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**Plasma electrolytic oxidation of Ti-6Al-4V
alloy to improve tribological and
mechanical properties**

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IN THE NAME OF GOD

PLASMA ELECTROLYTIC OXIDATION OF Ti-6Al-4V ALLOY TO IMPROVE TRIBOLOGICAL AND MECHANICAL PROPERTIES

BY

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Abstract

Plasma electrolytic oxidation of Ti-6Al-4V alloy to improve tribological and mechanical properties

By

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In the present work, plasma electrolytic oxidation of Ti-6Al-4V is investigated. The process was studied previously. In this study, the author tried to study some new aspects of the process. To do it, an electrolyte similar to that of used in other studies was selected. The constituents of the electrolyte were: sodium chloride, sodium hydroxide, sodium silicate, and hydrogen peroxide. These materials were used in electrolytes previously. In the present study, all of these constituents are used together to use the advantages of all materials. The application of all these materials together increases the growth rate and modifies the wear resistance of the coatings. Besides these constituents, starch was added. The primary objective of starch addition was to increase the viscosity and stability of the electrolyte against boiling and splashing out, but during the study it was clear that starch is effective in composition and wear resistance of the coating. The optimum concentrations of each of the electrolyte constituents were obtained by design of experiments via Taguchi method. One of the objectives of the present study was producing coatings comparable to the coatings of other researches while using very simple equipment and conditions. All materials used in the electrolyte were completely available and inexpensive and no expensive or complex were used. Tribological examination of the specimens coated in the optimized electrolyte showed that plasma electrolytic oxidation increased the wear resistance (reduction of friction coefficient and weight loss) of the alloy.

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List of Abbreviations

Title	Page
AC	Alternative current
Adj MS	Adjusted mean of squares
Adj SS	Adjusted sum of squares
ANOVA	Analysis of variance
Bcc	Body centered cubic
CAE	Cathode arc evaporation
CVD	Chemical vapor deposition
DC	Direct current
DF	Degree of freedom
DOE	Design of experiments
EPT	Electrolytic plasma technology
Hcp	Hexagonal close packed
MAO	Micro-arc oxidation
Min	Minute
MSD	Mean squared deviation
PED	Plasma
PEO	Plasma electrolytic oxidation
PES	Plasma electrolytic saturation
PET	Plasma electrolytic twins
PVD	Physical vapor deposition
QC	Quality characteristics
Rpm	Revolution per minute
S/N ratio	Signal to noise ratio
SBF	Simulated body fluid
SEM	Scanning electron microscopy
Seq. SS	Sequential sum of squares

Title	Page
S-N curve	Stress versus number of cycles curve
SS	Sum of squares
XRD	X-ray diffraction

Introduction

Ti-6Al-4V is a well-known alloy for many applications such as aerospace, automotive, and biomedical industries. Many studies has been carried out on improving the tribological behavior of the alloy via a variety of techniques such as physical vapor deposition [Ref1,Ref75, Ref119], ion implantation [Ref115], laser alloying [Ref116, Ref124], thermal oxidation [Ref140], sol-gel [Ref147] etc. one of the recently developed methods of improving tribological performance of this alloy is plasma electrolytic oxidation which has been developed rapidly in recent years. The process is a new generation of conventional anodizing which differs from the latter in applying high potentials [Ref13]. The high voltage leads to formation of a plasma envelope which appears as luminescent sparks on the surface of the anode [Ref]. The formation of plasma enhances the tribological properties of the coatings [Ref13].

Many research works studied the tribology of PEO coatings on Ti-6Al-4V and investigated the effect of porosity [Ref52], electrolyte composition [Ref105], current mode [Ref6, Ref58], sparking voltage [Ref126] and other factors on the microstructure, chemical composition, tribology and mechanical properties of PEO coatings.

The main objectives of the present work were:

- Lowering applied voltage
- Simplifying the process
- High wear resistance

Simplifying of the process was carried out by using simple equipment and familiar, available, and inexpensive materials. All materials were abundant and the process of making the electrolytes was easy. The power source used for PEO was a simple DC source with no extra attachments.

For lowering of the applied voltage, a combination of sodium chloride and sodium hydroxide was used and sodium silicate was added to the electrolyte for increasing the growth rate of the coatings.

To obtain high wear resistance, starch was added to the electrolyte and an optimum composition of the electrolyte was calculated using a statistical analysis by Taguchi method.

To evaluate the wear properties of the coatings wear tests were carried out by a pin-on-disk apparatus. Since one of the important applications of Ti-6Al-4V is production of biomedical implants, some wear tests were carried out in the presence of ringer's solution as the simulated body fluid. Results showed a better wear resistance for PEO coated alloy under dry conditions. In the presence of ringer's solution, however the uncoated alloy showed a better performance than the PEO coated alloy.

Fatigue tests showed that PEO is not a suitable method to enhance the wear properties of the alloy because of the brittle nature of the coatings and increasing the probability of fatigue crack initiation and propagation.

Section One

Theory

1. Titanium and its alloys

1.1. History of Titanium

Titanium is present in the earth's crust at a level of about 0.6% and is therefore the fourth most abundant structural metal after aluminum, iron, and magnesium. The most important mineral sources of titanium are ilmenite (FeTiO_3) and rutile (TiO_2). The first suspicion of a new, unknown element presents in a dark, magnetic iron sand (ilmenite) in Cornwall (UK) was expressed in 1791 by Gregor, a clergyman and amateur mineralogist. In 1795, Klaproth, a German chemist, analyzed rutile from Hungary and identified an oxide of an unknown element, the same as the one reported by Gregor. Klaproth named the element titanium after the Titans, the powerful sons of the earth in Greek mythology [1,2].

Wilhelm Justin Kroll from Luxembourg is recognized as the father of titanium industry. In 1932, he obtained significant quantities of titanium by combining TiCl_4 with calcium. He demonstrated that titanium could be extracted commercially by reducing TiCl_4 by changing the reducing agent from calcium to magnesium at the U.S. Bureau of Mines. Today this is still the most widely used method and is known as the “Kroll process”. After the Second World War, titanium-based alloys were soon categorized as main materials for