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Development of bioactivity and pull-out torque control technology on Ti implant surface and its application for cold thread rolled bone screw

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Abstract

The influence of cold thread rolling conditions and a production method of titania (TiO_2) films on surface bioactivity and pull-out torque of a titanium bone screw was investigated. The bone screw with micro surface roughness distribution was formed by cold thread rolling with a pair of parallel dies. The die shape and surface roughness distribution were changed to the 3 grades. The TiO_2 films were coated on the surface of the bone screw using anodizing in aqueous solutions (hydro-coating). We introduced the rolled bone screws into tibia of rats for two weeks and examined the effects of the combinations of the surface morphology and the TiO_2 film on osteoconductivity in an in-vivo experiment. As the results, it is found that we could control the bioactivity and pull-out torque by controlling the surface roughness at the bottom of the screw root.

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Keywords: Thread rolling; Surface modification; Bone screw

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1. Introduction

The world has been aging, and the number of the osteoporosis and the other orthopaedic diseases is increasing. Some implant products, such as bone screws and artificial hip joints, therefore have often been used in surgical operations. It is important to heal in a short time, however the formation of new bone on the surface of implants needs a long time. In addition, implant products sometimes drop out from the bone and patients are forced to bear the burden of retreatment and its pain. To overcome the problems, an enhanced bioactive film had to be developed to coat the surface of the metallic implants to promote high comprehensive bone growth (osteoconductivity). The osteoconductivity was accordingly evaluated on hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$:HAp), titania (TiO_2) by Kuroda et al. (2006, 2007) and Yamamoto et al. (2011), HAp-TiO_2 composite films (Kuroda et al., 2007) and the other coating materials. Moreover the influence of processing method of the bioactive films was investigated by Okido et al. (2011), however its optimum condition has not yet been revealed. Ramsden et al. (2007) have showed the importance of the selection of implant material and the surface treatment. Taddei et al. (2013) have been researching bioactivity of titanium (Ti) and fluorinated apatite coatings for orthopaedic implants. Xu et al. (2009) have evaluated the surface bioactivity of a calcium phosphate coated magnesium alloy. Kumar et al. (2007) focused on bioinert stainless steel screws and applied plasma processing for inducing bioactivity. Denkena and Lukas (2007) studied about biocompatible magnesium alloys and developed a method of controlling the surface and subsurface properties by machining processes. Guo and Salahshoor (2010) also studied about magnesium-calcium alloy and the surface integrity which was created by dry milling was characterized.

However, most biomaterial surface treatments are chemical or mechanical methods and their processing time is generally long and the productivity is relatively low. Alloys have a probability of corrosion in a human body, which phonomenon might have harmful effects. In addition, practical metallic implants have been produced without much consideration of the surface morphology and its optimization.

Hence, we selected pure Ti as the target material and focussed on thread rolling process. The target material has advanced corrosion resistance and has already been certified as a biomaterial. This process is meant to obtain products with a micro asperity in same quality on the surface in a short term. The micro asperity realizes the anchor effect, preventing the dropping-out of the products from bone. Further more it is not difficult to form bioactive titania films on the surface.

In the former study, the pure Ti rod with a micro asperity on the surface had been produced (Yoshida et al., 2012). The surface asperity was created by transcribing the asperity, which was created by shot blasting or electric discharging, on the surface of a flat parallel die. After the thread rolling, a titania film was formed by means of an anodizing. The rods were implanted into tibiae of rats, and observed after 2 weeks. Effects of the combinations of the surface asperity and anodizing condition on osteoconductivity and pull-out torque were investigated. The results showed that the bioactivity and the pull-out torque of the Ti implant surface were able to be controlled by optimizing the surface micro asperity and anodizing. However, the product property of implant should be evaluated not only on the Ti round bars but also on bone screws.

In this study, authors have produced the Ti bone screws with surface asperity distribution and titania coating. The screws are implanted into the tibiae of rats and the bioactivity and the pull-out torque of the screws are estimated. In addition, the influence of the surface asperity distribution and the coating on pull-out torque have been investigated.

2. Experimental methods

2.1. Cold thread rolling of bone screw with surface asperity distribution and TiO_2 surface treatment

A two-stage thread rolling method was developed for the production of the bone screws that have a surface asperity distribution on the surface of the products. The method consists of two thread rolling processes. In the first rolling, a pure Ti rod of 8 mm in length was formed to a screw of 2 mm in nominal screw diameter by means of a pair of parallel dies. Because the surface of the dies has been polished, the surface roughness (Ra) of the screws accordingly is 0.05μm on average.

In the second rolling, the screw is rolled again in another pair of parallel dies with surface asperity distribution. The asperity distribution is transcribed on the surface of the formed screws in the rolling. Fig. 1 shows the two-stage thread rolling and an example of screw produced in this method with TiO_2 bioactive film. We had produced 3 kinds of bone screws and TiO_2 coating was conducted after the rolling (Table 1). The surface asperity was measured by a laser microscope VK-8500 and the roughness was Ra=0.05 and 0.15μm at fine surface area and rough surface area, respectively. In order to produce TiO_2 film, which is a bioactive material, on the surface of the bone screws, an anodizing treatment was performed. In the treatment, the formed Ti screws were anodized in the solution of H_3PO_4 (Fig. 2) in which the molarity of the acid was 1.0 M. The potential was applied at 0.1 V/s between a Ti screw anode and a Pt coil cathode, the total anodizing time was 1000 s.

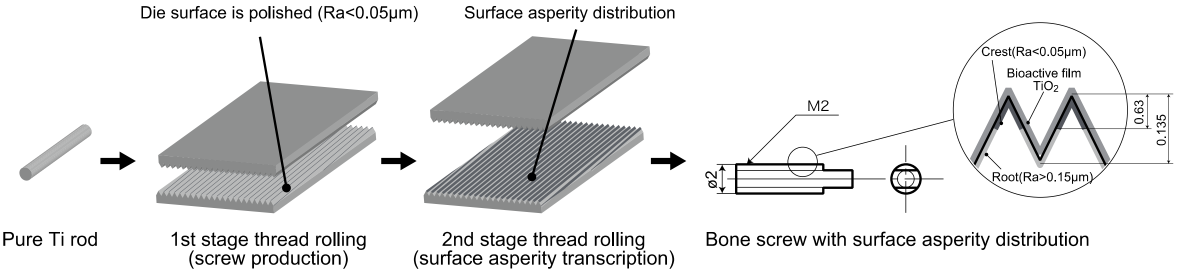


Fig. 1. Schematic diagram of two stage thread rolling and example of bone screw with surface asperity distribution (ex. Condition 2B, asperity on crest is fine and root is rough with TiO_2 film).

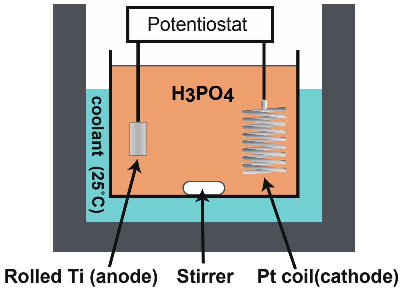


Fig. 2. Schematic diagram of hydro-coating (anodizing).

Table 1. Conditions of cold thread rolling and surface treatment.

Condition	Asperity	Surface treatment
1A	Crest: fine, Root: fine	non coated
1B	Crest: fine, Root: fine	TiO_2 coated
2A	Crest: fine, Root: rough	non coated
2B	Crest: fine, Root: rough	TiO_2 coated
3A	Crest: rough, Root: fine	non coated
3B	Crest: rough, Root: fine	TiO_2 coated

fine: Ra=0.05μm, rough:Ra=1.50μm

2.2. Bioactivity evaluation

A rolled and titania coated Ti screw was implanted into each tibia of a 9 week-old rat. Table 2 shows the implantation conditions. First of all, a rat had been anesthetized and the skin and the muscle of a leg were cut open. Then a 2 mm diameter hole was drilled at a tibia and the Ti screw was inserted into the hole, and after that, the skin and the muscle were sutured. The same operation was conducted for another leg. The rat had been grown for 2 weeks with the Ti screws (Fig. 3). Then the implanted Ti screws and the surrounding bone tissue were sliced perpendicular to the bone longitudinal direction and it was reduced the thickness to 20 μm by polishing using emery paper (Fig. 4). The sliced samples were stained with toluidine blue.

The bone structure consists of cortical bone tissue and cancellous bone tissue. The cortical bone is consisted of hard tissue on the surface of a bone and carries the mechanical load. In the cancellous bone, the 30% of the hard tissue is included and the bone metabolism is active.

Bioactivity was estimated by a bone-implant contact ratio ($R_{B\text{-}I}$) that defined by the next equation.

$$R_{B-I}(\%) = \frac{\text{Hard tissue contact length}}{\text{Total contact length}} \times 100 \tag{1}$$

The $R_{B\text{-}I}$ was estimated in the both of the bone tissue regions. The same 5 samples were implanted and the mean value of $R_{B\text{-}I}$ was used as bioactivity.

2.3. Pull-out torque evaluation

A thread rolled Ti screw was implanted in a tibia with its tenon out of the tibia surface. The bone was fixed in a vice and a torque meter is set on the tenon. After that, the meter was rotated and pull-out torque was output as voltage signals from the meter. At the beginning of the rotation, the value of torque increases and shows the maximum. We defined the pull-out torque as the maximum value.

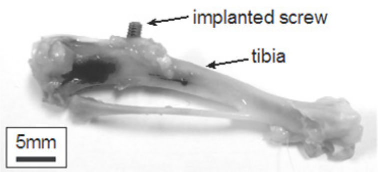
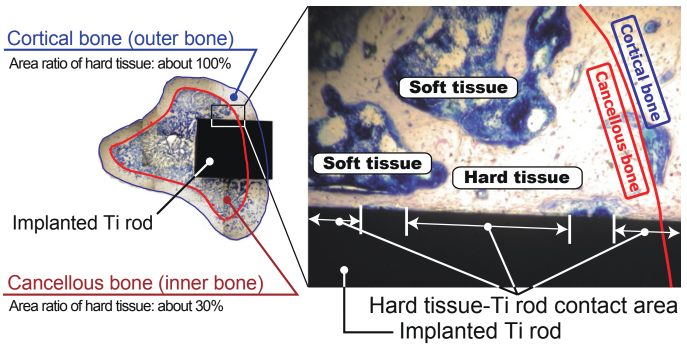


Fig. 3. Tibia with an implanted Ti screw for torque evaluation.

Table 2. Ti screw implantation conditions.

	Condition
Rat	SD-IGS Rat, 9 week old, bull
Installation	both feet, tibia
Temperature	23˚C
Period	2 weeks
Specimen	bone screw (M2), 1A~3B
Sliced sample	thickness: 20µm, staining: toluidine blue
Bioactivity estimation	Bone-Implant contact ratio ($R_{B\text{-}I}$)



(a) Cross section of tibia and implanted Ti rod (b) Enlarged illustration of hard tissue-Ti rod contact surface

Fig. 4. Definition of bone-implant contact area.

3. Results and discussions

3.1. Influence of surface morphology and coating on bioactivity

Fig. 5 shows sliced samples of tibia with the implanted Ti screw in the case of 2A and 2B. In the former sample without titania film, hard tissue formation was not active in cancellous bone and cortical bone. New hard tissue formation however occurred in the case of 2B in which titania coating was conducted. As a result, the titania coating is effective for hard tissue formation on the surface of bone screws.

3.2. Influence of surface morphology and coating on pull-out torque

Fig. 6 shows pull-out torque of Ti bone screws. Pull-out torque in the case of 1A was lower than that of 2A and 3A. Titania coating is not conducted in these conditions and the surface asperity in 1A overall is smooth, under 0.05μm. The torque of 2A and 3A is high because of anchor effect with rough surface on crest or root of the screw. On the other hand, pull out torque is the maximum in the case of 2B in which root of screw is rough and titania coating is conducted. This result shows the advanced effect of the coating and high bioactivity for hard tissue formation at the bottom of screw root. Titania coating is effective in the increase of bioactivity for all condition of surface roughness distribution.

3.3. Relationship between bioactivity and pull-out torque

Fig. 7 shows the relationship between bioactivity $R_{B\text{-}I}$ and maximum pull-out torque. We found that the titania coating is effective for the bioactivity, in particular, at the root of bone screw. In addition, it is showed that pull-out torque also is increased with increase of bioactivity at the root, comparing the result between 3B and 2B. On the other hand, bioactivity at the crest of screw doesn't change by the titania coating.

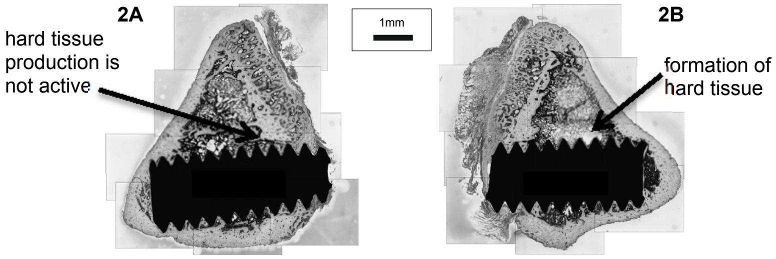


Fig. 5. Sliced sample of tibia with implanted Ti screw (a) with as-rolled surface (2A) and (b) with TiO_2 surface treatment (2B).

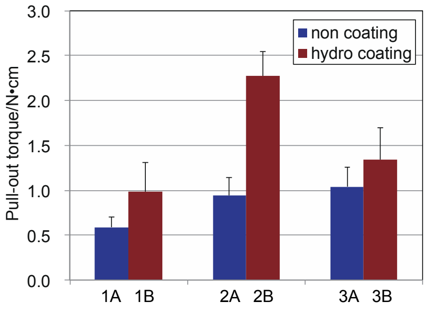


Fig. 6. Pull-out torque of Ti bone screws.

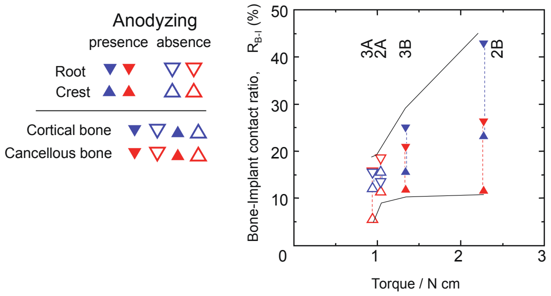


Fig. 7. Relationship between bioactivity $R_{B\text{-}I}$ and pull-out torque.

4. Conclusion

Surface asperity distributions and a titania film were created on the surface of the pure Ti screws in two-stage cold thread rolling. The processed screws were implanted into tibiae of rats and the bioactivities and the pull-out torques were evaluated. The results is concluded as follows,

(1) Titania coating is effective for hard tissue formation on the surface of bone screw.
(2) Titania coating is effective for the bioactivity, in particular, at the root of bone screw.
(3) Pull-out torque also is increased with increase of bioactivity at the root of bone screw.
(4) The pull-out torque of bone screw can be controlled by combination of surface roughness distribution and titania coating.

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