Thermal-shock resistance (1300°C – water), heat changes for specimens measuring, mm:

diam. 10, height 12 ...................... > 100
50 x 23 x 10 ......................... 70

The resistivity was determined in accordance with GOST 64332—71. The dielectric properties were studied in a waveguide device using a short-circuiting method at a frequency of $10^{10}$ Hz.

It follows from the above data that the new ceramic has good electrophysical properties and thermal-shock resistance.

CONCLUSIONS

The possibility of significantly improving the thermal-shock resistance of an AlN ceramic has been established. This is done by using compounds of a coarser grain composition and by adding Y$_2$O$_3$ and SiO$_2$ additives.

The physicomechanical, electrophysical, and dielectric properties and the thermal-shock resistance of the newly developed ceramic have been determined. This ceramic can be taken as a promising material for high temperature electrical insulation purposes.

LITERATURE CITED

4. USA Patent, No. 3833389.

GAS CHROMATOGRAPHIC DETERMINATION OF
THE CONCENTRATION OF CARBONATE
IN RESIN-BONDED REFRACTORIES

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Resin-bonded refractories may contain carbonates, and these have not previously been quantitatively determined. The concentration of carbonates in the material can be determined from the amount of carbon dioxide liberated when the specimen is dissolved in a mineral acid [1, 2].

The quantitative determination of the CO$_2$ in rocks is normally carried out using an absorption-weighing method which takes 4 h. The indirect method of determining the concentration of CO$_2$ involving a calcimeter used for the mass analysis of carbonate rocks is also well known. The indirect method gives less accurate (too low) results than the absorption-weighing method.

The concentration of CO$_2$ in a material can also be determined using various physicochemical methods, one of which is the simple, rapid, and highly sensitive method of gas chromatography.

We have determined the CO$_2$ in the carbonates of resin-bonded refractories using gas chromatography in the LKhM-8MD laboratory chromatograph previously used in [3] to determine the concentration of carbon in electrosmelted refractories.

The carbonates were decomposed in a 75 cm$^3$ glass flask with two outlets, with the end of one outlet almost at the bottom of the flask. The flask had a ground glass stopper with a capillary funnel whose end also came almost to the bottom of the flask. In order to speed up the decomposition of the carbonates, the
Fig. 1. Apparatus for the decomposition of carbonates: 1) column containing an absorbent for removing traces of CO₂; 2) glass flask for the decomposition of the carbonates; 3) capillary funnel; 4) absorbing flask; 5) graduated glass tube; 6) reservoir.

Fig. 2. Calibration graph for the determination of the concentration of CO₂ in resin-bonded refractories.

Acid treatment was done after heating to 100°C and with stirring by a magnetic stirrer. In order to extract the CO₂ from the flask, we blew a stream of air from which the CO₂ had been removed, or N₂, or O₂ through the flask. In the present study the decomposition was carried out in a stream of O₂. The apparatus for the decomposition of the carbonates is shown in Fig. 1.

A portion of the experimental material (0.5 g) milled to a grain size 0.063 mm was placed for decomposition on the bottom of the flask containing a magnet and the stopper was inserted. Into the funnel 40 cm³ of dilute (1:1) HCl was poured. The tap on the funnel was closed. CO₂-free O₂ was blown through the flask, thus removing the air from the system and carrying it into the chromatograph. The absence of a CO₂ peak on the chromatography patterns indicated the absence of CO₂ in the system.

The acid was slowly dripped, drop by drop, from the funnel into the flask and the stirring and heating was started. The mixture of gases leaving the apparatus was collected in the graduated tube which was connected to the reservoir filled with a saturated solution of sodium chloride. In front of the graduated tube was an absorbing bottle one-third filled with a solution of 4 g iodine and 12 g sodium iodate in 10 ml of water to absorb any H₂S liberated if sulfides were present in the original material.

One liter of gas was collected in the tube; the tube was agitated and the gas transferred via the reservoir to the chromatograph for analysis.

We obtained a chromatograph under the following conditions: strength of detector current, 140 mA; temperature of column and detector, 35°C; length of column, 3 m; consumption of gas carrier (helium), 45

| Table 1. Results of the Gas Chromatographic Determination of CO₂ in a Standard Specimen (concentration of CO₂, 2.05%) |
|---|---|---|---|---|---|---|
| i | x₁ | t₁ | y₁ | x₂ | t₂ | y₂ |
| 1 | 2.22 | 0.006 | 0.000 | 2.15 | 0.13 | 0.016 |
| 2 | 2.12 | 0.006 | 0.010 | 2.18 | 0.13 | 0.024 |
| 3 | 2.10 | 0.008 | 0.006 | 2.16 | 0.13 | 0.025 |
| 4 | 1.88 | 0.014 | 0.019 | 1.96 | 0.07 | 0.015 |
| 5 | 1.86 | 0.016 | 0.026 | 1.96 | 0.23 | 0.023 |
| 6 | 1.86 | 0.016 | 0.026 | 1.96 | 0.23 | 0.023 |
| 7 | 1.85 | 0.013 | 0.019 | 1.96 | 0.07 | 0.015 |
| 8 | 1.85 | 0.013 | 0.019 | 1.96 | 0.07 | 0.015 |
| 9 | 1.85 | 0.013 | 0.019 | 1.96 | 0.07 | 0.015 |
| 10 | 1.85 | 0.013 | 0.019 | 1.96 | 0.07 | 0.015 |

*The average value x₀ = 2.02; mean-square deviation, S = 0.146 % (abs); confidence interval for a 95% probability, x₀ ± Δx = 2.02 ± 0.07%.