Journal of the Mechanical Behavior of Biomedical Materials 86 (2018) 77–83

Contents lists available at ScienceDirect

Journal of the Mechanical Behavior of Biomedical Materials

journal homepage: www.elsevier.com/locate/jmbbm

Evaluation of the micro-mechanical and bioactive properties of bioactive glass-dental porcelain composite

Anastasia Beketova[a], Savvas Varitis[b], Eleana Kontonasaki[a], Triantafillia Zorba[b], Lambrini Papadopoulou[c], Nikolaos Kantiranis[c], Georgios S. Theodorou[b], Konstantinos M. Paraskevopoulos[b], Petros Koidis[a,*]

[a] School of Health Sciences, Dentistry Department, Aristotle University of Thessaloniki, Thessaloniki 54124, Greece
[b] Physics Department, Aristotle University of Thessaloniki, Thessaloniki 54124, Greece
[c] Department of Geology, Aristotle University of Thessaloniki, Thessaloniki 54124, Greece

ARTICLE INFO

Keywords:
Bioceramic composites
Sol-gel synthesis
Mechanical testing
Microindentation
Knoop microhardness
Elastic modulus
Bioactivity

ABSTRACT

The aim of this study was to evaluate microhardness and elastic modulus of a novel sol-gel derived dental ceramic − 58S bioactive glass composite (BP67: Bioactive Glass:33.3%, Dental Ceramic:66.7%) BP67[1] material by micro-indentation and to investigate its microstructure and bioactivity. The research hypotheses were that the values of microhardness (1) and elastic modulus (2) of the novel bioceramic composite and the commercial dental ceramic will be of the same order. The experimental sol-gel derived ceramics showed similar microstructural characteristics to a commercial feldspathic porcelain, and presence of additional calcium phosphate phases, which contributed its bioactivity. The formation of an apatite-like layer on the materials' surface observed by Fourier Transform Infrared (FTIR)[2] spectroscopy, X-ray Diffraction (XRD)[3] and Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS)[4] techniques after 12 days of maintenance in Conventional Simulated Body Fluid (cSBF)[5] solution. The BP67 exhibited values of microhardness and modulus of elasticity which were not statistically significant different compared to dental ceramic, indicating the adequate mechanical integrity of the material. The results of this study suggest that the novel bioactive composite could be potentially applied in prosthetic dentistry, while its thermal and optical properties should be investigated in future studies.

1. Introduction

Bioactive Glass/Porcelain (B/P) composites have been proposed for their potential use as veneering materials at the marginal area of Metal Ceramic restorations (MCR) (Kontonasaki et al., 2003; Chatzistavrou et al., 2010; Goudouri et al., 2014). These materials contain bioactive glass, which is known for its excellent biocompatibility and extensive use for tissue regeneration (Jones et al., 2016). In body fluids, bioactive ceramics can form a layer of non-stoichiometric biological hydroxyapatite (HA) on their surface, which promotes further cellular steps and their direct bonding to the adjacent bone or, even to soft tissues (Hench et al., 1998; Hupa and Karlsson, 2016). Such hydroxyapatite surface in the marginal area of MCR, in an attempt of mimicking dental cementum, could provide attachment and proliferation of specific cell types of the marginal periodontium leading to periodontal tissue reattachment over the margins of prosthetic restoration (Chatzistavrou et al., 2012; Kokoti et al., 2001; Kontonasaki et al., 2007). As a result, the achievement of a complete biological sealing of the marginal gap could improve the longevity of fixed dental restorations (Montazerian and Zanotto, 2017). Moreover, it has already been shown that bioactive glass modified dental ceramics enhanced attachment and proliferation of human periodontal ligament fibroblasts in vitro (Kontonasaki et al., 2007).

Among various compositions, B/P materials with a high dental ceramic content (60–80 wt%) seem to be very promising since they could to combine both apatite forming ability and sufficient mechanical

* Correspondeing author.
E-mail address: pkoidis@dent.auth.gr (P. Koidis).

[1] BP67- bioactive glass 33.3%-dental porcelain 66.7% composite.
[2] FTIR-Fourier Transform Infrared
[3] XRD-X-ray Diffraction.
[4] -SEM-EDS- Scanning Electron Microscopy-Energy Dispersive Spectroscopy.
[5] cSBF- Conventional Simulated Body Fluid.

https://doi.org/10.1016/j.jmbbm.2018.06.019
Received 11 May 2018; Received in revised form 8 June 2018; Accepted 12 June 2018
1751-6161/

properties (Goudouri et al., 2011a). Several authors investigated flexural strength and Weibull modulii of such B/P materials by conventional 3-point bending tests, and showed the linear relationship between the material's strength and the increase of the amount of dental porcelain (Goudouri et al., 2011a; Abbasi et al., 2016). However, the disadvantages of this technique are large specimens' dimensions, which are far away from clinical reality and susceptibility of such ceramic specimens to flaws and defects that could also affect the results of the measurements (Chung et al., 2005).

On the other hand, it was reported the micro-indentation tests are appropriate for studying of small-scale mechanical properties of brittle ceramic materials. The Knoop micro- indentation is one the most the most commonly used techniques, providing information about material's hardness and elastic modulus, required for clinical applications (Marshall et al., 1982). In particular, hardness is defined as resistance to plastic deformation under contact loads. The reported values of hardness of dental enamel range from 3 to 6 GPa, depending on its location, chemical composition and prism orientation (Cuy et al., 2002; Mahoney et al., 2000). The elastic modulus, or Young's modulus is a ratio of stress to the corresponding degree of deformation in the elastic region, which for enamel is between 70 and 100 GPa (Park et al., 2008)

Based on aforementioned the objective of this study was to evaluate microhardness and elastic modulus of a novel BP67 material by micro-indentation. An additional purpose of this study was to investigate its microstructure and bioactivity. The research hypothesis were that the values of microhardness (1) and elastic modulus (2) of the novel bioceramic composite and the commercial dental ceramic will be of the same order.

2. Materials and methods

The experimental procedures included the preparation and characterization of experimental bioactive glass-dental ceramic composite material BP67, containing 33.3 wt% of 58 S bioactive glass and 66.7 wt % of dental ceramic (IPS Inline Margin, Ivoclar) and its bioactivity control. Further, fabrication of square shaped (10 mm × 10mm × 1.5 mm) sintered specimens made of BP67 composite and control specimens made of commercial ceramic for margins (P) for evaluation of their microhardness and elastic moduli followed. A total of 20 specimens were fabricated, 10 per group.

2.1. Preparation of BP67 material by sol-gel method and fabrication of ceramic specimens

The BP67 material was prepared by sol-gel method by mixing in order of 9.5 ml distilled water, 1.6 ml nitric acid, 13.2 ml tetraethoxysilane (TEOS), 0.7 ml tri-ethyl-phosphate (TEP) and 5.8 gr of calcium nitrate tetra-hydrate ($Ca(NO_3)_2$ $4H_2O$), as described for preparation of bioactive glass 58 S (SiO_2 60 wt%, CaO 36 wt% and P_2O_5 4 wt%) (Zhong and Greenspan, 2000; Goudouri et al., 2011b). Dental porcelain powder (IPS InLine Margin, Ivoclar, Schaan Liechtenstein) was added in a ratio of 66.7%. After mixing of the components, the sol was sintered at 60 °C for 55 h, dried, and underwent firing cycle up to 700 °C in a programmable furnace (Nabertherm, Germany), so that the material remained at the stabilization temperature (700 °C) for 18 h. The resulting products were pulverized and sieved to powders of < 40 μm.

For the fabrication of the specimens, the powders of BP67 and dental ceramic were mixed with modeling liquid (IPS Classic Margin Build-Up Liquid, Ivoclar), using the same liquid to powder ratio for each material (0.335), transferred into silicone moulds, condensed using a vibrator, in order to remove the excess of liquid and dried with warm air. The square specimens were taken out by gentle hand pressure and sintered in oven Programat P95-Ivoclar (Schaan, Liechtenstein) at 930 °C according to manufacturers' instructions of dental porcelain. Samples were polished by grit silicon carbide 600–2000 suspended in water on a glass plate and then by polishing diamond paste of 6 μm, 3 μm and 1 μm by Strauers A/S (Denmark) polishing device with rotary speed 150 rpm.

2.2. Identification of microstructure of the BP67 materials powder and sintered specimens

2.2.1. FTIR analysis

The FTIR transmittance spectra of the BP67 material powders before and after sintering were obtained by the KBr technique using a Perkin-Elmer Spectrometer Spectrum 1000 in MIR region (4000–400 cm^{-1}) with a resolution 4 cm^{-1} and a number of 32 scans per spectrum.

2.2.2. XRD analysis

The XRD analysis of BP67 powders and crushed sintered specimens was performed using a Philips PW1710 diffractometer with Ni-filtered CuKα radiation on the surface of the studied samples. The counting statistics were: start angle 5°, end angle 75° (2θ), step size 0.05° (2θ), time per step 1 s and scan speed 0.01°2θ/s. The ICDD PDF-4 + (2009) database was used for the identification of the phases contained in the studied samples. The semi-quantitative estimation of the abundance of the phases was derived from the XRD data, using the intensity of a certain reflection for each phase, while the percentage of amorphous material was achieved by comparing the area of each broad background hump representing the amorphous material in samples with the analogous area in XRD patterns of standard mixtures of minerals and different contents of natural amorphous material scanned under the same conditions (Kantiranis et al., 2004).

2.3. Evaluation of bioactive behavior

The bioactive behavior of BP67 powders was tested through their immersion in conventional Simulated Body Fluid (SBF) solution which is a common technique for assessment of in vitro bioactivity of materials (Kokubo and Takadama, 2006). SBF was prepared by weighing and dissolving NaCl, $NaHCO_3$, KCl, $K_2HPO_4{\cdot}3H_2O$, $MgCl_2{\cdot}6H_2O$, $CaCl_2$, and $NaSO_4$ in distilled water, as previously described (Kokubo and Takadama, 2006). The pH of the solution was adjusted to 7.3–7.4 at a temperature of 36.5 °C in a water bath by the addition of 1.0 M hydrochloric acid (HCl) and tris-hydroxymethylaminomethane $(CH_2OH)_3CNH_2$.

The powder specimens (triplicates) were immersed in the SBF solution at 37 °C with powder to liquid ratio 1 mg/1 ml. The solution was renewed after 6 h, 24 h and then every 2 d. (Zhong and Greenspan, 2000). After 1 d, 3 d, 6 d, 9 d and 12 d of maintenance in solution the specimens were removed, rinsed with acetone and distilled water and characterized by FTIR transmittance and XRD. For topographical evaluation and analysis of surface elemental composition the specimens were carbon coated with an average thickness of 200 Å, using a vacuum evaporator JEOL-4 × and were observed using a Scanning Electron Microscope (JEOL J.S.M. 840A, Tokyo, Japan) with associated EDS (Oxford INKA-300). All analyses were done with a 20 kV accelerating voltage and 0.4 mA probe. The images were taken in magnification x1000.

2.4. Mechanical properties evaluation

2.4.1. Microhardness

Microhardness was determined using Knoop indenter geometry using an Anton Paar MHT-10 microhardness tester attached on a Zeiss Axiolab-A metallographic microscope. The indentation load was such as to produce crackles and easily measureable indentation prints. For each sample group a number of 20 indentations were performed at room temperature, 2 indentations per sample. The load was set at 1 N, contact time 10 s, slope 0.2 N/s and Knoop hardness was determined using the following equation: