ASIATRIB 2002International Conference
JEJU ISLAND, KOREA

Deposition and evaluation of MoNx films deposited by magnetron sputtering

Yajun Ma[1], Shenghua Li[1], Yuansheng Jin[1], Guoshun Pan[1]
Yucong Wang[2] and Simon C. Tung[2]

[1]*State Key Laboratory of Tribology, Tsinghua Univeristy, Beijing 100084, China*
[2]*Gernal Motors Corporation, Detroit 48090-9055, USA*

Molybdenum Nitrided (MoNx) films were deposited by DC planar magnetron sputtering. Silicon wafers and real nitrided stainless steel piston rings are employed as substrates. 12 different combinations of nitrogen and argon partial pressure, from 1:7 to 7:1, were applied to deposit MoNx films. X-ray diffraction (XRD) was used to determine the phase structures of films. When nitrogen vs. argon partial pressure is 1:7, the film is mainly Mo_2N phase. With increase of nitrogen partial pressure, MoN phase emerges, but Mo_2N phase still exists. Composition analysis with atomic emission spectrometry (AES) also agreed with this. The films have very high nanohardness (max 2400Hv) and good adhesion to the substrates.

Keywords : Molybdenum nitride, Film, Magnetron sputtering

1. INTRODUCTION

Molybdenum Nitrided (MoNx) films were deposited by DC planar magnetron sputtering. The sputtering target is 371x71x3 rectangular molybdenum board, whose purity is 99.9%. Silicon wafers and real nitrided stainless steel piston rings were employed as substrates. The substrates were ultrasonically cleaned in acetone and ethanol. The base vacuum pressure was 5.7x10-3Pa or less ensured by diffusion pump. The circumstance temperature is controlled at 230℃.

Before deposition, substrates were bombarded and sputter etched with Ar+ for 10 min and 5 min in sequence. MoNx films were deposited at different partial pressure ratio of nitrogen and argon. And the total pressure was kept at about 2.5Pa by adjusting the argon pressure. During deposition, the target power was 420W and the bombarding voltage was 160V.

Structure of deposited films was investigated with Leitz Wetzlar optical microscope and Opton CSM950 scanning electro microscope (SEM). Dmax-RB X-ray diffractometry (XRD) using Cu Kα radiation was employed to determine phase structure of films. The composition of deposited films was analyzed with PHI680 Auger electron spectroscopy (AES). The nano-hardness of films was measured with CSEM nanoindenter. Adhesion of films was judged with scratch test carried out on MTS XP nanoindenter.

2. RESULTS AND DISCUSSION

The thickness of deposited films is about 4 μm and they have dense structure. Figure 1 and figure 2 are respectively optical microscope and SEM images of MoNx film.

X-ray diffraction indicates that when nitrogen vs. argon partial pressure is 1:7, the film is mainly Mo_2N phase. With increase of nitrogen partial pressure, MoN phase emerges, but Mo_2N phase still exists. Even under the greatest nitrogen partial pressure (partial pressure ratio of nitrogen vs. argon is 7:1), no pure MoN film was found.

From analysis results of AES, with partial pressure ratio of nitrogen and argon changed from 1:7 to 7:1, the Atomic ratio of Mo and N descends, as shown in figure3. It indicates that MoN phase contents increased as nitrogen partial pressure increasing.

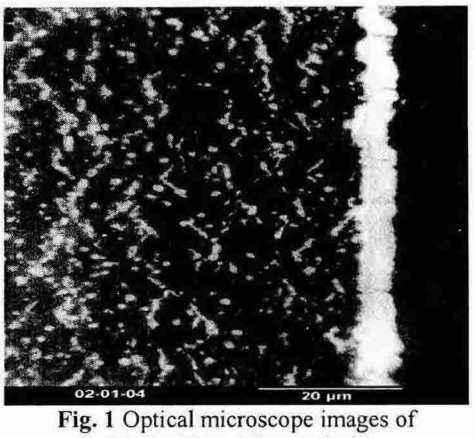

Fig. 1 Optical microscope images of MoNx films (after etched)

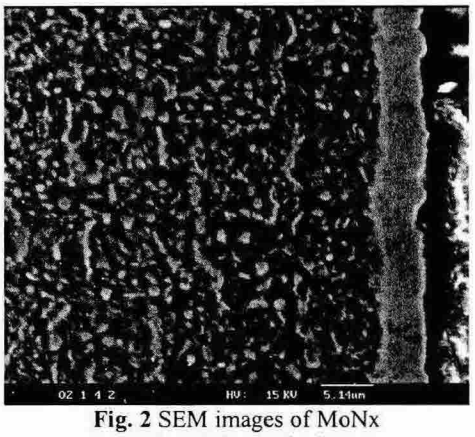

Fig. 2 SEM images of MoNx films (after etched)

The hardness of films correlates with the Mo/N atomic ratio. As shown figure 4, the nanohardness of films descend with increase of Mo/N atomic ratio.

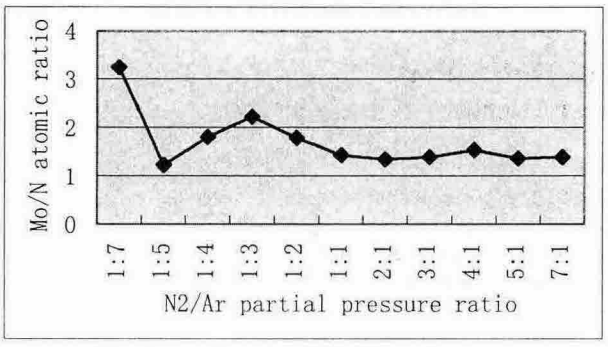

Fig. 3 Atomic ratio of Mo/N as a function of N_2/Ar partial pressure ratio

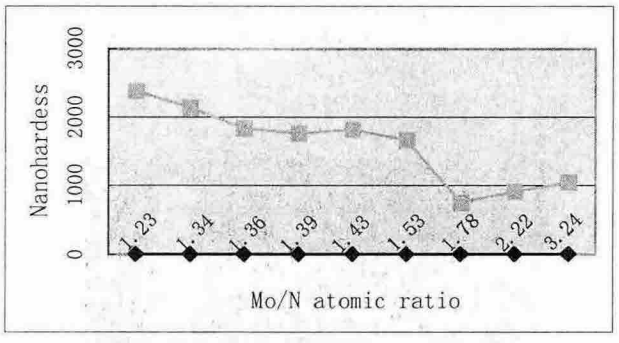

Fig. 4 Nanoharness of films as a function of Mo/N atomic ratio

The adhesion of films was judged by scratch tests. The maximum scratch load is 150 mN. The scratch length is 500μm and the scratch velocity is 10 μm/s. The results indicate the films have good adhesion to substrates and the adhesion of films decrease with increase of used nitrogen partial pressure. The film deposited at nitrogen vs. argon partial pressure ratio of 1:4 almost did not encounter flaking. The critical loads of film deposited at nitrogen vs. argon partial pressure ratio of 1:1 and 4:1 are respectively 118mN and 56mN.

7. REFERENCES

[1] E.P. Donovan, G.K. Hubler, M.S. Mudholkar and L.T. Thompson, "Iom-beam-assisted deposition of molybdenum nitride films," Surface and Coatings Technology, Vol.66, pp. 499-504, 1994

[2] W.D. SproulE, "High-rate reactive DC magnetron sputtering of oxide and nitride superlattice coatings," Vacuum, Vol.51, No.4, pp. 641-646, 1998

[13] I. Safi, "Recent aspects concerning DC reactive magnetron sputtering of thin films: a review Surface and Coatings Technology, Vol.127, pp. 203-219, 2000