

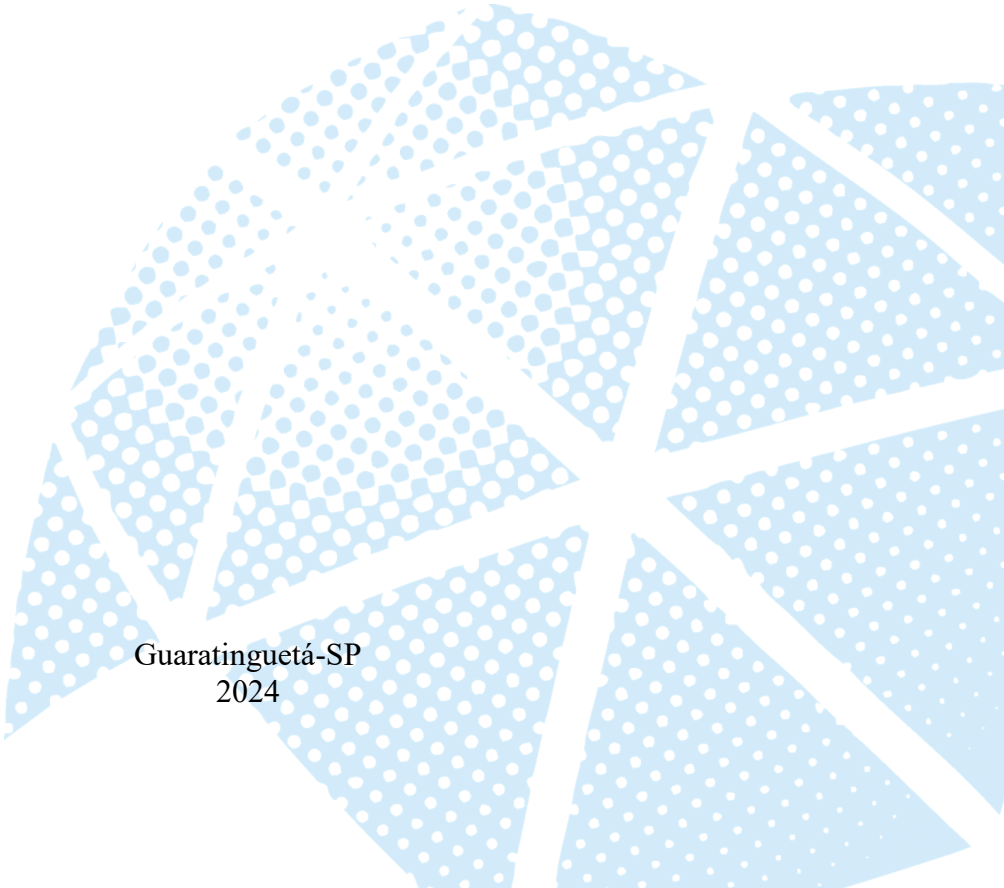
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UNIVERSIDADE ESTADUAL PAULISTA – UNESP
Faculdade de Engenharia e Ciências - Campus de Guaratinguetá

MARTIN FERREIRA FERNANDES

**INVESTIGATION OF THE FATIGUE AND DWELL-FATIGUE BEHAVIOR OF THE
Ti-6Al-4V ALLOY WITH Cr/CrN MULTILAYER COATING AT ROOM
TEMPERATURE AND 350 °C**



Guaratinguetá-SP
2024

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Tese apresentada à Universidade Estadual Paulista (UNESP), Faculdade de Engenharia e Ciências, Guaratinguetá, para obtenção do título de Doutor em Engenharia Mecânica.

Área de Concentração: Propriedades Mecânicas dos Metais e Ligas

Orientador: Prof. Dr. Herman Jacobus Cornelis Voorwald

Coorientadora: Profa. Dra. Verônica Mara de Oliveira Velloso

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IMPACTO POTENCIAL DESTA PESQUISA

O impacto esperado desta pesquisa é o potencial tecnológico de aplicar tratamentos superficiais em ligas de titânio para aplicações aeronáuticas, especialmente nos componentes de motores de turbina a gás. Os mecanismos de fadiga e a influência de revestimentos de múltiplas camadas foram investigados para a liga Ti-6Al-4V.

POTENTIAL IMPACT OF THIS RESEARCH

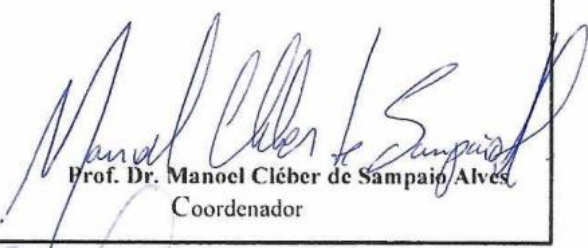
The expected impact of this research is the technological potential of applying surface treatments on titanium alloys for aeronautical applications, especially in the turbine gas engine components. The fatigue mechanisms and the influence of a multilayer coating were investigated for the Ti-6Al-4V alloy.

MARTIN FERREIRA FERNANDES

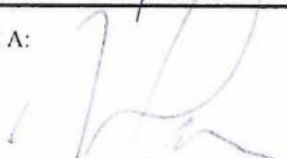
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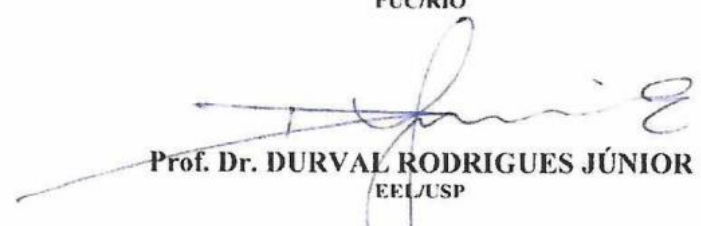
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ABSTRACT

The Ti-6Al-4V alloy is applied in the aerospace industry to produce fan blades and disks of the first stage of gas turbine engines that operate in the temperature range from 20 °C to a temperature range of (250 – 450) °C. During operation, these components are exposed to airborne particles and environmental deterioration responsible for wear, erosion, corrosion and oxidation damage mechanisms. Based on the recent literature, the Cr/CrN multilayer coating improves the corrosion and wear resistance of titanium alloys. However, coatings can be detrimental to the fatigue properties of Ti alloys such as Ti-6Al-4V. The present work aims to investigate the fatigue behavior of the uncoated and Cr/CrN multilayer coated Ti-6Al-4V alloy. The first part of the project was performed with an equiaxed microstructure Ti-6Al-4V alloy substrate and Cr/CrN coating with 8 bilayers (16 layers). The second part of the project was performed with a lamellar Ti-6Al-4V alloy substrate and Cr/CrN coating with 3 bilayers (6 layers). Characterization of crack initiation sites was carried out using SEM and EBSD to identify microstructural configurations prone to crack nucleation and clarify damage mechanisms for the different conditions. In addition, interrupted fatigue tests and in situ tensile tests inside of a scanning electron microscope were performed to elucidate the role played by the coating. The fatigue and dwell-fatigue mechanisms of crack nucleation and the damage mechanisms of the Cr/CrN coating were extensively investigated. The results showed that the coating thickness and number of layers played a role in the fatigue behavior of Ti-6Al-4V alloy since the Cr/CrN coating with 8 bilayers and 5 μm significantly reduced the fatigue strength, while the Cr/CrN coating with 3 bilayers and 1.5 μm nearly maintained the fatigue properties at room temperature. However, the Cr/CrN coating with 3 bilayers also reduced the fatigue life of the titanium alloy at 350°C. Cumulated plastic strain both for fatigue and dwell-fatigue tests were associated with the fatigue mechanism that led to crack initiation, and dwell-fatigue cycles were associated with higher cumulated plastic strain than fatigue cycles. EBSD analysis of the crack nucleation sites revealed that the $\langle a_1 \rangle$ and $\langle a_2 \rangle$ basal slip systems were responsible for the fatigue crack initiation of the lamellar Ti-6Al-4V alloy both at room temperature and 350°C for the uncoated and coated alloy. The early stages of damage in the coating were identified through in situ tensile tests inside of a SEM.

KEYWORDS: Titanium alloys; fatigue; dwell-fatigue; surface treatments; Ti-6Al-4V; Cr/CrN coating.

RESUMO

A liga Ti-6Al-4V é aplicada na indústria aeroespacial para produzir pás e discos do primeiro estágio de motores de turbina a gás que operam de 20 °C a um intervalo de temperaturas de (250 – 450) °C. Durante operação, estes componentes estão expostos a partículas em suspensão e deterioração ambiental responsáveis por mecanismos de dano de desgaste, erosão, corrosão e oxidação. Baseado na literatura recente, o revestimento Cr/CrN de múltiplas camadas melhora a resistência à corrosão e desgaste de ligas de titânio. Contudo, revestimento podem ser prejudiciais às propriedades de fadiga de ligas de Ti como a liga Ti-6Al-4V. O presente trabalho tem o objetivo de investigar o comportamento em fadiga da liga Ti-6Al-4V não recoberta e com recobrimento Cr/CrN de múltiplas camadas. A primeira parte do projeto foi realizada com a liga Ti-6Al-4V equiaxial e um revestimento Cr/CrN com 8 bicamadas (16 camadas). A segunda parte do projeto foi realizada com a liga Ti-6Al-4V lamelar e recobrimento Cr/CrN com 3 bicamadas (6 camadas). A caracterização dos locais de nucleação de trincas foi realizada usando MEV e EBSD para identificar as configurações microestruturais propensas a nucleação de trincas e clarificar os mecanismos de danos para as diferentes condições. Além disso, ensaios de fadiga interrompidos e ensaios de tração in situ dentro de um microscópio eletrônico de varredura foram realizados para elucidar o papel desempenhado pelo revestimento. Os mecanismos de fadiga e de fadiga com período *dwell* para nucleação de trinca e os mecanismos de dano do revestimento Cr/CrN foram extensivamente investigados. Os resultados mostraram que a espessura do revestimento e o número de camadas influenciaram o comportamento em fadiga da liga Ti-6Al-4V pois o revestimento Cr/CrN com 8 bicamadas e 5 µm reduziu significativamente a resistência à fadiga, enquanto o revestimento Cr/CrN com 3 bicamadas e 1,5 µm manteve aproximadamente as propriedades de fadiga em temperatura ambiente. Contudo, o revestimento Cr/CrN com 3 bicamadas também reduziu a vida em fadiga da liga de titânio na temperatura de 350°C. A deformação plástica acumulada para ambos os ensaios de fadiga e fadiga com *dwell* foram associados com maiores deformações plásticas do que ciclos de fadiga. A análise EBSD dos locais de nucleação de trinca revelaram que os sistemas de deslizamento basais $\langle a_1 \rangle$ e $\langle a_2 \rangle$ foram responsáveis pela nucleação de trincas de fadiga na liga Ti-6Al-4V com microestrutura lamelar na temperatura ambiente e 350°C. Os estágios iniciais de dano no revestimento foram identificados através de ensaios de tração in situ em um MEV.

PALAVRAS-CHAVE: Ligas de titânio; fadiga; *dwell*-fadiga; tratamentos superficiais; Ti-6Al-4V; revestimento Cr/CrN.

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1 CHAPTER I: INTRODUCTION

Titanium alloys are applied in the structural area of the aerospace sector due to their low specific mass, high specific mechanical strength, and good corrosion resistance (CUI et al., 2011). The Ti-6Al-4V alloy is used in the production of disks and propellers applied in the first stage of jet engines with operating temperatures that can reach up to 350 °C (DAI et al., 2016). During operation, these components are exposed to airborne particles (wear/erosion), environmental deterioration (corrosion/oxidation), dynamic loadings (fatigue), constant loadings (creep), and the combination of dynamic and constant loadings (dwell effect) (ALTENBERGER et al., 2012; WHITTAKER et al., 2013; WIECIŃSKI et al., 2014a; ZHENG; BALINT; DUNNE, 2017). However, Ti-6Al-4V titanium alloy has elevated reactivity at high temperatures (DAI et al., 2016; DONG et al., 2017) and a high friction coefficient (OLIVEIRA et al., 2014). Therefore, hard coatings are frequently used to improve tribological, corrosion, and oxidation properties at high temperatures (YONEKURA; FUJITA; MIKI, 2015).

The Ti-6Al-4V alloy application in the aeronautical industry increases fuel efficiency, engine thrust-to-weight ratio, payload, and performance of an aircraft by reducing the specific mass (DAI et al., 2016). However, the application of titanium alloys on some components is restricted without the application of surface treatments. Multilayer coatings composed of metal and ceramic layers (e.g., Cr/CrN multilayer coating) are modern coatings that can achieve an outstanding combination of properties. Chromium nitride (CrN) is a hard coating with resistance to friction, erosion, corrosion, and oxidation with thermal stability at temperatures up to 600 °C, while chromium (Cr) is capable of absorbing plastic deformation. Multilayer coatings are also interesting regarding the fatigue behavior since the interface between ductile/brittle layers can suppress the fatigue crack propagation.

The potential of the Cr/CrN multilayer coating to improve the wear resistance and tribological properties of titanium alloys was previously investigated in the literature (ARIAS et al., 2015; MARTINEZ et al., 2003; WIECINSKI et al., 2011; WIECIŃSKI et al., 2014a, 2014b). However, there are several variables that need further investigation to understand the effect of the Cr/CrN coatings on the fatigue behavior of the Ti-6Al-4V alloy during the aeronautical application in the first stage of jet engines. This thesis attempts to address these research gaps by investigating the fatigue and dwell-fatigue behavior of the Ti-6Al-4V alloy with a Cr/CrN multilayer coating at room temperature and 350 °C. Chapter II presents a literature review on the titanium alloy investigated, the fatigue and dwell-fatigue mechanisms, and the effect of surface treatments on the fatigue strength. Chapter III details the experimental

procedures developed in this work. Chapter IV corresponds to the Part I of the results and discussion that was developed in the São Paulo State University (UNESP), Brazil, with an equiaxed Ti-6Al-4V alloy with 8-bilayers Cr/CrN multilayer coating. Chapter V corresponds to the Part II of results and discussion that was developed in Insitut Pprime (P'), France, with a fully lamellar Ti-6Al-4V alloy with 3-bilayers Cr/CrN multilayer coating. Chapter VI summarizes the main conclusions of this work.

1.1 OBJECTIVES

The present thesis aims to investigate the fatigue and dwell-fatigue behavior of the uncoated and Cr/CrN multilayer coated Ti-6Al-4V alloy with equiaxed and lamellar microstructures at room temperature and 350 °C for aeronautical applications. The fatigue mechanisms at room temperature and 350 °C of the titanium alloy, the influence of the Cr/CrN multilayer coating on the fatigue behavior and the combination of dynamic and constant loadings (dwell-fatigue) were investigated in the present work.

6 CHAPTER VI: MAIN CONCLUSIONS

This thesis investigated the fatigue and dwell-fatigue behavior of the uncoated and Cr/CrN multilayer coated Ti-6Al-4V alloy for aeronautical applications with equiaxed and fully lamellar microstructures at room temperature and 350 °C. The Cr/CrN coatings are capable of improving the tribological properties of the titanium alloys according to the recent literature. However, the fatigue damage mechanisms that lead to crack nucleation on the coated titanium alloy need to be investigated to ensure structural integrity. The main conclusions can be summarized as the following:

- The coating thickness and number of layers directly influenced the fatigue behavior of the Cr/CrN multilayer coated Ti-6Al-4V alloy. The deposition of a Cr/CrN coating by pulsed DC magnetron sputtering with 3 bilayers and thickness of 1.5 μm resulted in better fatigue behavior than the coating with 8 bilayers and 5 μm .
- The deposition of Cr/CrN multilayer coating with 8 bilayers and 5 μm significantly reduced the fatigue strength of the equiaxed microstructure Ti-6Al-4V alloy for all stress levels tested. The coating damage mechanism was the premature nucleation of cracks on the brittle layers of chromium nitride that propagated without deflections to the substrate.
- The fatigue life at room temperature of the fully lamellar microstructure Ti-6Al-4V alloy with the Cr/CrN coating of 3 bilayers and thickness of 1.5 μm was slightly increased for the high-stress level of 90% of the yield strength compared to the uncoated material. At the low-stress level of 80% of the yield strength, a slight fatigue life reduction was observed. The experimental results suggest that the Cr/CrN multilayer coating with 3 bilayers could be applied to the Ti-6Al-4V alloy to improve tribological surface properties while maintaining good fatigue properties at room temperature.
- The fatigue damage mechanism at room temperature of the lamellar Ti-6Al-4V alloy was correlated to the plastic strain accumulation during the fatigue process. In general, high-cumulated plastic in the first fatigue cycles were associated to a lower fatigue life. The Cr/CrN coating did not influence the plastic strain behavior of the titanium alloy during the fatigue life compared to the uncoated alloy.
- The dwell-fatigue damage mechanism at room temperature of the lamellar Ti-6Al-4V alloy was related to high plastic strain accumulation during dwell-fatigue cycles, especially the very first dwell-fatigue cycles. The mechanism that explains the significant plastic strain accumulation at room temperature due to the dwell periods of 120 seconds for each dwell-fatigue cycle is the cold creep of titanium alloys.

- The deposition of a Cr/CrN coating with 3 bilayers and thickness of 1.5 μm reduced the fatigue life at 350°C of the lamellar Ti-6Al-4V alloy for the stress levels of 105% and 108% of the yield strength. The coated samples sustained a shorter fatigue life at 350°C than uncoated samples even for similar plastic strain levels at the first fatigue cycle. The cumulated plastic strain of fatigue and dwell-fatigue tests at 350 °C nearly stabilized after the first 100 fatigue cycles. The dwell-fatigue tests at 350 °C occurred at significant higher plastic strain levels than fatigue tests.

- The fatigue damage mechanism of the crack nucleation colonies for the fatigue tests of uncoated and coated lamellar Ti-6Al-4V alloy were investigated using EBSD. The maximum Schmid factor was always associated with the basal plane, which led to the assumption that the basal slip was responsible for fatigue crack nucleation mechanisms at both room temperature and 350 °C for the uncoated and coated Ti-6Al-4V alloy. Furthermore, the fatigue crack nucleation occurred only in colonies favorably oriented for $\langle a_1 \rangle$ and $\langle a_2 \rangle$ basal slip systems.

- In situ tensile tests inside of a scanning electron microscope and the SEM observation of the coatings after interrupted fatigue and dwell-fatigue tests revealed the early stages of damages on the coating that evolve as the number of cycles or stress level of tests increased. The observed coated damage can be correlated to the fatigue strength reduction of Cr/CrN coated specimens.

6.1 FUTURE WORK

The summary of some of the main potential directions for further investigation are listed below:

- Very high-cycle fatigue regime investigation of the Cr/CrN coated Ti-6Al-4V alloy. The fatigue and dwell-fatigue behavior and mechanisms of the coated alloy under real application operational stress.

- Further investigation of the dwell-fatigue mechanisms of Ti-6Al-4V alloy at 350 °C.

- Study of the effect of thickness and number of layers of Cr/CrN coatings on the fatigue strength of Ti-6Al-4V alloy. Experimental fatigue and in situ tests with lower thickness multilayer coatings or superlattices could have potential scientific interest.

- Study of the effect of a ductile interlayer with lower hardness than the titanium substrate in a multilayer coating.

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