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CRITICAL POINTS IN ISO 14577 PART 2 AND 3 CONSIDERING THE UNCERTAINTY IN MEASUREMENT

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Abstract – Based on the estimation of uncertainty the present requirements of the standard are discussed. Additional requirements needed for improving the reliability and the reproducibility are proposed. Especially the compliance of the machine affects strongly the parameters E_{IT} and HM_s in the macro range.

Keywords: Indentation, standard, uncertainty

1. INTRODUCTION

The ISO standard on the instrumented indentation test for hardness and other materials parameter was published in October 2002 [1]. Additional information on the history and the metrological problems of the standard are given in [2,3]. Appropriate reference materials needed for the verification of the testing machine is the object of the contributions on the micro range [4] and on the macro range [5]. Although the ISO 14577 represents a great progress in the local elastic-plastic characterisation of materials some points for improving the standard have been appeared since the publication. New ideas were given on the determination of the contact point [6], on the force calibration [7], and on the determination of the machine compliance [8]. Based on the estimation of uncertainty in the instrumented indentation testing [9] the present paper is aimed to improve the verification and calibration of testing machines as well as the calibration of reference materials.

2. METROLOGICAL REQUIREMENTS

The main requirements concern the uncertainty of the test force F and the displacement h' because the procedure of the instrumented indentation test is primary aimed to the measurement of the indentation curve (for instance Fig. 1). However, the raw data of the displacement are not the indentation depth h. It is needed to determine the zero depth h_0' (contact point) and to take into account the compliance of the machine C_m. The indentation depth h reads

$$h = h' - h_0' - C_m * F \quad (1)$$

While the ISO 14577 defines an upper limit of the uncertainty of h_0' ($u(h_0') < 0.01 h_{max}$) the requirement for the uncertainty of the machine compliance C_m is missing. The requirements given in the standard are collected in Table I.

TABLE I. Requirements for the verification and calibration of testing machines (ISO 14577 part 2) and for the reference materials (ISO 14577 part 3).

	Range	Part 2	Part 3
Tolerance			
Force	Macro	1.0%	0.25%
Force	Micro	1.5%	0.5%
Maximum permissible error			
Displacement	Macro	1% of h	0.5% of h or 30 nm
Displacement	Micro	1% of h	1% of h or 5 nm
Apex angle	Macro	0.3°	0.15°
Apex angle	Micro	0.3°	0.15°
Repeatability			
Hardness HM, H_{IT}	h>1µm	2%	2%
Indentation modulus E_{IT}	h>1µm	5%	5%

To find further requirements needed for the best reliability and reproducibility of the instrumented indentation test the uncertainty of the parameters calculated from the indentation curve is estimated.

3. FUNCTIONAL RELATIONSHIPS

To start with, the materials parameters of the instrumented indentation test are introduced. Martens hardness HM is directly calculated from the measured test force F and indentation depth $h = h_e + h_c$

$$HM = \frac{F}{A_s(h)} \quad (2)$$

$A_s(h)$ is the surface of the indenter from the tip up to the indentation depth $h=h_e+h_c$, especially $A_s(h)=G_1*h^2$ for pyramids and h>6µm. Using the slope of the indentation curve in the plot square root of force against depth the Martens hardness HM_s reads

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$$HM_s = \frac{\left(\frac{\partial \sqrt{F}}{\partial h}\right)^2}{G_1} \quad (3)$$

The initial unloading slope can also be used for separating the elastic part h_e of the indentation depth $h=h_c+h_e$. In this way the indentation hardness H_{IT} can be calculated as follows

$$H_{IT} = \frac{F_{max}}{A_P(h_c)} \quad with \quad h_c = h_{max} - \frac{\varepsilon F_{max}}{S} \qquad (4)$$

$A_p(h_c)$ is perpendicular to the test force and is named projected area at the plastic indentation depth h_c. In analogy to A_s, $A_p = G_2 * h_c^2$ for pyramids and h>6µm. The constant ε is dependent on the geometry of the indenter (ε=0.75 for Vickers and Berkovich indenter).

The initial unloading slope S (stiffness)

$$S = (\tfrac{dF}{dh})_{h=h_{max}} \qquad (5)$$

allows to calculate the elastic property. The indentation modulu s E_{IT} reads

$$E_{IT} = \frac{1-\nu_s^2}{\frac{1}{E_r} - \frac{1-\nu_i^2}{E_i}} \qquad (6)$$

with

$$E_r = \frac{S * \sqrt{\pi}}{2 * h_c * \sqrt{G_2}} \qquad (7)$$

E_i is the Young's modulus of the indenter, ν_i and ν_s are the Poisson ratio of the indenter and the specimen.

Three interrelations for calculating the uncertainty must be added to (3), (4), and (7). The uncertainty of the initial unloading slope is traced to the uncertainty of the slopes in the calibration of force and displacement.

$$\left(\frac{u(\frac{dF}{dh})}{\frac{dF}{dh}}\right)^2 = (u(\frac{dF}{dF_{St}}))^2 + (u(\frac{dF_{St}}{dh_{St}}))^2 + (u(\frac{dh}{dh_{St}}))^2 \qquad (8)$$

F_{st} and h_{st} are the standards in force and displacement. The uncertainty of the slope $d\sqrt{F}/dh$ is traced to the uncertainty of dF/dh.

$$\frac{dh}{d\sqrt{F}} = \frac{dh'}{dF} 2\sqrt{F} \qquad (9)$$

Finally, in all equations the indentation depth h must be inserted as a function of the raw displacement h' after (1) to take into account the correction by the machine compliance C_m. Using the compliance C instead of the stiffness S=1/C the derivates becomes

$$(\frac{dh}{dF}) = (\frac{dh'}{dF}) - C_m \qquad (10)$$

and

$$\frac{dh}{d\sqrt{F}} = \frac{dh'}{d\sqrt{F}} - 2C_m\sqrt{F} \qquad (11)$$

The reciprocal initial unloading slope reads

$$(\frac{dh}{dF})_{F=F_{max-}} = (\frac{dh'}{dF})_{F=F_{max-}} - C_m = C_T - C_m \qquad (12)$$

and the plastic indentation depth h_c after (7) reads

$$h_c = h'_{max} - h_0' - (1-\varepsilon)F_{max}C_m - \varepsilon F_{max} C_T \qquad (13)$$

Inserting (12) and (13) into (6) the effect of compliance on the resulting modulus E_r is given instead of (6)

$$\frac{\sqrt{\pi}}{2E_r\sqrt{G_2}} = (C_T - C_m)(h'_{max} - h_0 - (1-\varepsilon)F_{max} * C_m - \varepsilon * F_{max} * C_T) \qquad (14)$$

4. SOURCES OF UNCERTAINTY

The macro range, F>2N, is the main object of this paper. Some effective sources of uncertainty are illustrated in Figure 1. Because the indentation curve is a second order polynomial in the macro range (geometrical similarity of the pyramidal indentation) the determination of the zero point h_0 (contact point) can lead to an important source of uncertainty [6, 11]. The compliance of the machine affects not only the indentation depth but also the initial unloading slope. Additional problems concerning the calculation of the initial unloading slope (specific choose of fitting function and fitting range) should be noted.

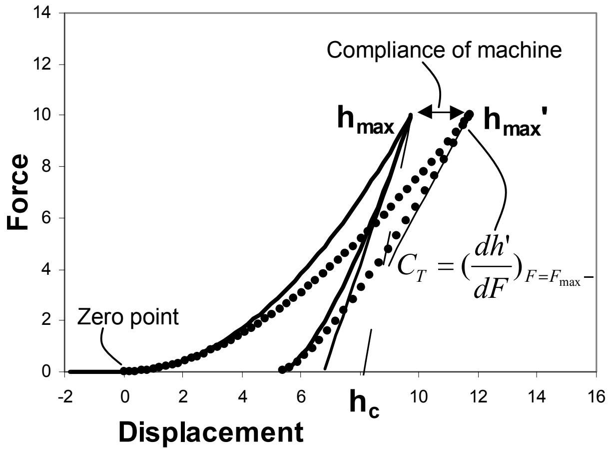

Fig. 1. Additional sources of uncertainty for the materials parameters of the instrumented indentation test

The sources of uncertainty needed for estimating the combined uncertainty of the materials parameters can be concluded from the equations of the last caption. As can be seen the additional input values are the uncertainties of the slopes in the calibration of force and displacement for increasing and decreasing quantities as well as the uncertainty of the compliance. The input values for the calculation of the combined uncertainty are collected in Table II. For simplification, all distributions are assumed to be normally. For instance, the use of rectangular distributions for the force and the displacement do not change the concluded proposals in caption 6.

The uncertainty of the slopes of increasing force and decreasing force were determined by an extended verification of an available testing machine. Regarding the calibration of the displacement at the same machine the results have agreed with the requirements of ISO 14577 part 3 (Table I). The determination of the machine compliance is a separate topic of a paper [8]. As a result the chosen value in Table II, C_m = 0.0005 µm/N, is the smallest one of the realistic range. The uncertainty of the geometrical factors G_1 and G_2 (for the indenter area functions $A_s(h)$ and $A_p(h_c)$ of the ideal Vickers pyramid, h>6µm) was evaluated by the required uncertainty of the apex angle. The uncertainty of ε in Table II represents the reliability of the used model for separating the elastic part of the indentation.

The uncertainty of the contact point is formed by the density of the data acquisition, the knowledge of the type of function for the extrapolation, and the fitting range [11]. For simplification, the uncertainty in Table II is the above limit required in ISO 14577 part 1.

To take into account the behaviour in the micro range the different uncertainties of that range are also collected in Table II. The values for the force and the displacement are given by the requirements of the standard (Table I). Because the force generation is based on electromagnetic actors no hysteresis in the force measurement is assumed. Most of the machines for the nano and micro range are designed in such a way that a larger correction of the machine compliance (including frame compliance) is needed. Therefore a higher uncertainty of the correction (about 5%) must be considered.

An important source of uncertainty is introduced by the use and determination of the specific indenter area function needed for h<6µm. Dependent on the direct or indirect procedure for the determination of the indenter area function the uncertainty can vary strongly. Besides of the scattering area, the real indentation process is more complex than it is represented by the simplificated hardness definition, force per area. For all of that reasons the twice uncertainty is assumed for the micro range but a more detailed study seems to be needed urgently.

TABLE II. Input values (u absolute uncertainty, w relative uncertainty) for the calculation of uncertainty (k=1, normal distribution)

Uncertainty		Makro	Mikro
w(force, increasing)	%	0.25	0.5
w(force slope, increasing)	%	0.15	0.3
w(force slope, decreasing)	%	0.3	0.3
u(displacement, increasing)	µm	0.03	0.005
u(displacement slope, increasing)	%	0.5	1
W(displacement slope, decreasing)	%	0,5	1
$u(C_m)$	µm/N	0,0005	0.005
$u(G_1, G_2)$	-	0.35	0.7
u(ε)	-	0.01	0.01
$w(h_0)$	%	1	1
w(dF/dh)	%	2	2
$w(E_i)$	%	10	10
$w(\nu_i)$	%	10	10
$w(\nu_s)$	%	5	5

5. RESULTS OF THE COMBINED UNCERTAINTY

The uncertainty of HM, HM_s, H_{IT}, and E_{IT} is calculated according to the GUM [10] using the equations of the caption 3. The calculation is implemented step by step using Microsoft EXCEL. For instance, the sequence for calculating the uncertainty of the indentation modulus is $u(h_{max}-h_0)$ after (1), $u(1/(E_r\sqrt{G_2}))$ after (14), and $u(E_{IT})$ after (6). The more detailed description of the calculation is given in [9].

The results of the combined uncertainty based on the sources according to Table II is demonstrated as a function of the test force in Fig. 2 for the four materials parameters HM, HM_s, H_{IT}, and E_{IT}. The jumps at 2 N are caused by the different input values of the micro and macro range. Fig. 2 shows that the relative uncertainty of the materials parameter is partially greater than the repeatability required by ISO 14577 parts 2 and 3 (Table I) although the experimental scatter has not been involved in the calculation. To reduce the uncertainty in the future the knowledge of the individual contributions to the combined uncertainty of the materials parameter is of great interest. Obviously, the amount of the contributions are dependent on the material, especially of the indentation hardness and the indentation modulus.

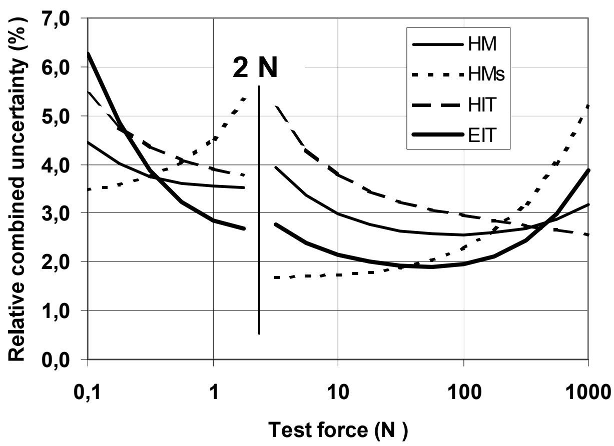

Fig. 2. Relative combined uncertainty of the materials parameter as a function of the test force for a material with HM=1000 N/mm² and E_{IT}=200000 N/mm².

Four representative materials according to Table III have been used. The materials are ordered by the increasing ratio of H_{IT}/E_{IT}. The curves in Fig. 2 are calculated for the material like WC.

TABLE III. Four typical combinations of Martens hardness and indentation modulus in the order of H_{IT}/E_{IT} .

HM	N/mm²	1000	700	16000	4200
E_{IT}	N/mm²	200000	70000	400000	80000
H_{IT}/E_{IT}	-	0.005	0.011	0.064	0.092
$\sqrt{H_{IT}}/E_{IT}$	µm/√N	0.005	0.013	0.013	0.034
Example material		Fe	Al	WC	Glass

The individual contributions to the combined uncertainty at 1 N (micro range) and 100 N (macro range) are presented in Fig. 3. The diagrams demonstrate the strong variation of the sensitivity coefficients which are not only dependent on the test force but also on the hardness and Youngs modulus of the material. While for soft steel the uncertainty of the indentation modulus is mainly affected by the uncertainty of

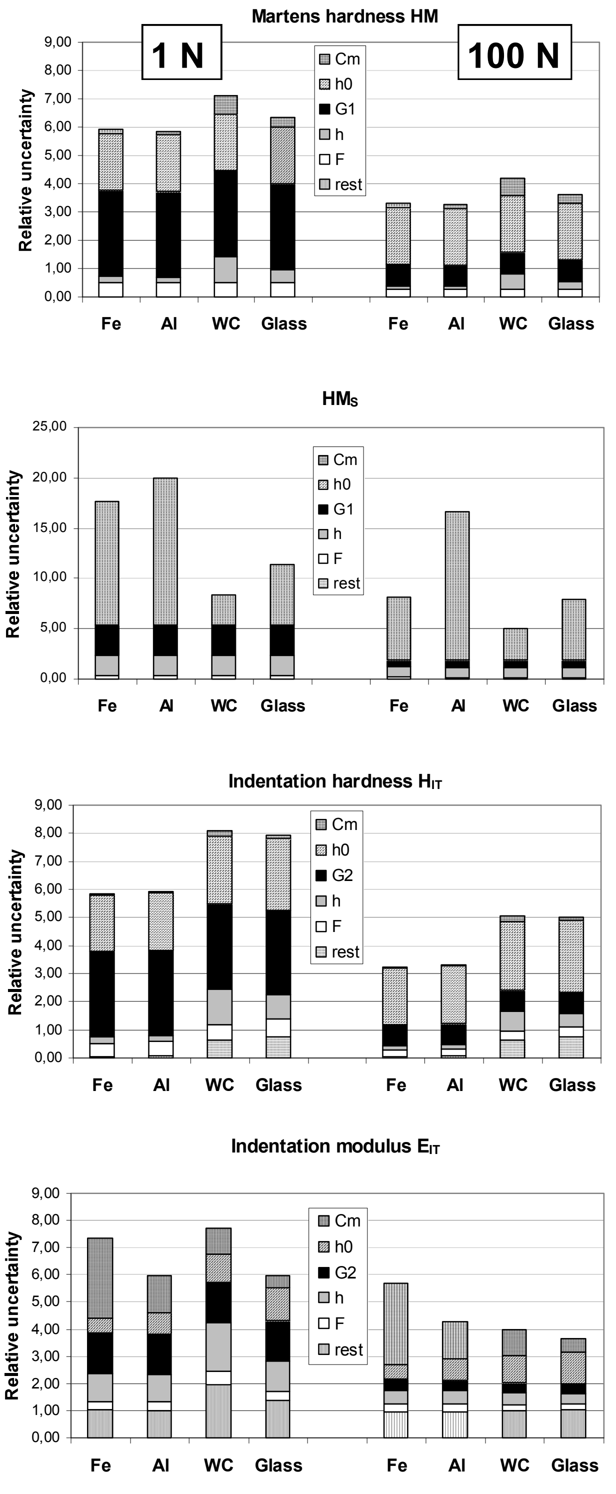

Fig. 3. Contributions to the combined uncertainties of the materials parameters HM, HM_s, H_{IT}, and E_{IT} in the micro range (left) and the macro range (right) for the four representative materials after Table III.

the machine compliance the uncertainty of the zero point is the main part regarding glass. The uncertainty of the zero point is also determining for the Martens hardness and the indentation hardness. However, the uncertainty of the Martens hardness which is determined from the slope without the use of the zero point is strongly affected by the uncertainty of the compliance of the machine. The uncertainty of force is unimportant.

In the micro range the uncertainties of the geometric factor (indenter area function) or of the displacement affect the Martens hardness and the indentation hardness or the indentation modulus, respectively.

6. COMMENTS ON THE ISO 14577

The comments are based on the uncertainties which are partially demonstrated in the last caption. However, some proposals are concluded from further detailed variations of the input values.

- The uncertainty of the compliance affects the calculated materials parameter with increasing test force stronger and stronger. Therefore the upper limit of the macro range in ISO 14577 part 1 (F < 20 kN) is not practicable.
- The required quality of the reference materials in ISO 14577 part 3 can only be fulfiled if the uncertainty of the compliance is limited (for instance to 0.0005 µm/N).
- The upper limit of the uncertainty regarding the zero point in ISO 14577 part 1 (1%) shall be reduced for reference materials in part 3.
- The required tolerance of the test force is too strong in ISO 14577 part 3. Other sources of uncertainty affect the combined uncertainty much more.
- The verification of the testing machine according to ISO 14577 part 2 should be extended to the check whether the zero point and the indenter area function (micro range) are determined precisely by the software of the machine.
- The calibration of force and displacement should be extended to the determination of the uncertainty of the slopes.
- A tolerance of the determined compliance should be involved into ISO 14577 part 2. The use of several values of the compliance in different ranges of the force should be permitted if the compliance response of the machine is not linear.

Obviously, the estimation of uncertainty of the materials parameters calculated from the indentation curve is very complex and circumstantial. Such a extended calculation is needed for the calibration laboratories if reference materials of higher level are missing. The verification of the testing machine in testing laboratories is based on the reference materials according to ISO 14577 part 3. The reference materials should be supplied with information on the uncertainty. Using this input values the testing laboratories can estimate the uncertainty of the results in analogy to the procedure which has been involved into the standards of the conventional hardness techniques. However, the instrumented indentation test needs the specific use of the reference materials. The rules can be concluded from the results of this contribution. Table IV shows some proposals for the best check on the measurement of force and displacement, on the precisely determined zero point as well as on the correct compliance and indenter area function.

TABLE IV. Specific use of the reference materials. The name of the material in the column M indicates only the values of HM and E_{IT} in Table III.

Object of verification	Parameter	M
Force and displacement at force application	HM_s	WC
Force and displacement at force removal	E_{IT}	WC
Zero point of indentation depth	HM	all
Compliance of the machine in macro range	HM_s	Al
Compliance of the machine in micro range	E_{IT}	Fe
Indenter area function	H_{IT}	Fe

7. CONCLUSION

It has been shown the estimation of uncertainty is an appropriate tool for the validation of the standard. Using realistic input values of the sources of uncertainty the most important contributions to the combined uncertainty can be detected. In this way the requirements of the present standard can be evaluated. The results of this paper may stimulate further discussions on the improvement of the standard. Based on the results of this paper a new calibration machine for reference materials of the instrumented indentation test in the macro range is under construction at BAM.

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