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Uncertainty Evaluation of the Thin-Film Indentation System

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Abstract – Center for Measurement Standards (CMS) had established a thin-film indentation testing system based on Akashi MZT-522. The electronic balance was made use of calibrating the testing force and the internal depth sensor was calibrated by laser hologauge. Five other uncertainty was also evaluated in the paper. Finally, the uncertainty of the thin-film indentation testing system reached a total of 5.02 %.

Keywords: Uncertainty, Indentation.

1. INTRODUCTION

Depth-sensing indentation (DSI) is a popular technique for estimating the elastic, plastic, and fracture properties of metals, ceramics, polymers, biological materials and thin films. In contrast to traditional hardness testing systems, DSI instruments determine hardness and material properties by means of continuous monitoring of the force and the depth of indentation over a complete loading cycle. Additionally, the extremely small force and displacement resolutions (i.e. lower than micronewton and nanometre respectively) are combined with very large ranges of testing load and depth, from millinewton to micronewton and from micrometer to nanometre respectively, to permit the determination of mechanical properties of all types of materials by one single instrument.

In Taiwan, with developments in computer science and technology, microelectronics, thin films, precision engineering, and micro-mechanics, many engineering components with small scales and tight tolerances must be measured with high accuracy to ensure their mechanical properties. A Nano Indenter XPW (MTS Systems Corp.) was purchased in 2003 and set up at NanoTechnology Research Center, a unit of Industrial Technology Research Institute, to fulfill industrial demands. Regarding the characterization of the nano-scale materials, the role of CMS is to provide the traceability and the service of instrument calibration. A micro-zone testing system, model MZT-522 manufactured by Akashi Corp., was purchased and established in 2002 and a TriboIndenter (Hysitron Corp.) is now considered to be set up as the standard measurement system before the end of this year.

The investigation into the uncertainty evaluation of MZT-522 facilitates the realization of the coming standard measurement system, even though its resolutions are not really perfect. In this paper, the procedure and analysis of the uncertainty evaluation of DSI instrument MZT-522 are given. The result shows that the relative expanded uncertainty (k=2) is 5.02 %.

2. EXPERIMENT BACKGROUND

2.1. System setup

MZT-522 consists of the indenter, the microscope, the indent screen, the controller, and the central control PC. Related conditions of the indentation were set in the central control PC. The measurement process and data are respectively controlled and captured by the controller. The data are analysed by the central PC. In addition, the indentation could be observed by the microscope and displayed on the indent screen. The system was showed in Fig. 1.

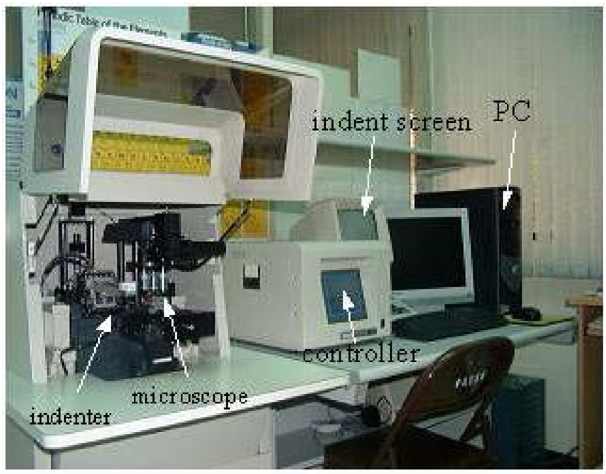

Fig. 1. Akashi MZT-5 system setup

The magnitude of loading force can be applied from 0.098 mN to to 980 mN and its resolution is 0.916 μN. The travel of penetration depth ranges from 0 μm to 20 μm and its resolution was 0.1 nm. The mechanism of load force is showed in Fig. 2. The load lever was born in a balanced position by the frictionless support. The loading force was produced by the load generator and the force was transferred through the load lever to the indenter, which was thus depressed into the specimen. The amount of the depression, or indentation was measured by the indentation depth gauge [1].

The standard indenter for the MZT-522 system is the triangular pyramid indenter, Berkovich indenter, and its constituent material is diamond. The geometrical configuration is illustrated in Fig. 3.

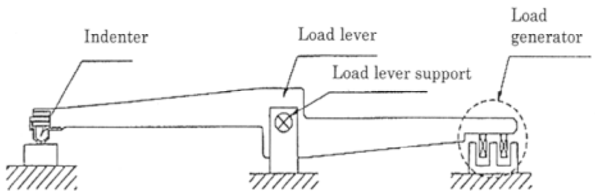

Fig. 2. Load application of test force mechanism [2]

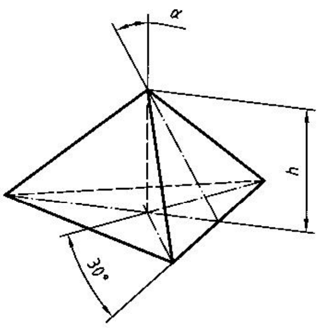

Fig. 3. Berkovich indenter [3]

2.2. Measurement theorem

In the testing procedure, the loading force, F, was first increased until both of the maximum force, F_{max}, and penetration depth, h_{max}, simultaneously reached. Then, the test force was gradually released down to zero. The data of testing force versus penetration depth were collected and the typical schematic is shown in Figs. 4 and 5.

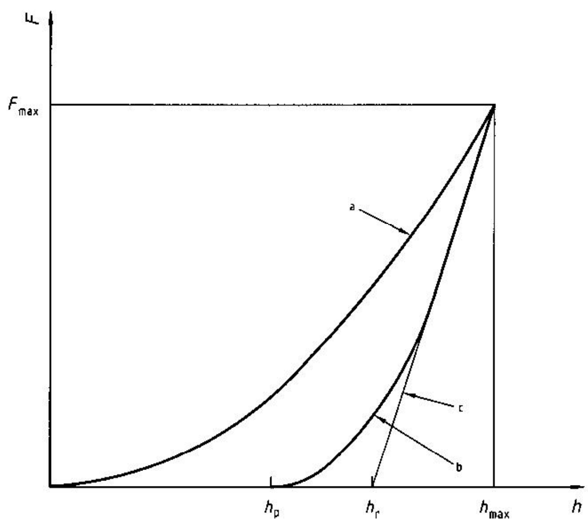

Fig. 4. The load (F) and the penetration (h) relation [3]

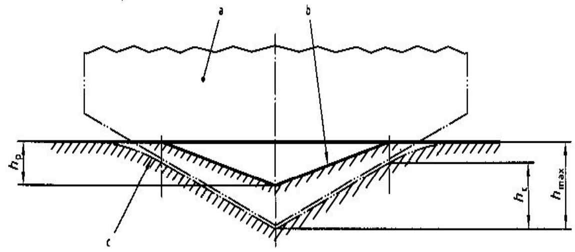

Fig. 5. The indent profile [3]

Martens hardness (HM) was chosen for the measurement scale in the system. Martens hardness (HM) is defined as follows [3]:

$$HM = \frac{F}{A_s(h)} \tag{1}$$

where

HM: Martens hardness unit;

F: the test force in N;

$A_s(h)$: the surface area of the indenter penetrating in mm^2.

In Fig. 3, the surface area, $A_s(h)$, penetration, h, and the angle between indentation direction and pyramidal surface, α, have the following relationship.

$$A_s(h) = \frac{3\sqrt{3} \times \tan\alpha}{\cos\alpha} \times h^2 \tag{2}$$

In this paper, the angle , α, is 65.03° and substitute (2) into (1). It gives

$$HM = \frac{F}{26.43 \times h^2} \tag{3}$$

3. UNCERTAINTY ANALYSIS

From aforementioned equations and [4], there were five parameters in the uncertainty evaluation as follows:

a. force measurement error (ΔF) of the force sensing system.
b. the penetration depth measurement error (Δh) of the depth sensing system.
c. the indenter angle error $(\Delta \alpha)$
d. the initial zero-position error (Δh_0) of the depth sensing system.
e. the tip radius error (Δr) of the indenter.
f. the compliance error (Δh_m).
g. the area function error (Δh_A).

3.1. Force measurement error (ΔF) uncertainty

From (3), it gives

$$\left(\frac{\Delta HM}{HM}\right)_{\Delta F} = \frac{\Delta F}{F} \tag{4}$$

The electronic balance, model WZ215-CW produced by Sartorius Corp., was employed to calibrate the force sensor of MZT-522. First, the balance was fixed on the table of MZT-522. Ten calibration points were selected, i.e. 0.1 gf, 0.2 gf, 0.5 gf, 1 gf, 2 gf, 5 gf, 10 gf, 20 gf, 50 gf and 100 gf. For every point, three experiments were performed. The indenter was lowered to press the pan of WZ215-CW until its reading reaches to the calibration point. At this time, the reading of MZT-522 recorded. Here, the maximum force relative uncertainty was adopted among ten calibration points [5]. Hence,

$$\left(\frac{\Delta HM}{HM}\right)_{\Delta F} = 7.66E-4 \tag{5}$$

In this paper, local gravity g is 9.78914 and the standard gravity is 9.80665. The uncertainty caused by gravity is 0.2%, and thus it can be neglected.

3.2. Depth measurement error (△h) uncertainty

From (3), it yields

$$\left(\frac{\Delta HM}{HM}\right)_{\Delta h} = -2\times\frac{\Delta h}{h} \tag{6}$$

The laser hologage, model L-DD-01 produced by TOKYO Lazax Corp., was employed to calibrate the depth sensor of MZT-522. Antecedently, the hologage sensor was fastened to focus on the moving stage installed on the table of MZT-522. The moving stage is model P-611.3S fabricated by Physik Instrumente Corporation. Six calibration points were selected i.e. 0.5 µm, 1.0 µm, 1.52 µm, 2.52 µm, and 3.02 µm. By comparing readings of L-DD-01 and MZT-533 at these positions, the parameters of MZT-522 control software are modified. Finally, the maximum depth relative uncertainty was utilized among six calibration positions [6]. Hence,

$$\left(\frac{\Delta HM}{HM}\right)_{\Delta h} = 1.39E-2 \tag{7}$$

In this paper, hologage was calibrated by use of a semiconductor laser. The related uncertainty we anticipated was small, so it can be neglected.

3.3. Indenter angle error (△ α) uncertainty

From (3), it gives

$$\left(\frac{\Delta HM}{HM}\right)_{\Delta\alpha} = -\left(\frac{1+\sin^2\alpha}{\sin\alpha\times\cos\alpha}\right)\times\Delta\alpha \tag{8}$$

Substituting α (65.03 °) into (8), then

$$\left(\frac{\Delta HM}{HM}\right)_{\Delta\alpha} = -4.755\times\Delta\alpha \tag{9}$$

According to the Berkovich indenter certificate, the angle is 65.03°±15'. It is assumed that the error probability is rectangular distribution. Substitute the related values into (7), then

$$\left(\frac{\Delta HM}{HM}\right)_{\Delta\alpha} = -1.20E-2 \tag{10}$$

3.4. Initial zero-position error (△h_0) uncertainty [2]

The electromagnetic force-based force generator attached to the load lever has two coils. The indenter force coil was used to apply test force load to the specimen. The indenter zero coil moved the indenter closer to the specimen surface.

To detect the specimen surface, the indenter zero coil was applied with minute current in steps. With the application of current, the indenter lowered toward the specimen at a constant rate with each additional step of current, until it touched the specimen. Once the indenter touched the specimen, the rate of lowering of the indenter changes. This point of change (i.e., the point on a plot at which the slope of lowering line changed) was taken as the specimen contact point (the initial zero-position).

Considering the initial zero-position *(h_0)* of the depth sensing system, (3) can be rewritten as follows:

$$HM = \frac{F}{26.43\times(h-h_0)^2} \tag{11}$$

From (11), it gives

$$\left(\frac{\Delta HM}{HM}\right)_{\Delta h_0} = 2\times\frac{\Delta h_0}{h} \tag{12}$$

The test specimen was polished in advance and it is assumed the zero-position error due to surface roughness is 3 nm. Substitute this value into (12), so

$$\left(\frac{\Delta HM}{HM}\right)_{\Delta h_0} = 2.00E-3 \tag{13}$$

3.5. Tip radius error (△r) uncertainty

In fact, the indenter tip is not perfect. Instead of the ideal shape, a ball shape was assumed. A profile is shown in Fig. 6. The penetration depth of the indenter mentioned above was h′ and the ball radius was r. It is assumed that the ball tip was tangent with the indenter surface, thus

$$\frac{r}{r+(h-h')} = \sin\alpha \tag{14}$$

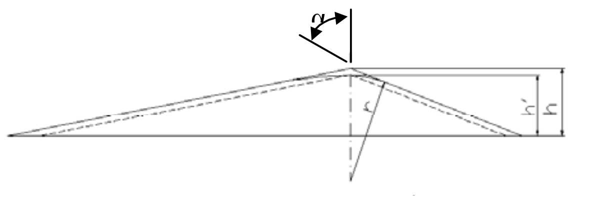

Fig. 6. The profile of the ball shape tip indenter

Substitute α=65.03 ° and (14) into (3), it gives

$$HM = \frac{F}{26.43 \times (h' + 0.103109r)^2} \qquad (15)$$

From (15), it yields

$$\left(\frac{\Delta HM}{HM}\right)_{\Delta r} = \frac{-2 \times 0.103109}{h} \times \Delta r \qquad (16)$$

The radius error was assumed to be ±0.25 μm, meanwhile, the penetration depth reached 3 μm. If the error probability was rectangular distribution, substitute these values into (16). It gives

$$\left(\frac{\Delta HM}{HM}\right)_{\Delta r} = -9.92E-3 \qquad (17)$$

3.6. Compliance error (Δh_m) uncertainty [7]

Consider the penetration depth influence (h_m) of the instrument compliance of the depth sensing system, it can be rewritten (3) as follows:

$$HM = \frac{F}{26.43 \times (h - h_m)^2} \qquad (18)$$

From (18), it gives

$$\left(\frac{\Delta HM}{HM}\right)_{\Delta h_m} = 2 \times \frac{\Delta h_m}{h} \qquad (19)$$

In this paper, the tested specimen is fused quartz and its Young's modulus and Poisson's ratio are 72 GPa and 0.170, respectively. Fifteen indentations experiments were carried out and the maximum load range was from 60 mN to 900 mN with the identical difference of 60 mN. The loading and unloading data were recorded and the following power equation was used to fit the unloading data.

$$P = \beta(h - h_f)^m \qquad (20)$$

where P is indenter load, β and m are the undetermined constants, h is the penetration depth and h_f is the contact depth when indenter load is fully released. Then, the following relationship was utilized to calculate the contact depth, h_c, and the corresponding area, A.

$$A = \frac{\pi}{4}\left(\frac{S}{E_r}\right)^2 \qquad (21)$$

where S is the initial unloading contact stiffness and E_r is the reduced modulus.

To account for elastic displacement of the load-frame of the instrument, the machine compliance, C_m must be added to the contact compliance C_c. The total compliance of the testing system is

$$C_{total} = C_m + C_c = C_m + \frac{\sqrt{\pi}}{2E_r\sqrt{A}} \qquad (22)$$

Finally, a plot of *1/measured stiffness* versus $A^{-0.5}$ for 15 indents was plotted and a linear equation was used to fit the experimental results. From (22), it is easy to understand that the machine compliance can be obtained from the intercept of *1/measured stiffness* axis. In this paper, calculated machine compliance is 1.1 nm/mN. According to authors' earlier work [5], the maximum force error is 3.83E-1 mN. It is assumed that the penetration depth was 3 μm and substitute theses values into (19). Then

$$\left(\frac{\Delta HM}{HM}\right)_{\Delta h_m} = 2.81E-4 \qquad (23)$$

3.7. Area function error (Δh_A) uncertainty

In this paper, the following area function was adopted [7] to describe the blunting of the indenter tip:

$$A(h_c) = C_0h_c^2 + C_1h_c + C_2h_c^{1/2} + C_3h_c^{1/4} + C_4h_c^{1/8} + C_5h_c^{1/16} \qquad (24)$$

Where C_i are the fitting constants. Substitute (24) into (1), then

$$\left(\frac{\Delta HM}{HM}\right)_{\Delta h_A} = \frac{-\left(2C_0h_c + C_1 + \frac{C_2}{2}h_c^{\frac{-1}{2}} + \frac{C_3}{4}h_c^{\frac{-3}{4}} + \frac{C_4}{8}h_c^{\frac{-7}{8}} + \frac{C_5}{16}h_c^{\frac{-15}{16}}\right) \times \Delta h}{\left(C_0h_c^2 + C_1h_c + C_2h_c^{\frac{1}{2}} + C_3h_c^{\frac{1}{4}} + C_4h_c^{\frac{1}{8}} + C_5h_c^{\frac{1}{16}}\right)} \qquad (25)$$

From the antecedent experiments, a plot of calculated contact depth versus area was plotted and tip area function was obtained as follows:

$$A = 26.43h_c^2 + (4.714E-3)h_c - (6.815E-5)h_c^{1/2} + (9.805E-6)h_c^{1/4} - (2.946E-6)h_c^{1/8} + (6.574E-7)h_c^{1/16} \qquad (26)$$

It is assumed that the error probability was rectangular distribution. From [6], substitute these values and (26) into (25). The maximum relative uncertainty is

$$\left(\frac{\Delta HM}{HM}\right)_{\Delta h_A} = 1.39E-2 \qquad (27)$$

3.8. Calculation of relative combined standard uncertainty and relative expanded uncertainty

First, the relative combined standard uncertainty (u_{rc}) was calculated by collecting all aforementioned uncertainty.

$$u_c = \sqrt{\begin{aligned}&(7.66E-4)^2 + (1.39E-2)^2 + \\ &(-1.20E-2)^2 + (2.00E-3)^2 + \\ &(-9.92E-3)^2 + (2.81E-4)^2 + \\ &(1.39E-2)^2\end{aligned}} = 2.52E-2 \qquad (28)$$

Finally, the coverage factor *(k)* is 2 at the confidence of level of 95%. Hence, the relative expanded uncertainty (U_{exp}) can be obtained as follows:

$$U_{exp} = k \times u_{rc} = 2 \times (2.52E-2) = 5.04E-2 \qquad (29)$$

4. RESULTS AND DISSCUSSION

The results are showed in TABLE I. When the penetration depth was less than 3.0 µm, the relative expanded uncertainty of MZT-522 is 5.04 %. This was close to the Hermann's result [4] by use of Vickers indenter in PTB.

TABLE I. Total uncertainty analysis

Uncertainty	Probability distribution	Degrees of freedom	Relative standard uncertainty
force	t	4.9E5	7.66E-4
depth	rectangular	∞	1.39E-2
angle	rectangular	∞	-1.20E-2
zero-position	rectangular	∞	2.00E-3
radius	rectangular	∞	-9.92E-3
compliance	rectangular	∞	2.81E-4
area function	rectangular	∞	1.39E-2
The relative combined standard uncertainty (u_{rc})			2.52 %
The effective degrees of freedom (ν_{eff})			≈ ∞
The coverage factor (k)			2
The relative expanded uncertainty (U_{exp})			5.04 %

The results show that the uncertainty is strongly influenced by the depth and area function error. Besides, they are affected deeply by the depth measurement. Hence, it is suggested that improving the depth sensing is the appropriate strategy to reduce relative expanded uncertainty of MZT-522. In addition, the approximation of the indent surface area was adopted. A more precision measurement could be further employed to improve the uncertainty.

5. CONCLUSION

In this paper, the uncertainty of a thin-film indentation testing system, Akashi MZT-522, was evaluated. The results show that the uncertainty of MZT-522 system is 5.02%. Besides, it is suggested that improving the depth sensing is the appropriate strategy to reduce relative expanded uncertainty of MZT-522.

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