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SPECIFIC HEAT OF RHENIUM AT HIGH TEMPERATURES

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The results of measurements of the specific heat of a wire sample of rhenium in the temperature interval 1600-2400°K and also data on the electrical resistivity and the integrated degree of blackness are reported.

To date, insufficient attention has been given to the specific heat of rhenium at high temperatures.

Taylor and Finch [1] cited data on the specific heat up to 3100°K, although in fact the experimental material is obtained up to 2000°K, since the electrical resistivity required for the treatment of the corresponding data is measured only up to this temperature and then extrapolated. The results of this extrapolation cannot be regarded as particularly reliable, since it involves the continuation of a curve whose slope is strongly dependent on temperature (it would be more logical to extrapolate the specific heat itself — a weaker function of temperature). The accuracy of the data in Taylor and Finch's paper could also do with being improved upon. In order to find the specific heat in the pulse method of measurement used by these authors, it is necessary to determine the time derivative of a curve produced on the screen of an oscilloscope, a procedure that is associated with a large error.

The measurements reported in [2] are coarse. The data are obtained by photographing the variation in time of the readings of a photoelectric pyrometer on the screen of an oscilloscope; the specific heat is obtained via measurements of the integrated degree of blackness, and the results are presented in the form of a large-scale graph.

The specific heat measurements of rhenium reported in [3] relate to a single crystal sample. They span the temperature interval up to 2500°K, but, as noted previously [3, 4], are of a tentative character, since they were obtained ignoring anisotropy of the single crystal.

In the work reported here the specific heat of wire samples of rhenium was measured using the method previously developed in the Department of Molecular Physics and Mechanics of the Physics Faculty of Moscow State University [5]. This method consists, essentially, in heating the investigated sample by the sum of dc

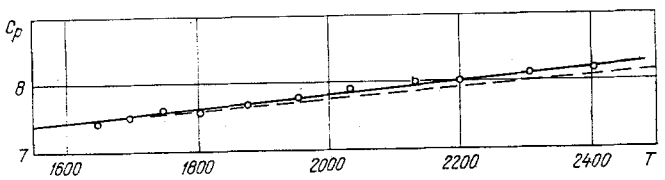

Fig. 1. Measured specific heat of rhenium. The dashed curve was calculated from (5). C_p is in cal/g-atom · °K; temperature T is in °K.

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TABLE 1. Results of Measurements of Specific Heat, Electrical Resistivity, and Integrated Degree of Blackness of Rhenium

T, °K	c_p, cal/ g-atom • °K	$\rho \cdot 10^8$, Ω•m	ε
1620	7,45	93,0	0,225
1680	7,51	94,3	0,230
1740	7,60	96,6	0,240
1795	7,64	99,2	0,250
1865	7,68	101,5	0,275
1940	7,76	104,0	0,280
2020	7,91	107,0	0,290
2115	7,97	110,5	0,305
2195	7,99	114,0	0,320
2305	8,10	117,5	0,325
2400	8,14	121,0	0,330

and ac currents and measuring the temperature oscillations of the sample by a contactless photoelectric method through the oscillations of its luminosity.

The theory behind the method is presented in detail in [5]. The method rests on the formula

$$C_p = \frac{2V_{=}V_{\sim}}{Rm\omega|\Theta|} . \tag{1}$$

Formula (1) is derived on the assumption that there are no temperature gradients along the axis and radius of the wire, which does not impose any significant constraints on the experiment. The correction to formula (1) due to heat transport from the surface of the wire for frequencies of tens of hertz is negligibly small [5].

The following formula is used to measure the amplitude of the temperature oscillations $|\Theta|$ by the photoelectric method:

$$|\Theta| = \frac{\bar{T}^2}{\alpha} \cdot \frac{I_{\sim}}{I} . \tag{2}$$

Formula (2) is valid for a linear recording system. The coefficient of proportionality α is found from the angle of slope of the straight line in the variables ln I and 1/T [4]. Inserting (2) into (1) gives the following formula for the specific heat:

$$C_p = \frac{2V_{=}\alpha I}{Rm\omega\bar{T}^2} \cdot \frac{V_{\sim}}{I_{\sim}} . \tag{3}$$

In the experimental setup the wire sample is located horizontally within a vacuum chamber provided on the top with a flat glass window. One end of the sample is rigidly gripped in a stand; the other is tensioned by a spring. In the middle part of the sample, at points a few centimeters apart, lie probe potential leadouts made from 30-μ-diameter tungsten wire, the ends of the leadouts being tensioned by loads.

The sample circuit contains the following in series: a source of dc voltage (two type VS-26 instruments); a matching transformer supplying an ac voltage from a GZ-34 audio-frequency generator (the 20-Hz frequency was determined by a PS-10,000 counter); and the sample resistance (0.1 Ω), which was used to measure the dc component of the current through the sample. The dc current through the sample and the voltage removed from the probe leadouts were measured using an R-307 potentiometer with an F116 photocompensated galvanometer as a null instrument. The ac voltage was measured using a VZ-7 cathode voltmeter.

The circuit measuring the temperature pulsations incorporates a photomultiplier and a differential-type amplifying stage. The stage was balanced so that the dc voltage at the output equalled zero for no light to the photomultiplier, thereby balancing the dark current and the anode voltage in the amplifying stage. The ac voltage at the output of the system was balanced with part of the ac voltage on the potential leadouts from the sample taken from a divider. The arms of the divider thereby determine the ratio of those voltages entering into formula (3). This method of determining the ratio $V_{\sim}/I_{\sim}$ eliminates the errors associated with readings of the scales of pointer instruments and so increases the accuracy of measurement. A dc voltage proportional to the luminosity being measured is determined using a separate potentiometer circuit similar to that used in the heating circuit. The construction of the setup and the measuring circuit are described in more detail in [6]. The measurements of the specific heat were preceded by the determination of the coefficient α and the determination of the limit of linearity of the measuring system, characterized by the maximum value of the

signal, I_{max}. During measurements at high temperatures, when the values of I approach this limiting value, a neutral attenuating light filter was placed in front of the photomultiplier.

One of the most important questions in the measurement of the specific heat by the present method is that of determining the absolute temperature. In the above setup an OPM-19 optical pyrometer was used to determine the brightness temperature. The pyrometer was sighted on the sample through a total reflecting prism placed on the glass of the chamber. The pyrometer readings were corrected for absorption in the glass of the chamber and the prism. In order to convert the brightness temperature into absolute temperature we require to know the spectral degree of blackness; the latter can be taken from [7].

Another way of determining absolute temperature is to construct a scale of temperature in terms of resistance, to which end use must be made of a black-body model [5]. This method has the advantage that it renders the experiment closed; no recourse need be made to literature data. As far as the present experiment is concerned, there is, however, the disadvantage that the resistance of rhenium at high temperatures is not particularly stable. We were able to show that the resistance of rhenium in a vacuum of 10^{-5} mm Hg depended considerably on the temperature at which the sample was annealed and on how long the sample was held at low temperatures. In this work we have translated the brightness temperature into absolute on the basis of tabulated values of the degree of blackness; also, for control purposes, an experiment was performed in the black-body model. This experiment, which was carried out by S. N. Banchila, consisted in the following.

A wire of the investigated rhenium was positioned along the axis of a thin-walled tantalum cylinder of diameter ~15 mm and length ~50 mm. The ends of the cylinder were closed with caps of sheet tantalum. The wire was connected with current and potential leadouts. The middle of the cylinder was provided with a small hole on which an optical pyrometer was sighted. The entire system was placed within the inductor of a high-frequency furnace.

The objects of investigation were rhenium wires of diameters 0.2 and 0.3 mm made by the powder metallurgy method. The rhenium content in the samples was 99.99%.

The wire was annealed in a vacuum of 10^{-5} mm Hg for a few hours. The mass of a known length of wire was determined before and after the experiment by weighing.

The calculations were carried out using formula (3).

The results of the calculations are shown in Fig. 1 and in Table 1. Random measurement error is characterized by a mean-square deviation of ~0.06 cal/g-atom · °K in a series of 10 measurements; the confidence interval of the results is ~0.14 cal/g-atom · °K at 0.95 reliability. The systematic error in the experimental data, by the estimates of [3, 4], is 3-5%. The main sources of error are the imprecise measurement of T (contribution up to ~2%), the error in the determination of α (~1%), and the error in the measurement of the ratio $V_{\sim}/I_{\sim}$ [see formula (3)]. The experimental data on the specific heat C_p are in good agreement with the results of calculations via Filippov's generalized formula [4]:

$$C_p = 6 + 3\frac{T}{T_m}. \quad (4)$$

In the course of the experiments we also determined the electrical resistivity and the integrated degree of blackness of rhenium (see Table 1); the systematic errors in the cited values are, respectively, ~1 and ~5%.

NOTATION

$V_{=}$, $V_{\sim}$, dc and ac components of voltage across a segment of the wire; m, R, mass and the electrical resistance of the segment; ω, angular frequency of the ac voltage; $|\Theta|$, amplitude of the temperature oscillations; $I_{\sim}$, amplitude of the alternating signal at the output of the recording device, which is proportional to $|\Theta|$; I, dc component of the signal at the output of the device, which corresponds to the mean temperature of the sample $\bar{T}$; c_p, specific heat; C_p, specific heat per g-atom; T_m, melting point of the sample; α, coefficient of proportionality.

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THERMAL CONDUCTIVITY AND ELECTRICAL RESISTIVITY OF NiTi IN THE TEMPERATURE INTERVAL 90-450°K

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Results of measurements of the thermal conductivity and the electrical resistivity of the metallide NiTi are reported.

The aim of the work reported here was to determine experimentally the thermal conductivity and the electrical resistivity of the metallide compound NiTi, which possesses the property of being able to "remember its shape."

The "shape-memory effect" is based on a martensitic transition and is expressed in the ability of a sample of this compound deformed at a temperature below the transition temperature T_{tr} to completely recover its shape after it is heated above T_{tr} [1-5].

According to most papers on the subject, the metallic NiTi possesses a complex crystal structure that varies depending on temperature. At a temperature of around 650°C an ordering of the structure of the high-temperature phase occurs, and below this temperature the compound has an ordered structure of CsCl type. The ordering of the high-temperature phase is accompanied by a reduction in the electrical resistivity [6]. At 60-120°C a martensitic-type phase transition occurs as a result of which the lattice of the metallide readjusts to form a more complex structure, a consequence of which is the appearance of hysteresis in the temperature dependences of the properties of NiTi. The temperature of the martensitic transition of NiTi, and so also the temperature at which shape recovery occurs, is essentially dependent on the chemical composition of the compound [3].

The properties of the metallide NiTi (51.5 at. % Ni) were measured using samples of length 80 mm and diameter 8 mm containing impurities (weight %): Co—0.19; Al—0.05; Fe—0.09; Cr ≤ 0.01; C—0.057; Si—0.09; S—0.01; Mn ≤ 0.01. The density of the material of the samples at 22°C was 6.51 g/cm^3.

The thermal conductivity was measured using an apparatus designed on the basis of the method of steady longitudinal heat flow [7]. A feature of the apparatus is the presence of a screen with an independent heater

TABLE 1. Thermal Conductivity and Electrical Resistivity of the Metallide NiTi

T, °K	90	100	150	200	250	300	350	400	450
λ, W/m·deg	10,5	12,0	16,5	18,5	18,7	18,3	—	—	—
ρ, μΩ·m	—	—	0,490	0,567	0,643	0,715	0,760	0,775	0,795

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