# Oxygen Plasma Ion Implantation of Biomedical Titanium Alloy

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***Abstract*—Titanium alloys have attracted more attention as biomaterials. Plasma ion implantation is utilized in this paper to improve the bioactivity, wear resistance, and corrosion resistance of Ti6Al4V alloy. As an effective surface-modification technique, plasma ion implantation eliminates the limitation of light of sight compared to conventional beam ion implantation. The plasma is excited by an RF power ranging from 200 to 400 W with sample bias of 20 kV. The results show the improvement in hardness, corrosion resistance, and tribological properties. Longer treatment time or higher RF power leads to a higher wear resistance. The friction coefficient rapidly increases at 500 s with the sample treated with the RF power of 400 W, while it changes abruptly at 1500 s with the sample processed with the RF power of 600 W. After the treatment, the corrosion resistance is considerably improved, which demonstrates that the potential of all the samples shift positively while the corrosion current decreases substantially. The corrosion current may decrease by a factor of six compared to that of the control sample. Precipitates containing phosphorous and calcium appear, indicating a better activity while nothing grows on the untreated sample.**

***Index Terms*—Bioactivity, biomaterials, oxygen plasma, plasma ion implantation, titanium.**

## I. Introduction

HARD tissues are often damaged due to accidents, aging, and other causes. It is a common practice to surgically substitute the damaged hard tissues with artificial replacements. Titanium and titanium alloys are widely used as hard-tissue replacements in artificial bones, joints, and dental implants. However, disadvantages such as a relatively poor wear resistance and poor bioactivity have limited its application to some extent. Increasing the tissue-bonding properties of titanium or titanium alloy implants for use in orthopedics and dentistry has recently become an active area of interest [1]. To enhance the surface bioactivity, elements such as Ca, Na, P, etc., have been utilized and implanted into titanium alloys [2]–[6]. Maitz *et al.* [6] performed sodium ion implantation and deposition using a high-voltage glow discharge plasma of evaporated sodium. The surface becomes active, as indicated by more calcium-phosphate precipitation. Hanawa *et al.* has investigated early bone formation on calcium-ion-implanted titanium, which is inserted into a rat tibia. Their results reveal that the $Ca^{2+}$ implanted titanium is superior to the unimplanted titanium from the perspective of bone conduction [2]. Krupa *et al.* [4] investigated the effects of a dual implantation of calcium and phosphorus on the structure, corrosion resistance, and biocompatibility of titanium. It was found that the $(Ca + P)$-implanted titanium possessed improved corrosion resistance and biocompatibility.

Of these elements biologically suitable for hard tissue replacements, oxygen is seldom investigated, which may be due to the fact that the native $TiO_2$ film on the surface of titanium alloys is inert. In this paper, oxygen ion was implanted into titanium alloys to increase the bioactivity, in addition to the improvement of the wear and corrosion resistances. Oxygen implantation was performed using a plasma immersion method. Plasma immersion ion implantation (PIII) is more preferred due to its capability to treat irregularly shaped components without manipulation of the holder compared to conventional ion-beam ion implantation [7]–[9]. It is attributed to a special acceleration mechanism of the plasma sheath during PIII processes and the plasma sheath being conformal to the components to be treated to some extent. In this way, the practical components such as a tooth, hip, and artificial heart can be treated uniformly and easily. In fact, another advantage that is more interesting is the batch-treatment capability. For conventional ion-beam implantation, the components have to be implanted one by one. In contrast, many components may be implanted simultaneously during PIII. Consequently, the process efficiency may be higher.

## II. Experiment

The samples were a commercial titanium alloy (Ti6Al4V), with composition of Al: 6.6 wt.%, V: 4.2 wt.%, Si: 0.07 wt.%, Fe: 0.3 wt.%, C: 0.03 wt.%, O:0.14 wt.%, N: 0.015 wt.%, H: 0.003 wt.%, and Ti: balance. The sample dimensions were $20 \times 20 \times 1$ mm. One side of the samples were polished to a mirror finish. After ultrasonic cleaning, the samples were loaded into a vacuum chamber. Before PIII, the samples were sputter cleaned with argon plasma

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TABLE I
INSTRUMENTAL PARAMETERS DURING PIII PROCESSES

| Sample No. | Gas pressure /pa | Bias voltage /kv | Pulse frequency /Hz | Pulse duration /μs | rf power /w | Treatment time /h |
|---|---|---|---|---|---|---|
| 1 | 0.4 | 20 | 100 | 20 | 400 | 1 |
| 2 | 0.4 | 20 | 100 | 20 | 200 | 1 |
| 3 | 0.4 | 20 | 100 | 20 | 600 | 1 |
| 4 | 0.4 | 20 | 100 | 20 | 400 | 1.5 |
| 5 | 0.4 | 20 | 100 | 20 | 400 | 0.5 |

ion bombardment. The pretreatment instrumental parameters were: RF power $= 400$ W with a reflected power of around 10 W, pulsed voltage applied to the sample $= 10$ kV, pulse frequency $= 100$ Hz, pulse duration $= 20$ $\mu$s, gas flow $=$ 5 sccm, and cleaning time $= 30$ min. Afterwards, oxygen was bled into the vacuum chamber and oxygen plasma was sustained by the RF power supply. The dominant ions were $O_2^+$ but some $O^+$ were also implanted [10]. The plasma density was maintained at about $1.0 \times 10^{15}$ ions/m$^3$, and the electron temperature was 2–5 eV. The PIII instrumental parameters are shown in Table I. Two series of experiments were conducted to disclose the effects of the RF power and treatment time on the surface properties. After the treatment, the samples were characterized for their surface hardness, tribological properties, corrosion resistance, and bioactivity, etc. The hardness was measured using a digital microhardness tester (HVS-1000). The applied load was 10 g with a press time of 20 s. To evaluate the corrosion behavior, potentiodynamic-polarization tests were conducted. The tests were carried out in a 0.9-wt.% NaCl solution and the scanning rate was 2 mV/s. The friction coefficients of all the samples were obtained using a ball on disk with 70-g load and rotation speed of 50 r/min. The friction couple was GCr15 with a diameter of 6.35 mm. To evaluate the bioactivity of the treated samples, a simulated-body-fluid (SBF) test was performed. After ultrasonically cleaned in acetone and rinsed in deionized water, the samples were soaked in the SBF solution. The SBF solution was buffered at pH 7.4 with trimethanol aminomethane-HCl, and the ionic concentrations in the solution are nearly equal to those in the human-blood plasma. If a bonelike apatite can form on the surface after soaked in the SBF for a period, it is thought to be bioactive [11].

## III. RESULTS

Oxygen plasma ion implantation effectively improves the surface hardness, as shown in Fig. 1. The hardness using a nanoindentor for the sample 1 is shown in Fig. 2 and compared to results of Ueda *et al.* [12]. The hardness of the titanium oxide is found to be lower than that of titanium nitride. The hardness improvement reported here may be attributed to the irradiation effect and formation of titanium oxide [10]. The surface hardness has been reported to improve by 2.2 times after oxygen implantation into the titanium with a dose of $3.0 \times 10^{17}/\mathrm{cm}^2$. A higher RF power is helpful in improving the surface hardness while the effect of a longer treatment time is not evident [13], [14] . The tribological properties as evaluated using the ball-

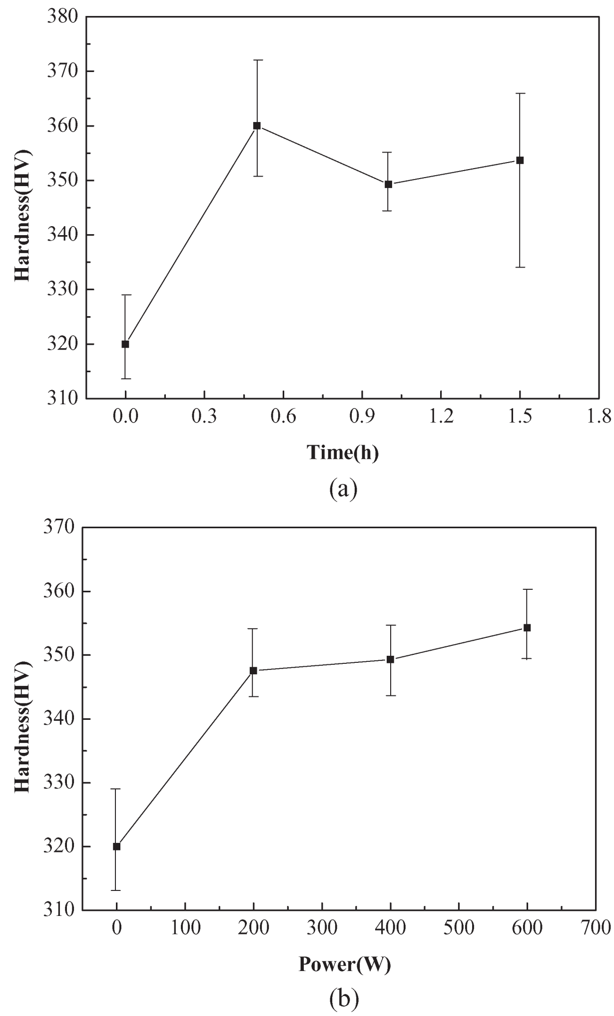



Fig. 1. Effect of processing parameters on a surface hardness. (a) Treatment time. (b) RF power.

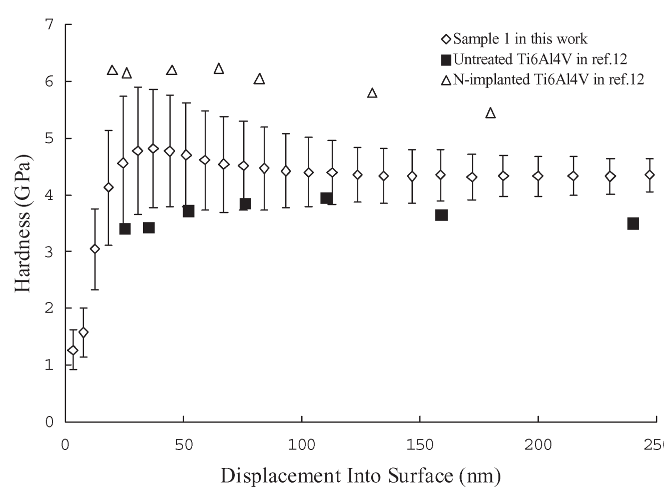



Fig. 2. Surface hardness of Sample 1 evaluated using nanoindentor.

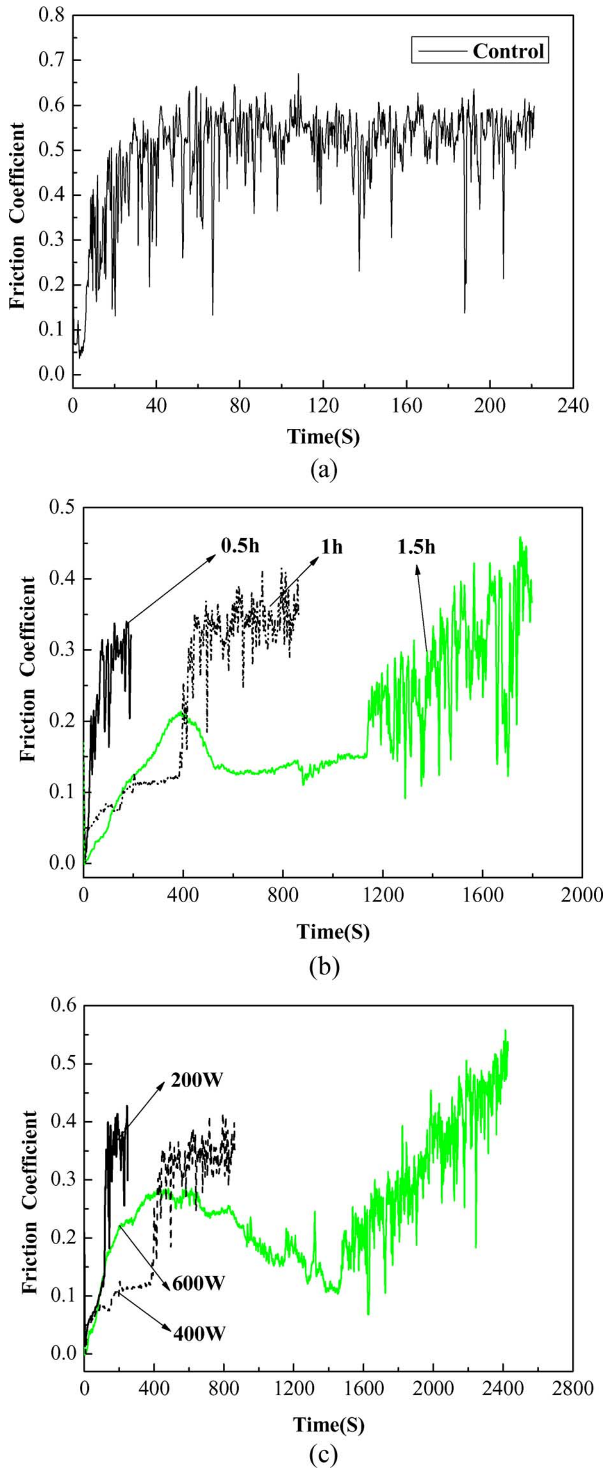



Fig. 3. Effects of processing parameters on a surface hardness. (a) Untreated sample. (b) Effect of the treatment time. (c) Effect of the RF power. (Color version available online at http://ieeexplore.ieee.org.)

on-disk tests are shown in Fig. 3. For the control sample, the friction coefficients rapidly rise to nearly 0.5–0.6 and show small fluctuations until the end of the test. The wear track discloses adhesion, tear, and oxidation, and the track boundary is evidently observed. In contrast, the treated samples demonstrate improved tribological properties. A lower friction coefficient be achieved even if the top surface is rapidly damaged for the sample treated for 30 min, as shown in Fig. 3(c). The treatment time has a critical influence on "punchthrough" The "punchthrough" occurs earlier for the sample treated for less time (e.g., 30 min). In contrast, the longer treatment time (e.g., 90 min) yields enhanced tribological properties demonstrated by the delayed "punchthrough." As shown in Fig. 3(b), the "punchthrough" is substantially influenced by the RF power. A higher RF power helps to improve the tribilogical properties. In fact, increase of the RF power or the treatment time means a larger implantation dose, which may be responsible for the enhancement of the surface properties.

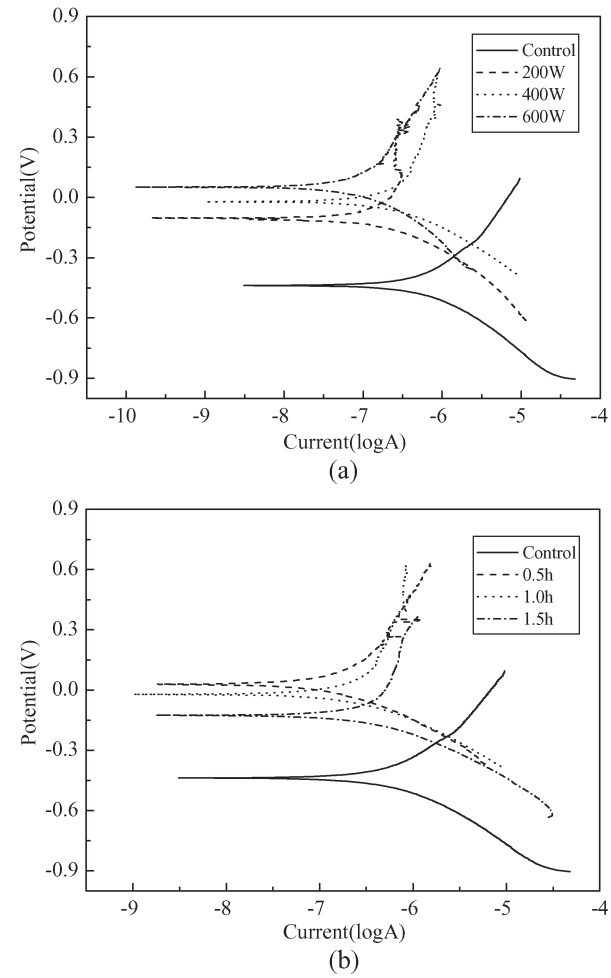
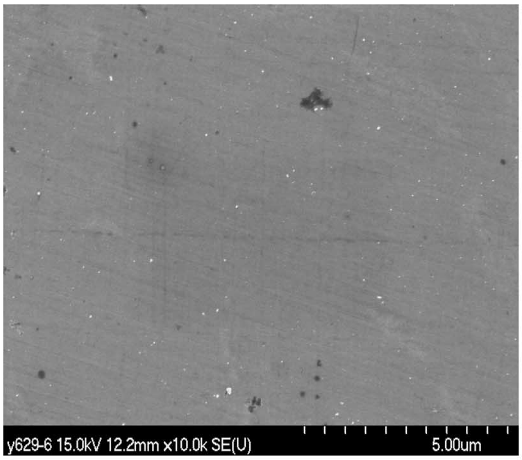
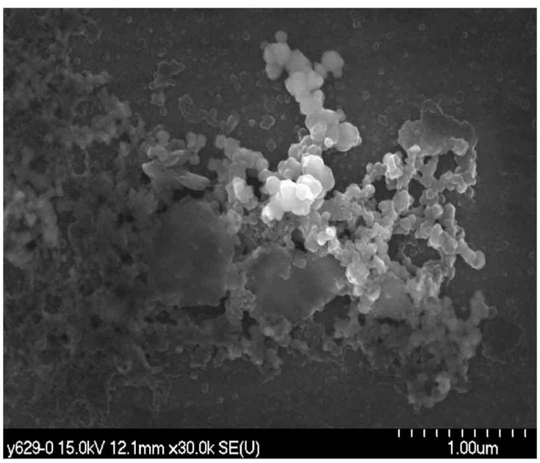



Fig. 4. Polarization curves obtained in a 0.9-wt.% NaCl solution. (a) Effect of the RF power. (b) Effect of the treatment time.

Polarization tests were used to evaluate the corrosion resistance of all the samples. Oxygen plasma ion implantation improves the corrosion resistance of the treated samples. The corrosion potential of all the treated samples shifts positively while the corrosion current decreases compared to that of the untreated sample. The increase of the RF power has a positive effect on the corrosion resistance. As shown in Fig. 4(a), the higher the RF power, the more positive is the corrosion potential. For example, the corrosion potentials are $-0.102$, $-0.022$, and 0.05 V for the samples treated with the power of 200, 400, and 600 W, respectively, while the corrosion potential of the untreated sample is $-0.438$ V. In contrast, the longer treatment time is not beneficial, as shown in Fig. 4(b). For example, the corrosion potentials of the samples treated for 30, 60, and




(a)




(b)

Fig. 5. SEM morphology showing the oxygen-implantation-induced bioactivity. (a) Untreated sample. (b) Treated sample.

90 min, respectively are 0.029, −0.022, and −0.126 V. Of these three samples, the sample treated with the longest time (90 min) also demonstrates the largest corrosion current.

After the samples were immersed in the SBF fluid in six days, the sample surface was observed using scanning electronic microscopy (SEM) as shown in Fig. 5. The unimplanted titanium surface is quite smooth, and only very small particles sparsely distributed can be observed. In contrast, the implanted titanium samples possess a rough surface, and balllike or chainlike precipitates can be observed. The bioactivity has been improved after the oxygen plasma ion implantation. Oxygen implantation induces more rapid precipitation compared to the untreated sample. The precipitates contain calcium and phosphorous as indicated by an energy-dispersive-spectroscopy (EDS) analysis, although the amount is small, but it may be due to a short immersion time.

## IV. Discussion

Oxygen plasma ion implantation has been demonstrated to effectively improve the surface properties of biomedical titanium alloys. The ion range is very small, (only 17–18 nm in this paper according to a TRIM program) and diffusion of the injected ions is small due to the low temperature (lower than 250 °C). However, it leads to improvements in surface hardness, although the penetration depth of the indenter approaches 1 $\mu$m, which is much larger than the ion range. This improvement, which is to a much deeper region than the ion penetration range has also been found elsewhere. The TiO may be formed in cases of a low processing temperature and low implantation dose described in this paper [12]. It was reported that TiO formed at a lower temperature while $TiO_2$ formed at a higher temperature [14], [15]. Meanwhile, TiO-$TiO_2$ transformation is difficult at a low temperature even if a large dose is implanted. Pin-on-disk tests show that oxygen ion implantation improves the tribological properties of the treated samples [15]. The longer the implementation time or higher the RF power, the more evident is the wear resistance improvement. This is consistent in that a higher dose is beneficial for mechanical properties. The titanium oxide film is responsible for the enhancement of the corrosion resistance [13], [14]. As reported by Armstrong and Quinn, the oxide layer (thinner than 1.5 nm) may effectively block the charge transfer at the beginning of the oxidation processes. A thick oxide layer is formed after oxygen ion implantation [16]. This thick layer hinders the charge transfer more effectively as reflected by the reduced corrosion current of the treated samples.

Oxygen is one of the frequently used elements to strengthen the surface properties such as wear, corrosion, outgassing, etc. Titanium oxide is known to have varying stoichiometries, and the common compounds are $Ti_3O$ to $Ti_2O$, $Ti_3O_2$, TiO, $Ti_2O_3$, $Ti_3O_5$ and $TiO_2$. Rutile titanium oxide ceramics, $TiO_2$ and $TiO_x$ films, which are prepared by thermal oxidation, ion beam-assisted deposition (IBAD), or ion implantation [18]–[22], generally have blood compatibility better than that of clinically biomaterials, such as low-temperature isotropic carbon (LTIC) [17]. It has been suggested that the TiO film possesses better blood compatibility than LTIC because of its suitable surface (interface) energy properties and the behaviors of adsorbed proteins. Leng *et al.* [21], [22] suggest that it is related to the semiconductor behavior (n-type) of the TiO films. TiO films have become more interesting as blood-contact materials [21]. However, oxygen implantation has seldom been reported to enhance the bioactivity of biomedical titanium alloys used in hard tissue replacements. The native oxide films are inert and have no bioactivity as demonstrated here and in other works in the literature. However, the top surface may be activated after oxygen plasma implantation. As such, ion implantation is not a steady-state process, and many effects may be achieved. Based on the assumption of the semiconductor behavior (n-type) of the oxygen-implanted titanium surface with proper processing parameters [21], it can be understood more easily that oxygen implantation may achieve better bioactivity. This n-type semiconductor effect is consistent with the assumption of apitate formation. In water with a neutral pH, a small negative charge forms on the surface of the titanium due to a fraction of the acidic hydroxides being deprotonated, while almost all of the basic and a large part of the acidic groups are still present in a neutral form [23]. In this way, the calcium ions from the body fluid adsorb onto the titanium surface because of a coulombic attraction. When more calcium ions adsorb, more phosphate ions are subsequently attracted, thereby accelerating the formation of the calcium phosphate. From this viewpoint, the semiconductor behavior of the oxygen-implanted titanium is helpful in improving the surface bioactivity.

## V. Conclusion

Oxygen plasma ion implantation has been utilized to improve the surface properties of biomedical titanium alloys. After the treatment, the surface hardness is increased depending on the processing parameters. The treated samples demonstrate better tribological properties, which are indicated by lower friction coefficients. The corrosion resistance is also improved by oxygen plasma ion implantation, which is demonstrated by a lower corrosion current and more positive corrosion potential compared to those of the untreated sample. The bioactivity of the titanium alloys is effectively improved. It may be explained by the semiconductor behavior of the treated surfaces. Further work has to be done to disclose the mechanism and optimize the processing parameters.

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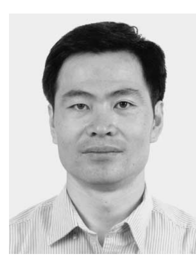

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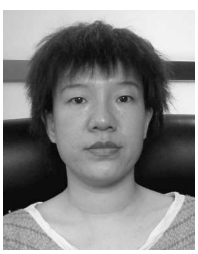

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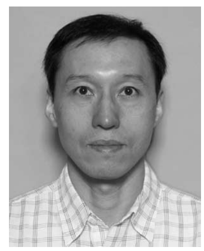

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