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Nguyen Minh QUANG^{1*}, Nguyen Ngoc QUAN¹,
Nguyen Trong MAI¹, Le Thi Phuong THANH¹,
Nguyen Tien TUNG¹, Tran Ngoc TAN¹,
Ha Thanh HAI¹, Nguyen Duy TRINH¹

A NEW ENVIRONMENTALLY FRIENDLY CHEMICAL MECHANICAL POLISHING METHOD APPLIED FOR SURFACE FINISHING Ti-6Al-4V ALLOY

A new eco-friendly slurry has been developed for the chemical mechanical polishing process with a solution of malic acid, deionized water, and an oxidizing agent hydrogen peroxide (H₂O₂). The surface quality of Ti-6Al-4V workpieces with the proposed chemical mechanical polishing slurry with optimal parameters include oxidizers (H₂O₂), colloidal (SiO₂) slurry, and deionized water by weight 8%, 45%, and 47% respectively, the pH concentration is adjusted 4 through the malic acid content present in the slurry. Experimental results obtained with the proposed chemical mechanical polishing method show a more improved surface quality than previous studies when applying for polishing Ti-6Al-4V alloy. The developed chemical mechanical polishing method's polishing results under optimal conditions obtain an ultra-fine surface quality with Ra = 0.696 nm over a measuring area of 53×70 μm². X-ray photoelectron (XPS) and electrochemical measurements were used to study the chemical reaction mechanisms in the proposed chemical mechanical polishing process. The chemical mechanical polishing processes for the surface of the Ti-6Al-4V alloy workpiece with the H₂O₂ oxidizing agent showed high suitability with the reactants formed on the surface such as Ti, V, and Al oxide. With the proposed oxidant and the established chemical mechanical polishing slurry, the feasibility and surface quality of the super smooth Ti-6Al-4V workpiece formed after polishing were demonstrated. The established chemical mechanical polishing method shows high applicability in environmental protection and Ti-6Al-4V alloy ultra-precision machining industries.

1. INTRODUCTION

Among Ti alloys, Ti-6Al-4V alloy is the most common and also one of the most important engineering materials in Ti alloys [1–3]. Ti-6Al-4V alloy is widely used in many different industries such as medical equipment, sporting goods, petrochemical, aerospace, automotive, and many other industrial fields. Due to the relatively low density of Ti-6Al-4V alloy and high biocompatibility, chemical corrosion resistance and high strength [4, 5]. With outstanding fatigue strength at high temperatures, Ti-6Al-4V alloy has been used in gas turbines in the aerospace industry with heat resistance up to 600°C [6, 7]. With the parts subjected to temperature and cyclic rotational loads, the scratches caused by machining

¹ Faculty of Mechanical Engineering, Hanoi University of Industry, Hanoi city, Vietnam

* E-mail: nmqy1984@gmail.com; ngocquan1982002@gmail.com

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processes source and initiation of failure cracks. The study by [8] showed that more than 85% of the service life of Ti-6Al-4V alloy is affected by cyclic loading. Under the action of cyclic loads, the scratches left by the machining processes form fatigue cracks that grow larger and lead to failure during use. This process significantly reduces the performance of Ti-6Al-4V alloy. Fatigue cracks are less likely to occur when scratches on the surface of the Ti-6Al-4V alloy workpiece are less. In addition, Ti-6Al-4V alloy is also used in the fabrication of microelectromechanical systems (MEMS) [9, 10]. The higher the surface roughness, the more scratches are left on the workpiece surface by machining. When surface roughness is high, MEMS systems used by Ti alloy components typically reduce fatigue life, mechanical wear, cracking and wear. As the surface quality is improved, the durability and usability of the devices are significantly improved [11]. Ti-alloy devices working under high-performance conditions have exceptionally high requirements for surface quality, where the lowest level of roughness is achieved in the form of micrometers. The technological processes in machining Ti alloys to produce ultra-precision surface quality pose a significant challenge due to their unique physicochemical properties and intricate machining of Ti alloys.

Traditional polishing processes apply to the surface finishing Ti alloy materials such as mechanical polishing, chemical polishing and electrochemical polishing [12, 13]. Mechanical polishing processes are commonly used by abrasives of aluminium oxide (Al_2O_3) and silicon carbide (SiC). The average surface quality obtained by mechanical polishing processes with surface roughness according to Ra at 300 nm [14, 15]. Electrochemical and chemical polishing methods are carried out to further improve the surface quality by using oxidizing hydrogen peroxide (H_2O_2), colloidal silica (SiO_2) and chemicals including hydrochloric acid (HClO_4), hydrochloric acid (HCl), hydrofluoric acid (HF), sulfuric acid (H_2SO_4) and cyanide [16]. When applying different chemical using for electrochemical and chemical polishing methods, the surface roughness according to Ra can be improved to 90 and 7 nm [17]. Electrochemical polishing processes applied to Ti alloys with chemicals used ethylene glycol and ethyl alcohol in hydrodynamic atmospheres after a 45 min polishing time yielded a surface quality of 1.9 nm over the measured area $9 \times 9 \mu\text{m}^2$ [18]. Combining the advantages of the mechanical polishing process with the chemically reactive surfaces, the electromechanical polishing model is established in the Ti alloy polishing processes to improve the surface quality further [19]. After chemical mechanical polishing (CMP) with established chemicals including oxidizing agent H_2O_2 , silica slurry, and disodium ethylenediaminetetraacetic acid in a pH medium at 9.5, average surface roughness was obtained at 1.899 nm with a measured area of $5 \times 5 \mu\text{m}^2$ by atomic force microscope (AFM) on the surface of Ti alloy workpiece [20]. Another solution was performed with CMP at a pH of 9 together with oxidant H_2O_2 and emulsifiable oils giving a surface quality according to Ra up to 0.96 nm obtained after polishing and cleaning the Ti alloy with ethanol [19]. However, the polishing mechanism and measuring area are unclearly mentioned.

The above analysis shows that with the mechanical polishing method to remove deep scratches and effectively used by abrasive particles with large particle sizes, the surface roughness obtained with Ra is achieved at hundreds of nanometers. With chemical and electrochemical polishing processes surface quality and root mean square (RMS) are obtained at tens of nanometers. Although electrochemical and chemical methods enhance surface quality, the chemicals commonly used are toxic or hazardous to humans and the environment.

In addition, the electrochemical and chemical polishing processes increase costs due to the time spent in storing, transporting, operating, and handling chemicals and cyanides. Besides, the measuring area at 5×5 and $9 \times 9 \mu\text{m}^2$ used in the CMP polishing process is much smaller than the area $53 \times 70 \mu\text{m}^2$ using in industry metrology. Surface roughness is higher when the measuring area is as large as the surfaces to be measured in industrial production $53 \times 70 \mu\text{m}^2$, with surfaces used in industry it is difficult to machine surfaces smaller than nanometers. Traditional CMP polishing processes also use solvents because strong acids, detergents and industrial salts harm the environment and people. Therefore, creating an environmentally friendly CMP polishing compound is necessary to apply to the Ti and Ti alloy processing industry.

To overcome the analytical characteristics above, this study refers to a new approach to creating a CMP by environmentally friendly mixtures with an RMS and surface roughness smaller than nanometers. Along with that, the results of X-ray spectroscopy analysis and electrochemical analysis are proposed to find out the polishing mechanism of the developed CMP model.

2. DESCRIPTION AND EXPERIMENTAL SETUP

A new eco-friendly slurry has been developed for the CMP polishing process with a solution of malic acid, deionized water and an oxidizing agent H_2O_2 . The SiO_2 slurry was established with a nanometer size and contained 45% by weight and an oxidizing agent solution with 18% by weight H_2O_2 . The pH concentration is adjusted through the malic acid content present in the mixture.

Ti-6Al-4V alloy workpieces are set to a diameter of 12 mm in size with 10 mm in height. The workpieces were carried out on a PLAN-SMART polishing machine, with a SiC abrasive aqueous solution with particle sizes of 600, 1000, 1500 and 3000. Workpieces are fixed on jigs, and SiC sandpaper is mounted on 200 mm diameter Al alloy discs. During polishing, the SiC polishing plates rotate, and the workpieces are 90 and 60 rpm, respectively. The polishing force acting on the workpiece corresponds to the level of 30 N, and deionized water plays the role of rinse water. After polishing the workpiece and sandpaper are cleaned with deionized water. After that, non-woven polishing cloth is substituted for sandpaper plate. The new eco-friendly polishing slurry carries out the CMP polishing processes on the same polishing machine. In polishing processes with CMP suggested, the rotation speed of the polishing plate and workpiece is 55 and 65 rpm respectively, the polishing force is 50 N and is carried out for 30 mins. After polishing, the workpieces are cleaned with deionized water and a dryer.

The polished workpieces were observed surface morphology by optical microscope Olympus - MX 40. Surface roughness was performed on ZYGO 7100 non-contact surface roughness measuring device with a measuring area of $70 \times 53 \mu\text{m}^2$. The three-electrode electrochemical measurement system used in electrochemical measurements, Ti workpiece reference electrodes, platinum electrodes and Ag/AgCl auxiliary electrodes along with a potential polarization curve measured by the instrument PARSTAT 2273. With a scan rate of 1 mV/s and a variable scan range in the 0.25 V to 1.6 V region. An Ultra DLD-AXIS spectrophotometer obtained XPS spectra. Nanoparticles in SiO_2 slurry were investigated by transmission electron microscope FEI Tecnai F20.

3. RESULTS AND DISCUSSION

The results of transmission electron microscopy (TEM) analysis with Silica slurry particles are shown in Fig. 1. After polishing by the CMP proposed applied to the Ti-6Al-4V workpiece surfaces, the resulting surface morphology as shown in Fig. 2, with the pH concentrations in the CMP slurry described on Table 1. The polishing processes are performed on the PLAN-SMART polishing machine. Table 2 shows the CMP process parameters. Experimental procedures were performed on a PLAN-SMART polishing machine, as shown in Fig. 2. The surface of the Ti-6Al-4V workpiece obtained after polishing with the CMP proposed shows that the chemicals affect the polished surface quality. As shown in Figs. 3a and 3b, the surfaces improved and approached the mirror surface according to different pH values adjusted by different malic acid concentrations. The illustrations obtained in Fig. 2d are shown on the surface of the workpiece when the pH value is 10, a more improved surface with a pH value of 7 as shown in Fig. 3c. The NaOH content is used to raise the pH value. The surfaces obtained in Figs. 3a and 3b show smooth surfaces and almost no indentation is obtained, for which the pH value should be used to correspond to level 4.

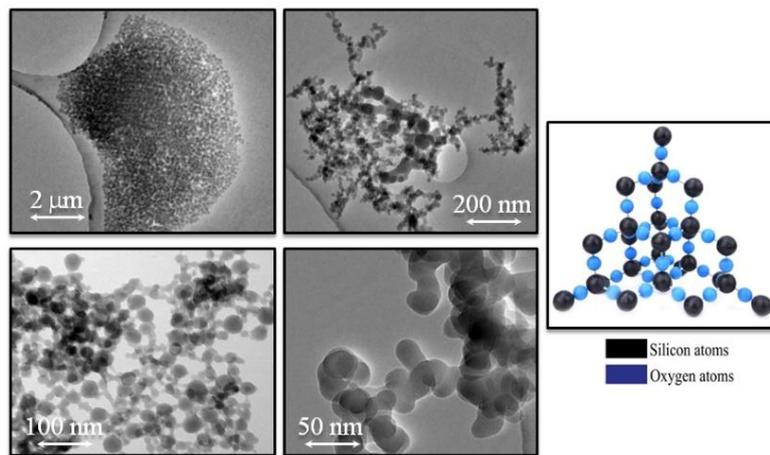


Fig. 1. Molecular structure of SiO₂ and TEM analysis results

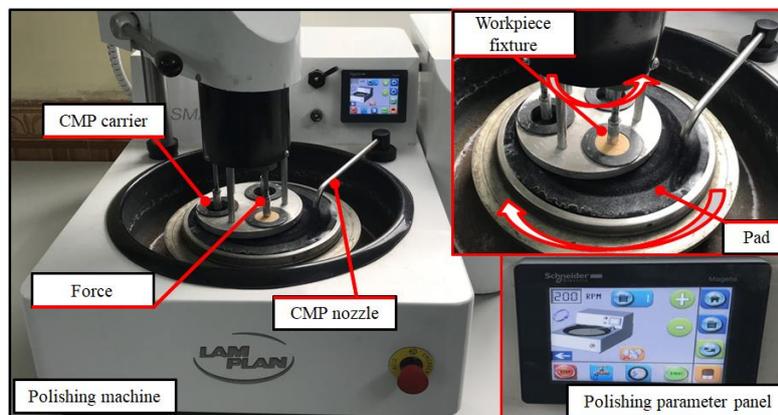


Fig. 2. Image of CMP experiment equipment

The surface quality of the Ti-6Al-4V alloy workpiece after polishing by CMP as shown in Fig. 4 with the percentage of H₂O₂ oxidizing agent by weight is 0; 5; 8 and 15% respectively, as shown in Table 3. In the absence of an oxidizing agent, the obtained surface consists of many protrusions as shown in Fig. 4a. When the oxidant content was raised to 5%, the surface quality was significantly improved, almost no dents were seen, and only a few black dots and minor scratches appeared as shown in the Fig. 4b. With the oxidant content increased to 8% a smooth surface and showing no scratches were obtained. While if continue to increase the oxidant content to 15% after polishing the workpiece appears small protrusions as depicted in Fig. 4d. The above analysis shows that the CMP slurry concentration set as listed in Table 4 gives the best surface quality.

The surface quality of Ti-4Al-4V workpieces polished with the proposed CMP slurry with parameters as shown in Table 4 is shown in Fig. 5. The surface roughness according to Ra and RMS from the peaks and valleys (PV) obtained on the surface of the workpiece as depicted in Fig. 5a is 1.490, 2.363 and 65.997 nm, respectively. For other workpieces, the surface quality obtained in Fig. 5b shows that the parameters Ra, RMS and PV are determined by 1.368, 2.179 and 53.203 nm, respectively. It can be seen that the surface quality according to Ra and RMS obtained by polishing results on two different workpieces is smaller than a nanometer with a measuring area of 53×70 μm². The surface over a measured area of 53×70 μm² with Ra = 0.696 nm was obtained on the surface of Ti alloy, the surface quality is better than in the previous study [21].

Table 1. Chemical composition in the proposed CMP

Characteristics	Oxidizers H ₂ O ₂	Colloidal SiO ₂ slurry	Deionized water	pH level (Malic acid)
Parameter	8% by weight	45% by weight	47% by weight	3.5; 4; 7; 10

Table 2. Machining parameters when polishing on the PLAN-SMART machine

Technological	process Pad composition	Rotation speed (rpm)	Force (N)	CMP flow speed (ml/min)
Polishing	Polyurethane IC1000	90	25	8

Table 3. Change of H₂O₂ oxidant concentration in CMP slurry developed

Characteristics	Oxidizers H ₂ O ₂	Colloidal SiO ₂ slurry	Deionized water	pH level (Malic acid)
Parameter	0; 5; 8; 15 % by weight	45% by weight	55; 50; 47; 40% by weight	4

Table 4. Optimal CMP composition in Ti-6Al-4V alloy polishing

Characteristics	Oxidizers H ₂ O ₂	Colloidal SiO ₂ slurry	Deionized water	pH level (Malic acid)
Parameter	8% by weight	45% by weight	47% by weight	4

The proposed total polishing time in the CMP polishing processes is 30 min, and the machining time is smaller than the electrochemical machining time of 45 min [22]. In previous studies, after polishing was cleaned with ethanol [23].

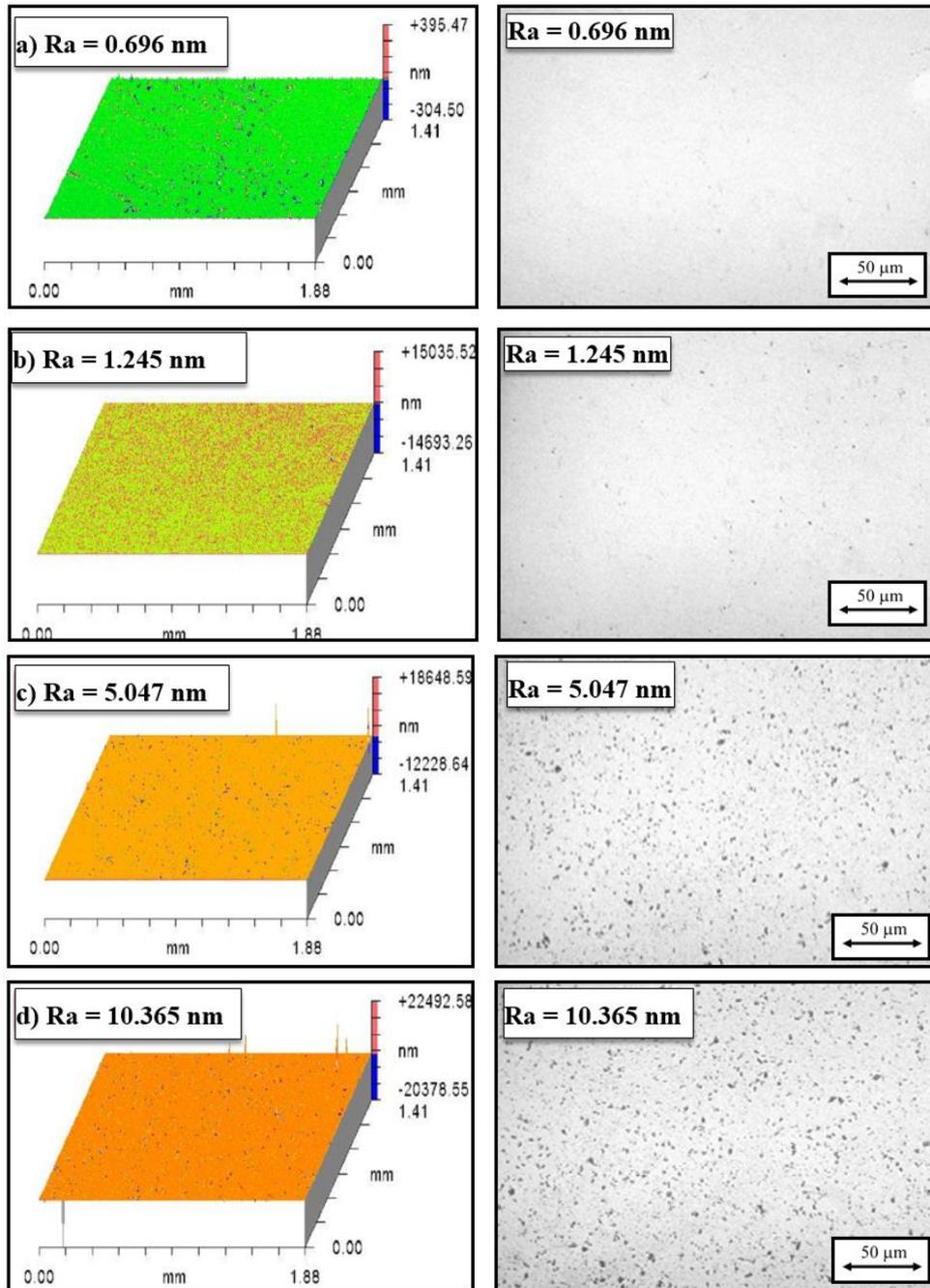


Fig. 3. Surface morphology of Ti-6Al-4V workpiece after polishing with the CMP slurry proposed with different pH concentrations 0; 5; 8 and 15%

With the proposed eco-friendly CMP, the Ti-6Al-4V workpieces do not have to be cleaned after polishing. The eco-friendly proposed polishing mix consists of malic acid, an H_2O_2 oxidizing agent, SiO_2 slurry, and deionized water. The malic acid in the food and medical industries exists in natural and environmentally friendly fruits. With the origin distributed in nature with the main components of rock, quartz and existing in the earth's crust, SiO_2 is therefore environmentally friendly. The substance is used environmentally friendly in food and disinfect human skin, with H_2O_2 used as an oxidizing agent that can slowly decompose into oxygen and water in the air.

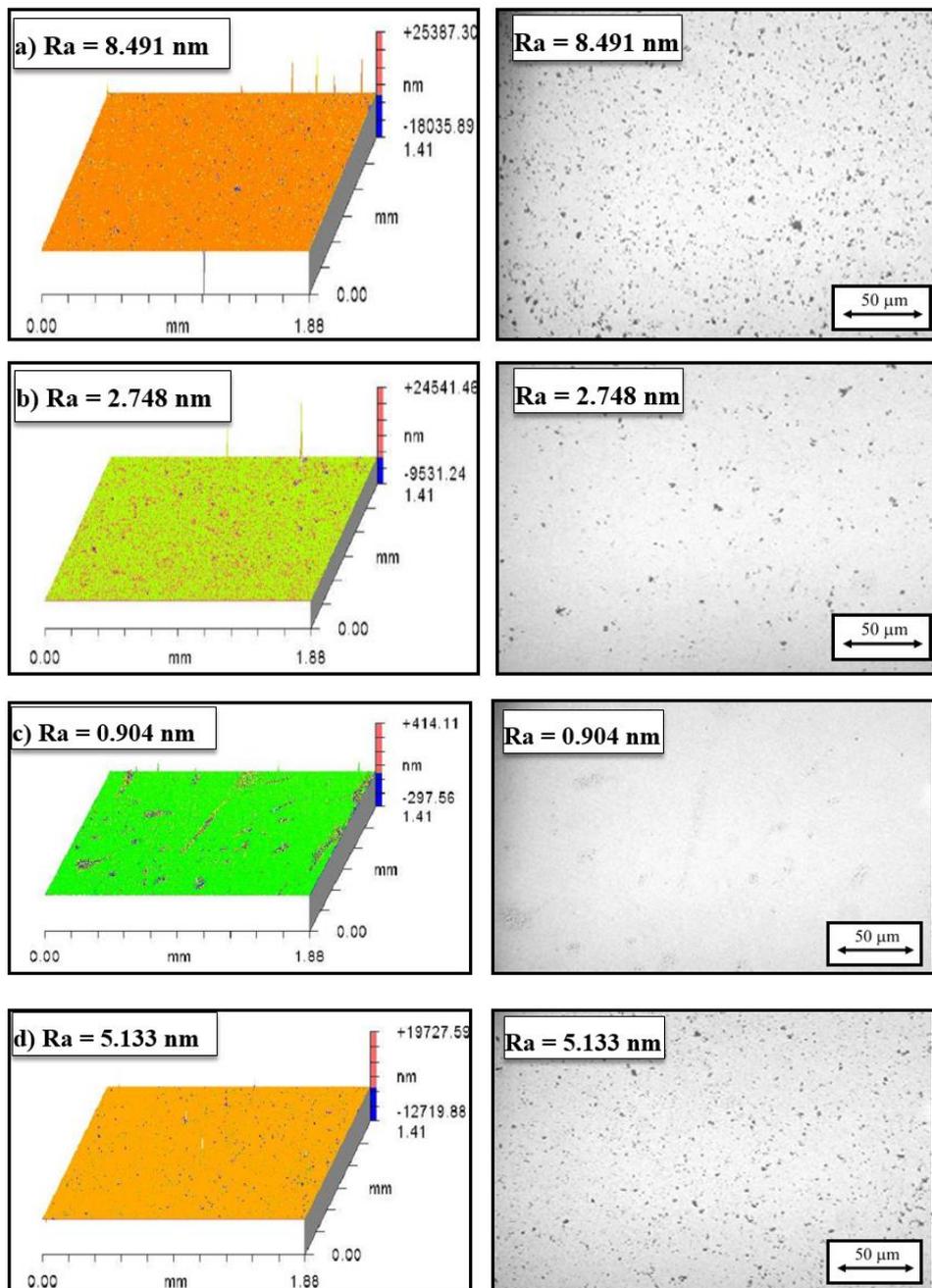


Fig. 4. Surface morphology of Ti-6Al-4V workpiece when polishing with different concentrations of oxidizing agents

All components in CMP developed environmentally friendly, so CMP developed is environmentally friendly. The process of adjusting the content of oxidizing agent H_2O_2 and the pH adjustment is selected as the most suitable for the polishing process of Ti-6Al-4V alloy. The surface quality obtained with surface roughness and PV measured over an area of $53 \times 70 \mu m^2$ achieved 1.368 and 53.203 nm, respectively. It is interesting to note that the surface roughness obtained with sub-nanometer roughness compared with the roughness achieved by the AFM processes. A new approach to using CMP environmentally friendly benefits Ti-alloy machining industry and industry polishing. Based on the attractiveness

of the proposed model, the proposed CMP polishing mechanisms are analyzed by the XPS spectrum and electrodynamic polarization curve.

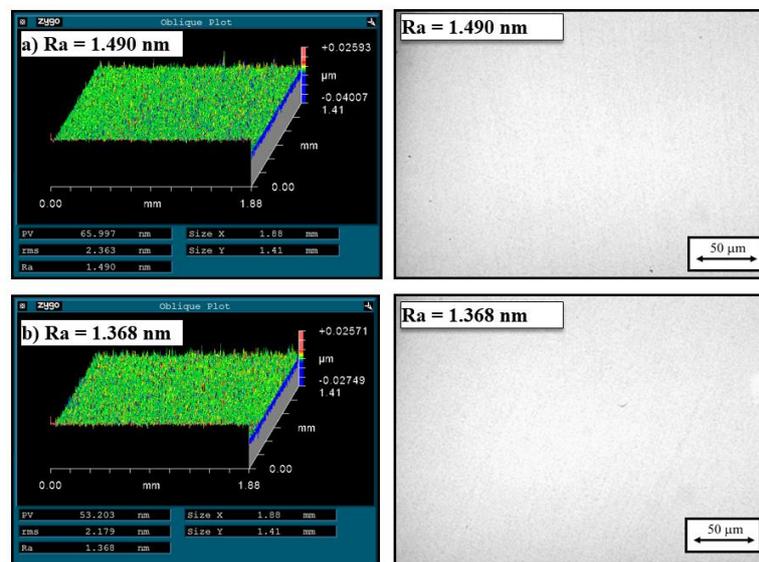


Fig. 5. The surface quality of Ti-6Al-4V workpiece after polishing by CMP in Table 4

The CMP mixtures were developed in Tables 1 and 2 as dynamic potential polarization curves on the surface of the Ti-6Al-4V workpiece as depicted in Fig. 6. The analysis results in Fig. 6a show that the corrosion current (I_{corr}) and the corrosion potential (E_{corr}) with the pH value of 3.5; 4; 7 and 10 correspond to 3.8; 1.7; 0.6; 0.59 μA ; and 210; 253; 63; -136 mV, respectively. As the pH increases, the monotonous corrosion current value decreases, indicating a decrease in the corrosion rate. The corrosion potential corresponds to the passivation layer and the thicker layer, the better the polishing surface quality. Analytical results show that the highest potential value corresponds to a pH of 4. Therefore, the best surface quality is obtained with a pH of 4, the analytical results are similar to those shown in Fig. 3b. The roughest Ti-6Al-4V workpiece surface obtained at a pH value of 10 together with the lowest corrosion potential obtained shows a highly consistent result corresponding to Fig. 2d. To improve the surface quality as well as the ability to remove materials, the authors used H_2O_2 oxidizing agent for CMP slurry. With the advantage of strong oxidizing ability of H_2O_2 and containing no ions, it is not harmful to the environment and humans. The environmentally friendly CMP slurry is therefore developed with the oxidizing agent H_2O_2 . Figure 6b shows the results of the analysis of the change of the I_{corr} electrodynamic polarization curves with the increasing content of the H_2O_2 oxidizing agent solution at 0, 5, 8 and 15% by weight, respectively. 0.0016, 0.76, 1.7 and 4.4 μA correspond to E_{corr} at 46, 118, 253 and 192 mV, respectively. The results clearly show that the strong oxidizing capacity of the H_2O_2 solution compared with the mixture without H_2O_2 is shown by I_{corr} to increase by two to three orders of magnitude [24]. The thinnest and thickest surface-formed passivation layer on the Ti-6Al-4V workpiece surface was obtained with the lowest and highest E_{corr} with 0 and 8% oxidant content, respectively. The obtained results are consistent with the description in Figs. 4a and c, respectively.

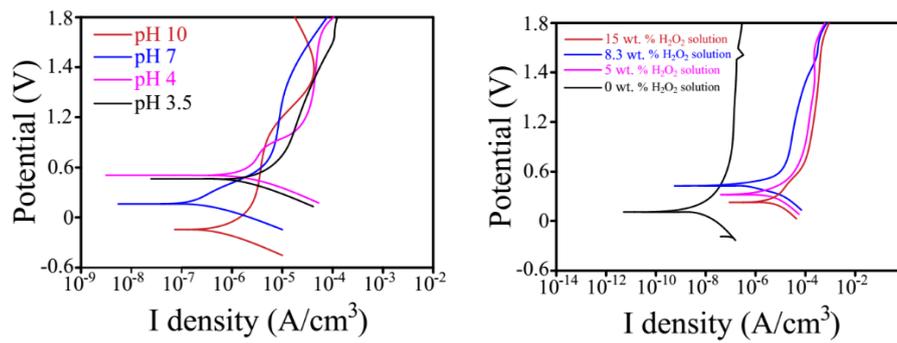


Fig. 6. Dynamic potential polarization curve against pH and oxidizing agent H_2O_2

The results of XPS analysis when polishing by the proposed CMP mixture for the most optimal case give the results shown in Fig. 7 with the lists shown in Table 5. The chemical composition of O 2p, C 1s and N 1s corresponding to the atomic percentage is 46.12; 33.42 and 1.34%. The C 1s of element C has an energy level of 284.8 eV that corresponds to the organic matter malic acid and the absorption of CO_2 in the air. The O 2p component is generated from the oxidation of the Al and Ti elements present in the Ti-6Al-4V alloy and the O in the air and in organic substances. The N 1s composition corresponds to the nitrogen content in the air and organic substances. After being corroded the chemical composition of O 2p, C 1s and N 1s decreased to 17.56; 4.56 and 0% due to the reduction of organic matter, oxidation. The composition V 2p, Ti 2p, Al 2p and increased to 1.65; 69.36 and 6.87 from 0%; 15.95; and 3.15, respectively.

Table 5. Ti-6Al-4V surface after polishing with CMP suggested

Characteristics	O 2p	Ti 2p	C 1s	Al 2p	N 1s	V 2p
At 10 nm depth	46.12	15.95	33.42	3.25	1.34	0
Surface layer	17.56	69.36	4.56	6.87	0	1.65

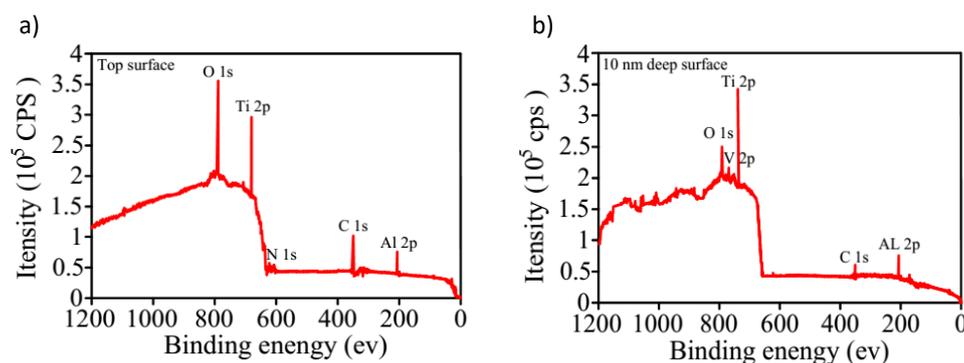


Fig. 7. XPS analysis results of Ti-6Al-4V workpiece at the outermost surface layer (a) and 10 nm depth (b) after polishing by the proposed CMP slurry as described in Table 4

XPS spectra of elements Al, Ti and V in Ti-6Al-4V alloy at the position on the outermost surface layer and a depth of 10 nm after polishing by the proposed CMP as depicted in Fig. 8. Figure 8a shows that the energy levels are 453.3 eV, 458.6 eV and 464.3 eV corresponding

to the peaks of Ti0 2p3/2, Ti4+ 2p3/2 and Ti4+ 2p1/2 [25], respectively. The peaks at a depth of 10 nm correspond to the energies 453.3 eV and 459.4 eV representing the peaks of Ti0 2p3/2 and Ti0 2p1/2 [26, 27]. The results in Fig. 8c show that the maximum energy levels are 511.6 eV, 515.4 and 523 for the elements V0 2p3/2, V4+ 2p3/2 and V4+ 2p1/2 [28, 29], respectively. The energy peaks with a depth of 10 nm are represented by 511.5 eV and 519.3 eV, for V0 2p3/2 and V0 2p1/2 [30], respectively.

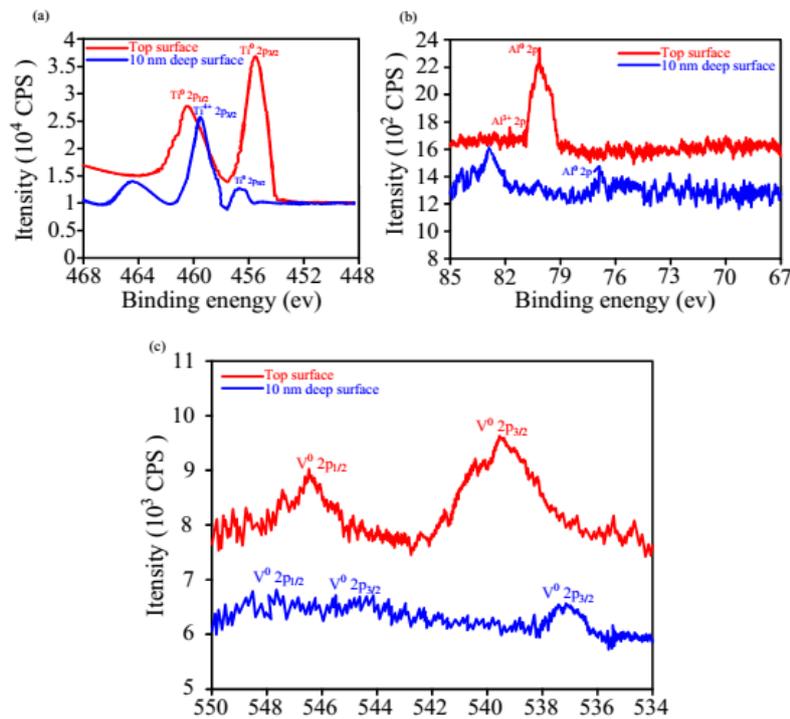


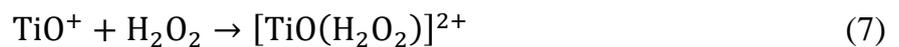
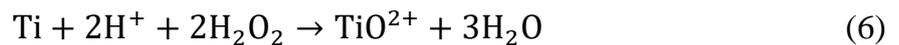
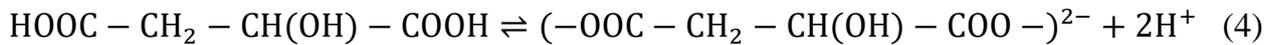
Fig. 8. XPS analysis results at 10 nm depth and on the outermost surface when polishing Ti-6Al-4V alloy proposed by CMP

Analytical results after polishing Ti-6Al-4V alloy by CMP proposed that on the surface of the workpiece, oxide layers of Ti, Al and V elements are formed corresponding to TiO₂, Al₂O₃, and VO₂. Thus, in the CMP polishing process, it is proposed that the elements present in the alloy are oxidized, and the O element exists on the surface after polishing as shown in Fig. 6a. The results are also consistent with the corrosion flow after polishing by CMP as shown in Fig. 5b and Table 4. When the H₂O₂ oxidizer content is added, the process creates an oxidation layer on the surface of Ti-6Al-4V alloy with a high oxidation rate of H₂O₂. The process creates an oxidation layer on the surface of Ti-6Al-4V alloy with a high oxidation rate of H₂O₂. Fig. 8 shows Ti, Al and V elements in the surface layer with a depth of 10 nm. In addition, when the pH increases from 3.5 to 10 the magnitude only increase by one order as shown in Fig. 5a. The results show that the corrosivity of CMP for Ti-6Al-4V alloy is dominant, and malic acid plays a role in the corrosion process. The proposed chemical reactions in the proposed CMP polishing process from the optimal mixture developed from the results of the electrochemical and XPS analysis can be described as follows:





Malic acid participates in the corrosion process and supports H_2O_2 to participate in the reactions in the CMP polishing process as described below:



Under the mechanical force of the buffer, the reactants were dissolved in the developed CMP slurry, thereby obtaining the super-smooth surface of the Ti-6Al-4V alloy.

4. CONCLUSIONS

This work develops an environmentally friendly chemical mechanical polishing slurry based on hydrogen peroxide oxidant (H_2O_2), malic acid, deionized water, and colloidal suspension (SiO_2). XPS analysis and electrochemical measurements were used to study the chemical reaction mechanisms in the proposed chemical mechanical polishing process. The proposed oxidant and an established chemical-mechanical polishing suspension, the feasibility, and the ultra-fine surface of the Ti-6Al-4V alloy workpiece formed after polishing has been demonstrated. The main conclusions of the study are presented as follows:

- The XPS analysis when polishing by the proposed CMP with optimal case gives the chemical composition of O 2p, C 1s, and N 1s corresponding to the atomic percentage is 46.12; 33.42, and 1.34%. After being corroded the chemical composition of O 2p, C 1s and N 1s decreased to 17.56; 4.56, and 0% due to the reduction of organic matter and oxidation. The composition V 2p, Ti 2p, Al 2p and increased to 1.65; 69.36, and 6.87 from 0%; 15.95; and 3.15, respectively. Analytical results after polishing Ti-6Al-4V alloy by CMP proposed that on the surface of the workpiece, oxide layers of Ti, Al, and V elements are formed corresponding to TiO_2 , Al_2O_3 , and VO_2 . When the H_2O_2 oxidizer content is added, the process creates an oxidation layer on the surface of the Ti-6Al-4V alloy with a high oxidation rate of H_2O_2 . The proposed chemical reactions in the proposed CMP polishing process from the optimal mixture developed from the results of the electrochemical and XPS analysis.
- The proposed CMP experiments were carried out with a polishing mixture established from environmentally friendly substances including malic acid, H_2O_2 , SiO_2 slurry, and deionized water. With H_2O_2 in natural conditions, it will decompose into oxygen and water, along with SiO_2 which is distributed and available in nature. Malic acid exists in edible fruits, creating a polishing mixture with environmentally friendly CMP. After polishing, the workpieces were cleaned with deionized water and a dryer. The polishing and cleaning processes in the study were all environmentally friendly while still producing the best corrosion resistance and surface quality obtained with Ti-6Al-4V alloy workpieces.

- The result after 30 mins of polishing the Ti-6Al-4V alloy workpiece by the CMP proposed method under optimal conditions, obtained an ultra-smooth surface with $R_a = 0.696$ nm over large measuring areas such as industrial production surfaces $53 \times 70 \mu\text{m}^2$ which previous studies have not achieved. The proposed CMP slurry with optimal parameters including H_2O_2 , SiO_2 slurry, and deionized water by weight is 8%, 45%, and 47%, respectively, the concentration pH was adjusted to 4 through the malic acid content present in the suspension. With the proposed method, which has created super-smooth surface quality for Ti-6Al-4V alloy, the proposed model is beneficial to humans and the environment as well as applied to industrial production.

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