

# DIRECT CRYSTALLIZATION OF SILICATE- PHOSPHATE GLASS FROM NaMgPO<sub>4</sub>-SiO<sub>2</sub> SYSTEM

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## Introduction

In many cases of trauma and injuries to the skeletal system, the particular need for biomaterials with superior properties is required. One of the most attractive materials are bioactive silico-phosphate glasses, which have the ability to form strong chemical bonds with living tissue when they are in contact with biological fluids [1]. Application of glasses as biomaterials is limited mainly due to their very low strength and chemical stability. One of the best ways to improve the mechanical properties of the glasses is to carry out their partial devitrification that allows to obtain glass-crystalline materials [2]. However, this process needs to be highly controlled because the appearance and growth of the crystalline phase may result in loss of bioactivity. In order to fully control the direct crystallization process, it is necessary to know the structure, microstructure and thermal properties of the glassy precursor [3,4]. The most accurate description of microstructure and structure of silico-phosphate glasses provides the opportunity to correctly plan direct crystallization process in order to obtain glass-crystalline materials with premeditated phase composition and crystallite size.

## Materials and Methods

The standard sol-gel method was chosen to obtain the materials of the highest possible homogeneity and to reduce the volatility of the individual components. TEOS (SiO<sub>2</sub>), Na<sub>3</sub>PO<sub>4</sub>·12H<sub>2</sub>O (Na<sub>2</sub>O), Mg(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O (MgO) and H<sub>3</sub>PO<sub>4</sub> (P<sub>2</sub>O<sub>5</sub>) were used to introduce particular oxides. The obtained gels were dried at room temperature (30 days) and then at the temperature of 80°C. To obtain the glassy samples the gels were melted in platinum crucible in the temperature of 1700°C (stabilized for 2 h) and then rapidly cooled. The gradient method was used to perform the direct crystallization process.

The obtained material was examined by thermal methods, X-ray diffractometry, infrared spectroscopy and scanning electron microscopy with the energy-dispersive X-ray spectroscopy compartment. The samples were heated in a gradient furnace for 2 h at the temperature determined on the basis of DSC analysis. In order to identify the type of crystallizing phases the detailed X-ray studies of the materials were carried out.

## Results and Discussion

In all studied glasses liquation phenomenon takes place. It means that it was observed glassy spherical droplets (inclusions) in glassy matrix. EDX analysis indicated that chemical composition of inclusions and matrix is different, therefore they must also have different crystallization temperature [5].

The liquation phenomenon seems to be beneficial as it may help to limit the problem associated with the uncontrolled growth of crystalline phases during devitrification. Phase boundary between inclusions and matrix should be a barrier, which can limit growth of crystals. FIG. 1 presents DSC curve of 3Mg glass.

The crystallization process runs in several stages. This is evidenced by four separated exothermal peaks, what proves that four different phases crystallize.

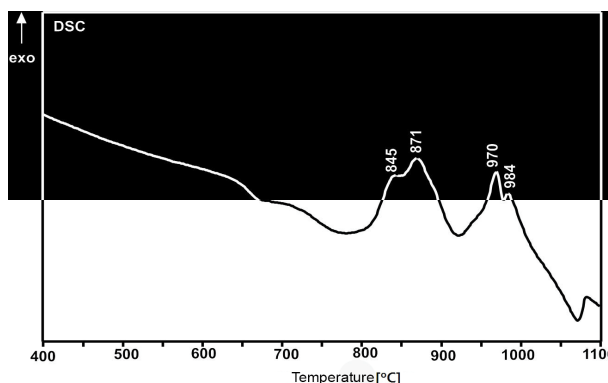


FIG. 1. DSC of the 3Mg glass.

After heating to identify the type of crystallizing phases, obtained materials were analyzed by XRD and MIR methods. Figure 2 presents X-ray pictures of 3Mg glass heated between 800-1000°C. The crystalline phases were  $\alpha$ -cristobalite,  $\beta$ -cristobalite, sodium magnesium silicate and sodium magnesium phosphate.

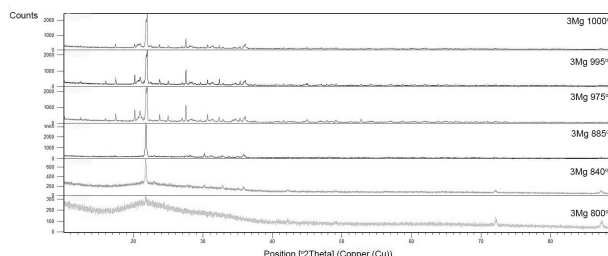


FIG. 2. XRD of the 3Mg glass after annealing at indicated temperatures.

## Conclusions

Structural studies of materials obtained after heating in gradient furnace shown that in the first stage cristobalite crystallized and then sodium magnesium silicate and sodium magnesium phosphate. Thus, based on the investigated glasses it is possible to obtain glass-crystalline materials by appropriate selection of crystallization conditions.

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