Electrical resistivity of zirconium carbide at 1200–2500 K

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Abstract. Zirconium carbide ZrC produced from nano-sized powders is a new material; its properties have not, in fact, been studied. We present the experimental results on the electrical resistivity of the zirconium carbide within the temperature range of 1200–2500 K. The ZrC specimens were prepared by spark plasma sintering from nano-size particles at a temperature of 2100 K. The goal of the present work is to compare the obtained results of the electrical resistivity with data for ZrC produced by traditional methods.

1. Introduction

The relevance of this work is associated with the intensive introduction of nanomaterials into modern technological processes. New materials that are made on the basis of powders with particle sizes of 10–50 nm are being actively studied [1–3]. However, the stage of transition to production on an industrial scale is always preceded by a stage of exploratory research, which is carried out on samples made in small quantities. Our work relates to exploratory studies of the electrical resistivity of zirconium carbide, which is made of nanosized powder. Zirconium carbide is widely applied as an abrasive for tool production. This material is actively applied as covering for nuclear fuel: uranium and thorium dioxides. Zirconium carbide is a gray metallic powder with a cubic lattice of the NaCl type. Zirconium carbide produced from nano-sized powders is a new material; its properties have not studied. The goal of the present work is to study the electrical resistivity of the ZrC produced from nano-sized powders and to compare the results with data obtained by traditional methods.

2. Experimental

The disk-shaped blanks were produced from zirconium carbide nano-sized particle powder with the size of particles 50–70 nm (chemically obtained) by spark plasma sintering. The blanks were annealed for an hour at a temperature of 2100 K. This blank production method did not include the high pressure pressing; thus, the density of the obtained ZrC, $\rho = 6030 \text{ kg/m}^3$. This value turns out to be lower than the density of $\rho = 6390 \text{ kg/m}^3$ [4] and higher than $\rho = 5990 \text{ kg/m}^3$ [5]. We cut a specimen in the form of the hollow cylinder from the blank. The first specimen had outer and inner diameters $D = 7.9 \text{ mm}$ and $d = 4.98 \text{ mm}$, length of $36.0 \text{ mm}$; the second sample—$D = 8.06 \text{ mm}$, $d = 4.0 \text{ mm}$ and length of $36.1 \text{ mm}$. 
Figure 1. Electrical resistivity of zirconium carbide; markers correspond to experimental data: 1 and 2—$D/d = 7.9/4.98$ and $8.06/4.0$, this work; 3—hot pressing [6]; 4—hot pressing [7]; 5—pyrolytic ZrC [8]; 6—hot pressing [9]; 7—cold pressing [4]; 8—cold pressing [5].

The principal layout of the measuring setup included a chamber, gas-vacuum and diagnostics systems [10]. We fastened the specimen horizontally between the current contact jaws. The chamber was filled with high-purity argon at a pressure of 0.1–0.15 MPa. The specimen was heated by direct electric current. Preliminary experiments to determine the isothermal section length consisted of the obtainment of the distribution of the brightness temperature over the specimen length. We drilled two holes 1.0 mm in diameter in the center of the isothermal section on the distance $l_{pr}$ from each other. Depth of each hole equals to thickness of a specimen wall. Graphite dampers were inserted into each hole. We drilled a through hole 0.3 mm in diameter along the axis of each damper and a 0.138 mm tungsten wire loop was fastened in the hole at a distance of $l_{pr} = 8.00$ mm for sample 1 and $l_{pr} = 8.01$ mm for sample 2. The second end of the tungsten wire was used to measure the voltage drop. A hole was drilled (1.0 mm in diameter with depth equal to the wall thickness) in the center between these two probes [10]. This hole served as a blackbody (BB) model to determine the temperature on the internal cylinder surface. We established plugs at the hollow cylinder ends in order to enhance the degree of perfection of the BB model. We measured the BB model temperature through the chamber window with an automatic micro pyrometer with a vision spot diameter of 0.3 mm. Micro pyrometer operated at a wavelength of 0.65 µm. We introduced a correction for absorption of the chamber wall glass, for the perfection of the BB model geometry sizes [11] and determined the true temperature on the internal cylinder surface. During the experiment, we measured the temperature on the internal specimen surface, the current $I$, and the voltage drop $U$ over the section between the probes. The hollow cylinder specimens with different wall thicknesses and two-cylinder method made it possible to estimate the true temperature on the external specimen surface [10]. We took the arithmetical mean value
between the true temperatures on the internal and on the external surfaces of the specimen as the reference temperature for the electrical resistivity.

3. Results and discussion
The electrical resistivity \( r \) of the zirconium carbide was calculated by equation

\[
r = \frac{\pi U (D^2 - d^2)}{4I_{pr}}.
\]

Figure 1 shows the temperature dependence of the electrical resistivity of the ZrC. The present values are plotted in figure 1, together with previously published values [4–9]. The electrical resistivity increases with increase in temperature throughout the temperature range covered by the experiments. Samples had the form of the continuous cylinder with diameter of 5.3 [4], 9.0 [5], 6.07 mm [9] or the hollow cylinder with \( D = 50.8 \) mm and \( d = 12.7 \) mm [6]. The experimental values of electrical resistivity have enough wide scatter. It is caused by manufacturing techniques of samples. Cold pressing used for production of ZrC in our country [4,5,7,8], hot pressing applied abroad is more often [6,7,9]. The samples received by cold pressing of powder ZrC at pressure of \( 2 \times 10^3 \) kg/cm\(^2\) with subsequent sintering in the environment of argon at temperature 2700 K [7] or in the environment of nitrogen at \( T = 2800 \) K [4] were investigated. The samples received by hot pressing and subsequent sintering in vacuum at \( T = 2400 \) K were investigated in works [6,9]. Hot pressing and the subsequent sintering in vacuum at pressure 50 kg/cm\(^2\) used for production of sample to work [7]. The received samples had porosity of 12.6 vol\% [4], 12–16\% [5]. Results of researches have shown, that \( r \) has higher values if samples are received by hot pressing unlike the samples made by cold pressing. Our results in figure 1 generally appear above received by hot or cold pressing traditional methods [4–9] and agree with the data (hot pressing) [7]. We studied the chip of the samples by scanning electron microscopy before experiment. The chip surface had a grain structure with an average grain size of about 3 \( \mu m \) and included pore-like defects. The size of particles in our experiments (3 \( \mu m \)) has appeared smaller than the size of particles of initial samples in work [4] (15 \( \mu m \)) and in work [6] (50 \( \mu m \)). Probably this fact has basic value for an explanation of higher values \( r \) received in our experiments.

4. Conclusion
The indicated technology for producing of the zirconium carbide from nanoscale particles and the results of studying electrical resistivity were obtained for the first time. An analysis of the experimental data shows that electrical resistivity depends on many factors [8,12], therefore it is required to continue experimental researches.

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