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## Mass Spectrometric Study of the Interaction of $Y_2O_3$ with Carbon at High Temperatures

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**Abstract**—The evaporation of  $Y_2O_3$  in the presence of carbon was studied by high-temperature differential mass spectrometry. In the temperature range 1950–2200 K, the vapor above the  $Y_2O_3$ –C system contains YO and atomic yttrium. At elevated temperatures, the partial pressure of YO vapor over the  $Y_2O_3$ –C system decreases compared to the partial pressure of YO vapor over individual  $Y_2O_3$ , leading to a decrease in the activity of yttrium oxide in the system.

**Keywords:** high-temperature mass spectrometry, evaporation,  $Y_2O_3$ –C system

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Ceramic products made from individual highly refractory oxides are widely used in various modern high-temperature processes and especially at temperatures above 2100 K in contact with various materials in various gas environments, for example, with ceramics, refractories, metals, and metal and oxide melts.

Modern lost wax casting technology for special-purpose parts with a directional structure, including blades of aircraft gas turbine engines, involves the use of electric vacuum furnaces with graphite heaters [1]. The use of new generation composite alloys based on refractory metals as an alternative to heat-resistant nickel alloys is associated with the necessity of directional crystallization at temperatures exceeding 2000 K [2]. In industrial furnaces, directional crystallization is generally performed in a reducing carbon-containing atmosphere, which can lead to irreversible phase and structural changes in the ceramic casting mold, disrupting the technological characteristics of the process. Currently, alumina is widely used as the main component of ceramic molds for directional crystallization of heat-resistant nickel alloys. Studies of  $Al_2O_3$  evaporation in the presence of carbon at high temperatures showed that the reduction of alumina is initiated at temperatures above 1800 K and occurs both with direct contact of  $Al_2O_3$  with carbon and with participation of gas phase components [3–9]. Note that casting composite alloys based on refractory metals requires the use of more thermally stable ceramic materials compared to  $Al_2O_3$ . As shown earlier [10, 11], ceramic molds made from  $Y_2O_3$  may be an alternative to alumina ceramics in this case. However, the

information on the high-temperature behavior of yttrium oxide under reducing conditions is very limited [9, 12].

Let us consider the available data on the evaporation of yttrium oxide, which was studied by many authors [13–22]. It was established that yttrium oxide  $Y_2O_3$  evaporates at high temperatures according to the equation:

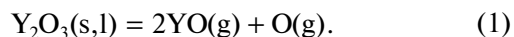


Table 1 summarizes the literature data [13–19] about the partial pressures of YO over  $Y_2O_3$  according to Eq. (1) up to a temperature of 2940 K.

It should be emphasized that in the vapor above  $Y_2O_3$ , atomic yttrium was also identified in addition to yttrium monoxide, and the ratio of partial pressures was  $p(YO) : p(Y) = 60$  at 2500 K, as shown in [20]. In a study of the evaporation of the  $Y_2O_3 + Y$  mixture from tungsten chambers [12] in the temperature range 1833–2100 K, the following temperature dependences of the partial pressures (Pa) of YO and atomic yttrium were found:

$$\log p(YO) = -(26460)/T + 11.28, \quad (2)$$

$$\log p(Y) = -(17650)/T + 9.09. \quad (3)$$

It should be emphasized that the ratio of partial pressures  $p(YO) : p(Y)$  at 1833 K is equal to 411, and at 2100 K it decreases to 101, which is apparently due to the selective evaporation of yttrium metal from the mixture.

**Table 1.** Coefficients  $A$  and  $B$  in the equation for the temperature dependence of the partial pressure (Pa) of YO vapor over  $Y_2O_3$ ,  $\log p(\text{YO}) = -A/T + B$  and the partial pressure of YO over  $Y_2O_3$  at 2200 K according to [13–15, 17–19] and the data obtained in the present study

$T$ , K	Effusion chamber material	$A$	$B$	$p(\text{YO})$ , (2200 K)	Refs.
2492–2697	W	$31\,295 \pm 2032$	$12.0 \pm 0.8$	$6.0 \times 10^{-3}$	[13]
2509–2720	W	$32\,376 \pm 1412$	$12.2 \pm 0.5$	$3.0 \times 10^{-3}$	[14]
2220–2650	W	$34\,570 \pm 160$	$13.1 \pm 0.1$	$2.4 \times 10^{-3}$	[15]
2691–2940	W	$34\,676 \pm 1295$	$12.3 \pm 0.5$	$3.4 \times 10^{-4}$	[17]
2300–2550	—	34467	13.1	$2.7 \times 10^{-3}$	[18]
2257–2703	W	$35\,387 \pm 388$	$13.5 \pm 0.2$	$2.6 \times 10^{-3}$	[19]
1946–2175	Mo	$43\,724 \pm 1388$	$17.2 \pm 0.7$	$2.2 \times 10^{-3}$	This study

The partial pressures of the molecular forms of vapor in the  $Y_2O_3$ –C system at high temperatures were not found in the literature.

Thus, the purpose of the present study was to investigate the evaporation processes of  $Y_2O_3$  at high temperatures in the presence of carbon to identify the processes in the  $Y_2O_3$ –C system, and to assess the temperature at which reliable operation of ceramic molds of yttrium oxide under reducing conditions is possible.

## EXPERIMENTAL

The present study was performed by Knudsen effusion mass spectrometry on an MS-1301 mass spectrometer designed to study evaporation processes and thermodynamic properties of highly volatile substances. The samples of yttrium oxide and yttrium oxide with carbon were evaporated from a dual single-temperature molybdenum chamber heated by electron bombardment. The temperature was measured with an EOP-66 optical pyrometer with an accuracy of  $\pm 10$  K in the temperature range 1500–2600 K. To determine the nature of ions in the mass spectra of vapor over the samples, the energies of appearance of ions in the mass spectra of vapor were measured by the vanishing ion current method. Gold with a known ionization energy of 9.2 eV was chosen as a standard for determining the energies of appearance of the ions under study [23].

The samples were evaporated as follows. One of the cells of the dual effusion chamber was charged with a mixture of yttrium oxide with activated carbon BAU-A (GOST (State Standard) 6217-74); the other (reference) cell was charged with individual yttrium oxide (special purity grade) with at least 99.9995 wt %  $Y_2O_3$ . A sample containing  $Y_2O_3$  and C was prepared by grinding a mixture of these components in a 1 : 1 ratio in an agate mortar. The partial pressures of the molecular forms of vapor over  $Y_2O_3$  and the  $Y_2O_3$ –C mixture were determined by comparing the ion currents using

the gold standard for the partial vapor pressure [24] according to the equation

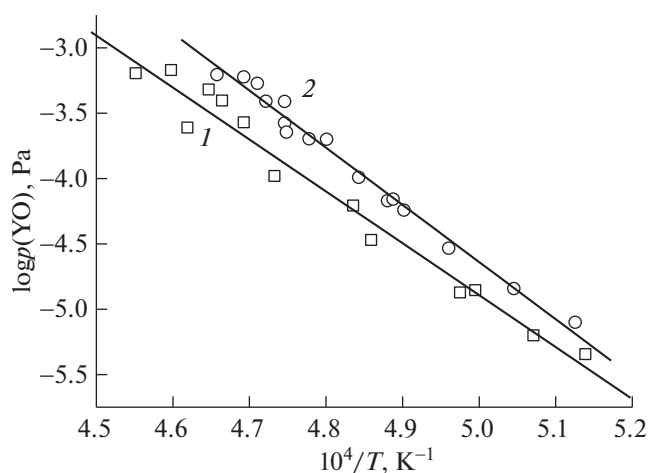
$$p_1 = \frac{p_2 I_1 T_1 \sigma_2 \gamma_2}{I_2 T_2 \sigma_1 \gamma_1}, \quad (4)$$

where  $I_i$  is the total ion current arising during ionization of the  $i$  molecular form of vapor  $I$ ;  $T_i$  is the temperature (K);  $\sigma_i$  is the ionization cross section of the  $i$  molecular form of vapor;  $\gamma_i$  is the conversion factor of the secondary electron multiplier, equal to the square root of the molecular mass of the corresponding  $i$  molecular form of vapor;  $p_i$  is the partial pressure of the  $i$  molecular form of vapor; and the subscripts 1 and 2 refer to the sample and standard, respectively.

The equipment was pre-calibrated against the  $CaF_2$  vapor pressure, adopted according to the recommendations given in [25]. Details of the equipment used and procedure for identification of the gas phase composition and determination of the partial pressures of the molecular forms of vapor were published and discussed previously, in particular, in [26–28].

In the mass spectra of vapor over the samples of individual  $Y_2O_3$  and the  $Y_2O_3$ –C system,  $Y^+$  and  $YO^+$  ions were identified starting from the temperature of 1950 K; the intensity ratio of these ions did not exceed 0.01–0.02 in both cases. To determine the molecular precursors of  $Y^+$  and  $YO^+$ , the energies of appearance of the indicated ions in the mass spectra of vapor over individual  $Y_2O_3$  and the  $Y_2O_3$ –C system were measured by the vanishing ion current method. The energies were found to be 11.5 eV and 6.0 eV, respectively. It was shown that the determined energy of appearance of the  $YO^+$  ion in the mass spectra of the vapor under study coincided, within the measurement error, with the ionization energy of the gaseous YO molecule, and the energy of appearance of the  $Y^+$  ion was significantly higher than the ionization energy of atomic yttrium [29].

Thus, from the analysis of the mass spectra of vapor over the samples of individual yttrium oxide and the



**Fig. 1.** Dependences of the logarithm of the partial pressure of YO on the reciprocal temperature in the vapor over the samples of the (1)  $Y_2O_3$ -C system and (2) individual  $Y_2O_3$ , determined in this work.

$Y_2O_3$ -C system, the temperature dependences of ion current intensities of  $Y^+$  and  $YO^+$ , as well as the energies of appearance of  $Y^+$  and  $YO^+$  ions, we can conclude that the vapor over the samples of individual yttrium oxide and the  $Y_2O_3$ -C system contains YO and atomic oxygen. However, the  $O^+$  ion current was not measured in this work in view of the significant background of residual gases in the vacuum part of the mass spectrometer.

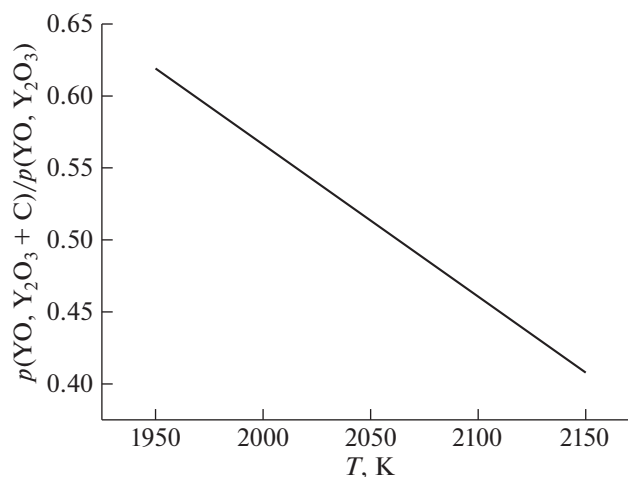
Measurement of the dependences of  $YO^+$  ion current intensities in the mass spectra of vapor over the samples of individual  $Y_2O_3$  and the  $Y_2O_3$ -C system, as well as determination of the partial pressures of YO over the indicated samples by comparing the ion currents, made it possible to obtain the temperature dependence of the partial pressure of yttrium monoxide over  $Y_2O_3$  and the  $Y_2O_3$ -C system in the temperature range 1946–2175 K (Fig. 1):

$$Y_2O_3: \quad \log p, (\text{Pa}) = -\frac{43724 \pm 1388}{T} + (17.22 \pm 0.67), \quad (5)$$

$$Y_2O_3\text{-C}: \quad \log p (\text{Pa}) = -\frac{397381256}{T} + (14.97 \pm 0.96). \quad (6)$$

Figure 2 shows the temperature dependences of the ratio of partial pressures of YO in the vapor over the samples of the  $Y_2O_3$ -C system and individual yttrium oxide obtained in this work. The ratio of the partial pressures of YO vapor decreases with increasing temperature.

The decrease in the partial pressure of YO vapor over the  $Y_2O_3$ -C system compared to that of individual yttrium oxide is evidently associated with the for-



**Fig. 2.** Temperature dependence of the ratio of the partial pressures of YO in the vapor over the samples of the  $Y_2O_3$ -C system and individual yttrium oxide, obtained in this work.

mation of thermally stable compounds at increased temperature in the condensed phase, for example, yttrium carbides or oxycarbides. It should be noted, however, that at 2150 K the mass spectrum of vapor above the  $Y_2O_3$ -C system did not contain  $YC^+$  and  $YC_2^+$  ions characteristic of yttrium carbide evaporation, as reported in [30]. The absence of these ions in the mass spectrum of vapor over the  $Y_2O_3$ -C system was possibly associated with the negligible partial pressure of  $YC_2$  vapor beyond the sensitivity of the mass spectrometer.

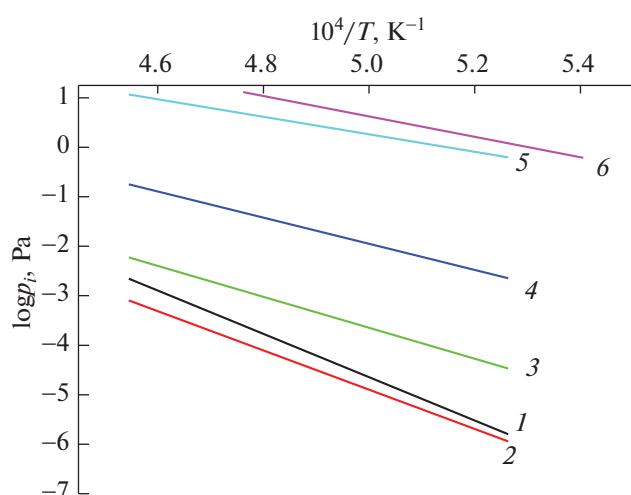
Figure 3 compares the data obtained in the present work with the temperature dependences of the partial pressures of YO vapor over individual  $Y_2O_3$  and the  $Y_2O_3$ -Y system and of atomic yttrium in the vapor over the samples of metallic yttrium and the  $Y_2O_3$ -Y system published in [12–19].

A comparison of the results given in Table 1 and Fig. 1 shows that the partial pressures of YO vapor over individual yttrium oxide obtained in this work are close to those determined in [13–19].

Note that the interaction of yttrium metal with  $Y_2O_3$  [12] leads to an increase in the partial pressure of YO vapor as a consequence of the partial reduction of yttrium oxide and to a decrease in the partial pressure of atomic yttrium compared to the Y vapor pressure over metallic yttrium.

In the temperature range 1946–2175 K for the  $Y_2O_3$ -C sample containing 50 mol % C, the activity of  $Y_2O_3$  ( $a_i$ ) was determined by the equation

$$a(Y_2O_3) = \frac{p(Y_2O_3)}{p_0(Y_2O_3)} = \frac{p^2(YO)p(O)}{p_0^2(YO)p_0(O)}, \quad (7)$$



**Fig. 3.** Temperature dependences of the partial pressures of YO vapor over the samples of the (1)  $Y_2O_3$ –C system and (2) individual  $Y_2O_3$  obtained in this work and those given in the literature for the following molecular forms of vapor: (3) YO over  $Y_2O_3$  [13], (4) and (5) YO and Y over the  $Y_2O_3$ –Y sample [12], and Y over the yttrium metal [25].

where are  $p_i$  and  $p_{0i}$  are the partial pressures of the molecular forms of vapor over the  $Y_2O_3$ –C system and individual yttrium oxide, respectively.

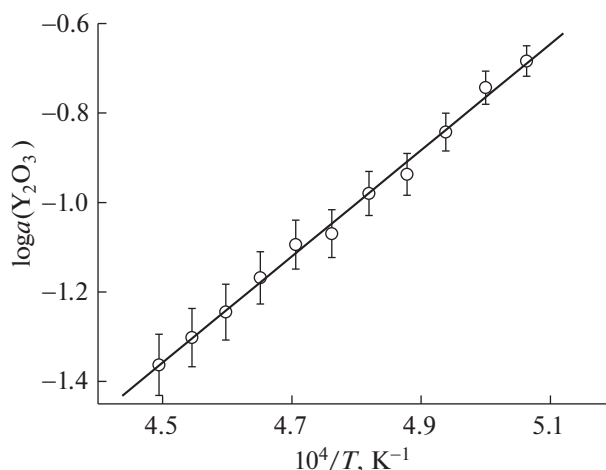
The resulting temperature dependence of the activity of  $Y_2O_3$  for the  $Y_2O_3$ –C sample containing 50 mol % C in the temperature range 1946–2175 K indicates that the activity of  $Y_2O_3$  decreases with increasing temperature according to the equation (Fig. 4):

$$\log a(Y_2O_3) = -\frac{11054 \pm 496}{T} + (6.32 \pm 0.24). \quad (8)$$

## RESULTS AND DISCUSSION

The thermodynamic data for the  $Y_2O_3$ –C system in the temperature range of 1950–2200 K obtained for the first time in the present study is unique thermodynamic information; it reflects one of the most promising trends in modern high-temperature chemistry, which is associated with the study of processes during the interaction of oxide and carbide systems, as well as materials based on them. Experimental studies in this direction are very scanty at present, despite their relevance for solving a wide range of technological problems and modeling phase equilibria using modern thermodynamic databases based on the CALPHAD approach.

The most striking illustration of the importance of this research are the results of a high-temperature experimental study [31] of the interaction of the chemically prototype metal and oxide components of the fuel-containing melt of VVER (PWR) reactors with  $Zr_2AlC$  carbide ceramics and also with composite



**Fig. 4.** Dependence of the logarithm of activity of  $Y_2O_3$  on the reciprocal temperature for the  $Y_2O_3$ –C sample containing 50 mol % C in the temperature range 1946–2175 K, obtained in this work.

ceramics based on zirconium dioxide and  $Zr_2AlC$ ,  $Zr_3AlC_2$ ,  $Ti_3SiC_2$ ,  $Ti_2SiC$ ,  $Ti_3AlC_2$ , and  $Ti_2AlC$ . Both the carbide and the composite materials containing  $Zr_2AlC$  exhibited high chemical resistance up to the temperature of 2773 K. Based on the results of [31], materials based on the  $ZrO_2$ – $Zr_2AlC$  system were first recommended for further study as coatings and individual elements of the active core having exceptional stability in the melt of the nuclear reactor core.

A study of  $SiC$ – $Al_2O_3$  and  $SiC$ – $Al_2O_3$ – $Y_2O_3$  systems performed by Chatillon and co-workers [9] was one of the pioneering works on the high-temperature interaction of oxide and carbide systems involving the gas phase. The results of the present study are qualitatively different from the data given in [9] when we compared the partial pressure of YO over individual  $Y_2O_3$  and the  $SiC$ – $Y_2O_3$  system. The temperature dependences of the partial pressure of YO over the systems in question were regrettably presented in [9] only in graphical form, which hinders data interpretation. When discussing the results of the present study, it should be emphasized that yttrium oxide in the  $SiC$ – $Y_2O_3$  system can be reduced not only with carbon, but also with silicon, increasing the partial pressure of YO compared to the pressure of yttrium monoxide over individual  $Y_2O_3$ . It was repeatedly shown that in the presence of reducing agents, in particular, carbon, the thermal stability of some oxides decreases. This is evidenced, for example, by the results of partial pressure determination of  $SiO$  vapor over  $SiO_2$  [32–35], as well as of Al and  $Al_2O$  over  $Al_2O_3$  [3–8], obtained by high-temperature mass spectrometry and illustrating an increase in the partial pressures of the indicated molecular forms of vapor in the presence of carbon. A similar increase in the partial pressure of YO vapor was observed during the interaction of  $Y_2O_3$  with

metallic yttrium [12]. Studies of the high-temperature interaction of alumina with carbon revealed the kinetic factors that hinder the initial stage of vaporization of alumina, carbon, and atomic aluminum [4, 5].

One of the factors responsible for the discrepancy between our data and the results of [9] concerning the observed tendency in the behavior of the activity of yttrium oxide with temperature in the  $Y_2O_3-C$  system may be associated with the difference in the composition of the condensed phase formed during the interaction of yttrium oxide with carbon compared to the  $SiC-Y_2O_3$  system. In the present work, it was impossible to control the change in the composition of the condensed phase at increased temperature during the evaporation of the  $Y_2O_3-C$  system because of the methodological limitations of the approach used within the framework of the high-temperature mass spectrometric experiment. However, it was assumed that the decrease in the partial pressure of YO over the  $Y_2O_3-C$  sample with increasing temperature is a consequence of the formation of yttrium carbides and their coexistence with  $Y_2O_3$  in the condensed phase. The correctness of this assumption can be illustrated by the formation of uranium carbide as an example, which was previously observed during the interaction of  $UO_2$  with carbon [36–38].

To summarize, the composition of vapor over the  $Y_2O_3-C$  system was identified for the first time, and the partial pressures of YO and the activity of  $Y_2O_3$  in the temperature range 1950–2200 K were determined. The obtained values of the thermodynamic properties of the  $Y_2O_3-C$  system at high temperatures can be recommended for inclusion in databases for calculating the phase diagrams of multicomponent systems using the CALPHAD approach.

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#### CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

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