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# Temperature activated self-lubrication in CrN/Mo<sub>2</sub>N nanolayer coatings

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# 1. Introduction

Hard coatings for cutting tools have been in use for many decades, [1-6], and have evolved from the initial, simple nitride and oxide coatings (such as TiN and Al<sub>2</sub>O<sub>3</sub>) applied by CVD and PVD in the 1970s, to more complex, high-performance alloy nitrides (such as TiAlN) employed today. More recently, the interest in green manufacturing has spurred development of coatings for dry machining, [7-10]. If coatings can effectively tolerate high temperatures or can help to reduce them, then coolant usage can be reduced. If the friction level due to the cutting process can be minimized, then lubricant usage can be reduced along with power requirements. In an earlier study the authors [11] characterized the deposition conditions and bulk

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## ABSTRACT

CrN/Mo<sub>2</sub>N multilayers were deposited and TEM, SEM, EDS, XPS and TGA studies were carried out. TEM studies show crystalline layers with sharp interfaces. TGA studies indicate that the CrN protects the underlying Mo<sub>2</sub>N layers from oxidation. On varying the periodicity the hardness of the films are in the 20–30 GPa regime, within the rule of mixtures of its constituents. Friction tests at high temperature shows the films to have a temperature activated self lubrication mechanism. Similar friction tests in a near nitrogen atmosphere shows the same temperature activated lubrication mechanism operating with the friction being in a lower regime. MoO<sub>3</sub> is isolated as the predominant oxides phase that evolves from the surface and at temperatures in excess of 400 °C acts as an *in situ* lubricant.

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properties of CrN and Mo<sub>2</sub>N coatings and their multilayers. In this paper the intent is to describe our work to develop and characterize multilayered, CrN/Mo<sub>2</sub>N coatings of different ratio and bi-layer periodicities. The intent is also to characterize the tribological behaviour of these coatings at high temperature and prove that CrN/Mo<sub>2</sub>N coatings do form hard coatings that are self lubricating at high temperatures. It is proposed to use the materials in a nano-layered structure that will be hard, tough, adherent, and lubricious at the service temperature (<1000 °C). The basic system of CrNx and MoNx to form multi (nano)-layered coatings offers some advantages as a starting point. Cr(N) is suggested because of its excellent wear and oxidation resistance to about 800 °C, [12,13] and Mo(N) [14] because it is likely to oxidize at about 500 °C, forming its low friction oxide, MoO<sub>3</sub>. The presumption is that the Mo-oxide will reduce the cutting friction and consequently, the heat generated by high-speed contact (relative to a coating without the Mo). Past research showed that plasma sprayed composites of Cr<sub>2</sub>O<sub>3</sub> and MoO<sub>3</sub> benefit from the addition of MoO<sub>3</sub> and exhibit a reduction of sliding friction from ~.4-.5 at RT to ~.2 at 450 °C (self-mated or against Cr-plated discs) [15]. A schematic representation of the structure conceived as a self

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Fig. 1. Schematic representation of a cutting tool with multilayers. Alternating layers of the lubrication Mo<sub>2</sub>N phase and the CrN phase.

lubricating tool is depicted in Fig. 1. This approach of using a lubricious oxide has also been explored by others in the cutting tool application, but they chose to explore TiOx, [16,17], and VOx, [18,19], as the active components. The oxide formation at temperatures above 400 °C appears to offer promise for the approach. In our case, added characteristics of the selected materials are that they are both hard phases (~20–30 GPa) and they are expected to be immiscible in their up to 800 °C [20], this could result in the films retaining their layered structure (and strength) at temperatures exceeding 800 °C.

## 2. Experimental details

The samples were prepared by sputter deposition in a closed-field dual-cathode unbalanced-magnetron system. The cryo-pumped system has a base pressure of  $4 \times 10^{-7}$  Torr and includes a high vacuum load lock

chamber. There are two vertically mounted 12.8 cm × 40.6 cm planar magnetron cathodes facing each other on opposite sides of the substrate holder and 10 cm from the substrates. The hexagonal substrate holder is just large enough to eliminate the cross contamination from the other cathode. The substrate holder can be rotated at 5–15.2 rpm to produce nano-layered materials with controlled layer thickness. All coatings were 1–1.5 µm in total thickness. The substrates were single crystal Si (001), glass sides, and polished M50 tool steel discs (Ra ~10 nm). Sapphire substrates were used in cases where high temperature anneals were to be carried out. Prior to deposition, the samples were cleaned in an ultrasonic bath of methanol. High purity N<sub>2</sub> (99.99%) and Ar (ultra high purity) were used during the deposition process.

To analyze the chemical composition, XPS analysis was performed on the wear debris with an Omicron ESCA probe, which was equipped with an EA125 energy analyzer. Photoemission was stimulated by a monochromated Al K-alpha X-ray (1486.6 eV) with an operating



**Fig. 2.** Cross-section TEM micrograph of the multilayers in bright field mode. The mild contrast is due to the different phases present as well as local strains and variations in lattice orientation with respect to beam direction. The lattice ordering is also visible showing a continuity across layer boundaries (area A).



**Fig. 3.** Cross-section STEM micrograph of the multilayers. The sharp bright and dark contrast is due to the difference in atomic number. The brighter regions are from the heavier element (Mo) which has a high scattering angle.

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