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타이타늄 합금에 코팅처리된 TiN,ZrN 및 WC피막의 기계적 성질에 관한 연구

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A study on mechanical properties of TiN,ZrN and WC coated film on the titanium alloy surface

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오동준

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임플란트 보철물을 고정체나 상부구조에 연결하는 방법에는 시멘트에 의해 유지를 얻는 방법과 나사에 의해 유지를 얻는 방법이 있다. 유지방법에 따른 각각의 장점이 있으나, 임상에서는 시멘트 유지방식보다 나사 유지방식이 더 선호되며, 그 이유는 철거가능성 때문이다. 그러나 나사 유지방식은 나사 풀림등의 문제점을 보인다.

나사의 풀림을 최소화하기 위한 시도로 지대주 나사에 순금이나 테프론을 표면처리한 제품을 사용하고 있으나, 반복적인 나사의 조임과 풀림과정에서 이들 코팅재료들의 마모와 손상등이 관찰되기도 한다.

따라서 마찰저항을 최소화하고, 동시에 표면의 안정성과 강도를 부여하기 위한 표면처리법의 연구가 필요하다. 최근 TiN, ZrN 및 WC의 코팅이 연구되고 있으며, 이들을 금속표면에 코팅시 마찰계수의 감소와 부식에 대한 저항, 물리적 취약함의 해소 등이 보고되고 있다.

따라서 본 연구의 목적은 PVD(Physical vapor deposition)를 이용해 TiN, ZrN 및 WC를 타이타늄 합금시편에 코팅 처리한 후 기계적 성질을 평가 코저하였다.

16개의 지름 12mm, 두께 1mm인 타이타늄 합금시편이 본 실험에서 사용되었으며 4개의 그룹으로 나뉘었다. 3개의 그룹은 PVD법을 이용해 TiN, ZrN 및 WC를 타이타늄 합금시편에 코팅한 실험군이며, 1개의 그룹은 코팅을 하지않은 대조군으로서, 마찰계수, 비커스경도, 코팅의 접합강도 및 코팅표면을 평가하여 다음과 같은 결과를 얻었다.

1. 3가지 코팅시편 모두 마찰계수의 유의성있는 감소를 보였으며, ZrN을 코팅한 시편에서 가장 낮은 마찰계수가 측정되었다.

2. 3가지 코팅시편 모두 비커스경도의 유의성있는 증가를 보였으며, 특히 TiN과 ZrN을 코팅한 시편에서 높은 비커스 경도를 보였다.

3. 코팅피막의 접합강도는 TiN (25.3N±1.6), ZrN (14.8N±0.6), WC (18.4N±0.7)으로 TiN을 코팅한 시편에서 가장 높은 수치를 보였으며, ZrN을 코팅한 시편에서 가장 낮았다.

4. SEM을 이용해 코팅 표면을 관찰한 결과, ZrN과 WC가 코팅된 시편은 매우 균일하고 매끈한 표면을 보였으나, TiN이 코팅된 시편의 경우는 거친 표면이 관찰되었다.

결론적으로 TiN, ZrN 및 WC를 지대주 나사에 코팅시 마찰저항의 감소로 인한 전하중의 증가와 침하효과의 감소로 지대주 나사의 풀림에 대한 저항이 증가될것으로 사료된다. 그러나 마찰저항의 감소는 또한 풀림 토크를 감소시켜 나사 풀림을 유발하는 양면성을 지니기 때문에 이에 대한 연구가 더 필요할 것으로 사료된다.

I. Introduction

Recently, dentists are administering prosthetic treatments using dental implants more than ever before on many occasions and indeed, implant prosthesis can restore stomatognathic function close to a normal level.

Implant prostheses can be attached to their superstructure or fixtures in two different ways: cement retained type and screw retained type. Although each type has its own merit comparing with the other, clinicians has preferred to use the screw retained type because of the retrievability. Although the screw retained type deserves its credit for the structure can be removed more easily, it may also cause problems such as screw loosening.

Jemt[1,2]reported that mobile prostheses caused by the loosened gold screws was the most common problem after the final tightening.[1,2] Worthington et al.[3] also reported that abutment screw had more problems such as loosening and fracture than any other components. Minimizing the screw loosening is essential for long term successful rates. Therefore, it is essential for clinicians to understand the mechanics and physical properties of the screw.

Forces attempting to disengage the parts are called "joint separating forces". The force keeping the parts together can be called the "clamping force". Screw loosening occurs when the joint separating forces acting on the screw joint are greater than the clamping forces holding the screw unit together.[4]

To achieve secure assemblies, screw should be tensioned to produce greater clamping force than separating force. Clamping load is usually proportionate to the tightening torque. Applied torque developing force within a screw called "preload". Preload is the initial load in tension on the screw.[4] This tensile force on the screw develops a compressive clamping force between the parts. Increasing the torque will increase the preload. Increasing preload will maximize the stability of the screw joint by increasing clamping threshold that separating forces must overcome to cause screw loosening.[4,5]

But the torque applied to the screw joint during tightening is not entirely translated into screw preload since part of this torque is expended to overcome friction. About 50% of the energy transmitted by torque tightening is expended to overcome the friction between the abutment screw head and the abutment-seating surface. About 40% of the applied torque is used to overcome thread friction and only 10% produces the screw tension.[6-8] Thus we have to maximize preload by minimizing the loss of torque for the stability of screw joint assemblies.

In an attempt to reduce frictional resistance, dry lubricant coating has been applied to the abutment screw. The notable products were GoldTite(Implant Innovation, 3i) coated with 0.76㎛ thickness of pure gold and TorqTite(Nobel Biocare, Steri-oss) coated with teflon.[9]

Martin et al.[10] reported that GoldTite and TorqTite abutment screw with enhanced surfaces produced greater rotational angles and preload values than the conventional gold alloy and titanium alloy screws. However, under repeated tightening and loosening procedures, low wear resistance and adhesion strength of coating material produced free particles on the surface of abutment screw and increased frictional resistance resulting in screw tightening problems. Therefore, further studies for surface treatment of abutment screw would be need to minimize frictional resistance as well as increase surface stability and strength.

TiN (titanium nitride), ZrN (zirconium nitride) and WC(tungsten carbide) coated surface has been considered as the solution of the surface treatment problems. Some studies have reported that TiN, ZrN, WC coatings could decrease the coefficient of friction and improve resistance of corrosion and physical properties.[11,12]

The aim of this study was to compare friction coefficient, adhesion strength, vickers hardness and evaluate coating surface of titanium alloy specimens coated with TiN, ZrN and WC.

II. Materials and methods

1. Materials

Titanium alloy(Ti-6Al-4V) discs of 12mm in diameter and 1mm in thickness were prepared for TiN, ZrN and WC coating and divided into 4 groups in this study. TiN, ZrN and WC was coated for the specimens of 3 groups respectively, and those of 1 group were not coated. Each group was made up of 4 specimens(Fig. 1, Table 1).

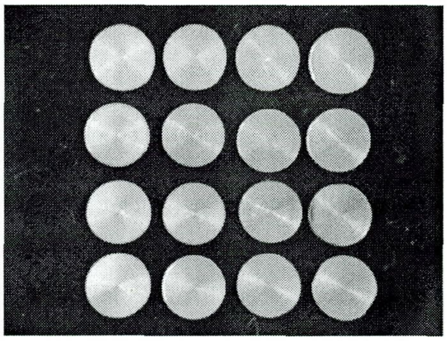


Fig. 1. Titanium alloy discs used in this study

Table 1. Kinds of coated materials used in this study

Group of specimen	Coated materials	Diameter * Length of specimen
A	TiN	∅12 * 1mm
B	ZrN	∅12 * 1mm
C	WC	∅12 * 1mm
D	None	∅12 * 1mm

2. Methods

1) Mounting of titanium alloy disc

Each specimen was mounted in liquid unsaturated polyester as shown in fig. 2. The mouting media(Epovia, CrayValley Inc. Korea) was a 2-part system made up of a resin and hardener. Two components were mixed together and poured to be cured overnight.

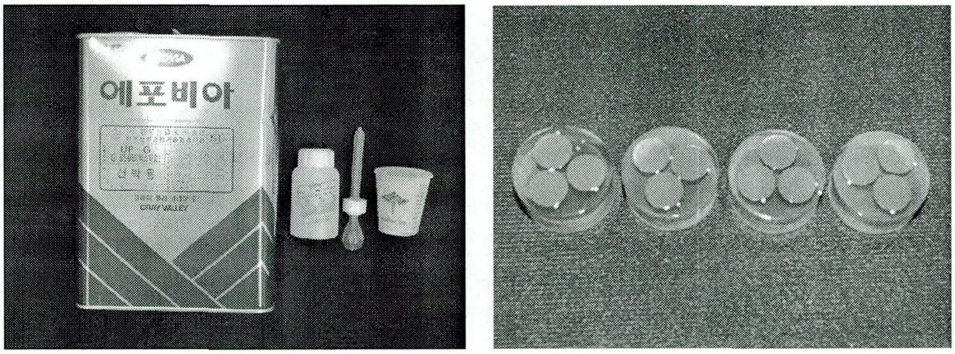


Fig. 2. Mounting media(Epovia, CrayValley Inc. Korea) and specimens which were mounted

2) Polishing and ultrasonic cleansing of mounted specimens

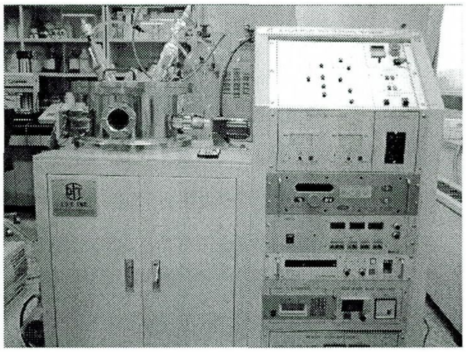
All specimens were polished with automatic polishing unit (Mecapol P 260, Presi, France) in which 200, 600, 1000 and 1200 grit silicon carbide paper were used in order. Micropolishing was finally done with Al_2O_3(0.3 ㎛)(Fig. 3). The polished specimens were cleansed in the solution of alcohol and aceton by ultrasonic cleaner (Branson 3510, Bransonic, USA) for 10 minutes(Fig. 4).

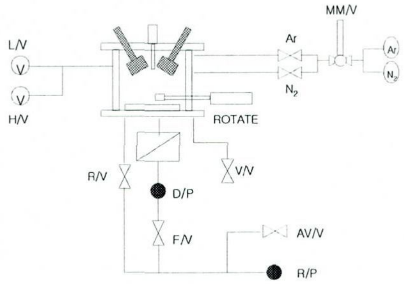
Fig. 3. Polishing unit (Mecapol P 260, Presi, France)

Fig. 4. Ultrasonic cleaner (Branson 3510, Bransonic, USA)

3) TiN, ZrN and WC film coating using RF sputtering

TiN, ZrN and WC was coated by magnetron sputtering equipment. Table 2 shows coating conditions of TiN, ZrN and WC.





<Magnetron sputtering equipment>

Fig. 5. Magnetron sputtering equipment and flow chart

Table 2. Coating conditions of TiN, ZrN and WC

	TiN	ZrN	WC
Target	Pure Ti	Pure Zr	Pure W
Base pressure	$3.0X10^{-5}$Torr	$3.0X10^{-5}$Torr	$3.0X10^{-5}$Torr
Working pressure	$2.0X10^{-2}$Torr	$2.0X10^{-2}$Torr	$2.0X10^{-2}$Torr
Gas	N_2(35sccm) Ar(5sccm)	N_2(35sccm) Ar(5sccm)	C_2H_2(35sccm) Ar(5sccm)
Operation Temperature	150°C	150°C	150°C
Pre-sputtering	20min	20min	20min
Deposition Time	15min	15min	15min
Power supply	100W	100W	100W

4) Measurement of friction coefficient for coated specimens

Mobile friction meter(SF-2, FACE, Japan) were used to measure friction coefficient of the coated specimens(Fig. 6). Each specimens were measured

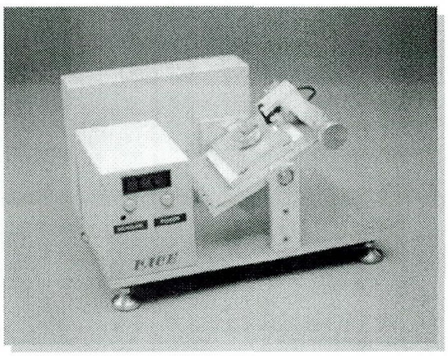


Fig. 6. Mobile friction meter

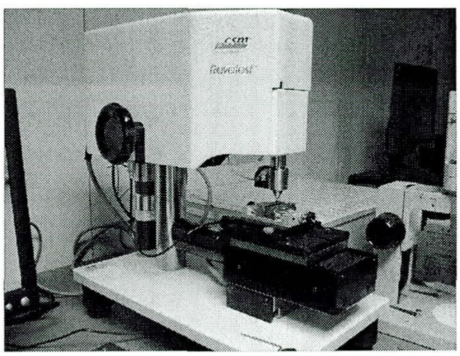


Fig. 7. Scratch tester

10 times.

5) Measurement of adhesion strength of coated specimen

Adhesion strength were measured with scratch tester(Revetest, CSM instrument, Switzerland), in which adhesion strength is evaluated by the analysis of friction coefficient and acoustic emission(Fig. 7). One specimen in each group were randomly selected, and adhesion strength was measured 5 times for each specimen.

6) Vickers hardness test of each specimen

One specimen in each group were randomly selected, and vickers hardness was measured 5 times for each specimen(Fig. 8).

7) Surface roughness investigation using FE-SEM instruments

The coated surfaces of specimens were observed by FE-SEM(Field Emission Scanning Electron Microscopy, XL 30 SFEG, Phillips Co., Netherlands) with the magnification of 1000 times(Fig. 9).

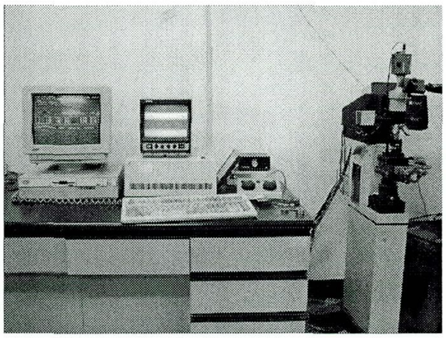


Fig. 8. Vickers hardness tester (MXT70, Matszawa Seiki Co. Japan)

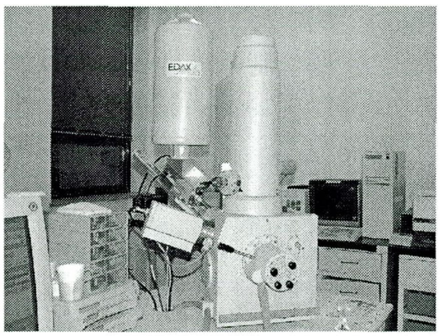


Fig. 9. FE-SEM instruments (XL 30 SFEG by Phillips Co.)

8) Statistical analysis

SPSS version 10.0.7 program has been utilized for statistics. Statistical significances were tested by oneway ANOVA(analysis of variances) among groups and significant difference between groups was tested by Tukey's multiple comparison test.

III. Results

1. Friction coefficient

The friction coefficient for each coating condition are shown in Table 3. As shown in Table 3, all coating conditions showed the decrease of the friction coefficient with the statistically considerable difference as compared with that of the control group. Especially, ZrN coating showed the lowest coefficient of friction.

Table 3. Friction coefficient for each coating condition

Coated materials	Mean	S.D.	P-value
TiN	0.39	0.02	<0.05
ZrN	0.24	0.01	
WC	0.31	0.03	
None	0.48	0.01	

2. Vickers hardness

Vickers hardness of each group are shown in Table 4. Vickers hardness of coated groups was increased with the statistically considerable difference as compared with that of the control group. As shown in Table 4, TiN coating shows the highest vickers hardness.

Table 4. Vickers hardness of each group

Coated materials	Mean (Hv)	S.D.	P-value
TiN	1865.2	33.8	<0.05
ZrN	1814.4	18.6	
WC	1008.5	35.9	
None	313.9	18.9	

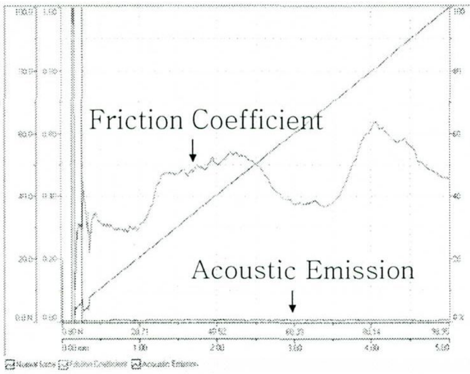
3. Adhesion strength

Adhesion strength for each coating condition are shown in Table 5. As shown in Table 5, TiN coating shows the highest adhesion strength, however ZrN coating has the lowest adhesion strength.

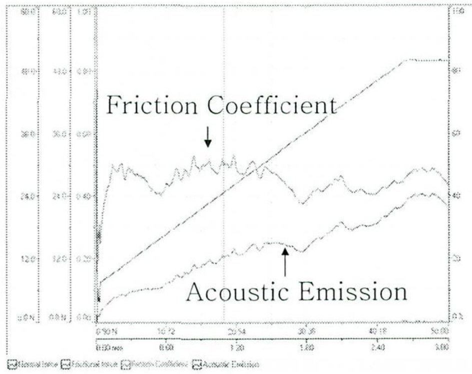
Fig. 10 showed the relationship between the friction coefficient and acoustic emission with the progressive increase of load to the TiN, ZrN or WC coated specimens. Adhesion strength was evaluated from the analysis of the graph in Fig. 10.

Table 5. Adhesion strength of each group

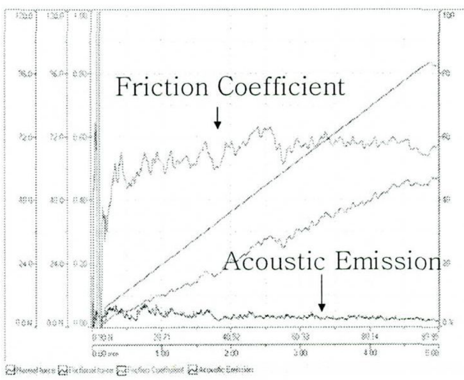
Coated materials	Mean (N)	S.D.	P-value
TiN	25.3	1.6	<0.05
ZrN	14.8	0.6	
WC	18.4	0.7	



a



b

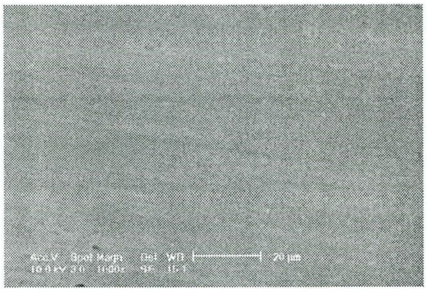


c

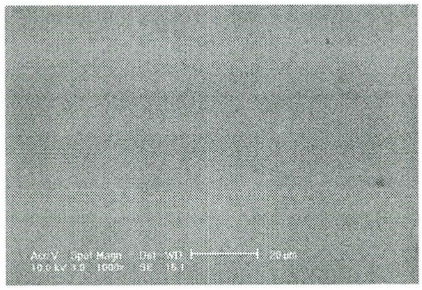
Fig. 10. Relationship between the friction coefficient and acoustic emission with the progressive increase of load (a : TiN, b : ZrN, c : WC)

4. Coating surface investigation

The coating surface was observed and evaluated by FE-SEM. The SEM micrographs of TiN, ZrN or WC coating surface are shown in Fig. 11. The ZrN or WC coated specimen show a homogeneous and smooth surface, however the rough surface was observed for TiN coating.



a



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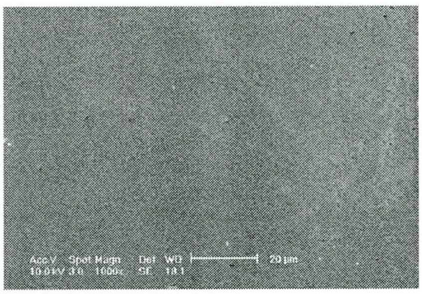

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Fig. 11. SEM investigation of TiN, ZrN and WC coated film
(a : TiN, b : ZrN, c : WC)

IV. Discussion

Previous studies have been reported that the most common problem associated with dental implant system is loosening of the screw that holds the prosthesis and implant together.[1,2,3)]

To solve the screw loosening problems, 3i (Implant Innovations Inc) altered the palladium-gold abutment screw surface itself, adding a solid lubricant to decrease the friction coefficient and increase preload values. Steri-Oss (Nobel Biocare) also altered the surface of its titanium abutment screw through a treatment process to decrease friction coefficient and increase fatigue strength.[9,13)]

Under repeated tightening and loosening procedures, however, free particles on the surface of abutment screw were produced because of the low wear resistance and adhesion strength of coating material. The particles increased frictional resistance and lead to the problems in tightening the screw.[10)]

From those reasons, improvement of coating material has became a prominent figure to get better mechanical properties.

Thin film coatings are achieved by using CVD or PVD. Thin hard coatings deposited by chemical vapor deposition (CVD) or physical vapor deposition (PVD) are used to improve metallic surface properties for industrial purposes. Its high hardness, low coefficient of friction and resistance to adhesive wear are favourable, for example, for drills and cutting tools.[12,14-16)]

CVD deposition takes place at a temperature of about 1000°C, and thus its range of applications is limited. Much lower deposition temperature can be achieved with PVD processes between 150°C and 500°C.[14)]

Valvoda et al.[17)] mentioned that such a high stress was unlikely able to originate from the different thermal expansion coefficients of the coatings and the substrate with respect to the low deposition temperature.

In this study, sputtering method was used among the PVD techniques available for TiN, ZrN and WC coatings. Sputtering has been one of the most commonly used method because dense structure films and better adhesion could be obtained comparing with the CVD coating.

Sputtering is a physical process whereby atoms in a solid target material are ejected into the gas phase due to bombardment of the material by energetic ions.[11] Sputtering is largely driven by momentum exchange between the ions and atoms in the material, due to collisions. Although the first collision pushes atoms deeper into the cluster, subsequent collisions between the atoms can result in some of the atoms near the surface being ejected away from the cluster. The number of atoms ejected from the surface per incident ion is called the "sputter yield" and is an important measure of the efficiency of the sputtering process. Other things the sputter yield depends on are the energy of the incident ions, the masses of the ions and target atoms, and the binding energy of atoms in the solid.

The ions for the sputtering process are supplied by a plasma that is induced in the sputtering equipment. In practice a variety of techniques is used to modify the plasma properties, especially ion density, to achieve the optimum sputtering conditions, including usage of RF alternating current, utilization of magnetic fields, and application of a bias voltage to the target. The sputtered atoms, those ejected into the gas phase, are not in their thermodynamic equilibrium state. Therefore, they tend to condense back into the solid phase upon colliding with any surface in the sputtering chamber. This results in deposition of the sputtered material on all surfaces inside the chamber. This phenomenon is used extensively in the semiconductor industry to deposit thin films of various materials onto silicon wafers.

One important advantage of sputtering as a deposition technique is that the deposited films have the same composition as the target material.

Friction coefficient

To achieve optimum joint stability, preload should be as high as possible and frictional resistance causing loss of torque should be minimize.[18]

For that reason, it is important to reduce frictional resistance while tightening screw. Frictional force depends on the properties of the two contact materials, lubricant and preload.

Martin et al.[10] mentioned that abutment screw with enhanced surfaces reduced the coefficient of friction resulting in greater rotation angles and preload values than the conventional abutment screw. Elias et al.[7] also mentioned that the greater the preload applied to a screw joint, up to a maximum equal to the ultimate strength, the greater the resistance to loosening and the more stable the joint.

As frictional force is proportionate to friction coefficient, low friction coefficient of abutment screw could produce greater rotational angle and preload, which decrease the screw loosening.[5,10]

From the results of this study, the coated groups had lower coefficient of friction than non-coated control groups. In particular, the ZrN coating showed the lowest coefficient of friction. When TiN, ZrN and WC coating applied to the abutment screw, frictional resistance would be reduced, as a result, the greater preload and prevention of the screw loosening could be expected.

However, accoriding to Elias[7], this results might have adverse effects on the stability of abutment screws in a lower opening torque.

Hagiwara and Chashi[19] noted that abutment screw removal torque values decreased with a reduction in the friction coefficient. Therefore, further studies should be needed whether too high reduction of coefficient of friction promotes screw loosening.

Vickers hardness

A significant mechanism that results in screw loosening of implant prostheses is the settling effect. The settling effect[5,20], which plays a critical role in screw stability, is the result of no surface being completely smooth. Settling occurs when the microsurface irregularities of the contacting component surfaces become burnished flat from functional loads and vibrations. Wear of the contact areas brings the two surfaces closer together. Sakaguchi et al.[21] reported that 2% to 10% of the initial preload is lost as a result of settling. Consequently, the torque necessary to remove a screw is less than the torque initially used to place the screw. When settling exceeds the elastic elongation of the screw, the screw loosening occurs because there are no longer any contact forces to hold it in place.[20,22]

The extent of settling depends on the initial surface roughness, surface hardness, and magnitude of the loading forces.[5,20,22] Therefore, high surface hardness of the abutment screw could diminish settling effect.

In this study, TiN, ZrN, WC coating experimental groups showed the significant increasing Vickers hardness comparing with the control group. Particularly, from the result that TiN and ZrN had high Vickers hardness in this measurement. TiN or ZrN coated abutment screw could be expected to reduce the settling effect.

SEM investigation of coating surface

Mezger et al.[23] mentioned that although TiN coating certainly had remarkable properties, more improvements would be needed, especially regarding to the continuity of the coated surface such as absence of pores or microcracking. In resent study about comparison with TiN and ZrN surface roughness, TiN surface showed more roughness than ZrN surface.[12]

In this study, while ZrN and WC showed smooth and homogeneous surface, TiN had rough surface with defects. The results, TiN had more rough surface than ZrN, was compatible with the previous study.

It has been known that coated surface roughness influences to the frictional coefficient. Moreover, smooth coating surface could be expected to reduce the settling effect.

Adhesion strength

In mechanical aspects, the coating adhesion to the surface of the substrate is one of the most important properties.[14] Adhesion strength of coatings is widely studied by using the scratch test. This consists of introducing stresses by deforming the surface by means of indentation of a moving diamond tip. The applied load is increased continuously until film detachment. The smallest load at which the coating is damaged is called the critical load.

Milic et al.[11] noted that hard coatings exhibited good corrosion, abrasion and wear resistance. For this reason, the coatings should have good adhesion to the substrate.

In the measurement of TiN, ZrN and WC coating adhesion strength, TiN showed the highest adhesion strength and ZrN had the lowest adhesion strength. When the titanium substrate with bodycentered cubic lattice(bcc) is coated with TiN, good structural match is shown.[24] Therefore, it is considered that the match will bring the high adhesion strength.

However, to get more objective information about adhesion strength, further studies with comparative control group would be needed. In addition, study for the clinically acceptable adhesion strength also might be needed.

V. Conclusion

In this study, friction coefficient, adhesion strength and vickers hardness of TiN, ZrN and WC coated film on the titanium alloy surface were evaluated respectively, and the coating surface of titanium alloy specimens were also observed by FE-SEM.

The results were as follows ;

1. For all three coating conditions, friction coefficient was significantly decreased. Especially, ZrN coated surface showed the lowest value.

2. TiN coating showed the highest adhesion strength, however ZrN coating had the lowest value.

3. Vickers hardness of all three coatings was remarkably increased as compared with that of none coated specimen. TiN coating had the highest Vickers hardness, however WC coating showed the lowest value.

4. The ZrN or WC coated specimen showed a homogeneous and smooth surface, however the rough surface with defects was observed for TiN coating.

Conclusively, when TiN, ZrN and WC coating applied to the abutment screw, frictional resistance would be reduced, as a result, the greater preload and prevention of the screw loosening could be expected.
And TiN, ZrN and WC coated abutment screw expected to reduce the settling effect.

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