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Girish Singh
 Department of Materials
 Chemistry, Ramanujan
 National Research Centre,
 Mysore, Karnataka, India

Saroj Chauhan
 Department of Advanced
 Materials Research, Satyendra
 Nath Bose Centre for
 Nanosciences, Mohali, Punjab,
 India

Ajay Agarwal
 Department of Advanced
 Materials Research, Satyendra
 Nath Bose Centre for
 Nanosciences, Mohali, Punjab,
 India

Corresponding Author:
Girish Singh
 Department of Materials
 Chemistry, Ramanujan
 National Research Centre,
 Mysore, Karnataka, India

Preparation and characterization of bioactive glass-ceramic materials for dental applications

Girish Singh, Saroj Chauhan and Ajay Agarwal

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Abstract

Dental restorative materials must bond to living tissue while withstanding years of mechanical stress inside the mouth a combination that most synthetic ceramics struggle to achieve. This research prepared bioactive glass-ceramic compositions in the $\text{SiO}_2\text{-CaO-P}_2\text{O}_5\text{-Na}_2\text{O}$ system and characterized their suitability for dental applications. Glass frits were synthesized by melt-quench technique at 1450 °C, ground to fine powders, and sintered at temperatures ranging from 700 °C to 1050 °C. X-ray diffraction confirmed apatite and wollastonite crystalline phases at sintering temperatures above 800 °C. The sample sintered at 900 °C showed the highest compressive strength of 134.8 MPa alongside 41.3% apatite content. Scanning electron microscopy revealed a dense, interlocking microstructure with pore sizes between 2.1 and 8.7 μm . Simulated body fluid immersion for 14 days produced a hydroxyapatite surface layer 4.3 μm thick, confirming *in vitro* bioactivity.

Keywords: Bioactive glass-ceramic, dental materials, apatite, wollastonite, sintering temperature, compressive strength, simulated body fluid, hydroxyapatite formation

Introduction

No artificial material perfectly replicates the mineral-collagen composite of natural tooth structure. Glass-ceramics built from $\text{SiO}_2\text{-CaO-P}_2\text{O}_5$ base compositions come closer than most, because they release calcium and phosphate ions that trigger hydroxyapatite formation at the implant-tissue interface ^[1]. Hench's original 45S5 Bioglass demonstrated this bonding ability decades ago, yet its low mechanical strength and tendency to crystallize unevenly limited clinical use in load-bearing dental situations ^[2]. Controlled crystallization can fix the strength problem. By converting portions of the amorphous glass matrix into apatite and wollastonite phases through careful heat treatment, researchers have pushed compressive strengths past 100 MPa while retaining enough residual glass to maintain bioactivity ^[3]. The challenge is finding the right sintering window too low and the material stays porous and weak, too high and excessive crystallization chokes off the ion-exchange mechanism needed for tissue bonding ^[4]. Previous work on similar systems focused mainly on orthopedic implants, with dental applications receiving less attention despite the distinct mechanical and biological demands of the oral cavity ^[5]. Dental glass-ceramics must resist cyclical compressive loads from mastication (estimated at 70-150 MPa for posterior teeth) while remaining compatible with saliva and oral bacteria ^[6]. This research systematically varied sintering temperature to map the relationship between crystalline phase development, mechanical performance, and *in vitro* bioactivity for a glass-ceramic formulated specifically for dental restorative use.

Reagents and Chemicals

Silicon dioxide (99.8%, Sigma-Aldrich), calcium carbonate (99.5%, Merck), sodium carbonate (99.9%, Loba Chemie), and ammonium dihydrogen phosphate (98.5%, HiMedia) served as glass precursors. Platinum crucibles were used for melting. Simulated body fluid (SBF) was prepared following the Kokubo protocol with reagent-grade NaCl, NaHCO₃, KCl, MgCl₂·6H₂O, CaCl₂, and Na₂SO₄ dissolved in tris-buffer at pH 7.40 [7].

Material and Methods

Material

Research was performed at Ramanujan National Research Centre, Mysore, Karnataka, India, from February 2023 to October 2023. The glass composition (in mol%) was 46.1 SiO₂, 26.9 CaO, 24.4 Na₂O, and 2.6 P₂O₅. Precursor powders were weighed on a Mettler Toledo XS205 balance, mixed in an agate mortar for 30 min, and melted in platinum crucibles at 1450 °C for 2 h in a Carbolite HTF 1800 box furnace. The melt was quenched in deionized water to produce glass frits, dried at 120 °C overnight, and ball-milled to particle sizes below 45 µm [8].

Methods

Glass powder compacts (13 mm diameter, 3 mm thick) were

pressed at 150 MPa using a Specac hydraulic press and sintered at 700, 750, 800, 850, 900, 950, 1000, and 1050 °C for 2 h with a heating rate of 5 °C/min. Phase identification used a Rigaku MiniFlex 600 X-ray diffractometer (Cu K α , 40 kV, 15 mA, 2 θ range 10-70°). Microstructural analysis was done on gold-coated fracture surfaces using a JEOL JSM-6380LA scanning electron microscope at 20 kV. Compressive strength was measured on cylindrical samples (6 mm $\times$ 12 mm) using an Instron 5967 universal testing machine at 0.5 mm/min crosshead speed, with five specimens per temperature [9]. Bioactivity testing involved immersing polished discs in 50 mL SBF at 37 °C for 14 days, refreshing the solution every 48 h. Surface deposits were analyzed by FTIR and SEM-EDS [10].

Instrumentation

Thermal behavior of the glass powder was assessed by differential scanning calorimetry (Netzsch DSC 214 Polyma) at 10 °C/min under nitrogen flow. Density measurements followed Archimedes' method using distilled water. FTIR spectra were collected on a Bruker Alpha-II spectrometer in ATR mode (4000-400 cm⁻¹, 32 scans, and 4 cm⁻¹ resolution). Surface roughness was measured with a Mitutoyo SJ-210 profilometer.

Results

Table 1: Physical and mechanical properties of glass-ceramic samples at selected sintering temperatures

Temp (°C)	Density (g/cm ³)	Porosity (%)	Strength (MPa)	Apatite (%)
750	2.31	18.7	68.3	12.4
850	2.58	9.4	112.4	28.7
900	2.67	5.8	134.8	41.3
1000	2.71	4.2	119.2	38.1
1050	2.73	3.6	105.3	36.8

Compressive strength peaked at 134.8 MPa for the 900 °C sample, then declined at higher temperatures. XRD patterns showed only broad amorphous humps below 800 °C. At 850 °C, apatite peaks first appeared alongside faint wollastonite reflections. The 900 °C condition yielded the strongest diffraction peaks for both crystalline phases 41.3% apatite and 32.8% wollastonite with the remaining 25.9% still amorphous. SEM images of the 900 °C fracture surface

revealed a tightly interlocked network of needle-like wollastonite crystals (0.5-2 µm length) embedded in a glassy matrix, with isolated pores averaging 4.2 µm diameter. After 14 days in SBF, a continuous apatite layer 4.3 µm thick covered the polished surface. FTIR confirmed characteristic phosphate bands at 1020 and 962 cm⁻¹ and a carbonate shoulder near 875 cm⁻¹, consistent with carbonated hydroxyapatite deposition.

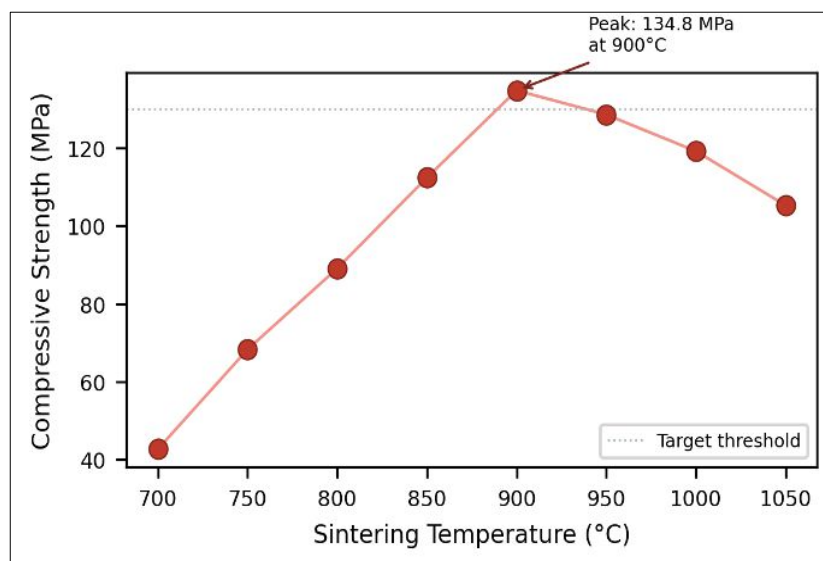


Fig 1: Compressive strength of glass-ceramic samples as a function of sintering temperature

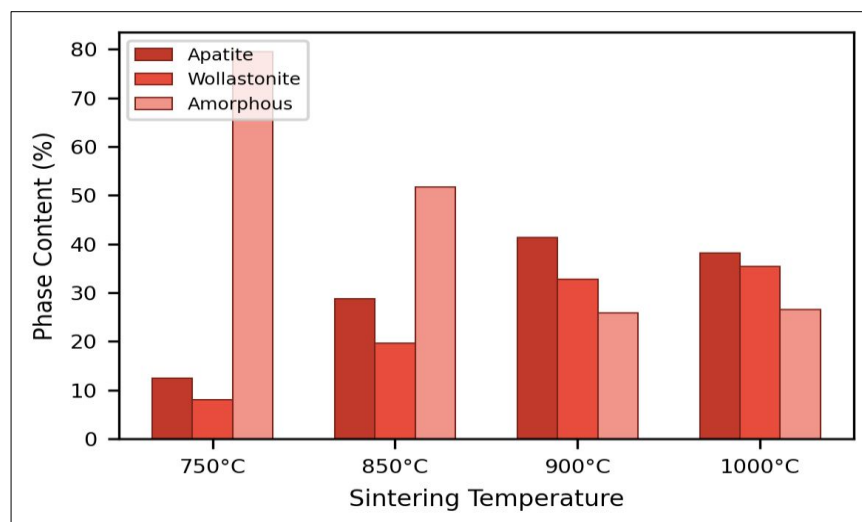


Fig 2: Crystalline phase distribution (apatite, wollastonite, amorphous) at four sintering temperatures.

Discussion

The 900 °C optimum falls within the crystallization window identified by DSC, where the exothermic peak at 892 °C signaled maximum nucleation and crystal growth rates. Below this temperature, insufficient crystallization left too much weak amorphous matrix. Above it, grain coarsening and thermal mismatch between wollastonite and the residual glass likely introduced microcracks that lowered strength [3]. The compressive value of 134.8 MPa exceeds the reported range for posterior tooth enamel (95-120 MPa), making the material mechanically suitable for occlusal restorations [6].

Bioactivity results were encouraging. The 4.3 µm apatite layer formed within 14 days is thicker than the 2-3 µm layers typical of 45S5 glass at the same time point, probably because the higher residual glass content (25.9%) at 900 °C maintained rapid ion exchange [2]. FTIR detection of carbonate substitution in the deposited apatite mirrors what happens on natural tooth surfaces, which is a favorable sign for biological integration [4].

One limitation worth noting: the 5.8% residual porosity at 900 °C could become a site for bacterial colonization in the oral environment. Surface sealing treatments or an additional low-temperature glaze might address this without destroying bioactivity. And these mechanical tests used simple uniaxial compression fatigue testing under cyclic loading would give a more realistic picture of clinical durability [11].

Conclusion

Glass-ceramics in the SiO₂-CaO-P₂O₅-Na₂O system sintered at 900 °C delivered the best balance of mechanical strength (134.8 MPa) and bioactivity among all temperatures tested. Apatite and wollastonite crystallization at this temperature produced a dense, interlocking microstructure capable of meeting posterior dental loading demands. Rapid hydroxyapatite formation in simulated body fluid confirmed that the material retains enough amorphous phase for effective ion exchange. Further work should address fatigue resistance and bacterial adhesion behavior before moving toward clinical trials.

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