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GLASS-CERAMIC BINDER OF CORDIERITE COMPOSITION FOR LOW-TEMPERATURE SINTERING OF HIGH-STRENGTH CERAMIC MATERIALS BASED ON OXYGEN-FREE SILICON COMPOUNDS

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The mechanical properties of composite ceramic materials obtained based on oxygen-free silicon compounds are largely determined by the properties of the glass binder. This paper presents the results of studies aimed at determining the most fusible glass in the pseudo-binary system $2MgO{\cdot}2Al_2O_3{\cdot}5SiO_2$—$2MnO{\cdot}2Al_2O_3{\cdot}5SiO_2$ with a high tendency to crystallize as a glass-ceramic binder for low-temperature sintering of high-strength ceramic materials based on oxygen-free silicon compounds. The crystallization tendency of the experimental melts decreases with an increase in in the content of manganese cordierite, as confirmed by X-ray and infrared spectroscopic studies. Based on experimental studies, a melting diagram was constructed, which was used to determine the ratio between magnesium cordierite and manganese cordierite (50:50 wt.%), ensuring a minimum melt temperature of 1275^{0}C. The melting point of the glass of the specified composition is 1450^{0}C. The synthesized glass is characterized by a softening point of 800^{0}C and crystallizes intensively at 1030^{0}C. The The thermal coefficient of linear expansion of the crystallized glass samples is $20.8{\cdot}10^{-7}$ $^0C^{-1}$. X-ray diffraction and electron microscopic studies have shown that the developed glass is almost completely crystallized during heat treatment for 2 hours, forming a cordierite solid solution $2(Mg,Mn)O{\cdot}2Al_2O_3{\cdot}5SiO_2$. The size of the cordierite phase crystals ranges from 0.5 to 3.0 μm. Due to its fusibility and high crystallization tendency, the developed glass, can be proposed as a promising glass-ceramic binder for the production of high-strength ceramic materials (wear and impact resistant) based on SiC and Si_3N_4 with reduced sintering temperatures.

Keywords: glass binder, magnesium cordierite, manganese cordierite, phase diagram, crystallization, phase composition, microstructure.

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Introduction

Among the high-strength materials based on oxygen-free silicon compounds (SiC and Si_3N_4), glass-ceramic dispersion-strengthened compositions occupy a special niche among low-cost wear-resistant materials. This is because classical ceramic technology is used for their manufacture, and the sintering temperature does not exceed 1250^{0}C. The main disadvantage of such materials is their relatively low compressive strength (no more than 700 MPa), limited primarily by the properties of the glass binder that forms the matrix of the glass-ceramic composite [1,2].

One of the most obvious solutions to increase the strength of glass-ceramic composites is to use a glass binder that is prone to crystallization and capable of forming a synthesized structure as a matrix. Glass-ceramic materials have significantly higher mechanical strength than basic glasses [3,4]. At the same time, an important requirement is compatibility in terms of the temperature coefficients of linear expansion (TLCE) of the filler (SiC, Si_3N_4), glass crystallization products, and the residual glass phase [5,6]. This excludes from consideration most glass-forming systems containing alkali metal oxides. In fact,

aluminosilicate systems with the participation of alkaline earth metal oxides remain promising for the synthesis of glass binders that crystallize efficiently, in particular, the $MgO-Al_2O_3-SiO_2$ (MAS) system, which produces glass crystalline materials of cordierite composition [7–9]. An obvious disadvantage of glasses of cordierite composition of the MAS system is their high refractoriness [10–12]. According to the phase diagram of the MAS system, eutectic compositions are quite far removed from the composition of stoichiometric cordierite [13]. This leads to the fact that in the process of heat treatment of glass ceramics, either a significant amount of glass phase will be remained or accompanying crystalline phases will be released. Both cases can lead to weakening material structure and decreasing mechanical strength. In our opinion, glass binders prone to the most complete crystallization with the formation of crystalline phases that are close to the TLCE values of crystalline fillers are more promising. It is possible to reduce the melting point and viscosity of magnesium-aluminum-silicate glass melts of the cordierite composition system by introducing manganese(II) oxide, which acts as a melting agent in silicate glass systems. In addition, Mg^{2+} and Mn^{2+} ions exhibit rather close values of ionic radii of 0.74 Å and 0.91 Å, respectively [14], which allows forming a wide range of isomorphic solid solutions between magnesium cordierite and manganese cordierite. Therefore, the search for effective glass binders involves investigations in the pseudo-binary system of magnesium $Mg_2Al_4Si_5O_{18}$ (MgAS) and manganese $Mn_2Al_4Si_5O_{18}$ (MnAS) cordierites, as compounds close in their crystal structure.

In view of the above, the aim of the work was to construct a melting diagram in the pseudo-binary system MgAS–MnAS and search for the most fusible glass composition, with its further investigations as a promising glass binder for low-temperature sintering of high-strength ceramic materials based on oxygen-free silicon compounds.

Materials and methods

To obtain the prototypes, we used SiO_2, Al_2O_3, MgO, and MnO oxides of chemically pure qualification as raw materials. Raw material mixtures corresponding to the chemical composition of MgAS and MnAS were prepared by joint grinding of the components in porcelain drum mills to a residue on the mesh No.0063 of no more than 1%. According to the experimental plan, the raw material mixtures were investigated in the range of MgAS:MnAS ratios from 10:90 to 90:10 in increments of 10 wt.%. The final mixing of the components of the raw material mixtures was carried out by grinding in a planetary mill until passing through the mesh No.0045. Chalcedony drums were used. The coding of the experimental raw material mixtures was made in accordance with the conditional content of MnAS in their composition. The composition No. 1 corresponded to the MnAS content of 10 wt.%, No. 2 corresponded to 20 wt.%, ..., No. 9 corresponded to 90 wt.%.

The heat treatment of the experimental raw material mixtures was carried out in an electric resistance furnace with silicon carbide heaters in the temperature range of 1250–1450^{0}C in air. The experimental charges were placed in 5 cm^3 corundum crucibles, heated to the maximum temperature, and held for 2 hours. The conclusion of melting of the raw material mixture was based on visual observation. The heat-treated samples were cooled together with the furnace. The cooling rate to a temperature of 700^{0}C was 60^{0}C/h, which allowed us to qualitatively assess the ability of the test melts to crystallize.

The glass was melted in an electric resistance furnace with carbide-silicon heaters in air at a temperature of 1400^{0}C with a homogenization time of 1 hour. Corundum crucibles were used for melting.

Standard experimental methods were used to determine the properties of glasses and glass crystalline materials. The TLCE in the range of 20–400^{0}C was measured on a quartz vertical dilatometer at a heating rate of 4–5^{0}C/min. Thermal effects related to the glass softening point (TSP) and its crystallization were determined by differential thermal analysis on a Q-1500 D derivatograph in the temperature range of 20–1100^{0}C when a heating rate was 10^{0}C/min. The mineralogical composition of the crystallized samples was determined by X-ray diffraction (XRD) using a Philips APD-15 diffractometer in Co-K_α radiation. The infrared (IR) absorption spectra of the test glasses were determined using a Thermo-Nicolet Avatar 370 FT-IR Spectrometer. Electron microscopic studies of the samples in the fracture were performed on a TESCAN Mira 3 LMU scanning electron microscope.

Results and discussion

One of the basic principles for choosing a specific glass composition as a glass-ceramic binder for glass-ceramic materials is the minimum temperature of its melting, which ensures efficient sintering of materials at low temperatures. In this regard, the most low-melting compositions in the pseudo-binary system MgAS:MnAS were searched in the temperature range of 1250–1450^{0}C. All the samples obtained were divided into groups, in particular, those that form or do not form melts at the temperature of the study. Based on the results of the research, a melting diagram was constructed (Fig. 1), which shows a single, clearly pronounced temperature minimum at a ratio of

MgAS:MnAS≈50:50 wt.% corresponding to the temperature of 1275°C.

Most of the melted samples visually show a tendency to crystallize to a greater or lesser extent. At the same time, the crystallization ability is decreased when moving from compositions with a higher MgO content to compositions with a higher MnO content.

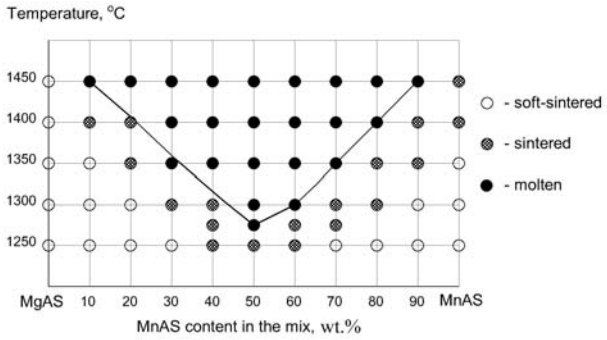


Fig. 1. Melting diagram in the pseudo-binary system MgAS–MnAS

For samples No. 1 and No. 5 (with a MgAS:MnAS ratio being 90:10 and 50:50, respectively) synthesized at 1450°C, compared to sample No. 9 (with a MgAS:MnAS ratio of 10:90), clearer absorption maxima in the infrared range are observed (Fig. 2). This is due to the structural heterogeneity of the samples due to distribution of the microcrystalline phase in the amorphous component and is consistent with the data of the XRD analysis (Fig. 3).

For the experimental samples, the main broad double absorption band in the frequency range of 830–1250 cm^{-1}, as well as a band of about 500 cm^{-1}, is characteristic of all silicates. These bands can be attributed to the valence vibrations of Si–O–Si bonds in [SiO_4] tetrahedra. When moving from sample No. 1 to sample No. 9, the main absorption band becomes smoothed, indicating the dominance of the amorphous phase. The intense absorption band with a maximum in the range of 770–800 cm^{-1} is typical of six-membered ring silicates, and in our case corresponds to aluminosilicate rings [$AlSi_5O_{18}$]. In the case of sample No. 9, the absorption band at 770 cm^{-1}, characteristic of six-membered ring structures [$AlSi_5O_{18}$], is manifested only by a weak inflection in the IR spectrum. The valence vibrations of Si–O–Si in six-membered cycles are responsible for the weak intensity absorption bands at 600 and 670 cm^{-1} (spectra of samples No. 1 and No. 5).

Al–O bonds are appeared as absorption bands at 950 cm^{-1}, which is typical of tetrahedra [AlO_4]. The absorption band at 570 cm^{-1} can be attributed to the valence vibrations of Me–O bonds, in particular

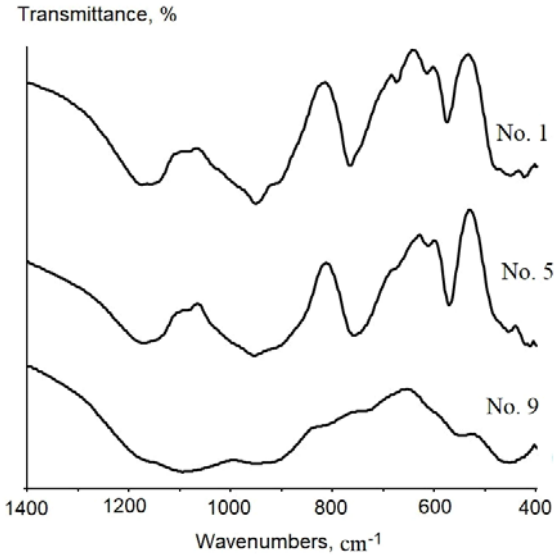


Fig. 2. Infrared spectra of experimental glass crystalline materials synthesized at 1450°C

Mg–O and Al–O [15]. The presence of these absorption bands is also characteristic of the cordierite structure.

The XRD patterns of the experimental samples (Fig. 3) fully confirms the conclusion of decreasing tendency to crystallization of glass melts when increasing the content of manganese (II) oxide in them. While intensive crystallization is observed for composition No. 1 with a MnAS content of 10 wt.%, the content of the crystalline phase is decreased significantly for compositions No. 5 and No. 9.

The main crystalline phase for the prototypes is cordierite. For sample No. 1, it is magnesium cordierite. Within the admissible error of experiments, the main reflexes on the diffractogram in Fig. 3, No. 1 ($d \cdot 10^{10}$=8.42; 4.87; 4.09; 3.371; 3.133; 3.027; 2.636; 2.098; and 1.689) correspond to the PDF No. 82-1884 $Mg_2(Al_4Si_5O_{18})$. The small reflexes observed in the diffractogram of Fig. 3, No. 9 ($d \cdot 10^{10}$=8.64; 4.09; 3.403; and 2.675) correspond to the PDF No. 29-0882 $Mn_2Al_4Si_5O_{18}$. In a diffractogram of the intermediate composition, Fig. 3, No. 5, the reflexes ($d \cdot 10^{10}$=8.35; 4.06; 3.371; 3.133; 3.018; 2.526; and 1.684) are shifted relative to both manganese and magnesium cordierite. This indicates the formation of a single solid solution between MgAS and MnAS, given the proximity of the ionic radii of Mg^{2+} (0.74 Å) and Mn^{2+} (0.91 Å) [14] and, as a result, isomorphic substitution of each other in the cordierite crystal lattice. In samples No. 5 and No. 9, in addition to the cordierite solid solution, a small amount of manganese garnet, spessartine $3MnO \cdot Al_2O_3 \cdot 3SiO_2$, is detected.

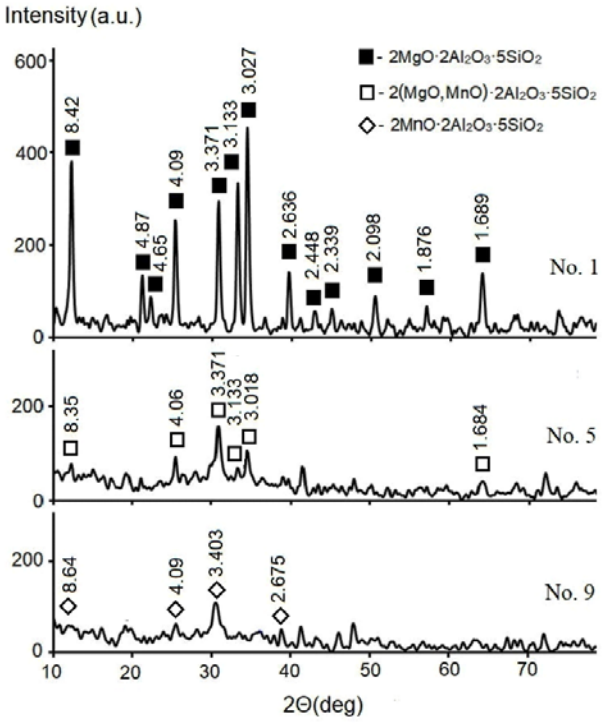


Fig. 3. XRD patterns of samples of experimental glass crystalline materials synthesized at a temperature of 1450°C

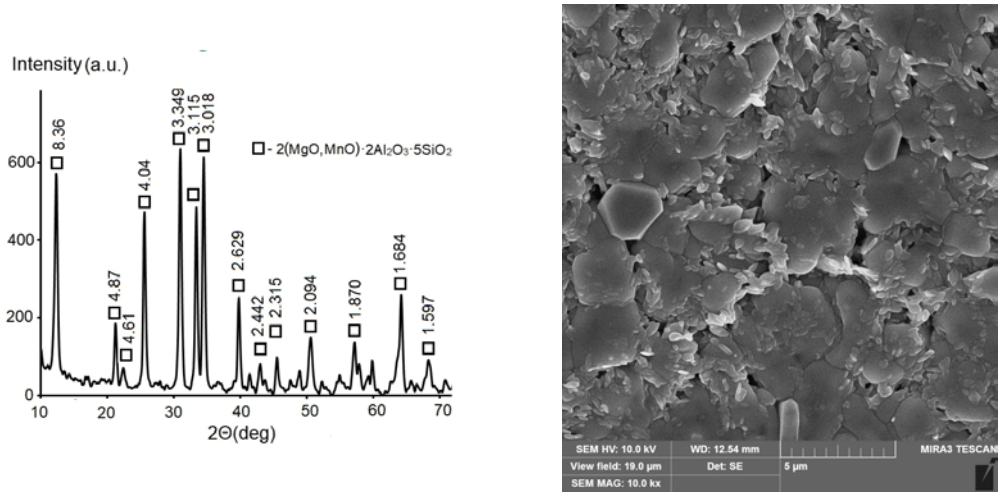
To determine the main properties of the glasses, we chose sample composition No. 5, as it exhibited the minimum temperature of homogeneous melt formation and high crystallization ability under conditions of fairly rapid cooling. The glass corresponding to the composition with the ratio MgAS:MnAS≈50:50 (wt.%) was melted at a temperature of 1450°C with a homogenization time of 2 hours. Samples were cast from the glass mass to determine the glass properties. For the sample of composition No. 5, the TLCE in the range of 20–400°C is $31.7 \cdot 10^{-7}$ °C^{-1}. According to the data of differential thermal analysis, the temperature when glass softening begins is 800°C, and intense crystallization starts at 1030°C.

In accordance with the results of the differential thermal analysis, additional heat treatment of samples of composition No. 5 was carried out at a temperature of 1030±5°C for 2 hours. The TLCE of the crystallized glass samples was determined to be $20.8 \cdot 10^{-7}$ °C^{-1}.

The shift of reflexes on the XRD patterns relative to the reflexes of pure manganese and magnesium cordierites (Fig. 4,a) and SEM image (Fig. 4,b) indicate almost complete crystallization of the experimental glass with the formation of a cordierite solid solution $2(Mg,Mn)O \cdot 2Al_2O_3 \cdot 5SiO_2$. The size of the crystals is ranged from 0.5 to 3.0 μm.

Conclusions

Based on the results of the experimental investigations, a melting diagram in the pseudo-binary system MgAS–MnAS was constructed. The ratio between magnesium cordierite and manganese cordierite (50:50 wt.%) was determined, which ensures a minimum melt formation temperature of 1275°C. The melting point of the glass of the specified composition is 1450°C. The synthesized glass has a softening point of 800°C and crystallizes intensively at a temperature of 1030°C. The TLCE of the crystallized glass samples is $20.8 \cdot 10^{-7}$ °C^{-1}. X-ray diffraction and electron microscopic researches have shown that the developed glass almost completely crystallizes during heat treatment for 2 hours to form a cordierite solid solution $2(Mg,Mn)O \cdot 2Al_2O_3 \cdot 5SiO_2$.



a **b**

Fig. 4. XRD pattern (a) and SEM image (b) of the crystallized sample of composition No. 5

The size of crystals of the cordierite phase is ranged from 0.5 to 3.0 μm. The developed glass, due to its low-melting and high tendency to crystallization, can be offered as a promising glass-crystal binder for obtaining high-strength ceramic materials (wear and impact resistant) based on SiC and Si_3N_4 with reduced sintering temperatures.

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СКЛОКРИСТАЛІЧНА ЗВ'ЯЗКА КОРДІЄРИТОВОГО СКЛАДУ ДЛЯ НИЗЬКОТЕМПЕРАТУРНОГО СПІКАННЯ ВИСОКОМІЦНИХ КЕРАМІЧНИХ МАТЕРІАЛІВ НА ОСНОВІ БЕЗКИСНЕВИХ СПОЛУК КРЕМНІЮ

С.Г. Положай, О.В. Зайчук, А.Г. Положай, О.А. Амеліна, Л.Л. Руднєва

Механічні властивості композиційних керамічних матеріалах, що одержані на основі безкисневих сполук кремнію, значною мірою визначаються властивостями склозв'язки. У роботі наведені результати досліджень щодо визначення найбільш легкоплавкого скла в псевдобінарній системі MgAS–MnAS з високою схильністю до кристалізації як склокристалічної зв'язки для низькотемпературного спікання високоміцних керамічних матеріалів на основі безкисневих сполук кремнію. Схильність дослідних розплавів до кристалізації зменшується при зростанні вмісту марганцевого кордієриту, що підтверджується даними рентгенофазових і ІЧ-спектроскопічних досліджень. На базі експериментальних досліджень побудовано діаграму плавкості, за допомогою якої визначено співвідношення між магнієвим кордієритом і марганцевим кордієритом (50:50 мас.%), що забезпечує мінімальну температуру утворення розплаву 1275^{0}C. Температура варіння скла визначеного складу становить 1450^{0}C. Синтезоване скло характеризується температурою розм'якшення 800^{0}C і інтенсивно кристалізується при температурі 1030^{0}C. Температурний коефіцієнт лінійного розширення закристалізованих зразків скла складає $20{,}8\cdot10^{-7}$ $^0C^{-1}$. Рентгенофазові і електронно-мікроскопічні дослідження показали, що розроблене скло практично повністю кристалізується при термічній обробці протягом 2 годин з утворенням кордієритового твердого розчину $2(Mg,Mn)O\cdot2Al_2O_3\cdot5SiO_2$. Розмір кристалів кордієритової фази коливається в межах 0,5–3,0 мкм. Розроблене скло, завдяки легкоплавкості та високій схильності до кристалізації, може бути запропоновано як перспективна склокристалічна зв'язка для одержання високоміцних керамічних матеріалів (зносо- і ударостійких) на основі SiC і Si_3N_4 зі зниженими температурами спікання.

Ключові слова: склозв'язка, кордієрит магнієвий, кордієрит марганцевий, діаграма плавкості, кристалізація, фазовий склад, мікроструктура.

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