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Microstructural and Tribological Properties of ZrO_2 Film Grown by DC Magnetron Sputtering Technique

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Anahtar Kelimeler

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Graphical/Tabular Abstract (Grafik Özet)

ZrO_2 film was deposited on the surface of CP-Ti material by DC magnetron sputtering technique. The wear resistance of ZrO_2 film was examined using pin-on-disc tester. After the wear test, SEM images were taken and the wear mechanism formed on the surfaces was determined. The wettability properties of ZrO_2 film were analyzed./ CP-Ti malzemesi yüzeyine DC magnetron sıçratma tekniği ile ZrO_2 filmi biriktirilmiştir. ZrO_2 filminin aşınma direnci pin-on-disk test cihazı kullanılarak incelenmiştir. Aşınma testi sonrası SEM görüntüleri alınarak yüzeylerde oluşan aşınma mekanizması belirlenmiştir. ZrO_2 filminin ıslanabilirlik özellikleri analiz edilmiştir.

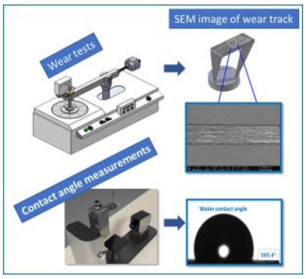

***Figure A**: The wear and contact angle measurements of ZrO_2 coated CP-Ti material*

***Şekil A:** ZrO_2 kaplı CP-Ti malzemenin aşınma ve temas açısı ölçümleri*

Highlights (Önemli noktalar)

- *ZrO_2 coatings have been deposited to improve the tribological properties of CP-Ti material in biomedical applications. / ZrO_2 kaplamalar, biyomedikal uygulamalarda CP-Ti malzemesinin tribolojik özelliklerini iyileştirmek için biriktirilmiştir.*
- *According to the XRD analysis of the ZrO_2 coated film, it was determined that both monoclinic and tetragonal oxide structures were formed. / ZrO_2 kaplı filmin XRD analizine göre, hem monoklinik hem de tetragonal oksit yapıları oluştuğu belirlenmiştir.*
- *It has been observed that the wear resistance increases with the ZrO_2 layer deposited after the coating process with the magnetron sputter system. / Magnetron sputter sistemi ile kaplama işlemi sonrasında biriktirilen ZrO_2 tabakası ile aşınma direncinin arttığı görülmüştür.*

Aim (Amaç): *The aim of this study is to investigate the microstructural, tribological and wettability properties of ZrO_2 thin films deposited on CP-Ti substrate using DC magnetron sputtering technique. /Bu çalışmanın amacı, DC magnetron sıçrama tekniği kullanılarak CP-Ti taban malzemesi üzerine biriktirilen ZrO_2 ince filmlerin mikroyapısal, tribolojik ve ıslanabilirlik özelliklerini araştırmaktır.*

Originality (Özgünlük): *The wear resistance of the material was increased and it showed hydrophobic properties with the 2.15 µm thick ZrO_2 film formed on the surface of the CP-Ti material. / CP-Ti malzemesi yüzeyinde oluşturulan 2.15 µm kalınlığındaki ZrO_2 film ile malzemenin aşınma direnci artmış ve hidrofobik özellik göstermiştir.*

Results (Bulgular): *The lowest friction coefficient value of 0.25±0.04 and also the lowest wear rate of 1.20±0.10 mm^3/Nm were found from the oxide deposited surfaces. / En düşük sürtünme katsayısı değeri 0,25±0,04 ve ayrıca en düşük aşınma oranı 1,20±0,10 mm^3/Nm olarak oksit biriktirilmiş yüzeylerden bulunmuştur.*

Conclusion (Sonuç): *It was concluded that the ZrO_2 film deposited on the CP-Ti material used as a biomaterial by the DC magnetron sputtering technique has hydrophobic properties and reduces the wear rate. / Biyomalzeme olarak kullanılan CP-Ti malzemesine DC magnetron sıçratma tekniği ile biriktirilen ZrO_2 film hidrofobik özellikte olup, aşınma oranını azalttığı sonucuna varılmıştır.*

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Abstract

To improve the mechanical and tribological properties of biomedical materials, biocompatible coatings are obtained on surfaces using various methods. In this study, ZrO_2 thin films were deposited on CP-Ti material used as biomaterial by DC magnetron sputtering method. In this context, structural, mechanical, tribological and wettability properties of ZrO_2 film coated on CP-Ti base material were investigated. X-ray diffraction method (XRD), scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDS) were used to characterize the crystallographic structure and surface morphology of the obtained film. Microhardness and wear tests were carried out to investigate the mechanical and tribological properties of the coatings. Additionally, contact angle measurements were made with pure water and ethylene glycol to determine the hydrophobicity and oleophobicity properties of the ZrO_2 coated samples. The water contact angle and the oil contact angle of the film are 105.4° and 73.2, respectively. The results showed that ZrO_2 coating film deposited on surfaces by DC magnetron sputtering method was more resistant to wear compared to bare CP-Ti.

DC Magnetron Sıçratma Yöntemi ile Büyütülen ZrO_2 Filminin Mikroyapısal ve Tribolojik Özellikleri

Makale Bilgisi

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Öz

Biyomedikal malzemelerin mekanik ve tribolojik özelliklerini iyileştirmek için çeşitli yöntemler kullanılarak yüzeylerde biyouyumlu kaplamalar elde edilmektedir. Bu çalışmada, biyomalzeme olarak kullanılan CP-Ti malzemesi üzerine DC magnetron püskürtme yöntemi ile ZrO_2 ince filmler biriktirilmiştir. Bu kapsamda, CP-Ti taban malzemesi üzerine kaplanan ZrO_2 filminin yapısal, mekanik, tribolojik ve ıslanabilirlik özellikleri incelenmiştir. Elde edilen filmin kristalografik yapısını ve yüzey morfolojisini karakterize etmek için X-ışını kırınımı yöntemi (XRD), taramalı elektron mikroskobu (SEM) ve enerji dağılımlı X-ışını spektroskopisi (EDS) kullanılmıştır. Kaplamaların mekanik ve tribolojik özelliklerini incelemek amacıyla mikrosertlik ve aşınma testleri gerçekleştirilmiştir. Ayrıca, ZrO_2 kaplı numunelerin hidrofobik ve oleofobik özelliklerini belirlemek için saf su ve etilen glikol ile temas açısı ölçümleri yapılmıştır. Filmin su temas açısı ve yağ temas açısı sırasıyla 105,4° ve 73,2'dir. Sonuçlar, DC magnetron püskürtme yöntemi ile yüzeylere biriktirilen ZrO_2 kaplama filminin, işlemsiz CP-Ti'ye göre aşınmaya karşı daha dirençli olduğunu göstermiştir.

1. INTRODUCTION (GİRİŞ)

Titanium and its alloys are used more in the biomedical field compared to stainless steel and cobalt alloys due to their high corrosion resistance, long fatigue life and high biocompatibility [1-3]. Despite the advantages of Ti and Ti alloys, their poor wear resistance properties limit their use in biomedical applications [4]. Although the most preferred commercially pure titanium (CP-Ti) material has good corrosion resistance and biocompatibility properties, many studies are being conducted on improving its low tribological performance [5]. Various surface treatments are applied to the surfaces to improve the low tribological properties of commercially pure titanium [6-8]. Magnetron sputtering technique is one of these surface treatment methods and this method is preferred due to its many advantages such as ease of sputtering of any metal, alloy or compound, high deposition rate, ease of automation, high purity films, high adhesion and high wear

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resistance of films [9-11]. Many oxide types of bioceramic films (TiO_2, Al_2O_3, Nb_2O_5, Ta_2O_5, ZrO_2) can be deposited on titanium material by magnetron sputtering technique [12-14]. Zirconium dioxide (ZrO_2) one of the bioceramic oxide types, are widely used in metal cutting tools, nuclear energy reactors, and biomedical applications due to their high corrosion resistance and mechanical properties [14,15]. Some of the experimental studies in the literature examining the mechanical and wettability properties of ZrO_2 films are summarized as follows: ZrO_2 thin films were deposited by radio frequency magnetron sputtering system in order to improve hardness and corrosion properties of 316L stainless steel samples. It was determined that the film properties changed depending on the applied plasma parameters and the monoclinic phase was formed according to the XRD result [16]. ZrO_2 nanotubes were produced on Ti–6Al–7N biomaterial under different conditions by anodization method. Zr was coated on the material by physical vapor deposition magnetron sputtering method and then subjected to anodization process. Coated materials were annealed at 450◦C and 650◦C and tetragonal oxide structure was determined in the formed film. After annealing at 850◦C and 900◦C, the formed phase structure was monoclinic [17]. Zirconium oxide was deposited on silicon and glass substrates by PVD method. A monoclinic oxide layer was formed on the surface and the highest contact angle was obtained by the coating process carried out at 400°C [18]. Zirconium dioxide films were formed by magnetron sputtering with different Ar partial pressure values. As the partial pressure value of Ar gas increased, the thickness of the oxide film increased and the minimum transmittance value above 63% was found in all optical properties [19]. In another study, the wear resistance of ZrO_2 coating on Al and tool steel by electrolysis method was investigated. In the wear tests, it was observed that the coefficient of friction and wear resistance of the coated pins were lower compared to the uncoated ones [20]. Zirconium oxide films were coated on glass substrates at different operating pressures using the RF magnetron sputtering method. When the operating pressure decreased from 3.5 Pa to 1 Pa, the contact angle value of the films also decreased. As a result of the wettability test, it was determined that the film was hydrophobic and delayed ice formation compared to the bare Ti [21].

In this study, the structural, mechanical and wettability properties of the ZrO_2 film formed on the surface by DC magnetron sputtering technique on the CP-Ti material were investigated. For this purpose, the structural properties of the samples were determined by XRD, SEM, EDS and microhardness tester, and the tribological properties of bare and ZrO_2 coated samples were determined by a pin-on-disc tribo-tester. The contact angle measurements of the surfaces were made using pure water and ethylene glycol solutions. The hydrophobicity and oleophobicity of the obtained ZrO_2 film on CP-Ti sample were observed.

2. MATERIALS AND METHODS (MATERYAL VE METOD)

2.1. Experimental Equipment (Deneysel Ekipman)

CP-Ti plates used in the field of biomaterials were used as the base material. For the cleaning process of the samples, sanding was done with 80, 220, 400, 800, 1000 and 1200 grid abrasive papers. Then, fine polishing process was applied with 5 µm and 1 µm diameter alumina powders. In the final cleaning process, the samples were washed with ethyl alcohol and ultrapure water in an ultrasonic cleaning device, respectively, and dried with alcohol. In addition, magnetron sputter coating was performed on glass materials for some structural analyzes. The cleaned substrates were coated with oxide films using 99.95% purity zirconium (Zr) target material with a magnetron sputtering device (Figure 1). Oxygen gas (purity 99.99%) was used to obtain 1 target material and ZrO_2 films in the system. Ion cleaning process was applied to the samples placed in the system with high purity Argon gas with 80 W DC power for 25 minutes. The parameters that obtained a homogeneous and continuous oxide layer using various experimental parameters are shown in Table 1.

Table 1. Deposition parameters of ZrO_2 thin film (ZrO₂ ince filminin biriktirme parametreleri)

Working gas pressure	5 mTorr
DC power	300 Watt
O_2 gas flow rate	20 sccm
Coating Time	4 h
Coating Temperature	100 °C

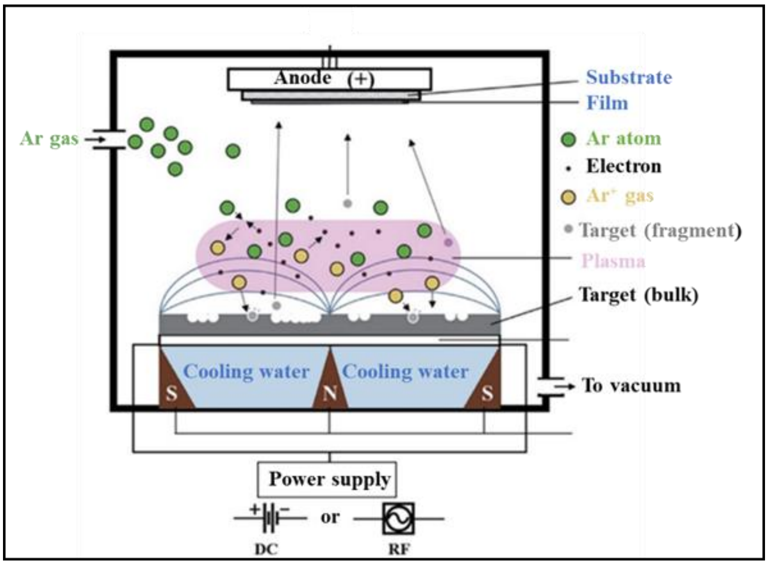

Figure 1. Schematic representation of magnetron sputtering method [23] (Magnetron sıçratma yönteminin şematik gösterimi)

In order for the deposited films to have a crystalline structure, the samples were annealed at 500°C in a Protherm furnace. To analyze the crystal structure of the coating film, the Grazing Incidence X-Ray Diffraction (GIXRD) method was used in the Panalytical Empyrean-XRD device; the surface image, thickness and chemical composition of the film were measured with the FEI Quanta FEG-450 SEM-EDS device. In order to determine the mechanical properties of the samples, the hardness of the sample surface was measured with the Bruker UMT-2 mechanical testing device (ASTM E92) with a Vickers tip for 15 s. The surface roughness of the ZrO_2 film on the surface was determined with a Bruker Contour GT-K profilometer brand 3D profilometer.

In order to determine the tribological properties of bare CP-Ti and oxide film coated samples, wear tests were performed for 1200 s under a 2N load on the Turkyus PODWT&RWT wear device. Aluminum oxide (Al_2O_3) ball with a diameter of 6 mm was used as the pin for the wear tests. The friction coefficient values were measured with a load cell connected to the wear device and the data were read from the computer. Wear scars were examined with a 3D non-contact optical microscope, and then the wear volumes were found. The wear rates of the samples were then calculated according to the equation (1).

$$W = \frac{V}{P \times D} \qquad (1)$$

where W: wear rate (mm^3/N.m), V: wear volume (mm^3), P: normal load (N) and D: sliding distance (m) [22].

Static contact angle measurements with water and oils were performed to estimate the hydrophobic properties and oleophobic of the coating film. The wettability of the sample surface was determined by measuring the contact angle (CA) of water and ethylene glycol (drop volume of 5 µl) on the surface of the sample using the Attension Theta Lite device. Five measurements were made at different locations for each sample and their averages were taken as the contact angle value.

3. RESULTS (BULGULAR)

According to the XRD result of the film formed on the surface, it was determined that it was amorphous and therefore the samples were annealed for the crystallization of the ZrO_2 film. After the annealing process, the GIXRD graph of the sample surface film was taken. According to the GIXRD result given in Figure 2, the 1st phase (111), 4th phase (220), 5th phase (311) and 7th phase (400) planes formed in the coating film correspond to the tetragonal phase of ZrO_2 (JCPDS: 27-0997) [24]. In the 2nd phase (200) plane specified in Figure 2, ZrO_2 corresponds to the monoclinic phase. In addition, the α (alpha) phase coming from the phase structure of the CP-Ti material used as the base material is also seen in the XRD results [6].

Figure 3a shows the surface image of the bare CP-Ti material, while Figure 3b shows the structure of the ZrO_2 film after annealing at 500°C. It was observed from the image of the coating film that the oxide film was formed in the form of homogeneous and clustered ball-like particles. Especially after the annealing process at 500°C, the ZrO_2 film on the surface was crystalline. The thickness of the ZrO_2 film was detected as approximately 2.15 µm from the SEM images. EDS analysis results showed that the coated material consisted of O and Zr with atomic concentrations of 66.45% and 33.55%, respectively (Figure 4). The microhardness of the bare and ZrO_2 coated samples are given in Table 2. The lowest surface hardness and roughness values were observed for Ti with values of 3.4 ± 0.18 GPa and 0.15 ± 0.25 µm, respectively. The microhardness and roughness values of the ZrO_2-coated CP-Ti sample ranged between 6.5 ± 0.20 GPa and 0.35 ± 0.10 µm, respectively.

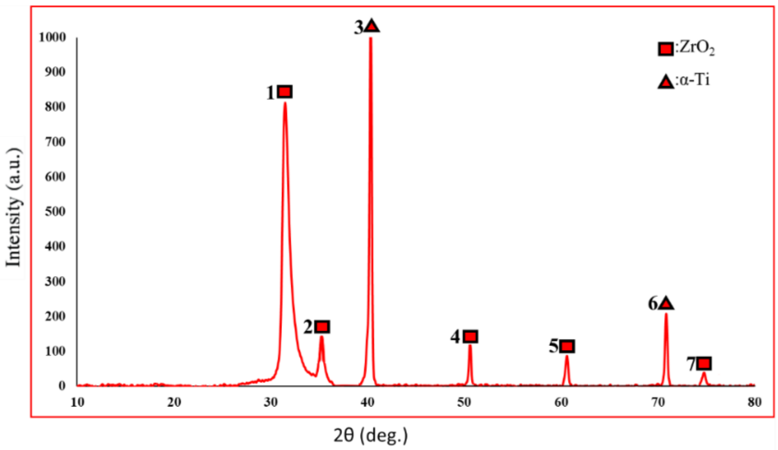

Figure 2. Grazing incidence XRD analysis result of ZrO_2 film (ZrO₂ filminin küçük geliş açılı XRD analiz sonucu)

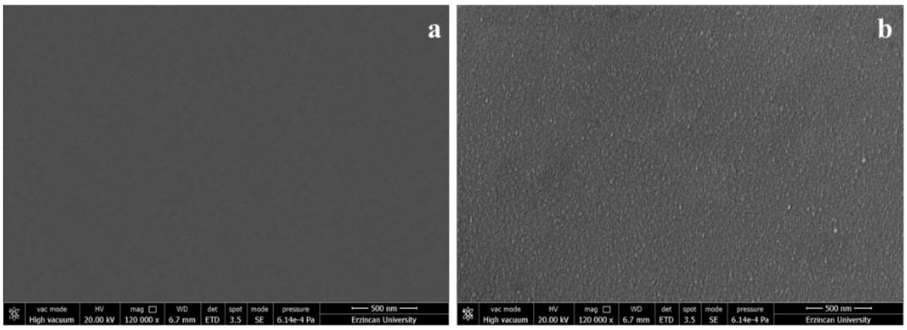

Figure 3. SEM surface images of samples (a) before and (b) after ZrO_2 coating (Numunelerin SEM yüzey görüntüleri (a) öncesi ve (b) ZrO_2 kaplama sonrası)

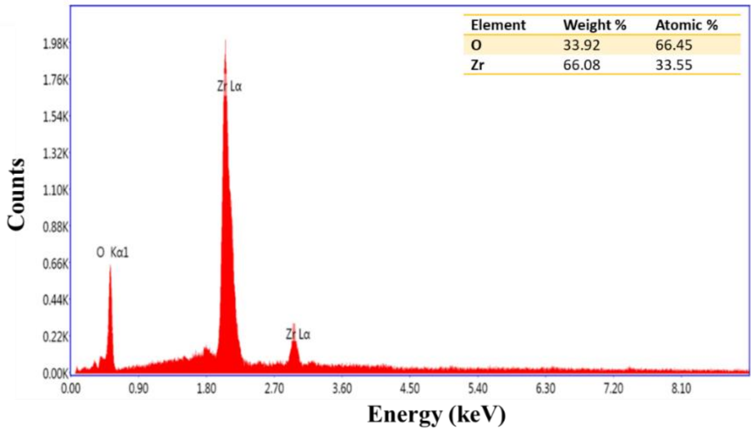

Figure 4. EDS analys result of ZrO_2 film (ZrO₂ filminin EDS analiz sonucu)

Figure 5 shows the coefficient of friction (CoF) graph obtained under 2N load for the bare and ZrO_2 coated CP-Ti sample. According to the friction graphs obtained from the wear test performed under 2N load, the CoF values increased as the surface roughness increased. A more oscillatory curve was obtained in the CoF graph obtained from the bare sample. The highest coefficient of friction value was calculated as 0.58 ± 0.05 on average from the bare CP-Ti sample. As shown in Figure 5, the coefficient of friction of the ZrO_2 film annealed at 500 °C was determined as approximately 0.25±0.04 after the alumina ball contacted the surface for an average of 600 s. As a result, the coefficient of friction of the bare CP-Ti material decreased significantly after the coating with the oxide film obtained by magnetron sputtering method. The film deposited on the surface by magnetron sputtering increased the surface roughness of the material. The hard oxide phases formed in the sample also change the wear resistace of the material [23].

The wear rates of bare and ZrO_2 coated CP-Ti samples are given in Table 2. The ZrO_2 film coated by sputtering method significantly increased the surface hardness of the material. The wear rate also decreases with the increased load carrying capacity due to the increase in surface hardness [25]. The wear rate value decreased significantly due to the stable structure of the tetragonal and monoclinic ZrO_2 layer formed on the surface after the coating process (Table 2). It was determined that the ZrO_2 film deposited on the surface has a protective feature against wear due to the increase in the wear life of the coated sample compared to the bare sample.

The wear rate of the oxide coated film is quite low compared to the bare sample. This is due to the stability of the ZrO_2 structure deposited on the surface and the sufficient thickness of the film. While the wear rate of the bare sample was 2.60±0.15 mm^3/Nm, the wear rate of the ZrO_2 coated sample was 1.20±0.10 mm^3/Nm (Table 2). The relationship between the wear test results of the bare and ZrO_2 coated samples using Al_2O_3 balls and their hardness values is given in Figure 6. It is seen that the wear rate decreases as the hardness value of the coated sample increases.

Figure 7 shows the wear marks obtained as a result of the wear test performed under a normal load of 2N in dry environment on the bare CP-Ti and ZrO_2 coated CP-Ti samples, respectively. It is observed that plastic deformation occurs in the bare CP-Ti sample and adhesive wear type occurs. The wear mark width was obtained from the bare CP-Ti sample at the widest. During the wear applied to the ZrO_2 film deposited on the surface, the wear mechanism turned into abrasive wear due to the film breaking and the particles being included in the wear. The wear mark width decreased significantly on the ZrO_2 coated surface compared to the bare sample. It was clearly seen in the SEM images after the wear that the wear resistance increased with the zirconium oxide layer deposited after the coating process with magnetron sputter system (Figure 7b).

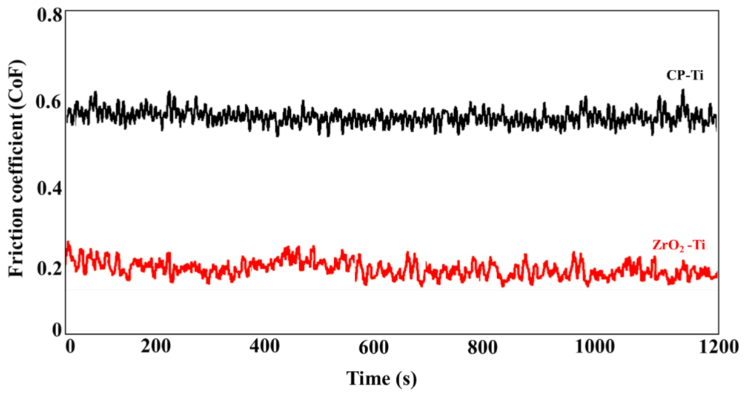

Figure 5. Friction coefficient-time graph for bare and ZrO_2 coated CP-Ti samples (Kaplamasız ve ZrO_2 kaplı CP-Ti numuneler için sürtünme katsayısı-zaman grafiği)

Table 2. The surface roughness, hardness, coefficients of friction (CoF) and wear rate values (Yüzey pürüzlülüğü, sertlik, sürtünme katsayısı (CoF) ve aşınma oranı değerleri)

	Surface roughness, Ra (µm)	Microhardness (GPa)	Average CoF (µm)	Wear Rate x10^{-3} (mm^3/Nm)
CP-Ti	0.15 ± 0.25	3.4 ± 0.18	0.58 ± 0.05	2.60 ± 0.15
ZrO_2-Ti	0.35 ± 0.10	6.5 ± 0.20	0.25 ± 0.04	1.20 ± 0.10

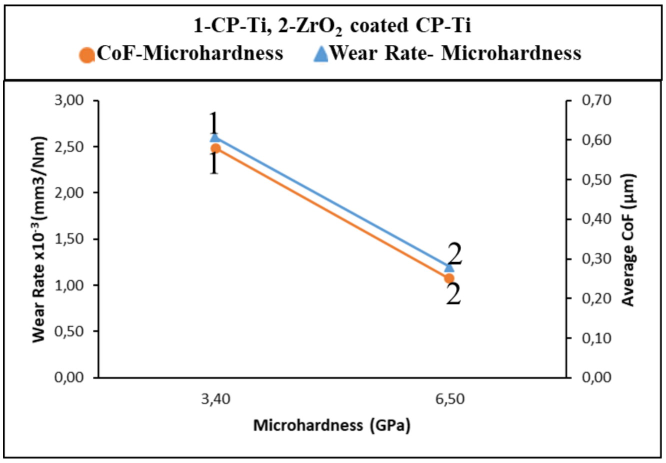

Figure 6. Relationship of wear rate and friction coefficient to hardness of bare and ZrO_2 coated CP-Ti samples (Kaplamasız ve ZrO_2 kaplı CP-Ti numunelerin aşınma oranı ve sürtünme katsayısının sertlikle ilişkisi)

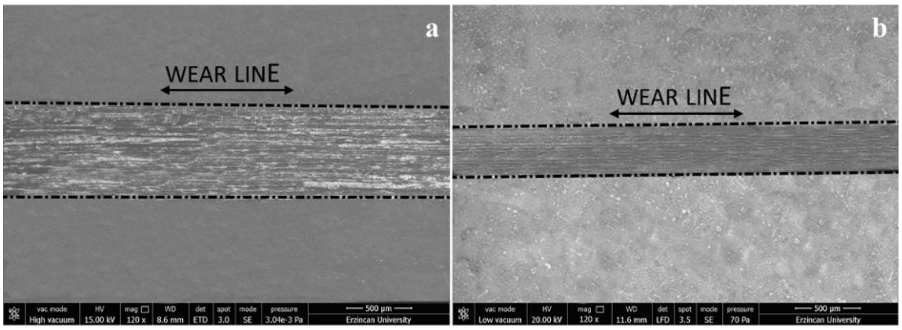

Figure 7. SEM images of wear tracks: (a) bare CP-Ti, (b) ZrO_2 coated CP-Ti (Aşınma izlerinin SEM görüntüleri: (a) kaplamasız CP-Ti, (b) ZrO_2 kaplı CP-Ti)

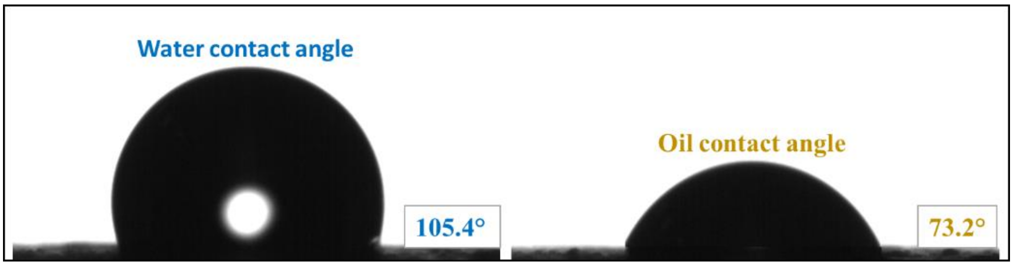

Figure 8. Water and oil contact angles of ZrO_2 coated CP-Ti (ZrO₂ kaplı CP-Ti'nin su ve yağ temas açıları)

The wettability properties of the ZrO_2 coating film with water and oil solutions were found by the contact angle measurement results (Figure 8). In our previous study [6], the contact angle measurement values of bare CP-Ti with water and oil were found 89° and 98°, respectively. The water contact angle value measured from the surface after the oxide film deposited on the surface was 105.4°. The water drop left on the surface is greater than 90 degrees, the surface shows hydrophobic properties [1]. Surface hydrobicity increased significantly compared to the bare sample due to the increase in surface roughness of the ZrO_2 film deposited on the CP-Ti surface (Table 2). The ability to repel oil drops left on the surface is defined as oleophobic [26]. Although it showed oil repellency after the coating process with ZrO_2 film (73.2°), the oleophobicity decreased slightly after the coating compared to the one without the process. As a result, the formed ZrO_2 film on CP-Ti, which increased the wear resistance, showed hydrophobic properties on the surface. It has been found that the CP-Ti material used as a biomaterial with its self-cleaning surface film contributes positively to the wear and hydrophobicity properties.

4. CONCLUSIONS (SONUÇLAR)

In this study, microstructural, microhardness, tribological, wettability and behavior of ZrO_2 films on CP-Ti material used as biomaterial were investigated by DC magnetron sputtering method. The obtained results are summarized below:

- According to the XRD result of the ZrO_2 film annealed at 500 °C, both monoclinic and tetragonal oxide structures were observed on deposited sample.
- As a result of the elemental analysis of the CP-Ti deposited film, the atomic weights of the coating components were measured as 33.55% and 66.45% for zirconium and oxygen, respectively.
- The average microhardness and roughness values of the ZrO_2-coated CP-Ti sample ranged between 6.5 ± 0.20 GPa and 0.35 ± 0.10 µm, respectively.
- The lowest friction coefficient value of 0.25±0.04 and also the lowest wear rate of 1.20±0.10 mm^3/Nm were found from the oxide deposited surfaces.
- According to SEM images of wear tracks, the adhesive wear was observed on the bare CP-Ti and it was determined that the mechanism turned into abrasive wear after ZrO_2-coating.
- The contact angle value of the ZrO_2-coated CP-Ti sample with water was found 105.4° and the value measured with oil was found 73.2°. The ZrO_2-coated CP-Ti surface was becomes hydrophobic.

DECLARATION OF ETHICAL STANDARDS (ETİK STANDARTLARIN BEYANI)

The author of this article declares that the materials and methods they use in their work do not require ethical committee approval and/or legal-specific permission.

Bu makalenin yazarı çalışmalarında kullandıkları materyal ve yöntemlerin etik kurul izni ve/veya yasal-özel bir izin gerektirmediğini beyan ederler.

AUTHORS' CONTRIBUTIONS (YAZARLARIN KATKILARI)

Mevra ASLAN ÇAKIR: She conducted the experiments, analyzed the results and performed the writing process.

Deneyleri yapmış, sonuçlarını analiz etmiş ve makalenin yazım işlemini gerçekleştirmiştir.

CONFLICT OF INTEREST (ÇIKAR ÇATIŞMASI)

There is no conflict of interest in this study.

Bu çalışmada herhangi bir çıkar çatışması yoktur.

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