



Effect of gaseous carburizing thermochemical treatment on tribological behavior of Ti-6Al-4V alloy

Amar Talhi, Mohamed Zine Touhami

*Metallurgy and Materials Engineering, Foundry laboratory,
Badji Mokhtar University - Annaba, Algeria
talbiamaryacine@gmail.com*

Kamel Fedaoui

*University of Constantine 1, Constantine, Algeria
Kamel.fedaoui@umc.edu.dz, <https://orcid.org/0000-0003-0885-6914>*

ABSTRACT. This study concerns the improvement of performance of resistance to wear phenomena of the Ti-6Al-4V alloy surface by means of Gaseous carburizing thermochemical treatment. Three-thermochemical treatment durations (2h, 4h, and 6h) were chosen for investigation of the effect of such treatment on this alloy. The hardness test under an indentation load of 0.05 kgf with a Vickers pyramidal indenter revealed that the surface hardness is 335 HV for the untreated samples. The hardness reaches approximately 1500 HV during gas cementation at 930 °C for variable times (2h, 4h, 6h) followed by quenching at 840 °C in an oil medium, which was accompanied by a significant improvement in wear resistance. The characterization of the modified surface layers was studied by means of a microscopic analysis and by X-ray diffraction. The case-hardening made it possible to obtain a wear resistance greater than that of the alloy not treated, minimal loss of mass by dry friction and an improvement in roughness as well as a good coefficient of friction.

KEYWORDS. Ti-6Al-4V; Tribological behavior; Carburizing; Wear; Hardness.



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INTRODUCTION

Titanium-Aluminum-Vanadium Ti-6Al-4V alloy is one of the most used alloy in industrial application .these properties include better corrosion resistance, biocompatibility and the possibility of working in a wide temperature range. these specific characteristics make this alloy a product used in 50% of the industrial market [1]. Titanium alloys are materials of choice in aeronautics in particular because of their low densities (4.5 g/cm³ for titanium and 4.4 g/cm³ for Ti-6Al-4V).



Ti-6Al-4V alloy offers excellent properties, which are advantageous after doing the casehardening. We note the increases of mechanical characteristics such as hardness, better resistance to fatigue and the borne of new complex phases resultants of diffusion of carbon during the interaction with the basic elements.

Due to its low tribological property, Ti-6Al-4V is known to have poor wear and friction behavior. For this reason, it is rarely used in industrial applications where the parts are often subjected to movements causing friction or sliding.

Therefore, various techniques of surface modification by thermochemical processes such as carburization or carburizing [2-4], nitriding [5-6] and carbonitriding [7] have been used to improve wear resistance of titanium alloys. As the results, carbides and nitrides newly formed with carbon and nitrogen are difficult and stable at high temperatures, which will improve the wear resistance and surface hardness of this alloy [8-9].

The damage to the material gradually leads to the establishment of three areas of research carried out respectively by:

- The mechanics who deal with the influence of fretting on cracking use for this purpose fretting tests [10].
- Researchers in materials sciences are developing new surface treatments and coatings, which allow high surface hardening and an improvement in tribological properties [11].
- Tribologists try to understand the mechanisms of action of the interfacial layers or debris which in turn increases friction, Field [12], Zahavi [13] and Daoud [14], studied the influence of roughness on the fatigue strength and demonstrated that the fatigue strength increases when the surface roughness decreases.

Our work can be classified in the research of a new treatment to improve of the tribological properties of such alloy. We focused our experimental investigation to study the role of treatment on the mechanical, tribological and structural properties of the Titanium-Aluminum-Vanadium Ti-6Al-4V alloy.

In this paper, we present an experimental investigation of the effect of time of material hardening on the mechanical and microstructural properties of the surface and sub-layers for untreated and carburizing state. Titanium-Aluminum-Vanadium Ti-6Al-4V alloy is the subject of this study. The techniques used to study the tribological behavior of this alloy are the mass losse, the surface roughness, the coefficient of friction; therefore, we have also shown by SEM the morphology of the friction tracks created by the ball, as well as the hardness as a function of time hardening.

MATERIAL STUDIED AND EXPERIMENTAL TECHNIQUES

The alloy used in this study is Titanium-Aluminum-Vanadium Ti-6Al-4V alloy, which was delivered in the form of a plate of dimensions (100x60x5 mm). This alloy has undergone hot rolling and the structure of which is completely recrystallized. It is a two-phase alloy of alpha + beta type ($\alpha + \beta$). Compared to others designation of titanium alloys, this alloy has the highest mechanical strengths [15]. As long as parts made of Ti6Al4V alloy are exposed to high conditions (temperature, oxidation medium, friction ...), can undergo rapid degradation and to improve it we must proceed with a case-hardening to be able to give a better tribological behavior.

It is found that in a situation of friction, the Ti-6Al-4V shows poor wear resistance behavior, thus causing a loss of material following its degradation. The chemical composition of the titanium alloy is shown in Tab. 1:

Elements	O	C	Fe	Al	V	H	N	Ti
Mass (%)	0.13	0.08	0.25	6	4	0.02	0.05	Bal.

Table 1: Chemical compositions of the titanium alloy Ti-6Al-4V studied [16].

The aim of this study is carrying out a gas carburizing treatment with a view to improving the tribological behavior of the titanium alloy "Ti6Al4V", known for its low resistance to wear. Carburizing is one of the most used surface treatments and more widespread. It improves certain properties of materials, notably their hardness, wear resistance and their corrosion resistance.

Surface hardening treatment by quenching after carburizing was done in an Aichelein-type oven at the level of the thermochemical treatment workshop of the EPE / Spa ETRAG Company in Constantine- Algeria.

The properties of carburizing gas are:

- Gas components: CH₄- NH₃
- Air / Gas ratio: 2.8
- Flow rate: 0.2 - 0.3 m³Nh⁻¹



- Dew point $\pm 2^\circ$
- Carbon potential: 0.8%
- Cracking temperature: 1040°C

The treatment was carried out according to the operating conditions set for the needs of the company's thermochemical treatment workshop: a treatment temperature of 930°C, for three variable times (2h, 4h, 6h) followed by a soaking at 840°C in an oil medium.

Scanning electron microscope is used to visualize the layers formed on the surface of the alloy after gas carburization. The same surface preparation described above was carried out.

To estimate the hardness of Ti-6Al-4V, we performed the Vickers micro-hardness tests before and after the cementation treatment, using a Micro Vickers Hardness Tester ZWICK - ROELL ZHV. The hardness tests of Ti-6Al-4V are carried out by applying a load of 0.05 kgf for 15 seconds.

Wear or loss of mass test was done at the metallography laboratory (metallurgy department of University of Annaba-Algeria). The device used is composed of a sample holder system fixed on a variable speed polisher. The load applied to the parallelepiped sample (10x7x5) mm is fixed to 5N for 1 minute. The sample rotate with angular speed of 65 rpm and the grade of silicon carbide paper is 800. Weight loss is measured after each minute using an electronic microbalance whose measurement accuracy is around 10^{-3} g. Note that the samples are cleaned with acetone before each weight gain.

X-ray diffraction (XRD) is one of the techniques commonly used to identify the crystal structures present in a metal sample. From the diffraction diagram, it is possible to determine the nature, the mesh parameters or even the quantity of each of the phases present in a multi-phase alloy for example.

X-ray diffraction (XRD) can also inform us about possible states of stress and crystallographic textures. The principle of this technique is based on the measurement of the inter-reticular distances relative to the different families of crystalline planes (in agreement with the Bragg relation). The diffractograms are recorded with the radiation ($\text{CuK}\alpha = 1.5406 \text{ \AA}$), in the case of a simple identification of the phases, a range from 0° to 100° (in 2θ). All the diffraction spectra were performed on a PANalytical 'X'Pert PRO "type diffract-meter at the University of Bejaia- Algeria.

The surface roughness was carried out on the surface of the samples before and after treatments using a MITUTOYO type roughness tester. The effect of the cementation conditions on the surface roughness was measured according to the average roughness values (Ra). For the tribology test and according to previous work [16-17] that treated the same material, the following parameters were chosen, see Tab. 2. All the tests are carried out without lubrication and in air.

Course	40 m
Rotation diameter	6 mm
The applied load	10 N
The material of the ball	Alumina (Al_2O_3).
Ball diameter	10 mm
The rotation speed	3 cm/s

Table 2. Parameters of tribology test.

RESULTS AND DISCUSSIONS

In this section, results from different technics of characterization of material used to study the effect of treatment on the mechanical and structural properties of Ti-6Al-4V alloy were presented.

Microstructures of the Ti 6Al 4V alloy

The sample alloys were cut, with a precision chainsaw, coated in section and polished. A chemical attack was made with the attack reagent consisting of 5% HF, 5% HNO_3 and 90% distilled water. The microstructures were observed under an optical microscope and the SEM. We observe on the (Fig.1) untreated state, an alpha + beta type structure ($\alpha + \beta$) and cemented layers According to the carburizing time (Fig.2).

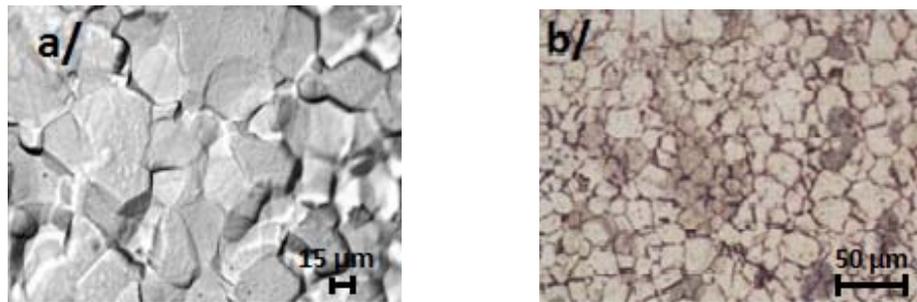


Figure 1: Structures by Optical microscope (untreated Ti6Al4V, a-b).

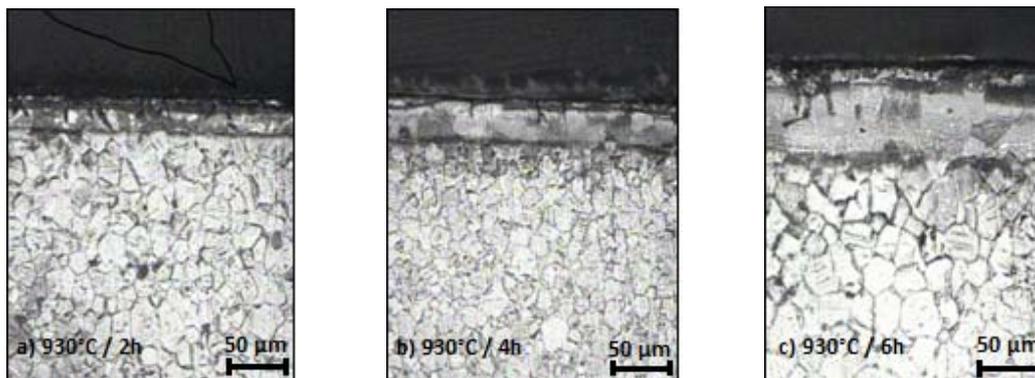
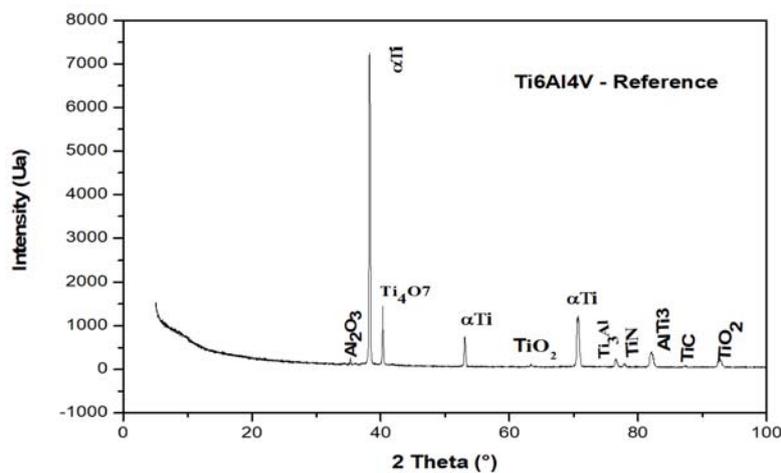


Figure 2: Microstructures of Ti 6Al 4V carburizing at 930 °C - a) for 2h, b) for 4h and c) for 6h.

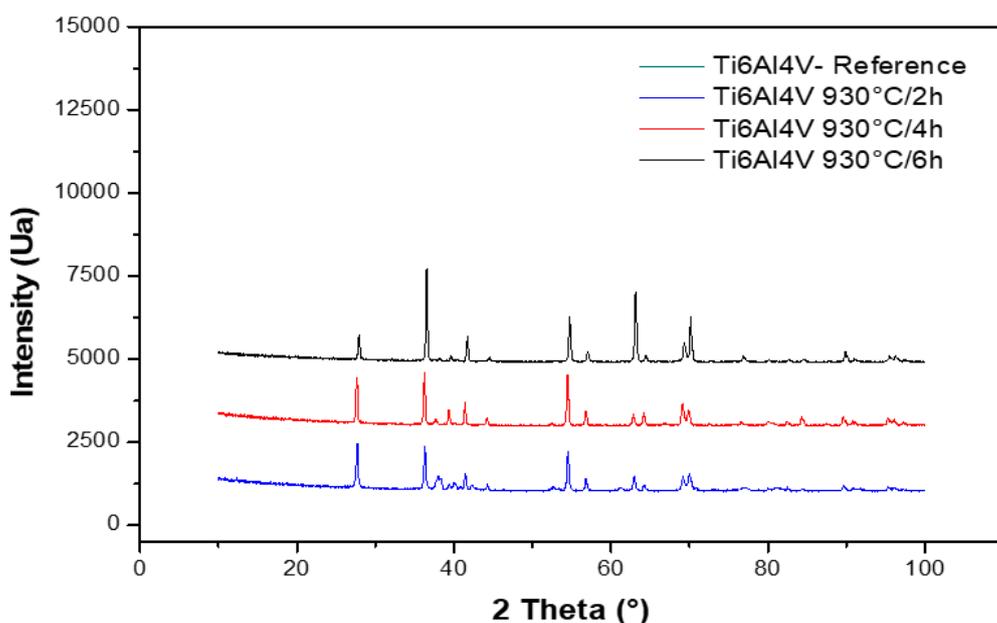
We note that the carburizing depths is about: for 2h = 71.98 μm, 4h = 123.66 μm and for 6h = 160.04 μm. From the figures, we see that the effect of the treatment times is clearly visible, the saturation of the cemented area with the presence of carbon and other elements give birth to hard and complex phases such as titanium carbide and carbide of vanadium (TiC, ViC ...) confirmed by Hailiang Du et all [18].

X-RAY DIFFRACTION

X-ray diffraction will remain, for a long time, one of the techniques used to the identification of crystallographic structures and the phases formed. The microstructures show the presence of layers of mixed and complex carbides and nitrides (TiN + TiNx, TiC, AlC, VC, AlTi, ...).



a)



e)

Figure 3: XRD of a) untreated state- Reference and Carburizing state 930°C – b) for 2h, c) 4h, d) 6h and e) for all the cases.

We note that the effect of the carburizing after decomposition of the carrier gas during the diffusion and under the action of the dissolution of the elements (carbon, nitrogen and oxygen) have an affinity to be dissolved with the whole elements (titanium, aluminum and vanadium). We observe that the peaks of titanium are the most dominant from the point of view of quantity.

Note: For the superposition of the XRD cannot be identical since the untreated sample and the three other samples are not realized with the same parameters such as the scanning step. We can clearly see in the figure that the Reference sample the main peak (α Ti) is shifted with respect to the three others at the angle $2\theta = 38^\circ$.

TRIBOLOGICAL BEHAVIOR

Weight loss of Ti-6Al-4V alloy

For each sample, we weigh its mass before and after the tribological test using a precision balance up to 10^{-3} g. We note that the loss of mass by degradation of the surfaces by friction of the treated sample for 6 h have a constant mass loss over 300 m. The sample with 4 h present an increase in weight loss after the 200 m. The same observation is valid for the sample of 2 hours where an increase in mass loss from 120 m is recorded.

The original sample without treatment, presented a linear and remarkable degradation. By comparison, to the other samples, a gain in mass was observed for the cemented samples unlike the original sample without treatment.

Several slip regimes and different wear mechanisms have been observed depending on the applied load [19-21]. Abrasion phenomenon was observed at different case hardening times for the applied load. The wear mechanism by delamination and by fatigue appears during the hardening times (2h, 4h and 6h). In fact, the increase in the loss of mass in the wear track results from a dissipation of the friction energy in the contact zone [22].

ROUGHNESS

The effect of the cementation conditions on the surface roughness was explored according to the average roughness values (Ra) after seven tests for each sample. We note here that cementation technic is a good technique for improving roughness surfaces (Fig.5).

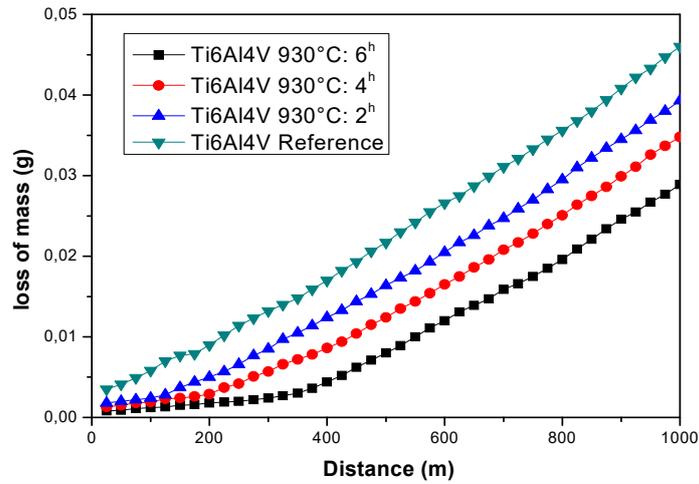


Figure 4: Variation in mass loss (Untreated State -Reference and Carburizing 930°C : 2h, 4h, 6h).

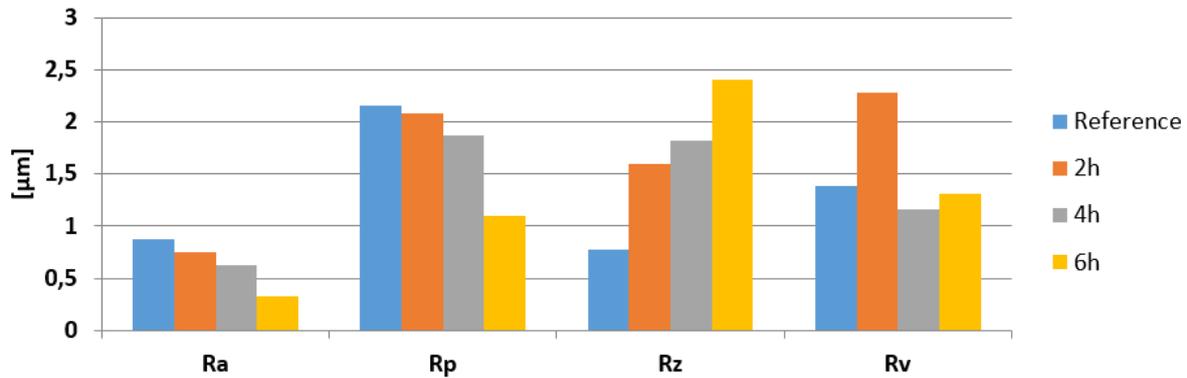


Figure 5: Roughness evolution (Ra : arithmetic mean height, Rp: maximum height of peaks,Rz : maximum height of the profile and Rv: maximum depth of the valleys).

WEAR RESISTANCE (COEFFICIENT OF FRICTION)

In order to have an idea of the behavior in the face of wear, we carry out, on each case hardened samples, a tribological test with identical conditions.

A first analysis of these results (Fig.6) shows that the coefficient of friction increases from the first cycles of friction; this is defined as the transition or running-in period observed during the test.

Friction can be classified into three characteristic periods of the evolution of friction as a function of time. For the first one, it represents the incubation time, which corresponds to the adaptation of the two surfaces by elimination of the surface oxides to give rise to the ceramic - metal interaction, hence a rapid increase in the coefficient of friction. The second is a period of modification of the friction properties of surfaces, which corresponds to the transition from friction to the stabilized state with formation of the 3rd body; the last one corresponds to the stabilized condition of friction.

At room temperature, the coefficient of friction of the Ti-6Al-4V alloy and the alloy coating varied with the wear time for different loads [24 23]. When the load was 3 N, the coefficient of friction of Ti-6Al-4V alloy increased with test time in the first 15 min, then stabilized after 15 min and the coefficient of friction was kept between 0.47 and 0.53. When the load was 6 N, the pre-grinding period was reduced to 10 min, then stabilized, and the coefficient of friction was kept in the range of 0.40 ~ 0.47. When the load increases to 9N, there was no pre-grinding period; it was directly in the stable period. Here coefficient of friction decreased and maintained in the range of 0.39-0.44. Finally, this observation confirms our parameters

and our results as the 10N load and the Al₂O₃ ball under the same conditions at ambient temperature without lubrication and with air. This achieved a good improvement in the coefficient of friction by addition of the thermochemical-hardened layer (gaseous cementation).

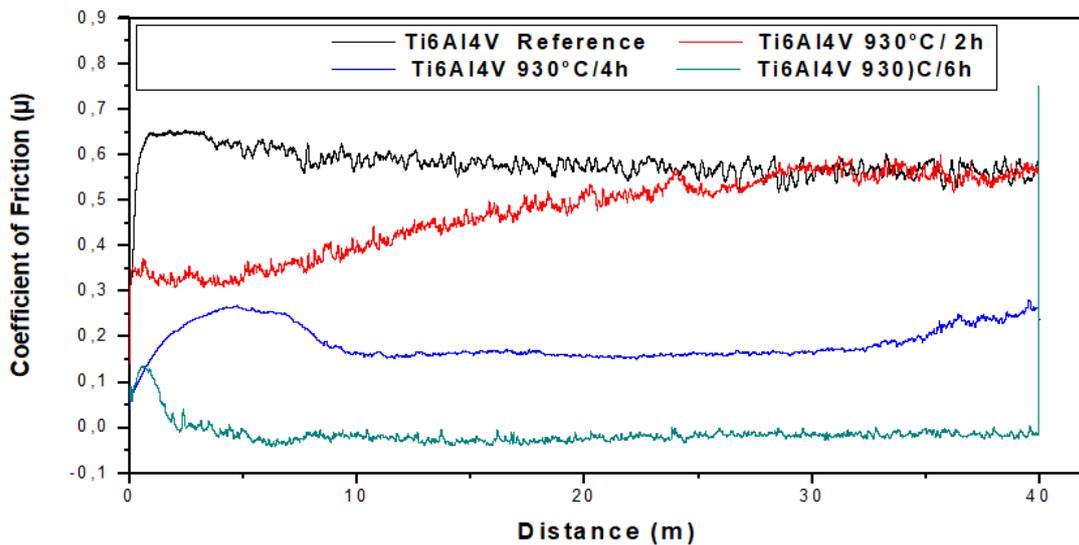


Figure 6: Coefficient of friction (Untreated and Carburizing 930°C - 2h, 4h, 6h).

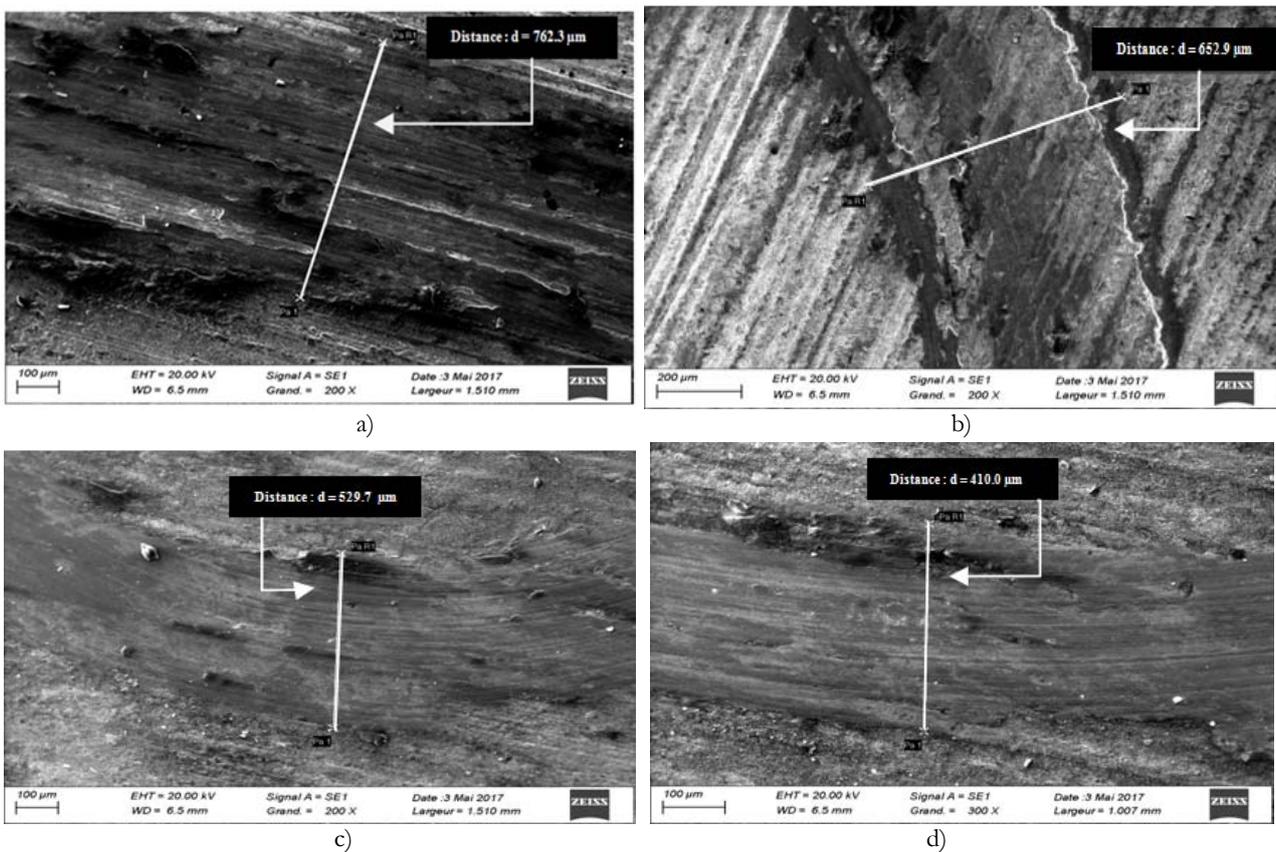


Figure 7: Morphological state of the samples after wear test using a Scanning Electron Microscope. a) untreated; b) carburized at 930°C - 2h; c) carburized at 930°C - 4h; d) carburized at 930°C - 6h.



From all these results, we note that the improvement in the coefficient of friction depend on the quantity of carbon diffused in the alloy; we observe that the tribological performances for (6h) are superior to those of the other samples (Fig.7).

The sliding of the ball on the surfaces of the hardened samples is carried out under a load of 10N in order to quantify the wear and the coefficient of friction. A streaks observed on the facies, on the friction track, scratches and / or streaks are caused by the hard particles of the third body which indicates a real deterioration of the surfaces as well in width which is very clear in Fig. 7, (Reference = 762.3 μm , 2h = 652.9 μm , 4h = 529.7 μm and for 6h = 410 μm).

Noting therefore that the sample of Fig. 7-d, presents a better behavior from the point of view of resistance to wear, hardness and depth of cementation.

We previously knew that oxygen was mainly found on the surface of the sample layer (titanium oxide TiO) without it forming crystalline precipitates. We can assume that oxygen impairs good tribological behavior by causing a three-body wear regime more quickly (Fig.8).

The diffusion of carbon in the titanium alloy by gas carburizing makes it possible to significantly improve both the hardness and the tribological performance of Ti-6Al-4V, see work of J.C. Sánchez-López et al [24].

Observation by scanning electron microscope (Fig.7-8) shows the presence of plowing grooves corresponds to the same abrasive wear mechanism.

The width of the passage of the ball for the untreated state is greater compared to the other case-hardened samples, (the values are respectively: untreated state = 762.3 μm , 2h = 652.9 μm , 4h = 529.7 μm and for 6h = 410 μm).

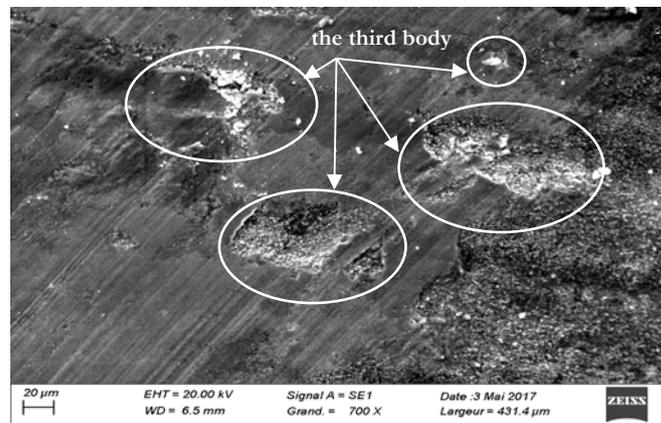


Figure 8: Appearance of the asperities of the third body (white).

Depending on the number of cycles, the particles detached from the surface participate in the kinetics of wear in the contact. In fact the type of contact passes each time from a two-body contact; Al_2O_3 -Ti 6Al 4V to that with three bodies; Debris - Al_2O_3 - Ti 6Al 4V; (Fig.8), which brings us to have the same phenomenon which are in agreement with those of [19, 25-26].

HARDNESS PROFILE

To study the effect of cementation on the mechanical properties of samples, Vickers Microdurometer with a 0.05 kgf of load is used. Vickers method (Hv) is simple and can provide some information's on the hardness property of samples and their depths. The presence of complex phases in the case hardened layers improves the fatigue strength of the treated titanium alloy. Therefore, it is often necessary to carry out a surface treatment on titanium alloys in order to improve their behavior in friction according to [27].

The hardened layers have a high surface hardness, close to 1500 Hv for the sample of the 6 hours of cementation while the hardness of the sample without treatment is 335 Hv. We note here a great increase of 5 times in surface hardness implies a high resistance to wear, friction, abrasion and seizure. We assume that the diffusion distance is only determined by two factors (one is the speed of diffusion, and the other is the driving force of diffusion), (see fig 9).

We deduce that the role played by the cementation time on the increase of hardness can be important in specific cases, but other factors such as mechanical, microstructural and contact surface characteristics (material / ball) have an influencing role, see [28-30].

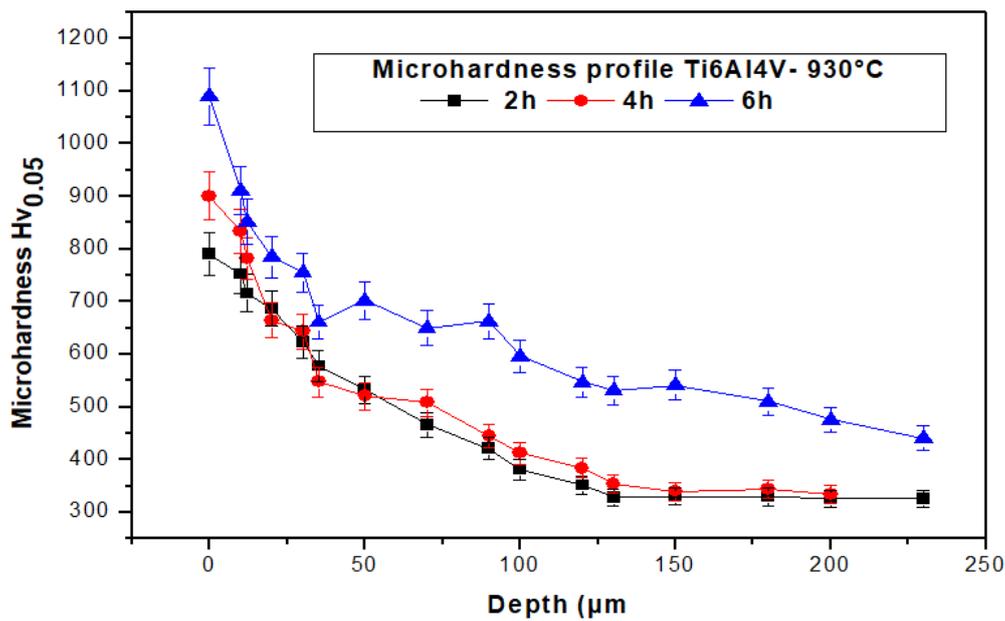


Figure 9: Micro hardness profile for different Carburizing Durations (2h, 4h, 6h).

The variation of surface hardness as a function of the carburizing time depends on the growth of the carburizing time relative to the surface hardness, which increases at the same time, (see Fig.10).

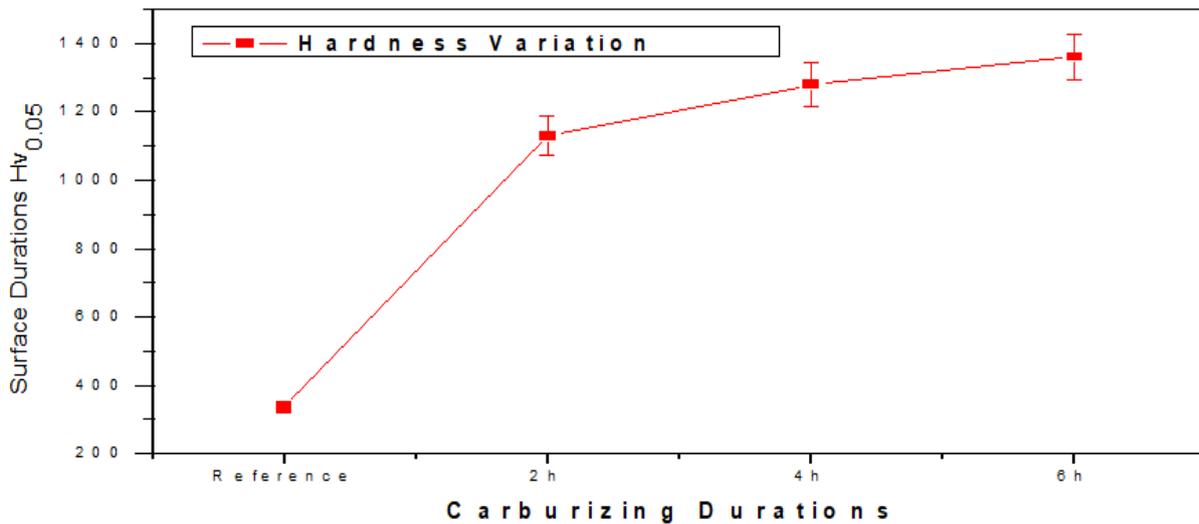


Figure 10: Surface hardness (Untreated State -Reference and carburizing 930°C - 2h, 4h, 6h)

CONCLUSIONS

This study was carried out with the aim of having a global vision on the effects of gas carburizing at high temperature and by the diffusion of carbon after decomposition of the carrier gas with variation of treatment durations. The microstructural study taught us that these improvements were due not only to the presence of a layer of titanium carbide but also to the formation of phase clusters. The samples of the cemented titanium alloy were characterized by optical microscope, SEM and XRD, which allowed us to delimit the cemented layers and showed that the depth of the cementation layer depended on the diffusion time. Studies have revealed that the cementation layer has simple and complex phases with a mixed microstructure of (TiC, α -Ti, Al₂O₃, VC, Ti₃Al₂C₂, ...).



Improving tribological performance has therefore answered many questions, but it has also opened the way to new areas of investigation like corrosion tests. Indeed, according to the results from the literature and with others surface treatment processes intended for titanium alloys; the origins of the improvements in corrosion resistance are the same as the increase in tribological performance. From the microstructural view, the wear resistance behavior study of the Ti-6Al-4V alloy before and after treatment by hardness, friction and wear tests, resulted in a significant improvement in the hardness and wear behavior of the alloy. All this confirms the effectiveness of the treatment carried out according to the conditions presented.

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