Investigation of thermoradiation properties of carbides of refractory metals at high temperatures by pulse electrical heating

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Abstract. Experimental study of thermoradiation properties of refractory metal carbides by the pulse heating method was performed. Quasi-stationary emission spectra of the eutectic system MoC$_{0.82}$ at high temperatures are obtained, the normal spectral emissivity of this carbide material at two wavelengths is calculated. The efficiency of the chosen technique of spectral pyrometry for studying the thermoradiation properties of refractory carbides at high temperatures is demonstrated.

1. Introduction
Refractory carbides, in particular TaC, HfC and their mixtures, have anomalously high melting temperatures of about 4000 K (which is higher than the melting point of the most refractory metal-tungsten) and retain high strength and durability under extreme thermal loads.

Knowledge of the thermoradiation properties of refractory carbides is extremely important, both in experimental thermophysical studies and in industry. Today in the literature there is practically no data on the thermoradiation properties of these carbides at extremely high temperatures, common in emergency operation modes of the equipment, which makes modeling and control of such regimes more difficult. Some experimental techniques based on combination of rapid electrical heating and polarimetric methods to measure optical constants for metals should be noted [1, 2].

In this paper, we describe a technique for investigation of the thermoradiation properties of refractory materials at high temperatures and in the melting range by pulse electrical heating [3,4]. The data obtained by concerned technique can be useful both for theoretical studies [5,6] and for solving the problems of high-temperature engineering, primarily in connection with the creation of new coating materials for aerospace application and aircraft engines, operating at extremely high temperatures, for innovative plasma processing technologies, and for designing next-generation nuclear reactors.

2. Experimental technique
Such technique as laser heating is often used for high-temperature studies of the thermophysical and optical properties of substances, but because of the large temperature gradients in the bath of the melt on the surface, the carbide composition may differ from the original, which
Figure 1. Scheme of the experimental setup: $T_{0.862}$ and $T_{0.65}$—brightness temperatures for the wavelengths involved; $U(t)$—voltage; $i(t)$—current; $L(t)$—width of the specimen.

complicates the interpretation of such measurements. Probably for this reason practically no information is available in the literature on the optical properties of high-temperature carbides in the temperature range of 2500–3000 K.

The proposed approach and the experimental implementation of a new original system for measuring radiative properties using a millisecond spectrometer and a multichannel microsecond pyrometer make it possible to investigate the spectra of thermal radiation in the range 0.5–0.8 µm, and also to control the homogeneity of sample heating and its integrity.

The millisecond duration of the experiment makes it possible to exclude the interaction of the sample with the external medium and makes it possible to reach extremely high temperatures up to the melting point. Knowledge of thermoradiation properties will essentially improve the accuracy of temperature measurement during the study of thermophysical properties of carbides, will make it possible to refine solutions of radiation-conductive heat transfer problems describing heating to extremely high temperatures. The results obtained will be useful for solid state physics, the calculation of thermodynamic properties in the melting range, high-temperature power engineering and plasma heat treatment.

The experimental setup was described in [7], the modification of this setup consists in the use of additional high-speed spectrometer. Figure 1 shows a schematic diagram of the experimental setup.

The setup is equipped with the AvantesAvaspec-2048 spectrometer with a spectral range of 240–795 nm, the exposure time in the experiments was 1.05 ms. The spectrometer was calibrated against the black body model at a temperature range of 1000–2500 °C. The spectrometer is equipped with a 2048 pixels linear image sensor and fast 16-bit adc.
3. Experimental results

During the experiments the emission spectra of the molybdenum–carbon system of the eutectic MoC\(_{0.82}\) composition heated to 2330 K were obtained. The carbide sample was heated to the required temperature of 2330 K in 1.2 ms. Within 800 ms after the end of heating, which was required to prevent any possible temperature inhomogeneities, the spectrometer was launched. From the beginning of the experiment, a high-speed video camera controls the sample position (and thus controls the aiming accuracy of the spectrometer), as well as the absence of electrical breakdown along the sample. In addition, the values of two brightness temperatures of the sample, the voltage drop and the current flowing through the sample were recorded during the experiment. To prevent the chemical oxidation of the sample surface at high temperatures the experiments were carried out in the atmosphere of the inert gas (argon) at a pressure of 700 bar. The thermograms obtained for one of the experiments for two brightness temperatures at wavelengths of 0.650 and 0.862 \(\mu\)m are shown in figure 2.

Figure 3 shows the experimentally obtained spectra of thermal radiation of the MoC\(_{0.82}\) sample at the true temperatures of 2330 and 1973 K. These spectra were obtained on a temperature plateau, measurements were carried out from 2 to 3 ms. Brightness temperature values on the temperature plateau for the true temperature of 2330 K gives \(T_{0.65} = 2102\) K and \(T_{0.862} = 2035\) K with errors \(\pm 24\) and \(\pm 32\) K respectively.
To determine the temperature of a heated body via the emission spectrum, the method of polychromatic pyrometry was used [8, 9]. It is convenient to express the radiance dependence via Wien coordinates, i.e. by the dependence of $\ln(I\lambda^5)$ upon $C_2/\lambda$. The slope of the line in these coordinates determines the true temperature of the heated body. The dependence of the emissivity on the wavelength only shifts the given line, but does not change its slope angle. In the case of weakly linear spectral dependence of emissivity in a given wavelength interval, the accuracy of such method for a large number of channels is acceptable [9], e.g. typical accuracy for 256 channels equals 50 K (our setup is equipped by the spectrometer with 2048 wavelengths).

Approximation of the experimental data for true temperature of 2330 K by a straight line using the method of least squares gives the value of $R$ square of 0.99964, which reflects the high accuracy of the approximation and, thereby, confirms the initial assumptions about the absence of a pronounced dependence of the emissivity of the substance under study on the wavelength. Another argument for the assumption of weakly linear spectral dependence of emissivity in a given wavelength interval for the involved molybdenum–carbon system is that such behavior of spectral emissivity is typical for the refractory carbide materials [10].

Assuming this temperature to be the true temperature of the heated sample, it is possible to calculate the value of normal spectral emissivity at two wavelengths of the pyrometer. Experiments demonstrate that for both wavelengths 0.650 and 0.862 µm it is close to 0.4 for these temperatures. Since there are no emissivity values for such carbide in the literature, it is reasonable to compare the obtained values of spectral emissivity with pure molybdenum. According to [10], the spectral emissivity of molybdenum at 2300 K equals 0.369 for 0.65 µm, which agrees well with the values obtained for carbide.

Figure 3. Spectrum of thermal radiation of the sample MoC$_{0.82}$ at a temperature of 2330 K.
4. Conclusion

Approbation of the technique of spectral pyrometry on refractory metal carbides with unknown thermoradiation properties at high temperatures is described. Experiments on the modernized setup for studying the thermoradiation properties of refractory electrically conductive substances by the pulse heating method were performed. Quasi-stationary emission spectra of the eutectic system MoC_{0.82} at 1973–2330 K are obtained. Under the assumption of weakly linear spectral dependence of emissivity the normal spectral emissivity of this carbide material at a given temperature at two wavelengths of 0.650 and 0.862 µm is calculated. The applicability of the chosen technique for studying the thermoradiation properties of refractory carbides at high temperatures is demonstrated.

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