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Assessment of adhesion between thin film and silicon based on a scratch test†

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Abstract

Thin film coatings are commonly utilized to prevent wear, modify surface properties, and manipulate the frictional behavior of various mechanical systems. The behavior of a coating has a direct effect on the life as well as performance of the system. However, the coating itself is subject to damage, and the quality of the coating is related to the adhesion characteristics between the coating and the substrate. Therefore, a quantitative assessment of the adhesion properties of thin film is important to guarantee the reliability of not only the thin film but also the mechanical system. In this study, ramp loading scratch tests were performed to assess the adhesion characteristics of Ag and ZnO thin films coated on a silicon wafer. Silver thin film, deposited by sputtering, and ZnO thin film, fabricated by a sol-gel method, were used as scratch specimens. Scratch tests using a diamond tip were performed with a continuously increasing normal force. During the scratch test, the normal and frictional forces were monitored to assess the integrity of the film. The Benjamin and Weaver model commonly used for obtaining the horizontal force during the scratching of films coated on a substrate showed large discrepancies with the experimental results. In this work, the model was modified with a plowing term to minimize the difference between the experimental and theoretical results. Using the modified model, the experimental results could be predicted with an accuracy of about 10%.

Keywords: Adhesion; Benjamin and Weaver model; Plowing model; Scratch test; Thin film

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1. Introduction

With the development of various current and future precision technologies, the demand for a higher durability of mechanical components has been increasing steadily. Particularly for systems that experience contact against a counter surface or object, tribological issues related to surface wear and friction become important. Also, in miniature devices where tight physical tolerance and dimensional accuracy affect the efficiency of system operation, wear that occurs at contacting surfaces ultimately dictates the life of the system.

In order to tackle the problems that arise due to wear, surface coatings have been extensively utilized in various tribological applications. Coatings have been used to change the physical and chemical characteristics of the surface and improve the durability of the system without changing the bulk material [1-3]. Especially for high precision systems, such as data storage systems, semi-conductor devices, machining tools, bearings, gears, space application devices, bio components, and MEMS, applications of coatings have been increasing gradually [4, 5]. There are a variety of coatings used in tribological applications. For example, carbon-based coatings such as diamond-like carbon (DLC) films have been applied to improve the tribological properties of tools, optical elements, moving mechanical components in MEMS, and other micro-scale systems [6-9].

The properties of coatings are not only affected by their material but also by their thickness. Thin film coatings may have a thickness in the range of several nanometers to tens of micrometers. The adhesion between the thin film and the substrate significantly influences the durability of the thin film [10]. Therefore, characteristics and measurement methods related to the adhesion between the thin film and substrate have been extensively investigated in the past. One method to evaluate the adhesion of film is the tape peeling method, where tape is adhered to the coating surface and then peeled off. By noting the degree of film that has been removed by the tape, the adhesion of the film can be assessed. Although this method is simple and useful for industrial applications, quantitative measures of film adhesion cannot be readily derived from the results. Another method is the abrasion test method, where an abrasive tip is used to scratch the film surface until the substrate is exposed. Although this method is adequate to evaluate the abrasive resistance of the film, it does not provide direct information regarding the adhesion of the film. In this study, the adhesion of Ag and ZnO thin films deposited on silicon was investigated by the ramp loading scratch test. The experimental results were used to analyze film adhesion based

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on a modified Benjamin and Weaver model [10].

2. Experimental details

2.1 Preparation of specimens

Ag and ZnO films were deposited on a silicon wafer to investigate the adhesion characteristics between the film and the substrate. An Ag thin film was deposited on a silicon wafer using the DC-magnetron sputtering method. As shown in Fig. 1, the thickness of the Ag film, which was measured by an Atomic Force Microscope (AFM), was about 130 nm. The ZnO thin films were deposited by the sol-gel method, and the thickness was about 280 nm. The ZnO films were annealed at temperatures of 450, 550, and 800 ℃ in order to vary the hardness of the films.

2.2 Experimental set-up and conditions

Ramp loading scratch tests were performed using a custom-built micro-scratch tester as shown in Fig. 2. The tester consisted of a precision stage, linear actuators, diamond tip for scratching the surface, and load cells to measure the friction and normal forces during scratching. The motion of the tip was controlled by three linear actuators with a resolution of 60 nm and a maximum stroke of 25 mm. X and Y direction stages were used to generate the lateral motion to create a relative motion between the tip and the specimen. Normal force between the tip and the specimen was applied by moving the Z direction stage. The normal force was monitored by a precision scale located at the bottom of the specimen. The normal force was gradually increased during the scratch test to generate a ramp loading condition. The frictional force was also monitored during the scratch test. For this purpose, a load cell with a resolution of 0.01 mN was used. A conical-shaped tip made of diamond was used for scratching the specimen surface. The high hardness and superior durability of the diamond assured uniform tip geometry during the entire experiment. Also, the diamond tip could readily penetrate the film surface and create a clear wear track from which the adhesion properties of the film could be assessed. The radius of the tip was 5 μm, and the sliding velocity of the tip was set to 2.5 μm/s. The experimental conditions are summarized in Table 1.

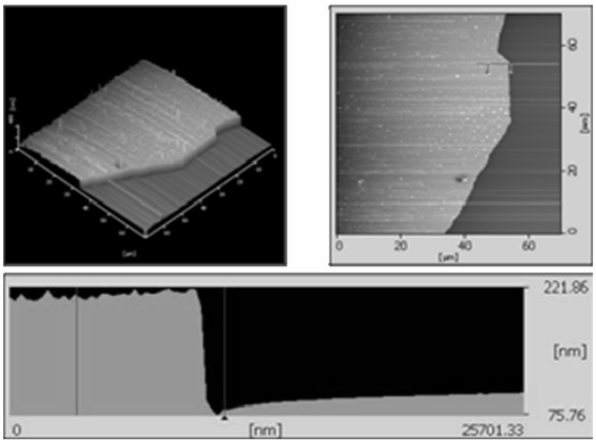


Fig. 1. AFM image of Ag thin film deposited on a silicon wafer.

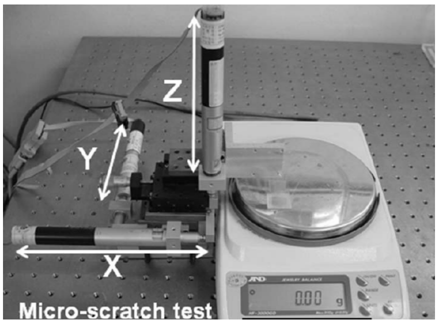


Fig. 2. Photograph of the custom-built micro-scratch tester.

3. Experimental results

Wear tracks created by the ramp loading scratch test were measured by an optical microscope and AFM. The depth and width of the wear track with respect to the normal force and sliding distance were measured. Moreover, the frictional force and normal force during the scratch test were also obtained. Fig. 3 shows the graph of a typical normal force variation with respect to sliding distance. The depths of the wear tracks measured by AFM for both Ag and ZnO films are shown in Figs. 4 and 5. From the results, it was clear that the width and depth of the wear track increased with sliding distance, as expected. For the Ag film, when the normal force exceeded 0.5 gf, the depth of the wear track was approximately 130 nm, which was the thickness of the Ag thin film. Therefore, the normal force of 0.5 gf could be considered as the “critical force” at which the film is delaminated from the silicon substrate. Tables 2 and 3 list the results of the scratch tests, including the frictional forces of the Ag and ZnO films. In the case of the ZnO film, the critical force increased as the annealing temperature increased.

Table 1. Experimental conditions for the ramp loading scratch test.

	Ag	ZnO
Normal force	0~10 gf	0~5 gf
Sliding velocity	2.5 μm/s	2.5 μm/s
Film depth	130 nm	280 nm

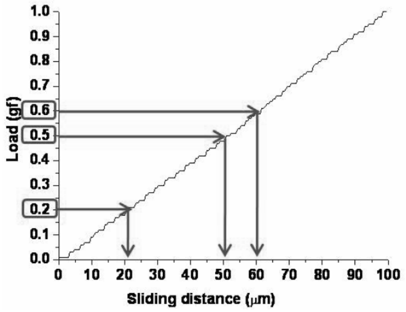


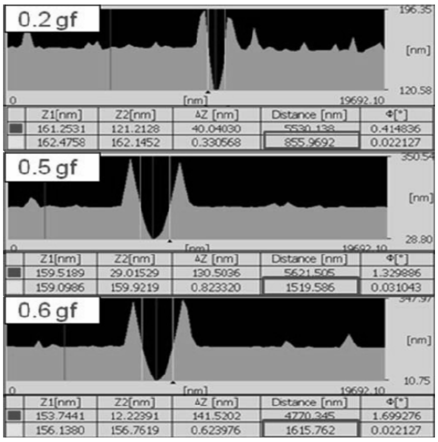
Fig. 3. Normal force variation with respect to sliding distance.

Table 2. Results of the scratch test on the Ag thin films.

Normal force (gf)	Scratch depth (nm)	Scratch width (μm)	Frictional force (gf)
0.2	40	0.9	0.01
0.5	130	1.5	0.1
0.6	140	1.6	0.14

Table 3. Results of the scratch test on the ZnO thin films.

Annealing temp. (℃)	Critical force (gf)	Scratch depth (nm)	Scratch width (μm)	Frictional force (gf)
450	1.5	280	2.5	0.3
550	2.6	280	2.6	1.3
800	3.5	280	3.1	2.5

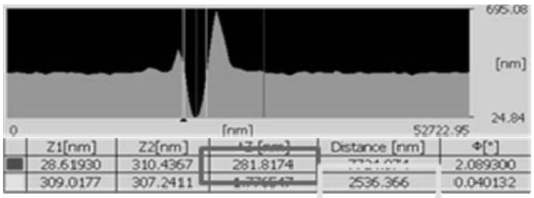


	Z1[nm]	Z2[nm]	ΔZ [nm]	Distance [nm]	Φ[°]
■	161.2531	121.2128	40.04030	5530.138	0.414836
□	162.4758	162.1452	0.330568	855.9692	0.022127

	Z1[nm]	Z2[nm]	ΔZ [nm]	Distance [nm]	Φ[°]
■	159.5189	29.01529	130.5036	5621.505	1.329886
□	159.0986	159.9219	0.823320	1519.586	0.031043

	Z1[nm]	Z2[nm]	ΔZ [nm]	Distance [nm]	Φ[°]
■	153.7441	12.22391	141.5202	4770.345	1.699276
□	156.1380	156.7619	0.623976	1615.762	0.022127

Fig. 4. Results of the depth profiling of wear track on the Ag thin films.



	Z1[nm]	Z2[nm]	[illegible]	Distance [nm]	Φ[°]
■	28.61930	310.4367	281.8174	[illegible]	2.089300
□	309.0177	307.2411	[illegible]	2536.366	0.040132

Fig. 5. Results of the depth profiling of wear track on the ZnO thin films.

Table 4. Results of the theoretical analysis for the Ag thin films.

Normal Force (gf)	P_1 (gf)	P_2 (gf)	T (gf)	F_H (gf)
0.2	-	5.4x10^{-4}	-	5.4x10^{-4}
0.5	-	2.9x10^{-3}	0.048	0.051
0.6	4.4x10^{-5}	3.1x10^{-3}	0.062	0.065

Table 5. Results of the theoretical analysis for the ZnO thin films.

Temp (℃)	Force (gf)	P_2 (gf)	T (gf)	F_H (gf)
450	1.5	9.8x10^{-3}	0.11	0.12
550	2.6	1.0x10^{-2}	0.75	0.76
800	3.5	1.3x10^{-2}	1.76	1.77

4. Theoretical analysis

Benjamin and Weaver (1960) reported an analytical model to obtain the horizontal force assumed by scratching a film coated on a substrate [10]. Essentially, the horizontal force, F_H, needed to remove the film from the substrate may be obtained by the following equations:

$$F_H = P_1 + P_2 + T \qquad P_2 = dtp'$$

$$P_1 = d^3 p / 12r \qquad T = \frac{1}{4}\tau\pi d^2$$

$$\tau = \frac{W}{2\pi d^2}\left\{(4+\nu_1)\frac{3\pi\mu}{8} - (1-2\nu_1)\right\}\frac{d}{r}$$

where P_1 is the force needed to plow the substrate, P_2 is the force required to push aside the thin film, d is the width of the wear track, p is the hardness of the substrate, r is the tip radius, p' is the hardness of the thin film, t is the thickness of the thin film, T is the shear force per unit area of the interface needed to detach the film from the substrate, τ is the interfacial shear stress between the substrate and the thin film, ν_1 is the Poisson's ratio of the thin film, W is the vertical force, and μ is the friction coefficient. The values in the equations were obtained from experimental results or the literature.

The experimental results were applied to the above equations to investigate the adhesion characteristics of the thin film. The results of the theoretical analysis for the Ag and ZnO films are summarized in Tables 4 and 5, respectively. The horizontal force, F_H, can be considered as the frictional force measured in the scratching experiment. In the case of the Ag film, it was found that the frictional force is significantly different from the measured values shown in Table 2. In the case of the ZnO films, there was no P_1 component since the calculation was performed at the critical force where the tip did not penetrate into the substrate. Also, P_2, the force required to push aside the thin film, was too small as compared to the shear force T. As a result, F_H, which is the sum of P_2 and T, is relatively smaller than the frictional force given in Table 3.

To minimize the discrepancy between the experimental and analytical results, the Benjamin and Weaver model was modified. Specifically, a plowing model was applied to replace the terms P_1 and P_2, since the terms were used to calculate the plowing force between the tip and the substrate or the thin film. In the modified model, the force F required to create a wear track and normal force L are represented as shown in the following equations:

$$F = HA_h = H \cdot a \cdot d \qquad \mu = \frac{F}{L} = \frac{2 \cdot d}{\pi \cdot a}$$

$$L = HA_r = H \cdot \frac{\pi \cdot a^2}{2} \qquad F = L \cdot \frac{2 \cdot d}{\pi \cdot a}$$

where H is the hardness of the substrate which was obtained by using the Vickers hardness tester, A_h is the contact area between the tip and the substrate in the horizontal direction, A_r

Table 6. Results of applying the plowing model for the Ag thin films.

Normal Force (gf)	F (gf)	T (gf)	F_H (gf)
0.2	0.011	-	0.011
0.5	0.055	0.048	0.1
0.6	0.068	0.062	0.13

Table 7. Results of applying the plowing model for the ZnO thin films.

Temp(℃)	Normal Force (gf)	F (gf)	T (gf)	F_H(gf)
450	1.5	0.21	0.11	0.32
550	2.6	0.35	0.75	1.1
800	3.5	0.39	1.76	2.15

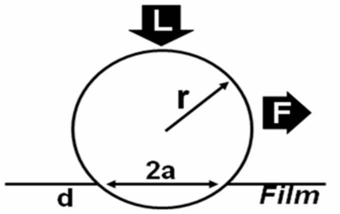


Fig. 6. Diagram of the plowing model used instead of the modified Benjamin and Weaver model.

is the contact area in the vertical direction, a is the radius of the tip at the contact region, and d is the penetration depth. The hardness in the equations was obtained by using the Vickers hardness tester. The diagram for understanding the variables of the plowing model is shown in Fig. 6. The results of the modified Benjamin and Weaver model analyses for the Ag and ZnO films are shown in Tables 6 and 7, respectively. As can be seen from the calculated values, the horizontal force (F_H) values were very similar to the values obtained from the experimental results, with discrepancies in the order of 10%.

Thus, it is clearly demonstrated that the modified model proposed in this work is more suitable for analyzing the adhesion characteristics of the thin films.

5. Conclusion

Ramp loading scratch tests were performed to assess the adhesion characteristics of the Ag and ZnO thin films coated on the silicon wafer. The Ag (thickness~130 nm) and ZnO (thickness~280 nm) films were deposited on the silicon wafers by the sputtering and sol-gel method, respectively. A diamond tip with a radius of 5 μm was used for scratching. The normal force and frictional force were monitored during the scratch tests. Also, the width and depth of the wear track were measured with an AFM following the scratch tests. The critical force at which the Ag film failed was 0.5 gf. As for the ZnO film, and the critical force was 1.5 gf, 2.6 gf, and 3.5 gf for annealing temperatures of 450℃, 550℃, and 800℃, respectively. The higher critical force observed for a higher annealing temperature was attributed to the increase in film hardness with annealing temperature.

In theoretical analysis, the Benjamin and Weaver model was initially used to compare with the experimental results. However, the theoretically calculated results showed large discrepancies from the experimental results. The discrepancies could be minimized by modifying the Benjamin and Weaver model with a plowing term. Using the modified Benjamin and Weaver model, the horizontal force needed to remove the Ag film of 130 nm thickness was calculated to be 0.1 gf. This value matched the frictional force obtained during the scratch test at which the Ag film failed. For all other cases, the differences between the experimental frictional force obtained during the scratch test and the theoretical analysis using the modified Benjamin and Weaver model were less than 10%. Therefore, to estimate the adhesion characteristics between a thin film and substrate, the modified Benjamin and Weaver model proposed in this work is appropriate.

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