

Specific Heat and Electrical Resistivity of 53% Niobium-47% Titanium Alloy Measured by Subsecond Calorimetric Technique¹

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This paper presents results of measurements of the specific heat and electrical resistivity of a 53%Ni-47%Ti superconducting alloy. Both properties were measured by a contact variant of the millisecond-resolution pulse calorimeter. W5%Re/W25%Re thermocouple thermometry enabled study from ambient temperature to 2000 K. Results are discussed, and their uncertainty is estimated.

KEY WORDS: electric conductors; electrical resistivity; high temperatures; niobium titanium alloy; specific heat capacity.

1. INTRODUCTION

The specific heat and electrical resistivity of a 53%Ni-47%Ti superconducting alloy have been investigated at temperatures from ambient to 2000 K, using a contact thermometry variant of the millisecond-resolution pulse calorimeter. Samples from the same batch were used for specific heat measurements at the National Institute of Standards and Technology in the high temperature range, employing a high-temperature variant of the millisecond-resolution pulse calorimeter. Experimental results of current measurements are presented and compared to the NIST high temperature data.

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2. MEASUREMENTS

2.1. Method

The method, apparatus, experimental procedure, and measurement uncertainties have been described in detail in previous papers [1, 2]. The method is based on rapid heating of a sample in the form of a thin rod or wire, from ambient to the desired maximum temperature. During the heating of the sample, the data on the current and voltage drop over the effective measurement zone of the sample and the thermocouple electromotive force, *EMF*, are collected. The thermocouple is welded intrinsically at the center of the effective zone. The *EMF* of the thermocouple is also determined during the initial part of the sample cooling period for calculation of the total hemispherical emissivity.

The basis for calculations is the energy balance of the effective part of the sample during heating:

$$UI = mC_p \left(\frac{\partial T}{\partial t} \right)_h + P_{\text{loss}} \quad (1)$$

where m is the mass of the effective sample, I is the current passing through the sample, U is the voltage drop over the effective sample length, C_p is the specific heat capacity, and $\left(\frac{\partial T}{\partial t} \right)_h$ is the first derivative of the temperature signal during the heating period. The radiative power loss, P_{loss} , is determined from the initial portion of the sample cooling period:

$$-mC_p \left(\frac{\partial T}{\partial t} \right)_c = P_{\text{loss}} \quad (2)$$

where $\left(\frac{\partial T}{\partial t} \right)_c$ is the first derivative of the temperature signal during the initial part of the cooling period.

As the experiments are carried out under vacuum of the order of 10^{-3} Pa, and the heating rates amount to $2800 \text{ K} \cdot \text{s}^{-1}$, heat losses due to convection and conduction can be neglected. This leaves radiative heat losses only, which are accounted for in Eq. (1) and defined in Eq. (2).

The specific heat is then determined from

$$C_p = \frac{UI - \varepsilon_t \sigma A (T^4 - T_0^4)}{m \left(\frac{\partial T}{\partial t} \right)_h} \quad (3)$$

where T is the sample temperature and T_0 is the ambient temperature. The total hemispherical emissivity, ε_t , is computed using the expression,

$$\varepsilon_t = \frac{UI}{\sigma A(T^4 - T_0^4) \left[1 - \left(\frac{\partial T}{\partial t} \right)_h / \left(\frac{\partial T}{\partial t} \right)_c \right]} \quad (4)$$

where σ is Stephan-Boltzmann's constant and A is the surface area of the effective part of the sample. The temperature derivative is computed for both the sample heating and cooling, as a function of the temperature.

The electrical resistivity is computed from Ohm's law,

$$\rho = \frac{US}{IL} \quad (5)$$

where S is the cross-sectional area of the sample and L is the effective sample length. The voltage drop is measured between the potential leads, mounted at 15 mm separations on each side of the thermocouple, and the current I is measured for a standard resistor in series with the sample.

2.2. Experimental

Three specimens of 53%Ni-47%Ti superconducting alloy wire provided by the late Dr. Ared Cezairliyan, NIST, were in the form of a wire, nominally 1.5 mm in diameter and 250 mm in length. For more details on the specimens' chemical composition and purity, the reader is directed to the manufacturer (the NIST reference for the specimen manufacturer is Astrolite Alloys, Via Alondra, Camarillo, California 93010).

dc pulses were provided by two heavy duty 12 V batteries connected in series. Pulses lasted from one to two seconds, depending on the desired heating rate. Dynamic specimen temperatures during pulse experiments were measured by 0.1 mm diameter W5%Re/W25%Re thermocouples. Details on derivation of true temperature from measured EMF are given in Ref. 1. Thermometry was based on the International Temperature Scale of 1990.

In order to stabilize the material crystal structure, the specimens were preheated at 1300 K before the pulse experiments.

3. RESULTS

The electrical resistivity and specific heat capacity were measured in 30 experiments, sixteen of them reached a temperature close to the maximum

of 2000 K. The average current ranged between 100 and 150 A. Each individual experiment resulted in a continuous set of electrical resistivity and specific heat data, covering a part of or the complete temperature range. All the raw data were used to obtain a single least-squares fitted polynomial, both for electrical resistivity and for specific heat capacity.

The polynomial representing the electrical resistivity in the range 300 to 2000 K has the form:

$$\rho = 6.0765 \times 10^{-7} + 4.3652 \times 10^{-10}T - 5.3132 \times 10^{-14}T^2 \quad (6)$$

and the specific heat capacity, in the same temperature range, is represented by a polynomial:

$$Cp = 382.4131 + 0.0863T - 4.1515 \times 10^{-5}T^2 + 2.5565 \times 10^{-8}T^3 \quad (7)$$

Table I. Thermophysical Properties of a 53%Niobium-47%Titanium Alloy

Temperature (K)	Specific heat ($\text{J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$)	Electrical resistivity ($\mu\Omega \cdot \text{m}$)		Hemispherical total emissivity
290.85		0.7229 ^c	0.7301 ^a	
291.45		0.7323 ^b	0.7304 ^a	
300	405.3		0.734	
400	411.9		0.774	
500	418.4		0.813	
600	424.8		0.850	
700	431.2		0.887	
800	437.9		0.923	
900	445.1		0.957	
1000	452.8		0.991	0.2268
1100	461.1		1.024	0.2358
1200	470.3		1.055	0.2448
1300	480.6		1.085	0.2538
1400	492.0		1.115	0.2627
1500	504.7		1.143	0.2717
1600	518.9		1.170	0.2807
1700	534.7		1.196	0.2896
1800	552.3		1.221	0.2986
1900	571.8		1.245	0.3076
2000	593.5		1.268	0.3166

^a Values from fitted polynomial.

^b Room-temperature value measured using the four-probe method with current reversal, before the first experiment.

^c Room-temperature value measured using the four-probe method with current reversal, after the final experiment.

Deviations of electric resistivities obtained in individual experiments from Eq. (6) are about $\pm 0.4\%$ at 300 K and about $\pm 0.5\%$ in the middle and upper parts of the measuring range.

Corresponding deviations of specific heat capacities from Eq. (7) were about $\pm 1.7\%$ in the middle of the range, and about $\pm 3\%$ at both ends of the measuring range.

The electric resistivity of one of the specimens was measured also at ambient temperature using stationary state four-probe-current-reversal method, before and after the end of pulse experiments. These measurements gave $0.7323 \mu\Omega \cdot \text{m}$ at 291.45 K before, and $0.7229 \mu\Omega \cdot \text{m}$ at 290.85 K after, comparing well with respective values of 0.7304 and $0.7301 \mu\Omega \cdot \text{m}$ computed from Eq. (6) at these temperatures.

The hemispherical total emissivity, which was necessary for calculation of C_p was calculated using data collected in the high-temperature range and in the initial part of the cooling period. The average hemispherical total emissivity follows a linear function in the range of 1000 to 2000 K, and is represented by

$$\varepsilon = 0.1371 + 8.9727 \times 10^{-5} T \quad (8)$$

Numerical values of the measured quantities are presented in Table I at 100 K increments.

4. DISCUSSION

The electrical resistivity function Eq. (6) is shown in Fig. 1. This figure also contains recently published high temperature data of Basak et al. [3], which was the only available data set of this particular Nb-Ti composition.

Both data sets have the same general pattern. The NIST data lie about 1.3% below our results; this difference is probably a result of the present electrical resistivity data not being corrected for thermal expansion. In this sort of experimental setup, the material specimen in the form of a thin wire can sometimes suffer significant thermal deformation, which depends on the nature of the investigated material and its behavior during intensive heating. Visual observation during the experiments showed a tendency of mechanical deformation due to specimen thermal expansion at higher temperatures. The same tendency was also detected in measurements on pure titanium [4]. Rough calculations showed that an increase of the effective length of the sample of 0.5 mm could result in a decrease of the electrical resistivity of about 1.5 to 2%, which would reduce the differences between the two data sets.

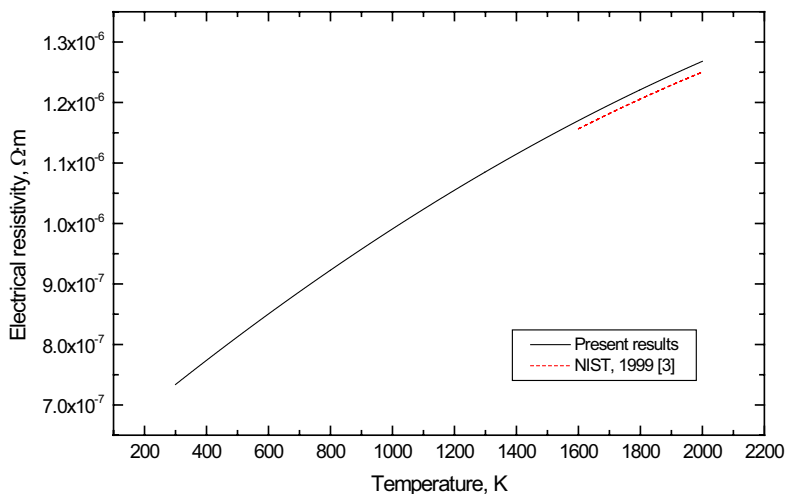


Fig. 1. Electrical resistivity of 53%niobium-47%titanium as a function of temperature.

Electrical resistivity measurements carried out on one specimen before and after the pulse experiments using stationary state four-probe-current-reversal method showed a decrease in the room temperature electrical resistivity of 1.2%.

The specific heat capacity function Eq. (7) is shown in Fig. 2, again together with high temperature data of Basak et al. [3]. The latter lie

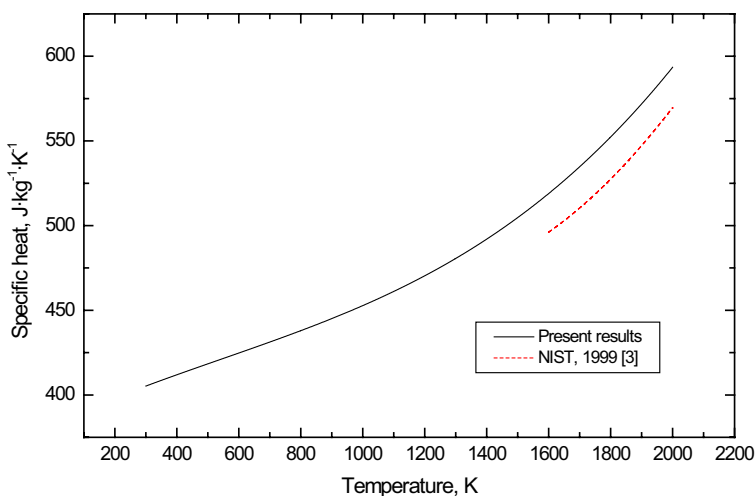


Fig. 2. Specific heat capacity of 53%niobium-47%titanium as a function of temperature.

about 4% lower. Both sets suggest contributions from vacancy formation at high temperatures.

The estimated maximum uncertainties in determining the electrical resistivity, specific heat capacity, and emissivity by the millisecond-resolution pulse technique were estimated at ± 1 , ± 3 , and $\pm 5\%$, respectively [1], which are in fair agreement with standard deviations determined for this set of experimental results. Uncertainties have maximum values close to the lower and upper limits of the measurement range. A paper with a new evaluation of the method, which will contain a more elaborate consideration of measurement uncertainties, is expected to appear within the next year.

5. CONCLUSION

The electrical resistivity and specific heat capacity data of 53%niobium-47%titanium alloy obtained in this research should contribute to a better understanding of thermal properties of this particular alloy in the complete temperature range from room temperature to 2000 K. Considering possible superconducting applications of this material, it would be of interest to have respective data of those properties below room temperature.

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