ASIATRIB 2002International Conference
JEJU ISLAND, KOREA

Tribological performance of a sputtered MoS_2 film having an oxidized surface layer

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An oxidized surface layer was intentionally formed on a sputtered MoS_2 film by introducing oxygen gas in the final stage of sputtering process. The film showed longer life than the normal Ar-sputtered film when the surface layer was slightly oxidized. A XPS analysis revealed co-existence of MoS_2 and MoO_3 in the surface layer, suggesting that the existence of some amount of oxides in the surface layer had beneficial effect. A confusing result was obtained: the life was much shorter than normal Ar-sputtered film when the film was exposed to O_2 environment for 1 minute after normal Ar-sputtering, although almost no oxide was detected in XPS analysis.

Keywords : MoS_2, Sputtered film, Surface oxide layer , Vacuum test

1. INTRODUCTION

Sputtered MoS_2 films were reported to show longer life when the operating environment contained a small amount of oxygen or water vapor [1,2]. The good tribological performance was attributed to stronger adhesion of a transfer film of MoS_2 to the counterpart surface, and the existence of oxide had a role to strengthen the adhesion. If this is the case, better tribological performance might be obtained when the surface layer is oxidized during film deposition process.

In this study, an oxidized surface layer was intentionally formed on a sputtered MoS_2 film by introducing oxygen gas in the final stage of sputtering process, and their tribological performance was evaluated in vacuum.

2. TEST SPECIMENS, TEST PROCEDURE ANDCONDITIONS

Sputtered MoS_2 films were deposited on a 440C stainless steel disk using a magnetron RF sputtering apparatus. The sputtering apparatus has two independent gas-supply systems, and thus the operating gas with an arbitrary mixed ratio of O_2/Ar can be introduced into the sputtering vacuum chamber.

Four oxidizing methods were employed in this study: (a) O_2 gas was introduced in the final stage of sputtering process. The time duration of O_2 introduction was changed from 0 to 3 minutes, and the mixture ratio of O_2/Ar was changed from 1% to 100%. (b) O_2 gas was introduced after normal Ar-gas sputtering process, and the film was exposed to O_2 gas environment for 1 minute. (c) O_2 plasma was activated after normal Ar-gas sputtering process, while the shutter in front of the specimen was closed. The shutter prevented the film from direct exposure to O_2 plasma. (d) Sputtering was carried out throughout in an O_2/Ar mixed gas with the O_2/Ar ratio of 1% and 10%. Only the surface layer will be oxidized in the methods of (a) (b) (c), while whole film will be oxidized in (d).

The film deposition conditions were RF power of 200 W, pressure during sputtering of 1.3 Pa, and the sputter duration of 30 minutes. Measured film thickness ranged from 500 to 700 nm.

Tribological performance was examined using a vacuum ball-on-disk friction tester. The counterpart specimen was 440C stainless steel ball with a diameter of 7.94 mm (5/16"). Rubbing conditions were a load of 10 N, a sliding speed of 0.5 m/s. The pressure during the tests was below 5×10^{-5} Pa. The life was defined as the time when the coefficient of friction exceeds 0.3.

3. RESULTS

Figure 1 shows the life of sputtered MoS_2 film against O_2 sputtering time in the last stage of sputtering process. When the O_2 sputtering time was 0.5 minute, life was almost the same as that of Ar-sputtered film. The life was increased to 1.5 times when the O_2 sputtering time was 1 minute, but it decreased rapidly as the time increased over 2 minute. Figure 2 shows the effect of O_2/Ar ratio, where O_2/Ar mixed gas was introduced for 1 minute in the last stage of sputtering process. The life decreased drastically when sputtered at an oxygen concentration of 1%, whereas the life gradually increased as the concentration increased.

Figure 3 shows the life of the films when the film was exposed to O_2 gas just after Ar sputtering. The pressure was 1.3 Pa, the same as during sputtering. The life drastically decreased to 1/50 by exposing O_2 gas for only 1 minute. However, when O_2 plasma was activated while the shutter in

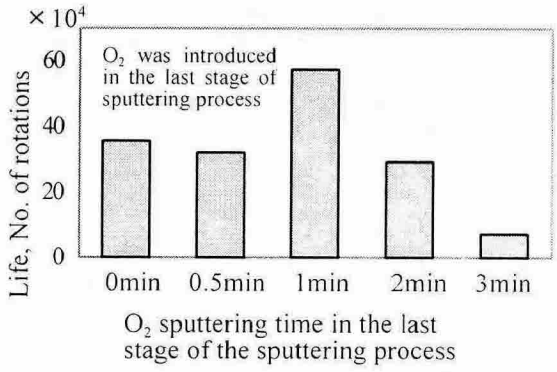


Fig.1 Life against O_2 sputtering time in the last stage of the sputtering process

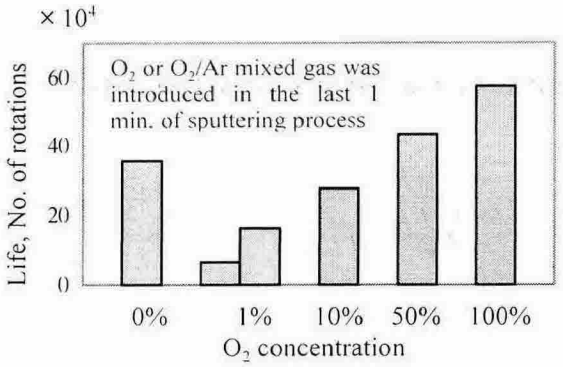


Fig.2 Life against O_2 concentration in the last 1 min. of sputtering

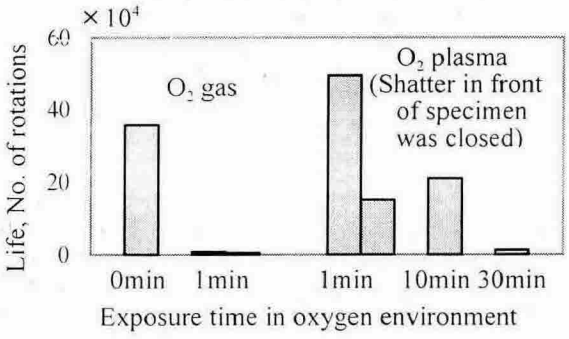


Fig.3 Life against exposure time in oxygen environment after sputtering

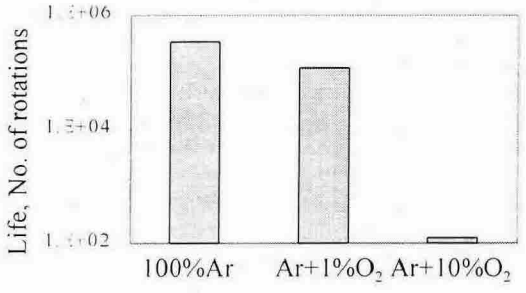
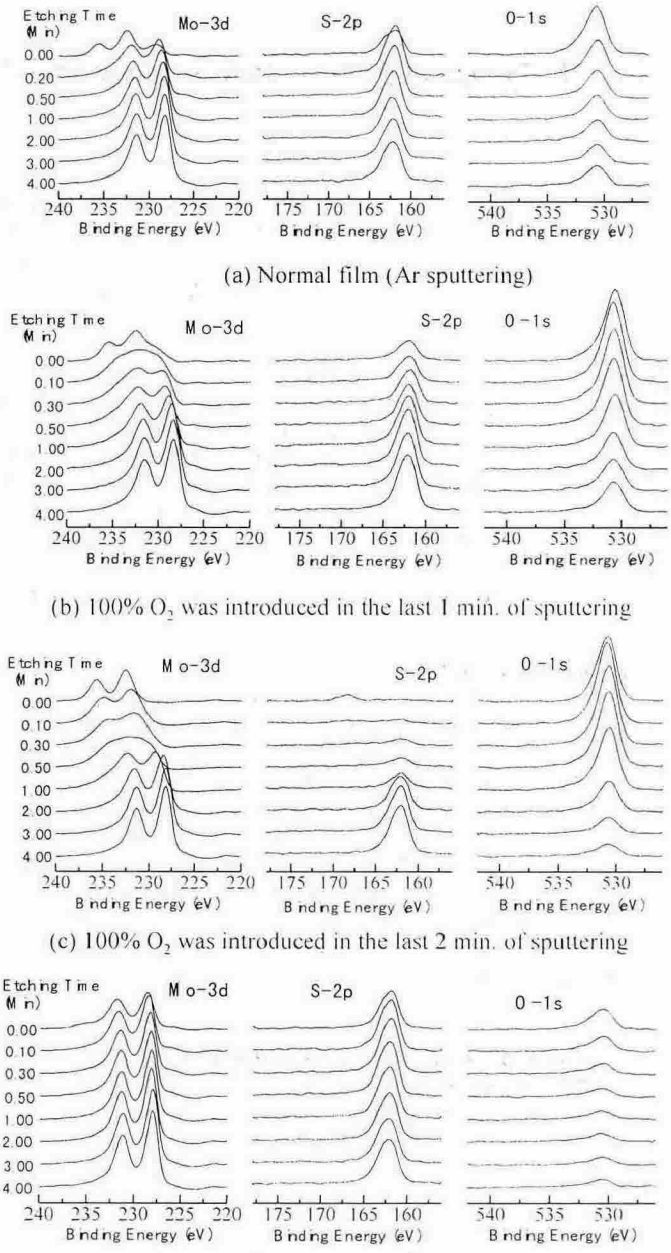


Fig.4 Life against O_2/Ar ratio during sputtering

front of the specimen was closed, exposure for 1 minute caused longer life, but further exposure resulted in shorter life.

Figure 4 shows the results for the films deposited using O_2/Ar mixed gas throughout the sputtering. Inclusion of only 1% oxygen into Ar resulted in shorter life of about 1/6. When oxygen concentration was increased to 10%, the film failed just after the start of the test. In the latter case, XPS analysis showed that no MoS_2 remained and the film was almost oxide.

Figure 5 shows XPS spectra for some films. Existence of MoO_3 was apparent for normal Ar-sputtered film until the etching time reached 0.5 minute, as shown in Fig.5 (a). When oxygen gas was introduced for 1 minute in the final stage of sputtering, Fig.5 (b), co-existence of MoS_2 and MoO_3 was confirmed until the etching time reached 2 minute. The etching rate was estimated to be about 3.5 nm/min from surface profile measurement. Thus the thickness of the oxide layer was estimated to be several atomic layers. When



(a) Normal film (Ar sputtering)

(b) 100% O_2 was introduced in the last 1 min. of sputtering

(c) 100% O_2 was introduced in the last 2 min. of sputtering

(d) Exposed to O_2 environment for 1 min. after sputtering

Fig.5 XPS spectra for several films

the O_2 sputtering time was increased to 2 minute. Fig.5 (c), there existed no S in the surface layer, and thus the surface layer seemed to consist of only oxide. This may be the cause of shorter life.

Fig 5(d) shows XPS spectra of the film exposed to O_2 gas for 1 minute after normal Ar sputtering. This film showed very short life as shown in Fig.3. Compared with the Ar-sputtered film, Fig5 (a), oxygen amount was rather lower. It is not clear why this film showed short life.

4. REFERENCES

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