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Apatite formation on CO_2 laser irradiated titanium oxide films

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Abstract

Biomimetic apatite-forming ability on CO_2-laser-irradiated titanium oxide films was examined. Sol–gel-derived amorphous titanium oxide films were crystallized after irradiation of a continuous wave CO_2 laser at the power of 5 W. The irradiation induced crystallization of anatase. The apatite-forming ability on the irradiated titanium oxide film was investigated by soaking in a solution with ion concentration 1.5 times those of simulated body fluid (1.5SBF). X-ray diffractometry showed that apatite formed on the laser-irradiated titanium oxide film within 5 days in 1.5SBF, while no apatite formed on the non-irradiated (amorphous) film. The apatite forming ability can be enhanced and given at a desired position by using the CO_2 laser-irradiation.
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Keywords: Titanium oxide; Anatase; CO_2 laser; Apatite formation

1. Introduction

Titanium and titanium-based alloys have been widely used as implant materials [1]. Titanium presents better physico-chemical stability and mechanical integrity of the processed implants as compared to other conventional metal-based implants. This advantageous interaction between bone tissues and titanium implants is believed to be linked to titanium oxide on their surface [2,3].

Numerous studies for evaluation of biomimetic apatite formation on Titanium oxide in simulated body fluid (SBF) or the biocompatibility of titanium metals and its alloys as implants have been carried out. Most of them are concentrated to improve the surface properties of the samples [4]. For example, when the metallic titanium is soaked in NaOH aqueous solution [5] and then heated at 600 °C, an amorphous sodium titanate layer is formed around its surface [6,7]. Na^+ ion is released from the surface layer in the SBF and exchanged with H_3O^+ ion in the fluid. As a result, numerous Ti–OH groups are formed on their surfaces, inducing the apatite nucleation [8]. Treatment of metallic titanium with H_2O_2 was also reported to enhance the apatite formation in SBF [9].

Titanium oxide containing anatase and sol–gel-derived anatase were shown in a number of studies to be effective in inducing apatite formation [10,11]. Maeda et al. has found that apatite formed on the composites of calcium carbonate/anatase and calcium carbonate/rutile in SBF [12] which supports the findings of Kasuga et al. [13] where the enhancement of apatite formation on the UV-irradiated anatase powders [13]. The apatite-forming ability of the anatase phase is shown to be higher than that of the rutile phase [14,15]. Thus the excellent apatite-forming ability on titanium oxide is closely related to the presence of Ti–OH group and/or anatase phase.

Although many methods have been investigated to enhance the apatite formation, little attention has been paid to the pulsed laser deposition. In the present work, attention was concentrated to crystallize titanium oxide films using CO_2 laser irradiation. When the laser irradiation to the film could induce crystallization of anatase, the apatite formation would be enhanced. This technology shows the potential to be in the preparation of artificial hip joints or dental implants.

2. Experimental procedures

Titanium oxide thin films were prepared through a hydrolysis of titanium (IV) *n*-butoxide with a mixed solution of water, ethanol and acetyl acetone. The acetyl acetone was used as a chelating agent at room temperature [16]. The molar

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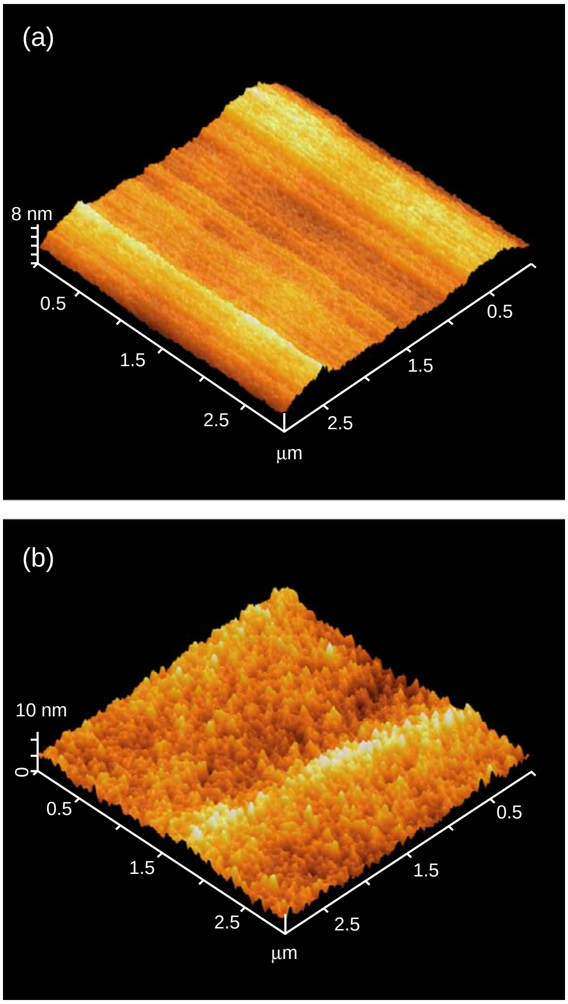

Fig. 1. AFM images of (a) the as-prepared titanium oxide films dried at 250 °C sample and (b) the films after CO_2-laser-irradiation.

ratio of titanium n-butoxide : acetyl acetone : ethanol : water was 1 : 1 : 26.5 : 1. The resulting solution was clear and colorless. The film was deposited on a silica glass substrate by withdrawing at a speed of 3 mm/s, followed by annealing at 250 °C in air for 15 min. The film was then cooled to room temperature. This heating–cooling process was repeated three times. The obtained films were irradiated with CO_2 laser at 10.6 µm wavelengths, with a fixed frequency 5 KHz. The laser was operated at 5-W power setting using a focused continuous waveform reflected from gold mirror at a scan speed 10 µm/s. The distance between mirror and the target surface was fixed at 7 cm. The calculated laser power density was 52 W/cm^2 and the working time was 5.8 min.

In order to know the possibility of the apatite formation, simulated body fluid (SBF) was used. The SBF is already supersaturated concerning the apatite. In the present work, modified SBF, whose supersaturation was increased and used to confirm the apatite formation. The apatite formation was examined by soaking the sample for 5 days at 37 °C in 100 ml of an aqueous solution with ion concentrations 1.5 times of those SBF (1.5SBF). The 1.5SBF includes of Na^+ 213.0, K^+ 7.5, Mg^{2+} 2.25, Ca^{2+} 3.75, Cl^- 222.45, HCO_3^- 6.3, HPO_4^{2-} 1.5 and SO_4^{2-} 0.75 mM. 1.5SBF was prepared by dissolving regent grade chemicals of NaCl, Na_2SO_4, KCl, $MgCl_2.6H_2O$, $CaCl_2$, Na_2HPO_4 and $NaHCO_3$ in ion-exchanged water and buffered at pH 7.40 with tris-hydroxymethylamminomethane $(CH_2OH)_3$ CNH_3 and diluted HCl.

The obtained films were characterized by atomic force microscopy (AFM), thin film X-ray diffractometry (TF-XRD), laser Raman spectroscopy and scanning electron microscopy (SEM) incorporating X-ray energy dispersive spectrometry (EDS).

3. Results and discussion

The surface morphologies of titanium oxide films deposited on silica glass were observed by AFM before and after laser irradiation. Fig. 1(a) shows the surface morphology of the as-deposited titanium oxide film heated at 250 °C for 15 min; a relatively smooth surface is seen. After the laser irradiation, a slightly rough surface was formed as shown in Fig. 1(b). Keshmiri et al. [17] proposed that the apatite formation on the titanium oxide surface was affected by the surface morphology. Considerably higher apatite formation rates on the anatase microspheres was found in comparison with to those on the sol–gel-derived titanium oxide thin films demonstrating the nucleation and growth of apatite on bioactive materials soaked in SBF which are influenced by their surface morphologies [18]. Therefore, the change in the surface morphology (rough surface) as shown in Fig. 1(b) may increase the number of apatite nucleation sites.

Fig. 2 shows TF-XRD patterns of the film surfaces. Fig. 2(a) illustrates an amorphous pattern of the film heated at 250 °C. After the laser-irradiation [Fig. 2(b)], the peaks at $2\theta \approx 25.30$ and 36° corresponding to anatase crystal are seen. After soaking in 1.5SBF, the peaks of apatite are seen at 2θ ≈ 26.00 and 32.00 degrees. This pattern is similar to that of the biomimetic apatite formed by the soaking in SBF as shown in references [3,5–13]. No anatase peaks are seen.

The laser Raman spectroscopy of the titanium oxide films are shown in Fig. 3. The film dried at 250 °C shows no anatase peaks detected on the titanium oxide surface as shown in Fig. 3(a). After the laser-irradiation, peaks at 143, 395, 519, and 640 cm^{-1} corresponding

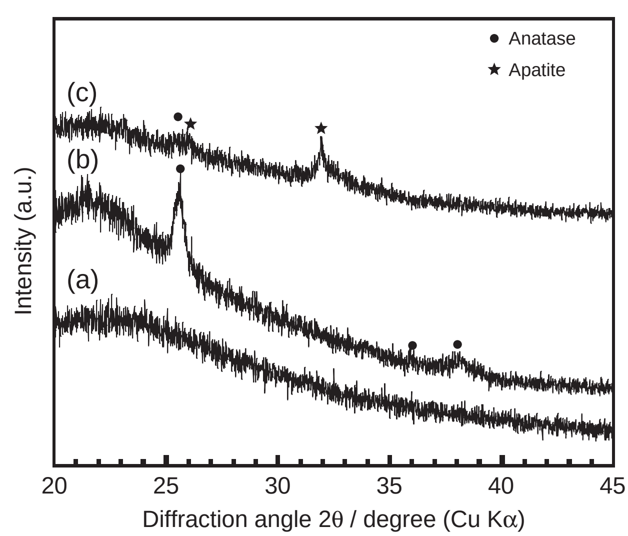

Fig. 2. TF-XRD patterns of (a) the as prepared titanium oxide films dried at 250 °C, (b) after CO_2-laser-irradiation at 5 W, and (c) after 5 days of soaking the irradiated sample in 1.5 SBF.

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