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The Effect of the Parameters on Al 7075 Coated with MAO Method by Adding Nano Ti-Powder

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Keywords

Micro Arc Oxidation
Wear
Coating
AA7075
Titanium

Abstract

Coating processes are carried out in many areas of industry to improve the surface properties of materials such as wear and corrosion resistance or appearance. One of these coating processes is ceramic-based surface coatings. In order to obtain harder, denser and more uniform coatings, the substrate material is coated with a ceramic-based material at the desired thickness. In this study, 7075 series aluminum was coated with nano Ti powder and non-additive micro arc oxidation (MAO) method in a solution consisting of KOH, $NaAlO_2$ and Na_3PO_4, and the effects of process parameters on coating thickness and wear behavior were experimentally investigated and the results were analyzed. According to the results obtained, it was observed that as the coating voltage, frequency and coating time increased, the wear rate decreased and a harder structure against wear is obtained. In addition, it was determined that the samples with nano titanium powder were less worn than the non-additive samples. According to SEM, EDX and XRD analyses, Al_2O_3 phase was observed in the coatings made without adding titanium particles; It has been observed that in addition to the Al_2O_3 main phase, TiO_2 and SiO phases are also formed in titanium doped coatings.

Cite

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1. INTRODUCTION

The primary goal of most materials science researchers is to improve the properties of metals and metal alloys, such as corrosion, wear and fatigue resistance, to minimize losses from wear and corrosion. Losses from corrosion and wear in the world constitute 3-5% of national income on average. Today, a significant part of the processes performed to eliminate these losses and improve the main properties of materials such as wear resistance consists of surface coating processes.

Coating with micro arc oxidation (MAO, also called as PEO, plasma electrolytic oxidation) method is based on the principle of coating the surface with a thick, hard, wear and corrosion resistant layer thanks to the plasma discharges caused by the electrochemical reactions of the substrate immersed in an electrolyte solution (Huang et al., 2004). The substrate material is connected to the power supply as the anode and the tank as the cathode. The substrate material is immersed in the electrolytic bath in the tank, which also acts as a cathode, and voltage is applied. Due to this voltage, electrodes surround the plasma and the oxide formed passes to the substrate and forms a film layer. It is possible to change the composition of the film layer in coatings with MAO by adding different chemicals to the electrolyte fluid (Li et al., 2013). MAO, an environmentally friendly coating method, has very different mechanical and physical structures. The most important advantage of coating with MAO method is lower investment cost compared to other coating methods (Zhu et al., 2007). Due to this advantage, the MAO method is frequently used in the automotive, aerospace, machinery, manufacturing and

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textile industries (Nie et al., 1999; Arslan et al., 2009; Shen et al., 2013; H.-Y. Wang et al., 2014; J.-H. Wang et al., 2014; Shao et al., 2014).

Arslan et al. (2009) coated AA2014 material with Al_2O_3 using the MAO method and investigated the high temperature wear behavior. In the study, micro hardness measurements were also made; The structure of the coating was examined by SEM, EDX and XRD analysis. While sometimes mounds were seen in the microstructure of the coating, pits have also occurred at the same time. When the surface roughness values were examined, the surface roughness of the substrate material was Ra = 0.1 micron, while the surface roughness after coating was obtained as Ra = 0.87 micron.

Nie et al. (1999) coated the Al6082 substrate with Al-Si-O using the micro arc oxidation method and examined the hardness and wear behavior of the coatings. It has been reported that the wear resistance of the obtained coating is very good and its hardness has a value of 2400 HV. It has also been stated that with the increase in coating thickness, the wear resistance increases. Zhu et al. (2007) investigated the wear behavior of samples by coating Al-Si substrate in an alkali-silicate solution with a PH value of 10 by micro arc oxidation method. Wear tests of the samples were carried out under 50 N normal load and it was determined that delamination wear mechanism occurred as a result of wear. It was stated that the wear resistance increased when compared to the coated substrate material. Shokouhfar and Allahkaram (2017) coated the titanium-based substrate with micro arc oxidation method in their study. In the study, nano-sized SiC, Al_2O_3 and TiO_2 powders were added to the coating solution and the corrosion and wear behavior of the samples were investigated. It has been reported that the nanoparticles added to the solution reduce the wear rate, friction coefficient, corrosion current density and surface roughness.

Ma et al. (2022a), made coatings in silicate electrolyte on different Mg-Li alloy substrate materials by plasma electrolytic oxidation method. Structural analysis, coating thickness, corrosion and wear resistance of these coatings were investigated. According to the results obtained, the addition of Al and Y phases to the substrate caused the formation of finer grains and caused the formation of AlLi and Al2Y phases. In addition, Mg-Li alloys caused a more resistant structure against corrosion. It was observed that the coating with Mg-14Li-3Al-1Y backing material was the most corrosion resistant combination with 7.485 × 10 icorr.

Ma et al. (2022b), made coatings by adding boron carbide and carbon particles on the Mg-Li substrate by the plasma electrolytic oxidation method. The effect of the added B4C and C particles on the microstructure was investigated. It was observed that the B4C coating added to the results of the experiments closed the micropores in the structure. The added carbon particles form protrusions on the coating surface, which indicates that the added powders have been successfully integrated into the coating. It was seen that both reinforcing powders increased the coating thickness and helped to obtain a more non-porous structure by reducing the pores in the structure. This contributed positively to wear and corrosion resistance. As a result, B4C and C supplements added to the electrolyte liquid of the coating made with the PEO method improved the coating properties in a positive way.

There are many studies on the coating of the cutting tool in machining technology, where cutting tool wear is frequently encountered (Salimiasl & Rafighi, 2017; Rafighi, 2021; Şahinoğlu & Rafighi, 2021; Das et al., 2022). When these studies are examined, it is seen that a material with both more economical and desired properties is obtained by adapting the coating processes to the existing material.

In the literature search, it has been seen that there are many studies that have been coated with the MAO method. The difference of this study from the others made a use of solution that has not been used before consisting of KOH, $NaAlO_2$ and Na_3PO_4 by adding Ti powders into the solution during coating, and the effect of these solution elements on the coating performance was examined. In the current study, the effect of Ti powder on the structure and mechanical properties of the coating was examined, and a final product with the desired properties was obtained. It has been an original study, different from other studies, with the effect of the solution used and the supplementation.

2. MATERIAL AND METHOD

In this study, AA7075 aluminum was coated with micro arc oxidation method and the effect of test parameters on coating thickness and wear was investigated using MAO coating device powered by AC current (Table 1). The sample names given depending on the parameters are also given in Table 1. In the coating process, an electrolytic solution was used, unlike other studies, a mixture of 4 g/l Na_3PO_4 (Sodium Phosphate), 1 g/l KOH (Potassium Hydroxide) and 3 g/l $NaAlO_2$ (Sodium Aluminate). In Ti added coatings, nano-titanium powder (0.8 g/l) was added to the same coating solution and mixed homogeneously (Table 2). The mixing of titanium powder into the solution was done with a mechanical mixer. The AA7075 substrate material was immersed in the solution to become the anode through the hole drilled on the sample holder in the MAO coating device. The AA7075 substrate material is connected to the sample holder in the MAO coating device as an anode through a hole. The coating chamber was connected to the positive pole as the cathode, and the sample was made ready for the oxide coating process. The system, whose anode and cathode were adjusted as mentioned, was made ready for coating and the coating process was carried out using parameters such as voltage, amperage and time. As seen in Figure 1a, after the sample was connected as an anode, the coating process started by giving the appropriate current and voltage to the parameters and the 25.4x25.4 mm sample was coated. Olympus Tokyo brand optical microscope was used for coating thickness measurements of coated substrate materials and SEM images of the coatings were examined and EDX and XRD analyzes were made.

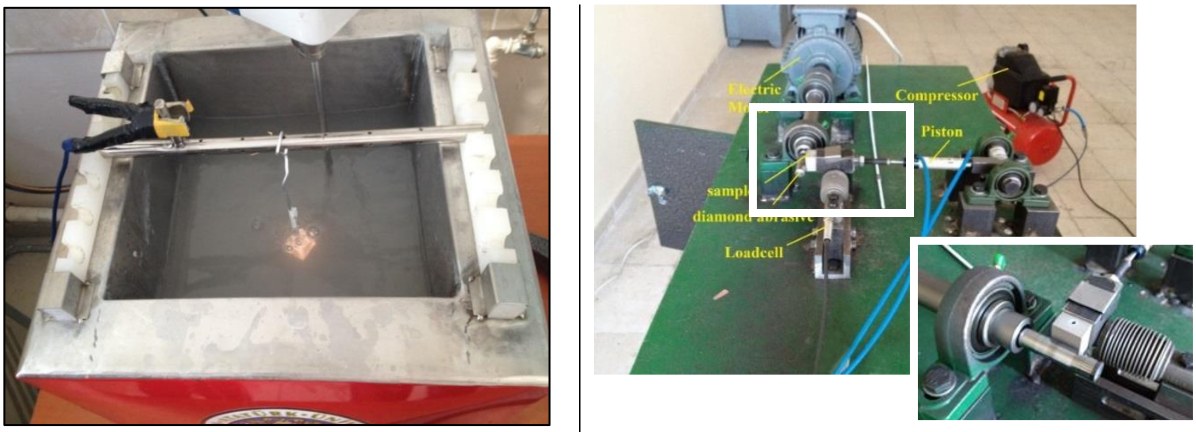
Table 1. *Process parameters and sample names*

Sample Name	Coating Time (min)	Frequency (Hz)	Voltage (Volt)
K1,K2,K3	10	100	450,500,550
K4,K5,K6		350	450,500,550
K7,K8,K9	20	100	450,500,550
K10,K11,K12		350	450,500,550
K13,K14,K15	30	100	450,500,550
K16,K1,K18		350	450,500,550
K19,K20,K21	10	100	450,500,550
K22,K23,K24		350	450,500,550
K25,K26,K27	20	100	450,500,550
K28,K29,K30		350	450,500,550

Wear tests were carried out on a diamond abrasive disc in accordance with ASTM G99 standard, using a 50 rpm rotation speed and 2 kgf (~2 daN) force on a Block-on-disc test device. The test sample was contacted with the pneumatic system on the abrasive shaft and the contact force was adjusted with the help of the valve in the system and controlled by the load cell (Figure 1b). At the end of 500, 1000 and 1500 meters of wear distances, the samples were weighed with a precision of 10^{-5} g and the amount of wear was obtained. The experimental study plan is given in Figure 2.

Table 2. *Chemicals used in the experiments*

Chemical	Amount (g/l)
KOH (Potassium Hydroxide)	1
$NaAlO_2$ (Sodium Aluminate)	3
Na_3PO_4 (Sodium Phosphate),	4
Ti (only Ti added coatings)	0.8



a b

Figure 1. a) *MAO coating process,* ***b)*** *Wear tester*

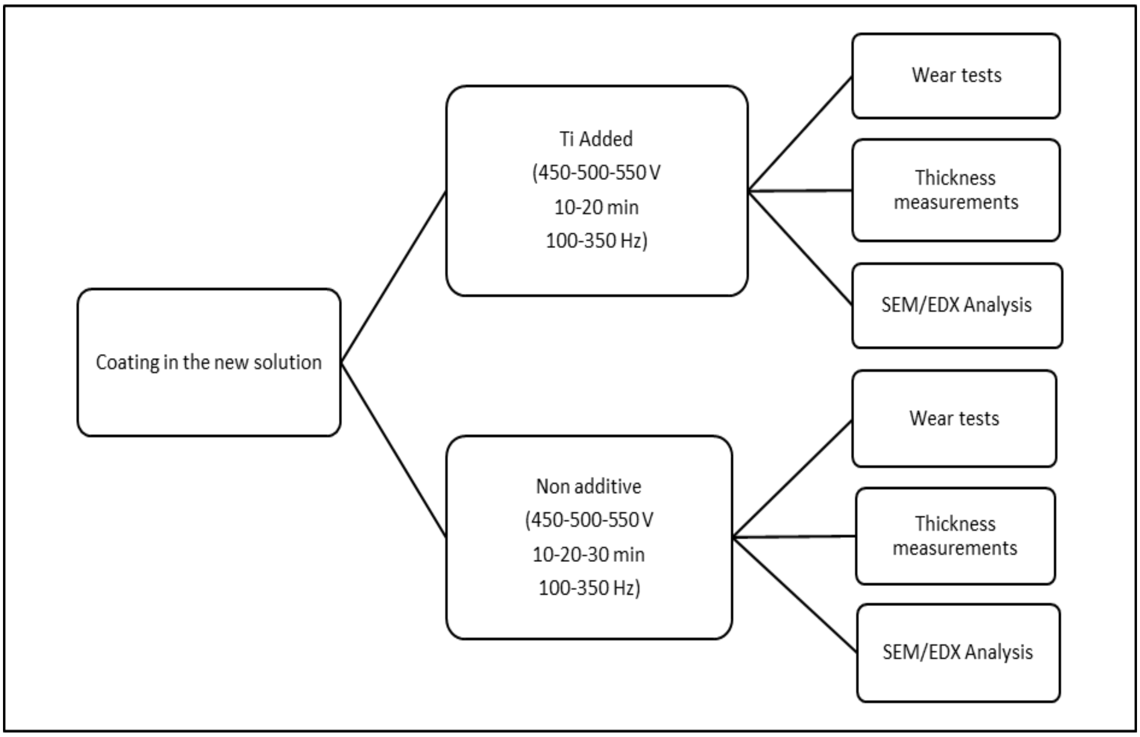


Figure 2. *Scheme of experimental Plan*

3. RESULTS AND DISCUSSION

After the coating processes, microstructure examinations and wear tests of the samples were made and the results were as follows.

The thickness values of the coatings carried out in Ti added and non-added solutions were given in Figure 3. The thickness values of the coatings made without Ti addition at 100 Hz frequency and 450, 500 and 550 Voltage were respectively measured as 3.2, 7 and 8 μm, while the coatings made at 350 Hz and 450, 500 and 550 V were measured respectively as 3.9, 9 and 10 microns. (Figure 3a). When the thickness of the Ti-added coatings was examined, the thickness value of the coating made at 100 Hz and 450 V was 3.6 micron, while 8 microns at 550 V were obtained. Likewise, the thickness value of the coating made at 350 Hz was 5.1 microns at 450 V, while 12 microns were obtained at 550 V (Figure 3b). In coatings without Ti addition, the increase in voltage increased the coating thickness. While there was no significant change in the coating thickness at 450 and 500 V values in Ti added coatings, the coating thickness increased at 550 V, and the highest coating thickness value was obtained (Figure 3b). In all coatings, the highest coating thicknesses were obtained at 350 Hz and 550 V.

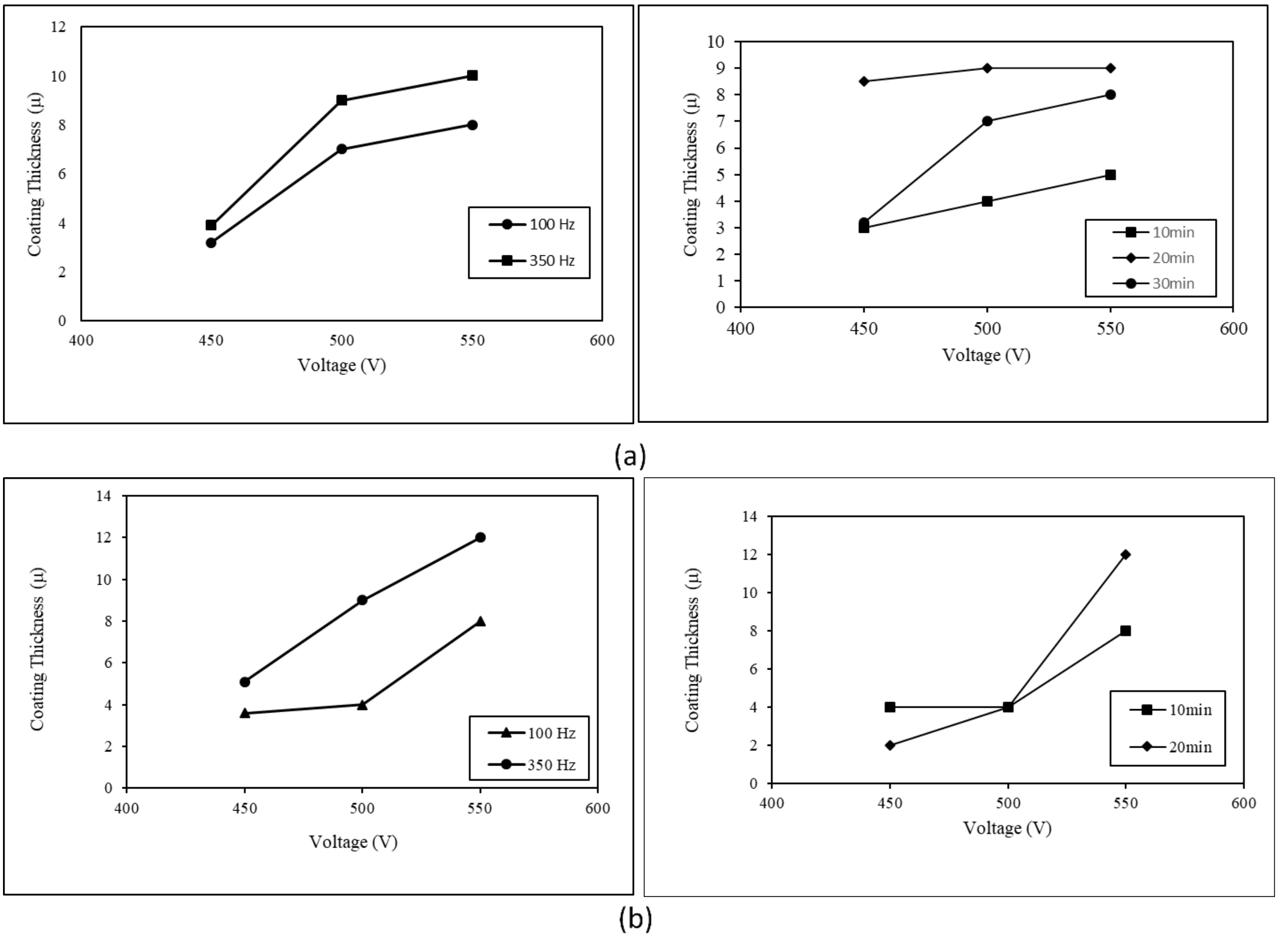


__Figure 3.__ Effect of coating parameters on coating thickness __a)__ without Ti addition, __b)__ with Ti addition

According to Equation 1, arc power increases in direct proportion to the square of the voltage. Since the increase in voltage increases the arc power, it increases the coating thickness (Zhu et al., 2007). Since the voltage changes as a function of time, the arc occurs at the peaks of the functional curve. At the same time, the increase in voltage and frequency increases the peaks on the graph, increasing the amount of arc per unit time.

$$P = \frac{V^2}{R} \tag{1}$$

P; power (watt), V; the voltage (Volt) and R symbolize the resistance (ohm). When the effect of coating time on coating thickness was examined, it was determined that coating thickness first increased and then decreased with the in-crease of coating time (Figure 3b). When the thickness of the 450V coated samples was examined, it was measured as 3 microns on coating for 10 minutes, 8 microns for 20 minutes and 3.2 microns for 30 minutes. When the thickness of the coatings made at 550 V was examined, it was measured as 5 microns in the coating for 10 minutes and 9 microns in the coating for 20 minutes, while the coating thickness of the sample coated for 30 minutes was measured as 8 microns. Increasing the coating time increases the coating thickness by increasing the amount of arc formed on the substrate per unit time (Dudareva & Gallyamova, 2019). On the other hand, the excessive arc that occurs in long coating times disrupts the coating structure. In high frequency coatings, the coating process could not be done because the coating device draws excessive current and causes it to work noisy.

The increase in the coating time increases the thickness of the oxide layer on the surface of the substrate by accumulating a larger amount of coating material on the substrate surface and a more resistant structure is obtained (Figure 4) (Nie et al., 1999; Shokouhfar & Allahkaram, 2017).

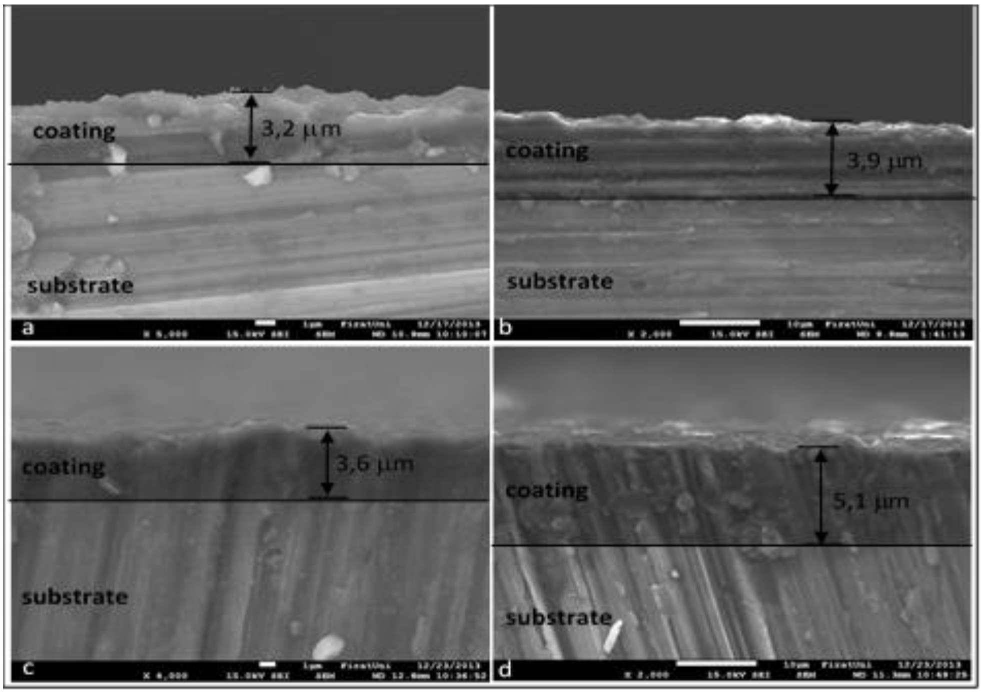


Figure 4. *SEM images of* ***a)*** *10min, 100 Hz, 450 V, Ti-free,* ***b)*** *10 min, 350 Hz, 450 V, Ti-free,* ***c)*** *10 min, 100 Hz, 450 V, Ti-added,* ***d)*** *10 min, 350 Hz, 450 V, Ti-added*

The wear amount of the coatings without the addition of nano titanium powder was given in Figure 5a. According to Figure 5a, increasing the coating time decreased the amount of wear. When the effect of coating time on the wear amount for 500 m wear distance was examined, the wear amount of the samples coated for 10 minutes was 120x10^{-5} g; The amount of wear was 102x10^{-5} g in the samples coated for 20 minutes, and 57x10^{-5} g was obtained in the samples coated for 30 minutes by continuing to decrease. When the wear amounts of Ti added and non Ti added coatings were com-pared (Figure 5c) the Ti added sample worn 102x10^{-5} g while the Ti added sample worn 68x10^{-5} g, at 1000 m the Ti added sample was 206x10^{-5} g while the Ti added sample was worn for 500 m. It was 147x10^{-5} g worn. On the 1500 m wear distance, it was observed that the samples with Ti addition of 317x10^{-5} g and 195x10^{-5} g were less worn. In addition, in wear above a certain wear distance for thin coatings, the coating material was worn by the abrasive disc in a short time and the disc contacts the softer substrate material (Lin et al., 2003). When the wear tests of the coating samples made with the addition of Nano Ti powders were examined, it was seen that the additional powder significantly reduced the wear rate of the coating. This is due to the formation of wear-resistant titanium dioxide and silicon dioxide phases with the aluminum oxide phase formed on the substrate during coating (Figure 6, Figure 7b). Free form nano Ti particles increase the wear resistance of the coating (Li et al., 2013; Tosun et al., 2019; Odabasi & Odabasi, 2020).

In Figure 5b, the wear amounts of the samples coated at frequencies of 100 and 350 Hz are given. According to Figure 5b, the wear amount of the samples coated at 450 V voltage and 100 Hz frequency was 226x10^{-5} g, while the wear amount of the sample coated at 450 V voltage and 350 Hz frequency was measured as 161x10^{5} g. While the wear amount of the sample coated at 500 V voltage and 100 Hz frequency was 187x10^{5} g, the wear amount of the sample coated at 500 V voltage and 350 Hz was 156x10^{-5}. Likewise, the wear amount of the sample coated at 550V voltage and 100 Hz frequency was 180x10^{-5} g, while the wear amount of the coating made at 350 Hz frequency was 149x10^{-5} g. The increase in frequency has decreased the amount of wear by increasing the coating thickness. Increasing frequency value increases the amount of arc per unit of time. By increasing the number of peaks in the voltage-time change graph at the same time, the coating thickness increases and the amount of wear decreases. When the SEM images of the non-additive coatings were examined, the coating thickness of the sample coated at 100 Hz frequency was 3.2 microns, while the coating thickness of the sample coated at 350 Hz frequency was 3.9 microns (Figure 4a, 4b). According to the SEM images of Ti added coatings, the coating thickness was 3.6 microns at a frequency of 100 Hz, while 5.1 microns at a frequency of 350 Hz (Figure 4c, 4d).

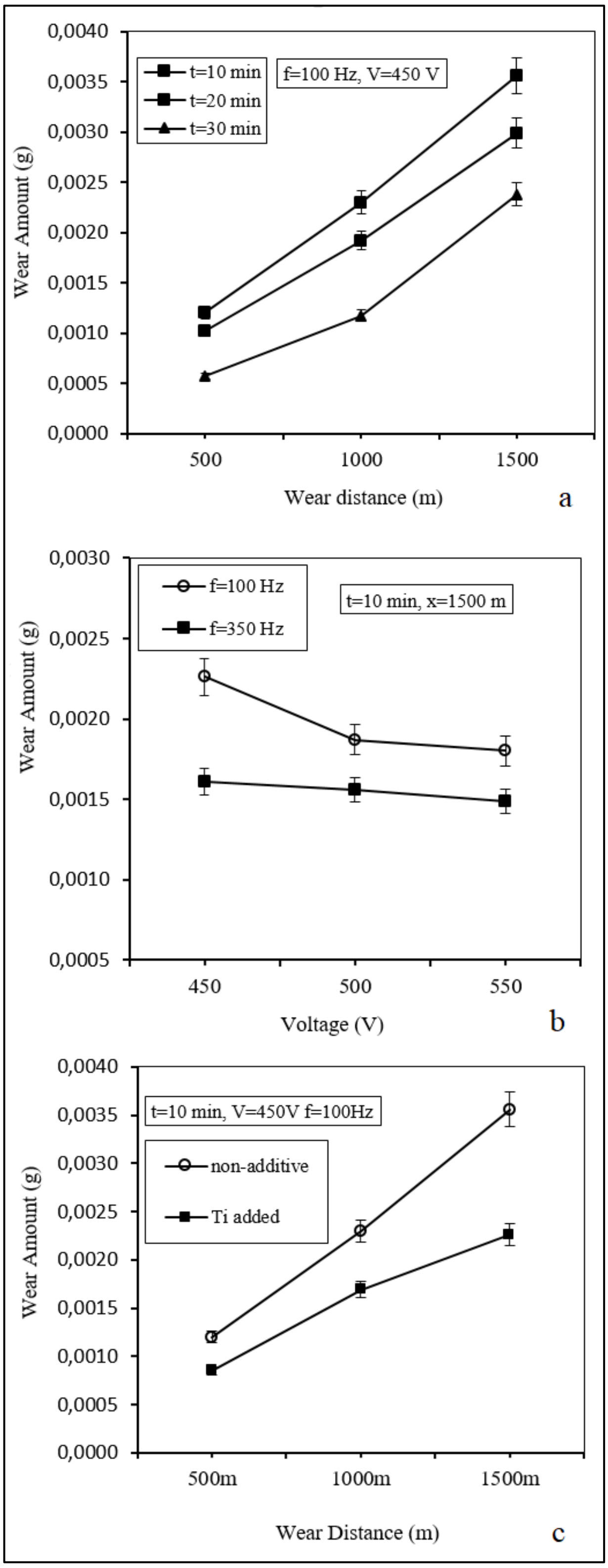
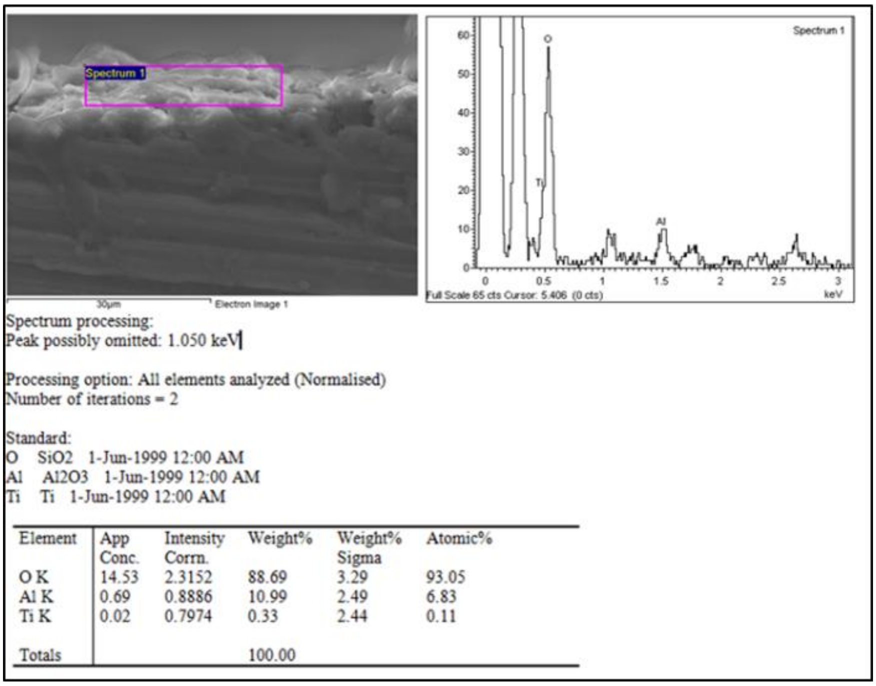


Figure 5. *The effects of parameters on wear amount, **a)** Wear distance – wear amount at different times, **b)** Voltage – Wear amount, **c)** Wear distance – Wear amount of non-additive and Ti added solution*

When the effect of the voltage on the wear rate was examined in Figure 5b, it was seen that the wear rate decreased as the voltage increased. In the wear tests performed at a frequency of 100 Hz, the wear rate for 450 V voltage was 226x10^{-5} g, for 500 V it was 187x10^{-5} g and for 550 V the wear rate was reduced to 180x10^{-5} g. In the experiments conducted at 350 Hz, the amount of wear at 450 V was 161x10^{-5} g, while at 500 V it was 156x10^{-5} g, decreasing at 550 V. Increasing arc power creates a more non-uniform coating structure on coating area (Zhu et al., 2007). At the same time, the structure formed as non-uniform and porous increases the surface roughness and friction coefficient (Lin et al., 2003). With the increasing thickness of the coating, a more irregular structure is formed and the wear resistance increases. In addition, high voltage values disrupt the structure of the coating and create an irregular coating thickness structure.

In Figure 5c, the wear rates of the coatings made in the solution with and without the addition of Ti powder are compared. Considering the wear amount for 1000 m wear distance of the samples, the wear amount of the samples coated with Ti-free solution was 230x10^{-5} g and the wear amount of the Ti added samples was 169x10^{-5} g. Similarly, for the samples coated with a Ti-free solution for 1500 m wear distance, 356x10^{-5} g was obtained, and 226x10^{-5} g was obtained for samples coated with a Ti-added solution. While the thickness of Ti added coatings made in 10 minutes was 3.6 microns, the thickness of Ti added coatings made under the same conditions was 3.2 micron (Figure 4a, 4c). Free Ti particles added to the solution adhere to the substrate with the arc formed during coating, increasing the coating thickness. Furthermore, in Figure 4 the porosity distribution within the coating structure was observed as homogeneous.

As a result of the regional EDX analysis of the Ti doped coatings, it was observed that the main component of the coating was Al_2O_3, along with the formation of SiO and TiO_2 compounds (Figure 6). At the same time, it was observed that Ti particles were in free form in the coating structure. Li et al. (2013) stated that when Ti is added at an appropriate concentration, free nano Ti particles are formed in the structure of the coating. When the EDX analysis was examined on the basis of elements, it was observed that there was 10.99% aluminum and 0.11% titanium according to the weight of oxygen at 88.69%. The presence of a high amount of oxygen during EDX analysis indicated that the micro arc oxidation coating was monolithic and composed predominantly of Al_2O_3 (Muhaffel et al., 2021). In addition to Al_2O_3, TiO_2 and SiO compounds were also observed according to the analysis result as a result of high oxygen component.



Spectrum processing:
Peak possibly omitted: 1.050 keV

Processing option: All elements analyzed (Normalised)
Number of iterations = 2

Standard:
O SiO2 1-Jun-1999 12:00 AM
Al Al2O3 1-Jun-1999 12:00 AM
Ti Ti 1-Jun-1999 12:00 AM

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
O K	14.53	2.3152	88.69	3.29	93.05
Al K	0.69	0.8886	10.99	2.49	6.83
Ti K	0.02	0.7974	0.33	2.44	0.11
Totals			100.00		

Figure 6. *EDX analysis result (Powder: Ti-doped, Frequency:350 Hz, Voltage:550 V, Time: 20 min)*

According to the XRD analysis of the sample coated with a normal solution (Ti-free), it was determined that the aluminum oxide phase, which is the main component of both substrate and coating, was seen in the microstructure (Figure 7a). The aluminum oxide phase was located at 2θ = 38,360° and 2θ = 78,159°, while the Fe phase was observed at $2\theta = 44,580^{o}$ and $2\theta = 64,960^{o}$. It was observed that TiO_2 and SiO phases were also formed along with the main phase of aluminum oxide in samples coated with the solution containing Ti (Figure 7b).

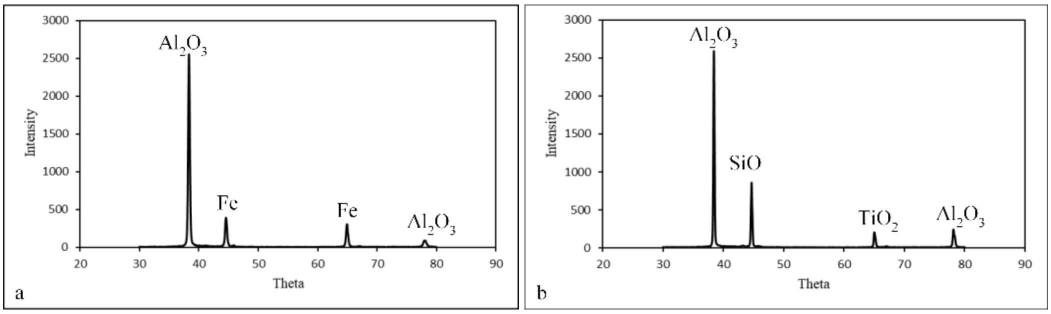


Figure 7. *XRD analysis results of* ***a)*** *Ti free, 500 V, 10 min, 100 Hz,* ***b)*** *Ti added, 500 V, 10 min, 100 Hz*

While aluminum oxide phases were seen at $2\theta = 38.440^{o}$ and $2\theta = 78.1$, the silicon oxide phase was observed at $2\theta = 44.680$ and the titanium dioxide phase at $2\theta = 65,001$. Nano Ti particles added to the coating solution combined with oxygen to form the TiO_2 phase with high wear resistance.

In Figure 8, SEM images of the samples with/without Ti added, taken after wear tests, were given. While the adhesive and abrasive wear mechanism were seen together in the worn zone of the Ti added sample, which was coated at 450 V voltage value (Figure 8a), the abrasive wear mechanism was predominantly observed in the Ti added sample (Figure 8b). In line with the experiments and literature information, it has been shown that the main wear mechanism of the TiO_2 coating is abrasive wear (Mu et al., 2013). When the SEM image of the sample without Ti added coated at 550V voltage was examined, it was seen that the dominant wear mechanism was the adhesive wear mechanism (Figure 8c). Abrasive wear was predominantly prominent in Ti- added coating (Figure 8d). It has been determined that the predominant wear type in the wear mechanisms of Ti-added samples is abrasive wear (Ding et al., 2018; Özler et al., 2020).

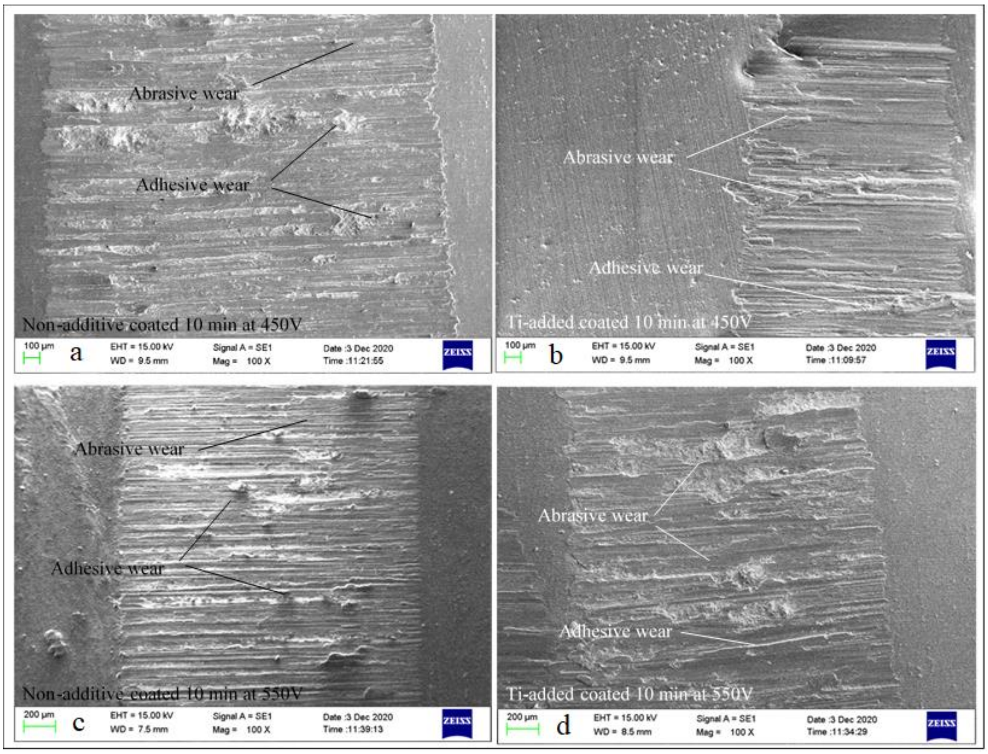


Figure 8. *SEM images of samples after wear tests,* ***a)*** *Non additive coated 10 min at 450 V,*
b) *Ti-added coated 10 min at 450 V,* ***c)*** *Non-additive coated 10 min at 550 V,*
d) *Ti-added coated 10 min at 550 V*

4. CONCLUSION

In this study, 7075 series aluminum material was coated by micro arc oxidation method with and without Ti addition, and the following results were obtained by examining the effect of coating parameters on thickness and wear:

- As the applied voltage increased during coating, the amount of wear of the coating decreased. Increasing the amount of voltage increases the thickness of the coating. For this reason, the rate of wear has decreased.
- As the frequency increased during coating, the thickness of coating part increased and the amount of wear of the coating decreased. Increasing the frequency increases the number of electrons passing per unit volume and causes more coating material to be deposited on the substrate surface. When the coatings made at 100 Hz and 350 Hz frequency values were compared, it was seen that thicker coatings were obtained with the increase in the frequency value. This accumulation has created a structure more resistant to wear in the coated substrate material.
- As the coating time increased, the coating thickness increased and the amount of wear decreased accordingly. The longer the time, the longer the substrate material will be exposed to the arc and the more coating material will be deposited on the surface and the higher the wear resistance.
- According to the EDX analysis of Ti added sample, SiO compound was found together with the main phase of Al_2O_3. However, it was also seen that Ti particles were in free form in the coating.
- According to the regional XRD analysis taken from the Ti added coating surface, it was observed that TiO_2 and SiO compounds were formed in addition to the main phase of Al_2O_3.
- The addition of Ti added to the coating solution reduced the amount of wear of the coated samples. This is due to the Al_2O_3 phase deposited on the substrate during the coating and the TiO_2 and SiO phases formed next to the main phase. While the Ti phase is free in the microstructure of the coating, the nano-sized Ti particles have increased the resistance to wear during the coating.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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