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Fracture mechanics based analysis of the scratch resistance of thin brittle coatings on a soft interlayer

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ABSTRACT

The scratch resistance of thin brittle coatings deposited on rigid substrates with a soft intermediate layer has been characterized by instrumented nanoscratch testing in order to identify the damage mechanisms. Several "hard thin film/polymer/rigid substrate" multilayers have been addressed with varying compositions and thicknesses. The relevance of the use of nanoscratch with respect to end-user performances has been confirmed by the analysis of the damage and cracking mechanisms triggered by more empirical tests. The scratch wear resistance behavior is interpreted in the light of fracture mechanics since the dominant damage mode is the cracking of the brittle film. This behavior is related to the mechanical properties of the individual layers, e.g. elastic modulus, hardness, fracture toughness, internal stress, and to the soft interlayer thickness. The influence of the soft interlayer on the enhancement of the cracking energy release rate in the coating plays a major role, depending directly on the elastic mismatch and adhesion characteristics. The fracture toughness and adhesion of the hard coating jointly determine the morphology and the extension of the crack density and the amount of delamination, whereas the soft interlayer thickness controls the scratch depth. The constraint effect induced by the rigid substrate for thinner interlayer thicknesses favors the cracking of the hard coating despite lower scratch depths.

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1. Introduction

Following the trend towards the development of high performance multifunctional composite materials, the development of more efficient coatings goes along with the use of multilayers in order to take advantage of the different properties of several constituents. The mechanical properties of a multilayer do not result from a simple law of mixture of the individual layer properties and very different responses and load transfers between the layers result in complex phenomena, with damage mechanisms and competing effects that are not encountered within bulk or monolayer systems. A better understanding of the relationships between the mechanical properties of each layer and the scratch and wear resistance of the overall stack is of major importance to guarantee and improve the reliability and lifetime of various technologies that are based on such multilayered systems.

This research focuses on a particular range of functional multilayers which are of the type "hard brittle thin film/polymer interlayer/rigid substrate" (see Fig. 1). Surface protection (paints) and advanced optics are two mainstream fields of application where such multilayers are encountered. Multilayer systems involving thin hard brittle films and polymer layers are also encountered in novel flexible electronics applications, e.g. [1].

The systems under interest in this study are characterized by the significant stiffness and hardness mismatch between the upper hard layer and the soft interlayer. This configuration favors the cracking of the upper film due to the enhancement of the crack energy release rate associated to the total elastic energy stored in the soft layer, e.g. [2,3]. The fracture of the hard coating is therefore expected to play a crucial role in the scratch and wear resistance of such systems. The fracture properties of the top film are expected to influence the damage mechanism during scratching. In addition, the presence of

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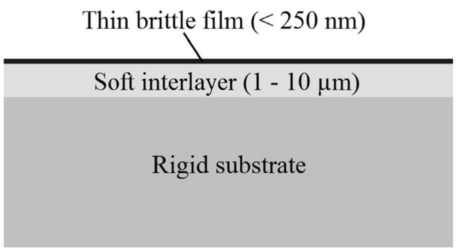

Fig. 1. Schematic illustration of the "hard brittle thin film-polymer interlayer-rigid substrate" configuration.

the rigid substrate below the soft interlayer constrains its deformation compared to the case of a bulk polymer. As a consequence, the stresses generated in the top hard layer also differ from the case of a coated bulk polymer. Hence, we analyze also the effect of the polymer thickness on the damage mechanisms in order to unravel the impact of the constraint effect induced by the underlying rigid substrate as a function of the soft interlayer thickness.

Model systems composed of a few hundred nanometer thick brittle layer, namely either chromium nitride or zinc oxide have been deposited on polymer layers of various thicknesses ranging between 1 and 10 µm, themselves deposited on a rigid substrate, either steel or silicon. In the present paper, the focus is mainly on the chromium nitride film. The observations on the zinc oxide film are given only to show that the results are not specific to one system but can be indeed generic to the class of "hard brittle thin film/polymer interlayer/rigid substrate" systems.

The mechanical properties of the film and of the polymer have been characterized using various techniques involving nanoindentation, a so-called "lab-on-chip" method for testing freestanding films and wafer curvature measurements. In parallel, the scratch resistance has been investigated by nanoscratch measurements completed with scanning electron microscopy in order to identify the damage mechanisms. The validity of the approach has been confirmed by the analysis of the damage and cracking mechanisms occurring during empirical tests that are commonly used in the industry to evaluate the scratch and wear resistance of products.

The experimental procedures and materials are presented in Section 2. Section 3 describes the experimental results, followed by a discussion in Section 4, before final conclusion.

2. Materials and methods

2.1. Sample preparation

The brittle coatings are made of chromium nitride (CrN). The chromium based films have been deposited by physical vapor deposition in a reactive magnetron sputtering chamber at room temperature using a continuous pilot line process with a chromium target. The target power was set to 580 W and the substrates were grounded. The nitrogen concentration in the inflowing gas has been varied in order to generate three different compositions: chromium (Cr), CrN with low nitrogen content (CrNlow) and CrN with high nitrogen content (CrNhigh). The chemical composition measured by X-ray photoelectron spectroscopy (XPS) for the films deposited on steel and on silicon is 56 at% Cr, 36 at% N and 8 at% O and 50 at% Cr, 42 at% N and 8 at% O, respectively for the CrNlow and the CrNhigh films. The composition is constant over the film thickness. X-ray diffractograms obtained by transmission electron microscopy indicate that the CrN films are essentially amorphous, except for the presence of some Cr_2O_3 crystallites. Nevertheless, it cannot be excluded that very fine nanocrystalline CrN domains are present.

The films were deposited on two different substrates. The first one is an industrial sheet composed of steel already pre-coated with a 1-µm thermoset polymer, namely polysilazane (ThS1-steel). The second one is a "model system" composed of a silicon wafer that has been spin-coated with various thicknesses of polyimide (PI). An adhesion promoter has been used to enhance the adhesion of the polyimide on the wafer. The first family of samples with the ThS1-steel substrate has been mainly used for analyzing the influence of the hard film properties on the scratch and wear behavior whereas the second family of samples with polyimide–silicon substrates has been used for assessing the influence of the soft interlayer thickness. The thicknesses of the different samples are given in Table 1.

In addition to these "hard thin film/polymer interlayer/rigid substrate" samples, other samples without the soft interlayer have been produced for characterization purposes. First, specimens have been processed by depositing the hard film directly on a bare silicon wafer (Si). Secondly, using photolithography techniques, freestanding 200 nm thick CrNlow and CrNhigh beams have been produced for testing following the "lab-on-chip" concept. This technique described in details in [5–8] allows performing tensile tests on a wide range of thin film materials with very good control of the alignment. It was not possible to fabricate the same samples with the chromium film due to very high internal stress which led to the cracking of the film during the process.

2.2. Mechanical properties of the layers

The mechanical properties of the brittle top film and of the soft interlayer have been measured using a combination of various techniques.

The internal stress in the brittle films has been evaluated by Stoney's method which is based on the curvature change of the silicon wafer induced by the deposition of a film under stress [9,10].

The Agilent G200 nanoindenter has been used for the determination of Young's modulus, hardness and fracture toughness. Young's modulus of the soft interlayers has been measured using the standard XP head, whereas all indentation experiments on the brittle films have been performed using the high precision DCMII head (Dynamic Contact Module). The two heads have been equipped with a Berkovich-shaped diamond tip and the modulus measurements were performed using the continuous stiffness mode (CSM). Young's modulus of the soft interlayers has been determined using the Oliver and Pharr method [11], while Young's modulus of the brittle films has been extracted based on the Li and Vlassak correction [12] to account for the substrate effect. The high modulus difference between the polymer and the upper film does not allow extracting Young's modulus and hardness of the film

Table 1
Description, composition and dimensions of the specimens tested in this work.

Reference	Rigid substrate	Soft interlayer		Brittle top layer	
	Material	*Material*	*Thickness* (µm)	*Material*	*Thickness* (nm)
Cr-Si	Silicon	–	–	Cr	100 ± 5
CrNlow-Si	Silicon	–	–	CrNlow	100 ± 5
CrNhigh-Si	Silicon	–	–	CrNhigh	100 ± 5
CrNhigh-PI1-Si	Silicon	Polyimide	0.8	CrNhigh	100 ± 5
CrNhigh-PI3-Si	Silicon	Polyimide	2.9	CrNhigh	100 ± 5
CrNhigh-PI6-Si	Silicon	Polyimide	6.3	CrNhigh	100 ± 5
CrNhigh-PI9-Si	Silicon	Polyimide	9.7	CrNhigh	100 ± 5
Cr-ThS1-steel	Steel	Thermoset	1	Cr	100 ± 5
CrNlow-ThS1-steel	Steel	Thermoset	1	CrNlow	100 ± 5
CrNhigh-ThS1-steel	Steel	Thermoset	1	CrNhigh	100 ± 5

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