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# Nanoindentation study of magnetron-sputtered CrN and CrSiN coatings

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#### **ABSTRACT**

CrN and CrSiN coatings were deposited on stainless steel substrate by reactive magnetron sputtering. The coatings were characterized for phases, chemical composition, microstructure, and mechanical properties by X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM)/energy dispersive spectroscopy (EDS), atomic force microscopy (AFM), and nanoindentation technique, respectively. The cubic phase was the only phase observed in both the coatings as observed in XRD results. A dense morphology was observed in these coatings deposited with high nitrogen and Si contents, 50:50 and 18.65 at.%, respectively. Nanoindentation measurement of CrN coatings, with  $Ar + N_2$  proportions of 60:40, showed maximum hardness (H) and modulus (E) of  $21 \pm 0.85$  GPa and  $276 \pm 13$  GPa, respectively. The CrN coatings deposited in pure N<sub>2</sub> atmosphere showed H and E values of  $27 \pm 1.62$  and  $241 \pm 10$  GPa, respectively. The measured H and E values of CrSiN coatings were found to be  $28 \pm 1.40$  GPa and  $246 \pm 10$  GPa, respectively. The improved hardness in both the coatings was attributed mainly to a reduction in crystallite size, decrease in surface roughness, and dense morphology. The incorporation of Si into the CrN coatings has improved both hardness and Young's modulus.

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## 1. Introduction

Transition metal nitrides such as CrN and CrSiN coatings are widely preferred for tribological applications owing to their excellent properties such as high hardness, high wear resistance, and high temperature oxidation resistance. An addition of alloying elements such as Al or Si into CrN coatings improve its tribological and hardness properties further as reported in the literature [1– 4]. In the case of M–Si–N systems (where M is (Cr, Ti, W)), the addition of Si leads to the formation of two-phases containing  $MN_x$ nanocrystallites, surrounded by the  $\text{SiN}_x$  amorphous phase [5–7]. The segregation of  $X$  ( $X = Si$ ,  $Ge$ ) to the grain boundaries limits the crystallite growth in the M–X–N system, through the formation of an amorphous phase [8]. The hardness  $(H)$  and modulus  $(E)$  of the sputtered CrSiN coatings, with 2.3 at.% silicon content, measured, by using depth sensing indentation were found to be 24 GPa and 240 GPa, respectively [9]. Lee and Chang [10] reported that the surface roughness, grain size, hardness, and friction coefficient of pulsed DC-reactive magnetron sputtered CrSiN coatings decreases with increasing Si contents from 0.7 to 12.9 at.%. They reported high hardness (24.6 GPa), low friction coefficient, and high wear resistance, for the CrSiN coatings containing 10.0 at.% Si. Mercs et al. [11] have investigated the sputtered CrN and CrSiN

coatings deposited under different nitrogen flow rates (6 and 10 sccm), keeping constant argon flow rate (20 sccm). It showed a maximum hardness (24.5 GPa) and low Young's modulus (298 GPa), measured by nanoindentation technique, with the increase of the silicon content up to 2.5 at.% into the coatings. A further increase of the Si content led to the decrease of  $H$  and  $E$  in the coatings. It was reported in their further work [12] on the CrN and CrSiN coatings produced with at.%  $Si \le 0.92$ , in Ar/N<sub>2</sub> mixture atmosphere, that the formation of amorphous silicon nitride phase has occurred at the grain boundaries of CrN which lead to a higher nanohardness (30 GPa at 2.2 at.% silicon). Martinez et al. [13] investigated the electrical, optical, and mechanical properties of reactive magnetron sputtered CrN and CrSiN coatings as a function of N and Si contents and observed that the hardness of CrN coatings is influenced by the coating morphology. The coatings with less than 3 at.% Si form fcc CrSiN compounds with a maximum nanohardness of 22 GPa while coatings containing more than 3 at.% of Si shows segregation of a  $\text{SiN}_x$  amorphous phase and columnar morphology [14]. With increasing Si contents, the crystallite size of the coatings reduces gradually and finally to nanosized crystals, which get dispersed in the amorphous phase of  $Si<sub>3</sub>N<sub>4</sub>$  [15]. The coatings hardening of co-sputtered CrSiN and nc- $CrN/a-Si<sub>3</sub>N<sub>4</sub>$  multilayer is due to the incorporation of Si atoms in the CrN lattice, which leads to solid solution formation. It contributes for the high microhardness of 32 GPa at  $Cr/(Cr + Si)$  ratio of 34% [16]. The nanohardness values of CrN coating with preferred



