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Investigation of wear, surface, metallurgical and mechanical properties of Ti6Al4V alloys coated with PVD method

PVD yöntemi ile kaplanan Ti6Al4V alaşımlarının aşınma, yüzey, metalurjik ve mekanik özelliklerinin incelenmesi

***Yazar(lar) (Author(s))**: Hakan ADA[1], Ahmed Qays Jabbar EL RUBAYE[2], Elif AŞIKUZUN TOKEŞER[3], Ahmet MAVİ[4], Yavuz KAPLAN[5], Sinan AKSÖZ[6],*

ORCID[1]: 0000-0002-0412-5789

ORCID[2]: 0000-0003-3588-8268

ORCID[3]: 0000-0003-1850-7080

ORCID[4]: 0000-0003-0339-2639

ORCID[5]: 0000-0002-3144-9332

ORCID[6]: 0000-0003-4324-5043

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Investigation of Wear, Surface, Metallurgical and Mechanical Properties of Ti6Al4V Alloys Coated with PVD Method

Highlights

- *Cylindrical Ti6Al4V specimens coated by PVD method were produced.*
- *Ti6Al4V alloys were coated with AlCrN, TiAlN, TiN, TiSiN coatings*
- *Profilometer, microhardness, and wear tests were applied to the coated specimens.*

Graphical Abstract

This study applied TiAlN, AlCrN, TiSiN and TiN coating processes to the samples using Ti6Al4V alloy as the base material by the PVD method with two different parameters (temperature and time).

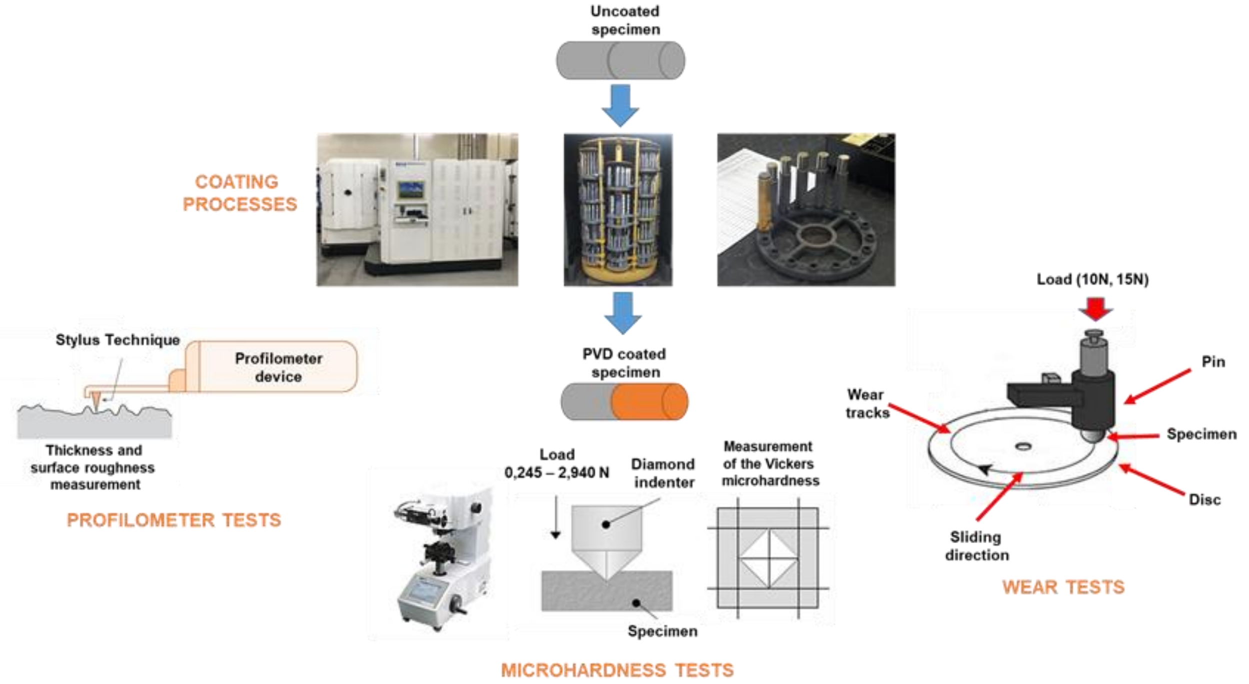

Figure. Coating and test processes of the specimens

Aim

It is aimed to improve the microhardness and wear resistance of Ti6Al4V alloys coated with the PVD method.

Design & Methodology

Profilometer, microhardness, and wear tests were applied to Ti6Al4V coatings produced by the PVD method.

Originality

This study was carried out because it was realised that there was a need for more publications on PVD coating processes where different coating types are applied at different temperatures and times.

Findings

Thin-coated specimens gave better results than thick-coated specimens.

Conclusion

TiAlN coating had the best performance in microhardness and wear tests.

Declaration of Ethical Standards

The author(s) of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

Investigation of Wear, Surface, Metallurgical and Mechanical Properties of Ti6Al4V Alloys Coated with PVD Method

Araştırma Makalesi / Research Article

Hakan ADA[1,2*], Ahmed Qays Jabbar EL RUBAYE[3], Elif AŞIKUZUN TOKEŞER[4], Ahmet MAVİ[5], Yavuz KAPLAN[6], Sinan AKSÖZ[7]

[1]Teknoloji Fakültesi, Metalurji ve Malzeme Mühendisliği Bölümü, Gazi Üniversitesi, Türkiye
[2]Mühendislik ve Mimarlık Fakültesi, Makine Mühendisliği Bölümü, Kastamonu Üniversitesi, Türkiye
[3]Fen Bilimleri Enstitüsü Makine Mühendisliği ABD, Kastamonu Üniversitesi, Türkiye
[4]Mühendislik ve Mimarlık Fakültesi, Metalurji ve Malzeme Mühendisliği Bölümü, Kastamonu Üniversitesi, Türkiye
[5]Teknoloji Fakültesi, İmalat Mühendisliği Bölümü, Gazi Üniversitesi, Türkiye
[6]Teknoloji Fakültesi, İmalat Mühendisliği Bölümü, Pamukkale Üniversitesi, Türkiye
[7]Teknoloji Fakültesi, Metalurji ve Malzeme Mühendisliği Bölümü, Pamukkale Üniversitesi, Türkiye

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ABSTRACT

In this study, the PVD method coated cylindrical specimens produced from Ti6Al4V alloy with AlCrN, TiAlN, TiN, TiSiN coatings at different thicknesses. Profilometer tests were applied to determine the surface properties, such as coating thickness and surface roughness of the coated specimens, and microhardness and wear tests were applied to the specimens to determine the mechanical properties. In the profilometer measurements, the coating thicknesses were measured between 1-5 µm and the surface roughness values between 0.4 and 0.8 µm. From the microhardness and wear results, it was determined that thin-coated specimens gave better results than thick-coated specimens. While TiAlN had the best performance in microhardness and wear tests, this coating was followed by TiSiN, TiN and AlCrN coatings, respectively.

Keywords: Ti6Al4V, PVD Coating, Surface Treatments, Microhardness, Wear.

PVD Yöntemi ile Kaplanan Ti6Al4V Alaşımlarının Aşınma, Yüzey, Metalurjik ve Mekanik Özelliklerinin İncelenmesi

ÖZ

Bu çalışmada, Ti6Al4V alaşımından üretilen numuneler PVD yöntemi ile farklı kalınlıklarda AlCrN, TiAlN, TiN, TiSiN kaplamalarla kaplanmıştır. Kaplanan numunelerin kaplama kalınlığı ve yüzey pürüzlülüğü gibi yüzey özelliklerini belirlemek için profilometre testleri, mekanik özelliklerini belirlemek için ise numunelere mikrosertlik ve aşınma testleri uygulanmıştır. Profilometre ölçümlerinde kaplama kalınlıkları 1-5 µm arasında, yüzey pürüzlülük değerleri ise 0,4 ile 0,8 µm arasında ölçülmüştür. Mikrosertlik ve aşınma sonuçlarından, ince kaplanmış numunelerin kalın kaplanmış numunelere göre daha iyi sonuçlar verdiği, en iyi performansı TiAlN kaplamanın gösterdiği, bu kaplamayı sırasıyla TiSiN, TiN ve AlCrN kaplamaların takip ettiği tespit edilmiştir.

Anahtar Kelimeler: Ti6Al4V, PVD Kaplama, Yüzey İşlemleri, Mikrosertlik, Aşınma.

1. INTRODUCTION

Titanium alloys are widely used in the aerospace, automotive, petrochemical, and biomedical industries due to their low density, low modulus of elasticity, high specific strength/weight ratio, excellent corrosion resistance, high-temperature resistance, and biocompatibility [1-7]. Ti6Al4V alloy, more commonly known as Ti64, is one of the titanium alloys containing mainly α phase and a small amount of β phase (α + β), which attracts great attention due to the high strength, low density, favourable corrosion resistance and biocompatibility properties of titanium alloys [5-13]. Ti6Al4V alloy is one of the most widely used titanium alloys, commonly used in the aerospace industry to produce aircraft components such as landing gear, turbine parts, beams, etc., and implant materials [9-12]. However, Ti alloys also have some disadvantages. These materials are expensive, exhibit poor tribological properties, and have low wear resistance [9-17].

Due to the high cost of titanium alloys, the feature-enhancing technologies and application areas applied to the components of the material are increasing day by day

**Sorumlu Yazar (Corresponding Author)*
e-posta : hakanada@gazi.edu.tr

[9]. In practical applications, coating technology has been widely used in recent years to improve the surface hardness and wear resistance of materials produced from the Ti6Al4V alloy. Many technologies such as nitriding, carburising, oxidation, physical vapour deposition (PVD), chemical vapour deposition (CVD), and ion implantation are some surface treatment techniques developed to eliminate the mentioned disadvantageous situations [5,18]. The PVD method has been favoured for producing hard coatings since the 1980s [19]. PVD coatings can be produced at high temperatures in various properties, ranging from very soft coatings [20] to tough and fatigue-resistant coatings [21,22]. However, besides its high hardness, high elastic modulus, high breaking strength, good fatigue strength, good oxidation, thermal resistance, and pleasing appearance, PVD coating exhibits superior tribological properties thanks to its high wear resistance and low friction coefficient [23-31]. PVD coatings are widely used in instruments and bearings due to their high hardness, low coefficient of friction, and low thermal resistance, on blades of gas turbines or steam turbines on their excellent oxidation and thermal resistance, and as decorative coatings for their pleasing appearance [32].

Many studies are in the literature on coating Ti6Al4V alloy with the PVD methods and examining its wear properties. Still, the need for more publications on PVD coating processes that apply different coating types at different temperatures and durations has attracted attention. Therefore, in this study, TiAlN, AlCrN, TiSiN and TiN coating processes were applied to the samples using Ti6Al4V alloy as the base material by PVD method with two different parameters (temperature and time). Microhardness tests were applied to determine the mechanical properties of the specimens and abrasion tests were applied to determine the tribological properties. Energy Dispersion Spectrometer (EDS) and Field Emission Scanning Electron Microscope (FESEM) analyses were performed on the worn specimens in metallurgical investigations. Profilometer analysis was also performed to determine the specimens' coating thickness and surface roughness. During the study, the wear rate, as well as the mechanical and metallurgical properties of the surfaces, which were formed by different coating processes (TiAlN, AlCrN, TiSiN, and TiN) applied to Ti6Al4V alloys, were examined in detail and this study also attempted to determine the effects of different coatings on the alloy.

2. MATERIAL and METHOD

TiAlN, AlCrN, TiSiN, and TiN coating processes were applied to the specimens, in which Ti6Al4V alloy was used as the base material at two different parameters (450/500 ºC temperature, duration of 240/360 min.) by the PVD method. Typical processing temperatures for PVD coatings are between 250 and 450 °C, sometimes up to 600 °C. Coatings can be deposited in single, multiple, and staggered layers. Coating thickness generally ranges from 2 to 5 µm, but it can be as thin as a few hundred nanometers or as thick as 15 µm. The parameters in this study were based on data from various studies. [33-36]. The chemical composition of the Ti6Al4V alloy is given in Table 1. Specimens with dimensions of Ø12x30 mm were preferred for the coating processes.

Table 1. The chemical composition of the Ti6Al4V alloy.

Elements	Al	V	C	Fe	O	N	H	Ti
Percent by Weight	5,5-6,75	3,5-4,5	<0,1	<0,3	<0,2	<0,05	<0,015	Remaining

PVD coatings (AlCrN, TiAlN, TiSiN, and TiN) were applied on the Ti6Al4V base material, which was determined as the substrate using the cathodic arc method. Coating processes were carried out on the ISYS i90A brand and model device in the Inan Coating company (Figure 1.a). An 8-stage ultrasonic cleaning process was performed before the specimens were coated. Before the PVD coating processes, the specimens were placed in the coating boxes, as shown in Figures 1b and c, and then in the coating device.

Two different coating thicknesses were created at different temperatures and durations for each coating under 0.5 MPa pressure. Coating processes were carried out for 240 minutes and 360 minutes at 450ºC for TiAlN and TiN coatings and at 500ºC for AlCrN and TiSiN coatings. The images of the specimens after the coating process are given in Figure 2.

The thickness and surface roughness of the produced coatings were measured using a Veeco Dektak 150 profilometer device. Contact Stylus profilometry techniques were applied to measure the thickness and surface roughness. The Stylus profilometry technology and method, the accepted standard for surface topography measurements and for measuring roughness and step size, is to trace the topography of a film-substrate step. The Vickers method was applied in the microhardness analysis using the Shimadzu brand and HMV model microhardness device. The results in the Vickers hardness test were obtained by measuring the diagonal lengths created by immersing the penetrating tip on the surface of the material for 10-15 seconds under 0.245 N, 0.490 N, 0.980 N, 1.960 N and 2.940 N loads selected according to the type of material. The wear test was performed on the pin-on-disc wear test device by the ASTM G99 standard. The disc made of AISI 52100 material with a hardness of 60 HRC was used as an abrasive. The abrasion tests were carried out at room temperature under 10 N and 15 N loads for 60 minutes at a speed of 1.2 m/s.

Figure 1. a) PVD coating machine (ISYS Intelligent System I 90A), b), c) The specimens were placed in the coating boxes

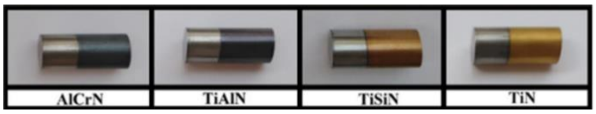


Figure 2. The images of the specimens after the coating process

3. RESULT AND DISCUSSIONS

3.1 Coating Morphology

The thickness and surface roughness values obtained from the profilometer measurements after the coating process have been given in Table 2.

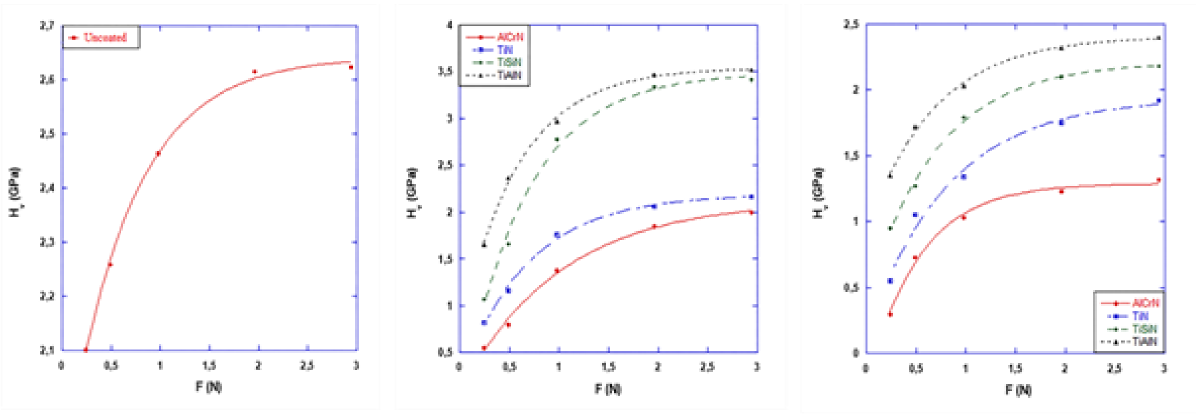
Table 2. The thickness and surface roughness values obtained from the profilometer measurements

Spec. No	Coating type	Temp. of coating (ºC)	Time (Minute)	Thickness (µm)	Surface roughness (µm)
1	TiAlN (thin coated)	450	240	1.46	0.40
2	TiAlN (thick coated)	450	360	2.21	0.89
3	AlCrN (thin coated)	500	240	0.93	0.38
4	AlCrN (thick coated)	500	360	2.81	0.45
5	TiSiN (thin coated)	500	240	1.41	0.25
6	TiSiN (thick coated)	500	360	2.31	0.32
7	TiN (thin coated)	450	240	2.14	0.85
8	TiN (thick coated)	450	360	5.10	0.89

It has been determined from the measurements that the coating thicknesses varied between 1 and 5µm. While the thickness values of the thin-coated specimens varied between 1 and 2 µm, the thickness values of the thick-coated specimens varied between 2-5 µm. It was determined that the densest and thickest film group among the coatings was TiN (Specimens 7 and 8), while the thinnest film thickness was determined to occur in the AlCrN (Specimen No. 3) coating in the thin coatings, and in the TiAlN (Specimen No. 2) coating in the thick coatings. When thin and thick specimens were evaluated together, it was determined that the surface roughness ranged between 0.4 and 0.9 µm. While the coating type with the highest surface roughness was the TiN and TiAlN coatings, the lowest surface roughness value was measured in the TiSiN coating type. It was determined that there was a slight increase in the roughness values with the increase of the coating thickness in all coating types. However, it was observed that there was a severe increase in the surface roughness with the increase of coating thickness in the TiAlN-coated specimens. This situation is thought to occur because the PVD effect is more effective in TiAlN cathode, depending on the temperature and time. The highest surface roughness values were measured on TiN, TiAlN, AlCrN and TiSiN coatings, respectively. When we ordered the coating thicknesses from the highest to the lowest, respectively, it was observed that the ordering was comprised of TiN, TiSiN, TiAlN, and AlCrN coatings in the thin coatings group. In contrast, the TiN, AlCrN, TiSiN, and TiAlN coatings ranged from the highest to lowest in the thick coatings group.

3.2 Microhardness Profile

Microhardness test results are provided in Figure 3 by separately evaluating thin coatings (specimens 1,3,5 and 7) and thick coatings (specimens 2, 4, 6, and 8). In the literature, two different types of behaviour are mentioned when interpreting the microhardness values of materials. The first effect is the Indentation Size Effect (ISE). According to this, When the load is applied to the material's surface, the specimen's microhardness value decreases [37, 38]. The elastic and plastic deformation is observed in these specimens. The other effect is the Reverse Indentation Size Effect (RISE). If the specimen exhibits RISE behaviour, the material shows an increasing microhardness value against the applied load. Additionally, these materials exhibit only plastic



a b c

Figure 3. Microhardness test results a) Base material b) Thin coated specimens c) Thick coated specimens

deformation. Elastic deformation is not observed or is very small compared to plastic deformation.

In the study, the microhardness values increased with the increase of applied load. That is, the material exhibited RISE behaviour. Moreover, the microhardness values of all specimens did not change after approximately $2N$. This region where the hardness doesn't change much is called the "saturation or plateau region" [39]. In other words, in the study, there was no significant change observed in the microhardness of the material at loads greater than $2N$. When the coating thicknesses were considered, the microhardness values of the thicker specimens were smaller. This decrease is related to the state and irregularities of the impurity phases. These factors weaken the strong bonds, resulting in the reduction of microhardness [40]. In addition, this decrease in the Hv value causes an increase in the interparticle resistivity, a decrease in the interparticle transmission surface, and a weakening at the grain boundaries. Here, it was observed that the material behaviour had not changed and that only the microhardness values had changed with the coating process.

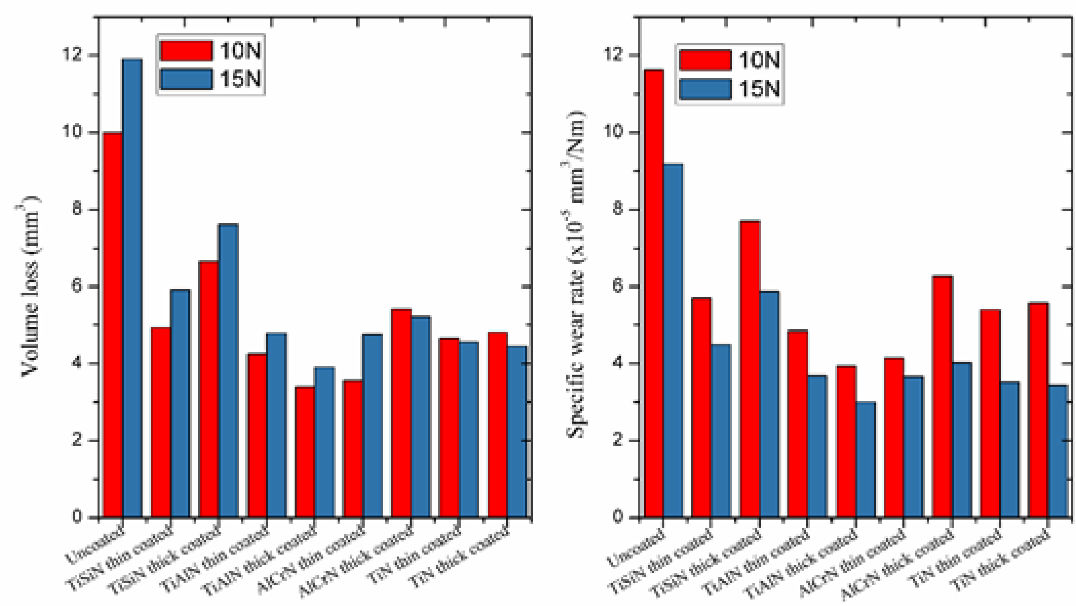


Figure 4. Volume loss and specific wear rate of coated Ti6Al4V

The results showed that the hardness values of the thin specimens were higher than those of the thick specimens. While the highest hardness values were measured in TiAlN coated specimens in both coating thicknesses, TiSiN, TiN, and AlCrN coatings followed in hardness values, respectively. It was also observed that the hardness values of all specimens increased with the increase of applied load.

3.3 Tribological properties of coated Ti6Al4V

Figure 4 shows volume loss and specific wear rate (SWR) graphs. The lowest volume loss and SWR were obtained with the thick-coated TiAlN specimen in all wear test conditions. Therefore, the thick-coated TiAlN specimen has the highest wear resistance. The uncoated Ti specimen obtained the highest volume loss, wear rate, and lowest wear resistance. Thin and thick-coated TiSiN specimens exhibited the worst wear performance among the coated Ti alloys. Although the volume loss is slightly higher at 15N test load than at 10N, except for uncoated Ti, there is no significant difference. It can be said that all applied coatings protect the Ti alloy against wear in both loads. However, SWR values are higher at 10N than at 15N. The SWR depends on mass, density, load and sliding distance. It is preferred for accurately describing the wear properties of metals, alloys, and composites. It is used as a precise indicator of the wear properties of materials under different loads, speeds and sliding distances. The higher SWR obtained at a lower load is due to the increase in volume loss with a 50% increase in applied load. In other words, the applied load increased 50% from 10N to 15N, but volume loss increased much less. In addition, the thick-coated TiAlN specimen showed the best wear performance in SWR results, similar to volume loss results. Similar studies determined that higher performances were obtained from TiAlN coatings applied to different materials [41-43].

The friction the average friction coefficient values are given in Table 3 and the coefficient graphs obtained during the wear tests of the coated Ti6Al4V specimens are given in Figure 5.

Table 3. Average friction coefficient (CoF) values

Spec. No	Coating type	Average of CoF
1	TiAlN (thin coated)	0.178
2	TiAlN (thick coated)	0.165
3	AlCrN (thin coated)	0.250
4	AlCrN (thick coated)	0.190
5	TiSiN (thin coated)	0.210
6	TiSiN (thick coated)	0.190
7	TiN (thin coated)	0.200
8	TiN (thick coated)	0.189

The highest coefficient of friction among the coated specimens was obtained in the thin-coated AlCrN specimen (specimen no. 3). There was no significant change between the friction coefficient values of the other coated specimens. When the average CoF was examined, the highest CoF was obtained in the uncoated Ti6Al4V specimen with a value of 0.42 and the lowest CoF was obtained as 0.165 in the thick-coated TiAlN specimen (specimen no.2). However, there are no significant differences between these results. When these results are evaluated with similar studies, it was also determined that the coatings applied to the Ti6Al4V specimen positively affected the CoF [44].

Figures 6, 7, and 8 show the worn surface FESEM images of the coated and uncoated Ti6Al4V alloy—the surface of the uncoated Ti6Al4V specimen in Figure 6 shows signs of adhesive wear. Adhesive wear occurs when a titanium surface comes into contact with the surface of most engineering materials, such as metal or ceramic, under force and motion.

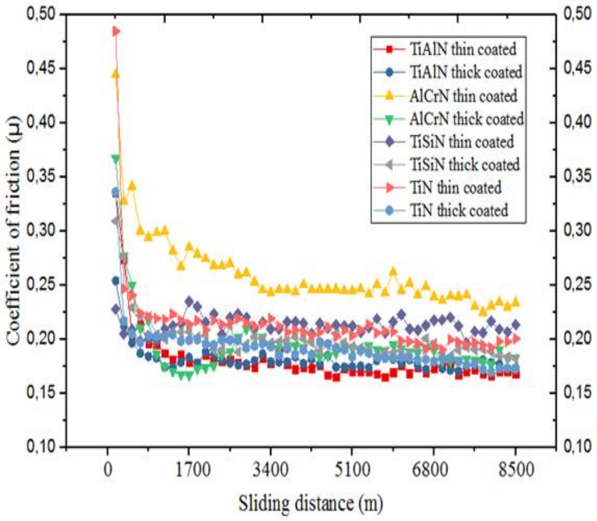


Figure 5. Coefficient of friction-coated Ti6Al4V depending on sliding distance

Therefore, titanium alloys are particularly prone to adhesive wear, leading to seizure and galling [45]. EDS analysis also confirms the adhesive wear. It shows that iron (Fe) and carbon (c) from abrasive disc AISI 5200 adhere to the Ti6Al4V specimen during the analysis.

The FESEM images are given In Figure 7 for the eroded surface of coated TiAlN and TiN specimens (specimens 1, 2, 7, and 8) and in Figure 8 for the coated TiSiN and AlCrN specimens (specimens 3, 4, 5, and 6). When the images were examined, it was observed that deep cavities were formed on the surface of the TiAlN thin-coated specimen (specimen no. 1). When the coating thickness increased, the volume loss and wear coefficient were the lowest in the thick-coated TiAlN specimen (specimen no. 2), and a smoother surface was obtained.

While wear debris and wear tracks were formed on the surface of the thin-coated TiN specimen (specimen no. 7), fractures occurred on the surface of the thick coating (specimen no. 8). As mentioned above, TiN-coated Ti6Al4V specimens had the highest wear rate and volume loss, and thus, it was observed that these were one of the specimens with the lowest wear resistance. The worn surface images also confirmed this. Also, in Fig 7. (b), the effect of the coating is visible. It is observed that the wear marks start on the base material and continue to the coating area but disappear in the coating area. When the worn surfaces of the thin and thick-coated TiSiN and

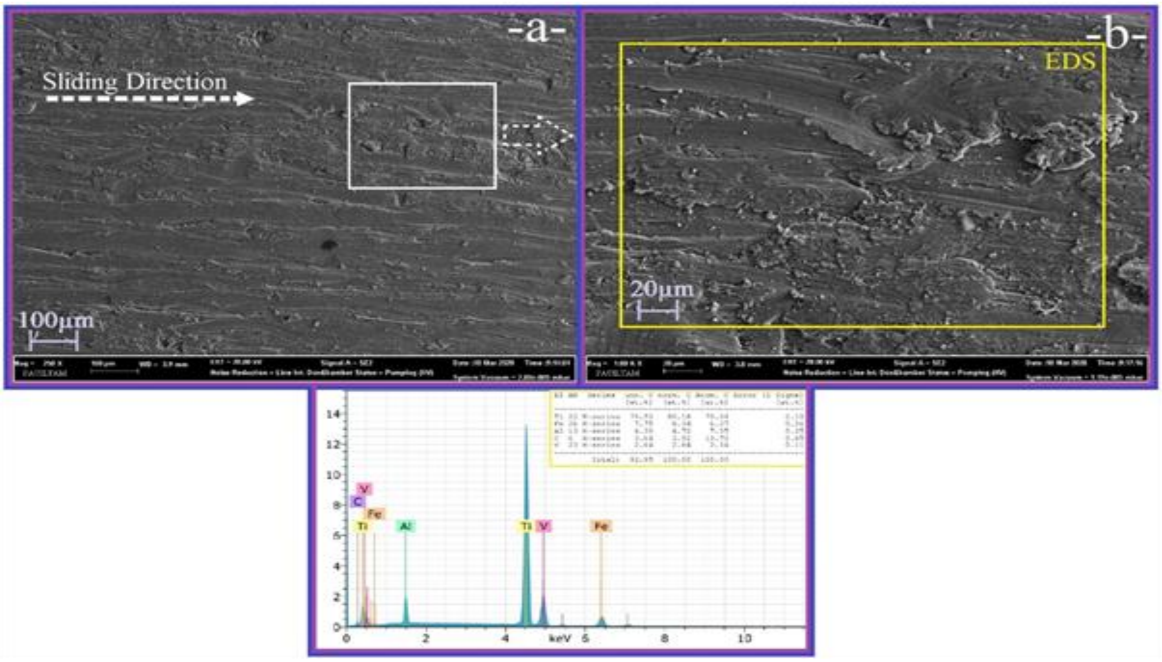


Figure 6. Worn surface FESEM images of uncoated Ti6Al4V a) 250x b) 1000x c) EDS analysis of surface

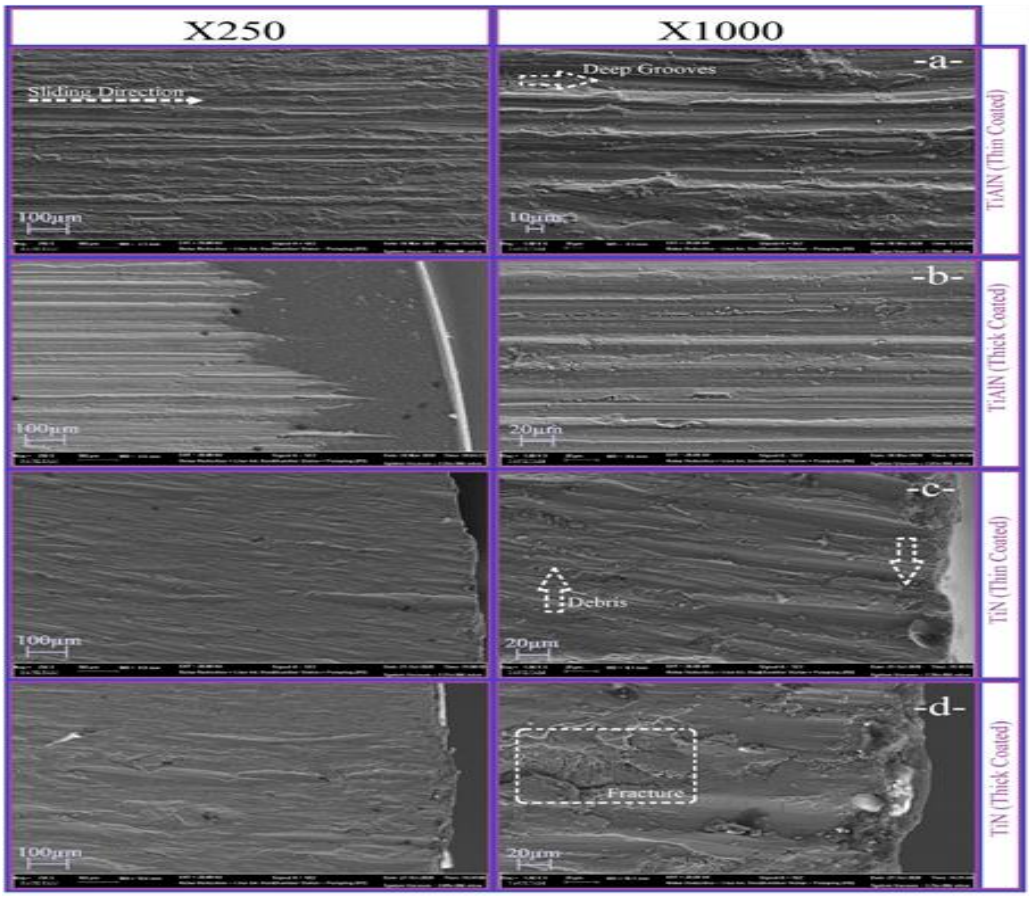


Figure 7. FESEM images of different magnifications a) TiAlN Thin Coated, b) TiAlN Thick Coated, c) TiN Thin Coated, d) TiN Thick Coated

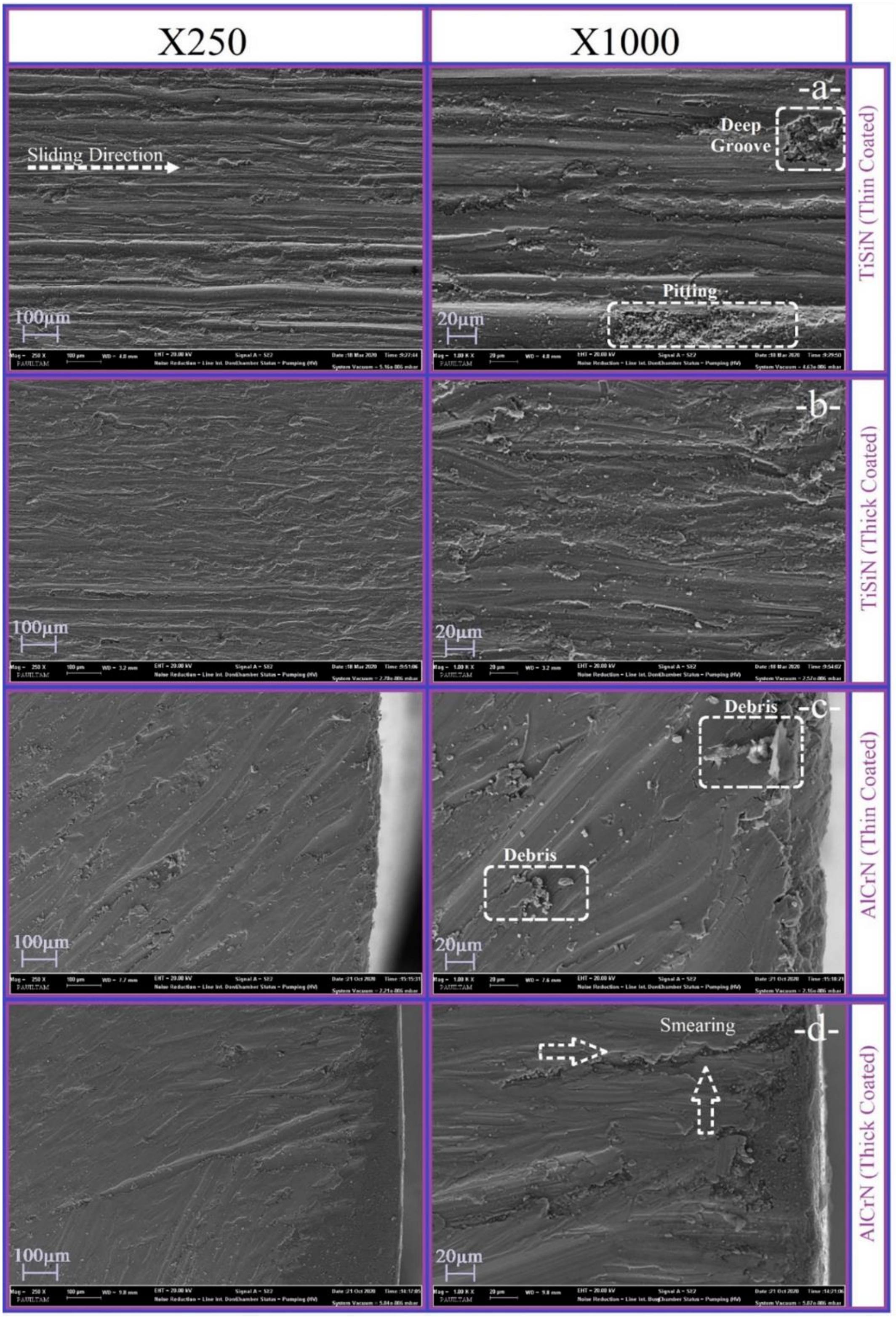


Figure 8. FESEM images of coated Ti6Al4V worn surface with different magnification a) TiSiN thin coated, b) TiSiN thick coated, c) AlCrN thin coated, d) AlCrN thick coated

AlCrN specimens given in Figure 8 are examined, pitting, wear debris, smearing, and deep groove mechanisms are seen. Pitting and wear debris generally occurred in thin coatings, while smearing and sticking ruptures occurred in thick coatings with increasing coating thickness. This is consistent with the results from similar studies [46-54]. The map image of the TiAlN thick-coated Ti6Al4V alloy (specimen no 2) is given in Figure 9.

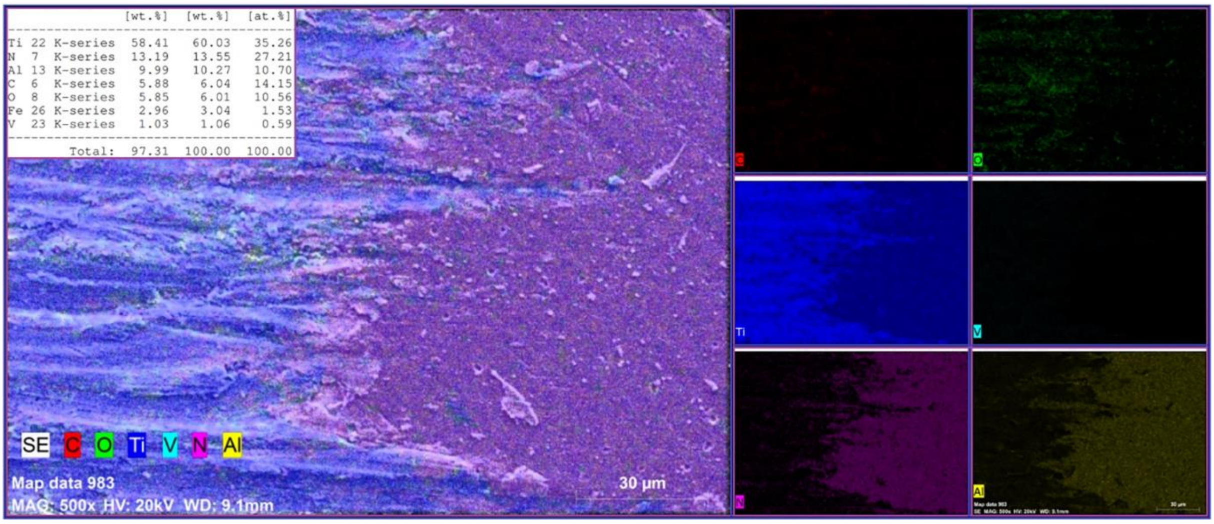


Figure 9. FESEM, EDS, and MAP analyses of C, O, Ti, V, N, Al Elements of TiAlN thick-coated

The image on the right in the map image is the image of the coating effect. It is seen that the O structure is homogeneously distributed, and the Al and N contents are generally located on the right half of the microstructure.

4. CONCLUSION

According to the analyses, the obtained results are given below:

1. It has been determined that there was a slight increase in the roughness values with the increase of the coating thickness in all coating types. The results showed that the thin specimens' microhardness values were higher than those of the thick specimens. The highest microhardness values were measured in the TiAlN-coated specimens in the coating processes performed on two parameters. TiSiN, TiN, and AlCrN coatings followed this specimen in hardness values, respectively.

2. The coating type with the highest surface roughness was the TiN and TiAlN coatings, while the lowest surface roughness value was measured in the TiSiN coating type.

3. Among the coated specimens, the highest coefficient of friction was obtained in the thin-coated AlCrN specimen.

4. The lowest volume loss and SWR were obtained with thick-coated TiAlN specimen in all wear test conditions.

5. The surface of the uncoated Ti6Al4V specimen shows signs of adhesive wear. While deep cavities were formed on the surface of the TiAlN thin-coated specimen, when the coating thickness was increased, the thick-coated TiAlN specimen was determined as the specimen with the lowest volume loss and wear coefficient, and a smoother surface was obtained in this specimen.

While wear debris and wear tracks were formed on the surface of the TiN thin-coated specimen, fractures occurred on the surface of the TiN thick-coated specimen. Pitting, wear debris, smearing, and deep grooves mechanism can be seen when the worn surfaces of thin and thick-coated TiSiN and AlCrN specimens are examined. Pitting and wear debris generally occurred in thin coatings, while smearing and adherence ruptures occurred in thick coatings.

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DECLARATION OF ETHICAL STANDARDS

The author(s) of this manuscript declare that the materials and methods used in their studies do not require ethics committee approval and/or legal-specific permission

AUTHORS' CONTRIBUTIONS

Hakan ADA: Contributed to conceptualisation, methodology, writing, reviewing and editing.

Ahmed Qays Jabbar EL RUBAYE: Contributed to performing experimental studies.

Elif AŞIKUZUN TOKEŞER: Contributed to performing microhardness tests.

Ahmet MAVİ: Contributed to performing profilometer tests.

Yavuz KAPLAN: Contributed to performing wear tests.

Sinan AKSÖZ: Contributed to conceptualisation and performing wear tests.

CONFLICT OF INTEREST

There is no conflict of interest in this study.

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