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Measurement of Melting Point, Normal Spectral Emittance (at Melting Point) and Electrical Resistivity (Near Melting Point) of Some Refractory Alloys*

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A subsecond duration pulse heating method is used to measure the melting point, normal spectral emittance (at the melting point, corresponding to 650 nm), and electrical resistivity (near the melting point) of the following refractory alloys: 90 Ta–10 W, 99 Nb–1 Zr, and 80 Nb–10 Ta–10 W (numbers indicate nominal composition in percentage by weight). The melting point and the normal spectral emittance are: 3286 ± 15 K and 0.396 for 90 Ta–10 W, 2737 ± 10 K and 0.351 for 99 Nb–1 Zr, and 2814 ± 10 K and 0.333 for 80 Nb–10 Ta–10 W. The inaccuracy of the normal spectral emittance and electrical resistivity results is estimated to be 3 percent and 0.5 percent, respectively.

Key words: Alloys; electrical resistivity; high temperature; melting point; normal spectral emittance; refractory materials; thermophysics.

1. Introduction

A millisecond resolution pulse heating technique was used earlier for the measurement of the melting point, normal spectral emittance (at the melting point), and electrical resistivity (near the melting point) of niobium [1][1]. In the present study, the same technique is used for similar measurements on the refractory alloys: 90 Ta–10 W, 99 Nb–1 Zr, and 80 Nb–10 Ta–10 W (numbers indicate nominal composition in percentage by weight).

The technique is based on resistive self-heating of the specimen from room temperature to its melting point in less than one second and measuring current through the specimen, potential difference across the specimen, and specimen temperature. These quantities are measured and recorded digitally every 0.4 ms with a full-scale signal resolution of approximately 1 part in 8000. The details regarding the construction and operational characteristics of the measurement system are given in an earlier publication [2].

2. Measurements

Melting point and electrical resistivity measurements were performed on specimens in the form of tubes having the following nominal dimensions: length, 102 mm; outside diameter, 6.3 mm; and thickness, 0.5 mm. A small rectangular hole (0.5 × 1 mm) fabricated in the wall at the middle of the specimen approximated blackbody conditions for temperature measurements. Normal spectral emittance measurements were performed on specimens in the form of rods having the following nominal dimensions: length 76 mm; diameter, 3.1 mm. The surfaces of all specimens were polished. The composition of the alloys is given in table 1.

TABLE 1. *Composition of the alloys*

Alloy	Major constituents (% by weight)	Total impurities (% by weight)
90 Ta–10 W	Ta, 90.45; W, 9.45	0.10
99 Nb–1 Zr	Nb, 98.78; Zr, 1.05	0.17
80 Nb–10 Ta–10 W	Nb, 80.94; Ta, 9.70; W, 9.30	0.06

Before starting the experiments, the specimens were heat treated by subjecting them to 30 heating pulses in the temperature range 2200 to 3000 K. The experiments were conducted in a vacuum environment of 10^{-4} to 10^{-5} torr. Duration of current pulses (duration for the specimen to heat from room temperature to its melting point) was in the range 380 to 750 ms.

For each alloy two experiments were performed for the determination of the melting point and one experiment was performed for the determination of normal spectral emittance and electrical resistivity. Designation of the specimens and the results of some of the

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

measurements are given in table 2. All measurements reported in this paper are based on the International Practical Temperature Scale of 1968 [3].

TABLE 2. *Specimen designations and the results of individual experiments*

Specimen number	Substance	Geometry	Pulse length (ms)	Melting point			Radiance temp. at melting point		
				Number of temp.	Melting point (K)	Stand. dev. (K)	Number of temp.	Rad. temp. (K)	Stand. dev. (K)
1	90 Ta–10 W	tube	480	12	3287.0	1.3			
2	90 Ta–10 W	tube	750	15	3285.1	0.8			
3	90 Ta–10 W	rod	600				26	2888.7	0.4
4	99 Nb–1 Zr	tube	380	23	2737.0	1.9			
5	99 Nb–1 Zr	tube	390	24	2737.6	1.3			
6	99 Nb–1 Zr	rod	380				21	2423.2	0.8
7	80 Nb–10 Ta–10 W	tube	630	21	2816.1	0.3			
8	80 Nb–10 Ta–10 W	tube	660	30	2812.8	0.6			
9	80 Nb–10 Ta–10 W	rod	610				8	2469.2	0.2

2.1. Melting Point

Temperature of the tubular specimens was measured near and during the initial melting period. A plateau in temperature indicated the transition from solid to liquid phase. Measured temperatures at the plateau for specimens are shown in figure 1. The melting point for each specimen was obtained by averaging the temperature points on the plateau. The results are presented in table 2. The standard deviation of an individual point was in the range 0.3 to 1.9 K. As may be seen in table 2, the difference between the results for the two specimens of the same alloy was in the range 0.6 to 3.3 K. The average melting points of the alloys are given in table 3.

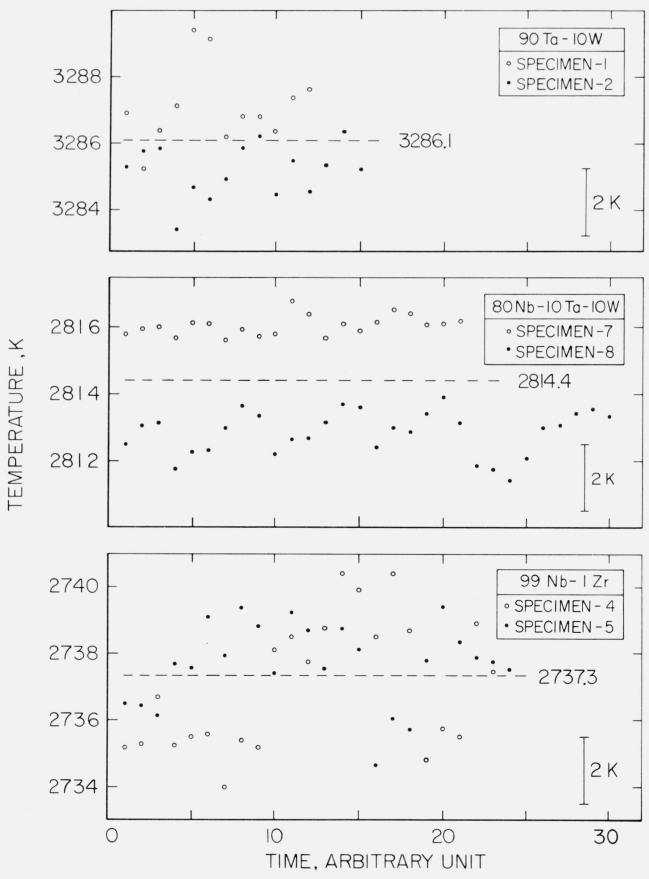

FIGURE 1. *Variation of the temperature of various refractory alloys as a function of time at their respective melting points (1 time unit = 0.833 ms).*

TABLE 3. *Melting point (T_m), radiance temperature (T_s) at the melting point, and normal spectral emittance (ϵ) at the melting point of some refractory alloys*

Substance	T_m(K)	T_s(K) (at 650 nm)	ϵ (at 650 nm)
90 Ta–10 W	3286	2889	0.396
99 Nb–1 Zr	2737	2423	0.351
80 Nb–10 Ta–10 W	2814	2469	0.333

2.2. Normal Spectral Emittance

Normal spectral emittance at the melting point of each alloy was determined from the measurements of the radiance temperature of the specimen (in rod form) and the average melting point obtained from the measurements on the tubular specimens. Based on the Wien radiation equation, the relation between emittance and temperature is

$$\epsilon = \frac{1}{\exp\left[\frac{c_2}{\lambda}\left(\frac{1}{T_s}-\frac{1}{T_m}\right)\right]}$$

where ϵ is the normal spectral emittance, T_m the melting point, T_s the radiance temperature, c_2 the second radiation constant (1.4388×10^{-2} m K), and λ the effective wavelength of the optical system. The measurements were performed at 650 nm which corresponds to the effective wavelength of the pyrometer's interference filter. The bandwith of the filter was 10 nm. The circular area viewed by the pyrometer was 0.2 mm in diameter.

The experimental results of the radiance temperature of 90 Ta–10 W and 99 Nb–1 Zr alloys near

and at the respective melting points are shown in figure 2. Similar results for the alloy 80 Nb–10 Ta–10 W are shown in figure 3. It may be seen (figure 2) that both 90 Ta–10 W and 99 Nb–1 Zr samples behaved normally, that is, radiance temperature increased and exhibited a plateau upon melting. However, 80 Nb–10 Ta–10 W results (figure 3) do not indicate the existence of any well-defined plateau, with the exception of a short segment immediately after reaching the melting point. The results of radiance temperature at the melting point of the alloys and corresponding values for the normal spectral emittance are given in table 3.

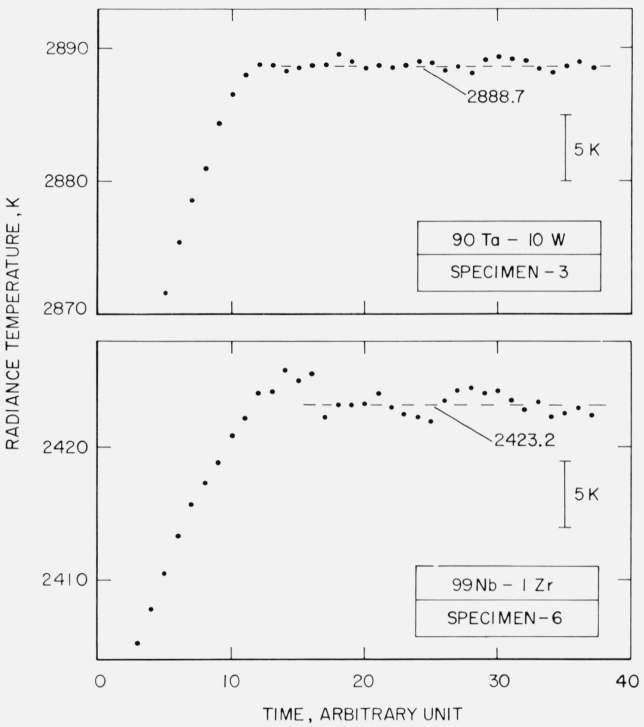

FIGURE 2. *Variation of the radiance temperature (at 650 nm) of the alloys* 90 Ta–10 W *and* 99 Nb–1 Zr *as a function of time near and at their respective melting points (1 time unit = 0.833 ms).*

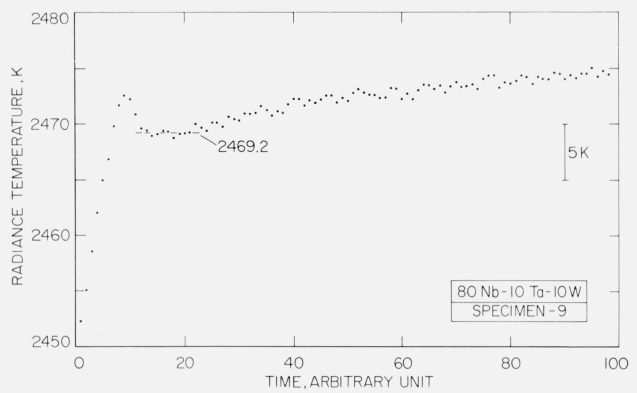

FIGURE 3. *Variation of the radiance temperature (at 650 nm) of the alloy* 80 Nb–10 Ta–10 W *as a function of time near and at its melting point (1 time unit = 0.833 ms).*

2.3. Electrical Resistivity

Electrical resistivity of the tubular specimens was calculated using the relation $\rho = RA/L$, where R is the resistance, A the cross-sectional area, and L the length of the specimen between the potential probes. The dimensions were based on their room temperature values; the cross-sectional area was determined from the measurement of weight and density. The results for the electrical resistivity of the alloys near and at their respective melting points are shown in figures 4, 5, and 6. The values for the 100 K range below the melting points are given in table 4. The results indicate that electrical resistivity of the alloys 90 Ta–10 W (figure 4) and 99 Nb–1 Zr (figure 5) changed almost abruptly at the start of melting. However, in the case of the alloy 80 Nb–10 Ta–10 W (figure 6) the change in the electrical resistivity was gradual, starting approximately 15 K below the melting point. Below the melting point electrical resistivity for each alloy was fitted, using the least squares method, to a linear function in temperature. In all cases, the standard deviation of an individual point from the smooth function was less than 0.06 percent.

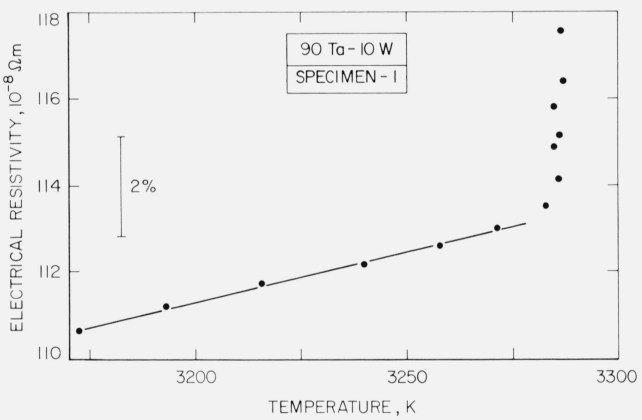

FIGURE 4. *Electrical resistivity of the alloy* 90 Ta–10 W *near and at its melting point.*

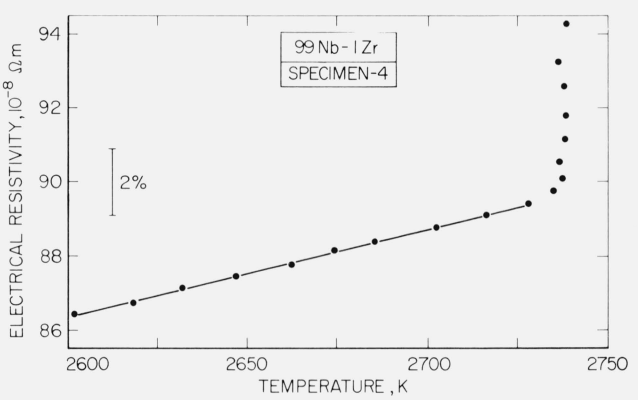

FIGURE 5. *Electrical resistivity of the alloy* 99 Nb–1 Zr *near and at its melting point.*

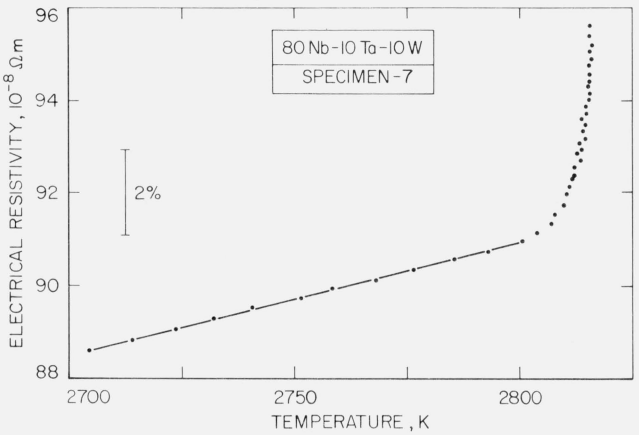

FIGURE 6. *Electrical resistivity of the alloy* 80 Nb–10 Ta–10 W *near and at its melting point.*

TABLE 4. *Electrical resistivity of some refractory alloys near their melting points*

Alloy	Temp. (K)	Elec. res.* (10^{-8} Ω m)
90 Ta–10 W	3180	110.88
	3230	112.03
	3280	113.17
99 Nb–1 Zr	2630	87.06
	2680	88.25
	2730	89.43
80 Nb–10 Ta–10 W	2700	88.49
	2750	89.70
	2800	90.92

*Based on ambient temperature (298 K) dimensions.

2.4. Estimate of Errors

Sources and estimates of errors in experiments similar to the ones conducted in this study are given in earlier publications [4, 5]. Specific items in the error analysis were recomputed whenever the present conditions differed from those in the earlier publications.

As it may be seen from table 2, the imprecision[2] of true (blackbody) and radiance temperature measurements was 0.3–1.9 K and 0.2–0.8 K, respectively. A somewhat higher imprecision in the case of the blackbody temperature measurements may be attributed to the movements of the sighting hole in the specimen during melting. The inaccuracy[3] of the melting points is estimated to be approximately 10 K for the alloys 99 Nb–1 Zr and 80 Nb–10 Ta–10 W, and 15 K for 90 Ta–10 W.

The combination of inaccuracies in the temperature measurements, taking into account likely common errors in both blackbody and radiance temperature measurements, indicates that the inaccuracy in the normal spectral emittance is approximately 3 percent.

The imprecision of the electrical resistivity results is 0.05 percent and the estimated inaccuracy is 0.5 percent.

3. Discussion

The agreement of the melting points of the specimens of the same alloy were in the range 0.6 to 3.3 K, indicating reasonably good reproducibility of the measurements. Radiance temperature and electrical resistivity of the binary alloys 90 Ta–10 W and 99 Nb–1 Zr behaved normally, that is exhibited an almost abrupt change at the beginning of melting. However, in the case of the ternary alloy 80 Nb–10 Ta–10 W the change in electrical resistivity was more gradual, starting approximately 15 K below the melting point. Radiance temperature of the ternary alloy exhibited a peak before melting, a short plateau immediately after the start of melting, and a continuous increase as melting progressed. The peak may be due to the roughness of the solid surface, and the increase in radiance temperature during melting may be attributed to the increase of the normal spectral emittance during melting. With the present system it was not possible to follow the entire melting process because the specimens collapsed and opened the electrical circuit prior to the completion of melting.

4. References

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[2] Imprecision refers to the standard deviation of an individual point as computed from the difference between measured value and that from the smooth function obtained by the least squares method.

[3] Inaccuracy refers to the estimated total error (random and systematic).