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LOW-TEMPERATURE DUCTILITY AND HYDROGEN EMBRITTLEMENT OF URANIUM - A LITERATURE REVIEW

H. Inouye and A. C. Schaffhauser

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H. Inouye and A. C. Schaffhauser

JULY 1969

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ABSTRACT

The ductile-to-brittle transition temperature of uranium, which ranges between 100 and −50°C, is influenced by the grain size, strain rate, and the grain structure. Thus, the ductility of uranium at room temperature depends upon the heat treatment, fabrication procedures, and the testing conditions. Aluminum, silicon, and carbon impurities in concentrations below 1000 ppm impair the ductility.

A literature review of the hydrogen embrittlement of uranium was made. Threshold concentrations between 0.2 and 0.5 ppm H present as β-UH_3 are the probable causes of the severe impairment of the ductility at room temperature. The morphology of UH_3 and microcracks associated with this phase also influence the degree of embrittlement. The hydrogen in heat-treated uranium is derived from the reaction of uranium with water vapor in the annealing media.

INTRODUCTION

Alpha uranium, which is stable below 663°C, has an orthorhombic crystal structure. The elastic constants vary greatly with crystallographic orientation resulting in very anisotropic behavior. In polycrystalline material, very high stress concentrations are produced because of the anisotropy of deformation, and localized yielding occurs at low applied stress. Below 450°C the predominant mode of deformation is {172}, {130}, and {176} twinning depending on the crystallographic orientation with some {010}⟨100⟩ slip. Cross slip on {001}⟨100⟩ and slip on {011}⟨100⟩ to accommodate twins is also observed. The crystallography of twinning has been examined in detail by Cahn[1] and Crocker.[2]

[1]R. W. Cahn, Acta Met. 1, 49 (1953).

[2]A. G. Crocker, J. Nucl. Mater. 16, 306 (1965).

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The atom displacements are large, resulting in considerable distortion and stress at the twin boundary (i.e., the twin boundary energy is high).

Twinning increases with decreasing temperature and increasing strain rate since the twinning stress is lowered and slip becomes more difficult. The twinning stress (σ_T) is predicted to increase with decreasing grain size analogous to the Hall-Petch relation:[3]

$$\sigma_T = \sigma_{0(T)} + K_T d^{-\frac{1}{2}} \tag{1}$$

where

σ_T = twinning stress

$\sigma_{0(T)}$ = stress to propagate a twin within a grain,

K_T = stress concentration factor, and

d = grain size.

These considerations are important in assessing the effect of metallurgical and testing variables on the ductility of uranium.

Uranium exhibits a transition in ductility from 20 to 60% elongation to 2 to 5% elongation in the temperature range of 100 to −50°C. The sharpness and average temperature of this transition in ductility are sensitive to minor changes in processing history or testing procedures. This behavior often results in large variations in the room-temperature ductility.

The common impurities Fe, Si, Ni, Cr, Al, C, O, N, and H are soluble in appreciable quantities in the gamma phase of uranium, but are essentially insoluble in the alpha phase, and parts-per-million quantities produce precipitation of uranium compounds. The solubility and the effects of these impurities on high-temperature mechanical properties have been studied extensively, but limited information is available near ambient temperatures except in the case of hydrogen embrittlement by salt bath annealing. Hydrogen is one of the more important impurities in uranium as evidenced by the extensive amount of work devoted to the

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[3] D. Hull, Acta Met. 9, 105 (1961).

determination of its effects. The effect of only a few parts-per-million hydrogen is readily recognized by the severe impairment of the tensile properties near room temperature.

Previous studies of impurity effects at low temperatures are of questionable value because the thermal treatments given uranium did not exclude hydrogen as a variable.

The first part of this report reviews and analyzes the literature data on the ductile-to-brittle transition temperature (DBTT) of uranium to determine the effect of metallurgical and testing variables on the low-temperature ductility. The second part attempts to determine the status of our knowledge of the factors responsible for the hydrogen embrittlement of uranium through an interpretative review of published data.

PART I. LOW-TEMPERATURE DUCTILITY OF URANIUM

Effect of Grain Structure

Grain Size

The ductility of uranium is a very sensitive function of grain size in the temperature region of the DBTT. This is clearly illustrated in Fig. 1 with the data of Taplin[4,5] for vacuum-annealed reactor-grade uranium (approximately 500 ppm total impurities). The transition from a ductile-to-brittle behavior is more gradual with increasing grain size, which raises the mean DBTT. The room-temperature ductility as a function of grain size (Fig. 2) shows the marked reduction in ductility at the larger grain sizes. Also, in high-purity uranium (less than 100 ppm total impurities), Lemogne and Lacombe[6] showed a decrease in room-temperature elongation from 18 to 3.3% with an increase in grain size from 0.015 to 0.086 mm (Fig. 3). Taplin's data for material having a grain size of

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[4] D.M.R. Taplin and J. W. Martin, J. Less-Common Metals 7, 89 (1964).

[5] D.M.R. Taplin, J. Australian Inst. Metals 12(1), 32 (1967).

[6] A. Lemogne and P. Lacombe, J. Nucl. Mater. 16, 129 (1965).

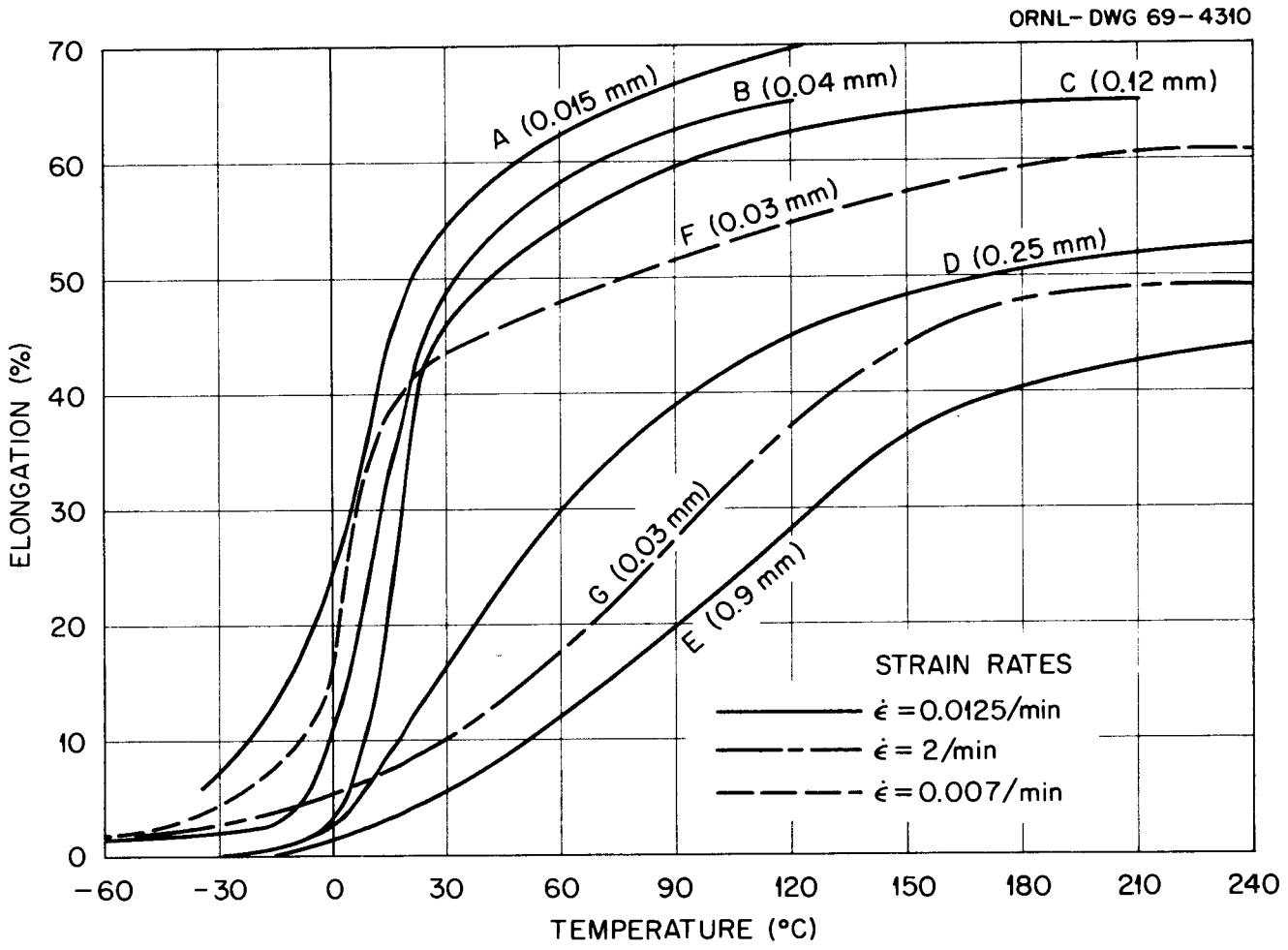

Fig. 1. Elongation of Reactor-Grade Uranium as a Function of Temperature, Grain Size, and Strain Rate. Based on: D.M.R.Taplin and J. W. Martin, J. Less-Common Metals 7, 89 (1964) and D.M.R. Taplin, J. Australian Inst. Metals 12(1), 32 (1967).

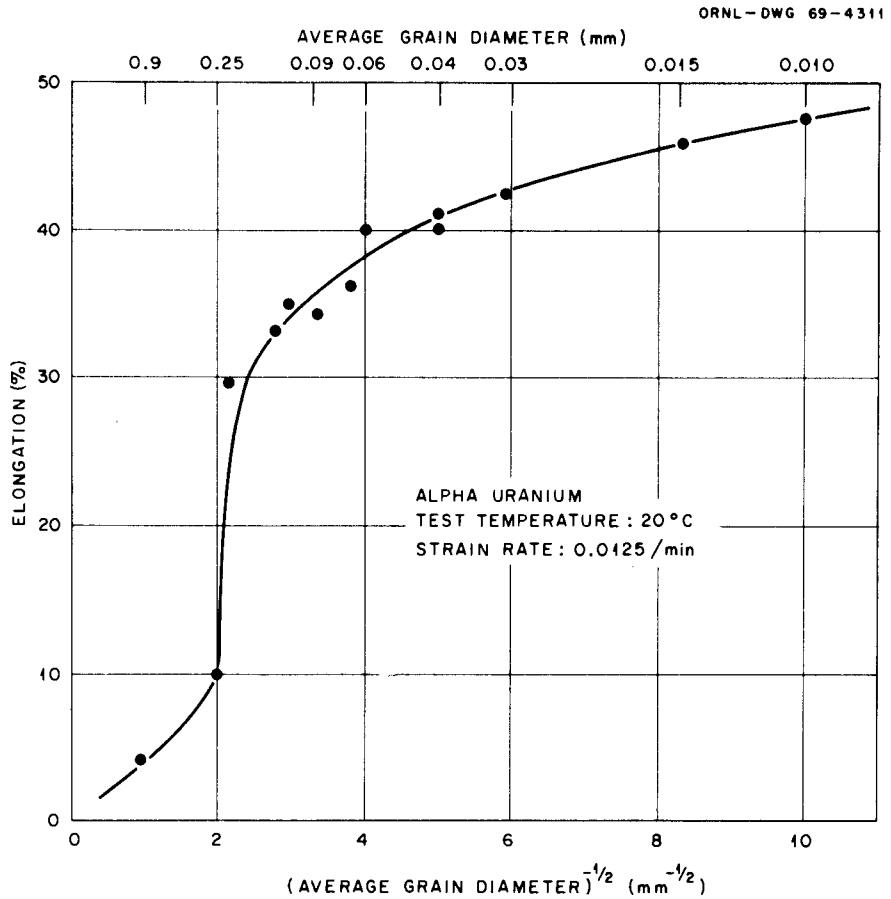

Fig. 2. The Effect of Grain-Size on the Tensile Elongation of Reactor-Grade Uranium (99.9% Purity) at 25°C. Based on: D.M.R. Taplin, J. Australian Inst. Metals 12(1), 32 (1967).

0.030 mm and tested at a strain rate of 2 min^{-1} (curve G, Fig. 1) compares very closely with the data of Lemogne and Lacombe[6] at a strain rate of 0.6 min^{-1}. These investigators, using materials of different purity, produced the different grain sizes by different rolling and annealing schedules which could have influenced the texture and distribution of precipitates. However, the good correlation of the data indicates that the grain size has the largest effect on ductility for material in this purity range.

From his observation that extensive microcracking occurs only in coarse-grain specimens, Taplin concluded that fracture is controlled by crack nucleation for his test conditions. In fine-grain material the twinning stress is high, the stress concentration at the tip of a twin is lower; therefore, cracks are difficult to nucleate and ductile failure

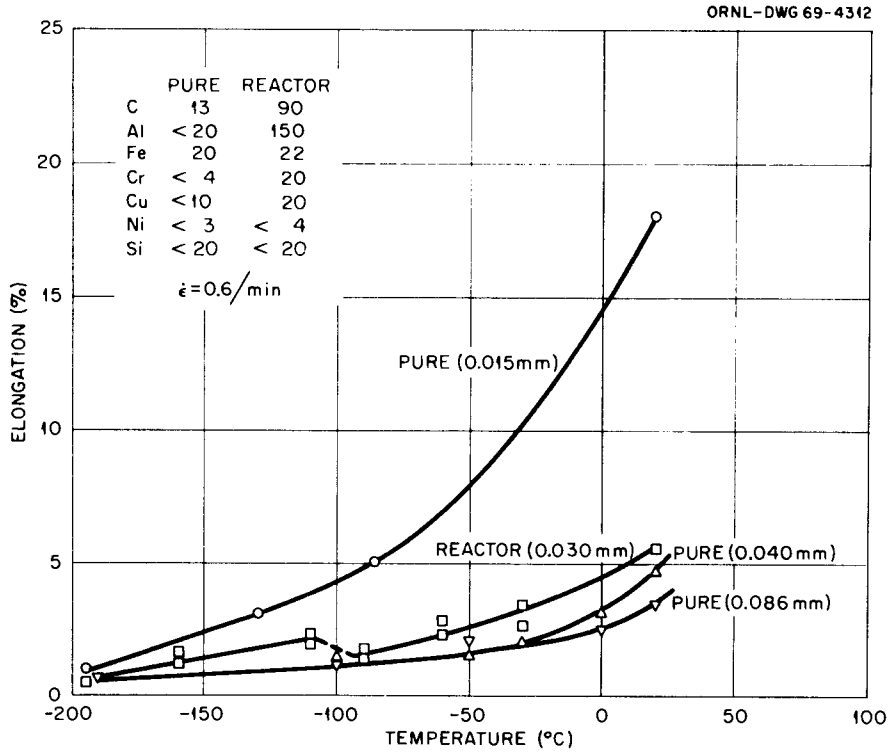

Fig. 3. Elongation at 25°C of Pure and Reactor-Grade Uranium for Different Grain Sizes. Based on: A. Lemogne and P. Lacombe, J. Nucl. Mater. 16, 129 (1965).

occurs only after extensive deformation. In the coarse grain material cracks are nucleated because of more profuse twinning and brittle fracture results when the cracks exceed a critical size.

The decreased slope of the DBTT with increasing grain size (Fig. 1) can also be explained using this argument. At high temperatures, cracks are nucleated in the coarse grain material but their growth to a critical size for propagation is difficult because the stress at the tip of the crack can be relieved by slip. As the test temperature is decreased, cracks can grow more easily and fracture occurs at lower strains. In fine grain material the DBTT occurs at lower temperatures because crack nucleation is more difficult and the DBTT is very sharp because the first crack which is nucleated can propagate easily and initiate fracture.

Cold Work and Texture

Although numerous room-temperature tensile tests have been performed on cold worked uranium, very little data have been reported on the DBTT of this material. The data of Taplin and Martin[7] (Fig. 4) show the effect of vacuum annealing temperature in the alpha phase on the room-temperature ductility of reactor-grade uranium rod which was rolled 28% at 300°C before annealing. The elongation shows minor changes after recovery and partial recrystallization and varies between 35 and 48% for the as-rolled and recrystallized conditions.

In a later study Taplin and Martin[4] determined the DBTT of this material. The significant differences in ductility from −100 to 100°C between the as-rolled and material annealed at 550°C are shown in Fig. 5. While the annealed material has greater ductility at room temperature and above, the ductility of the rolled material is greater at lower temperatures.

[7] D.M.R. Taplin and J. W. Martin, J. Less-Common Metals 6, 430 (1964).

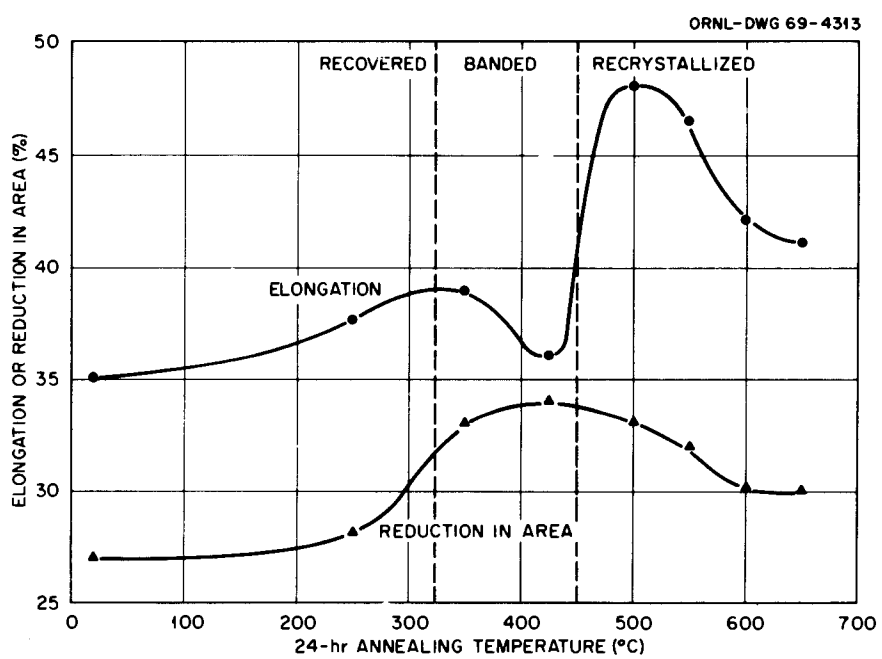

Fig. 4. Elongation and Reduction in Area at 25°C as a Function of Annealing Temperature for Reactor-Grade Uranium Rolled 28% at 300°C. Based on: D.M.R. Taplin and J. W. Martin, J. Less-Common Metals 6, 430 (1964).

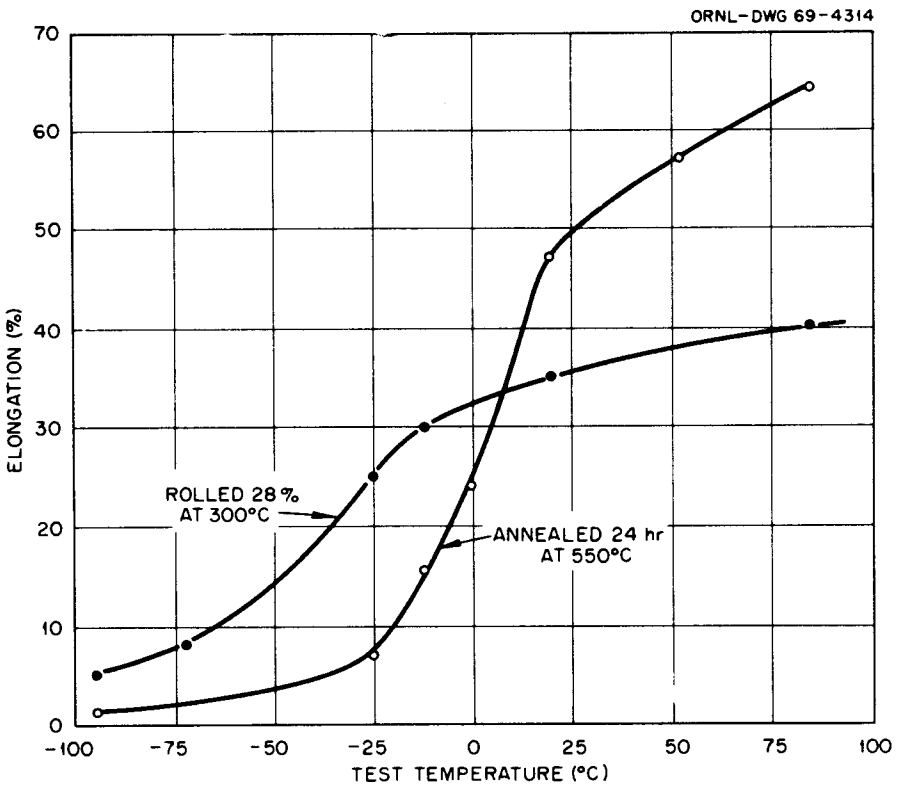

Fig. 5. Elongation of Rolled and Annealed Reactor-Grade Uranium as a Function of Test Temperature. Based on: D.M.R. Taplin and J. W. Martin, J. Less-Common Metals 7, 89 (1964).

Apparently the rolling temperature also has an effect on the ductility of uranium. Marsh et al.[8] tested biscuit (purity not given) uranium rolled from a salt bath at 550°C. The grain structure and high tensile strength of this material showed that the temperature drop during rolling allowed a large amount of warm work to be retained. As seen in Fig. 6 the ductility of the warm rolled material was greater than vacuum-annealed material at all test temperatures, which is not consistent with data of Taplin and Martin[4] (Figs. 4 and 5).

Tilbury[9] observed a difference in the ductility of hot rolled rod and hot cross-rolled plate which he attributed to a difference in

[8]L. L. Marsh, G. T. Muehlenkamp, and G. K. Manning, Effect of Hydrogen on the Tensile Transition in Uranium, BMI-980 (Feb. 14, 1955).

[9]P. D. Tilbury, Impact Testing of Uranium, AWRE-O-36/66 (March 1966).

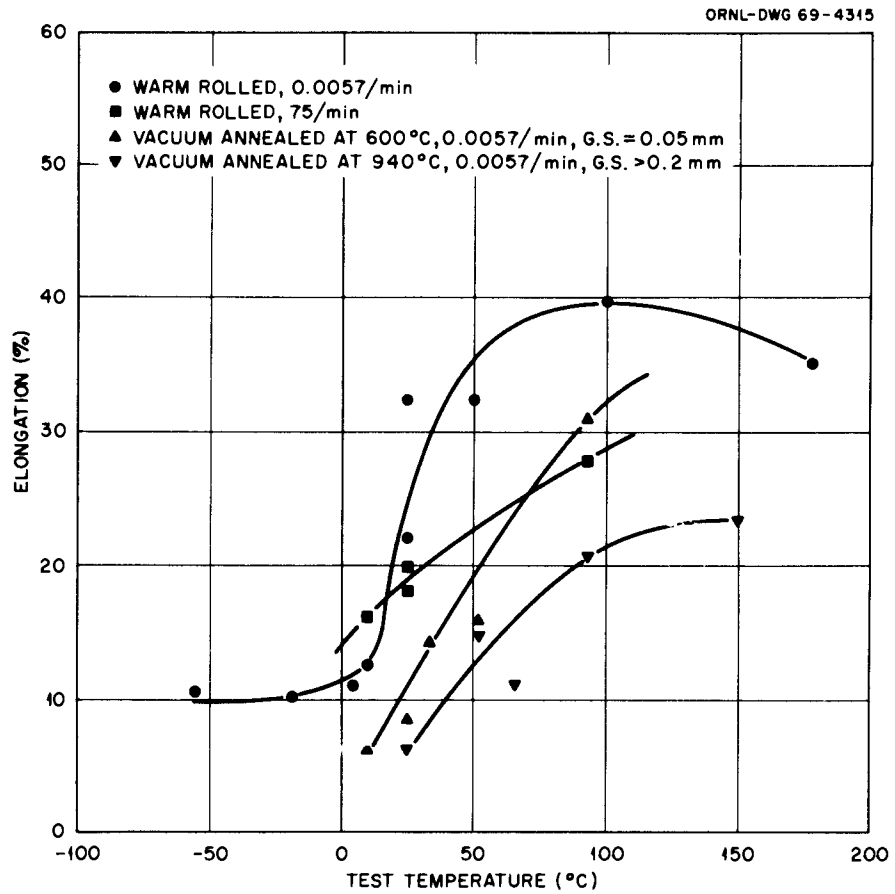

Fig. 6. Elongation of Warm-Rolled and Vacuum-Annealed Biscuit Uranium as a Function of Test Temperature. Based on: L. L. Marsh, G. T. Muehlenkamp, and G. K. Manning, Effect of Hydrogen on the Tensile Transition in Uranium, BMI-980 (Feb. 14, 1955).

preferred orientation. The data in Fig. 7 for the hot rolled rod show that the low-temperature ductility of the vacuum-annealed material is higher than that of the salt bath annealed material, but the DBTT is about the same for both slow strain rate and tensile impact testing. Figure 8 shows that the ductility of the plate material above and below the DBTT are the same for the vacuum-annealed and salt bath annealed conditions, but the DBTT is about 25°C lower for the vacuum-annealed material.

Other evidence of the effect of preferred orientation on the mechanical properties is discussed later in the section on impact tests.

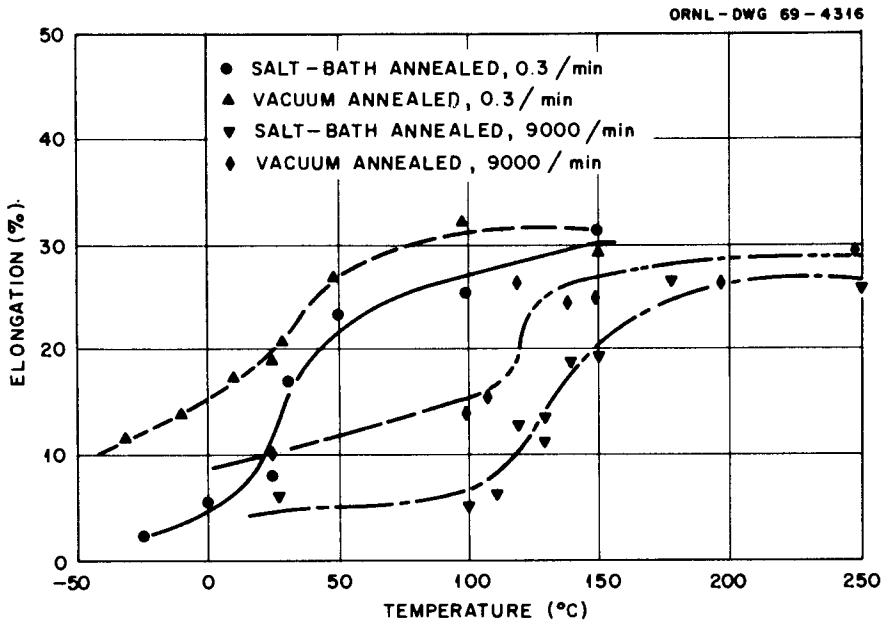

Fig. 7. Comparison of the DBTT of Salt-Bath-Annealed and Vacuum-Annealed Uranium for Tensile Impact and Slow Strain Rate Tests. Based on: P. D. Tilbury, Impact Testing of Uranium, AWRE-O-36/66 (March 1966).

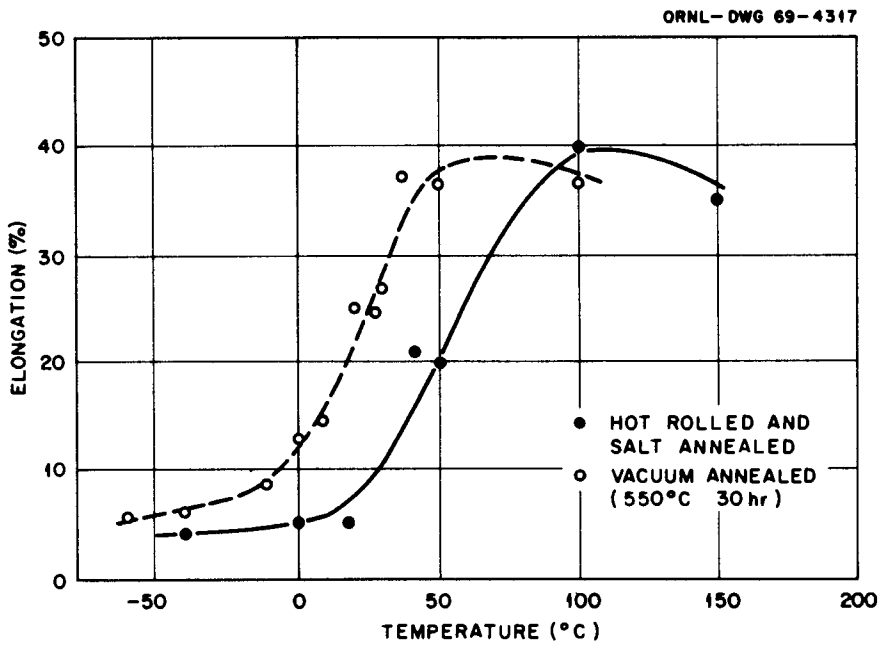

Fig. 8. Ductile-to-Brittle Transition Temperature of Vacuum-Annealed and Salt-Bath-Annealed Hot-Rolled Reactor-Grade Uranium Plate. Based on: P. D. Tilbury, Impact Testing of Uranium, AWRE-O-36/66 (March 1966).

Castings

A study of high assay uranium (97.5% ^{235}U) by Legacy and Yaggi[10] showed that the mechanical properties were uniform within and between castings. The average tensile ductility of specimens machined from four ingots vacuum cast in heated graphite molds (Fig. 9) is consistently lower than wrought uranium at the same temperature (Fig. 5).

The effect of the grain shape on the tensile transition behavior of alpha-uranium[11] is shown in Fig. 10. Cast specimens quenched from the beta phase, then annealed at 470°C, had the most irregular alpha grains, while specimens quenched in mercury, then annealed at 600°C, had a recrystallized and smooth grain structure. Specimens with irregular grains had a higher DBTT. Taplin[11] suggests that irregular boundaries would increase the tendency for crack propagation.

Effect of Strain Rate

Tensile Tests

As with most materials exhibiting a DBTT, the strain rate of the test can have a marked influence on the ductility of uranium. Increasing the strain rate of alpha-annealed uranium raises the mean DBTT and produces a more gradual transition as shown by curves F and G in Fig. 1. This behavior is similar to that produced by increasing the grain size of the material. From these results Taplin concluded that a high strain rate promotes crack nucleation because stress relaxation by twinning is easier than slip. Fracture is controlled by crack propagation under these conditions because of greater difficulty in relieving the stress at the tip of a crack. At low strain rates the DBTT is lower due to decreased crack nucleation. Therefore, fracture is nucleation controlled since the first crack can easily propagate at these lower temperatures.

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[10] D. J. Legacy and W. J. Yaggi, Determination of Selected Mechanical and Physical Properties of Cast Very-High-Assay Uranium, Y-1593 (Jan. 29, 1968).

[11] D.M.R. Taplin and J. W. Martin, J. Nucl. Mater. 10(2), 134 (1963).

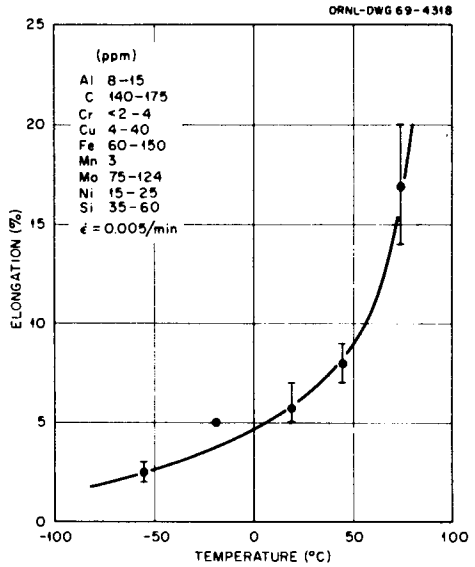

Fig. 9. Tensile Elongation of Vacuum Induction Cast Uranium as a Function of Test Temperature. Based on: D. J. Legacy and W. J. Yaggi, Determination of Selected Mechanical and Physical Properties of Cast Very-High-Assay Uranium, Y-1593 (Jan. 29, 1968).

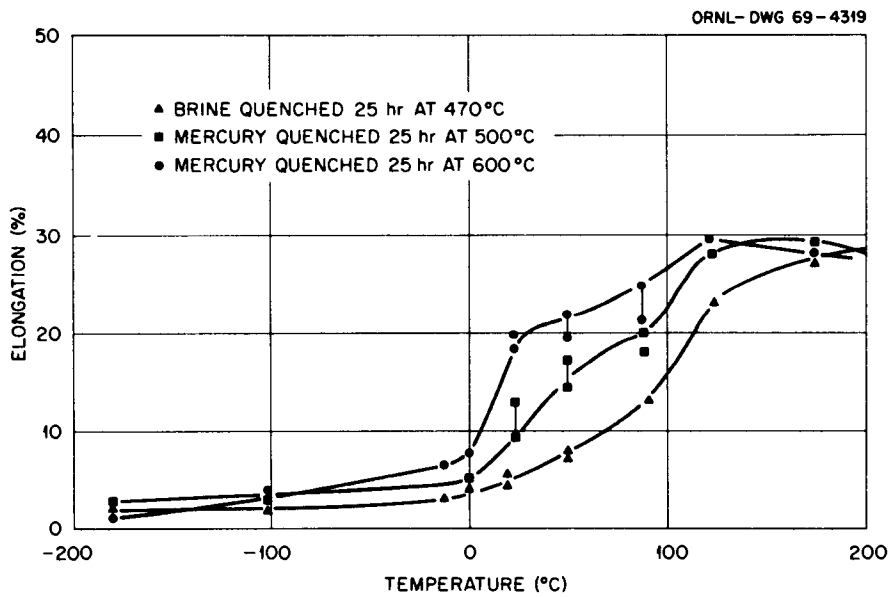

Fig. 10. Tensile Elongation of Beta-Quenched and Alpha-Annealed Cast Reactor-Grade Uranium as a Function of Test Temperature. Impurity contents: 600 ppm C, 200 ppm Al, 75 ppm Fe, and 65 ppm Si. Based on: D.M.R. Taplin and J. W. Martin, J. Nucl. Mater. 10(2), 134 (1963).

A comparison of data of different investigators shows that metallurgical variables such as grain structure and purity also influence the strain rate sensitivity. In the early work at Battelle,[8] the DBTT of hot rolled uranium was determined for strain rates of 0.0057 and 75 min^{-1}. The data in Fig. 6 show a gradual decrease in ductility with decreasing temperature at the higher strain rate, but the shift in the DBTT is not as large as that observed by Taplin for a smaller increase in strain rate. However, as previously discussed, the material retained a significant amount of warm work, had an inhomogeneous grain structure, and had a higher purity than Taplin's material. It is impossible to assess the importance of these differences because of the lack of a complete characterization of the starting material. Other investigations by Newman[12] and Loretan and Murphy[13] showed no consistent effect of strain rates between 0.00078 and 1.56 min^{-1} on the room-temperature ductility of vacuum, hydrogen, or salt bath annealed uranium.

Tilbury[9] compared the DBTT of hot rolled and annealed reactor-grade uranium rod at strain rates of 0.3 and 9000 min^{-1}. The very high tensile strain rate was obtained with a modified pendulum-type impact tester. The data in Fig. 7 show that the DBTT of both the vacuum and salt bath annealed material was raised from 25 to 120°C by the large increase in strain rate.

These results and other unpublished work were analyzed by Tilbury in terms of the Stroh[14] relationship between DBTT and strain rate

$$\frac{1}{T_c} = -\left(\frac{k}{u}\right) \ln \dot{\epsilon} + C \ , \qquad (2)$$

where

T_c = DBTT, °K

$\dot{\epsilon}$ = strain rate,

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[12]G. T. Newman, Ph.D. Thesis, University of Birmingham (1966).

[13]P. A. Loretan and G. Murphy, Influence of Rate of Loading on the Tensile Properties of Normal Alpha Uranium, IS-757 (August 1963).

[14]A. M. Stroh, Advan. Phys. 6, 418 (1959).

u = activation energy to free the most easily operated source when the dislocation pileup is of sufficient size to initiate a crack,

k = Boltzman constant, and

C = a constant independent of T_c and $\dot{\epsilon}$.

The data are plotted in Fig. 11 along with that of Taplin[15] and Marsh et al.[8] The data of Taplin and Tilbury for alpha-annealed uranium are in reasonable agreement with an activation energy of about 1.09 ev whereas Marsh et al. show a much lower strain rate sensitivity for warm rolled uranium.

[15]D.M.R. Taplin, J. Australian Inst. Metals 12(1), 32 (1967).

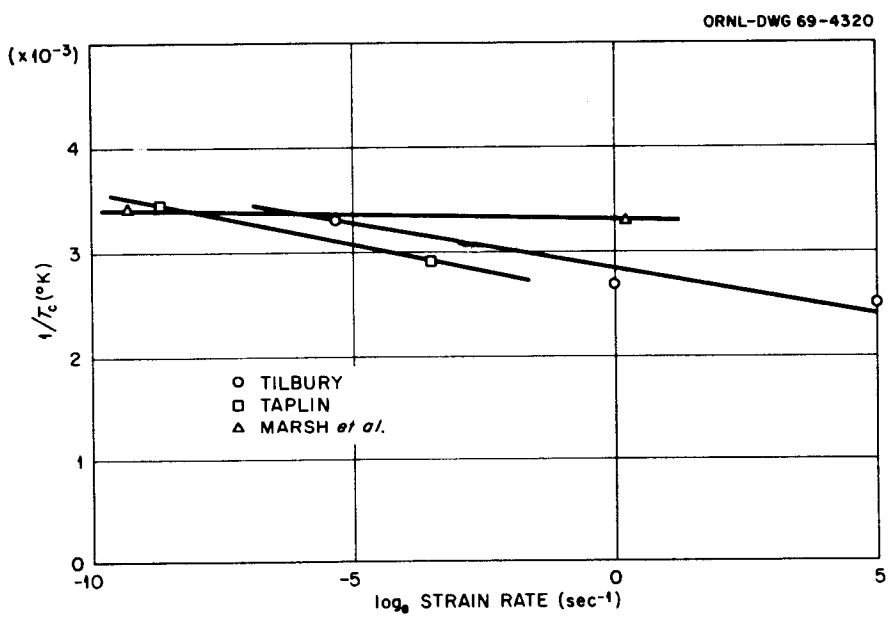

Fig. 11. Relationship Between DBTT and Strain Rate. Based on: P. D. Tilbury, Impact Testing of Uranium, AWRE-O-36/66 (March 1966); L. L. Marsh, G. T. Muehlenkamp, and G. K. Manning, Effect of Hydrogen on the Tensile Transition in Uranium, BMI-980 (Feb. 14, 1955); and D.M.R. Taplin, J. Australian Inst. Metals 12(1), 32 (1967).

Charpy V-Notch Impact Tests

Impact data on standard Charpy V-notch specimens from four investigators are compared in Fig. 12. Marsh et al.[8] observed a gradual increase from 5 to 20 ft-lb in the impact values between −75 and 110°C, then a sharper increase to greater than 40 ft-lb between 110 and 170°C for material hot rolled from a salt bath at 550°C. Similar material tested by Tilbury[9] showed a similar transition behavior but had higher impact

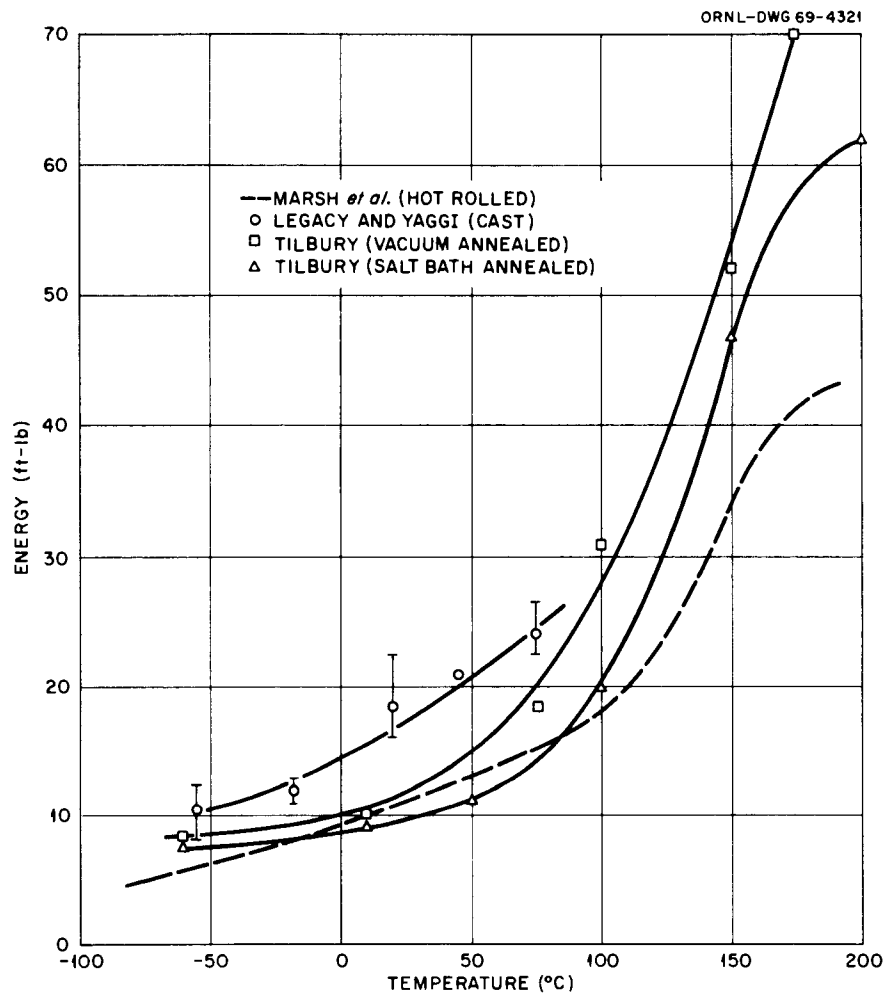

Fig. 12. Charpy V-Impact Values of Uranium as a Function of Test Temperature. Based on: L. L. Marsh, G. T. Muehlenkamp, and G. K. Manning, Effect of Hydrogen on the Tensile Transition in Uranium, BMI-980 (Feb. 14, 1955); P. D. Tilbury, Impact Testing of Uranium, AWRE-O-36/66 (March 1966); and D. J. Legacy and W. J. Yaggi, Determination of Selected Mechanical and Physical Properties of Cast Very-High-Assay Uranium, Y-1593 (Jan. 29, 1968).

values at the higher temperatures. The vacuum-annealed material tested by Tilbury had a transition temperature about 25°C lower than the salt annealed material. This difference is about the same as that observed for the tensile tests and is assumed to be due to hydrogen pickup during the salt bath anneal.

Impact tests by Legacy and Yaggi[10] on specimens cut from large vacuum-induction castings were not performed at high enough temperatures to determine a transition temperature; however, this material had the highest impact values below 75°C. These results are surprising in view of the large grain size and low tensile strength and elongation (Fig. 9) of the cast material compared to hot rolled and annealed material. The preferred orientation of the hot rolled and annealed material could be responsible for the lower impact properties. The impact specimens cut from the hot rolled plate always have the V notch perpendicular to the rolling plane, whereas in tensile tests the tensile axis is always parallel to the rolling plane. The specimens cut from the cast material had a random orientation and specimens longitudinal and transverse to the mold axis had similar properties.

Effect of Metallic Impurities

There has been no systematic investigation of the effect of a specific metallic impurity on the low-temperature ductility of vacuum-annealed uranium. A compilation of the tensile properties of several lots of uranium sheet having different iron, silicon, and carbon impurity concentrations is given in Table 1 (ref. 16). These data suggest that silicon increases the strength and lowers the ductility of uranium. However, the grain size and test conditions for these materials were not given, so it is difficult to draw a firm conclusion from these results. Table 2 lists the impurity concentrations in three grades of uranium and their corresponding ductilities as a function of the test

[16]G. S. Hanks *et al.*, *Effect of Annealing Media on the Mechanical Properties of Uranium*, LA-1619 (August 1953).

Table 1. Tensile Properties of Several Lots of Uranium Sheet 0.035-in. Thick after Vacuum Annealing for 30 min at 600°C[a]

Lot Number	Impurity Concentration, ppm			Strength, psi		Percent Elongation (in 2-in. Increments)
	Silicon	Carbon	Iron	Ultimate Tensile	Yield	
				$\times 10^3$	$\times 10^3$	
D-544-B	20	320	70	133.0	47.0	21.6
E-235	30	310	> 100	131.3	45.2	19.0
D-427[b]	50	350	> 100	152.5	56.2	10.2
D-427[b]	50	350	> 100	133.0	45.3	18.5
E-288	100	385	> 100	146.1	55.2	14.8
E-288	100	385	> 100	146.1	55.2	13.2
D-599-A	100	290	> 100	143.9	53.7	13.8

[a]G. S. Hanks et al., Effect of Annealing Media on the Mechanical Properties of Uranium, LA-1619 (August 1953).

[b]Contained 100 ppm Mo.

Table 2. Impurity Concentration of Uranium for Data in Figs. 13 and 14

Grade	Impurity Concentration, ppm			
	Iron	Aluminum	Silicon	Carbon
A	50	50	65	570
B	500	250	30	590
C	1000	500	70	780

temperatures are plotted in Figs. 13 and 14 (ref. 17). These latter data show that the DBTT of uranium is raised as the iron, aluminum, and carbon contents are increased. Although these impurities had a grain-refining effect, it is noted that the grain size effect on the transition temperature is opposite to that observed in Fig. 1.

[17]D.M.R. Taplin and J. W. Martin, J. Nucl. Mater. 12(1), 50 (1964).

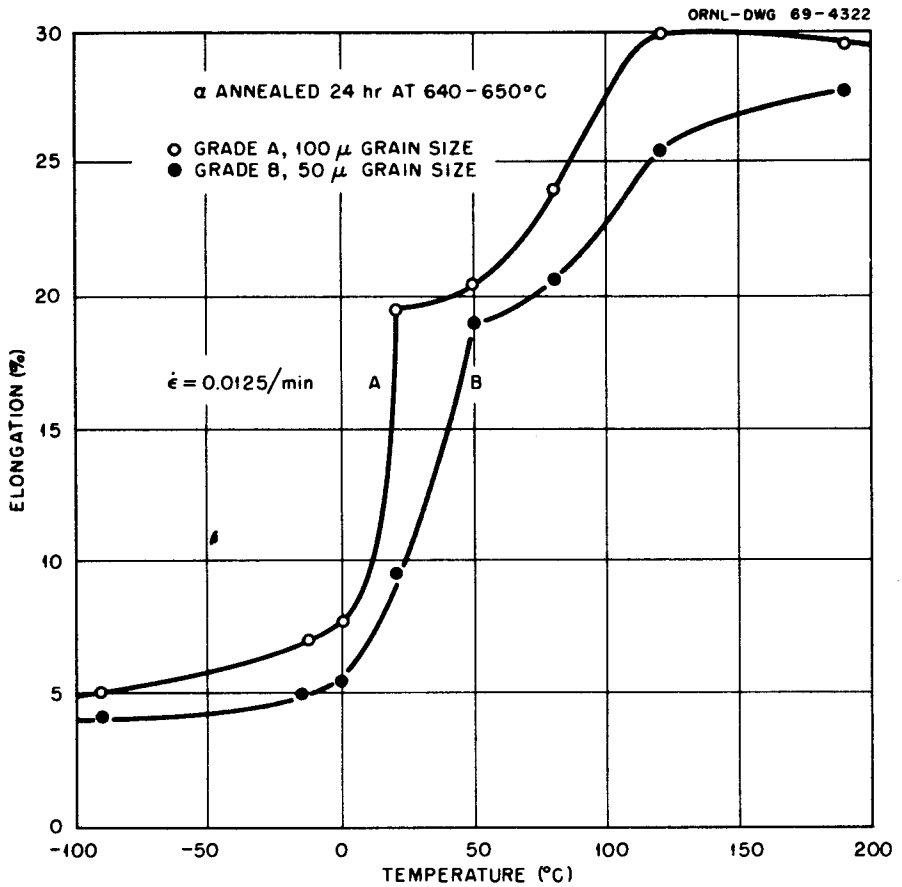

Fig. 13. Elongation of Alpha-Annealed Uranium as a Function of Test Temperature. Based on: D.M.R. Taplin and J. W. Martin, J. Nucl. Mater. 12(1), 50 (1964).

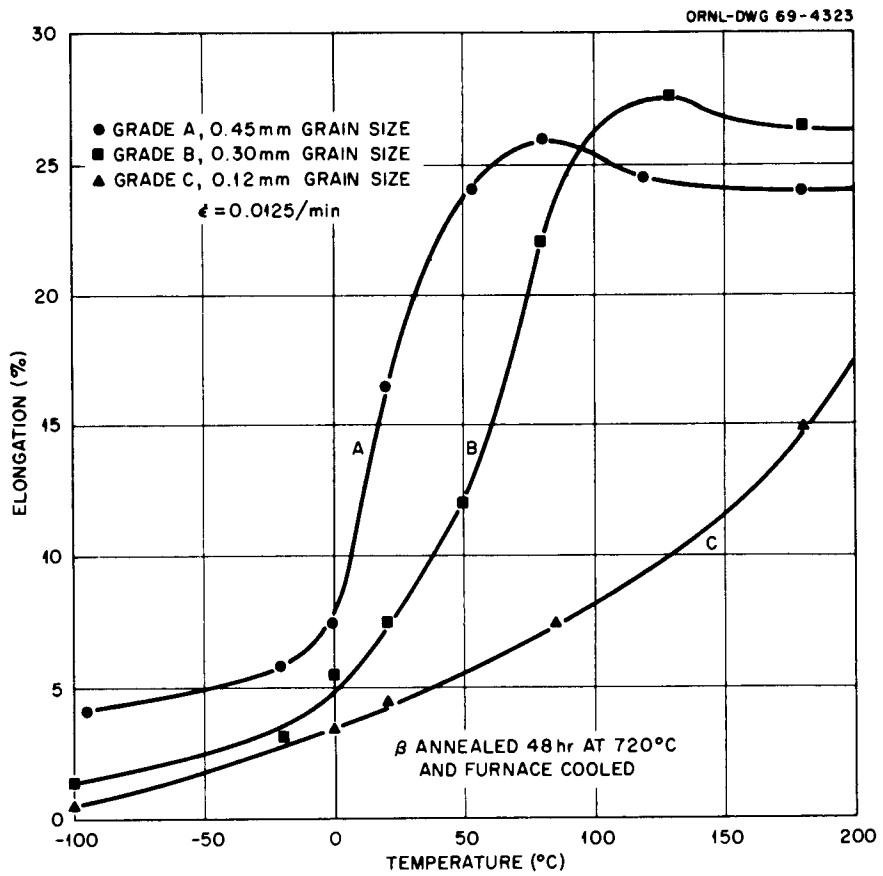

Fig. 14. Elongation of Beta-Annealed Uranium as a Function of Test Temperature. Based on: D.M.R. Taplin and J. W. Martin, J. Nucl. Mater. 12(1), 50 (1964).

PART II. HYDROGEN EMBRITTLEMENT OF URANIUM

The Nature of Hydrogen in Uranium

Two sets of literature data on the solubility of hydrogen in uranium are available and cover the interval from 100 to 1400°C (refs. 18, 19). These data, listed in Table 3, show that the solubility is extremely low below 300°C (ref. 20) and increases modestly with temperature; however, a comparatively large and abrupt increase in solubility occurs at 662, 769, and 1129°C. These temperatures correspond to the $\alpha \rightarrow \beta$, $\beta \rightarrow \gamma$, and $\gamma \rightarrow$ liquid transformation in uranium, respectively. In general, the disagreement between these data is mainly in the values of the hydrogen solubility in the alpha phase of uranium.

The permeability, solubility constant, and the diffusivity of hydrogen in uranium are given in Table 4 (ref. 19). The temperature dependence of the diffusivity of hydrogen in alpha uranium based on the tabulated data is given by the equation

$$\log D\ (\mathrm{cm}^2/\mathrm{sec}) = -1390/T(^\circ\mathrm{K}) - 2.85\ . \qquad (3)$$

Somewhat lower values are reported by Mallett and Trzeciak[21] whose data can be represented by the equation

$$\log D\ (\mathrm{cm}^2/\mathrm{sec}) = -2420/T(^\circ\mathrm{K}) - 1.71\ . \qquad (4)$$

The difference between these data may be due to the observation that the values of the diffusivity and permeability were markedly lowered if the surface was contaminated with oxides or nitrides.[19] Davis, whose data are shown in Table 4, recognized this problem and the values of P and D were obtained for a clean uranium surface.

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[18] P. Cotterill, Progr. Mater. Sci. 9(4), 269 (1961).

[19] W. D. Davis, Solubility, Determination, Diffusion, and Mechanical Effects of Hydrogen in Uranium, KAPL-1548 (Aug. 1, 1956).

[20] Since uranium forms UH_3 below 516°C at 1 atm hydrogen pressure, solubility data below this temperature are calculated values.

[21] M. W. Mallett and M. J. Trzeciak, Trans. Am. Soc. Metals 50, 981 (1958).

Table 3. Solubility of Hydrogen in Uranium at 1 atm Hydrogen Pressure

Temperature (°C)	Hydrogen Solubility, ppm	
	Cotterill[a]	Davis[b]
100	0.0006	
200	0.02	
300	0.2	
400	1.1	0.54
500	1.8	0.91
600	2.0	1.65
662(α-U)	2.2	
662(β-U)	7.8	
700	8.5	7.5
769(β-U)	9.7	
769(γ-U)	14.7	
800	15.0	14.0
900	15.6	
1000	16.2	
1100	16.7	
1129(γ-U)	16.9	
1129(liquid U)	28.1	
1200	29.3	
1300	31.1	
1400	32.7	

[a]P. Cotterill, Progr. Mater. Sci. 9(4), 269 (1961).

[b]W. D. Davis, Solubility, Determination, Diffusion, and Mechanical Effects of Hydrogen in Uranium, KAPL-1548 (Aug. 1, 1956).

Table 4. Permeability, Solubility Constant, and Diffusivity of Hydrogen in Uranium

Temperature (°C)	Permeability $\left[\frac{cm^3 \text{ of } H_2 \text{ (NTP)}}{(sec)(cm)(atm)^{\frac{1}{2}}}\right]$	Solubility Constant[a] $\left[\frac{cm^3 \text{ of } H_2 \text{ (NTP)}}{(cm^3)(atm)^{\frac{1}{2}}}\right]$	Diffusivity (cm^2/sec)
	$\times 10^6$		$\times 10^5$
450	2.0	0.118	1.71
475	2.6	0.143	1.82
500	3.9	0.172	2.28
550	6.4	0.251	2.55
600	12.8	0.343	3.73
650	18.9	0.454	4.17
680	64.0	1.71	3.75
700			5
800			10.2

[a]0.236 cm^3 H_2 (NTP) = 1 ppm H (by weight); NTP = 0°C and 760 torr.

The dependence of the hydrogen content of uranium on the hydrogen pressure is given by $C = S_0 \sqrt{P}$, where C is the equilibrium hydrogen content at pressure P, and S_0 is the solubility constant; that is, the solution of hydrogen in uranium obeys Sievert's law. The hydrogen content of the alpha phase of biscuit uranium as a function of the pressure is given in Fig. 15 and of the beta and gamma phases of uranium in Fig. 16 (ref. 19). Extrapolation of the data of Fig. 15 to zero pressure does not pass through the origin as it should, but intersects the ordinate between 0.1 and 0.2 ppm H. A similar extrapolation of the data of Fig. 16 shows the expected normal behavior. Measurements of the hydrogen content in alpha uranium at low hydrogen pressures are presented in Fig. 17. The values show an abnormally large hydrogen content in biscuit uranium (that saturates at about 4 torr of hydrogen) compared to dingot and high-purity uranium. Cotterill[18] and Davis[19] attribute this behavior to impurities which may be present as a separate phase in the biscuit uranium (possibly as a grain-boundary precipitate) rather than solute impurities. Table 5 compares the hydrogen contents of the three grades of uranium represented in Fig. 17 after equilibration at 600°C and 4 torr of hydrogen. The impurities suspected to be responsible for the anomalous solution behavior are carbon and iron. Evidence that carbon increases the hydrogen solubility is given by the observation that the hydrogen content of uranium (after exposure to high-pressure hydrogen) was much higher than the expected $\sqrt{P}$ dependence above 800°C when the carbon content was more than 700 ppm (ref. 22). A high-pressure phase corresponding to U_3CH_5 was found to be responsible for the higher content. The area surrounding UC inclusions has been observed to be the precipitation site for UH_3, which has been attributed to the fact that UC has an appreciable solubility for hydrogen at high temperatures.

Solutions suitable for the metallographic preparation of uranium are listed in Table 6. A newly polished surface reveals UH_3 as a brown-gray phase and when allowed to tarnish in air, the particles have a "halo" around them. Halos also form around UC, which is distinguished

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[22]R. Smith, The Behavior of Hydrogen in Uranium, AERE-M/R-2799 (February 1959).

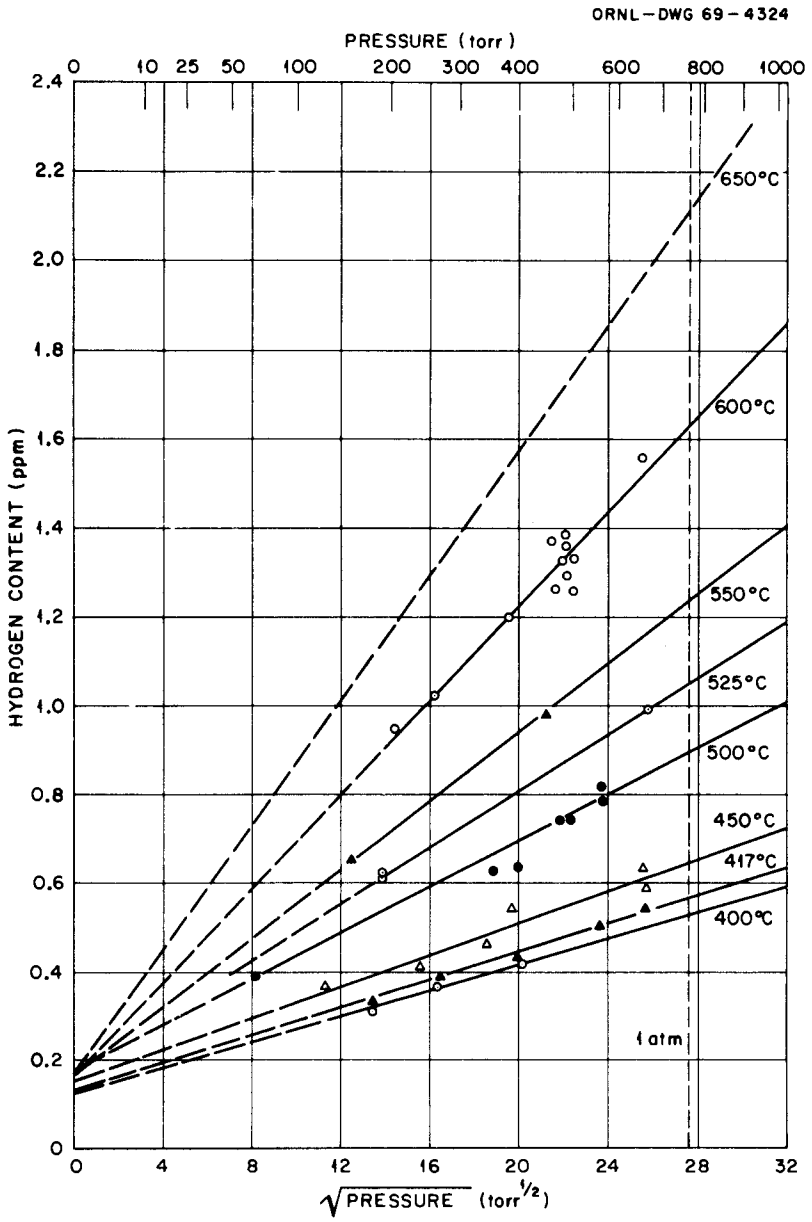

Fig. 15. Hydrogen Content of Alpha Uranium as a Function of Square Root of Hydrogen Pressure. Based on: W. D. Davis, Solubility, Determination, Diffusion, and Mechanical Effects of Hydrogen in Uranium, KAPL-1548 (Aug. 1, 1956).

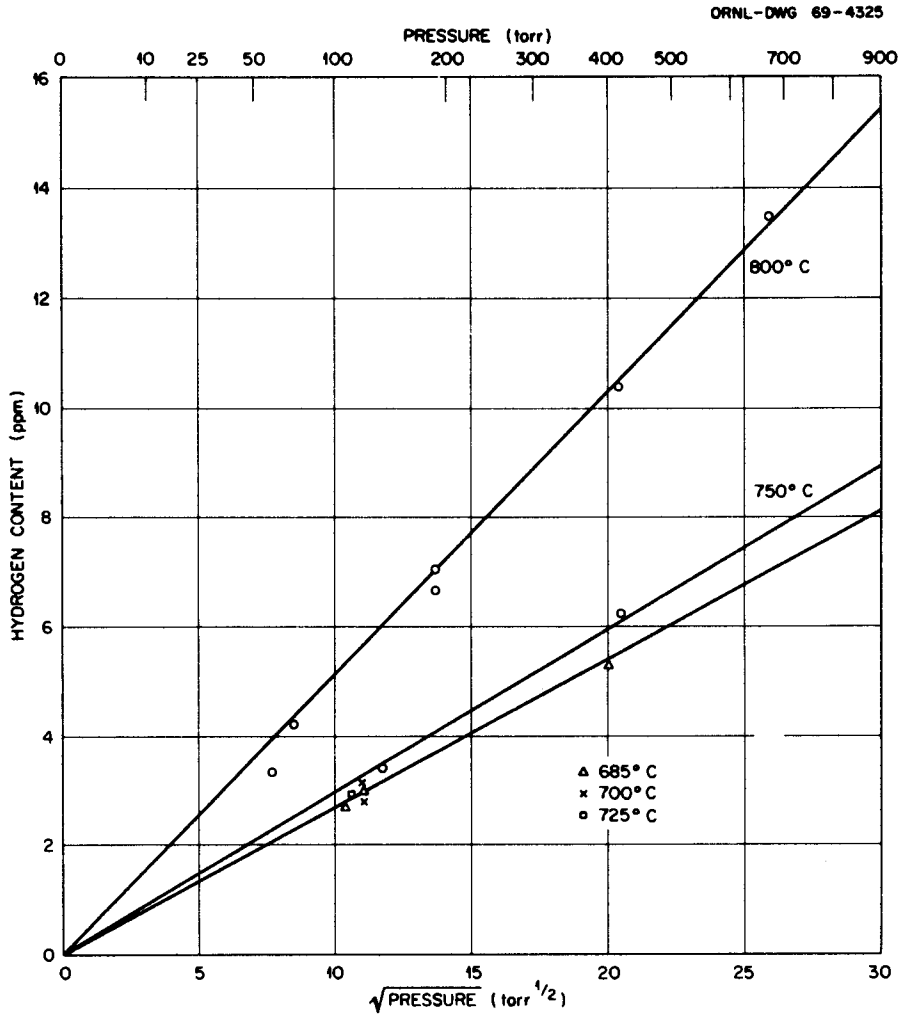

Fig. 16. Hydrogen Content of Beta and Gamma Uranium as a Function of Square Root of Hydrogen Pressure. Based on: W. D. Davis, Solubility, Determination, Diffusion, and Mechanical Effects of Hydrogen on Uranium, KAPL-1548 (Aug. 1, 1956).

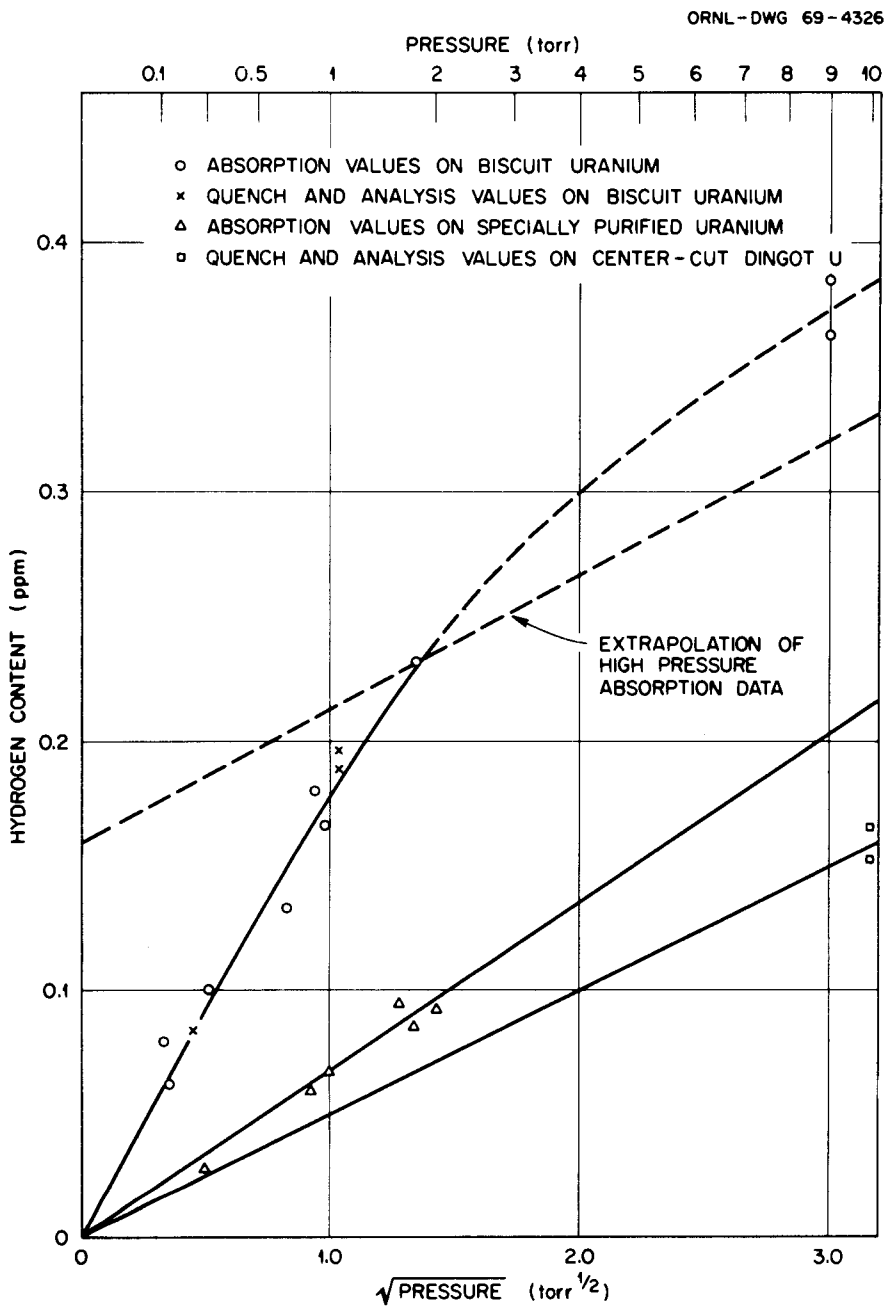

Fig. 17. Hydrogen Content of Biscuit, High-Purity, and Dingot Uranium at 600°C as a Function of Square Root of Hydrogen Pressure. Based on: W. D. Davis, Solubility, Determination, Diffusion, and Mechanical Effects of Hydrogen on Uranium, KAPL-1548 (Aug. 1, 1956).

Table 5. Comparison of the Hydrogen Content of Dingot, High-Purity, and Biscuit Uranium at 600°C and 4 torr of Hydrogen Pressure

Type of Uranium	Analysis, ppm														
	H	C	V	Cr	Si	Mo	Zr	Fe		Al	Mn	Mg	Ca	Cu	N
Dingot	0.09	200			150	40	40	20	20	5	5	3	2	2	60
High purity	0.13	65	0.8	16	35			80	20	5	8	12	< 10	20	
Biscuit[a]	0.31	450	< 10	7	50			> 100	25	20	3	2	< 10	15	

[a]Typical analysis of this type of uranium. Analysis of metal tested was not given.

Table 6. Solutions for Metallographic Preparation of Uranium Surfaces

Operation	Solution Composition	Conditions
Attack polish	100-g chromic acid, 15-ml nitric acid, 100-ml water	Immersion or swab
Electrolytic etch	10-g citric acid, 2.5-ml nitric acid, 500-ml water	0.75 v
Electrolytic polish	100-ml sulfuric acid, 50-ml orthophosphoric acid, 100-ml water	5 to 7 v

from UH_3 by a characteristic blue-gray color. Two forms of UH_3 exist: α-UH_3 is cubic with a_0 = 4.16 A and is considered to be metastable; β-UH_3 is also cubic with a_0 = 6.63 A and an x-ray density of 10.9 g/cm^3. The latter form is stable if formed above room temperature. The heat of formation for the reaction $U + 3/2\ H_2 \rightarrow UH_3$ is 30.8 kcal/mole UH_3 and the dissociation pressure of UH_3 (300 to 600°C) is given by the equation

$$\log P_{H_2}\ (\text{torr}) = -4495/T + 9.25\ . \qquad (5)$$

β-UH_3 is nonstoichiometric and is ferromagnetic below 181°K.

Solubility data indicate that nearly all of the hydrogen in uranium near ambient temperatures is present as UH_3 at equilibrium. The state of the hydrogen in processed uranium, however, can be influenced by heat treatment. Davis[19] and Smith[22] report that UH_3 precipitation can be suppressed by quenching. Beevers and Newman[23] report UH_3 in the microstructures of quenched wires. Based on an investigation of Gardner and Riches,[24] UH_3 was observed in uranium containing approximately 3 ppm H but not in uranium containing 0.12 to 0.36 ppm when quenched from above 600°C at a rate of 380°C/sec. On the other hand, when specimens containing 0.12 to 0.36 ppm H were cooled at a rate of approximately 10^{-3}°C/sec UH_3 was observed in the microstructure.

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[23]C. J. Beevers and G. T. Newman, J. Nucl. Mater. 23, 10–18 (1967).

[24]H. R. Gardner and J. W. Riches, Trans. Am. Soc. Metals 52, 728–747 (1960).

The morphology of UH_3 is influenced by cooling rate and aging time. In general, very fine particles precipitate at grain boundaries, subgrains, and UC inclusions in quenched specimens. Slower cooling rates increase the UH_3 particle size and occasionally large, isolated needles are observed. Under isothermal conditions the UH_3 particles coarsen with aging time, and evidence of continued precipitation at room temperature was observed in specimens aged for one year.[22]

The hydrogen content of uranium can be reduced when the P_{H_2} in the heat-treating environment is less than the equilibrium pressure (e.g., during a vacuum anneal). In this respect, the mode of decomposition of the hydride particles is of interest. Smith[22] reports that the hydride particles gradually decrease in size and number (upon vacuum annealing at 550°C) until after 15 to 20 min they completely disappear. Mogard and Cabane,[25] however, observed voids in specimens vacuum annealed at 600°C which were attributed to the decomposition of hydride particles. Gardner and Riches[24] found unexplained microcracks in uranium degassed in a dynamic vacuum of 10^{-5} torr at 600°C for 15 hr. These cracks had the same shape as UH_3 needles.

Low-Temperature Mechanical Properties of Uranium

Figure 18 compares the effect of various annealing media on the tensile strength and ductility of uranium at room temperature (grain size and strain rate not specified but assumed to be constant). The bar graph shows that the tensile strength is essentially unaffected but large reductions in ductility occur for annealing media such as salt, air, impure helium, and hydrogen. The data of Fig. 19 show that the deleterious effect of the salt bath (35% $LiCO_3$–65% K_2CO_3) can be removed by a 30-min vacuum anneal at 600°C and re-embrittled by a subsequent salt bath anneal, et cetera.[26] The embrittling influence of the various annealing media and the subsequent restoration of the ductility on

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[25] H. Mogard and G. Cabane, Rev. Met. 51, 617 (1954).

[26] G. S. Hanks et al., Effect of Annealing Media on the Mechanical Properties of Uranium, LA-1619 (August 1953).

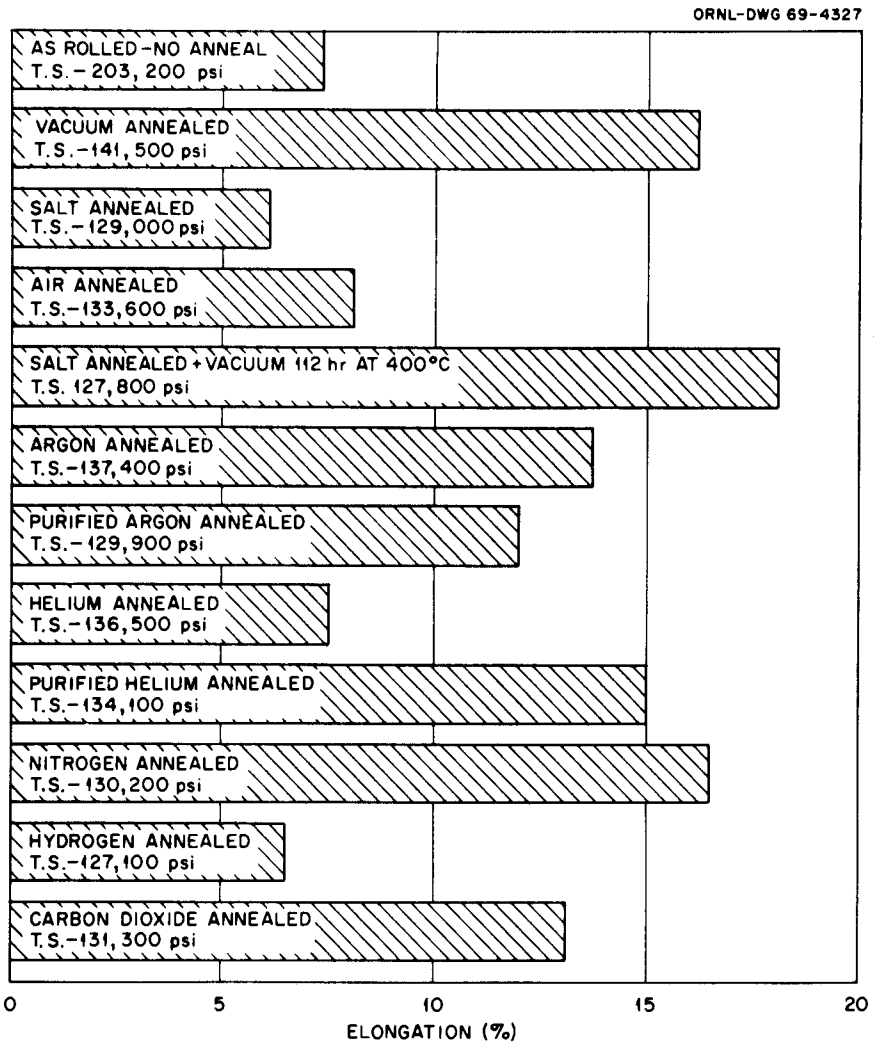

Fig. 18. Ductility of Uranium Annealed in Various Media. Each sample was annealed at 600°C for 30 min in the indicated medium. Normal uranium, lot E-480-A, 0.035-in. sheet, hot rolled 72%, warm rolled 85% at 340°C. Each point is the average of five samples with 2-in. gage lengths. Based on: G. S. Hanks et al., Effect of Annealing Media on the Mechanical Properties of Uranium, LA-1619 (August 1953).

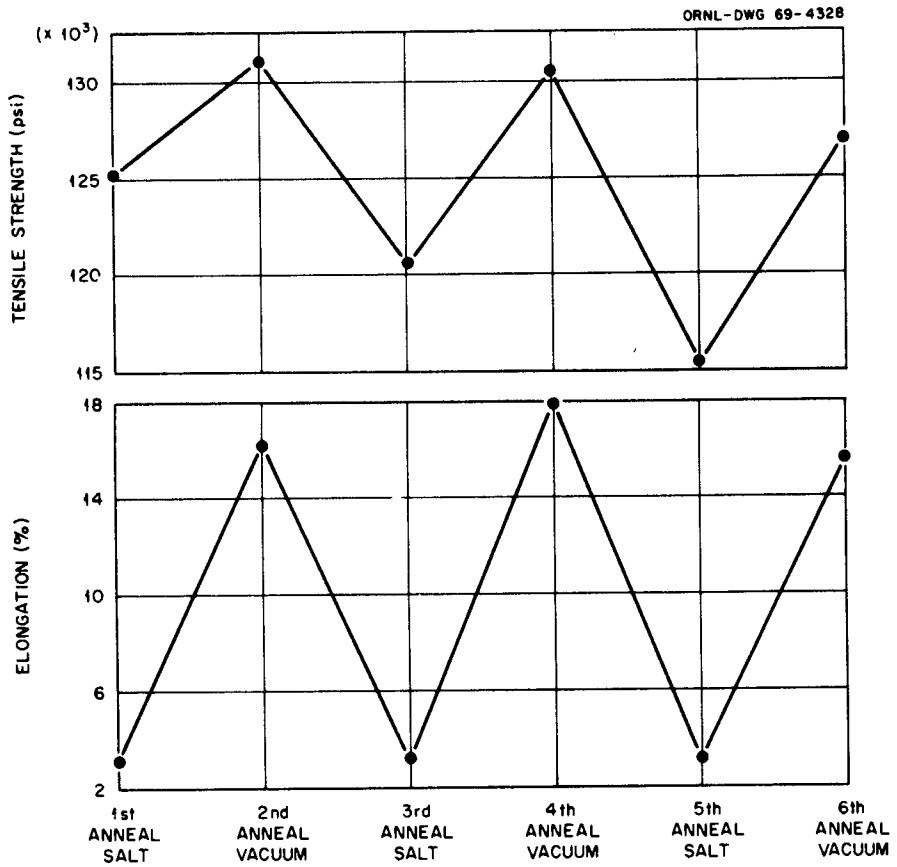

Fig. 19. Ductility of Uranium Alternately Annealed in Salt and Vacuum. Gage length 2 in. All samples were first annealed in salt at 600°C for 30 min. Then three were pulled and the remainder annealed in vacuum for 30 min. Another three were pulled and the remainder annealed in salt, etc. Based on: G. S. Hanks et al., Effect of Annealing Media on the Mechanical Properties of Uranium, LA-1619 (August 1953).

vacuum annealing suggest a gaseous impurity. This impurity has been found to be hydrogen.[27]

Some characteristics of the hydrogen embrittlement resulting from salt bath annealing are as follows: annealing times of 20 to 120 min in 35% $LiCO_3$–65% K_2CO_3 at 600°C show no important difference on the degree of embrittlement according to the data in Table 7 (ref. 26). The tensile data suggest that the hydrogen in uranium attains a saturation or a critical concentration in less than 20 min in sheets 0.035 in. thick. The hydrogen content of these specimens was not determined.

A large fraction of the hydrogen in uranium is introduced early in the fabrication schedule, and subsequent heat treatments have little or no influence on its concentration. As an example, dingot uranium (high purity) rolled to a 1.34-in.-diam rod from a molten 46% Li_2CO_3–54% K_2CO_3 salt then beta quenched after 14 min at 720 to 740°C in Houghton's 980 molten salt (48% $BaCl_2$–28% KCl–24% NaCl) contained 3.3 ppm H. Tensile specimens 0.250 in. in diameter machined from this rod and reannealed four additional minutes at 730°C plus 5 min at 650°C resulted in a hydrogen content of 3.2 ppm H (ref. 24).

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[27] J. T. Waber, Effect of Annealing Practice on the Hydrogen Content of Uranium, LA-1605 (November 1953).

Table 7. Effect of Annealing Time in a Salt Bath on the Tensile Properties of Uranium[a]

Time (min)	Strength, psi		Elongation (%)
	Ultimate Tensile	Yield	
	$\times 10^3$	$\times 10^3$	
20	104.0	47.5	1.8
20	125.1	43.2	7.3
30	125.2	47.9	3.2
120	117.0	39.8	3.5

[a] Salt bath composition was 35% $LiCO_3$–65% K_2CO_3 at 600°C [ref., G. S. Hanks et al., Effect of Annealing Media on the Mechanical Properties of Uranium, LA-1619 (August 1953)]. Specimen thickness 0.035 in. sheet.

The distribution of hydrogen in as-rolled uranium is not uniform as Table 8 shows. The listed data are based on analyses of a rod originally 1.355 in. in diameter that was machined to the tabulated diameters. The higher hydrogen content near the surface is attributed to the presence of a large number of microcracks introduced by the quenching stresses, machining tears, and rolling defects.[28] The presence of discontinuities near the surface is supported by the data in Table 9 which shows that the ductility of uranium sheet increases with the specimen thickness. This dependence should be evident in both vacuum-annealed or salt annealed specimens. The lower ductility of the corresponding salt annealed specimen shows that the embrittling effect of hydrogen becomes more pronounced as the specimen thickness decreases. This behavior is consistent with the observation that the hydrogen is concentrated at the metal surface.

As indicated previously (Fig. 19) the tensile ductility of salt annealed uranium can be dramatically improved by vacuum annealing. The effect of the annealing time at 550°C in vacua of 10^{-3} torr on the ductility of rod-type specimens of uranium is shown by the data in Table 10 and for an argon atmosphere[29] in Table 11.

[28] W. E. Ray and H. C. Bowen, The Hydrogen Content of Fabricated Uranium, HW-30116 (Nov. 30, 1953).

[29] P. D. Tilbury, Experiments to Improve the Mechanical Properties of Wrought Uranium, AWRE Report No. 038/68.

Table 8. Hydrogen Gradient in a Hot-Rolled Uranium Rod[a]

Sample Diameter (in.)	Hydrogen-to-Uranium Ratio	Hydrogen (ppm)
1.355	0.352	1.49
1.305	0.302	1.28
1.253	0.255	1.08
1.204	0.246	1.04
1.154	0.235	0.99
1.100	0.235	0.99

[a] W. E. Ray and H. C. Bowen, The Hydrogen Content of Fabricated Uranium, HW-30116 (Nov. 30, 1953).

Table 9. Room-Temperature Tensile Properties of Uranium Sheet as a Function of Thickness[a]

Lot Number	Specimen Thickness (in.)	Anneal	Temperature (°C)	Time (min)	Strength, psi		Percent Elongation (2 in.)	Number of Tests Averaged
					Ultimate Tensile	0.2% Yield		
					$\times 10^3$	$\times 10^3$		
D-417[b]	0.119	Salt	600	45	102.5	38.7	14.3	5
E-235[c]	0.049	Salt	600	30	125.2	47.5	8.9	5
E-235	0.036	Salt	600	20	104.3	47.5	1.8	4
D-417	0.119	Vacuum	600	45	115.0	41.0	38.7	5
E-235	0.049	Vacuum	600	30	132.4	49.9	24.2	5
E-235	0.036	Vacuum	575	30	131.3	45.2	19.0	4

[a]G. S. Hanks et al., Effect of Annealing Media on the Mechanical Properties of Uranium, LA-1619 (August 1953).

[b]Analysis in parts per million: 330 C, 4 Mg, 2 Al, 40 Si, < 10 V, 8 Cr, 4 Mn, > 100 Fe, 10 Ni, and 15 Cu.

[c]Analysis in parts per million: 310 C, 3 Mg, 5 Al, 30 Si, < 10 V, 5 Cr, 4 Mn, > 100 Fe, 30 Ni, and 5 Cu.

Table 10. Effects of Vacuum-Annealing Time at 550°C on Tensile Properties of Uranium[a]

Time (hr)	Tensile Strength (psi)	Percent Elongation	Reduction in Area (%)
	$\times 10^3$		
0	101.0	10.6	11.4
3	109.0	32.0	31.0
5	108.2	34.1	36.4
10	104.4	42.6	41.5
15	104.6	43.2	47.2
20	108.0	38.1	33.6
30	103.8	35.5	36.2
40	102.4	39.2	41.1

[a]British Standard No. 5 Tensile specimens (dimensions not specified).

Table 11. Effects of Argon Annealing Time at 550°C on Tensile Properties of Uranium[a]

Time (hr)	Tensile Strength (psi)	Percent Elongation	Reduction in Area (%)
	$\times 10^3$		
0	101.0	10.6	11.4
10	101.2	12.7	11.0
20	108.4	32.7	29.0
30	108.8	29.6	26.6
50	107.8	34.9	33.8

[a]British Standard No. 5 Tensile Specimens (dimensions not specified).

Assuming that the ductility improvement is due to the decrease in the hydrogen content, these data show that the degassing rate of uranium is greater in vacua than in argon and that 15 hr in vacuum appears to be the optimum. Figure 20 shows the effect of the vacuum-annealing temperature on the ductility of uranium which was previously salt annealed.[30] Annealing temperatures between 100 and 150°C reduce the ductility of salt annealed specimens still further. Beginning at about 200°C, the ductility improves rapidly with temperature and reaches a maximum at about 400°C.

Table 12 is one of the first sets of data that establish the relationship between the tensile properties of uranium and the hydrogen content.[30] The ductility as a function of the hydrogen content is shown in Fig. 21 (curve I) together with the data of other investigators for comparative purposes. The data of curves II and III are based on bar specimens of biscuit uranium, beta quenched, and reheated either in vacua or hydrogen at 600°C. The material representing curve II contained about 450 ppm C. The data of curve III are based on high-purity uranium containing 37 ppm C. Curve IV represents the ductility of biscuit uranium heat treated at different temperatures.[31] The specimen

[30]G. S. Hanks et al., Effect of Annealing Media on the Mechanical Properties of Uranium, LA-1619 (August 1953).

[31]L. L. Marsh, G. T. Muehlenkamp, and G. K. Manning, Effect of Hydrogen on the Tensile Transition in Uranium, BMI-980 (Feb. 14, 1955).

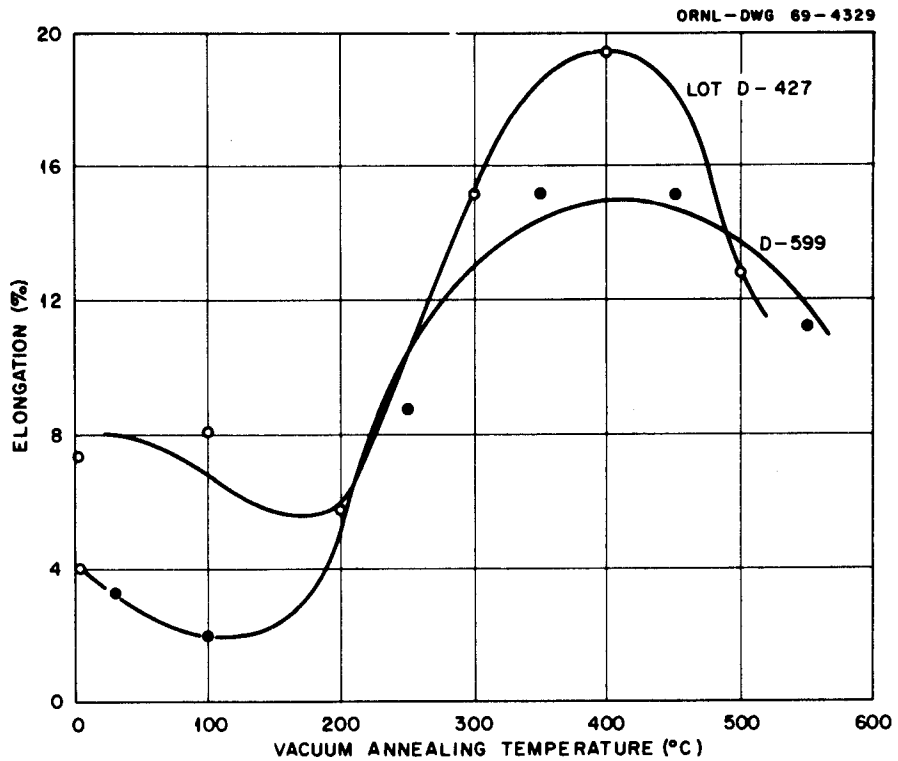

Fig. 20. Effect of Vacuum Annealing Temperature on Ductility of Salt-Annealed Uranium. Specimens were annealed 10 hr at indicated temperature in vacuum after a 30-min anneal in salt at 600°C. Gage length 2 in. Based on: G. S. Hanks et al., Effect of Annealing Media on the Mechanical Properties of Uranium, LA-1619 (August 1953).

containing the lowest hydrogen content (assumed to be about 0.1 ppm) was gamma annealed in vacuum at 938°C; the 0.3 ppm specimen was vacuum annealed at 600°C, water quenched, and aged 1 hr at 350°C; the 0.8 ppm specimens were tested after rolling at 550°C (27% elongation) or alpha annealed 10 hr at 600°C (8% elongation). It is apparent from Fig. 21 that hydrogen is not solely responsible for the embrittlement of uranium. Other important factors appear to be the carbon content, grain size, state of the hydrogen (see data points of curve IV containing 0.8 ppm H), the strain rate, test temperature, and the likely presence of microcracks. Beevers and Newman report that the maximum embrittlement of uranium occurs at a hydrogen content of about 2.5 ppm in quenched wires[32] for a given grain size and a wide range of strain rates to reinforce the observation that factors other than the hydrogen content contribute to uranium embrittlement.

[32] C. J. Beevers and G. T. Newman, J. Nucl. Mater. 23, 10–18 (1967).

Table 12. Room-Temperature Tensile Properties of Three Lots of Uranium Containing Hydrogen[a]

Lot Number	Anneal	Temperature (°C)	Time (min)	Hydrogen Content (ppm)	Strength, psi Ultimate Tensile	Strength, psi 0.2% Offset	Percent Elongation (2 in.)
					$\times 10^3$	$\times 10^3$	
D-544-B[b]	Salt	600	30	1.50	115.1	40.4	4.5
D-544-B	Vacuum	600	30	0.50	133.0	47.0	21.6
D-550-B[c]	Hydrogen	575	30	1.80	134.7	66.1	4.3
D-550-B	Vacuum	600	30	0.53	144.0	64.5	12.8
E-235-4[d]	Salt	600	30	0.72	125.2	47.5	8.9
E-235-4	Vacuum	600	30	0.32	132.4	49.9	24.2

[a]G. S. Hanks *et al.*, *Effect of Annealing Media on the Mechanical Properties of Uranium*, LA-1619 (August 1953).

[b]0.035 in. sheet: analysis in parts per million – 320 C, 2 Mg, 2 Al, 20 Si, < 10 Ca, < 10 V, 4 Cr, 3 Mn, 70 Fe, 15 Ni, and 2 Cu.

[c]0.035 in. sheet: normal uranium, no analysis.

[d]0.049 in. sheet: analysis in parts per million – 310 C, 3 Mg, 5 Al, 30 Si, < 5 Ca, < 10 V, 5 Cr, 4 Mn, > 100 Fe, 30 Ni, and 5 Cu.

Sources of Hydrogen in Uranium

The major source of hydrogen in heat-treated uranium appears to be the water vapor in the annealing media (see Fig. 18). Hydrogen is also derived from pickling baths[30] and electrolysis.[32] Metallic uranium and water (or steam) form UO_2 and hydrogen according to the reaction

$$U + 2\ H_2O \rightarrow UO_2 + 2\ H_2 \qquad (6)$$

The equilibrium constant for the reaction given in Eq. (6) is 5×10^{15} at 600°C based on the free energies of formation of UO_2 and H_2O. Thus, the conversion of water vapor to hydrogen at equilibrium is complete. This reaction proceeds at a constant rate between 35 and

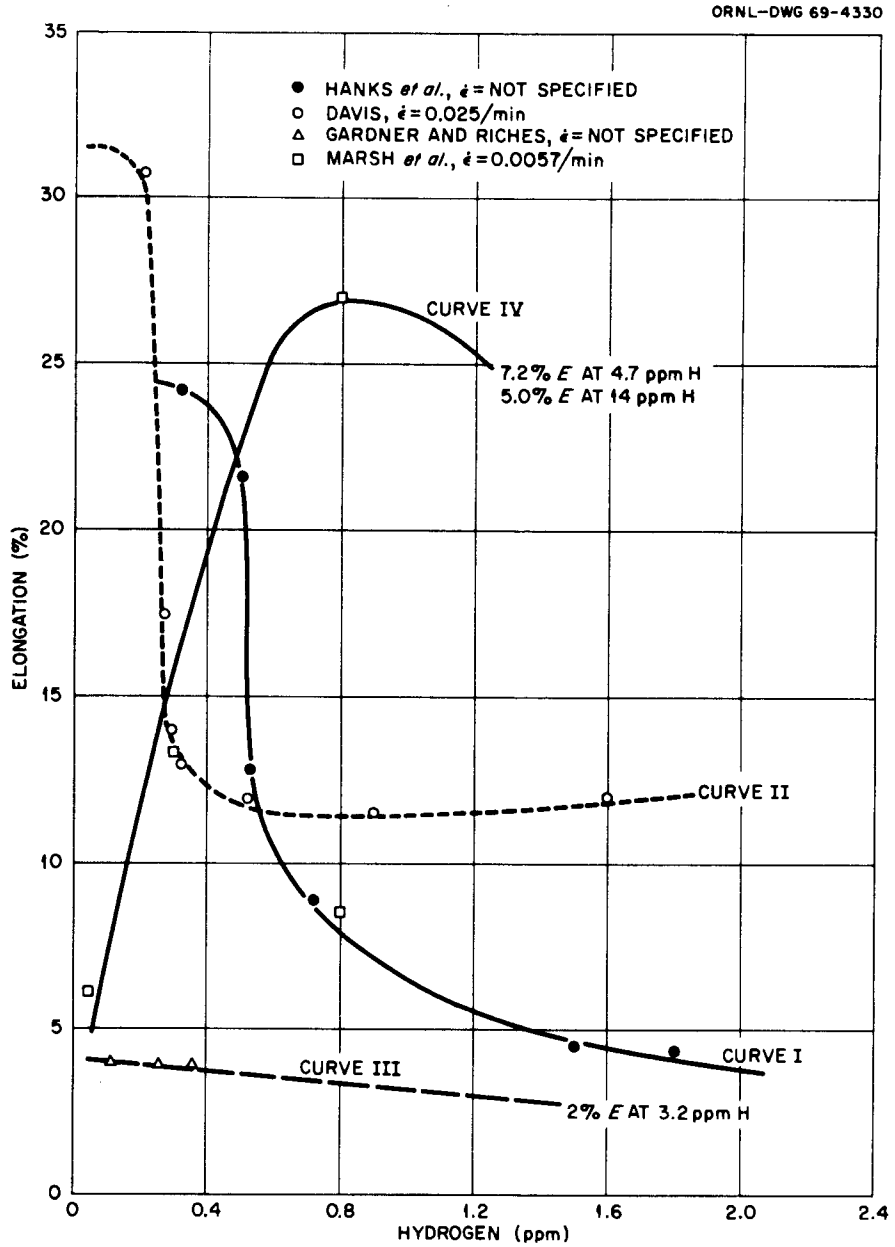

Fig. 21. Effect of Hydrogen Content on Elongation of Uranium at Room Temperature. Based on: W. D. Davis, Solubility, Determination, Diffusion, and Mechanical Effects of Hydrogen in Uranium, KAPL-1548 (Aug. 1, 1956); G. S. Hanks et al., Effect of Annealing Media on the Mechanical Properties of Uranium, LA-1619 (August 1953); L. L. Marsh, G. T. Muehlenkamp, and G. K. Manning, Effect of Hydrogen on the Tensile Transition in Uranium, BMI-980 (Feb. 14, 1955); and H. R. Gardner and J. W. Riches, Trans. Am. Soc. Metals 52, 728–747 (1960).

240°C (ref. 33) and between 600 and 1200°C the rates are parabolic.[34] The generation rate of hydrogen at the uranium surface in 1 atm of steam between 600 and 1200°C is given by

$$V^2 = 1.95 \times 10^{-5}\ t\ \exp(-18{,}600/RT)\ , \qquad (7)$$

where

V = volume hydrogen evolved, ml(STP)/cm^2, and

t = reaction time, minutes.

The liberated hydrogen is then dissolved by uranium.

The water vapor must be soluble in salt baths as evidenced by the hydrogen pickup in this media. As a first approximation alpha-annealed uranium in a salt bath contains about 2 ppm H at saturation[30] and beta-annealed uranium about 3.3 ppm H (ref. 35). From Sievert's solubility relationship and the value of S_0 (Table 4) the effective hydrogen pressure in the salt bath is calculated to be a surprising 1.12 atm at 600°C and 0.675 atm at 680°C.

SUMMARY

Uranium exhibits a ductile-to-brittle transition behavior in the range 100 to −50°C. Since the sharpness and the temperature of the transition in ductility are sensitive to the processing history and the testing procedures, large variations in the ductility can occur in the same material at room temperature.

In general the ductile-to-brittle transition temperature increases with the grain size (except Figs. 13 and 14) and the strain rate. At a given temperature, the ductility decreases as these variables increase. Variations in the grain structure introduced by phase transformations

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[33] M. Baker et al., Trans. Faraday Soc. 62(5), 2513–2524 (1966).

[34] R. E. Wilson et al., Nucl. Sci. Eng. 25, 109–115 (1966).

[35] H. R. Gardner and J. W. Riches, Trans. Am. Soc. Metals 52, 728–747 (1960).

and fabrication procedures also influence the ductility of uranium. Although impurities such as aluminum, silicon, and carbon at concentrations less than 1000 ppm refine the uranium grains, the ductility is lowered by their presence.

The probable state of most of the hydrogen in uranium at room temperature is β-UH_3 whose morphology and location vary with the hydrogen content and heat treatment. Carbon appears to be an important companion impurity because hydrogen has an appreciable solubility in UC.

In general, the ductility of uranium for a given heat treatment and strain rate decreases drastically when the hydrogen exceeds a threshold content of about 0.2 to 0.5 ppm; however, because the ductility varies significantly with the heat treatment and the carbon content, it is concluded that the morphology of UH_3 also plays an important role in the embrittlement. Microcracks generated from fractured UH_3 particles or degassed UH_3 could be another important variable; however, evidence of their contribution to embrittlement is indirect. The hydrogen embrittlement of uranium is further complicated by several equally important factors discussed in Part I.

The hydrogen in heat-treated uranium is derived from the reaction of uranium with water vapor in the annealing media. Based on the typical values of hydrogen in uranium, the effective hydrogen pressure at the heat-treating temperature due to the water vapor can be over 1 atm.

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