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Multilayered and nanolayered hard nitride thin films deposited by cathodic arc evaporation. Part 1: Deposition, morphology and microstructure

C. Ducros [a,*], C. Cayron [b], F. Sanchette [a]

[a] *CEA Grenoble, DTEN/STN/LTS, Laboratoire de Technologies des Surfaces, 17 rue des Martyrs 38054 Grenoble Cedex, France*
[b] *CEA Grenoble, Laboratoire Analyses, Mécanismes et Modélisation, 17 rue des Martyrs 38054 Grenoble Cedex, France*

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Abstract

TiN/AlTiN, TiN/CrN and CrN/AlTiN multilayer coatings have been deposited by the cathodic arc evaporation technique. The period is in the range 7–200 nm for a total thickness of 3 μm. The period's control of the nanoscaled hard films is achieved, a priori, by way of a simple geometrical calculation and, a posteriori, via both X-ray diffraction and transmission electron microscopy on cross-sections. Microstructure of the as-deposited coatings has been investigated by means of X-ray diffraction and transmission electron microscopy in connection with the decrease of the period λ. For lower periods (multilayered coatings), the fcc structures which derive from each nitride are observed while only the superlattice structure is found for nanoscale layered films (nanolayered coatings). Microstructure evolution with the period is investigated for the three systems and the differences are comment. Tribological behaviours and cutting performances are studied in [C. Ducros, F. Sanchette, submitted to Surf. Coat. Technol.].
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Keywords: Arc evaporation; Superlattice; TEM; X-ray diffraction

1. Introduction

The demand for PVD hard coatings exhibiting properties such as high wear and oxidation resistance has grown enormously during the last decades particularly for mechanical applications in severe environment as machining. Evolvement of both machining techniques and materials to be machined led to the development of multilayered coatings and, recently, to super-hard superlattice structure [2–4]. It is well established that the bond structure in transition metal nitrides is responsible for high hardness, high wear resistance and chemical inertness. Moreover, AlTiN is well known for its high oxidation resistance enabling improved performances in hard material machining [5–7]. Therefore, AlTiN based superlattice coatings, which have been already extensively investigated [3,8,9], are good candidates for cutting difficult-to-machine materials such as nickel based alloys.

The purpose of this study was to control multilayer or superlattice coatings deposition on cemented carbide cutting tools in an industrially sized arc-PVD system. Morphology and micro-structure of TiN–(Al,Ti)N, TiN–CrN and CrN–(Al,Ti)N as deposited coatings with different periods λ were also investigated in order to be correlated with tribological behaviours and cutting performances which are studied in [1]. The main goal was to establish the relationships between deposition conditions, micro-structure, hardness and wear/friction measurements and, ultimately, the Ni based alloys cutting performances.

2. Experimental

All the coatings were deposited by cathodic arc evaporation in an IMD 700 Plassys system equipped with 4 random arc BMI sources (100 mm in diameter) and with a threefold rotating substrate holder [10]. AlTiN, TiN and CrN were elaborated from AlTi (60 : 40 at.%), pure Ti and pure Cr targets. Hardened M2 HSS and glass substrates were cleaned in a heated alkaline solution, degreased in acetone and rinsed in alcohol prior to deposition. A first ion etching is performed at 3 Pa in pure argon (bias: −800 V) during 30 min prior to a metal etching (chromium or titanium ions are used when CrN or TiN is

* Corresponding author. Tel.: +33 4 38 78 97 86.
E-mail address: Cedric.ducros@cea.fr (C. Ducros).

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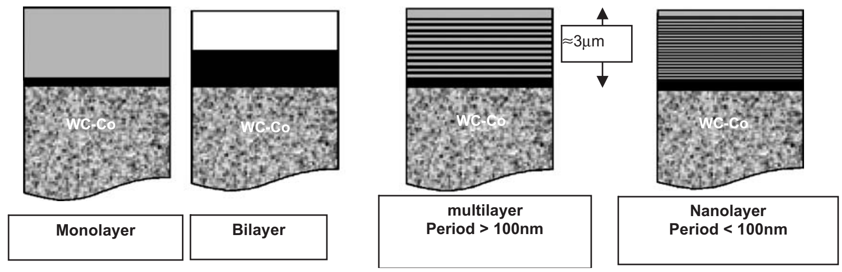

Fig. 1. Scheme of the different coatings architectures. The total thickness is kept constant at 3 µm.

deposited, respectively) performed in pure argon at 1 Pa (bias: −600 V) during 10 min. During the deposition stage, nitrogen pressure (pure nitrogen atmosphere), arc intensity and substrate bias voltage are 1 Pa, 100 A and −150 V. Deposition temperature is kept constant at 400 °C.

TiN or AlTiN are deposited as top layer for TiN/CrN or TiN/AlTiN and CrN/AlTiN coatings, respectively and the interlayer is a thin pure metal (Cr or Ti) film. This interface, which has an intermediate hardness, allows accommodation between the substrate and the hard coating and thus favours adhesion.

Two face to face pairs of cathodes with Ti/AlTi, Cr/AlTi and Ti/Cr targets are used to deposit multilayered coatings with different periods from bilayered film to multilayered coatings (12 to 52 layers) and then to the superlattice structure (Fig. 1). The total thickness is kept constant at 3 µm. The period λ is controlled via the speed of the turntable (700 mm in diameter) of the threefold rotation substrate holder. In this study, a two rotations mode is used to coat cemented carbide inserts. For nanolayered coatings deposition, the four targets are evaporated simultaneously and the speed of turntable rotation is in the range 1.5 to 4 rpm. For multilayered coatings, the targets are evaporated alternatively and the period is controlled via evaporation duration of each material. Interface between each elementary layer of multilayered coatings is a mixture of the two elements in order to ensure a good cohesion.

X-ray diffraction is performed by means of a D8 advance Bruker type goniometer (λ CuKα = 1.5418 Å) in the $\theta/2\theta$ mode. Scanning electron Microscopy observations are made in a Philips XL30 and roughness examination was performed with Perthometer Perthen.

Coatings have been observed by conventional transmission electron microscopy (TEM) on a Jeol-2000FX microscope (200 kV). High resolution chemical maps and measurements have been realized by Energy Filtered Transmission Electron Microscopy (EFTEM) with a Gatan Energy Filtering (GIF) on Jeol-3010 microscope (300 kV) of the Department of Fundamental Research (CEA). Information about this technique is reported in [22]. The cross-section TEM samples have been prepared by mechanical grinding and polishing (down to 30 µm) followed by Ar^+ ion milling on a Fischione 1010 polisher (accelerating voltage of 4 kV with ion current of 5 mA).

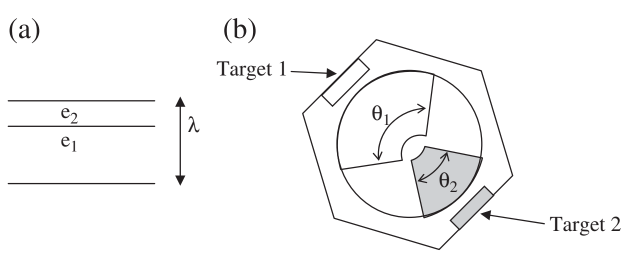

Fig. 2. Period λ defined by the sum of two elementary layers (a) and deposition zones defined by angle θ_i for two different materials (b).

3. Results and discussion

3.1. Period's control

Period's control of a multilayered coating is not a problem whereas calculation is needed for nanolayered films. A very

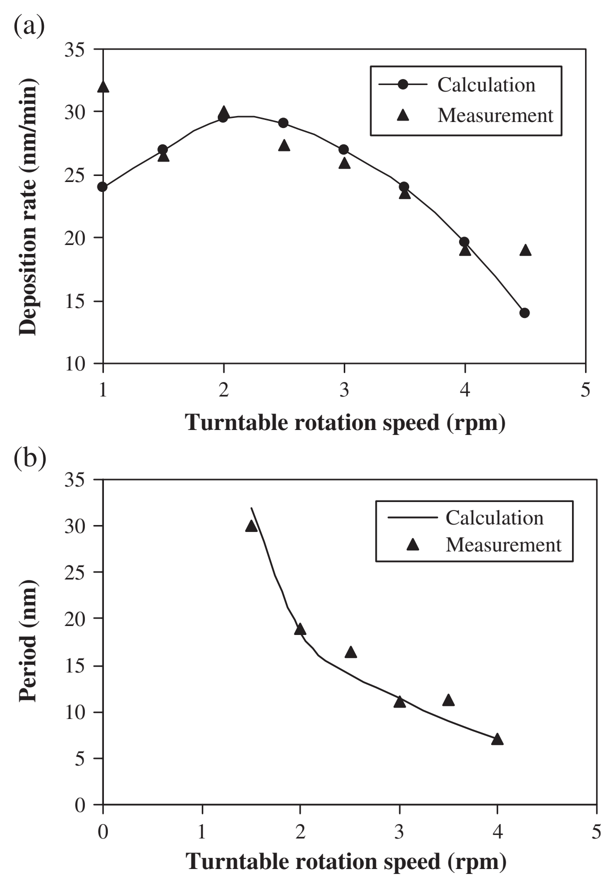

Fig. 3. Effect of the turntable rotation speed on deposition rate (a) and period (b) of TiN/AlTiN nanolayered coatings. Measurements come from the position of the satellite peaks of the (111) reflection of the superlattice structure.

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