## Simultaneous Measurement of Specific Heat, Electrical Resistivity, and Hemispherical Total Emittance of Niobium-1 (Wt. %) Zirconium Alloy in the Range 1500 to 2700 K by a Transient (Subsecond) Technique<sup>\*</sup>

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Simultaneous measurements of specific heat, electrical resistivity, and hemispherical total emittance of niobium-1 (wt. %) zirconium alloy in the temperature range 1500 to 2700 K by a subsecond duration pulse heating technique are described. Estimated inaccuracy of measured properties are: 3 percent for specific heat and hemispherical total emittance, and 0.5 percent for electrical resistivity. Properties of the alloy are compared with the properties of pure niobium. It was found that specific heat and emittance of the alloy were approximately 0.5 percent and 1.5 percent, respectively, higher than those of pure niobium. Electrical resistivity of the alloy was 0.5 percent lower than that of pure niobium. Like niobium, the alloy showed a negative departure from linearity in the curve of electrical resistivity versus temperature.

Key words: Electrical resistivity; emittance; high-speed measurements; high temperature; niobiumzirconium alloy; specific heat; thermodynamics.

## 1. Introduction

In this paper, application of a transient technique to the simultaneous measurements of specific heat, electrical resistivity, and hemispherical total emittance of the alloy niobium-1 (wt. %) zirconium in the temperature range from 1500 to 2700 K is described.

The method is based on rapid resistive self-heating of the specimen from room temperature to any desired high temperature (up to its melting point) in less than one second by the passage of electrical currents through it; and on measuring, with millisecond resolution, experimental quantities, such as current through the specimen, potential drop across the specimen, and specimen temperature. Details regarding the construction and operation of the measurement system, the methods of measuring experimental quantities, and other pertinent information, such as formulation of relations for properties, etc. are given in earlier publications [1, 2].<sup>1</sup>

### 2. Measurements

The specimen was a tube of the following nominal dimensions: length, 102 mm; outside diameter, 6.3

mm; and wall thickness, 0.5 mm. Zirconium content of the specimen was 1.05 percent by weight. The total amount of impurities was less than 0.17 percent; the major impurity was tantalum with 0.09 percent. Photomicrographs of the specimen, shown in figure 1, indicate that considerable grain growth took place as the result of pulse heating to high temperatures.

To optimize the operation of the high-speed pyrometer, the temperature interval (1500 to 2700 K) was divided into six ranges. One experiment was performed in each range. Before the start of the experiments, the specimen was annealed by subjecting it to 30 heating pulses (up to 2500 K). The experiments were conducted with the specimen in a vacuum environment of approximately  $10^{-4}$  torr.

To optimize the operation of the measurement system, the heating rate of the specimen was varied depending on the desired temperature range by adjusting the value of a resistance in series with the specimen. Duration of current pulses in the experiments ranged from 360 to 410 ms; and the heating rate ranged from 4500 to 6600 K s<sup>-1</sup>. Radiative heat loss from the specimen amounted to approximately 1 percent at 1500 K, and 9 percent at 2700 K of the input power.

#### 3. Experimental Results

The thermophysical properties reported in this paper are based on the International Practical Temper-

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<sup>&</sup>lt;sup>1</sup> Figures in brackets indicate the literature references at the end of this paper.

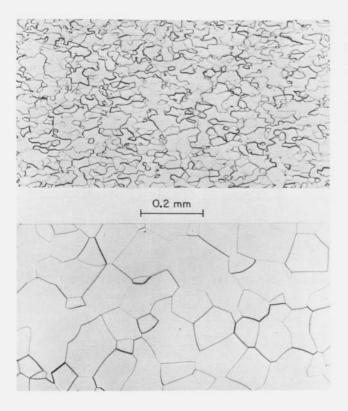


FIGURE 1. Photomicrographs of the niobium-1 (wt. %) zirconium specimen.

Upper photograph, specimen as received; lower photograph, specimen after the entire set of experiments.

ature Scale of 1968 [3]. In all computations, the geometrical quantities are based on their room temperature (298 K) dimensions. The experimental results for properties are represented by polynomial functions in temperature obtained by least squares approximation of the individual points. The final values on properties at 100 degree temperature intervals computed using the functions are presented in table 1. Results obtained from individual experiments, by the

 TABLE 1. Specific heat, electrical resistivity, and hemispherical total emittance of the alloy niobium-l (wt. %) zirconium

Temp. K	$c_p J g^{-1} K^{-1}$	$\begin{array}{c} \rho^*\\ 10^{-8}\Omega\mathrm{m} \end{array}$	$\epsilon^*$
1500	0.3207	57.36	
1600	.3263	60.13	
1700	.3322	62.87	0.218
1800	.3385	65.59	.232
1900	.3455	68.27	.245
2000	.3535	70.93	.257
2100	.3627	73.56	.268
2200	.3735	76.16	.278
2300	.3861	78.74	.287
2400	.4007	81.29	.295
2500	.4177	83.81	.303
2600	.4373	86.30	.309
2700	.4598	88.76	

\*Based on ambient temperature (298 K) dimensions.

method described previously [2], are given in the appendix (tables A–1 and A–2). Each number tabulated in these tables represents results from over 50 original data points.

Specific Heat: Specific heat was computed from data taken during the heating period. A correction for power loss due to thermal radiation was made using the results on hemispherical total emittance. The function for specific heat (standard deviation = 1%) that represents the results in the temperature range 1500 to 2700 K is:

$$c_p = 7.073 \times 10^{-2} + 3.783 \times 10^{-4} T - 2.091 \times 10^{-7} T^2 + 4.532 \times 10^{-11} T^3$$
(1)

where *T* is in K, and  $c_p$  is in J g<sup>-1</sup>K<sup>-1</sup>.

*Electrical Resistivity:* The electrical resistivity was determined from the same experiments that were used to calculate the specific heat. The function for electrical resistivity (standard deviation = 0.06%) that represents the results in the temperature range 1500 to 2700 K is:

$$\rho = 12.50 + 3.199 \times 10^{-2} T - 1.387 \times 10^{-6} T^2$$
 (2)

where T is in K, and  $\rho$  is in  $10^{-8} \Omega$  m. The measurement, before the pulse experiments, of the electrical resistivity of the specimen at 293 K with a Kelvin bridge yielded a value of  $16.2 \times 10^{-8} \Omega$  m.

Hemispherical Total Emittance: Hemispherical total emittance was computed using data taken during both heating and initial free radiative cooling periods. The function for hemispherical total emittance (standard deviation = 1%) that represents the results in the temperature range 1700 to 2600 K is:

$$\epsilon = -1.647 \times 10^{-1} + 3.056 \times 10^{-4}T - 4.749 \times 10^{-8}T^2$$
(3)

where T is in K.

## 4. Estimate of Errors

The details for estimating errors in measured and computed quantities in transient experiments using the present measurement system are given in an earlier publication [2]. In this paper, the specific items in the error analysis were recomputed whenever the present conditions differed from those in the earlier publication. The results for imprecision<sup>2</sup> and inaccuracy<sup>3</sup> in the properties are: 1 percent and 3 percent for specific heat, 0.06 percent and 0.5 percent for electrical resistivity, 1 percent and 3 percent for hemispherical total emittance.

## 5. Discussion

The specific heat, electrical resistivity, and hemispherical total emittance of the niobium-1 (wt. %)

<sup>&</sup>lt;sup>2</sup> 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.

<sup>&</sup>lt;sup>3</sup> Inaccuracy refers to the estimated total error (random and systematic).

zirconium alloy measured in this work are presented in figure 2. A comparison of the present results for the alloy with those for pure niobium [4] obtained using the same method is given in figure 3. It may be seen that specific heat and hemispherical total emittance of the alloy were approximately 0.5 percent

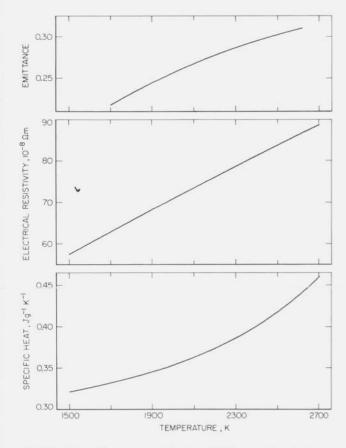


FIGURE 2. Specific heat, electrical resistivity, and hemispherical total emittance of niobium-1 (wt. %) zirconium alloy.

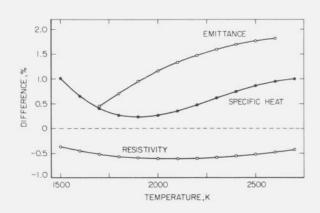


FIGURE 3. Differences in specific heat, electrical resistivity, and hemispherical total emittance of niobium-1 (wt. %) zirconium alloy from those of pure niobium.

The zero line corresponds to results of pure niobium[4].

and 1.5 percent, respectively, higher than those of pure niobium. The difference in specific heat cannot be accounted for by the additivity law. Electrical resistivity of the alloy was 0.5 percent lower than that of pure niobium. However, one should not place too much significance to these differences since their magnitudes are less than the combined estimated errors in the measurements for the alloy and for the pure metal.

At 293 K, electrical resistivity of the alloy  $(16.2 \times 10^{-8} \Omega \text{ m})$  is higher than the resistivity of pure niobium  $(15.9 \times 10^{-8} \Omega \text{ m})$  [4]. However, at high temperatures (fig. 3) the resistivity of the alloy is lower than that of niobium. A similar trend was also observed in the electrical resistivity of the alloy tantalum-10 (wt. %) tungsten [5]. Like niobium, at high temperatures the alloy showed a negative departure from linearity in the curve of electrical resistivity versus temperature.

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## 6. Appendix

TABLE A-1. Experimental results on specific heat and electrical resistivity of the alloy niobium-1 (wt. %) zirconium

T K	$J g^{-1} K^{-1}$	$\Delta c_p^*$	$\begin{array}{c} \rho \\ 10^{-8}\Omega \ \mathrm{m} \end{array}$	${\Delta  ho st m} _{\%}^{*}$
1500	0.3198	- 0.21	57.37	+ 0.01
1550	.3206	-0.87	58.75	-0.01
1600	.3275	+0.37	60.13	0.00
1650	.3344	+1.57	61.53	+ 0.03
1700	.3311	-0.31	62.90	+0.04
1750	.3392	+1.19	64.27	+0.05
1800	.3339	-1.34	65.59	+ 0.01
1850	.3416	-0.06	66.92	-0.02
1900	.3480	+0.74	68.26	-0.03
1950	.3419	-2.14	69.58	-0.03
2000	.3505	-0.82	70.88	-0.07
2050	.3587	+0.26	72.19	-0.09
2100	.3666	+1.08	73.50	- 0.09
2150	.3739	+1.62	74.81	-0.08
2200	.3700	-0.91	76.25	+ 0.10
2250	.3779	-0.42	77.51	+0.06
2300	.3859	0.00	78.77	+ 0.03
2350	.3943	+ 0.33	80.03	+0.01
2400	.4030	+0.60	81.28	-0.01
2450	.4024	-1.59	82.68	+ 0.16
2500	.4217	+0.98	83.79	-0.02
2550	.4228	-1.01	85.11	+0.07
2600	.4426	+1.24	86.28	-0.02
2650	.4461	-0.43	87.52	-0.02
2700	.4594	-0.07	88.69	-0.08

\*The quantities  $\Delta c_p$  and  $\Delta \rho$  are percentage deviations of the individual results from the smooth functions represented by eqs (1) and (2), respectively.

Т	e	$\Delta \epsilon^*$
K		%
711	0.218	- 0.44
711	.223	+1.71
711	.217	- 1.01
712	.223	+1.76
899	.242	- 1.01
899	.242	- 0.93
899	.240	- 1.76
900	.241	- 1.50
2055	.264	+ 0.36
2056	.265	+0.62
2056	.266	+1.05
2057	.266	+1.22
2322	.287	- 0.69
2324	.289	-0.11
2324	.290	+0.14
2325	.291	+0.64
2667	.312	-0.31
2670	.312	-0.11
2670	.313	+ 0.02
672	.314	+ 0.20

TABLE	A-2.	Experimental results on hemispherical total emittance
		of the alloy niobium-1 (wt. %) zirconium

# \*The quantity $\Delta \epsilon$ is percentage deviation of the individual results from the smooth function represented by eq (3).

## 7. References

- 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 (1971).
- [2] 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 (1970).
- [3] International Practical Temperature Scale of 1968, Metrologia, 5, 35 (1969).
- [4] Cezairliyan, A., High-speed (subsecond) measurement of heat capacity, electrical resistivity, and thermal radiation properties of niobium in the range 1500 to 2700 K, J. Res. Nat. Bur. Stand. (U.S.), 75A (Phys. and Chem.), 565 (1971).
- [5] Cezairliyan, A., High-speed (subsecond) simultaneous measurement of specific heat, electrical resistivity, and hemispherical total emittance of tantalum-10 (wt. %) tungsten alloy in the range 1500 to 3200 K, High Temperatures-High Pressures, in press.

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