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Research Article

Tribological Properties of Films Coated on CP-Titanium and CoCrMo Alloy by Cathodic Arc Deposition Method

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Abstract

The improvement of tribological properties of biomaterials in load-bearing implants is very important. In this study, the tribological properties of TiN and ZrN films deposited on surfaces by CA-PVD method on two different types of biomaterials (CP-Ti and CoCrMo alloy) were compared under dry wear conditions and 1N and 3 N loads. In addition, the microstructural and mechanical properties of TiN and ZrN films deposited on the surface on CP-Ti and CoCrMo materials were investigated. The crystal structure, elemental composition and surface morphology of TiN and ZrN coated CP-Ti and CoCrMo materials were determined using XRD, SEM and SEM-EDS analyses, respectively. According to the test results conducted in a dry environment after the wear test under 1N load, the lowest friction coefficient was found in the untreated CoCrMo sample at approximately 0.35, while the highest friction coefficient was found in the ZrN-coated CoCrMo sample at 0.55. While the lowest wear rate under 3N load was $0.38\text{x}10^{-6}$ mm^3/Nm in the ZrN/CCM sample, the highest wear rate was $2.10\text{x}10^{-6}$ mm^3/Nm in the untreated Ti sample. As a result, it was determined that the microhardness values and wear resistance of the TiN and ZrN-coated CP-Ti and CoCrMo samples increased.
Keywords: CA-PVD; CP-Ti; CoCrMo; Microhardness; Wear resistance.

Keywords: CA-PVD, CP-Ti, CoCrMo, Microhardness, Wear resistance.

CP-Titanyum ve CoCrMo Alaşımı Üzerine Katodik Ark Biriktirme Yöntemi ile Kaplanmış Filmlerin Tribolojik Özellikleri

Öz

Biyomalzemelerin yük taşıyan implantlarda tribolojik özelliklerinin iyileştirilmesi oldukça önemlidir. Bu çalışmada iki farklı türdeki biyomalzeme üzerine (CP-Ti ve CoCrMo alaşımı) CAPVD yöntemiyle yüzeylere biriktirilen TiN ve ZrN filmlerin kuru aşınma şartlarında, 1N ve 3N'luk yük altında tribolojik özelliklerinin kıyaslanması yapılmıştır. Ayrıca yüzeyde biriktirilen TiN ve ZrN filmlerinin CP-Ti ve CoCrMo malzemesi üzerindeki mikroyapısal ve mekanik özellikleri incelenmiştir. TiN ve ZrN kaplı CP-Ti ve CoCrMo malzemelerinin kristal yapısı, elementel bileşimi ve yüzey morfolojisi sırasıyla XRD, SEM ve SEM-EDS analizleri kullanılarak belirlenmiştir. 1N'luk yük altında yapılan aşınma testi sonrası kuru ortamda yapılan test sonuçlarına göre en düşük sürtünme katsayısı yaklaşık 0,35 değerinde işlemsiz CoCrMo numunesinde görülürken, en yüksek sürtünme katsayısı ise 0,55 değerinde ZrNkaplı CoCrMo numunesinde bulunmuştur. 3N'luk yük altında en düşük aşınma oranı ZrN/CCM numunesinde 0,38x10-6 mm^3/Nm iken, en yüksek aşınma oranı değeri işlemsiz Ti numunesinde 2,10 $\text{x}10^{-6}$ mm/Nm'dir. Sonuç olarak, TiN ve ZrN kaplı CP-Ti ve CoCrMo numunelerin mikrosertlik değerleri ve aşınma direncinin arttığı belirlenmiştir.

Anahtar Kelimeler: CA-PVD, CP-Ti, CoCrMo, Mikrosertlik, Aşınma direnci.

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1.Introduction

CP-Ti (commercially pure Ti) and CoCrMo (cobalt-chromium-molybdenum) alloys used as biomaterials are among the prominent materials in the biomedical field due to their superior mechanical properties, corrosion resistance and biocompatibility. CP-Ti material has become a type of biomaterial mainly used in dental and orthopedic applications due to its high strength-to-weight ratio, excellent corrosion resistance and high biocompatibility with bone tissue. It is also used in applications where wear resistance is required. CoCrMo alloys are widely used in load-bearing biomedical applications such as hip and knee prostheses due to their superior strength [1-3]. Although CP-Ti and CoCrMo alloys are widely preferred in biomaterial applications due to their superior corrosion resistance and biocompatibility properties, their limited tribological performance (especially insufficient wear resistance and undesirable friction behavior) can negatively affect the long-term mechanical stability and implant life of these materials [4,5]. Various surface coating processes are applied to these materials to overcome the negative tribological properties that occur in these materials [6-8].

Physical vapor deposition (PVD) techniques stand out as highly efficient methods for modifying the surface morphology and functional properties without disrupting the macroscopic structural integrity of the substrate [9]. Cathodic arc physical vapor deposition (CAPVD), one of the PVD coating methods, enables the obtainment of high adhesion and tribologically superior coatings under low thermal loading. The CAPVD method enables the deposition of nitride, carbide and carbon-based hard ceramic films, providing a significant increase in surface hardness, reducing the coefficient of friction and greatly improving the resistance of the surface to wear [10,11]. By varying the target materials and reactive gases (e.g., nitrogen) used in the Cathodic Arc PVD process, a wide variety of coatings can be deposited on surfaces, including TiN, ZrN, CrN, TiAlN and DLC (diamond-like carbon) each offering different advantages in reducing wear and friction in materials under different loading conditions [12,13]. Some studies in the literature on the tribological properties of TiN and ZrN coated CP-Ti and CoCrMo materials using PVD methods are given below.

CoCrMo alloy as the substrate was coated with TiN film using PVD method for 6 hours. XRD results showed that PVD coated TiN films exhibited (111) preferred orientation, while SEM analysis showed a very uniform and quite dense TiN coated layer with a columnar growth mode reaching from the substrate to the coating surface. Also, scratch test determined that there was sufficient adhesion strength between TiN film and CoCrMo substrate [14]. In a study investigating the properties of TiN coatings on CoCrMo and Ti6Al4V alloys to increase wear resistance, arc evaporation and high-power pulsed magnetron sputtering (HiPIMS) methods were used. The performance of the coatings was evaluated in standard wear tests against ultra-high molecular weight polyethylene (UHMWPE) in bovine serum lubricant and in the presence of abrasive PMMA bone cement particles in the lubricant. The results indicated that the samples showed significant wear reduction after the coating process [15]. In a study investigating the effect of TiN coating on CoCrMo material, TiN films coated by high-power pulsed magnetron sputtering (HiPIMS) method were optimized in terms of surface roughness and hardness on CoCrMo and ceramic surfaces; CoCrMo samples coated at 200°C and ceramic samples coated at 100°C exhibited the highest hardness and smoothest surface morphology, improving the material mechanical properties [16]. In another study where TaN, ZrN and TaZrN coatings were synthesized on CoCrMo alloy by magnetron sputtering, mechanical, adhesion and wear properties of the coatings were investigated. All coatings showed excellent adhesion properties on CoCrMo alloy. While smaller grains of TaN coating caused lower surface roughness, larger grains and nanopores of ZrN coating contributed to high surface roughness values [17]. In this study, microstructural, mechanical and tribological properties of biomedical materials produced from CP-Ti and CoCrMo alloys were investigated by deposition of TiN and ZrN coatings on their surfaces using the CAPVD method.

2. Material and Methods

In this study, forged CoCrMo alloy (Ø15 mm × 4 mm) and commercially pure-Grade 2 titanium (CP-Ti) (15 × 15 × 4 mm) were used as base materials. Before coating, all samples were sanded with 80, 400, 600, 800 and 1200 grit SiC sandpapers, respectively, and then subjected to mechanical polishing using alumina suspensions with 1 μm and 5 μm particle sizes. The polished samples were cleaned with ethyl alcohol and pure water in an ultrasonic bath for 10 minutes, respectively. TiN and ZrN coatings were made by a local commercial company (Titanit Ultra Hard Coatings Ltd., Istanbul, Türkiye). TiN and ZrN coating films were deposited on a Platit PL-1011 model PVD device. The films of the samples coated at the company are of approximately similar thickness, around 1.5 μm. Before deposition, all samples were exposed to Ar ion bombardment for 20 min to remove impurities from their surfaces. The temperature was kept in the range of 400–430 °C during the deposition process of both coating films. For TiN film deposition, the bias voltage was gradually reduced with time from -120 V to -40 V as the cathode current was set at 70 A and the N_2 pressure was increased from 0.5 Pa to 3 Pa. For ZrN film deposition, the bias voltage was gradually reduced with time from -150 V to -100 V as the cathode current was set at 80 A and the N_2 pressure was increased from 3 Pa to 6 Pa (Table 1).

Table 1. Deposition parameters of coating films

Coatings	Deposition temperature (°C)	Current of target evaporation (A)	Bias voltage (V)	Working pressure (Pa)
TiN	400-430	70	40-120	0.5-3
ZrN	400-430	80	100-150	3-6

For the microstructure analysis of the films deposited on the surface, Panalytical Empyrean-XRD device was used; for the surface morphology of the coated materials and wear marks after the wear test, scanning electron microscope FEI Quanta FEG-450 SEM-EDS device was used and the surface hardness of the samples was determined with a hardness device using Bruker brand UMT-2 model. Wear tests to determine the tribological properties of untreated, TiN and ZrN coated surfaces of CP-Ti and CoCrMo samples were carried out on the Bruker UMT-2 mechanical testing machine. The wear tests were carried out under room conditions, using the linear reciprocating ball-on-flat method under 1N and 3N loads, and for these tests, the samples were abraded with a 6 mm diameter Al_2O_3 (alumina) ball under dry friction conditions. In this study, the abbreviations TiN/Ti for TiN-coated CP-Ti, ZrN/Ti for ZrN-coated CP-Ti, TiN/CCM for TiN-coated CoCrMo and ZrN/CCM for ZrN-coated CoCrMo were used to define the coated samples, respectively.

3. Results and Disscussion

3.1. SEM, XRD and EDS analysis

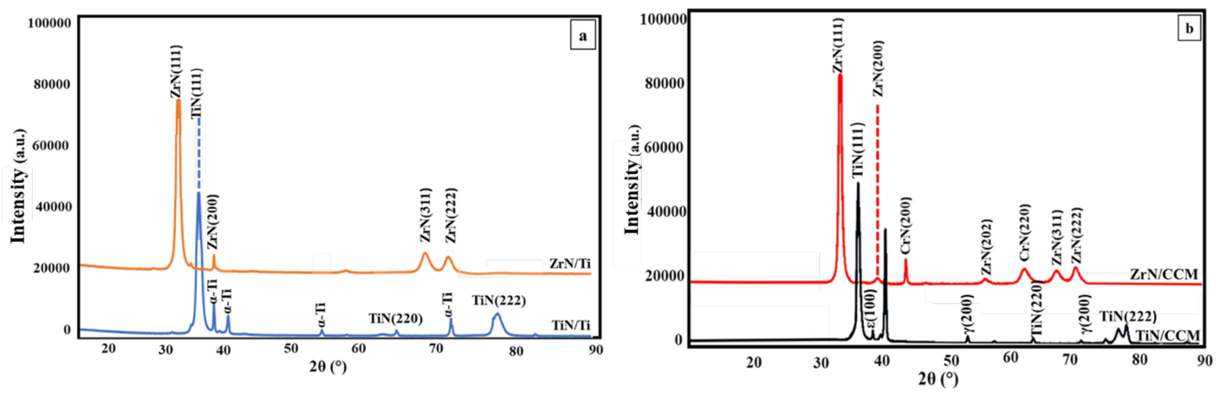

Fig. 1. XRD graphs of a) TiN/Ti and ZrN/Ti, b) TiN/CCM and ZrN/CCM samples

According to the XRD analysis in Fig.1(a), the dominant phase in the TiN-coated CP-Ti sample is TiN, and it shows a distinct orientation especially in the (111) plane. TiN (220), TiN (222) and α-Ti phases belonging to the base material were also detected in this sample. In the ZrN-coated CP-Ti sample, the ZrN phase exhibited (111), (200), (311) and (222) orientations; it was determined that the dominant phase was ZrN (111) and this orientation was compatible with the ICDD 35-0753 standard. Fig.1(b) shows the XRD patterns of the TiN/CCM and ZrN/CCM samples. In the XRD analysis of the CoCrMo sample obtained after the TiN coating process, the (111), (220) and (222) planes of the TiN phases belonging to the coating, as well as the ε (100) and γ (200) phases originating from the base alloy, were detected. In the ZrN-coated CoCrMo sample, the XRD results show that the ZrN phase exhibits crystallographic orientations in the (111), (200), (202), (311) and (222) planes; in addition, the CrN (200) and CrN (220) phases were also formed [18,19].

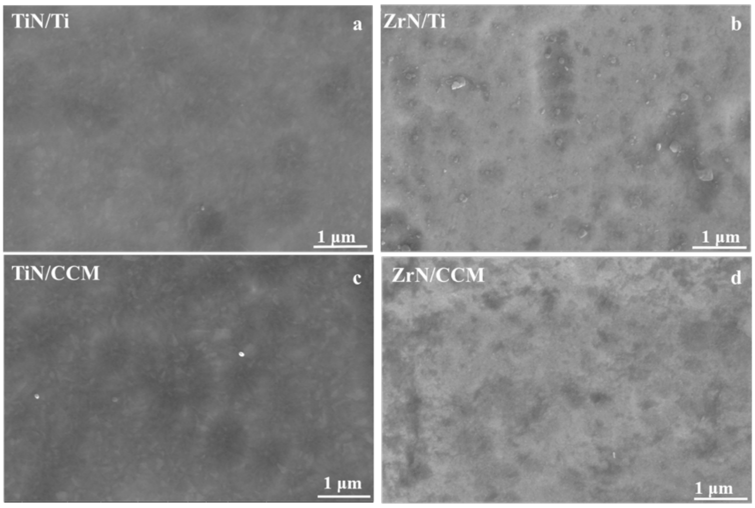

Fig. 2. Surface SEM images of a) TiN/Ti and ZrN/Ti, b) TiN/CCM and ZrN/CCM samples

SEM images of the surface topography of TiN and ZrN coated CP-Ti and CoCrMo samples are shown in Fig. 2. As can be seen, the morphological structure of the surfaces changed and the surface roughness increased with the CA-PVD process on both TiN coated and ZrN coated surfaces. It was observed that the surface was coated homogeneously in the TiN/Ti coating presented in Fig. 2(a). In Fig. 2(b) and 2(d), micro droplet formations, which are commonly encountered in coatings performed with the CA-PVD method, were detected in the ZrN coated samples. It is evaluated that these droplets are caused by Zr particles that break away from the Zr cathode during coating and reach the surface without sufficient interaction with the nitrogen gas. In addition, it was determined that as a result of these droplets hitting and holding on to the base surface, void or pit structures were formed in the lower regions of the droplets due to the shadowing effect of the ion flow [20]. The average vickers microhardness of the untreated CP-Ti sample was 150-200 HV, while this value increased to approximately 1750-1800 HV after TiN coating and 1800 HV after ZrN coating. This increase shows that hard ceramic coatings significantly improve the surface hardness and increase the mechanical strength of the surface. Similarly, the microhardness value of the untreated CCM sample was 420-470 HV, while this value reached 1850-1900 HV with TiN coating and 1950-2000 HV with ZrN coating. These results show that TiN and ZrN hard coatings applied on both CP-Ti and CoCrMo substrates provide approximately 4-5 times increase in surface hardness. Additionally, it was observed that ZrN coatings provided higher hardness compared to TiN coatings, which may be related to the denser crystal structure and harder phase properties of ZrN.

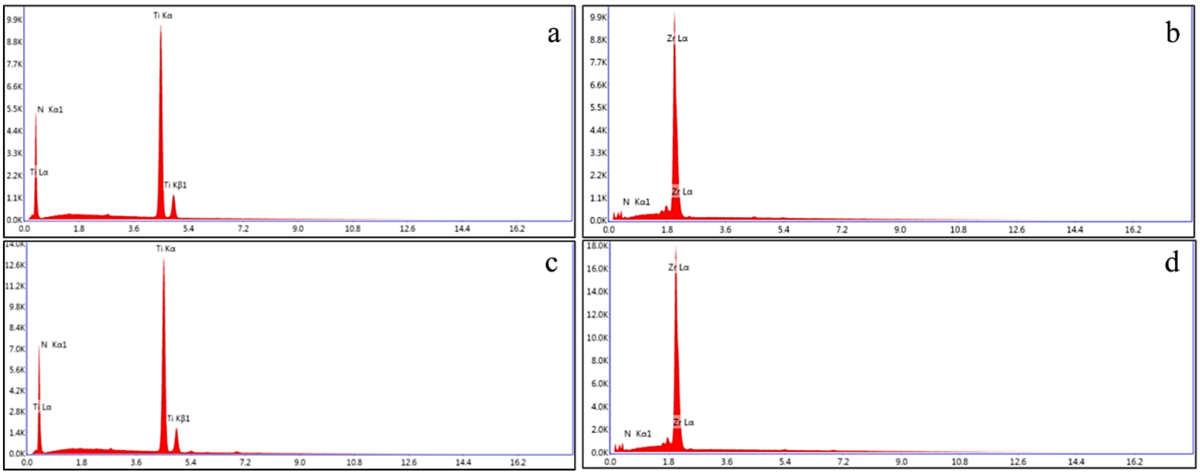

Fig. 3. EDS analysis result graphs of a) TiN/Ti and ZrN/Ti, b) TiN/CCM and ZrN/CCM samples

The surface chemical composition of TiN and ZrN coatings was analyzed by energy dispersive X-ray spectroscopy (EDS). EDS spectra of TiN and ZrN films coated on CP-Ti and CoCrMo substrates are presented in Fig.3 and Table 2. In TiN films coated on CP-Ti, the atomic ratios of the coating layer were measured as 53.22% Ti and 46.78% N. In the ZrN coated CP-Ti sample, these ratios were 39.09% Zr and 60.91% N. EDS results of the films coated on CoCrMo substrate, the atomic ratios were determined as 53.29% Ti and 46.71% N in the TiN coating and 39.4% Zr and 60.6% N in the ZrN coating (Table 2).

Table 2. Deposition parameters of coating films

Parameters	Element	Weight %	Atomic %
TiN /Ti	N Ti	20.44 79.56	46.78 53.22
ZrN/Ti	N Zr	19.31 80.69	60.91 39.09
TiN/CCM	N Ti	20.40 79.60	46.71 53.29
ZrN/CCM	N Zr	19.10 80.90	60.6 39.4

3.2. Tribological Test Results

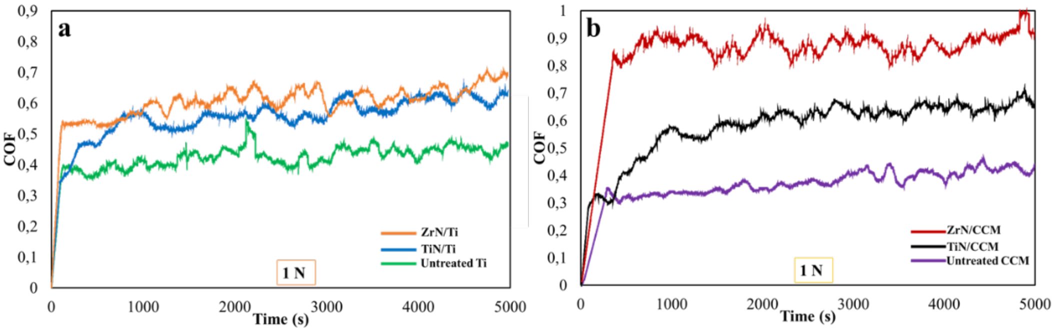

Fig. 4. Friction coefficient-time graphs after wear tests under 1N load,

The data obtained after the wear tests of untreated, TiN and ZrN coated CP-Ti and CoCrMo samples under 1N and 3N loads in a dry environment are given in Table 2.1. The coefficient of friction (COF)-time graphs obtained after the wear tests under 1N load are shown in Figure 4. While the average friction coefficient of the untreated CP-Ti sample under 1N load is 0.45 in Fig. 4 (a), the friction coefficient values increased due to the effect of the TiN and ZrN films obtained after the CAPVD coating process. According to the test results of the untreated and TiN and ZrN coated CoCrMo alloy under 1N load in a dry environment in Fig. 4 (b), the lowest friction coefficient is seen in the untreated CCM sample with a value of approximately 0.35, while the highest friction coefficient is seen in the ZrN/CCM sample with a value of 0.90. One of the most important factors affecting the coefficient of friction is the surface roughness [21]. The coefficient of friction values obtained from TiN coated samples are 0.60 in the TiN/Ti sample and 0.57 in the TiN/CCM sample.

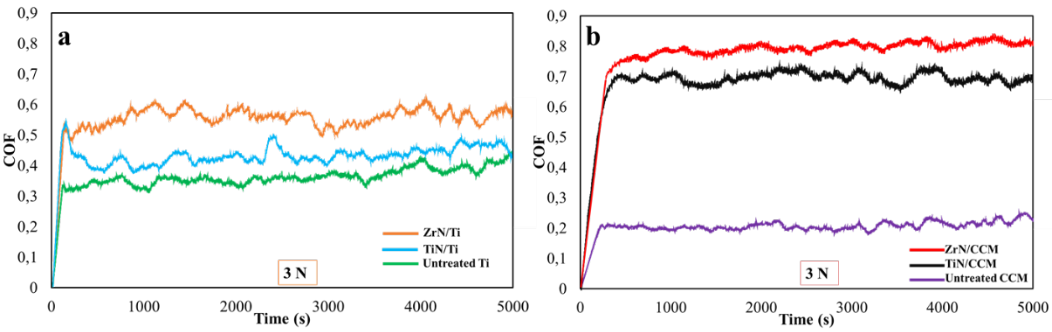

Fig. 5. Friction coefficient-time graphs after wear tests under 3N load.

The friction coefficient-time graphs obtained after the wear tests performed under 3N load are shown in Fig.5. According to the values obtained under 3N wear load in Figure 5, the values were found to be 0.36 for the untreated Ti sample, 0.42 for the TiN/Ti sample and 0.55 for the ZrN/Ti sample. As a result of the experiments performed under the same wear load, the increase in the COF values obtained from the TiN and ZrN coated samples increased depending on the increase in surface roughness. Since the highest surface roughness values were obtained from the ZrN coated samples, the highest wear rates were also found from these samples. As seen in the graphs after the wear test performed under 3N wear load shown in Fig. 5, the lowest friction coefficient value (0.21) was obtained from the untreated CCM sample, while the highest friction coefficient (0.86) was measured from the ZrN/CCM sample.

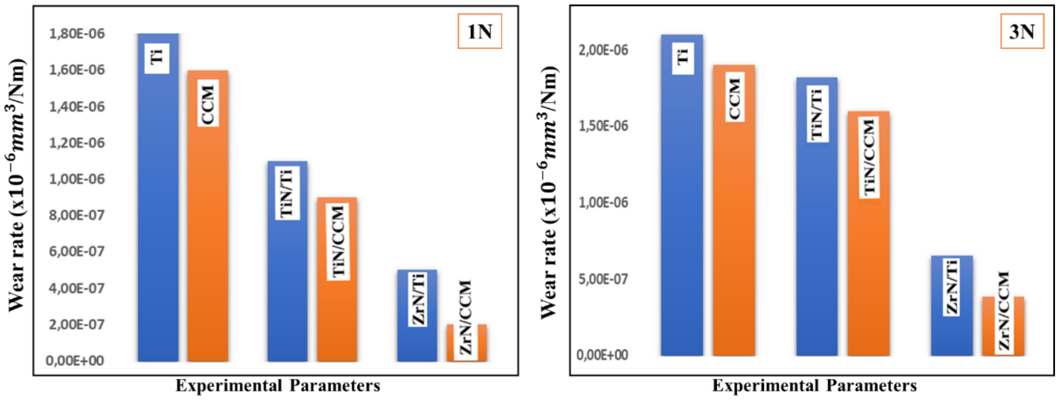

Fig. 6. Wear rate graphs of untreated, TiN and ZrN coated samples obtained under 1N and 3N loads

Table 3. Wear test results of untreated, TiN and ZrN coated samples obtained under different wear loads

Parameters	Average Coefficient of Friction (μ)		Average Wear rate (x10^{-6}mm^3/Nm)	
	1N	3N	1N	3N
Untreated Ti	0,45	0,36	1,85	2,10
TiN/Ti	0,60	0,42	1,10	1,80
ZrN/Ti	0,62	0,55	0,50	0,65
Untreated CCM	0,35	0,21	1,60	1,90
TiN/CCM	0,57	0,67	0,90	1,60
ZrN/CCM	0,90	0,86	0,52	0,38

Fig. 6 shows the wear rates obtained after the wear tests of untreated, TiN and ZrN coated samples under 1N and 3N wear test loads. In addition, the wear rate values calculated for each sample are presented in Table 3. While the highest wear rate under 1N load was 1.85 x10^{-6} mm^3/Nm in the untreated Ti sample, the lowest wear rate was calculated as 0.50x10^{-6} mm^3/Nm in the ZrN/Ti sample. Under 1N load, the wear rate in the CP-Ti sample after TiN coating is approximately 41% lower than the untreated CP-Ti sample. While the wear rate value of the untreated CCM sample under the same load was 1.60x10^{-6} mm^3/Nm, this value was determined as 0.52x10^{-6} mm^3/Nm after ZrN coating. After TiN coating, the wear rate value under 1N load in CoCrMo alloy is 44% lower compared to the untreated CCM sample. It was determined that the wear resistance of TiN coated samples under 1N load is higher in CoCrMo sample compared to CP-Ti sample. From this, it was concluded that TiN film is more resistant to wear in CoCrMo alloy. Since the values obtained in both ZrN coated samples under 1N load are close to each other, it can be said that ZrN film shows similar wear rate in both samples. It was found that the wear resistance of ZrN coated samples under 1N load increased compared to untreated samples. While the lowest wear rate under 3N load was 0.38x10^{-6} mm^3/Nm in ZrN/CCM sample, the highest wear rate value was found as 2.10x10^{-6} mm/Nm in untreated Ti sample. Under a load of 3N, the wear rate of the CP-Ti sample after ZrN coating is approximately 69% lower than that of the untreated CP-Ti sample. Under the same load, the wear rate value of the untreated CCM sample is 1.90x10^{-6} mm^3/Nm. Under a load of 3N, the wear rate of the TiN-coated CoCrMo sample is 16% lower than that of the untreated sample, while this rate reached approximately 80% in the ZrN-coated sample. According to the results of the wear test conducted under a load of 3N, it was concluded that the ZrN film was more effective in wear resistance than the TiN film in samples with CoCrMo as the base material. The reduced wear rates observed in the ZrN-coated materials under both loading conditions can be attributed to the formation of protective oxide layers on the surfaces.

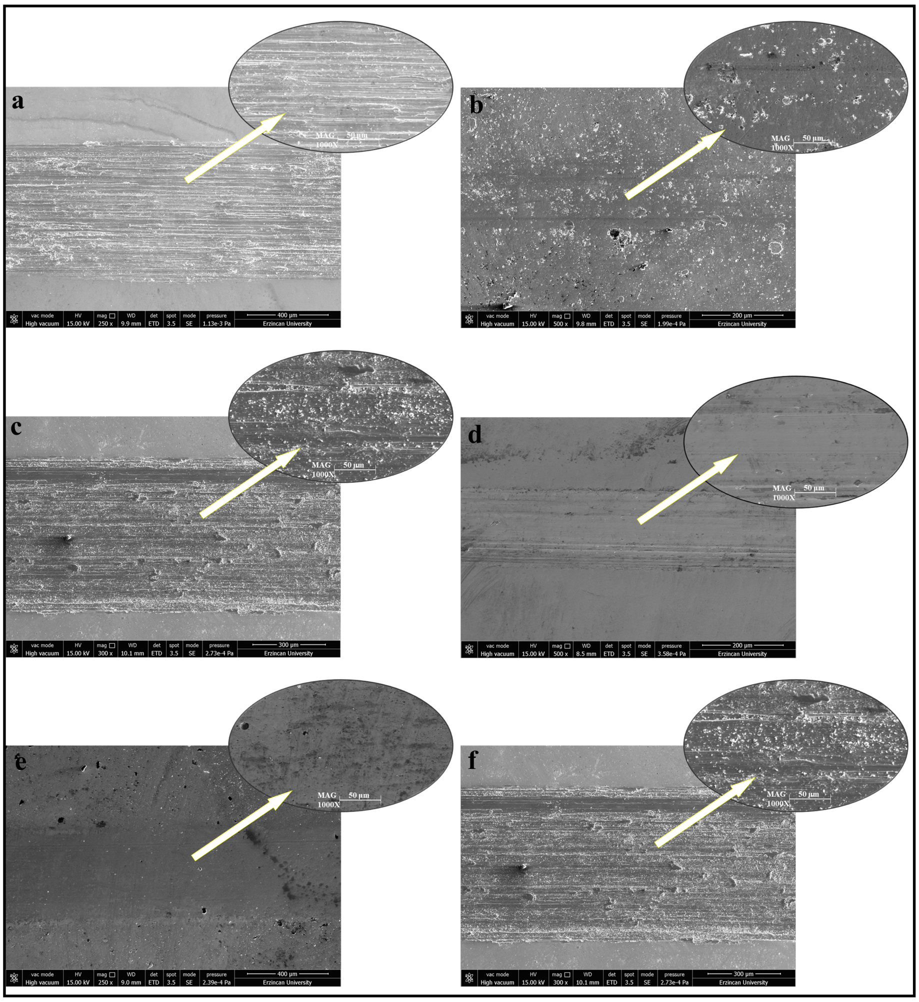

Fig. 7. SEM images obtained after wear under 1N load; a) Untreated Ti, b) TiN/Ti, c) ZrN/Ti, d) Untreated CCM, e) TiN/CCM, f) ZrN/CCM

Fig. 7 shows SEM images of wear marks after wear tests performed in dry conditions using alumina (Al_2O_3) balls under 1N load. In SEM analyses after wear, the common wear type in untreated CP-Ti and untreated CoCrMo materials under 1N load is abrasive wear. Since the hardness of the alumina ball used during the wear test (approximately 2000 HV) is very high, it causes scratching and micro material removal effects on the material surface even at low load, causing abrasive wear type [22]. Plate-like wear products formed in the wear mark of CP-Ti material after TiN coating under 1N load indicate adhesive wear. It is observed that micro particles that break off during wear in ZrN/Ti material create thin scratches in the wear mark. It was observed that the abrasive wear determined in the ZrN coated CP-Ti material was determined by the microscopic particles separated during the tribological interactions, causing microscopic fine scratches in the morphology of the wear trace. It is seen in Figure 7 (e,f) that under 1N load, the adhesive wear mechanism is dominant in the TiN coated CoCrMo material and the abrasive wear mechanism is dominant in the ZrN coated CoCrMo sample.

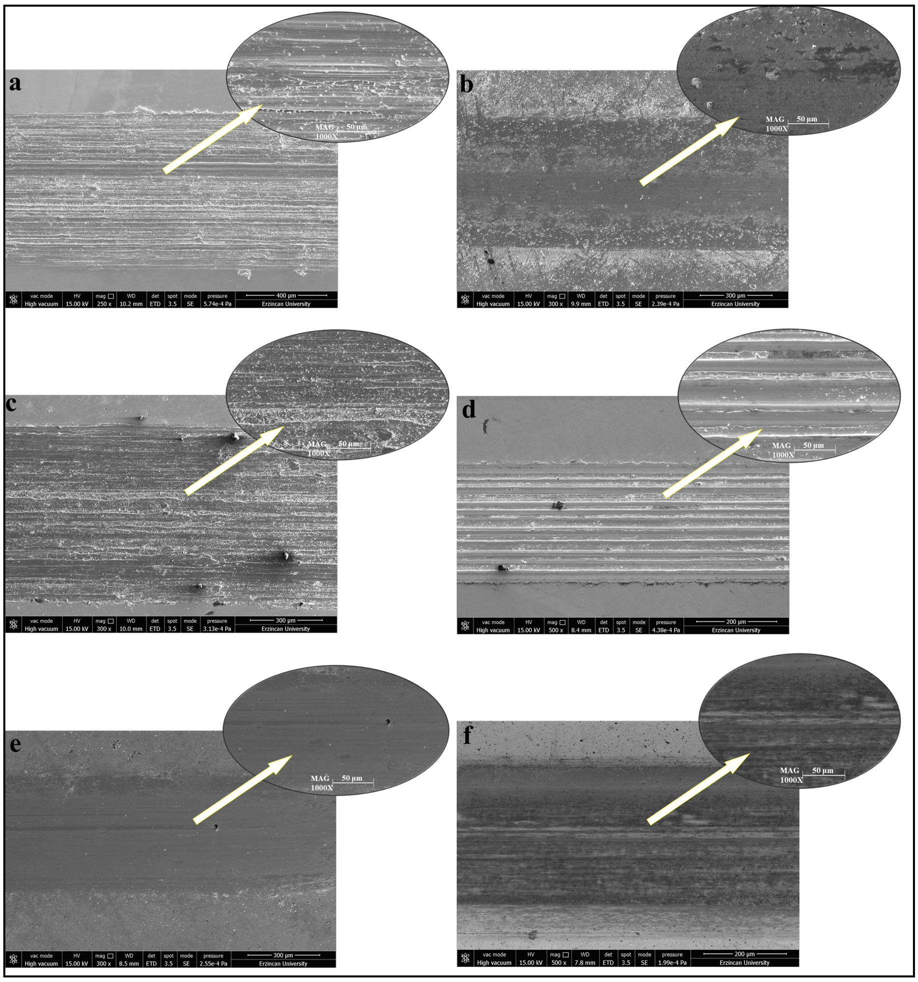

Fig. 8. SEM images obtained after wear under 3N load; a) Untreated Ti, b) TiN/Ti, c) ZrN/Ti, d) Untreated CCM, e) TiN/CCM, f) ZrN/CCM

Fig. 8 shows the SEM images of the wear marks after the wear tests performed in dry conditions using alumina (Al_2O_3) balls under a 3N load. In untreated CP-Ti and CoCrMo samples, it is clearly seen that the type of wear is abrasive wear with increasing load in Fig.8 (a,d). The reason for this is that with the increase in the applied load during the wear test, it was determined that the particles separated at a higher rate activate the abrasive mechanism on the wear surface. It was determined that the type of wear in TiN coated CP-Ti and CoCrMo materials is similarly adhesive wear. In addition, it is more clearly seen that abrasive wear occurs in ZrN coated samples compared to the wear marks formed under a 1N load. In the wear tests performed using alumina (Al_2O_3) balls under a 3N load, it was determined that the wear mark widths increased compared to the wear mark width formed under a 1N load, similar to the studies conducted in the literature [23].

4. Conclusion

In this study, XRD, SEM, EDS, microhardness and wear tests of TiN and ZrN and coated films were performed on CP-Ti and CoCrMo materials, which are frequently used as biomaterials, with CA-PVD technique under two different loads in dry conditions. The results are as follows:

- For the XRD results, TiN was identified as the dominant phase, particularly oriented along the (111) plane in TiN/Ti sample. The ZrN/Ti sample exhibited prominent ZrN peaks with (111), (200), (311), and (222) orientations. In the TiN/CCM alloy, the (111), (220), and (222) planes of TiN were detected, alongside ε (100) and γ (200) phases from the base alloy. For the ZrN/CCM sample, XRD patterns revealed ZrN crystallographic orientations in the (111), (200), (202), (311), and (222) planes. TiN and ZrN films coated with CA-PVD method showed significant changes in surface morphology and increased roughness. While a homogeneous surface was obtained in TiN coatings, micro droplet formations were observed in ZrN coatings.
- As a result of the wear tests, uncoated samples showed lower friction coefficients, while these values increased with TiN and ZrN coatings. Under 1N load, the lowest friction coefficient was observed in the uncoated CoCrMo sample with ~0.35, while the highest value was measured in the TiN/Ti sample with 0.60. In the wear tests performed under 3N load, the friction coefficient increased after coating. The lowest value was measured as 0.21 in the uncoated CoCrMo sample, and the highest value was measured as 0.86 in the ZrN coated CoCrMo sample.
- In the SEM analysis after the wear tests performed under 1N and 3N loads, the dominant wear type in untreated samples was abrasive wear. Adhesive wear was observed in TiN coated samples and abrasive wear type was determined in ZrN coated samples. Especially in ZrN coatings, microparticles broken off by tribological interactions created fine scratches.

Ethics in Publishing

There are no ethical issues regarding the publication of this study.

Author Contributions

Mevra ASLAN ÇAKIR: Writing – review & editing, Writing – original draft, Project administration, Methodology, Investigation, Resources.

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