

[Article ID] 1003- 6326(2002) 01- 0078- 05

# Comparison of tribological properties of industrial low friction coatings<sup>①</sup>

WANG Jihui(王吉会)<sup>1,2</sup>, Hua Chen<sup>3</sup>, E. Wieers<sup>2</sup>, L. M. Stals<sup>2</sup>, J. P. Celis<sup>3</sup>

(1. School of Materials Science and Engineering, Tianjin University, Tianjin 300072, China;

2. Institute for Materials Research, Limburg University Centrum, B- 3590 Diepenbeek, Belgium;

3. Katholieke Universiteit Leuven, Dept. MTM, B- 3001 Leuven, Belgium)

**[Abstract]** MX<sub>2</sub> (M= Mo, W; X= S, Se) and DLC ( $\alpha$ C: H and WC/C) are the two kinds of typical low friction coatings widely used in industry. The friction and wear properties of these two kinds of coatings marked as MOVIC, MOST, MoSe<sub>2</sub>/Ni, WSe<sub>2</sub>,  $\alpha$ C: H and WC/C coatings were determined by fretting tests in ambient air of different humidity. The results show that the coefficient of friction of MX<sub>2</sub> coatings increases when the relative humidity of air increases whereas the coefficient of friction DLC coatings decreases with the increasing of relative humidity. MOVIC and WSe<sub>2</sub> coatings have a poor friction and wear resistance because of non basal planes (100) and (101) parallel to the surface in the MOVIC coating, or the rough and porous surface of WSe<sub>2</sub> coatings. Among these six coatings, MoSe<sub>2</sub>/Ni and WC/C coatings have the highest wear resistance which seems to be unaffected by the relative humidity.

**[Key words]** low friction coatings; fretting; relative humidity

**[CLC number]** TG 174.44

**[Document code]** A

## 1 INTRODUCTION

Although lubricants can be used to reduce friction and wear of sliding contact surfaces, in more and more cases liquid lubrication or greases are forbidden. Possible reasons are the risk of contamination in food, medical and textile industries, and they are not applicable in hostile environments such as high temperature or vacuum conditions either. Low friction coatings are of increasing interest for reasons of environmental compliance<sup>[1]</sup>.

By referring to low friction coatings, it normally means materials with a coefficient of friction of 0.4 or below. Among the layered metal dichalcogenides MX<sub>2</sub> (M= Mo, W; X= S, Se), MoS<sub>2</sub> is the most popular coating<sup>[2]</sup>. Among the diamond-like carbon (DLC)<sup>[3]</sup>, the pure DLC coating, hydrogenated DLC ( $\alpha$ C: H) and metal-doped or carbide-doped coatings can be classified. Tungsten carbide/ carbon (WC/C) coating is such a carbide-doped DLC coating which has been used successfully in engineering on bearings, pumps, compressors, gears and tools<sup>[4,5]</sup>.

Over the past 20 to 30 years, several papers on the tribological properties of MX<sub>2</sub> and DLC coatings have been published<sup>[6,7]</sup>. But only in a few papers both MX<sub>2</sub> and DLC coatings were investigated and compared, and the industrial applications of these coatings were discussed<sup>[1,8]</sup>. Moreover the tribologi-

cal tests used in these papers differ from each other, so that it is difficult to compare the tribological properties of these coatings. On the other hand, it seems that low coefficients of friction are achieved only in specific conditions such as MoS<sub>2</sub> coatings operated in dry air or in vacuum<sup>[6,7,9]</sup>. This imposes the testing of the friction and wear behaviour of these low friction coatings at different relative humidity.

## 2 EXPERIMENTAL

### 2.1 Preparation of coatings

MOST is a trademark of a MoS<sub>2</sub>/Ti multilayer developed by Teer Company (GB)<sup>[10]</sup>. The MoS<sub>2</sub>/Ti coating (0.74  $\mu$ m) was deposited by sputtering from three MoS<sub>2</sub> targets and a titanium target simultaneously in unbalanced magnetron sputtering ion-plating system. MOVIC coating (1  $\mu$ m) is a MoS<sub>2</sub> based coating including 14 other alloying elements developed by using magnetron-sputtering methods at 120 °C.

The MoSe<sub>2</sub>/Ni coating (1  $\mu$ m) is from MSAI Company, and prepared by an electrochemical deposition method (Ni layer) and pulsed laser deposition method (MoSe<sub>2</sub> layer). The WSe<sub>2</sub> coating produced by MSAI Company (50  $\mu$ m) was prepared as follows: firstly WSe<sub>2</sub> powder was dispersed in organic silicon vanish and then sprayed onto the substrate.

The diamond-like carbon coating (named  $\alpha$ C: H or DLC, 1.2  $\mu$ m) was obtained from VITO (B) by

<sup>①</sup> [Foundation item] Project (BIL 96/35) supported by the Flanders Government and the P. R. China Government

[Received date] 2001- 03- 27; [Accepted date] 2001- 07- 05

PACVD with using a  $\text{CH}_4\text{-CO}_2$  gas mixture. The WC/C coating from Balzers Company ( $3.7 \mu\text{m}$ ) was produced by physical vapour deposition<sup>[3, 4]</sup>. The WC phase was grown using DC magnetron sputtering of WC target, whereas the carbon phase was grown simultaneously from a hydrocarbon gas (acetylene,  $\text{C}_2\text{H}_2$ ) and argon plasma.

All coatings were deposited on substrates made of H40C. The substrates were thoroughly degreased with acetone and ethanol followed by warm air-drying.

## 2.2 Testing procedures

The fretting tests were performed at  $23^\circ\text{C}$  in ambient air with a relative humidity (RH) of  $< 10\%$ ,  $50\%$  and  $90\%$ . Corundum balls with a diameter of  $10\text{ mm}$  were used as counter-bodies. The corundum balls were loaded on top of the coated flat samples at a normal load of  $1\text{ N}$ . A linear displacement stroke of  $100\mu\text{m}$  was used and a vibration at a frequency of  $10\text{ Hz}$  was applied to the tribocouples. The test duration was 50 000 cycles. The contact displacement and the tangential force were measured online during the fretting tests. The tangential force-displacement hysteresis loops were acquired at regular time intervals and stored. The coefficient of friction was calculated from these data<sup>[11]</sup>.

After fretting tests, the wear scar was determined by laser profilometry (Rodenstock RM600). Depth profiles were acquired along the fretting wear scar normal to the sliding direction. A set of equally spaced depth profiles covering the whole wear track area were used to evaluate the volumetric wear as described previously<sup>[11]</sup>.

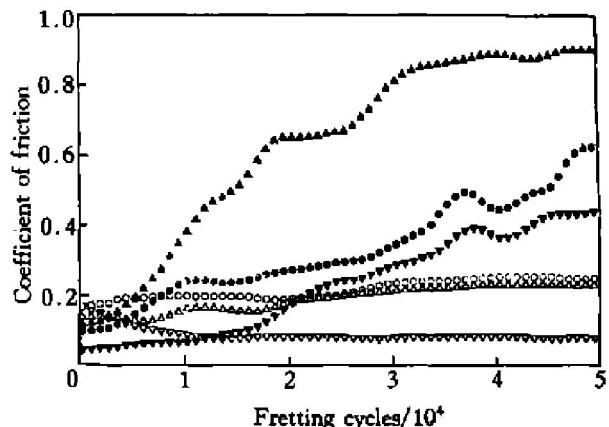
The morphology and structure of coatings were determined by respectively Philips XL30 scanning electron microscopy and Siemens D5000 X-ray diffractometer.

## 3 RESULTS

### 3.1 MOST and MOVIC coatings

The coefficient of friction of MOST and MOVIC coatings at different relative humidity and at a normal load of  $1\text{ N}$  is shown in Fig. 1. At less than  $10\%$  RH, MOST coating possesses a coefficient of friction of only about  $0.04\sim 0.07$ . But when the relative humidity increases to  $50\%$  or  $90\%$  RH, the coefficient of friction of MOST coating increases to  $0.14\sim 0.25$ . There is only a small difference in the coefficient of friction noticed between  $50\%$  and  $90\%$  RH.

Unlike MOST coating, the coefficient of friction of MOVIC coating increases with increasing test cycles. At the beginning of the fretting tests, the coefficient of friction is only about  $0.05$  for tests done at  $< 10\%$  RH or  $0.10\sim 0.13$  at  $50\%$  and  $90\%$  RH. As the MOVIC coating was quickly worn away especially



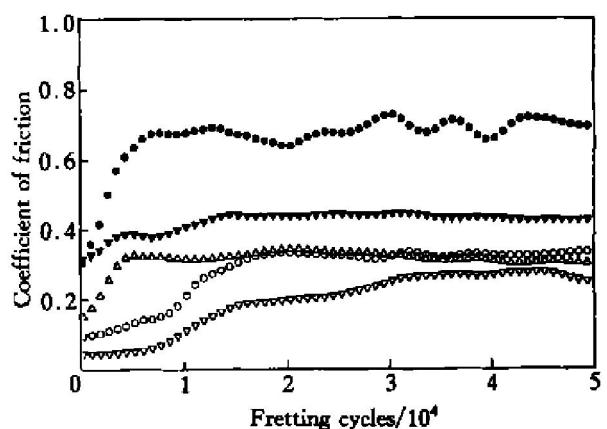
**Fig. 1** Coefficients of friction of MOST and MOVIC coatings sliding against corundum in air of different relative humidity and at a normal load of  $1\text{ N}$

▼—MOST, RH <  $10\%$ ; ○—MOST, RH =  $50\%$ ;  
△—MOST, RH =  $90\%$ ; ▼—MOVIC, RH <  $10\%$ ;  
●—MOVIC, RH =  $50\%$ ; ▲—MOVIC, RH =  $90\%$

at high relative humidity, the friction actually occurred between the substrate and the transfer film on the corundum ball at the final period of fretting process. At 50 000 cycles, the coefficient of friction of MOVIC coating has increased to  $0.42$  at  $< 10\%$  RH,  $0.62$  at  $50\%$  RH and  $0.90$  at  $90\%$  RH. Similar to the MOST coating, the coefficient of friction of MOVIC coating increases largely on the going of relative humidity from  $10\%$  RH to  $90\%$  RH.

### 3.2 MoSe<sub>2</sub>/Ni and WSe<sub>2</sub> coatings

Fig. 2 shows the coefficient of friction of MoSe<sub>2</sub>/Ni and WSe<sub>2</sub> coatings with test cycles under different relative humidity. After the running-in period, the average coefficient of friction of MoSe<sub>2</sub> / Ni coating



**Fig. 2** Coefficients of friction of MoSe<sub>2</sub>/Ni and WSe<sub>2</sub> coatings sliding against corundum in air of different relative humidity and at a normal load of  $1\text{ N}$

▼—MoSe<sub>2</sub>/Ni, RH <  $10\%$ ; ○—MoSe<sub>2</sub>/Ni, RH =  $50\%$ ;  
△—MoSe<sub>2</sub>/Ni, RH =  $90\%$ ; ▼—WSe<sub>2</sub>, RH <  $10\%$ ;  
●—WSe<sub>2</sub>, RH =  $50\%$ ; ▲—WSe<sub>2</sub>, RH =  $90\%$

stabilises at 0.25 for tests at < 10% RH or at 0.32 for tests at 50% and 90% RH. From the value of wear volume and the worn track observation by SEM after fretting process, it is proved that there is still  $\text{MoSe}_2$  coating at the bottom of worn track after 50 000 cycles' fretting. Compared with MOST and MOVIC coatings, there is only a small difference in the coefficient of friction of  $\text{MoSe}_2/\text{Ni}$  coating with relative humidity.

For  $\text{WSe}_2$  coating, the coefficient of friction at 50% RH is about 0.70, which is about twice as high as that at less than 10% RH. If we considered this value of the coefficient of friction,  $\text{WSe}_2$  coatings could not be called a low friction coating. The high friction originates from its preparation method, i. e. the rough and porous surface of the coating.

### 3.3 $\alpha\text{C: H}$ and $\text{WC/C}$ coatings

The coefficient of friction of  $\alpha\text{C: H}$  and  $\text{WC/C}$  coatings tested against corundum at different relative humidity and a normal load of 1N is shown in Fig. 3. For  $\alpha\text{C: H}$  coating, the coefficient of friction decreases rapidly with the increase of relative humidity. At 50 000 cycles, the coefficient of friction is separately 0.42 (< 10% RH), 0.23 (50% RH) and 0.14 (90% RH). As for  $\text{WC/C}$  coating, There is only a small difference noticeable in the coefficient of friction at the three levels of the relative humidity, the coefficient of friction is around 0.21~0.23.

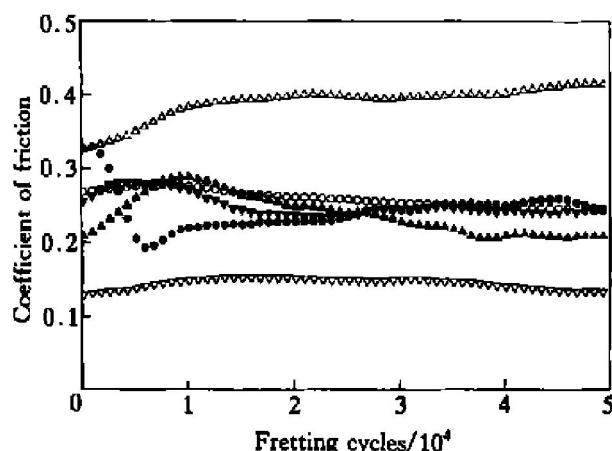


Fig. 3 Coefficients of friction of  $\alpha\text{C: H}$  and  $\text{WC/C}$  coatings sliding against corundum in air of different relative humidity and at a normal load of 1N  
 △— $\alpha\text{C: H}$ ,  $\text{RH} < 10\%$ ; ○— $\alpha\text{C: H}$ ,  $\text{RH} = 50\%$ ;  
 ▽— $\alpha\text{C: H}$ ,  $\text{RH} = 90\%$ ; ▼— $\text{WC/C}$ ,  $\text{RH} < 10\%$ ;  
 ●— $\text{WC/C}$ ,  $\text{RH} = 50\%$ ; ▲— $\text{WC/C}$ ,  $\text{RH} = 90\%$

### 3.4 Comparison of six coatings

The coefficient of friction and wear volume for the different coatings are summarized in Fig. 4 and Fig. 5. From these figures, it can be observed that the coefficient of friction and the wear volume of the four  $\text{MX}_2$  coatings increase obviously with increasing

humidity. Among the four  $\text{MX}_2$  coatings, the MOST and  $\text{MoSe}_2/\text{Ni}$  coatings have a lower friction and higher wear resistance than the MOVIC and  $\text{WSe}_2$  coatings. But the friction and wear properties of MOST coatings are affected by relative humidity, whereas the  $\text{MoSe}_2/\text{Ni}$  coating is hardly sensitive to the relative humidity in the ambient air.

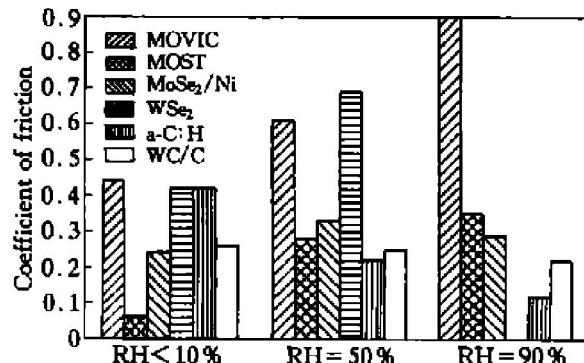


Fig. 4 Comparison of coefficient of friction of low friction coatings at three relative humidities after 50 000 cycles' fretting tests (10Hz, 100  $\mu\text{m}$  displacement amplitude, 1N normal load)

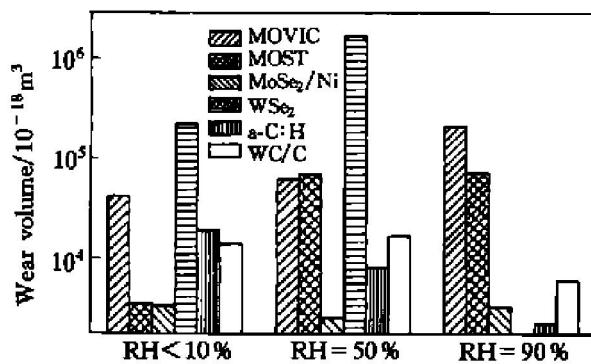


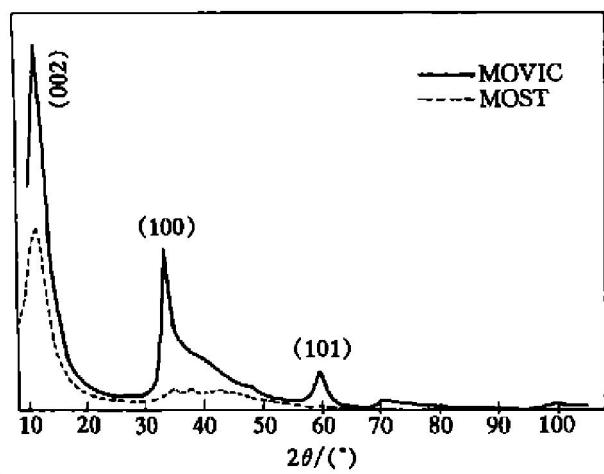
Fig. 5 Comparison of wear volume of six low friction coatings at three relative humidities after 50 000 cycles' fretting tests (10Hz, 100  $\mu\text{m}$  displacement amplitude, 1N normal load)

For  $\alpha\text{C: H}$  and  $\text{WC/C}$  coatings, the general trend is that the coefficient of friction and the wear volume decrease with increasing relative humidity. This conclusion is the same as that for graphite and carbon materials<sup>[12, 13]</sup>. The friction and wear resistance of  $\alpha\text{C: H}$  coating at higher relative humidity such as 50% RH or 90% RH, are better than the ones of  $\text{WC/C}$  coatings. The superiority of the  $\text{WC/C}$  coating is related to the fact that the performance of this coating is not sensitive to relative humidity.

## 4 DISCUSSION

The friction and wear properties of materials normally depend on their surface morphologies, structures and test conditions. For  $\text{MX}_2$  and graphitic carbon materials that have crystal layer structure, the

bonds within the layer such as between the Mo and S atoms are covalent and strong, whereas the interlayer attractions between the adjacent layer (adjacent S layer) are weak and consist basically of weak Van Der Waals forces. It is this weak interlayer bonding that contributes to the low shear strength during sliding, which in turn is reflected in the low coefficient of friction<sup>[7, 14]</sup>. So if the basal plane (002) in the coating is parallel to the surface (sliding direction), the coating will display excellent lubricating properties with a very low coefficient of friction. If the basal plane is perpendicular to the surface or the non-basal planes (100) and (110) (edge orientation) are parallel to the surface, the coefficient of friction of the coating will be higher even though the coating can be reoriented the crystals with its basal planes parallel to the sliding surface during the sliding process. Seitzman<sup>[15]</sup> showed that the ratio of edge-to-basal plane orientation is related to tribological properties of the coating. A low ratio is shown to promote coatings with a long lifetime and low coefficient of friction, whilst the high ratio has the opposite effect. Fig. 6 shows the X-ray diffraction pattern of MOVIC and MOST coatings. In the pattern of the MOVIC coating, not only the basal plane (002) but also non-basal planes such as (100) and (101) planes are noticed, whereas only basal plane (002) is appearing in the MOST, as it is the case for WSe<sub>2</sub> and MoSe<sub>2</sub>/Ni coatings too. So the high coefficient of friction of the MOVIC coating can be related to its unfavourable crystallographic orientation.



**Fig. 6** XRD patterns of MOVIC and MOST coatings investigated in this study

As for the surface morphology, we know that the friction force is a sum of two terms<sup>[16]</sup>, namely the product of the contact area and the shear strength, and a ploughing term associated with the translation of a harder penetrating material through a softer material. For thin and smooth solid lubricating coatings such as MOST and MoSe<sub>2</sub>/Ni, the ploughing term is negligible; but for the thick and rough

coating, the ploughing term could not be neglected. Maybe this term is even larger than the product of contact area and shear strength. It was observed by scanning electron microscope that the WSe<sub>2</sub> coating has a large grain size of about 50  $\mu$ m and a very rough and porous surface. This characteristic results from the preparation method used in which ion bombardment is needed to smoothen and densify the surface. In contrast to WSe<sub>2</sub> coating, the MOST, MoSe<sub>2</sub>/Ni and MOVIC coatings have a fine grain size and a very smooth surface, thanks to an ion bombardment or a low growth rate during the deposition.

Another important factor that greatly affects the tribological properties of solid lubricating coatings is humidity. For MX<sub>2</sub> structure<sup>[7, 17]</sup>, the basal plane is extremely resistant to the reactions with oxygen and water vapours. However the sites of edge planes exhibit a preferential affinity for polar compounds. At the edge sites, Mo can exist in a number of different oxidation states. The adsorption of oxygen or water vapour forms thin layers of MoO<sub>3</sub>. Since the formation of MoO<sub>3</sub> hinders the easy shearing, thus increasing the coefficient of friction. From the results of MOST and MOVIC coatings (Fig. 1), it can be seen that the increment of friction coefficient of MOVIC coating with the humidity is much bigger than that of MOST coating. This difference is related to their different structures. Contrast to MX<sub>2</sub> coatings, graphitic carbon coatings require the presence of humidity to produce a lubricating effect<sup>[18]</sup>, so the coefficient of friction of WC/C and  $\alpha$ C: H coatings decreases with the increasing of humidity.

## 5 CONCLUSIONS

The friction and wear properties of six low friction coatings currently available on the market were investigated under fretting in air of different relative humidity. The results can be summarised as follows.

- 1) The coefficient of friction of MX<sub>2</sub> type coatings (MOVIC, MOST, MoSe<sub>2</sub>/Ni and WSe<sub>2</sub>) increases when the humidity increases. On the contrary, the coefficient of friction of DLC type coatings ( $\alpha$ C: H and WC/C) decreases with increasing humidity.
- 2) Among these six coatings, the MOST and  $\alpha$ C: H coatings have the lowest coefficient of friction and the highest wear resistance; and MoSe<sub>2</sub>/Ni and WC/C coatings are not sensitive to the humidity in the ambient air.

## [ REFERENCES ]

- [1] Hirvonen J P, Koskinen J, Jervis J R, et al. Present progress in the development of low friction coatings [J]. Surf Coat Technol, 1996, 80: 139– 150.
- [2] Cohen S R, Rapoport L, Ponomarev E A, et al. The tri-

biological behavior of type II textured  $\text{MX}_2$  ( $\text{M} = \text{Mo, W; X} = \text{S, Se}$ ) films [J]. *Thin Solid Films*, 1998, 324: 190–197.

[3] Wanstrand O, Larsson M, Hedenqvist P. Mechanical and tribological evaluation of PVD WC/C coatings [J]. *Surf Coat Technol*, 1999, 111: 247–254.

[4] Roth D, Rau B, Roth S, et al. Large area and three dimensional deposition of diamond-like carbon films for industrial application [J]. *Surf Coat Technol*, 1995, 74/75: 637–641.

[5] Derflinger V, Brandle H, Zimmermann H. New hard/lubricant coating for dry machining [J]. *Surf Coat Technol*, 1999, 113: 286–292.

[6] Fleischauer P D, Bauer R. Chemical and structural effects on the lubrication properties of sputtering  $\text{MoS}_2$  films [J]. *Tribol Trans*, 1988, 31(2): 239–250.

[7] Spalvins T. A review of recent advances in solid film lubrication [J]. *J Vac Sci Technol*, 1987, A5(2): 212–219.

[8] Donnet C. Advanced solid lubrication coatings for high vacuum environments [J]. *Surf Coat Technol*, 1996, 80: 151–156.

[9] Hilton M R, Fleischauer P D. Applications of solid lubricant films in spacecraft [J]. *Surf Coat Technol*, 1992, 54–55: 435–441.

[10] Fox V, Hampshire J, Teer D.  $\text{MoS}_2$ /metal composite coatings deposited by closed field unbalanced magnetron sputtering: tribological properties and industrial uses [J]. *Surf Coat Technol*, 1999, 112: 118–122.

[11] Mohrbacher H, Celis J P, Roos J R. Laboratory testing of displacement and load induced fretting [J]. *Tribol Inter*, 1995, 28(5): 269–278.

[12] Halling J. *Principles of Tribology* [M]. London: The Macmillan Press Ltd, 1975. 82.

[13] Grill A, Patel V, Meyerson B S. Optical and tribological properties of heat-treated diamond-like carbon [J]. *J Mater Res*, 1990, 11(5): 2531–2537.

[14] Bowden F P, Tabor D. *The Friction and Lubrication of Solids* [M]. Oxford: Clarendon Press, 1964. 192.

[15] Seitzman L E, Bolster R N, Signer I L. Effects of temperature and ion to atom ratio on the orientation of IBAD  $\text{MoS}_2$  coatings [J]. *Thin Solid Films*, 1995, 260: 143–147.

[16] Bowden F P, Tabor D. *Friction: An Introduction to Tribology* [M]. Garden City, NY: Anchor Press, 1973. 91.

[17] Feng H C, Chen J M. Effects of low-energy argon ion bombardment on  $\text{MoS}_2$  [J]. *J Phys*, 1974, C7: L75–L78.

[18] Lancaster J K. Transitions in the friction and wear of carbons and graphites sliding against themselves [J]. *ASLE Trans*, 1975, 18(3): 187–201.

(Edited by YUAN Sai-qian)