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Effect of thermal treatment on physical properties of bioactive glass

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Abstract

The bioactive glass of composition $53SiO_2$–$6Na_2O$–$22CaO$–$11K_2O$–$5MgO$–$2P_2O_5$–$1B_2O_3$ (1–98) has been prepared using the melt method. The ultrasonic velocities, attenuation and elastic properties measurements have been made operated at a fundamental frequency of 5 MHz at room temperature on bioactive glass 1–98 before and after different thermal treatment conditions. Further, the frequency dependence of the attenuation was fitted into a linear relation $\alpha = af^N$. The observed linear increase in velocities, density and elastic properties of bioactive glass as a function of thermal treatment temperature, indicates that the densification in bioactive glass is due to the change in network bond per unit volume and not due to the structural/cross-linkage changes. Thus, thermal treatments of bioactive glasses lead to changes in elastic properties of bioactive glass. Furthermore, a long-term thermal treatment at higher temperatures also seemed to cause such changes to the glass surface that the formation of Ca,P-layer was inhibited during a 48 h immersion in simulated body fluid (SBF).

Keywords: Bioactive glasses; Thermal treatments; Bioactivity; Ultrasonic measurements; Elastic properties

1. Introduction

Biomaterials such as bioactive glasses and glass-ceramics have been developed for different newer applications in the field of medicine. Recent studies [1–7] reveal numerous successful implementations of bioactive glasses, such as the replacement of damaged or diseased body parts. Further, in dental prosthetics and orthopedics, bioactive glasses have been used as a filler material in bone defects. However, a better understanding of the microstructure, physico-chemical and mechanical properties of bioactive glasses would widen the range of the options for clinical applications of this material. The above properties of bioactive glass depend not only on the compositions of the glass, but also on thermal treatment conditions. Bioactive glass implants are exposed to thermal treatment for several hours, i.e., a heat sterilising of surgical bioactive glass implants or any medical device containing bioactive glass. In addition, when developing new clinical applications of bioactive glasses, one has to understand thoroughly the mechanical interactions between the implant and the surrounding tissues, the knowledge of the bone stiffness and elastic properties of bioactive glasses [8], etc.

Recently [9–12], attention has been focused on the development of new bioactive glasses due to different applications based on the preparation methods, the change in chemical compositions and different heat treatment conditions. For instance, the crystallisation behaviour and the phase formation order of different crystals with increase in temperature have been explored by Aboud et al. [13] on SiO_2–P_2O_5–Al_2O_3–MgO–Na_2O glasses. The changes in microstructure, mechanical and chemical properties of Al_2O_3–K_2O–Na_2O–CaO–P_2O_5 glass ceramics [14] with different heat treatment conditions lead to an important application in dental restoration. Attempt has been made [15] on the crystallisation and microstructural changes on high mechanical strength bioactive glass ceramic, which has the ability to form tight chemical bonds with living bone.

Several techniques, such as static bending, resonance, ultrasonic and impulse methods have been employed to measure the elastic properties of biomaterials [16,17]. Ultrasonic

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non-destructive testing (NDT) technique has been widely accepted as a unique tool for materials characterisation [18,19]. This may be possible due to the interaction of ultrasonic waves with macro, micro and sub-microscopic particles during the propagation of ultrasonic waves into the bioactive glass, and also the availability of multimode vibrations and a wide range of frequency selection. In the present investigation, an attempt has been made to study the effect of thermal treatments on ultrasonic properties on 1–98 bioactive glass. Further, the effect of the thermal treatment on the in vitro bioactivity of the glass was also determined by immersion of the 1–98 bioactive glasses in a simulated body fluid (SBF) for 48 h at 310 K. The observed results have been discussed in terms of the change in structure, stability, mechanical properties and in vitro bioactivity of the bioactive glass.

2. Experimental

The bioactive glass of composition $53SiO_2$–$6Na_2O$–$22CaO$–$11K_2O$–$5MgO$–$2P_2O_5$–$1B_2O_3$ (hereafter named as 1–98) was prepared from commercially available raw materials using the melt method. The components include Belgian quartz sand, Na_2CO_3, K_2CO_3, MgO, $CaCO_3$, H_3BO_3 and $CaHPO_4{\cdot}2H_2O$. All were of analytical grade and used without any further purification. The mixture was melted in a platinum crucible for 3 h at 1633 K in an electric furnace. Then the glass melt was cast into a pre-heated graphite mould giving a plate (90 mm × 60 mm × 20 mm) and annealed at 793 K or 1 h using subsequent furnace cooling. After that, the glass plate was allowed to cool overnight at a gradually descending temperature to 298 K. The glass plate was crushed and remelted to improve homogeneity of the bioactive glass. Finally, the homogenised melt were recast to a shape of a rectangular glass. The thermal behaviour of bioactive glass has been studied using the differential thermal analysis (DTA). The T_g value is determined from the DTA curve as shown in Fig. 1. Additionally, the theoretical value of T_g for the glass 1–98 was also calculated using the method developed by Karlsson and Rönnlöf [20].

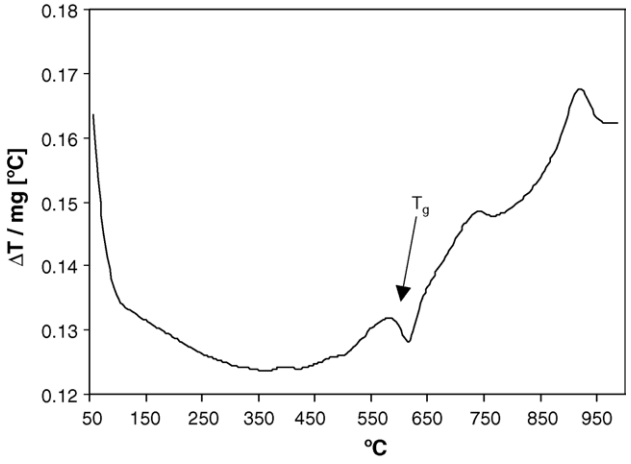

Fig. 1. DTA curve for 1–98 bioactive glass.

For ultrasonic velocities and attenuation measurement, six bioactive glasses (rectangle) were cut using a diamond saw. One of the bioactive glasses (termed as A) was used as such for the room temperature measurements, while the remaining five bioactive glasses were subjected to thermal treatment at different temperatures, namely 623, 673, 723, 773 and 823 K. Out of the remaining five bioactive glasses, the first bioactive glass (termed as B) was subjected to thermal treatment at 623 K, the second bioactive glass (termed as C) at 673 K. Similarly, the remaining three bioactive glasses termed as D, E and F were subjected to thermal treatment at temperatures 723, 773 and 823 K, respectively. The thermal treatment process consists of heating the bioactive glass up to a fixed temperature (say 623, 673, 723, 773 and 823 K for bioactive glasses B, C, D, E and F, respectively) under atmospheric condition keeping the material for an hour at each temperature. Then the bioactive glass is allowed to cool to room temperature in the furnace itself. The thermal treatment temperatures were selected from the thermal analysis of 1–98 bioactive glass and are well below the glass transition temperature T_g.

An important factor to be considered in the measurement of precise velocity and attenuation is the plane parallelism between the opposite faces of bioactive glasses. All bioactive glasses were shaped in the form of disc of 10 mm diameter and 6–7 mm thickness for the present investigation. The opposite faces of the disc shaped bioactive glasses were highly polished using lapping papers. The foreign particles residues were removed by rinsing with acetone, followed by rinsing with ethanol. The plane parallelism between the surfaces of the bioactive glass was checked employing a surface plate and dial gauge. The variation in bioactive glasses thickness is ±5 μm. The thickness of the respective bioactive glass after the thermal treatment at different temperatures was measured and no change was found.

3. Density measurements

Archimedes principle was employed to measure the density of all bioactive glass using CCl_4 as buoyant. The density of bioactive glass was obtained using the relation,

$$\rho = \frac{W_a}{W_a - W_b} \times \rho_b \quad (1)$$

where W_a is the weight in air, W_b the weight in buoyant and ρ_b the density of buoyant. All the weight measurements have been made using a digital balance (M/s. Sartorius, Model: BP221S, USA) having an accuracy of ±0.0001 g. The experiment was repeated for five times to get the accurate value of density. The overall accuracy in the density measurement is ±0.5 kg m^{-3}. The percentage error in the measurement of density is ±0.05%.