CONCLUSIONS

A connection between the acoustic (propagation of ultrasonic vibrations) and the structural anisotropy of refractories has been established.

Monitoring the anisotropy of the USV propagation velocity can be used to estimate the uniformity of the articles and the extent to which technological factors influence the structure-formation process.

It seemed reasonable to study the connection between the structural anisotropy and the properties of the refractories which determine its behavior during service.

LITERATURE CITED


CHEMICAL PHASE ANALYSIS OF ALUMINOSILICATE REFRACTORIES

A. N. Aizenberg

A method based on the dissolution of the glass phase in HF is used to determine the concentration of glass phase and the total content of crystal phases. Several variants of this method [1-7] are known which differ in the conditions of the acid treatment of the material.

All these methods are unsuitable for analyzing materials which have been fired at 1300°C or below since the mullite formed under such conditions is a fine-crystal material and dissolves significantly in the HF and this leads to rather low results for the glass-phase concentration. The solubility of fine-crystal mullite in HF has already been pointed out in [8-11]. A correction for the mullite solubility was established for the determination of mullite [12] but is valid only for the actual mullite obtained under specific conditions. In a study of the solution of individual components of a porcelain material [13], solution curves were obtained from which it was possible by extrapolation to determine the concentration of individual phases.

The HF treatment is also unsuitable in the determination of the glass phase in materials containing free silica (quartz and cristobalite) because of the significant dissolution of the free silica.

The present study deals with the development of a method for making a chemical-phase analysis of aluminosilicate refractories with a particular concentration of glass phase, mullite, and total of quartz plus cristobalite.

For the study we chose special minerals obtained from various raw materials. The glass was obtained from a mixture of oxides at 1650°C; the mullite was precipitated from Prosymkovka kaolin fired at 1500°C and from clay fired at 1280°C; quartz, from quartz sand; and cristobalite, from quartz sand fired at 1580°C. All the materials were milled and separated into fractions; the glass, quartz, and cristobalite were treated with a mixture of hydrochloric and nitric acids; and the fired kaolin and clay were treated first with HF and then with HCl. The residues were carefully washed by decantation, filtered, then washed many times in the filters, and dried at 120°C.

All-Union Institute of Refractories. Translated from Ogneupory, No. 11, pp. 57-60, November, 1980.
TABLE 1. Chemical Composition of the Purified Minerals, %

<table>
<thead>
<tr>
<th>Mineral</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>TiO₂</th>
<th>Fe₂O₃</th>
<th>CaO</th>
<th>MgO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass from kaolin</td>
<td>75.65</td>
<td>14.67</td>
<td>2.00</td>
<td>0.92</td>
<td>1.03</td>
<td>2.10</td>
</tr>
<tr>
<td>Mullite from kaolin</td>
<td>27.87</td>
<td>70.46</td>
<td>0.56</td>
<td>1.18</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Mullite from clay</td>
<td>27.76</td>
<td>68.46</td>
<td>2.27</td>
<td>1.80</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Quartz</td>
<td>99.63</td>
<td>0.29</td>
<td>0.059</td>
<td>0.10</td>
<td>0.15</td>
<td>0.029</td>
</tr>
<tr>
<td>Cristobalite</td>
<td>99.22</td>
<td>0.16</td>
<td>0.074</td>
<td>0.06</td>
<td>0.14</td>
<td>0.028</td>
</tr>
</tbody>
</table>

*Here and henceforth mass fractions are given.
† Concentration of K₂O, 2.60%; of Na₂O, 0.90%.

The purified materials were then examined under a microscope* (using immersion liquid) and chemically analyzed; where the cleaning was inadequate the specimens were given a repeat treatment. The results of the chemical analysis of the purified materials are given in Table 1.

No crystal phases were observed in the glass when examined in the immersion liquid; single grains of quartz were encountered in the cristobalite; only aggregations of pure mullite were found in the mullite produced from kaolin; and in the clay mullite, aggregations of fine grained mullite.

We used HF and fluosilicic acid as the solvent for the glass. Different variants of the treatment with these acids were tested on pure minerals and on the working samples. The insoluble residues were examined in the immersion liquid; according to the microscopy data, the residues contained no glass phase.

Table 2 gives the results obtained when the materials were treated with the solvents. In the HF treatment or in the treatment with the HF + HCl mixture, the quartz, cristobalite, and fine-grained mullite are dissolved to a significant extent; at the same time, in the treatment with the fluosilicic acid, only the glass phase dissolves and virtually none of the crystal phases.

Separate determination of the quartz and cristobalite concentrations using selective dissolution proved impossible since the chemical properties of these materials are very similar; when treated, the same amounts of quartz and cristobalite were transferred into solution. Therefore, we determined the total concentration of these components. In order to separate the quartz and cristobalite, we used the method described in [14] according to which the material is fused with potassium pyrosulfate and then treated with an alkali. After such treatment, mullite is transferred into solution but the quartz and cristobalite remain almost entirely in the residue; the glass phase dissolves partially (Table 2).

It must be pointed out that the solubility of mullite depends on the regularity of the crystal structure to a greater extent than on the degree of dispersion of the material; mullite separated from kaolin fired at 1500°C (according to the microscopy data, it consists of aggregations of pure mullite) in fractions < 0.063 mm is dissolved only 6.3% while the mullite separated from clay fired at 1280°C (fine crystal) is 41% dissolved even in a 0.15-0.20 mm fraction.

The treatment of 0.15-0.20-mm fractions recommended in the old methods cannot give a correct result since an average sample is not obtained when this fraction is selected for analysis. Errors are also possible because of the incomplete dissolution of the glass phase which occurs inside the coarse grains. It is recommended that for the analysis the material should be milled to a grain size < 0.063.

As an example, we give the results of an analysis of a chamotte specimen from a mass of the following composition: 65% chamotte for buckets; 20% unfired pyrophillite; 15% Sherekhovskii clay. When 0.15-0.20-mm fractions were treated in 20% HF, the results of the determination of the concentration of undissolved residue were rather low and were not very reproducible (32, 36, 39%). When the material in fractions finer than 0.063 mm was treated in H₂SiF₆ in accordance with the proposed method, the amount of undissolved residue was 56.2 and 56.8% (according to the microscopy data, the residue contains no glass phase).

On the basis of these experiments, the following recommendations can be made for determining the concentration of glass phase and undissolved residue in aluminosilicate refractories:

the material to be analyzed should be milled to a grain size < 0.063 mm;

*Microscopy carried out by E. V. Petrova and G. G. Mel'nikova.