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Nada Štrbac
Dragana Živković

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on Mining and Metallurgy

Editors:
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University of Belgrade - Technical Faculty in Bor
Prof. dr Dragana Živković
University of Belgrade - Technical Faculty in Bor

Technical Editors:
Doc. dr Ljubiša Balanović
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3D BIOACTIVE PHOSPHATE GLASS-CERAMIC SCAFFOLDS PREPARED BY THE FOAM REPLICA TECHNIQUE

Vladimir Topalović[1], Srđan Matijašević[1], Jelena Nikolić[1], Marija Đošić[1], Snežana Grujić[2], Sonja Smiljanić[2]

[1]Institute for the Technology of Nuclear and Other Mineral Raw Materials, 86 Franchet d' Esperey St, 11000 Belgrade, Serbia

[2]Faculty of Technology and Metallurgy, University of Belgrade, Karnegijeva 4, 11000 Belgrade, Serbia

Abstract

Glass-ceramic scaffolds similar to bone structure were obtained by the foam replica technique using a polyurethane (PU) foam and powdered polyphosphate glass containing strontium. The parent glass was prepared by the standard melt-quenching method. Polyurethane foam coated with glass particles (< 5μm) was thermally removed and the glass was sintered during 1h at 650 ^{0}C. The microstructure, morphology and phase composition of the as-prepared glass-ceramic scaffolds were investigated using the SEM and XRD methods. 3-D porous glass-ceramic scaffold containing bioactive β-$Ca_3(PO_4)_2$ and β-$Ca_2P_2O_7$ crystalline phases was fabricated.

Keywords*: bioactive glasses, glass-ceramic scaffolds, foam replica technique, β-$Ca_3(PO_4)_2$,, β-$Ca_2P_2O_7$*

1. INTRODUCTION

A number of techniques including starch consolidation, incorporation of volatile organic particles, foaming and replication of a polymeric sponge have been developed to fabricate 3 D bioactive porous glass–ceramic scaffolds for application in bone tissue engineering [1-5]. Among these techniques, the foam replication method revealed a number of advantages that enable to replicate the natural macroporous structure of the bone tissue [6]. By this method the scaffolds are prepared by sintering of the powders of bioactive glasses previously coated on polyurethane (PU) foam which serves as a sacrificial template. Beside the bioactive silicate glasses (Hench glasses), the polyphosphate-based glasses derived from a base P_2O_5-CaO-Na_2O glassy system became a promising material for such purpose. These glasses are preferable than silica-based glasses because of their more controllable dissolution rate [7, 8]. Usually, the properties of a base phosphate glass including bioactivity is regulated by addition of small content of different oxides as a TiO_2. [9, 10]. Also, a small content of metallic cations (Sr, Zn, Cu, Ag) with therapeutic effect in bone healing can be added [11]. In this paper, the 3D macroporous bioactive glass–ceramic scaffold based on $42P_2O_5$•40CaO•5SrO•$10Na_2O$•$3TiO_2$ (mol %) glass was prepared by PU foam replication technique and analyzed.

2. EXPERIMENTAL

2.1 Synthesis of starting glass

The raw materials used for glass synthesis were reagent grade $(NH_4)_2$ HPO_4, Na_2CO_3, $CaCO_3$, $SrCO_3$ 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 air. The glass samples were transparent, without visible residual gas bubbles. Powder X-ray diffraction analysis confirmed the quenched melts to be vitreous. In order to obtain desired particle size (< 5μm), the glass

sample was ground at 400 rpm during 1h in a SFM-1 Desk-Top Planetary Ball Miller, MTI Corporation.

2.2 Scaffolds fabrication

The polymeric template used for scaffolds preparation was a commercial PU sponge, Vapen S (apparent density ~20 kg m^{-3}). The polymer was cut into 10.0 x 10.0 x 10.0 mm^3 cubes and then soaked into a water-based slurry. The weight composition of slurry was: 30% glass, 64% distilled water and 6% polyvinyl alcohol (PVA), which was used as binding agent to optimize the ability of glass particles to uniformly coat the template. The sponge cubes were soaked into the glass slurry for 60 s, taken back and compressed (20 kPa for 1 s) to remove the exceeding slurry. This infiltration-compression process was repeated for several times. Finally, the samples were dried at room temperature for 6 h and thermally treated in order to remove the organic phase and to sinter the glass particles, thus obtaining macroporous glass-ceramic scaffolds. The sample was heated in an electric furnace Carbolite CWF 13/13 with automatic regulation and temperature accuracy of ± 1 ºC at a rate of 10 °C min^{-1} up to T = 650 ^{0}C , maintained at this temperature for 1h and then cooled. The sintering temperature for glass was chosen according to DTA and HSM experiment performed previously [12].

2.3 Scaffolds characterization

Scaffolds structure and morphology were analysed by optical and scanning electron microscopy (SEM). Stereo microscope (EU Instruments) and a scanning electron microscope (MIRA X 3 TESCAN) were employed. The samples for SEM analysis were gold coated in a Leica SCD005 device. The phase composition of the sintered scaffold was determined by XRD analysis using a Philips PW-1710 automated diffractometer with Cu tube operated at 40 kV and 30 mA.

3. RESULTS AND DISCUSSION

3.1 Scaffolds structural and morphological characterization

Figure 1 shows the structure of PU sponge, used as scaffolds template that exhibits 3-D network of pores, which vary in size from 100 to 600 μm. In Figure 2, the SEM micrographs of PU skeleton coated with glass particles are shown. As may be seen in Figure 3, the morphology of the scaffold sintered at T = 650 ^{0}C for 1h remains higly porous without significant decrease of the pore size. The images revealed that the pore struts are well sintered.

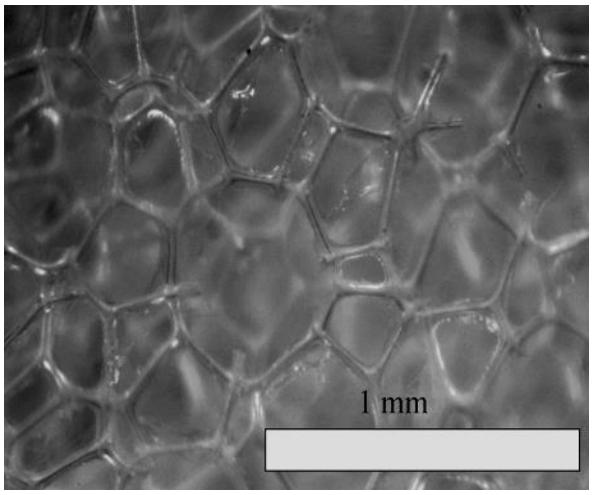

Figure 1 - Bare PU sponge

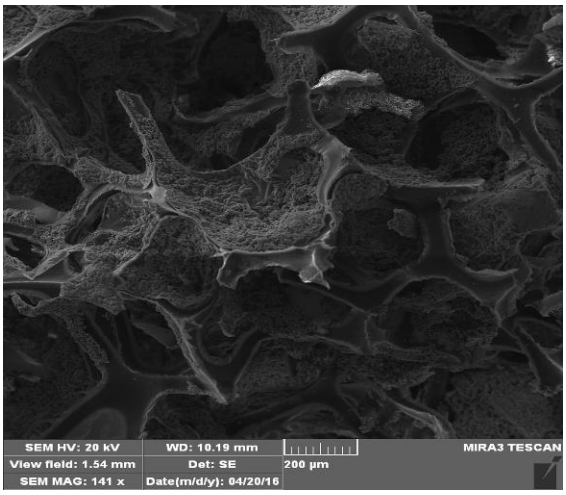

Figure 2 - SEM micrographs of PU sponge covered with glass particles

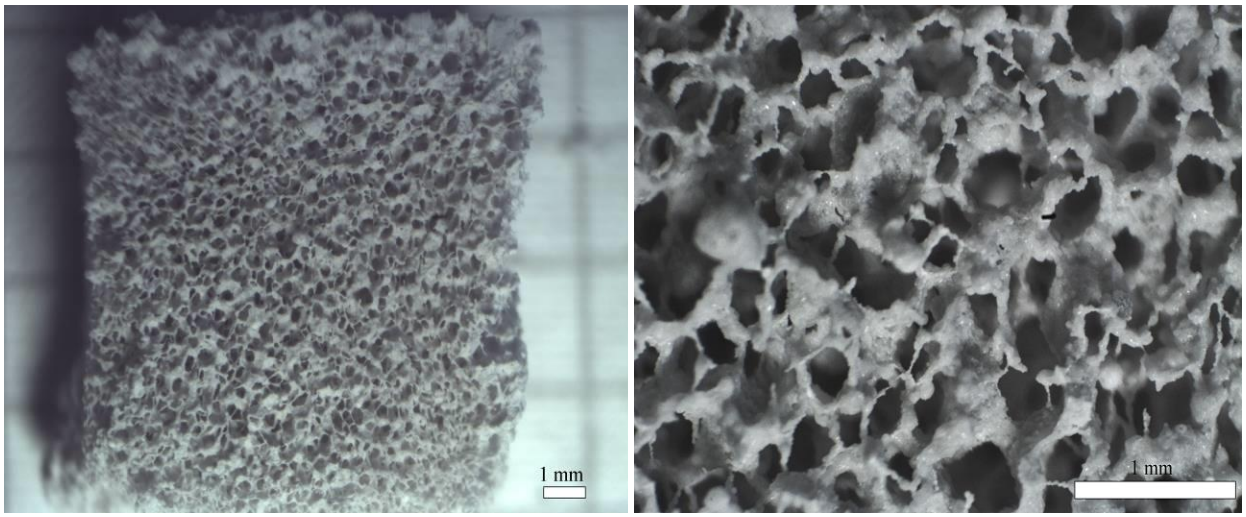

Figure 3 - Sintered glass-ceramics scaffold structure

XRD analysis of the powdered scaffold showed that during sintering the glass particles crystallized and the crystalline phases determined are: $Ca(PO_3)_2$, β-$Ca_3(PO_4)_2$, α-$Ca_2P_2O_7$ and β-$Ca_2P_2O_7$ (Figure 4). For β-$Ca_3(PO_4)_2$ and β-$Ca_2P_2O_7$ the bioactivity, i. e. the ability to promote the formation of apatite (HAP) layer after reaction with the surrounding body fluid has been reported [13].

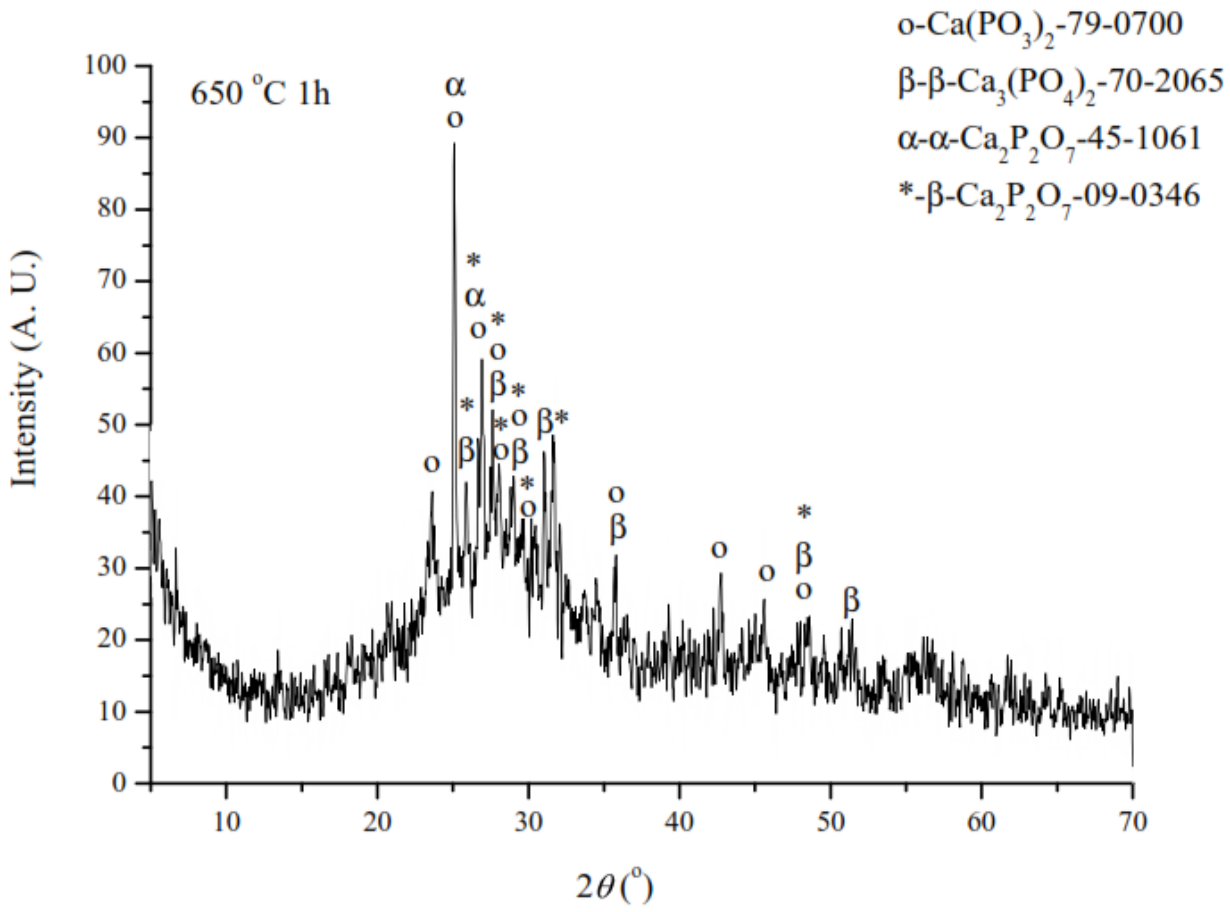

Figure 4 - XRD pattern of the sintered scaffold

4. CONCLUSIONS

For fabrication of 3D glass-ceramics scaffold the parent polyphosphate glass $42P_2O_5$•40CaO•5SrO•$10Na_2O$•$3TiO_2$ (mol %) was prepared by standard melt-quenching method. A commercial polyurethane (PU) sponge as a template and glass powder grain size (< 5μm) were used. Highly porous 3D glass-ceramics scaffold containing bioactive crystalline phases, β-$Ca_3(PO_4)_2$ and β-$Ca_2P_2O_7$ was fabricated by sintering of glass powder at T = 650 ^{0}C for 1h. The crystalline phases contained Sr and Ti were not detected. The obtained phase composition and the microstructure of as-prepared scaffold indicated its possible application as a bioactive material for bone tissue engeenering.

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