Slag resistant and refractory ceramic compositions in MgO–Al₂O₃–SiO₂ system on the base of local raw materials of Uzbekistan

Z A Babakhanova, M Kh Aripova
Department of silicate materials and rare noble metals, Tashkent Chemical-Technological Institute, 32 Navoi street, Tashkent 10011, Uzbekistan

Corresponding author e-mail: zebo.babakhanova@gmail.com

Abstract. Forsterite and spinel-corundum ceramic materials were obtained in the MgO-Al₂O₃-SiO₂ system based on local raw materials of Uzbekistan - talc-magnesite of Zinelbulak deposit, kaolin of Angren deposit and inorganic additives (fused periclase PPE-87, electric fused corundum). The formations of various phases were studied using XRD and SEM-EDS. Study of chemical-mechanical properties of ceramic samples showed that spinel-corundum composition have density from 2520 to 2770 kg/m³, water absorption rate from 10.52 to 11.71%, open porosity from 27 to 31%, apparent density from 2420 to 2750 kg/m³, compressive strength from 100 to 120 MPa, characterized by increased refractoriness (it was 1750 °C for forsterite ceramic materials, for spinel-corundum more than 1800 °C) and slag resistance under metallurgy slags. The synthesis of spinel in optimal compositions occurs at 300 °C lower than the traditional sintering temperature of spinel products, which contributes to significant energy savings in the synthesis of highly refractory spinel-corundum materials.

1. Introduction
The synthesis of refractories based on local raw materials is an urgent task for an effective development of the metallurgical industry of the Republic of Uzbekistan. More than 900 metallurgical enterprises are operating in Uzbekistan, widely using refractory ceramic materials in the processes of casting liquid metals and linings of metallurgical furnaces. For instance, ferrous and non-ferrous metallurgy consumes up to 70 % of the refractory ceramic materials produced around the world [1-2].

There are fireclay and acid-resistant refractory materials produced at several ceramic enterprises of Uzbekistan. Due to the limited availability of suitable raw materials, highly needed high-alumina, magnesite, magnesia-chrome, chrome refractories are not produced in Uzbekistan. In this regard, scientists are currently working intensively on development of methods of enrichment of raw materials and comprehensive study of new types of raw materials [3]. To increase the refractoriness and slag resistance of fireclay refractories, the new compositions of composite materials are being developed, in particular, special attention is paid to the selection of certain binders and special additives [4].

Currently, compositions of new generation refractory materials are obtained on the basis of fine and highly purified oxides [5], nano- and other advanced materials [6-7].

S. Pagliosa et al. [8] have developed refractory products for metallurgical industry based on periclase-alumina-carbon system, as a carbon component nanographite was used to increase the efficiency of the synthesized masses. Compared to natural graphite, the oxidation resistance of
nanographite is 50% higher due to the presence of nanized particles. At the same time, the MgO – Al₂O₃ – SiO₂ system is of considerable interest for obtaining ceramic materials with high refractoriness, heat and slag resistance [9]. The study of the phase diagram of the MgO – Al₂O₃ – SiO₂ system show the presence of such refractory compounds as corundum, periclase, quartz, forsterite (melting temperature 1890 °C), mullite (1850 °C), spinel (2100 °C) [10].

Spinel-corundum, mullite and corundum products, including those with carbon content, are used in metallurgy for preparation of refractory crucibles, melting tanks and furnace lining. The effect of spinel-corundum refractories on the environment is less than that of periclase-chromite refractories, since they do not contain chromium compounds [11-12]. In this regard, spinel corundum products are also widely used in rotary kilns in the production of cement and lime.

In this research work the crystallization regions of forsterite, corundum and spinel in MgO-Al₂O₃-SiO₂ system were chosen in order to obtain ceramic materials with increased refractoriness and slag resistance.

**Forsterite crystallization area.** Forsterite compositions (42 w.% SiO₂) cannot be fired prior to sintering up to very high temperatures, because the liquid phase does not form in a system at temperatures below 1850 °C. In the compositions lying in forsterite-enstatite region, containing mainly forsterite, the liquid is formed at 1557 °C. Since the liquidus curve in this case has a steep slope, the amount of liquid present in the system changes insignificantly with temperature. Therefore, such compositions have a wide sintering range and can be easily sintered. In contrast, in compositions containing mainly enstatite (55, 60, and 65 % SiO₂), even at low temperatures, a large amount of liquid is formed, and its content rapidly increases with increasing of temperature. These compositions have a narrow sintering range.

In the MgO-Al₂O₃-SiO₂ system, MgO, MgSiO₃, MgAl₂O₄ (spinel) and Mg₅Al₆Si₆O₁₈ (cordierite) coexist in equilibrium with forsterite at 1350 °C. Spinel forms a simple binary eutectic system with forsterite, tₑ = 1710 °C. The resulting melt makes it difficult to achieve equilibrium and refractories of the spinel-forsterite composition are practically not obtained. In the forsterite - cordierite system, the lowest eutectic temperature is 1370 °C. Cordierite is not refractory (incongruent decomposition temperature is 1465 °C), but it has a relatively low thermal expansion coefficient of 1.5·10⁻⁵. Thus, cordierite is included in the composition of some ceramic refractories as a binder to increase their heat resistance [13].

**Spinel crystallization area.** In the technology for the production of refractory ceramics, spinel compositions take an important place, while two types of spinels are distinguished: normal spinel AB₂O₄, where A²⁺ ions are located in tetrahedral regions, and B³⁺ ions are located in octahedral regions. This structure was observed for ZnFe₂O₄, CdFe₂O₄, MgAl₂O₄, FeAl₂O₄, CoAl₂O₄, NiAl₂O₄, MnAl₂O₄, and ZnAl₂O₄. In reverse spinels, the A²⁺ ions and half of the B³⁺ ions are located in octahedral regions; the other half of B³⁺ is at tetrahedral sites, B(AB)₂O₄. This is a more common structure and is observed for FeMgFe₂O₄, Fe₃O₄, ZnSnZnO₄, FeNiFe₂O₄, and many other ferrites that are important for obtaining ceramics with magnetic properties [14].

Magnesium aluminates spinel is of great importance as a structural ceramic due to its use in refractories. It provides a combination of the desired physical, chemical and thermal properties at both normal and elevated temperatures. Spinel melts at 2135 °C, exhibits high resistance to most acids and alkalis and has low electrical losses. Due to these properties, spinel and ceramics with spinel structure have a wide range of applications in the chemical, optical, metallurgical and electrical industries. They are used as refractory lining in steel furnaces, transition and sintering zones of cement rotary kilns [15], glass furnace regenerators [16], side walls and bottom of steel ladles in glass furnaces and melting tanks.

Well known methods for the synthesis of spinel MgO·Al₂O₃ are the traditional solid-phase reaction (SSR) [17], joint processing [18] and the process of gelation. Ping et al [19] reported that the traditional SSR method is the most used in spinel preparation. However, it has several disadvantages such as longer processing times, several calcination steps, the need for very high sintering temperatures, accompanied by uneven and abnormal grain growth, and residual porosity.
Periclase and spinel form a certain range of solid solutions; spinel forms with Al₂O₃ an extensive region of spinel solid solutions, which reaches a maximum at 85 wt. % Al₂O₃ at 1860 °C, and a more limited region of MgO solid solution in spinel, reaching 62 wt.% Al₂O₃ at 1995 °C. There is also a solid solution of Al₂O₃ in periclase at elevated temperatures. On the other hand, in the corundum field for MgO, the solid solution field is not shown, since the solubility of MgO in corundum is no more than a few hundred parts per million.

Mixtures of magnesia spinel with periclase or corundum are used for production of highly refractory spinel, spinel-periclase and spinel-corundum materials with a melting point of at least 1925 °C [20]. The tendency of spinels to form solid solutions is a positive factor due to ability of flux oxides (Fe₂O₃, K₂O, Na₂O), common in the composition of local raw materials to form solid substitution solutions. As a result, the negative effect of flux oxides on the physical-chemical characteristics of refractory materials could be significantly reduced.

Magnesia-alumina spinel was synthesized by Krasny B. L. [21], using a finely dispersed mixture as an alumina-containing component, in wt. %: talc - 5-20, electrocorundum - 80-95, as a binder - lignosulfonate. The firing of products was carried out at a temperature of 1600 ± 50 °C with isothermal holding for the time required for the formation of an equilibrium amount of magnesium-alumina spinel. However, the material obtained was characterized by low compressive strength (45-50 MPa). This disadvantage is due to the fact that the products were manufactured in a one-stage method, in which there is a linear and volumetric expansion of the material due to the spinel reaction. Authors reported that in this way it is impossible to obtain products based on an alumina-magnesia spinel with zero open porosity, density close to theoretical, and a high level of strength.

The objective of this research work was development of high refractory and slag resistant ceramics with forsterite and spinel-corundum structure based on the MgO-Al₂O₃-SiO₂ system using raw materials of Uzbekistan.

2. Research methods

For the synthesis of slag-resistant refractory materials in the MgO-Al₂O₃-SiO₂ system, regions of crystallization of forsterite, spinel, and corundum were selected. Kaolin of Angren Region and talc-magnesite of Zinelbulak deposit were used as local raw materials of Uzbekistan.

The compositions of raw materials and calculated ceramic masses were reduced to a three-component system to ensure the mathematical planning of the properties of refractory compositions. Analysis of the regions in state diagram of MgO-Al₂O₃-SiO₂ make it possible to determine the main crystalline phases and the amount of the liquid phase at the refractory operating temperature.

To identify crystalline phases in the synthesized materials, a Bruker AXS D8 Advance diffractometer, Bruker, Germany was used (Cu-Kα - cathode, step - 0.05, shooting speed 2 sec). Identification of crystalline phases was accomplished by means of a Match! program package (Crystal Impact GbR, Bonn, Germany).

The structure of materials was studied by scanning electron microscopy on an electron microscope (Carl Zeiss, Germany) with an energy-dispersive elemental analyzer (Oxford Instruments, Great Britain).

An oxidation level and thermal stability of graphite-containing ceramic materials was determined by heat treatment of a sample at a temperature of 1000-1500 °C in an oxidizing environment, sawing along the axis with measurement of the decarbonized zone. For a qualitative assessment of the slag resistance of ceramic compositions in relation to a specific type of slag, a static method for assessing slag resistance at 1500-1550 °C was chosen. Slag corrosion was evaluated by the depth and area of the refractory corrosion. This method also takes into account the influence of the porosity of the product on its slag resistance. Additionally, the chemistry of the interaction of slag with refractories was studied by electron microscopy to detect new phases in the material.

Refractoriness of materials was determined by the method of pyrometric cones in conditions of trade production company “Ogneupor” (Tashkent city). Standard methods were used according to GOST 2409-2014, ASTM C20-00, C356-03, C773-88 to determine the apparent porosity, water
absorption, specific gravity, density, linear shrinkage and mechanical compressive strength of ceramic samples.

Analysis of the given compositions obtained on the basis of two main raw materials - kaolin AKF-78 of the Angren deposit and talc-magnesite from the Zinelbulak deposits showed that all compositions (compositions No. 40-49 in figure 1) are situated on the state diagram of the MgO-Al₂O₃-SiO₂ system in the region of the initial crystallization of non-refractory corderite and sapphirine.

In this regard, for synthesis of refractory and slag-resistant ceramic materials in the MgO-Al₂O₃-SiO₂ system, the compositions were calculated in crystallization region of forsterite (compositions No. 51-58 in figure 1), as well as in crystallization area of corundum and spinel (No. 70-79 in figure 1).

![Figure 1. Arrangement of calculated compositions on MgO-Al₂O₃-SiO₂ system state diagram.](image-url)

To obtain refractories of forsterite composition (No. 51-58), enriched talc-magnesite of the Zinelbulak deposit (Uzbekistan), AKF-78 kaolin (Uzbekistan) and fused periclase of the PPE-87 grade (Russian Federation) were used as raw materials. The samples were fired in a laboratory furnace with silit rods at 1450 °C for 2 hours.

To obtain high-alumina compositions, area of interest were found in the crystallization field of MgAl₂O₄ spinel with a melting point of 2105 °C. Ceramic masses were synthesized with an electrocorundum content (Kazogneupor) from 90 to 70 wt.%, Zinelbulak talc-magnesite from 5 to 30 wt. % and enriched graphite concentrate of Zakhechakhona (Uzbekistan) from 5 to 10 wt. % (compositions No. 70-82).

The chemical and mineralogical composition of the initial local raw materials, as well as methods of enrichment of local graphite and the high-temperature processes of phase formation were studied earlier and are presented in our previous works [22]. The samples were fired in a silit furnace at 1500-1520 °C for 2 hours.

3. Results and its discussion

Mineralogical composition of calcined ceramic samples of forsterite and spinel composition have been studied by semi-quantitative XRD- and SEM-EDS analysis.

3.1. Study of phase formation in forsterite-enstatite region of MgO-Al₂O₃-SiO₂ system

The results of studying the mineralogical composition of fired samples of forsterite composition (compositions No. 51-58) by semi-quantitative XRD-analysis (figure 2) showed that the main crystalline phase in composition No. 51-52 is represented by enstatite; in compositions No. 53-54 and No. 57-58 by forsterite; and compositions No. 55-56 contain a significant amount of non-refractory corderite.

In compositions No. 51-53, lying in the forsterite-enstatite region, containing mainly forsterite, the liquid is formed at 1557 °C. In this regard, compositions No. 51-53 are characterized by formation of a
large amount of glass phase. Composition No. 54 is characterized by a predominant amount of forsterite in the crystalline phase; according to the state diagram of the system, the liquidus curve in this case changes slightly with temperature: composition No. 54 has a fairly wide sintering range, is easily sintered and promising composition for production. Physical and mechanical properties of ceramic samples are given in Table 1.

Table 1. Physical and mechanical properties of ceramic samples synthesized in forsterite-enstatite region.

| No | Open porosity, % | Water adsorption, % | Specific gravity, kg/m³ | Main crystalline phases (according to XRD analysis) |
|----|-----------------|---------------------|-------------------------|-----------------------------------------------|
| 51 | 5.35            | 3.34                | 2920                    | Enstatite, forsterite, periclase               |
| 52 | 5.57            | 3.87                | 2900                    | Enstatite, forsterite, periclase               |
| 53 | 8.80            | 5.88                | 2850                    | Forsterite, enstatite, periclase               |
| 54 | 9.20            | 6.05                | 2840                    | Forsterite, periclase, enstatite, spinel       |
| 55 | 3.21            | 2.35                | 2950                    | Forsterite, cordierite, periclase, spinel      |
| 56 | 3.52            | 2.78                | 2920                    | Forsterite, cordierite, periclase, spinel      |
| 57 | 8.51            | 6.45                | 2830                    | Forsterite, periclase, cordierite, spinel      |
| 58 | 8.92            | 7.20                | 2800                    | Forsterite, enstatite, periclase               |

Analysis of the properties, structure and phase composition of the ceramic samples synthesized in forsterite-enstatite region showed that the optimal composition for obtaining refractory materials is composition No. 54, the phase composition is represented by forsterite - 87, periclase - 11 and spinel - 2% (figure 2). Refractoriness of the optimal composition was 1750 °C, which refers these compositions to high refractory materials, the temperature of the onset of deformation under load (0.02 kN/cm) - 1600 °C; heat resistance - 7 thermal cycles; residual change in dimensions when heated at 1350 °C - 0.6 cm; porosity - 7 %. A technological scheme and technological regulations for the production of refractory forsterite products were developed, synthesized forsterite refractory materials recommended to use at heating units, regenerators, open-hearth furnaces, etc.

3.2. Phase formation in the area of spinel crystallization

Study showed that in compositions No. 70-82 calcined at 1000 °C the microstructure of materials is loose and contains forsterite, enstatite and corundum phases; at 1200 °C in MgO-Al₂O₃-SiO₂ system in the presence of a small amount of impurities, fusible compounds is formed and crystallization of spinel accelerated. According to the results of semi-quantitative XRD-analysis, the mineralogical composition of materials synthesized at 1500-1520 °C is represented by corundum Al₂O₃ - from 60 to 70% and spinel MgAl₂O₄ - from 30 to 40% (figure 3). The parameters of the crystal cell of the cubic spinel are a = 0.8078 nm (8.0775 Å), which is in good agreement with the reference standard No. 00-075-1796 (Match, a = 8.078 Å, space group Fd3m).
Figure 2. Mineralogical composition of ceramic samples No. 51-58 synthesized in forsterite-enstatite region at 1450 °C.

Figure 3. Diffraction pattern of the optimal composition of ceramic sample No. 77 synthesized in spinel crystallization region at 1500 °C.

The crystalline phase in calcined at 1500 °C ceramic composition No. 77 presented by corundum (65 wt.%) and spinel (35 wt.%) (figure 3), the binder is represented by a high-alumina-silicate phase. The pores in the ceramic sample are generally isolated, rounded, ranging in size from 10 to 50 µm. The formation of a spinel phase in compositions No. 75-78 leads to an increase in the slag resistance of refractory compositions, in contrast to graphite-chamotte refractories. At the same time, the synthesis of spinel in compositions No. 75-78 occurs at much lower temperatures (at 1500 °C) than the traditional sintering temperature of spinel products (1750 °C) [23].

Formation of binding in ceramic materials requires much more reactions than the crystalline phase formation processes. The binder phase in spinel-corundum refractories is formed during the decomposition of talc and magnesite, followed by the interaction of periclase with the silicate phase. As a result, a magnesium-containing alumina silicate phase is formed (spectrum 6 in figure 4), which binds individual crystals of corundum and spinel increasing mechanical strength of the samples. A binder in refractories is the most important factor that determines the processing methods and properties of the synthesized material; it is represented in the material by round, irregularly shaped grains with a size of <25 µm (figure 4).

Figure 4. Electron microscopic image and EDS of a ceramic sample No. 77 synthesized at 1500 °C: formation of spinel phase.

The main compaction of materials is performed at the stage of pressing; therefore, when synthesizing refractories, it is extremely important to obtain materials with a higher density and lower values of water absorption and porosity. An increase in the density of the material contributes to an increase in the chemical and thermal resistance of the refractory material. In this regard, when determining the optimal compositions, the main attention was paid to the analysis of the density and porosity of ceramic samples.

The study of the physical and mechanical properties of the synthesized materials showed that ceramic materials of spinel-corundum composition No. 70-82 have a density from 2520 to 2770 kg/m³, water absorption from 10.52 to 11.71%, an apparent specific gravity from 2420 to 2750. The compressive strength of the samples was from 100 to 120 MPa. The temperature of the beginning of deformation under load (0.02 kN / cm) - more than 1800 °C; heat resistance - 9 thermal cycles; residual change in dimensions when heated at 1350 °C - 0.2 cm. The synthesized materials are high refractory with a melting point of more than 1800 °C. Compared with the ceramic materials given in [21], the synthesized spinel-corundum materials were distinguished by a higher mechanical strength: 100-120 MPa versus 45-50 MPa. This indicates the possibility of using the synthesized materials for the production of high refractory ceramic products with the required mechanical strength.
Determination of slag resistance of materials in relation to metallurgical slag of the Almalyk Mining and Metallurgical Complex (AMMK) by the static method for 2 hours at 1550 °C showed that compositions No. 75-77 are demonstrated a higher resistance to molten metals.

Chemical composition of metallurgical slags from copper-smelting production of AMMK, %: Cu - 0.95; Pb - 0.2; Zn - 0.77; S - 1.19; Fe - 35.15; SiO_2 - 34.24 [24-25]. To determine the zones of penetration of metallurgical slag, studies were carried out using an electron microscope on the distribution of elements over the surface of a ceramic sample. The results are shown in figure 5.

![Figure 5](image)

**Figure 5.** Electron microscopic image and spectral analysis of sample No. 77 after slag resistance test: (a) general view, (b) – spinel composition with Si and Fe content.

The main crystalline phases in a ceramic specimen after testing for slag resistance are represented by crystals of corundum (crystal sizes 10-25 mk), fine-crystalline spinel phase (crystals sizes 1-5 mk) (figure 5). The amount of alumina silicate binder increased, and penetration of metallurgical slag occurs in small amounts along the phase boundary and pores of the sample. The area of penetration of slag in specimens No. 75-77 was 0.25-0.3 cm, under the same conditions the depth of penetration of slag in fireclay refractories was 1.5-2.2 cm. To determine the oxidation of graphite-containing ceramic materials, fired specimens were sawn and the inner sections were examined microscopically to determine the decarbonized area. Studies have shown that the optimal compositions No. 75-77 with a carbon content of 7% are distinguished by the best indicators of resistance to oxidation (the smallest thickness of the decarbonized layer).

4. Conclusion
In the MgO-Al_2O_3-SiO_2 system based on local raw materials and inorganic additives, refractory materials of forsterite (compositions No. 51-58) and spinel-corundum composition (compositions No. 70-79) with high refactoriness (1750-1800 °C) and slag resistance in relation to metallurgical slag were synthesized. The formation of a spinel bond in corundum refractories at a temperature of 1500 °C, which is lower than the traditional sintering temperature of spinel products (1750 °C), has been established. This contributes to significant energy savings in the synthesis of highly refractory materials of spinel-corundum composition. A higher resistance to metallurgical slag of refractories of spinel-corundum composition, which is also distinguished by higher refactoriness (1800 °C), has been established. In this connection, spinel-corundum compositions No. 75-77 with a carbon content of 7% can be recommended for use in crucial processes of metallurgical production, for operation in direct contact with metallurgical slag.

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