

SURFACE STRUCTURE OF TiN/Ti MULTI-LAYERED THIN FILMS DEPOSITED ONTO Ti-6Al-4V ALLOY BY MAGNETRON SPUTTERING

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Ti-6Al-4V alloy attracting attention as a biomaterial features excellent mechanical properties and corrosion resistance, and super plasticity, so enables the forming of denture bases of complicated shapes. However, this alloy contains aluminum and vanadium liable to do serious harm to human bodies [1,2], so actual use will require prevention of direct contact with biological tissues. In our previous study, a method of coating the alloy surface with pure titanium film of excellent biocompatibility as a barrier layer was developed by DC sputtering in argon atmosphere, to improve the dental applicability of the alloy [3]. The pure titanium barrier layer prevents such harmful substances from leaching out to biological tissues. The alloy coated with titanium layer is, however, not suitable for the medical materials exposed to the blood stream such as artificial heart valves, because titanium is not only poor in the blood compatibility but also soft and easily damaged [4]. Hence, it is necessary to improve both the hardness and the blood compatibility of the barrier layer. Y.Mitamura, et al. reported the good blood compatibility of titanium nitride films [5]. Therefore, the deposition of TiN/Ti multi-layered thin films onto Ti-6Al-4V alloy substrates by magnetron sputtering in Ar gas atmosphere was examined, aiming at the application of pure titanium to the material for totally implantable artificial hearts, in order to improve not only the adhesion between the deposited film and the alloy but also the surface smoothness of titanium nitride coatings and thereby the blood compatibility of the alloy coated with the titanium nitride film. The effects of the thickness of the TiN(titanium nitride) layer on the surface structure of the multi-layered film were investigated, in order to develop TiN coatings with higher blood compatibility [6].

A planar magnetron sputtering system with a 200mm-diameter, 130mm-high stainless steel chamber was used. The planar targets used for this study were a 100mm-diameter pure titanium disk and a titanium nitride disk. Ti-6Al-4V alloy substrates ($14 \times 14 \text{ mm}^2$, thickness 0.55 mm) were mounted on the water-cooled substrate holder. The magnetron sputtering to deposit TiN/Ti multi-layered films was carried out in the atmosphere of argon (Ar). The sputtering conditions examined in this study were as follow. Discharge voltage and discharge current for DC sputtering the pure titanium target were 500V and 1.2A respectively. On the other hand, the electric power for RF sputtering of the titanium nitride target was 600W. A Ti layer and a Ti-N layer were deposited onto the same Ti-6Al-4V alloy substrate in sequence by sputtering pure titanium target and titanium nitride one respectively. Thus Ti-N/Ti multi-layered films were formed through the accumulation of these layers during the sputter-deposition. Depositing time for DC sputtering of the pure titanium target was 2min to adjust so that its corresponding layer might be 200nm thick, while that for RF sputtering of the titanium nitride target was 9min or 24min to adjust so that its corresponding layer might be 300nm thick or 800nm thick respectively, thereby the multi-layered film might be 500nm thick or 1000nm thick in total. For the comparison, we also performed the sputter-deposition of Ti-N monolithic films of 500nm thick onto the alloy substrate under the same sputtering conditions as for depositing the Ti-N layer of the multi-layer, except the depositing time. The thickness of deposited film was measured by tracing the substrate-film step using a surface roughness tester [7]. The surface morphology of the obtained films was studied on AFM

images both under the scope of nanometer-scaled area and under the scope of micrometer-scaled area. Then the surface roughness σ_{RMS} was also measured using AFM both under the area of micrometer scale and under the area of nanometer scale.

Under visual observation, the obtained TiN/Ti multi-layered films looked yellow gold and appeared to be uniform and adhesive, and each thickness of the films was approximately 500nm or 1000nm. On the other hand, Ti-N monolithic films deposited directly onto the alloy substrate under the same sputtering conditions peeled off partly. Therefore it was found that the multi-layered thin films were more adhesive to the alloy than the monolithic films. According to AFM images for both of the films, the surface morphology of each TiN layer observed under the scope of nanometer-scaled area, where the area of image was $505\text{nm} \times 505\text{nm}$, was rough with pits and bumps (Fig.1), while that under the scope of micrometer-scaled area, where the area of image was $20\mu\text{m} \times 20\mu\text{m}$, was smooth without such pits and bumps (Fig.2). Furthermore on the basis of AFM measurements, the surface roughness σ_{RMS} measured under the area of nanometer scale above-mentioned were found to be 3.06nm and 11.10nm for the TiN layers of 300nm thick and 800nm thick respectively, while the surface roughness σ_{RMS} measured under the area of micrometer scale above-mentioned were found to be 33.49nm and 34.39nm for the TiN layers of 300nm thick and 800nm thick respectively. Therefore it was found that the surface roughness σ_{RMS} measured under the area of nanometer scale was found to increase with the increase of the thickness of the TiN layer in the multi-layered film, although that measured under the area of micrometer scale was assumed to be independent of the thickness of the TiN layer. And it was guessed that the blood compatibility of the TiN/Ti multi-layered films might depend on the thickness of their TiN layer, because the surface roughness in scope of nanometer changed with the thickness of TiN layer deposited under thickness range of several hundreds nanometer scale.

References:

- [1] S. G. Steinemann and S. M. Perren, *Titanium Science Technology*, **2** (1985) 1327.
- [2] P. Galle, *Comptes rendus*, **299** (1984) 536.
- [3] M. Kato and T. Sonoda, *J. Iron and Steel Institute of Japan*, **77** (1991) 348.
- [4] Y. Mitamura, *J. Surface Finishing Society of Japan*, **43** (1992) 739.
- [5] Y. Mitamura, T. Yuhta and T. Mikami, *High Tech. Ceramics*, **1** (1987) 127.
- [6] J. Y. Park, C. H. Gemmell and J.E. Davies, *Biomaterials*, **22** (2001) 2671.
- [7] M. Numoto, *J. Surface Finishing Society of Japan*, **40** (1989) 241.

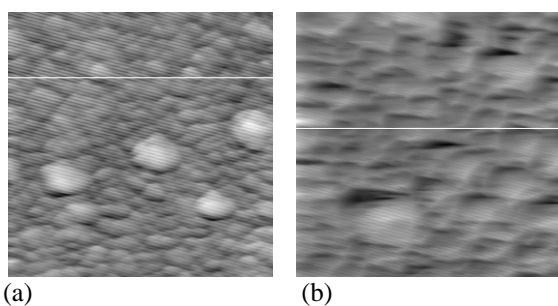


Fig.1. AFM images for the surface of TiN/Ti multi-layered films with the thickness of $0.3\mu\text{m}$ (a) and $0.8\mu\text{m}$ (b), where the area of each image is $505\text{nm} \times 505\text{nm}$.

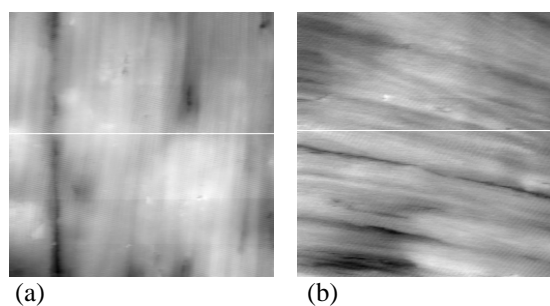


Fig.2. AFM images for the surface of TiN/Ti multi-layered films with the thickness of $0.3\mu\text{m}$ (a) and $0.8\mu\text{m}$ (b), where the area of each image is $20\mu\text{m} \times 20\mu\text{m}$.