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EFFECT OF MODIFIED GLASS MATRICES ON THE STRENGTHENING OF GLASS CRYSTALLINE COATINGS

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Досліджено можливість зміцнення склокристалічних покриттів при одночасній дії трьох видів модифікаторів (ZnO, TiO_2, SnO_2). Експериментально виявлено вплив концентрації кожного з трьох оксидів-модифікаторів на фазові перетворення покриттів в процесі термообробки. Запропоновано механізм дії оксидів-модифікаторів на утворення та зростання зародків кристалів отриманих фаз. Тонкі кристалічні фази, які іммобілізовані до скломатриці, сприяють утворенню однорідної ситалоподібної структури. При цьому досягається співмірність часток, яка дозволяє одержати матеріал із високими міцносними і термомеханічними властивостями

Ключові слова: скломатриця, оксиди-модифікатори (ZnO, TiO_2, SnO_2), тонкі кристалічні фази, міцносні і термомеханічні властивості

Исследована возможность упрочнения стеклокристаллических покрытий при одновременном действии трех видов модификаторов (ZnO, TiO_2, SnO_2). Экспериментально выявлено влияние концентрации каждого из трех модификаторов на фазовые превращения в процессе термообработки покрытий. Предложен механизм действия оксидов-модификаторов на образование и рост зародышей кристаллов образующихся фаз. Тонкие кристаллические фазы, которые иммобилизованы в стекломатрицу, способствуют образованию однородной ситаллоподобной структуры. При этом достигается соразмерность частиц, позволяющая получить материал с высокими прочностными и термомеханическими свойствами

Ключевые слова: стекломатрица, оксиды-модификаторы (ZnO, TiO_2, SnO_2), тонкие кристаллические фазы, прочностные и термомеханические свойства

1. Introduction

Glass-crystalline coatings with increased strength are a new class of high resource materials, characterized by unique technological and operational properties. Microstructure of these materials is determined by technological parameters of their obtaining, by the type of modifying additives, even distribution of phases in a glass matrix.

Introduction of modifying additives affects primarily regulation of the processes of phase creation, obtaining of microcrystalline dense structure of the material with the required strength of glass matrices.

A special place in the creation of high-strength glass-crystalline coatings is taken [1] by the compounds of refractory d-transitive metals of groups II, IV of the periodic table of elements.

This is linked to the peculiarities of their crystallochemical structure:

– these metals lack electrons on the internal s, p and d-orbitals; this leads to the fact that they can easily take away electrons from any source, which might be internodular atoms of oxygen;

– most transitive metals have large enough atomic resources and during formation of compounds with non-metal atoms may be subject to the Hagg's rule [2]. According to the rule, the ratio of the radii of atoms of non-metal to metal must be less than the value of 0.59. Compounds of metals of groups II, IV with oxygen can be considered as the most resistant (in terms of thermodynamics) material for glass-crystalline coatings.

In this regard, the relevance of this work consists in the management of the process of controlled volumetric crystallization for obtaining coatings with microstructure, which provides high strength and thermomechanical properties.

2. Analysis of scientific literature and the problem statement

Strength is the most important characteristic of material. In glass-crystalline materials it is usually larger than in glass. Larger strength of glass-crystalline materials is due to their high module of elasticity ($(8–14)\cdot10^4$ MPa) compared with glass ($7\cdot10^4$ MPa). Larger strength of glass-crystalline materials is due to the presence of homogeneous fine-grained microstructure, which takes on the role of the frame of rigidity [3]. Formation of microstresses in the volume of glass-crystalline material leads to the occurrence and increase of microcracks. Average size of microcracks and mechanical strength are determined by the structure of glass-crystalline material. With even distribution of crystals in the volume of glass material with sufficiently large volume of a crystalline phase, the size and distribution of microcracks can be limited by the space between crystals. The same mechanism for "blocking" cracks by the obstacles is applied in sitalls. But if the degree of crystallizing for sitalls may amount to 60÷90 %, then for glass-crystalline coatings an optimal quantity of crystalline phase should be in the range of 30÷55 % [4], which is why the properties of glass-crystalline coatings depend on the number and type of phases that are formed.

As far as the influence of individual components on the degree of crystallization is concerned, there is no consensus of researchers; the unequivocal conclusion is only that the increase in the rate of formation of crystallization nuclei and decrease in the rate of their growth occur when catalysts of crystallization are introduced to the composition of modifiers. Crystal phase can consist of crystals of one or more types, defined by both chemical composition of glass and the conditions of its thermal treatment. Using glassy oxygen silicon grid of oxides of *sp*-elements, including ZnO, TiO_2, SnO_2, as modifiers will help control creation of dense structure of the material, which must differ by increased strength. The mechanism for strengthening of the original frame of a glass matrix comes down to embedding (intercalation) of modifying groups of the type $[RO_x]$ to cybotaxic associations of short-range order and the beginning of formation of the nuclei of a new crystalline phase of the type of solid solutions. The strengthening or loosening of the structure of a glass composition as a whole depends on how dimensions of the embedded group deviate from the main grid of glass (a greater or lesser side) [5].

There has been growing interest recently in tin dioxide as a component, which plays various role in the compositions of silicate glasses. The authors [6, 7] obtained optically transparent monodispersed glassceramic with high density. The issue of the role of SnO_2 in silicate systems and its influence on the physic-chemical and crystallizing properties is given little attention in the scientific literature. The papers [8, 9] provide data on the impact of SnO_2 on the processes of liquation and crystallization. It was found that SnO_2 can not isomorphically replace SiO_2 in the structure of silicate glass, which is why it easily forms a separate phase – cassiterite – during crystallization. The contribution of SnO_2 to the indicators of properties of glasses is quite complicated and depends on the coordination state of tin, which, in turn, is determined by its concentration, conditions of synthesis and composition of glasses. In accordance with the correlation rule of ionic radii of cations and anions, it is possible for the ion $[SnO]^{4+}$ to have both four and six coordination, but the size of the tetrahedron $[SnO_4]$ is considerably larger than the size of the tetrahedron $[SiO_4]$, so embedding of the groups $[SnO_4]$ to oxygen silicon grid is problematic, even very limited. The more stable is the six coordination $[SnO]^{6+}$, as a result of which SnO_2 is easily released from the melt as in the form of crystals of cassiterite [8]. These studies are performed with regard to the fact that glass-crystalline materials are obtained on their basis to create products, the main requirements to the technologies of which are provision of high speed of forming the defined phases and finely crystalline structure at relatively low (up to 1200 °C) temperatures.

Still unsolved part of the problem is identifying the influence of compositions of certain oxides-modifiers on the processes of phase creation and microstructure when obtaining high-strength glass-crystalline coatings.

3. The purpose and objectives of the study

The purpose of the study is to obtain high-strength glass-crystalline coatings by the directed process of phase creation and formation of microstructure when using three types of additives.

To achieve the set goal, the following tasks are to be solved:

– to determine the effect of modifying components containing ZnO, TiO_2, SnO_2, and examine concentration limits of their presence in the compositions of glass-crystalline coatings;

– to determine peculiarities of formation of phase composition and microstructure of coatings and their influence on the characteristics of strength.

4. Materials and methods of the study of physical and mechanical properties of glass-crystalline coatings

When designing glass compositions, to optimize their formulations by the criterion of the largest inclination to crystallization, the calculation method of forecasting crystallizing capacity of glasses by the set of structure-sensitive coefficients was used [10].

The measurements of strength limit at bending was carried out on the samples in the form of parallelepipeds with dimensions 35×70×10 mm at the laboratory device MII-100. In the moment of destruction of the sample at its bending we recorded its destructive force and calculated bending tensile strength by the following formula:

$$\sigma = \frac{3Pl}{2bh^2}, \qquad (1)$$

where P is the destructive force at the moment of bending, H; l is the distance between supporting prisms, mm; b is the width of the studied sample, mm; h is the thickness of the studied sample, mm.

Temperature coefficient of linear expansion (TCLE) was determined at the vertical quartz dilatometer DKV-5a, using the samples of 50 mm in length and 3 mm in diameter in the temperature range of 20–600 °C. The values of TCLE were calculated by the formula:

$$\mathrm{TLCE} = \frac{1000 \cdot \Delta l_0 + k \cdot l}{1000 \cdot (t-20) \cdot l}, \qquad (2)$$

where l is the length of the sample at room temperature; Δl_0 is the change in the length of the sample; (t–20) is the difference between final and initial temperatures of the interval; k is the correction for expansion of parts of dilatometer of quartz glass.

Determining thermal resistance of the coatings was conducted according to DSTU B V.2.7-118-2002 on ceramic samples with coating by heating from 125 °C (for 30 minutes) with increasing the temperature after every 25 °C with subsequent cooling in water of the temperature 20 °C for 10 minutes. A coating is considered thermally resistant if no damage to the surface is revealed after testing. The final temperature reached when tested is the measure of crack resistance of the coating.

Microhardness of glass-crystalline coatings was determined at the device PMT-3 by the method of pushing a diamond pyramid into the surface of the coating and subsequent calculation of the value of microhardness by the imprint that remained on the surface of the sample [11].

Phase composition of the coatings was determined using the method of X-ray phase analysis (XPA) using the diffractometer DRON-3M with CuKα-radiation and nickel filter at standard conditions of its work. To identify the phases, we used the American database [12].

Petrographic studies were carried out on the polished ground joints (polished sections) in the retracted light under the microscope NU-2E with the magnification $80–320^x$ and in immersion preparations in transmitted polarized light under the microscope MIN-8 with the magnification $100–480^x$.

To calculate the viscosity (lg(η)), surface tension (σ) and TCLE of the model glass compositions, the Delphi-based software program "Properties of glaze " [13] was developed at the Department of Ceramics (Kharkiv, Ukraine), which allows calculating these properties by the chemical composition of the coating.

5. Results of the study of influence of modified glass matrices on the formation of phases and structural organization of coatings

The output glass matrix was synthesized by the technology of coatings and limited by the content of oxides, mass %: Al_2O_3 – 12,5÷17,5; SiO_2 – 43,5÷53,5; R_2O in the amount of 5.5 mass % and RO in the amount of 8.5 mass %. The compounds of intercalation of the elements of groups II and IV were chosen [14] as modifiers, whose characteristics are given in Tables 1, 2.

Based on the data specified in tables, and in accordance with the Hagg's rule, intercalations are the phases of variable composition and their corresponding chemical formulas usually characterize the maximum content of metalloids in them. Based on the phases of intercalation, solid solutions with defective lattice are easily formed. High strength of these phases is explained by the large strength of inter-atomic bonds, additional bonds that occur between non-metal and metal atoms, and by low magnitudes of inter-atomic distances as a result of replacement of metalloid atoms.

Table 1

Atomic characteristics and physical properties of the elements of groups II and IV

Name of properties	Chemical elements			
	Zn	Si	Ti	Sn
Atomic number	30	14	22	50
Atomic mass, u	65,370	28,086	47,956	118,690
Radius of ion by Belov and Bokiy, nm	0,139	0,039	0,064	0,067
Radius of atom by Belov and Bokiy, nm	0,083	0,134	0,146	0,158
Degrees of oxidation	2	4; 2; –4	4; 3	4; 2
Density, g/cm^3	7,133	2,33	4,505	7,29
Melting temperature, K	692,5	1696	1838	505

Table 2

Physical and chemical characteristics of technical oxide materials

Indicators of physical properties	Names of oxide materials		
	Zinkite ZnO	Rutile TiO_2	Cassiterite SnO_2
Moos hardness	4–5	6–6,5	6–7
Density, g/cm^3	5,64–5,68	4,2	6,950
Microhardness, MPa	2340	10000	9885–12043
Melting temperature, °C	1950–2000	1850	1625
TCLE, K^{-1}	$316 \cdot 10^{-8}$ (at 40 °C, ∥ axis c) – $539 \cdot 10^{-8}$ (at 40 °C, ⊥ axis c)	8,19 (0–500 °C)	$3{,}76 \cdot 10^{-6}$ (20–650 °C)

Based on the data indicated in Table 1, 2, we selected modifiers to ZnO, TiO_2, SnO_2 and examined the compositions of glass-crystalline coatings with their participation.

Table 3 displays chemical compositions, Table 4 presents calculated and experimental values of the properties of melts and glazed coatings, which are parched at the temperature 1200 °C.

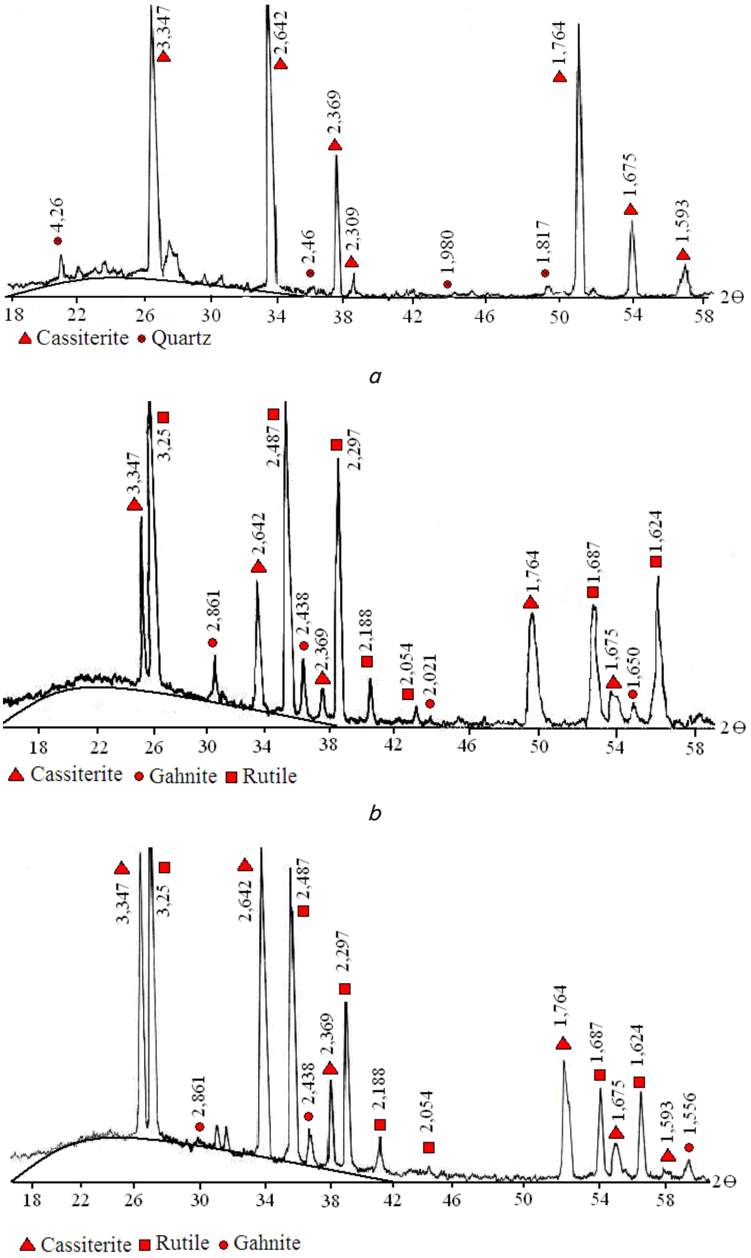
Table 3

Chemical composition of glass-crystalline coatings

Code of composition	Content of oxides, mass %									
	SiO_2	Al_2O_3	Na_2O	K_2O	MgO	CaO	BaO	ZnO	TiO_2	SnO_2
1	53,5	17,5	2.0	3,5	1,5	2,5	4,5	–	–	15,0
2	51,0	15,0	2,0	3,5	1,5	2,5	4,5	5,0	10,0	5,0
3	43,5	12,5	2,0	3,5	1,5	2,5	4,5	10,0	10,0	10,0

The study of the characteristics of the properties of the melt of the given compositions revealed that the composition 2 is optimal by the values of logarithm of viscosity and surface tension. The composition 3 is optimal by the thermal mechanical and strength indicators. Comprehensive evaluation of phase creation was carried out by the method of X-ray phase analysis and petrographic method.

Fig. 1 presents radiographs of experimental compositions of coatings.



a

b

c

Fig. 1. Radiographs of coatings: *a* is the composition 1; *b* is the composition 2; *c* is the composition 3

We can conclude from Fig. 1 that while using SnO_2 as a modifier (composition 1, Fig. 1, *a*), intense crystallization of the phase of cassiterite occurs. The phases of quartz are observed in the additive quantity.

Phase composition 2 differs by the presence of clearly defined peaks of rutile, cassiterite, gahnite. In this case, the area of amorphousness grows by 33 % compared to the composition 1.

Phase composition 3 includes a large number of cassiterite, rutile, gahnite, and in this case, the area of amorphousness grows by 16 % compared to the composition 2 (Fig. 1, *b*).

Petrographic studies have shown that a coating consists of colorless transparent isotropic glassy substance, in which evenly distributed "inclusions" of phases are observed. The particles, despite their very small size, are irregular in shape, often isometric, to a lesser extent – elongated. Characteristic of the phase composition of glass-crystalline coatings is shown in Table 5.

Table 4

Calculated and experimental properties of melts and glazed coatings

Name of properties of coatings	Code of composition		
	1	2	3
Logarithm of viscosity, lg η (Pa·s)	3,94	1,81	2,69
Surface tension, $\sigma \cdot 10^3$ N/m	311,80	312,28	323,15
TCLE, 10^{-6} degree^{-1}	5,14	5,82	6,18
Heat resistance, °C	200	225	>275
Bending tensile strength, MPa	158	196	230
Microhardness, MPa	7680	7750	8080
Modulus of elasticity, HPa	86	92	104

Table 5

Phase composition of glazed coatings

Code of composition	Composition of crystalline phase	Average grain size, microns	Quantity of glass phase	Index of light refraction ±0,003
1	cassiterite, quartz	20	12,04	1,35
2	cassiterite, gahnite, rutile	10	19,89	1,65
3	cassiterite, gahnite, rutile	less than 2	21,6	2,21

Analyzing the data from Tables 4, 5, one can see that the maximum indicators of thermal resistance, microhardness and bending strength characterize the coating of composition 3 (Fig. 1, *c*), which significantly exceeds the data known about compositions of non-fritted coverings [15].

6. Discussion of the results of study of dependency of strength and thermomechanical properties on the total concentration of oxides-modifiers

The studies have shown that the largest effect of strengthening of glass-crystalline coatings, is produced by the composition 3, in which the total concentration of oxides-modifiers (ZnO, TiO_2, SnO_2) is 30 %. We discovered the main feature of phase composition of the specified coating. According to the XPA, the zone of amorphousness in the compositions 2 and 3 (Fig. 1, *b*, *c*) increases by 40–50 % compared to the composition 1. But the indicators of strength and thermal resistance (Table 4) do not decrease but significantly grow at that. This peculiarity is associated with a comprehensive influence of the oxides-modifiers that are replaced as a result of the growth of grains of basic phases beyond the border of these grains – to a glass phase, reinforcing it.

According to the X-ray phase analysis, the samples that were calcined at 1200 °C, with the content of oxides-modifiers at 20–30 % (compositions 2, 3), are composed of the mixture of three phases: cassiterite, gahnite, and rutile. The availability of the specified phases is the cause of the increase in physical and mechanical properties of the coatings that increase depending on the concentration of cations-modifiers. The data from Table 5 indicate that with increasing concentration of oxides-modifiers the average size of crystals of newly formed phases (less than 2 microns) is reduced. Such coatings must withstand mechanical loads of pressure and friction, which can be compared to sitall products.

Based on the obtained data, one can draw a conclusion about the influence of oxides-modifiers on the properties of glass matrices and provide a mechanism for their action, which comes down to the following:

– in the silicate melts with three cations-modifiers, non-static distribution of cations-modifiers of various types is observed among the silicate anions of varying degree of polymerization, which comply with the principles of acid-base interaction in silicate melts. The mechanism of strengthening of a glass matrix is intercalation (embedding) of the modifying groups (ZnO and TiO_2) in the grid of glass and the beginning of formation of the nuclei of a new crystalline phase of the type of solid solutions;

– the possibility of a broad change in phase composition and microstructure under the action of three modifiers is a peculiarity and advantage of the coatings that allows changing their properties in a given direction. Thus, the study of the mechanism of formation of fine crystalline volumetric structure of the coatings with three modifiers revealed a sharp difference in the processes of nucleation and growth of crystals while introducing SnO_2, ZnO, TiO_2. Thus, SnO_2 in the amount of 15 % does not activate the process of phase creation but it takes on the role of an ordinary modifying oxide, additional introduction of zinc and titanium oxides contributes to formation of volumetric crystallization with the release of phases of gahnite and rutile, which contributes to significant strengthening of the coatings.

New formulations of glass-crystalline coatings with increased (by 50 %) strength and thermal resistance (by 70 %) may find wide application in engineering, medicine, various industries: chemical, machine-building and construction.

7. Conclusions

It was established as a result of the performed study:

– the most efficient way of structural organization of a glass-crystalline material is the use of three types of oxides-modifiers (ZnO, TiO_2, SnO_2), the total concentration of which is not lower than 30 %. We examined the peculiarities of formation, role and concentration limit of each of them. The introduction of TiO_2 (5–10 %) and ZnO (5–10 %) promotes the formation of rutile and gahnite, respectively, which increase physical and mechanical properties of coatings (bending tensile strength, microhardness, thermal resistance). The introduction of SnO_2 does not activate the process of phase creation but its presence (not lower than 15 %) inhibits the growth of crystals of newly formed phases;

– comprehensive application of the three oxides-modifiers (not lower than 30 %) promotes the formation of fine crystalline coating, with the size of particles not larger than 2 μm, at a relatively low (up to 1200 °C) temperatures of calcination;

– the obtained results can be used for the synthesis of glass-crystalline coatings with required phase composition and microstructure.

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TECHNOLOGICAL AND KINETIC REGULARITIES OF FORMATION OF SUSPENSION AND ITS INTERACTION WITH NITRIC ACID WHILE OBTAINING CALCIUM NITRATE

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Розроблено технологію одержання кальцієвої селітри підвищеної концентрації. Встановлено граничні концентрації вапна в розчинах кальцієвої селітри, теоретично розраховано процес гідратації оксиду кальцію в цих розчинах. Досліджено механізм загустіння приготованої суспензії (суспензія $Ca(OH)_2$ в розчині $Ca(NO_3)_2$), яка утворюється на виробництвах кальцієвої селітри. Встановлено залежності концентрації $Ca(NO_3)_2$ в розчині після нейтралізації від концентрації HNO_3 та вмісту вапна в транспортному розчині. Запропоновано технологічну схему одержання кальцієвої селітри збагачену додатковими живильними компонентами

Ключові слова: кальцій нітрат, вапно, гідратація, концентрація, суспензія, нейтралізація, нітратна кислота, технологія

Разработана технология получения кальциевой селитры повышенной концентрации. Установлены предельные концентрации извести в растворах кальциевой селитры, теоретически рассчитан процесс гидратации оксида кальция в этих растворах. Исследован механизм загустения приготовленной суспензии (суспензия $Ca(OH)_2$ в растворе $Ca(NO_3)_2$), которая образуется на производствах кальциевой селитры. Установлены зависимости концентрации $Ca(NO_3)_2$ в растворе после нейтрализации от концентрации HNO_3 и содержания извести в транспортном растворе. Предложена технологическая схема получения кальциевой селитры обогащенной дополнительными питательными компонентами

Ключевые слова: кальций нитрат, известь, гидратация, концентрация, суспензия, нейтрализация, азотная кислота, технология

1. Introduction

By the scope of application and economic results, mineral fertilizers are one of the important factors of agriculture intensification and increasing the quantity of food products. The volume of production and domestic consumption of mineral fertilizers in Ukraine indicate that only the use of nitrogen fertilizers is approaching the necessary scientifically based amount, whereas phosphate and potassium fertilizers are used less because the raw materials are very expensive.

The scope of using calcium nitrate in other industries, except agriculture, is expanding, in particular, as the basis of anti-ice mixtures, corrosion inhibitors, additives in the construction mixtures, as one of the components of emulsion explosives, accumulative solutions in refrigeration technology, etc.

The chemical industry, including the production of mineral fertilizers, is the industry which requires substantial material and energy consumption and the main trend of its development now is resource saving. Development of energy-saving technologies of the production of calcium nitrate containing