INFLUENCE OF TECHNOLOGICAL PARAMETERS ON THE PROPERTIES AND WEAR RESISTANCE OF PERICLASE–CARBON REFRACTORIES

K. V. Simonov, V. V. Zagnoiko, G. V. Burdina,
V. I. Sakk, and A. I. Katunin

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One of the most progressive ways of developing the refractories industry is to develop a technology and master the production of highly resistant periclase-carbon refractories for converters with combined blow, power electric furnaces, and plant for the vacuum treatment of steel (UVS).

Abroad many types of periclase-carbon refractories have been developed, differing in their concentrations of residual carbon, type of carbon, periclase, bond, special additives, and designed above all for converters with bottom blow, and high-power electric furnaces, and UVS [1].

Work has been done in the USSR aimed at developing wear-resistant periclase-carbon refractories using domestic materials [2-4]. Such work was done in order to determine the relationship between their physical properties (compressive strength σ_{comp}, open porosity P_{op}, apparent density ρ_{app}, wear resistance) and the type and amounts of bond, the grain-size and chemical compositions of the starting materials, the content of graphite, and the fabrication pressure. The properties were determined on laboratory and industrial specimens. The fillers consisted of fused and sintered periclase powders in fractions of 3-5, 3-1, 1-0.5, and minus 1, and minus 0.063 mm; and the carbon – in the form of graphite; as the bond, phenol powder (SFP), and a combination with liquid phenol-formaldehyde bonds of the resol type (liquid bakelite grade BZh-3, bakelite lacquer grade LBS-1), nontoxic water solutions of lignosulfonates (LST), and sodium polyphosphate (PFN).

The bodies were prepared in a runner mill mixer. The specimens were pressed in a hydraulic press and heat treated at 180 and 220°C in a drying cupboard with different rates for the temperature rise and soaking and at a maximum temperature for 4 h; and also at 800°C in a coke filling in a muffle with the same soaking at maximum temperature.

When all the tested types of periclase were used in the batch it was noted that all the specimens containing the bond in the form of 3% SFP + 5% BZh-3 have a high strength (Table 1).

The specimens pressed from the crushed periclase scrap (unfired and fired) possess the highest strength but with some reduction in the density, which indicates the possibility of partial use of scrap products in the batch of periclase-carbon refratories. The specimens made from fused pariclase have a higher density. The dimensions of the crystals of fused periclase are one order higher than that of the sintered. Since the coarse crystalline fused periclase has fewer boundaries through which the slag can penetrate downwards, then the corrosion resistance is higher than for the finely crystalline sintered material [5].

With an increase in the porosity of the sintered powders and an increase in the content of calcium oxide, there is a marked fall in the apparent density, and a rise in the porosity of the articles. It is known that with an apparent density in the refractory of less than 2.82 g/cm^3, and with 20% graphite there is a sharp rise in the rate of wear [5]. To obtain high-grade periclase-carbon articles with an apparent density of not less than 2.85 g/cm^3 it is necessary to use dense periclase powders whose open porosity does not exceed 8%.

During a study of the influence of the bond on the properties of the articles, the amount of graphite was kept constant (10-12%), the amount of finely milled periclase at 20-23%, the granular material (fractions 5-0.063 and 3-0.063 mm) 67-70%. The concentration of SPF was constant and equaled 3%. The specimens were pressed at 100 N/mm^2 and heat-treated with the LST at 180 and 800°C, with LBS-1 and BZh-3 at 220 and 800 °C. Figure 1 shows the influence of the amount and form of liquid bond on the strength and porosity of the articles.

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TABLE 1. Characteristics of the Original Periclase and the Properties of the Periclase-Carbon Specimens

Periclase	Characteristics of periclase					Properties of articles*		
	fractions, mm, minus	open porosity, %	mass %			σ_{comp}, N/mm^2	P_{op}, %	σ_{app}, g/cm^3
			MgO	CaO	SiO_2			
Fused	5	3,0	95,3	1,9	0,6	46,6/16,7	2,7/10,5	2,86
	3	3,1	95,3	1,9	0,6	51,1/22,2	3,1/11,7	2,87/2,85
Sintered	5	6,8	91,2	3,4	2,0	30,1/16,6	4,3/10,0	2,80
	3	5,6	92,1	2,6	1,9	42,7/14,8	4,3/12,5	2,84/2,81
	5	16,7	82,7	10,8	2,3	40,9/17,4	11,4/19,9	2,61/2,60
	5	20,3	92,5	3,0	1,9	49,2/17,1	6,9/16,9	2,69/2,63
Crushed from scrap								
fired	5	16,7	92,2	2,7	2,4	60,5/29,4	5,5/16,0	2,69/2,67
not fired	5	7,3	91,4	3,0	2,5	66,6/25,0	4,3/10,9	2,66/2,63

*The numerator shows the properties of specimens after heat treatment at 220°C; and the denominator at 800°C.

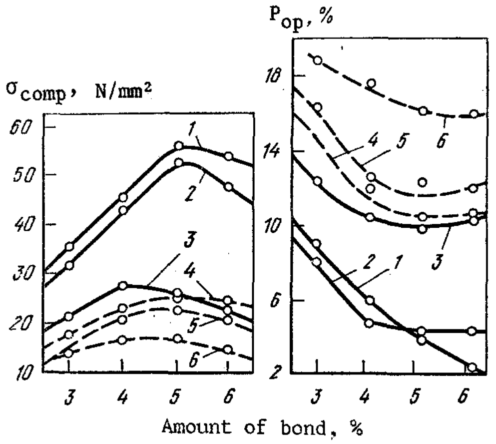

Fig. 1

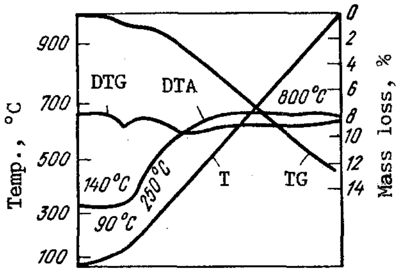

Fig. 2

Fig. 1. Change in σ_{comp} and P_{op} of periclase-carbon specimens in relation to amounts and form of liquid bond: 1, 4) BZh-3; 2, 5) LBS-1; 3, 6) LST; ——) heat treatment at 180 and 220°C; -----) at 800°C.

Fig. 2. Thermograms of periclase-carbon specimens.

With increase in the amount of liquid bond (BZh-3, LBS-1, and LST) from 3 to 5%, the strength of the articles rises sharply, and the porosity is reduced; with more than 5% bond the converse is true. Specimens with BZh-3 and LBS-1 have 1.2-2 times the strength, and a lower porosity compared with specimens containing LST. The liquid resin (LBS-1, BZh-3) wets the powdered resin, and SFP is partly dissolved in the liquid resin, yielding a solution which possesses good adhesive properties, strongly bonding the grains, and forming a strong coke framework during heating.

With the use of LST at first during heating the water evaporates, then the SFP melts, enveloping the grains of powder, and together with LST attaches them to each other, giving strength to the articles. With increase in the heat treatment temperature to 800°C the strength is reduced, and the porosity is approximately 1.5-2.5 times higher (see Fig. 1) as a result of the thermal destruction of the organic bond. The start of thermal destruction is fixed on the thermogram* at 250°C (Fig. 2). The total mass loss is 12.3% including 1% loss connected with moisture removal, and 11.3% with the combustion of the bond and the graphite.

The influence of the content of SFP with constant amounts of BZh-3 and LST on the strength and porosity of the specimens is shown in Fig. 3, and the apparent density of the greenware in Fig. 4a. With increase in the amount of SFP from 2 to 4% in combination with BZh-3, we note a rise in the strength of the speicmens heated at 220°C (180°C). Further increase in the SFP concentration in the batch reduces the strength of the articles.

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*Carried out by the East Institute of Refractories.

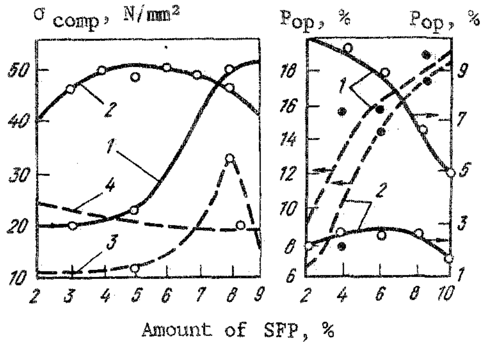

Fig. 3. Relationship between σ_{comp} and P_{op} of periclase-carbon specimens and the amount of SPF and form of liquid bond: 1, 3) specimens containing LST; 2, 4) with BZh-3. ———) heat treated at 180 and 220°C. -----) at 800°C.

The porosity of the specimens, regardless of the liquid bond, with rise in the SFP amount in the batch, is reduced in specimens heated at 220°C (180°C), and is increased in specimens heated at 800°C.

It is known that an increase in the graphite content increases the corrosion resistance of the refractories.

In determining the influence of the amount of graphite on the strength of the specimens, we used liquid bakelite, BZh-3, as the bond, and a solution of lignosulfonates. The powdered phenol bond was added in amounts of 4 and 8%, respectively, in combination with BZh-3 and LST. The relationship between the apparent density of the green brick and the strength of the specimens, and the graphite content and form of bond are shown in Fig. 4b and in Fig. 5.

With an increase in the graphite content in the batch to 30%, the apparent density of the green brick falls continuously. An increase in the amount of graphite to 15% leads to the most significant reduction in the strength of the specimens (by a factor of 1.3-1.7), when heated at 180-220°C. With incerase in the amount of graphite from 15 to 30% the fall in strength is less marked. During heating of specimens at 800°C the influence of the graphite on the strength is sharply reduced (see Fig. 5). With increase in the content of graphite from 10 to 15%, and especially above 15%, there is a sharp increase in the overpressing of the articles. Without using special methods for its elimination the optimum amount of graphite added to the articles should be 10-20%. With the LST bond overpressing develops more strongly than when BZh-3 and LBS-1 bonds are employed.

The amount of finely milled periclase also has an effect on overpressing and the quality of the goods. The effect was detected on specimens pressed with the use of 5% LST + 8% SFP as bond. The addition of finely milled periclase to the batch in amounts of more than 25% leads to a reduction in the strength of the specimens due to the appearance of overpressing cracks.

The rise in the strength of specimens is noted with an increase in the body in the amount of finely milled periclase to 20-25% (Fig. 6); the porosity of the specimens is increased, and the apparent density of the green brick is reduced (Fig. 4c). The rise in the fabrication pressure from 100 to 160 N/mm² leads to an increase in the strength, and a fall in the porosity (Fig. 7), and in the apparent density of the specimens (Table 2).

The increase in the fabrication pressure (>120 N/mm²) of the bodies with a high content of graphite (>20%) has a slight effect on the strength and density of the goods. With an in-

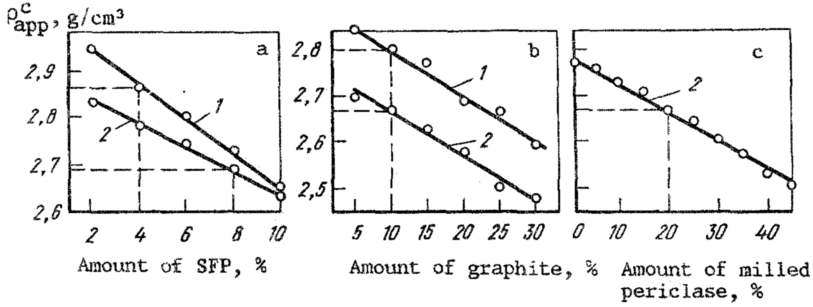

Fig. 4. Change in the apparent density of the green brick ρ^c_{app} in relation to the amount of SFP (a), graphite (b), and finely milled periclase (c): 1) specimens with BZh-3; 2) with LST.

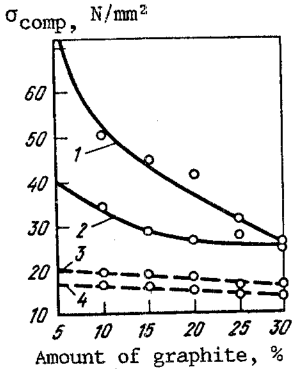

Fig. 5

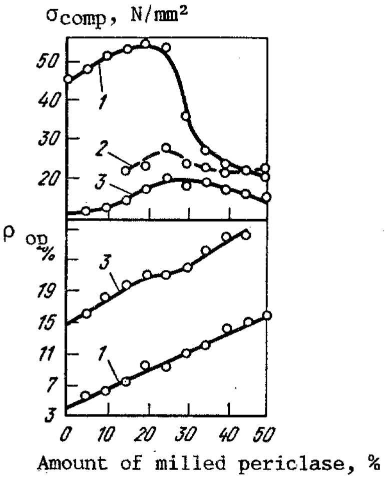

Fig. 6

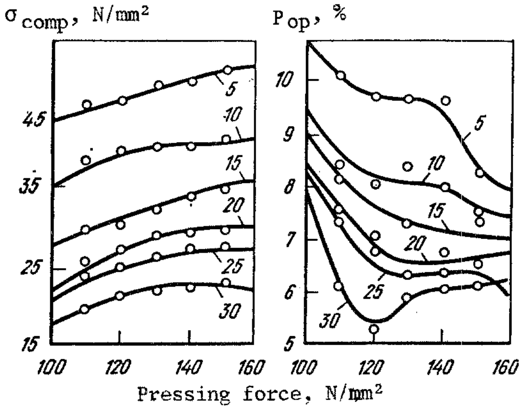

Fig. 7

Fig. 5. Relationship between σ_{comp} of periclase-carbon specimens and the amount of graphite: 1, 3) specimens with BZh-3; 2, 4) with LST. ——) Heated at 180 and 220°C; -----) at 800 °C.

Fig. 6. Change in σ_{comp} and P_{op} of periclase-carbon specimens in relation to the amount of milled periclase and the type of bond: 1, 2) specimens with BZh-3; 3) with LST; ——) heated at 180 and 220°C; -----) at 800 °C.

Fig. 7. Relationship between σ_{comp} and P_{op} of periclase-carbon specimens and the pressing force and amount of graphite (stated on curves, %).

TABLE 2. Change in the Apparent Density of the Green Brick and Those Heat Treated in Relation to the Amount of Graphite and the Stressing Force

Fraction pressure, N/mm²	Apparent density of the green articles, g/cm³, with a graphite content, %						Apparent density of specimens, g/cm³, with a graphite content, %					
	5	10	15	20	25	30	5	10	15	20	25	30
100	2,86	2,84	2,77	2,68	2,67	2,63	2,82	2,80	2,71	2,62	2,61	2,57
110	2,88	2,87	2,79	2,74	2,69	2,64	2,85	2,83	2,75	2,68	2,65	2,61
120	2,86	2,83	2,79	2,73	2,68	2,65	2,86	2,79	2,74	2,69	2,61	2,59
130	2,89	2,87	2,82	2,74	2,68	2,64	2,87	2,86	2,78	2,71	2,65	2,57
140	2,91	2,88	2,84	2,75	2,71	2,65	2,86	2,83	2,78	2,70	2,65	2,56
150	2,93	2,90	2,84	2,75	2,72	2,67	2,87	2,83	2,75	2,65	2,63	2,59
160	2,95	2,89	2,85	2,76	2,73	2,69	2,88	2,83	2,79	2,70	2,66	2,61

crease in the amount of SPF, the apparent density of the articles is reduced (Fig. 8). The maximum value of the apparent density is attained with a pressing force of 140 N/mm², and a batch content of 2% SFP.

Together with the liquid bonds of organic composition we also tested a mineral composition — an aqueous solution of sodium polyphosphate (PFM) having a density of 1.55 g/cm³. With a combination of powdered phenol resin SFP and a solution of PFN we traced the same relationship between the properties and the technological parameters as for the combination of SFP + LST. With rise in the amount of graphite there are falls in the strength and porosity of the specimens (Fig. 9). The maximum values for the strength of the specimens heated at 220 and 800°C are noted respectively at 8 and 2% SFP. The sodium polyphosphate was used here not only as a bond but also as in antioxidant, reducing the oxidation of the carbon and weakening the articles during heating [6].

As a result of laboratory and semiindustrial investigations, we selected the optimum batch compositions which were taken as the basis for producing an industrial batch of articles.

Using a scrap-free technology, we prepared an experimental batch of articles and large blocks from fused periclase with mass proportions of more than 95% MgO (fractions 3-1, 1-0, and <0.063 mm) with additions of 12-15% graphite, 15% carbon-containing pellets (scrap from

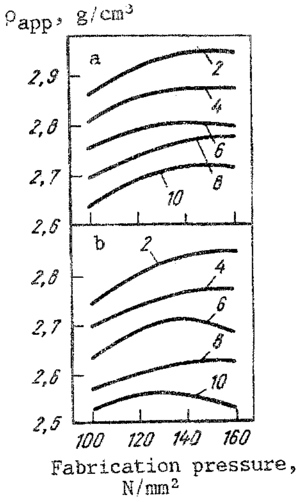

Fig. 8

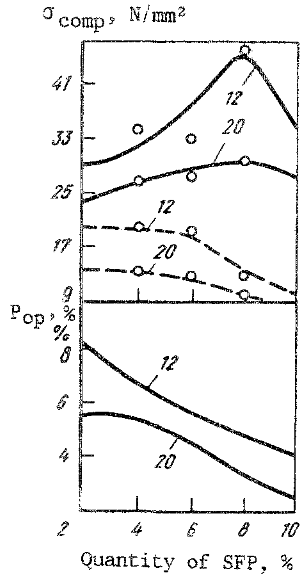

Fig. 9

Fig. 8. Relationship between ρ_{app} of periclase-carbon specimens and the pressing force, type of bond, amount of SFP (stated on curves, %); a) specimens with BZh-3; b) with LST.

Fig. 9. Change in σ_{comp} and P_{op} of periclase-carbon specimens containing PRN in relation to the amount of SFP and gaphite (indicated on the curves, %): ———) heated at 220°C; -----) at 800°C.

periclase-carbon articles), and waste products from the melting of periclase in the block (skin), using a bond made of SPF + LBS-1 and SFP + LST.

The second batch of articles was prepared also with the scrap-free method, from sintered periclase with more tham 91% (mass) MgO (fractions 3-1, 1-0, and <0.063 mm) with an addition of 10-15% graphite and a bond of SFP + LST without antioxidant and with it in the form of a finely dispersed mixture of combined milled periclase and iron [7, 8]. The sintered powders were used, both after preliminary hydration aging, and also without the aging. The use of dry powders without aging yielded articles with higher apparent density (by 0.1 g/cm³) and strengths, compared with articles made of moist powders after aging. The open porosity of the grains of fused periclase was 2-5%, and for sintered 8-10%. The sintered powders were used without aging.

The body from fused periclase was prepared in runner mills, and that made of sintered periclase in high-speed centrifugal mixers, using the known mixing schedule [9]. The body made in the runner mill was more uniform, without lumps and rolled-out pieces, compared with the body obtained in the centrifugal mixer. In the latter the body was additionally heated, and many rolls were formed with the use of the finely milled periclase at temperatues above 35°C. This body needs to be passed through a screen-abrader.

With the use of the liquid bonds BZh-3 and LBS-1, the concentration of phenol, formaldehyde, and dust on the mixer platform and the press exceeded the planned factors. Therefore, in producing the second batch of articles, instead of the toxic LSB-1 and BZh-3 liquid bonds we used the nontoxic, water solution of LST, in combination with the powdered phenol SFP resin [8]. The liquid bond LST in combination with SFP ensures a high plasticity, a long life and good pressibility in the body; adequate strength in the greenware, resistance to deformation during heating, and also safe working conditions.

The articles were pressed on hydraulic presses at a pressure of 125-150 N/mm² with a double and a triple-staged schedule of increasing pressure. The apparent density of the green brick varied within the range 2.83-2.94 g/cm³.

The articles were heat processed in a tunnel dried with a double-staged schedule first to remove the moisture and then up to the setting state of the phenol resin [8]. The heat-

TABLE 3. Characteristics of the Periclase-Carbon Articles

Factors	Articles	
	from fused periclase with SFP+ LBS-1	from sintered periclase with SFP + LST
Mass proportion, %:		
MgO	80,86	78,26
C (from Δm_{calc})	16,60	13,05
Compressive strength, N/mm²	51,8	35,2
Open porosity, %	4,9	5,8
Apparent density, g/cm³	2,95	2,87
Refractoriness under load 0.2 N/mm², °C	>1700	

ing rate was about 10 °C/h [10]. The larger the articles the stronger the influence of the heating rate on their quality. The properties are shown in Table 3.

The articles have relatively good properties. Those made from fused periclase with the SFP + LBS-1 bond have better properties in terms of strength and density than the articles made from the sintered periclase with SFP + LST bond. The temperature of initial softening of both types of refractory is high.

The periclase-carbon refractories made of sintered periclase were used in the linings of the walls of 100-ton arc steel melting furnaces (DSP) at KMK steel combine from the level of the slag belt to the water-cooled panels, instead of the fired PKhP refractories made of fused periclase and periclase-chromite. It should be noted that with the use of the water-cooled panels in the walls and roof of modern DSPs there is a reduction in the consumption of refractories by a factor of 5-10, and for graphitized electrodes by 10-15% [11]. The periclase-carbon articles were laid on edge, dry (without mortar). In the rings of the structure we obtained several joints up to 10 mm thick because of the inadequate wedge-shaped nature of the articles (380 × 150 × 85 × 75 mm). The joints were finished with magnesite powder.

During the experimental campaigns meltings were produced of tool, alloyed carbon, and chrome grades of steels. The average melting time was 3 h 4 min. The metal temperature at discharge was 1560-1630°C. During the campaign the wall linings were fettled with a mixture of magnesite powder and scrap products, slaked with water glass. The service results of the periclase-carbon articles compared with PKhP refractories are shown in Table 4.

The periclase-carbon refractories made from sintered periclase demonstrated a high resistance in the linings of the walls of 100-ton steel furnaces, and 29% less wear than the fires PKhP refractories made of fused materials. These results were obtained with a 16% higher consumption of oxygen for the periclase-carbon lining compared with PKhP linings. The latter is the same conditions of service show 1.24-2.2 times higher resistance in the lining of the walls of arc furnaces compared with PKhS [12].

It should be noted that there is a lower consumption of mixture of magnesite powder and scrap for fettling the linings made of periclase-carbon refractories than for the single melt repair of the linings made from PKhP brick.

The wear of the PKhP articles occurred mainly through scaling and partly fusing; the periclase-carbon — by fusing of the reaction zone 0.5-2.0 mm thick, formed as a result of the combustion of the carbon and corrosion of the periclase. The use in the latter of periclase-carbon articles measuring 460 × 150 × 79 × 68 mm enables us to eliminate the wide joints, increase the resistance of the lining, and release scarce, fusion-granular refractories, PKhP, for use in units processing steel by vacuuming.

The periclase-carbon blocks 920 mm in length, part of whose fused periclase is replaced with scrap from electric melting, and also prepared with nontoxic bond SFP + LST have been tested in service in the tuyere locations of the lining of the bottoms of converters as the combined blow at ZSMK and showed the same wear resistance as the blocks of fused periclase

TABLE 4. Service Results of Periclase-Carbon and Periclase-Chromite Refractories

Refractories	Campaign	Resistance, heats	Lining thickness, mm		Rate of wear per heat, mm	Consumption per ton of steel		
			original before service	residual after service*		oxygen, m^3	mixture of periclase powder and scrap	electric energy, thousand kW·h.
PKhP	1	1160	460	Not det'd.	—	42,0	12,5	553,1
	2	903	460	» »	—	46,7	16,1	542,7
Periclase-carbon	3	999	380	» »	—	53,7	12,4	531,0
	4	1140	380	» »	—	52,6	11,7	518,5
Periclase-carbon and PKhP	5	590†	380‡	$\frac{195}{150-250}$	0,30	52,0	11,8	506,0
			460**	$\frac{214}{200-230}$	0,42			

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*The numerator shows the averages and the denominator the ranges.
†The furnace was stoopped for planned repairs with a large residual thickness in the lining. The average life of the PKhP material was 1031 heats, of the periclase-carbon 1064 heats.
**Length of periclase-carbon articles, mm.
‡Length of PKhP articles, mm.

carbon blocks of sintered periclase, or from a mixture of it with fused material. It is also necessary to identify the influence of additions of antioxidants on the existence and service of periclase-carbon refractories.

CONCLUSIONS

A study was made of the influence of the type of periclase, the form and amount of bond, the concentration of graphite and milled periclase, the pressing pressure, and the firing temperature on the compressive strength, apparent density, and open porosity of periclase-carbon refractories.

We developed an industrial, scrap-free technology for the manufacture of periclase-carbon refractories from fused and sintered periclase and graphite with organic bonds SFP + LSB-1 and nontoxic SFP + LST, with the use of scrap from electromelting, and scrap products.

We determined the technological parameters for obtaining high-quality periclase-carbon refractories, including those with nontoxic bonds, with good strength, porosity, and refractoriness under load factors.

The resulting periclase-carbon materials ensure high wear resistance and efficiency to the lining of walls in high-capacity arc, steel-melting furnaces with water-cooled panels, in comparison with PKhP refractories and PKhS materials, which facilitates a reduction in the specific consumption of refractories, including fused. Replacing PKhP and PKhS materials with periclase-carbon will eliminate loss in firing and the consumption of fuel for preparing the goods, and it will also release fusion-grain refractories PKhP for UVS units.

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SILICON CARBIDE PRODUCTS FOR FIRING PORCELAIN

E. A. Gerasimova, I. Ya. Guzman,
G. E. Karas', and V. P. Kryuchkova

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The firing of porcelain in tunnel kilns is characterized by a variety of temperature schedules and gaseous media [1, 2]. The severe conditions of operation due to the cyclic action of the thermal and mechanical loadings in the range from 20 to 1420°C, in conditions of oxidizing-reducing media, place increased demands on the quality of the silicon carbide refractories used (tiles, pillars, etc.). The maximum service temperature of such kiln furniture is 1600°C [3].

Silicon-carbide slabs suitable for porcelain production are produced by the Semiluksk refractories factory. The manufacturing technology for grade KA-3, as specified by GOST 10153-70, specifies the use of clay in the form of a mixture of materials obtained by combined grinding in tube mills with fine fractions of SiC and scrap SiC products. The plasticizer-bond material consists of concentrates of technical lignosulfonate (LST) obtained in the process of biochemical desugaring of sulfite lye [4].

Cellulose-paper industries produce several grades of liquid lignosulfonates. Refractories producers use concentrate type A with improved properties, the organic part of which consists of the sodium salts of lignosulfonic acids [5]. According to chemical analysis data of the dry residue of lignosulfonate, the mass part of sodium oxide equals from 45 to 50%.

The firing of silicon carbide articles KA-3 is done in an oxidizing atmosphere. In the fired articles the silica content varies from 10 to 15% by weight. According to the phase diagram in the system Na_2O-SiO_2 at 100-1100°C from 17 to 20% liquid phase is formed, containing 78.2% SiO_2 and 21.8% Na_2O on account of the vigorous reaction of the sodium oxides and silica. The substantial quantity of Na^+ ions on the surface of the articles is confirmed by analytical data obtained on the laser microanalyzer.

The densification of the articles in the process of oxidation and the formation of a liquid phase leads to its extrusion in amounts of from 25 to 40% on the surface of the articles, and the appearance of surface defects in the form of bubbles and melt-outs occupying from 30 to 50% of the surface of the articles as a whole. The surface defects impair the quality of the SiC products; and the use of items with defects increases the loss of the fired porcelain, and increases the consumption of engobe coatings on the SiC slabs.

The aim of the combined work of the Eastern Institute of Refractories and the Semiluksk refractories factory was to eliminate surface defects on KA-3 products. For this, in industrial conditions various bonds were tested: polyvinyl alcohol (PVS), carboxymethyl cellulose (KMTs), carbophenol-formaldehyde resin (KFMT-300), and ammonium lignosulfonate (LST grade D).

Some properties of the regular and experimental articles are shown in Table 1. The data indicate that the bonds not containing sodium ions (compositions Nos. 2-5) do not cause the surface defects. It is found that the use of polyvinyl alcohol, carboxymethyl cellulose, and carbophenol-formaldehyde resins in production is technically difficult, since the pressed greenware has low strength, which hinders the removal of items from the press. Therefore, as a temporary bond it is best to use lignosulfonate grade D containing ammonium compounds.

CONCLUSIONS

The cause of the development of surface defects (bubbles and meltouts) on silicon carbide articles grade KA-3 at the Semiluksk refractories factory is the use, as temporary bond, of

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