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SCIENTIFIC RESEARCH

EVALUATION OF THE SERVICE CHARACTERISTICS OF THE EXPERIMENTAL BATCHES OF INDUCTION-MELTED ELECTRICAL-ENGINEERING PERICLASE

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This paper presents the results of our experimental studies on the service characteristics of electrical-engineering periclase that was obtained by induction melting in a cold crucible (IMCC).

The previous studes showed that it is promising to use the clean (uncontaminating) method of IMCC for obtaining high quality electrical engineering periclase (in conjunction with its heat treatment [1] and antihydration treatment [2]).

In order to determine the service properties, we took three experimental batches of electrical engineering periclase, viz., No. 1 – periclase containing Ga_2O_3 addition with subsequent high-temperature firing; No. 2 – periclase heat treated after melting; and No. 3 – unheat-treated periclase that was treated with the GKZh-94 silicone (organosilicon) liquid for protecting it against hydration during its storage. Preparation of the periclase powders obtained by IMCC was carried out according to the procedure described elsewhere [1].

Microstructural studies carried out on periclase according to the methods of x-ray diffraction and microscopic analysis showed that periclase of the Nos. 1 and 2 batches consists of colorless or pale yellow grains. The content of the impurities present in the form of simple and complex oxides is insignificant. CaO is virtually absent (<0.01 %). SiO_2 is present in the form of cristobalite, enstatite ($MgSiO_3$), wollastonide ($CaSiO_3$), and forsterite (Mg_2SiO_4).

The cristobalite content amounts to 1.0-1.5% (it is somewhat lower in the No. 2 batch) and the Fe_2O_3 and Al_2O_3 contents amount to 0.005 and 0.048%, respectively. The Ga_2O_3 addi-

TABLE 1. Granulometric Composition of the Experimental Batches of Periclase Obtained by IMCC

Periclase	Residue on sieve, %, with the sieve number						
	05	04	025	016	0063	004	<004
Batch							
№ 1	0,7	9,2	27,1	11,9	20,0	11,3	19,8
№ 2	0,4	5,7	29,5	11,5	20,3	12,1	20,5
№ 3	1,2	12,0	27,3	10,7	38,3	6,9	3,6
According to GOST 13236—83	—	<2	>30	—	>30	<8	<4
PPE-1M	—	1,2	29,8	32,4	31,7	3,3	1,6
'Dinaterm'	—	—	33,2	30,1	30,5	5	1,2

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TABLE 2. Electrical Resistivity of the Experimental Batches of the Electrical-Engineering Periclase Obtained by IMCC

Batch number	Resistivity, Ω·cm, at a temp., °C		Quality grade according to GOST 13236-83, class
	1000	800	
1	$2.0 \cdot 10^7$	$4.0 \cdot 10^8$	I
2	$2.3 \cdot 10^7$	$5.6 \cdot 10^8$	I
3	$1.1 \cdot 10^7$	$7.2 \cdot 10^7$	II

tive introduced into the No. 1 batch creates secondary formations on its grains that occupy 5-10% of their surface area and appear as irregular isotropic grains or as their extensions (concretions) having a refractive index corresponding to that of the $MgGa_2O_4$ spinel.

The No. 3 batch is characterized by a higher (up to 2%) silicon oxide content (as compared to the Nos. 1 and 2 batches) owing to the presence of a silicone film on the periclase grains. This semitransparent film covers 10-20% surface area of the periclase grains and is also observed along the edges of the grains and along the boundaries of their growth within the blocklike (coarse) grains.

Table 1 shows the granulometric composition of the experimental powders that was determined according to the procedure given in GOST 13236-83. For the purpose of comparison, the granulometric compositions of periclase according to GOST 13236-83, the PPÉ-1M grade periclase obtained at the Bogdanovsk Production Complex of refractory materials, and the 'Dinaterm' (FRG) grade periclase are also presented.

Table 1 shows that treating periclase with the GKZh-94 liquid leads to particle coarsening due to agglutination: the number of the particles belonging to the minus 0.04 mm fraction decreases approximately from 20 up to 3% and this leads to an increased quantity of the particles finer than 0.063 mm.

The differences observed in the granulometric compositions of the periclase batches obtained by IMCC and the periclase powders used in the Soviet and the foreign industries can be attributed to the use of the capron sieves whose mesh size does not coincide with that of the commonly used wire sieves. Using capron sieves is preferable since they do not introduce impurities that adversely affect the electrophysical properties of periclase. The grain shape of the induction-melted periclase is characterized by certain features: many crystals are acicular and their borders (facets) are not rounded.

All the aforementioned factors led to the differences in the technology of making the specimens from the periclase obtained by IMCC and the experimental specimens from the periclase used in the Soviet and the foreign industries.

The electrical resistivity of the aforementioned batches of electrical engineering periclase was measured according to GOST 13236-83 (Table 2). We note that before carrying out the measurements, periclase of the No. 3 batch was subjected to calcination in order to remove the organic constituent of GKZh-94.

The fact that periclase of the No. 3 batch corresponds to the II class according to GOST 13236-83 owes to use of the powders that were not heat treated before applying the GKZh-94 coating on them. Redetermination of the electrical resistivity of this batch of periclase after storing it for one year showed that its resistivity did not decrease; this fact indicates the high effectiveness of the antihydration treatment of periclase with the GKZh-94 liquid. The previously conducted experiments showed that the electrical resistivity of unheat-treated periclase decreases significantly after prolonged storage [2].

Accelerated high-temperature testing of the electrical engineering periclase obtained by IMCC was carried out according to the procedure recommented by Aver'yanov et al. [3]. The tests were conducted under the conditions of high voltages at a constant power which ensured a constant average spiral (coil) temperature.

In order to carry out the test, specimens were prepared according to the well known technology [3]. The specimens measure 210 mm in length and the resistor element is in the form of a coil made from a wire measuring 0.4 mm in diameter.

The following are the main parameters of the accelerated tests: the nominal power 800-850 W, the specific power 10.5 W/cm^2, the average temperature of the jacket (920±10)°C, and the cycle time 12 h. The specimens were not hermetically sealed.

During the tests, we measured the ohmic resistance at the room temperature. The tests were continued until not less than three out of nine test specimens became unusable (failed). Based on the experimental data, we calculated the average service period of the specimens τ_{av}:

$$\tau_{av} = \frac{\sum_{i=1}^{3} t_i + (9-3)t_2}{9},$$

where t_i is the service period of the specimens rejected before completing the tests; and t_2 is the service period of the specimens that were not rejected before completing the tests.

The average service period of a specimen depends on the resistivity, the leak currents, the breakdown voltage, the thermal conductivity of periclase, the characteristics of the jacket and the coil of the specimen, and their interaction with periclase. One can consider the magnitude of the average service period as the main (generalizing) characteristic of the periclase powder (charge) as well as the specimen as a whole.

We note that the average service period is a quality index of periclase provided that the other materials of the system (the resistor and the tube-jacket) remain unaltered. In order to ensure 'purity' of the experiment, it is necessary to exclude the effect of the technological rejections; during the tests, this was achieved using x-ray defectoscopy of the specimen and using a component of simple configuration.

Based on the conducted tests, we determined the average service period of the specimens made from periclase of the Nos. 1, 2, and 3 batches. It was found to be 560, 670, and 640 h for the Nos. 1, 2, and 3 batches, respectively. The average service period of the 'Dinaterm' grade periclase and the PPÉ-1M grade electrical engineering periclase were found to be 410 and 610 h, respectively.

In order to understand the effect of the method of removing the organic constituent of GKZh-94 and the effect of the dissociation products of GKZh-94 on the coil of the specimen, we made specimens using periclase of the No. 3 batch without calcining before testing. In this case, burn-off of the organic constituent occured in the specimen.

Before carrying out the life (resource) test, five specimens were subjected to active heating (power 600 W, surface temperature approximately 800°C, heating period 13 h; heating was out according to a cyclic regime); the remaining five specimens were heated in a muffle furnace at a temperature of 800°C for 5 h (excluding the heating period and the time required for cooling the furnace). In the first case, the average service period was found to be 690 h and in the second case, it amounted to 600 h. The obtained results show that active heating is more effective since the dissociation products of GKZh-94 are carried away from the coil due to diffusion. In the case of passive heating in a muffle furnace, the coil existed in contact with the products of thermal dissociation for a longer period which led to erosion of the nichrome coil. The thermal (temperature) regime of removing the products of thermal dissociation of GKZh-94 requires additional work although it is already clear that one can recommend active heating of the components that are not hermetically sealed before using them.

The interaction processes occuring in the system 'periclase obtained by IMCC – Nichrome coil' were studied on the periclase specimens drawn from the already used components according to the methods of x-ray diffraction, spectral analysis, and petrographic examination. Periclase was selected from four zones viz., the interior of the coil, the vicinity of the coil, the center of the specimen, and near its jacket. The studies, established that the $NiCr_2O_4$ spinel (observed in the zones of the periclase charge that are directly adjoining the coil) forms the product of aging the coil.

On increasing the service period of the component, the concentration of the chromium-nickel spinel in periclase increases owing to its diffusion into periclase. Besides this, the failed (burnt) specimens also showed nontransparent and dark colored magnetic particles consisting of the oxides of the metals entering the composition of the coil (Fe, Ni, Cr) and, possibly, their solid solutions. Diffusion of these oxides into periclase during the service period of the component leads to the formation of colored periclase grains whose content is maximum in the samples drawn from the neighborhood of the coil. We observed an insignificant number of particles having different colors (grayish green reddish brown) which are also the products of interaction between periclase and the coil; they were identified as the spinels $MgCr_2O_4$ and $MgFe_2O_4$.

CONCLUSION

Comparative accelerated tests were carried out on three batches of periclase obtained by IMCC.

It was established that treating periclase with the GKZh-94 silicon organic liquid does not reduce its resource-characteristics (life). We recommend active heating of the components prior to putting them into service for removing the organic constituent of GKZh-94.

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