Available online at **www.ijmrhs.com**

ISSN No: 2319-5886

International Journal of Medical Research & Health Sciences, 2018, 7(6): 114-122

Fabrication and Investigation of Commercial Pure Titanium/Bioactive Glass Ceramic as a Material for Dental Implant

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ABSTRACT

Background: *The osseointegration is a major factor influencing the success of the dental implant. The optimum circumstances of the osseointegration are the biointegration which means there is an intimate contact between bones and implant material without intervening space. The study aims at the fabrication of functionally graded material from bioglass at the surface and commercially pure titanium as a core of cylindrical implant specimen.* ***Material and method:*** *The functionally graded materials were fabricated, by using powder metallurgy method from bioglass 45S5 as a surface layer of the cylindrical specimen and commercially pure titanium as the core of cylindrical specimen. The compacted specimens were divided into three groups according to sintering temperature 800ºC, 900ºC and 1000ºC, with 3 hours holding time. The specimens of each group were analyzed by using scanning electron microscope and the energy dispersive X-ray spectroscopy. The porosity percentage was calculated and then mechanical evaluations were done by using Vickers hardness test and compressive strength test.* ***Results:*** *The scanning electron microscope examination shows a fusion of powder particles with an increase in the sintering temperature and decreases in the pores size among particles. And, also shows the diffusion of commercially pure titanium towards the bioglass at the interface at micrometric scale 100µm, 50 µm, and 10 µm. The results of energy dispersive X-ray spectroscopy also revealed that diffusion of commercially pure titanium towards bioglass at interface increased with an increase in the sintering temperature.* ***Conclusion:*** *Powder metallurgy method is used to obtain the synthetic functionally graded materials as implant material with the diffusion of the CPTi to the Bioglass at the interface.*

Keywords: Functionally graded material, Commercial pure titanium, Bioglass 45S5

INTRODUCTION

Dental implants are one of the most important dental treatments for replacing the missing tooth/teeth nowadays, providing both an aesthetic and functional replacement. Dental implants are inserts used for the substitutes of a missing natural tooth. Implants may be inserted in the maxilla or mandible. If designed properly, dental implants bond with a bone over time and act as an anchor for dental prostheses [1]. A major concern for implant design is the developing of materials that are biologically and physically compatible with bone. In ideal circumstances, bone should respond to the implant material as compatible material by osseointegration and remodel around it, and if it doesn't respond to the implant material it is used as a foreign body by encapsulating it with fibrous tissue [2]. Titanium and its alloys are broadly used as a material for a dental implant, due to their adequate properties such as biocompatibility, thermal conductivity, corrosion resistance, non-toxicity and fatigue strength [3]. However, the fact that all titanium alloys are considered as bioinert materials but they don't permit bone growth on their surface resulting in weak bonding between the implant materials and bone [4]. Natural biomaterials often have the structure of functionally gradient materials (FGM) which allows them to satisfy these requirements. FGMs supply the structure with which synthetic biomaterials should be formed essentially. To overcome this problem, usually, bioactive materials are used as a coating material for the metallic implants. The improvement in the fixation properties of Ti implant, coated with bioactive materials, has been reported in the previous studies [5,6]. On the other hand, it has been reported that the layer of bioactive materials coatings is often absorbed out in the tissue or are mechanically broken down after implantation. The bulk FGMsare used in the fabrication of the dental implant to overcome such problems of coating [7,8]. Bioactive glass is

reported to have the ability to stimulate more bone regeneration than other bioactive ceramics. Bioglass 45S5 is one of the bioactive glasses which form a strong bond with bone which leads to the breakage of the bone during its removal rather than breaking the bond [9].

PATIENTS AND METHODS

Specimen Design

Cylindrical specimens were prepared with 10 mm height and 5 mm diameter with two layers: the outer layer formed from bioglass with 1.75 mm thickness and the inner layer 'core of the cylinder' formed from commercial pure titanium (CPTi) with 1.5 mm diameter (Figure 1).

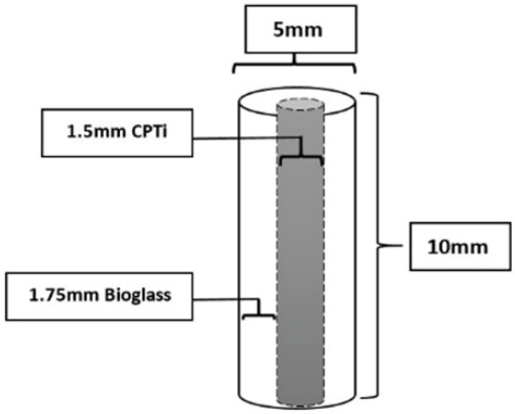

Figure 1 Specimen design

Mold Design

The mold was made from steel tool material, which consists of four parts: die, base, punch guide and punches.

- **Die:** The die was a hollow cylindrical steel mold of 25 mm height, 5 mm interior diameter and 30mm outer diameter.
- **Base:** The base was a steel metal disc of 5 mm height and 30 mm in diameter, at the center of the base there was a short solid rod with 4 mm height and 5 mm diameter. This rod was inserted inside the canal of the mold when the die seated on the base. The notch was prepared on the surface of the short solid rod for centralization of the solid steel rod during bioglass compaction.
- **Punch guide:** The punch guide was a hollow cylindrical steel mold of 10 mm height, 5 mm interior diameter and 30 mm outer diameter seated on the die for centralization of the punch during press and prevention punch bending.
- **Punches:** The two punches were used and were made from a steel solid rod, long and short punches each one had two ends, the pressing end, and the working end. The pressing end had 7 mm diameter and 15 mm length. The short end according to which the punches were divided into the short punch which had a short working end for pressing the powder inside the die with 5 mm diameter and 15 mm length, and the long punch which had long working end for ejecting the specimen outside the die with 5 mm diameter and 25 mm length.

The solid steel rod which was used during bioglass compaction was of 1.5 mm diameter and 26 mm in length.

Specimen Preparation

Wetting of the powder: The bioglass powder was wetted with 2% polyvinyl alcohol as a binder by Planetary Mono Mill Pulverisette 6 classic line. The reverse mode was used according to the manufacturer instructions to improve the homogeneity of the sample at 300 rpm for 1 hour as uniform mixing was obtained [10].

Compaction: The parts of the mold was lubricated with paraffin, the die was seated on the base and the solid steel rod was fitted in a notch present in the solid rod of the base, and the wet bioglass (0.55g) was compacted in the die in

such a manner that leaves the cylindrical space for Ti powder by using the dental condenser of size 0.5 mm.Then the punch guide was seated on the die, after that the bioglass was compacted by using the modified short punch, which had a canal in the working end with 1.5 mm diameter and 5 mm length, with the presence of the solid steel rod which was inserted inside the canal of the modified punch during the pressing with hydraulic press at 10MPa for 2min after that the solid steel rod drawn to leave a cylindrical space. Later on, this space was filled with dry Ti powder (0.25g) and was compacted with a solid steel rod.

Pressing

It was started with seating the punch guide on the die before pressing to prevent the bending of the punch during pressing the powders, then the powders were pressed inside the die by uniaxial cold pressing, with short punch by using the hydraulic press with 130Mpa for 10min holding time then the punch guide and the base was removed. The specimen was ejected by using the long punch. The specimens were left for drying for 24 hours at room temperature.

Sintering

The sintering process was done by placing the specimens in a quartz tube of argon furnace. Three groups were prepared according to the sintering temperatures (800ºC, 900ºC, 1000ºC) with 3hours holding time for each temperature group and the heating rate was 5ºC/min. The specimens were left in the furnace in the inert atmosphere inside the furnace, with the flow of argon gas, for 24hours for cooling at the room temperature as shown in Figure 2 [11].

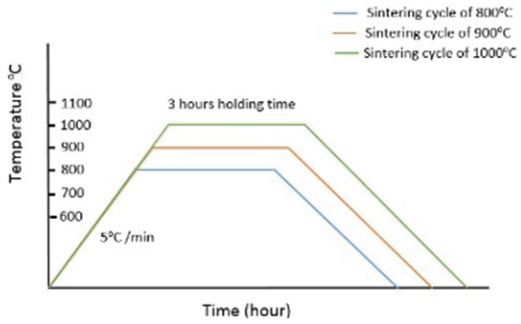

Figure 2 Sintering cycles representation

Total 90 specimens were prepared and were subdivided into three groups according to the temperature of the sintering. Group 1 (1000ºC) was composed of 30 specimens which sintered at (1000ºC), Group 2 (900ºC) was composed of 30 specimens which sintered at (900ºC), and Group 3 (800ºC) was also composed of 30 specimens which sintered at (800ºC). Each group was subdivided into three subgroups according to the group porosity test, group microhardness test, and group compression test, each was composed of 10 specimens.

Specimens' Preparation for Tests

The specimens were prepared by flattening the ends of the cylindrical specimen with 10 mm height and 5 mm diameter by using the prosthetic engine with the cutting disc.

Scanning Electron Microscopy (SEM)

Scanning electron microscope SEM "Hitachi S4700 EDAX ApolloX Genesis software" was used for identifying the interface of two materials used in the specimens of each temperature group. The magnifications used were 36X, 500X, 1000X and 5000X for all the specimens. The SEM device was also used to determine the atomic percentage of elements by EDS map, and to detect the elements along the cross-section of the specimen.

Porosity Test

The density and porosity of the consolidated specimens of each temperature group were measured by using Archimedes principle and by weighing the specimens by electronic balance at different conditions. First, the dry specimens were weighed and were then immersed in acetone for 24 hours, after that the specimens were removed from the acetone and were weighed and suspended by a wire in acetone. Equations 1-5 show the formula used for the calculation of the porosity.

Bulk density, $\rho b = \frac{W_a}{W_c - W_b} * \rho dl$ (1)

Where:

Pb: The density of the sintered specimen measured by Archimedes method (g/cm^3)

Wa: Weight of the dry specimen

Wb: Weight of the suspended immersed specimen

Wc: Weight of the saturated (after removing from acetone) specimen

ρdl: The density of the tested liquid (0.7845 g/cm^3 at 25ºC)

$$\rho_{tb} = \sum_{i=1}^{n} W_1 t^* \rho_1 + W_2 t^* \rho_2 + W_3 t^* \rho_3 + ... + W_n t^* \rho_n \quad (2)$$

Where:

ρ_{tB} : Theoretical density of the blended powders (g/cm^3)

n: No. of elemental powders

W_t : Weight percent (%).

$\rho_{1,2,3,....n}$: Density of elemental powder (g/ cm^3).

$$Open\ porosity\ = \left(\frac{w_c - w_a}{w_c - w_b} * 100 \right) \quad (3)$$

$$Total\ porosity = 1 - \frac{bulk\ density\ \rho b}{theoretical\ density\ \rho_{tb}} * 100 \quad (4)$$

Closed porosity=Total porosity-Open porosity (5)

Microhardness Measurements

Microhardness Vickers test was done according to ASTM (E92-82)for specimens of each group, with microhardness Vickers tester type (Digital display microhardness tester model MHV-2000S) [12]. The Vicker's microhardness (HV) is specified as follows:

HV=1.854 p/d2 (6)

Where:

p=applied load (Kg), d=average length of diagonal (mm)

The cylindrical specimens were used in this test so according to the ASTM (E92-82), the correction factors were applied to the Vickers hardness values were obtained on the test cylindrical surfaces of the specimens.

Compression Test

The compression test was done according to ASTM (E9-89a) at the room temperature [13]. The specimens of each group were tested at a crosshead speed of 0.1 mm/min and load of 1000N using the universal testing machine.

RESULTS

Scanning Electron Microscope Analysis (SEM)

Surface morphology: The surface morphology of sintered samples was determined using SEM images. Figure 3 showed the top view image of each sintered group with a magnification of 36X. Figures 4-6 showed the cross-section of specimens of each sintered group with magnification 500X, 1000X and 5000X respectively, and showed the adhesion of small powder particles in micrometric scale between 50 µm and 10 µm of bioglass and CPTi while small

pores were observed being trapped between the adhered particles. These findings were observed in the specimens of each sintered group.

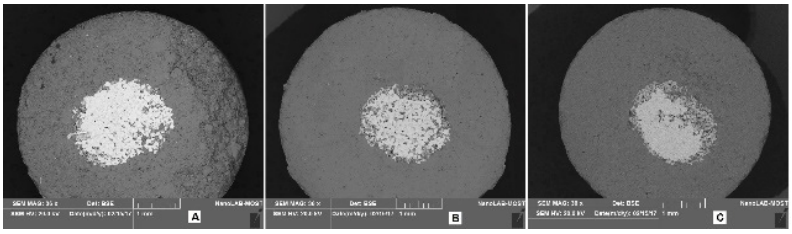

Figure 3 SEM top view images of specimens of sintered groups with 36X magnification, a) 800ºC, b) 900ºC,c) 1000ºC

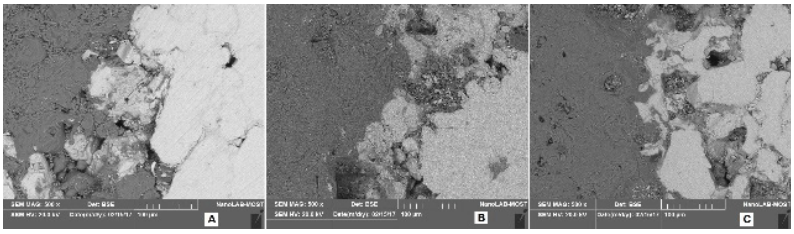

Figure 4 SEM cross-section images of specimens of sintered groups with 500X magnification, a) 800ºC, b) 900ºC,c) 1000ºC

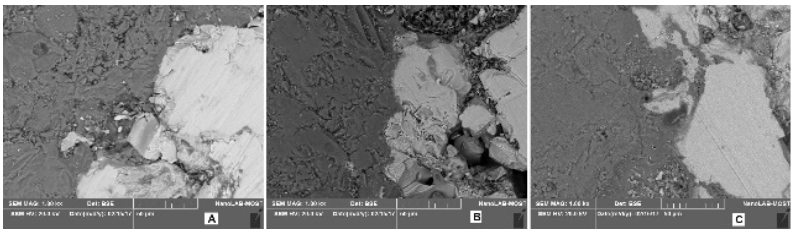

Figure 5 SEM cross-section images of specimens of sintered groups with 1000X magnification, a) 800ºC, b) 900ºC,c) 1000ºC

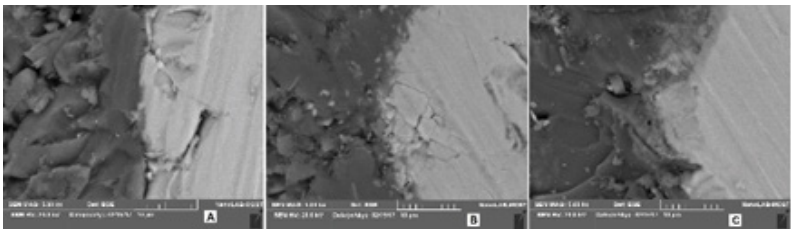

Figure 6 SEM cross-section images of specimens of sintered groups with 5000X magnification,a) 800ºC, b) 900ºC,c) 1000ºC

Elemental composition: Energy dispersive X-ray spectroscopy (EDS) analysis was done at the interface between bioglass and CPTi for 1 mm distance at both the sides. The interface is located at the zero point of this line distance. Using the line analysis, the chemical composition variation can be characterized in each point distance from the edge representing the atomic mobility in both the directions. Hence, the diffusion mapping could be quantitatively clarified. Here, for each sintered group the EDS maps showed the main components in both sides. Ca, Na, Si, P, O, and Ti, were detected with different concentration gradient depending on the sintering temperature as shown in Figures 7-9, respectively. It could be demonstrated that the diffusion of Ti towards bioglass is dominant at almost all sintering temperatures. In other words, the diffusion rate of Ti is increased with increasing the treating temperature.

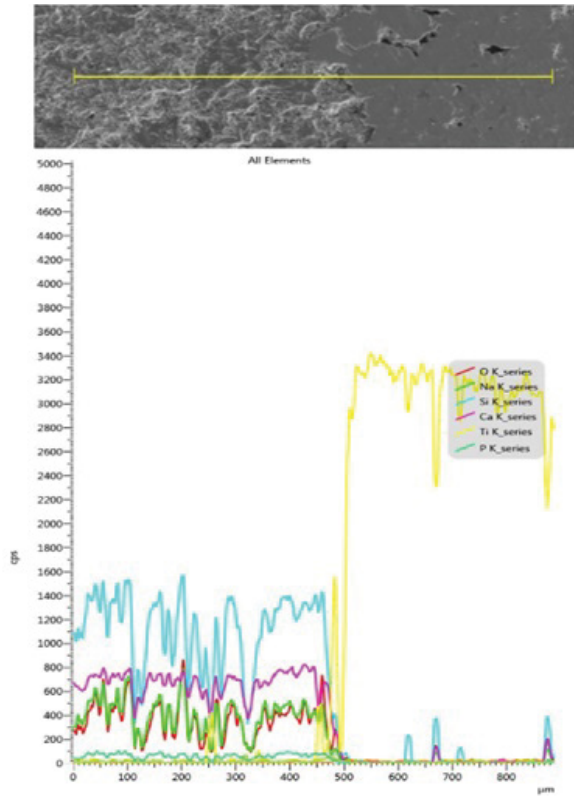

Figure 7 EDS map of specimen sintered with 800°C

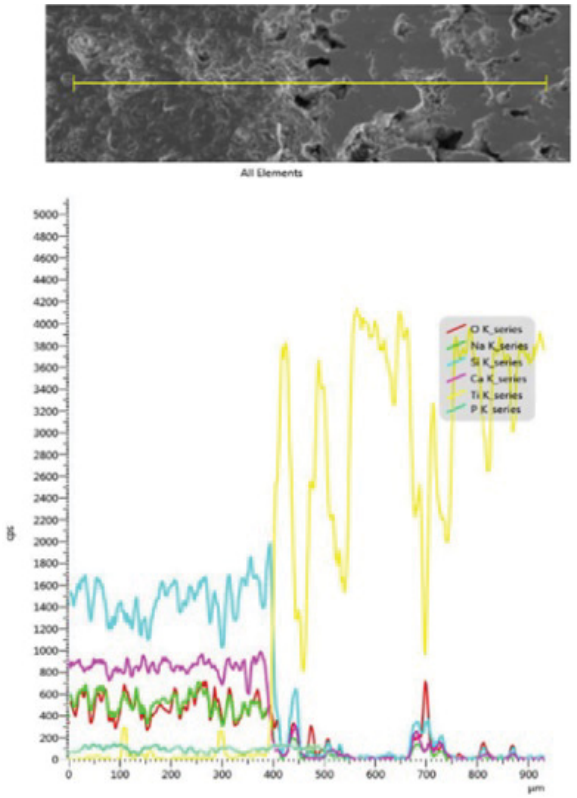

Figure 8 EDS map of specimen sintered with 900°C

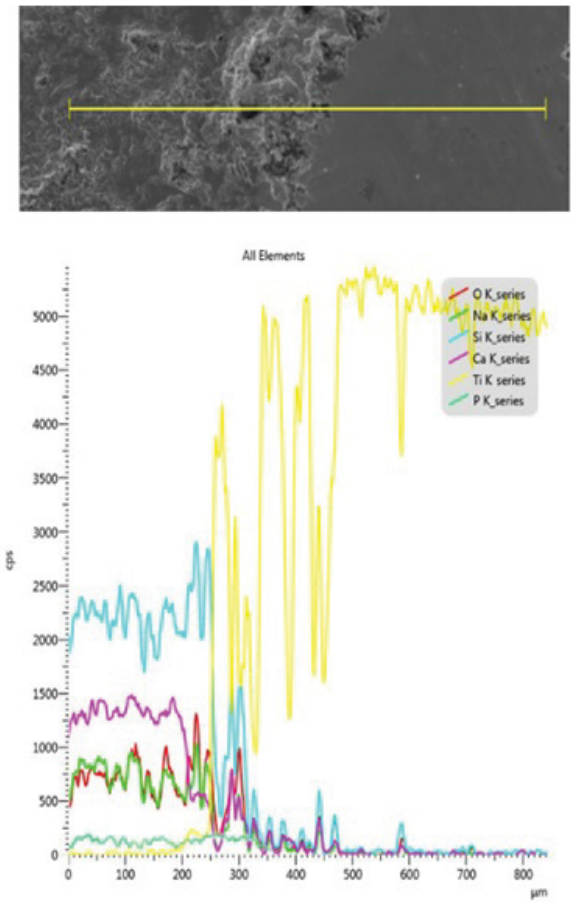

Figure 9 EDS map of specimen sintered with 1000ºC

Porosity, Microhardness and Compression Tests

The results of porosity percentage values, Vickers hardness values and compressive strength values at different sintering temperature groups were statistically analyzed using descriptive and analytical analysis. In Tables 1-6, the results of ANOVA test with post-hoc analysis using Bonferroni test.

Table 1 Mean values of porosity percentage % for all tested groups and ANOVA test

Sintering Temperatures	N	Mean (%)	SD	F-test	Significance
1000ºC	10	5.66348%	0.03958813	14660.843	0
900ºC	10	6.71128%	0.02457205		
800ºC	10	7.73149%	0.00411493		

Table 2 Post-hoc/Bonferroni test among different tested groups

Sintering Temperatures			Mean Difference (I-J)	Significance
Bonferroni	1000ºC	900ºC	-1.04780-*	0
		800ºC	-2.06801-*	0
	900ºC	800ºC	-1.02021-*	0
*The mean difference is significant at the 0.05 level				

Table 3 Mean values of Vickers hardness (Kg/mm²) for all tested groups and ANOVA test

Sintering Temperatures	N	Mean (Kg/mm²)	SD	F-test	Significance
1000ºC	10	674.007	9.90549	1308.905	0
900ºC	10	520.487	7.01528		
800ºC	10	459.85	12.6147		

Table 4 Post-hoc/Bonferroni test among different tested groups

Sintering Temperatures			Mean Difference (I-J)	Significance
Bonferroni	1000°C	900°C	153.51988*	0
		800°C	213.99328*	0
	900°C	800°C	60.47340*	0
*The mean difference is significant at the 0.05 level				

Table 5 Mean values of compressive strength (Mpa) for all tested groups and ANOVA test

Sintering Temperatures	N	Mean (Mpa)	SD	F-test	Significance
1000°C	10	5.146497	0.124815	1768.719	0
900°C	10	2.802548	0.110076		
800°C	10	2.318471	0.10539		

Table 6 Post-hoc/Bonferroni test among different tested groups

Sintering Temperatures			Mean Difference (I-J)	Significance
Bonferroni	1000°C	900°C	2.34395*	0
		800°C	2.82803*	0
	900°C	800°C	0.48408*	0
*The mean difference is significant at the 0.05 level				

DISCUSSION

Living tissues like teeth and bones have a characterization of functionally graded material from nature, to substitute these tissues. A biocompatible material is required to replace the original bio-tissue. The perfect candidate for this purpose is functionally graded material. FGM is used broadly in the orthopedic and dental applications for bone and teeth substitutions [14].

The SEM images in this study showed the adhesion of small powder particles in micrometric scale between 50 μm and 10 μm of bioglass and CpTi while small pores were observed being trapped among the adhered particles. The size of the pores reduced as there was an increase in the sintering temperature. This resulted from necks that formed at the contact regions between adjacent particles during the initial sintering stage, in addition, within each neck a grain boundary forms, and pore forms from every interstice between particles. With the progress of sintering, the pores become more spherical in shape and smaller in size. Sintering was accomplished by diffusion of atoms from the bulk particles to the neck regions [15]. While the EDS analysis showed the diffusion of CpTi towards bioglass was dominant at almost all sintering temperatures. In other words, the diffusion rate of CpTi was increased with an increase in the treating temperature. This diffusion of CpTi may be attributed to atomic and molecular interactions at the interface. These interactions resulted from asymmetrical coordination, the differences in coordination between the atoms at the interface of the different materials. Also, the progression of a diffusion bond in the solid state depends on both the diffusion controlled mass transfer and thermal activation. Mass transfer and surface diffusion include the interfacial voids elimination, while an adhesion process gives the interface boundary strength [16].

The atoms with thermal activation contained excess energy and this is considerably higher for metal atoms as the chemical bonds are the metallic bonds which are less in strength than that of the ionic bonds of the ceramics. This resulted in mass transfer and surface diffusion of the CpTi towards the bioglass more than that of the bioglass towards the CpTi.

The mass transfer and surface diffusion of the CpTi towards the bioglass increased with increasing sintering temperature which may be attributed to the increase in the atomic energy with an increase in the temperature. The melting point of the bioglass was (1200ºC) which less than that of CpTi (1668ºC) but with increasing temperature the CpTi diffused more in bioglass and not vice versa. This may be resulted from the high affinity of CpTi to interact with O_2.

The porosity percentage was reduced with the increasing sintering temperature, while the microhardness of specimens increased with increasing the sintering temperature. This attributed to the densification and reduction of pores size with the progression of sintering, i.e. with increasing the sintering temperature [11,15].

The compressive strength of specimens increased with increasing the sintering temperature. This may be attributed

to the increasing the densification and decreasing the porosity with increasing the sintering temperature[11,17]. In addition to that joining of CpTi with bioglass might lead to strengthening the ceramic material by metal.

CONCLUSION

By using the powder metallurgy method a successful synthetic functionally graded materials as implant material with the diffusion of the CPTi to the bioglass at the interface can be obtained. Mechanically, the specimens that sintered at 1000ºC revealed best mechanical properties than that of specimens that sintered at 900ºC or 800ºC.

DECLARATIONS

Conflict of Interest

The authors have disclosed no conflict of interest, financial or otherwise.

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