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HYDROXYAPATITE GRANULE IMPLANTED Ti-ALLOY

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Abstract

To obtain a biomaterial that has both biological affinity and high mechanical strength, hydroxyapatite granules were implanted into the surface of pure titanium film coated titanium alloy. The film was coated by reactive DC sputtering method on the alloy substrate.

Hydroxyapatite granules (32-38 ㎛ in diameter) were spread over titanium alloy substrate and pressed to implant the granules in the substrate.

They can be implanted into substrate under 17MPa at 800℃ for 10minutes. The only tops of the granules were exposed and they were firmly stuck in substrate. The hydroxyapatite implanted titanium alloy composites were expected to be useful for biomaterials as artificial bones and dental roots.

Key words : *Hydroxyapatite, granule, Implant, superplastic, titanium alloy, sputtering*

1. INTRODUCTION

Having biological affinity, hydroxyapatite (hereafter named HAp) is suitable for biological hard tissues. However, because of its poor mechanical properties, particularly low fracture toughness, it cannot be used under a heavy load and its application is limited. If HAp can be coated onto a metallic material, such a composite may be a good biomaterial for its affinity for a living body and high mechanical strength[1].

As artificial hip joints and artificial roots need intensities, metals such as stainless and titanium are mainly used as their materials. However, metals do not have satisfactory affinities for living bodies. Recently, we have developed the method to implant HAp granules into the surface of superplastic Ti-4.5Al-3V-2Fe-2Mo-alloy[2, 3].

However, titanium alloy contains aluminium and vanadium, which are potentially harmful to human bodies[4, 5]. In a previous study[6], we developed a method of coating the alloy surface with pure titanium film by DC sputtering. In this study, as shown in fig. 1, HAp granules were implanted into the surface of titanium coated titanium alloy.

2. EXPERIMENTS

2. 1 Reactive DC suputtering

A planar magnetron sputtering system (ANELVA Corp. type SPE-210H) with a stainless steel chamber 200㎜ in diameter and 130㎜ in height was used. The planar target used for this study was a 99.99 mass% pure titanium disk 100 ㎜ in diameter.

Ti-4.5Al-3V-2Fe-2Mo-alloy substrate (NKK, SP-700, hereafter named Ti-A) of size 0.49㎜× 20㎜×20㎜ cleaned with organic solvent was mounted on a water-cooled substrate holder.

The argon gas was introduced into the chamber which was evacuated to 1×10^{-2} Pa or loss in advance. The pre-sputtering between the target and the shutter in the atmosphere of pure argon was carried out to clean the target surface. Titanium film was coated by reactive DC sputtering for 18min. Discharge voltage and current were at 400V and 1A, respectively. (hereafter named Ti-Ti-A)

2. 2 Implantation

HAp was granulated with the spray-dryer. And the granules were heated for 3hours at 1200℃, and were sifted 32-38㎛. The specimens were coated with grease and granules adhered to its surface. By using a hot press, HAp granules were implanted into the Ti-A substrates. Speicimens were set in the hot press, and pressed under a pressure of 17 MPa for 10 min at 750℃ or 800℃.

Diameter of granules (b) was measured using

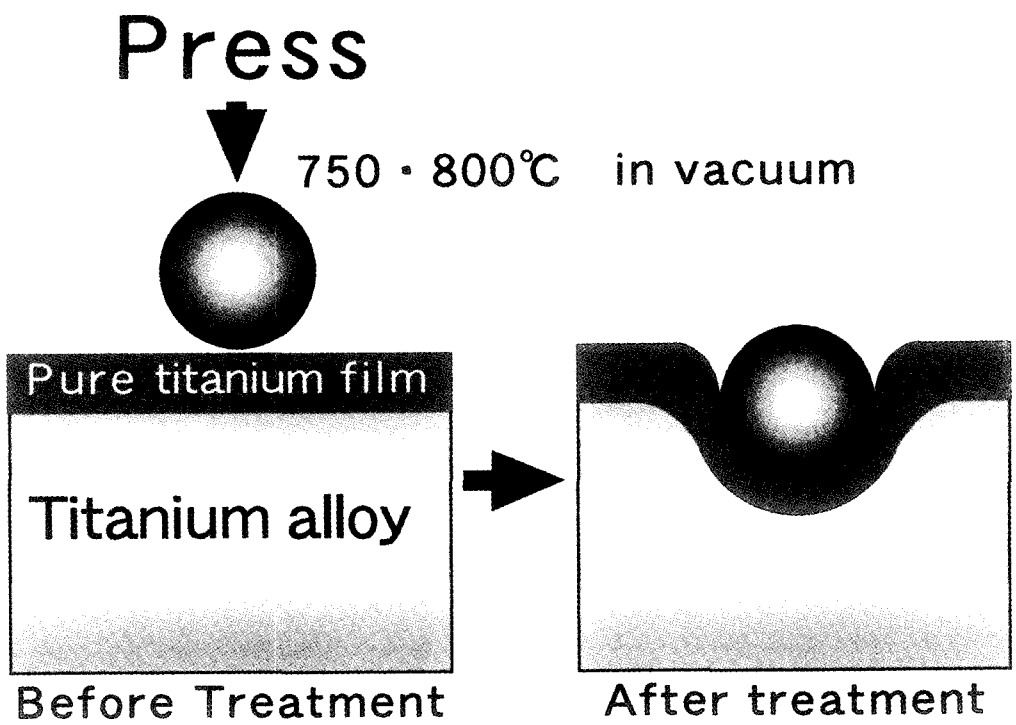

Fig. 1 Experimental apparatns for implantation of HAp granules into titanium film deposited titanium alloy.

an optical microscope. The height of projected part of granules (a) was measured using surface analyzer. The implantation ratio (%) of granules was calculated by the following Eq. (1). 30 granules were measured to get an average of data points.

$$\text{Implantation ratio}(\%) = (b-a)/b \times 100 \quad (1)$$

3. RESULTS AND DISCUSSIONS

3. 1 Appearance and surface Morphology of Ti-Ti-A

The film appeared to be uniform and adhesive. The surface of the Ti-Ti-A showed a metallic gloss, and observed to consist of uniformly accumulated submicron-sized particles. Upon more detailed observation of the topography of the accumulated deposit, pits and bumps on the order of several microns were found. These might reflect pits and bumps existing originally on the substrate surface. The film might be deposited on the alloy substrate with high topographic fidelity to the substrate surface. The thickness of the film was approximately 3㎛.

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3. 2 Implantation

Granules were not deformed at a uniform implantation of every specimen. The surface of specimen showed a metallic gloss, and a reaction wasn't observed with shape. Aluminium and iron were detected on the surface of HAp implanted Ti-A by EDS spectra. On the other hand, only titanium was detected on the surface of HAp implanted Ti-Ti-A

The implantation ratio of HAp granules on Ti-A surface was 100% at 750℃, but that of Ti-Ti-A was 35%, As the ductility of pure titanium is lower than that of titanium alloy. Therefore it is considered that those granules were prevented from being implanted. Considerable cracks were found around the implanted granules, the part where is hollowed by implanting the granules as shown in Fig. 2.

In side of the cracks, al and Ti were detected by the line analysis. Therefore, it is clear that titanium alloy is exposed on the surface. It is considered that the cracks are generated only on the titanium film. It is supposed that the film was deformed locally by implanting granules, and then it tore.

On the other hand, the implantation ratio of granules was 100% and no cracks were found in Ti-Ti-A which was implanted granules at 800℃. Fig. 3 presents SEM micrograph for the speci-

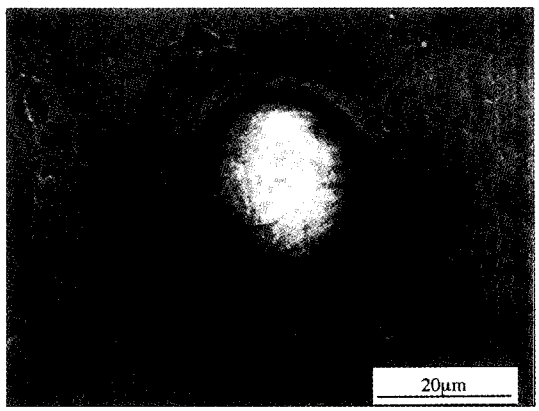

Fig. 2 SEM photographs of titanium deposited titanium-alloy(Ti-Ti-A). HAp granules were implanted at 750℃ for 10min with 17 MPa.

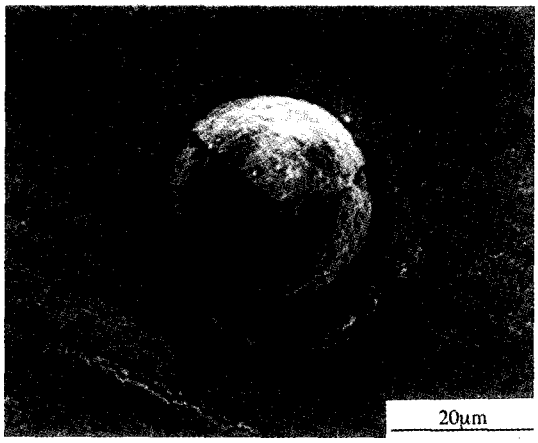

Fig. 3 SEM photographs of titanium deposited titanium-alloy(Ti-Ti-A). HAp granules were implanted at 800℃ for 10 min with 17 MPa.

men. Generally, pure titanium is deformed super-plastically under hihg temperature, which is more than 800℃. The ductility of pure titanium at 800℃ is higher than at 750℃, the film was expanded equally. As the mentioned above, HAp granules could be implanted suitably at 800℃ into the Ti-Ti-A.

4. CONCLUSIONS

Pure titanium film was coated on the surface of titanium alloy by reactive DC sputtering, and implantation of HAp granules was examined.

1) Deposited titanium film was approximately 3㎛ in thickness, and the film might be deposited on the alloy substrate with high topographic fidelity to the surface. The film appeared to be uniform and adhesive.

2) The surface of the alloy showed metallic gloss. Only titanium was detected by EDX, after HAp granules were implanted.

3) Considerable cracks were observed around the granules which had been implanted at 750℃. However, at 800℃, no cracks were found and the granules were implanted suitably.

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