International Journal of Engineering Sciences &

Research Technology

(A Peer Reviewed Online Journal) Impact Factor: 5.164





Chief Editor Dr. J.B. Helonde

Executive Editor Mr. Somil Mayur Shah



JESRT

[Nanimina* *et al.*, 9(2): February, 2020] ICTM Value: 3.00

INTERNATIONAL JOURNAL OF ENGINEERING SCIENCES & RESEARCH TECHNOLOGY

ISSN: 2277-9655

CODEN: IJESS7

 \odot

Impact Factor: 5.164

CHARACTERIZATION AND ANALYSIS OF BIOMEDICAL GRADE ALLOY MACHINED SURFACE IN ALUMINUM POWDER MIXED EDM

A. M. Nanimina^{*1}, Djimako BONGO¹, Nandiguim LAMAÏ¹, Togdjim Jonas¹, Saka GONI², Alain MATIBEYE³, & Ahmad Majdi ABDUL-RANI⁴

¹Mechanical Engineering department, Higher National Institute of Sciences and Techniques of Abéché (INSTA), 130, Abéché, Ouaddai, Chad

²Mechanical engineering department, Polytechnique University of Mongo, 4377, Mongo, Guerre,

Chad

³Physic department, University of Sarh, Sarh, Chad

⁴Mechanical Engineering department Universiti Teknologi PETRONAS (UTP), 32610 Seri Iskander, Perak, Darul Ridzuan, Malaysia

DOI: 10.5281/zenodo.3692946

ABSTRACT

The use of metallic powder mixed electrical discharge machining (PMEDM) is an alternative of machining hard and difficult materials. Titanium alloy is one of difficult to machine materials. Titanium alloy is employed in various domains including biomedical. The characterization of titanium alloy biocompatibility of titanium alloy Ti-6Al-4V in terms of corrosion, phase analysis and surface sensitivity are prior analysis to its usage as implant. The objective of this research study is to investigate the effect of PMEDM on Ti-6Al-4V machined surface. Corrosion analysis was conducted using linear polarization resistance (LPR) test. XRD was used to examine changes on the nano aluminum PMEDM machined surface and compounds formed using Cu Ka radiation. Xray photoelectron spectroscopy (XPS) techniques were used to analysis the chemical composition and oxidizing amounts of the outermost machined surface and evaluate if any contamination from foreign particles. The results show that the corrosion of nano aluminum PMEDM on titanium alloy is about 42% reduced as compared to the corrosion of conventional EDM on titanium alloy machined surface. Electric sparks generated during machining decompose the kerosene dielectric fluid into C and H and is resulting in C deposited onto the machined surface. The results of the titanium alloy machined surface show difference in term of machined surface compositions.

KEYWORDS: Characterization, PMEDM, Corrosion, phase structure, surface sensitivity.

1. INTRODUCTION

Biomedical grade alloy such Ti-6Al-4V is applied in various domains including the usage in implant. In general, machining titanium alloy is difficult when employing conventional process due to due to the mechanical properties leading to non-desired shape [1]. Adding metallic powder to electrical discharge machining (EDM) dielectric can improve the machined surface quality in terms of corrosion resistance, fatigue performance, phase structure and surface sensitive [2]. Powder mixed EDM (PMEDM) process is of potential application for machining difficult to machine materials for machined surface modifications but the complexity of the process motivate further research since several process parameters are involved [3]. The objectives of this research study are to investigate the output responses including phase analysis, corrosion rate and surface sensitivity. Corrosion analysis is an important parameter indicator for industry and biomedical engineering because corrosion can affect the mechanical properties and biocompatibility of materials. The analysis of phase structure is to evaluate its change on mechanical properties od machined surface.

http://www.ijesrt.com© International Journal of Engineering Sciences & Research Technology
[124]



2. MATERIALS AND METHODS

Subheading

Peak current, ON-time, gap voltage, powder concentration are main variable parameters selected the process. Corrosion analysis was conducted using linear polarization resistance (LPR) test with three electrodes as shown in Figure 1, according to ASTM G3 – 89, ASTM G59 and ASTM G31.



Figure 1: LPR corrosion cell [4]

The use of a three-electrode potentiostat with a separate reference and counter electrode allows the potential and the current at the working electrode (WE) to be measured with little or no "interference" or "contribution" from the other electrodes. Potentiotat polarization measurements were used to study the effect of corrosion on Ti-6Al-4V.

The three electrodes are immersed in a simulated body fluid, Hank's solution as the electrolyte [5, 6]. The linear LPR method uses a small voltage of about ± 20 mV between the electrodes and the current is measured. The linear polarization resistance was done in 3 hours setting time with 6 reading points. Ecor (mV) was ranging from -10 mV to 10 mV with scan rate of 10mV/s and sample area of 0.33cm2.

In LPR method, a small current is applied (few μA) therefore, there will be change in electrode potential and current. The potentiostat will measure the potential current changes, plot the overvoltage and versus current and calculate the slope. The measured resistance is inversely related to the corrosion rate.

The titanium alloy specimens as received and machined using EDM and nano aluminum PMEDM processes were immersed in Hank's solution, the body fluid. The results of 3 hours linear polarization resistance with 6 reading points Ecor (mV) is ranging from -10 mV to 10 mV with scan rate of 10mV/s and sample area of 0.33cm2.

Phase analysis is a quantitative and qualitative non-destructive analysis which is to identify crystalline phases and orientation, to determine structural properties such as strain, grain size, phase composition and thermal expansion, to determine atomic arrangement. XRD model ME510LA2 of Rigaku Corporation Japan available at Universiti Teknologi PETRONAS provides detailed information on the crystallographic structure and physical properties of materials.

Due to thermal energy during PMEDM process, specimen was heated and melted. Some of melted material was flushed away and some was resolidfied and deposited on machined surface. Therefore, phase modifications of

http://www.ijesrt.com© International Journal of Engineering Sciences & Research Technology
[125]

Attribution 4.0 International Licen



machined surface can be examined. The effect of PMEDM process on phase changes was analyzed using X-ray diffraction (XRD) analysis. XRD provides detailed information on the crystallographic structure and physical properties of materials [7]. XRD measures the average distance between layers or rows of atom and the correspondent intensity. A pattern was obtained in terms of peak position, peak width and peak intensity which was compared with known standards in the Joint Committee on Powder Diffraction Standards (JCPDS) file.

Surface sensitivity analysis provides valuable quantitative and chemical information from the machined surface. Foreign materials from PMEDM process or from environment can be detected on machined surface. It is important to run surface sensitivity analysis to identify and quantify foreign materials. Surface sensitivity analysis in biomedical engineering is to examine if there is any contamination from foreign elements on machined surface [8]. XPS was used to characterize the surface chemical composition of machined surfaces. X-ray photoelectron (XPS) was used to characterize the surface chemical composition of some selected samples.

XPS principle is based on the X-ray photon effect that measures the kinetic energy (KE) of collected electrons. The kinetic energy of electron depends upon the photon energy (hv) and the binding energy (BE) of electron. By measuring the kinetic energy of the emitted electrons, it is possible to determine which elements are near a material's surface, their chemical states and the binding energy of electron [9].

3. RESULTS AND DISCUSSION

Corrosion Rate on Titanium Alloy

The titanium alloy specimens as received and machined using EDM and nano aluminum PMEDM processes were immersed in Hank's solution, the body fluid. The results of 3 hours linear polarization resistance with six (6) reading points and the average corrosion rates are presented in Figure 2.



Figure 2: Average corrosion of as received, EDM and PMEDM on Ti-6Al-4V

Ecor (mV) is ranging from -10 mV to 10 mV with scan rate of 10 mV/s and sample area of 0.33cm2. The maximum corrosion rate is found for the conventional EDM on Ti-6Al-4V alloy compared to PMEDM. The

http://www.ijesrt.com© International Journal of Engineering Sciences & Research Technology
[126]



Ο



Ο

electrochemical corrosion results show that corrosion resistance of the specimens which are machined by EDM is about two times more than the specimens which are machined using PMEDM.

Corrosion is induced during the EDM on titanium alloy but corrosion rate is reduced with PMEDM and slightly closed to as received titanium alloy. The average corrosion rate of PMEDM of 2.28266 x 10-4 mm/year on titanium alloy is closed to the accepted corrosion rate for implants which is about 2.5 x 10-4 mm/y [10] compared to the average corrosion rate of EDM of $3.98554 \times 10-4 \text{ mm/year}$. With added nano aluminum, the corrosion of nano aluminum PMEDM on titanium alloy is about 41.97% reduced as compared to the corrosion rate is due to improvement of machined surface irregularities and microstructure by powder-mixed dielectric fluid.

Phase Analysis of Nano Aluminum PMEDM on Titanium Alloy

XRD was used to examine changes on the nano aluminum PMEDM machined surface and compounds formed using Cu Ka radiation. Figure 3 presents the XRD patterns of as received and nano aluminum PMEDM on titanium alloy. It can be examined from Figure 3 that most of the peaks match titanium peaks. The XRD pattern of machined titanium alloy by copper tungsten electrode indicates the formation of TiO2, TiC and WO2 including titanium phases as it be seen in Figure 3. Electric sparks generated during machining decompose the kerosene dielectric fluid into C and H and is resulting in C deposited onto the machined surface. The transfer and deposit of alloying elements improve the machined surface properties particularly mechanical properties.



Figure 2: XRD pattern of nano aluminum PMEDM on titanium alloy

Surface Sensitivity of Nano Aluminum PMEDM on Titanium Alloy

X-ray photoelectron spectroscopy (XPS) techniques were used to analysis the chemical composition and oxidizing amounts of the outermost machined surface and evaluate if any contamination from foreign particles. Figure 4 presents the XPS of titanium element of as received, EDM machined surface PMEDM respectively. As analyzed from the Figure 4, the XPS analysis shows changes in peak position and intensity of elements on the machined surfaces. Ti-6Al-4V alloy revealed the Ti2p doublet peaks at 459.38eV and at 465.8eV attributed to the Ti-Ti bond. Machined surfaces of EDM and PMEDM on titanium alloy showed these peaks at slightly lower binding energies of 455.28eV, which were attributed to Ti-O due to the release of oxygen during EDM and PMEDM process. Titanium oxide layer forms on the titanium alloy implant surface protect the original metal from further oxidation and improves the osseointegration [11]. Ti-O is useful for the development of

http://www.ijesrt.com© International Journal of Engineering Sciences & Research Technology
[127]



ISSN: 2277-9655 Impact Factor: 5.164 CODEN: IJESS7

 \odot

[Nanimina* *et al.*, 9(2): February, 2020] ICTM Value: 3.00

biocompatible coatings of human implants [12]. The binding energy for EDM and PMEDM on titanium remains slightly the same for the various peaks.



Figure 2: XPS of EDM (IP=27A, ON=64µs, GapV=80V and PMEDM (IP=27A, ON=64µs, GapV=80V, Pcon=1g/l) on titanium alloy

The difference in EDM and PMEDM on titanium alloy is that PMEDM shows a pronounced intensity of photoelectron and it measures how much of Ti2p is at the surface. EDM and PMEDM processes induce surface modification due to erosion of sparks. The presence of carbon is from EDM and PMEDM processes which release carbon from the burning cycle. After XPS examinations of EDM and PMEDM machined surfaces, the results of the titanium alloy machined surface show difference in term of machined surface compositions. For PMEDM machine surface, the increase in the amount of nano aluminum and aluminum oxide by XPS measurements may be the proof of improvement of PMEDM machined and responsible for corrosion resistance compared to conventional EDM.

4. CONCLUSION

The use of aluminum powder mixed electrical discharge machining process improves the machined surface in terms of corrosion rate, phase structure and surface sensitivity. This improvement strengthens the properties of the titanium alloy grade due to transfer and deposit alloying elements. This transfer and deposit of the elements improve the osseointegration of the machined surface for the biomedical application.

REFERENCES

- F. Klocke, M. Schwade, A. Klink, D. Veselovac, and A. Kopp, "Influence of Electro Discharge Machining of Biodegradable Magnesium on the Biocompatibility," Procedia CIRP, vol. 5, pp. 88-93, 2013.
- [2] Y.-F. Tzeng and C.-Y. Lee, "Effects of powder characteristics on electrodischarge machining efficiency," The International Journal of advanced manufacturing technology, vol. 17, pp. 586-592, 2001.
- [3] A. Abdul-Rani, A. Nanimina, T. Ginta, T. Rao, and S. Pedapati, "APPLICATION OF NANO ALUMINUM IN MODIFIED EDM: PMEDM," 2006.
- [4] A. Standard, "G5-94 Standard Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements," Annual book of ASTM standards, vol. 3, 1994.
- [5] H. R. A. Bidhendi and M. Pouranvari, "Corrosion study of metallic biomaterials in simulated body fluid," Metalurgija-M J o M, vol. 17, pp. 13-22, 2011.

http://www.ijesrt.com© International Journal of Engineering Sciences & Research Technology
[128]



ISS	N: 22	77-9	9655
Impact	Facto	or: 5	.164
CO	DEN:	IJF	SS7

- [6] N. Zaveri, G. D. McEwen, R. Karpagavalli, and A. Zhou, "Biocorrosion studies of TiO2 nanoparticlecoated Ti–6Al–4V implant in simulated biofluids," Journal of Nanoparticle Research, vol. 12, pp. 1609-1623, 2010.
- [7] N. C. Miller, E. Cho, M. J. Junk, R. Gysel, C. Risko, D. Kim, et al., "Use of X-ray diffraction, molecular simulations, and spectroscopy to determine the molecular packing in a polymer-fullerene bimolecular crystal," Advanced Materials, vol. 24, pp. 6071-6079, 2012.
- [8] C. J. Powell and A. Jablonski, "Progress in quantitative surface analysis by X-ray photoelectron spectroscopy: Current status and perspectives," Journal of Electron Spectroscopy and Related Phenomena, vol. 178, pp. 331-346, 2010.
- [9] M. Mohai, "Development and applications of quantitative X-ray photoelectron spectroscopy," PhD Thesis, Institute of Materials and Environmental Chemistry, Chemical Research Center, Hungarian Academe of Science, 2005.
- [10] M. Mohanty, S. Baby, and K. Menon, "Spinal fixation device: A 6-year postimplantation study," Journal of biomaterials applications, vol. 18, pp. 109-121, 2003.
- [11] G. A. Castilho, M. D. Martins, and W. A. Macedo, "Surface characterization of titanium based dental implants," Brazilian Journal of Physics, vol. 36, pp. 1004-1008, 2006.
- [12] Z. Li, Y. Wu, and S. Miyake, "Metallic sputtering growth of high quality anatase phase TiO 2 films by inductively coupled plasma assisted DC reactive magnetron sputtering," Surface and Coatings Technology, vol. 203, pp. 3661-3668, 2009.

