Trace elements on the surface of titanium implants extracted from rat bone

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Abstract In order to make clear the relationship between the surface properties of a dental implant and biocompatibility, trace elements on the titanium surface of the implant extracted from a rat bone were examined. A cpTi bullet, as a prototype, of 1.1 mm diameter and 3.5 mm length had a flat surface on its cylindrical side. The flat area was sandblasted by glassy particle and cleaned by argon sputtering. The surfaces of the implants extracted from the femur of Wistar rats were cleaned ultrasonically and examined by X-ray photoelectron spectroscopy (XPS). The detected main elements were Ti, O, C, and N. Ti peaks arise from the base material and the others indicate presence of bio-molecules including proteins. Trace elements, such as P, Ca and Si, were also detected. However, the Ca/P ratios were very small compared to those of calcium phosphate compounds, such as calcium diphosphate. This fact indicates that calcium phosphate compounds, including hydroxyapatite, don't form on the titanium implant in rat bone.

Key words: dental implant, biocompatibility, X-ray photoelectron spectroscopy, calcium, phosphorus, bone formation

Introduction

Dental implants are used to replace missing teeth by anchoring prosthesis to the mandible or maxilla. Many different types of dental implants have been tried over the years [1]. To date, the most successful single-tooth implant is a screw-shaped device made from commercial pure titanium (cpTi). It is well known that the cpTi shows excellent biocompatibility and its major reason is attributed to the surface protective oxide [2].

Many researchers reported from the experiments *in vitro* that a calcium phosphate similar to hydroxyapatite, which is the main component of bone and teeth, is naturally formed on cpTi in a simulated body fluid involving a neutral Hank's solutions [3]. In addition, it is believed that this calcium phosphate is responsible for excellent biocompatibility of cpTi. Recently, however, Serro et al. have reported [4] that when the Hanks' balanced solution contained 0.05 mg/mL protein (far below the concentration in blood), the formation of a calcium phosphate layer was strongly inhibited. This fact suggested that the calcium phosphate formation may not occur in biological environment. Murakami et al. pointed out [5] that it was doubtful that calcium phosphate precipitation on the surface had any responsibility for bio-compatibility. Therefore, to elucidate the reason of the good biocompatibility of cpTi, the surface analysis of the sample extracted from the bone is very important.

The purpose of this study is to make clear the surface modification of the cpTi implant extracted from a rat bone by means of X-ray photoelectron spectroscopy.

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Materials and Methods

A part of cylindrical side of cpTi bullet, having 1.1 mm diameter and 3.5 mm length, was prepared to flat surface. The flat area was sandblasted by glassy particle, rinsed with acetone in ultrasonic cleaner for 5 min, and

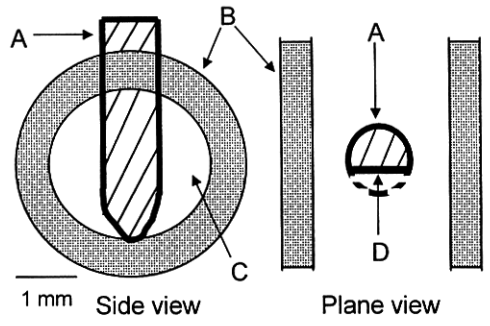

Figure 1. Schematic representation of implantation of the cpTi bullet to rat femur
A: cpTi bullet, B: Cortical bone, C: Bone marrow, D: Analytical surface

cleaned by radio-frequent argon sputtering. The bullet was inserted to the femur of young male Wistar rats (8 weeks old), as shown in Fig.1. After 3 hours or 7 days, the extracted samples were washed ultrasonically for 4 minutes in pure water (Distilled Water, Wako, 043-16785) and dried by gas blowing. XPS (Quantum 2000, ULVAC-PHI) were done using Al monochromatic X-rays. To check the XPS peak intensity of the calcium and phosphorus, standard compounds were used.

Results and Discussion

Wide scans of the sample implanted for 3 hours and 7 days were shown in Fig. 2 and partly enlarged spectra were shown in Fig.3. The main peaks are those of Ti, O, C, and N. Ti peaks arise from the base material and the others indicate presence of bio-molecules including proteins. Trace elements, such as P, Ca and Si, were also detected. There have been some reports that Si is an important element in the early step of bone formation [6, 7]. However, the exact role of Si is unknown yet.

Fig.4 shows partial spectra of two typical compounds that include equal ratio of calcium and phosphorus atoms. As for such compounds, the intensity of Ca2p peak is 3.3 ($Ca_2P_2O_7$) or 4.3 ($CaHPO_4 \cdot 2H_2O$) times larger than that of the P2p peak. Fig.5 shows the scatter diagram of the multi point quantitative analyses of the sample implanted for 7 days. From this diagram, the correlation factor was less than 0.3, which indicates weak relationship between calcium and phosphorus. In addition, calcium and phosphorus ratio is very small compared to that of the typical calcium phosphate compounds. These facts imply that even after 7 days from the implantation, few calcium phosphate compounds exist on the titanium oxide. From the experiment *in vitro*, Frauchiger et al.

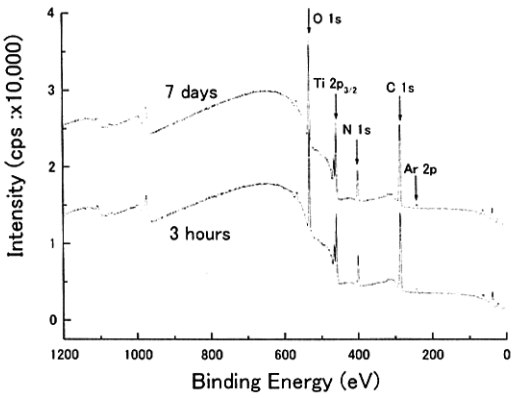

Figure 2. XPS wide spectra of the cpTi bullet implanted for 3 hours or 7days

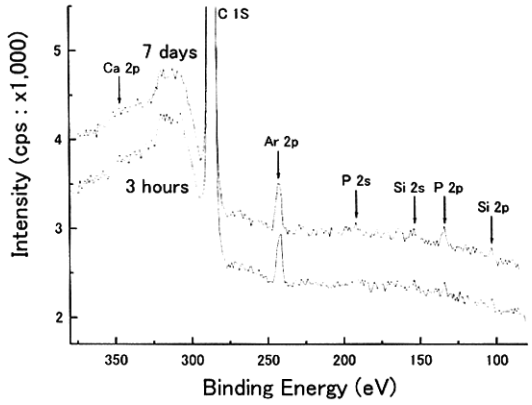

Figure 3. Enlarged spectra of figure 2

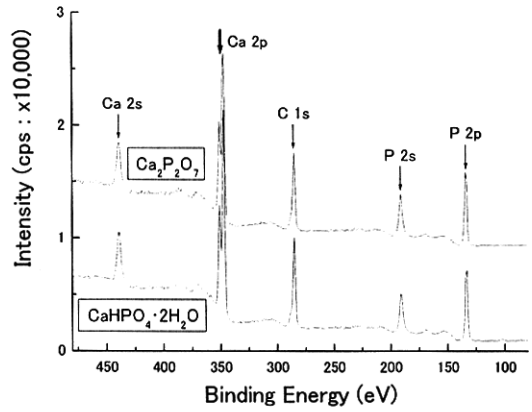

Figure 4. XPS spectra of standard compounds containing Ca and P

obtained the result that the Ca/P ratio increased with time and approached that of brushite after 71 days [8]. Longer implantation experiment seems to be required to clarify the mechanism of excellent biocompatibility of cpTi.

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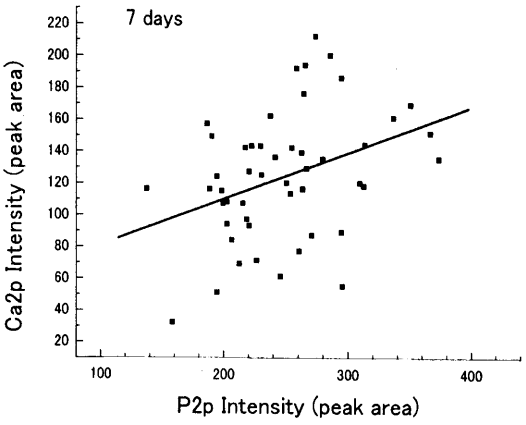

Fig.5 Scatter diagram of the multi point quantitative analyses of the bullet implanted for 7 days

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