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UNCERTAINTY EVALUATION OF THE NANOINDENTATION SYSTEM

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Abstract: In this paper, the uncertainty of nanoindentation system was evaluated. The laser interferometer and electronic balance were respectively adopted to calibrate the quantities of displacement and load of nanoindeantion system. The results reveal that the relative expanded uncertainty of indeantion hardness, U_H, is 8.1%. The relative expanded uncertainity of reduced modulus, U_{Er}, is 5.4%.

Keywords: uncertainty, nanoindentation system

1. INTRODUCTION

During the recent decades, the thim films are widely used in semiconductor, microelectromechanical system (MEMS), etc. The reliability of the these components is considerably influenced by the mechnaical properties of thin films. Therefore, it is an important issue to characterize the mechnaical properties of the constituent materials of thin films.

Since the thickness of the thin films are rather thin, their mechanical properties are quite different than those of bulk materials. Besides, the traditionally experimental techniques cannot be employed to measure the mechanical properties of thin films. Although some novel experimental techniques have been developed to understand the mechanical properties of thin films, either their experimental prepare or procedures are almost tedious. In contrast to the aforementioned methods, nanoindenation method is an attractive technique due to its easy and convenient procedure. In the nanoindenation experiment, it is only necessary to coat the thin film onto the substrate. Thus, the nanoindentation method is adopted by more and more researchers.

Regarding the nanoindentation test, the experiment is accomplished by employing an indenter to press into the surface of tested material. The experimental data of displacement versus applied load are recorded through the precise actuator and sensor. Since the mechanical properties of constituent material of indenter is known, the indentation hardness and reduced modulus of tested material can be obtained through the unloading behavior and theory of contact mechanics. The contact mechanics was first originated from the investigation of contact behaviors between the distinct bodies by Boussinesq [1] and Hertz [2, 3]. Sneddon [4] developed a theory to predict the contact behaviors for any punch with solid revolution of a smooth function. He discovered that the relationship between displacement and load can be appropriately described by power-law function and it has been used in the calculation of reduced modulus of nanoindentation test. Because the residual imprint of indentation test is rather small, its projected area is difficult to observe directly from optical microscope. To remedy this requirement, Oliver and Pharr [5] proposed the area function to describe approximately the relationship between the projected area and contact depth. In this paper, the uncertainty of nanoindentation system was attempted to evaluate [6-8].

2. THEORY OF NANOINDEANTION

The definition of indentation hardness is as follows:

$$H = \frac{P_{\max}}{A} \tag{1}$$

where $P_{\max}$ is the maximum load and A is the projected area. Oliver and Pharr [6] discoveried that the power-law relationship is appropriate to describe the unloading behavior, i.e.

$$P = \alpha(h - h_f)^m \tag{2}$$

where P is the applied load, h is the total displacement, h_f is the residual depth after removing the applied load, α is the constant, and m is the order of the power-law equation. According to the theory of contact mechanics, it gives

$$E_r = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A}} \quad (3)$$

$$h_c = h_{max} - \varepsilon \frac{P_{max}}{S} \quad (4)$$

where $E_r = \frac{1-\nu_1^2}{E_1} + \frac{1-\nu_2^2}{E_2}$ is the reduced modulus, ν_1 and E_1 are the Poisson's ratio and Young's modulus of indenter, ν_2 and E_2 are the Poisson's ratio and Young's modulus of the tested materials, and ε is the geometrical constant of indenter. It is defined that the contact stiffness (S) is the slope of unloading curve while the maximum load is applied. Thus,

$$S = \frac{dP_{\max}}{dh_{\max}} = m\alpha\left(h_{\max} - h_f\right)^{m-1} \quad (5)$$

Oliver and Pharr [5] proposed the following area function to describe the projected area of the indenter.

$$A(h_c) = C_0\, h_c^2 + C_1\, h_c + C_2\, h_c^{1/2} + C_3\, h_c^{1/4} + C_4\, h_c^{1/8} + C_5\, h_c^{1/16} \quad (6)$$

3. EXPERIMENTAL PROCEDURE

Based on the aforementioned description, the experimental result of nanoindentation test will be influenced by many parameters. To evaluate the unceraint of nanoindeantion system, the emphasis should be first placed on the fundamental quantities, i.e. displacement and load. For this reason, in this paper, the displacement and load were respectively calibrated by the appropriate techniques. The laser interferometer (LAZAX L-DD-01, TOSEI ENGIINEERING Co.) was utilized to calibrate the displacement provided by the nanoindeantion system and the schematic of experimental setup is shown in Fig. 1. The electronic balance (WZ215-CW, Sartorius) was used to calibrate the load of nanoindeantion system (Fig. 2). After the uncertainties of displacement and load of nanoindeantion system were determined, the uncertainties of other factors, i.e. thermal drift, zero point determination, machine compliance, area function, are also evaluated.

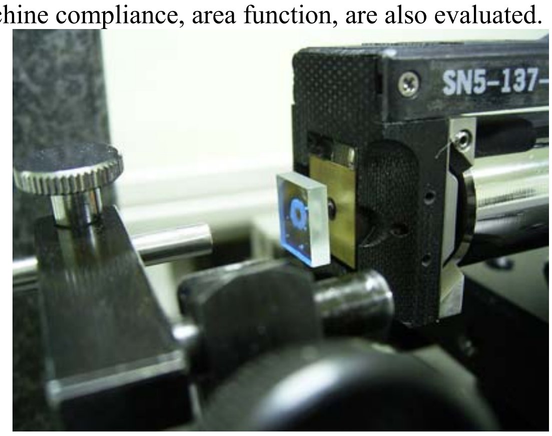

Fig. 1 The schematic of displacement calibrated system


Fig. 2 Fig. 1 The schematic of load calibrated system

4. THE EVALUATION OF UNCERTAINTY

4.1 Force uncertainity

Regarding the evaluation of force uncertainty, the indentation mode of load control was performed. The applied loads were gradually applied on the electronic balance up to 10 mN with the identical interval for 500 μN. The experimental data for force calibration are listed in Table 1.

Table 1 The experimental data for force calibration

F	$\overline{\Delta_F}$	$u(I_m)$	$u(g)$	$u_{\Delta F}$	u_{Fr}	$u(F)$	$u(F)/F$ (%)
500	13.382	0.016	0.00002887	3.863	0.882	3.965	0.793
1000	28.168	0.016	0.00002887	8.131	1.060	8.202	0.820
1500	43.574	0.016	0.00002887	12.578	1.028	12.621	0.84
2000	60.644	0.016	0.00002887	17.506	1.194	17.547	0.877
2500	76.801	0.016	0.00002887	22.170	1.277	22.207	0.888
3000	94.621	0.016	0.00002887	27.314	1.843	27.377	0.912
3500	113.616	0.016	0.00002887	32.798	2.866	32.923	0.940
4000	129.51	0.016	0.00002887	37.386	1.798	37.430	0.935
4500	147.234	0.016	0.00002887	42.503	1.588	42.533	0.945
5000	166.099	0.016	0.00002887	47.948	2.127	47.996	0.959
5500	184.344	0.016	0.00002887	53.215	1.941	53.251	0.968
6000	202.262	0.016	0.00002887	58.388	2.139	58.427	0.973
6500	220.866	0.016	0.00002887	63.758	2.075	63.792	0.981
7000	239.307	0.016	0.00002887	69.082	2.051	69.112	0.987
7500	258.269	0.016	0.00002887	74.556	2.297	74.591	0.994
8000	277.330	0.016	0.00002887	80.058	1.923	80.081	1.001
8500	296.129	0.016	0.00002887	85.485	2.202	85.513	1.006
9000	315.255	0.016	0.00002887	91.006	2.210	91.033	1.01
9500	333.957	0.016	0.00002887	96.405	2.091	96.427	1.01
10000	353.246	0.016	0.00002887	101.97	2.014	101.99	1.019

It has the relationship for the nomial magnitudes between nanoindentation measurement system (F) and the electronic balance (I_m), i.e.

$$F = I_m \times g + \Delta_F \quad (7)$$

where g is the gravitational acceleration , and Δ_F is the gauge value. Using the uncertainty propagation law, it gives

$$[u(F)]^2 = [g \times u(I_m)]^2 + [I_m \times u(g)]^2 + [u_{\Delta F}]^2 + [u_{Fr}]^2 \quad (8)$$

where $u(F)$ is the uncertainty of force, $u(I_m)$ is the uncertainty of electronic balance, $u(g)$ is the gravitational uncertainty, $u_{\Delta F}$ is the uncertainty of gauge value, and u_{Fr} is the standard deviation estimated form the three experiment results.

4.2 Depth uncertainty

The gauge values of displacemnts between the nanoindentation mesurement system (h) and interferometer (I_H) have the following relationship, i.e.

$$h = I_H + \Delta I_h \tag{9}$$

Using the propagation law of uncertainity, it yields

$$[u(h)]^2 = [u(I_H)]^2 + [u(\Delta I_h)]^2 + [u_{Ihr}]^2 + [u_T]^2 + [u_Z]^2 + [u_C]^2 \tag{10}$$

where $u(h)$ is the uncertainty of the depth, $u(I_H)$ is the uncertainty of optical interferometer, $u(\Delta I_h)$ is the uncertainty of gauge value, u_{Ihr} is the standard deviation obtained from three experiments, u_T is the uncertainty resulted form the thermal drift, u_Z is the uncertainty in determining the zero point, and u_c is the uncertainty caused by the machined compliance. The experimental data for displacement calibration is shown in Table 2. The uncertainty and relative uncertainty related to the contact depth is also listed in Table 3.

Table 2 The experimental data for displacement calibration

I_{H1}	ΔI_{h1}	h_2	I_{H2}	ΔI_{h2}	h_3	I_{H3}	ΔI_{h3}	$\overline{\Delta I_h}$	$s(\overline{\Delta I_h})$
510	-4.4304	506.408	505	1.407	504.364	515	-10.636	-4.552	3.4773
1040	-35.007	1006.8	1015	-8.204	1007.29	1020	-12.709	-18.640	8.286
1520	-19.112	1501.34	1520	-18.662	1500.91	1515	-14.090	-17.288	1.604
2020	-16.601	2003.52	2020	-16.483	2005.572	2025	-19.427	-17.504	0.962
2530	-28.767	2505.13	2590	-84.869	2505.198	2535	-29.801	-47.812	18.530
3100	-90.026	3009.07	3035	-25.932	3009.058	3080	-70.941	-62.300	19.000
3615	-111.641	3503.82	3540	-36.182	3507.146	3545	-37.853	-61.892	24.879
4045	-43.320	4004.38	4045	-40.615	4004.019	4050	-45.981	-43.305	1.549
4550	-47.591	4503.19	4560	-56.811	4502.688	4550	-47.312	-50.571	3.120
5055	-69.156	4988.4	5055	-66.601	4987.431	5050	-62.568	-66.108	1.917

Table 2 The uncertainty and relative uncertainty related to the contact depth

(Unit：nm)								
Depth	$u(I_H)$	$u(\Delta I_h)$	u_{Ihr}	u_T	u_Z	u_C	$u(h)$	$u(h)$ / h (%)
510	2.887	1.314	3.477	0.958	0.2413	3.175	5.763	1.130
1040	2.887	5.381	8.286	0.958	0.2413	3.175	10.817	1.040
1520	2.887	4.990	1.604	0.958	0.2413	3.175	6.846	0.450
2020	2.887	5.053	0.962	0.958	0.2413	3.175	6.771	0.335
2530	2.887	13.802	18.530	0.958	0.2413	3.175	23.522	0.929
3100	2.887	17.984	19.000	0.958	0.2413	3.175	26.529	0.855
3615	2.887	17.866	24.879	0.958	0.2413	3.175	30.944	0.856
4045	2.887	12.501	1.549	0.958	0.2413	3.175	13.344	0.329
4550	2.887	14.598	3.120	0.958	0.2413	3.175	15.564	0.342
5055	2.887	19.083	1.9174	0.958	0.2413	3.175	19.679	0.389

4.3 Area function uncertainty

From the Eqn. (6) it is easy to obtain the following relationship between the uncertainty and contact depth.

$$[u(A_P)]^2 = \left[\left(2C_0 h_C + C_1 + \frac{1}{2}C_2 h_C^{-1/2} + \frac{1}{4}C_3 h_C^{-3/4} + \frac{1}{8}C_4 h_C^{-7/8} + \frac{1}{16}C_5 h_C^{-15/16}\right) \times u(h_C)\right]^2 + [u_\Delta]^2 \tag{11}$$

where $u(h_c)$ is the uncertainty of contact depth and u_Δ is the residuals of fitting curve. The uncertainty of contact depth in Eqn. (11) can be derived from Eqn. (4) and it is

$$[u(h_C)]^2 = [u(h_{max})]^2 + \left[\frac{\varepsilon}{S} u(F_{max})\right]^2 + \left[\frac{\varepsilon}{S^2} F_{max} u(S)\right]^2 \tag{12}$$

where $u(h_{\max})$ is the uncertainty of maximum contact depth, $u(F_{\max})$ is the uncertainty of maximum applied load and $u(S)$ is the uncertainty of contact stiffness. This magnitude can be obtained from Eqn. (5) and it is

$$[u(S)]^2 = \left[mK(m-1)(h_{max}-h_p)^{m-2}\right]\left[u(h_{max})^2 + u(h_p)^2\right] \tag{13}$$

From the aforementioned relations, we can obtain the uncertainty of contact depth ($u(h_c)$), contact stiffness (S), and the relative uncertainty of projected area are as follows:

$$u(h_c) = 3.252nm \tag{14}$$

$$u(S) = 1.225nm \tag{15}$$

$$[u(A_p)/A_p] = 3.9\% \tag{16}$$

4.4 Evalutation of uncertainty of indentation hardness

The uncertainty of indentation hardness (u_{IT}) can be obtained from Eqn. (1) and it is

$$[u(H_{IT})]^2 = \left[\frac{1}{A_P} u(F_{\max})\right]^2 + \left[\frac{F_{\max}}{A_P^2} u(A_P)\right]^2 \tag{17}$$

This equation can be further deduced as the follows:

$$\left[\frac{u(H_{IT})}{H_{IT}}\right]_S^2 = \left[\frac{u(F_{\max})}{F_{\max}}\right]^2 + \left[\frac{u(A_P)}{A_P}\right]^2 \tag{18}$$

Therefore, the relative expanded uncertainty of indentation hardness under the 95 % confidence ($k = 2$) is

$$U_{HS} = 2 \times \left[\frac{u(H_{IT})}{H_{IT}}\right]_S = 8.1\% \tag{19}$$

4.5 Evaluation of uncertainty of reduced modulus

The uncertainty of reduced modulus can be deduced from Eqn. (3) and it is

$$[u(E_r)]^2 = \left[\frac{\sqrt{\pi}}{2}\frac{1}{\sqrt{A_P}} u(S)\right]^2 + \left[\frac{\sqrt{\pi}}{4}\frac{S}{A_P^{3/2}} u(A_P)\right]^2 \tag{20}$$

Re-arrange this equation, the relative expanded uncertainty of reduced modulus is as follows:

$$\left[\frac{u(E_r)}{E_r}\right]^2=\left[\frac{u(S)}{S}\right]^2+\left[\frac{1}{2}\frac{u(A_P)}{A_P}\right]^2 \qquad (21)$$

After some calculation, the relative expanded uncertainty of reduced modulus under the 95 % confidence ($k=2$) is

$$U_{Er}=2\times\left[\frac{u(E_r)}{E_r}\right]_S=5.3\% \qquad (22)$$

4. RESULTS AND DISCUSSIONS

In this paper, the uncertainty of nanoindeantion system was evaluated. The indenation hardness and reduced modulus obtained from nanoindentation system were respectively estimated. The results reveals that the relative expanded uncertainty of indeantion hardness, U_{HS} , is 8.1%. The relative expanded uncertainity of reduced modulus, U_{Er} , is 5.4%.

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