



# Substrate effect on wear resistant transition metal nitride hard coatings: Microstructure and tribo-mechanical properties

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## Abstract

Four types of different hard transition metal nitrides (TMN:ZrN, CrN, WN and TiN) coatings were deposited on Si (100) and 316LN stainless steel substrates using DC magnetron sputtering. A comprehensive study of microstructure and substrate dependent tribo-mechanical properties of TMN coatings was carried out. Higher hardness ( $H$ ) and elastic modulus ( $E$ ) were obtained for WN ( $H=40$  GPa and  $E=440$  GPa) and TiN ( $H=30$  GPa and  $E=399$  GPa) coatings. This is related to the formation of (100) and (111) preferred orientations in WN and TiN coatings, respectively. However, the less hardness and elastic modulus were obtained for ZrN and CrN coatings where (200) orientation is preferred. Remarkably, low friction coefficient (0.06–0.57) and higher wear resistance in the coatings deposited on steel substrates are directly associated with the higher resistance to plastic deformation ( $H^3/E^2$ ) and the presence of intrinsic compressive stress. Three body wear modes enhanced the friction coefficient (0.15–0.62) and the wear rate in the coatings deposited on Si substrates. This is primarily associated with low fracture toughness of brittle single crystalline Si (100) substrates. Steel-on-steel contact was dominated in ZrN/steel sliding system. This occurs due to the severe adhesive wear mode of steel ball, whereas, the abrasive wear modes were attained for the CrN, WN and TiN coatings sliding against steel balls.

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## 1. Introduction

Low friction coefficient and high wear resistance are eventually required for the enhancing energy efficiency of moving mechanical components in semiconductor industries, cutting tools, spacecraft and biomedical implant industries [1–5]. It is known that due to the high friction and wear, the sustainability and work efficiency of the mechanical components reduces. Therefore, improving the hardness, elastic modulus and fracture toughness of materials are the key parameters for enhancing tribological properties of mechanical components [6]. The designing of nanocrystalline hard coating is an important way for improving the tribo-mechanical properties [7]. TMN coatings in binary phase are another important class of materials for several tribological applications owing to their

excellent physicochemical, mechanical and tribological properties [8–12]. Among them, the tribo-mechanical properties of nanocrystalline TiN coatings deposited by several techniques have been extensively studied [13–16]. However, the reports on the microstructure, mechanical and tribological properties of other TMN coatings (CrN and ZrN) are limited [4,17,18]. The WN coating is another important refractory material with extreme hardness and barrier properties. This coating is largely studied for the micro-electronic applications, but less attention is paid for tribological point of view [9,19,20]. In general, the tribo-mechanical properties are inherently related to the composition and microstructural characteristics, which can be typically controlled by the deposition conditions [21]. The PVD [22] techniques are useful to deposit the TMN coatings, amongst them, the magnetron sputtering is commonly applied due to its versatility, uniformity and its large industrial applications [23,24]. Unfortunately, there is still a lack of knowledge to understand the substrate dependent microstructure

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and tribo-mechanical characteristics of TMN coatings deposited by PVD techniques.

The main aim of the present work was to deposition and characterization of ZrN, CrN, WN and TiN coatings on single crystalline Si (100) and polycrystalline 316LN stainless steel substrates using reactive DC magnetron sputtering. Si substrate was chosen owing to its extensive applications in semiconductor industries; while, the stainless steel is applicable in cutting tool industries. The observed results reveal an interesting correlation with the composition and microstructure; and it provides a remarkable relationship between the microstructure and tribo-mechanical behavior of the coatings. The substrate dependent friction and wear behavior of these coatings were also elucidated and explained on the basis of coating microstructure.

## 2. Experimental details

### 2.1. Coatings deposition

ZrN, CrN, WN and TiN coatings were deposited on Si (100) and 316LN SS substrates using DC magnetron sputtering technique (Hind High-Vacuum, Bangalore, India) at the substrate temperature ( $T_s$ ) of 250 °C. Commercially available polycrystalline AISI 316LN SS ( $15 \times 15 \times 1.0 \text{ mm}^3$ ) and Si (100) ( $10 \times 10 \text{ mm}^2 \times 500 \mu\text{m}$ ) substrates were used for the deposition. The polishing and surface cleaning process for 316LN SS substrates were reported elsewhere [25]. The target-substrate distance was kept constant approximately 70 mm for all the samples. High pure (99.997%) Zr, Cr, W and Ti disks (50 mm diameter and 3 mm thickness) were used as target materials procured from Taewon Scientific Co. Ltd., Korea. Typical base pressure of the vacuum chamber was achieved around  $9 \times 10^{-6}$  mbar. In order to remove the surface contamination, each target surface was pre-sputtered

for 5 min. During deposition, ultra high pure (99.999%) Ar and reactive  $\text{N}_2$  gases were fed into the chamber and the total deposition pressure was maintained around  $2 \times 10^{-3}$  mbar. For this, the Ar gas flow rate was kept constant at 30 sccm; whereas, the  $\text{N}_2$  flow rate was varied for different layers (5 sccm for ZrN and TiN and 8 sccm for CrN and WN coatings). The nitrogen flow rates in TMN coatings were individually optimized by various characterization techniques, and the optimized flow rate (as mentioned above) was used to deposit TMN coatings. To improve the adhesion between the coating and substrate, the Ti metal interlayer with the thickness of 150 nm was deposited for 5 min at the sputtering power of 150 W. For all the samples, the constant sputtering target power and deposition time were 150 W and 60 min, respectively. The typical thickness of all the coatings was in the range of  $\sim 1.8\text{--}2.0 \mu\text{m}$  as observed by in-situ Quartz-crystal digital thickness monitor.

### 2.2. Characterization of the coatings

The surface topographies of the coatings were analyzed using an atomic force microscope (AFM, park system XE-70). Crystallography of the coatings were characterized by X-ray diffraction (XRD, Rigaku- Miniflex II) with a  $\text{Cu K}\alpha$  radiation ( $\lambda = 1.5406 \text{ \AA}$ ), step size of  $0.02^\circ$ . The qualitative analyses of chemical behavior of the coatings were characterized by X-ray photoelectron spectroscopy (XPS-SHIMADZU-ESCA3400), using Mg radiation with the acceleration voltage of  $0 \sim 12 \text{ kV}$ . Low ion energy sputtering was carried out for all the coatings to remove the surface contamination before the XPS analysis. The nanomechanical properties of the coatings were evaluated by Hysitron Nanoindentation technique (Triboindenter TI950, USA) using Berkovich diamond indenter with the tip curvature of 150 nm. The maximum load of 6 mN was applied for all the nanoindentation measurements. The

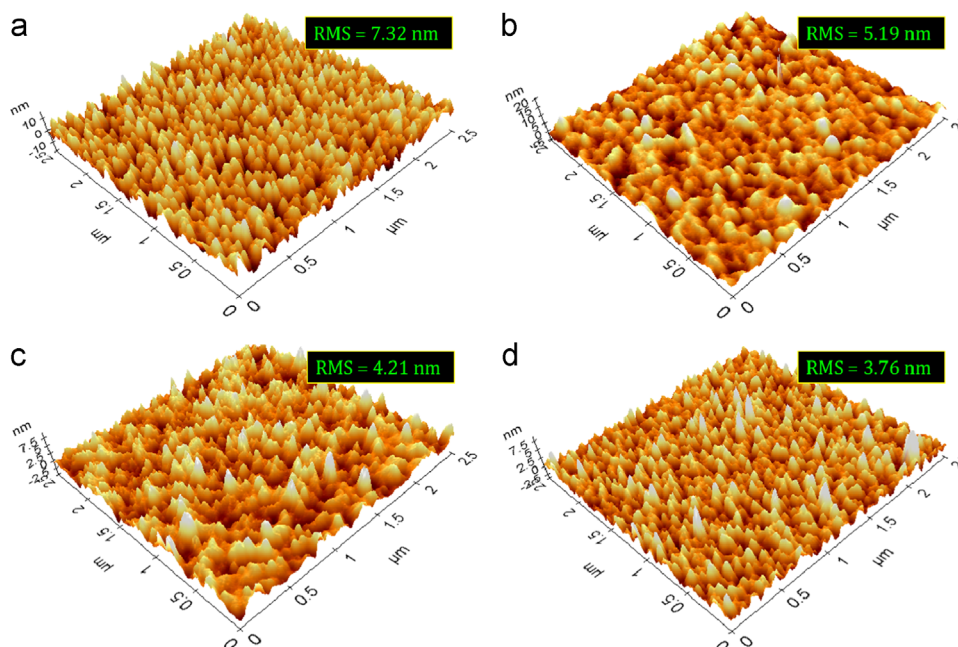


Fig. 1. AFM topographies of (a) ZrN, (b) CrN, (c) WN and (d) TiN coatings deposited on Si substrate.

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