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Effect of contact stiffness and machine calibration in nano-indentation testing

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Abstract

Instrumented indentation tests enable evaluation of several material parameters. Preliminary investigations underlined the influence of contact stiffness in the nano-range. ISO 14577-1 recommends two methods for contact stiffness evaluation, which, however, present shortcomings mainly related to the disregard of actual shape of unloading indentation curve. Given the relevance not only at the extent of material characterization, but also at testing machine calibration, alternative evaluation procedures have been developed, showing better agreement with experimental data and lower uncertainty in the evaluation of contact stiffness. In this work, shortcomings of standard and alternative methods in the evaluation of material parameters are highlighted, and innovative procedures are proposed to overcome them.

Keywords: Instrumented indentation test; Nano-range; Contact stiffness; Machine Calibration

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1. Introduction

Hardness test, being often considered non-destructive, easy to perform, inexpensive and enabling wide range of mechanical properties to be measured, such as yield strength, creep and relaxation [1], is broadly applied in a number of industrial fields, such as aeronautics and high segment automotive production, where qualification of finished component is required. Additionally, the definition of depth sensing techniques, such as instrumented indentation test (IIT), enabled the characterization of material properties down to nano-load range, overcoming constraints conventionally set by optical instrument resolution [2,3]. This is achieved by the continuous measurement over the complete loading and unloading cycle of both applied force and indenter displacement and a simple relationship, the area shape function, between the contact surface of the indenter and its displacement into the material [4].

In the present work, estimation of elastic properties, such as indentation modulus, from IIT in the nano-range is considered. In section 2 performances of methods, both established in standards and available in literature, are briefly discussed. Section 3 proposes new methodologies to improve estimation of the contact stiffness and results are presented in section 4. Finally, conclusions are drawn in section 5.

2. Instrumented indentation test overview

2.1. General background

Instrumented indentation test consists of indenting up to plastic deformation a test piece by loading a diamond indenter

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in a direction orthogonal to the sample surface. In the case of the most common force control, shown in Fig. 1, alike to traditional hardness testing, force increases from zero to a maximum value over a specified time interval, then it is held at constant value for a certain period, to compensate for creep, and load is gradually removed over a specified time, down to zero [4]. Time history of applied force and indenter displacement is recorded and resulting point clouds are processed to obtain material characteristics.

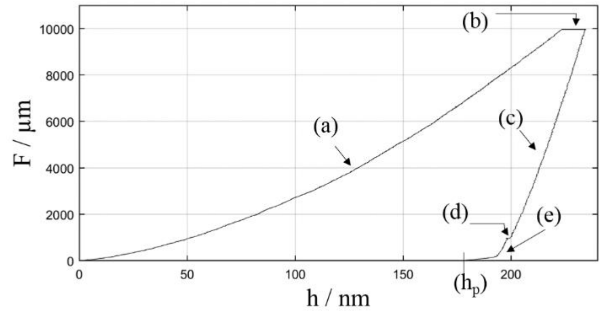


Fig. 1. Example of IIT force vs displacement curve: (a) is the application of the test force, (b) is the hold phase, (c) is the removal of test force up to the (d) second held to compensate for drift, and (e) is the final unloading with residual indentation h_p.

The main outcomes of IIT are instrumented hardness H_{IT}, and indentation modulus E_{IT}, an indication of elastic properties of material [4]. The latter, as expressed in Eq. (1), is obtained from contact stiffness S, contact area at maximum depth $A_p(h_{max})$ and other parameters, such as sample Poisson's modulus ν_s, indenter's Poisson's modulus and Young's modulus ν_i and E_i. Contact stiffness is defined, according to Eq. (2), as the slope of the force-displacement $F(h)$ unloading curve at the onset of unloading and it is relative to the sample, see ISO 14577-1:2015 [4].

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

$$S = \left.\frac{\partial F}{\partial h}\right|_{h_{\max}} \tag{2}$$

Therefore, material properties depend on, amongst the other, measured indenter displacement h and geometrical characteristics of the $F(h)$ represented by S. However, raw data shall be corrected, according to Eq. (3), to cater for several contributions, such as zero error h_0 in the displacement scale, the displacement due to elasticity of the sample surface (where ε accounts for tip geometry) and the frame compliance C_f.

$$h_{\max} = h_{m,\max} - h_0 - [C_f + \frac{\varepsilon}{S}]F_{\max} \tag{3}$$

$$\frac{1}{S_m} = C_f + \frac{1}{S} \tag{4}$$

Frame compliance requires to be calibrated; according to ISO 14577-2:2015 [5], it is sufficient a simple modelling of the indentation system as made up of two compliant objects, i.e. the sample and the testing equipment, as in Eq. (4). Therefore, overall (or measured) stiffness, S_m can be computed by applying Eq. (2) on raw data and S follows after C_f calibration. Similarly, area shape function parameters require calibration, which, when it is not performed by means of an atomic force microscope, can rely on an iterative procedure which contemporarily achieves calibration for both C_f and these parameters [5,6]. Consequently, accurate and precise evaluation of S is necessary considering its importance on both characterisation of the materials and testing equipment calibration [7].

2.2. Review of contact stiffness evaluation

Analysis of methods proposed in two main related standards, ISO 14577-1:2015 [4] and ASTM E2546-15 [8], underlines some limits, liable to yield non-compatible results.

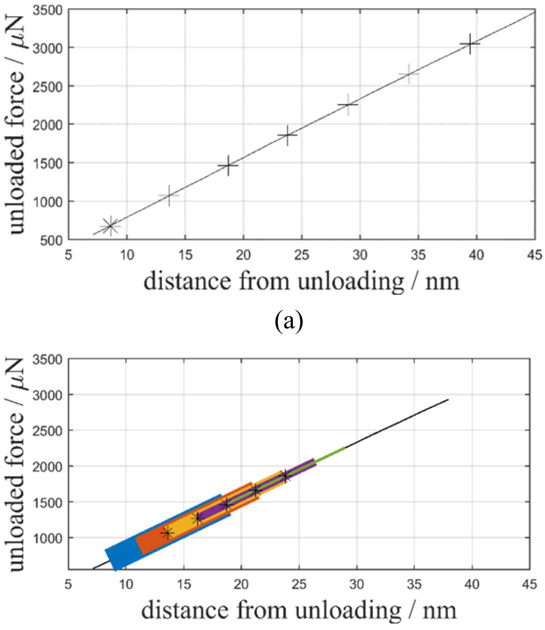
In order to estimate material's elastic properties, these standards entail fitting experimental unloading points according to given models, followed by differentiation to estimate contact stiffness. Two mathematical models are considered, the linear extrapolation method (LE) and the power law method (PL). The first model, defined by Doerner and Nix [9], adopts a linear fitting of the first portion of the unloading curve, according to experimental evidence, supported by Hertz's solution in the case of indentation by a flat punch. The second model, PL, has been later introduced by Oliver and Pharr [10]. In order to cater for the inherent non-linearity of unloading curve, see Fig. 1, PL suggests fitting a power law, which is compliant to Sneddon's solution of Boussinesq's problem for conical indenters [11]. However, as already highlighted elsewhere [7,12], both PL and LE presents shortcomings. In fact, LE tends to determine the secant of the unloading curve rather than its tangent. Therefore, even though it can produce results associated to low measurement uncertainty, severe bias may occur, mostly due to curvature. On the other hand, PL inherent curvature characteristics showed to limit the adequacy of the non-linear fitting and resulting in discrepancies from theoretical solution [13] and on a dependence on considered portion of the unloading curve [7]. Moreover, the model definition due to the adoption of h_p may generate larger measurement uncertainty [12]. Therefore, alternative methods were proposed [7,12]. They are the sinus (SN) and the logarithmic method (LN), which are in better agreement with experimental data as in the neighbourhood of the origin (a change of reference system is performed to bring the unloading onset at the origin) they can be properly approximated by linear function. However, when coming at a throughout comparison, either methods are affected by bias (LE and SN) or high uncertainty (PL) or show little agreement with each other (LN is particularly critical) [7].

3. Proposed methodologies for contact stiffness evaluation

Contact stiffness definition requires the evaluation of the indentation curve derivative at the maximum penetration depth. Methodologies proposed thus far suggest fitting such a curve with a pre-defined mathematical model, according to contact theory, which then requires to be differentiated. However, this presents two inherent criticalities. The first, the fitting ensures consistency with the interpolated data, but it cannot guarantee any mathematical property of the derivative. The second, fitting is an operation whose adequateness is evaluated on the whole set of data; thus, there might be local curvatures that are not properly interpolated. Since the fitted model has to be differentiated, and the contact stiffness requires the evaluation of the derivative in the first point of the curve, corresponding to the onset of unloading, significant errors may be introduced. Therefore, to provide a metrological consistent evaluation of contact stiffness with its definition, direct derivative evaluation methods are addressed.

In the following, two methodologies are proposed to estimate the trend of the derivative, directly from experimental points, which describe the unloading indentation curve, to eventually achieve evaluation of *S*. Both methods exploit the robustness of the secant evaluation of the *F(h)* unloading curve. According to Sneddon, by means of linear regression, the secants, d_i, at different positions, can be evaluated; these are now interpolated to extract the derivative trend. In particular, two methodologies have been developed.

In the first method (named S1), secants at different position are evaluated as the slope of the regression line of a portion, or window, of unloading indentation curve that yields from the onset of unloading to an increasing distance from it, see Fig. 2(a). Instead, the second approach (named S2) performs the linear fitting on window of the unloading indentation curve of the same width, expressed in terms of number of considered points, but centered on different positions, see Fig. 2(b).

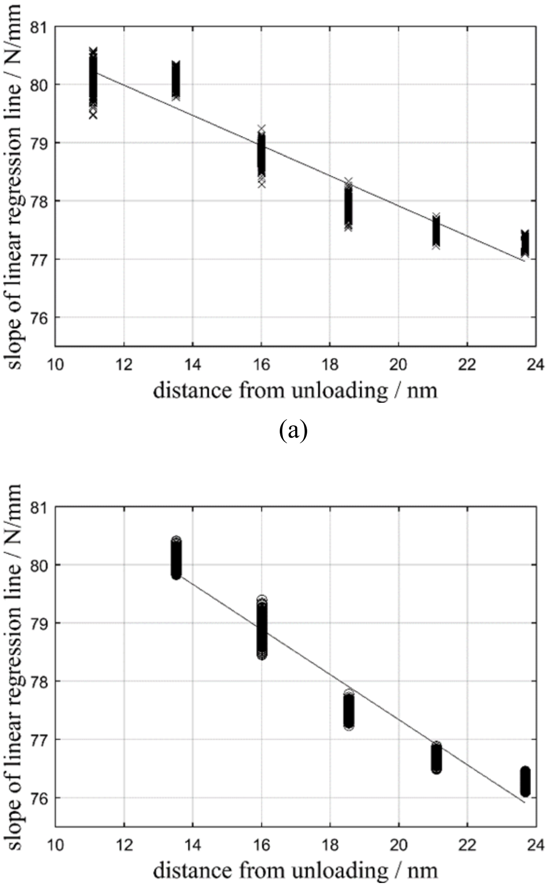


(a)

(b)

Fig. 2. (a) Method S1 applied to unloading indentation curve: * start of windows, + end of windows at different distances from onset of unloading. (b) Method S2 applied to unloading indentation curve: * centre of windows, different colours highlight position of different windows. Reference system has been changed according to methodology proposed for SN and LN. Test performed on fused silica at 10 mN.

Once the secants have been computed, they have to be interpolated; however, uncertainty associated to secant evaluation at different positions from the linear regression has to be properly catered for. In fact, standard deviation of the slope of the regression line, $s(d_i)$, depends on the number of fitted points. Moreover, because onset of unloading is a transient condition, force and displacement signals are affected by higher noise content, therefore, secant evaluation nearby the start of unloading, will be associated to higher measurement uncertainty. Thus, linear regression to extrapolate derivative is applied to a set of data points that has been constructed ad hoc to introduce uncertainty effect, as Fig. 3 shows. At each location where secants are evaluated, a set of one hundred points extracted from a normal distribution $N(d_i, s(d_i))$ are considered. Regression according to a linear model has been adopted according to Sneddon's solution of Boussinesq's problem for conical indenter [11].



(a)

(b)

Fig. 3. Constructed data set for the interpolation of secants computed with methods (a) S1 and (b) S2. Sample indentation on fused silica at 10 mN. Slope of the unloading curve as a function of distance from onset of unloading is shown.

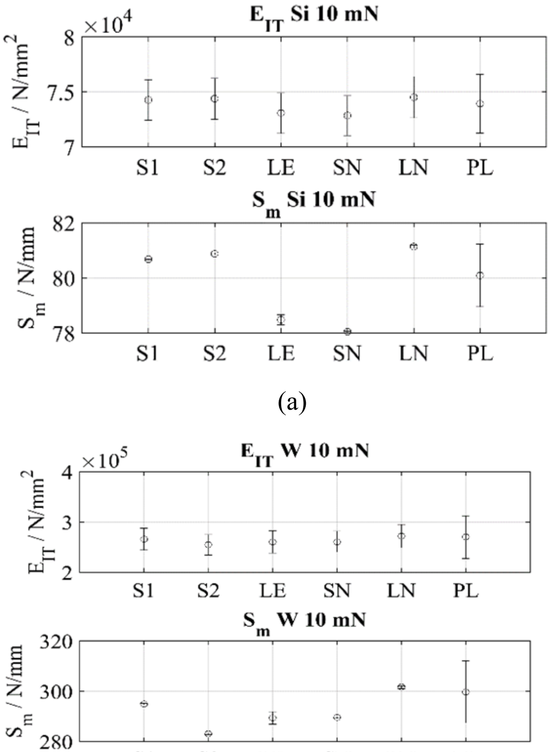
The preliminary application of mobile-average filter to the measured force and displacement signals was required to eliminate measurement noise and outliers along with the verification of absence of significant systematic, which allow supporting the assumption of normal distribution. Moreover, it is worth to stress that the mobile average filter was applied to unloading indentation curve from the 98% of F_{max}, in line with general standard prescription [4]. Consequently, as Fig. 2

shows, unloading curve does not begin from zero unloaded force.

In both cases, to be consistent with linear derivative approximation, only the initial part of unloading indentation curve is considered, by properly choosing the width and position of curve portion, i.e. centering windows from 2.5% to 15% with steps of 2.5%, which are exploited for secant evaluation. Considering previous discussion about the requirement of a trade-off between accuracy and precision, for S2, a window width of 10% of unloading curve length was considered appropriate. Similarly, trial and error suggested a symmetric window wide ten points per side, for the application of mobile average filter.

4. Results discussion

In order to compare effectiveness of the two proposed methodologies, S1 and S2, with the four already presented in literature, LE, PL, SN and LN, these were applied to evaluate S_m and E_{IT} on the measurement performed on fused silica and tungsten, which are standard materials, at load ranging from 0.5 mN to 10 mN [7]. S_m is examined to assess methods behaviour excluding contribution from C_f calibration. Fig. 4 shows results, provided with measurement uncertainty evaluated according to GUM [14], in the representative case of indentation performed at 10 mN as maximum load.



(a)

(b)

Fig. 4. Indentation modulus and contact stiffness of (a) fused silica and (b) tungsten indented at 10 mN. Comparison of new methods S1 and S2 with standards and literature methods.

In the case of fused silica, S1 and S2 provide measured contact stiffness evaluation compatible with each other, differently from the case of tungsten, which suggest a material effect. Moreover, although the new evaluations fluctuate within the largest uncertainty range provided by formerly defined methods, due to derivative direct evaluation dependence on spikes and singularities of experimental curve, they are compatible with previously presented methods and are associated to limited measurement uncertainty, as far as contact stiffness is concerned. Therefore, despite signal noise hinders from concluding on general behaviour and robustness of S1 and S2, the proposed methodologies are capable of providing precise results. E_{IT} results are non-conclusive due to measurement uncertainty, which include contribution of calibration of testing machine and S_m.

5. Conclusion

The present work outlines main criticalities related to contact stiffness evaluation for material characterisation by instrumented indentation testing. Considered its relevance in both the characterisation and the testing equipment calibration and given the limitations of several available techniques, new methodologies aimed at improving methodologies for estimating the contact stiffness are proposed. These directly evaluate the derivative to achieve a metrological consistent assessment of contact stiffness. Although measured force and displacement noise hampers from concluding on the robustness of proposed methodology, these are promising and capable of providing precise results. Therefore, future work, along with improving presented methodologies to achieve higher accuracy, will investigate further approaches, for example based on numerical methods, to directly evaluate derivative from experimental data. This will benefit on the application of instrumented indentation test, particularly for the nano-range, by reducing measurement uncertainty and providing procedure with a metrological robust framework.

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