



Tribological Behaviour of Strained Harden Commercially Pure Titanium with Nano Structured Diamond like Coatings



Song Jeng Huang¹, Aqeel Abbas^{1*}, Dawit Bogale Alemayehu¹, Tsun Yueh yang¹, Vladimir Semenov² and Manohoran Thangavel¹

¹Department of Mechanical Engineering, National Taiwan University of Science and Technology, Taiwan

²Department of Mechanical Engineering, Ufa State Aviation Technical University, Russia

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*Corresponding author: Aqeel Abbas, Department of Mechanical Engineering, National Taiwan University of Science and Technology, No. 43, Keelung Rd., Sec. 4, Da'an Dist., Taipei, Taiwan, R.O.C., Email: Engr:aqeel14@gmail.com

Abstract

In this research, the tribological tests for coarse grained and variant surface prepared commercially pure Titanium (CP Ti) grade 4 were performed under plastic friction and elastic contact conditions. Coefficients of frictions were calculated for samples with and without coating in commercially pure Titanium-Tool Steel friction pair. After sever plastic deformation via equal channel processing, mechanical testing was performed for commercially pure Titanium with ultra-fine grains. Metallographic studies of coating applications were conducted for samples with ultra-fine grained structure. From the results it can be concluded that commercially pure Titanium grade 4 with Multi arc oxidation coatings presents less coefficient of friction and remains almost constant with increasing no of cycles. It was observed that strength of commercially pure Titanium is increased by 35% which corresponds to VT6 and after Sever Plastic Deformation through Equal Channel Angular Processing grain size is reduced to 300nm.

Keywords: SPD processing; Zones; Tribometer; Properties; Chromium steel

Abbreviations: UFG: Ultra Fine Grain; SPD: Sever Plastic Deformation; ECAP; Equal Channel Angular Pressing; CP Ti: Commercially Pure Titanium

Introduction

At the present time, it is observed that interest in studies related to strength enhancing in metals has been increased. The ultra-fine grain (UFG) structure can be obtained by sever plastic deformation (SPD) processing [1], specifically by equal-channel angular pressing (ECAP) [2,3] and ECAP-Conform [4]. Development of industrial SPD processes is hindered, in particular, by the lack of systematic studies related to the preparation of billet surface prior to SPD processing.

The feature of the SPD processing via ECAP-Conform is that in the deformation site there are distinct tension zones and compression zones, as well as stick zones and slip zones. Thus, in order to increase the efficiency of the strain hardening process in metals via ECAP-Conform and produce good-quality high-strength semi-products, it is required to fulfill two competing requirements: to provide a low friction coefficient in slip zones and, at the same time, to preserve the integrity of the billet in tension zones and a high friction coefficient in such zones.

The present knowledge in the area of tribology involves a number of fundamental theoretical and experimental regularities enabling solution of applied tasks related to dry and boundary friction, aerodynamic, hydrodynamic and elasto hydrodynamic lubrication, which are realized in the conditions of metals processing [5-8].

Materials and Methods

Commercially pure Titanium (CP Ti) grade 4 was selected as basic element for strain hardening process and coating of Micro arc oxidation and Ion-plasma spraying was developed for surface preparation.

Coefficient of friction under plastic condition

The initial samples of Grade 4 Titanium were settled in ring shape with dimension of outer diameter, $D_0 = 18\text{mm}$; inner diameter, $d_0 = 9\text{mm}$; height, $H_0 = 6\text{mm}$. As the under layer coating, an aqueous salt composition based on sodium tetra

fluoroborate was used on surface of samples. The ring-shaped samples were strained on flat anvils with a surface finish not lower than 0.25µm. The upsetting ratio was

50%. After deformation, [9,10] the coefficient of external friction is determined according to the nomogram.

Coefficient of friction under Elastic condition

The reciprocating movement was used to evaluate the friction coefficient in the pair «Grade 4 CP Ti - Fe-1.5Cr-1.0C chromium steel» on a Nanovea TRB-1 tribometer with spherical *diameter* of 3mm. parallelepiped-shaped samples with a length of 25mm and a section of 9.5 x 9.5mm were used. The displacement amplitude under a normal load of 5N was 20mm under 5000 cycles. The total friction path was 200m under a displacement speed of 30 cycles per min. The testing results were processed on a computer, using the appropriate software. Before and after the tribological tests, the micro hardness $H\mu$ of the studied CP Ti samples was tested on a *Micromet*-5101 device under a load of 0.98 N with a holding time under load of 15s.

The adhesion of the coatings was analyzed using a Nanovea Nano scratch tester. The testing conditions were as follows: the scratching speed was 0.77mm/min; the line length was 3mm; the initial and maximum loads were 5 and 200mN, respectively. The rate of load increase was 50N/min. A conical diamond indenter with an apex angle of 120° and an apex radius of 20µm was used.

SPD processing of initial samples

To produce a high-strength material with a UFG structure, a six-cycle deformation processing was conducted. After getting the UFG structure, mechanical tests were performed by measuring micro hardness across the billet's section in 15 points. The load was 1 N and the holding time was 10s. The method of micro hardness measurement provides a rather full picture to evaluate the mechanical properties of the tested material, in particular, its strength.

Results and Discussion

Coefficient of friction under plastic condition

The variation in internal *diameter* of the ring during the upsetting with flat anvils serves as an indicator of the value of frictional forces. When the coefficient of external friction (f) tends towards zero, the internal *diameter* increases; when the coefficient of external friction tends to the maximum value (≈ 0.57), the internal *diameter* decreases; and when the friction coefficient is $f=0.05-0.06$, the internal *diameter* remains practically unchanged.

When determining the coefficient of external friction under plastic deformation of Grade 4 CP Ti, it was found that under the deformation of a ring-shaped sample without any lubricant, the average value of the friction coefficient amounted to 0.35, and with under layer coating in combination with a lubricant based on solid fine-dispersed filler, it amounted to 0.25. Also, in the

case of «dry friction» an adhesive interaction between the tool (anvils) and the billet's material was noted.

Coefficient of friction under elastic condition

The initial roughness of the contacting surfaces of the tested samples and the indenter for both procedures was 0.06-0.16µm in the Ra scale. The results of tribological tests in accordance with the first principle in the following friction pairs:

«Grade 4 CP Ti without coating - Fe-1.5Cr-1.0C chromium steel»;

«Grade 4 CP Ti with an IPS coating - Fe-1.5Cr-1.0C chromium steel»;

«Grade 4 CP Ti with MAO coating - Fe-1.5Cr-1.0C chromium steel», is shown in Figure 1. The investigated material was in the as-annealed state and had a coarse-grained structure [11].

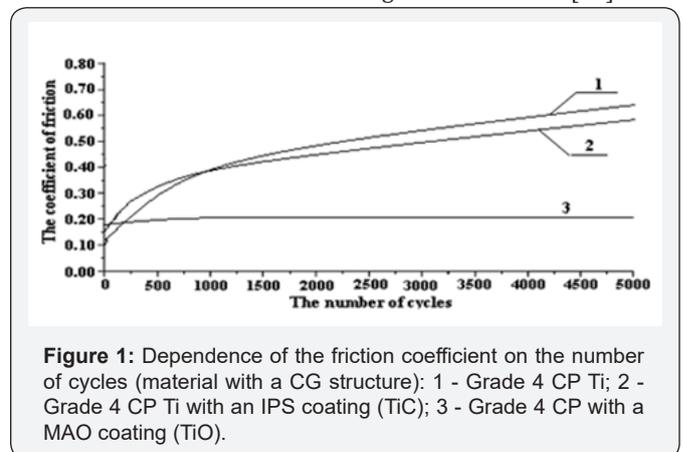


Figure 1: Dependence of the friction coefficient on the number of cycles (material with a CG structure): 1 - Grade 4 CP Ti; 2 - Grade 4 CP Ti with an IPS coating (TiC); 3 - Grade 4 CP Ti with a MAO coating (TiO).

As it can be seen from the presented diagram, the friction coefficient values for the annealed samples from CP Ti (curve 1) are higher than those for the samples with coatings (curves 2 and 3). It is also noted that in the course of testing of all the samples, the onset of the regime close to the steady-state one is in the range of 750 to 1000 cycles. It is visible that the running-in stage for the samples without coating (curve 1) has a flatter character. This indicates a smoother change of the friction regime when reaching the steady-state regime. It was found that for the uncoated CP Ti samples with a CG structure (curve 1) and the samples with a TiC coating applied by ion-plasma spraying (curve 2) the friction coefficient has the largest values in the investigated friction pair. Within the confidence interval, the variation dynamics and quantitative values of the friction coefficients presented in Figure 1 by curves 1 and 2 differ insignificantly. Evidently, during the testing of the sample with a TiC coating on a tribometer in accordance with the selected principle,

A rapid abrasion of the coating is observed.

Figure 2 shows the friction tracks after testing of the samples with different variants of surface preparation. The smallest values of the friction coefficient are observed for the sample with

a surface treated by MAO, producing TiO titanium oxide (curve 3). From analysis of Figure 2 it follows that there is observed a similarity between the friction tracks on the samples from Grade 4 CP Ti without coating (a) and with ion-plasma coating (TiC) (b). This is evidently related to the fact that in the accepted conditions of the physical experiment, there occurs intensive abrasion of the coating and exposure of the substrate material - Grade 4 CP Ti. The friction track displayed in (Figure 2), c and formed on the sample with a TiO coating produced by MAO represents an even trace without any disruptions. This indicates the preservation of integrity of the studied coating and its high

strength. From analysis of Figure 2 it is observed a similarity between the friction tracks on the samples from Grade 4 CP Ti without coating (a) and with ion-plasma coating (TiC) (b). This is evidently related to the fact that in accepted conditions of the physical experiment, it occurs intensive abrasion of the coating and exposure of the substrate material - Grade 4 CP Ti. The friction track displayed in Figure 2, c and formed on the sample with a TiO coating produced by MAO represents an even trace without any disruptions. This indicates the preservation of integrity of the studied coating and its high strength.

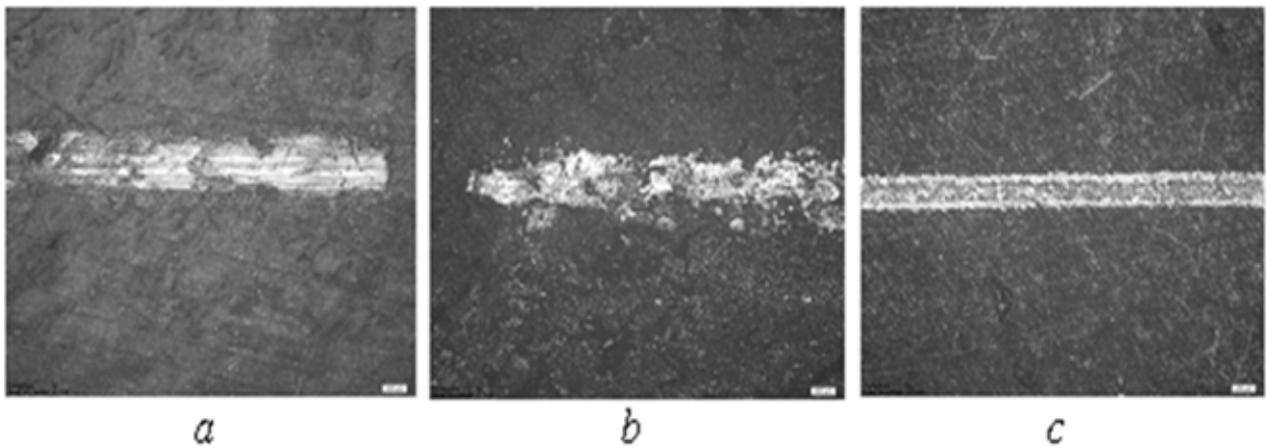


Figure 2: Friction tracks obtained during the tribological testing in accordance with the principle involving linear reciprocating movement:
 a. Grade 4 CP Ti without coating;
 b. Grade 4 CP Ti with an IPS coating (TiC);
 c. Grade 4 CP Ti with a MAO coating (TiO).

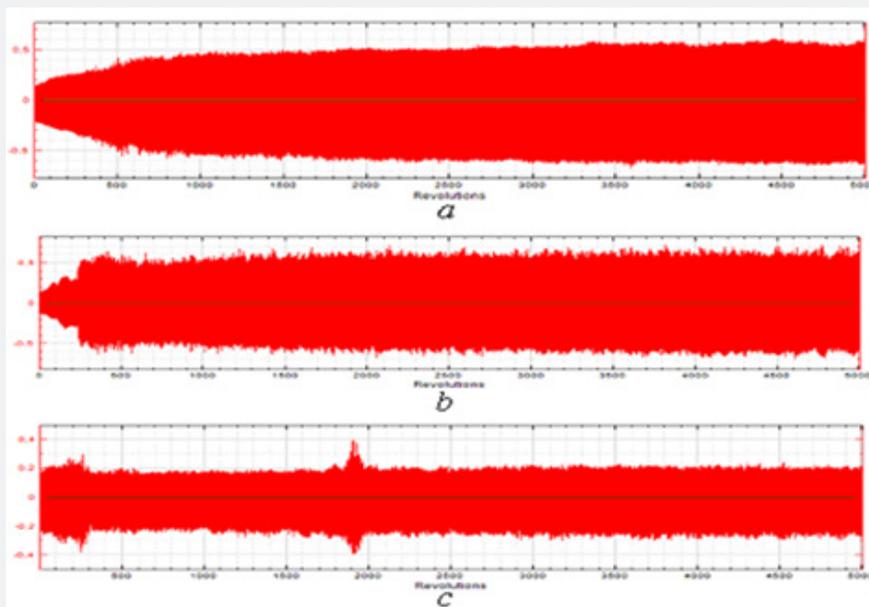


Figure 3: Machine diagrams produced during the tribological testing in accordance with the principle involving linear reciprocating movement:
 a. Grade 4 Ti without coating;
 b. Grade with an IPS coating (TiC);
 c. Grade 4 Ti with coating produced by MAO (TiO).

Thus, from the point of view of tribological efficiency, the TiO coating produced by micro arc oxidation is of most interest. Figure 3 shows the machine diagrams produced in the real-time mode during the testing in accordance with the principle involving linear reciprocating movement.

(Figure 3) Machine diagrams produced during the tribological testing in accordance with the principle involving linear reciprocating movement:

- a. Grade 4 Ti without coating;
- b. Grade with an IPS coating (TiC);
- c. Grade 4 Ti with a coating produced by MAO (TiO).

Surface analysis on a nano scratch tester

As it can be seen from the presented results of physical experiments, in this case the nano scratch tester enabled

producing, in fact, profilograms of the investigated In Figure 3, the samples of Grade 4 CP Ti without coating (a) and with an ion Plasma spraying (IPS) coating (b) are shown. It is observed a much rougher surface in the whole studied area, both at the initial moment of contact and in the process of the indenter's penetration with increasing load. For the sample with a TiO coating produced by MAO (c), there is observation of practically constant curve in the process of scratching of the surface by the indenter, as the load and the depth of the needle's penetration into the coating increase. This may indicate a high shielding ability and hardness of the coating produced by MAO. This observation is in good agreement with the results of tribological testing (Figure 1, curve 3), obtained earlier, and demonstrating the smallest friction coefficient values and a practically absent running-in segment (Figure 4).

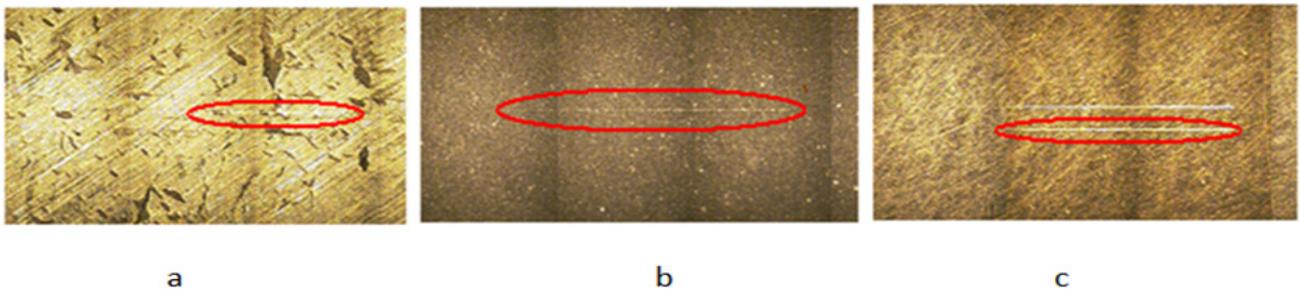


Figure 4: Surface analysis of the samples using a nanoscratch tester:

- a. Grade 4 CP Ti without coating;
- b. Grade 4 CP Ti with an IPS coating (TiC);
- c. Grade 4 CP Ti with a coating produced by MAO (TiO).

The red color indicates the areas of the indenter's action on the object of study.

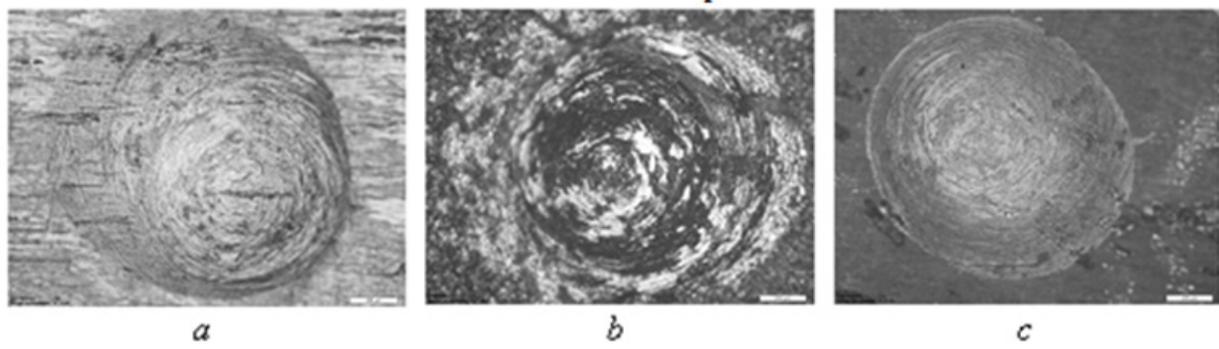


Figure 5: Indentations made by the indenter on the coarse-grained samples from Grade 4 CP Ti with different variants of surface preparation:

- a. without coating;
- b. with a TiC coating applied by ion-plasma spraying;
- c. with a TiO coating produced by MAO.

It is visible from Figure 4 that in the indentation cup on the sample surface without coating (a), the exposed surfaces are observed due to the formation of adhesive bridges with the indenter's material. The sample TiC surface coated with ion-plasma spraying (b) has exposed visible areas (bright fragments

in the photo). These areas are caused by the adhesive interaction between the materials of the sample and of the indenter (Figure 5) it is clear that the most even and clean indentation was made on the sample surface with a TiO coating (c) produced by MAO (Table 1).

Table 1: Adhesive component of the friction coefficient in coarse-grained Grade 4 CP Ti in the contact pair with an indenter from Fe-6W-5Mo steel.

Sample Material	Tribotechnical Characteristics				
	p_r , MPa	τ_n , MPa	τ_n / p_r	β	τ_0 , MPa
Without coating	1605	376	0.235	0.208	43
With a TiC coating applied by ion-plasma spraying	2158	447	0.207	0.189	39
With a TiO coating produced by micro arc oxidation	2480	189	0.076	0.064	31

Where β is the strengthening coefficient of molecular bonds under the action of compressing pressure, τ_0 is the shear strength of adhesive bonds in the absence of a normal load.

It follows from analysis of the data obtained earlier that the smallest value of the adhesive component of the friction coefficient, from the investigated options, is observed in the contact pair «indenter from Fe-6W-5Mo steel - coarse-grained Grade 4 CP Ti with surface coated by oxide, using MAO». For the two other samples, with a TiC coating and without coating, the adhesive components of the friction coefficient are 2.5 and 3 times higher, respectively. Thus, from the point of view of frictional interaction under elastic contact sliding conditions, the most preferable from the tested options is the oxide coating produced by MAO on the surface of coarse-grained Grade 4 CP Ti. Mutually correlated results were obtained both when determining the integral quantity of the friction coefficient and when evaluating the adhesive component of the friction coefficient.

SPD processing of initial Samples

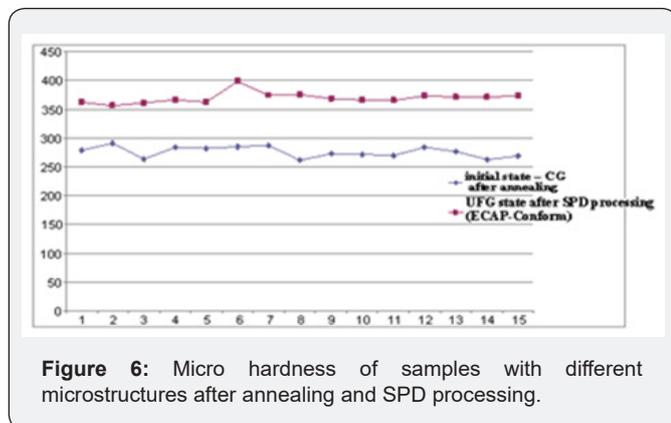


Figure 6: Micro hardness of samples with different microstructures after annealing and SPD processing.

As a result of the testing, the SPD-processed samples demonstrated the average micro hardness value of 372.12 MPa. Figure 6 presents the results of comparative micro hardness measurements for CP Ti.

It can be analyzed for the results of deformation processing that the strength of CP Ti is increased by about 35%, which corresponds to the strength of the titanium alloy VT6 (or Grade 5). Figure 7 shows the microstructure of CP Ti with a sub microcrystalline (SMC) structure.

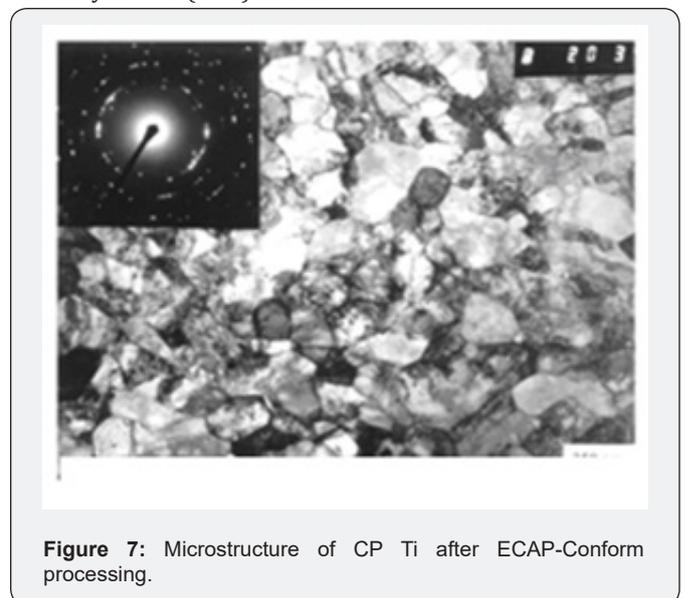


Figure 7: Microstructure of CP Ti after ECAP-Conform processing.

Metallographic studies revealed that after SPD processing via ECAP-Conform, [12,13] the microstructure of CP Ti represents an equiaxed structure with a mean grain size of 300nm. From the Hal-Petch effect grain size is directly related with strength of material. Mechanical properties can be elaborated in table below.

Table 2: Mechanical properties of CP Ti.

Material	Mechanical Properties				
	Yield Stress, $\sigma_{0.2}$, MPa	Ultimate Tensile Strength, σ_u , MPa	Elongation, δ , %	Relative Reduction of Area, δ_r , %	Elastic Modulus, E , GPa
CP Ti	730	820	20	45	117

Conclusion

From the point of view of frictional interaction under elastic sliding contact conditions, the most preferable from the tested options is oxide coating on the surface of coarse-grained Grade 4 CP Ti, produced by MAO. Mutually correlated results were obtained both when determining the integral quantity of the friction coefficient and when evaluating the adhesive component of friction; After deformation processing, it is observed that the strength of CP Ti is increased by about 35%, which corresponds to the strength of the titanium alloy VT6 (or Grade 5);

Metallographic studies reveal that after SPD processing via ECAP-Conform, the microstructure of CP Ti represents an equiaxed structure with a mean grain size of 300 nm;

The type of the applied coating (ion-plasma deposition and micro arc oxidation) has practically no effect on the extent of corrosion damage of the CP Ti specimens.

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