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Contact damage and fracture micromechanisms of multilayered TiN/CrN coatings at micro- and nano-length scales

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ABSTRACT

In this study, systematic nanomechanical and micromechanical studies have been conducted in three multilayer TiN/CrN systems with different bilayer periods (8, 19 and 25 nm). Additionally, experimental work has been performed on corresponding TiN and CrN single layers, for comparison purposes. The investigation includes the use of different indenter tip geometries as well as contact loading conditions (i.e. indentation/scratch) such to induce different stress field and damage scenarios within the films. The surface and subsurface damage under the different indentation imprints and scratch tracks have been observed by atomic force microscopy, field emission scanning electron microscopy and focused ion beam. Multilayer TiN/CrN coated systems are found to exhibit higher adhesion strength (under sliding contact load) and cracking resistance (under spherical indentation) than those coated with reference TiN and CrN monolayers. The main reason behind these findings is the effective development of microstructurally-driven deformation and cracking resistant micromechanisms: rotation of columnar grains (and associated distortion of bilayer period) and crack deflection of interlayer thickness length scale, respectively.

1. Introduction

A variety of multilayered transition metal nitride coatings have been extensively studied during the last two decades because of their promising properties to improve the lifetime of tools and components for engineering applications, e.g. cutting and forming tools. The fact that physical vapor deposition of both TiN and CrN is a well-established technique has promoted the interest on investigating TiN/CrN multilayers by different research groups [1–13]. In this regard, several issues have been addressed: microstructural characterization [2,3,6–10], chemical stability [3,8], thermal stability [4], residual stress assessment [5,7,8], in-situ deformation mechanisms [12], friction/wear behavior [1,4,7,8,11, 13], and surface fatigue [7,8]. However, although bilayer period (chemical modulation period) is commonly recognized as key parameter for defining final performance of multilayered TiN/CrN systems, only few of the referred studies have considered it as an experimental variable (e.g. Refs. [1,5,10]), this being particularly true concerning their mechanical response and induced damage under contact load.

In this study, a systematic nanomechanical and micromechanical study has been conducted in three multilayer TiN/CrN systems with different bilayer periods (8, 19 and 25 nm). Additionally, experimental work has been performed on corresponding single layers, for comparison purposes. In all the cases, a commercial high speed tool steel has been used as substrate. The investigation includes the use of different indenter tip geometries as well as contact loading conditions (i.e. indentation/scratch) such to induce different stress field and damage scenarios within the films. Special attention has been paid to document and analyze main damage and fracture mechanisms at the micrometric and nanometric length scales: deformation features and crack paths, and their interaction with microstructure and layer assemblage, interlayer and/or coating/substrate interface. In doing so, advanced microscopy techniques, as atomic force microscopy (AFM) and field emission scanning electron microscopy (FE-SEM) have been used. Furthermore, sectioning using focused ion beam (FIB) and FIB-tomography has been implemented to assess the local surface damage under the different indentation imprints and scratch tracks. It is found that mechanical response under contact loads of multilayered TiN/CrN is enhanced, with respect to that exhibited by TiN and CrN monolayers, by effective

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development of microstructurally-driven deformation and cracking resistant micromechanisms.

2. Experimental procedure

2.1. Coating deposition

Substrate material is a commercial high speed steel (HSS, M2/EN-1.3343) thermally hardened prior to deposition (63 HRC — about 8 GPa), and polished to a 0.5 μm surface finish. Five different coatings (TiN/CrN with three different bilayer periods: 8, 19 and 25 nm; as well as monolayer TiN and CrN) were deposited in an industrial scale METAPLAS MZR323 system. It was done using multisource cathodic reactive arc evaporation with an arc current of 60 A and a burning voltage of 200 V. Cathodes of Cr and Ti, located in each side of the reactor (Fig. 1) were used to produce CrN and TiN films with different bilayer periods. Before loading into the chamber, substrates were cleaned with alkaline detergent as well as in an acetone ultrasonic bath. The ion cleaning was carried out by arc-enhanced glow discharge in argon plasma. Prior to the multilayer deposition process a Cr-rich interlayer at the substrate-coating interface was deposited, in order to improve coating adhesion as well as to reduce stresses at the interface. Depositions were conducted in a 1.10 Pa N_2 atmosphere using a substrate bias of -200 V, and a substrate temperature of 450 °C. Substrates were mounted in two rows on the rotating substrate holding drum (see Fig. 1). Periodicity of the multilayer is controlled by the substrate rotation; thus, rates of 2, 4 and 8 rpm resulted in bilayer periods of 25, 19 and 8 nm, respectively, yielding a coating thickness of 2 μm for all the specimens.

2.2. Glow discharge optical emission spectroscopy

Chemical profiles were determined by glow discharge optical emission spectroscopy (GDOES) using a Jobin-Yvon 10000RF equipment with a 2 mm copper anode. The analysis was carried out with an incident radio frequency power of 20 W inside an argon atmosphere at a pressure of 620 Pa. The equipment was calibrated for quantification by material standards.

2.3. Mechanical response under contact loading

The mechanical characterization of the coated systems included the evaluation of their effective hardness (H) and elastic modulus (E) through instrumented indentation. It was performed using a nanoindenter XP (Agilent Technologies) equipped with a continuous stiffness measurement module, the latter allowing a dynamic determination of hardness and elastic modulus during the indentation [14]. Indentations were organized in a regularly spaced array of 16 indentations (4 by 4) at 2000 nm penetration depth (or until reaching maximum applied load, i.e. 650 mN) with a constant distance between each imprint of 50 μm in order to avoid any overlapping effect, and the results were averaged. The strain rate was held constant at 0.05 s^{-1}. The indenter shape was carefully calibrated for true penetration depths as small as 60 nm by indenting fused silica samples of accurately known Young's modulus (72 GPa). The values of hardness and elastic modulus were calculated by the Oliver and Pharr method [15], assuming a constant Poisson ration for the specimens of study equals to 0.25 [16].

Indentations with a cube-corner tip were done to produce cracking in the coatings at different loads of 50, 100, 150, 200, 250 and 500 mN; with a distance of 30 μm between each imprint.

Damage induced under contact load was assessed by means of spherical indentation (Hertzian tests) in order to identify and document damage emergence at the surface of coated substrates (e.g. circumferential cracks, cohesive spalling and/or adhesion failure). Tests were conducted in a servohydraulic testing machine (Instron 8511) using hard metal indenters of 2.5 mm of curvature radii [17–19]. Applied load ranged from 200 to 1200 N, at a loading rate of 30 $N \cdot s^{-1}$ and a holding time of 20 s.

Sliding contact tests were done at both nanometer and micrometer length scales. The former was carried out by means of a nanoscratch fixture attached to the nanoindenter system referred above. A Berkovich indenter was employed to scratch the coating surface under increasing load, up to a maximum value of 500 mN, at a velocity of 10 μm/s. Three different scans were done in each sample with a scratch length of 500 μm and in an interval length of 500 μm.

Microscratch tests were performed in a Scratch tester unit (CSM-Intruments) with a spherical diamond indenter of 200 μm in radius. Scratches were made under linearly increasing load, from 0 to 100 N,

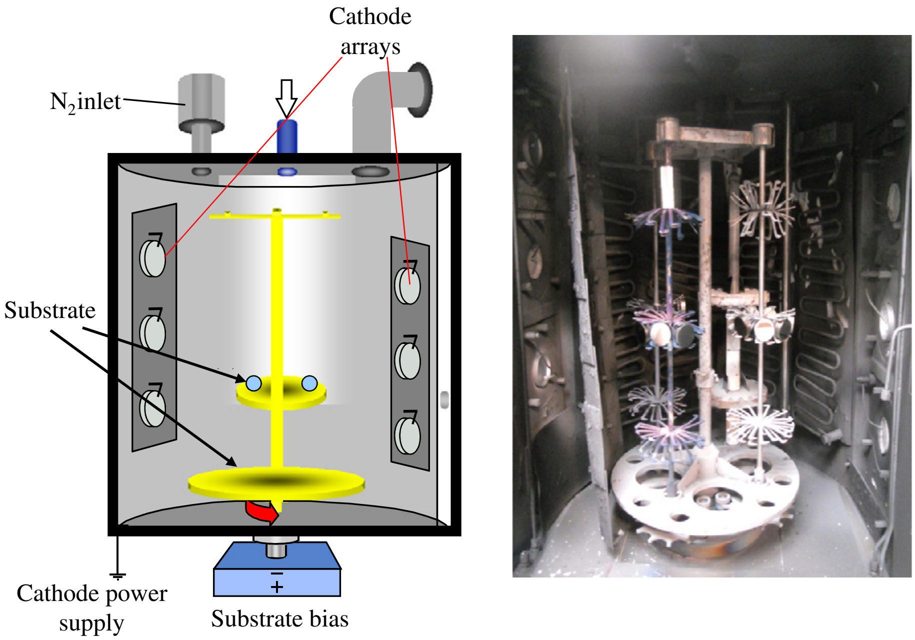

Fig. 1. Schematic and real image of the coating deposition system used in this investigation.

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