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Mechanical and tribological properties of sputtered Mo–Se–C coatings

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ABSTRACT

Transition metal dichalcogenides belong to the more developed class of materials for solid lubrication. However, the main limitation of these materials is the detrimental effect of air humidity causing an increase in the friction. In previous works, molybdenum diselenide has been shown to be a promising coating retaining low friction even in very humid environment. In this study, Mo–Se–C films were deposited by sputtering from a C target with pellets of $MoSe_2$. Besides the evaluation of the chemical composition, the structure, the morphology, the hardness and the cohesion/adhesion, special attention was paid to the tribological characterization.

The C content varied from 29 to 68 at.% which led to a progressive increase of the Se/Mo ratio. As a typical trend, the hardness increases with increasing C content. The coatings were tested at room temperature with different air humidity levels and at temperatures up to 500 °C on a pin-on-disc tribometer. The friction coefficient of Mo–Se–C coatings increased with air humidity from ~0.04 to ~0.12, while it was as low as 0.02 at temperature range 100–250 °C. The coatings were very sensitive to the elevated temperature being worn out at 300 °C due to adhesion problems at coating–titanium interface.

1. Introduction

In late 1990s, a new concept of coatings based on the alloying of transition metal dichalcogenides (TMDs) with carbon started to attract the attention of various scientific groups. The original idea was to join the excellent frictional behavior of TMD in vacuum and dry air with the tribological properties of DLC coatings. Moreover, an increase in the coatings compactness in relation to TMD and an improvement of the mechanical properties, particularly the hardness, could be expected.

Voevodin and Zabinski [1] prepared W–S–C coatings either by magnetron-assisted pulsed laser deposition (MSPLD—target WS_2) or by laser ablation of a composite target made of graphite and WS_2 sectors. The friction coefficient in dry air was lower than that measured in humid air (0.02 and 0.15, respectively), and the frictional stability during an environmental cycling was considered as the most interesting feature. The low friction in dry air increased in the presence of humid air and fell down when the atmosphere was dried again.

Cavaleiro et al deposited W–S–C coatings by magnetron sputtering from carbon target with embedded WS_2 pellets [2,3]. The maximum coating hardness was around 10 GPa, i.e. about one order of magnitude higher than that of pure sputtered WS_2. The tribological behavior of the coatings was tested under different conditions, such as temperature, air humidity or load. The friction decreased significantly with load varying in the range 5–48 N from 0.2 to 0.07 in humid air; the cyclic change of the air humidity showed the same “chameleon” behavior as referred to above.

However, both referred W–S–C systems still show relatively high wear and friction coefficients in humid environment. To remedy this lack, it was decided to select other member of the TMD family, the molybdenum diselenide, which showed low friction almost independently of the air humidity [4], and to study the mechanical and tribological properties of Mo–Se–C coatings prepared by non-reactive magnetron sputtering from a carbon target with $MoSe_2$ pellets.

2. Experimental details

The coatings were deposited on both silica wafers and steel substrates (chromium steel, quenched and tempered with a final hardness of 62 HRC) polished to final roughness Ra ≤ 30 nm. The thin titanium interlayer (~300 nm) was deposited in order to improve the adhesion of the coatings to the substrate. The coatings were deposited by magnetron sputtering in argon atmosphere from a carbon target with pellets of $MoSe_2$. The pellets (99.8% pure) were

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positioned in the erosion zone of the carbon target with a diameter of 100 mm. The dimensions of pellets were 1.5 mm × 3 mm × 4 mm, the number of pellets varied between 16 and 72. The discharge pressure and the power density were 0.75 Pa and 8 W cm^{-2}, respectively. The deposition time was 1 h.

The coating microstructure was studied by X-ray diffraction (XRD—Philips diffractometer, Bragg-Brentano configuration, Co Kα radiation (λ = 0.178897 nm); the chemical composition was determined by electron probe micro-analysis (EPMA—Cameca SX-50). The hardness (H) and Young's modulus (E) of the coatings were evaluated by depth-sensing indentation technique using a Fischer Instruments-Fischerscope.

Wear testing was done using a high temperature pin-on-disc tribometer (CSEM Instruments) adapted to work in controlled atmosphere; sliding partners were steel 100Cr6 balls with a diameter of 6 mm and a 5 N load. The number of laps was 1000, if not stated otherwise. The air humidity (RH) was controlled by a precise hygrometer; the atmosphere with relative air humidity 5% is referred in this study as dry air. The friction tests were carried out as well at elevated temperature up to 500 °C. The morphology of the coating surface, ball scars, wear tracks and wear debris were examined by scanning electron microscopy (SEM—Philips); the chemical analysis of the wear tracks and the wear debris was obtained by energy-dispersive X-ray analysis (EDS). The profiles of the wear tracks were measured by mechanical profilometer. The wear rate of the coating was calculated as the worn material volume per sliding distance and normal load. The average value of three profiles measured on one wear track was used to calculate the coating wear rate.

3. Results

3.1. Main characteristics of Mo–Se–C coatings

The coatings were deposited with different number of MoSe$_2$ pellets implanted in carbon target in order to achieve different chemical compositions, as presented in Fig. 1. The carbon content in the coatings decreased linearly with increasing number of pellets, while the deposition rate increased due to the higher sputtering rate of MoSe$_2$ compared to carbon. Since the deposition time was kept constant (60 min), the coatings thickness increased as well from 2.2 (68 at.% C) to 4.0 μm (29 at.% C). As it was expected, a monotonic decrease of the C content with increasing number of MoSe$_2$ pellets was observed with the simultaneous drop of the Se/Mo ratio from 2.0 to 1.7.

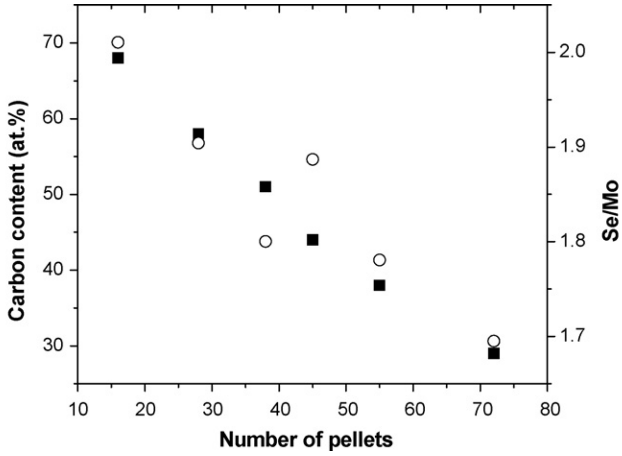

Fig. 1. Carbon content (full symbol) and Se/Mo ratio (open symbol) as a function of number of MoSe$_2$ pellets embedded into carbon target.

The coating with the lowest carbon content showed a columnar structure typical for pure transition metal dichalcogenides [5]. The columns length decreased with increasing carbon content and the morphology started to be amorphous-like for 58 at.% C (see Fig. 2). The XRD diffractograms of Mo–Se–C coatings were almost identical to those of W–S–C system discussed in detail elsewhere [6] (see typical example in Fig. 2); therefore only brief information is given in this paper. The well-defined peak at ∼37° corresponded to (1 0 0) orientation and it was followed by a peak at ∼43° with a long tail representing a turbostratic stacking, i.e. the presence of (10 L) orientations, where L = 1, 2, 3, ... The peak at ∼67° could be attributed to the (1 1 0) orientation.

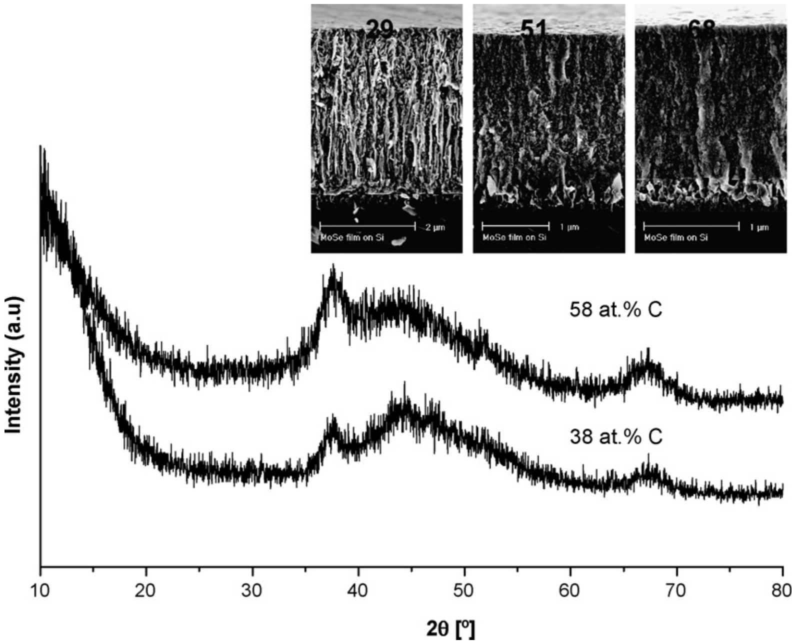

Fig. 2. XRD diffractograms and cross-section morphologies of a Mo–Se–C coatings.