

Melting Point, Normal Spectral Emittance (at the Melting Point), and Electrical Resistivity (above 1900 K) of Titanium by a Pulse Heating Method*

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A subsecond duration pulse heating method was used to measure the melting point, the normal spectral emittance (at the melting point), and the electrical resistivity (above 1900 K) of 99.9 + percent pure titanium. The results, based on the International Practical Temperature Scale of 1968, yield a value of 1945 K for the melting point. The normal spectral emittance (at 653 nm) at the melting point is 0.40. Estimated inaccuracies are: 5 K in the melting point, 5 percent in the normal spectral emittance, and 3 percent in the electrical resistivity.

Key words: Electrical resistivity; emittance; high-speed measurement; high temperature; melting point; radiance temperature; titanium.

1. Introduction

Because of the reactive nature of the Group IV B metals, it is important that their properties at high temperatures are measured by techniques which eliminate (or at least minimize) contact with other substances. Recently, the melting points and other related properties of zirconium [1],¹ of hafnium-3 (wt. %) zirconium [2] and of several other refractory metals [3-5] were measured using a subsecond-duration pulse heating technique in which the "effective" specimen melts without being in contact with other substances that are likely to react with the specimen. In the present study, the same technique was used for the measurements of the melting point, the normal spectral emittance (at 653 nm) at the melting point, and the electrical resistivity (above 1900 K) of titanium.

The method involves measuring the specimen temperature, the current through and potential difference across the specimen as it undergoes rapid resistive self-heating from room temperature to its melting point in less than one second. The experimental quantities are recorded digitally every 0.4 ms with a full scale resolution of about 1 part in 8000. Details regarding the construction and operation of the measurement system, the methods of measuring experimental quantities, and other pertinent information, including error analysis, are given in earlier publications [6, 7].

2. Measurements

The measurements were performed on three tubular specimens fabricated by an electro-erosion technique from cylindrical rods. Nominal dimensions of the tubes were: length, 75 mm; outside diameter, 6.4 mm; wall thickness, 0.5 mm. The pyrometric temperature measurements were made by sighting through a small rectangular hole (1 × 0.5 mm) in

the wall at the middle of the specimen thereby approximating blackbody conditions. The heat loss due to thermal radiation was reduced by polishing the outer surface of each specimen. The results of a typical analysis furnished by the manufacturer indicated that the material was 99.9 + percent pure with the following impurities present (in ppm by weight): O, 360; Zr, 30; Fe, Ni, Cu, 20 each; C, 15; Al, S, 10 each; Si, V, Mn, Sn, 5 each; Cr, 3; H, 3; N, 2; all other detected elements were less than 1 ppm each.

The experiments were conducted with the specimens in an argon environment at atmospheric pressure. The average heating rate for each specimen was about 2400 K·s⁻¹, corresponding to a heating period (from room temperature to its melting point) of about 600 ms.

The high-speed pyrometer [8] was calibrated prior to starting the experiments, using a tungsten filament reference lamp which in turn was calibrated against the NBS "Temperature Standard."

An oscilloscope trace photograph showing the variation in radiation from a specimen, as seen by the pyrometer, is presented in figure 1. The high and low plateaus correspond to the true (blackbody) temperature and the radiance temperature² (at 653 nm), respectively, of the specimen during melting. Radiance temperature plateaus were observed only in two (out of three possible) cases because of the unpredictable collapse of the melting specimens.

The true temperatures, obtained from digitally recorded data, of a specimen during the transition from solid to liquid phase are plotted in figure 2. The dashed line indicates the mean melting temperature as well as the segment of the plateau included in computing the average. Similar results for the radiance temperature of a specimen are shown in figure 3.

All temperatures reported in this paper, except where explicitly noted, are based on the International Practical Temperature Scale of 1968 [9].

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¹ Figures in brackets indicate the literature references at the end of this paper.

² Radiance temperature (sometimes referred to as brightness temperature) is the apparent temperature of the specimen surface corresponding to the effective wavelength of the measuring pyrometer.

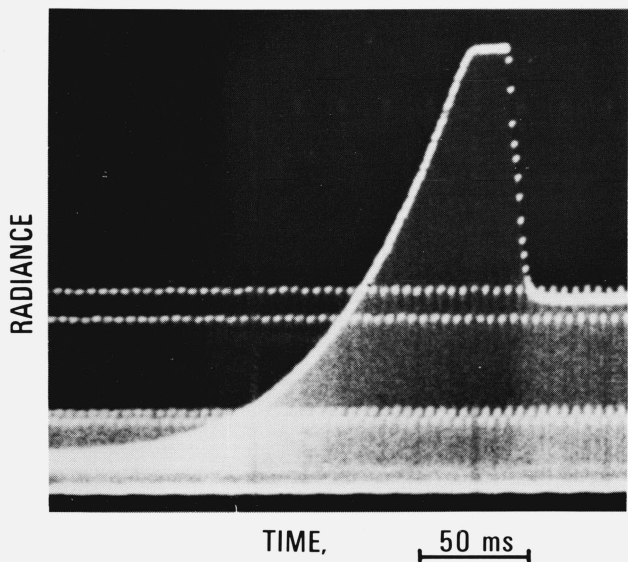


FIGURE 1. Oscilloscope trace photograph of specimen radiance (as observed by the pyrometer) near and at the melting point of titanium (Specimen 3).

Dots forming the long horizontal lines correspond to radiances from a reference source.

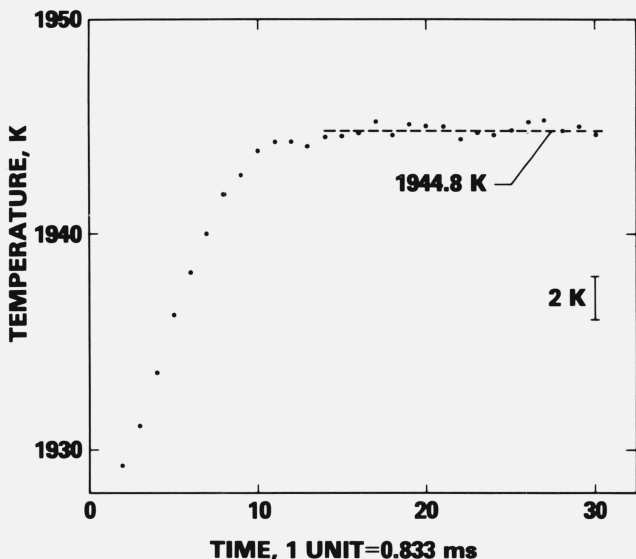


FIGURE 2. Variation of the true temperature as a function of time near and at the melting point of titanium (Specimen 1).

The points are temperatures obtained from individual pyrometer readings. The time-axis origin is arbitrary.

3. Results

Melting Point: The true temperatures at the plateau of each specimen were averaged to obtain its melting point. The same procedure was adopted to obtain the radiance

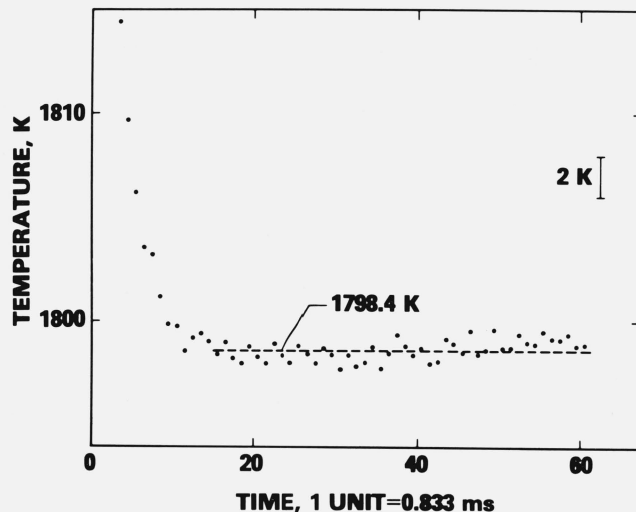


FIGURE 3. Variation of the radiance temperature as a function of time at the melting point of titanium (Specimen 3).

The points are temperatures obtained from individual pyrometer readings. The time-axis origin is arbitrary.

temperature at the melting point. The final results, presented in table 1, indicate that the average melting point and average radiance temperature at the melting point for the specimens studied in this work are 1945.3 K and 1799.0 K, respectively, with an average absolute deviation from the mean of 0.4 K in each case. It may be concluded that the melting point of titanium is 1945 K and that its radiance temperature (at 653 nm) at the melting point is 1799 K.

TABLE 1. Experimental results for the melting point and the radiance temperature (at 653 nm) at the melting point of titanium

Specimen number	Melting point			Radiance temp. at melting point		
	Number of temp.	Melting point (K)	Standard dev. (K)	Number of temp.	Radiance temp. (K)	Standard dev. (K)
1	17	1944.8	0.3	—	—	—
2	11	1945.5	0.4	19	1799.4	0.4
3	9	1945.7	0.2	46	1798.6	0.5

Normal Spectral Emittance at the Melting Point: From the true temperature (1945 K) and the radiance temperature (1799 K), Planck's radiation equation yields a value of 0.40 for the normal spectral emittance (at 653 nm) of titanium at its melting point.

Electrical Resistivity: The electrical resistivity of titanium near its melting point was determined from the measured resistance and the room temperature dimensions of the specimens. It was found that repeated heating and cooling through the solid-solid phase transformation point at about 1166 K [10] produced permanent distortions (elongations) in the specimens. The resistivity values were corrected for

these geometrical changes which, at temperatures near the melting point, gave rise to systematic shifts of less than 2 percent. The (corrected) results for a specimen are shown in figure 4. The maximum difference between the measured values of the resistivity for the three titanium specimens in the range 1900 to 1940 K was approximately 1 percent. The final results, obtained by averaging the (corrected) resistivity values for the three specimens, are $160.7 \mu\Omega \cdot \text{cm}$ at 1900 K and $161.5 \mu\Omega \cdot \text{cm}$ at 1940 K.

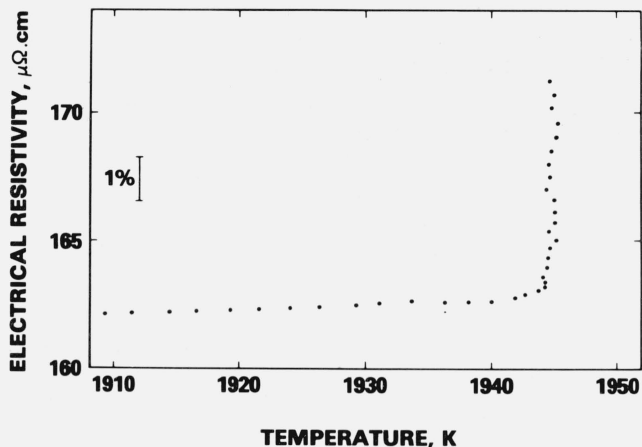


FIGURE 4. Variation of the electrical resistivity as a function of temperature near and at the melting point of titanium (Specimen 1).

Estimate of Errors: A detailed analysis of errors in such experimental quantities as temperature, voltage and current measured using the present pulse heating system was given in an earlier publication [7]. Specific items in the error analysis were recomputed whenever the present conditions differed from those in the earlier publication. The resultant estimated maximum errors in the reported values are: 5 K in the true temperature and radiance temperature at the melting point, 5 percent in the normal spectral emittance and 3 percent in the electrical resistivity.

4. Discussion

Results for the titanium melting point as reported in the literature are given in table 2 along with the corresponding values based on IPTS-68 for comparison with the present work. The measurements prior to 1953 [11-13] yield melting points which exceed the most recent values by considerably more than the combined reported uncertainties in the experiments. The discrepancies appear to arise from specimen contamination and/or inaccurate temperature determinations insofar as all of the measurements involved high purity titanium (99.9% or better). To overcome these difficulties, some of the recent investigators [15, 17, 19, 20] have used the classical method of approximating blackbody conditions³

³ The temperature is measured pyrometrically at the base of a small deep hole (depth $\geq 5 \times$ diameter) in the uniformly heated specimen.

TABLE 2. Values of the melting point of titanium reported in the literature

Investigator	Ref.	Year	Melting point (K)	
			As reported	On IPTS-68
Burgess and Waltenberg	11	1913	2068 ± 15	
Hansen et al.	12	1952	1993 ± 15	1996
Adenstedt et al.	13	1952	1973 ± 15	1976
Maykuth et al.	14	1953	1953 ± 10	1956
Schofield and Bacon	15	1953	1933 ± 10	1936
Oriani and Jones	16	1954	1945	1948
Deardorff and Hayes	17	1956	1941 ± 10	1944
Bickerdike and Hughes	18	1959	1940 ± 8	1943
Rudy and Progulski	19	1967	1941 ± 8	1944
Berezin et al.	20	1974	1939 ± 4	1941
Present work			1945 ± 5	1945

for pyrometric temperature measurements. In addition, Deardorff and Hayes [17], Rudy and Progulski [19], and Berezin et al. [20] have all utilized experimental techniques which preclude contact between the molten titanium and other substances likely to react with the specimen. The average value of the melting points obtained in the above three investigations is 1943 K, the average and maximum difference from the mean being 1 K and 2 K, respectively. The melting point determined by the present study is 2 K higher than the above average.

Bonnell et al. [21] have measured the radiance temperature of melting titanium to be 1814 K at an effective wavelength of 645 nm. Using the above value for the radiance temperature and an average melting point (1946 K) from the literature, they obtained a value of 0.434 for the normal spectral emittance. Subsequent measurements by Berezin et al. [20] of the true temperature (1941 ± 4 K) and radiance temperature (1801 ± 4 K) at the melting point yield an emissivity value of 0.412 ± 0.017 for 650 nm, which is 3 percent higher than the value of the present work. Recently, Righini et al. [22] have determined the emissivity at 653 nm to be 0.401, using their measurement of radiance temperature (1800 ± 6 K) at the melting point and the melting temperature (1945 ± 5 K) obtained in the present study.

There appears to be no data in the literature for electrical resistivity of titanium in the range 1900 to 1940 K. For comparison, measurements by Wyatt [23], Zhorov [24], and Arutyunov et al. [25], on specimens with respective purities of 99.74 percent (and 99.96%), 99.82 percent, and 99.8 percent extending respectively to temperatures of 1775 K (and 1624 K), 1800 K and 1700 K were extrapolated linearly to the temperature range of the present work; the extrapolated values were found to be 2.7 percent (and 12.3%), 3.3 percent and 2.8 percent, respectively, above those obtained in the present study. With one exception, the agreement among all of the resistivity values above 1900 K is consistent with the combined experimental and extrapolation errors.

5. References

- [1] Cezairliyan, A., and Righini, F., Measurement of melting point, radiance temperature (at melting point), and electrical resistivity (above 2100 K) of zirconium by a pulse heating method, *Rev. Int. Hautes Tempér. et Réfract.* **12**, No. 3, 201-207 (1975).
- [2] Cezairliyan, A., and McClure, J. L., Measurement of melting point and radiance temperature (at melting point and at 653 nm) of hafnium-3 (wt. %) zirconium by a pulse heating method, *J. Res. Nat. Bur. Stand. (U.S.)* **80A**, (Phys. and Chem.) No. 4, 659-662 (1976).

- [3] Cezairliyan, A., Morse, M. S., and Beckett, C. W., Measurement of melting point and electrical resistivity (above 2840 K) of molybdenum by a pulse heating method, *Rev. Int. Hautes Temp., et Réfract.* **7**, No. 4, 382-388 (1970).
- [4] Cezairliyan, A., Measurement of melting point and electrical resistivity (above 3600 K) of tungsten by a pulse heating method, *High Temp. Science* **4**, No. 3, 248-252 (1972).
- [5] Cezairliyan, A., Measurement of melting point, normal spectral emittance (at melting point), and electrical resistivity (above 2650 K) of niobium by a pulse heating method, *High Tem.-High Press.* **4**, 453-458 (1972).
- [6] Cezairliyan, A., Design and operational characteristics of a high-speed (millisecond) system for the measurement of thermophysical properties at high temperatures, *J. Res. Nat. Bur. Stand. (U.S.)* **75C**, (Eng. and Instr.), 7-18 (1971).
- [7] Cezairliyan, A., Morse, M. S., Berman, H. A., and Beckett, C. W., High-speed (subsecond) measurement of heat capacity, electrical resistivity, and thermal radiation properties of molybdenum in the range 1900 to 2800 K, *J. Res. Nat. Bur. Stand. (U.S.)* **74A**, (Phys. and Chem.) 65-92 (1970).
- [8] Foley, G. M., High-speed optical pyrometer, *Rev. Sci. Instr.* **41**, 827-834 (1970).
- [9] International Practical Temperature Scale of 1968, *Metrologia* **5**, 35-44 (1969).
- [10] Cezairliyan, A., and Müller, A. P., Thermodynamic study of the $\alpha \rightarrow \beta$ phase transformation in titanium by a pulse heating method, *J. Res. Nat. Bur. Stand. (U.S.)*, in press.
- [11] Burgess, G. K., and Waltgenberg, R. G., *Schmelzpunkte Refraktärer Elemente. I. Elemente von Atomgewicht 48 bis 59*, *Z. anorg. Chem.* **82**, 361-372 (1913).
- [12] Hansen, M., Kessler, H. D., and McPherson, D. J., The titanium-silicon system, *Trans. Am. Soc. Metals* **44**, 518-535 (1952).
- [13] Adenstedt, H. K., Pequignot, J. R., and Raymer, J. M., The titanium-vanadium system, *Trans. Am. Soc. Metals* **44**, 990-1003 (1952).
- [14] Maykuth, D. J., Ogden, H. R., and Jaffee, R. I., Titanium-tungsten and titanium-tantalum systems, *Trans. Am. Inst. Min. Met. Eng.* **197**, 231-237 (1953).
- [15] Schofield, T. H., and Bacon, A. E., The melting point of titanium, *J. Inst. Metals* **82**, 167-169 (1953).
- [16] Oriani, R. A., and Jones, T. S., An apparatus for the determination of the solidus temperatures of high melting alloys, *Rev. Sci. Instr.* **25**, No. 3, 248-250 (1954).
- [17] Deardorff, D. K., and Hayes, E. T., Melting point determination of hafnium, zirconium, and titanium, *J. Metals* **8**, 509-511, (1956).
- [18] Bickerdike, R. L., and Hughes, G., An examination of part of the titanium-carbon system, *J. Less-Common Metals* **1**, 42-49 (1959).
- [19] Rudy, E., and Progulski, J., A Pirani furnace for the precision determination of the melting temperatures of refractory metallic substances, *Planseeber. Pulvermet.* **15**, 13-45, (1967).
- [20] Berezin, B. Ya., Kats, S. A., Kenisarin, M. M., and Chekhovskoi, V. Ya., Heat and melting temperature of titanium, *High Temperature (USSR)* **12**, No. 3, 450-455 (1974).
- [21] Bonnell, D. W., Traverton, J. A., Valerga, A. J. and Margrave, J. L., The Emissivities of Liquid Metals at Their Fusion Temperatures in the book *Temperature, Its Measurement and Control in Science and Industry 1972*, H. H. Plumb, Ed., Vol. 4, part 1 (ISA, Pittsburgh) pp. 483-487.
- [22] Righini, F., Rosso, A., Coslovi, L., Cezairliyan, A. and McClure, J. L., Radiance Temperature of Titanium at Its Melting Point, to be published in the Proceedings of the Seventh Symposium on Thermophysical Properties. ASME, N. Y.
- [23] Wyatt, J. L., Electrical Resistance of Titanium Metal, *J. Metals* **5**, No. 7, 903-905 (1953).
- [24] Zhorov, G. A., Relation between the emissive power and specific electrical resistivity in metals, *High Temperature (USSR)* **5**, No. 6, 881-888 (1967).
- [25] Arutyunov, A. V., Banchila, S. N., and Filippov, L. P., Properties of titanium at temperatures above 1000 K, *High Temperature (USSR)* **9**, No. 3, 487-489 (1971).