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Influence of Deposition Conditions on the Adhesion of Sputter-deposited MoS_2-Ti Films

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Abstract

MoS_2-Ti films were deposited on SKD-11 tool steel substrate by a D.C. magnetron sputtering system. The influence of deposition parameters on the adhesion of the films was investigated by the scratch test. Cross-section morphology was evaluated using FE-SEM. The plasma etching played an important role on the adhesion of the films. The appropriate etching conditions roughened the surface, resulting in the improved adhesion of the film. The adhesion of the film increased with the interlayer thickness up to 110 nm and then decreased slightly with further increasing of interlayer thickness. The adhesion was highest at a bias voltage of −50 V. Further increase of the bias voltage decreased the film adhesion.

Keywords : MoS_2-Ti film, D.C. magnetron sputtering, Adhesion, Hardness

INTRODUCTION

Coatings of sputter-deposited molybdenum disulfide have been used in high vacuum and aerospace applications for lubrication purposes where low friction is desirable. However, when MoS_2 is exposed to H_2O/O_2 atmosphere, MoS_2 oxidizes to MoS_3 which shortens its life time[1,2]. Recently, Kim *et al.*[3] and other researchers[4,5] reported that small addition of titanium to MoS_2 based matrix not only prevent the oxidation of MoS_2 but also improve the adhesion of the film.

The critical load determined by a scratch tester represents the adhesion between the film and the substrate and is very important mechanical parameter for the thin film[6,7]. Critical load is sensitive to the deposition parameters including etching parameters, the interlayer and the substrate bias voltage. Much work has been done on the performance (friction and wear properties) of the MoS_2-Ti films[3-5]. However, little works have been published on the influence of deposition conditions, especially the effect of the interlayer thickness and the plasma etching parameter on the adhesion of MoS_2-Ti films. Additionally, the substrate bias voltage plays a very important role on mechanical properties of the MoS_2-Ti film such as hardness. However, few studies have been performed on the relationship between the hardness and the bias voltage of the MoS_2-Ti film. The main objective of this work was to study the influence of plasma etching parameters, the interlayer thickness and the substrate bias voltage on the adhesion of the MoS_2-Ti film deposited on SKD-11 tool steel by D.C. magnetron sputtering. The effect of bias voltage on the films' hardness was also discussed.

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EXPERIMENTAL PROCEDURE

The MoS_2-Ti films were deposited on SKD-11 tool steel and Si wafer substrates by an unbalanced D.C. magnetron sputtering system. The sputtering system contained two facing magnetrons on the lateral face of the chamber. Titanium target (99.995% pure) and MoS_2 target (99%) with diameter of 75 mm were mounted on each magnetron sputter source. The sample holder, which could rotate and apply bias voltage, was located at the center of the chamber. The distance between the sample holder to target was about 60 mm.

After the chamber was evacuated to 1.3×10^{-3} Pa, argon was introduced to maintain the working pressure of 0.3 Pa. Before deposition, the substrates were cleaned for 5~20 min by argon plasma. The etching power and the etching time were varied in the range of 0~250 W and 0~20 min, respectively. The titanium target was then sputtered to deposit an interlayer. Afterward, the MoS_2 and titanium target were sputtered simultaneously to form the composite MoS_2-Ti film by rotating the sample holder between two targets. The rotation speed was 5 rpm. Co-sputtering time was fixed at 30 min to maintain MoS_2-Ti film with thickness of 1.3~1.5 μm. Temperature measured by a thermocouple located close to the sample during the deposition process was less than 60°C.

The adhesion of the film was evaluated by a commercially available scratch testing equipment (Revetest, CSEM, Switzerland), under the standard condition. The diameter of the diamond tip was 200 μm. The scratch speed and load speed were 10 mm/min and 10 N/min, respectively. From the signal of acoustic emission vs. applied load, the critical load was determined at the load of abrupt change of acoustic emission. Optical microscopy examination of coatings after scratch test was used to confirm this result.

The cross-section micrograph was determined by FE-SEM (JEOL, JSM-6500F). Hardness of the MoS_2 film was measured by Vicker's hardness tester (MVK-H1). A load of 10 g was used for Vicker's hardness tests.

RESULT AND DISCUSSION

The sputtering power on MoS_2 and titanium target was 240 and 110 W, respectively, during co-sputtering. The titanium and MoS_2 deposition rates determined by FE-SEM were 8 nm/min and 33 nm/min, respectively. Titanium content determined by EPMA was about 16 at. % in the MoS_2-Ti composite film.

Fig. 1(a) shows the effect of the etching power on the adhesion of MoS_2-Ti films. The etching power was varied from 0 to 250 W, while the etching time and the substrate bias voltage was fixed at 10 min and –50 V, respectively. Without etching, the critical load was 14 N. The critical load increased with etching power and reached a maximum of 21 N at the etching power of 150 W. Further increase of etching power to 250 W decreased the critical load remarkably to 11 N. Fig. 1(b) shows the effect of the etching time on the adhesion of MoS_2-Ti films. The etching time was varied from 0 to 20 min with the etching power fixed at 150 W. The critical load reached a maximum of 21 N at the etching time of 10 min. Subsequent experiments were carried out with this etching time.

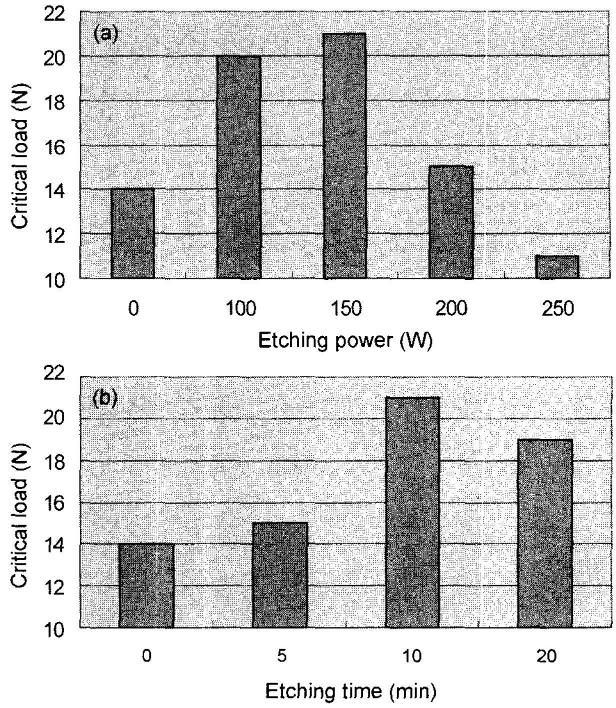

Fig. 1. Effect of etching condition on the critical load (Lc) of MoS-Ti films (a) etching power (b) etching time.

Fig. 2 shows the morphology of etched surface. The etching time was fixed at 10 min, and the etching power was varied from 100 to 200 W for Fig. 2(a), (b) and (c). Fig. 2(a) shows that less etching was done on the surface with the etching power of 100 W. The etched surface became rough with the etching power of 150 W (Fig. 2(b)) resulting in the good adhesion of the film. With the increase of the etching power to 200 W, the surface was non-uniform (Fig. 2(c)), which decreased the adhesion of MoS_2-Ti film.

The etching power was fixed at 150 W and the etching time was varied from 5 to 20 min. When the etching time was 5 min, the surface was less etched (not shown). The increase of the etching time to 10 min roughened the surface (Fig. 2(b)). Further increase of the etching time to 20 min made the etched surface non-uniform. Therefore, the MoS_2-Ti film deposited with 5 and 20 min etching, the adhesion was lower than that of the 10 min etching.

Titanium was used as an interlayer to improve the adhesion between the film and the substrate when depositing the MoS_2-Ti film. Fig. 3 shows the influence

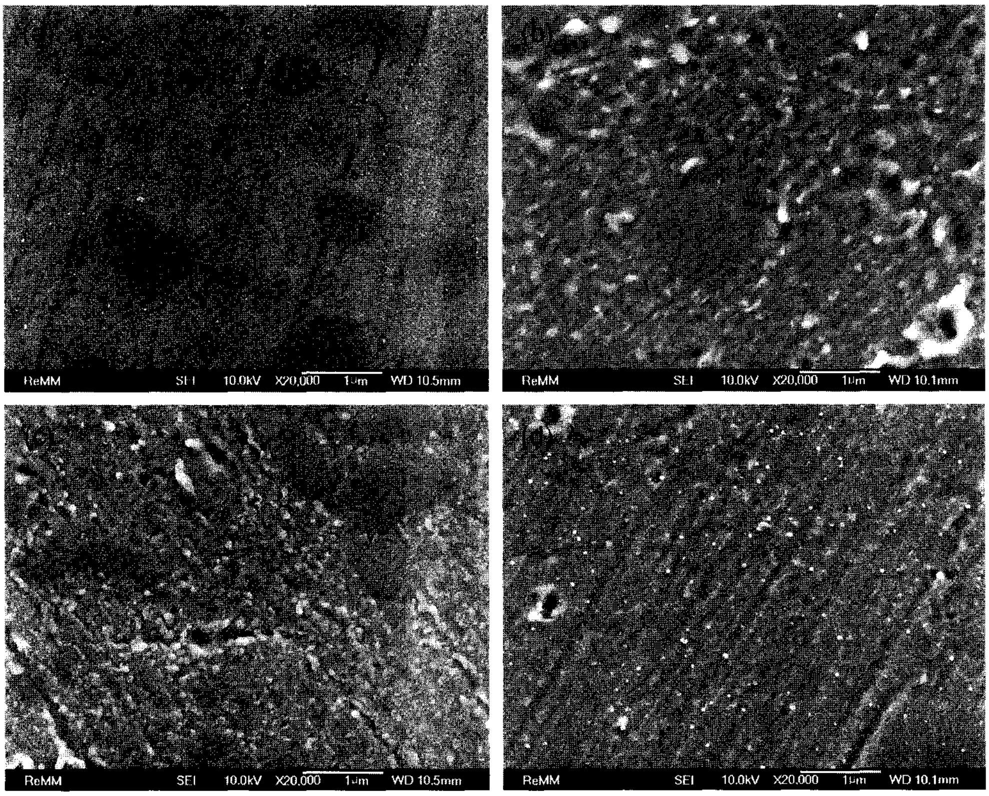

Fig. 2. FE-SEM micrograph of the morphology of etched surface of SKD 11 substrate (a) 100 W, 10 min (b) 150 W, 10 min (c) 200 W, 10 min (d) 150 W, 20 min.

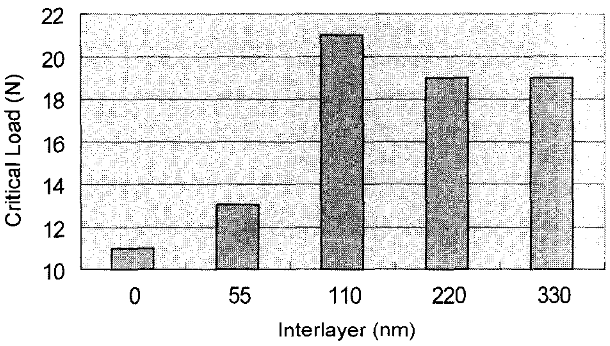

Fig. 3. Effect of interlayer on the critical load (Lc) of MoS_2-Ti films.

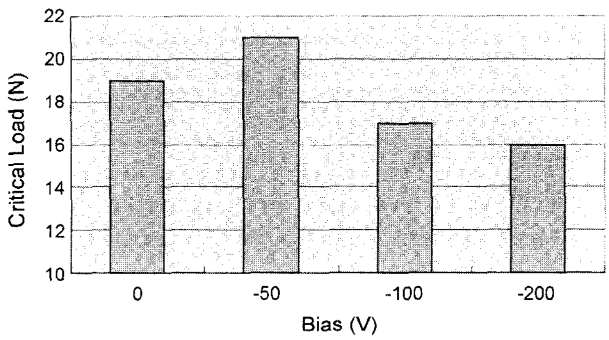

Fig. 4. Effect of bias voltage on the critical load (Lc) of MoS-Ti films.

of interlayer thickness on the adhesion of MoS_2-Ti films. The films were deposited with the etching power of 150 W, the etching time of 10 min and the substrate bias voltage of −50 V. The critical load of the film was 11 N without an interlayer. The critical load was highest (21 N) with an interlayer of 110 nm. Further increase of the interlayer decreased the critical load slightly. The optimum thickness of interlayer for the good adhesion of the film was about 100 nm. Less than 100 nm, the interlayer may not completely cover the substrate, while too thick interlayer can be easy deformed by mechanical load during the scratch test. Similar results were obtained by Renevier *et al.*[4,5].

The plasma etching condition was fixed at etching power of 150 W and etching time of 10 min, respectively. Fig. 4 shows the effect of the substrate bias voltage on the adhesion of MoS_2-Ti films. The critical load slightly increased with change of the substrate bias voltage from 0 to −50 V. Further increase of the substrate bias voltage decreased the critical load. Fig. 5 shows FE-SEM cross-section micrograph of MoS_2-Ti film deposited at bias voltage of −50 V. The MoS_2-Ti film was non-columnar and there was no separate

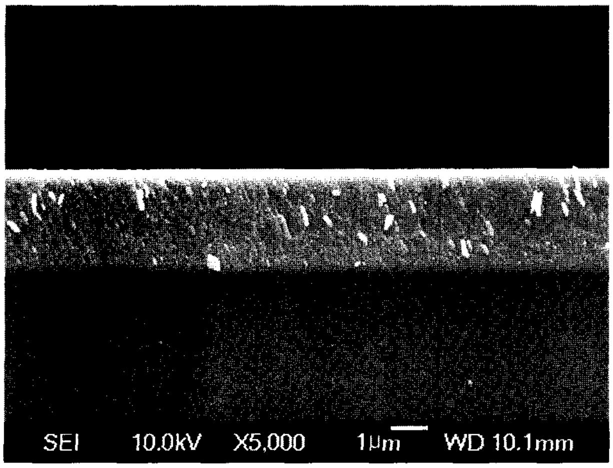

Fig. 5. FE-SEM micrograph of cross-section (2.7 mm after 1 hr. sputtering).

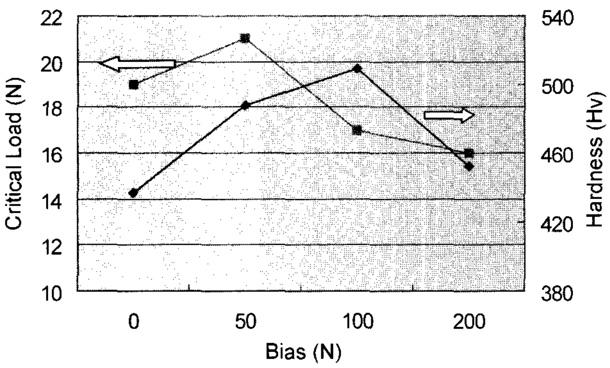

Fig. 6. Hardness and critical load of MoS_2-Ti film as a function of substrate bias voltage.

titanium and MoS_2 layers in the film, which indicated the MoS_2-Ti film grew homogeneously with Ti and MoS_2 during sputtering. At high bias voltage, the ion bombardment is strong and the film stress becomes high, resulting in low critical load.

The relationship between the hardness and the substrate bias voltage is shown in Fig. 6. Without a substrate bias voltage, the hardness of the film was 437 $Hv_{0.01}$ and increased with the substrate bias voltage. The hardness value was highest (509 $Hv_{0.01}$) at the substrate bias voltage of −100 V, and then decreased to 452 $Hv_{0.01}$ at the substrate bias voltage of −200 V. The film hardness is largely influenced by the residual stress. Without the substrate bias voltage, the residual stress of the film is low because the energies of ions and atoms bombarding on the growing film are very low. As the substrate bias increases to −50 and −100 V, the increase of film hardness is thought to be due to the densification of the film and increase of the residual stress by ions and atoms bombardment. However, at bias voltage of −200 V, the film was damaged by severe ion bombardment, resulting in the decrease of film hardness. Similar results were obtained in the deposition of TiN films[8] and NbN[9] films.

CONCLUSION

MoS_2-Ti thin films were deposited by a D.C. magnetron sputtering method. The influence of deposition parameters on the films adhesion was studied by scratch testing. The plasma etching parameters including the etching power and the etching time played an important role on the film adhesion. The optimized etching parameters were 150 W of the etching power and 10 min of the etching time. The Ti interlayer thickness and the substrate bias voltage influenced the films adhesion. The optimum thickness of the interlayer was about 110 nm. A bias voltage of −50 V improved the film adhesion. The hardness of the film was varied with the substrate bias voltage. The film hardness was highest with a substrate bias voltage of −100 V.

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