Experimental study of thermal expansion of chromium near its melting point

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Abstract. Experimental study of thermal expansion of chromium by millisecond pulse heating technique was performed. The results obtained allow calculating the density of Cr 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, especially chromium, are still insufficiently investigated at this temperature region.

In this paper, we use a modification of technique for investigation of the thermal expansion of refractory materials at high temperatures and in the melting range by pulse electrical heating [1]. This modification was suggested in [2] as applicable to refractory carbides. Some motivation for this modification is discussed in detail. This work contains the experimental study of the temperature dependences of thermal expansion of chromium at high temperatures and by pulse electrical heating. The data obtained by present technique can be useful for theoretical studies [3], constructing wide-range equation of state for various refractory metals [4, 5] and for solving the problems of high-temperature engineering, because the literature data for thermophysical properties of chromium at the melting point and in the liquid phase are obviously insufficient. For example even the melting temperature shows some discrepancy: 2131±5 [6], 2120–2150 [7], 2180 K [8].

2. Experimental technique
The developed method consists in fast heating of the sample up to the melting temperature and above in a time of about 2–4 milliseconds due to homogeneous volumetric heat release when an electric current pulse passes through it. Heating is carried out under isobaric conditions in a high-pressure chamber at a static pressure of a buffer inert gas (Ar) up to 2 kbar. 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 from [2] was used, 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% [1]. 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 for almost an order of magnitude.

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 is mounted on it, and a laser with a wavelength of 532 nm and a power of up to 0.5 W is used for illumination. The scheme of the experimental technique was presented in [2].

It is important to note that due to the use of the backlight of the pointer allocated to the heated sample, the possibility of optical distortions due to heating of the dense gas layer near the sample is excluded. As was shown earlier [2], these distortions can lead to errors in the measurement of thermal expansion comparable with the magnitude of the expansion itself. The nature of these distortions is explained in details in figure 1.

During the experiment, the heated sample heats up the adjacent gas layer through a thermal conductivity process. The heated layers become less dense, and, therefore, have a lower refractive index than unheated. It changes the path of the laser beam, visually increasing the shadow of the sample in the light of the laser backlight.

Figure 1. Scheme of the optical distortion when measuring the thermal expansion of the sample using laser backlight.
Table 1. Residual elements for material under study, weight percent; aggregate percent of K, Li, Mg, Mo, Mn, Na, W is not more 0.01.

|   | Fe  | Al  | S   | Ni  | Si  | Cu  | N   | C   | O   |
|---|-----|-----|-----|-----|-----|-----|-----|-----|-----|
|   | 0.008 | 0.001 | 0.002 | 0.001 | 0.004 | 0.001 | 0.002 | 0.004 | 0.001 |

For estimation of the of the deviation angle $\alpha$ one can use equation from [9] for deviation of light in the medium with changing refraction index $n$:

$$\alpha = \frac{l}{R} = \frac{l}{n} \frac{dn}{dx},$$

where $l$ is the typical size of the heated region, $R$ is the radius of the deflection curve, $x$ is position on the deflection curve. Assuming that for the dense gas refraction index is proportional to the gas density due to Gladstone–Dale relation and because the density is reversely proportional to the gas temperature, one can use the solution of one-dimensional heat-transfer equation for semi-infinite body with a constant boundary temperature to find the gradient of the refraction index [10]:

$$\frac{dn}{dx} = \frac{2\sqrt{\pi\chi t}}{n_2 - n_1} \exp\left(\frac{x^2}{4\chi t}\right),$$

where $\chi$ is thermal diffusivity, $t$ is time of heating, $n_1$ and $n_2$ are refraction indexes of heated and unheated gas respectively. Now taking as refraction index $n$ for each point average value of it in the heated area one can obtain estimation for deflection angle:

$$\alpha = \frac{l}{n} \frac{2\sqrt{\pi\chi t}}{n_2 - n_1} \exp\left(\frac{x^2}{4\chi t}\right) = \frac{2l}{n_2 + n_1} \frac{2\sqrt{\pi\chi t}}{n_2 - n_1} \exp\left(\frac{x^2}{4\chi t}\right) = \frac{4l\sqrt{\pi\chi t}}{n_2^2 - n_1^2} \exp\left(\frac{x^2}{4\chi t}\right).$$

Based upon the values of density, heat capacity and thermal conductivity of argon at the considered pressures and temperatures from [11], a typical heating time of 1 ms and the typical size of the heated region of 0.5 mm, it is possible to obtain the estimation of the deflection angle of around $7 \times 10^{-5}$ rad. The estimation was done for Ar gas pressure of 1 kbar and the sample temperature of 2000 K.

According to the optical scheme of shadow photography the distance between the sample and the camera lens is equal to 0.2 m, that ultimately leads to the value of the distortion of the sample size of 0.014 mm for each edge or the errors more than 100% at 2000 K. The obtained estimation is confirmed by experimental results, which are of the same order of magnitude.

At high surrounding gas pressure applying new elongation measurement technique based on absolute elongation of the heated sample (for the refractory metals it can reach up to 0.5 mm near melting temperature) allows effectively reduce the measurement errors in comparison with the method based on the analysis of thermal images of the cross section of the sample that was used in previous works [12, 13]. In addition, the use of continuous laser illumination makes it possible to carry out measurements beginning immediately from the start of heating using the minimum possible exposure. That, along with the increased time of the experiment, also leads to an increase in the number of shooting frames in a single experiment.

3. Experimental data
The samples had a form of parallelepiped, the size was about $0.8 \times 1.3 \times 10$ mm$^3$, 20 experiments were conducted. Chromium with purity of 99.95% was used, the chemical composition is presented in table 1.
Figure 2. Experimental dependence of density of Cr in comparison with literature data [6–8,15]. Black dashed line depicts melting temperature from [6].

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 specific electrical resistivity and specific enthalpy of chromium at high temperatures.

The true temperature was calculated from two brightness temperatures at wavelengths 0.650 and 0.862 \( \mu \text{m} \) under the assumption of a nearly constant emissivity of material in the temperature range 1500–2200 K, the value of the emissivity for red spectral region at these temperatures \( \varepsilon = 0.39 \) was taken from [14]. Electrical resistivity dependence shows good agreement with the literature data [8].

The experimental data on the density of chromium are presented in figure 2. The literature data for the solid phase from [6–8,15] are also depicted.

4. Conclusion
Modification of technique for investigation of the thermal expansion of refractory materials at high temperatures by pulse electrical heating and motivation for this modification were discussed in the article. Experiments with millisecond pulse electrical heating of chromium were performed. During the experiments, data on the electrical resistivity of chromium up to the temperatures close to the melting point were obtained. The thermal dependence of density for Cr at these temperatures was also obtained. The experimental data are in good agreement with those available in the literature.

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