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# Experimental and numerical wear studies of porous Reactive Plasma Sprayed Ti–6Al–4V/TiN composite coating $\stackrel{\mathackar}{\sim}$

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## ABSTRACT

The influence of porosity on the wear behaviour of a reactive plasma spray Ti-6Al-4V/TiN coating is investigated through a comparison between experimental results and numerical simulations by finite elements. Samples have been coated using optimized plasma spray conditions and then tested in linear reciprocating sliding against a Ti-6Al-4V ball under different normal loads and number of cycles. Wear tracks were investigated using a combination of scanning electron microscopy, wavelength dispersive spectroscopy and profilometry to assess friction and wear mechanisms. Finite element analysis of the wear problem has been tackled with an iterative 2D model using remeshing to simulate wear and including some of the microstructural features of the coating such as the actual porosity shape and size distribution. Finite element simulations are able to reproduce the wear kinetics observed experimentally. In addition both experimental and numerical analyses reveal that pores within the coating layer may represent weak points for the wear resistance.

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# 1. Introduction

The combined high strength and low density of titanium alloys and their excellent resistance to corrosion make them well suited for applications in aerospace and automotive industry. However, the poor wear resistance of titanium alloys precludes their use for many components subjected to sliding contact [1,2]. Surface engineering technologies, as plasma spraying for thick coatings or physical vapor deposition for thin films, are increasingly seen as being keys to enable the use of titanium in applications traditionally not compatible with the material characteristics. As such, there is a growing need for techniques which are capable of providing reliable information regarding the performance of thermochemically treated and/or coated alloys.

One of the interesting deposition techniques is Reactive Plasma Spraying (RPS) which allows us to *in situ* achieve a metal matrix composite material as a thick layer. A better understanding of the relationship between tribological behaviour and the microstructure of these composite coatings is a key requirement for industrial applications. Porosity and surface roughness are the most relevant features for thermally sprayed coatings and have strong influence on the coating response under contact loading.

RPS produces knowingly porous coatings, so how the porosity affects the wear performance of materials is of importance to materials design and processing. There are multiple variables concurrently involved in this process; elastic modulus, plasticity, reinforcing phase, matrix/reinforcement interfacial bond strength, porosity size, shape and distribution all need to be considered, which makes it difficult to take a close look at the wear process and to elucidate the experimentally determined overall wear performance of materials under the influence of porosity [3]. For instance, the influence of porosity and roughness on the wear behaviour of materials has been reported as sometimes beneficial or detrimental in several studies and is thus not clearly identified yet. Chen et al. investigated the influence of porosity on solid-particle erosion of composite materials with the aim of clarifying relevant unclear issues [3]. They showed that the porosity could be beneficial to the performance of composite materials under some conditions. A study combining neutron scattering and X-ray tomography showed the importance of the layered spatial distribution of the pores in thermal spray coatings as opposed to a more homogeneous distribution in RPS coatings [4]. A study conducted by Branco et al. showed that, to some extent, pores in materials were beneficial to the wear resistance of ductile materials but harmful for brittle materials [5]. Simchi and Danninger investigated effects of porosity on wear of sintered plain iron and also observed a positive influence of porosity on its wear behaviour [6]. They explained that the positive effect of porosity on wear could be attributed to a decrease in the contact pressure and resulting plastic deformation near the pores. Besides, pores could also act as a trap for wear debris to reduce abrasion.





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The presence of pores usually results in detrimental influence on the performance of materials. For instance, through experimental and finite element analysis, Hardin and Beckermann demonstrated an apparent reduction in elastic moduli of steel due to the presence of pores [7]. Experiments performed by Deshpande and Lin showed that the porosity in WC/Cu composites decreased their wear resistance [8]. The influence of porosity on the wear behaviour of materials depends on their microstructures and the wear condition. The presence of porosity can be very detrimental to the wear resistance of composite materials [9–11]. The potentially negative effect of porosity on the wear resistance is influenced by pore size, shape and density as they may act as stress risers to facilitate damage and crack initiation.

In a preliminary work [12], the influence of chamber pressure on *in situ* nitridation has been studied. Two spherical Ti–6Al–4V (25–45  $\mu$ m and 45–75  $\mu$ m) powders were sprayed using High-Pressure RPS and conventional RPS modes in a Controlled Atmosphere Plasma Spraying (CAPS) system (Sulzer-Metco AG, Wohlen, Switzerland). The coatings exhibit fine titanium and rather coarse nitrides' reinforcements which formed during spraying.

The complexity of sliding surfaces interaction is a limitation in wear modelling even if more than a hundred wear models are listed in [13]. Two approaches have traditionally been adopted: on one hand, a macroscopic estimation of the total wear volume using an energetic approach, and on the other hand local wear rate determination which allows us to predict the wear profile. Most of the wear models use a modified Archard's law to assess either the local or global wear rate [14]. Some local wear models adopt semi-analytic methods to predict wear [15–17]. This has been used for instance to define the optimum geometry regarding fretting loading, for three-dimensional industrial problems. Other local wear models implement a modified Archard's law into a FEM code [18,19] and are able to reproduce the geometry evolution of the system for problems with reasonable size. In this paper, a local FE wear solution is adopted as described in Section 4.2.

The present paper investigates the influence of RPS coatings porosity on their wear behaviour and features a comparison between experimental results and numerical simulations by finite elements. Coated samples have been tested under reciprocating sliding to study the wear response. Wear tracks were studied using a combination of SEM, WDS, and profilometry to investigate the friction and wear mechanisms. Coating life is analyzed using a dissipated energy criterion and related to the experimental loading conditions. A numerical model using 2D plane strain finite element analysis was developed to evaluate the possible effect of the pores as stress concentration and wear accelerators. Numerical results are compared to experimental tests.

#### 2. Experimental methods

### 2.1. Materials

The material investigated in this study is Ti-6Al-4V, which titanium-based composite coatings were achieved by spraying of

Ti-6Al-4V powder using High-Pressure Reactive Plasma Spraying (HPRPS) in nitrogen. Plasma spraying experiments were carried out using the CAPS system of the Centre for Plasma Processing at MINES ParisTech with a F4-MB plasma gun. The equipment has an 18 m<sup>3</sup> chamber, which can operate in a controlled atmosphere of air, argon or nitrogen from 2 to 350 kPa. Ti-6Al-4V (25-45 µm) powder (PyroGenesis Inc., Canada) was sprayed using HPRPS in nitrogen at the pressure of 200 kPa onto a Ti-6Al-4V substrate [20]. Coating obtained under these spraying conditions has been studied previously by XRD phase analysis and nanoindentation on cross-section [12,21,22]. During the coating process, sand-blasting is used to clean the surface before deposition and to increase the substrate roughness to obtain a larger contact area leading to a higher adhesion. Ti-6Al-4V specimens were grit blasted with 350 µm white alumina grits and cleaned with ethanol in an ultrasonic bath. The average measured (Hommel surface tester T500) surface roughness  $(R_a)$  of the grit-blasted plates was 3.0–3.5  $\mu$ m. Coatings exhibited  $\alpha$ -Ti and  $\delta$ -TiN, which is the sign of actual in situ formation of titanium nitrides when spraying. Moreover, the formation of nitrided compounds was studied using standard X-ray diffraction and electron probe microanalysis with the help of scanning and transmission electron microscopy (TEM) techniques to assess the pressure assisted nitriding of the Ti-6Al-4V [12]. For TEM observations, foils parallel to the coating surface were ion-thinned and analyzed with a 300 kV EM430-T TEM from FEI. the Netherlands.

A typical cross-section observed by optical microscopy is shown in Fig. 1a. As-sprayed roughness is evaluated to  $R_a =$  $5.1 \pm 0.2 \,\mu\text{m}$  and  $R_t = 39.6 \pm 2.5 \,\mu\text{m}$  and thickness  $151 \pm 11 \,\mu\text{m}$ . Etching with Kroll reagent revealed a typical Ti–6Al–4V/TiN lamellar microstructure. The titanium nitride content modifies the image grey level: dark grey regions correspond to highly nitrided lamellae, whereas light grey lamellae are considered as plain Ti–6Al–4V and termed as "Ti–6Al–4V matrix", Table 1. Some nitrided lamellae showed a typical dendritic microstructure with coarse TiN dendrites. Apart from dendrites, nitrided lamellae showed a homogeneous distribution of nanosized TiN precipitates in a Ti–6Al–4V matrix as seen on TEM micrographs (see Fig. 1b). This is due to high rates in the nitriding and solidification process in reactive plasma spraying.

#### 2.2. Wear tests

All coated specimen surfaces were carefully polished using SiC grid paper and diamond suspension  $(1 \ \mu m)$  before testing to avoid any influence of the surface quality on the wear results. Wear tests were carried out at room temperature using a CETR UMT-3 tester in ball-on-disk configuration with linear motion. The upper section of the testing unit has both vertical and lateral positioning systems to control the location and loading force of the upper test specimen. The normal-load sensor provides feedback to the vertical motion controller, actively adjusting the sample position



Fig. 1. (a) Optical micrograph showing a typical microstructure of the studied coating, etched with Kroll's reagent; (b) TEM micrograph of a rich TiN lamellae showing the nano sized TiN precipitates.

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