XIX IMEKO World Congress
Fundamental and Applied Metrology
September 6−11, 2009, Lisbon, Portugal

SUB-MILLIGRAM WEIGHT SUBDIVISION AND APPLICATION IN FORCE CALIBRATION OF NANOINDENTER

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Abstract − In this time of flourishing nanotechnology research and the miniaturization of relevant products, the mechanical properties, such as hardness and tensile strength, of relevant materials must be tested in order to ensure product reliability. Because such testing requires the measurement of miniscule forces, relevant testing systems must be traceable to tiny masses of less than one milligram. This paper chiefly investigates induction of the use of a set of weights ranging from 1 mg to 0.1 mg, and describes how 0.5 mg, 0.2 mg, and 0.1 mg weights can be traced from a 1 mg standard weight. This paper explains how such a weight set can be used to calibrate a nanoindenter in micro-forces range and estimate the uncertainties.

Keywords: subdivision, traceability, nanoindenter

1. INTRODUCTION

The center for measurement standards (CMS, Taiwan) has consequently established a micro-/nano mechanical system to measure the properties of material. Our system comprises two sets of equipment [5, 7]: one is the micro-/nano tensile testing system and the other is the nanoindenter. The nanoindenter micro-force measurements are traceable to micro-masses. As we used the smallest traceable standard mass unit is 1 mg, so we have consequently formulated a sub-milligram weight set, it's 552211 specification comprising 0.5 mg, 0.2 mg, and 0.1 mg weights (weights comply with OIML R111[4] requirements), to derive these masses. Using this process, we can ensure that the measurement of micro-forces can traceable to SI units in the application of nanoindenter.

We use a microbalance (METTLER／UMT5) with 5 g capacity and 0.1 μg resolution to measure the nanoindenter in micro-forces ranging from 1 μN to 10 μN. The microbalance was calibrated using sub-milligram weight set traceable to IPK. The mass subdivition method was used to define the mass values of the sub-milligram weights by 1 mg. The 4 weights of 0.1 mg, 0.2 mg, 0.5 mg, and 1 mg were used for micro-forces measured ranging from 1 μN to 10 μN.

The traceability chain for micro-forces measured is shown in Fig. 1.

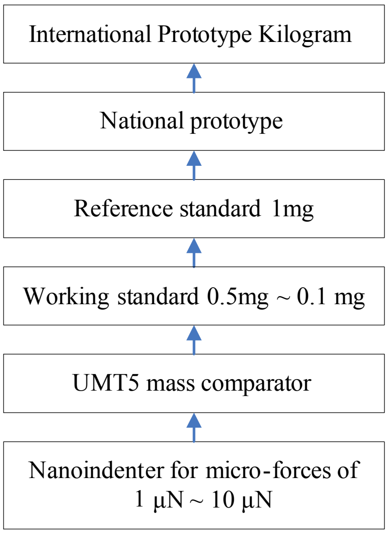

Fig. 1. Traceability chain for micro-forces measured

2. CALIBRATION METHODS

2.1. Mass subdivision method

A complete sub-milligram weight set consists of seven weights (k=7), 1 mg, 0.5 mg, 0.5 mg*, 0.2 mg, 0.2 mg*, 0.1 mg, and 0.1 mg*. The 1 mg as a reference weight, and 10,5,5,2,2,1,1was used as the weighting design[1,2,3], comparison was performed ten times, n=10, the ten observed values is $y_1, y_2, \cdots y_n$, which are the mass difference $\Delta m_i = (m_A - m_B)_i$.

The weighing cycle ABBA was used, the number of weighing cycles is six cycles. The effect of air buoyancy is minimal, where air buoyancy correction is estimated to be negligible.

Letting the observed values $y_i = \Delta m_i$, and the masses of *k* weights $\beta_1, \beta_2, \cdots \beta_k$, we used the least squares method in conjunction with the NBS T.N.952 [1] matrix model to obtain the estimated mass difference for individual weights. X is the replacement weight position and order matrix in the assessment process. The weighing scheme matrix is as follows:

$$X\beta = \begin{bmatrix} +1 & -1 & -1 & 0 & 0 & 0 & 0 \\ 0 & +1 & -1 & 0 & 0 & 0 & 0 \\ 0 & +1 & 0 & -1 & -1 & -1 & 0 \\ 0 & 0 & 0 & +1 & -1 & 0 & 0 \\ 0 & 0 & 0 & +1 & 0 & -1 & -1 \\ 0 & 0 & 0 & 0 & 0 & +1 & -1 \\ 0 & +1 & 0 & -1 & -1 & 0 & -1 \\ 0 & 0 & 0 & +1 & -1 & 0 & 0 \\ 0 & 0 & 0 & 0 & +1 & -1 & -1 \\ 0 & 0 & 0 & 0 & 0 & +1 & -1 \end{bmatrix} \begin{bmatrix} \beta_1 \\ \beta_2 \\ \beta_3 \\ \beta_4 \\ \beta_5 \\ \beta_6 \\ \beta_7 \end{bmatrix} \tag{1}$$

The observed values is:

$$y = \begin{bmatrix} -0.0000800 \\ -0.0004800 \\ -0.0013400 \\ -0.0015800 \\ -0.0009500 \\ -0.0016700 \\ -0.0022900 \\ -0.0039900 \\ +0.0005600 \\ -0.0021700 \end{bmatrix} \tag{2}$$

$$y = X\beta + e \tag{3}$$

The estimates of the unknown masses, $\hat{\beta}$, are calculates as:

$$\hat{\beta} = \left(X^T X\right)^{-1} X^T y \tag{4}$$

Because $\left(X^T X\right)$ is a singular matrix, its inverse matrix $\left(X^T X\right)^{-1}$ does not exist, and it can therefore be solved by imposing a constraint

$$r_1\beta_1 + r_2\beta_2 \cdots\cdots + r_k\beta_k = m_R \tag{5}$$

m_R : mass of reference weights, and r_i are matrix coefficients of the reference weights used in the assessment design. Lagrangian multipliers and the least squares method are used as follows to obtain the extreme values:

$$\Phi(\beta,\lambda) = \sum (e_i)^2 + 2\lambda\left(r_1\beta_1 + \cdots + r_k\beta_k - m_R\right) \tag{6}$$

The matrix can be expressed as:

$$\begin{bmatrix} X^T X & r \\ r^T & 0 \end{bmatrix} \begin{bmatrix} \hat{\beta} \\ \lambda \end{bmatrix} = \begin{bmatrix} X^T y \\ m \end{bmatrix} \tag{7}$$

Let

$$\begin{bmatrix} X^T X & r \\ r^T & 0 \end{bmatrix}^{-1} = \begin{bmatrix} C & h \\ h^T & 0 \end{bmatrix} \tag{8}$$

Then The estimates of the unknown masses, $\hat{\beta}$, is

$$\hat{\beta} = \begin{bmatrix} CX^T & h \end{bmatrix} \begin{bmatrix} y \\ m_R \end{bmatrix} \tag{9}$$

2.2. micro-forces calibration

The micro-forces measurement of nanoindenter was completed by indenter tip loaded in the center of the weighing pan. Four forces loading of 1 μN, 2 μN, 5 μN and 10 μN was set because it correspond to weights of 0.1 mg, 0.2 mg, 0.5 mg, and 1 mg, the number of each loading measurement is 3 times. The X-Y table of nanoindenter must was removed, then the UMT5 microbalance was placed on the platform of nanoindenter as shown in Fig. 2.

The calibration model is

$$\mathrm{F} = \mathrm{I} \times \mathrm{g} + \mathrm{d}_\mathrm{F} \tag{10}$$

The F is loading of nanoindenter, the I is reading of average of microbalance, the g is gravity, the d_F is Error value.

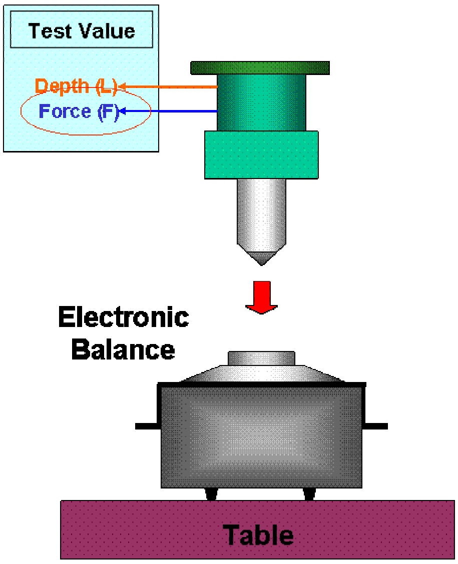

Fig. 2. The skeleton diagram of forces calibration of nanoindenter

3. UNCERTAINTY ANALYSIS

3.1. Sub-milligram weigh

Referring to OIML R111 Part I [4], the main sources of measurement uncertainty consist of weighing uncertainty $u_w(\beta_j)$, reference weight uncertainty $u_r(\beta_j)$, air buoyancy uncertainty u_B, and the balance uncertainty u_{ba}. These are described as follows. Air buoyancy uncertainty u_B has very little effect on this experiment and can therefore be neglected.

The equation (9), where h is the weight ratio used in subdivision. The C matrix is an important matrix, because the variance-covariance matrix of weights can be expressed as

$$V_\beta = s^2 C \tag{11}$$

Where the degree of freedom of the weighing process $f = n - k + r$, and r is the number of reference weights.

In the case of each weight, the uncertainty contributed by combined weighing can be estimated using the variance-covariance matrix V_β. Matrix V_β is a square symmetrical matrix; its diagonal elements v_{jj}, j=1…k are the variances of the individual weights β_j. As a consequence, the weighing uncertainty $u_w(\beta_j)$ of the individual weights β_j is equal to the square root of v_{jj}:

$$u_w(\beta_j) = \sqrt{v_{jj}},\ j = 1 \ldots k \tag{12}$$

The reference weight uncertainty consists of the root-mean-square of twice the uncertainty derived from the reference weight plus the weight stability uncertainty:

$$u(m_r) = \sqrt{\left(\frac{U(m_r)}{k}\right)^2 + \left(\frac{D(m_r)/2}{\sqrt{3}}\right)^2} \tag{13}$$

In this equation, $U(m_r)$ is expanded uncertainty in the reference weight traceability report, and k is the reference weight expansion coefficient.

When the combined assessment method is used to perform weighing, the effect of reference weight uncertainty $u(m_r)$ on each weight is allocated in accordance with the ratios of the nominal values of the weights M_j, M_r. h is therefore the ratio matrix, with elements $h_j = M_j / M_r$. The uncertainty for each weight due to the reference weight during weighing is therefore:

$$u_r(\beta_j) = h_j u(m_r),\ j = 1 \ldots k \tag{14}$$

Uncertainty due to the balance is affected by the balance display resolution (d). A rectangular distribution is employed to estimate the balance resolution uncertainty u_d

$$u_{ba} = \left(\frac{d/2}{\sqrt{3}}\right) \times \sqrt{2} \tag{15}$$

The foregoing types of uncertainty are mutually independent and uncorrelated. As a result, the total uncertainty of the weight will be [6]

$$u_c(\beta_j) = \sqrt{u_w^2(\beta_j) + u_r{}^2(\beta_j) + u_{ba}{}^2(\beta_j)},\ j = 1 \ldots k \tag{16}$$

Taking k=2 as the expansion coefficient, the expanded uncertainty will be

$$U(\beta_j) = k u_c(\beta_j) \tag{17}$$

Sub-milligram weight measurement results are as shown in Table 1.

Table 1. Sub-milligram weight measurement results

nominal		0.1mg.	0.1mg	0.2mg.	0.2mg	0.5mg.	0.5mg
mass(mg)		0.1012	0.0995	0.2018	0.1993	0.5002	0.4997
u_w (mg)	A	0.00047	0.00047	0.00053	0.00053	0.00071	0.00071
u_r(mg)	B	1.1E-05	1.1E-05	2.1E-05	2.1E-05	5.3E-05	5.3E-05
u_{ba}(mg)	B	0.00004082					
u_B(mg)	B	negligible					
u_c(mg)		0.00047	0.00047	0.00053	0.00053	0.00072	0.00072
k		2	2	2	2	2	2
U(mg)		0.00095	0.00095	0.00107	0.00107	0.00143	0.00143

3.2. micro-forces of nanoindenter

The calibration of microbalance was executed on eccentric loading, repeatability and linearity. The 0.1 mg, 0.2 mg, 0.5 mg, and 1 mg of microbalance were selected as calibration point to reduced human error. The measurement of eccentric loading and repeatability selected 1 mg as a measurement point for conservative estimate uncertainty. Generally the balance used to direct read the display, so the error of balance was estimated into uncertainty of microbalance. The uncertainty of microbalance includes eccentric loading, repeatability, microbalance error and reference weight, as shown in Table 2.

Table 2. Uncertainty analysis for the balance (mg)

Weight (nominal)			0.1	0.2	0.5	1
Repeatability	u_R	A	0.00012	0.00012	0.00012	0.00012
Error	Value		0.00094	0.00076	0.00042	-0.00002
	u_ε	B	0.00027	0.00022	0.00012	0.00001
eccentric loading	u_E	B	0.00002	0.00002	0.00002	0.00002
Reference weight	u_r	B	0.00047	0.00054	0.00072	0.00028
	u_c		0.00056	0.00059	0.00074	0.00030
	k		2	2	2	2
	U		0.0012	0.0012	0.0015	0.00061

The uncertainty of micro-forces measurement of nanoindenter has been estimated following sources:

1. Standard uncertainty of microbalance
2. Standard uncertainty of loading repeatability
3. Standard uncertainty of error of nanoindenter measurement

Due to practical considerations, the error of nanoindenter didn't correction and the nanoindenter error is included uncertainty sources. Assuming that the nanoindenter error has a rectangular distribution, the uncertainty analysis of the nanoindenter is listed in Table 3.

Table 3. Uncertainty analysis for nanoindentor in micro-forces

Force		(μN)	1	2	5	10
Repeatability		(mg)	5.5E-04	2.3E-03	3.0E-03	1.8E-03
Repeatability Standard uncertainty	A	(μN)	5.4E-03	2.3E-02	2.9E-02	1.8E-02
Balance uncertainty		(mg)	1.2E-03	1.2E-03	1.5E-03	6.1E-04
Balance Standard uncertainty	B	(μN)	5.9E-03	5.9E-03	7.3E-03	3.0E-03
Error value		(μN)	-3.4E-02	-5.5E-02	-6.3E-02	-1.9E-01
Error Standard uncertainty	B	(μN)	9.7E-03	1.6E-02	1.8E-02	5.6E-02
Combined Standard uncertainty		(μN)	1.3E-02	2.9E-02	3.5E-02	5.9E-02

5. CONCLUSIONS

With regard to assessment of the uncertainty of sub-milligram weight induction results, it can be seen from the sub-milligram weight measurement results in Table 1 that almost all uncertainty is due to weighing. This is chiefly because of the difficulty of working with small sub-milligram weights, human error, and the effect of air currents. Although strenuous efforts are made to control the environment and prevent human error during calibration, experimental results are still the most significant source of uncertainty. In addition, the relative nominal value of the combined uncertainty estimated from balance calibration is also relatively high, and this is mainly derived from reproducibility.

With regard to the measurement of micro-forces, the Hysitron TriboIndenter's balance weighing results are increasingly influenced by external factors as the force becomes smaller. This causes reproducibility to deteriorate, and is the largest source of uncertainty. Consequently, when performing micro-force calibration tracing of the system, as can be seen from the TriboIndenter's load uncertainty in Table 3, when the balance uncertainty relative to the reproducibility of the TriboIndenter's load measurements is small, then the combined standard uncertainty u_F of the TriboIndenter's load will be within the acceptable measurement range.

ACKNOWLEDGMENTS

This work is supported by Bureau of Standards, Metrology and Inspection (BSMI), Taiwan, R.O.C.

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