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Damage phenomena of thin hard coatings submitted to repeated impacts: Influence of the substrate and film properties

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ABSTRACT

To evaluate the surface fatigue resistance of some thin nitride films obtained by Physical Vapour Deposition (PVD) techniques, repeated impact tests have been performed under controlled impact conditions. Short and long duration tests have revealed the occurrence of an original damage phenomenon likely linked to a mechanical blistering of the films. As these blisters appear to be the first damage step, their formation has to be understood in order to be avoided in industrial applications. In particular, the role of the mechanical properties of the substrate has to be clarified as thin protective coatings may be used on pieces prepared using various heat treatments. finite element method (FEM) analysis has been conducted in order to better understand the specific mechanical conditions in the substrate and at the film–substrate interface that could lead to such blistering phenomena. Correlations with the experimental results have been evidenced. From the modelling results the substrate properties have been shown to be of significant influence on the blister formation. However as they do not fully explain the origin of this phenomenon, the influence of the substrate microstructure has also been studied and the presence of vanadium carbides appears to be of major effect.

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1. Introduction

Thin hard coatings are today well known solutions to improve surface performances and increase the component endurance life. Among them, nitride based films are one of the most popular coatings family due to their interesting mechanical properties. If various elaboration methods can lead to the realization of thin hard coatings on complex industrial parts, dry processes like Physical Vapour Deposition (PVD) or Chemical Vapour Deposition (CVD), are now widely used to coat mechanical parts submitted to severe tribological conditions. However, depending on the elaboration process and the particular deposition conditions, these films may exhibit very different performances. There is therefore a need for reliable characterisation methods, which could really give valuable information about the possible coatings in-service behaviour. Usual characterization methods like scratch or indentation tests are widely used but cannot satisfactorily describe the coatings long term performance [1,2]. To enable a better endurance life prediction, repeated impact testing strategies have been developed. Most of the research groups in domain are using high

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number of impacts of high energy to establish global S–N curves but without real insights in the damage origins [3,4]. In our case, attention has been focussed on the origin of the materials damage in order to be able to understand the initial phenomenon and its evolution law. Using this specific approach, not only endurance life curves (S-N) can be obtained [5,6] but also valuable information about the way to improve the coating performances. The present paper will focus on this last point.

During the first testing period, where no apparent damage can be evidenced, blisters appeared on some thin hard coatings tested under specific impact conditions. In the literature, blistering of films is mainly studied in relation with the surface preparation or elaboration process conditions [7-10]. Blisters of various geometrical shapes (round, telephone cord ...) may then be observed [11,12]. In the case of Thermal Barrier Coatings/oxides, uncontrolled elaboration parameters can lead to the creation of extremely severe compressive stresses in the growing coating that can induce spontaneous coating delamination and blistering. In extreme situations, complete spalling can be observed as soon as the deposition process stops [7,8]. Blistering phenomena may also be observed during oxidation experiments when the oxide films reach critical thickness values [13]. Mechanical models, related to the mechanical properties and thickness of the film, have been proposed to explain this blisters formation [11-14].

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In [13], Strawbridge and Evans reviewed the different possible cases (thermal or mechanical origin, various blister shapes). In the case of a round (radius *R*) already fully de-laminated area, buckling is observed if the compressive residual stress in the film reaches a critical value σ_c that can be determined by the following equation from [13]

$$\sigma_c = \frac{122E_d}{1 - v_d^2} \left(\frac{e}{R}\right)^2 \tag{1}$$

where e is the thickness, E_d the Young modulus, v_d the Poisson coefficient of the film and *R* the radius of the de-laminated area (insufficient adherence). From this equation, He. et al. [9] and Wang and Evans [15] proposed a buckling index $\Pi = (1-v^2)/(\sigma/E)(R/e)^2$ that characterizes the buckling risk for a delaminated coating. It is to be noted that this equation and the Π index are not directly related to the reason of the film de-lamination that can be linked to an inappropriate elaboration process or to progressive crack propagation under mechanical conditions. Therefore, the strength of the coating/ substrate interface does not appear in Eq. (1) and is not used in numerical models [16–18]. However, this interfacial strength determines the blister stability that can be predicted using an adhesion index \sum defined by Evans and co-workers in [19] as

$$\sum = \sigma \sqrt{e(1-v)/E\Gamma_i} \tag{2}$$

As all blister formed under our experimental impact conditions are round and stable, one can deduce from [19] that $\sum < 1.85$ but no real estimation of the interfacial strength of our coating to be made. However, if the mechanical behaviour of a pre-delaminated film can be fully predicted [9–13] and local stress relief induced in films by buckling can be estimated [20], the specific conditions that lead to buckling under repeated impacts have to be clarified. In particular, the influence of the substrate hardness remains quite uncertain. If preliminary tests on M2 steels seem to indicate that a high hardness inhibits blister formation, results obtained on ductile C48 steel (AISI 1050) appear somehow contradictory. One unquestionable fact is that no blisters are observed on untested samples whatever the steel substrate nature and heat treatment. This suggests that the buckling conditions are induced by the impacts repetition.

The objective of the present work is then to better understand the conditions of the blister formation on various thin PVD hard nitride coatings and to evidence the influence of the substrate mechanical properties and/or microstructure on this blistering mechanism. Depending on the results, heat treatments or hardness, recommendations will be provided in order to avoid thin film blistering under impact conditions.

2. Experimental

2.1. Impact tester

The principle of the impact tester used in this study is sketched on Fig. 1 [5,6,21]. A rigid indenter, ended by a hemispherical tip electromagnetically is accelerated and pushed on the sample surface under normal incidence.

During this work, 100Cr6 (AISI 52100, which has the base composition of 1C–0.3Si–0.3Mn–1.5Cr(wt%)) steel balls of 10 mm radius were used as the impacting tip leading to a total indenter mass of 165 g. As a constant acceleration is generated by the electromagnets, the indenter velocity and kinetic energy just before the impact can be adjusted using the indenter initial position above the sample surface.

This incident velocity was also checked using a laser diode displacement sensor recording the tip position during each impact cycle [22]. Impact speeds ranging from 0.05 to 0.3 m/s were used



Fig. 1. Principle of the impact tester.

Table 1		
Typical	testing	conditions

Parameter	Range of values
100Cr6 ball radius	10 mm
Kinetic energy (ball 165 g)	0.2-8 mJ
Impact Force	50-2500 N
Number of Impacts	$1-2 \times 10^6$ impacts
Impact Speed (m/s)	0.05-3 m/s
Impact Frequency	10 Hz

during this study, leading to kinetic energies ranging from 0.2 to 8 mJ. As the induced impact load depends not only on the kinetic energy of the indenter but also on the mechanical properties of the tested sample and the geometry of the indenter, it has to be determined for each testing condition. Due to the normal incidence and the limited penetration depth during each cycle (confirmed in the following by experimental and numerical results), only the normal component of the load (Fn) induced in the impacted material was considered and measured during each cycle using a piezoelectric transducer. Typical testing conditions are summarized in Table 1.

A set of reference testing conditions was chosen to be used for both modelling and experimental work in order to enable valuable comparisons. For these reference tests, the testing parameters were adjusted to ensure an incident kinetic energy of 0.8 mJ to enhance the influence of the mechanical properties of the substrate as well as the blistering phenomenon. Unless otherwise mentioned all results presented in the following were obtained under these reference conditions.

2.2. Materials

CrN thin coatings were elaborated at the LERMPS laboratory on annealed and treated (oil quenching+1h30 tempering at 550 °C) M2 steel in order to achieve low (230 Hv₅₀) and high (790 Hv₅₀) substrate hardnesses. Each coating series were elaborated using the same experimental protocol (mirror polishing of the substrate, cleaning and deposition steps). The resulting film thickness was determined using a 3D optical profilometer. The Young modulus of the coatings was deduced from nanoindentation tests using a Nanoindenter XP2

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