

# Effect of substrate temperature on the mechanical and tribological properties of W/WC produced by DC magnetron sputtering

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## Abstract

W/WC bilayers were grown using the DC magnetron sputtering technique and varying substrate temperature. The mechanical and tribological behaviors were characterized using the nanoindentation and pin-on-disk techniques. The hardness and Young's modulus tended to increase, while the coefficient of friction tended to be stable with increasing substrate temperature. Moreover, better mechanical and tribological performances were observed for all of the coated systems compared with the uncoated steel. Furthermore, the inclusion of a W interlayer did not significantly influence the hardness; nevertheless, this interlayer dramatically improved the coating tribological behavior, thus producing less coating damage and decreasing the wear rate.

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**Keywords:** C. Hardness; Coefficient of friction; Metallic interlayer; Wear; Young's modulus

## 1. Introduction

Transition metal carbides (TMCs), which normally include all 3d elements and 4d/5d elements of groups 3–6 of the early transition metals, exhibit special physical and chemical properties. For instance, TMC compounds usually exhibit high hardness, brittleness, melting points and electrical and thermal conductivities, which make them attractive for technological applications, such as cutting tools and hard-coating materials [1]. Tungsten carbide (WC) is a very attractive refractory material for industrial applications because it has an excellent combination of properties, such as high hardness, elastic modulus, corrosion resistance, oxidation temperature and a low coefficient of friction (COF) [2–6]. WC films have been synthesized using physical vapor deposition (PVD) and chemical vapor deposition (CVD) techniques [7–10]. Magnetron sputtering of tungsten in a hydrocarbide environment has been used to produce stable and metastable WC crystalline phases at lower deposition temperatures than those used in CVD techniques [11]; nevertheless, in most thin film

production processes, the coatings are mainly dominated by the cubic phase. The reported microstructures  $WC_{1-x}$  films are either very fine nanocrystalline or poorly crystalline grains in an amorphous carbon matrix [12]. Many tungsten properties have been thoroughly investigated and reported in the literature, including its high melting point, thermal shock resistance, thermal diffusivity and corrosion resistance. Tungsten has often been used for wear prevention, e.g., to protect the bores of large caliber tank guns, and selective tungsten coatings have been used on MEMs devices to improve their wear resistance [13]. Although there are several applications of tungsten that are related to its tribological properties, more studies of its tribological behavior, especially at elevated temperatures, and of the processing–microstructure–property relationship are required. Therefore, it is important to investigate the tribological properties of this material for suitable applications [14]. WC thin films have been studied by several authors. For instance, Abdelouahdi et al. [15] presented a study of WC that was grown by the RF magnetron sputtering technique with an Ar/CH<sub>4</sub> gas mixture. The produced coatings exhibited a cubic  $WC_{1-x}$  phase, a maximum hardness of 22 GPa and a plastic deformation parameter ( $H^3/E^2$ ) of approximately 0.08. Other studies by Rincón et al. [16] showed that synthetic cubic

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WC<sub>1-x</sub> was produced by the RF magnetron sputtering technique using two environments: pure Ar and an Ar/CH<sub>4</sub> gas mixture. The WC that was grown in a pure Ar environment had a maximum hardness of 19.5 GPa and a coefficient of friction (COF) of 0.39, while the coatings that were grown in a mixture of Ar/CH<sub>4</sub> exhibited hardness and COF of 18.5 GPa and 0.12, respectively. These results show that the mechanical and tribological properties of thin films are strongly influenced by the process conditions.

More recently, multilayer coatings have been used for mechanical applications to improve the wear resistance of materials [17,18]. The wear resistance results from a specific, favorable combination of hardness and toughness, which is difficult to obtain with a single coating [19]. The toughness of hard ceramic coatings can be improved by matching the coatings with a ductile layer. The introduction of a metal interlayer achieves the following: (i) an enhancement of the plastic deformation of the coating, (ii) stress relaxation, (iii) a reduction in the porosity and (iv) crack deflection [20,21]. It is very common to find WC coatings with interlayers such as Ni [22], Co [23] and C [24]; however, it is not common to use W as the metallic interlayer. On the other hand, in a previous work carried out by our group, structural and morphological studies of W/WC coatings produced using the magnetrons sputtering technique and varying the temperature were presented [25]. Diffraction analysis of W/WC bilayers deposited on 316L stainless steel at RT, 100 °C, 200 °C and 300 °C revealed the peaks corresponding to the bcc structure of W (1 1 0), and to the fcc-type NaCl structure of WC (1 1 1), (2 0 0), (2 2 0) and (3 1 1), showing a preferred orientation in the (200) direction. Moreover, Raman analysis revealed the presence of W–O, W–C and W–C bonds. Morphology studies carried out at different substrate temperatures using AFM, SEM and profilometry techniques showed that as the substrate temperature increased, a slight decrease in roughness and grain size was observed. Moreover, the W and WC monolayer thicknesses were approximately 780 and 730 nm. The W/WC bilayers that were deposited at increasing  $T_s$  exhibited slight increase in the thickness; however, the sample that was grown at 300 °C exhibited a decrease in the thickness.

The present work describes a study of W/WC bilayers deposited on stainless steel substrates using the DC magnetron sputtering technique with W and WC cathodes for various substrate temperatures. The W interlayer was deposited to improve the mechanical and tribological properties. The effects of the deposition temperature on the mechanical and tribological properties are studied. Also, their relationship with microstructural features reported previously [25] is analyzed.

## 2. Experimental setup

The deposition system used for thin films production was a DC magnetron sputtering. W/WC bilayers were grown on 316L stainless steel substrates using tungsten (99.995%) and 50/50 tungsten carbide (99.995%) as targets 5 cm in diameter. The substrate temperature ( $T_s$ ) was varied as follows: room temperature (RT), 100 °C, 200 °C and 300 °C. The base pressure was  $10^{-4}$  Pa,

and, before the deposition process, a 5-min plasma cleaning was conducted. The production conditions were as follows: a working pressure of 0.6 Pa, a power density of 5 A/cm<sup>2</sup>, an Ar gas flow of 20 sccm and deposition time of 60 min for each layer [25]. The mechanical properties were obtained by a Nanovea IBIS Technology nanoindenter using the Oliver and Parr method, a diamond Berkovich indenter and a penetration depth below 10% of the total coating thickness to disregard substrate effects on the obtained measurements [26]. Tribological characterization was performed with a CSEM tribometer, using an alumina counterface ball of 6 mm in diameter, a 1 N normal applied load and a 10 cm/s linear velocity. The wear coefficient was obtained from Archard's law, which related the wear area (as measured by profilometry) to the work done on the material.

## 3. Results and discussion

### 3.1. Mechanical properties: hardness and elastic modulus

Nanoindentation was used to measure the hardness and elastic moduli of the substrate and the W, WC and W/WC films according to the Oliver and Parr method [27]. The W/WC load–unload curves are shown in Fig. 1. An elastic–plastic evolution was identified during the nanoindentation process. Moreover, stacking around the indenter was not observed [28]. Table 1 shows the hardness and elastic moduli for all of the samples. Three samples grown at each temperature were employed to determine the hardness and elastic modulus values.

The WC coatings that were produced at RT with and without a W interlayer exhibit a hardness of approximately 16 GPa. It can be concluded that the presence of an interlayer does not influence the hardness because the indenter penetration is less than 10% of the total layer thickness, thus avoiding the substrate effect. Nevertheless, as  $T_s$  increases, the hardness also increases. This behavior can be related to the decrease of the grain size as was reported in our previous work [25]. Using the Hall–Petch model [29,30], an increasing hardness can be predicted with decreasing grain size. L.P. Ward and P.K. Datta [31] found correlations among the surface morphology, grain size and roughness with the coating hardness; this study explained how a dense surface with fine grains

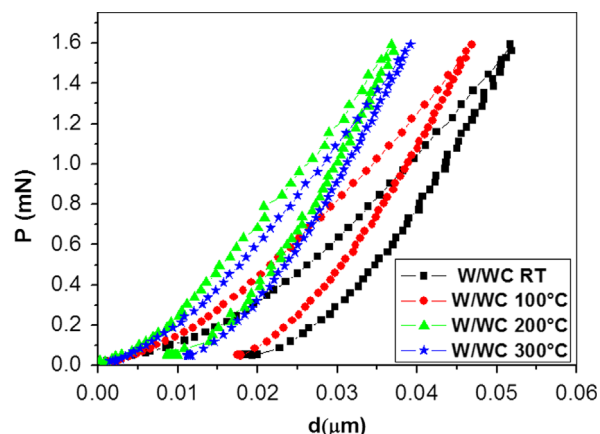


Fig. 1. Load–displacement curves for the W/WC bilayers with different substrate temperatures.

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