

Characterization of Apatite Glass-Ceramic Coatings on Ti-6Al-4V Substrate by Sol-Gel Method for Medical Application

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1- Introduction

A wide range of materials are used in implants and medical prostheses. Titanium alloys implants have weak biocompatibility and bioactivity properties and are usually coated with bioactive and biocompatible materials. The hydroxyapatite ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$) coatings stimulate osteoconductivity and osteoproduction of metal implants. The presence of anorthite ($\text{Ca}[\text{Al}_2\text{Si}_2\text{O}_8]$) compound along with the apatite-silica matrix improves bioactivity and biocompatibility, mechanical properties and reduces thermal expansion coefficient. The aim of this study is to analysis the double-layer apatite-anortite and single-layer apatite glass-ceramic coatings on titanium substrate.

2- Experimental

The plates of Ti-6Al-4V alloy specimens ($5 \times 5 \times 3$ mm) were grind with a 800 grit SiC abrasive paper. The cleaned substrates were soaked in a 1M NaOH solution at 60 °C for 1h. The air dried substrate dipped into the pre-prepared sols for 15s and then dried in a sealed container for 24h. Heat treatment was done at 600°C and 800°C for 3 h. Differential thermal analysis, DTA, was performed using 20 mg of powders with heating rate of 5 °C/min. Detecting of crystalline phases was carried out by a PW1730 X-ray diffraction. The morphology of the glass-ceramic coatings investigated by a scanning electron microscopy (SEM) operated at 20 kV. Microhardness Tests were done with a BUEHLER (1600-6125, USA) microhardness tester equipped with a vickers indenter, a 50 g load and a dwell time of 10 seconds. AFM analysis (AFM device (model no. 0101/A, Iran)) was used to determine the mean roughness of coatings.

3. Results and Discussion

Fig. 1(a-b) depicts DTA analysis of S0 specimen with heating rate of 10°C/min and 5°C/min, respectively. The endothermic peak over the temperature range from room temperature to 200°C is related to the evaporation of water and alcohols. The exothermic peaks between 200°C and 350°C, indicate oxidation process or dehydroxylation of the silanol groups. The exothermic peaks at around 800-1000°C are related to the crystallization and formation of apatite. XRD analysis of

S0 specimen after heat at 1100°C is shown in Fig. 2. XRD pattern affirms the formation of hydroxyapatite and anorthite in the silica amorphous matrix. SEM topography of coated samples are given in Fig. 3. The spherical hydroxyapatite morphology of S1 sample, derived from crystallinity of calcium phosphate glasses. The needle like morphology of first layer, is related to the non-equilibrium cooling rate of the first glassy layer.

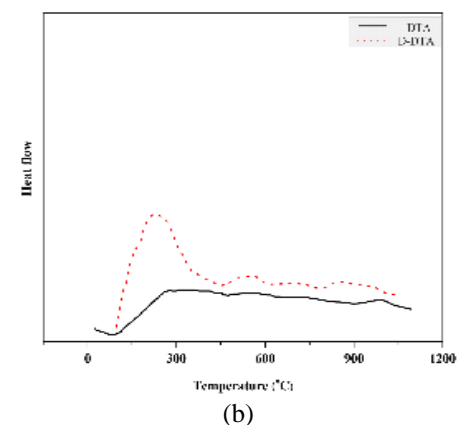
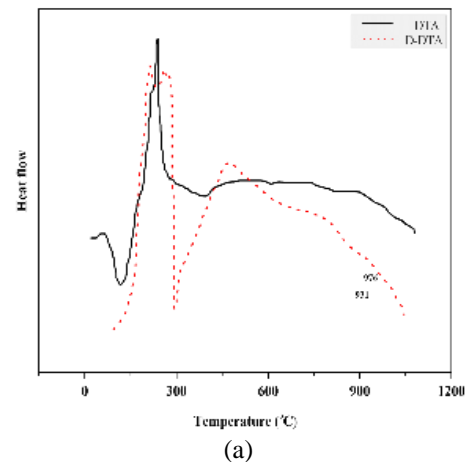


Fig. 1 DTA analysis of S0 specimen. a) Heat treatment with the rate of 10°C/min after drying at 100 °C. b) Heat treatment with the rate of 5°C/min after drying at 400 °C

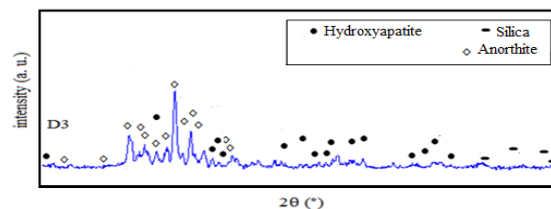


Fig. 2 XRD patterns of S0 sample

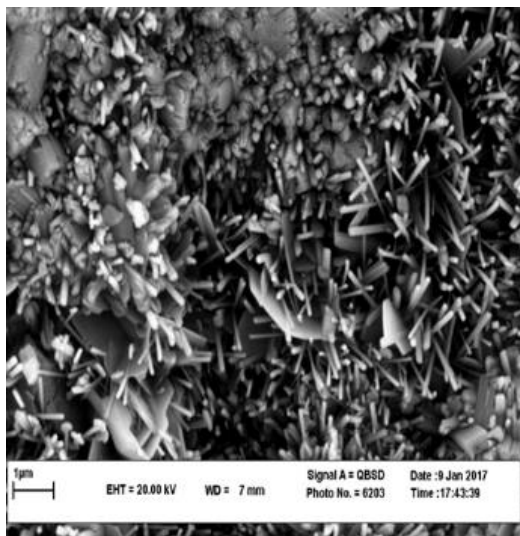
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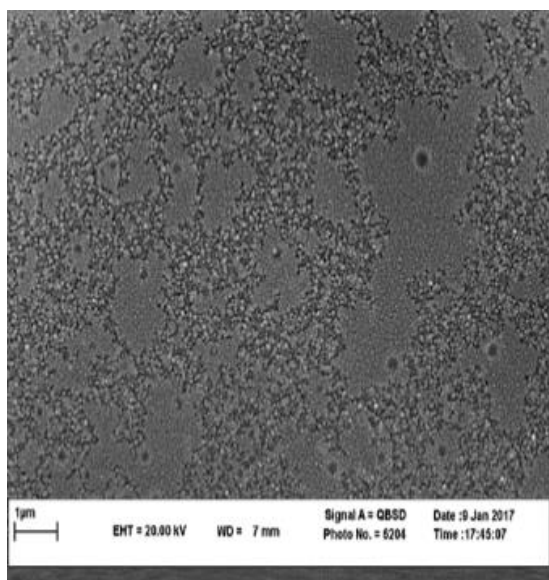
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(a)



(b)

Fig. 3 SEM micrographs of coated samples at different magnifications

The microhardness results of titanium coated substrates are shown in Table 1. It displays that coating process accelerates the microhardness of the titanium substrate. The microhardness of S2 film is more than the S1 sample, which is refer to the hard crystalline aluminosilicate compounds in the glassy matrix of this specimen. microhardness can be a criterion of wear resistant. Therefore, the coating process promotes the biological and mechanical properties of metal implants. Mean roughness amounts (R_{mean}) of coatings are illustrated in Table 2. All R_{mean} of samples are in the appropriate range of roughness which is used in dental application.

Table 1 Microharness results of Ti-6Al-4V surface treatment substrate and S1 and S2 samples

Analysis surface	surface treatment substrate	S1 film	S2 film
Microhardness (HV)	345.66	503.33	750.4

Table 2 Mean roughness values (R_{mean}) of S1 and S2 films

Film	S1	S2
R_{mean} (nm)	63.234	53.849

4- Conclusions

The apatite containing compounds have good bioactivity and biocompatibility. XRD results showed that apatite could effectively crystallized after controled thermal treatment at 1000 °C. Apatite containing glass-ceramic coatings improve the hardness and roughness values of the Ti-6Al-4V substrate. SEM results showed that applied coating had spherical morphology, which was effective in improving the mechanical properties.