Development of Sol-Gel Nano Hydroxyapatite Coatings on Ti-6Al-4V Implant for Biomedical Applications

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Keywords---*Sol-Gel, Hydroxyapatite and Electrophoretic Deposition*

I. INTRODUCTION

HYDROXYAPATITE is one form of calcium phosphate based bioceramic, its chemical formula is $Ca_{10}(PO_4)_6(OH)_2$ and shown as HAP. Due its good biocompatibility and bioactivity used in biomedical applications such as orthopaedic and dental fields [1]. Metallic biomaterials, such as titanium and its alloys are being extremely used as dental and orthopaedic implant materials due to their good biocompatibility, superior mechanical strength, and corrosion resistance in physiological environment [2, 3]. Electrophoretic Deposition (EPD) has been found to be an efficient technique to prepare ceramic coatings from powder suspensions and it is an easier process for obtaining nano structural deposits from colloidal solutions. It has been reported that nanosized crystals are very efficient to improve the contact and stability at the artificial/natural bone interface observed *in vitro and in vivo* environments.

II. MATERIALS AND METHODS

The calcium nitrate tetrahydrate and triethyl phosphite were taken as calcium and phosphorus sources respectively. A stoichiometric amount of calcium nitrate tetrahydrate was slowly added to the phosphorus solution in order to obtain a Ca/P ratio of 1.67, under vigorous stirring. Aged mixture of solution was refluxed by using oil bath for 16h. The gel was dried at 110°C for 5h, dried gel was grained and sintered at 900°C for 2h. the synthesized powder was characterized by FT-IR, XRD, and AFM analysis.The voltage was set at 60V and the deposition time was varied from 1, 2 and 3min respectively. The uniform coatings were obtained and dried at 85°C for 2h and sintered the coated implant at 500°C for1h.

III. RESULTS AND DISCUSSION

The calcium nitrate tetrahydrate acts as oxidizing agent and triethyl phosphite is a complexing agent. In the formation of sol, triethyl phosphite undergoes hydrolysis and condensed with calcium ion. Ca-O-P bond was formed due to strong reactivity between the calcium, phosphorus precursors to induce the formation of hydroxyapatite.

- *FT-IR Spectroscopy*

The FT-IR spectrum (Fig.1.) of raw powder sintered at 900°C exhibited a strong absorption in three regions: (i) the band at 3577 cm^{-1} and the vibration band at 636 cm-1 are originated from OH- group. (ii) The bands at 961 cm^{-1} are characteristic bands of PO_4 -3 ions (γ1 band), 1095 and 1047 cm^{-1} (γ3 band) and 566 and 602 cm-1 (γ4 band). (iii) The presence of 1407-1634 cm^{-1} peak confirms the presence of carbonate group. These characteristic peaks are indicates the presence of hydroxyapatite.

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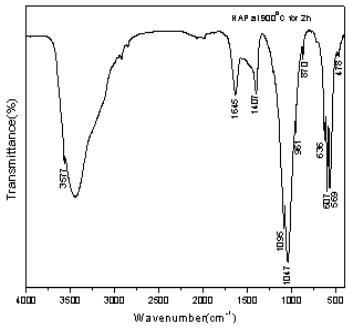


Fig.1 FT-IR Spectra of HAP Sintered at 900°C

- *Powder-XRD*

Sol gel synthesized HAP phase was investigated using powder XRD analysis and illustrated in Fig.2. The XRD pattern of nanosized HAP was compared with JCPDS≠99-100-8866 which confirmed the presence of the hydroxyapatite along with some trace amount of tri calcium phosphate (JCPDS#100-6972). The peaks at 27.2°, 28.73°, 31.03°, 34.10° belong to ß-TCP, indicated with star mark (*) and unmarked peaks are identified as HAP peaks.

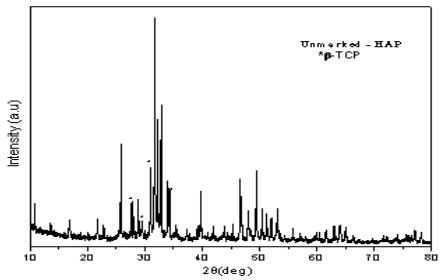
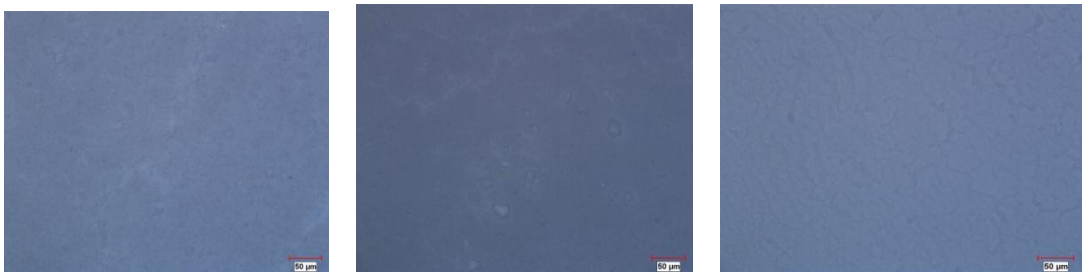


Fig.2. Powder XRD Pattern of HAP at 900°C for 2h

- *Optical microscopy*

The optical microstructures (Fig.3.) of HAP coated Ti-6Al-4V reveals the information about the porosity and uniformity. The 60V for 1min and 2 mins have shown high pores when compared to with the coating derived from 3mins. The adherent coating was obtained at 60V for 3 min (Fig.3c.) when compared with the other coatings.



(a) (b) (c)

Fig.3. Optical Micrographs of HAP Coated on Ti-6Al-4V (a) 60V for 1 min, (b) 60V for 2 mins, (c) 60V for 3 mins

Atomic Force Microscopy

The surface roughness and morphology of sintered HAP was studied by AFM (Fig.4.). From AFM analysis it was found that the HAP powders are nano sized particles with the size of 16.4 nm and was spherically arranged.

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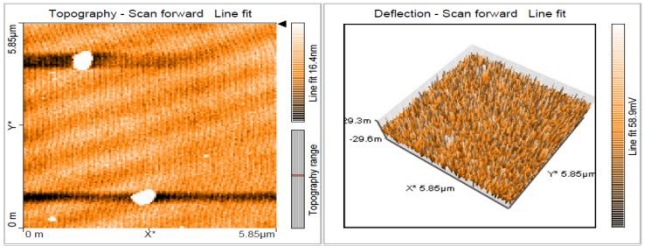

Fig.4. AFM Image of the Nano Spherical Powder

Further study is in progress for the evaluation of Corrosion resistance of the developed coating by electrochemical polarization and impedance analysis using Ringer's solution as an electrolyte medium.

IV. CONCLUSION

The nano sized HAP was synthesized by sol-gel method and its composition was found to be biphasic (HAP+ ß TCP) which was confirmed by powder XRD but the major phase was HAP and particle size was in nano as regime investigated by AFM. The nano sized HAP was coated on Ti-6Al-4V without using any additives by EPD technique. The HAP with ß TCP in the coating mimics the bone composition and gives an added advantage because it leads to enhanced bone bonding ability.

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