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# Comparison in microstructure and mechanical properties of nanocomposite CrWN and nanolayered CrN/WN coatings

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#### Abstract

Chromium–tungsten nitride (CrWN) and chromium nitride (CrN)/tungsten nitride (WN) multilayer coatings were fabricated by rf magnetron-reactive sputtering technique. The CrWN coating was deposited with dual gun cosputtering apparatus, while the CrN/WN coatings were manufactured by sequential CrN and WN sputtering to exhibit an alternating nanolayered feature. The microstructure of the nanocomposite and nanolayered coatings was evaluated by both scanning and transmission electron microscopy. The bilayer period of the multilayer was controlled ranging from 10 to 24 nm, with the average thickness of single nitride layer ranging from 5 to 12 nm. The nanolayered CrN/WN coatings exhibited a higher hardness of approximately 30 GPa, which was superior to that of the nanocomposite CrWN coating. The nanolayered structure which confined the grains of the nitrides in the nano range was beneficial to the enhancement of the hardness in the nanolayered coating.

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### 1. Introduction

In recent years, intensive investigation focused on nanostructure coating materials has attracted enormous effort for worldwide researchers to develop coatings with better performance in various characteristics. Among coating materials, nanocomposite or nanolayered nitride deposit systems have been most frequently investigated [1–3]. The transition metal nitrides were usually considered as hard protective coating due to their excellent performance in hardness, wear and corrosion resistance [4–6]. In the nitride systems, chromium nitride (CrN) has been proved as a promising candidate providing high hardness, excellent antioxidation, and good corrosion and wear resistances, as compared to other nitride systems [7,8]. Tungsten, classified as a refractory material, was another material frequently considered due to its high melting temperature and high

hardness [9]. Tungsten nitride coatings were usually used in optical and microelectronic applications, such as barrier

layer and electrode [10]. Nevertheless, only few discussions of its mechanical properties were found in literatures. Shih and Dove investigated the mechanical properties of multilayer films, including Ti/TiN, Hf/HfN, and W/WN [11]. It was concluded that the coatings with alternating configuration in pure metal and metal nitride exhibited superior mechanical properties than the single-layer films. Especially for the W/WN coatings, the hardness of the single WN film, 2400 kg/mm<sup>2</sup> (around 24 GPa), could be promoted up to 30 GPa by changing the structure into W/WN with an individual layer thickness down to nanoscale [11]. The tungsten nitride multilayer coating was thus proved as a potential candidate in nanolayered material for hard coating applications. In present study, the combination of chromium nitride and tungsten nitride coating was proposed to form a nanostructure coating system. The nanolayered CrN/WN and nanocomposite CrWN coatings were fabricated by rf sputtering technique. Studies focused on microstructure evaluation of the nanostructured coatings were carried out.

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The mechanical properties including hardness and Young's modulus of the coatings was also investigated. The microstructure and mechanical properties both of the multilayer CrN/WN and nanocomposite CrWN coatings were then taken into consideration for comparison.

## 2. Experimental procedures

The fabrication of the nanocomposite CrWN and nanolayered CrN/WN coatings was conducted by a dual gun sputtering apparatus using two metal targets, Cr and W, both in purity of 99.99%. The dual gun magnetron sputtering apparatus was schematically illustrated in Fig. 1. The coating chamber was first pumped down to  $2.0 \times 10^{-3}$  Pa, followed by the inlet of Ar and N2, both of 20 sccm, as plasma and reactive gas sources, respectively, to a working pressure of  $4.0 \times 10^{-1}$  Pa. The target-to-substrate distance was controlled at 60 mm. The nanocomposite CrWN coating was deposited by dual gun cosputtering, while sputtering of single element, Cr or W, was proceeded alternately to form the sequential CrN/WN multilayer coating. The input power on the Cr target was fixed at 150 W, while that for the W target was controlled at 35 W. The deposition time of individual nitride layer of the multilayer CrN/WN coating during sequential sputtering was modified from 100 to 240 sec. Detailed fabrication parameters and layer characteristics were summarized in Table 2. The coating thickness and cross-section image was observed with a scanning electron microscope (FESEM, JSM-6700, JEOL, Japan). A high-resolution transmission electron microscope (HRTEM, JEM-2010, JEOL, Japan)

was employed to investigate the detailed microstructure of the nanostructured coatings. The X-ray diffractometer (Rigaku Dmmax-B, Tokyo, Japan) using Cu K $\alpha$  radiation generated at 15 kV was adopted to identify the phases of the coatings. The hardness and Young's modulus of the coatings were analyzed with a nanoindentation apparatus (Tribo-Scope, Hysitron, Minneapolis, MN, USA). The data of hardness and modulus was calculated and averaged from 5 indentation tests for each sample. The maximum load adopted for all the coating was fixed at 3000  $\mu$ N.

#### 3. Results and discussions

### 3.1. Coating configuration

Through co-sputtering and sequential deposition techniques with controlled periods of time, the composite CrWN and multilayered CrN/WN coatings were accomplished. In the sequential deposition process for CrN/WN coatings, the deposition time interval for each gun was achieved by the control of shutters. Fig. 2 indicates the cross-sectional SEM image of the multilayer CrN/WN coating with a total film thickness of 650 nm. Smooth and dense microstructure was developed by rf magnetron sputtering technique. The homogeneity in coating thickness could also be revealed through SEM image. It was found that the film was composed of a layered configuration by alternating stacking darker CrN and lighter WN phases. The thickness of bilayer periods, one darker CrN and one lighter WN layers, in the CrN/WN nanolayered coatings were evaluated and listed in Table 1. The bilayer periods of the coatings were

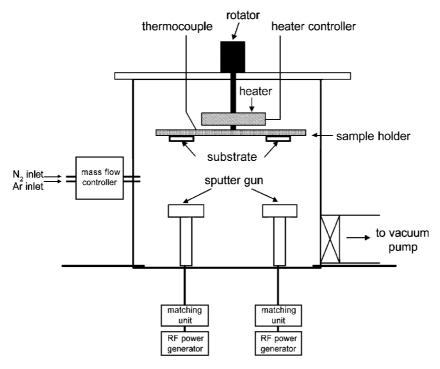


Fig. 1. Schematic illustration of the sputtering apparatus.

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