Effects of oxide layer formed on TiN coated silicon wafer on the friction characteristics

C. W. CHO [1] and Y. Z. LEE [2]

[1] *Graduate School of Mechanical Engineering, SungKyunKwan University*
300 ChunChun-Dong, JangAn-Ku, Suwon 440-746, KOREA
[2] *School of Mechanical Engineering, SungKyunKwan University*
300 ChunChun-Dong, JangAn-Ku, Suwon 440-746, KOREA

In this study, the effects of oxide layer formed on the wear tracks of TiN coated silicon wafer on friction characteristics were investigated. Silicon wafer was used for the substrate of coated disk specimens, which were prepared by depositing TiN coating with 1μm in coating thickness. AISI 52100 steel balls were used for the counterpart. The tests were performed both in air for forming oxide layer on the wear track and in nitrogen to avoid oxidation. This paper reports characterization of the oxide layer effects on friction characteristics using X-ray diffraction (XRD), scanning electron microscopy (SEM) and friction force microscope (FFM).

Keywords : Oxide layer, TiN coated silicon wafer, X-ray Diffraction analysis, FFM, Frictional force image

1. INTRODUCTION

Ceramic coatings can increase the life of machine elements due to their outstanding low friction characteristics and good wear resistance [1]. Therefore, ceramic coatings such as TiN (titanium nitride), CrN (chromium nitride) and DLC (diamond like carbon) are generally being applied to many machine elements including machine tools, bearings and shafts [1-2]. A problem that arises when such coatings are applied to a part which is counter acting with other material in air, is that friction and wear characteristics of the coating change according to characteristics of the oxide layer, which forms due to oxidative wear. When ceramic coatings are in contact with steel, it usually shows outstanding characteristics of low friction and wear resistance initially but as sliding motion continues, an oxide layer forms on sliding surfaces of both coating and steel due to wear particles from steel. In this case, the low friction characteristic of the coating may disappear with the formation of an oxide layer and the friction force between two materials increase causing wear of the steel to increase as contact condition changes from steel on coating to steel on oxide layer, oxide layer on coating or oxide layer on oxide layer.

In this study, the effect of oxide layer on the friction and wear characteristics of TiN coated silicon wafer and steel ball were investigated during sliding test. Sliding tests were conducted in both air and nitrogen environments to study the role of oxide layer in sliding motion as well as to investigate the effect of oxide layer on the ceramic coating.

2. EXPERIMENTAL DETAILS

2.1 Material and test conditions

A silicon substrate was used for the substrate of coated disk specimen and its thickness was 750μm. The silicon substrate is p-type and <100> oriented, whose surface hardness was 12.4GPa measured by nano-hardness measurement and surface roughness was Ra 0.0002μm. The coated silicon specimens were prepared by depositing TiN coating with 1μm thick of the coating using arc ion plating method. The surface hardness was 27.4GPa. For the upper specimens, AISI 52100 steel ball was used and its diameter was 10mm.

A sliding speed of 0.03m/sec (60rpm) and contact load of 1.0N were applied in all tests.

2.2 X-ray diffraction (XRD) analysis

X-ray diffraction (XRD) analysis was carried out on the wear tracks of the TiN coated silicon wafer tested in air and nitrogen, using *Bruker AXS D8 Discover X-ray diffractometer* with Cu-Kα radiation. The diffraction patterns were acquired in a two-theta angle range of 20° -80° at a scan speed of 4° /min.

2.3 Friction force microscope (FFM)

Surface topography of the wear tracks tested in air and nitrogen was characterized by AFM under vacuum conditions, using *Seiko SPA-300HV* system. All imaging was performed in contact mode using *Si-AF01* tips. Frictional force imaging was performed simultaneously with topographical imaging by scanning the tip in the direction orthogonal to the long cantilever axis. The constant scan rate is 1Hz.

3. RESULTS AND DISCUSSION

3.1 Friction and wear characteristic of uncoated and the TiN coated silicon wafer under sliding test

To investigate the friction and wear characteristic of the uncoated and the TiN coated silicon wafer, the changes in COF signal was measured in sliding motion against the steel balls in air and nitrogen environment. As shown in Fig. 1, typical dry friction behaviors, the sudden transition and the high friction were observed as the contact number of cycle increased for all test specimens and test environments. However, all friction signals showed constant values after 400 contact numbers of cycles. Under each test conditions, five sliding tests were carried out. The COF values were measured in the region where the values were stable and then they were averaged and those0 from the sliding tests with the TiN coated silicon wafer and the steel ball in air were larger than those in nitrogen. This is believed to be due to contact condition changes from steel on coating to steel on oxide layer, oxide layer on coating or oxide layer on oxide layer. Thus, it can be said that the oxide layer determines the high friction characteristics rather than the low friction characteristic of TiN coating.

The SEM micrographs of the wear tracks generated on the TiN coated silicon wafer tested up to 1800 cycles in nitrogen and in air are shown in Fig. 2 (a) and (b), respectively. The wear track generated from the test in nitrogen shows more severe abrasive wear than that generated during the test in air. As shown in Fig. 2 (b), oxide layers were formed due to severe adhesive wear.

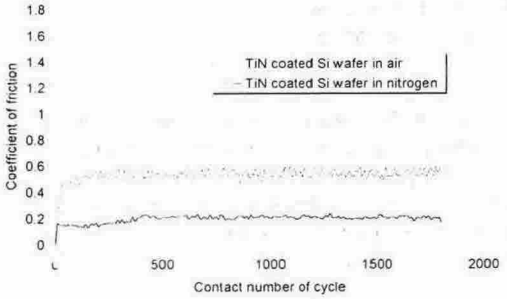
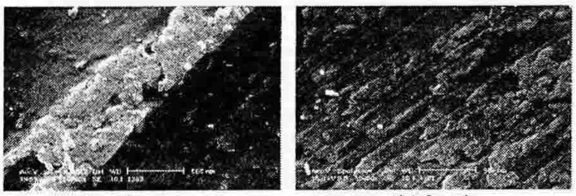


Fig. 1 Coefficient of friction signal from the sliding tests in various test conditions



(a) In nitrogen (b) In air

Fig. 2 SEM micrographs of the wear tracks on TiN coated silicon wafer generated during the sliding tests in various environments up to 1800 contact number of cycles

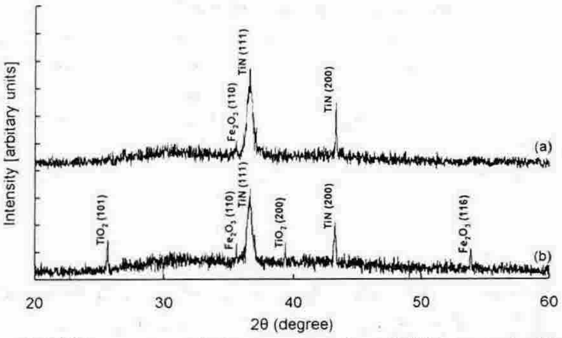
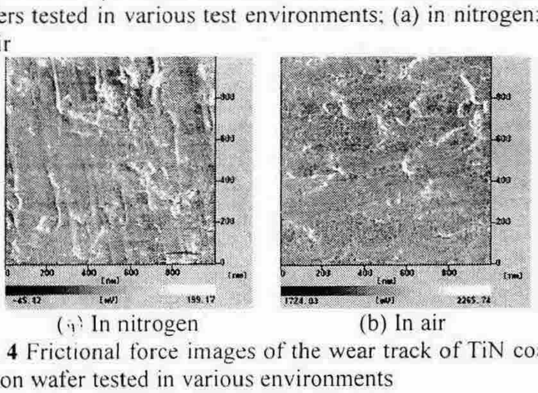


Fig. 3 XRD spectra of the wear tracks of TiN coated silicon wafers tested in various test environments; (a) in nitrogen; (b) in air



(a) In nitrogen (b) In air

Fig. 4 Frictional force images of the wear track of TiN coated silicon wafer tested in various environments

3.2 XRD for crystal phase analysis of the wear tracks tested in nitrogen and air

The structures of the wear tracks on the TiN coated silicon wafer tested in nitrogen and air were determined by XRD using Cu-Kα excitation line as shown in Fig. 3. The lattice parameters are compared to the powder diffraction database. Apart from the peaks ascribed to TiN present in the coating material, TiO_2 and Fe_2O_3 were identified on the wear track of the TiN coated silicon wafer tested in air as shown in Fig. 3 (b) but not on the wear tack in nitrogen as shown in Fig. 3 (a).

Most of related research results reported that titanium oxide (TiO_2) and iron oxide (Fe_2O_3) formed on the surfaces of two counter-faces or between the contacting surfaces can reduce the friction coefficient. It has been reported that the deposition of a soft thin coating such as titanium oxide and iron oxide on a hard substrate will reduce both the contact area and the shear strength at the contact, and thus result in very favorable tribological conditions with low friction [3-4]. However, hard coatings such as TiN in sliding with a steel ball have intrinsic frictional characteristics of low friction. Thus, the TiN coated surface with the titanium oxide and iron oxide shows relatively high frictional characteristics compared to that without theses oxide layers.

3.3 FFM results of the wear track

AFM surface topography and frictional force images of the wear tracks of the TiN coated silicon wafer tested up to 1800 cycles in nitrogen and air are shown in Fig. 4 (a) and (b), respectively. The frictional force images, which were generated by interactions between the silicon tip and the surface of wear tracks, as shown in Fig. 4 (a) and (b) do not show the exact values of the frictional force but the relative frictional force. As shown in these figures, the friction force of the wear track with oxide layer was several ten times greater than that of the wear track without oxide layer.

4. CONCLUSION

The effect of the oxide layer on the friction characteristics of the TiN coated silicon wafer and steel ball were investigated during sliding test, the following conclusions were obtained.

(1) The TiN coated surface with the titanium oxide (TiO_2) and iron oxide (Fe_2O_3) shows relatively high frictional characteristics compared to that without the oxide layers from the macro- and micro-friction point of views.

(2) The oxide layer formed on the wear tracks of the TiN coated silicon wafer in air protects the base material from wear.

5. REFERENCES

[1] Holmberg, K., Mattews, A., "Coatings Tribology", Elsevier, pp. 172-189, 1994.

[2] Bhushan, B., Gupta, B. K., "Handbook of Tribology", McGraw-Hill, pp. 9.1-9.121, 1991.

[3] Fu, Y., Zhu, X., Xu, K., and He, J., Journal of Materials Processing Technology, Vol. 83, pp. 209-216, 1998.

[4] Zhou, Y. M., Asaki, R., Higashi, K., Soe, W. H., and Yamamoto, R., Surface and Coatings Technology, Vol. 130, pp. 9-14, 2000.

ACKNOWLEGEMENTS

The authors are grateful for the support provided by a grant from the Safety and Structural Integrity Research Center at SungKyunKwan University.