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Resistance-Temperature Relation and Thermoelectric Properties of Uranium [1]

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The paper describes measurements made on the resistance-temperature relation and the thermoelectric properties of several samples of uranium. The resistance-temperature relation showed sharp breaks at about 650° and 770° C, indicating the existence of transformation points. The very rapid change in resistance occurred at a somewhat higher temperature on heating than on cooling. Only slight irregularities in the thermoelectric properties were noted in the region of the transformation points.

I. Introduction

In connection with the work of the Manhattan District, Corps of Engineers, United States Army, on the development of atomic energy, the National Bureau of Standards was requested among other things to measure certain physical properties of uranium. This paper describes those portions of the work done in the pyrometry laboratory of the Bureau's Temperature Measurements Section on the resistance-temperature relation, the location of the transformation points, and the thermoelectric properties of uranium.

II. Resistance-Temperature Relation of Uranium

The electrical resistance of all pure or nearly pure metals increases with temperature. The temperature coefficient of electrical resistance is often used as a criterion for purity of the metal. In general, the higher the coefficient, the higher the purity, providing the samples have been subjected to the same physical treatment. The relatively high sensitivity of this test, together with the comparatively simple application to metals in the form of wire or strip, makes it very useful in determining the relative purities of samples of the same metal.

For some metals the resistance-temperature curve is known to exhibit irregularities at transformation points. In the case of nickel and some of its alloys, a change in slope occurs at the magnetic transformation point. Burgess and Kellberg [2] used this phenomenon to locate the critical ranges of high-purity iron. Their work indicates that the method is reliable and sensitive to a high degree. The test is particularly applicable to material in wire form and therefore was suggested for the study of the transformations in uranium.

No report of previous work on the resistance-temperature relation of uranium has been found in the literature.

1. Materials

Samples taken from five lots of uranium were available for the present work. The metal was obtained from three sources and ranged in purity from about 99.85 to 99.94 percent with carbon and iron as the major impurities. The preparation of the samples from the cast ingots into wires approximately 0.03 in. in diameter was carried out in the Metallurgy Division of the Bureau.[3]

2. Experimental Procedure

A diagram of the experimental setup is shown in figure 1. A 15-in. length of uranium wire, 0.03 in. in diameter was bent double, as shown in figure 1. Temperature gradients within the furnace were minimized by a heavy-walled silver tube extending to within 1 in. of the end of the furnace

[1] The information covered in this paper will appear in volume 11B, Division VIII, of the Manhattan Project Technical Series as part of the contribution of the National Bureau of Standards.

[2] G. K. Burgess and I. N. Kellberg, Bul. BS 11, 457 (1915) S236.

[3] Acknowledgment is made to H. E. Cleaves for this work.

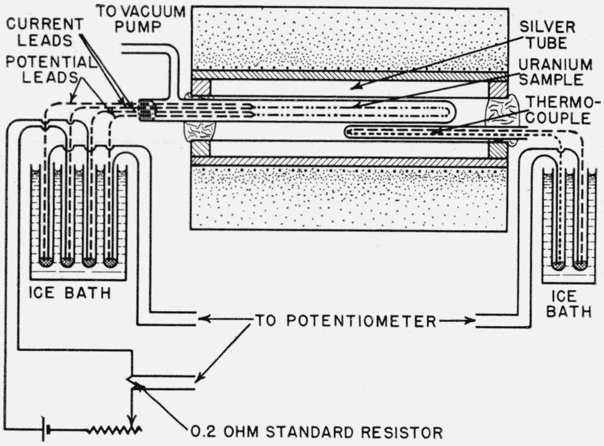

FIGURE 1.—*Diagram of apparatus*

tube. Two platinum—10-percent rhodium wires, 0.015 in. in diameter, were spot-welded to each end of the sample, one of each pair serving as current leads and the other as potential leads. The sample and a portion of the leads were contained in a gastight porcelain protection tube, the leads extending through a wax seal to the measuring circuit. To prevent oxidation of the sample during heating, the porcelain tube was evacuated, the pressure being maintained at <0.001 mm Hg.

The electrical resistance of the test specimen was obtained by comparing the potential drop across the specimen with the potential drop across a 0.2-ohm resistor in series with the specimen. A current of 10 ma was maintained in the circuit. The temperature of the specimen was measured by means of a Pt–Pt-Rh thermocouple located close to the specimen. The measurement of the resistance of the specimen at 0° C was made with the porcelain tube containing the sample immersed in an ice bath. The specimen was under atmospheric pressure during this test. Simultaneous readings of the resistance and the temperature of the specimen were obtained by the use of two potentiometers, one measuring the emf of the thermocouple and the other the potential drop across the test specimen. The galvanometer deflections were registered on a single scale.

The uranium wire samples as received had been annealed in helium for 30 minutes at 600° C. Preliminary tests showed that this treatment had not completely stabilized the electrical properties at temperatures above 600° C. Therefore, the samples were further annealed in vacuo for 30 minutes at 910° C, the heating being carried out after the specimen had been mounted in the apparatus shown in figure 1. In order to obtain a measure of the change brought about by this additional heat treatment, the resistance of the sample at 0° and 100° C was determined before and after the anneal at 910° C.

Preliminary observations on a number of specimens revealed that abrupt changes in electrical resistance occurred in the ranges of about 640° to 670° C and 760° to 775° C. For this reason observations were made at temperature intervals varying from about 0.5 to 2 deg C over these ranges. Outside of these temperature ranges observations were made at about 50° to 100 deg C intervals. The rate of temperature rise or fall during an observation did not exceed 1 deg C a minute.

3. Results

The resistance of the test specimen ranged from about 0.18 to 0.21 ohm at 0° C. The use of the ratio R_t/R_0 rather than the absolute value of the resistance facilitates the intercomparison of the changes in resistance of the specimen with temperature. Figure 2 shows the values of R_t/R_0 for the various samples. It should be noted that the vertical scale has been shifted by various amounts for the different specimens to permit all observations to be plotted without having the curves cross.

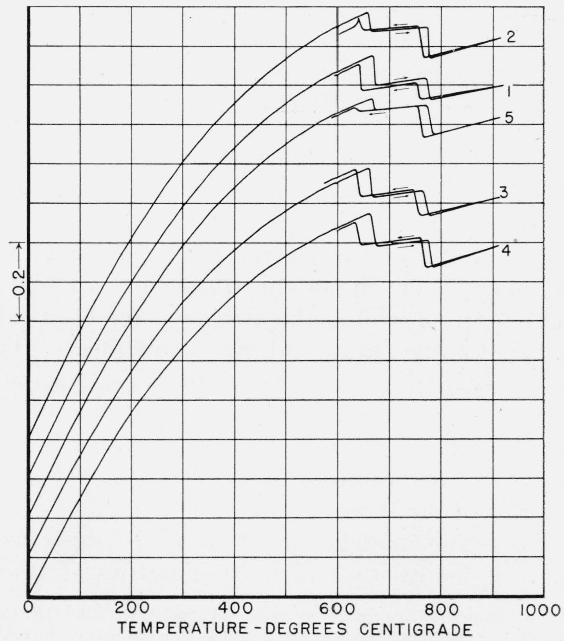

FIGURE 2.—*Resistance-temperature relation for uranium.*

Using the curve for sample 1 as an example, it is seen that the value of R_t/R_0 (necessarily 1 at $t=0°$ C for all samples) increased at a gradually decreasing rate until a temperature of about 670° C was reached. At this temperature an abrupt decrease in R_t/R_0 occurred, the value changing from 2.070 to 2.000 over a temperature interval of 5° C. Upon further heating R_t/R_0 again increased with temperature but at a linear and somewhat lower rate than at temperatures below 670° C. At 772° C, another sharp decrease in R_t/R_0 occurred, the value changing from 2.017 to 1.945 over a 5 deg C temperature interval. Thereafter a linear increase in R_t/R_0 continued to 900° C, the highest temperature at which observations were made. Resistance-temperature measurements made on cooling from 900° C closely approximated those obtained on heating, except that the abrupt changes in resistance occurred at somewhat lower temperatures, namely 760° and 644° C. Subsequent runs with the same sample duplicated closely the original resistance-temperature curve.

Table 1 gives the values of the temperature coefficient, $(R_{100}-R_0)/100R_0$, of the samples before and after the anneal in vacuo for 30 minutes at 910° C.

TABLE 1.—*Mean temperature coefficient, 0° to 100° C*

Sample	Temperature coefficient $(R_{100}-R_0)/100R_0$: After 30 minutes at 600° C	Temperature coefficient $(R_{100}-R_0)/100R_0$: After 30 minutes at 910° C
1	0.00282	0.00271
2	281	272
3	268	249
4	270	246
5	278	265
Mean value	.00276	.00261

Table 2 gives corresponding values of temperature, R_tR_0, and the temperature coefficient $(1/R_0)(dR_t/dt)10^3$ for each of the specimens obtained on heating.

The temperature at which the resistance-temperature curve began to show a definite change in slope was taken as the transformation point. The transformation at the lower temperature is known as the alpha-beta transformation and that at the higher temperature as the beta-gamma transformation. Hysteresis averaged 8 deg C at the beta-gamma and 22 deg C at the alpha-beta transformation.

TABLE 2.—*Resistance-temperature relation of uranium, 0° to 900° C*

Temperature	Sample 1		Sample 2		Sample 3		Sample 4		Sample 5	
	$\frac{R_t}{R_0}$	$\frac{1}{R_0}\frac{dR_t}{dt}\times10^3$	$\frac{R_t}{R_0}$	$\frac{1}{R_0}\frac{dR_t}{dt}\times10^3$	$\frac{R_t}{R_0}$	$\frac{1}{R_0}\frac{dR_t}{dt}\times10^3$	$\frac{R_t}{R_0}$	$\frac{1}{R_0}\frac{dR_t}{dt}\times10^3$	$\frac{R_t}{R_0}$	$\frac{1}{R_0}\frac{dR_t}{dt}\times10^3$
0	1.000	2.89	1.000	2.92	1.000	2.72	1.000	2.68	1.000	2.83
50	1.140	2.69	1.142	2.73	1.131	2.52	1.130	2.48	1.136	2.64
100	1.269	2.49	1.274	2.54	1.252	2.32	1.248	2.28	1.264	2.46
150	1.388	2.29	1.396	2.34	1.363	2.13	1.358	2.08	1.382	2.28
200	1.498	2.10	1.508	2.14	1.464	1.94	1.456	1.89	1.492	2.09
250	1.599	1.91	1.610	1.93	1.557	1.76	1.548	1.71	1.591	1.90
300	1.690	1.71	1.701	1.72	1.641	1.57	1.628	1.53	1.682	1.71
350	1.770	1.51	1.782	1.52	1.715	1.37	1.701	1.35	1.762	1.52
400	1.839	1.31	1.853	1.33	1.778	1.18	1.763	1.17	1.834	1.33
450	1.899	1.13	1.915	1.15	1.832	1.00	1.818	1.00	1.895	1.14
500	1.951	0.96	1.968	0.97	1.878	0.85	1.863	0.85	1.948	0.96
550	1.995	.80	2.012	.80	1.917	.71	1.902	.71	1.992	.79
600	2.031	.65	2.048	.65	1.950	.58	1.934	.58	2.027	.64
650	2.061	.51	2.077	.49	1.976	.46	1.960	.45	2.056	.50
700	2.004	.18	2.040	.15	1.921	.16	1.894	.16	2.038	.14
750	2.013	.18	2.047	.15	1.929	.16	1.902	.16	2.045	.14
800	1.952	.43	1.974	.45	1.870	.42	1.840	.39	1.971	.41
850	1.974	.43	1.996	.45	1.891	.42	1.859	.39	1.992	.41
900	1.995	.43	2.019	.45	1.912	.42	1.879	.39	2.012	.41

Table 3 summarizes the observations on the transformation temperatures of uranium samples.

TABLE 3.—*Transformation temperatures of uranium*

Sample	Alpha-beta transformation		Beta-gamma transformaton	
	On heating	On cooling	On heating	On cooling
	$^\circ C$	$^\circ C$	$^\circ C$	$^\circ C$
1	670	644	772	760
2	669	651	774	766
3	666	645	766	761
4	665	644	776	770
5	667	643	773	764
Mean value	667	645	772	764

4. Discussion of Results

No correlation of the resistance-temperature data with the purity of the samples has been established. The mean value of 0.00261 for the temperature coefficient over the fundamental interval, 0° to 100° C, for uranium annealed at 910° C is considerably lower than that reported for most high-purity metals. Furthermore, the temperature coefficient of most metals increases with high-temperature annealing, while in the case of uranium the coefficient decreases. There is a possibility that the uranium samples reduced some of the silica of the porcelain tube during the annealing at 910° C and were contaminated by the absorption of oxygen or silicon or both. This would account for lowering of the temperature coefficient. On the basis of the analyses of the original lots of material, sample 5 has the highest purity.

5. Summary

The resistance-temperature relation for 99.9 percent uranium has been determined over the range 0° to 900° C.

The average value of the temperature coefficient of electrical resistance, $(R_{100}-R_0)/100R_0$ for five samples of 99.9 percent uranium has been found to be 0.00276 for metal annealed at 600° C and 0.00261 for metal annealed at 910° C.

The irregularities occurring in the resistance-temperature curve for uranium have been used as a basis for locating the transformation points in uranium. The values for the transformation temperatures are

ON HEATING		ON COOLING	
Alpha-beta	667° C	Gamma-beta	764° C
Beta-gamma	772° C	Beta-alpha	645° C

III. Thermoelectric Properties of Uranium

The thermal electromotive-force (hereafter written as emf) of a material is generally determined by comparison thermoelectrically with pure platinum.

In general, the effect of a given change in the amount of impurity upon the thermal emf of a nearly pure metal increases as the purity is increased. For this reason, the thermal emf is often used as a test of the relative purity of samples of the same metal. The test is capable of high precision. For example, the thermoelectric properties of a sample of platinum give an even more senstitive indication of the purity of the material than spectroscopic tests. The smallest amount of impurity that can be detected spectroscopically in platinum corresponds to a change in the thermal emf of the material of 10 to 20μ at 1,200° C. The sensitivity of this test depends somewhat on the type of impurities present.

No report of previous work on the thermoelectric properties of uranium has been found in the literature.

1. Materials

Samples from the same lots of uranium as were used in the resistance-temperature tests reported above were used for the thermoelectric tests. The uranium was in the form of 0.03-in.-diameter wire.

2. Methods

The thermal emf of the uranium samples was determined against pure platinum as the reference standard. One end of the sample of uranium wire under test was spot-welded to one end of the platinum working standard. The platinum uranium thermocouple thus formed, contained in a porcelain protection tube, was placed in the tube furnace shown in figure 1. As uranium oxidizes readily, even at moderate temperatures, it was heated in vacuo. The pressure within the tube was maintained at <0.001 mm Hg during the test. The free ends of the platinum-uranium couple protruded through a wax seal and extended to the reference junction maintained at 0° C by means of an ice bath. Copper wires completed the circuit to the potentiometer. The temperature of the platinum-uranium junction was measured by means of a standard platinum to platinum-rhodium thermocouple placed adjacent to the junction.

Preliminary measurements on the emf of a platinum-uranium thermocouple showed small irregularities in slope in the region of the transformation points, when observations were made at intervals of 1 to 2 deg in these regions. However, it did not appear that this was a satisfactory method of locating transformation points, and no further investigation was made. Subsequent observations on the other samples were taken at 50-deg intervals from 0° to 900° C.

The observations of corresponding values of emf and temperature on the samples of uranium are plotted in figure 3. Uraninum is thermoelectrically positive to platinum on the basis of the generally accepted convention, namely: if, in a simple thermoelectric circuit the current flows from metal *A* to metal *B* at the colder junction, *A* is thermoelectrically positive to *B*.

3. Results

Such irregularities that may exist in the emf-temperature relation at the transformations were of such small magnitude as not to be discernable on the graphs shown in figure 3, or in figure 4, in

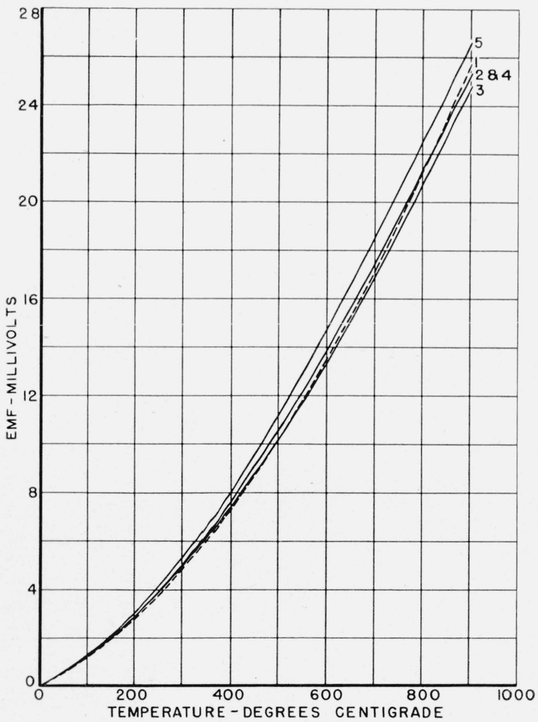

FIGURE 3.—*Thermal electromotive force of uranium versus platinum.*

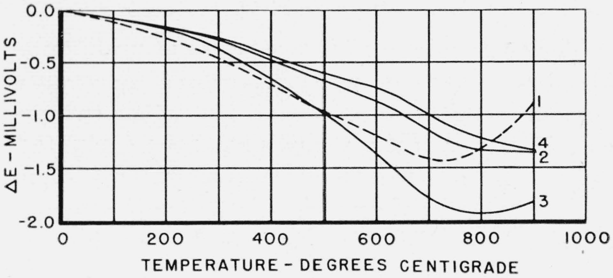

FIGURE 4.—*Differences in thermal electromotive force of uranium samples.*

which the differences in the thermal emf of each sample from sample 5 are plotted.

The relatively large effect of small variations in purity upon the thermal emf of a pure metal is illustrated in figure 4. A difference of about 1.92 mv at 800° C between the thermal emf of samples 3 and 5 is presumably due to differences of about 0.07 percent of Fe and 0.02 percent of O_2, the other impurities in the two samples being about the same. Table 4 summarizes the thermal emf of the various samples versus platinum. It is believed that values for sample 5 most nearly represent the thermal emf of pure uranium versus platinum. The last column of table 4 gives the mean value of the thermoelectric power de/dt for all the samples.

TABLE 4.—*Thermoelectric properties of uranium with respect to platinum*

Temperature	Electromotive force versus platinum					de/dt [a]
	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	
°C	*mv*	*mv*	*mv*	*mv*	*mv*	*μv/° C*
0	0.00	0.00	0.00	0.00	0.00	12
50	.52	.54	.54	.53	.58	14
100	1.15	1.19	1.19	1.17	1.27	17
150	1.89	1.96	1.96	1.94	2.08	19
200	2.76	2.86	2.85	2.85	3.02	22
250	3.75	3.88	3.85	3.89	4.10	24
300	4.85	5.02	4.93	5.04	5.30	26
350	6.04	6.26	6.11	6.28	6.62	28
400	7.33	7.57	7.38	7.61	8.04	30
450	8.72	8.99	8.75	9.05	9.56	32
500	10.23	10.51	10.21	10.59	11.18	34
550	11.84	12.14	11.75	12.24	12.90	35
600	13.54	13.87	13.37	13.99	14.72	36
650	15.32	15.65	15.07	15.79	16.64	37
700	17.15	17.43	16.81	17.60	18.57	38
750	19.10	19.26	18.64	19.41	20.52	40
800	21.19	21.18	20.59	21.29	22.51	41
850	23.40	23.19	22.64	23.27	24.53	42
900	25.69	25.23	24.75	25.25	26.58	43

[a] Mean value for all samples.

4. Summary

The thermal emf of five samples of uranium of about 99.9 percent purity have been determined over the range 0° to 900° C. The mean thermoelectric power of the samples has been computed.

Uranium has been found to exhibit only slight irregularities in thermoelectric properties at the alpha-beta and beta-gamma transformations.

Variations in the amount and nature of the impurities, when the total impurity is of the order of 0.1 percent, have been found to have a considerable effect on the thermal emf of uranium.

WASHINGTON, April 21, 1947.