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Fatigue behavior of PVD coated Ti–6Al–4V alloy

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ABSTRACT

The aim of the present work is to identify an environmentally clean coating process that will present lower influence in the Ti–6Al–4V fatigue strength. Axial fatigue tests of Ti–6Al–4V alloy TiN, CrN and WC:H (tungsten containing diamond-like carbon) Physical Vapor Deposition (PVD) coated was evaluated. Decrease in fatigue life was observed for coated specimens in comparison to base metal. Scanning Electron Microscopy technique was used to observe crack origin sites and coating thickness. DLC coating provides higher Ti–6Al–4V alloy PVD coated fatigue strength due to lower defects presence and the chromium interlayer, which acts as a barrier to fatigue crack propagation.

1. Introduction

Landing gears are components subjected to severe conditions such as aggressive environment and high stress levels [1,2]. Although high strength steels and 7XXX series aluminum alloys are still used for structural aircraft parts, titanium alloys have been finding increasing applications due to high strength/weight ratio, stiffness and corrosion resistance [3–8].

In mechanical components subjected to friction, the titanium low shear resistance in addition to low protection exerted by surface oxide layer restricts the use of the base material in the as received condition. Therefore a specific surface treatment is required [9–11].

Electroplated chromium coatings are a usually choice in engineering components, automotive, aerospace and decorative industries due to the high hardness (up to 880 $Hv_{0.01}$) and protection against corrosion and wear [12]. However the electroplating bath produces carcinogenic vapors of hexavalent chromium (Cr6+). Regulative agencies, such as Occupational Safety and Health Administration (OSHA) in the USA, are reducing the permissible exposure limit (PEL) to 5 μg of C6 (5 μg/m^3), which represents 10% of previous limit [13].

In order to assure their competitiveness and to find new possible applications, the research on coating processes are focused on environmentally friendly technologies, which guarantee protection against corrosion and wear. However, it is important to investigate the influence of new coatings on fatigue resistance of base material when the component is under cyclic loading [2,3,13–16].

Tungsten carbide (WC) coatings applied by High Velocity Oxygen Fuel (HVOF) process present better rotating bending fatigue strength than AISI 4340 steel chromium electroplated and it was appointed as a promising substitute to electroplated chromium in landing gears [6,17,18]. For titanium alloys, results showed that Ti–6Al–4V alloy axial fatigue strength for 10^7 cycles was 900 MPa and for Ti–6Al–4V WC–10%Co–4%Cr thermal spray coated, it decreases to 400 MPa [16]. Considering this strong reduction, other finishing alternative as Physical Vapor Deposition (PVD) was evaluated in this work.

PVD coatings are classified in two main groups: traditional wear resistant coatings, the nitrides and carbides of titanium and chromium as TiN, TiCN, TiAlN and CrN, which are suitable to avoid galling problems; and solid lubricating coatings, characterized by low coefficient of friction [19,20].

Studies in PVD coatings provided a useful analysis of the substrate alloy's mechanical properties and how they can be modified by coating treatments. Dobrzanski et al. [21] studying X37CrMoV5-1 TiN, TiN/(Ti,Al)N and CrN coated wear resistance, obtained higher adhesion for the CrN coating and low friction coefficient in high and low temperature tests for TiN. Nolan et al. [22] investigated TiN coating deposited by PVD and ion implantation process and concluded that both processes improved Ti–6Al–4V wear resistance.

Puchi-Cabrera et al. [23] evaluated the influence of TiN coating on mechanical properties of 316L stainless steel. Authors reported an improvement of 60 MPa in fatigue limit, without any significant changes in the yield and tensile strength. The increase in fatigue strength was attributed to compressive residual stresses inside the coating.

Sundaram [7] observed that wear and fatigue behavior of AISI 4340 steel DLC (diamond-like carbon) coated were superior when

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compared to AISI 4340 steel chromium coated; in addition an excellent adhesion of DLC film on the steel surface was achieved.

In this work, the axial fatigue strength of Ti–6Al–4V alloy PVD coated with TiN, CrN and DLC was evaluated.

2. Experimental procedures

2.1. Materials

The chemical composition of Ti–6Al–4V alloy used in this research was: 69.86% Ti, 6.03% Al, 4.58% V, 0.61% Fe wt.% obtained by atomic absorption spectrophotometer. It is characterized as a metallurgical duplex structure with a 30% volume fraction of equiaxied primary α and 70% correspond to a lamellar $\alpha + \beta$ structure. Titanium alloy specimens were obtained by grind machining, which represents surface roughness $Ra = 0.98 \pm 0.11$ μm, cut-off 0.8 mm. The most important mechanical properties are: ultimate tensile strength of 1270 MPa, microhardness 380 ± 28 $HV_{0.3\ kgf}$ for primary α and 354 ± 4 $HV_{0.3\ kgf}$ for lamellar $\alpha + \beta$ structure, in the annealed condition.

2.2. PVD coatings

The PVD coatings were performed in accordance to Bodycote Brasimet Co. specifications. In the present research three different coatings were studied. TiN, CrN and DLC based coatings were deposited using a HTC 1200 PVD unit manufactured by Hauzer Techno Coating Europe BV Venlo, The Netherlands. The cathodes are equipped with a cathodic arc deposition (TiN and CrN) and sputtering technique (DLC).

Before the process deposition, all specimens were cleaned in phosphoric acid, alkaline and detergent solution and deionized water in an ultrasonic cleaner system. This process is used to remove organic compounds and thin oxide layer, improving adhesion.

The TiN and CrN specimens were rotated and negatively biased with respect to the anode and the chamber and positive ions generated from the target were deposited onto the substrate under N atmosphere. The DLC coating was evaporated using a WC target under a argon + acetylene gas mixture plasma to deposit DLC (WC:H).

The process deposition followed four steps: pump down and heating, metal ion etch, reactive or magnetron sputtering deposition and cool down. Table 1 shows the typical deposition conditions.

The coating thickness measured for TiN and CrN was ca. 4 μm, and the surface roughness obtained $Ra = 1.06 \pm 0.04$ μm and $Ra = 1.08 \pm 0.15$ μm, respectively. The DLC coating shows thickness ca. 2.4 μm and first layer presented ca. 1 μm, the surface roughness was $Ra = 0.57 \pm 0.13$ μm. In the DLC process a thin chromium layer (<0.1 μm) was deposited to improve the adhesion and to act as a chemical barrier between carbon and titanium elements.

The average and standard deviation hardness for coatings studied are represented in Table 2.

Table 2
Average coating hardness.

Coating	Hardness (Hv)
TiN	2500 ± 100
CrN	2200 ± 150
DLC	1200 ± 100

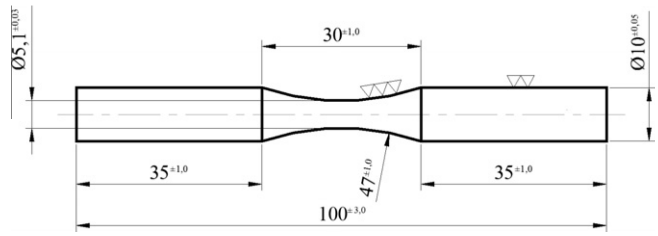

Fig. 1. Axial fatigue testing specimens.

Table 3
Specimens tested and replication ratio.

Specimen group	Number of specimens tested	Replication ratio (%)
Base material (Ti–6Al–4V)	13	23
Base material DLC coated	10	30
Base material CrN coated	9	22
Base material TiN coated	12	17

The TiN and CrN coatings were characterized as a monolayer or mono-block. This characteristic is because during deposition process the main parameters like bias, pressure and current of evaporation were maintained constant.

The DLC layer was produced in two steps using a gradient procedure for the gas flow. This means that in the first step of deposition de ratio of argon acetylene was 80/20 and in the second step the reaction was 60/40. The main objective is to produce a layer with higher hardness (1700 Hv) in step 1 and to reduce ca. 30% in the second step (1200 Hv). It is expected in the second step a carbon graphite structure formed with mainly sp2 bonds, which will increase DLC coating wear resistance [24,25].

2.3. Fatigue tests

Axial fatigue tests according to ASTM E 466 [26] were conducted using a sinusoidal constant amplitude load of frequency 20 Hz and stress ratio $R = 0.1$ at room temperature considering, as fatigue strength, specimens fractured or 10^7 load cycles. Four groups of fatigue specimens were prepared, according to Fig. 1. Preliminary tests were developed to obtain S–N curves for axial fatigue tests, according ASMT E 739 [27]. The number of specimens tested and replications for each condition are listed in Table 3.

Table 1
Deposition conditions.

Coating	Steps	Steps conditions	Pressure	Temperature
TiN, CrN, DLC	Pump down and heating	–	5×10^{-5} mbar	450 °C
TiN, CrN, DLC	Metal ion etch	Substrate voltage 900–1200 V Arc current 140 A	5 min	450 °C
TiN, CrN	Reactive deposition (cathodic arc)	Substrate voltage 80 V Arc current 140 A	3×10^{-3} mbar – N2	450°
DLC	Reactive deposition (magnetron sputtering)	Substrate voltage 20 V Cathode power 8 kW	100 sccm Ar 170 sccm C2H2	180 °C
TiN, CrN, DLC	Coll down	–	5×10^{2} mbar – N2	<200 °C

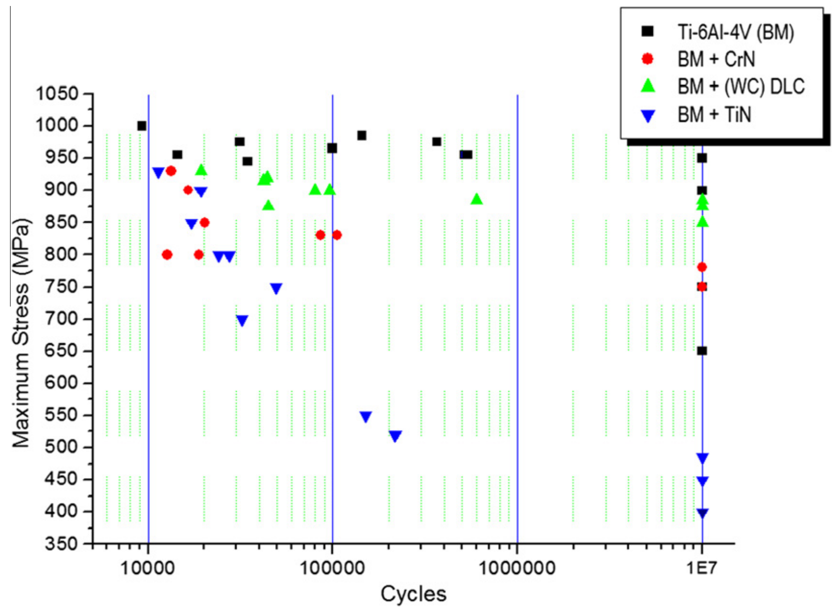

Fig. 2. Scratch test (a) CrN (b) TiN (c) DLC.

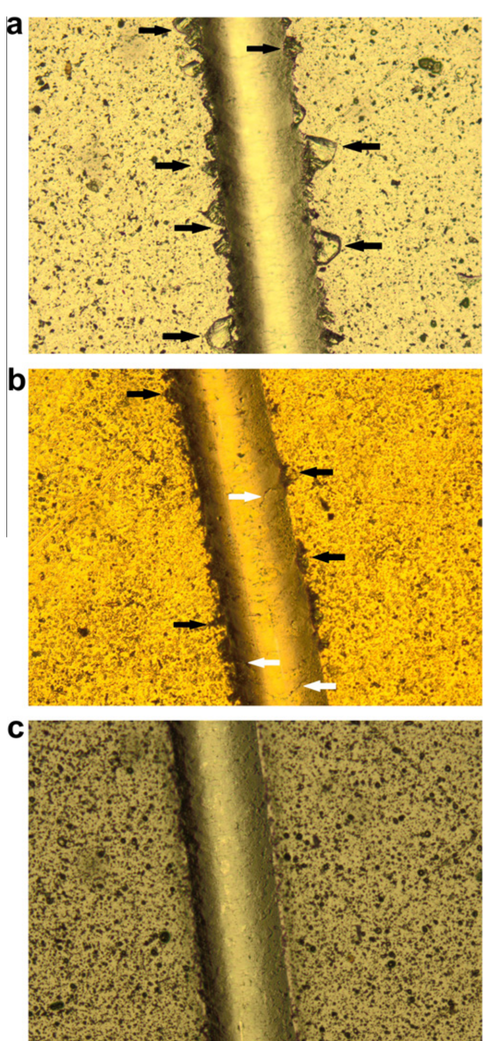

Fig. 3. SXN curves for Ti–6Al–4V alloy, Ti–6Al–4V alloy TiN coated, Ti–6Al–4V alloy CrN coated and Ti–6Al–4V DLC coated.

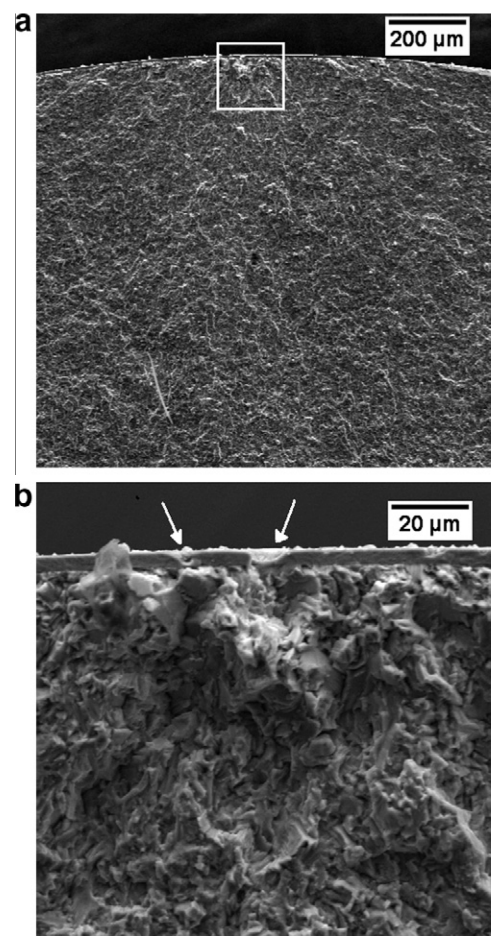

Fig. 4. Fracture surface of Ti–6Al–4V alloy CrN coated – σ_{max} = 830 MPa, 105,839 cycles. (a) 100× (b) 1000× – detail in (a).

Fracture planes of fatigue specimens were examined using a scanning electron microscope model JEOL JSM 5310 in order to identify the crack origin sites.

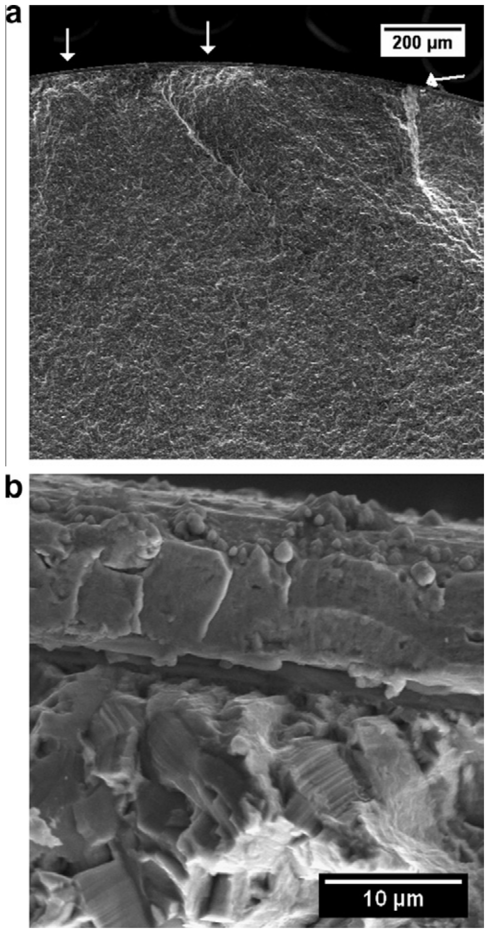

Fig. 5. Fracture surface of Ti–6Al–4V alloy CrN coated – σ_{max} = 800 MPa, 18,888. (a) 100× (b) 3500× – detail in (a).

2.4. Scratch test

The scratch test was performed according to DIN EN1071-3 using a scratch tester Revestest from CSEM. The specimens of TiN, CrN and DLC were scratch tested using a constant 70 N force. The analysis was classified according to the delamination pattern and the cracks around the scratch.

3. Results

Fig. 2 presents scratch test results, it can be observed DLC with less cracks when compared to TiN and CrN coatings. CrN coating shows small defects at the edge of the scratch showed by arrows. TiN coating presents cracks at the edge of the scratch (white arrows), with evidence of edge deterioration (black arrows). All coatings presented no delamination for 70 N.

Axial fatigue tests data and S–N curves for the base material and specimens TiN, CrN and WC:H (DLC) PVD coated, are shown in Fig. 3. Results indicate, clearly, that the influence of PVD is to reduce the base material fatigue strength for low and high cycle fatigue.

For maximum applied stress equal to 900 MPa, which represents 71% of the ultimate tensile strength, Ti–6Al–4V alloy as received presented a fatigue life of 10^7 cycles. For the same stress level, the number of cycles to failure for Ti–6Al–4V alloy TiN, CrN and DLC PVD coated was 19,212, 16,506 e 87,989 cycles, respectively. Experimental results showed better fatigue performance for Ti–6Al–4V alloy DLC coated.

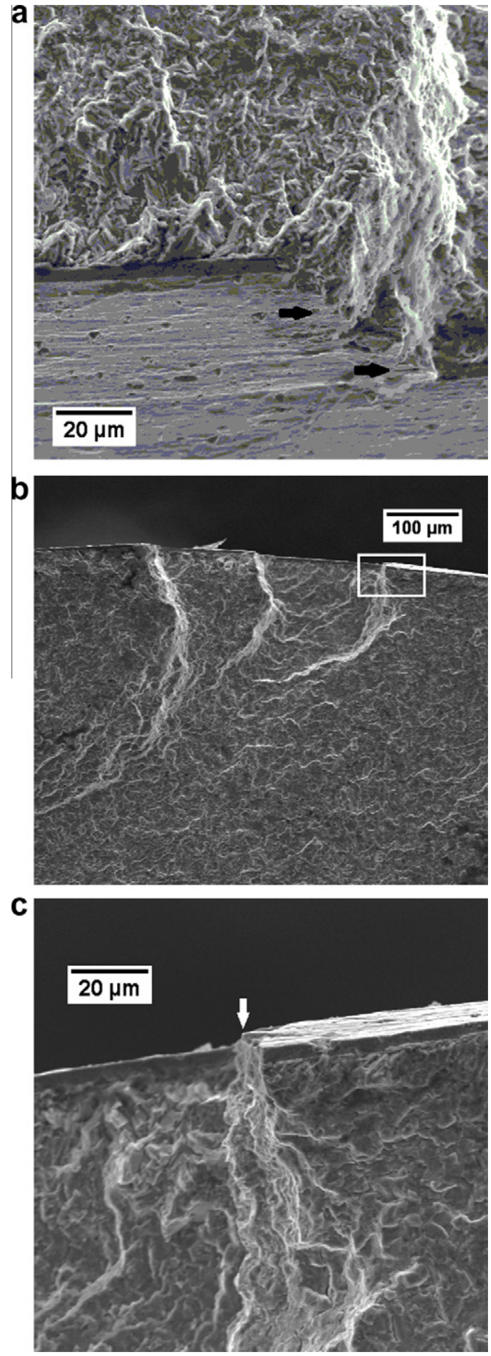

Fig. 6. Fracture surface of Ti–6Al–4V alloy CrN coated – σ_{max} = 930 MPa, 13,365 cycles. (a) 1000× (b) 200× (c) 1000× – detail in (b).

High cycle fatigue regime, 10^7 cycles, may be associated to ca. 750 MPa for Ti–6Al–4V/CrN coated; 450 MPa for Ti–6Al–4V/TiN coated and 850 MPa for Ti–6Al–4V/WC (DLC) coated. This means, respectively, 59%; 36% and 67% of the Ti–6Al–4V alloy ultimate tensile strength.

For the same number of cycles (10^7), reductions in maximum applied stress in comparison to base metal, to the PVD coatings are, respectively, 16.6%; 50% and 5.5%.

The scratch test result seems to be in agreement with the fatigue test, considering that the best result was obtained by the DLC coating and the worst associated to TiN coating. This means that the DLC absorbs better the sliding energy and reduce the free energy of fatigue sites.

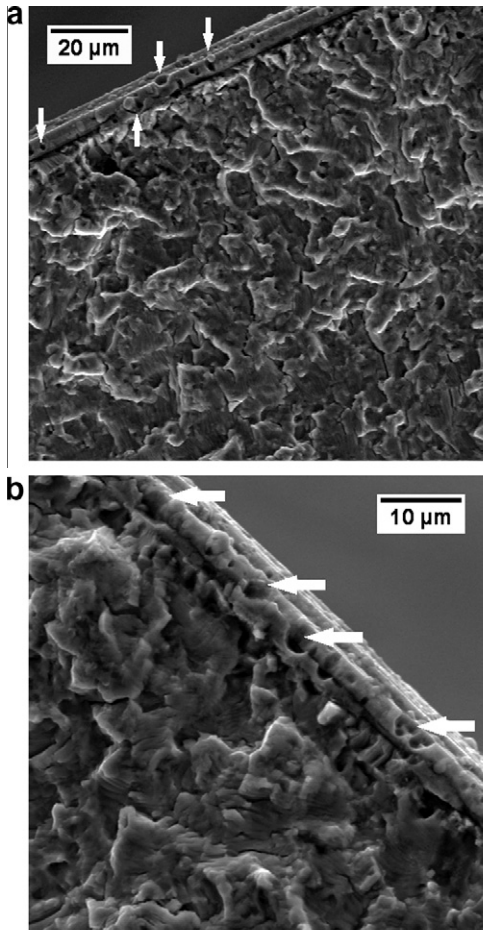

Fig. 7. Fracture surface of Ti–6Al–4V alloy TiN coated – σ_{max} = 520 MPa, 217,630 cycles. (a) 1000× (b) 3500.

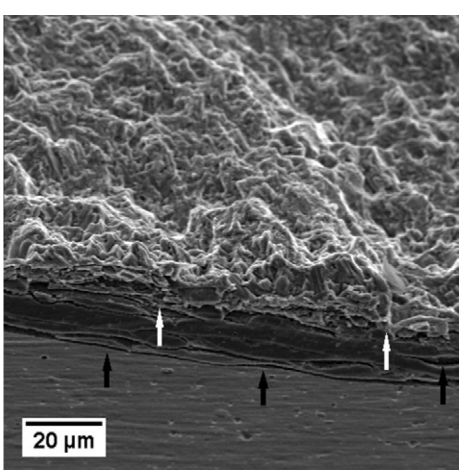

Fig. 8. Fracture surface of Ti–6Al–4V alloy TiN coated – σ_{max} = 750 MPa, 49,086 cycles.

Smith [28] mentioned that cathodic arc source of evaporation is not suitable for low defect density coatings due to the ejection of macro particles during process. This characteristic explains the lower fatigue resistance in comparison to magnetron sputtering process.

The fracture surfaces for Ti–6Al–4V alloy CrN coated are indicated in Figs. 4–6. Coating thickness measured was 4 μm.

The Ti–6Al–4V alloy CrN coated tested at 830 MPa, with 105,839 cycles until fracture is represented in Fig. 4a. Coating defects indicated by arrows in Fig. 4b were the fatigue nucleation sites.

The fracture surface of Ti–6Al–4V alloy CrN coated tested at 800 MPa with 18,888 cycles until failure is presented in Fig. 5. Three sites of fatigue nucleation are indicated in Fig. 5a. At higher magnification, Fig. 5b shows that cracks started at coating surface and propagated throughout base material. This result explains the decrease in cycles to failure in comparison to tests at 830 MPa.

Crack growth from coating throughout Ti–6Al–4V alloy is also represented in Fig. 6. From Fig. 6a, it is possible to observe that fatigue crack nucleation occurred at coating surface. Voorwald et al. [5] showed that considering a brittle coating and ductile substrate, fatigue crack may propagate through interface inside base metal. It was mentioned that the CrN coating and substrate hardness were, respectively 2000 Hv and 380 Hv. This behavior was observed in Fig. 6b and c.

Fracture surfaces of Ti–6Al–4V alloy TiN coated are represented in Figs. 7 and 8. The coating thickness is approximately 4 μm.

The presence of voids is observed in Fig. 7a and b, associated to fatigue crack propagation throughout base material. The TiN coating deposition resulted in reduction in the axial fatigue strength of Ti–6Al–4V alloy as indicated in Fig. 3. It is important to consider that TiN coating hardness was 2500 Hv.

It is interesting to observe in Fig. 8, which represents the fracture surface of a specimen TiN coated and tested at 750 MPa, the growth of multiple fatigue cracks into base material (black arrows) and parallel (white arrows) to the coating surface.

Baragetti et al. [29] investigated the influence of CrN coating on the 6082 aluminum alloy and 2205 stainless steel fatigue behavior. At high stress levels, the main fatigue mechanism observed was the surface hard film cracks in multiple points due to the impossibility to sustain the imposed strain as requested from the base material substrate.

in Figs. 9 and 10 show the fracture surfaces of a base metal specimen DLC coated. Cracks were originated from the coating surface. The coating thickness measured was about 2.4 μm for the DLC coating and 0.15 μm for chromium interlayer.

Fracture surface from base material DLC coated and tested at 930 MPa is presented in Fig. 9a, with the indication of three crack nucleation sites: A, B and C. Fatigue cracks nucleated inside the coating and propagated into the material base, as indicated in Fig. 9b–d. It is possible to observe fatigue striations as a stable fatigue crack propagation characteristic. One sees, from Fig. 9b and d, microcracks along the thickness of the DLC coating, which did not grow in direction to the substrate, inhibited by the ductile chromium interlayer (300–400 Hv) and Ti–6Al–4V alloy (380 Hv).

Fatigue data indicates the importance of the two step deposition of DLC coating as a barrier to crack propagation, despite the fact that microcracks were present in the DLC coating. The mechanical strength of materials involved is directly related to the fatigue crack growth. In this work, hardness sequence is: DLC coating second layer (1700 Hv), DLC coating first layer (1200 Hv), chromium interlayer (300–400 Hv) and Ti–6Al–4V alloy (380 Hv), as presented in Fig. 10. The presence of chromium interlayer, which with the base material is ductile in relation to DLC coating, acted as a barrier to fatigue crack propagation, allowing fatigue strength of Ti–6Al–4V DLC coated values near to base material [5].

Fig. 10 shows backscattering image from Ti–6Al–4V alloy DLC coated and tested at 900 MPa. There are two distinct colors in the coating. The brighter area is associated to the first step DLC coating with higher tungsten concentration, providing a hardness gradient between coating and substrate [30]. The pure chromium thickness is ca. 0.15 μm and in addition to the step by step adding

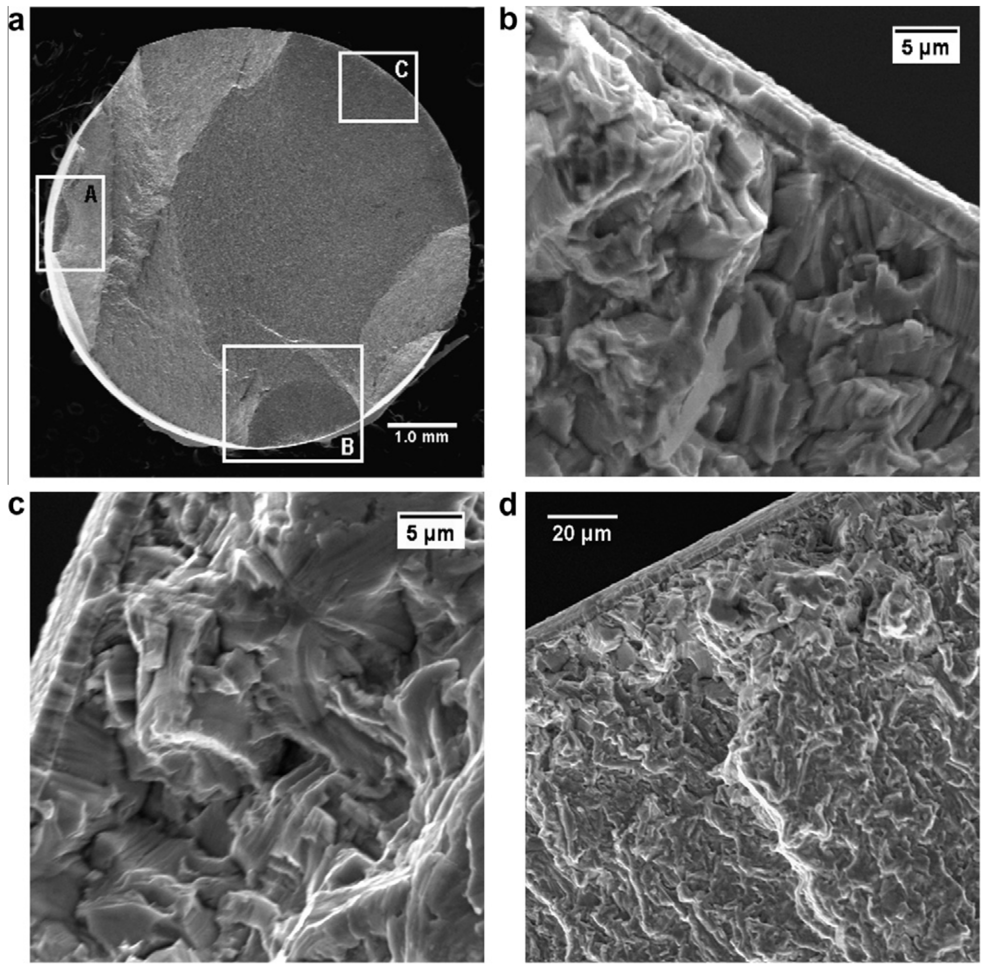

Fig. 9. Fracture surface of Ti–6Al–4V alloy DLC coated – σ_{max} = 930 MPa, 19,430 cycles – (a) 15×.

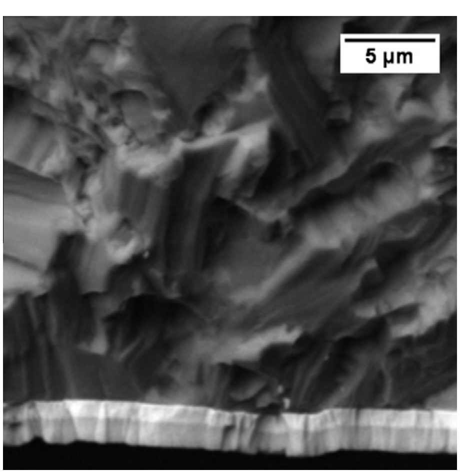

Fig. 10. Fracture surface of Ti–6Al–4V alloy DLC coated – σ_{max} = 900 MPa, 79,932 cycles – 5000×.

DLC creates a good hardness transition and reduces the stress between these two layers.

Sundaram [7] investigated the axial fatigue strength of Ti–6Al–4V alloy and AISI 4340 steel DLC coated. In this research, both base materials were previously shot peened before coating deposition. Shot peening is a useful treatment to improve fatigue behavior. This process prevents fatigue crack initiation and delay fatigue crack propagation by inducing a compressive residual stress field [31,32]. It was observed that the fatigue life for AISI 4340 steel was slightly improved by DLC coating with shot-peening pre-treatment and, for Ti–6Al–4V alloy, S–N data for DLC coated with shot-peening pre-treatment slightly decreased when compared with base material.

These results demonstrate that the use of shot peening as a surface treatment before DLC coating deposition are directly related to the fatigue strength obtained for coated specimens [7]. In this work, Ti–6Al–4V alloy was coated as machined and the effect of all coatings was to reduce fatigue strength of base material in high and low cycle regime.

For deposition parameters used in this work, higher axial fatigue strength for Ti–6Al–4V alloy DLC coated in comparison to TiN and CrN coatings is attributed to chromium interlayer which acted as a barrier to fatigue crack propagation and to film growth process, considering that sputtering technic (DLC) produces lower defect density coatings than cathodic arc source of evaporation (TiN and CrN) [28].

4. Conclusions

Experimental results showed that TiN, CrN and DLC coated by PVD reduced the Ti–6Al–4V axial fatigue strength.

CrN coating defects were the main cracks nucleation sites. Cracks started at the coating surface and propagated thought interface into base material due to the hardness difference between coating and substrate.

In the TiN coating, fatigue cracks initiation sites are associated to the presence of voids. The mechanical strength of coating and substrate is directly related to fatigue crack growth.

DLC coating provides higher Ti–6Al–4V alloy PVD coated fatigue strength. Parameters responsible are the magnetron sputtering film growth process, which allow lower defects presence and the chromium interlayer, which acts as a barrier to fatigue crack propagation.

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