Investigation of physical properties of 0.9ZrN + 0.1ZrO₂ ceramics at 2000–4500 K by current pulse heating

S V Onufriev¹, A I Savvatimskiy¹ and S A Muboyadzhyan²

¹ Joint Institute for High Temperatures (JIHT), Russian Academy of Sciences, Moscow, 125412, Russia
² National Scientific Research Institute of Aviation Materials (VIAM), Moscow, 105005, Russia
E-mail: s-onufriev@yandex.ru

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Abstract
The results of the study of zirconium nitride ceramics (0.9ZrN + 0.1ZrO₂) in solid and liquid phases are presented: temperature dependences of the specific input energy (enthalpy), specific heat, heat of fusion, electrical resistivity at temperatures of 2000–4500 K. To measure these properties the method of microsecond electrical current pulse heating was used. The specimens were obtained by spark plasma sintering method. Data for the liquid phase were obtained for the first time. The end of the nitride ceramics melting was 2700 ± 70 K at an estimated pressure of 5 MPa. A comparison with the literature data is presented.

1. Introduction
Zirconium nitride, along with zirconium carbide, is considered as a promising high-temperature material for nuclear technologies [1–3]. Both compounds have NaCl-type (rocksalt) structure in which metal and nonmetals form face-centered cubic sublattices. Zirconium nitride can have vacancies on metal and nonmetal sublattices, i.e., the ratio of N/Zr can be both less and more than 1, depending on in which sublattice there are more vacancies [4, 5]. In carbide, only carbon vacancies are possible.

This feature of nitride leads to the existence of compounds of different stoichiometry and to a variety of zirconium nitride phases. The possible existence of the variety of stable and metastable zirconium nitride phases with the different stoichiometry was predicted from the computational study [6].

Another difference between nitrides and carbides of group IVA metals is the change of their melting point depending on the pressure of nitrogen. Nitrogen pressure strongly affects the melting temperature of ZrN; with increasing pressure, the melting temperature increases [7]. It is also known that the properties of nitrides depend on oxygen impurity. For example, in the same work [7] it was found that 0.5–1 wt% oxygen lowers the melting point of ZrN by 200–300 K. It was reported [8], that during magnetron sputtering of zirconium nitride film, an increase in the flow rate of the reacting gas (N₂ + O₂) changes the electrical resistance of this film by 12 orders of magnitude, i.e. this nitride changes from metal conductivity to a dielectric as the oxygen content increases.

The most studied zirconium nitride compound is the zirconium mononitride ZrNₓ (0.55 < x < 1). In particular, the electrical, thermophysical, and optical properties of this nitride are known to ∼2600 K. At higher temperatures, as well as in the liquid phase, its properties have not been studied [9].

In this work, we present the results of a study of zirconium nitride ceramics (N/Zr ≈ 1.36) at high temperatures in the solid and liquid phases.

2. Methods

2.1. Measuring technique
Studies were carried out under heating of the sample by a single pulse current of microsecond duration. The method and instrumentation are described in detail elsewhere [10, 11]. Samples are heated by a current pulse when the capacitor battery is discharged. The sample is heated to a liquid state during 2–5 μs, which eliminates...
the effects of sample displacement, surface tension forces, changes in composition (section 4.3), and allows neglecting heat losses by radiation and thermal conductivity. The applied voltage, the current through the sample, and the pyrometer signal are recorded with a 4-channel digital oscilloscope. The sample temperature and temperature dependences of the specific Joule energy, specific heat, resistivity, and temperature coefficient of electrical resistance (TCR) are calculated using these signals presented in a discrete form.

Measurement uncertainties were estimated in previous works \cite{10, 11}. The relative uncertainty in measurements enthalpy ($H$) in the studied temperature range is 6% and originates from the measurement of Joule energy absorbed by the sample. The uncertainty of measurement of specific heat does not exceed 30% at 4000 K. The uncertainty of measurement of electrical resistivity (referred to initial dimensions of the sample) is less than 5%. The temperature coefficient of resistivity (TCR) is measured with 30% uncertainty at 4000 K. The uncertainty in temperature is $\sim$70 K below 2700 K and $\sim$140 K at 4000–4500 K.

2.2. Samples synthesis and characterization

Zirconium nitride powder was used to produce dense samples by SPS (Spark Plasma Sintering) method. The sintered disc, 60 mm in diameter and 3.5 mm thick, was cut by electro-erosive cutting into the plates with the dimensions of 0.5 $\times$ 3.5 $\times$ 15 mm. The plates were ground to a thickness of 0.14 mm. The surface cleaning and polishing of these samples were carried out manually on a diamond bar with the diamond grains of 5–7 microns.

The surface of the polished plate (figure 1(a) and microstructure of the fracture surface figure 1(b) were studied using a scanning electron microscope Nova NanoSem 650. The bright inclusions are enriched with tungsten.

The analysis of the sample composition by energy-dispersive x-ray spectroscopy showed 38.69 at% Zr and 52.60 at% N (N/Zr ratio $\approx$ 1.36). The sample also contained 7.25 at% O and impurities of Ca and W (0.48 and 0.39 at%), Al and Fe (0.3 at% each). These data are average values for three points on the fracture surface and three points on the sample surface.

Figure 2 shows the x-ray diffraction spectra obtained by EMPERYAN diffractometer (by PanAltyical). Registration of diffractogram was carried out in monochromatic CuK$_\alpha$ - radiation in the Bragg-Brentano geometry. To interpret the diffractogram the PDF-2 database was used. As can be seen from figure 2 the sample consisted of cubic NaCl-type ZrN (87 vol%), and tetragonal ZrO$_2$ (13 vol%). That is from 88.6 and 11.4 wt% and 90.1 and 9.9 mol% of these phases (the densities of 7.09 for ZrN \cite{12} and 6.1 g cm$^{-3}$ for t-ZrO$_2$ was used).

The density of the ceramics after SPS was 6.90 g cm$^{-3}$, calculated according to the data of x-ray phase analysis $-6.96$ g cm$^{-3}$.

A nitride plate was glued between two silica glass plates 3.5–4 $\times$ 8 $\times$ 15 mm in order to create a reliable electrical contact of the electrodes with a fragile sample with a mechanical clamp. UV curing glue was used to cover two sides of the sample. The lack of influence of the adhesive layer on the measured temperature was checked on samples of tantalum foil. The view of the cell with the sample is shown in figure 3. Indium gaskets ($\sim$0.1 mm thick) were used to provide reliable contact of the sample with electrodes. This configuration provided a closed volume, which led to an increase in the pressure during pulsed heating.

3. Results

The typical view of the waveforms on the display of oscilloscope is shown in figure 4.

The pressure arising from the heating of the sample was estimated (section 4.2) at 5–9 MPa.
3.1. Thermal properties

In the experiments the sample was melted at 2700 ± 70 K (figure 5), which is ≈550 K lower than the melting point of zirconium mononitride at atmospheric pressure (3253 K) [4].

The dependence of the Joule energy $E$ dissipated in the sample (enthalpy $H$, section 4.4) on temperature is shown in figure 6, curve 3. The melting plateau is smoothed, but the end of melting at 2700 K is determined quite clearly. The onset of melting is fixed on the dependence of the electrical resistivity on temperature (figure 8) at 2600 K. We determined the heat of fusion $\Delta E$ as the difference between the enthalpy of the liquid phase at the end of melting (2700 K) and the enthalpy of the solid phase at the start of melting (2600 K). Using figure 6, we obtain $\Delta E = 1.4 - 0.8 = 0.6 \text{ kJ g}^{-1}$.

The specific heat of nitride $C_p$ was calculated by formula [11]

$$C_p(T_{ref}) = \frac{E[T(t_{i+n})] - E[T(t_i)]}{T(t_{i+n}) - T(t_i)},$$

where $t_n, t_{i+n} (n = 150)$—points of oscilloscope sweep (10 $\mu$s) remote from each other by (10 $\mu$s)/10^4 × 150 = 0.15 $\mu$s; $E$—the Joule energy; $T$—the true temperature of the specimen; $T_{ref} = 0.5 \times [T(t_i) + T(t_{i+n})]$—the temperature, to which the calculated $C_p$ value relates. The temperature

Figure 2. Diffractogram of the sample obtained in CuKα—radiation. The PDF-2 database was used to interpret the diffractogram. The phase composition is given in vol%.

Figure 3. The photographs of quartz cells with a sample. Silica glass plates are connected by side textolite plates with cyanoacrylate glue-gel, which was also used together with thin mica strips to seal the side slots in the cell: 1, 3—silica glass plates; 2—the sample (0.14 × 3.5 × 15 mm) protruding from the cell due to the shortening the upper plate; 4—textolite plates.
dependence of the specific heat is shown in figure 7 (curve 3). The melting region extends from 2600 K to 2700 K (arrow 4 and corroborated by the measurements of electrical resistivity discussed below. The dashed part of line 3 is presented only for illustration the application of formula (1) to the melting region and is not used as $C_p$ data [11]. Arrow 5 indicates the area of assumed boiling.

3.2. Resistivity and temperature coefficient of electrical resistance

The change of the resistivity ($\rho$) with temperature shown in figure 8, referred to the initial dimensions of the samples. The melting region is clearly defined on the curve 1. The curve has three distinct segments, correlated with the resistivity of the solid phase (to the left of line 3; $T < 2600$ K), the melting region (between lines 3 and 4; $2600 \leq T \leq 2700$ K) and the liquid phase (to the right of line 4; $T > 2700$ K).
In the solid phase, before melting, the resistance is $120 \mu\Omega \cdot \text{cm}$, and at the end of melting is $180 \mu\Omega \cdot \text{cm}$. Thus, upon melting of the zirconium nitride (it has a metallic character of conductivity), the resistance increases by 50%, which is much higher than typical values for metals $\sim 5\%–10\%$. The sample resistance in the liquid phase increases linearly with increasing temperature (figure 8).

The dependence of the temperature coefficient of electrical resistance (TCR) on temperature is shown in figure 9. This coefficient was calculated using the $\rho(T)$ data and the same algorithm as $C_p(T)$ calculation.

4. Discussion

4.1. Temperature measurements

The surface temperature of the samples was measured using a high-speed pyrometer, calibrated with a tungsten temperature lamp, at a wavelength of 856 nm. The operation of the pyrometer and the method of measuring the temperature of the sample under pulsed heating were additionally checked by measuring the melting...
temperature of tantalum 12 μm thick. Ta foil with 1 wt% Nb was used for measurements. The measured melting point, 3260 ± 50 K, agreed with reference data for Ta [16] within estimated uncertainty.

The uncertainty of temperature measurement is mostly determined by the uncertainty of emissivity of nitride

\[ \delta T / T \sim (\lambda T / C_2) \delta \varepsilon_{\lambda, m} / \varepsilon_{\lambda, n} \]  

where \( C_2 \) is the second constant in Planck’s law; \( \varepsilon_{\lambda, m}, \delta \varepsilon_{\lambda, m} \) are the normal spectral emissivity and its uncertainty.

The data on \( \varepsilon_{\lambda, n} \) for zirconium mononitride [17, 18] were considered. Earlier, when studying deposited samples of zirconium nitride [19], we chose the value of \( \varepsilon_{\lambda, n} \) obtained in [17]—0.78 for the samples containing oxygen. In [19], the correctness of \( \varepsilon_{\lambda, n} = 0.78 \) value was confirmed by the coincidence of the results of experiments on flat samples and on samples in the form of a blackbody model. However, a more thorough analysis of the measurement results of \( \varepsilon_{\lambda, n} \) for ZrN [17] shows that they cannot be regarded as sufficiently reliable in view of the following: (i) it is not indicated in what atmosphere the sample was heated; (ii) the sample
was kept at a maximum test temperature of 2287 K for 8–16 h, as a result, the sample surface changed, its weight increased and its density decreased by ~6% (there is no analysis of the effect of these changes on \( \varepsilon_{\lambda, n} \)); (iv) the obtained dependences of \( \varepsilon_{\lambda, n} \) on the wavelength at different temperatures differ in shape from each other; (v) the temperature dependences of the normal total emissivity of ZrN calculated on the basis of the measured \( \varepsilon_{\lambda, n} \) differ sharply from the data of other works [20].

Considering all of this, we refused the data [17] and used the more reliable, in our opinion, data [18] - the value of \( \varepsilon_{\lambda, n} = 0.56 \) obtained for ZrN0.85 at a temperature of 2800 K and at a wavelength of 856 nm (installation and measurement method [18] are presented in [21]).

This value was accepted in our work in the entire temperature range, including the liquid phase. The error of the data [18] is 3%, however, when estimating the uncertainty of temperature measurement in our work, taking into account differences in the composition of our samples and samples [18], we increased this error to \( \delta \varepsilon_{\lambda, n} / \varepsilon_{\lambda, n} = 10\% \).

The reason for the difference in the values of \( \varepsilon_{\lambda, n} \) in [19] and the present work (the elemental composition of the studied zirconium nitride is approximately the same) is the microstructure of the surface layer. In [19] surface microstructure is less dense with well-developed grain boundaries.

4.2. Pressure assessment
As was mentioned above the properties of zirconium nitride (melting point) depend on the pressure [7]. So an estimation of the pressure under which the experiments were performed is important. The pressure in the sample increases during its rapid heating due to its thermal expansion within the confinement of the cell. We will refer to this as an inertial pressure.

During the rapid expansion of the sample, the upper and lower quartz plates are subjected to compressive stresses (the transit time of the compression wave through the quartz plate is 0.6–0.7 \( \mu \)s, the sound speed for quartz glass is 5.935 km s\(^{-1} \) [12]). Tensile stress acts on the side plates, and shear stress acts on glue joints of side plates during the sample expansion. For quartz glass, the compressive strength (under static and impulse load) is many times greater than the tensile strength of the PCB. In turn, the tensile strengths of the textolite plates are significantly greater than the shear strength of the adhesive used. Therefore, with the expansion of the clamped sample, the destruction of adhesive joints is most likely. The force causes the destruction of one glue joint is equal to

\[
F = \sigma s
\]

where \( \sigma \) - is the ultimate shear stress of adhesive; \( s \) - is the cross-sectional area of one glue joint.

Experiments show that for about 7 \( \mu \)s (\( T \approx 4500 \) K, figure 5), the top plate of cell through which the sample surface is sighted retain its integrity. This is indicated by smooth (without sharp jumps) curves of \( T(t), E(T), \rho(T) \). Due to the symmetry of the loading, it can be argued that the lower plates also remain intact. We assume that during the destruction of the cell, all four glue joints (figure 3) are broken simultaneously. In this case for the destructive pressure \( P \) in the cell (the pressure acting on the quartz plates of the cell) from equation (3) we obtain

\[
P = 4 \sigma s / S,
\]

where \( S \) is the surface area of the sample; \( s / S \approx 0.114 \).

For the CA adhesive used (Super Moment 'Henkel' glue, \( \sigma \sim 10 \) MPa) we obtain \( P \approx 4.6 \) MPa. The destruction of sealing glue joints occurs at the same pressure level. Thus, accounting for the integrity of the cell, the pressure in the specimen was \( 0.1 < P < 5 \) MPa.

We estimate the time \( \tau_p \) of inertial pressure equalization across the sample. The speed of sound \( v_s \) of zirconium nitride may be calculated by the formula [12]

\[
\gamma v_s^2 = B + \frac{4}{3} G
\]

where \( \gamma \) - sample density equals to 6.90 · 10\(^3 \) and 6.61 · 10\(^3 \) kg m\(^{-3} \) at temperatures \( T_0 = 300 \) K and \( T = 2500 \) K, respectively (the volume thermal expansion of nitride 1.97 · 10\(^{-5} \) K\(^{-1} \) was taken from the data of [4] for linear thermal expansion); \( B \) - bulk modulus of zirconium nitride equals to 255 and 187 GPa at the same temperatures [22]; \( G \) - shear modulus equals to 153 and 115 GPa at these temperatures [22]. We obtained 8.2 and 7.2 km s\(^{-1} \) at 300 and 2500 K that gives \( \tau_p \sim 0.01 \mu s \), which much smaller than the time of heating. Therefore, the distribution of inertial pressure in the sample during heating was uniform.

In addition to the inertial pressure from the plates, the sample is compressed by the electromagnetic forces (pinch effect). Using the solution of the problem for the cylinder [23], for the distribution of magnetic pressure over the thickness of an infinite plate we obtain
where \( j \) is the current density; \( \mu = 1, \mu_0 = 4\pi \times 10^{-7} \text{ H m}^{-1} \) — magnetic permeability for ZrN and vacuum; \( 2a \) — width of the plate; \( z \) — is the distance of the point from the plane of symmetry for the sample. The pressure increases according to the square law — from zero on the surface of the plate to the maximum value in the plane of symmetry. The thickness of the samples studied is much smaller than their width and length, which makes it possible to apply the formula (7) to estimate the pressure. For the sample with a thickness of \( 2a = 0.14 \text{ mm} \), the maximum of the current at the beginning of the melting was measured as \( I = 25.5 \text{ kA} \) (figure 5), which corresponds to current density, \( j = 5.2 \times 10^{10} \text{ A m}^{-2} \) and to the maximum pressure on the sample axis \( \approx 4 \text{ MPa} \). Thus, the heating of relatively thick ceramic samples leads to gradients of electromagnetic pressure across the thickness of the sample. The maximum pressure on the surface of the sample and in its central part could reach 5 and 9 MPa, respectively.

4.3. Assessment of changes in sample composition on heating

In our case, the heating process takes units of microseconds — \( t_h \approx 5–7 \mu \text{s} \).

Changes in the composition of the samples in the solid phase, during melting and in the liquid phase can occur only as a result of relatively slow diffusion of atoms and vacancies in the sublattice of metal and non-metal in the solid sample and diffusion of the atoms in the liquid phase.

Let us estimate the time \( t_d \) required for the diffusion of nitrogen atoms through the sample, and the thickness \( \delta \) of a layer, the composition of which may change during heating as a result of diffusion:

\[
t_d = \frac{a^2}{D_i},
\]

\[
\delta = \sqrt[4]{D_i t_d},
\]

where \( a \) is half of the thickness for the sample; \( D_i \) — is diffusion coefficient of nitrogen into zirconium nitride.

Data on the diffusion coefficient are given in [23, 24]. In [24], at a temperature of \( 2750 \text{ K} \), \( D \approx 5 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1} \) was obtained, which gives \( t_d \approx 10 \text{ s} \) and \( \delta \approx 0.05–0.06 \mu \text{m} \).

In [25], homogenization of samples made from a mixture of zirconium nitride and oxide, in particular, with a composition of 0.9 ZrN + 0.1ZrO2, was carried out at a temperature of 3000 K. The data of this work allow us to approximately estimate the diffusion coefficient of the components \( \approx 4 \times 10^{-2} \text{ cm}^2 \text{ s}^{-1} \), and by using formulas (8), (9) to estimate in our case the diffusion time \( t_d \approx 2.5 \text{ ms} \) and the layer thickness \( \delta \approx 5 \mu \text{m} \).

These estimates show that the time required to change the composition of the sample is much longer than the heating time, and the layer of the sample, in which its composition may change as a result of diffusion, is much thinner than the thickness of the sample. Thus, it can be argued that the composition of the sample did not change during heating. The negligible compositional change during pulse heating of the metals is stated in [26].

4.4. Thermal properties

The thermogram (figure 5) does not have a melting plateau. We assume this is due to the dependence of the melting temperature of nitride on pressure. The increase in pressure during melting (due to volume expansion) will lead to an increase in the melting temperature. The melting region of the samples is most clearly determined by the dependence of \( \rho(T) \) shown in figure 6. The reason for lowering the melting temperature compared to ZrN is the presence of oxygen in the samples [7].

We measure the specific energy of Joule heating dissipated in the sample \( E = \frac{1}{m} \int_0^t \mathbf{I}(U) dt \), where \( I, U \) is the current through the sample, and the active component of the voltage on the sample; \( m \) is the mass of the sample [10, 11]. The process of heating the sample is not isobaric — pressure varies from atmospheric \( P_0 \approx 0 \) to \( P \) (temperature varies from \( T_0 \approx 293 \text{ K} \) to \( T \)). We will show that for the solid phase the value of \( E \) in our conditions is approximately equal to the enthalpy of the sample \( H \). When heated, the sample passes from stage 1 \( (P_0, T_0, H_0, E_0 = 0) \) to stage 2 \( (P, T, E, H) \). In this case

\[
\delta E = dH = VdP; \quad E = \Delta H = \int_{P_0}^P V(T, P)dP
\]

Let us consider the transition from 1 to 2 as successive stages — first heating at \( P_0 \) to temperature \( T \), and then isothermal compression until pressure \( P \) is reached. In this case, for the first stage

\[
V^* = V_0(1 + \beta(T - T_0)),
\]

where \( \beta(T) = \left(1 / V_0 \right)(V - V_0) / (T - T_0) \) — is the mean thermal volume expansion coefficient at constant pressure \( P_0 \); \( V_0 \) — is the specific volume at \( P_0, T_0 \). For the second stage, after compressing the sample, its volume will be equal to
\[ V = V^* [1 - \chi_T (P - P_0)], \]

where \( \chi_T = -\left(1/V^*\right)(V - V^*)/(P - P_0) \) — is isothermal compressibility.

Substituting (11) into (12) and integrating (10) we obtain

\[ \Delta H - E = \frac{V_0 P}{E} \left[1 + \frac{T_0}{(T - T_0)} \right] \left(1 - \frac{P}{2B_T} \right) \]

where \( B_T = 1/\chi_T \) — is the isothermal bulk modulus.

Using pressure values \( P = 1–50 \text{ MPa} \) and \( T = 2600 \text{ K} \) and characteristic values for zirconium nitride [4, 27] (section 4.2): \( V_0 = 1.45 \cdot 10^{-4} \text{ m}^3/\text{kg}; T_0 = 1.97 \cdot 10^{-5} \text{ K}^{-1}; (T - T_0) = 2307 \text{ K}; B_T = 187 \text{ GPa}; E \approx 1.0 \cdot 10^6 \text{ J kg}^{-1} \) (see figure 6) the difference of enthalpy from the Joule energy is within 0.02%–0.8%. It is significantly less than the uncertainty in the measurements of Joule energy thus we consider Joule energy equals to the enthalpy.

The dependence of the enthalpy on the temperature is shown in figure 6. For comparison, figure 6 also shows the temperature dependences of the enthalpy of the ZrN mononitride presented in reference publications [13, 14]. It should be noted that the data given in [13, 14] were obtained by joint processing of experimental data on specific heat in the temperature ranges 53–297 K and 298–1073 K for the solid phase ZrN and their further extrapolation to 6000 K.

The range of change for heat capacity with temperature measured in this work are close to previous extrapolations [13, 14]. However, there is a significant difference in the values of enthalpy and temperature and reference data. The reason for such discrepancies is most likely the difference in the composition of ZrN and the studied zirconium nitride ceramics.

We obtained the heat of fusion of nitride 0.6 kJ g\(^{-1}\). In [13, 14], the calculated estimates of the heat of fusion of ZrN were 0.80 and 0.64 kJ g\(^{-1}\), respectively. In [9], referring to [19], the value of the heat of fusion of zirconium nitride 0.988 kJ g\(^{-1}\) is given. Unfortunately, this is an inaccuracy: in [19], direct measurements of the heat of fusion were not performed and such data were not obtained.

The results of the specific heat measurements are shown on figure 7. It also shows the dependencies for ZrN, given in the reference publications [13, 14]. The end of the melting of the investigated nitride is shifted by \( \approx 550 \text{ K} \) down compared to the melting point for ZrN. However, the data on the specific heat for the liquid phase up to 3800 K (figure 7, curve 3) are in agreement with the reference data within the measurement uncertainties. Above this temperature, the heat capacity begins to grow, which is probably due to approaching the boiling point.

In [19] for \( C_p \) of zirconium nitride of approximately the same elemental composition as in the present work, a value of 1.25 J g\(^{-1}\)K\(^{-1}\) was obtained in the temperature range 2700–3200 K, which is in reasonable agreement with our results (0.8 J/g K). When comparing these data, it is necessary to take into account that in [19], samples were used in the form of a thin film (2.6 \( \mu \text{m} \)), leading to an increased error in measurements of sample mass and specific heat.

4.5. Resistivity and TCR

Zirconium nitride is a conductor with conductivity close to that of metals. At room temperature, its resistivity is equal to 7–21 \( \mu \text{ohm cm} \) [4]. At high temperatures, it behaves as metal also.

Our data on \( \rho(T) \) for zirconium nitride ceramics are compared with data on \( \rho(T) \) for zirconium mononitride [15] (squares in figure 8). In the study [15], the samples of pyrolytic zirconium nitride (N/Zr = 0.95 \( \div \) 1.0) were obtained in the form of tubes by the method of deposition from the gas phase. Prior to the experiments, the samples had a density of 7.26–7.37 g cm\(^{-3}\) (close to theoretical) and did not contain oxygen. The study was performed by a stationary current heating method in a vacuum of 1.3 \( \cdot \) \( 10^{-3} \) Pa. The temperature of the samples was measured by a pyrometer using a black body model. After the experiments, the oxygen content in the two of the four samples increased to 0.7–1.0 wt%. Our data (both in absolute values and in the slope of the curve) coincide with the data [15], which seems to be quite reliable.

According to [19], the resistivity of the deposited nitride samples in the solid phase is approximately 5–6 times higher than the data of this work. The main reason for this is the difference in the microstructure of the samples. In [19] the structure is less dense with pronounced grain boundaries, which corresponds to a lower density of samples (6.7 g cm\(^{-3}\)).

Linear change of resistivity with temperature in the liquid phase (figure 8) indicates that TCR in this phase is approximately constant and corresponds to the value \( \approx 4 \cdot 10^{-4} \text{ K}^{-1} \) (figure 9).
5. Conclusion

The developed heating method and cell design enable the melting of relatively massive samples of zirconium nitride ceramics in less than 10 μs, which avoids changes in sample composition and allows measurements of thermophysical and electrical properties of electrically conductive ceramics at ultra high temperatures.

For the first time, thermophysical properties of zirconium nitride ceramics (0.9ZrN + 0.1ZrO₂) were studied to 4500 K, providing temperature dependencies of the enthalpy, specific heat, electrical resistivity, and temperature coefficient of resistance in the liquid state.

The temperature dependence of the electrical resistance shows that the melting of such ceramics takes place in the temperature range of 2600–2700 K. The heat of fusion of ceramics is 0.6 kJ g⁻¹ and the specific heat of the liquid phase is ≈ 8 J/(g·K) (constant up to 3800 K) are close to the calculated reference data for zirconium nitride.

The electrical resistance of the studied ceramics at temperatures of 2300–2600 K coincides with the electrical resistance of zirconium nitride.

In general, this work demonstrates the wide possibilities for rapid heating of substances with a single current pulse. The method allows obtaining thermophysical and electrophysical properties of conducting substances at temperatures up to 5000–6000 K (carbon—up to 9000 K [28]).

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ORCID iDs

S V Onufriev © https://orcid.org/0000-0001-7826-125X
A I Savvatimskiy © https://orcid.org/0000-0003-4757-5672

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