FORMATION OF NANOCRYSTALLINE HYDROXYAPATITE THIN FILM BY ROOM TEMPERATURE SPRAY

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Introduction

Hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$, HA) is the major mineral component of bone and teeth [1]. HA coatings have shown promising effects on rapid bone remodeling and suitable functional life in orthopedic and dental applications [2]. However thermal sprayed coatings as plasma spray have some drawbacks such as poor adherence to the substrate, chemical inhomogeneity and high porosity [3]. Together with chemical inhomogeneity, thick coated layer (~60 mm) by plasma spraying could induce delamination and exfoliation of the coatings and thus decrease lifetime of the implants [4]. These problems are associated with the excessively high processing temperature.
Room temperature (RT) spray coating, which is also called aerosol deposition, is attractive as a novel fabrication technology for ceramic coating at room temperature without any heat damage between the substrate and the film. Furthermore, the raw ceramic powder is not decomposed on the molecular level during the deposition process, thus stoichiometry of the films can be obtained [5].
Xenogeneic bone is usually of bovine origin, easy to obtain, and available in unlimited supply with low cost [6]. HA materials manufactured from bovine bones have an advantage that it inherits some properties of the raw materials such as its chemical composition and structure [7]. Therefore, it seems to be an alternative for kinds of products based on synthetic HA. The aim of this study is to coat dense HA film derived from xenogeneic bone using AD method on the Ti6Al4V substrate.

Experimental

Bovine bone was used as a raw material to fabricate the HA thin films. The organics in bone ash were removed by soaking in a 0.1 M NaOH solution at 80 ˚C for 4 h and by calcination at 800 ˚C for 1 h. The HA precursor powder was finally obtained by ball milling of calcined bones for 8 h. Commercial Ti6Al4V discs were used as a substrate of coating. RT spray is a film fabrication method that utilizes an impact solidification phenomenon of submicron-sized particles [6]. The experimental conditions of coating process were fixed as gas flow rate of 5 l/min and deposition time was 5 min at room temperature. The pressure difference between the aerosol chamber and deposition chamber was 1 atm. The HA powder of 0.4 mm in sized was mixed and vibrated with a He carrier gas to form an aerosol flow in the aerosol chamber. The vibration speed for particle dispersion was 600 rpm. The distance between the nozzle and substrate was 10 mm, and orifice size of nozzle was 0.3 mm. The incident angle for deposition was changed from 0˚ to 60˚. The deposition working pressure was 3-4 Torr.

Results and Discussion

Fig. 1 shows the FE-SEM micrograph and XRD pattern of bovine bone-derived HA powder. The powders consisted of submicron-sized crystallites of 0.3-0.5 mm. The particles with weaken agglomeration had a circular structure with uniform shape. XRD pattern showed that the bovine bone-derived HA powder consisted of HA and small amount of MgO. It is well known that inorganic components of native bone mainly composed of HA and minor amounts of several elements such as magnesium, sodium potassium, etc. [8].

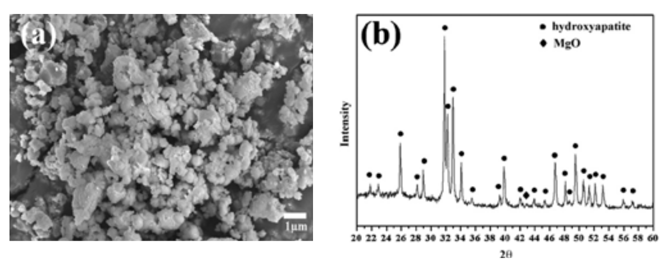

Fig. 1 FE-SEM micrograph and XRD pattern of bovine bone-derived HA powder.

Fig. 2 shows the FE-SEM micrographs of HA thin films formed at incident angles of 0˚, 45˚, and 60˚. In the film deposited with 0˚, many asperities and particles shape

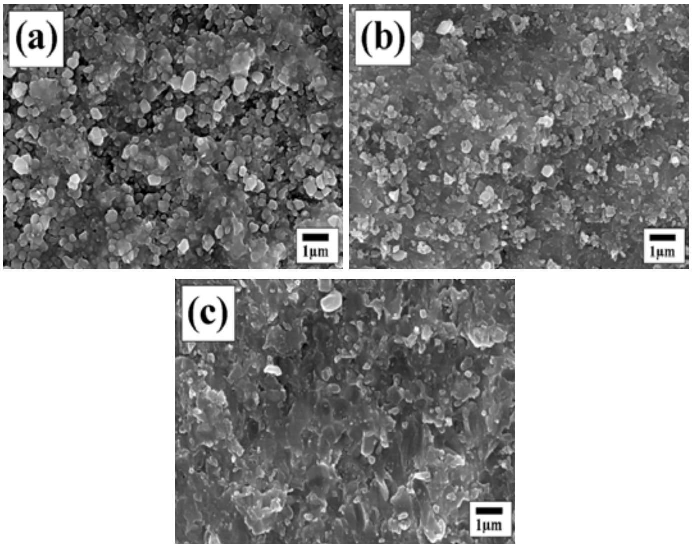

Fig. 2 FE-SEM micrographs of HA thin films at incident angles of (a) 0˚, (b) 45˚, and (c) 60˚.

were observed on the surface. As shown Fig. 2(b) and (c), the HA films with incident angles of 45˚ and 60˚ were smoother and denser than that of the 0˚ case.

HA film with the thickness of 1-2 mm is shown in Fig. 3 for the incident angle of the beam. Although the particle shape was observed for 0˚, it was not observed for 60˚. It was found that fine HA film be produced at oblique incidence of the beam.

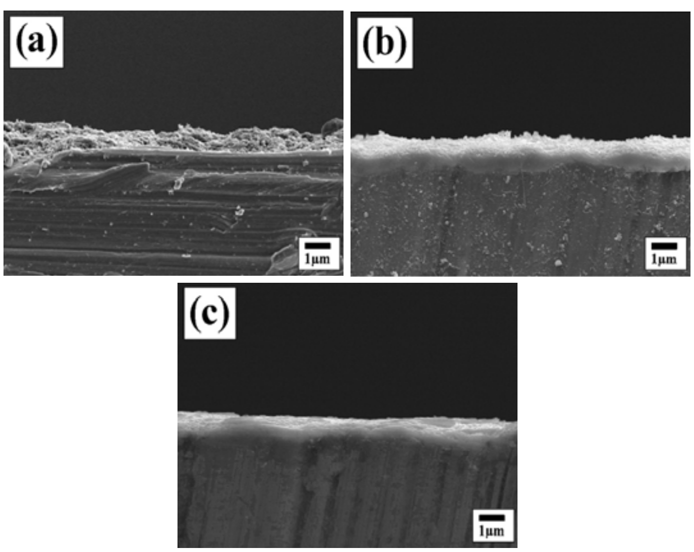

Fig. 3 FE-SEM micrographs of HA thin films formed at incident angles of at (a) 0˚, (b) 45˚, and (c) 60˚ cross-section.

EDS analysis presents that the film is composed of Ca, P and Mg. The semiquantitative analysis of the X-ray for the relative amount of Ca and P revealed that the Ca/P ratio was 1.78 as shown in Fig. 4.

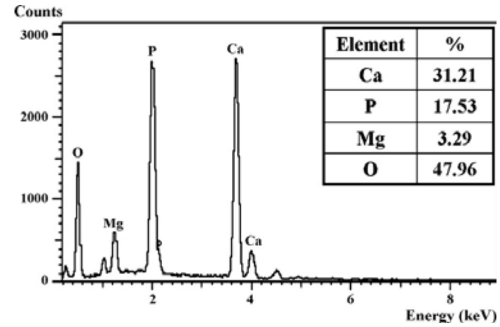

Fig. 4 EDS analysis of bovine bone-derived HA film.

Conclusion

The bovine bone-derived HA thin film with dense microstructure was successfully deposited on the Ti6Al4V substrate at room temperature. Homogeneous HA film was produced by flattering of particles for the incident angle of 60˚. The thickness of deposited film was 1-2 mm. Bovine bone-derived HA coatings can be an alternative to prevent delamination and exfoliation of plasma sprayed the HA coating in biological environment.

Acknowledgements

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