

The tribological behavior of W–S–C films in pin-on-disk testing at elevated temperature

T. Polcar^{a,b}, M. Evaristo^a, A. Cavaleiro^{a,*}

^aDep. Eng. Mecânica, ICEMS Faculdade de Ciências e Tecnologia da Universidade de Coimbra, Rua Luís Reis Santos, 3030-788 Coimbra, Portugal

^bDepartment of Applied Mathematics, Faculty of Transportation Sciences, CTU in Prague, Na Florenci 25, Prague 1, Czech Republic

Abstract

W–S–C films were deposited by magnetron sputtering in an Ar atmosphere with a Ti interlayer. A carbon target with several pellets of WS₂ incrustated in the zone of the preferential erosion was used. The number of pellets was changed to modify the carbon content in the films, which varied from 29 up to 70 at%. Doping W–S films with carbon led to a substantial increase of the hardness in the range 4–10 GPa; the maximum of hardness was obtained for coatings with the carbon content of 40 at%. X-ray diffraction (XRD) patterns showed that there was a loss of crystallinity with the increase of the carbon content in the film.

The coatings were tested by pin-on-disk from room temperature (RT) up to 400 °C. At RT, the friction coefficient was in the range 0.2–0.30. At temperatures higher than 100 °C, the friction is below 0.05 for all compositions. The tribological behavior of the coatings with increasing temperatures depended on the films carbon content. For low-carbon content up to 40%, the wear rate was almost independent of the temperature up to 300 °C, while it increased dramatically in the case of the coatings with high-carbon content. In general, the limiting temperature for W–S–C coatings is 400 °C.

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1. Introduction

Transition metal dichalcogenides (TMD) have excellent self-lubricant properties in dry air or vacuum; however, these films are easily rusted in the presence of moist environments and their reduced mechanical resistance makes them inappropriate for applications requiring high-load bearing capacity. On the other hand, hard coatings are often employed to protect from abrasive and erosive wear; however, they do not have low friction and they are frequently brittle. Therefore, there is a need to design coatings combining low friction with high-load bearing capacity; moreover, a good adhesion to the substrates is required. Our recent works showed that the synergetic effect of doping W–S films with carbon or nitrogen together with the deposition of a Ti interlayer could give rise to significant improvements of the mechan-

ical properties and the tribological behavior, particularly in the case of films alloyed with carbon [1,2].

The objective of this work was the development of coatings capable of being used in a wide range of applications, with different applied loads, temperatures and environments. In this study, the structural and mechanical properties of W–S–C coatings are presented together with their tribological behavior at elevated temperature.

2. Experimental details

All W–S–C coatings were deposited on 100Cr6 and M2 polished steel samples with hardness close to 5 and 9 GPa, respectively. The depositions were carried out in a radio-frequency (RF) magnetron sputtering Edwards ESM 100 unit, equipped with two cathodes ($\varnothing = 100$ mm). Prior to the depositions the substrates were sputter cleaned during 20 min by establishing the plasma close to the substrates electrode. Immediately after, a Ti interlayer was deposited with an approximate thickness of 300 nm. The

*Corresponding author. Tel.: +351 239 790 700; fax: +351 239 790 70.
E-mail address: albano.cavaleiro@dem.uc.pt (A. Cavaleiro).

main sputtering target was pure carbon, partly covered by WS_2 pellets placed in the preferentially eroded zone. The dimensions of the pellets were $4.1 \times 3.5 \times 1.5$ mm. The degree of target coverage determined the overall chemical composition of the sputtered film. The depositions were carried out with constant power density of 7.6 W cm^{-2} in the carbon target.

To evaluate the chemical composition of the films, a Cameca SX 50 electron probe microanalysis was used. The hardness was determined by depth-sensing indentation technique using a Fisherscope H100; the coatings deposited on M2 steel were used for these measurements.

The tribological tests were carried out with a pin-on-disk tribometer (CSM Instrument). 100Cr6 steel balls were used as sliding partners. The diameter of the ball was 6 mm which, together with an applied 5 N load, gives a maximum Hertz contact pressure of ~ 0.7 GPa. The coatings tribological behavior was evaluated in air with a relative humidity of 30% and in dry nitrogen. The friction coefficient reported in this study is the average value of the whole sliding test, unless stated otherwise. The standard number of laps during the pin-on-disk measurements was 1500.

3. Results and discussion

3.1. Chemical composition, structure and mechanical properties

Different carbon contents were achieved in the coatings by varying the number of WS_2 pellets in the C target. As it was expected, the carbon content of the films decreases almost linearly with the increase of the total area of pellets. The chemical composition of the deposited coatings varied from 29 to 70 at% C (hereinafter, denomination at% C represents carbon content in the coating); the coatings with carbon contents of 29, 40, 51 and 64 at% were tribologically tested. The S/W ratio varied in the range 1.2–1.45; being the slight increase observed with increasing carbon content. The sulfur deficiency is probably caused by the re-sputtering of sulfur atoms from the substrate and by the chemical reactions of sulfur with the residual atmosphere.

X-ray diffraction (XRD) patterns of W–S–C films showed a gradual loss of crystallinity with the increase of carbon in the coatings. The films with a low-carbon content presents the typical XRD patterns of Me–S (Me—transition metal) sputtered films with prominent peaks at $2\theta \approx 40^\circ$ with an extended shoulder corresponding to a turbostrating stacking of (10L) planes ($L = 0, 1, 2, 3$), and $2\theta \approx 70^\circ$ indexed as (1 1 0) planes. Weise et al. [3] demonstrated that referred XRD patterns could be explained by a two-dimensional (2D) organization of the basal plans which could have several tenths of unit cells dimension. With the progressive decrease of the lateral dimensions of the basal plans, either broadening or drop in the intensity of the (10L) plan occurred until a unique low intensity and broad peak typical of an amorphous structure was detected. This situation would arise when the lateral order

of the basal plans did not exceed a couple of lattice parameters [3]. In previous work, it was shown by high-resolution transmission electron microscopy (HRTEM) that W–S–C films with high C contents were formed by a nanocomposite structure which included WS_2 grains with only some nanometers size, in agreement with Weise et al. interpretation [4].

The hardness of W–S–C films increases with increasing carbon content, reaching a maximum at ~ 40 at% C. This trend can be related to the increase of the compactness of the films associated with the possible formation of nano-sized carbide phases, intrinsically harder than tungsten disulfide. With the addition of more carbon to the films, there is no more W available to establish W–C bonds and the formation of carbon phases leads to a decrease of the hardness (see Fig. 1). However, the hardness of W–S–C coatings is generally about one order of magnitude higher than that of pure tungsten disulfide [5].

3.2. Sliding properties

The easy intra and intercrystalline slip in the friction contact due to weak van der Waal's forces between the lamellae of S–M–S hexagonal basal planes of pure tungsten disulfide facilitates the friction by diminishing the tangential force necessary for the sliding. During a pin-on-disk test, the transfer of the coating material to the ball surface and the reorientation induced by the friction of the randomly oriented WS_2 grains, in the contact area, to the basal plane orientation, transforms the materials in contact. Consequently, the friction force originated in this case is due, predominantly, from the type of slipping referred to above. Zabinski et al. [6] showed for W–S–C system with 20 at% of sulfur content that the friction mechanism intervening in a sliding contact was very similar to that of pure tungsten disulfide and that the contribution of the carbon to the friction was negligible. In the present

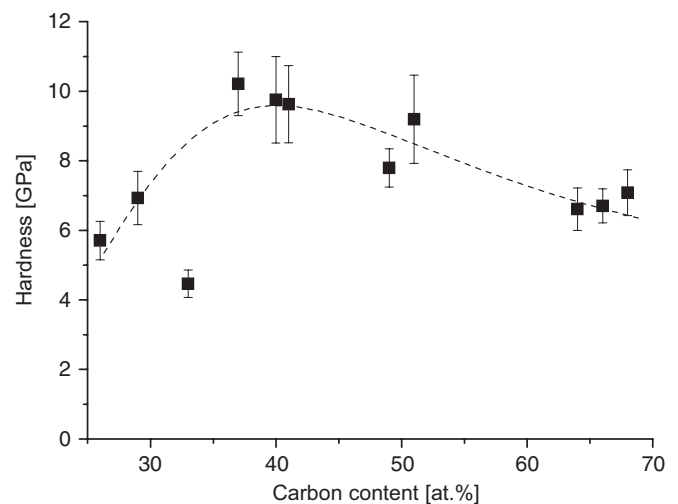


Fig. 1. Evolution of the hardness with the carbon content in the films. The line is a guide to the eye.

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