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# Fretting wear rate of sulphur deficient MoS<sub>x</sub> coatings based on dissipated energy

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The fretting wear of sulphur-deficient MoS<sub>x</sub> coatings with different crystallographic orientations has been investigated in ambient air of controlled relative humidity. The coefficient of friction and the wear rate of MoS<sub>x</sub> coatings sliding against corundum depend not only on fretting parameters like contact stress, fretting frequency, and relative humidity, but also strongly on the crystallographic orientation of the coatings. For randomly oriented MoS<sub>x</sub> coatings, the coefficient of friction and the wear rate increased significantly with increasing relative humidity. In contrast, basal-oriented MoS<sub>x</sub> coatings were less sensitive to relative humidity. The coefficient of friction of both types of MoS<sub>x</sub> coatings decreased on sliding against corundum with increasing contact stress and decreasing fretting frequency. A correlation between dissipated energy and wear volume is proposed. This approach allows detection in a simple way of differences in fretting wear resistance between random- and basal-oriented MoS<sub>x</sub> coatings tested in ambient air of different relative humidity.

## I. INTRODUCTION

MoS<sub>2</sub> is known for its excellent solid lubrication properties in reducing friction, wear, and energy loss. MoS<sub>2</sub> coatings have already been successfully used in high vacuum and aerospace environments.<sup>1</sup> The main obstacle to the use of MoS<sub>2</sub> under ambient condition is its high sensitivity to moisture.<sup>2,3</sup> Different methods have been tried out to improve the humidity resistance of MoS<sub>x</sub> coatings in air of high relative humidity, such as codeposition with other materials,<sup>4-6</sup> producing multilayers,<sup>7</sup> and postdeposition processes, e.g., ion beam bombardment<sup>8</sup> and laser beam treatment.<sup>9</sup> Until now, the tribological properties of MoS<sub>2</sub> coatings reported were mainly related to pin-on-disk wear tests.<sup>10-14</sup> However, coatings

are frequently subjected to oscillating contact conditions. Friction and wear data for MoS<sub>2</sub> coatings under such bi-directional contact conditions are not yet available. It is widely accepted that the coefficient of friction of MoS<sub>2</sub> coatings decreases with increasing contact stress. However, the effects of sliding speed, as well as the combined influence of load and speed on the coefficient of friction of MoS<sub>2</sub> coatings, have not been deeply investigated.

Most literature deals with the coefficient of friction of MoS<sub>2</sub> coatings, and only limited attention has been given to the wear resistance of MoS<sub>2</sub> coatings. Indeed, monitoring the coefficient of friction is often the method to evaluate a sliding contact during an experiment. Based on friction force, several criteria can be applied to compare the wear resistance of coatings, such as the increase of the friction up to a certain value, an abrupt increase of the friction force or the emission of noise. On the other

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hand, the coefficient of friction of MoS<sub>2</sub> coatings can be remarkably stable, making sensing of the coating loss based on friction or torque signals difficult.

Understanding and comparing the wear resistance could be more helpful in optimizing durability and confidence in the use of MoS<sub>2</sub> coatings. So, e.g., Wahl *et al.*<sup>15</sup> proved that the wear and lubrication failure of MoS<sub>2</sub> coatings can be investigated by the *in situ* measurement of the electrical contact resistance.

In this study, MoS<sub>x</sub> coatings with different crystallographic orientations were evaluated in fretting tests performed in air of different relative humidity. The effects of contact stress and fretting frequency on the coefficient of friction and the wear rate of MoS<sub>x</sub> coatings were analyzed. The correlation between dissipated energy and wear volume<sup>16</sup> was used to evaluate the wear resistance of sulphur-deficient MoS<sub>x</sub> coatings with different crystallographic orientations.

## II. EXPERIMENTAL

MoS<sub>x</sub> coatings were deposited by planar magnetron sputtering by Institute for Materials Research-Limburgs Universitair Centrum (IMO-LUC) (Diepenbeek, Belgium). Hardened and polished (Ra = 0.05 μm) 440C stainless steel substrates were used. The depositions were performed at a substrate temperature of about 80 °C. The distance between the flat substrates and targets was kept at 65 mm, the deposition time was 14 min, the target power 8 W/cm<sup>2</sup>, and the argon pressure was varied between 0.2 and 2.4 Pa. Depositions were carried out with a commercially available target and electrically floating substrate holder, unless noted otherwise. X-ray diffraction measurements were done via Cu K<sub>α</sub> radiation with a Siemens D5000 diffractometer. The morphology and the cross-sectional structure of the coatings were investigated with a Philips XL 30 Scanning Electron Microscope (SEM).

Fretting wear tests (mode I) were performed under gross slip conditions at 23° C in ambient air at relative humidities (RH) of 10%, 50%, and 90%. Corundum balls with a diameter of 10 mm and surface roughness of approximately 0.2 μm (Ra) were used as counterbody. The balls were loaded on top of coated flat samples at a normal load of 1.0–10.0 N applied by springs.<sup>17</sup> The relative hertzian contact stress was in the range of 530 to 1143 MPa. The flat samples were oscillated at 1, 2, 5, or 10 Hz for a given number of fretting cycles. The tangential linear displacement amplitude imposed on the flat samples was 100 μm while the counterbody remained in a fixed position. The dissipated energy during fretting wear tests was obtained by summing up the energy dissipated during the successive fretting wear cycles. This was obtained by integrating numerically the force–displacement loops recorded during the fretting wear test

cycles. The wear volume was determined by laser stylus profilometer (Rodenstock RM600). In this procedure, depth profiles were acquired along the fretting wear track normal to the sliding direction. A set of equally spaced depth profiles covering the whole wear track was used to evaluate the wear volume. The morphology of the wear track was investigated by light optical microscopy using Normarski contrast.

## III. RESULTS AND DISCUSSION

XRD spectra of the sulphur-deficient MoS<sub>x</sub> coatings deposited at five different Ar pressures are shown in Fig. 1. At Ar pressures below 0.4 Pa, only the peak related to basal planes (.002) appeared. These basal planes were parallel to the substrate surface. These coatings were referred to as basal-oriented coatings. At Ar pressures between 0.6 and 2.4 Pa, the diffraction peaks related to edge planes became stronger. These coatings were further identified as random-oriented coatings.

The S/Mo ratio of the coatings was 0.69, 0.90, 1.20, 1.29, and 1.27 when the Ar pressure was 0.2, 0.4, 0.6, 1.5, and 2.4 Pa, respectively, during the depositions. Characteristic loops of tangential force vs. displacement recorded during fretting tests are shown in Fig. 2 for basal- (0.4 Pa Ar pressure) and random- (1.5 Pa Ar pressure) oriented MoS<sub>x</sub> coatings. The area of the loop represents the energy dissipated during each fretting cycle. The coefficient of friction was the ratio of the average tangential force during sliding to the applied normal force. The average tangential force over one fretting cycle was calculated by numerically integrating the hysteresis loop and dividing the obtained work by the total displacement. All the energies dissipated during a certain fretting cycles were used to correlate with wear volume later on.

The coefficient of friction for basal- and random-oriented MoS<sub>x</sub> coatings sliding against corundum during fretting tests is shown in Fig. 3. During the initial 3000 to

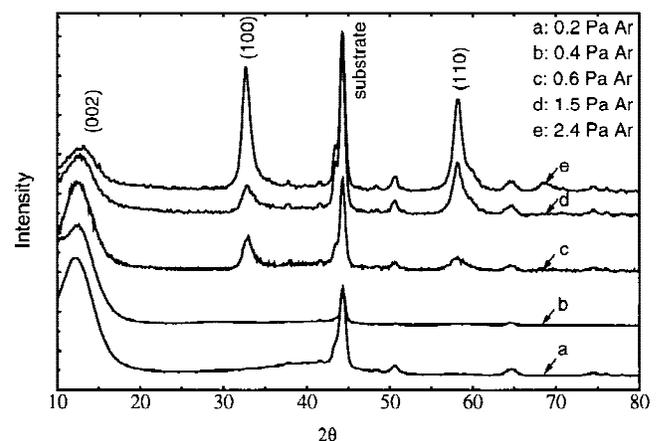


FIG. 1. XRD spectra of MoS<sub>x</sub> coatings deposited at different Ar pressures.

4000 cycles, the coefficient of friction gradually decreased for basal-oriented MoS<sub>x</sub> coatings and increased for random-oriented ones. After the running-in stage, the coefficient of friction became relatively stable, especially for the basal-oriented MoS<sub>x</sub> coatings, until wear-through of the coatings took place (not shown in Fig. 3). The coefficient of friction further reported in this paper referred to this stable stage and was the average of at least three identical tests repeated on a given sample with a fresh corundum counterbody each time.

The coefficient of friction of basal- and random-oriented MoS<sub>x</sub> coatings generally increased with increasing relative humidity (Fig. 4). For basal-oriented coatings deposited at an Ar pressure of 0.2 and 0.4 Pa, the coefficient of friction was lower in comparison to the

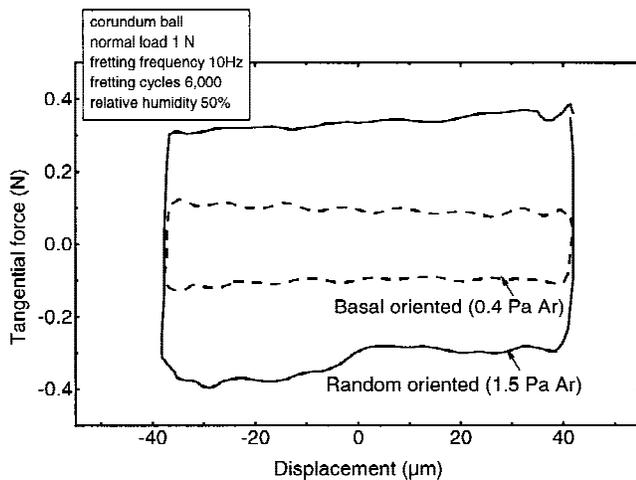


FIG. 2. Hysteresis loops of tangential force vs. fretting displacement in fretting tests performed in ambient air of 50% RH on MoS<sub>x</sub> coatings of different crystallographic orientation sliding against corundum.

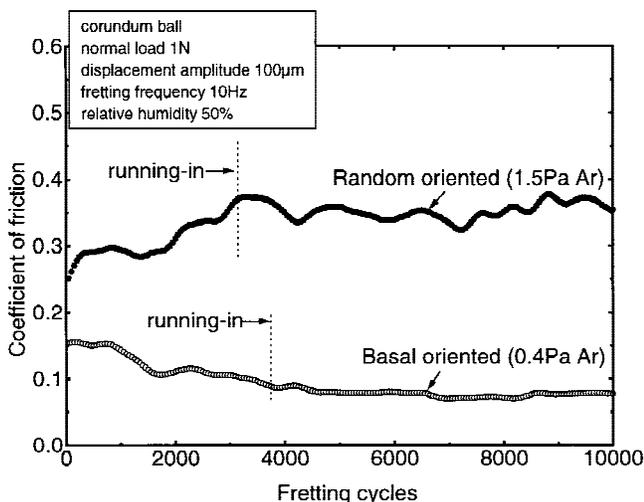


FIG. 3. The coefficient of friction versus fretting cycles for basal- and random-oriented MoS<sub>x</sub> coatings sliding against corundum in ambient air of 50% RH.

random-oriented coatings deposited at higher Ar pressure. This clearly appeared at the three levels of relative humidity tested. The lowest coefficient of friction at the three humidity levels tested was obtained with MoS<sub>x</sub> coatings deposited at an Ar pressure of 0.4 Pa. For random-oriented coatings, the coefficient of friction was not only high (above 0.2), but it increased significantly with increasing relative humidity. This implies that the random-oriented MoS<sub>x</sub> coatings are more sensitive to moisture than the basal-oriented MoS<sub>x</sub> coatings. This increase of the coefficient of friction of MoS<sub>x</sub> coatings with relative humidity was linked to the tribo-chemical reaction of MoS<sub>x</sub> with O<sub>2</sub> and H<sub>2</sub>O.<sup>18</sup> That reaction mainly takes place at the edge planes of MoS<sub>2</sub> crystals, because the vacant or dangling bonds of the edge planes are more reactive than the saturate bonds of basal planes. The more edge-oriented crystals were present in the as-deposited MoS<sub>x</sub> coatings, the more the tribo-chemical reaction would take place. For coatings deposited at an Ar pressure of 0.6 Pa, the fraction of edge-oriented crystals in the coatings was relatively low (see Fig. 1, curve c), and the tribo-chemical reaction reached a saturation level at a relative humidity of 50%. As a result, the coefficient of friction reached the highest level at 50% RH, and then decreased with a further increase of relative humidity. The coefficient of friction for coatings deposited at an Ar pressure of 2.4 Pa is not shown in Fig. 4 at a relative humidity of 90% because of the difficulties in identifying a stable friction stage resulting from the very limited wear life of the coatings leading to a premature wear-through.

The coefficient of friction of MoS<sub>x</sub> coatings sliding against corundum and recorded at different contact stresses and sliding speeds is shown in Fig. 5. When the sliding speed was kept constant, the coefficient of friction varied linearly with the inverse contact stress. The higher the contact stress, the lower the coefficient of

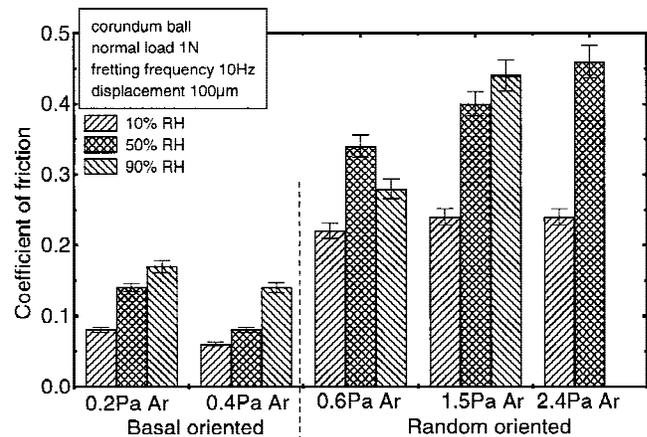


FIG. 4. The coefficient of friction of MoS<sub>x</sub> coatings deposited at different Ar pressures. Data obtained in fretting tests performed in ambient air of different relative humidity.

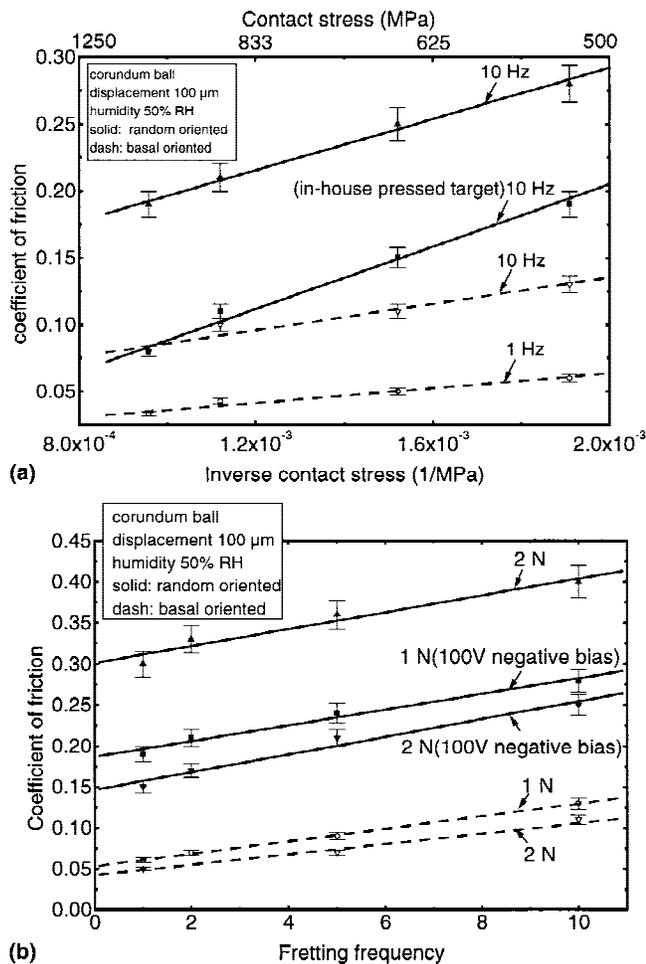


FIG. 5. The influence of (a) contact stress and (b) fretting frequency on the coefficient of friction of random- and basal-oriented MoS<sub>x</sub> coatings.

friction was [Fig. 5(a)]. The coefficient of friction also varied linearly with fretting frequency when the contact stress was kept constant. The lower the sliding speed, the lower the coefficient of friction was [Fig. 5(b)]. The contact stress model proposed by Singer *et al.*<sup>19</sup> suggests that the coefficient of friction  $\mu$  of MoS<sub>2</sub> coatings can be expressed as a function of contact stress  $P$ :

$$\mu = S_0/P + \alpha \quad (1)$$

with  $S_0$  the shear strength of MoS<sub>2</sub> coatings, and  $\alpha$  treated as a constant.  $S_0$  corresponds to the slope of each curve in Fig. 5(a).  $S_0$  appeared to be almost independent of sliding speed when the fretting frequency changed from 10 to 1 Hz for basal-oriented coatings. The same was noticed for random-oriented coatings (not shown in Fig. 5). The shear strength of MoS<sub>2</sub> coatings depended thus heavily on the crystallographic orientation of the MoS<sub>x</sub> coatings. For basal-oriented MoS<sub>x</sub> coatings,  $S_0$  was in the range of 30–50 MPa, while it increased to 90–120 MPa for random-oriented MoS<sub>x</sub> coatings in our fretting

(mode I) tests performed at 50% relative humidity under gross slip conditions. According to Singer *et al.*,<sup>19</sup>  $S_0$  was 24.8 MPa for sputter-deposited MoS<sub>2</sub> in dry air, and  $23 < S_0 < 33$  MPa for burnished MoS<sub>2</sub> in ambient air, but different values were published by Grosseau-Poussard *et al.*<sup>20</sup> He found that  $S_0$  increases from 40 MPa in dry air to 80 MPa at 60% relative humidity. The relative higher  $S_0$  value noticed in our tests is considered as resulting from the bi-directional sliding and the higher relative humidity used in our tests. But eventually it may also result from the specific orientations of our MoS<sub>x</sub> coatings.

The coefficient of friction depended not only on contact stress  $P$ , but also on sliding speed  $V$ , as can be seen from Fig. 5(b). As a first approximation, it can then be assumed that

$$\mu = S_0/P + \beta \times V \quad (2)$$

with  $\beta$  the velocity dependence of the coefficient of friction.  $\beta$  is the slope of the curves in Fig. 5(b).  $\beta$  appeared as independent of contact stress, since the curves in Fig. 5(b) were parallel for normal loads of 1 and 2 N for a given MoS<sub>x</sub> coating.  $\beta$  appeared to be insensitive to the crystallographic orientation of the MoS<sub>x</sub> coatings and was between  $1.0 \times 10^{-6}$ – $0.2 \times 10^{-5}$  (m<sup>-1</sup>s) in our fretting mode I tests performed under gross slip conditions.

The fact that the coefficient of friction decreased with decreasing sliding speed suggested that MoS<sub>x</sub> coatings offer a lower flow resistance at decreasing sliding speed. A similar result has been reported for compact graphite.<sup>21</sup> On the contrary, an increase of the sliding speed resulting in a decrease in friction has been reported for pin-on-disk tests<sup>22–24</sup> on MoS<sub>2</sub> coatings. However, this was attributed to a loss of moisture from the MoS<sub>2</sub> films induced by a frictional heating at higher speeds. The decrease of the coefficient of friction in that case was considered to result from a loss of moisture and not from the change in sliding speed. The contradiction between our results and literature may be due to the fact that the sliding speed in our fretting tests was much lower ( $2 \times 10^{-4}$  to  $2 \times 10^{-3}$  m s<sup>-1</sup>) than that in pin-on-disk tests reported in literature (0.02 to 0.2 m s<sup>-1</sup>). An increase in temperature at the sliding speed used in our fretting tests, can thus be neglected. Secondly, the effect of varying sliding speeds during fretting tests may play a more important role on the coefficient of friction than in unidirectional sliding conditions as pin-on-disk tests.

To fully understand the tribological behavior, the evolution of wear on MoS<sub>x</sub> coatings was recorded (Figs. 6 and 7). The dimensions of the wear tracks for both the basal- [Fig. 7(a)] and the random- [Fig. 7(b)] oriented MoS<sub>x</sub> coatings increased gradually with increasing fretting wear cycles. Some of the debris was ejected out of the wear tracks and accumulated mainly at both ends

of the fretting wear tracks in sliding direction (Fig. 6). A small amount of debris was found along the lateral sides of the wear tracks. This phenomenon was common in fretting wear tests.

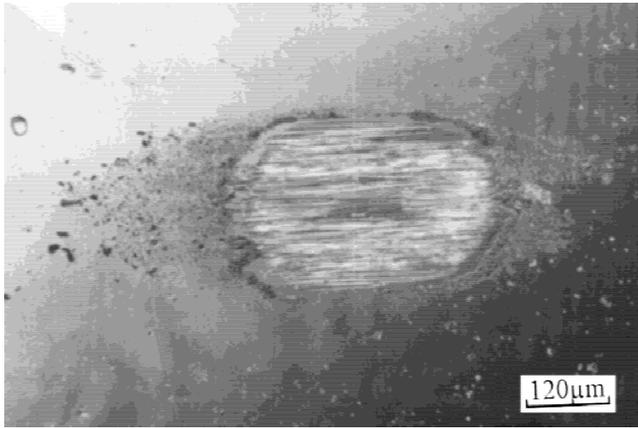
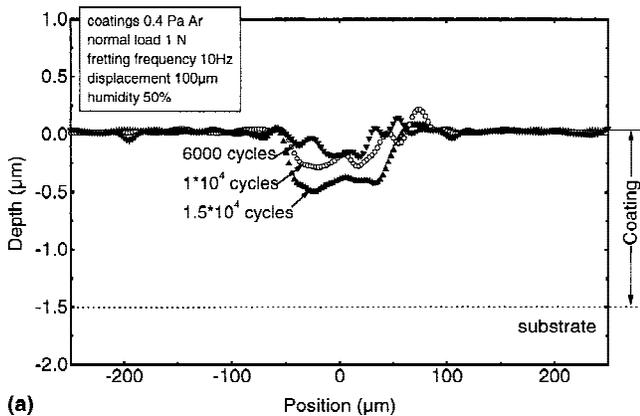
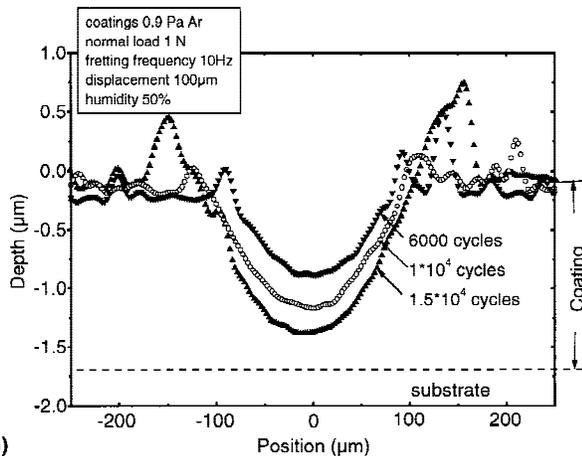


FIG. 6. Normarski micrograph of wear track on a random-oriented MoS<sub>x</sub> coatings deposited at an Ar pressure of 0.9 Pa and sliding against corundum in ambient air of 50% RH for 15,000 cycles (*s*, 100 μm; *F<sub>n</sub>*, 1 N; *f*, 10 Hz).



(a)



(b)

FIG. 7. Cross-sections of wear tracks perpendicular to the sliding direction on (a) basal- and (b) random-orientated MoS<sub>x</sub> coatings by laser stylus profilometer.

On comparing width and depth of the wear tracks, it was apparent that the size of wear tracks increased faster in their width than in their depth (Fig. 7). Therefore the wear resistance of MoS<sub>x</sub> coatings in fretting wear tests should be evaluated by reference based on wear volume value than on wear depth.

Such a comparison of the fretting wear volume on MoS<sub>x</sub> coatings tested in ambient air of different relative humidity, is given in Fig. 8. The wear volume on basal-oriented MoS<sub>x</sub> coatings (0.4 Pa Ar pressure) remained at a low level even at increasing relative humidity. Coatings deposited at even lower Ar pressure, 0.2 Pa, showed higher and larger scattering in wear volumes at the three relative humidity levels. In contrast, for random-oriented MoS<sub>x</sub> coatings (0.6 up to 2.4 Pa Ar pressure), the fretting wear volume was not only high in comparison with the basal-oriented MoS<sub>x</sub> coatings, but it also increased significantly with increasing relative humidity. For coatings deposited at an Ar pressure of 2.4 Pa, the wear resistance was so poor that at 50% and 90% relative humidity wear-through of the coatings occurred within 10,000 fretting cycles. On comparison of Fig. 4 with Fig. 8, it is evident that no conclusion can be drawn from the coefficient of friction on the wear volume after a given number of fretting cycles.

An appropriate and reliable method is thus needed to compare the wear resistance of MoS<sub>x</sub> coatings with different crystallographic orientations. The area of the loops of tangential force versus displacement, shown in Fig. 2, represents the energy dissipated during the corresponding fretting cycles. The total dissipated energy after a given number of fretting cycles was obtained by summing up these dissipated energies over all fretting cycles. On plotting that dissipated energy against the wear volume in fretting tests done at a constant relative humidity, a linear relation was obtained for each MoS<sub>x</sub> coating deposited at different Ar pressures (Fig. 9). The

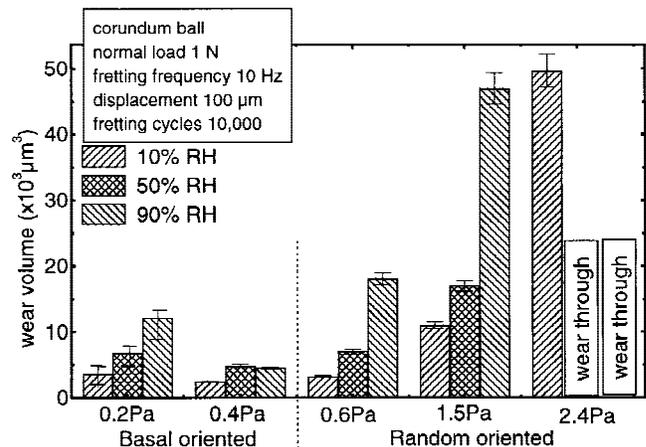


FIG. 8. Wear volume of MoS<sub>x</sub> coatings deposited at different Ar pressures. Fretting tests were done at different relative humidity for 10,000 cycles (*s*, 100 μm; *F<sub>n</sub>*, 1 N; *f*, 10 Hz).

higher the Ar pressures in the deposition processes, the higher the wear rate, expressed in  $\mu\text{m}^3/\text{J}$ , of the coatings. But the wear rate was higher for coatings deposited at 0.2 Ar pressure than that at 0.4 Ar pressure (not shown in Fig. 9). Such a linear dependence of wear volume on dissipated energy has already been reported for TiN coatings.<sup>16,25</sup> In that case no dependence on relative humidity was found, but for MoS<sub>x</sub> coatings, a dependence of the wear rate in fretting tests on relative humidity was noticed (Fig. 10). For basal-oriented coatings [Fig. 10(a)], the wear rate remained almost unchanged when the relative humidity increased from 10% to 50%, although a slightly higher wear rate was noticed at 90% relative humidity. For random-oriented MoS<sub>x</sub> coatings, the wear rate increased with increasing relative humidity [Figs. 10(b) and 10(c)]. The higher the Ar pressure during deposition, the lower the humidity resistance of the coatings was. Three reasons for this difference in resistance to humidity between basal- and random-oriented MoS<sub>x</sub> coatings can be put forward. The edge planes of MoS<sub>2</sub> crystals were more reactive than basal planes. The tribo-chemical reaction on random-oriented MoS<sub>x</sub> coatings occurred more easily under higher humid conditions than on basal-oriented MoS<sub>x</sub> coatings, and more MoO<sub>3</sub> particles were generated on the wear tracks and in the debris.<sup>18</sup> Secondly, the presence of brittle MoO<sub>3</sub> particles may cause an abrasive wear on these hard to slide edge planes. The formation of MoO<sub>3</sub> would thus affect more badly the tribological performance of the random-oriented coatings than in the case of basal-oriented ones. And finally, the basal-oriented coatings had a dense and noncolumnar structure as shown in Fig. 11(a). Cracks could not easily propagate through the cross-section of such basal-oriented coatings. In contrast, the random-oriented coatings [Figs. 11(b) and 11(c)]

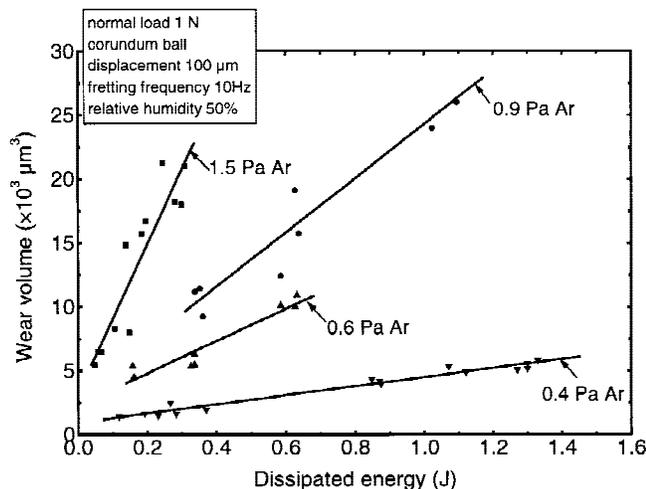


FIG. 9. Wear volume as a function of dissipated energy for MoS<sub>x</sub> coatings deposited at different Ar pressures and tested in fretting mode I.

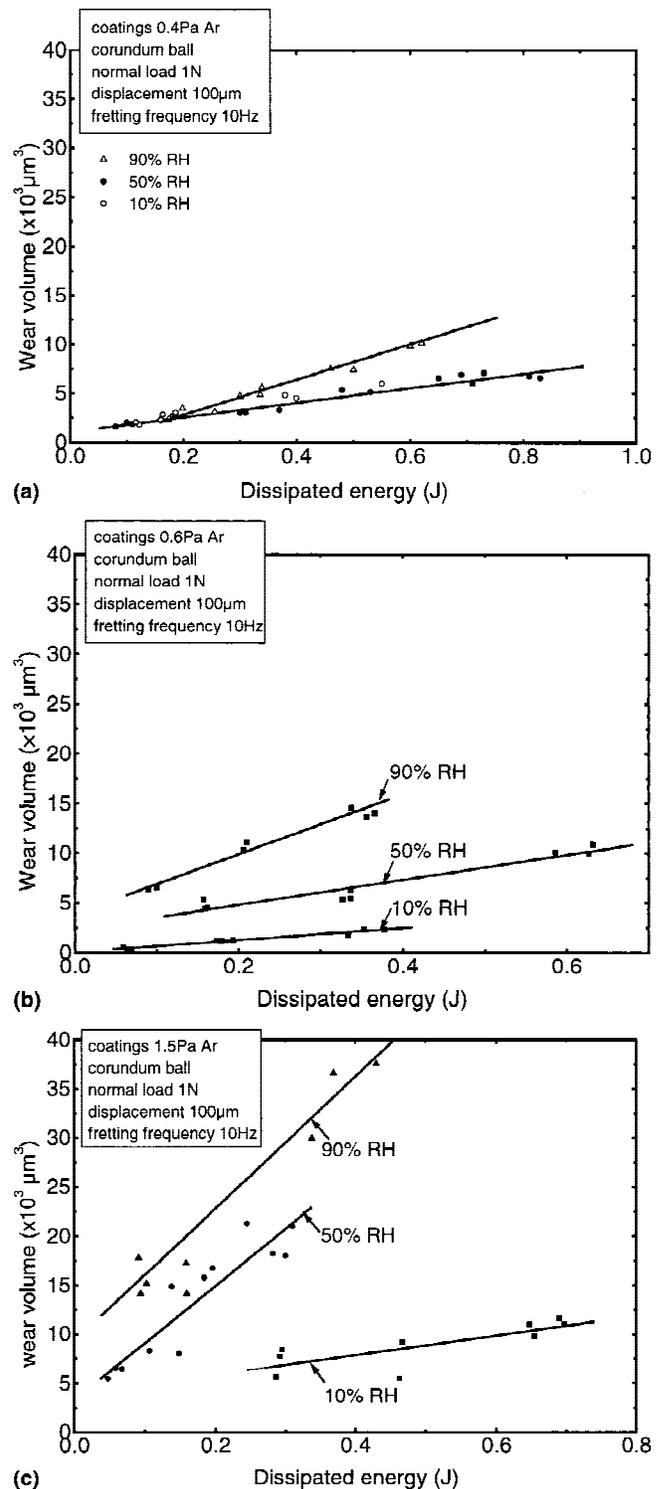


FIG. 10. Fretting wear volume as a function of dissipated energy for (a) basal- and (b,c) random-oriented MoS<sub>x</sub> coatings tested at different relative humidity levels.

possessed a porous columnar structure that became more pronounced at higher Ar pressure in the deposition process. The columnar structure could provide a path for moisture to easily penetrate the coatings.

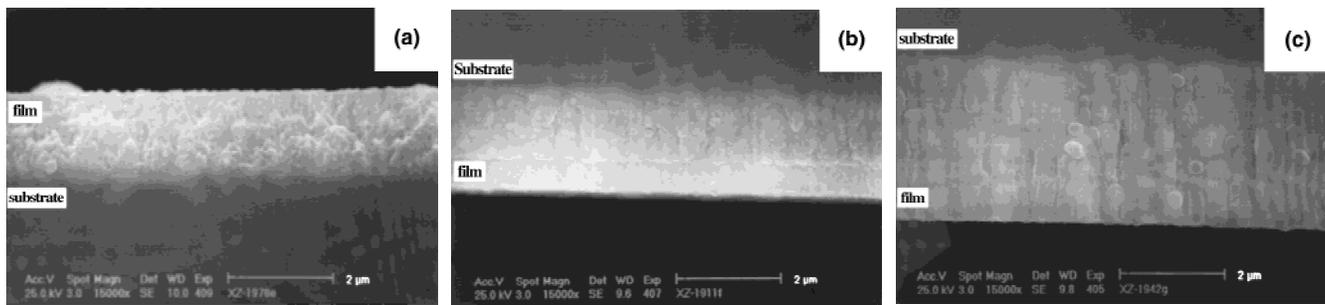


FIG. 11. Cross-section of MoS<sub>x</sub> coatings deposited at the Ar pressure of (a) 0.4, (b) 0.6, and (c) 1.5 Pa.

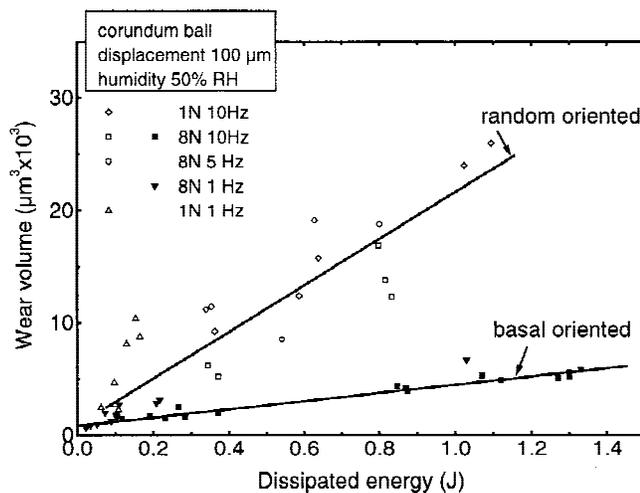


FIG. 12. The influence of contact stress and fretting wear frequency on wear rate of basal and random-oriented MoS<sub>x</sub> coatings.

An interesting finding was that the linear relation between wear volume and dissipated energy was valid for different contact stresses and fretting frequencies (Fig. 12). Xu *et al.*<sup>26</sup> have reported that increasing wear volume occurs with increasing fretting cycles under different sliding amplitudes and normal loads. These results indicated that, using load and sliding amplitude as reference parameters, the dependence of wear volume on load and sliding amplitude was complicated. From our results, it appeared that, on plotting wear volume versus dissipated energy (Figs. 10 and 12) that unclarity disappeared. The relationship between wear volume and dissipated energy was linear for both random- and basal-oriented coatings tested at different fretting testing conditions of relative humidity, normal load, and contact frequencies. Selecting dissipated energy as the governing parameter allows an easy and flexible comparison of fretting wear rate. Loading and fretting cycles affect independently wear rate. Thus dissipated energy appears to be a more appropriate tool for comparing wear rate of coatings in general, and MoS<sub>x</sub> specifically, tested under different fretting conditions.

It is interesting to compare our results on wear rate of MoS<sub>x</sub> coatings with different crystallographic orientation with literature. A few authors claimed that the wear life increased with the thickness of MoS<sub>x</sub> coatings<sup>27–29</sup> and that the wear volume increased gradually with the number of fretting cycles.<sup>26</sup> On the contrary, some literature data showed that the wear rate of MoS<sub>x</sub> coatings seemed to be very high at the beginning of wear tests<sup>30</sup> and that 2/3 of the film wore away in the running-in stage.<sup>31</sup> It was argued that MoS<sub>2</sub> coatings with a columnar structure might break up in between the upper columns and their base. As the columns collapsed, the base region acted as a lubricating layer, so the effective thickness of the lubricant was very limited.<sup>30</sup> In most cases, our tests showed that the wear volume of MoS<sub>x</sub> coatings increased gradually with increasing fretting cycles. On the contrary, an abrupt wear occurred in the case of random-oriented coatings subjected to a high contact stress at a low fretting frequency. So the contradictory results reported in literature might result from the combined effect of orientation and thickness of the coatings, contact stress, and sliding speed during the wear tests. High or abrupt wear loss would occur for random-oriented coatings at high contact stress and low sliding speed. When random-oriented coatings were tested under low contact stress and high sliding speed, the wear loss increased gradually as was noticed for the basal-oriented coatings under all test conditions.

#### IV. CONCLUSIONS

The coefficient of friction and the wear rate of MoS<sub>x</sub> coatings depend not only on the crystallographic orientation and relative humidity but also on the contact stress and frequency during fretting mode I wear tests. For random-oriented MoS<sub>x</sub> coatings, the coefficient of friction and the wear rate increased significantly with increasing relative humidity. The basal-oriented MoS<sub>x</sub> coatings showed little sensitivity to different levels of relative humidity. The coefficient of friction of MoS<sub>x</sub> coatings decreased with the increase of contact stress and the decrease of fretting frequency. The dependence of the

coefficient of friction ( $\mu$ ) on contact pressure ( $P$ ) and sliding speed ( $V$ ) can be expressed as  $\mu = S_0/P + \beta \times V$ .  $S_0$  was the shear strength of MoS<sub>x</sub> coatings, which depended strongly on the orientation of the coatings. The shear strength of basal-oriented MoS<sub>x</sub> coatings was lower than that of the random-oriented ones.  $\beta$  was the velocity coefficient of friction force, which was not sensitive to the orientation of the coatings. The wear resistance of MoS<sub>x</sub> coatings can be derived from the linear relation between dissipated energy and wear volume. That linear relation was valid for the basal- and random-oriented MoS<sub>x</sub> coatings tested at different contact stress, fretting frequency, and humidity level. This approach allows a more flexible and systematic comparison of the wear rate of different MoS<sub>x</sub> coatings tested at different fretting mode I test conditions.

## ACKNOWLEDGMENTS

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