Experimental investigation of thermal expansion of niobium at high temperatures

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Abstract. Experimental study of thermal expansion of niobium by millisecond pulse heating technique was performed. Using this technique, an experimental determination of the temperature dependence of thermal expansion coefficient, electrical resistivity, enthalpy and specific heat capacity at premelting region for niobium have been carried out. The results obtained allow calculating the density of Nb in the wide range of high temperatures up to the melting point.

1. Introduction
Millisecond electrical pulse heating is a prominent technique for investigation of refractory metals at high temperatures. Meanwhile, some properties of such materials are still insufficiently investigated at this temperature region. For niobium such properties as density change during melting is of special interest.

In this paper we describe a technique for investigation of the thermal expansion of refractory materials at high temperatures and in the melting region by pulse electrical heating [1–2]. This work contains the experimental study of the temperature dependences of thermophysical properties of niobium at high temperatures and in the melting region by pulse electrical heating. The data obtained by concerned technique can be useful both for theoretical studies in constructing wide-range equation of state for refractory metals [3] and for solving the problems of high-temperature engineering.

2. Experimental technique
The experimental method consists in fast heating of the wire sample up to the melting temperature and above in a time of about 3—4 milliseconds due to homogeneous volumetric heat release when the electric current pulse passes through it. Heating is carried out under isobaric conditions at a static pressure of buffer inert gas (Ar) of about 50 bar. The input energy or enthalpy change $\Delta H(T)$ can be determined by measuring the current $I(t)$ and the voltage drop $e(t)$ between the potential probes in the central part of the sample.

Temperature measurements are performed by a two-channel optical pyrometer that implements the spectral ratio method. Thus, by measuring the surface temperature of the sample during the experiment $T(t)$, current $I(t)$ and voltage $e(t)$, one can determine the dependence of the enthalpy change $\Delta H(T)$, as well as the heat capacity $C_p(T)$.

The expansion measurement technique used in previous papers [4] was modified, which led to a significant increase in accuracy. The measurements of the relative expansion of the sample using the data for cross section of the specimen had an error of the order of 10%. Current approach allows measuring also the relative elongation of the heated sample in a solid state with an accuracy of about
2%, which is better than cross section measurements for almost an order of magnitude. This experimental technique for thermal elongation measurements developed for refractory carbides is discussed in detail in [5].

The main principle of the experimental technique for thermal expansion measurements is the measurement of the absolute linear elongation of a heated sample by determining the changing position of one of the collets with which the sample is attached, while the second collet remains stationary. To determine the position of the collet, a pointer was mounted on it, and a laser with a wavelength of 532 nm and a power of up to 0.5 W is used for backlight illumination of the pointer during the heating.

3. Experimental data
The wire samples had cylindrical form with diameter of 0.8 and length of about 7 mm, 14 experiments were conducted. Niobium wire with purity of 99.99% (metal basis excluding Ta, Ta < 300 ppm) was used in the experiments.

During the experiments, measurements of such parameters of a heating pulse as current and voltage drop across the central part of the sample between the potential probes were made. Knowing these values, as well as the geometrical dimensions of the sample under study, it is possible to obtain the time and temperature dependences of the electrical resistivity of niobium at high temperatures. This dependence is shown in figure 1.

![Figure 1. Experimental data for dependence of the electrical resistivity of niobium on specific enthalpy in comparison with the literature data [6–8].](image-url)
In the temperature range 1750—2600 K this dependence can be approximated by the following polynomial (temperature in K, electrical resistivity in μOhm*m):

\[ \rho_{el} = 0.36831 + 1.07041 \times 10^{-4}T + 2.92738 \times 10^{-8}T^2 \]

The experimental data obtained allow us to measure the temperature dependence of the specific enthalpy of niobium up to the melting temperature. The true temperature was calculated from two brightness temperatures at wavelengths of 0.650 and 0.862 μm, using the temperature dependence of emissivity in the temperature range 1500—2200 K, the value of the emissivity at these temperatures was taken from [9]. Comparison of the values of specific enthalpy at the beginning of melting with the literature data is shown in table 1.

**Table 1.** Specific enthalpy of Nb at the beginning of melting.

| Source          | \( \Delta H \) (kJ g\(^{-1}\)) |
|-----------------|----------------------------------|
| This work       | 0.805                            |
| Wilthan [8]     | 0.795                            |
| Glushko [10]    | 0.833                            |
| Berezin [11]    | 0.773                            |
| Kirillin [12]   | 0.865                            |

The data on specific enthalpy also allow calculating the specific heat capacity of the material under study. The methodology used for this is considered in detail in [13]. The value of average specific heat capacity for the temperature 2000 K is presented in table 2.

**Table 2.** Average heat capacity of Nb for the temperature 2000 K.

| Source          | \( C_P \) (J g\(^{-1}\)K\(^{-1}\)) |
|-----------------|-----------------------------------|
| This work       | 0.353                             |
| Boboridis [6]   | 0.357                             |
| Righini [7]     | 0.361                             |
| Glushko [10]    | 0.366                             |
| Kirillin [12]   | 0.379                             |

Since niobium is an isotropic material, measurements of thermal expansion of it allow calculating the thermal dependence of density. The experimental data on density of niobium are presented in figure 2. The literature data from [14–16] are also depicted, the agreement between the experimental data and the literature data is reasonable.
Figure 2. Experimental dependence of density of niobium in comparison with literature data [14–16].

4. Conclusion
In this work the experimental data on the specific enthalpy, heat capacity and electrical resistivity of niobium up to the temperatures close to the melting point were discussed. The dependence of density of niobium at these temperatures was also calculated. The experimental data for these thermophysical properties of Nb are in good agreement with those available in the literature.

Acknowledgments
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