Tribological Properties of Mo$_2$N Films at Elevated Temperature

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Abstract: Mo$_2$N films were synthesized using the reactive magnetron sputtering system in a mixture of argon and nitrogen, and the tribological properties were investigated at different testing temperatures against an Al$_2$O$_3$ counterpart. The relative intensity ratio (RIR) method was used to calculate the weight fraction of the tribo-film (MoO$_3$) on the wear tracks of the films. The results showed that the average friction coefficient first increased from 0.30 at 25 $^\circ$C to 0.53 at 200 $^\circ$C, and then decreased to 0.29 at 550 $^\circ$C, while the wear rate decreased from 2.1 $\times$ 10$^{-6}$ mm$^3$/Nmm at 25 $^\circ$C to 5.3 $\times$ 10$^{-7}$ mm$^3$/Nmm at 200 $^\circ$C and then increased to 3.1 $\times$ 10$^{-5}$ mm$^3$/Nmm at 550 $^\circ$C. The weight fraction of tribo-film was mainly attributed to changes in the average friction coefficient and the wear rate. Besides this, the relative humidity also influenced the tribological properties at 25–200 $^\circ$C.

Keywords: reactive magnetron sputtering; Mo$_2$N films; elevated temperature; tribological properties

1. Introduction

Recently, Magnéli phases have been attracting an increasing level of interest due to their “easy sliding” crystallographic shear planes [1–4]. These phases were reported to reduce the average friction coefficient significantly [5–7]. For example, S.M. Aouadi et al. synthesized adaptive Mo$_2$N/MoS$_2$/Ag composite films which showed that the molybdenum oxide phase which formed on the wear track could play a lubricating role during the wear test [8,9]. Molybdenum nitrides and carbides deposited by physical vapor deposition (PVD) possess many unique physical and mechanical properties, such as high hardness, low average friction coefficients, good adherence to steel substrates, and low electrical resistivity [10–13]. Specifically, Mo$_2$N-based films synthesized using PVD were reported to exhibit excellent friction properties at room temperature [4,11,14]. Nowadays, the mechanical and tribological properties of molybdenum nitride-based composite films or multilayer films have been attracting the attention of hard film scientists and engineers. However, discussion in the literature of the tribological properties of binary Mo$_2$N films at elevated temperatures is relatively scarce. The testing temperature has a significant influence on the tribological properties of transition metal nitride films. For instance, our group deposited Nb-Al-N films using a magnetron sputtering system; the results showed that the tribological properties of the film against a Si$_3$N$_4$ counterpart were significantly influenced by the testing temperature, since the tribo-film on the wear track was influenced by the testing temperature [15]. A magnetron-sputtered TiAlN/VN film showed variation in the average friction coefficient in a ball-on-disc wear test against an Al$_2$O$_3$ counterpart when the testing temperature was increased from 25 $^\circ$C to 700 $^\circ$C [16]. However, some transition metal nitride-based films containing copper or silver possessed tribological properties which were slightly influenced by the testing temperature, due to the self-lubricating nature of copper or silver [17–19]. G. Gassner et al. reported [4] the self-lubrication mechanism of Mo$_2$N at testing temperatures above 500 $^\circ$C, while the tribological properties at <500
°C and the wear resistance properties of the films were barely mentioned. There are two aims in this paper: (1) to investigate the tribological properties at 25–550 °C, especially the friction coefficient and wear rate at <500 °C; and (2) to reveal the influence factor of the friction coefficient and wear rate at 25–550 °C.

In this work, Mo2N films were deposited using RF unbalanced reactive magnetron sputtering, and the tribological properties were investigated at different testing temperatures between 25 °C and 550 °C against an Al2O3 counterpart. The relative intensity ratio (RIR) method, which is one of several quantitative X-ray diffraction method, was used to calculate the weight fraction of the tribo-film (MoO3) on the wear tracks of the films. The effects of testing temperatures on the tribological properties will be discussed.

2. Experiment

Mo2N films were deposited onto stainless steel (06Cr19Ni10) substrates using the RF magnetron sputtering system. AMo (99.95%) target with a diameter of 75 mm was sputtered by the radio frequency power. Mirror-polished stainless steel substrates were ultrasonically cleaned in acetone and alcohol, and then mounted onto the substrate holder in a vacuum chamber. The substrate holder was electrically grounded with no applied bias voltage. The target-to-substrate distance was 10 centimeters. After the base pressure reached 6 × 10⁻⁴ Pa, Ar (99.999%) and N₂ (99.999%) were introduced into the chamber by means of two separate gas manifolds. Prior to deposition, the substrate was heated to about 200 °C. The Mo2N films were created by fixing the power of the Mo targets at 150 W while constantly maintaining a working pressure of 0.3 Pa and a nitrogen-to-argon ratio (flow ratio) of 10:3.

The microstructure of the films was characterized by X-ray diffraction (XRD, Bruker D500, Karlsruhe, Germany) at a 2-degree incidence angle with a Siemens X-ray diffractometer using Cu Kα radiation, operating at 40 kV and 35 mA. The microstructure of the film was further studied by transmission electron microscopy (TEM, JEM-2100F, JEOL, Tokyo, Japan) operating at an accelerating voltage of 200 kV. A 30 min wear test was carried out along a circular track of 10 mm diameter against a 9 mm diameter Al2O3 counterpart at 50-rpm under a constant normal load of 5 N in the atmosphere using a UTM-2 CETR tribometer. The selected testing temperatures were RT (25 °C; relative humidity was ~55%), 100 °C, 200 °C, 300 °C, 400 °C, 500 °C, and 550 °C. The wear test at each testing temperature was repeated three times to ensuring data reliability. The wear track cross-sectional areas at eight different locations were measured using a profilometer and the accompanying software (BRUKER, Dektak-XT, Karlsruhe, Germany) after the wear test, and their average was used to calculate the wear rate (Ws) using the following formula [20]:

\[ W_s = \frac{C \times S}{F \times L} \]  

where C is the perimeter of the wear track; its value is ~31 mm in this paper, S is the average area of wear track obtained for a profilometer, F is the normal load; its value is 5 N in this paper, and L is the total sliding distance; its value is ~47,100 mm.

Scanning electron microscopy (SEM, JEM-6480, Tokyo, Japan) was used to study the wear track of the films. The elemental compositions of the wear track at various testing temperatures were characterized using energy dispersive spectroscopy (EDS, Oxford, UK) on an EDAX DX-4 energy dispersive analyzer.

3. Results and Discussion

3.1. Microstructure and Mechanical Properties

Across-sectional SEM image of a Mo2N film, in which a typical columnar crystal structure is visible, is shown in Figure 1. The film thickness was ~2 µm.
Figure 1. Cross-sectional SEM image of Mo$_2$N film.

Figure 2 illustrates the XRD pattern of the Mo$_2$N film. As shown in Figure 1, the Mo$_2$N film exhibits a single face-centered cubic (fcc) $\gamma$-Mo$_2$N structure with four diffraction peaks corresponding to fcc-Mo$_2$N (111), (200), (220), and (311) (PDF card #25-1366).

Transmission electron microscopy (TEM) was used to investigate the crystal structure of the Mo$_2$N film; the results are shown in Figure 3. Figure 3a is the HRTEM image; it reveals a clear crystal lattice. The inserted Fourier transformation (FFT) image of the square region, as shown in the Figure 3a, represents a single cubic phase structure, and the inserted filtered inverse fast Fourier transformation (IFFT) clearly shows that the lattice spacing is 0.2408 nm, referring to the fcc-Mo$_2$N (111). The selected area electron diffraction (SEAD) pattern shown in Figure 3b confirms that a single phase of fcc-Mo$_2$N existed in the films.

Based on our previous result in Reference [21], the hardness and the oxidation resistance temperature of the Mo$_2$N film was ~29 GPa and ~500 °C.
3.2. Tribological Properties

The average friction coefficient of the Mo$_2$N film at different testing temperatures is shown in Figure 4. It can be clearly observed that the testing temperature has a significant effect on the average friction coefficient. When the testing temperature is below 200 °C, the average friction coefficient increases from 0.30 at 25 °C to 0.53 at 200 °C. A further increase in testing temperature decreases the average friction coefficient; the minimum average friction coefficient is 0.29 at 550 °C. Similar friction properties were also reported in other transition metal nitride films, such as TiAlVN [22], ZrAlN [23], and W-Si-N [5].

**Figure 3.** Image (a) and the corresponding SEAD pattern (b) of the Mo$_2$N film.
Figure 4. Friction coefficient and wear rate of Mo₂N film as a function of testing temperatures.

Figure 4 also presents the wear rate as a function of various testing temperatures. In contrast to
the tendency of the average friction coefficient, the wear rate decreased from 2.1 × 10⁻⁶ mm³/Nmm
at 25 °C to 5.3 × 10⁻⁷ mm³/Nmm at 200 °C. However, a further increase in the testing temperature
increased the wear rate gradually, reaching a maximum value of 3.1 × 10⁻⁵ mm³/Nmm at 550 °C. The
film was worn out after the wear test at 600 °C. Therefore, the influence of the testing temperature—in
the range of 25–550 °C—on the tribological properties is discussed in this paper.

Tribo-film is known to significantly influence the tribological properties of transition metal nitride
films, since tribo-oxidation has been reported to be a generalized phenomenon [6,7,11]. In order to
study the effects of testing temperature on the tribo-film, the wear tracks at various testing temperatures
were analyzed by XRD; the result is shown in Figure 5. The XRD patterns confirm that the MoO₃ phase
was detected in all wear tracks, regardless of the testing temperature. When the testing temperature
was in the range between 25 °C and 200 °C, Mo₂N could form MoO₃ by a complex tribo-chemical
reaction between the wear track and moisture in the air [16]. The possible reaction might be as follows:

\[
2\text{Mo}_2\text{N} + 12\text{H}_2\text{O} = 4\text{MoO}_3 + 2\text{NH}_3 + 9\text{H}_2,
\]

Figure 5. Patterns of wear tracks at different testing temperatures.
Although the oxidation resistance temperature of the Mo$_2$N film was about ~500 °C, both friction heat and environment temperature induced oxidation in the wear track. The possible reactions are as follows [24]:

\[
\begin{align*}
Mo_2N + 2O_2 &= 2MoO_2 + (1/2)N_2, \\
MoO_2 + (1/2)O_2 &= MoO_3,
\end{align*}
\]

According to the XRD data in Figure 5, the relative intensity ratio (RIR) method [25] was used to calculate the weight fraction of Mo$_2$N and MoO$_3$. The general definition of the RIR for phase $\beta$ to reference phase $\alpha$ is given by:

\[
RIR_{\alpha,\beta} = \left(\frac{I_\beta}{I_\alpha}\right) \left(\frac{X_\alpha}{X_\beta}\right) R_I,
\]

where $I$ is the intensity and $X$ is the weight fraction. Rearranging the above equation:

\[
X_\beta = \left(\frac{I_\beta}{I_\alpha}\right) \left(\frac{X_\alpha}{RIR_{\alpha,\beta}}\right).
\]

$RIR_{\alpha,\beta}$ can be obtained by the following equation:

\[
RIR_{\alpha,\beta} = \frac{RIR_{\alpha,\gamma}}{RIR_{\beta,\gamma}}.
\]

The weight fraction of Mo$_2$N and MoO$_3$ can be calculated using the following equations:

\[
X_{Mo_2N} = \left(\frac{I_{Mo_2N}}{I_{MoO_3}}\right) \left(\frac{X_{MoO_3}}{RIR_{MoO_3,Mo_2N}}\right),
\]

\[
X_{Mo_2N} + X_{MoO_3} = 100\%,
\]

\[
RIR_{MoO_3,Mo_2N} = \frac{RIR_{MoO_3,Al_2O_3}}{RIR_{MoO_3,Al_2O_3}}.
\]

where $RIR_{MoO_3,Al_2O_3} = 4.59$ (PDF 65-2421) and $RIR_{MoO_3,Al_2O_3} = 16.72$ (PDF 25-1366).

The weight fractions of Mo$_2$N and MoO$_3$ on the wear tracks as a fraction of testing temperatures are illustrated in Figure 6. The weight fraction of MoO$_3$ first decreased and then increased with the increase of testing temperatures; the minimum value was about 0.8 wt.% at 200 °C. When the testing temperature was below 200 °C, the decrease of the weight fraction of MoO$_3$ was attributed to a decrease of moisture; when the testing temperature was between 200 °C and 550 °C, both the environmental temperature and the friction heat induced the formation of tribo-film MoO$_3$.

![Figure 6](image_url)

**Figure 6.** The weight fractions of Mo$_2$N and MoO$_3$ on the wear tracks as a fraction of testing temperatures.
As reported in Reference [26], MoO$_3$ consists of double layers of distorted edge-sharing MoO$_6$ octahedra parallel to (010) planes. Successive layers are held together by weak Van der Waals forces, and MoO$_3$ has low shear strength. Thus, MoO$_3$ could be worn away easily by the counterpart during the wear test.

Figure 7 shows SEM images of the wear track at different testing temperatures. As shown in Figure 7a, most of the wear track area is smooth, while some deep scratches were also detected on both sides after the wear test at 25 °C. A tiny protrusion appeared on the outside of the wear track surface; this could be induced by bombardment with high energy particles during deposition. The counterpart first came into contact with the protrusions, and then crushed some of them, forming debris during the wear test. The hard debris moved along the wear track with the counterpart and scratched the film surface. Some complex tribo-chemical reactions induced the formation of the tribo-film MoO$_3$, which could lubricate the wear track to some extent. Moreover, moisture also lubricates the wear track; J.K. Lancaster [16] reported that the absorption of moisture can reduce the average friction coefficient.

![Figure 7. SEM images of the wear tracks at various testing temperatures: (a) 25 °C, (b) 200 °C, (c) 400 °C and (d) 550 °C.](image)

Increasing the testing temperature to 200 °C, as shown in Figure 7b, induces more obvious scratches and cracks, indicating the strong interactions between the counterpart and the wear track surface; based on the results shown in Figures 5 and 6, the lowest weight fraction of MoO$_3$ might be attributable to such interactions. Besides this, increasing the testing temperature also reduces the absorption of moisture; the disappearance of moisture may also be attributed to the increase in the average friction coefficient. Similar results were also reported in some other nitride hard films [27,28].

As for the SEM image at 400 °C (Figure 7c), although cracks occurred on the wear track surface similar to those of 200 °C, the interactions between the counterpart and wear track became gentle; no obvious deep scratch was detected and a lot of wear debris adhered to the wear track surface. The MoO$_3$ tribo-film formed by the environmental temperature and friction heat could be considered the main lubricating phase. The increase of the weight fraction of MoO$_3$ may be attributed to a drop in the average friction coefficient and an increase in the wear rate at this period.

A further increase in testing temperature to 500 °C induced a smooth wear track surface, and a lot of wear debris appeared on it. Based on the results shown in Figures 5 and 6, the weight fraction reached a maximum value. Therefore, the wear mechanism changed to oxidation. The MoO$_3$ tribo-film could play a lubricating role during the wear test. Therefore, the average friction coefficient was 0.29, i.e., lower than that of any other testing temperature, and the wear rate reached a maximum value at 550 °C.
Table 1 illustrates the elemental compositions on the wear track at different testing temperatures. The aluminum content was also investigated to compare the counterpart adhesive on the wear track. The aluminum content, regardless of the testing temperatures, was 0 at.%, indicating that the counterpart did not adhere to the wear track. With an increase in the testing temperature, the oxygen content initially decreased and then decreased gradually, reaching a minimum value of ~3.2 at.% at 200 °C. However, both the molybdenum and nitrogen contents showed the opposite trend. This result is in good agreement with that of Figures 5 and 6.

| Testing Temperature (°C) | Elemental Compositions (at.%) |
|--------------------------|-------------------------------|
|                          | Mo | N   | O   | Al |
| 25                       | 54.6 ± 2.7 | 38.7 ± 1.9 | 6.7 ± 0.3 | 0 |
| 200                      | 56.7 ± 2.8 | 40.1 ± 2.0 | 3.2 ± 0.2 | 0 |
| 400                      | 53.8 ± 2.7 | 35.4 ± 1.9 | 10.8 ± 0.4 | 0 |
| 550                      | 42.2 ± 2.1 | 18.3 ± 0.9 | 39.5 ± 2.0 | 0 |

Based on above results, other factors may have given rise to the observed tribological properties at 25–200 °C, since the weight fraction of MoO$_3$ is influenced only slightly by the testing temperature. The only change for the wear test at 25–200°C was the relative humidity of the testing environment. In order to investigate the effect of relative humidity on the tribological properties at 25–200 °C, the wear tester system without sample was first heated to 200 °C and cooled to 25 °C. Then, the tribological properties of the Mo$_2$N film at 25 °C were investigated; the average friction coefficient and wear rate were shown to be 0.45 and 7.9 × 10$^{-7}$ mm$^3$/Nmm, respectively. Therefore, both the environmental relative humidity and the weight fraction of MoO$_3$ could be considered as influencing factors of the tribological properties at 25–200 °C. As for the tribological properties at >200°C, the high testing temperature led to the disappearance of moisture, and the environmental relative humidity was shown to have little effect.

The changes in the average friction coefficient and the wear rate at various testing temperatures can be explained as follows: when the testing temperature increased from 25 °C to 200 °C, decreasing the absorption of moisture and the weight fraction of MoO$_3$ led to an increase in the average friction coefficient and a decrease in the wear rate with an increase of the testing temperature; further increasing the testing temperature, moisture disappeared; as such, increasing the weight fraction led to a decrease in the average friction coefficient and an increase in the wear rate with an increase of the testing temperature. In summary, the weight fraction of lubricious layered MoO$_3$ may mainly be attributed to changes in the average friction coefficient and the wear rate.

4. Conclusions

Mo$_2$N films were deposited using RF unbalanced reactive magnetron sputtering and the tribological properties were investigated at different testing temperatures between 25 °C and 550 °C against an Al$_2$O$_3$ counterpart. The relative intensity ratio (RIR) method was used to calculate the weight fraction of the tribo-film (MoO$_3$) on the wear tracks of the films. The Mo$_2$N film showed tribological properties that were significantly temperature-dependent. When the testing temperature increased from 25 °C to 200 °C, decreasing the absorption of moisture and the weight fraction of MoO$_3$ led to an increase in the average friction coefficient and a decrease in the wear rate; with a further increase in the testing temperature, increasing the weight fraction of MoO$_3$ led to a decrease in the average friction coefficient and an increase in the wear rate. In summary, the weight fraction of the lubricious layered MoO$_3$ may mainly be attributed to changes in the average friction coefficient and the wear rate.
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