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Surface modification of Ti 6Al4Valloy coated (HAp-collagen) by a spin coating method

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Abstract
Bone and blood are the most implanted tissue. The collagen and substituted hydroxyapatite are major solid components of human bone. In the present study, a modest attempt has been tried to use a different method. The techniques involve the coating materials (collagen and hydroxyapatite) and the base materials Ti alloy. The aim of the paper to use spin method to coating Ti6Al4V alloy with HAp (1, 2, 3) g + (0.5 collagen) The X-ray diffraction test results show nano-size crystal structure on the coated film. By Increasing in concentration of HAp leads to an increase in thickness of film, (25-40) μm and that is clear in the results of scanning electron microscopy SEM and optical microscopy. From SEM results, the aggregation HAp nanoparticles are confined to their nano size .Also collagen with graded porosity, the mean diameter of pore in nm this type of scaffold can be used to tissues that belong to the success of the solution preparation method and use spin coating method.

Keywords: Ti-6Al-4Valloy; Spin coating; Hydroxyapatite; Collagen.

1. Introduction

The life expectancy will Increase as well as arthritis, bone diseases d, generative, hip replacement and after the world wars, it was essential to the development of different types of biomaterials to substitute damaged of the parts [1]. In order to get artificial implants parts and to improvement tissue-engineering research or system with excellent mechanical properties, physical and chemical, it's necessary to collect materials nature with biocompatibility, high corrosion resistance and wear. Ti alloy has wildly used for dental implant and orthopaedic also as a substrate due mechanical properties are closer to those of bone [2]. It is necessary to modify the surface of Ti alloy such as polishing and coating before implants; this is because many fracture-related events, particularly fatigue cracks, porosity initiate at the surface of components, also to increase the biocompatibility of surface. The biomaterials ceramic always used for the coated purpose [3]. The natural solid bone has properties such as high strength and excellent mechanical properties which come from mixing nano mineral crystal within the collagen matrix. The bone matrix consists of 65% wt mineral material, 25% wt organic material (consisting proteins such as collagen include different types I, III, V, XXIV collagen) and 10% wt water. Collagen type must provide protein counting 97% .The hydroxyapatite is main in organic mineral phase, besides many types of elements include (Na, B, Mg, Si, Fe, Zn…etc.) present in the biological bone and shown it is important factor in growth bone [4]. The addition of components of the ceramic materials such as titanium or hydroxyapatite composite with collagen type I (HA/Col) shows effective in stimulating the activity of the bone cell and rising HA/Col osteoconductive [5, 6]. One of the ingredients bone graft artificial material involve the collagen/nano hydroxyapatite composite because it's very good structural similarity and compositional with natural bone [7, 8].

2. Materials

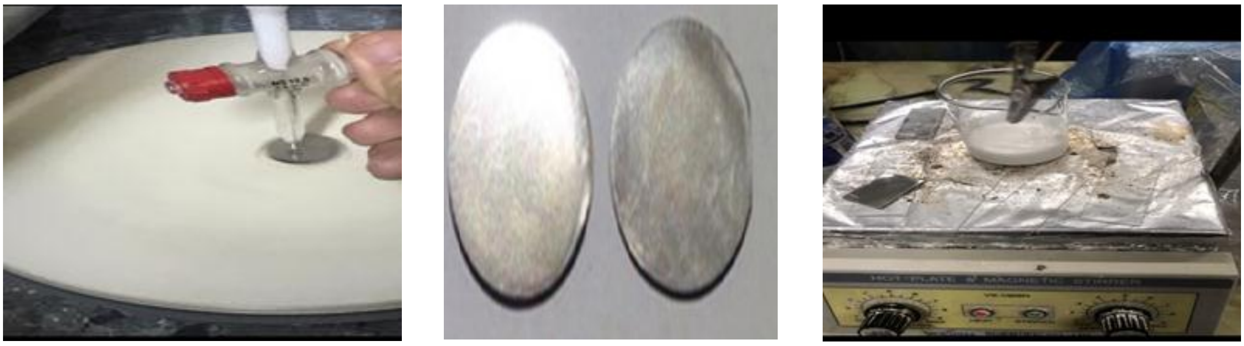
Related to the manufacturer of the titanium alloys (Ti6Al4V) GR2 ASTM F136 have a chemical composition shown in Table 1. Hydroxyapatite (HAp) has chemical formula Ca10(OH)2(PO4)6 were obtained from Riedel-De Haen Ag Seelze-Hannover Germany with purity 99.0%and particle size 40 nm. Collagen type I is a protein called pro-alpha1 have a spherical shape with size (75-125μm), white colour was obtained from Arkopharma Company.

Table 1. Illustrated the Chemical composition of Ti6Al4Valloy in weight percent.

Ti	Al	V	Fe	C	N	O	H
89.2	5.5-6.5	3.5-4.5	0.40	0.1	0.05	0.20	0.0125

3. Method

1- Solution (1): Gram chitosan was added to the acetic acid solution, and the mixture continued for two days to obtain a complete dissolution using magnetic stirrer.3ml of the solution above the first is added to the next second solution
2- Solution (2): 30 ml of absolute C2H5OH ethanol Additive:
HAp (1, 2, 3) g + (0.5 collagen) g. Mixing well mixed by a magmatic stirrer for one hour, it was prepared for coating titanium alloy, as shown in Figure (1a).
3- The specimen Ti 6Al4Valloys was grained by using various different grades of SiC paper and then polished by using a Struers DAP-U system, Denmark, as shown in Figure (1b).
4- The sample was coated by using a Spin coating method starting from the centre towards the edges for 1min with 75 rps. At a distance of 0.5cm between the substrates and source of solution coating. All the samples with thick film (solution from HAP & collagen) coated were heat treatment 400°C for one h using a furnace, as shown in Figure (1c) [8].



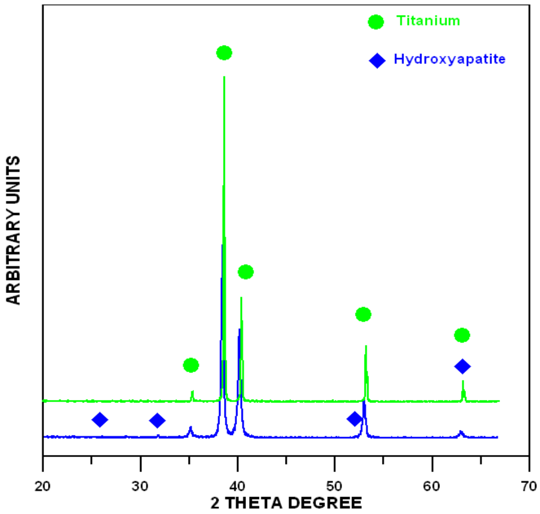
(a) (b) (c)

Figure 1. (a) Hap & collagen well mixed by magmatic stiller (b) The specimen Ti 6Al4Valloys, (c) The sample was coated by using a Spin coating method.

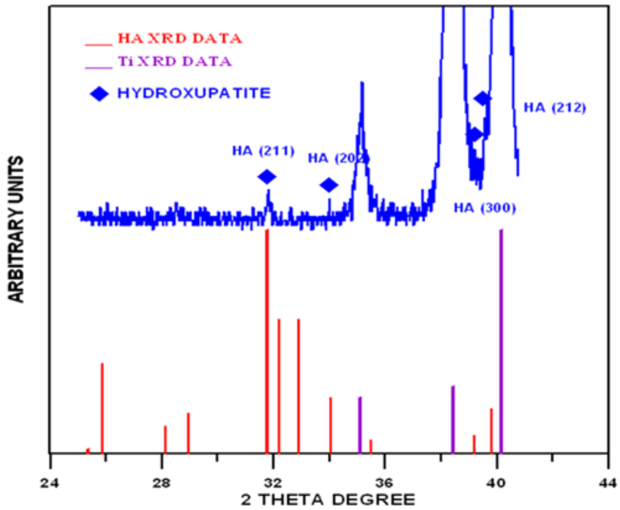
4. Results and discussion

4.1 X-ray diffraction analysis

The XRD x-ray diffraction analysis was used for the detection of plate components. Figure 2 shows an XRD pattern for the first sample (1gHAp+0.5g collagen). The crystal structure represented by sharp peak w with nanoparticle size and small grain size, There is more than one intensity of peak Hap (211, 202) around 2θ° (31.7, 34.2) also (300 and 212) around 2θ° (38.5 and 39.6) are overlapped of Ti substrate peak (002, 101) around 2θ° (38, 40.1).



(a)



(b)

Figure 2. XRD patterns data: (a) for HAp and Ti6Al4V alloy standard (b) for Ti6Al4V alloy coated with (1 g HAP &0.5g collagen).

4.2 Hydroxyapatite & collagen Thickness of

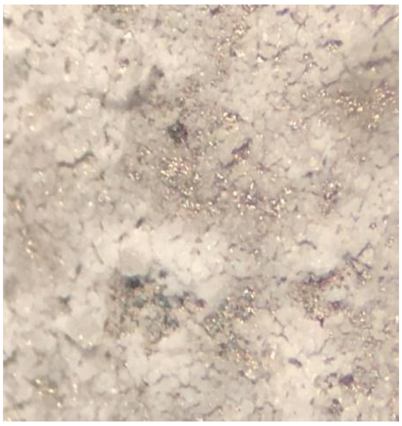
The thickness of (HAP & collagen) film has been determining by using the Minutest 3000 system. Table 2 shows the thickness increasing from 25µm to 40µm, with increasing the concentration of HAp.

Table 2. The thickness of (HAP &collagen) film coated on the substrate.

(HAp+collagen) film	Value
HAp (1) g + (0.5 collagen) g	25
HAp (2) g + (0.5 collagen) g	32
HAp (3) g + (0.5 collagen) g	40

4.3 Optical microscopy

The optical microscopy was used to detect the composite coated surface with magnification in 10 micrometres. Figure 3 (a, b and c) shows a uniform and fully coated of Ti-6Al-4V alloy with the spin-coated method. Figure (2a) for the first sample (1gHAp+0.5 g collagen) film-coated. The collagen particle is transparent with silver colour and circular shape, the cotton shape aggregation with whit colour belong to HAp. Figure 2 (b, c) for second and third sample (2g HAp + 0.5g collagen) and (3g HAp + 0.5g collagen) respectively show the collagen particle reduce with increase in the concentration of HAp. The optical photograph in agreement with results [9].



(a)

(b)

(c)

Figure 3. Top view of the microstructure of Ti-6Al-4V alloy (a) (1gHAp+0.5 g collagen coating) (b) coated of the (2gHAp+0.5 g collagen) (c) coated of the (3gHAp+0.5 g collagen).

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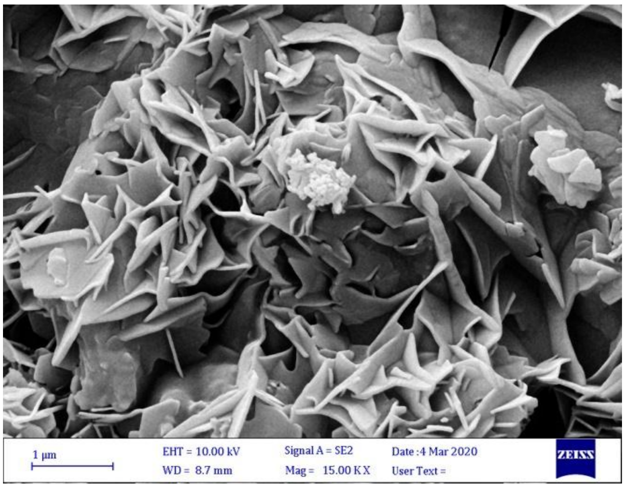
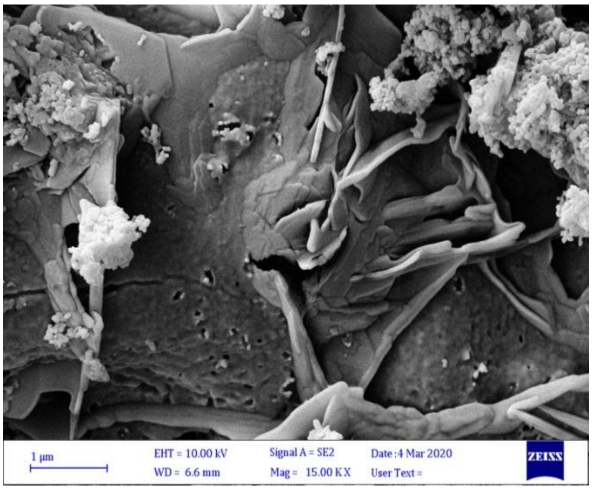
4.4 A scanning electron microscopy (SEM)

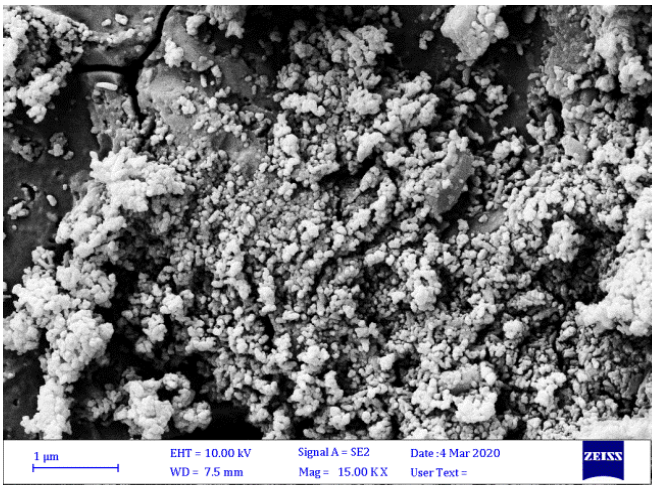
Figure 4 (a, b & c) represent the SEM images for Ti6Al4V alloy coated with (HAp+collagen). Image of collagen with graded porosity, sine the average diameter of the pore in nanometer this kind of scaffold could be used to tissues [10]. Figure (3a) show collagen particle is very clear with fins shape which agreement with results [11], the HAp particle are clumped like flowers. Figure (3.b) the collagen reduces with the emergence and spread of HAp is clear. At last Figure (3.c) HAp particle approximately cover all the surface with little visibility for collagen that's belong to increasing in concentration of HAp.



(a)



(b)



(c)

Figure 4. SEM picture of the Ti-6Al-4V alloy microstructure from the top view of (a) coated (1gHAp+0.5 g collagen) (b) coated of the (2gHAp+0.5 g collagen) (c) coated of the (3gHAp+0.5 g collagen).

5. Conclusions

1- Aspin coating method was successful in coating the Ti6Al4V ally with film from Hydroxyapatite and collagen.
2- X-ray diffraction results show the sharp peaks induct that the coated film contains a nano-size crystal structure and small grain size.
3- The Increasing in the concentration of Hydroxyapatite HAp leads to an increase in the thickness of film-coated (25-40) µm, and that is clear in the results of optical and SEM.
4- From SEM results, the aggregation nanoparticles of HAp particles are confined to their nano size. Also, collagen with graded porosity, the average diameter of the pore in nanometer this kind of scaffold could be used to tissues that belong to the success of the solution preparation method and use spin coating method.

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