

## SELECTION OF MELTING UNIT FOR FUZED ZIRCONIUM DIOXIDE PRODUCTION

V. A. Sokolov,<sup>1,4</sup> M. A. Gorbanenko,<sup>2</sup> A. B. Lisafin,<sup>2</sup> and M. D. Gasparyan<sup>3</sup>

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Data are provided for preparing fuzed and fuzed stabilized zirconia from baddeleyite and synthetic zirconia in induction, plasma, and electric arc furnaces. Results are discussed and prospects for carbothermal preparation of zirconium dioxide from zircon in an electric arc furnace are demonstrated. A favorable effect of oxidation melting in an electric arc furnace on quality and the process of fuzed product preparation is demonstrated. Data are provided for preparing fuzed zirconia from zircon in plasma furnaces.

*Keywords:* fuzed zirconium dioxide (FZD), fuzed stabilized zirconium dioxide (FSZD), baddeleyite, zircon, induction furnace, plasma furnace, electric arc furnace, oxidation melting, carbothermy.

Zirconium dioxide is a chemical compound of zirconium used most extensively which in natural form (baddeleyite) and synthetic material is used in various branches of industry, mainly for manufacturing refractories, commercial ceramics, and abrasives. In the last decade there has been a significant increase in the use of zirconium dioxide for producing materials for electronics, oxygen sensors, pigments for ceramics, catalysts, etc. A large industry requiring zirconium dioxide is the atomic industry for preparation of nuclear-clean zirconium metal ([www.chmz.net](http://www.chmz.net)).

Alongside natural and synthetic zirconium dioxide there is also considerable demand for its structural modifications, monoclinic and cubic, stabilized with calcium and yttrium oxides, in fuzed form. According to expert evaluation the world production of fuzed zirconium dioxide (FZD) is more than 50 thousand tons; in this case the proportion of stabilized grade does not exceed 20%.

The requirement within Russia for FZD due to a lack of in-house production is accomplished entirely due to imported supplies. In this case the users of FZD are enterprises of strategically important branches of industry (atomic, aviation, space rocket, metallurgical, etc.). Therefore creation of

large tonnage production of FZD is not only an important task, but it is also necessary in the case of occurrence of unforeseen external economic and political situations. Research for preparing FZD was quite widespread within the USSR [1, 2] and continues within Russia [3, 4]. Systematic comprehensive study for development of rational methods for preparing FZD is being conducted in NITU MISiS [-57], and also starting from 2010 in OOO Tekhnokeramika (Obninsk, Kaluga Region).

The choice of a melting method is a governing factor for producing FZD. In this case it is necessary to consider that FZD with use of various melting methods may be prepared either in cast form (castings) or in a dispersed form (granules). The form of FZD preparation determines its subsequent use. An example of preparing zirconium dioxide in a cast form is melted and cast refractories containing more than 90% ZrO<sub>2</sub> that the final saleable product. Fuzed granulated zirconium dioxide, apart from use as heat insulation material, is a raw material for semifinished product for subsequent processing, etc.

Consideration of heating methods used for preparing FZD makes it possible to take account of rational requirements for its quality and volume of product, and to determine the form of fusion unit. The main fusion methods during FZD preparation by remelting of synthetic zirconium dioxide or baddeleyite concentrate are induction and electric arc melting

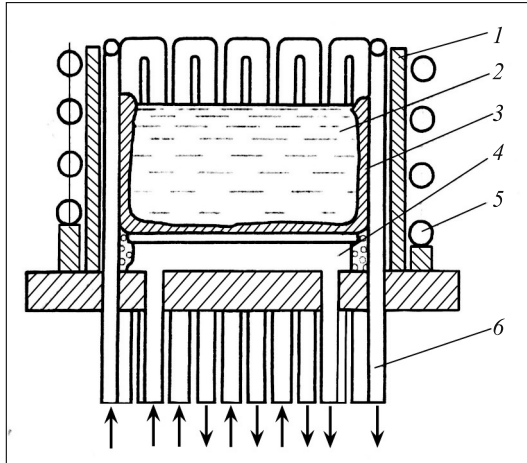
Induction melting is based on a method of direct HF heating and melting oxide materials in a cold crucible. A pe-

<sup>1</sup> FGAOU VO National Research Technological University MISiS, Moscow, Russia.

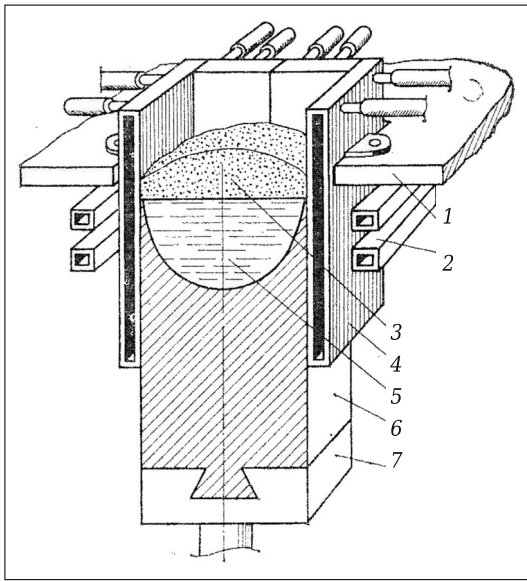
<sup>2</sup> OOO Tekhnokeramika, Moscow, Russia.

<sup>3</sup> FGBOU VO D. I. Mendeleev Russian Chemical Technology University, Moscow, Russia.

<sup>4</sup> sokolov235@yandex.ru



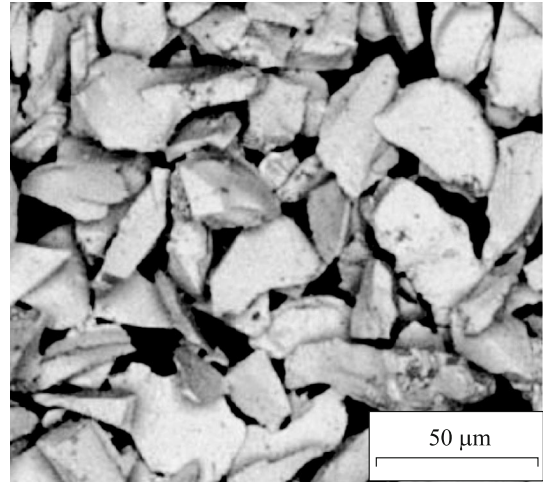
**Fig. 1.** Cold crucible for melting refractory oxide materials: 1) crucible; 2) melt; 3) skull; 4) inductor; 5) cooled bottom; 6) water cooling pipes.



**Fig. 2.** Layout of induction melting and crystallization unit with a rectangular shaft section; 1) assembly plate; 2) inductor; 3) charge; 4) melted charge water-cooled section; 5) melt bath; 6) melted block; 7) mobile hearth.

riodically operating cold crucible scheme is shown in Fig. 1. The production process includes starting heating of a charge (in this case powder of  $ZrO_2$  with additives) to the melting temperature, and an increase in the original melt volume to that prescribed followed by crystallization [8, 9].

A periodic operation induction melting and crystallization unit of the cold crucible type is used for producing  $ZrO_2$  single crystals with different additives in order to create heat-barrier protective coating powders in the aerospace industry and also in the jewellery industry (fianite) [10]. The requirements for producing fused oxide materials are most



**Fig. 3.** FSZD powder fraction 40 – 100  $\mu m$ .

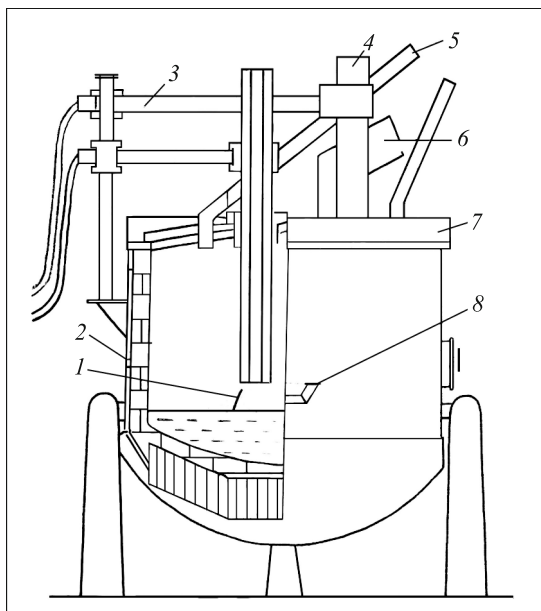
completely satisfied by induction continuous-sequential melting of ingots using a travelling melting and crystallization unit with a moving hearth (Fig. 2).

Features of this process are the continuous melting of an original charge at the molten bath surface and constant crystallization of melt within the bottom part of a bath. The ingot obtained moves upwards from the melting zone by means of a mobile hearth to which it is attached. The specific expenditure for melting in this case is significantly less than with melting in a cold crucible.

Induction melting technology in a cold crucible for powder FSZD, i.e., melting stabilized zirconium dioxide (with calcium and yttrium oxides) is conducted by the enterprise Tekhnokeramika. The FSZD powder (Fig. 3) contains  $CaO$  (4.6%),  $SiO_2$  ( $\leq 0.3\%$ ),  $Fe_2O_3$  ( $\leq 0.1\%$ ). Output is organized within the enterprise for powder fractions coarser than 5, 3 – 5, 1 – 3, 0.5 – 1, 0.2 – 0.5 mm, and 40 – 100  $\mu m$ , and also finely milled powder (finer than 45  $\mu m$ ) with an average particle size of 5 – 10  $\mu m$ .

Use of an induction heating unit is economically valid with use of raw material with high purity specifications (for example for fianite) making it possible to produce highly profitable product. This melt has low charge material losses, which is very important with use of expensive raw material. However, disadvantages of using these units for preparing a considerable volume of FZD is the low productivity and the difficulty of obtaining fine materials.

Electric arc melting, whose source of thermal energy is an electric arc, is used extensively in metallurgy and production of fused oxide materials [11, 12]. As a result of concentrated energy release within a small volume in an electric arc furnace high temperatures are achieved (2200 – 2700°C) required for melting charge based on refractory oxides ( $ZrO_2$ ,  $Al_2O_3$ ,  $MgO$ , etc.). An electric arc also provides a fast heating rate, increased unit productivity, and in the majority of cases the cleanliness required for melted material. Electric arc melting is used to prepare casting of fused and cast



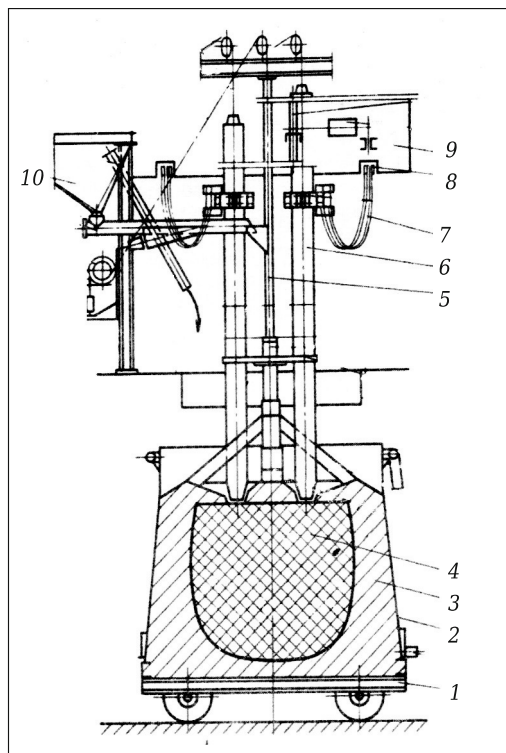
**Fig. 4.** Electric arc furnace for "pouring" melting; 1) arc; 2) body; 3) electrode holder; 4) electrode; 5) charge loader; 6) gas outlet; 7) lid; 8) taphole.

refractories containing  $ZrO_2$  up to 94% (refractory ER 1195). In addition, an advantage of electric arc melting is the possibility of preparing fuzed material in a dispersed form.

Three-phase electric arc furnaces with graphite electrodes and an air cooled vessel are used for melting refractory oxides. Furnaces intended for pouring melt into a casting mold and casting preparation, and also melt granulation, have an inclined furnace mechanism (Fig. 4). Electric arc furnaces are also used with melting "en bloc": a charge is melted within a mold located within the casing of a moving trolley. Casting cooling proceeds within the mold (Fig. 5).

During "en bloc" melting of charges consisting of baddeleyite and chalk in a three-phase electric furnace with power of  $400 \text{ kV}\cdot\text{A}$  (mold 900 mm in diameter and 1200 mm high) for 3 h blocks weighing 680 kg were melted [1]. The electric melting regime: current 3500–4000 A, voltage 82 V. The output of melted material is 68% and for unmelted material it is 32%. Melted material has a dark color as a result of partial  $ZrO_2$  reduction disrupting its stoichiometry and carbide formation. In order to restore  $ZrO_2$  stoichiometry melted material was subjected to oxidation firing at  $1500^\circ\text{C}$  and then ground in screw and beam crushers in order to obtain sizes less than 2 mm. The chemical composition of the ground material fraction 0.5–2 mm after screening and magnetic separation, %:  $ZrO_2$  94.3, CaO 5.5.

FSZD, stabilized with calcium oxide, has also been prepared in a single-phase electric furnace with power of 300 kVA with melting of baddeleyite concentrate with limestone. Melting was conducted "en bloc" with a voltage of 88 V and electrical energy consumption of  $2900 \text{ kW}\cdot\text{h/ton}$  [12, pp. 173, 174]. Data for preparing FSZD have also been



**Fig. 5.** Electric arc furnace for "block" melting; 1) trolley; 2) casing; 3) insulation; 4) melted material; 5) pipe for charge loading; 6) electrodes; 7) line; 8) current supply; 9) casing for gas removal; 10) bunker with charge.

provided in articles [14, 15]. In this case it has been demonstrated that the productivity of "pouring" melting is higher by a factor of 2–3 than "en bloc" melting.

Melting of zirconium dioxide due to a high melting temperature ( $2700^\circ\text{C}$ ) presents considerable difficulties and requires high energy expenditure. Creation of new electric arc units ÉPD-450 and

ÉPD-600M has made it possible to prepare FZD in an oxidation melting regime [16, 17]. In an ÉPD-450 electric arc furnace with power of  $450 \text{ kV}\cdot\text{A}$  melting of zirconium dioxide grade TsrO-1 with chemically pure calcium carbonate has been used to prepare casting weighing up to 32 kg (melting duration not more than 60 min). The composition of the crystalline phase of the VTs-2 material obtained according to x-ray microanalysis data, %:  $ZrO_2$  91.5–92.7, CaO 7.0–7.8, impurities 0.4 [18]. The structure of the material was  $ZrO_2$  crystals of cubic modification with sizes of 0.15–0.35 mm having a shear plane of polygonal isometric shape. A feature of this material is presence of a surrounding crystalline rim 0.002–0.003 mm wide around grains of basic crystallization. Probably this points to presence of small amount of  $ZrO_2$  of monoclinic modification that is indicated by weak peaks in the x-ray pattern for this substance. However, a high degree of  $ZrO_2$  stabilization is confirmed by the dilatometric test curves (Fig. 6) on which there is no hysteresis loop typical for materials of baddeleyite composition. A change



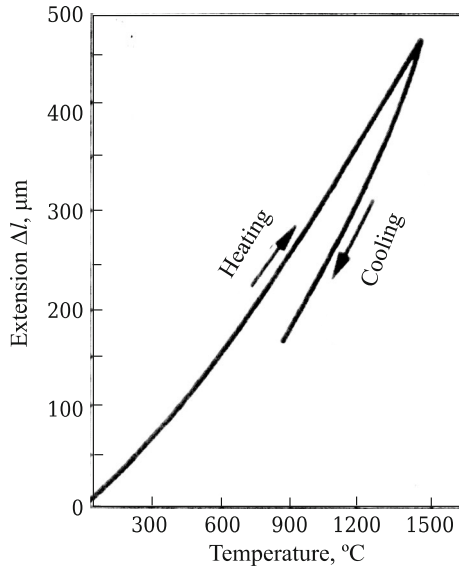


Fig. 6. FSZD VTs-2 dilatometric curves.



Fig. 7. Granulated FSZD.

should be noted in the color of material prepared from light grey to greenish as a result of an especially selected oxidation melting regime. In contrast to this according to data in [1] with arc reduction melting of a charge based on baddeleyite and chalk due to disturbance of  $ZrO_2$  stoichiometry castings acquired a color from dark grey to black. Therefore, for these materials additional firing was necessary in order to restore  $ZrO_2$  stoichiometry.

A modernized ÉPD-600M unit with improved energy properties has made it possible to implement an oxidation melting regime and to obtain a heated liquid flowing melt with the possibility of maximum pouring from the furnace. Therefore a systematic study of the FSZD preparation has been performed in an ÉPD-600M furnace with moving graphite electrodes 100 mm in diameter. The raw materials used were baddeleyite powder grade PB-0 containing 98.5%  $ZrO_2$  and chalk (GOST 8253). After charge melting the melt was held for some time in the furnace and then poured into a graphite mold with preparation of castings weighing up to 50 kg. A series of castings was also made with preparation of FSZD granules and aggregates with size from 1 to 10 mm (Fig. 7).

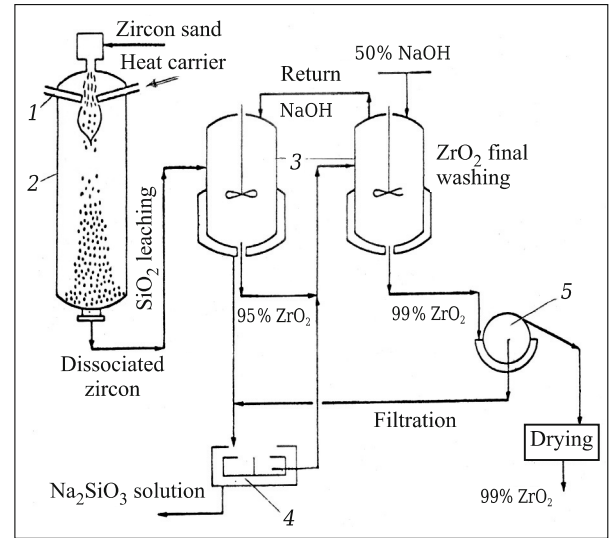


Fig. 8. Equipment layout for FZD preparation by zircon thermal breakdown within a plasma reactor: 1) plasmatron; 2) plasma reactor; 3) reactor-extractor; 4) centrifuge; 5) vacuum-filter.

According to spectrographic analysis data FSZD has a completely crystalline structure of quite coarse  $ZrO_2$  crystals. Castings have coarser crystals (0.40 – 0.60 mm) compared with granules (0.20 – 0.40 mm). The crystalline phase of granular FSZD according to x-ray microanalysis data contains 91.5 – 92.4%  $ZrO_2$  and 7.6 – 8.5% CaO. Results of x-ray phase analysis have confirmed that the sole crystalline compound of all melted castings and granules is a cubic modification of  $ZrO_2$ .

The examples provided of using induction and electric-arc melting point to the possibility of preparing FZD and FSZD by remelting quite expensive powder raw material, i.e., baddeleyite or synthetic  $ZrO_2$ . Synthetic  $ZrO_2$ , prepared by a prolonged and complex hydrometallurgical method is a highly pure and fine grained powder that is used most rationally in the preparation of structural and electronic ceramics. In order to produce the majority of types of refractories and commercial ceramics this form of raw material is quite expensive, which retards effective development. Therefore in order to reduce raw material expenditure methods have been developed for preparing FZD of monoclinic and cubic modifications from zircon material whose cost is an order of magnitude lower than synthetic  $ZrO_2$ . The main method for preparing  $ZrO_2$  from zircon, implemented practically, concerns electric arc and plasma induction melting.

Basic plasma and plasma-induction melting are dissociation of zircon introduced into a high temperature zone (7000 – 9000°C) of a plasma reactor within which it dissociates to  $ZrO_2$  and  $SiO_2$  followed by  $SiO_2$  removal by a hydrometallurgical method. The fundamental equipment layout of a pilot plant unit for producing FZD from zircon sand in a plasma furnace is shown in Fig. 8 [19, 21].

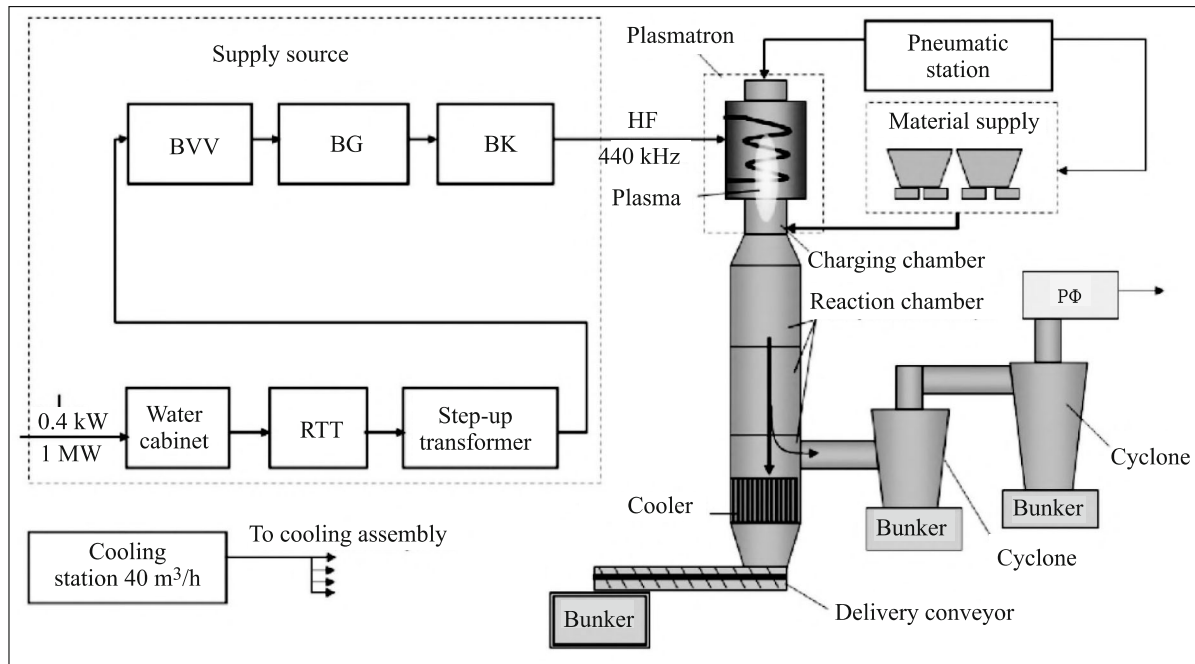
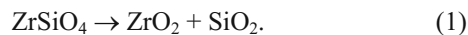


Fig. 9. Layout of HFI-plasma unit.

Zircon sand, ground to a size of 200  $\mu\text{m}$ , is fed uniformly into a plasma discharge created by an arc plasmatron. During fractions of a second of dwell time in a plasma there is melting and almost complete (99%) breakdown of zircon into oxides:



After cooling and solidification a product contains spherical particles of monoclinic  $\text{ZrO}_2$  coated with a layer of amorphous silica; the latter as a result of increased reaction capacity is selectively leached with 1% caustic soda solution at 120°C. After two-stage countercurrent leaching  $\text{ZrO}_2$  is obtained with an  $\text{SiO}_2$  content less than 1% and also sodium silicate solution being a useful subsidiary product. Zirconium dioxide consists of particles represented by complete spheres 0.1 – 0.2  $\mu\text{m}$  in diameter. Depending on user specifications for the product the process may be shortened by using just one leaching. According to data in [19] it is possible to obtain three forms of product containing 70%  $\text{ZrO}_2$  and 30%  $\text{SiO}_2$ , 96%  $\text{ZrO}_2$  and 4%  $\text{SiO}_2$  or 99%  $\text{ZrO}_2$  and 0,5%  $\text{SiO}_2$ . Other examples of FZD preparation from zircon by plasma decomposition have been provided in publications [22 – 24]. Serious disadvantages of arc plasmatrons are the impossibility of prolonged continuous operation with a power of more than 300 kW, significant electrode erosion, and use of argon of a plasma-forming gas.

Use of high-frequency induction (HFI) plasma units for preparing FZD is more effective compared with the arc plasma technique [4]. In a test HFI-plasma unit with power of 1 MV·A the Tekhnokeramika enterprise conducted experiments for thermal decomposition of zircon in an air plasma

(Fig. 9) [25]. A zircon particle is shown in Fig. 10 after plasma treatment represented by sphere consisting of a matrix of zirconium dioxide whose space is filled with amorphous silicon dioxide exhibiting high reaction capacity that accelerates the process of pure zirconium dioxide extraction. In addition, work has been conducted in treating zirconium concentrate decomposition product obtained in an air plasma followed by ammonium fluoride extraction and preparation of materials containing 99.9%  $\text{ZrO}_2$ . Pilot plant batches of  $\text{ZrO}_2$  product have been produced by technology for preparing  $\text{ZrO}_2$  with programmed treatment of plasma-dissociated zircon developed in the Tekhnokeramika enterprise together with the Tomsk Polytechnic University.

Electric-arc melting for FZD preparation by zircon carbothermal decomposition is based on zircon decomposition at high temperature to oxide by reaction (1) and reduction of the  $\text{SiO}_2$  separated to volatile  $\text{SiO}$  monoxide [26]:



Silicon monoxide volatilizes from the furnace working zone and is subsequently oxidized to  $\text{SiO}_2$  whose particles are captured in dust catchers. In order to implement carbothermal decomposition electric arc furnaces have been used with “en bloc” melting (see Fig. 3) and “pouring” (see Fig. 4). In order to prepare FSZD from zircon (65 – 67%  $\text{ZrO}_2$ ) reduction melting “en bloc” is used during which the  $\text{SiO}_2$  content is reduced from 33 to 0.5 – 0.9% [12, pp. 172, 173]. A charge included zircon, iron shavings and metallurgical coke. The melted product was ground, demagnetized and calcined at 1400 – 1450°C for carbide decomposition. FSZD composition, %:  $\text{ZrO}_2$  94 – 95, CaO 4 – 5.

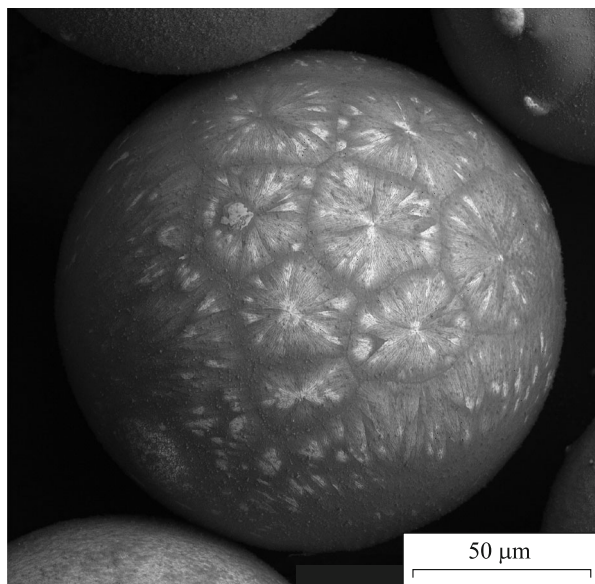


Fig. 10.  $ZrO_2$  particle after treating zircon in an HFI-air plasma.

Implementation of zircon carbothermal decomposition in an ÉDP-450 electric arc unit has made it possible in the first stage to reduce the amount of  $SiO_2$  in zircon by almost a factor of three and also to prepare raw semifinished products containing more than 80%  $ZrO_2$ . With use of an oxidation melting regime and casting coke as carbon reducing agent fused product has been prepared in the form agglomerates with sizes of 10 – 15 mm [27]. The high content of residual silica in fused product is explained by significant wear and combustion of coke in the furnace charging period, which led to a reduction on its reducing function. Achievement of a high degree of silicon removal requires appropriate adjustment of the technology (change in melting regime and conditions, increase in reducing agent efficiency, charge compaction, etc.).

It should be noted that although adequate research has been conducted for preparation of FZD and FSZD pilot plant verification in our country has not been completed due to liquidation of the pilot plant experimental bases as a result of the economic situation in the 1990s. In selecting a melting unit for industrial production of FZD and FSZD it is necessary to consider many years of practice of using electric arc furnaces for preparing fused oxide materials [11, 12]. On the basis of this equipment it is possible to adopt electric arc furnaces types DS-05, DSPM-1.5, and OKB 2126A [16] modernized in accordance with specifications of fused product and on a production scale.

## CONCLUSION

1. Results of analyzing melting units indicate that in order to create pilot plant production of FZD and FSZD from baddeleyite concentrate or synthetic  $ZrO_2$  it is more rational

to use an electric arc furnace with the possibility of performing an oxidation melting regime. Creation of a pilot plant electric arc unit based on the DS-0.5 equipment has made it possible to produce within a year by the melting method up to 1000 tons of fused granular  $ZrO_2$  of monoclinic modification and  $ZrO_2$  stabilized with calcium or yttrium oxides. This product is intended primarily for the main users, i.e., enterprises for manufacturing refractories, abrasives, ceramics, and pigments. In order to prepare structural and electronic ceramic oxide sensors with increased specifications with respect to raw material cleanliness it is possible to use induction and plasma units taking account of the economic expediency.

2. In order to prepare FZD and FSZD from zirconia raw material two versions are possible: electric arc carbothermal and plasma with use of hydrometallurgy. Use of an electric arc furnace for carbothermal treatment of zircon will make it possible to organize quite large production (several thousand tons a year) of FZD and FSZD. However, this requires special melting equipment, which besides the high temperature of the process should provide removal of considerable amount of silicon monoxide; in this case melt granulation during casting is required.

3. Use of plasma units will make it possible to organize medium tonnage production with output of individual batches of FZD and FSZD. The  $ZrO_2$  produced in this case exhibits good cleanliness and has a particle size suitable for use in various industries without additional crushing and grinding.

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