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MICROSTRUCTURE AND MECHANICAL PROPERTIES OF AMORPHOUS HYDROGENATED DLC-COATED Ti-6Al-4V ELI ALLOY WITH TiCN INTERLAYER PREPARED BY rf-PECVD

The low adherence of diamond-like carbon (DLC) films on titanium (Ti) alloys can be improved by using interlayer coatings. In this study, DLC (a-C:H) films were deposited using radio-frequency plasma-enhanced chemical vapor deposition (rf-PECVD), and a TiCN interlayer was applied between the extra low interstitial (ELI) grade of Ti-6Al-4V alloy and a-C:H film. The characteristics of the a-C:H-coated Ti-6Al-4V ELI alloy were investigated using field emission scanning electron microscopy, Vickers hardness, and scratch and wear tests. The DLC (a-C:H) films deposited by rf-PECVD had a thickness of 1.7 µm, and the TiCN interlayer had a thickness of 1.1 µm. Vickers hardness of the DLC (a-C:H) films were increased as a result of the influence of the TiCN interlayer. The resulting friction coefficient of the a-C:H-coated Ti-6Al-4V with the TiCN interlayer had an extremely low value of 0.07.

Keywords: DLC Coating, rf-PECVD, Ti-6Al-4V ELI Alloy, TiCN

1. Introduction

Titanium (Ti) alloys have excellent biocompatibility with high specific strength due to their thin and stable oxide films that have thicknesses of 3-5 nm on their surfaces [1]. The extended use of orthopedic implants in heavily stressed parts can cause the problem of abrasion. Surface modifications have been widely studied for Ti-based alloy implants to improve their wear characteristics [2,3].

Diamond-like carbon (DLC) films used for hard coatings have high hardness and good wear properties. However, thick DLC films easily delaminate from the substrate because of the poor bonding force of the DLC film [4,5]. Nitride or carbide as an interlayer has been applied to improve the adhesive force between the DLC film and substrate [6,7]. Nevertheless, one study to date has presented a case in which a TiCN was used as the interlayer for DLC (ta-C) films [8].

In this study, amorphous hydrogenated DLC (a-C:H) films were deposited using radio-frequency plasma enhanced chemical vapor deposition (rf-PECVD), and a TiCN interlayer was applied between the extra low interstitial (ELI) grade of Ti-6Al-4V alloy and DLC (a-C:H) film. The microstructure and mechanical properties of the DLC (a-C:H) coated Ti-6Al-4V ELI were investigated.

2. Experimental

In this study, Ti-6Al-4V ELI (ASTM Grade 23) specimens had a diameter of 15 mm and a thickness of 3 mm. TiCN interlayer films were deposited using a Nachi ion-plating system with a Ti purity of greater than 99.9%. The initial pressure of the chamber was less than 7.5×10^{-4} Pa, and the TiCN interlayer was deposited onto the substrate at a bias voltage of 300 V and a target power of 50 W in a mixture of gases of C_2H_2 and N_2 for 80 min. The a-C:H films were deposited using an rf-PECVD system with a working pressure of 1.5×10^{-4} Pa and a power of 60 W. The DLC (a-C:H) films were deposited onto the substrate in a CH_4 atmosphere at 14.25 standard cubic centimeters per minute for 60 min.

The thickness of the film was observed using a field emission scanning electron microscope (FE-SEM, S-4700, Hitachi, Japan). Vickers hardness was measured using a DUH-W201 micro-Vickers hardness tester (DUH-W201, Shimadzu Co., Japan) with a 100-g load for 10 s. Five random measurements were made on the specimen surfaces. The bonding force between the Ti-6Al-4V ELI and DLC (a-C:H) film with a TiCN interlayer were measured using a scratch tester (REVETEST, CSM Instruments, Switzerland). Wear properties of the DLC (a-C:H)-coated specimens were measured by a wear tester (RB

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102 PD, R&B, Korea) using a pin-on-disk method with a load of 20 N and a rotation speed of 150 rpm for 1800 s.

3. Results and discussion

Fig. 1 shows cross-sectional FE-SEM images of the DLC (a-C:H) films and the TiCN/DLC coated on the Ti-6Al-4V ELI alloy. As shown in Fig. 1(a), the DLC (a-C:H) films deposited using the rf-PECVD for 60 min had a thickness of approximately 1.7 μm. The sequence for the Ti-6Al-4V ELI/TiCN/DLC (a-C:H) coating layer is also shown in Fig. 1(b). The TiCN interlayer had a thickness of 1.15 μm. No defects such as pin holes or cracks in the films were observed, thus ensuring clean interfacial boundaries between the DLC (a-C:H) film and TiCN interlayer or between the TiCN interlayer and Ti-6Al-4V ELI.

Fig. 2 shows the micro-Vickers hardness of a substrate as well as a substrate + TiCN, substrate + DLC (a-C:H), and substrate + TiCN + DLC (a-C:H). The average values of Vickers hardness for Ti-6Al-4V ELI/TiCN and Ti-6Al-4V ELI/DLC (a-C:H) were 56 and 42.4 H_v respectively. The hardness of the TiCN interlayer was higher than that of the DLC (a-C:H) film, which was greater than 30%. The hardness of the Ti-6Al-4V ELI/TiCN/DLC (a-C:H) film was approximately 67 H_v, which represents an increase in hardness of 67% as compared to the non-coated Ti-6Al-4V ELI alloy. The presence of the TiCN interlayer effectively increased the hardness of DLC (a-C:H) films, and this could be attributed to the intrinsically high hardness of TiCN. In addition, the TiCN interlayer could affect the wear properties of the DLC layer because of a fully thick top-coated layer.

In general, the scratch test was used to evaluate the adhesion strength between a coated layer and a substrate. Fig. 3 shows scratch test curves of the DLC (a-C:H) films deposited using the rf-PECVD with optical scratch tracks. Fig. 3(a) shows that the first appearance of a crack on a layer of TiCN/Ti-6Al-4V ELI occurred at a load of 12.2 N and a displacement of 0.95 mm. Significant spallation or delamination was observed at a load of 21.8 N with a displacement of 2 mm. However, as Figs. 3(b) and (c) show, the first appearance of cracks on the films of DLC(a-C:H)/Ti-6Al-4V ELI and DLC(a-C:H)/ TiCN /Ti-6Al-4V ELI were not observed up to the critical loads for delamination, which were 28.1 N at 2.57 mm and 32.5 N at 3.25 mm, respectively. The critical load necessary for full delamination of the TiCN interlayered DLC (a-C:H) film was higher than that of the DLC (a-C:H) film without the TiCN interlayer. Cicek also reported that TiN/TiCN layers with the DLC coatings increased the load carrying capacity on Inconel substrates [9]. Thus, the TiCN interlayer played a major role in the development of adhesive DLC (a-C:H) films on a Ti-6Al-4V ELI with higher hardness.

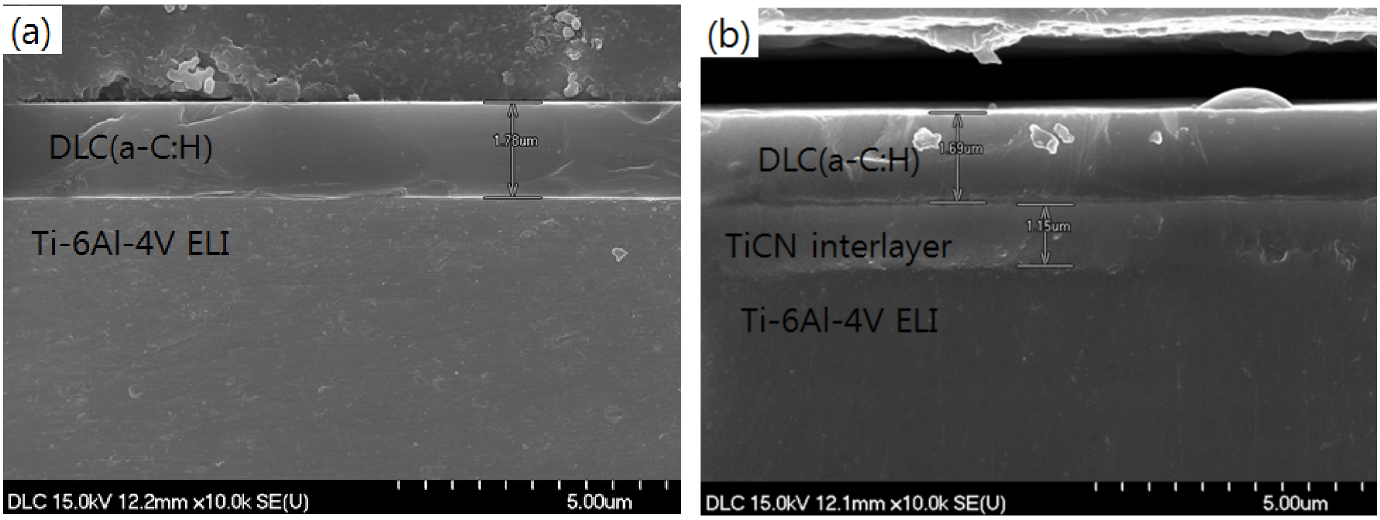


Fig. 1. Cross-sectional FE-SEM images of the DLC films deposited using an rf-PECVD: (a) Ti-6Al-4V ELI/DLC(a-C:H) and (b) Ti-6Al-4V ELI/TiCN/DLC(a-C:H)

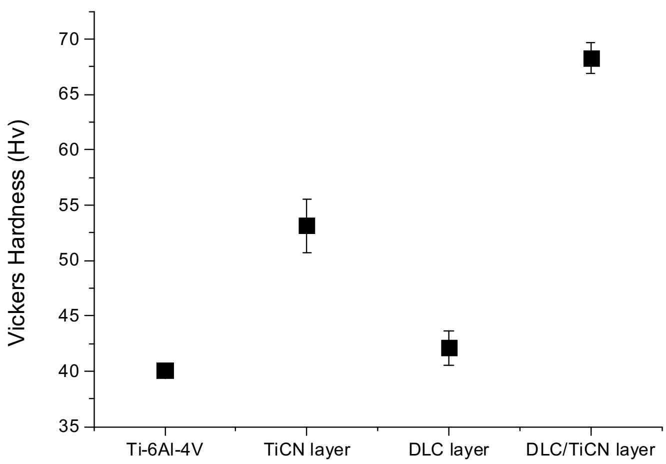


Fig. 2. Vickers hardness values of the Ti-6Al-4V ELI, Ti-6Al-4V ELI/TiCN, Ti-6Al-4V ELI/DLC (a-C:H), and Ti-6Al-4V ELI/TiCN/DLC (a-C:H) measured using a micro-Vickers hardness tester

Fig. 4 shows the results of the wear test using the pin-on-disk method for the DLC (a-C:H) films with and without the TiCN interlayer. A friction coefficient of the non-coated Ti-6Al-4V ELI alloy was 0.35, whereas that of the DLC

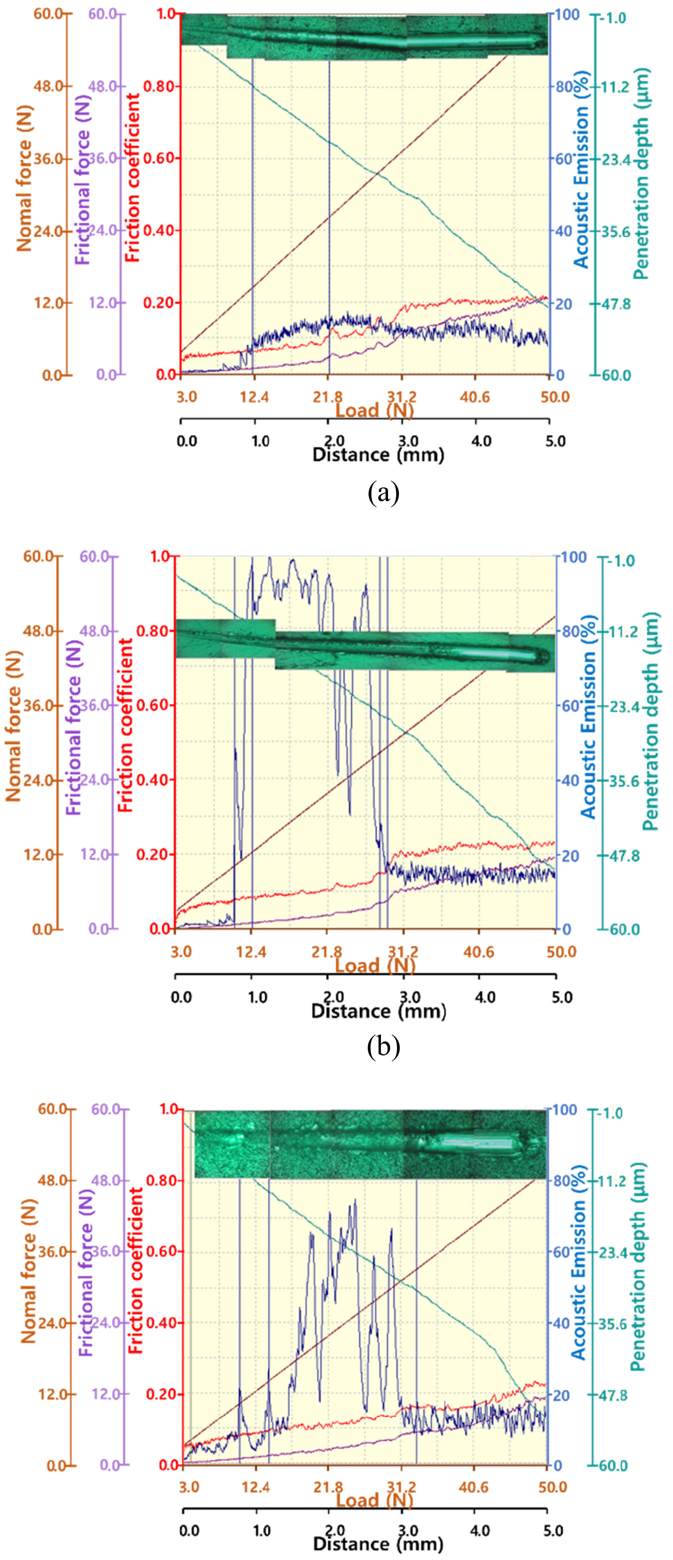


Fig. 3. Scratch test curves of the DLC (a-C:H) films deposited using an rf-PECVD with optical scratch tracks: (a) Ti-6Al-4V ELI/TiCN, (b) Ti-6Al-4V ELI/DLC(a-C:H) and (c) Ti-6Al-4V ELI/TiCN/DLC(a-C:H)

(a-C:H)-coatedTi-6Al-4V ELI alloy was 0.12. The wear behaviors at 4500 cycles showed regular wear patterns as compared to the initial conditions of the test. The friction coefficient of the DLC (a-C:H)-coated Ti-6Al-4V ELI alloy with the TiCN interlayer was obtained with an extremely low value of 0.07, which represents an excellent lubrication property for the application of heavily stressed implant parts. The low friction coefficient of the specimen derived from the strong adhesion between the DLC (a-C:H) film and the TiCN interlayer.

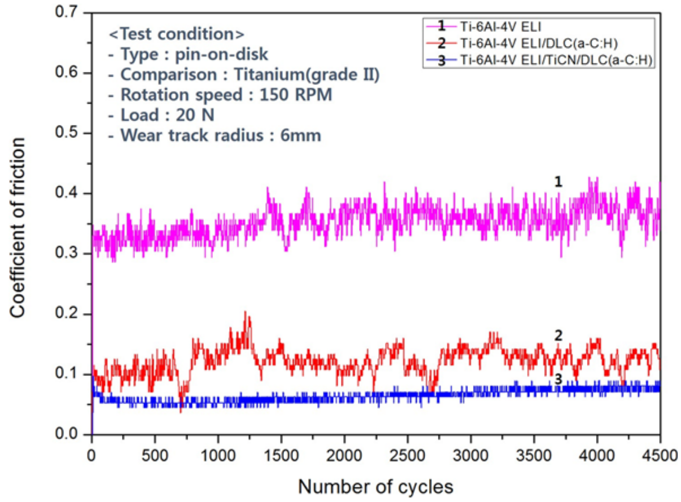


Fig. 4. Friction coefficients of the Ti-6Al-4V ELI, Ti-6Al-4V ELI/DLC(a-C:H), and Ti-6Al-4V ELI/TiCN/DLC(a-C:H) specimens measured using the pin-on-disk method

Fig. 5. shows wear track surface images of Ti-6Al-4V ELI/DLC (a-C:H) and Ti-6Al-4V ELI/TiCN/DLC (a-C:H) specimens after the pin-on-disk test. The DLC (a-C:H)-coated specimens with a TiCN interlayer appeared to be less worn after the wear test, which was consistent with the friction coefficient of specimens. As a result, the TiCN interlayer played a major role in improving the wear property of the DLC (a-C:H) film.

4. Conclusion

In this study, DLC (a-C:H) films were deposited using a rf-PECVD, and a TiCN interlayer was applied between the Ti-6Al-4V ELI and a-C:H film. The following results were derived from this study.

1. DLC (a-C:H) thin films were successfully coated on Ti-6Al-4V ELI with a TiCN interlayer using rf-PECVD. The DLC (a-C:H) films and TiCN interlayer had a thickness of 1.7 µm and 1.1 µm, respectively. An interface boundary between the TiCN interlayer and DLC (a-C:H) film was clearly observed with no cracks and pin holes.
2. The presence of the TiCN interlayer significantly contributed to increasing the hardness of DLC (a-C:H) films because of the intrinsically high hardness of TiCN.
3. A scratch test revealed that the TiCN interlayer played a major role in improving adhesion of DLC (a-C:H) films on Ti-6Al-4V ELI.
4. The resulting friction coefficient of the DLC (a-C:H)-coated Ti-6Al-4V ELI alloy with the TiCN interlayer had an extremely low value of 0.07.

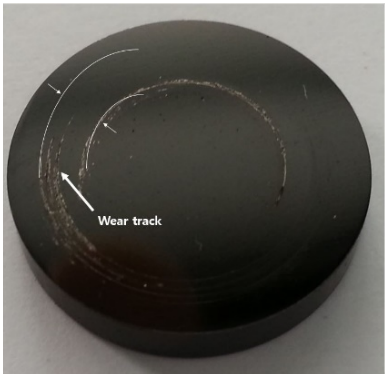
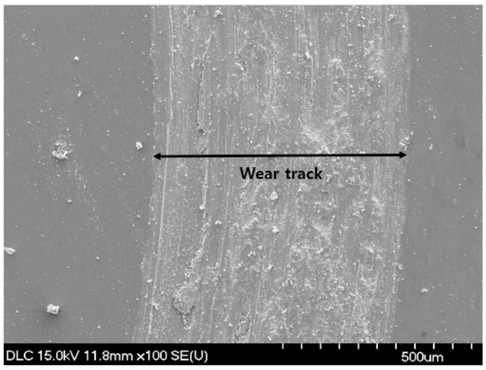
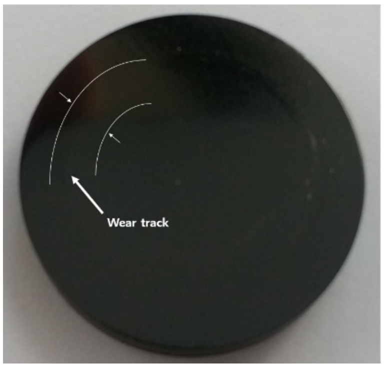
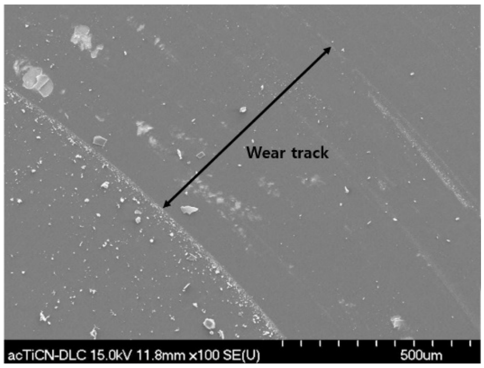
	Surface of specimen	SEM micrograph
Ti-6Al-4V ELI/DLC (a-C:H)		
Ti-6Al-4V ELI/TiCN/ DLC(a-C:H)		

Fig. 5. Wear track surface images of Ti-6Al-4V ELI/DLC(a-C:H) and Ti-6Al-4V ELI /TiCN/DLC(a-C:H) specimens after the pin-on-disk test

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