

HEAT CAPACITY OF TITANIUM IN THE TEMPERATURE RANGE 1500 TO 1900 K MEASURED BY A MILLISECOND PULSE-HEATING TECHNIQUE

E. Kaschnitz and P. Reiter

Österreichisches Giesserei-Institut, Parkstrasse 21, A-8700 Leoben, Austria

Abstract

Heat capacity of titanium in the temperature range 1500 to 1900 K was measured by a pulse-heating system, operating in the millisecond time regime. The measurement technique is based on self-heating of a tube-shaped specimen from room temperature to melting in approximately 500 ms and measuring current through the specimen, voltage drop along a defined portion of the specimen, and temperature of the specimen every 0.5 ms. A total of 12 measurements on 3 tube-shaped specimens was made. The reproducibility between individual measurements was 0.4%, between different specimens 0.7%. The extended measurement uncertainty (at a confidence level of 95%) is estimated to be 3%.

Keywords: high temperature, pulse heating, specific heat capacity, titanium, transient techniques

Introduction

Values reported in the literature for the heat capacity of titanium above the α - β transformation point are in considerable disagreement of up to 15%. This may be expected in view of the reactive nature of titanium as well as measurement difficulties associated with the phase transformation at high temperature. In order to reduce the effects of these problems, a subsecond-duration pulse-heating technique was used in the present work to measure the specific heat capacity of titanium in the temperature range from 1500 to 1900 K. This technique eliminates any significant contact between the specimen and the surroundings.

The method is based on rapid resistive self-heating of the specimen from room temperature to high temperature in a short time by the passage of an electrical current pulse through the specimen. Simultaneously, specimen temperature, current through the specimen, and voltage drop across a defined portion of the specimen are measured.

Experimental

The pulse-heating system uses heavy-duty batteries to supply the current. The current through the tubular specimen is determined by measuring the voltage across a stan-

standard resistor connected in series with the specimen. The voltage across the middle part of the specimen is measured between spring-loaded knife-edge probes. They are mounted to stationary clamps via a lever-mechanism that allows the probes to move in the axial direction to follow the thermal expansion of the specimen during the experiment. The temperature is measured by means of a high-speed single wavelength pyrometer, targeted at a small sighting hole machined through the wall of the specimen, thereby approximating a blackbody cavity. The target size of the pyrometer is 0.2 mm. The working wavelength of the pyrometer is 902 nm, selected by an interference filter with a bandwidth of 20 nm. The radiation is collected by a silicon PIN photodiode and converted to voltage by precision amplifiers. The pyrometer is calibrated by comparison with a tungsten-filament standard lamp, which in turn has been calibrated by the Deutscher Kalibrierdienst (DKD). Small corrections are made for the deviation from blackbody conditions (approx. 0.8 K) and for scattered light effects (approx. 6.3 K) [1].

The experimental quantities are recorded simultaneously every 0.5 ms by a data acquisition system with sample-and-hold amplifiers with full-scale resolution of 16 bit. Details regarding construction of the system are given in [2].

Measurements

A total of 12 measurements of specific heat capacity was performed on three tubular specimens in the temperature range from 1500 to 1990 K. The nominal dimensions of the tubes were: length, 75 mm; outside diameter, 10.2 mm; wall thickness, 0.8 mm. For the temperature measurements, a rectangular hole (0.5×1.3 mm) was machined through the wall. The departure from ideal blackbody conditions for the specimen was estimated to be smaller than 0.5% [3].

On the basis of typical analyses by the manufacturer, Goodfellow Metals, Cambridge, UK, the specimen material is reported to be 99.6% pure, containing the following impurities (in ppm per mass): O, 2000; Fe, 1500; Al, Si, C, 300 each; Sn, 200; N, 150; Mn, 100; H, 60; Cr, Ni, 50 each; Ca, 20; Cu, 5.

All experiments were performed either in an argon environment or under high-vacuum conditions to prevent problems associated with chemical reactions. All temperatures reported in this paper are based on the International Temperature Scale of 1990 (ITS-90) [4, 5].

Specific heat capacity was computed from data taken during the heating period of the millisecond experiments. A correction for power loss due to thermal radiation of up to 3% at the highest temperatures was made using data measured during the cooling of the specimen. From the power balance for the specimen during heating and cooling periods, the specific heat capacity c_p as a function of temperature T can be expressed as

$$c_p(T) = \frac{U(T)I(T)}{m \left[\left(\frac{dT}{dt} \right)_h(T) - \left(\frac{dT}{dt} \right)_c(T) \right]} \quad (1)$$

where U is voltage, I is current, m is effective mass, and dT/dt is the heating rate (subscript h) and cooling rate (subscript c), respectively. Heating and cooling rates are treated as independent variables and are obtained by a polynomial fit to the temperature vs. time data for the heating period and by an exponential fit for the cooling period.

Results

The variation of specific heat capacity as a function of temperature is shown in Fig. 1 and Table 1. The reproducibility of measurements for an individual specimen is 0.4% and that between different specimens is 0.7%. The least-squares fit to the specific heat capacity vs. temperature data, in the range $1500\text{ K} < T < 1900\text{ K}$, is

$$c_p = -1627.7 + 4.2327T - 2.6858 \cdot 10^{-3}T^2 + 5.9235 \cdot 10^{-7}T^3 \quad (2)$$

where c_p is in $\text{J kg}^{-1} \text{K}^{-1}$, and T is in K.

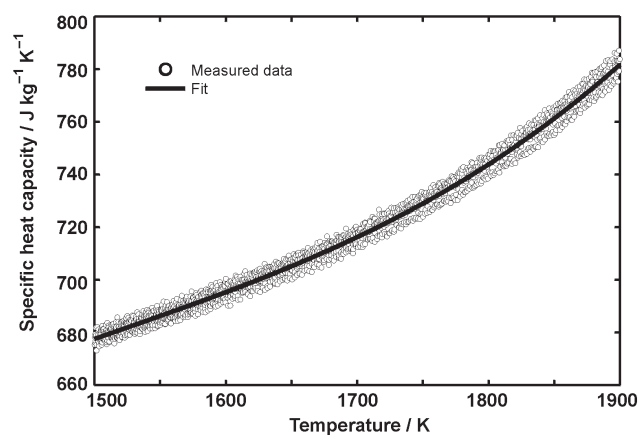


Fig. 1 Individual measurement data and smoothed heat capacity of titanium

Table 1 Specific heat capacity of titanium

T/K	$c_p/\text{J kg}^{-1} \text{K}^{-1}$
1500	677
1550	686
1600	695
1650	705
1700	716
1750	729
1800	744
1850	761
1900	782

Table 2 Sources of uncertainty in specific heat capacity measurements^a

Source of uncertainty	Estimated value	Standard uncertainty	Sensitivity coefficient	Uncertainty contribution/ J kg ⁻¹ K ⁻¹
Electrical measurement related:				
Shunt resistor	40.0 μΩ	0.12 μΩ	18.00 J kg ⁻¹ K ⁻¹ (μΩ) ⁻¹	1.80
Shunt voltage	76.12 mV	0.05 mV	9.46 J kg ⁻¹ K ⁻¹ (mV) ⁻¹	0.47
Voltage divider	0.500	0.001	1440 J kg ⁻¹ K ⁻¹	1.44
Voltage	2.405 V	0.002 V	299.4 J kg ⁻¹ K ⁻¹ (V) ⁻¹	0.60
Specimen related:				
Effective length	34.7 mm	0.1 mm	20.8 J kg ⁻¹ K ⁻¹ (mm) ⁻¹	2.08
Total length	75.11 mm	0.05 mm	9.59 J kg ⁻¹ K ⁻¹ (mm) ⁻¹	0.48
Total mass	7.439 g	0.005 g	96.8 J kg ⁻¹ K ⁻¹ (g) ⁻¹	0.48
Purity	99.6%	0.2%	7.23 J kg ⁻¹ K ⁻¹ (%) ⁻¹	1.45
Pyrometer related (values in K, coefficients in J kg ⁻¹ K ⁻²):				
Tungsten filament lamp	1700.0	1.5	1.64	2.46
Geometry differences	1700.0	1.0	1.64	1.64
Effective wavelength	1700.0	0.5	1.64	0.82
Non-linearity	1700.0	0.5	1.64	0.82
Calibration transfer	1700.0	1.0	1.64	1.64
Radiance source align.	1700.0	1.0	1.64	1.64
Noise and digitization	1700.0	0.2	1.64	0.33
Temperature measurement related (values in K, coefficients in J kg ⁻¹ K ⁻²):				
Secondary standard drift	1700.0	1.0	1.64	1.64
Window calibration	1700.0	0.5	1.64	0.82
Pyrometer calibration drift	1700.0	1.0	1.64	1.64
Blackbody quality	1700.0	0.3	1.64	0.49
Heating rate/K s ⁻¹	3709	44	0.194 ^b	8.56
Total uncertainty/J kg ⁻¹ K ⁻¹	720.0			10.4

^a The probability distribution is incorporated in the standard uncertainty

^b Unit of related coefficient is: J kg⁻¹ K⁻¹ (K s⁻¹)⁻¹

Estimate of uncertainties

An analysis of possible errors for specific heat capacity at 1700 K is given in Table 2. The uncertainty in the measured values of specific heat capacity is calculated as recommended in EA-4/02 [6] and is estimated to be less than ±3% for the entire temperature range from 1500 to 1900 K. The reported uncertainties are based on the standard uncertainty multiplied by a coverage factor of 2, providing a level of confidence of approximately 95%.

Discussion

Values for the heat capacity of β -phase titanium reported in the literature were derived from drop [7–11], modulation [12], adiabatic [13], and pulse calorimetry [14], and, as can be seen in Fig. 2, are in considerable disagreement. In drop calorimetry, the enthalpy difference between a chosen temperature and room temperature is measured. In most cases [7, 9–11], the obtained enthalpy data above the solid–solid transformation of titanium was fitted with a linear function, leading to a constant value for heat capacity. Temperature dependent values of Kothen [8] are up to 14% higher than the results of the present work. Heat capacity values of Holland [12], obtained by modulation calorimetry, show a significantly different trend from other reported values. Results of Kohlhaas *et al.* [13], obtained by adiabatic calorimetry are approximately the amount of the combined uncertainties lower than the results of the present work. There is a good agreement between our values and those of Cezairliyan and Müller [14], which were also obtained by pulse calorimetry. The average deviation between the present results and those of Cezairliyan and Müller is 0.4% with a maximum deviation of 0.9% at 1500 K.

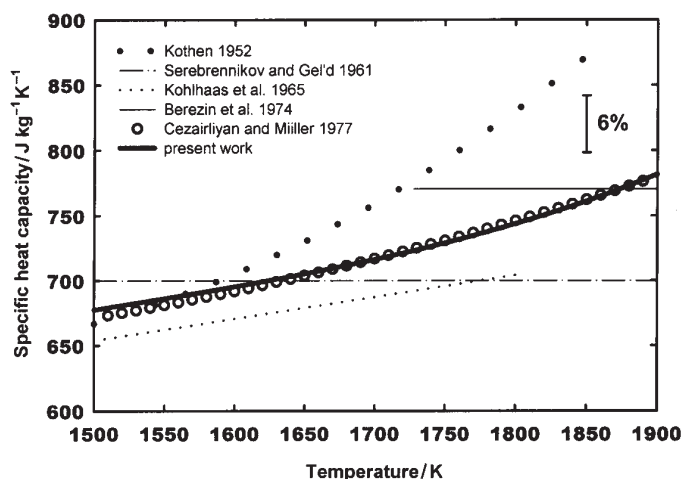


Fig. 2 Heat capacity of titanium: present work and data reported in the literature

It should be noted, that no conversion from IPTS-68 and older temperature scales has been made, since the effect on heat capacity is small and well within the uncertainties of these values. A value of 47.867 for the atomic mass of titanium [15] was used to express heat capacity in units of $\text{J kg}^{-1} \text{K}^{-1}$.

* * *

This work was supported by the Fonds zur Förderung der wissenschaftlichen Forschung (FWF), Vienna, Austria under contract No. P12804-PHY.

References

- 1 A. Cezairliyan, M. S. Morse, H. A. Berman and C. W. Beckett, *J. Res. Natl. Bur. Stand.*, 74A (1970) 65.
- 2 P. Reiter and E. Kaschnitz, *High Temp.-High Press*, in press.
- 3 J. C. De Vos, *Physica*, 20 (1954) 669.
- 4 H. Preston-Thomas, *Metrologia*, 27 (1990) 3.
- 5 H. Preston-Thomas, *Metrologia*, 27 (1990) 107.
- 6 EA-4/02, *Expression of the Uncertainty of Measurement in Calibration*, European co-operation for Accreditation, <http://www.european-accreditation.org/> 1999, p. 1.
- 7 F. M. Jaeger, E. Rosenbohm and R. Fonteyne, *Rec. Trav. Chim.*, 55 (1936) 615.
- 8 C. W. Kothen, Ph. D. thesis, Ohio State University, Columbus 1952, p. 1.
- 9 Yu. M. Golutvin, *Russ. J. Phys. Chem.*, 33 (1959) 164.
- 10 N. N. Serebrennikov and P. V. Gel'd, *Non-Ferrous Metall.*, 4 (1961) 80.
- 11 B. Ya. Berezin, S. A. Kats, M. M. Kenisarin and V. Ya. Chekhovskoi, *High Temp.*, 12 (1974) 450.
- 12 L. R. Holland, *J. Appl. Phys.*, 34 (1963) 2350.
- 13 R. Kohlhaas, M. Braun and O. Vollmer, *Z. Naturforsch., Teil A* 20 (1965) 1077.
- 14 A. Cezairliyan and A. P. Miiller, *High Temp.-High Press*, 9 (1977) 319.
- 15 *Atomic Weights of the Elements 1997*, *Pure Appl. Chem.*, 71 (1999) 1593.