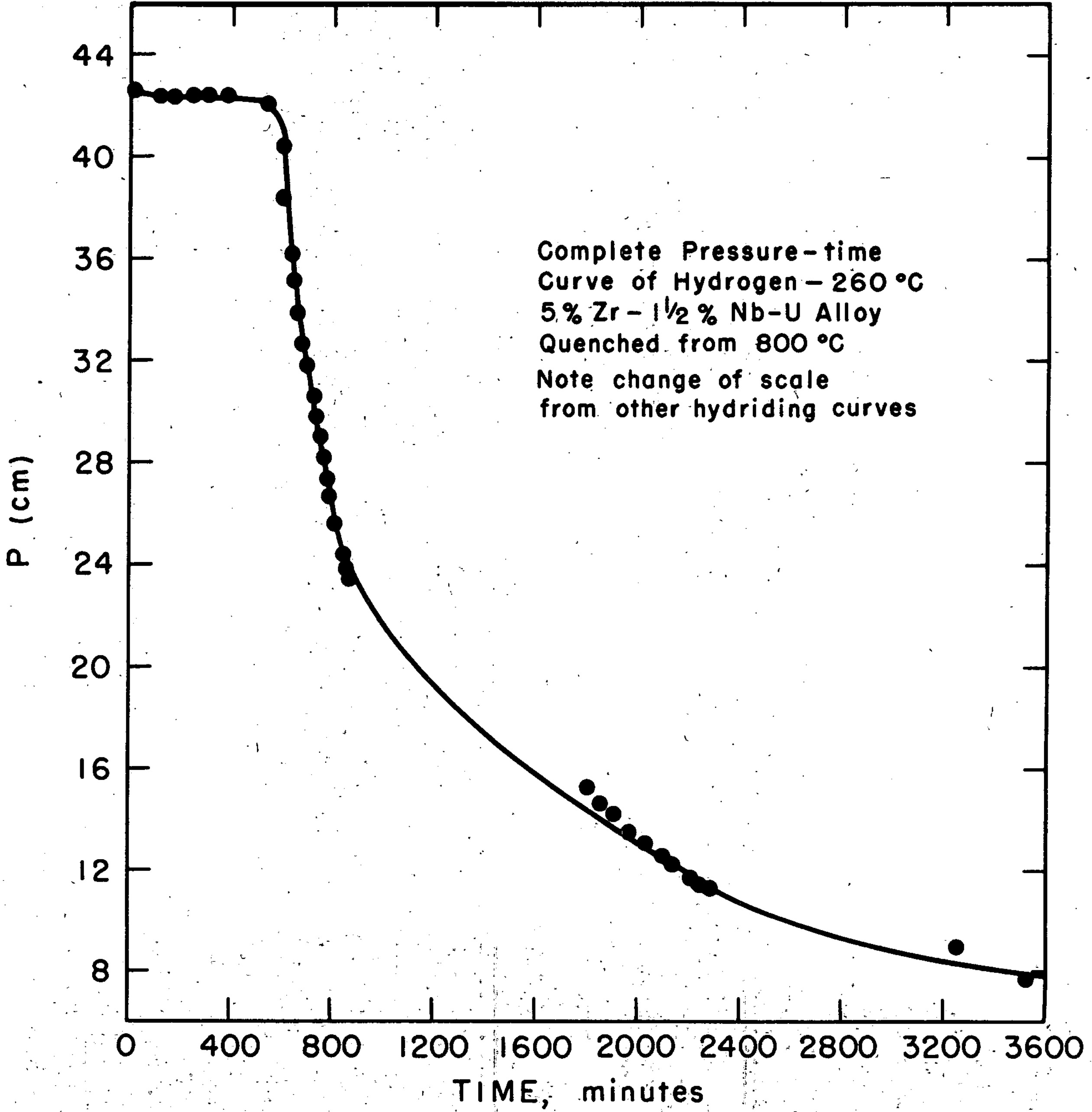

FIGURE 25.

TIME-PRESSURE CURVE FOR HYDROGEN AND 800 °C QUENCHED 1½ % Nb – 5% Zr – U ALLOY AT 260 °C.
(time scale not same as in others of this series)

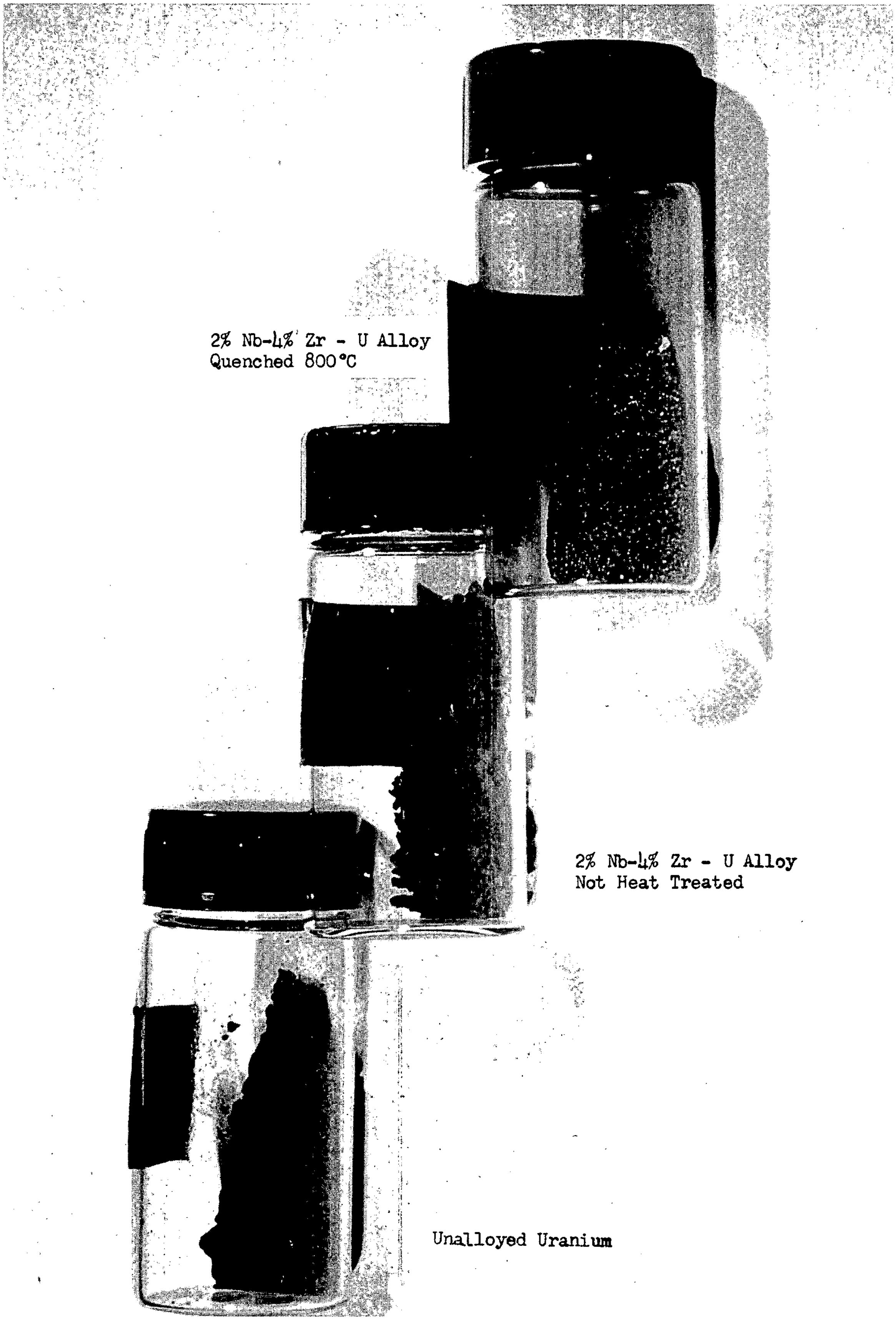
2% Nb-4% Zr - U Alloy
Quenched 800°C
2% Nb-4% Zr - U Alloy
Not Heat Treated
Unalloyed Uranium

Figure 26. Appearance of Uranium Hydride Made From Uranium And Some Alloys as Shown.

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