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Archives of Applied Science Research, 2013, 5 (3):137-141
(http://scholarsresearchlibrary.com/archive.html)

ISSN 0975-508X
CODEN (USA) AASRC9

Hydroxyapatite/Titania coating on titanium strip by brush coating

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ABSTRACT

Dual hydroxyapatite and titania coating was developed on titanium strip with single step by simple brush coating method. Sintering was done at 950 °C. The resultant coating was analyzed for Ca/P ratio, coating surface morphology by SEM, X-ray diffraction, Adhesion test. Uniform hydroxyapatite/titania coating was formed on metal surface after sintering. Scanning electron micrographs showed dense and crack free coating. EDAX analysis showed 1.66 Ca/P ratio, X-ray diffraction study gave peaks both for titanium dioxide and hydroxyapatite. Adhesion strength was found to be more than 35 MPa.

Key words: Hydroxyapatite, SEM, EDAX, XRD, Adhesion test

INTRODUCTION

Many biocompatibility studies have proved that hydroxyapatite has very similar chemical composition like the inorganic part of human hard tissue, such as bone and teeth [1]. The most important advantage of hydroxyapatite being a bioactive material is that bone will form a direct chemical bonding to hydroxyapatite implant without forming a collagen interface layer which is usually found in many bare metallic devices and other bio-inert materials after implantation [2]. The main disadvantage of hydroxyapatite lies with its poor mechanical properties, particularly fatigue properties it means hydroxyapatite can not be used in bulk for load bearing application such as orthopaedics [8]. Owing to the inferior mechanical properties of hydroxyapatite and poor osteo-inductive properties of titanium, SS, Co-Cr alloy implant, bioactive coating on metallic implant such as hydroxyapatite has been tried to enhance osseo-integration and initial fixation property of implant in dentistry and orthopaedic [3].

Different coating techniques are available for hydroxyapatite coating like; Plasma spray, Electrophoretic deposition, Electro-chemical deposition, Sol-gel, Magnetron sputtering. Plasma spray is the only process which is clinically used at present. Attempts have been tried for hydroxyapatite/titania coating by plasma spray and micro-arc oxidation [13-14].

In this study, Hydroxyapatite and titania coating was developed by simple brush coating technique. A brush coating process in which the wet coating mixture is applied on the surface by means of brushes. Sintered coated samples were analyzed by Scanning electron microscopy, EDAX, X-ray diffraction and Adhesion test.

MATERIALS AND METHODS

Preparation of hydroxyapatite slurry and coating

1 gm hydroxyapatite (Plasma biotech, USA) and 1 ml Tween 20 (Merck, India) were taken and blended together using glass rod. Wet hydroxyapatite slurry was spread over a surface of titanium strip (1cm x 1cm x 0.05 cm) by brush. The coated samples were placed for sintering.

Sintering of hydroxyapatite

Coated samples were put in programmable quartz tubular furnace. In table 01 showed that samples kept at 100 °C for 30 minutes for solvent evaporation. Again samples were kept for 15 minutes at 300 °C for Tween 20 evaporation. Sintering was done at 950 °C for 1hr without inert atmosphere. Then finally cooled to room temperature slowly. Rapidly cool down may create cracks in coating because heat capacity of both calcium phosphate ceramic and titanium are different.

Table 1: Temperature profile for sintering of hydroxyapatite

Time (hrs)	0.5	1	1.75	2.5	4	4.5	6	7	8.5	9.5	11	12	13	14	24
Temp °C	100	100	300	300	500	500	700	700	950	950	800	600	500	300	25

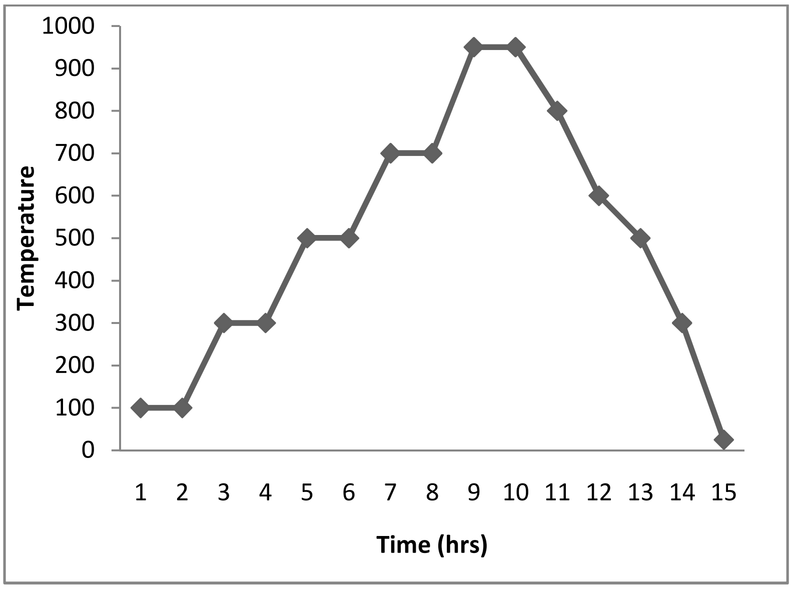


Figure 1: Temperature profile for sintering

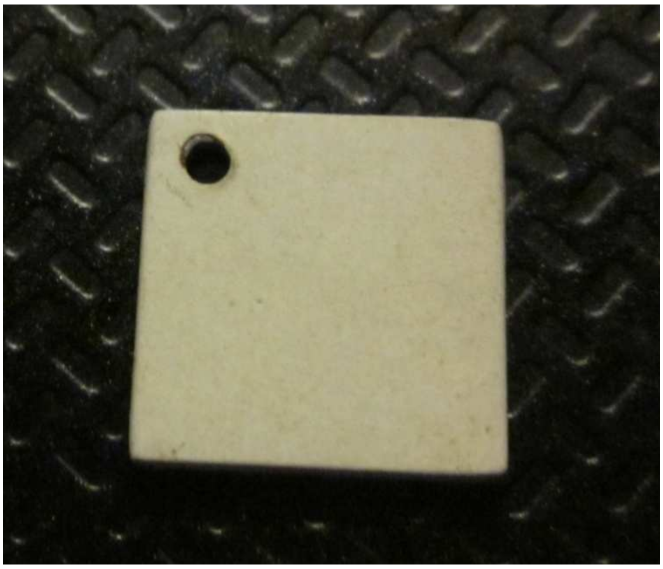


Figure 02: Hydroxyapatite/titania coating

The excess un-sintered hydroxyapatite powder was removed from the surface coating. Figure 02 showed the uniform hydroxyapatite/titania formed on titanium metal surface.

Scanning electron microscopy

Coated samples were used to analyze the coating morphology by SEM. Under SEM, thick coating was observed at 250 x magnification. Packed sintered crystals were seen at 1000 x. No crack was seen at all magnification (fig 03). Absence of crack was due to low rate cooling profile after sintering at 950 °C.

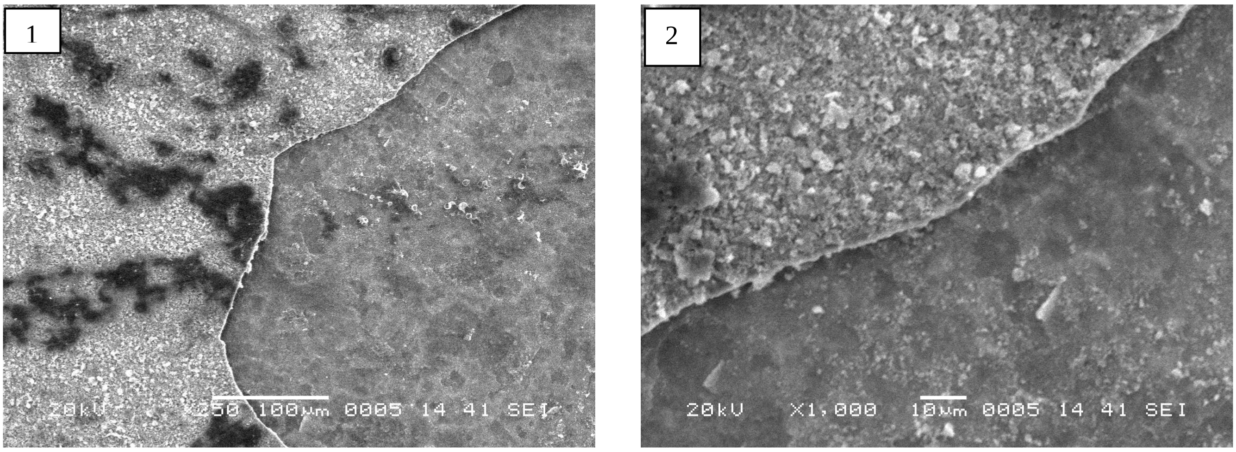


Figure 03: SEM images of hydroxyapatite coating done by brush method (1) coating morphology at low magnification 250x (2) coating morphology at 1000x

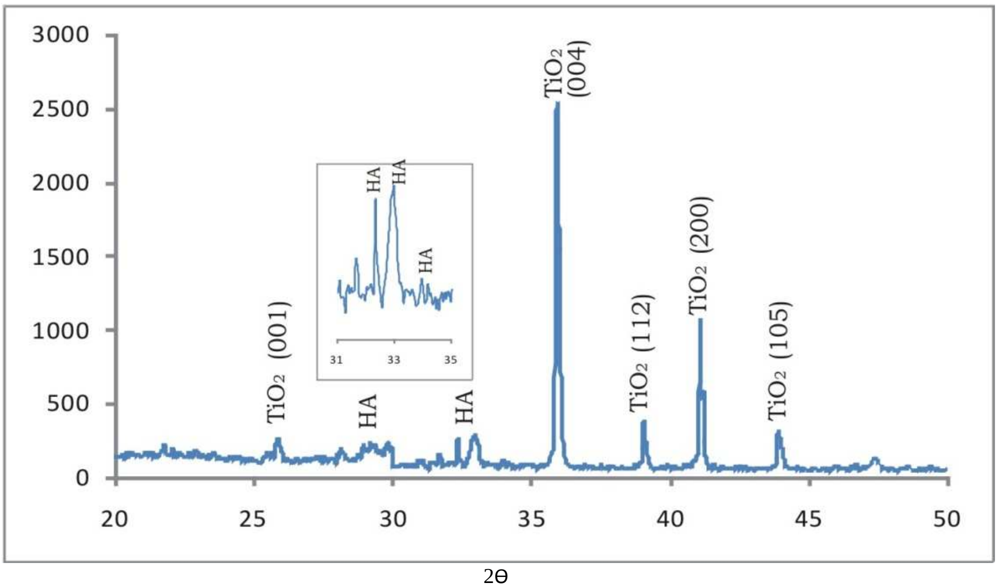
Energy dispersive analysis by X-ray (EDAX)

Sintered hydroxyapatite coated samples was analyzed for EDAX spectra. The peaks at 2.0 and 3.7 keV demonstrate that the relationships between Ca/P ratio. The EDAX spectrum of hydroxyapatite showed specific Ca and P (fig 04), which has an elemental distribution of 3.70 % and 2.23 % respectively in the material (table 02). From the pattern, it can be seen that the Ca/P ratio obtained 1.66 which was very closely to the theoretical value (1.67). The peak obtained for titanium due to titanium dioxide (titania) resulting due to oxidation of titanium.

Table 02: Weight and atomic % of Element

Element	Weight%	Atomic %
O K	45.06	70.07
P K	2.78	2.23
Ca K	5.96	3.70
Ti K	46.20	24.00
Totals	100.00	

Figure 04: EDAX for hydroxyapatite/titania coated strip



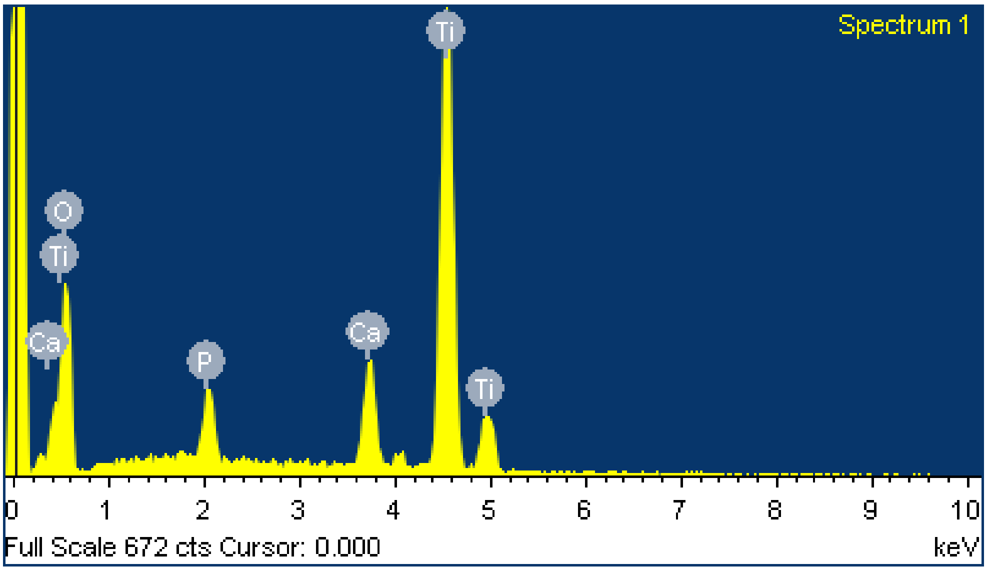


Figure 05: X-ray diffraction patterns for hydroxyapatite coating by brush coating

Hydroxyapatite coated strip was used X-ray diffraction analysis. The X-rays used are of the Copper Kα wavelength 1.54056 x 10^{-10} m, the scan was taken between 2ϴ of 10° and 2ϴ of 50° at increments of 0.05° with a count time of 0.02 second for each step. These angles were selected as this is where the important reflections lie for hydroxyapatite and other relevant impurities. The intensity of the X-rays was measured on the Y axis, and increasing values of 2ϴ are shown on the X axis.

There are several major peaks obtained between 31° to 35° which are identical to those of the reference HA structure (Fig 05)There are some major peaks i.e. (001), (004), (112), (200), (105) for titanium dioxide which is an Indication of hydroxyapatite/titanium dioxide composite formed at titanium surface.

Adhesion strength of hydroxyapatite coating on titanium

The bonding strength of the hydroxyapatite coating on titanium substrate was measured by indigenously developed testing system. 0.5 cm^2 area of the hydroxyapatite/titania coating was glued on to glued onto the bare metal side with epoxy resin and then cured at a 110 $^{\circ}$C in the oven for 24 hours; to be ready-to-pull sample. Pan was attached to bare metal using steel wire and hydroxyapatite/titania coated titanium was held fixed to wall using steel wire.

In testing, the sand beads were added in pan continuously till the coating pull-out from metal. The amount of sand was weighed that was accountable for coating failure. The tensile strength 35 MPa was evaluated for hydroxyapatite/titania coating.

CONCLUSION

Titania layer formed due to oxidation titanium metal. This coating technique is economical and has certain advantages because it eliminates any equipments or apparatus used in plasma spray, sol-gel, electrochemical deposition, electrophoretic deposition, sputtering. Brush coating is the simplest but faster than other. Brush coating technique is simpler and gives more adhesion strength of material.

REFERENCES

[1] DA Wahl, JT Czernuszka. DA Wahl. *Eur.* Cells. *Mater*. **2006,**11,43.
[2] Jong Kook Lee, Kyu Hong Hwang. *J. Ceram. Process. Res*.**2012**,13,2,158.
[3] Yang Shoufeng, leong kah-fai. *Tissue. Eng*.**2001**,7,6,679.
[4] Nayak Amit Kumar. *Int. J. ChemTech. Res*. CODEN(USA).**2010**,2,2,903.
[5] MJ Orbunoff. *Anal Biochem*.1**984**.136,2,425.
[6] MJ Gorbunoff. *Anal Biochem*. 1**984**.136,2,433.
[7] MJ Gorbunoff, Timasheff SN. *Anal Biochem*. 1**984**.136,2,440.
[8] Manivasagam Geetha, Dhinasekaran Durgalakshmi. Recent Patents on Corrosion Science. **2010**,2,40.
[9] Cong Wang, J. Ma, Wen Cheng, Ruifang Zhang. *Materials Letters*.**2002**,57,99.
[10] Ishikawa Masahiko, Nishikawa Kouji.*Mat. Res. Soc. symp. Proc*. **2002**,711, 1.

[11] Liu Dean-Mo, Yang Quanzu, *Troczynski Tom. Biomaterials*. **2002**,23,691.
[12] Liu Dean-Mo, Yang Quanzu, *Troczynski Tom. Biomaterials*. **2002**,23,1679.
[13] Li H, Khor KA, Cheang P. *Biomaterials*.**2003**,24, 949.
[14] X Nie, A Leyland, A Matthews. *Surf. Coat. Technol.* **2000**, 125, 407.