



Apatite-forming ability of titanium compound nanotube thin films formed on a titanium metal plate in a simulated body fluid

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ABSTRACT

We compared the apatite-forming ability of a sodium titanate nanotube thin film, an anatase-type titanium dioxide nanotube thin film, and a silver nanoparticle/silver titanate nanotube nanocomposite thin film, in simulated body fluid. The ability of the silver nanoparticle/silver titanate nanotube nanocomposite thin film is slightly higher than that of the anatase-type titanium dioxide nanotube thin film and significantly higher than that of the sodium titanate nanotube thin film. The high ability of the silver nanoparticle/silver titanate nanotube nanocomposite thin film is a newly observed phenomenon, which is probably due to the crystal structure of silver titanate – specifically, to the surface atomic arrangement, the large amount of Ti–OH formed on the nanotube surface, or both. The anatase-type titanium dioxide nanotube thin film and the silver nanoparticle/silver titanate nanotube nanocomposite thin film may have bright prospects for future use in implant materials such as artificial joints. The silver nanoparticle/silver titanate nanotube nanocomposite thin film is particularly promising for its antibacterial properties.

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1. Introduction

Kim et al. [1] first reported the formation of a sodium titanate thin film on a titanium metal plate by alkali and heat treatment and demonstrated the high apatite-forming ability of the obtained sodium titanate thin film. Since then, researchers have actively performed many *in vivo* and *in vitro* studies on the applications of sodium titanate thin films in implants [2–11]. Similar studies have also been performed on calcium titanate thin films [12–16], titanium dioxide thin films [17–20], and a nanohydroxyapatite thin film [21], and the excellent biocompatibilities of these films have been reported. Therefore, titanium compound thin films show tremendous promise for use as implant materials. Sodium titanate particles exhibit various nanostructures and morphologies, such as nanowires, nanosheets, and nanotubes [22]. These structures vary according to the types of titanium sources, concentrations of alkaline aqueous solutions (such as aqueous sodium hydroxide solution), and reaction temperatures. In particular, titanate and titanium dioxide nanotube particles synthesized by hydrothermal treatment have attracted much attention [23,24] because they have large specific surface areas, ordered one-dimensional internal

spaces with a diameter of approximately 4 nm, and various potential applications in photocatalysis, dye sensitizing solar batteries, hydrogen storage, electrochromism, proton conductors, electron field emissions, and photoinduced hydrophilicity. Extensive studies have been performed regarding the thin film formation of titanate nanotube thin films formed on titanium metal plates [25–28]. Our group has also reported the formation of various shapes of a sodium titanate nanotube thin film on titanium metal surfaces [27], and demonstrated the transformation of a sodium titanate nanotube thin film into a titanium dioxide nanotube thin film [29]. Our ultimate research goal is to develop antibacterial implant materials. To this end, we are investigating the formation of nanotube thin films on the surfaces of implants that are composed of titanium or titanium alloys, such as artificial joints. Since nanotubes can enclose various substances in their internal spaces, implants coated with nanotubes could be highly functionalized by incorporating substances that could stimulate osteogenesis or possess antimicrobial action.

Various reports of *in vivo* and *in vitro* apatite formation are relevant to this study. Principal among these is the study of Kasuga et al., who have reported the ability of titanate nanotubes to form *in vivo* and *in vitro* apatite [30–32]. However, although their patent [30] states that they reported the apatite-forming ability of “titanium dioxide” nanotubes, they particularly reported the apatite-forming ability of “titanate” nanotubes. In evaluating the

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in vivo apatite-forming ability of a material, researchers commonly perform experiments in simulated body fluid (SBF) [33], and indeed Kasuga et al. reported the apatite-forming ability of titanate nanotubes in SBF [30,32]. However, of the 14 cases that they examined, 13 concerned titanate nanotube powders and only a single case concerned a calcium titanate nanotube thin film, whose apatite-forming ability was reported to be very high because of elution of Ca^{2+} . But the characteristics of a nanotube thin film fixed onto a substrate still remain largely unknown. The process to fix the nanotube powders onto a substrate as a thin film is as follows: (1) preparation of a nanotube powder dispersion liquid, (2) coating of the prepared liquid on the substrate, and (3) fixation of the coated nanotube

powders onto the substrate by calcination. A large change in the apatite-forming ability is expected during this multi-step process. We have reported growing titanate nanotubes directly on the surface of the titanium metal plates by the hydrothermal treatment of titanium metal [27]. Since the synthetic method (raw material, reaction temperature, reaction time, washing, and ion exchange) and the thermal history of the titanium compound nanotube thin films reported in our present research are entirely different from those used by Kasuga et al. [30–32], the apatite-forming ability of our products is likely to differ from that reported by them.

Our research aims at developing antibacterial implant materials. We recently reported the formation of a silver nanoparticle/silver titanate nanotube nanocomposite thin film and the behavior of elution of Ag^+ from this film [34]. The film shows high antibacterial activity against methicillin-resistant *Staphylococcus aureus*. We synthesized the silver nanoparticle/silver titanate nanotube nanocomposite thin film from a sodium titanate nanotube thin film by a silver ion exchange treatment. During the exchange of Na^+ ions for Ag^+ ions, the layered structure of titanate transforms into a new three-dimensional crystal structure. The apatite-forming ability of a silver titanate nanotube thin film having this new crystal structure has not yet been reported. The present study primarily aims at evaluating the apatite-forming ability of titanium compound nanotube thin films by comparing the apatite deposition behaviors of a sodium titanate nanotube thin film [27], a titanium dioxide nanotube thin film [29], and a silver nanoparticle/silver titanate nanotube nanocomposite thin film [34], in SBF.

2. Experimental procedure

2.1. Synthesis of titanium compound nanotube thin films

We performed the formation of a sodium titanate nanotube thin film on titanium metal according to the methods put forward in our previous report [27,29,34]. By immersing a titanium metal plate in 20 mL of a 10 M NaOH solution and performing hydrother-

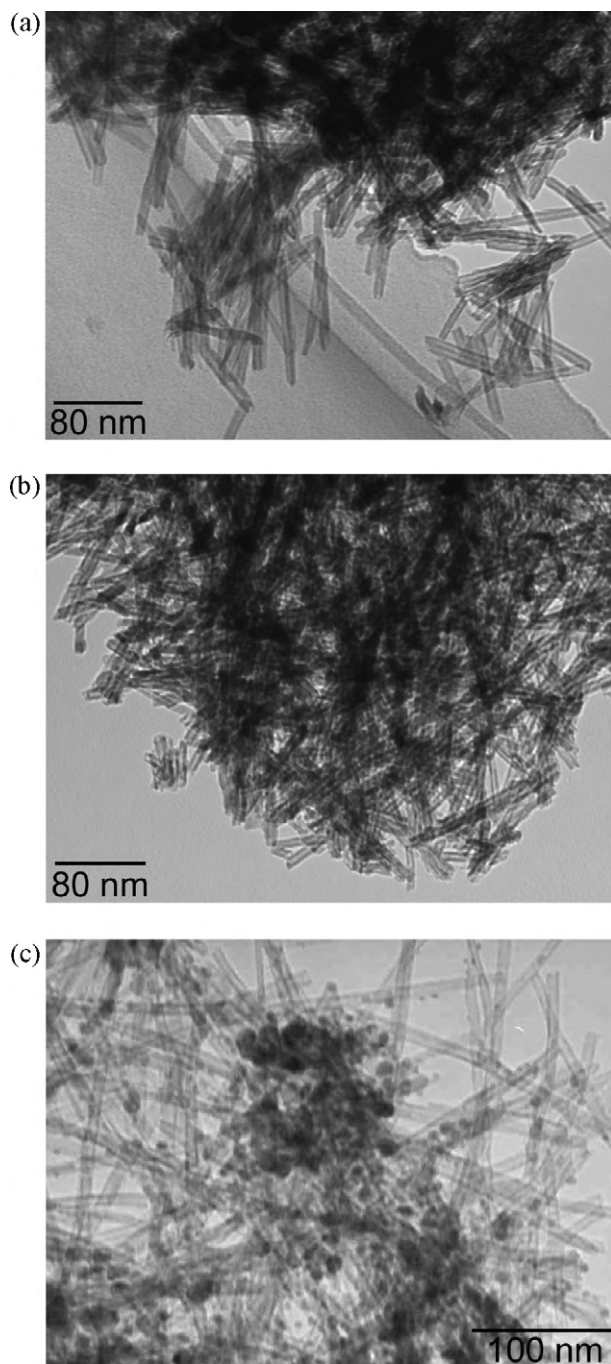


Fig. 1. TEM images of sodium titanate nanotube (a), titanium dioxide nanotube (b), and silver nanoparticle/silver titanate nanotube nanocomposite (c) thin films prior to immersion in SBF.

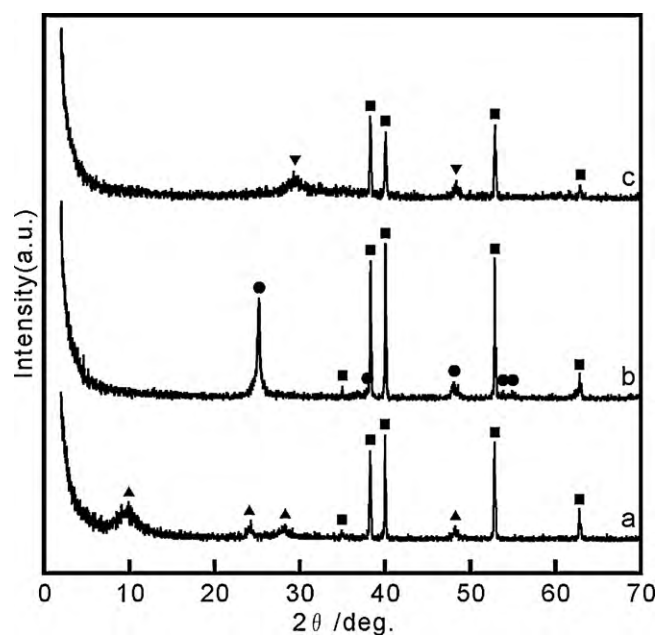


Fig. 2. XRD patterns of sodium titanate nanotube (a), titanium dioxide nanotube (b), and silver nanoparticle/silver titanate nanotube nanocomposite (c) thin films prior to immersion in SBF. Peak assignment: (■) α -titanium; (▲) sodium titanate; (●) anatase-type titanium dioxide; (▼) silver titanate.

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