GLASS-CRYSTAL MATERIALS ON THE BASIS OF $\text{CaO-P}_2\text{O}_5\text{-MgO-Al}_2\text{O}_3\text{-SiO}_2$ SYSTEM

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Abstract $\text{CaO-P}_2\text{O}_5\text{-MgO-Al}_2\text{O}_3\text{-SiO}_2$ system was researched. The biocompatible glass-crystal biomaterials were synthesized on the basis of the researched system. The phase compound, microstructure and physical-chemical properties of crystallized glasses were determined. The obtained glass-crystal biomaterials are intended for medical purposes, specifically as bone and tooth implants in reconstruction and orthopedic stomatology.

INTRODUCTION

The biocompatible glass-crystal materials on the basis of phosphates are finding a wide use in medicine as bone and tooth implants.

The interest to the bioglass-crystal materials lately has sharply increased, because of exceptional properties: high physical-technical indexes and their ability to biochemical interaction with a living tissue. Most impressionable results are reached by the authors.¹⁻³

The present work gives results of research of $\text{CaO-P}_2\text{O}_5\text{-MgO-Al}_2\text{O}_3\text{-SiO}_2$ system by synthesis of the glass-crystal biocompatible materials containing $\text{Mg}_3[\text{PO}_4]_2$ – $\text{CaAl}_2\text{Si}_2\text{O}_8$ as crystal phases. Study of the magnesium orthophosphate – anortite system was a subject of a separate research.

EXPERIMENTAL PROCEDURE

Determination of the melting temperatures as well as experiments with the annealing and quenching for definition of the liquidus and solidus points were carried out on the high-temperature microscope MHO-2. During study of phase equilibria the temperature of incandescence of crystallization was determined by the way of fixing temperature when crystals appeared from melt. For amplification of results an equilibrium was approached from "below": the specimens were held at the temperatures higher than the melting point, and as a temperature of crystallization there was taken the temperature by which disappearance of crystal from melt was observed. Precision of temperature was $\pm 10^{\circ}\text{C}$.

The glasses were melted in refractory crucibles put in furnace with silicon carbide heaters in laboratory conditions. The maximum temperature of melting of the glasses was $1350\text{--}1400^{\circ}\text{C}$ during 1 hour.

The crystallization ability of experimental glasses was studied by the methods of mass crystallization, DTA, electron-microscope and X-ray.

RESULTS AND DISCUSSION

Research of the phase equilibria in the system $\text{Mg}_3[\text{PO}_4]_2 - \text{CaAl}_2\text{Si}_2\text{O}_8$ showed that the studied system belongs to the simple eutectic type. Eutectic melts at the temperature 1220°C . The chemical compounds and solid solutions in the system don't appear. The eutectical point contains 53 mass.% of $\text{Mg}_3[\text{PO}_4]_2$ and 47 mass.% of $\text{CaAl}_2\text{Si}_2\text{O}_8$.

The crystal-optical and X-ray phase researches showed that compositions containing from 100 to 40 mass.% of $\text{Mg}_3[\text{PO}_4]_2$ as a primary phase educe crystals of anortite (Table 1).

The carried out electron-microscope researches of the glasses of eutectical composition confirms presence of the fields of non-mixing fluids. Microliquation is represented as drops in size of $0.1\text{--}0.5\text{ }\mu\text{m}$. The reason of exfoliation of glasses may be explained by differences between structure forming ions of Si^{+4} and P^{+5} which leads to isolation of the phosphorus-containing groups still at the temperatures, higher than the liquidus temperature.

TABLE 1.- The phase formation in the system $\text{Mg}_3[\text{PO}_4]_2\text{-CaAl}_2\text{Si}_2\text{O}_8$

Compound, mass. %		Temperature, °C	Time, min.	Phases
$\text{Mg}_3[\text{PO}_4]_2$	$\text{CaAl}_2\text{Si}_2\text{O}_8$			
90	10	1340	5	Magnesium orthophosphate+glass
		1330	4	Magnesium orthophosphate+glass
		1350	4	Glass
		1240	7	Magnesium orthophosphate+glass
80	20	1260	7	Magnesium orthophosphate+glass
		1380	7	Glass
		1260	4	Magnesium orthophosphate+glass
		1280		Magnesium orthophosphate+glass
70	30	1360	4	Glass
		1240	4	Magnesium orthophosphate+glass
		1220	4	Magnesium orthophosphate+ anortite+glass
		1380	4	Glass
40	60	1220	4	Anortite+glass
		1370	4	Glass
30	70	1240	4	Anortite+glass
		1385	4	Glass

Synthesis and melting of glasses on the basis of the tested system $\text{Mg}_3[\text{PO}_4]_2\text{-CaAl}_2\text{Si}_2\text{O}_8$ were carried out on the basis of reactive raw materials at the temperature

1350-1400°C during 1 hour. The crystallization ability of testing glasses was studied by the method of mass crystallization, results of the tests are shown in Table 2.

It was determined that in the studied system the temperature of crystallization of glasses of compounds 58-63 mass.% of $\text{CaAl}_2\text{Si}_2\text{O}_8$ was 1000°C during 2 hours. The crystallized specimens are characterized by the dense crystal structure, and the first and main crystal phase is $\text{Mg}_3[\text{PO}_4]_2$ in these compounds, anortite is represented in two modifications: – triclinic and orthorhombic.

TABLE 2. Crystallization ability of glasses

Composition No	Temperature, °C					
	650	700	800	900	1000	1100
1
2
3
4
5

The conditional indication of the degree of crystallization of specimens (visual examination).

- – Absence of signs of crystallization
- – Surface crystallization as separate fields
- – Surface crystallization as solid fine film
- – Surface crystallization as solid thick crust and with partial spreading of crystals.
- – Spreading of crystallization all over the volume.

Crystallized glasses have the milk-white color, and surface near to polished, which impart them the attractive view.

Research of processes of sintering of glass powders by the method of the ceramic technology showed that the synthesized bioceramics have following physical-chemical properties: Thermal Expansion Coefficient $(85-87) \cdot 10^{-7} \text{ degree}^{-1}$, density – 2750-2800 kg/m^3 , microhardness – 7310-7300 MPa.

For the purpose of studying the biocompatibility of the material there were carried out experiments on rabbits. The reaction of organisms of the animals was studied by adding bioceramics under the skin intramuscular in the field of the lower jaw of rabbit.

The carried out morphological researches of implant-surrounding bone tissues showed the good biocompatibility, changes in blood of tested animals were not determined. Tests made on the animals showed, that the glass-crystal materials, synthesized on the basis of the glasses of the system $\text{CaO-P}_2\text{O}_5\text{-MgO-Al}_2\text{O}_3\text{-SiO}_2$ may be effectively used for reconstruction and orthopedic stomatology as bone and tooth implants.

Using the synthesized glass-crystal materials as bone implants in human organism (jaw-face surgery) showed possibility of their effective application.

CONCLUSIONS

$\text{CaO-P}_2\text{O}_5\text{-MgO-Al}_2\text{O}_3\text{-SiO}_2$ system was researched. Results of the research of the system showed, that crystal phases exude $\text{Mg}_3[\text{PO}_4]_2$ and $\text{CaAl}_2\text{Si}_2\text{O}_8$.

Study of the phase equilibriums in the system, showed that $\text{Mg}_3[\text{PO}_4]_2\text{-CaAl}_2\text{Si}_2\text{O}_8$ system concerns the simple eutectical type. The eutectical point contains 53 mass.% of $\text{Mg}_3[\text{PO}_4]_2$ and 47 mass.% of $\text{CaAl}_2\text{Si}_2\text{O}_8$.

On the basis of the tested system the biomaterials were developed for using as an intrabone implants, for orthopedical needs, tooth crowns and metal crown covers.

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