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Apatite Formation on Titanium Containing Apatite

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Abstract

Some titanium containing apatite (TMA) ceramics prepared by wet synthesis was superior to Hydroxyapatite (HAp) ceramics of mechanical property and cutting performance. In this study, we investigated chemical reaction of TMA in a body environment. Each TMA or commercial Hydroxyapatite powder (HAp) was formed to circular pellets by uniaxial pressing. The specimens were heated in muffle furnace by conventional sintering process. Some TMA specimens were dispensed by alkali and heat treatment. After the treatments, the specimens were soaked in simulated body fluid (SBF: Kokubo's solution) at pH 7.25 at 37.0°C up to 7 days. Before and after soaking in SBF, the surfaces of the samples were characterized by XRD, SEM, and ICP. After soaking in SBF for 7days, some particles were precipitated on the surface of HAp ceramics and TMA ceramics with alkali and heat treatment. Ti-containing in the crystal structure of HAp affected apatite formation in the body environment.

Keywords: TMA specimens; HAp ceramics; TMA ceramics

Introduction

Hydroxyapatite (HAp) is one of the components of human bone and the HAp ceramics are used by bone substitutes [1]. But mechanical properties of the HAp ceramics were not fitted at human bone due to the brittleness. Thus some composite materials consisting of HAp and other compounds, and tricalcium phosphate ceramics as biodegradable materials have been investigated [2,3]. Titanium (Ti) and its alloy are attractive for biomaterials as its mechanical property, corrosion resistance and low toxicity [4]. Ti metal induced by alkali treatment was tested in a body environment and apatite was formed on the surface [5]. Some Ti containing apatite (TMA) ceramics, which powder prepared by wet synthesis, was superior to HAp ceramics of mechanical property and cutting performance. In this study, we investigated chemical reaction of TMA in a body environment.

Materials and Methods

Sample preparation

Each commercial Ti containing apatite powder as Ti medical apatite (TMA: $Ca_{10}(PO_4)_6TiO_3 \cdot nH_2O$, TMA*, Imuno Science, Co.Ltd.) or commercial hydroxyapatite powder (HAP: $Ca_{10}(PO_4)_6(OH)_2$, HAP100, Taihei Chemical Industrial Co., Ltd) was formed to circular pellets by uniaxial pressing. The specimens were heated in muffle furnace to 1000°C at a rate of 20°C/min, and kept for 10 h, followed by cooling to room temperature at the natural cooling rate of the furnace. These TMA and HAP specimens are denoted as TMA1000 and HAP1000, respectively. Some TMA1000 specimens were soaked in NaOH solution at 60°C for 6 hours, dried at 40°C for 24 hours, and heated at 400°C for 1 hour. These alkali treatment specimens are denoted as TMA-AT [5].

Body environmental evaluation

TMA1000, HAP1000, and TMA-AT were soaked in simulated body fluid (SBF: Kokubo's solution, Na⁺ 142.0, K⁺ 5.0, Mg²⁺ 1.5, Ca²⁺ 2.5, Cl⁻ 147.8, HCO₃⁻ 4.2, HPO₄⁻²⁻1.0, and SO₄⁻²⁻0.5 mmol/dm³) at pH 7.25 at 37.0°C up to 7 days [6,7]. These samples were taken out from the fluid, gently rinsed with pure water and dried at room temperature.

Sample evaluation

The starting powders and the surfaces of the samples before and after soaking in SBF were characterized by Powder (PW-XRD) or Thin Film (TF-XRD) X-ray diffraction (XRD: XRD-6100, Shimadzu Corp.), and scanning electron microscopic (SEM, VE-9800, Keyence Corp.) observation. The Ca and P concentration of SBF before and after soaked samples was evaluated by inductively coupled plasma (ICP, Optima 4300DV, Perkin Elmer Co., Ltd.).

Results and Discussion

TMA and HAP-100 powders were similar crystal phase (Figure 1). Crystallinity of TMA was lower than that of HAP-100. As TiO_3 groups in TMA were inside in the HAp structure, the crystallinity of TMA would be decreased.

After heating TMA at 1000°C, almost all of the peaks were attributed to β - TCP (Figure 2). A transition temperature from HAp to β -TCP might be lowered as containing Ti in HAp structure.



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After soaking in SBF for 3 days, no precipitates were formed on the surface of HAP1000. But for 7 days, precipitates were covered the surface on HAP1000 (Figure 3). After soaking in SBF for 1 day, crystal nuclei were formed on the surface of TMA-AT. Then, the crystal growth was observed on the surface of TMA-AT after soaking in SBF for 3 and 7 days. The peaks at 26° and 32° assigned HAp were detected in HAP1000 and TMA-AT. The precipitations were assigned low crystalline HAp. On the other hand, no precipitations were formed on the surface on TMA1000. The surface crystal phase of TMA1000 didn't change.

Ca and P concentrations in SBF after soaked TMA-AT and HAP1000 were gradually decreased (Figure 4). These changes match the surface HAp formation on TMA-AT and HAP1000. The precipitations were not formed on the surface of TMA1000 after soaking in SBF for 7 days nevertheless of the degradation of Ca and P concentration. In SBF after soaked TMA1000, homogeneous nucleation might be induced. The difference of the formation of deposits would be caused by surface chemical-bonding state. An adequate surface chemical-bonding state might bring Ti containing apatite in HAp formation in a body environment.

Conclusions

Low crystalline HAp was formed on the surface of Ti containing apatite dispensed by alkali treatment in a body environment. Ti and its alloy are attractive for biomaterials as its mechanical property, corrosion resistance and low toxicity. Therefore, Ti containing apatite ceramics were expected to be more safety and to match mechanical property to bone as bone substitutes.



After soaking

Figure 2: TF-XRD patterns of HAP1000, TMA1000, and TMA-AT before and after soaking in SBF for 7 days.



Figure 3: SEM photos of HAP1000, TMA1000, and TMA-AT before and after soaking in SBF for 1, 3, and 7days. Scale bar: 10 mm.



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