

# Enhancing mechanical and tribological performance of multilayered CrN/ZrN coatings

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## Abstract

The CrN/ZrN multilayered coatings with nanoscale bilayer period were synthesized at different substrate rotary speeds (4–11 rpm) and reaction gas flows in an unbalanced reactive dc magnetron sputter chamber. XRD, AES, XPS, Nano indenter and tribometer were employed to investigate the influence of substrate rotary speed, species of reaction gases and flows on microstructure, mechanical and tribological properties of the coatings. A layer structure with small modulation period synthesized by a proper percentage of NH<sub>3</sub> in N<sub>2</sub> reaction gas was proved to be of benefit to synthesize high hard (32 GPa) and low wear-resistant (wear rate:  $0.3865 \times 10^{-5}$  mm<sup>3</sup>/Nm) CrN/ZrN coatings. These properties were related to strongly mixed Cr–N and Zr–N preferred orientations and nanolayer structure.

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## 1. Introduction

Magnetron sputtering has developed rapidly over the last decade to offer the same functionality as much thicker films produced by other surface coating techniques. It now makes a significant impact in application areas including hard, wear-resistant coatings, low friction coatings, corrosion-resistant coatings, decorative coatings [1–3].

Nanoscale multilayers are well-known for increasing hardness as the period is reduced, usually reaching values that surpass those of their individual components, resulting in the improvement of the mechanical and tribological properties [4,5]. Transition metal nitride coatings, mainly based on titanium, chromium and zirconium, are widely used as protective coatings against wear and corrosion due to their desirable high melting point, high hardness, lower friction coefficient, high chemical stability and corrosion resistance [5–9]. However, few reports on nanoscale CrN/ZrN multilayered coatings can be found in recent literatures. In this work, we focus on this model grown in a dc reactive magnetron sputtering chamber by varying deposition parameters. Our aim is to obtain insight into the

significance of these process parameters on the structure and mechanical properties of the multilayered CrN/ZrN coatings.

## 2. Experimental detail

The CrN/ZrN multilayered coatings were synthesized in an unbalanced reactive magnetron sputter chamber. Prior to deposition, silicon (100) substrates were heated to 250 °C and then etched for 10 min by Ar<sup>+</sup> at –600 V. The base pressure was down to 10<sup>–4</sup> Pa. Ar, N<sub>2</sub>, and NH<sub>3</sub> gas flows were independently controlled using the mass-flow controllers. The deposition of the multilayered coatings started with the deposition of a couple 10 nm Zr buffer layer to increase coating adhesion. By alternately exposing the substrates to the Cr (99.9%) source and the Zr (99.9%) source set on opposing position using varying substrate rotation (4 to 11 rpm), the multilayered coatings with different layer modulation periods were grown. The total pressure was kept at 0.26 Pa no matter how change N<sub>2</sub> or NH<sub>3</sub> flows. DC power supplies were run in constant mode with 1 kW and 1.2 kW applied to Cr and Zr targets. A negative bias of 200 V was applied to the substrate during deposition.

The thicknesses (1–1.2 μm) of the coatings were measured using a XP-2 profiler. This system was also used to perform residual stress test. A Nano Indenter XP system was employed

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to perform nanoindentation and elastic modulus tests. In this measurement, the hardness and elastic modulus were measured as a continuous function of depth from a single indentation experiment. Wear tests were performed in a humidity of 30% at 23 °C using a MS-T3000 tribometer (ball-and-disk) with a Si<sub>3</sub>N<sub>4</sub> ceramic ball sliding on the specimen under an applied load of 2 N. The diameter of the wear track was 5 mm; the sliding velocity was held constant 1000 rpm. Wear track analysis was performed using the profiler.

XRD was used for structural analysis of the coatings using a D/MAX 2500 (Japan) diffractometer. The element compositions and their depth profiles of the coatings were investigated by Auger electron spectroscopy (AES, PHI-610, USA). XPS studies were performed with a VG ESCALAB 5 multi-techniques electron spectrometer, in order to investigate element chemical bonding states.

**3. Result and discussion**

Fig. 1 shows the XRD patterns of the CrN/ZrN and monolithic layers synthesized at gas flow of 0.63 sccm for N<sub>2</sub> and 0.17 sccm for NH<sub>3</sub> under identical other operation parameters. The structure investigation of CrN and ZrN monolithic layers reveal a typical face-centered-cubic structure. However, only a broad and weak ZrN(111) peak is found in the multilayer structure. The reason is that CrN layer periodic deposition suppresses crystal growth of ZrN. Another reason is due to thinner ZrN layer within a modulation period due to a lower deposition rate. The thicknesses of individual CrN and ZrN layers for all deposition parameters are calibrated using the film growth rate measured by the thickness of coatings grown using a profilometer. 70%/30% CrN/ZrN is controlled and achieved within each bilayer thickness for all multilayers. Therefore, individual ZrN thickness is estimated to be 0.7 nm for this case (see later analysis of low-angle XRD pattern). A new strong texture appears at 61.06° in the structure of this multilayer. This seems to reveal a phase transformation from cubic NaCl CrN to

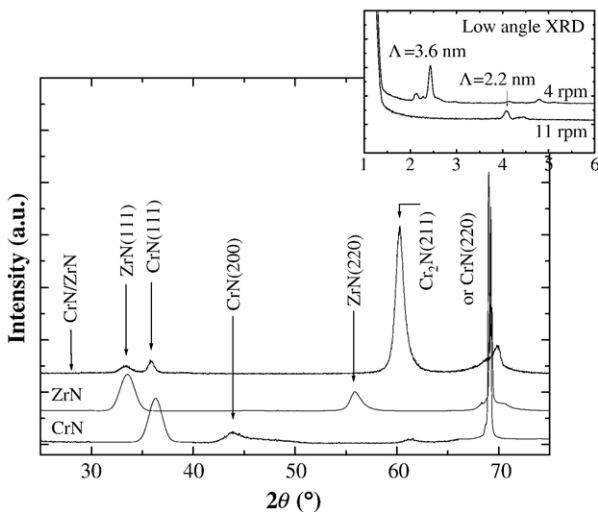


Fig. 1. High-angle and low-angle XRD patterns of CrN/ZrN and monolithic layers.

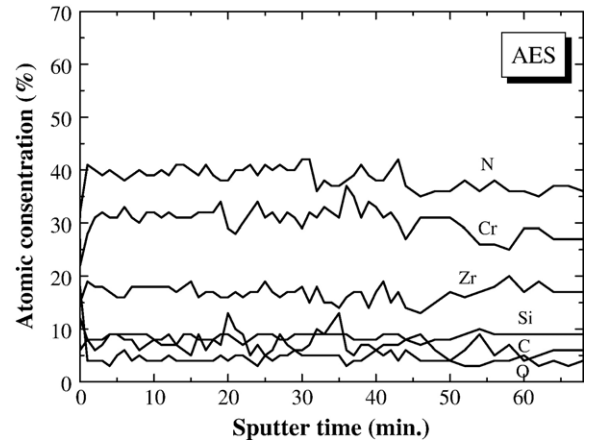


Fig. 2. Sputter depth profile of the CrN/ZrN coating.

hexagonal Cr<sub>2</sub>N due largely to stress change in the coating growth, but could not be identified conclusively. On the other hand, if the stress/strain relationship changes, the diffraction peak also shifts, according to Bragg’s law. Therefore, the peak at 61.06° might also correspond to shift CrN (220) texture. So, it could not be concluded whether the peak at 61.06° corresponded to the Cr<sub>2</sub>N (211) preferred orientation or shifted CrN (220) phase. But, it is believed that the presence of a proper amount of NH<sub>3</sub> in the process gas is able to produce a mixed polycrystalline

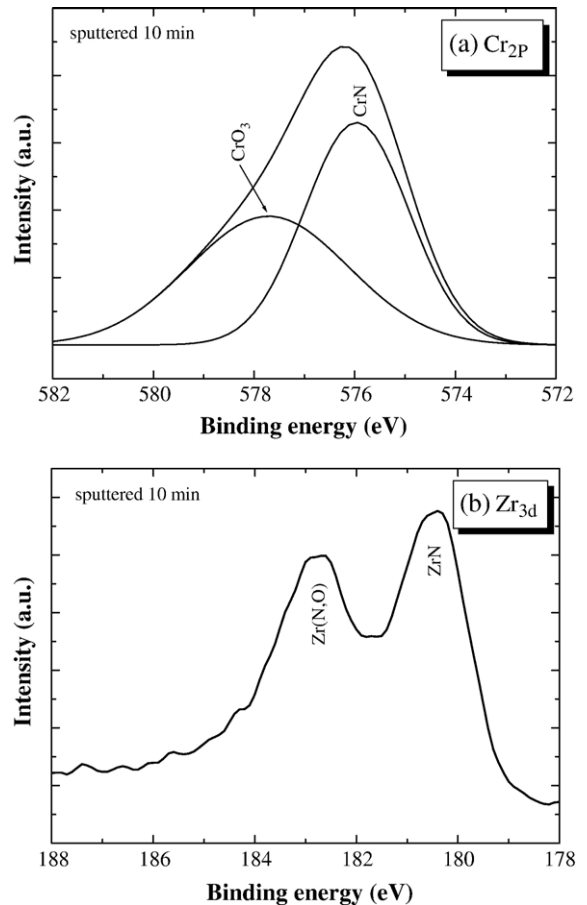


Fig. 3. XPS (a) Cr<sub>2p</sub> and (b) Zr<sub>3d</sub> spectra obtained from the CrN/ZrN coating.

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