

INVESTIGATION OF STRUCTURE FORMATION IN CALCIUMSILICOPHOSPHATE GLASS-CERAMIC COATINGS FOR DENTAL IMPLANTS

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Abstract. Viability of developing bioactive glass-ceramic coatings on titanium alloys as dental implants with short terms of fusion with bone tissue has been substantiated. Features of formation of calcium silicophosphate glasses on initial stages of nucleation have been studied. It has been established that the presence of sybotaxic metaphosphate and hydroxide groups in glass melt and formation of crystalline phase nuclei through spinodal separation will allow formation of strengthened structure of vitreous material containing hydroxyapatite and fluorapatite in conditions of short-term low-temperature thermal treatment. Developed glass-ceramic materials can be used as a base in obtaining resorptive strengthened glass-ceramic coatings on titanium alloys for dental implants.

Keywords: structure formation, calcium phosphate-silicate coatings, implant, dental implantation.

1. Introduction

Currently, due to deteriorating global environment conditions and imperfect nutrition, most population over 30 years old require complete or partial teeth replacement. The use of dental implants involves a multi-stage treatment, namely preparing mouth cavity, insertion of an implant, strong binding of an implant and tissue healing, choosing and manufacturing of prosthetic crown and its installation. Average period of entire dental prosthetic rehabilitation is about six months; its defining stage (2-3 months) is insertion of implant and formation of strong bone-implant binding [1]. In dental implantology, prolonged fusion period of an implant with bone significantly increases the expenses on patient's rehabilitation and is a constraining factor in providing normal life conditions to patient. Considering the abovementioned, development of bioactive implants with

shortened terms of fusion with bone is a priority task. One of the solutions to this problem is obtaining calcium phosphate-silicate glass-ceramic coatings on titanium alloys, which will provide required strength characteristics and bioactivity level needed to form a strong apatite-like layer during one month.

An important role in developing a new generation of functional glass-ceramic materials belongs to self-organization processes of their structure, which occurs both on the stage of crystalline phase nucleation, and during their further evolution [2]. The main aspect in the synthesis of the mentioned materials is provision of uniform fine volume crystallization of calcium phosphates, in particular due to the effect of nucleation by liquation mechanism. Occurrence of secondary separation as a stage of heterogeneous formation of crystal centers is due to formation of stabilized clusters – heterophase fluctuations that form self-organizing nanostructure.

Theoretical investigation of initial stages of glass crystallization was a subject of the works by N. Toropov *et al.* [3], Shelby Notes [4]. They have pointed out that occurrence of uniform glass crystallization when there are no catalyzing additives is due to segregation of initial melt into immiscible phases in the form of very small droplets, which are not able to increase. Another point of view on this subject was articulated by V. Filipovich [3], who puts in doubt the theory of mandatory existence of pre-crystallizing liquation and points out that due to liquid phase separation, phase interfaces develop significantly, diffusion processes are facilitated and as a result, the glass composition approximates to the composition of future crystals.

However, per B. Varshal [5], the main specific feature of the metastable liquation process is not the fact that in conditions of unlimited high temperature treatment of a melt it will necessarily crystallize, since the processes occur before the beginning of the crystallization, and in such conditions metastability does not affect them. The main specific feature of these processes is that they occur in conditions of high viscosity of both initial melts and each of two phases formed, *i.e.* they occur relatively

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slowly. Thus, it is possible to obtain two-phase macro-nonuniform materials, which consist of two sections of matter, different by composition and properties, which often possess a valuable complex of properties.

It is known that calcium phosphate-silicate systems have wide liquation regions. Mechanism of action of P_2O_5 as crystallization catalyst is based on separating a system into two liquid phases. Phosphorus pentoxide is incorporated into glass network and creates prerequisites to break Si–O–Si bonds, as a double bond between phosphorous and oxygen is formed. Such bond differentiates $[PO_4]$ tetrahedra from $[SiO_4]$. It is liquation mechanism of nucleation, which creates conditions for provision of uniform glass-ceramic structure with crystalline phases that are responsible for performance properties of glass ceramic materials and coatings [6].

The aim of this work is development of glass-ceramic materials on titanium alloys for dental implants and investigation of structure formation in the materials during thermal treatment.

2. Experimental

2.1. Investigation Methods

Presence of crystalline phase was investigated using XRD (“DRON-3”) and petrographic (polarizing microscope NU-2E) methods. Microstructure of glasses was investigated using EMB 100 AK electron microscope and Specord-M80 spectrophotometer. Temperature of thermal treatment and mechanism of glass crystallization was established using the methods of differential thermal analysis (Paulik-Paulik-Erday derivatograph).

Microhardness (H) and Vickers hardness (HV) were determined by indenting diamond pyramid under the load of 1.961 N for 10 investigations and 49 N for 5 investigations. Crack resistance parameter (K_{IC}) was determined using TMV-1000 device by indenting Vickers pyramid under the load of 49 N for 5 investigations and calculating using Niihari semi-empirical dependence [7].

Behavior of materials *in vitro* was determined by their solubility in distilled water with pH = 5.4–6.6 using gravimetric method.

2.2. Development of Compositions of Glass-Ceramic Materials for Dental Implants

Bioactive glass-ceramic coatings for dental implants on titanium alloys may be obtained by controlling the processes of structure and phase formation under low-temperature short-term single-stage thermal treatment by means of:

- process of the fine volume crystallization of the coatings with formation of bioactive phases of calcium phosphates by liquation mechanism of phase separation;

- provision of formation of sybotaxic groups of $[PO_4]^{3-}$, $[PO_3]_n^{n-}$ and $[F]^-$ in glass melt, with the ratios $CaO/P_2O_5 = 1.67$, similar to stoichiometric hydroxyapatite, $R_2O/P_2O_5 > 1.0$ and the amount of $CaF_2 > 1.0$ wt %;

- crystallization of hydroxyapatite (HAP) and fluorapatite (FAP) in total amount of 30–40 vol %.

Presence of bioactive HAP and FAP after thermal treatment, which strengthen the structure, allows to form a material with the structure and composition close to that of natural teeth. In order to provide a certain level of resorption and adhesion to the base coating, total amount of crystalline phase in glass-ceramic coating must be in the range of 30–40 vol %. Additionally, in order to provide biocompatibility, the amount of FAP crystalline phase must not exceed 10 % of the total amount of crystalline phase (1–4 vol %).

Development of the compositions of bioactive glass-ceramic materials and coating is based on calcium phosphate-silicate and silicate glasses, which contain crystallization catalysts. Presence of silicon in glass composition intensifies the process of cell adhesion to the surface of the material, their proliferation and differentiation, collagen and osteogenic markers synthesis, since Si–OH nucleators, due to their initiating effect, stimulate homogeneous and heterogeneous nucleation of hydroxyapatite crystals on the surface of abovementioned materials [8].

Provision of the above composition and structure of glasses will allow to obtain glass-ceramic coatings on their base with required level of performance characteristics, namely:

- certain level of coatings solubility required for the formation of apatite-like layer on their surface in physiological medium *in vitro*, which is a guarantee of a strong bond in “implant-bone” system;

- mechanical properties of developed glass-crystalline coatings on titanium alloys must be close to the properties of natural teeth ($H \approx 4.0$ GPa; $HV \approx 3.4$ –3.7 GPa).

Considering the experience up to date and the complex of requirements to glass-ceramic coatings used in dental implants, the system R_2O – RO – CaF_2 – R_2O_3 – P_2O_5 – SiO_2 has been chosen as initial one, where R_2O – Na_2O , K_2O , Li_2O ; RO – CaO , ZnO ; R_2O_3 – B_2O_3 , Al_2O_3 . In the region of defined concentration limits, the model glasses were chosen with the following ratio of phase forming components to provide HAP and FAP crystallization: $CaO/P_2O_5 = 1.67$ and $CaF_2 = 1.5$ –6.5 wt % (Table 1). In order to provide improved mechanical characteristics of

glass-ceramic materials and coatings in conditions of cyclic loads, ZnO has been chosen as a catalyst for crystallization of resistive crystalline phase – HAP. The presence of zinc oxide will allow obtaining glass-ceramic coatings with fine structure, which will strengthen the structure of the material and increase its reactivity. CaF₂ was selected as a crystalline phase catalyst and phase-forming component. Moreover, introduction of fluorite and boron oxide as fluxes will positively influence the decreasing of TCLE ($\approx 90 \cdot 10^{-7} \text{ deg}^{-1}$), glass melting and coating firing temperature, which is needed to prevent significant thickness of brittle alpha case from forming when obtaining quality glass-ceramic coatings for titanium and titanium alloys. In order to decrease viscosity, which is a guarantee of the formation of fluctuation structure, sodium, potassium and lithium oxides were introduced into the glass composition.

Synthesis of model glasses with the amount of SiO₂ ≤ 60 wt % leads to decreasing of the amount of cristobalite, that can be formed from free quartz during polymorphous transformations, which decreases the possibility of coating chipping during the cooling of metal-ceramic composition. Considering resource and energy saving requirements, domestic complex natural mineral raw material has been used as a main component to introduce silicon, aluminum and potassium oxides – feldspar from Maidan-Vilsk deposit.

Melting of model glasses has been carried out in corundum crucibles at the temperatures of 1473–1623 K, the rate of heating being approximately 5 K/min, during 6 h (Table 1). Fritting was carried out by dry method on a sheet of metal. After melting, the glasses were opacified and had blue and blue-and-white color. Coating on VT1-00 titanium alloy was obtained by slip technology.

Table 1

Chemical composition of model glasses, crystalline phases which form after their melting and thermal treatment

| GCM | Chemical composition of model glasses, wt % | | | | | Amount of crystalline phase, vol % | | Technological parameters | | Amount of crystalline phase in the coatings, vol % | |
|-------|---|-------------------------------|------------------|---------------------------|------|------------------------------------|------------------|--------------------------|---|--|-----|
| | Phase forming components | | | Crystallization catalysts | | | | Melting temperature, K | Thermal treatment temperature, K, at $\tau = 1.5$ min | | |
| | CaO | P ₂ O ₅ | SiO ₂ | | | ZnO | CaF ₂ | | | HAP | FAP |
| | | | | HAP | FAP | | | | | | |
| FAR-1 | 9.4 | 5.6 | 42.5 | 4.53 | 6.78 | 0 | 0 | 1473 | 973 | 24 | 1 |
| FAR-2 | 9.4 | 5.6 | 50.0 | 1.02 | 1.54 | 18 | 2 | 1573 | 1033 | 33 | 2 |
| FAR-3 | 16.3 | 9.8 | 42.5 | 1.38 | 2.08 | 17 | 3 | 1623 | 993 | 32 | 3 |
| FAR-4 | 9.4 | 5.6 | 46.3 | 2.77 | 4.17 | 13 | 2 | 1573 | 993 | 26 | 2 |
| FAR-5 | 12.8 | 7.7 | 46.3 | 1.20 | 1.81 | 12 | 3 | 1573 | 1023 | 37 | 3 |
| FAR-6 | 12.8 | 7.7 | 42.5 | 2.95 | 4.44 | 15 | 2 | 1573 | 1013 | 40 | 4 |

3. Results and Discussion

3.1. Investigation of Structure Formation and Phase Composition of Model Glasses and Glass-Ceramic Materials on Their Base During Thermal Treatment

According to the data of XRD and petrographic analysis, model glasses FAR-2, FAR-3, FAR-4, FAR-5, and FAR-6 contain HAP and FAP in the amount of 10–18 and 2–5 vol %, respectively, already after melting. Model glass FAR-1 is X-ray amorphous after melting, which is explained by the lowest amount of phase-forming components among the developed glasses. Large amount of crystalline phase of up to 18–20 vol % for FAR-2, FAR-3 and FAR-6 glasses is determined by the amount of crystallization catalyst ZnO (2–3 wt %). Decrease of the amount of crystalline phase to 15 vol % (after melting) for FAR-4 and FAR-5 glasses due to the amount of crystallization catalysts $\Sigma(\text{ZnO}, \text{CaF}_2) = 5.5$ and 2.4 wt %, respectively, creates prerequisites for obtaining a coating on their base with improved performance characteristics.

respectively, creates prerequisites for obtaining a coating on their base with improved performance characteristics.

In order to investigate the structure of glasses, FAR-5 model glass has been chosen as a base for obtaining glass-ceramic coating, and for comparison, FAR-2 glass, with the highest amount of crystalline phase after melting, was also used.

According to the data of IR-spectroscopy, the model glasses are characterized by wide absorption band of stretch (ν) oscillations (ν) over 900–1200 cm⁻¹ range having a deep maximum at 1050 cm⁻¹, and deformation oscillations (δ) at 450 cm⁻¹, which is the evidence of the high level of [SiO₄] tetrahedra polymerization (Fig. 1).

Presence of Al₂O₃ in model glasses favors formation of three-dimensional network with [AlO₄] and [SiO₄] tetrahedra. Occurrence of Si–O–Al bonds in glasses is confirmed by absorption band over the 720–780 cm⁻¹ range. Absence of oscillations over 580–680 cm⁻¹ range, which are intrinsic to [AlO₆] octahedra, indicates that aluminum exists in four-fold state with absorption band over 710–780 cm⁻¹ range, and isomorphically substitutes silicon, forming mixed aluminum-silicon-oxygen network.

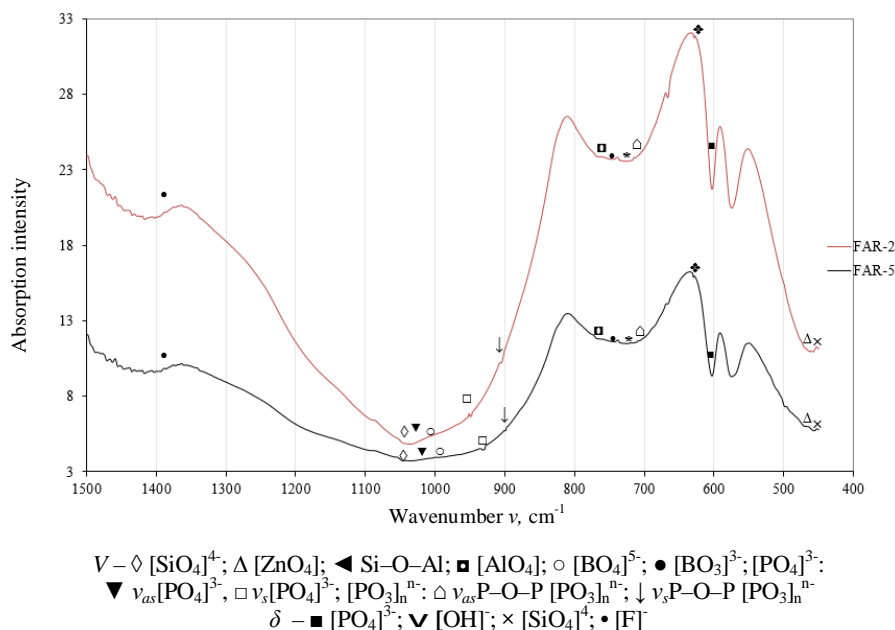


Fig. 1. IR-spectra of experimental glass materials after melting

In the model glasses, stretch oscillations of $[\text{BO}_3]^{3-}$, which are characterized by absorption band over the 1350–1400 cm^{-1} range, are nondistinct, which is the evidence of a more dominant tetrahedral coordination of boron. It is confirmed by the fact that all model glasses are characterized by $\Psi_B > 1$, in this case boron exists mainly in a fourfold coordination. Presence of fourfold boron and alkali metals in the glass, which together with $[\text{BO}_4]^{5-}$ tetrahedral form $[(\text{BO}_4)^5\text{Me}^+]^4$, increases the connectivity level of the anion network of glass. Thus, introduction of aluminum and boron oxides into model glasses will significantly increase their chemical resistance and hence reduce their solubility. Moreover, existence of $[\text{BO}_4]$, $[\text{AlO}_4]$ and $[\text{ZnO}_4]$ tetrahedral will allow to somewhat decrease TCLE of model glasses, which will have a significant impact when obtaining quality glass ceramic coatings on titanium alloys.

Absorption maxima of symmetrical oscillations ν_s of $[\text{PO}_3]_n^{n-}$ polyhadra bridges P–O–P $[\text{PO}_3]_n^{n-}$ over the range of 630–815 cm^{-1} and asymmetrical oscillations ν_{as} over the range of 875–1015 cm^{-1} are observed in model glasses, with particularly high intensity in FAR-5 model glass which contains > 7.0 wt % of phosphorous oxide. The presence of these groups in the glass matrix will allow providing the preset level of solubility. For a polyhedron of the group $[\text{PO}_4]^{3-}$ with absorption maximum of oscillation $\nu_{as}\text{PO}_4$ and $\nu_s\text{PO}_4$, oscillation over the ranges of 970–1100 cm^{-1} and 910–950 cm^{-1} are observed, being most intensive for FAR-2 glass.

Existence of absorption maxima at 747 and 630 cm^{-1} is the evidence of $[\text{F}]^-$ [9] and $[\text{OH}]^-$ [10] groups,

respectively, in the structure of glass, and are characteristic of HAP and FAP crystals.

Therefore, the presence of $[\text{PO}_4]^{3-}$ structural groups in the spectra of model glasses allows to confirm both presence of phosphate in the structure of model glasses and latent inner rebuilding of their structure with the formation of groups with short-range order and chemical bonds similar to those in crystals. The presence of metaphosphate and hydroxide groups in the structure of glasses as homogeneous nucleators of crystalline calcium phosphates phases, along with the presence of polyphosphates, is an important prerequisite of forming apatite layer on the surface of glass-ceramic coatings on titanium alloys *in vivo*, which is the main aspect of their bioactivity. Determined amount of fluoride-ions in the apatite structure allows approximating both phase composition and mechanical properties of the coating to those of teeth.

In order to study the mechanisms of nucleation and structure formation of model glasses in conditions of low-temperature thermal treatment, FAR-5 model glass was selected, which satisfies the requirements to the base glass the most. According to the results of differential-thermal analysis (Fig. 2), the following characteristic temperatures have been chosen for the glass to study the glass forming stages: temperature T_1 after melting; temperature T_2 after thermal treatment near softening onset T_E and temperature T_3 above the temperature of softening onset; temperature of “end” crystallization T_4 and temperature of glass-ceramic coating formation T_5 . To identify the structure of glass-material on initial stages of crystalline phases nucleation, areas free of crystallization were selected [3].

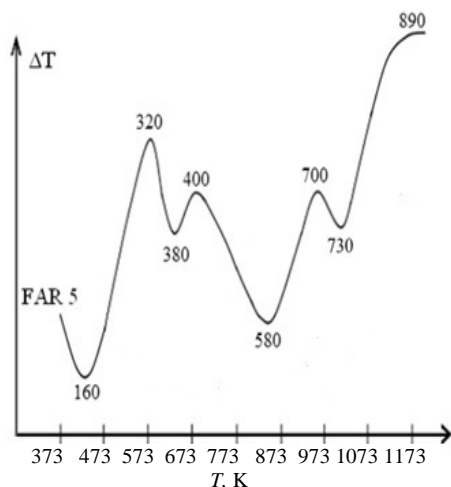


Fig. 2. Thermogram of FAR 5 model glass

According to the results of electron microscopy, FAR-5 glass sample is a multiphase system formed by mother glass and drop-like concave and convex entities. Small amounts of spherical nonuniformities with the size of 0.2–0.4 μm and significant amounts of fine globular particles with the size $\approx 0.005 \mu\text{m}$ uniformly distributed in the volume are observed on the surface of the glass (Fig. 3a). Short parallel microfractures are observed near drop-like inclusions on the breakage view of the sample. These microfractures is an evidence of fluctuational nature of nonuniformities, since as a result of breakage, microfractures are formed behind the particulate inclusions, parallel to each other and directed along the crystallization front.

An interesting fact, apart from sharpness of the microstructure displayed on the figure, is regular positioning of spherical nonuniformities on some areas, *i.e.* along the straight lines with formation of chains. Such microliquational structure with nonuniformities of an order of magnitude of 100–500 \AA is a transitional fluctuational structure, characteristic to glass as a single, although micrononuniform, phase. According to the minimum thermal potential change principle, fluctuation areas have a shape that is close to spherical; occurrence of elongated areas is mostly explained by their coalescence.

After treating the breakage of FAR-5 glass material in 0.5% solution of hydrofluoric acid, significant number of spherical nonuniformities with the sizes of 0.1–0.2 μm is observed (Fig. 3b). Drop-like nonuniformities have the shape of concavities after etching. This can be attributed to respective changes in composition from periphery of a drop to its center. After etching the surface of experimental samples, chemically resistant phase is located behind the protrusions, less resistant – behind cavities.

The increase of number of small nonuniformities with the size of 0.2 μm at the temperature of $T_3 = 893 \text{ K}$ after treating the sample in 0.5% hydrofluoric acid is explained by the increase of intensity of chemical

composition (Fig. 3d) and structure and its closeness to the temperature of phase transition $T_4 = 993 \text{ K}$, near which the transitional fluctuation reach their maximum (Fig. 3e). In this case, crystalline phases are formed, whose structure is most affined to the structure of glass in terms of short-range order. This will lead to the decrease of energy barriers for crystals nucleation, which can easily be overcome at relatively low temperatures used for initial crystallization of glass.

The structure of FAR-5 glass at $T_5 = 1013 \text{ K}$ is micrononuniform and represented by prismatic elongated HAP crystals with the size of $0.2 \times 1.0 \mu\text{m}$ and columnar crystals with hexagonal faceting with the size of $0.3 \times 0.5 \mu\text{m}$ with split ends (Fig. 3f), which, according to [12], may be the evidence of the effect of fluoride-ions on the HAP structure, *i.e.* of FAP crystals. Crystals oriented in a certain direction penetrate base matrix and assist in reinforcing the material.

3.2. Development of Glass-Ceramic Coatings and Investigation of Their Performance Properties

In order to assess the possibility of using FAR-5 model glass as a base in obtaining glass-ceramic coatings on VT1-00 titanium alloy, slip application technology has been chosen.

Surface pre-treatment of VT1-00 titanium alloy, used in obtaining bone implants in dental implantology, was done by sand-blasting technique, while adhering to the requirements to titanium-ceramic linings. To provide coating layer thickness of about 100–200 μm , needed to obtain bioactivity, sifting of the powder of FAR-5 model glass was done using a sieve with 10000 openings/ cm^2 with a cell size of 60 μm [13]. It has been established that optimal grinding fineness is 3.0–3.5°BS.

0.2 wt % solution of xanthum gum was selected as optimal dispersing medium for obtaining defect-free coating, the use of which allows to obtain acceptable values of slip density and covering power – 1.5 g/cm^3 and 3.2 g/dm^2 , respectively. Derived slips were applied by pouring onto specimens made of VT1-00 titanium alloy for use in medical equipment (ISO 5832/III) and dried at 353–393 K. Firing of the coatings was done in the temperature range of 1013–1023 K for 1–1.5 min. The indicated temperature regime allows to avoid active oxidation of titanium alloys with formation of brittle alpha case of significant thickness, as well as deformation of the item itself [14].

Results of studying performance properties of developed glass-ceramic coating based on FAR-5 model glass (Table 2) allow to state the formation of fine volume

crystallized structure with occurrence of crystalline phases of HAP and FAP in the amount of 37 and 3 vol %, respectively, allows to obtain a coating with preset

chemical and mechanical properties, enabling its use as a base in developing dental implants with shortened terms of fusion with bone.

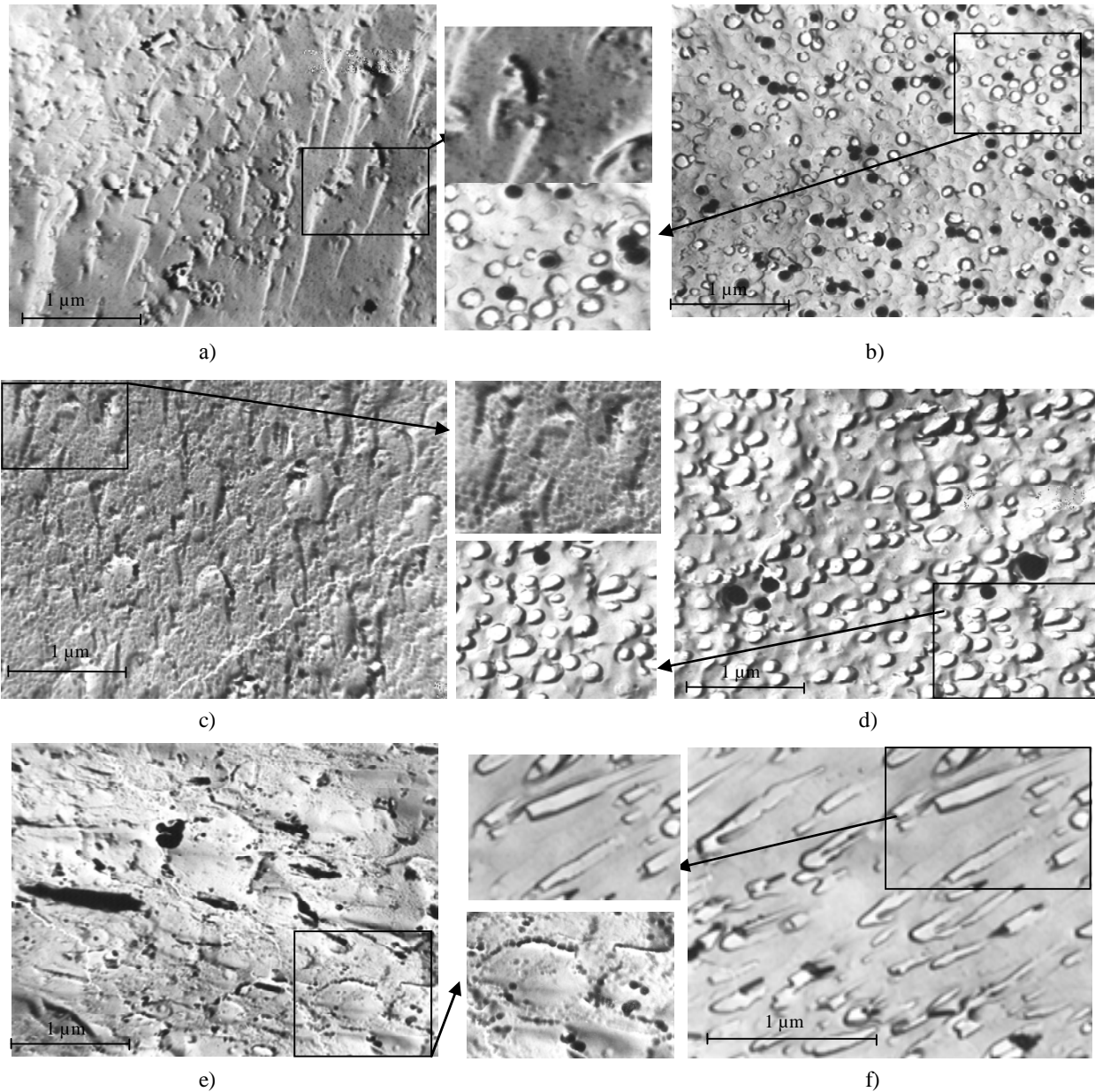


Fig. 3. Structure of initial FAR-5 glass (a), after treatment in HF (b) and after thermal treatment at: 853 K (c); 893 K (d); 993 K (e); 1013 K (f)

Table 2

Performance and technological properties of glass-ceramic coating FAR-5

| Indicator | Chemical properties | Mechanical properties | | | Technological properties |
|-----------|--|-----------------------------|-----------------------------------|---|--|
| | Mass loss after exposure to distilled water w , wt % | Vickers hardness, H , GPa | Vickers microhardness, HV , GPa | Crack resistance parameter, K_{Ic} , MPa·m ^{1/2} | TCLE, $(\alpha_{25-500} \cdot 10^7)$, deg ⁻¹ |
| Value | 0.528 | 6.8 | 6.6 | 2.6 | 89.3 |

4. Conclusions

Relevance and directions of development of bioactive glass-ceramic coatings on titanium alloys for dental implants with shortened resorption term has been established. A choice of basic $R_2O-RO-CaF_2-R_2O_3-P_2O_5-SiO_2$ system has been substantiated, within which model glasses have been synthesized and glass-ceramic coatings on VT1-00 titanium alloy have been obtained on their base using slip technology. Mechanism of structure and phase formation of calcium phosphate-silicate glass has been established, which consists in the following sequential processes: formation of $[PO_4]^{3-}$ and $[F]$ sybotaxic groups in the melt, provided the ratio of $R_2O/P_2O_5 > 2.0$ and amount of CaF_2 is 1.8 wt %, with subsequent formation of crystallization nucleators with sharp phase boundaries and crystal growth on cooling due to liquation (spinodal mechanism) with formation of the two-frame structure and secondary separation; occurrence of the volume fine crystallization in glass in conditions of short-term low-temperature thermal treatment at $CaO/P_2O_5 = 1.67$ with formation of hydroxyapatite and fluorapatite, which is a prerequisite of their bioactivity and high strength.

Developed calcium phosphate-silicate coating on titanium alloys can be used as a base in obtaining bioactive implants with shortened resorption term for dental implantology.

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ДОСЛІДЖЕННЯ СТРУКТУРОУТВОРЕННЯ В КАЛЬЦІЙФОСФАТОСИЛКАТНИХ СКЛОКРИСТАЛІЧНИХ ПОКРИТТЯХ ДЛЯ ДЕНТАЛЬНОГО ПРОТЕЗУВАННЯ

Анотація. Обґрунтовано перспективність створення біоактивних склокристалічних покриттів по сплавах титану як імплантатів для дентального протезування зі скороченим терміном зрощування з кістковою тканиною. Досліджено особливості формування структури кальційфосфатосилікатних стекол на початкових етапах зародкоутворення. Встановлено, що наявність сиботоксичних метафосфатних і гідроксидних груп у склорозпаві та утворення зародків кристалічної фази за механізмом спінодального розподілу дає можливість сформувати зміцнену структуру скломатеріалів з вмістом гідроксиапатиту та флюорапатиту за умов низькотемпературного короткочасного термічного оброблення. Розроблені склокристалічні матеріали можуть бути використані як основа при одержанні резорбційних зміцнених склокристалічних покриттів по сплавах титану для дентальних імплантатів.

Ключові слова: структуроутворення, кальційфосфатосилікатні склокристалічні покриття, імплантат, дентальне протезування.