CIRP Annals - Manufacturing Technology 69 (2020) 429–432

Contents lists available at ScienceDirect

CIRP Annals - Manufacturing Technology

journal homepage: https://www.editorialmanager.com/CIRP/default.aspx

Single-step calibration method for nano indentation testing machines

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ARTICLE INFO

Article history:
Available online 17 May 2020

Keywords:
Nano indentation
Calibration
Uncertainty

ABSTRACT

Nano indentation is an effective method for materials mechanical characterisation at grain scale. Literature underlined relevance of testing machine calibration to measurement uncertainty of the mechanical characterisation. ISO 14577-2 defines multi-step iterative methods for calibrating frame compliance and indenter area function that do not require high-resolution microscopes. Previous research demonstrated that standard's recommendations are unsatisfactory and result in high calibration uncertainty. This work defines an improved calibration method based on a single-step procedure that achieves, as proved by experimental tests, definite advantages in terms of implementation and measurement uncertainty of both calibration and mechanical characterisation with respect to current procedures.

1. Introduction

Currently, to cope with the more stringent demands of customers for enhanced performances and customisation, manufacturing is facing the development of novel processes, e.g. Additive Manufacturing [1], and advanced materials, e.g. innovative composites [2] and coatings [3]. This, within the framework of Industry 4.0 and the need to cope with big data, requires flexible and fast quality inspections that relies on thorough, accurate and precise characterisation methods [4].

Amongst several product properties, the characterisation of technological surfaces is core both to control the manufacturing process, as surfaces may feature distinctive manufacturing signatures, and to engineer the product [5]. In particular, the mechanical properties of technological surfaces are interesting as they ultimately affect tribological, wear and fatigue behaviour.

Instrumented Indentation Test (IIT) is one of the most appealing mechanical characterisation techniques. It consists of a semi-destructive test, which requires limited sample preparation and can be performed on the final product. It allows to achieve a thorough multiscale mechanical characterisation, i.e. ranging from grain to bulk properties, in terms of Young's modulus, hardness, creep and relaxation and stress-strain behaviour [6]. Nowadays, nano-indentation is exploited to optimise manufacturing processes by characterising related products. It finds application in characterising microstructures by quantitatively distinguishing different phases [7] and estimating the characteristic dimension of the microstructure [8], multi-layer materials by assessing mechanical properties decoupling the contribution of the coating and the substrates [9], estimating residual stresses [10] and characterising micro- and nano-structures, e.g. MEMS [11].

Considering the wide characterisation capabilities and its limited destructiveness, IIT seems suitable for online quality controls and rapid set-up of manufacturing process and was standardised by the ISO 14577, latest updated in 2015.

IIT consists in performing an indentation on a sample by applying a loading-holding-unloading cycle during which the applied force, F, and the related displacement of the indenter in the material, h, are measured. The characterisation of the material is then achieved by analysing the indentation curve (IC), i.e. the $F(h)$ curve, see Fig. 1.

The measurement of h during the whole test allows to resolve hardness, for which IIT was originally conceived, and other mechanical properties at nano-scales, where optical resolution hampers the application of traditional methods [6,12]. The synchronous measurement of F and resulting h is usually obtained by a high-accuracy three-plate capacitive transducer [12].

Amongst the several possible characterisation output, the indentation hardness, H_{IT}, and the indentation modulus, E_{IT}, i.e. an estimate of the Young modulus E of the material, are the most commonly reported and they are defined in Eq. 1 [13], where, ν_i and ν_s respectively are the Poisson's modulus of the indenter and tested material, E_i is the indenter Young's modulus, S is the contact stiffness, i.e. the sample stiffness, and A_p is the projection, on the sample surface, of contact area between the indenter and the specimen.

$$H_{IT} = \frac{F_{max}}{A_p(h_{c,max})} \tag{1.1}$$

$$E_{IT} = \frac{1-\nu_s^2}{\frac{2\sqrt{A_p(h_{c,max})}}{S\sqrt{\pi}} - \frac{1-\nu_i^2}{E_i}} \tag{1.2}$$

S can be computed by modelling the indenter-sample system as a couple of ideal springs representing for the testing machine and the sample, respectively with a compliance C_f and $1/S$ [6,14]. The system total compliance, $C_{tot} = C_f + 1/S$, is obtained as the reciprocal of the measured total stiffness, S_m, which is the slope of the tangent to the

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https://doi.org/10.1016/j.cirp.2020.03.015
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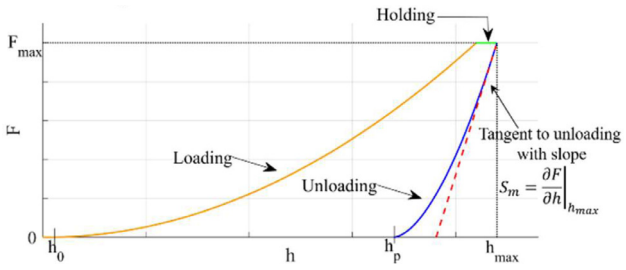

Fig. 1. Example of IC: loading from first contact h_0, holding at maximum load F_{max} for creep compensation, unloading from maximum penetration h_{max} and the residual indentation depth h_p.

unloading curve at its onset (see Fig. 1), and can be evaluated according to the standard power law method [12,13].

The measurement of h during the whole test allows to express A_p as a function of the corrected displacement h_c. In fact, literature requires to correct h, as per Eq. (2), to account for the zero error (h_0) and the elastic deformations respectively of the indentation testing machine (C_fF) and of the sample surface ($\varepsilon(C_{tot}-C_f)F$, where ε is a shape factor depending on the indenter type, e.g. for Berkovich indenter it is 0.75) [13,15].

$$h_c = h - h_0 - [C_f + \varepsilon(C_{tot} - C_f)]F \tag{2}$$

The functional form of A_p depends on the indenter geometry. In the most typical case of a modified Berkovich indenter, i.e. a tetrahedron with dihedral angle of 130.56°, it is $A_p(h_c) = 24.5 \bullet h_c^{\,2}$ [13]. However, due to wear and deviation from ideal geometry, e.g. tip blunting and offset, a more general form can be written as in Eq. (3) [16,17]:

$$A_p(h_c) = a_2 h_c^2 + a_1 h_c + a_0 \tag{3}$$

Furthermore, A_p is related to the reduced Young's modulus, E_r, according to the following equation [13]:

$$A_p(h_{c,max}) = \frac{\pi S^2}{4E_r^2} = \frac{\pi}{4E_r^2 (C_{tot} - C_f)^2} \tag{4.1}$$

$$1/E_r = \frac{1-\nu_s^2}{E_s} + \frac{1-\nu_i^2}{E_i} \tag{4.2}$$

Provided the industrial relevance of this characterisation technique, traceability, accuracy and precision are core to be achieved and ISO 14577-2:2015 establishes the calibration procedure for the testing machine to guarantee them and ISO 14577-1:2015 lists uncertainty contributions to final characterisation results. Barbato et al. [18] proved that C_f and the parameters of A_p are the major contributors to measurement uncertainty of E_{IT}. These are calibrated through a procedure described in the Annex D of ISO 14577-2:2015.

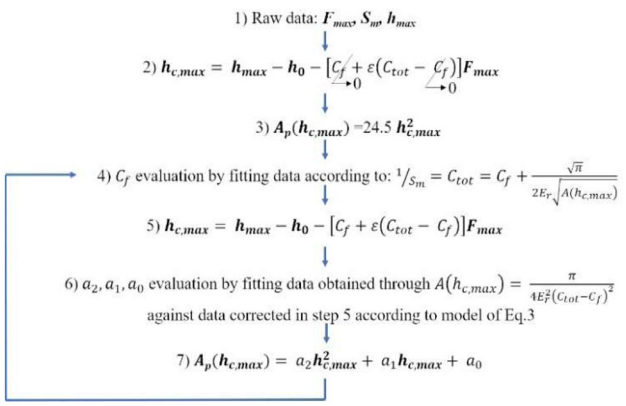

Fig. 2. Calibration iterative procedure workflow as per ISO 14577-2:2015. Quantities in bold are arrays built as $F_{max} = \{F_{max,\ i,j}\}$, where i counts the force levels and j the replications.

Five alternative methods are present. Methods no. 1, 3 and 5 calibrate C_f by preliminary calibrating the parameters of A_p through the measurement of the indenter tip by a metrological AFM. Despite the smaller calibration uncertainty, they are more expensive; thus, in academic and industry practice, method no. 2 and 4 are preferred. They both consist of an iterative procedure which achieves the calibration of C_f and A_p parameters by relying only on the results of a set of indentations. Although the widespread adoption of the method no. 2 and 4, the ISO 14577-2:2015, reference literature [16,19] and good practices of testing machine manufacturers provide a wide range of calibration recipes, which, however, demonstrate the method's unclear definition and comparison have shown to be unsatisfactory for accuracy, precision and consistency among each other [20].

To overcome the criticalities of the cheaper calibration approaches, the present paper proposes an improved calibration method based on a single-step procedure rather than on an iterative method to improve both implementation of the calibration and mechanical characterisation results. Section 2 discusses current calibration methods and proposes the novel approach, Section 3 analyses results based on experimental tests and Section 4 draws conclusions.

2. Nano-indentation testing machines calibration methods

2.1. Multi-step iterative standard methods

The Annex D of ISO 14577-2:2015 describes two iterative methods, whose workflow is depicted in Fig. 2, to achieve the concurrent calibration of C_f and A_p parameters.

The procedure requires to perform J replicated indentations at I levels of maximum force, F_{max}, over the application range of the instrument on a calibrated reference material [17]. The system is initialised at step 2 and 3, supposing ideal infinitely stiff machine and shape of the indenter. The procedure holds fixed ε and E_r, which ultimately enables for the calibration and follows the steps of Fig. 2. The steps are iterated until convergence is achieved.

Method no. 2 and 4 differ for exploiting, respectively, one and two calibrated reference materials. For the latter, a material with higher E, e.g. tungsten (W), shall be considered to calibrate C_f, whilst a material with lower E, e.g. SiO_2 or monocrystalline Al, enables the calibration of A_p parameters. Method no. 4 proved to be faster and more accurate as it decouples the parameters to be calibrated and reference materials [16,20], and it will be adopted and referred to in this work as best standard calibration practice. Literature [20] showed that different choices, all compatible with standard recommendations, for I and J yield significant variability of accuracy and precision to the calibration results which make them potentially ineffective to establish traceability and ensure results comparability amongst different platform and laboratories.

2.2. Single step method

To cope with shortcomings of ISO 14577-2:2015, this work proposes a single-step method to calibrate C_f and A_p parameters. Rearranging equations from Eq. (1) to (4), the multivariate function $f = f(F_{max}, h_{max}, S_m)$ in Eq. (5) is obtained.

$$f = \begin{cases} \frac{\pi}{4E_r^2} = \left(\frac{1}{S_m} - C_f\right)^2 \left\{ a_2 \left[h_{max} - h_0 - \left[C_f + \varepsilon\left(\frac{1}{S_m} - C_f\right)\right]F_{max}\right]^2 + +a_1 \left[h_{max} - h_0 - \left[C_f + \varepsilon\left(\frac{1}{S_m} - C_f\right)\right]F_{max}\right] + a_0 \right\} \\ H_{IT} = \dfrac{F_{max}}{a_2 \left[h_{max} - h_0 - \left[C_f + \varepsilon\left(\frac{1}{S_m} - C_f\right)\right]F_{max}\right]^2 + a_1 \left[h_{max} - h_0 - \left[C_f + \varepsilon\left(\frac{1}{S_m} - C_f\right)\right]F_{max}\right] + a_0} \end{cases} \tag{5}$$

Once J indentations at I force levels have been performed, the calibration can be achieved by performing a regression having as predictors the measured F_{max}, h_{max} and S_m and as responses the calibrated E_r and H_{IT} of a reference material. Because Eq. (5) is strongly nonlinear, and not linearisable, in the parameters and predictors variability is not negligible, as they are influencing factors to measurement uncertainty [17], a nonlinear Orthogonal Distance Regression (ODR) is necessary [21].

These non-trivial hypotheses have not been properly investigated in previous approaches [16,17,19], for this reason the standard assumes an Ordinary Least Square (OLS) to suffice for the regressions, thus neglecting predictors variability and affecting the estimate of parameters. In order to implement the ODR approach, Eqs. from 1 to 4 must be combined in the system reported in Eq. (5), which, due to the current use of OLS has never been proposed and investigated before in the scientific literature. This, with respect to the standard method, has twofold advantages. First, mathematical and statistical formality to the calibration problem is provided which avoids possible misinterpretation of the unclearly defined multi-step iterative algorithm and allows the adoption of a more appropriate statistical tool. Second, H_{IT} is introduced in the calibration pipeline: because calibrated parameters are exploited to characterise also hardness, conceptually it is core to include it in the calibration procedure. The only trivial requirement for this approach is that calibration laboratories upstream in the traceability chain should calibrate reference materials also in terms of H_{IT} via an independent technique, e.g. by calibrating A_p by a metrological AFM.

2.3. Uncertainty evaluation

Influencing factors for the calibration measurement uncertainty are sourced by measurements, i.e. F_{max}, h_{max}, S_m, which also contain measurement noise, by calibration certificates of reference materials, i.e. ν_s, E_r, H_{IT}, and by tabular values, i.e. ν_i and E_i. The standard approach, because of its iterative workflow, introduces as influencing factors C_f and A_p parameters, i.e. the calibration results themselves. Thus, closed formulae for the uncertainty propagation according to GUM [22] cannot be written. As regards the single step method, influencing factors are only the measured, calibrated and tabular ones. However, nonlinear regression does not guarantee that the outputs distribute according to a normal distribution. Therefore, the simple standard deviation of regression output cannot be assumed as the standard uncertainty of the calibrated parameters. To evaluate the expanded uncertainty at a 95% confidence level, a Monte Carlo Method (MCM) with at least 10^4 iterations shall be exploited to estimate distribution of C_f and A_p parameters. Inputs are the influencing factors, with measurement and calibrated sources assumed to distribute normally and tabular sources uniformly [23].

3. Analysis of calibration methods

3.1. Experimental data

Data were collected during last CIRP international comparison on nanoindentation [24] according to literature [16,17]: fifty ($I = 5$ and $J = 10$) indentations on W and SiO_2 calibrated reference materials at (0.1, 0.5, 1.0, 5.0, 10.0) mN; the setup is chosen to optimise standard method accuracy and precision. Indentations were performed by a Hysitron TriboScope, hosted in the facilities of the Oklahoma State University and equipped with a modified Berkovich indenter ($E_i = 1140$ GPa, $\nu_i = 0.07$ and $\varepsilon = 0.75$), see Fig. 3, was calibrated on calibrated samples, whose characteristics are summarised in Table 1. The testing equipment features a force-displacement transducer with resolution and noise floor, respectively, of 1 nN and 75 nN, on force, and of 0.006 nm and 0.2 nm, on displacement. This platform allows to neglect h_0.

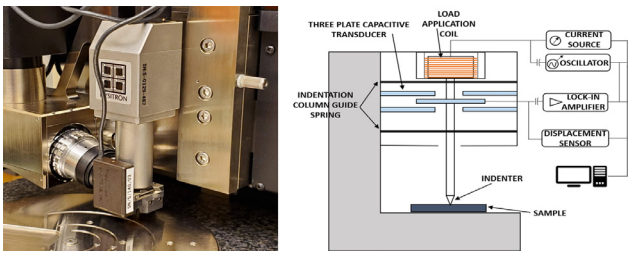

Fig. 3. Detail and scheme of the Hysitron TriboScope indentation head.

Table 1
Calibrated reference materials characteristics.

Material	Calibration body	E / GPa	ν
SiO_2	NPL	73.3 ± 0.6	0.161 ± 0.003
W	NPL	413.0 ± 2.8	0.281 ± 0.003

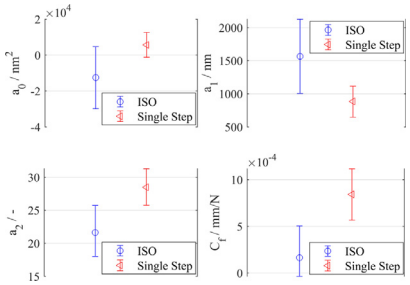

Fig. 4. ISO and single step method calibrated parameters comparison.

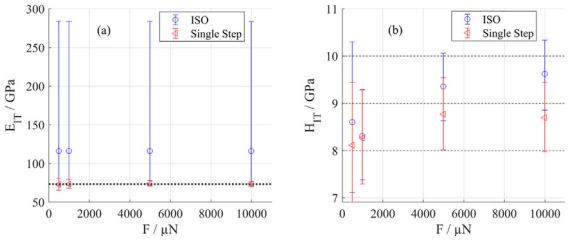

Fig. 5. ISO and single step method validation on SiO_2. (a) E_{IT}, (b) H_{IT}. Black dashed lines are calibrated references mean and uncertainty interval.

3.2. Results discussion

Results, as mean and expanded uncertainty, see Section 2.3, in terms of C_f and A_p parameters are shown in Fig. 4. Good practices prescribe to validate the calibrated parameter by characterising calibrated reference materials. E_{IT}, H_{IT} and F/S^2 (since it is independent from A_p [25]) of W and SiO_2, as these materials represent wide range of mechanical properties, are shown in Figures from 5 to 7. The single step method provides more accurate and precise results in most of the cases. With respect to the ISO method, the single step method shows higher robustness to possible outliers, which yield the high uncertainty in Fig. 5(a). In Fig. 5(a), the asymmetry of E_{IT} confidence interval when the system is calibrated according to the standard can also be seen. Improved accuracy with respect to the standard method is shown for H_{IT}, which is expected because of the method definition see Figs. 5(b) and 6(b). F/S^2 results in Fig. 7 show that most accurate and precise calibration of C_f is obtained by the single step method. Similar results were preliminary obtained performing same

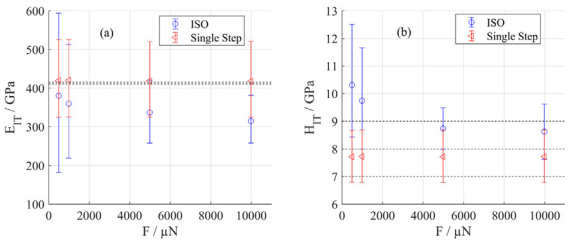

Fig. 6. ISO and single step method validation on W. (a) E_{IT}, (b) H_{IT}. Black dashed lines are calibrated references mean and uncertainty interval.

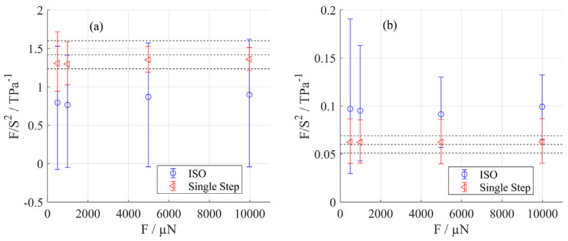

Fig. 7. ISO and single step method validation on F/S^2. (a) SiO_2, (b) W. Black dashed lines are calibrated references mean and uncertainty interval.

experiments on different indentation platforms, i.e. Hysitron TI 950 (owned by Istituto Italiano di Tecnologia in Turin – Italy) and Anton Paar NHT^3 (owned by Politecnico di Torino – Italy).

4. Conclusions

This work proposed a single step procedure to overcome the several shortcomings of ISO 14577-2:2015 iterative calibration method for nanoindentation testing equipment. The single step method has both conceptual and practical advantages. The former consist in providing rigorous mathematical formulation to the problem; in catering for variability of inputs by relying on an Orthogonal Distance Regression (ODR), and introducing the hardness as calibration reference. The latter have been proved, through experimental comparison, to be a greater accuracy and precision with respect to the standard approach, not only for the calibrated values but also for the validation on reference materials. Even if this approach requires calibration laboratories upstream in the traceability chain to calibrate indentation hardness by an independent technique, the related costs are negligible in comparison to the great procedural and metrological advantages. Future works will exploit the proposed single-step method to improve and simplify the calibration procedure by investigating the effect of reference materials when they are not calibrated in terms of H_{IT}. Future research will also address comparison of performances between this method and the more expensive alternatives based on AFM, and amongst different instrumented indentation machines.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgement

This work has been partially supported by “Ministero dell’Istruzione, dell’Università e della Ricerca”. Award “TESUN-83486178370409 finanziamento dipartimenti di eccellenza CAP. 1694 TIT. 232 ART. 6″.

The authors thanks Prof. D. A. Lucca from Oklahoma State University for sharing the data of the CIRP intercomparison [24].

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