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Experimental Investigation of FGM Dental Implant Properties Made from Ti/HA Composite

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ABSTRACT

Although titanium/hydroxyapatite composite is an attractive material for dental implants, it would be more useful if it could be produced as a functionally graded material (FGM). In this paper, microstructure and microhardness of a five-layer titanium/hydroxyapatite functionally graded material has been investigated. First, titanium and hydroxyapatite (HA) powders were mixed with the Ti to HA volume ratios of 100:0, 90:10, 80:20, 70:30 and 60:40. Next, the obtained powders were poured into a graphite mold functionally and then sintered using spark plasma sintering (SPS) method. Microhardness and microstructure of the samples was examined using Vickers microhardness test and optical microscopy. The results show that HA particles have an acceptable distribution in the matrix. Also, average matrix grain size increased in Ti/HA composites compared to the pure Ti layer. Moreover, the layers with 30 and 40 vol% HA have the highest and the lowest microhardness, respectively.

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1. Introduction

The concept of functionally graded material (FGM) was first proposed to decrease thermal stresses induced in a combination of materials (metal and ceramic) which could then function in high surface temperature applications (e.g. a hypersonic plane project)[1]. FGMs are advanced materials consisting of at least two continuous or discontinuous gradients in their composition. Chemical composition and/or properties changes allow these materials to have a wide range of applications in aerospace and medical industries [2]. In the medical industry, implants can be fabricated as FGMs. Using materials which are physically and biologically adapted with the human body is an important need in dental implant design. Austenitic stainless steel, Co–Cr alloys, and titanium (Ti) and its alloys are widely used as metallic implants in human bodies. Among them, titanium and its alloys are some of the most popular metals used in dental implants because of their biocompatibility and proper mechanical properties such as elastic module, strength, and toughness [3]. In recent years, different approaches have been used to improve osseointegration (direct structural and functional connection between living bone and the surface of a load-bearing artificial implant) in dental implants. To this end, implants have recently been made of biocompatible ceramics. Most human body hard tissues (60-70% of bone, 98% of enamel) contain hydroxyapatite (HA). Using HA in implants could accelerate osseointegration. Most properties of synthesized HA (except its crystal structure) are the same as HA which exists in human bone [4, 5]. There are two ways to produce HA: using raw materials containing HA such as bovine bone, fish scale, or egg shell etc.; or synthesize it chemically [6-8]. Ayatollahi et al. prepared nano-sized HA from bovine bone and used it in bone cement to improve its mechanical and tribological properties [9].Titanium/hydroxyapatite (Ti/HA) composite is an attractive material for implants in the human body. It combines the excellent biocompatibility of hydroxyapatite ceramic (with bioactive properties for new bone formation) and the desirable mechanical properties of titanium. However, it would be more useful if Ti/HA dental implants could be fabricated as functionally graded materials for biomedical purposes. For an FGM dental implant, the Ti/HA

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layer with a high HA volume fraction would be implanted in the jawbone for better osseointegration, while the Ti layer at the top could tolerate applied forces well.

There are several methods to fabricate FGMs including powder metallurgy (with different sintering procedures such as spark plasma sintering and pressure less sintering), casting methods (such as slip casting and centrifugal casting [10, 11]), and rapid prototyping (such as three-dimensional printing and selective laser sintering [12, 13]). The most common of these methods is powder metallurgy [2]. Spark plasma sintering (SPS) method is a high speed method to sinter materials rapidly by applying heating and mechanical pressure simultaneously. In the case of Ti/HA composition, SPS temperature decreases from 1300°C (which is usual in other methods) to 850°C. This temperature prevents decomposition of hydroxyapatite. Only 5 minutes are required to complete the sintering process [14, 15].

Shahrjerdi et al. [16] fabricated five-layer FGM Ti/HA by applying optimal pressure-less sintering procedure. The results showed that the third layer with 50% Ti and 50% HA had the highest hardness [16]. Chenglin et al. produced a six-layer FGM. According to the result, Vickers hardness and Young's modulus were strongly affected by the porosity, while bending strength and fracture toughness increased remarkably with the rise of Ti content [17]. Investigation of the biocompatibility of Ti/HA FGM showed that the FGM has a better biocompatibility than the pure titanium implant. Watari et al. fabricated Ti/HA FGMs three different ways: electric furnace, induction furnace, and SPS (which was the only method that produced a stable FGM sample). It was reported that the composite needed at least 30% HA content to induce sufficient change in its biocompatibility properties [14, 18, 19].

The purpose of the present work is to fabricate a five-layer sample of HA/Ti FGM using powder metallurgy and SPS method. Microstructure and microhardness of each layer will be investigated and discussed.

2. Experimental procedure

2.1. Material and method

The schematic configuration of a five-layer FGM dental implant is shown in Fig 1. Titanium powder (particle size <75μm) and hydroxyapatite powder (particle size <1μm) were used to produce the FGM.

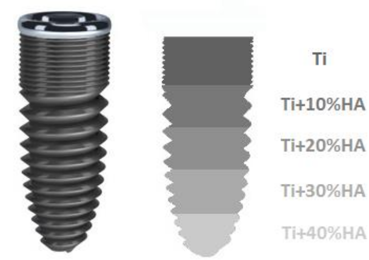

Figure 1. Schematic configuration of a five-layer FGM dental implant

The hydroxyapatite powder was derived from natural bovine bone. First, bone was boiled in distilled water for 6h to remove the bone marrow and tendons. The boiled bone was then dried in an oven at 100°C for 24 hrs in order to eliminate moisture. Next, dried specimens were calcined in an electric furnace at 900°C for 2 hrs to remove other organic substances. Finally, the cooled remained material was crushed into powder. The X-Ray diffraction pattern of the resulting powder showed a good match with existing HA patterns. SEM image and X-ray diffraction pattern of HA powder are shown in Fig 2. and Fig 3., respectively.

Fig.4 shows an SEM image of titanium powder. A planetary ball-milling machine was used to mix Ti and HA powders into different layers. The titanium to hydroxyapatite volume ratio selected for the five layers was 0:100, 10:90, 20:80, 30:70 and 40:60. The mixing process was carried out under argon atmosphere for 3 hours and with a rotational speed of 40 rpm. Finally, the mixed powders were stacked layer by layer in a graphite mold and sintered at 850°C for 5 minutes under the pressure of 40 MPa.

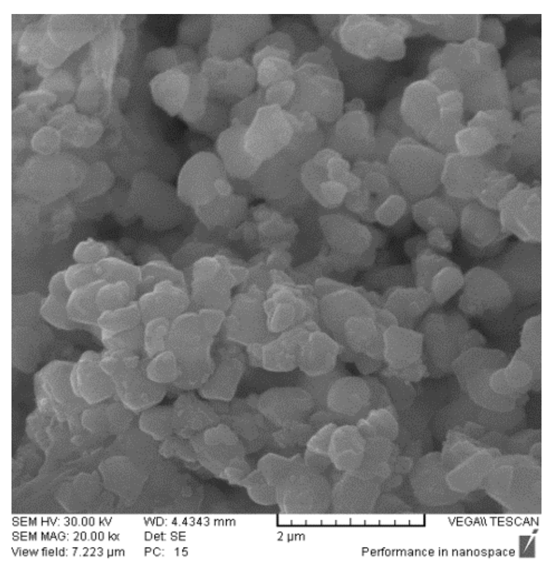

Figure 2. SEM image of hydroxyapatite powder

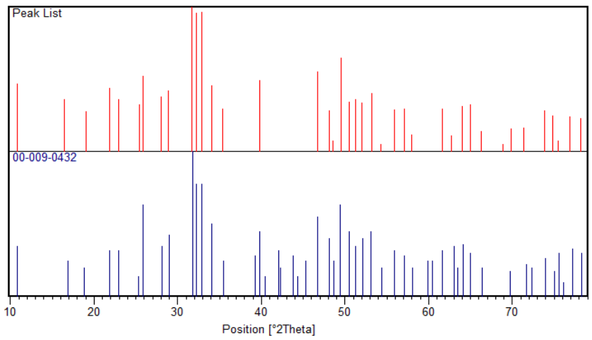

Figure 3. X-ray diffraction pattern of Hydroxyapatite powder

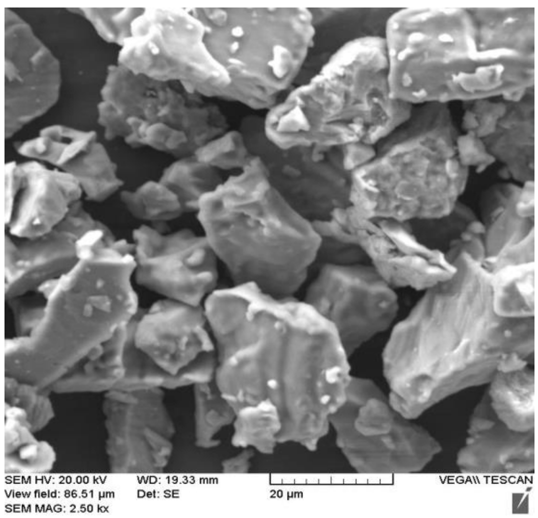

Figure 4. SEM image of Titanium powder

2.2. Characterization

For microstructural observations by optical microscope, the samples were ground, polished and etched (for grain size measuring). Vickers microhardness of samples was determined by applying a 200 g load to the polished surfaces for 10 seconds. The measurements were repeated in each layer at least 5 times to minimize errors.

3. Results and discussion

Fig.5 shows Ti/HA gradation in the fabricated FGM sample after grinding and polishing. HA particle distribution is observed in the five layers which have Ti to HA volume ratios of 0:100, 10:90, 20:80, 30:70 and 40:60. Light areas belong to the parts containing Ti and dark areas show the parts containing HA. The particle distribution here is acceptable in comparison to references [17-19], although agglomeration of HA or presence of porosity, especially on the implant surface, could be desirable for osseointegration. Increasing percentage of hydroxyapatite in Ti/HA composites leads to enhanced osseointegration. OHowever, according to the literature, samples made of high percentage HA are not stable and the pure HA layer does not have enough strength to carry occlusal forces (the force exerted on opposing teeth when the jaws are closed or tightened) [14].

The grain size of different layers is shown in Fig 6. The results show the grain size has been increased by enhancing the volume percentage of hydroxyapatite. There are two factors that affect grain size of the matrix: first, presence of reinforcement phase, and second, spark plasma sintering process. The presence of a reinforcement phase would prevent excessive increase of the grain size in the titanium matrix. But it also would decrease the effective cross section of conductive matrix in the composite layers. As the total amount of electrical current in SPS is constant for different layers of FGM, the temperature increases locally because of increasing matrix effective resistance. Moreover, the amount of thermal conductivity of composite layers is less than that of pure titanium layers. For this reason the material is kept at higher temperatures for longer time. These factors can increase the grain size of titanium matrix by enhancing the percentage of HA reinforcement. The hardness of titanium is lower than HA. So, based on the law of mixture, the hardness of Ti/HA composite should be enhanced by increasing HA volume fraction.

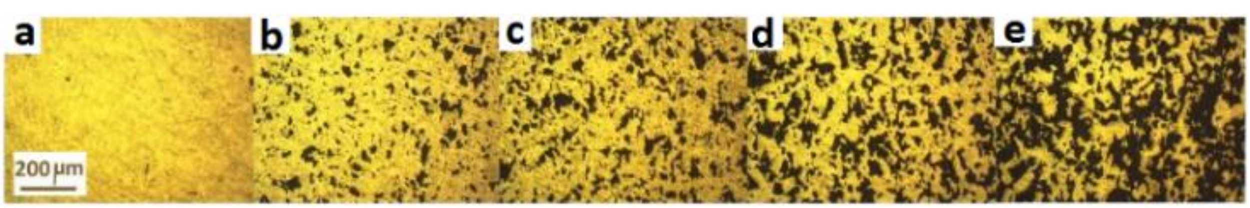

Figure 5. Different layers of the FGM sample; a) Ti, b) Ti/10 vol% HA, c) Ti/20 vol% HA, d) Ti/30 vol% HA and e) Ti/40 vol% HA.

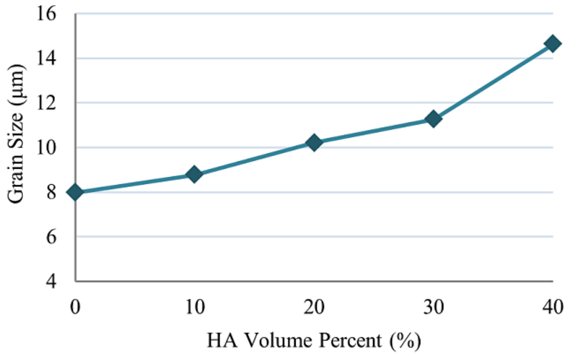

Figure 6. Grain size of the FGM layers with different HA volume fraction

Fig.7 shows Vickers microhardness for the different layers of the FGM. The results show that unlike the law of mixture, the hardness of the five-layer FGM sample is not directly correlated with the HA volume fraction. The highest hardness is obtained in the layer with 30 vol% HA, while the lowest is in the last layer with 40 vol% HA. There are four important reasons which affect the amount of microhardness. First, HA particles have a higher hardness in comparison to Ti and when they are distributed in Ti matrix, they can make local plastic deformation difficult (due to applied forces) in the matrix. As a result, the hardness in the matrix would be increased. Second, presence of second and intermetallic phases in the interface of matrix and reinforcement could increase microhardness because of brittle behavior and higher hardness. Third, the grain size has a direct effect on the amount of hardness. Decreasing average grain size results in increasing hardness and vice versa. Fourth, the porosity and particle agglomeration have a negative effect on hardness. Increasing the amount of porosity causes hardness to drop rapidly. Each of these reasons could affect the microhardness of the Ti matrix.

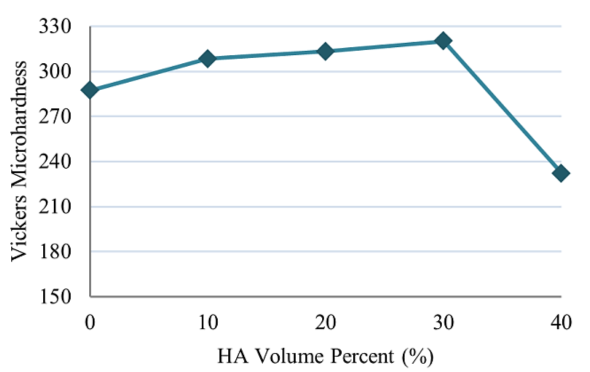

Figure 7. Vickers microhardness of the FGM layers with different HA volume fraction

In the four initial layers from Ti to Ti/30 vol% HA, the first and second reasons dominate and hardness increases continually, while the third and fourth reasons have more influence in the Ti/40 vol% layer.

4. Conclusion

A five-layer Ti/HA FGM sample was successfully fabricated using powder metallurgy and SPS method. The particle distribution of HA was acceptable and could improve and accelerate osseointegration. Increasing HA volume fraction increased the average matrix grain size in the Ti/HA Layers. The Vickers microhardness of the layers increased when increasing the amount of HA volume percentage from 0% to 30%. In the last layer with 40 vol% HA, the hardness dropped to the smallest amount in the FGM.

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