Materials Letters 218 (2018) 60–66

Contents lists available at ScienceDirect

Materials Letters

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

Enhanced bioactive glass-ceramic coating on Ti6Al4V substrate by microwave processing technique for biomedical applications

Sumana Ghosh [a,*], Saloni Nandi Majumdar [b], Mayur Shukla [a]

[a] Bio-ceramics and Coating Division, CSIR- Central Glass and Ceramic Research Institute, Kolkata 700 032, India
[b] Department of Biotechnology, Heritage Institute of Technology, Kolkata 700 107, India

ARTICLE INFO

Article history:
Received 6 December 2017
Received in revised form 18 January 2018
Accepted 27 January 2018
Available online 1 February 2018

Keywords:
Bioactive
Glass-ceramic coating
Microwave processing
Microstructure
Surfaces
Biomedical applications

ABSTRACT

The bioactivity of the microwave and conventionally processed coatings was investigated in terms of formation of fluroapatite layer onto the coating after immersion in SBF solution. The microwave and conventionally processed bioactive coatings were characterized by XRD, SEM and EDX analysis to identify the coating composition and morphology. It was observed that microwave processed coating favored the formation of fluroapatite layer on its surface compared to the conventionally processed coating and thereby, indicating enhancement in bioactivity of the microwave processed coating surface.

1. Introduction

Biocompatible materials like Ti6Al4V have found widespread use as a structural implant material. However, it is unable to bond with the bone due to lack of a specific biological response from the living tissues [1]. Bio-glasses first introduced by Hench et al. [2] have found extensive use in periodontology. Ti6Al4V is coated with bio-glass to aid ossteointegration [3]. Bio-glass coated Ti6Al4V substrate can be prepared by using conventional processing technique. Recently, microwave processing has become a new and efficient technique for material processing [4]. There is a considerable interest in apatite based materials for dental and medical applications. Studies on apatite-mullite based glass ceramics have been reported elsewhere [5]. Apatite-mullite based glass-ceramics have been developed by microwave plasma batch processing technique [6]. Microwave processing was employed to obtain hard glass-ceramic coating on nickel based superalloy substrate [7].

Garcia et al [8] studied bioactive coatings on titanium alloy. Kokubo et al. [9] attempted to produce apatite and wollastonite based glass-ceramics for bone substitution. A common characteristic of bioactive glasses and glass-ceramics is the time-dependent and kinetic modification of the surface upon implantation [10]. Bio-glass and bioactive glass-ceramics have the ability to bond with the host tissue. In particular, when implanted, they induce the formation of hydroxy-carbonate apatite (HCA) layer, which is very similar to the mineral component of bones [9]. This mechanism is known as bioactive fixation. Moreover, some specific bioactive materials are also able to bond with the collagen of soft tissues [2].

The objective of the present investigation is to prepare improved bioactive fluoroapatite and diopside based glass-ceramics coating on Ti6Al4V substrate using microwave processing technique and subsequently characterize them to check their suitability for biomedical applications.

2. Experimental details

SiO_2–Al_2O_3–CaO–P_2O_5–CaF_2 based glassy material was prepared by melting the glass batch at 1650 °C for 1 h. The glass composition has been given in Table 1.

Detailed glass frit making process have been reported in our earlier paper [1]. Bio-glass coatings were applied on the substrate by conventional enamelling technique. The glass slurry was sprayed on mechanically roughened and cleaned surface of Ti6Al4V substrates using hand spraying gun. The glass powder coated substrate was first air dried in ambient atmosphere and

* Corresponding author.
E-mail address: sumana@cgcri.res.in (S. Ghosh).

https://doi.org/10.1016/j.matlet.2018.01.155
0167-577X/

Table 1
Glass composition.

Sample	Constituents (in wt.%)							
	SiO_2	CaO	MgO	P_2O_5	CaF_2	Na_2O	B_2O_3	TiO_2
Glass	42	31	14	11	1.9	0.05	0.008	0.05

then dried in an air electric oven at 80 °C for overnight. The glass powder coated substrate was fired at 900 °C for 10 min and subsequently crystallized at 935 °C for 30 min in a conventional furnace as well as in a microwave furnace. The phase composition of the glass-ceramic coating was determined by X-ray diffraction (XRD) analysis (PW 1710, Philips Research Laboratory, Eindhoven, The Netherlands) using Cu Kα radiation (45 kV, 35 mA). Microstructures of the glass-ceramic coatings were studied by scanning electron microscope (PhenomTM Pro-X/Pro/Pure, Netherlands) while elemental composition of the coating surface was determined using the energy dispersive X-ray analysis (EDAX-SEM) using SEM Model (PhenomTM Pro-X/Pro/Pure, Netherlands) attached to EDAX unit. The bioactivity of both microwave and conventionally processed glass-ceramic coated substrates was studied by immersing them in a 50 ml of Tris-buffered simulated body fluid (SBF) solution with ion concentrations and pH nearly equal to those of human blood plasma at 37.4 °C for 7 and 14 days. The composition of SBF solution and detailed procedure has been reported elsewhere [1].

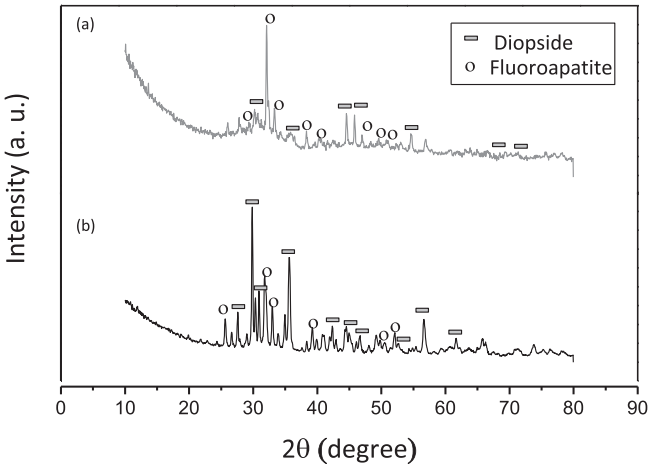

Fig. 1. XRD patterns of (a) microwave processed coating after 7 days immersion in SBF solution and (b) conventional processed coating after 14 days immersion in SBF solution.

3. Results and discussion

XRD, SEM and EDX analysis was performed for the microwave and conventionally processed glass-ceramic coatings after immersion in SBF solution. The XRD analysis of both bioactive coatings after immersion in SBF showed diopside and fluoroapatite crystalline phases (Fig. 1). XRD data confirmed that the microwave processed glass-ceramic coating showed the presence of fluroapatite as a major phase after immersion in SBF solution for 7 days whereas diopside crystalline phase was present as a major phase in the conventionally processed glass-ceramic coating immersed in SBF solution for 14 days. Therefore, it is quite evident that better deposition of fluoroapatite on the microwave processed glass-ceramic coating was occurred than that in the conventionally processed glass-ceramic coating even after immersion in SBF solution for 7 days. In case of both microwave and conventionally processed bioactive coatings during nucleation calcium phosphate precipitated in the SBF solution as well as onto the coating. As the Ti6Al4V substrate was already coated with fluroapatite and diopside based layer so nuclei were energetically more stable on the surface of coating. After reaching their critical size the precipitate started growing and transformed into fluroapatite and diopside crystals. Calcium and phosphate ions were responsible for the formation of thick fluroapatite based layer on the coated Ti6Al4V substrate [11].

SEM images of the conventionally processed and microwave processed bioactive coatings after immersion in SBF solution for 14 days and 7 days respectively and the corresponding EDX data are shown in Figs. 2 and 3. Characterization of conventionally processed coating before immersion in SBF has been performed and reported in our earlier paper [1]. The conventionally processed glass-ceramic coatings contained very low percentage of crystallites heterogeneously distributed in the glassy matrix after immersion in SBF for 14 days (Fig. 2(a)–(d)). The EDX analysis (Fig. 2(e)) demonstrated that the concentrations of Ca, P and F elements were lower in the conventionally processed coating as compared to those for the microwave processed coating and thereby, indicating the formation of greater amount of fluroapatite in the microwave processed glass-ceramic coating. Thus, EDX analysis confirmed the XRD data.

SEM microstructures of the microwave processed coatings showed the presence of mainly needle shaped fluroapatite crystals in the glassy matrix before immersion in SBF solution (Fig. 3(a)–(d)). Bioactivity study established that microwave processing greatly influenced the precipitation of fluroapatite onto the coating from the SBF solution even after 7 days immersion (Fig. 3(e)–(h)). It can be noticed from the SEM images that the microwave processed bioactive glass-ceramic coatings consisted of high percentage of crystallites that were uniformly distributed in the glassy matrix after immersion in SBF for 7 days only. Fig. 3(i) and (j) shows the EDX data of the microwave processed coating surfaces before immersion and after immersion in SBF for 7 days, respectively. Fig. 3(k) and (l) showed the microwave processed coating thickness before immersion and after immersion in SBF for 7 days. The corresponding EDX data have been given in Fig. 3(m). The coating thickness was ~45 μm ± 5 μm before immersion while it was ~50 μm ± 5 μm after immersion for 7 days. It was observed that the density of fluroapatite was quite high in case of microwave processed coating. Immersion in SBF solution had no significant effect on the thickness of the coating.