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Temperature dependence of tribological properties of MoS_2 and $MoSe_2$ coatings

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Abstract

Transition metal dichalcogenides are well known for their lubricating properties. MoS_2 is the most popular member of this family and it is widely used as a solid lubricant in vacuum and inert gases. However, the lubricating properties of MoS_2 are deteriorated in humid air. It is known that molybdenum diselenide ($MoSe_2$) has the same crystal structure and also exhibits suitable lubricating properties, but their dependence on air humidity has not been studied yet in details.

This paper is aimed to the comparison of tribological properties of MoS_2 and $MoSe_2$ coatings measured in air of different humidity and at elevated temperatures. Both coatings were prepared by non-reactive DC magnetron sputtering and tested with ball-on-disc high temperature tribometer. The results of measurements of friction coefficient and wear rate vs. tribometer revolutions and the resulting dependencies of friction coefficient and wear rate on ambient air relative humidity are presented.

These results show that the friction coefficient of $MoSe_2$ was not influenced by air humidity. Wear rate of $MoSe_2$ in dry air was substantially higher than that of MoS_2; in humid air, the MoS_2 wear rate increased rapidly while wear rate of $MoSe_2$ remained unchanged. The operating temperature of both coatings was limited to 350 °C.

Keywords: Friction; $MoSe_2$; MoS_2; Solid lubricant coatings

1. Introduction

Transition metal dichalcogenides (sulfides, selenides or tellurides of tungsten, molybdenum and niobium) are well known for their lubricating property [1]. Low friction coefficient is caused by the special layered crystal structure. These structures consist of stack of layers in which a layer of metal is surrounded with layers of chalcogen atoms. The attraction between molybdenum and chalcogen is strong covalent bonding while there is only weak van der Waals attraction between sandwich layers. Therefore a slip between lamellae takes place when friction occurs and it results in low coefficient of friction and other tribological phenomena, e.g. the transfer of coating material to opposing surface is possible.

MoS_2 is the most popular member of the above-mentioned family and it is widely used as a solid lubricant in vacuum and inert gases. Modern method of application is a magnetron sputtering, which improved tribological properties and provided the modification of chemical composition and crystal structure [2,3]. The main problem of MoS_2 used as a lubricant is the strong influence of humidity on the coating properties. Numbers of investigation have been done on that issue and a lot of different ways for solving the problem were shown [4,5].

There is lack of information on behavior of other dichalcogenides in humid air [6]. This paper is a contribution to comparison of the friction properties of the molybdenum disulfide (MoS_2) and molybdenum diselenide ($MoSe_2$) measured in the air of different humidity and at elevated temperatures. The comparatively high resistance of

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Table 1
Chemical composition of used materials (wt.%)

	Fe	C	Cr	Mn	Ni	P	S	Si
100Cr6		1	1.5	0.4	max 0.3	max 0.027	max 0.03	0.2
Substrate		2	12	0.3	max 0.5	max 0.03	max 0.035	0.35

$MoSe_2$ coatings to the ambient air relative humidity is presented.

2. Experimental details

The examined coatings were sputtered on substrates in the shape of discs 20 mm in diameter and 4 mm thick made from tool steel processed to the hardness of 60 HRC. The substrate chemical composition is in Table 1. The substrate surface was polished with diamond pastes to the roughness of Ra<30 nm using standard metallographic procedures. The samples were coated by DC magnetron sputtering in Ar atmosphere using planar cylindrical magnetron. The sputtering targets were MoS_2 resp. $MoSe_2$ (purity 99%) circular, 96 mm in diameter. Prior to deposition the apparatus was evacuated to 5×10^{-4} Pa and the deposition process was carried out at argon atmosphere of the pressure of 0.2 Pa. The relatively long target–substrate distance about 120 mm was chosen in order to achieve uniform film thickness on substrates. The samples were on earth potential during deposition.

The tribological tests were carried out with a high temperature ball-on-disc tribometer (CSM Instrument). This device allowed measurements of the friction coefficient continuously during sliding test at elevated temperatures from room temperature (RT) up to 800 °C in a controlled atmosphere. The counterparts used in these measurements were 100Cr6 bearing steel balls with 6 mm diameter . All measurements were provided with a load of 5 N and a sliding speed of 4 $cm \cdot s^{-1}$ on radius in the range from 3 to 6 mm. Because of the variable diameter, the time dependence of the friction coefficient was evaluated on the number of cycles instead of the sliding distance. Number of cycles was 2000 unless stated otherwise. Measurements were performed in both dry nitrogen and humid air with relative humidity 35% and 50%. The declared values of relative humidity were measured at room temperature.

The worn volume was evaluated by means of the wear track width measurement. Since there was no significant ball wear, the wear track was assumed to be circular-shaped and its area was evaluated from measured width and known ball diameter. The coating wear rate w was determined from the cross-section area A of the wear track, loading force F and number of cycles n as

$$w = \frac{A}{nF}. \quad (1)$$

The wear track width was measured by optical microscope.

3. Results and discussion

3.1. Coatings properties

The stoichiometry of the $MoSe_2$ film determined by means of EDX was 1:1.91. X-ray diffraction showed hexagonal structure. In the case of MoS_2, the structure was a mixture of rhomboidal and hexagonal ones. The value of critical force at the scratch test was 60 N for MoS_2 and 50 N for $MoSe_2$ coatings.

3.2. Friction tests

Fig. 1 shows the typical dependence of MoS_2 friction coefficient as a function of the number of loading cycles at two different temperatures. It could be clearly seen that the temperature effect on friction properties of MoS_2. In comparison with the results measured at room temperature, the value of the friction coefficient was three times lower at 100 °C and the curve turned to be smoother (the standard deviation of the friction coefficient decreased from 0.015 at RT to 0.003 at 100 °C). All curves measured at elevated temperatures were similar to that one measured at 100 °C.

The friction properties of $MoSe_2$ were unaffected by the temperature up to 200 °C. Typical friction trace of the $MoSe_2$ at room temperature is shown in Fig. 2 and it remained unchanged at higher temperatures. When comparing Figs. 1 and 2 mind the different scales. The standard deviation of the friction coefficient value was about 0.002 in the case of MoS_2 at elevated temperature and 0.005 in the case of $MoSe_2$ at RT.

The temperature dependence of the mean values of the friction coefficient measured at 50% relative humidity after 2000 cycles is presented in Fig. 3 for both coatings. The value of MoS_2 friction coefficient at RT in humid air was 0.14, at 100 °C it decreased to values typical for dry air (0.05). The reason was the decrease in relative humidity of the atmosphere surrounding the heated sample (because the humidity was measured at RT). In contrast to MoS_2, the

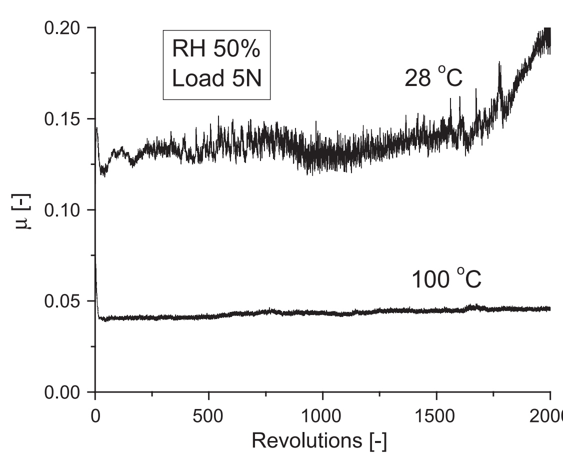

Fig. 1. Typical friction coefficient trace of MoS_2 measured at two different temperatures.