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Mechano-tribological properties and *in vitro* bioactivity of biphasic calcium phosphate coating on Ti-6Al-4V

R.R. Behera[a], A. Das[b], D. Pamu[b], L.M. Pandey[c], M.R. Sankar[a,*]

[a] *Department of Mechanical Engineering, Indian Institute of Technology Guwahati, Assam 781039, India*
[b] *Department of Physics, Indian Institute of Technology Guwahati, Assam 781039, India*
[c] *Department of Biosciences and Bioengineering, Indian Institute of Technology Guwahati, Assam 781039, India*

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ABSTRACT

Biphasic calcium phosphate (BCP) consists of hydroxyapatite (HA) and beta-tricalcium phosphate (β-TCP). BCP is mainly used in artificial tooth and bone implants due to higher protein adsorption and osteoinductivity compared to HA alone. Although, many studies have been investigated on radio frequency (RF) magnetron sputtering of HA on Ti and its alloy, however, limited studies are available on BCP coating by this process and its bioactivity and adhesion behavior. Thus, in order to obtain a better understanding and applications of BCP films, RF magnetron sputtering is used to deposit BCP films on Ti-6Al-4V in the present study. The effect of film thickness on wettability, mechanical properties and *in vitro* bioactivity at a particular set of sputtering parameters are investigated. BCP film thickness of 400 nm, 700 nm and 1000 nm are obtained when sputtered for 4 h, 6 h and 8 h, respectively. Although the phase compositions are almost same for all films, the surface roughness values varies around 112–153 nm with rise in film thickness. This in turn enhances hydrophilicity in accordance to Wenzel relation as the contact angle decreases from 89.6 ± 2° to 61.2 ± 2°. It is found that the 1000 nm film possess highest micro-hardness and surface scratch resistance. No cracking of film up to scratch load of 2.3 N and no significant delamination up to load of 7.8 N are observed, indicating very good adhesion between BCP films and Ti-6Al-4V substrate. There is a great improvement in wt% apatite layer formation on all films when dipped in simulated body fluid (SBF) for 14 days. Among these, 1000 nm sputtered film results the highest increase in wt % apatite layer from 44.87% to 86.7%. The apatite layer possess small globular as well as elliptical structure are nucleated and grew on all the BCP films. Thus, sputtering of BCP films improves wettability, mechanical properties as well as bioactivity of Ti-6Al-4V, which can be applied for orthopedic implants.

1. Introduction

Increasing demand of artificial implants encourages the researchers to develop and improve the quality of implant in terms of its functionality as well as durability. Because of lightness, inherent toughness, low density, high fatigue, impact strength and good corrosion resistance, pure Ti and its alloys are widely used in biomedical applications (Davim, 2014; Lauro et al., 2016; Veiga et al., 2013). Ti-6Al-4V is a preferred load bearing material for dental and orthopedic applications owing to its magnificent mechanical properties (Meng et al., 2015; Quek et al., 1999; Veiga et al., 2012). However, Hwang and Choe (2017) reported that Ti-6Al-4V is bio-inert, which cannot exhibit any positive influence on tissue and cell behavior. So, both the osteoblasts and new bone tissues cannot grow well. Therefore bonding between host tissues and the implants are not formed easily, which leads to poor osteointegration. As a result, Ti-based implant is detached from the host tissue in long-term implantation. On the other hand, hydroxyapatite (HA, $Ca_{10}(PO_4)_6(OH)_2$), a calcium phosphate (CaP) based biomaterial is widely used in different implants for repair or replacement of natural tissues due to its excellent osteoconductivity, biocompatibility and osteointegration (Saxena et al., 2018). Singh et al. (2018) revealed that HA has similar mineral constituents found in teeth and bones. Wei et al. (2015) demonstrated that HA has the potential to integrate with the surrounding tissues by forming strong bonds at the interface of tissue and implant. However, HA alone cannot be utilized for load bearing materials because of its higher elastic modulus and lower fatigue strength. Thus, surface modification of Ti-6Al-4V with HA is carried out in order to achieve superior mechanical performance of the metal as well as excellent bioactivity of HA simultaneously. From the findings of Branemark's work in 1969, Ti implant surface showed excellent

* Corresponding author.
E-mail addresses: rasmi@iitg.ernet.in (R.R. Behera), apurba12@iitg.ernet.in (A. Das), pamu@iitg.ernet.in (D. Pamu), lalitpandey@iitg.ernet.in (L.M. Pandey), evmrs@iitg.ernet.in (M.R. Sankar).

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osteointegration when embedded in rabbit bone (Branemark, 1969; Surmenev et al., 2014; Tomsia et al., 2011). However, later it was reported that, these metals have serious shortcomings for better and faster osteointegration of implants. Thus, in order to enhance the osteointegration of Ti and its alloy, CaP bioactive coating was done on the surface of implant (Surmenev et al., 2014). Jimbo et al. (2012) found better osteointegration along the Ti implant with three different CaP coatings compared to uncoated Ti. Meirelles et al. (2008) also observed significant increase in bone formation in nano-HA coated Ti implants after 4 weeks of healing compared to uncoated implants. Nano-crystalline HA coating on Ti accelerated as well as improved the osteointegration, i.e. strengthen the bone-implant integration at early as well as later stage of healing (Yamada et al., 2012).

For surface modification of the metal alloy, several deposition methods have been implemented to obtain HA films over biomedical implants. Dong et al. (2017) prepared HA coating on Ti-6Al-4V using plasma spraying process considering in situ dry ice blasting. Farnoush et al. (2015) investigated HA and HA/TiO_2 coating on Ti-6Al-4V substrate by processing electrophoretic deposition. Rabiei et al. (2006) deposited functionally graded HA coating on silicon substrate using ion beam assisted deposition method. Duta et al. (2017) fabricated HA and Ti doped HA coating on silicon as well as Ti substrate by using pulsed laser deposition. Ge et al. (2011) studied fluoridated HA nano coating on pure Ti plate by electrochemical deposition method. However, these processes have at least one of following shortcomings: (1) poor long term bonding of coating on substrate, (2) non-uniformity of coatings and (3) thermal decomposition of coating material. Therefore, the attention has been focused on obtaining HA coatings over metallic substrates resorting to conventional as well as modified radio frequency (RF) magnetron sputtering. Badea et al. (2016) reported that, this process is a physical vapor deposition method, known to generate uniform, dense, thin, low impurity and strong-adherent coatings with desired crystallinity. Different studies have been carried out to explore the influence of different input variables on different surface structures and mechanical properties of sputtered HA coating on Ti and its alloys. Bramowicz et al. (2016) established the relationship between surface morphology with the elastic modulus and hardness during sputtering of HA film at different sputtering temperatures. Surmenev et al. (2017) sputtered HA film on Ti as well as poly-tetra-fluorethylene substrate and compared surface morphology, Ca/P ratio and compositional phases evolved during sputtering. RF sputtered HA films not only eliminates the shortcomings of other techniques but also have shown excellent biological activity. Thian et al. (2005) reported the formation of extra-cellular matrix and apatite on rat bone marrow cells over films deposited by magnetron sputtering. Mello et al. (2007) found enhanced osteoblast cell adhesion and density on HA coated Ti as compared to HA coated Si substrate by magnetron sputtering.

Besides HA, other calcium phosphates also play important role in biomedical applications for different purpose. Brown et al. (2010) discussed the importance of synthetic bone graft as an addition or substitute to the already established grafting procedures. Biphasic calcium phosphate (BCP) is mainly used for preparation of synthetic bone graft materials. BCP is a mixture of beta-tricalcium phosphate (β-TCP, β-$Ca_3(PO_4)_2$) and HA, exhibits the combined properties of both β-TCP and HA. Though HA is highly stable and β-TCP can be easily resorbed in physiological environment, the combination of two allows us to gain control over the rate of resorption to some extent. Wang et al. (2012) reported that BCP has a higher hydrophobicity than HA, so it can absorb more protein and hence shows better osteoinductivity than HA. Zhu et al. (2009) also found more protein adsorption on porous BCP as compared to dense BCP. Zhang et al. (2011) illustrated the extensive use of BCP in delivery of hormones, antibiotics, drugs as well as repair of nasal septum. Zhu et al. (2008) reported the applications of BCP in artificial bone and tooth implants due to its excellent biocompatibility. Considering advantages of BCP over HA, Zhang et al. (2011) developed BCP/Ti nanocomposite coating on Ti-6Al-4V using laser coating. Benhayoune et al. (2010) used pulsed elecrodeposition to develop monophasic and biphasic calcium phosphate coating on Ti-6Al-4V. Recently, Prosolov et al. (2017) deposited BCP films by RF magnetron sputtering using targets of HA and TCP, sintered at different ratios. They found decrease of average element size of the coating surface with increase of TCP in the target. However, Ca content increased with the increase in TCP in the target.

Although, many studies have been investigated on magnetron sputtering of HA on Ti and its alloy, however, limited studies are available on BCP coating on Ti-6Al-4V by sputtering and its bioactivity and adhesion behavior. So, it is essential to deposit BCP films by magnetron sputtering for better understanding and application of BCP thin films through this process. Thus, in the present experimental investigation, a thin film of BCP coating is carried out on Ti-6Al-4V substrate by RF magnetron sputtering. The effect of film thickness on molecular phase, surface morphology and elemental composition is studied. In addition, the wettability, hardness and surface scratch resistance of different coated substrates are investigated and compared with uncoated Ti-6Al-4V. The *in vitro* bioactivity is investigated by soaking the sputtered substrates in simulated body fluid (SBF).

2. Materials and methods

2.1. Materials

Hydroxyapatite powder is used as precursor to develop the target for sputtering process. The powder is synthesized from abundantly available raw fish scales using method described by Kongsri et al. (2013), however, with slight modifications. After calcinating at 800 °C, HA powder is processed in high-energy ball mill (M/S Fritsch, pulverisette, Germany) for 10 h to get nano-HA powder. The synthesized nano-HA is then characterized for phase composition, particle morphology, elemental composition and particle size using X-ray diffractometry (XRD, M/S Rigaku TTRAX 3, USA), field emission scanning electron microscopy (FESEM, M/S Zeiss-Sigma, Germany), energy dispersive spectroscopy (EDS) and particle size analyzer (M/S Beckman Coulter Delsa Nano, USA), respectively.

Ti-6Al-4V plates with an area of 20 mm × 15 mm and 3 mm thickness are cut by wire electrical discharge machining process and used as substrate for sputtering process. Before coating, the substrates are mechanically polished using SiC abrasive paper up to 1200 grit, and then ultrasonically rinsed with acetone for 20 min in order to remove oil as well as other residues from the surface of the substrate.

2.2. Deposition of BCP films

Coating of HA is carried out on Ti-6Al-4V substrate by RF magnetron sputtering (M/S Advanced Process Technology, India) process. The schematic diagram and photograph of RF magnetron sputtering experimental set up are shown in Figs. 1 and 2, respectively. A cylindrical target of 62 mm diameter and 4 mm thickness is made by sintering the compressed HA powder at 1300 °C for 5 h. The selection of sintering temperature and time is done to obtain BCP films consisting of β-TCP and HA approximately in same proportion. The depositions are carried at room temperature. To begin with, the coating chamber is pumped down to a base pressure of 3×10^{-6} mbar. Maintaining the target to substrate distance approximately at 5 cm, the films are deposited at an RF power of 30 W.

Highly pure Argon gas (99.99%) is used as sputtering gas. With the help of mass flow controller, sputtering gas is introduced into coating chamber and a constant sputtering pressure of 3×10^{-2} mbar is maintained during the entire process. Three sets of film thickness are considered in the present study by varying the deposition times (4 h, 6 h and 8 h) and keeping all other parameters constant. The substrates are taken out for further characterization.

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