



## Extreme friction reductions during initial running-in of W–S–C–Ti low-friction coatings

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### ABSTRACT

The disulphides of tungsten and molybdenum are known for their low friction properties when used as solid lubricants. Due to their low hardness, their load bearing capacity when used as thin films is poor. When carbon is added to a WS<sub>2</sub> coating, both of these shortcomings are improved, and a structure consisting of nanocrystals of WS<sub>2</sub>, and possibly tungsten carbide, in a matrix of amorphous carbon is formed. In this study, an attempt is made for further increasing the hardness of such coatings, by addition of Ti, a strong carbide former. A number of W–S–C(–Ti) coatings were deposited using magnetron co-sputtering, and characterised with regard to chemical composition, structure and tribological properties. It was seen that addition of Ti significantly increased the hardness of the coatings, while maintaining their excellent low friction properties in dry atmosphere. However, the coatings with Ti showed extremely high initial friction, a feature not seen for the coatings without Ti. The mechanisms behind this running-in behaviour were investigated by studying surfaces at early stages of wear. It was observed that tribofilms formed during sliding for the coatings containing Ti consisted mainly of TiO<sub>2</sub>, with platelets of WS<sub>2</sub> appearing in the contact only after prolonged sliding. For the pure W–S–C coatings, WS<sub>2</sub> was observed in the sliding interface almost instantly at the onset of sliding.

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### 1. Introduction

Transition metal dichalcogenides, TMDs, are materials well-known for their solid lubricant properties. They have the general formula MX<sub>2</sub>, with the metal being Mo or W, and the chalcogen being S or Se. Their crystal structure is highly anisotropic, and can be described as layers of metals atoms sandwiched between layers of chalcogen atoms [1]. The layered structure with weak bonding between the layers makes these materials easily sheared, enabling their use as solid lubricants [2]. The TMDs can be used for lubrication purposes in various forms, either as additives in liquid lubricants [3,4] or as coatings. Coatings can be prepared by, for example, incorporation of fullerene-like particles [5] or by burnishing [6] but physical vapour deposition (PVD) such as sputtering has been extensively used for deposition of TMD coatings for tribological purposes [7–12].

However, the TMDs in themselves have very low hardnesses and low load-bearing capacities, as well as being porous and

easily oxidized [9,13]. One way to improve the mechanical properties is the addition of a third element, which is easily done during sputter deposition of coatings. Promising results have been presented for example for the W–S–C system, where coatings are seen to become denser and harder with the addition of carbon, while still showing low-friction properties [14–19]. The W–S–C films consist of nanocrystalline WS<sub>2</sub>, and possibly also some carbide grains, in an amorphous carbon matrix. A “chameleon behaviour”, where the WS<sub>2</sub> or the DLC-like matrix provides lubrication depending on the environmental conditions, was suggested by Voievodin et al. [14,15], Wu et al. [20] and Polcar et al. [21], although the results of Cavaleiro et al. indicate that the former is solely responsible for the low-friction properties [21]. Thus, the WS<sub>2</sub> phase provides low friction while the matrix, and carbide grains if present, provides stability and improved mechanical properties.

One possibility of further increasing the hardness of the coatings is to incorporate an additional phase, which adds to the hardness, such as a carbide. This can be done by the addition of Ti, which is known to be a strong carbide-former. We have presented results on the quaternary W–S–C–Ti system, showing that it is indeed possible to increase the hardness of WS<sub>2</sub>-based films while

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still maintaining low friction and TMD lubrication [22]. However, the tribological behaviour of the W–S–C–Ti films at some points differ from that of the W–S–C films, especially in the early stages of sliding, where notably higher friction levels are observed. The aim of this work is to study and compare the tribological behaviour of W–S–C and W–S–C–Ti coatings, especially the running-in process, to gain an understanding of the frictional mechanisms. This has been done by tribological testing of selected W–S–C and W–S–C–Ti coatings, followed by analysis of the wear tracks on the coatings and the wear scars on the counter-surfaces. The tests were either continued until reaching steady-state low friction, or interrupted during the initial running-in process, a method previously used to study the tribological behaviour at various stages of sliding [23].

## 2. Experimental

The coatings were deposited by non-reactive DC magnetron sputtering in a von Ardenne system, using a sintered WS<sub>2</sub> target (99.95%) and a C target (99.995%). A Ti foil (ASTM grade 1) was mounted onto the WS<sub>2</sub> target so as to cover part of the racetrack. The power on the WS<sub>2</sub>/Ti target was held at 450 W, while two different C target powers of 350 and 750 W were used in order to vary the C content. In the former case, the substrates were heated to approximately 300 °C during deposition by an infra-red heater, while no additional substrate heating was used for the coatings with higher carbon content. The coatings presented in this study were selected from a larger number of produced W–S–C(–Ti) coatings, presented elsewhere [22]. The coatings were deposited onto powder-metallurgical high-speed steel (ASP 2023) substrates, which were hardened to 9 GPa and polished to a surface roughness of about 10 nm R<sub>a</sub>. For characterisation purposes, the coatings were also deposited onto (100) silicon wafers. The substrates were ultrasonically cleaned prior to coating deposition, and cleaned by argon ion etching in the deposition chamber. The deposition was performed at a discharge pressure of 4 mTorr, with the substrates kept at floating potential, and with a rotating sample stage. The deposition time was varied between 75 and 110 min, resulting in coatings with a thickness of 1 μm.

The elemental composition of the samples was determined by energy-dispersive X-ray spectroscopy (EDS) using a Zeiss LEO 440 scanning electron microscope (SEM) with a LaB<sub>6</sub> crystal as electron source. The elemental composition was also studied, along with the chemical bonding, by X-ray photoelectron spectroscopy (XPS) on a PHI Quantum 2000 instrument with monochromatic Al Kα radiation. The sample surface was cleaned by sputtering with 200 eV Ar<sup>+</sup> ions, and the resulting spectra were fitted using Casa XPS software. The crystallinity and structure of the coatings were studied by grazing incidence X-ray diffraction (XRD) on a Siemens D5000 instrument with CuKα radiation, using parallel geometry and an incidence angle of 1°. As-deposited films, as well as the wear scars

on the coatings and balls, were studied by scanning electron microscopy (SEM) in a Zeiss LEO 1550 instrument with a field-emission gun. The topography of the unworn coating surfaces was also studied by atomic force microscopy (AFM), using a PSI AXE-50 instrument, operated in non-contact mode. The hardness and Young's modulus for each coating were determined by nanoindentation using an Ultra Nano Hardness tester from CSM instruments with a Berkovich diamond tip. The indentation was load-controlled with a maximum load of 900 μN, ensuring a penetration depth of less than 10% of the film thickness in each case. For each coating, 20 indentations were made and the results are calculated using the method presented by Oliver and Pharr [24].

The frictional properties of the coatings were tested by unlubricated sliding against ball bearing steel balls (100Cr6) with a diameter of 6 mm, under a normal load of 5 N (corresponding to a maximum Hertzian contact pressure of 1.1 GPa), in a rotating ball-on-disc setup with a rotational radius of 2.5 mm. The tests were performed in an atmosphere of dry air (relative humidity below 5%), achieved by a continuous flow of dry air through the test chamber. This atmosphere was chosen as TMD-based coatings are known to perform well in dry conditions. Tests with a duration of 1000 revolutions (sliding distance 15.7 m) were performed on all coatings, using a sliding speed of 0.1 m/s. In order to study the initial frictional behaviour in more detail, some coatings were also subjected to shorter tests of only 20 revolutions (sliding distance approx. 0.3 m). These tests were performed at a lower sliding speed of approximately 1.6 mm/s (or 0.1 revolutions/s). For detailed studies of the steady-state tribofilm appearance, a longer test of 5000 revolutions was performed for one coating at the higher sliding speed (0.1 m/s).

After tribological testing, the wear scars on the coatings and balls were studied by SEM in conjunction with EDS, as previously described, and by Raman spectroscopy on a Renishaw micro-Raman system with a 514 nm laser at a power of 10 mW. Cross-section samples from selected wear tracks were prepared by in-situ lift out in a focused ion beam (FIB) in a FEI Strata D235 instrument, and studied by transmission electron microscopy (TEM) in a FEI Tecnai F30 ST instrument equipped with a Gatan post-column energy filter, which was used to acquire EFTEM elemental maps at 300 kV. In addition, HR STEM EELS was performed on a FEI Titan 80-300 Cubed TEM equipped with a HR energy filter, a monochromator and a probe corrector. STEM images and EELS-Spectrum Imaging (SI) maps were acquired at 200 kV, with a camera length of 19 mm.

## 3. Results

### 3.1. Coating characterisation

The chemical compositions of the coatings, as determined by EDS with calibration by XPS, are presented in Table 1. It can be

**Table 1**  
Composition, structure and mechanical properties of the coatings.

	W–S–C350	W–S–C350–Ti	W–S–C750	W–S–C750–Ti
Composition (at%)				
W	48	19	37	16
S	36	23	31	20
C	16	15	32	36
Ti	0	43	0	36
Dep. temperature	300 °C	300 °C	Room temperature	Room temperature
Structure	Nanocrystal line WS <sub>2</sub>	X-ray amorphous	X-ray amorphous	TiC <sub>x</sub> S <sub>y</sub>
Hardness (GPa)	4.1 ± 0.5	18.3 ± 1.8	6.9 ± 0.5	8.3 ± 1.5
Young's modulus (GPa)	84 ± 6	222 ± 13	100 ± 4	149 ± 15
Surface roughness, R <sub>a</sub> (nm)	8.7	11.5	6.9	20.2

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