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Surface modification of Ti-6Al-4V alloy with Ti-C/Ti-N/Ti multilayered film by magnetron DC sputtering(•)

T. Sonoda*, A. Watazu*, J. Zhu*, W. Shi*, T. Yamada*, K. Kato* and T. Asahina*

Abstract

Coating of Ti-6Al-4V alloy with Ti-C/Ti-N/Ti multi-layered films was examined by magnetron DC sputtering, aiming at the application of the alloy to artificial joints, in order to improve not only the biocompatibility and the abrasion resistance of the alloy but also the adhesion between the deposited film and the alloy, preserving the high hardness of such ceramic coatings. According to AES in-depth profiles, it was confirmed that a Ti-C/Ti-N/Ti multi-layered film had formed on the alloy. The multi-layered film appeared to be uniform and adhesive while Ti-C monolithic film deposited directly onto the alloy peeled off partly, concluding that the multi-layered film was more adhesive to the alloy than the monolithic film.

Keywords Multi-layered film. Adhesion. Titanium alloy. Abrasion resistance. Sputtering.

Modificación de la superficie de aleación de Ti-6Al-4V con multicapa delgada de Ti-C/Ti-N/Ti por pulverización catódica de corriente continua de magnetrón

Resumen

El recubrimiento de la aleación Ti-6Al-4V con multicapas delgadas de Ti-C/Ti-N/Ti se examinó por medio de pulverización catódica (*sputtering*) magnetrón DC, con el objetivo de mejorar no sólo la biocompatibilidad y la resistencia al desgaste de esta aleación sino, también, la adherencia entre la capa y la aleación depositadas, preservando asímismo la extgrema rigidez de esta clase de revestimientos cerámicos. Según los análisis de profundidad de AES, se confirmó la formación de una multicapa de Ti-C/Ti-N/Ti en la aleación. Al parecer la multicapa fue uniforme y adhesiva, mientras que la monocapa de Ti-C depositada directamente en la aleación se despegó parcialmente. Así se llegó a la conclusión de que la multicapa presenta una mayor adherencia a la aleación que la monocapa.

Palabras clave Multicapa delgada. Adhesión. Aleación de titanio. Resistencia al desgaste. Pulverización catódica.

1. INTRODUCTION

Ti-6AL-4V alloy attracting attention as a biomaterial features excellent mechanical properties and corrosion resistance. However, this alloy contains aluminium and vanadium liable to do serious harm to human bodies[1-3], so actual use will require prevention of direct contact with biological tissues[4-10]. On the other hand, the alloy has poor abrasion resistance to be applied to an artificial joint, so its application will require some surface modification of the alloy such as coating with hard ceramic films. However such hard ceramic coating might have poor adhesion to the alloy, so it is necessary to form some interlayer between the hard coating and the alloy to relax the stress concentrated at the interface. Therefore we performed coating of the alloy substrates with Ti-C/Ti-N/Ti multi-layered films by magnetron DC sputtering, aiming at the application of the alloy to artificial joints. Because it is expected that coating with the multi-layered films will improve not only

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(*) National Institute of Advanced Industrial Science and Technology (AIST) 2266-98 Anagahora, Shimoshidami, Moriyama-ku, Nagoya, 463-8560, Japan.

the biocompatibility and the abrasion resistance of the alloy but also the adhesion between the deposited film and the alloy substrate preserving the high hardness of such a ceramic coating as titanium carbide coating (Ti-C).

2. MATERIALS AND EXPERIMENTAL PROCEDURE

A planar magnetron sputtering system with a 200 mm diameter, 130 mm high stainless steel chamber was used. The planar targets used for this study were a 100 mm-diameter pure titanium disk, a titanium nitride disk and a titanium carbide disk. Ti-6Al-4V alloy substrates (14 × 14 mm^2, thickness 0.55 mm) were mounted on the water-cooled substrate holder. DC sputtering to deposit Ti-C/Ti-N/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 the sputtering were 400 V and 1 A respectively. A titanium (Ti) layer, a Ti-N layer and a Ti-C layer were deposited onto the same Ti-6Al-4V alloy substrate in sequence by sputtering pure titanium target, titanium nitride one and titanium carbide one respectively. Thus Ti-C/Ti-N/Ti multi-layered films were formed through the accumulation of these layers during the sputter-deposition. Depositing time for sputtering each target was adjusted so that its corresponding layer might be 1 μm thick, thereby the multi-layered film might be 3 μm thick in total. For the comparison, we also performed the sputter-deposition of Ti-C monolithic films of 3 μm thick onto the alloy substrate under the same sputtering conditions as for depositing the Ti-C layers of the multi-layers, except the depositing time.

The surface morphology of the obtained films was studied on SEM images. In-depth profiles of the chemical composition of the films were analysed by AES with an ion sputter etching method using argon ion beam. Characterization of the films were accomplished by X-ray diffractometry (XRD). Hardness of the films was measured using a Vickers hardness tester under 10 g load.

3. RESULTS AND DISCUSSION

Under visual observation, the obtained multi-layered film appeared to be uniform and adhesive and their thickness were approximately 3 μm. On the other hand, the Ti-C monolithic films deposited directly onto the alloy substrate peeled off partly. Therefore it was found that the multi-layered films were more adhesive to the alloy substrates than the monolithic film.

Under SEM, the surface of the multi-layered films was found to have fine particles in a uniform accumulated deposit.

AES in-depth profiles of Ti, Ti+N, C and Al are shown in figure 1, where the in-depth profile of Ti+N is substituted for that of nitrogen, because the principal Auger nitrogen transition peak (N-KLL) overlaps with one of titanium peaks (Ti-LMM). According to these in-depth profiles, it was confirmed that a Ti-C/Ti-N/Ti multi-layered film had formed on the alloy substrate.

Figure 2 shows the X-Ray diffraction pattern of the obtained multi-layered film. On the basis of XRD, it was found that titanium carbide, titanium nitride and α-titanium phase were formed in the multi-layered film, with TiC (111), TiN (111) and Ti (002) dominant respectively.

Vickers hardness of the multi-layered film deposited onto the alloy substrate was shown in figure 3, compared with those of the monolithic films of Ti, Ti-N and of Ti-C. The obtained Ti-C/Ti-N/Ti multi-layered film approximately indicated Hv = 1100, which was between the Vickers hardness of the Ti-C monolithic film and that of the Ti-N one. Thus the multi-layered film could be expected to feature good mechanical properties.

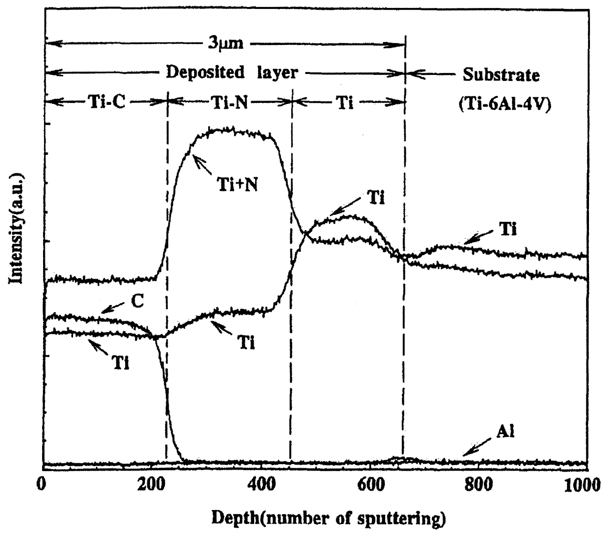

Figure 1. AES in-depth profiles of Ti, Ti+N, C and Al for the deposited multi-layered film and substrate.

Figura 1. Análisis de profundidad (suputtering) de AES a Ti, Ti+N, C y Al para la multicapa y capas inferiores depositadas.

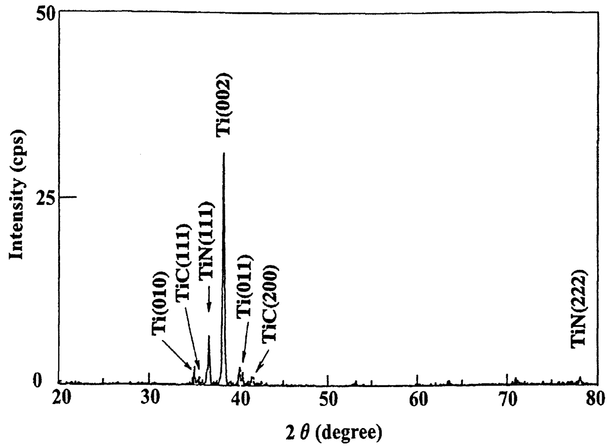

Figure 2. X-Ray diffraction pattern of the multi-layered film obtained in this study.

Figura 2. Patrón de difracción de rayos equis de la multicapa obtenido en este estudio.

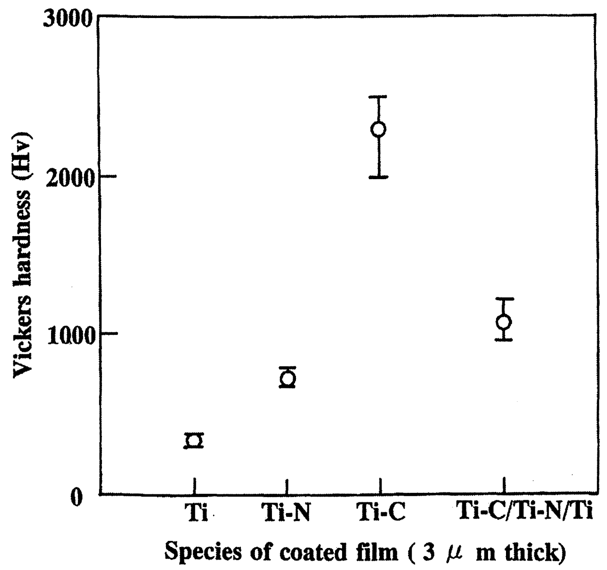

Figure 3. Vickers hardness of the Ti-C/Ti-N/Ti multi-layered film obtained in this study, and monolithic films of Ti, Ti-N and Ti-C of 3 μm thick deposited onto the same alloy substrates.

Figura 3. Dureza Vickers de la multicapa de Ti-C/Ti-N/Ti obtenida en este estudio, así como de las monocapas de Ti, Ti-N y Ti-C con 3 μm de espesor depositadas sobre las capas inferiores de la misma aleación.

4. CONCLUSIONS

Coating of Ti-6Al-4V alloy with Ti-C/Ti-N/Ti multi-layered films was examined by magnetron DC sputtering, aiming at the application of the alloy to artificial joints, in order to improve not only the biocompatibility and the abrasion resistance of the alloy but also the adhesion between the deposited film and the alloy with preserving the high hardness of such ceramic coatings.

Under visual observation, the obtained films appeared to be uniform and adhesive, while Ti-C monolithic films deposited directly onto the alloy substrate under the same sputtering conditions peeled off partly. Therefore it was found that the multi-layered films were more adhesive to the alloy substrates than the monolithic film.

Under SEM, the surface of the multi-layered films was found to have fine particles in an uniform accumulated deposit.

According to AES in-depth profiles, it was confirmed that a Ti-C/Ti-N/Ti multi-layered film had formed on the alloy.

On the basis of XRD, it was found that titanium carbide, titanium nitride and α-titanium phase was formed in the multi-layered film, with TiC(111), TiN(111) and Ti(002) dominant respectively.

Furthermore the good mechanical performances of the multi-layered film could be expected, based on the comparison with the mechanical properties of the monolithic films of Ti, Ti-N and Ti-C.

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