

08 Nov 2013

An Investigation into the Structure and Properties of CaO-ZnO-SiO₂-TiO₂-Na₂O Bioactive Glass/hydroxyapatite Composite


Chokchai Yatongchai

Mark R. Towler

Missouri University of Science and Technology, mtowler@mst.edu

Anthony W. Wren

Follow this and additional works at: https://scholarsmine.mst.edu/che_bioeng_facwork

 Part of the [Biochemical and Biomolecular Engineering Commons](#), and the [Biomedical Devices and Instrumentation Commons](#)

Recommended Citation

C. Yatongchai et al., "An Investigation into the Structure and Properties of CaO-ZnO-SiO₂-TiO₂-Na₂O Bioactive Glass/hydroxyapatite Composite," *Proceedings of the IEEE Annual Northeast Bioengineering Conference, NEBEC*, pp. 102 - 103, article no. 6574378, Institute of Electrical and Electronics Engineers, Nov 2013.

The definitive version is available at <https://doi.org/10.1109/NEBEC.2013.51>

This Article - Conference proceedings is brought to you for free and open access by Scholars' Mine. It has been accepted for inclusion in Chemical and Biochemical Engineering Faculty Research & Creative Works by an authorized administrator of Scholars' Mine. This work is protected by U. S. Copyright Law. Unauthorized use including reproduction for redistribution requires the permission of the copyright holder. For more information, please contact scholarsmine@mst.edu.

An Investigation into the Structure and Properties of CaO-ZnO-SiO₂-TiO₂-Na₂O Bioactive glass/ Hydroxyapatite Composite

Chokchai Yatongchai, Mark R. Towler, Anthony W. Wren.

Inamori School of Engineering

Alfred University

Alfred, New York

cy3@alfred.edu

Abstract— Effect of the addition of CaO-ZnO-SiO₂-TiO₂-Na₂O glass as a reinforcing phase on properties of hydroxyapatite/glass composites was investigated. The addition of the glass caused the decomposition of HA phase to tricalcium phosphate, inhibiting densification in the HA/glass composites. However, depending on the glass concentration, some of the glass can act as a sintering aid enhancing the densification in the composites.

Keywords—hydroxyapatite; calcium zinc silicate; glass; composite.

I. INTRODUCTION

Hydroxyapatite (HA) is widely investigated bioceramic for medical and dental applications because it has good biocompatibility [1]. HA is chemically similar to the mineral phase of bone and is capable of direct-bonding [2]. However, its mechanical properties are currently the limiting factor in its widespread utilization. Attempts have been made to improve the mechanical properties of HA. A suitable method of improving mechanical properties of HA is based on the synthesis of composites which are made of HA and bioactive glasses. Glasses are considered to be good candidates for the HA matrix composites because they usually have quite low softening point enhancing densification in HA composites. Calcium zinc silicate glass (CaO-ZnO-SiO₂) is a bioactive glass formulated for use in orthopedic applications [3]. The release of zinc ions from zinc-based glass has the ability to increase DNA synthesis in osteoblasts, resulting in increased bone mass in osteoporotic patients. In this work, the effect of the addition of a CaO-ZnO-SiO₂-TiO₂-Na₂O glass as a reinforcing phase on properties of the HA composites was investigated.

II. MATERIALS AND METHODS

Hydroxyapatite powder with Ca/P ratio of 1.667 was produced at a synthesis temperature of 25 °C based on a chemical precipitation method [4]. 40 g of calcium nitrate hydrate (Ca(NO₃)₂·4H₂O) in 304 ml of water was made basic by the addition of ammonium hydroxide (NH₄OH). The mixture was stirred vigorously and heated to the temperature of 25°C.

13.42 g of di-ammonium hydrogen orthophosphate ((NH₄)₂HPO₄) was made basic in deionized water by the addition of 13 ml of NH₄OH. The Ca(NO₃)₂·4H₂O and (NH₄)₂HPO₄ solutions were brought to 25°C and stirred vigorously to ensure the reagents were completely dissolved. Under continued stirring, the (NH₄)₂HPO₄ solution was drop-wise from a glass funnel into the Ca(NO₃)₂·4H₂O solution. Throughout synthesis, the pH was kept above 10.0 by addition of NH₄OH at constant intervals. The precipitate was then left to stand for 24 h. the supernatant was removed and replaced with fresh deionized water. This procedure was undertaken three times to remove any unwanted residue from the precipitation. The suspension was then filtered under vacuum. The filter cake was dried in an oven at 75°C for 24 h.

A CaO-ZnO-SiO₂-TiO₂-Na₂O glass with composition shown in Table 1 was prepared by conventional quench method. A glass batch was melted at 1500°C for 1 hour in a platinum crucible and then quenched into water. The resulting frit was dried and ground to obtain a glass powder (<45µm). The glass ranging from 5 to 25 wt% was added and then the powders were compacted uniaxially. The compacts were sintered at temperature from 1000-1200°C for 2 hours.

TABLE I. GLASS COMPOSITION (MOL%)

SiO ₂	TiO ₂	Na ₂ O	ZnO	CaO
0.38	0.12	0.17	0.28	0.05

For X-ray diffraction analysis, sintered samples were analyzed using a Siemens D5000 diffractometer. Apparent porosities of the sintered samples were performed in water using the Archimedes principle. The biaxial flexural strength was determined to find characteristic strength of the composites.

III. RESULTS AND DISCUSSION

Fig. 1 shows XRD patterns for HA/glass composites sintered at 1200°C. β-TCP peaks appear at 5wt% and the intensity of the peaks increases with the addition of glass. In contrast, the intensity of HA peaks decreases

with increasing glass content. This indicates that calcium zinc silicate glass causes decomposition of HA to β -TCP. It is also found that the peaks which belong to Ca_2SiO_4 and $\text{Na}_2\text{Si}_2\text{O}_5$ appear at 25wt%. This is due to the crystallization of the calcium zinc silicate system.

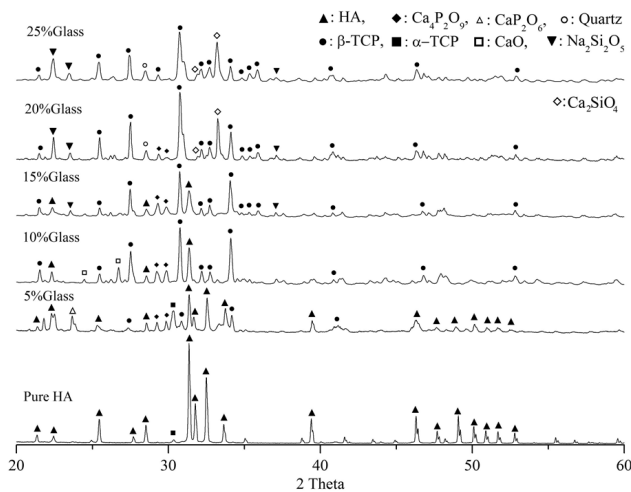


Figure 1. X-ray diffraction patterns for HA/glass composites sintered at 1200°C.

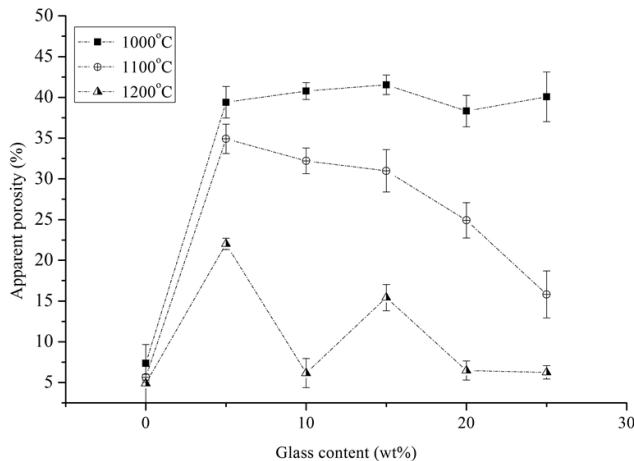


Figure 2. Apparent porosity of HA/glass composites sintered at different temperatures.

Fig. 2 shows apparent porosity of the HA/glass composites. The porosity dramatically increases at 5 wt% glass and then decreases with increasing the glass content. The increase in the porosity may have been due to the occurrence of β -TCP, α -TCP and CaP_2O_6 , which have lower density compared to HA. This could inhibit the densification leading to the increase in

porosity. However, with increasing the glass content, some of the glass can act as a sintering aid enhancing the densification leading to the decrease of the porosity.

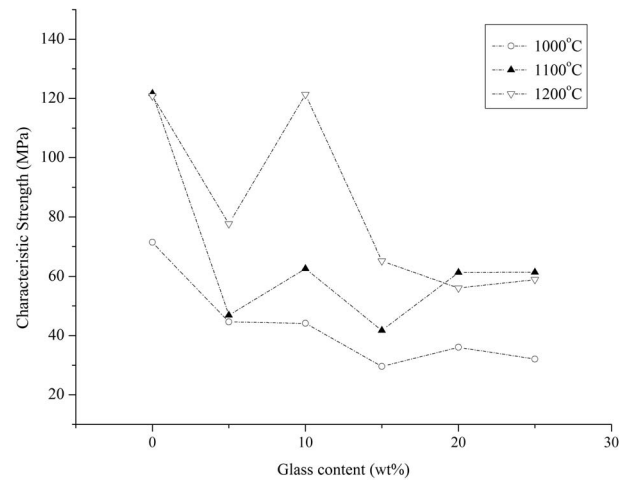


Figure 3. Characteristic strength of HA/glass composites sintered at different temperatures.

Fig. 3 shows the characteristic strength of the HA/glass composite. The presence or absence of porosity in ceramics significantly affects the mechanical properties. A great increase in porosity at 5wt% glass causes a sharp decrease of the strength. By increasing the glass content from 10-25 wt%, the strength of the composites should have increased. However, in this system, it is further complicated by the occurrence of decomposition and phase changes that can also have a detrimental effect on the mechanical properties.

REFERENCES

- [1] Hench, L.L. and J. Wilson, An Introduction to bioceramics. Advanced series in ceramics1993, Singapore ; River Edge, N.J.: World Scientific. x, 386 p.
- [2] Mobasherpour, I., et al., Synthesis of nanocrystalline hydroxyapatite by using precipitation method. Journal of Alloys and Compounds, 2007. 430(1-2): p. 330-333.
- [3] Hien, N., Glass-Ionomer Cements as Restorative and Preventive Materials. Dental Clinics of North America, 2010. 54(3): p. 551-563.
- [4] Curran, D.J., et al., Mechanical parameters of strontium doped hydroxyapatite sintered using microwave and conventional methods. Journal of the Mechanical Behavior of Biomedical Materials, 2011. 4(8): p. 2063-2073.