MINING AND METALLURGY INSTITUTE BOR

and

TEHNICAL FACULTY BOR, UNIVERSITY OF BELGRADE

IOC 2018

International October Conference

50th International October Conference
on Mining and Metallurgy

PROCEEDINGS

Editors:

Ana Kostov
Milenko Ljubojev

30th September – 3rd October 2018

Hotel "Jezero" Bor Lake, Serbia

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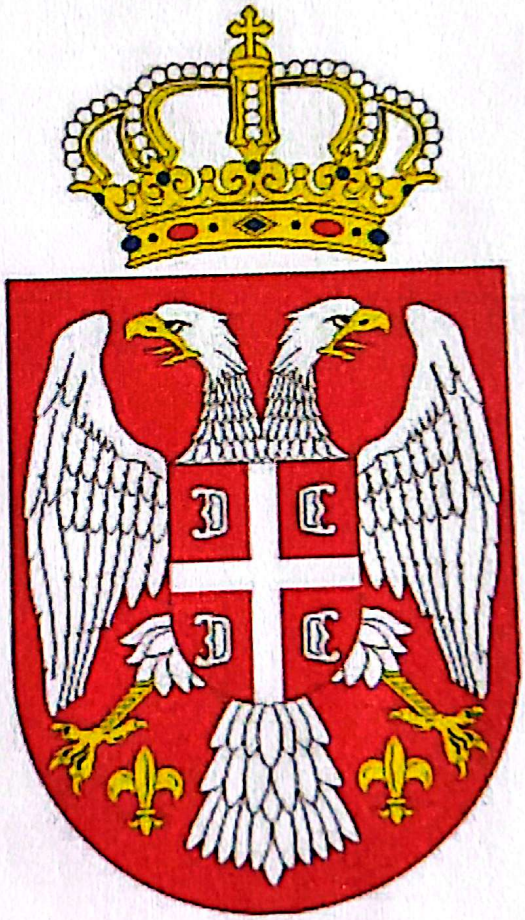

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LANTHANUM-DOPED PHOSPHATE GLASS FOR BIOMEDICAL APPLICATION

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Abstract

Glass-ceramic with the composition $42P_2O_5 \cdot 40CaO \cdot La_2O_3 \cdot 10Na_2O \cdot 3TiO_2$ (mol %) was obtained by the standard melt-quenching method. The crystallization and sintering behavior of glass have been studied using the DTA and XRD methods. The sintered phosphate glass-ceramic, containing bioactive β-CaP_2O_6, α-$Ca_3(PO_4)_2$ and β-$Ca_3(PO_4)_2$ phases, was successfully prepared.

Keywords: *polyphosphate glass, bioactive glass-ceramic, β-CaP_2O_6, α-$Ca_3(PO_4)_2$, β-$Ca_3(PO_4)_2$.*

1 INTRODUCTION

Due to their potential biomedical application, different types of phosphate based glasses have been extensively studied. [1-4]. Recently, they have been evaluated for the drug delivery applications and bone tissue engineering. They showed highly positive and promising results [5-7]. The addition of lanthanum to modified apatite was found to improve its biocompatibility and lower the cytotoxicity against osteoblast [8]. Also, lanthanum substituted β-tricalcium phosphate (β-$Ca_3(PO_4)_2$) was developed employing the precipitation technique. The anti-bacterial efficiency of modified β-$Ca_3(PO_4)_2$ was confirmed against both *Staphylococcus aureus* and *Escherichia coli* [9]. In this paper, the glass-ceramic with the composition $42P_2O_5 \cdot 40CaO \cdot La_2O_3 \cdot 10Na_2O \cdot 3TiO_2$ (mol %) was obtained by the standard melt-quenching method. The crystallization and sintering behavior of glass samples was analyzed using the DTA and XRD methods. The sintered phosphate glass-ceramic, containing bioactive β-CaP_2O_6, α-$Ca_3(PO_4)_2$ and β-$Ca_3(PO_4)_2$ phases, was successfully prepared.

2 EXPERIMENTAL

2.1 Synthesis of Starting Glass

The raw materials used for glass synthesis were the reagent grade $(NH_4)_2HPO_4$, Na_2CO_3, $CaCO_3$, La_2CO_3 and TiO_2. The appropriate batch composition was melted at 1250°C for 0.5 h in a Pt crucible, after which the melt was cast on a steel plate and cooled in the air. The glass sample was transparent, without visible residual gas bubbles. The powder X-ray diffraction analysis has confirmed the quenched melt to be vitreous.

2.2 Glass Crystallization Experiments

To examine the glass crystallization, the non-isothermal crystallization was studied using a DTA-Netzsch STA 409 EP instrument with Al_2O_3 powder as a reference material. The powder samples (100mg) were prepared by crushing and grinding the bulk glass in an agate mortar and sieving thus prepared specimen up to the appropriate grain size of less than 0.048 mm. The glass was heated from 20 to 800°C at a heating rate of $\nu = 10°C\ min^{-1}$.

The XRD technique was used to identify the phase composition of the crystallized bulk glass samples. Samples were crystalized at the appropriate temperature according to the DTA analysis. The XRD patterns were obtained using a Philips PW-1710 automated diffractometer with a Cu tube operated at 40 kV and 30 mA. The instrument was equipped with a diffracted beam curved graphite monochromator and Xe-filled proportional counter. The diffraction data were collected in the 2θ Bragg angle range of 5 - 70°, counting for 1 s.

3 RESULTS AND DISCUSSION

3.1 Differential Thermal Analysis

Figure 1 shows the DTA curve of powder glass sample (< 0.048 mm), recorded at a constant heating rate of $10°C\ min^{-1}$.

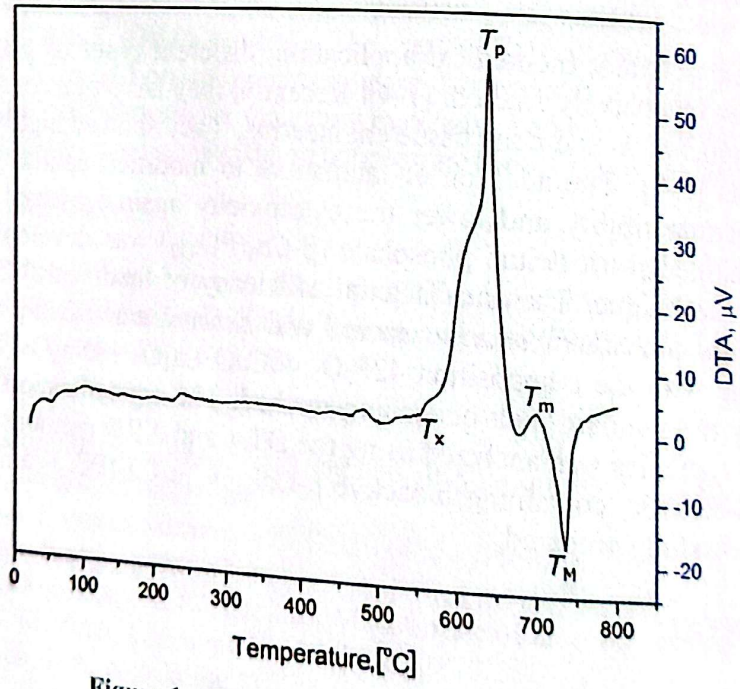

Figure 1 *DTA curve for the glass powder sample*

The glass crystallization was featured on the DTA curve by an exothermic peak with the onset crystallization temperature of $T_x = 585°C$ and maximum crystallization peak temperature $T_p = 640°C$. A well-defined endothermic peak, represented the melting of sample, was observed after the crystallization peak. The onset melting temperature of $T_m = 700°C$ and maximum peak temperature of $T_M = 735°C$ were marked on the DTA curve (Figure 1).

3.2 XRD Analysis

The bulk glass sample was isothermally heated at T = 640°C for 3 h. The phase composition of sample was determined by a XRD analysis and the results are presented in Figure 2. The XRD analysis of the powdered sample showed that during sintering the glass particles crystallized and determined crystalline phases are: β-CaP_2O_6, α-$Ca_3(PO_4)_2$, β-$Ca_3(PO_4)_2$, $Na_{1,8}Ca_{1,1}P_6O_{17}$ and $NaCa(PO_3)_3$. For β-CaP_2O_6, α-$Ca_3(PO_4)_2$ and β-$Ca_3(PO_4)_2$, the bioactivity, i.e. the ability to promote the formation of apatite (HAP) layer after reaction with the surrounding body fluid, has been reported [10].

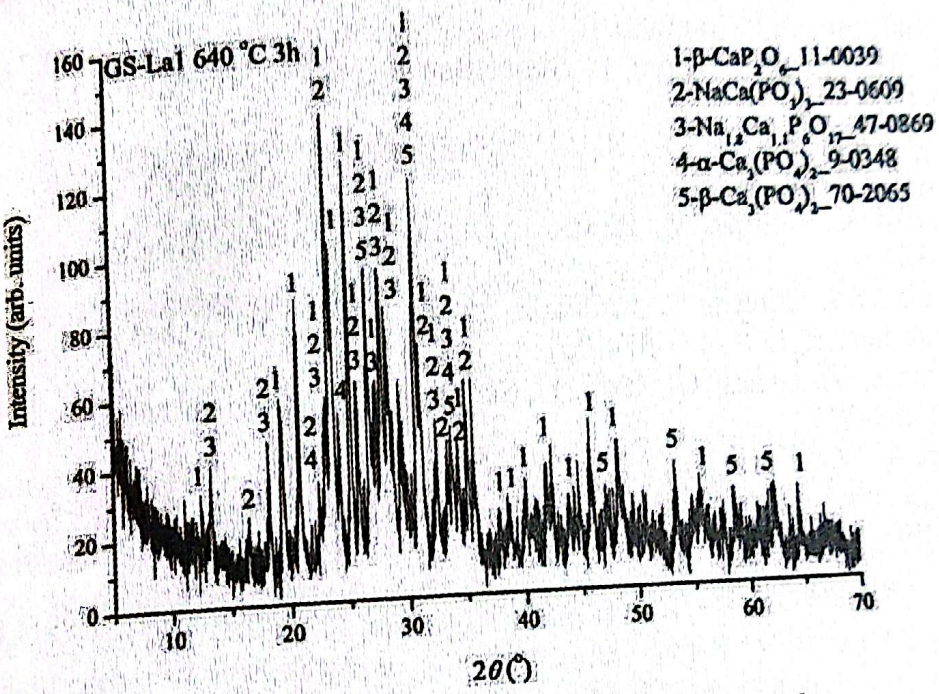

Figure 2 *XRD pattern of the sintered glass sample*

Tricalcium phosphate, (CaP_2O_6, TCP) and β-calcium phosphate (β-CaP_2O_6), found in the analyzed glass samples, are one of the most biologically compatible materials to the human bone in the bio-ceramics field. Also, the TCP can be used as a tissue replacement for repairing bony defects.

4 CONCLUSION

For synthesis of the sintered glass-ceramic, the parent $42P_2O_5 \cdot 40CaO \cdot La_2O_3 \cdot 10Na_2O \cdot 3TiO_2$ (mol %) glass was prepared by the standard melt-quenching method. The glass-ceramic, containing three bioactive crystalline phases, β-CaP_2O_6, α-$Ca_3(PO_4)_2$ and β-$Ca_3(PO_4)_2$, was prepared by heating the compacted glass powder at T = 640°C for 3h. The crystalline phases, containing La and Ti, were not detected. The obtained phase composition of the glass-ceramic has indicated its possible application as a bioactive material for the bone tissue engeenering.

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