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CERAMIC PROTECTIVE COATINGS FOR CORDIERITE-MULLITE REFRACTORY MATERIALS

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Abstract. The issue of cordierite-mullite refractories protection from the influence of aggressive factors is considered. The interaction between the components of protective coatings has been studied. It has been investigated that in the systems based on poly(methylphenylsiloxane) filled with magnesium oxide, alumina and quartz sand, the synthesis of cordierite (2MgO·2Al₂O₃·5SiO₂), mullite (3Al₂O₃·2SiO₂) or magnesium aluminate spinel (MgO·Al₂O₃) is possible. The basic composition of the protective coating, which can be recommended for the protection of cordierite-mullite refractory, is proposed.

Keywords: cordierite-mullite refractory materials, high temperature corrosion, protective coatings.

1. Introduction

Taking into account the short life of cordierite-mullite refractory materials, associated with destructive processes caused by aggressive factors in their surface layers [1], it is advisable to protect the surface of refractories by coating. The use of known coating compositions for the protection of cordierite-mullite refractories is unacceptable due to the significant difference between the thermal coefficients of linear expansion (TCLE) of these coatings and the substrate ((6–10)·10⁻⁶ K⁻¹) [2-6]. The coating for the protection of cordierite-mullite refractories should have a composition that will provide a TCLE value close to that of cordierite ceramics, *i.e.*, within ((2.0–3.5)·10⁻⁶ K⁻¹).

Lithium aluminosilicates have a low value of TCLE (0.5–2.0)·10⁻⁶ K⁻¹, but the most acceptable crystalline phase in the composition of such coatings is magnesium aluminosilicate – cordierite. From the practice and theoretical studies of coatings for high-temperature protection it is known [7-15] that the maximum protective effect and minimum energy consumption can be achieved by forming the required phase component in the coating

layer during coatings pre-burning or operation. Therefore, the main attention in the studies was paid to the synthesis of cordierite α -phase directly in the coating.

The most technological binder materials for creating protective compositions are organosilicon polymer varnishes, which allow the use of conventional paint technology for the preparation and application of coatings. When burning, these materials are converted into a ceramic material [16, 17]. The advantage of organosilicon varnishes over other binders is the ability to form reactive amorphous silica during thermooxidative degradation. Silica is capable to actively react with other components of the coating and form new crystalline phases [18, 19]. By directly adjusting the original composition, it is possible to obtain a material with predetermined properties.

There are no studies on coatings with MgO that would ensure the formation of α -cordierite in the coating compositions, so it is important to study the peculiarities of the interaction in the MgO-Al₂O₃-SiO₂ system with the participation of the organosilicon component.

2. Experimental

2.1. Materials

The following materials were used to obtain ceramic protective coatings: magnesium oxide of PA grade (Sigma Aldrich, Switzerland), SALOX M-MSG (ALO-DN-10) alumina with α -Al $_2$ O $_3$ content of 95 wt % (SILKEM HUNGARY Kft., Hungary) and VS-030-MO quartz sand used in the glass industry with SiO $_2$ content of 98.5 % (JSC "United Mining and Chemical Company", Ukraine). A KO-08 organosilicon heat-resistant varnish (Silicon Polymer Factory, Ukraine) was used as a silicone component. It is a solution of poly(methylphenylsiloxane) (methyl phenyl polysiloxane, PMPS) resin in toluene with a concentration of 30 %.

2.2. Investigation Methods

Differential thermal analysis of the samples of oxide compositions and coatings was performed using a

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Q-1500 derivatograph (Paulik, Paulik, Erdey system) in the air with a temperature rate of 10 K/min. Burnt alumina of PA grade was used as a standard. Radiographs were recorded in the range of 2θ equal to $10-70^{\circ}$, with a scanning step of 0.02°. Identification of crystalline phases was performed by comparing the interplanar distances and intensities of the diffractograms maxima with the data of the ASTM file [20]. Electron microscopic investigations were performed using a scanning electron microscope REM-106I (resolution 4 nm, magnification range from 15 to 300,000 times, measuring range $0.2-5000 \mu m$). The thermal coefficient of linear expansion (TCLE) was using a quartz dilatometer DKV-5A. determined Coagulation thixotropic structure formation organosilicon mixtures was evaluated at a temperature of 293±0.5 K by measuring the relative viscosity using a viscometer VZ-4 with a nozzle diameter of 4 mm in the speed range of 20–24 s⁻¹.

3. Results and Discussion

The mixture 1 (Fig. 1) was chosen for the study of the influence of silicate component nature on the physicochemical processes during the heating in the system MgO–Al₂O₃–SiO₂. The mixture composed of MgO (13.7 wt %); Al₂O₃ (34.9 wt %) and SiO₂ (51.4 wt %) was obtained using a mechanical mixing of magnesium oxide, alumina and quartz sand. For the similar mixture 1k a combined silicate component is represented by a quartz sand in the amount of 34.13 wt %, and amorphous SiO₂ component due to the introduction of KO-08 varnish (25 wt %). This organosilicon varnish provides optimal rheological properties of the composition, namely the viscosity within 20–24 s determined by the VZ-4 viscometer.

The starting magnesium oxide, alumina and quartz sand were pre-calcined at a temperature of 1123 K, followed by a dry grinding of the components in a ball mill until complete passage through a sieve No.0063. The samples-cylinders with a diameter of 10 mm were formed from the mixture using a hydraulic press (compacting pressure 20 MPa). The pressed samples were burnt in an electric furnace at 1573, 1623 and 1673 K; the heating rate was 5 K/min and isothermal keeping at a maximum temperature was 60 min. The mixture with organosilicon was prepared by grinding the components in a ball mill, followed by transferring the samples to corundum crucibles and burnt according to the above procedure.

The DTA curve of the model mixture 1 (Fig. 2) is characterized by the presence of an endothermic effect at 413 K, which is associated with the release of adsorbed moisture from the components and the corresponding weight loss (2.9 %). The endothermic effect at 663 K is

caused by the release of chemically bound moisture from $Al_2O_3 \cdot 3H_2O=2Al(OH)_3$ (hydrargillite) and is characterized by a weight loss of 1.9 %; the endothermic effect at 763 K is associated with the release of chemically bound moisture from $Al_2O_3 \cdot H_2O=2AlO(OH)$ (boehmite), which causes a weight loss up to 1 %. A significant endothermic effect at 1523 K is caused by the intensive melting of the components. The exoeffect at a temperature of 1623 K indicates the proceeding of crystal formation of α -cordierite in the studied model system, which is confirmed by X-ray phase analysis (Fig. 4b).

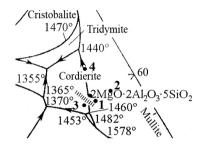


Fig. 1. The area of the studied compounds on the state diagram of the MgO–Al₂O₃–SiO₂ system

Thermal analysis of the model mixture 1k, with 22.37 % of organosilicon varnish shows the presence of endothermic effects at 383, 653, 833, 1523 and 1583 K (Fig. 3). The first endothermic effect at 383 K is due to the release of solvent from the composition, and the next two endothermic effects, as well as the presence of corresponding weight loss (12.4%, and the total weight loss of the sample is 15.2 %) on the TG and DTG curves at 653 and 833 K, indicate the thermal destruction of poly(methylphenylsiloxane). The endoeffect at 653 K is due to the detachment of methyl radicals, and the endothermic effect with a maximum at 833 K is caused by detachment of phenyl radicals from poly(methylphenylsiloxane) chain.

A characteristic feature of thermal analysis of materials containing organosilicon polymers is a pronounced exothermic effect in the temperature range of 383-973 K. The exothermic effect for the mixture 1k is due to the gradual oxidation of the poly(methylphenylsiloxane). According to [21], the of thermooxidative process destruction of polyorganosiloxanes occurs in the temperature range of 473–1073 K, after which they are completely converted into inorganic compounds. The exothermic effect at a temperature of 1083 K is explained by the beginning of nucleation of magnesium aluminate spinel crystals, and the exothermic effect in the temperature range of 1183-1293 K is associated with the beginning of crystal formation of the mullite phase. The stepwise nature of the DTA curve in the temperature range of 1293–1583 K is caused by recrystallization of spinel and mullite phases into cordierite α -phase and secondary mullite. One can see from the DTA curves (Figs. 2 and 3) that the amorphous silicate component obtained by the introduction of organosilicon varnish KO-08 significantly intensifies the

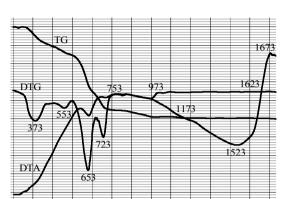


Fig. 2. Differential thermal analysis of the model stoichiometric mixture of cordierite composition (mixture 1)

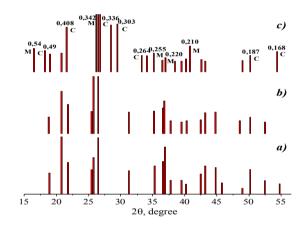


Fig. 4. Stick diagram of the model mixture of stoichiometric cordierite composition after burning at 1573 K (a), 1623 K (b) and 1673 K (c). C– cordierite; M –mullite

The burning of the mixture 1 at 1623 K (Fig. 4b) leads to the disappearance of α -quartz reflexes on the pattern, to the increase in the α -cordierite lines intensity and corresponding decrease in the intensity of mullite and magnesium aluminate spinel maxima. A significant increase in the intensity of maxima with d/n = 0.467 nm and 0.344 nm should be noted, which can be associated with the overlapping of reflexes of α -cordierite, magnesium aluminate spinel and mullite. The data at a temperature of 1673 K indicate the appearance of more intense double maxima of α -cordierite with d/n = 0.313 and 0.303 nm in the 28.5–30 range of 20 angles. This means the maximum crystal formation in the system under study. Crystallization of α -cordierite in the oxide mixture is accompanied by a decrease in the amount of

interaction between components in the model system MgO–Al₂O₃–SiO₂, as evidenced by the shift of crystal formation thermal effects toward the region of low temperatures (Fig. 3). This phenomenon is confirmed by X-ray phase analysis (Fig. 5).

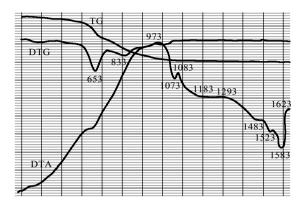


Fig. 3. Differential thermal analysis of the mixture with organosilicon (mixture 1k)

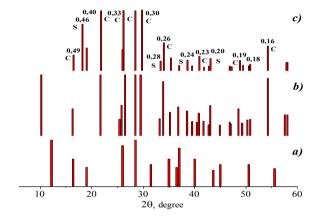


Fig. 5. Stick diagram of the organosilicon mixture 1k after burning at 1573 K (a), 1623 K (b) and 1673 K (c).

C- cordierite; S – spinel

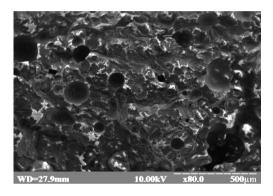
magnesium aluminate spinel, which is an intermediate compound during the formation of α -cordierite (Fig. 4c). Thus, the heating of oxide mixtures of stoichiometric composition to 1573–1673 K leads to the formation of crystalline phases: cordierite, mullite and spinel. The phase composition of the mixture $1\mathbf{k}$, burnt at a temperature of 1573 K (Fig. 5a) is represented by the maxima of α -cordierite and mullite. The maxima of magnesium aluminate spinel are also observed. Increasing the temperature to 1623 K leads to a decrease in the intensity of mullite and magnesium aluminate spinel maxima and the appearance of α -cordierite maximum with simultaneous disappearance of α -cordierite reflex, which indicates the material recrystallization with the formation of stable cordierite α -phase (Fig. 5b). The phase

composition of the mixture 1k with an amorphous silicate component, burnt at a temperature of 1673 K (Fig. 5c), is represented by the reflexes of α -cordierite and magnesium aluminate spinel. Low intensity of mullite reflexes may indicate the participation of this phase in the process of cordierite synthesis, and that mullite serves as an intermediate phase in the process of cordierite formation (Fig. 5c).

The microstructure of the mixture without organosilicon after burning at 1673 K is characterized by a significant porosity (Fig. 6). The introduction of poly(methylphenylsiloxane) into the mixture promotes the formation of a more sintered material; the crystals of α -cordierite and spinel are evenly distributed in the material. The structure of such mixture after burning at 1673 K is characterized by a minimum number of closed pores (Fig. 7).

Comparison of the phase compositions of the samples provides an intensive formation of the cordierite α -phase. So, in the MgO–Al $_2$ O $_3$ –SiO $_2$ system the synthesis of crystalline phases based on magnesium, aluminum and silicon oxides is possible due to a partial replacement of the quartz component with poly(methylphenylsiloxane) varnish KO-08, which forms a reactive, amorphous SiO $_2$ component during the thermodestruction.

Peculiarities of phase formation during heating the mixture 1k are confirmed by the given thermodynamic calculations, which indicate the theoretical possibility of the cordierite formation at first, and then the formation of mullite, spinel, forsterite and enstatite (Table 1).



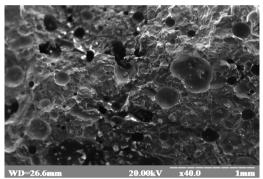
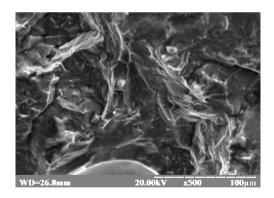


Fig. 6. The microstructure of the model mixture of cordierite composition after burning at 1673 K



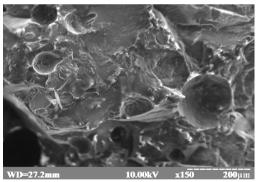


Fig. 7. The microstructure of the mixture with organosilicon after burning at 1673 K

Table 1

Change of isobaric-isothermal potential depending on temperature

Compound	ΔG (kJ/mol) at the temperature							
Compound	1373 K	1473 K	1573 K	1623 K	1673 K	1723 K		
Cordierite	-9170.35	-9174.53	-9178.3	-9180.81	-9182.49	-9185.0		
Mullite	-7075.69	-7192.92	-7218.04	-7232.7	-7243.16	-7259.91		
Spinel	-2418.3	-2426.67	-2434.62	-2439.65	-2443.0	-2447.18		
Forsterite	-2307.76	-2317.39	-2326.6	-2332.05	-2336.23	-2340.42		
Enstatite	-1641.23	-1647.51	-1654.62	-1657.97	-1661.32	-1663.0		

When the sample consisting of magnesium, silicon and aluminum oxides is heated, the formation of cordierite passes through the hexagonal form of cordierite $-\mu$ -cordierite, which turns into rhombic α -cordierite with the increase in temperature to 1573 K. The most intense formation of cordierite occurs in the temperature range of 1573–1673 K [22]. The presence of a high content of magnesium aluminate spinel in the phase composition of the coating is undesirable due to its TCLE index approx. 9.0·10⁻⁶ K⁻¹ [23]. To choose a protective coating with an increased content of α -cordierite, the model mixtures 2, 3 and 4 (Fig. 1) were studied. To increase the modulus of basicity of the material, the content of magnesium oxide was increased in the direction of the field of cordierite stability in the MgO–Al₂O₃–SiO₂ state diagram.

During the coatings preparation, a part of the quartz sand, which was injected with silicon oxide, was replaced by poly(methylphenylsiloxane) varnish KO-08 in the amount of 25 wt %. The composition of the model systems was chosen in such a way that the maximum ratio of components for the synthesis of cordierite α -phase should be provided. For this purpose we investigated the region in the MgO-Al₂O₃-SiO₂ system, which lie at the interface of the cordierite-mullite phases in the stability fields of these crystalline compounds (Fig. 1). Accordingly, the mixture 1k corresponds to the stoichiometric content of cordierite, the mixture 2k corresponds to the oxide composition of cordierite-mullite refractories, and the mixture 3k is characterized by the increased magnesium oxide content and lies in the stability field of cordierite, whereas the mixture 4k is characterized by the increased SiO₂ content. To ensure a more complete interaction between the components in the studied mixtures, the starting materials were subjected to a mechanical grinding in a porcelain ball mill till 5 % residue on a sieve No.0063.

The interaction between the components of the model systems during heating to a temperature of 1623 K and isothermal keeping for 60 min at this temperature was studied by X-ray phase analysis. The mixture 1k (Fig. 8b) is represented by reflexes of α -cordierite, magnesium aluminate spinel and low-intensity reflexes of mullite. The diagram of the mixture 2k is characterized by the presence of α -cordierite and magnesium aluminate spinel maxima (Fig. 8a).

The phase composition of the model mixture 3k, the oxide content of which is in the field of cordierite stability, is represented by more intense reflexes of α -cordierite (Fig. 8d). Studies of the phase composition of the model mixture 4k revealed significant differences in a phase formation compared to previous coatings. On the diagram (Fig. 8c) the reflexes of mullite and α -quartz are observed. The phase composition of this mixture is characterized by a decrease in the intensity of the characteristic maxima of α -cordierite and the complete

absence of magnesium aluminate spinel, but the formation of α -quartz can cause undesirable volume changes in the material structure and a corresponding increase in the thermal expansion coefficient. To characterize more fully the processes occurred during the heating of the systems under study and to determine the technological characteristics of the mixtures with optimal composition, the ratios between the oxides in the ternary system were calculated (Table 2). It has been established that in the systems based on poly(methylphenylsiloxane) filled with magnesium oxide, alumina and quartz sand, the synthesis of α-cordierite (2MgO·2Al₂O₃·5SiO₂), mullite (3Al₂O₃·2SiO₂) or magnesium aluminate spinel (MgO·Al₂O₃) is possible with only definite components ratio. Thus, the maximum content of the cordierite phase corresponds to the composition with the following ratios: Al₂O₃/MgO < 2.0, $SiO_2/MgO < 3.0$, and $(Al_2O_3 + SiO_2)/MgO < 5.0$.

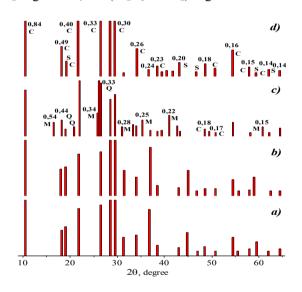


Fig. 8. Stick diagram of the phase composition of the mixtures 2k (a), 1k (b), 4k (c) and 3k (d) after burning at 1623 K. C- cordierite; M -mullite; S - spinel; Q - α -quartz

The highest content of magnesium aluminate spinel corresponds to the following ratios: $(Al_2O_3 + SiO_2)/MgO =$ = 6.29; Al₂O₃/MgO = 2.54; SiO₂/MgO = 3.75. The mixture 4k deserves attention because simultaneously with the low content of α -cordierite and the complete absence of spinel phase, the highest content of mullite and the presence of residual α-quartz are observed. The oxide for components ratio 4k is the following: $= 5.66; Al_2O_3/MgO =$ (Al₂O₃ + SiO₂)/MgO $SiO_2/MgO = 3.66$. When comparing the oxide and phase compositions of the studied systems, it can be stated that the maximum content of the cordierite α -phase has the model mixture 3k (Table 2). So, it can be considered as an optimal mixture and be recommended for further research for the preparation of protective coatings.

Table 3

Characteristics of the oxide and phase compositions of model systems after burning at 1623 K

	Conditional intensity of crystalline phase reflexes				Ratio (w/w)		
Model mixtures	Cordierite $d \cdot 10^{-9}$, $m = 0.848$	Mullite $d \cdot 10^{-9}$, $m = 0.34$	Spinel $d \cdot 10^{-9}$, $m = 0.468$	Quartz $d\cdot 10^{-9}$, $m = 0.33$	(Al ₂ O ₃ +SiO ₂)/MgO	Al ₂ O ₃ /MgO	SiO ₂ /MgO
1k	0.9	_	0.6	_	6.29	2.54	3.75
2k	0.8	0.3	0.8	_	11.5	4.75	6.75
3k	1.0.	0.1	0.4	_	4.88	1.94	2.94
4k	0.7	0.5	_	1.0	5.66	2.0	3.66

Note: d is an interplanar distance of characteristic diffraction maxima of crystalline phases of the greatest intensity, m⁻⁹(nm)

Technological and operational properties of coatings after burning at 1623 K

Model mixtures	Synthesis temperature, K	Viscosity, s	TCLE·10 ⁻⁶ , K ⁻¹
1к	1623	20–24	3.56
2к	1623	20–24	4.26
3к	1623	20–24	3.37
4к	1623	20–24	5.49

Protective coatings were prepared by mechanical activation of fillers in the organosilicon medium in a ball mill, which provides grafting of the polymer to the filler particles, and creates the conditions for obtaining sedimentation-resistant compositions [24]. In the process of forming the material, the strong bonds, including chemical ones, are formed between the mixture components, resulting in the formation of a cross-linked spatial structure. During dispersion the condensation reactions between oxide and polymer components also occur due to the surface OH groups and silanol groups of polyorganosiloxane [25]. The process of physical adsorption, destruction of the crystal chemical lattice of the filler, chemical grafting of the polymer and its diffusion into the depth of the oxide contributes to more complete interaction of oxides with organosilicon binders in the process of mechanical treatment. The weight of the polymer grafted on the oxide surface may be 4.2-5.9 wt % [26]. The interaction of polyorganosiloxanes with oxides and silicates during dispersion significantly affects not only the formation of protective coatings structure, but also their technological and operational properties (Table 3). The compositions of the protective coatings were burnt in an electric furnace with a heating rate of 5 K/min to a temperature of 1623 K. The properties of the obtained coatings (Table 3) are closely correlated with the phase composition of individual experimental mixtures.

The highest value of TCLE (5.49·10⁻⁶ K⁻¹) for the mixture $4\mathbf{k}$ is provided by the content of α -quartz and

complete absence of the spinel phase. The latter indicates the inadmissibility of preserving residual or newly formed α -quartz during burning of coatings. A significant difference in the coefficient of expansion of α -quartz and α -cordierite leads to the formation of a microcracked structure, which increases the permeability of the coating. An increase in the magnesium oxide content of the coating leads to an increase in the content of α -cordierite in the burnt coating due to the binding of α -quartz. The lowest value of TCLE (3.37·10⁻⁶ K⁻¹) is observed for the mixture 3k, which is characterized by the highest content of α -cordierite.

Thus, the mixture 3k (MgO 17.0 wt %; Al₂O₃ 33.0 wt %; SiO₂ 50 wt %) was found to be the optimal coating. To ensure the specified oxide composition the following charge composition of the coating was chosen (wt %): KO-08 22.37; magnesium oxide 15.21; alumina 29.53 and quartz sand 32.88. This composition can be recommended as a protective coating for cordierite-mullite refractories.

4. Conclusions

The regularities of phase formation during heating of model systems in the field of cordier formation have been investigated. The influence of the silicate component of organosilicon varnish KO-08 on the phase formation during heating has been studied. The effect of the oxide composition on the ratio of mullite, cordierite and spinel phases which were formed during heating has been

investigated. The boundary relations between oxides were determined, which provided intensification of cordierite formation and inhibition of spinel phase crystallization, achieved at $Al_2O_3/MgO < 2.0$, $SiO_2/MgO < 3.0$, and $(Al_2O_3 + SiO_2)/MgO < 5.0$.

The quantitative phase composition of the experimental coatings was estimated relative to the conditional intensity of the crystalline phase diffraction maxima after burning. Technological parameters for the production of organosilicon mixtures of paint type (viscosity, density, residue on the sieve) were proposed. The highest value of TCLE ($5.49 \cdot 10^{-6} \, \text{K}^{-1}$) was observed for the mixture containing α -quartz, and the lowest one ($3.37 \cdot 10^{-6} \, \text{K}^{-1}$) – for the mixture with the highest content of α -cordierite. The charge composition of the optimum coating was (wt %): KO-08 22.37; magnesium oxide 15.21; alumina 29.53 and quartz sand 32.88.

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КЕРАМІЧНІ ЗАХИСНІ ПОКРИТТЯ ДЛЯ КОРДІЄРИТОМУЛІТОВИХ ВОГНЕТРИВІВ

Анотація. Розглянуто питання захисту кордієритомулітових вогнетривів від впливу агресивних чинників. Досліджено процеси взаємодії між компонентами захисних покриттів. Досліджено, що в системах на основі поліметилфенілсилоксану, наповненого магнію оксидом, глиноземом та піском кварцовим, можливий синтез кордієриту $(2MgO\cdot 2Al_2O_3\cdot 5SiO_2)$, муліту $(3Al_2O_3\cdot 2SiO_2)$ чи алюмомагнезіальної шпінелі $(MgO\cdot Al_2O_3)$ лише за певного відношення оксидних складових. Запропоновано базовий склад захисного покриття, яке можна рекомендувати для захисту кордієритомулітових вогнетривів.

Ключові слова: кордієритомулітові вогнетриви, високотемпературна корозія, захисні покриття.