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# UNCERTAINTY EVALUATION OF MODULUS AND HARDNESS FOR THE NANOINDENTATION MEASUREMENT SYSTEM

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**Abstract**- The uncertainties of nanoindentation measurement system were evaluated in this paper. A precise electonic balance and an optical interferometric system were respectively utilized to calibrate the force and displacement of nanoindentation measurement system. The uncertainties of reduced modulus and indentation hardness providing from the naoindentaion measurement were respectively obtained.

**Keywords:**Uuncertainty, nanoindentation

## 1. INTRODUCTION

The nanoindentation measurement system was frequently employed to characterize the mechanical properties of thin films. For instance, the benefits of surface modification of thin films are evaluated through the nanoindentation measurement results. It should be emphasized that the measurement difference of the investigated thin films comes from the inherent uncertainty of system and the exhibiting characteristics of thin films. It is not appropriate to conclude the performance of surface modification from the nanoindentation measurement results while their differences are smaller than system uncertainty. For this reason, it is indeed an important issue to evaluate the system uncertainty of nanoindentation system such that meaningful difference of measurement result can be clarified. This paper aims to evaluate the system uncertainty of nanoindentation measurement system and experimental procedures were followed suggestion in Refs. [1-3]. The uncertainty relationships were expressed from the naoindentaiton theory. A precise electronic balance was used to calibrate the force of nanoindentation measurement system. An optical interferometer was established to calibrate the displacement of nanoindentation measurement system.

## 2. CALIBRATION AND UNCERTAINTY EVALUATION

### 2.1 Force calibration and uncertainty evaluation

The uncertainty of nanoindentation measurement system (TriboIndenter, Hysitron Inc., U.S.A.) was evaluated and the uncertainty expressions were derived in accordance with ISO GUM (Guide to the Expression of Uncertainty in Measurement) [4]. The precise electronic balance (WZ 215-CW, Sartorius) was employed to calibrate the force of nanoindentation measurement system. The experimental arrangement is shown in Fig. 1. The relationship between the force magnitudes obtained respectively from nanoindentation ($F$) and electronic balance ($I_m$) can be expressed as

$$F = I_m \times g + \Delta_F \qquad (1)$$

where $g$ is the local acceleration of gravity where the nanoindentation system was established and $\Delta_F$ is the indication error. Since the measurement result would not be corrected by indication error in the further, the standard force uncertainty ($u(F)$) of nanoindentation system can be expressed as

$$u^2(F) = [u(I_m) \times g]^2 + [I_m \times u(g)]^2 + u^2(F_n) + u^2(F_r) + u^2(\Delta F) + u^2(F_z) + u^2(F_{repeat}) \qquad (2)$$

where $u(I_m)$ is the standard uncertainty of electronic balance, $u(g)$ is the standard uncertainty of acceleration of gravity, $u(F_n)$ is the standard uncertainty resulting from the noise, $u(F_r)$ is the standard uncertainty resulting from the resolution of force transducer, $u(\Delta F)$ is the standard uncertainty of indication error, $u(F_z)$ is he standard uncertainty of zero point and $u(F_{repeat})$ is the standard force uncertainty of force repeatability.

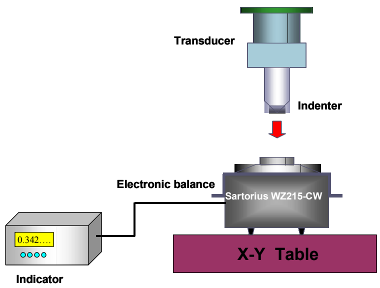


Fig.1 The experimental arrangement of force calibration

Prior to force calibration, the electronic balance was calibrated from 1 mg to 1 g by the traceable weights and the result gives [5]

$$u(I_m) = 0.000033\ \text{g} \qquad (3).$$

In addition, the absolute acceleration of gravity at Center for

Measurement Standards (CMS) had been measured and it is $9.789137281 \pm 2.5 \times 10^{-8}$ (m/s$^2$) [6]. Consider the uncertainty distribution of acceleration of gravity is rectangular and thus the standard uncertainty of acceleration of gravity ( $u(g)$ ) is

$$u(g) = \frac{2.5 \times 10^{-8}}{\sqrt{3}} (\text{m/s}^2) \quad (4).$$

The resolution of transducer of nanoindentation system is 1 nm [7]. Suppose the uncertainty resulting from resolution of transducer is rectangular distributed and it yields

$$u(F_r) = \frac{10^{-9}}{2\sqrt{3}} \ (\text{nN}) \quad (5).$$

The fused silica and indenter with Berkovich geometry were employed to respectively demtermine the standards uncertainties of noise floor and zero point. First, the indenter was placed on the surface with 0 N for 20 seconds. The force data were recorded during this period and their standard deviation is treated as standard uncertainty of noise floor for this experiment. The afromentioned experiment was repeated for ten times, and the maximum magnitude in the nosie floor experiment was selected to be the standard uncertainty of noise floor ( $u(F_n)$ ), i.e.

$$u(F_n) = 0.063271 (\mu\text{N}) \quad (6).$$

Berkovich indenter as well as fused silica were also used to determinate the standards uncertainty of zero point ( $u(F_z)$ ). Let the indenter apply load on the surface of fused silica up to 20 μN within 20 seconds. The displacement and force data were simultaneously recorded ,and the standards uncertainty of zero point ( $u(F_z)$ ) was selected by the froce magnitude while its corresponding displacement is zero. The afromentioned experiment was repeated for ten times, and the maximum magnitude in the experiments of standards uncertainty was selected to be the standard uncertainty of zero point ( $u(F_z)$ ), i.e.

$$u(F_z) = 0.274617 \ (\mu\text{N}) \quad (7).$$

On the other hand, the force of nanoindentation system was calibrated from 500 μN to 1000 μN with equal increment of 500 μN by electronic balance. The experiment was repeated three times and the experimental results are listed in Table 1. In this table, the standard force uncertainty of repeatability ( $u(F_{repeat})$ ) is calculated by the standard deviation of indication error of three experiments, i.e.

$$u(F_{repeat}) = \left[ \sum_{i=1}^{n} \frac{(\Delta F_i - \overline{\Delta F})}{n-1} \right]^{1/2} / \sqrt{n}, \ n = 3 \quad (8).$$

The standard uncertainty of indication error ( $u(\Delta F)$ ) is the mean value of the three experiments. The details about the force calibration results are listed in Table 1 in which $\overline{F}$ is the mean of three time experiments of nanoindentation system, $\overline{I_m}$ is the mean of three time experiments of electronic balance, and $\overline{\Delta F}$ is the mean of three time experiments of indication error. Besides, it can also be seen in Table 1 that the relative standard uncertainty of force ( $u(F)/F$ ) for the nanoindentation system investigated is 2.21%.

| F (μN) | $\overline{F}$ (μN) | $\overline{I_m}$ (g) | $\overline{I_m} \times g$ (μN) | $\overline{\Delta F}$ (μN) | $u(F_{repeat})$ (μN) | $u(\Delta F)$ (μN) | u(F) (μN) | u(F)/F % |
|---|---|---|---|---|---|---|---|---|
| 500 | 499.895 | 0.05060 | 495.29772 | 4.59718 | 0.40089 | 4.59718 | 4.62635 | 0.925 |
| 1000 | 999.849 | 0.10070 | 985.76612 | 14.08292 | 1.52205 | 14.08292 | 14.16876 | 1.417 |
| 1500 | 1499.815 | 0.15093 | 1477.44186 | 22.37302 | 1.66689 | 22.37302 | 22.43744 | 1.496 |
| 2000 | 1999.784 | 0.20102 | 1967.77975 | 32.00387 | 1.30823 | 32.00387 | 32.03229 | 1.602 |
| 2500 | 2499.761 | 0.25113 | 2458.31341 | 41.44759 | 2.44452 | 41.44759 | 41.52091 | 1.661 |
| 3000 | 2999.729 | 0.30118 | 2948.32500 | 51.40434 | 2.96075 | 51.40434 | 51.49058 | 1.716 |
| 3500 | 3499.703 | 0.35120 | 3437.91238 | 61.79110 | 3.16550 | 61.79110 | 61.87300 | 1.768 |
| 4000 | 3999.675 | 0.40113 | 3926.68401 | 72.99064 | 3.71064 | 72.99064 | 73.08564 | 1.827 |
| 4500 | 4499.645 | 0.45113 | 4416.14087 | 83.50422 | 3.86525 | 83.50422 | 83.59428 | 1.858 |
| 5000 | 4999.618 | 0.50104 | 4904.71671 | 94.90154 | 4.53848 | 94.90154 | 95.01057 | 1.900 |
| 5500 | 5499.614 | 0.55080 | 5391.82418 | 107.78959 | 5.16180 | 107.78959 | 107.91362 | 1.962 |
| 6000 | 5999.641 | 0.60074 | 5880.72633 | 118.91475 | 5.51668 | 118.91475 | 119.04310 | 1.984 |
| 6500 | 6499.608 | 0.65057 | 6368.48641 | 131.12117 | 5.55828 | 131.12117 | 131.23933 | 2.019 |
| 7000 | 6999.561 | 0.70039 | 6856.18123 | 143.38024 | 6.14413 | 143.38024 | 143.51220 | 2.050 |
| 7500 | 7499.563 | 0.75024 | 7344.20235 | 155.36073 | 6.33809 | 155.36073 | 155.49031 | 2.073 |
| 8000 | 7999.544 | 0.80001 | 7831.40772 | 168.13674 | 6.89113 | 168.13674 | 168.27822 | 2.103 |
| 8500 | 8499.537 | 0.84981 | 8318.87412 | 180.66247 | 8.39574 | 180.66247 | 180.85775 | 2.128 |
| 9000 | 8999.509 | 0.89913 | 8801.70700 | 197.80237 | 11.41988 | 197.80237 | 198.13203 | 2.201 |
| 9500 | 9499.500 | 0.94927 | 9292.53435 | 206.96548 | 8.31780 | 206.96548 | 207.13282 | 2.180 |
| 10000 | 9999.476 | 0.99899 | 9779.21762 | 220.25807 | 8.91230 | 220.25807 | 220.43855 | 2.204 |

Table 1 The experimental data for force calibration

## 2.2 Displacement calibration and uncertainty evaluation

An optical interferometric system was established to calibrate the displacement of nanoindentation measurement system. The schematic of experimental arrangement is illustrated in Fig. 2 First, a cloumn was self-prepared to assembly with the tranducer. Then a piece of silicon wafer coated with the gold film was adhered to the end the column. The gold film aims to increase the light intensity reflected from the silicon wafer. The He-Ne laser with 633nm wavelength was utilized as the light souce and it is traceable to SI unit through the dimensional standards established at CMS. The relative standard uncertainty of wavelenght ( $u(\lambda)/\lambda$ ) is $3.55 \times 10^{-10}$ [8]. In Fig. 3, four measurement beams are directed to the wafer surface by the high resolution interferometer (HP10716A). In addition, the axis board (HP10885A) was used to analyze the data so that measurement resolution can be down to 2.5 nm.

Let h and $I_L$ be the displacements respectively obtained from nanoindentation system and optical interferometric system, and the relationship of displacement calibration can be expresses as

$$h = I_L + \Delta h \quad (9)$$

where $\Delta I$ is the indication error. Since the measurement result would not be corrected by indication error in the further, the displacement uncertainty ( $u(h)$ ) of nanoindentation system can be expressed as

$$u^2(h) = u^2(I_L) + u^2(I_R) + u^2(h_r) + u^2(h_n) + u^2(h_T) + u^2(h_c) + u^2(h_{repeat}) + u^2(\Delta\bar{I}) \quad (10)$$

where $u(I_L)$ is the standard uncertainty of optical interferomertic system, $u(I_R)$ is the standard uncertainty resulting from the resolution of optical system, $u(h_r)$ is the standard uncertainty resulting from the resolution of transducer of naoindentation system, $u(h_n)$ is the standard uncertainty resulting from the noise, $u(h_T)$ is the standard uncertainty of thermal drift, $u(h_c)$ is the standard uncertainty resulting from the uncertainty of machine compliance of nanoindentation system, $u(h_{repeat})$ is the standard uncertainty of displacement repeatability, and $u(\Delta\bar{I})$ is the standard uncertainty of indication error.

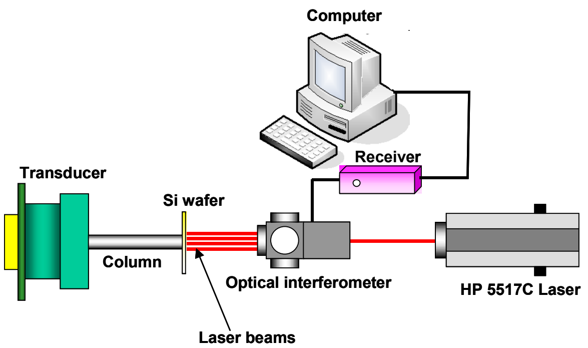


Fig. 2 The schematic of experimental arrangement for depth calibration

It had been indicated that the resolution of optical interferometric system adopted in this investigation is 2.5 nm, and the displacement resolution of transducer of nanoindentation system is 0.04 nm [7]. Suppose the standard uncertainties resulting from resolutions of optical interferometric and nanoindentation system are both rectangular distributed and it gives

$$u(I_R) = \frac{2.5}{2\sqrt{3}} = 0.721687836\ (\text{nm}) \quad (11)$$

$$u(h_r) = \frac{0.04}{2\sqrt{3}}\ (\text{nm}) \quad (12).$$

The fused silica and Bekovich indenter were also employed to determine the displacement noise, thermal drift, and machine compliacne of naoindentation system. First, the surfae of fused silica was applied a constant load of 1.5 μN by indeter. Then the indenter was scanned by the area of 1 nm×1 nm with frequency of 1 Hz, and the dispacement was simultaneously recorded. The root mean square (RMS) in the scanning image of diplacment is calculated to be the displacement noise for this experiment. The experiment was repeated for then times, and the maximum magnitude of displacement noise is slected to be the standard uncertainty of dipslacement noise ( $u(h_n)$ ) and it is

$$u(h_n) = 0.242965\ (\text{nm}) \quad (13)$$

The determineation of stnadard uncertainty of thermal dift must first measure the thermal drift rate of nanoindentation system. Turn off the function of drift correction in the operation software of nanoindentation system and apply an load of 0 N on the surface of fused silica for 60 seconds. Record the data of displacement versus time during this period, and then thermal drift rate is determined from the slope of fitting line of the data of displacement versus time. Repeat the above-mentioned, and select the maximum thermal drift rate as the thermal drift rate of nanoindentation system. Suppose the amount of operation time in the further will not longer than 60 seconds, and thus the standard uncertainty of thermal drift of nanoindentaiton system ( $u(h_T)$ ) is

$$u(h_T) = 0.0092\ (\text{nm/s}) \times 60\ (\text{sec}) = 0.552\ (\text{nm}) \quad (14).$$

The determination of standard uncertainty resulting from the uncertainty of mechanice complinace ( $u(h_c)$ ) has to first measure the mechine compliance of nanoindentation system. The loads were increasly applied from 5000 μN to 10000 μN. Details of the experimental procedure of machine compliane can be found in Ref. [7]. Repeat the same experiment of the determination of mahcine compliance for for five times, and their standard deviation is selected as the standard uncertainty of mechanice compliance, i.e. 0.158114 (nm/mN). Because the maxumum load can be provied by the system is 10000 μN, the standard uncertainty resulting from the uncertainty of mechanice complinace ( $u(h_c)$ ) is

$$u(h_c) = 0.158114(\text{nm/mN}) \times 10000(\mu\text{N}) = 1.58114(\text{nm}) \quad (15)$$

For the experiment of displacement calibration, the displacement of nanoindentaion system was calibrated by the opitcal interferometric system from 50 nm to 300 nm with the same increasement of 50 nm. The experiment was repeated for three times. It had been mentioned that the relative standard uncertainty of wavelenght ( $u(\lambda)/\lambda$ ) is $3.55\times10^{-10}$, and thus the standard uncertainty of optical interferometric system is

$$u(I_L) = L_G \times u(\lambda)/\lambda =_G \times 3.55 \times 10^{-10} \quad (16)$$

where $L_G$ is the calibration displacement magnitude. The experimental result for displacement calibration is listed in Table 2 in which $\bar{h}$ is the mean of three time experiments of nanoindentaiton system, $\bar{I}$ is the mean of three time experiments of the optical interferomertic system, and $\overline{\Delta I}$ is the mean of three time experiments of indication error. Besides, the standard uncertainty of indication error ( $u(\Delta F)$ ) is calculated from the mean value of the three experiments. In this table, the standard displacement uncertainty of repeatability ( $u(h_{repeat})$ ) is calculated by the standard displacement deviation of indication error of three experiments, i.e.

$$u(h_{repeat}) = \left[ \sum_{i=1}^{n} \frac{(\Delta h_i - \Delta \bar{h})}{n-1} \right]^{1/2} / \sqrt{n},\ n = 3 \qquad (17)$$

where $\Delta \bar{h}$ is the mean value of displacement of the three experiments for displacement calibration. The relative standard displacement uncertainty of nanoindentation system ( $u(h)/h$ ) evaluated in this investigation is shown in Table 2, and it varies with the displacement value.

Table 2 The experimental result for displacement calibration

| h (nm) | $\bar{h}$ (nm) | $\bar{I}$ (nm) | $\overline{\Delta I}$ (nm) | $u(I_L)$ (nm) | $u(h_{repeat})$ (nm) | $u(\Delta I)$ (nm) | u(h) (nm) | u(h)/h (%) |
|---|---|---|---|---|---|---|---|---|
| 50 | 51.040 | 54.959 | 3.918 | 1.951E-08 | 1.46745 | 3.91824 | 4.57101 | 9.14202 |
| 100 | 99.179 | 103.545 | 4.366 | 3.6758E-08 | 0.72226 | 4.36583 | 4.79274 | 4.79274 |
| 150 | 149.627 | 152.132 | 3.687 | 5.4007E-08 | 1.49670 | 3.68710 | 4.38440 | 2.92293 |
| 200 | 197.293 | 201.515 | 5.939 | 7.1538E-08 | 3.06209 | 5.93926 | 6.93104 | 3.46552 |
| 250 | 249.339 | 252.968 | 3.629 | 8.9804E-08 | 0.77891 | 3.62901 | 4.14302 | 1.65721 |
| 300 | 299.728 | 303.467 | 3.738 | 1.0773E-07 | 0.46246 | 3.73817 | 4.19237 | 1.39746 |

### 2.3 Area function calibration and uncertainty evaluation

The Berkovich indenter and fused silica were employed to calibrate the area function of indenter tip. The loads were increasingly applied on the surface of fused silica from 50 μN to 5000 μN at the distinct locations while the displacement and load were simultaneously recorded. Details of the experimental procedure can be found in Ref. [7]. Because the projected area using to calculate the indentation hardness and reduced modulus is provided from the fitting curve in the further, the standard uncertainty of area function ( $u(A)/A$ ) corresponding to their contact depth is calculated from the area difference between fitting curve and data point divided by the area of data point. If the uncertainty evaluation is claimed to that the contact depth is deeper than 100 nm, and it is

$$u(A)/A = 2.262868\ \% \qquad (18).$$

### 2.4 Contact stiffness calibration and uncertainty evaluation

The uncertainty expression of contact stiffness ( $u(S)$ ) can be

$$[u(S)]^2 = \left[ mK(m\text{-}1)(h_{max}\text{-}h_p)^{m\text{-}2} \right] \left[ u(h_{max})^2 + u(h_p)^2 \right] \qquad (19)$$

where $u(h_{max})$ and $u(h_p)$ are the standard uncertainties of the maximum displacement and residual depth, respectively. The $u(h_{max})$ and $u(h_p)$ can be obtained referred to the standard displacement uncertainty in Table 2. The Berkovich indenter and fused silica were also employed to determine the standard uncertainty of contact stiffness. The amount of indentation is 20 times, and the loads were increasingly applied from 500 μN to 10,000 μN with the same increasement. If the uncertainty evaluation is claimed to that the contact depth is deeper than 100 nm, the relative standard uncertainty of contact stiffness ( $u(S)/S$ ) is

$$u(S)/S = 3.195891\ \% \qquad (20).$$

### 2.5 Uncertainty evaluation of indentation hardness

The uncertainty expression of indentation hardness can be expressed as

$$[u(H_{IT})]^2 = \left[ \frac{1}{A_P} u(F_{max}) \right]^2 + \left[ \frac{F_{max}}{A_P{}^2} u(A_P) \right]^2 \qquad (21).$$

The relative standard uncertainty of indentation hardnes ( $\frac{u(H_{IT})}{H_{IT}}$ ) can be calculated by re-arranging this equation and the aforementioned results, i.e.

$$\left[ \frac{u(H_{IT})}{H_{IT}} \right]^2 = \left[ \frac{u(F_{max})}{F_{max}} \right]^2 + \left[ \frac{u(A_P)}{A_P} \right]^2 = [0.0221]^2 + [0.02262868]^2 = 0.0010005 \qquad (22)$$

Thus provided that the maximum displacement is deeper than 100 nm, the relative standard uncertainty of indentation hardness corresponding to a level of confidence of 95 % ( $U_H$ ) is

$$U_H = 2 \times \left[ \frac{u(H_{IT})}{H_{IT}} \right] = 6.3\% \qquad (23).$$

### 2.6 Uncertaint evaluation of reduced modulus

The uncertainty expression of reduced modulus can be expressed as

$$[u(E_r)]^2 = \left[ \frac{\sqrt{\pi}}{2} \frac{1}{\sqrt{A_P}} u(S) \right]^2 + \left[ \frac{\sqrt{\pi}}{4} \frac{S}{A_P{}^{3/2}} u(A_P) \right]^2 \qquad (24).$$

The relative standard uncertainty of reduced modulus ( $\frac{u(E_r)}{E_r}$ ) can be calculated by re-arranging this equation and the aforementioned results, i.e.

$$\left[ \frac{u(E_r)}{E_r} \right]^2 = \left[ \frac{u(S)}{S} \right]^2 + \left[ \frac{1}{2} \frac{u(A_P)}{A_P} \right]^2 = [0.03195891]^2 + [\frac{0.02262868}{2}]^2 = 0.001145 \qquad (25).$$

Thus provided that the maximum displacement is deeper than 100 nm,the relative standard uncertainty of reduced modulus corresponding to a level of confidence of 95 % ( $U_{Er}$ ) is

$$U_{Er} = 2 \times \left[ \frac{u(E_r)}{E_r} \right] = 6.8\,\% \qquad (26).$$

## 3. Conclusion

The uncertainties of indentation hardness and reduced modulus for the nanoindentation measurement system investigated in this paper were evaluated. A traceable electronic balance and an optical interferometric system were respectively employed to calibrate the force and displacement of naoindentation measurement system. Furthermore, the uncertainty expressions were derived in this study. The results show that the relative expanded uncertainties of the indentation hardness and reduced modulus corresponding to a level of confidence of 95 % were respectively 6.3 % and 6.8 % provided that the maximum dsiplacent during indentation is deeper than 100 nm. The result enables one to understand that the measurment difference among investigation for the indentation hardness as well as reduced modulus is meaningful or just results from system uncertainty. Although the system uncertainty depends on the different system to be utilized, the proposed methodology gives the detailed insight into the uncertainty evalution of nanoindentation measurement system.

REFERENCES

1. ISO 14577-1：Metallic Materials-Instrumented Indentation Test for Hardness and Materials Parameters-Part 1: Test Method, 2002.
2. ISO 14577-2：Metallic Materials-Instrumented Indentation Test for Hardness and Materials Parameters-Part 2: Verification and Calibration of Testing Machines, 2002.
3. ISO 14577-3：Metallic Materials-Instrumented Indentation Test for Hardness and Materials Parameters-Part3: Calibration of Reference Blocks, 2002.
4. Guide to the Expression of Uncertainty in Measurement, International Organization for Standardization (ISO), 1995.
5. Testing Report, Center for Measurement Standards, No. 960828, 2007. (In Chinese)
6. R. K. Chang, Final Report for the Investigation of Re-measurement of absolute gravity and measurement of relative gravity, Center for Measurement Standards, 1994. (In Chinese)
7. TriboIndenter Users Manual, Hysitron Inc., 2001.
8. Calibration Report, National Measurement Laboratory, No. B960384, 2007. (In Chinese)

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