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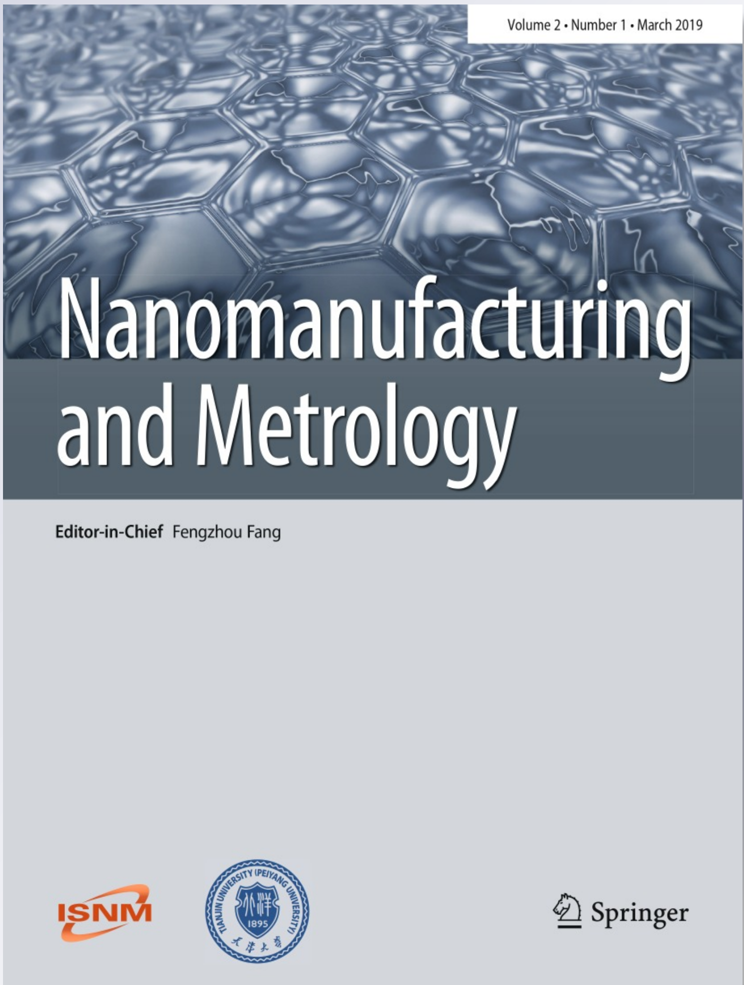

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ORIGINAL ARTICLES

Instrumented Indentation Test: Contact Stiffness Evaluation in the Nano-range

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Abstract

Instrumented indentation test is a non-conventional hardness test, relevant for performing both hardness measurement and estimating the elastic modulus. The indentation modulus, which depends on the contact stiffness, estimates the elastic modulus. To evaluate contact stiffness, the literature requires fitting force–displacement curve according to mathematical models, which are hardly representative of the curve. This research aims at proposing alternatives, which estimate contact stiffness directly from experimental data. Proposed methods are applied to nano-indentations performed on a reference material and a high-speed bearing steel; indentation modulus is evaluated along with uncertainty contribution of contact stiffness. Comparison with standard methods is provided.

Keywords Instrumented indentation test · Nano-range · Elastic modulus · Contact stiffness · Fitting model

1 Introduction

Hardness measurements can be considered to be semi- or non-destructive test, which allows for the final component to be characterised. Therefore, the lack of the need of properly shaped specimen, along with ease and low cost of testing, enabled these characterisation procedures to find application in a number of industrial fields. Hardness is defined as the capability of a material to resist to indentation up to the onset of permanent deformation or cracking, respectively, for plastic or fragile materials. A loading and unloading cycle is performed on the sample by means of an indenter at a certain maximum load; when the load has been completely removed, a residual indentation will be present, and its surface can be related to the material hardness, which can be computed as the ratio between the maximum test load and the residual indentation area.

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Hardness test was first introduced by Brinell in the 1900, later, alternatives, such as Rockwell (1922), Vickers (1935), Knoop (1939) and Martens (2000) hardness, were developed, featuring different indenter shapes and procedures to characterise the material at low loads, down to micro-scale [1, 2]. However, when nano-indentions were introduced, optical methods to determine the area of residual indentation proved either to be non-effective due to limited lateral resolution or extremely time-consuming [3]. Therefore, a further hardness test was defined. It is the instrumented indentation test (IIT), which can overcome limits set by optical instruments by means of a continuous measurement during the whole loading–unloading cycle of both the applied force (F) and the indenter displacement (h), which can be considered an estimation of indentation depth [4, 5].

Moreover, hardness measurements found technological and metallurgical application because not only tribological properties, but even relationship between measured quantities and material characteristics, such as yield strength, elastic modulus, creep and resilience, can be determined. In fact, Brinell hardness test was first aimed at assessing material elastic property through a simpler and non-destructive procedure than tensile test [1]. However, it is relevant to carry in mind the arbitrary attribute of hardness scales which results from the definition of the scale itself, in terms of how, depending on the indenter geometry, the residual indentation should be evaluated and the load applied. Consequently, the relationships between hardness and other material characteristics were found to depend on the scale and material itself, so that caution in the use of conversion tables proposed by standard organisations, i.e. ISO and ASTM, is recommended. In fact, hardness measurements yield conventional values, as evidenced by the symbols adopted for different scales.

In the case of IIT, material elastic properties are expressed in terms of the indentation modulus (E_{IT}), which depends on the contact area (A) evaluated at the onset of the unloading and on the contact stiffness (S), which is defined as the slope of the unloading $F(h)$ curve at the maximum force. Moreover, IIT, by enabling hardness test to be performed at loads ranging from macro- to nano-level, allows for from bulk, i.e. average properties, to local material characterisation, i.e. microstructural characterisation, which arose interest in providing robust metrological framework to this technique. Amongst several issues related to IIT, the evaluation of contact stiffness proved to be a major source of measurement uncertainty to the indentation modulus and to significantly affect the calibration of the testing machine [6, 7].

In the present work, alternative methods are introduced to evaluate the contact stiffness aiming at reducing the measurement uncertainty of indentation modulus and solve main issues related to methods available thus far: in Sect. 2, adopted methodology is discussed, in Sect. 3 results are examined, and finally in Sect. 4 conclusions are drawn.

2 Methodology

Instrumented indentation test consists of indenting a sample through a diamond indenter, whose shape can be either Vickers or Berkovich, orthogonally to the surface of the test piece. The test requires a loading–unloading cycle to be performed, an example of which is shown in Fig. 1. The cycle is generally force-controlled and reaches the maximum test force in a specified time interval, and then it is held at constant value for a certain period, to compensate for creep phenomena and, finally, load is completely removed, in a certain time interval [4]. Material characterisation can be obtained by properly processing the force and the indenter displacement, which are measured by the means of transducers and define an experimental point cloud.

Even though the literature shows that IIT can provide quantitative and qualitative information about microstructure [8, 9] and evaluate material flow properties [10], according to the standard ISO 14577-1 [4], IIT, along with assessing the hardness, achieves assessment of the creep and relaxation behaviour and the elastic modulus of the tested material. The latter is estimated by means of the indentation modulus, E_{IT}, which depends on the contact area at maximum depth, $A(h_{c,\max})$, the contact stiffness, S, the sample Poisson's modulus, ν_s, and the Poisson's modulus and the Young's modulus of the indenter, respectively, ν_i and E_i, see Eq. (1).

The contact area is evaluated as a function of the indentation depth, and the functional form of this relationship depends on the geometry of the indenter tip; for example, conical, Vickers and Berkovich indenters yield to quadratic relationship [3]. This functional dependence enables, in practice, to overcome constraints set by optical instrument lateral resolution and to cope with nano-indentations.

The contact stiffness is defined, see Eq. (2), as the slope of the force–displacement unloading curve at the onset of the unloading. Its evaluation is critical because it resulted to be often one of the major sources of uncertainty for this estimation [6, 7] and because it further has an indirect contribution from the calibration of the testing equipment.

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

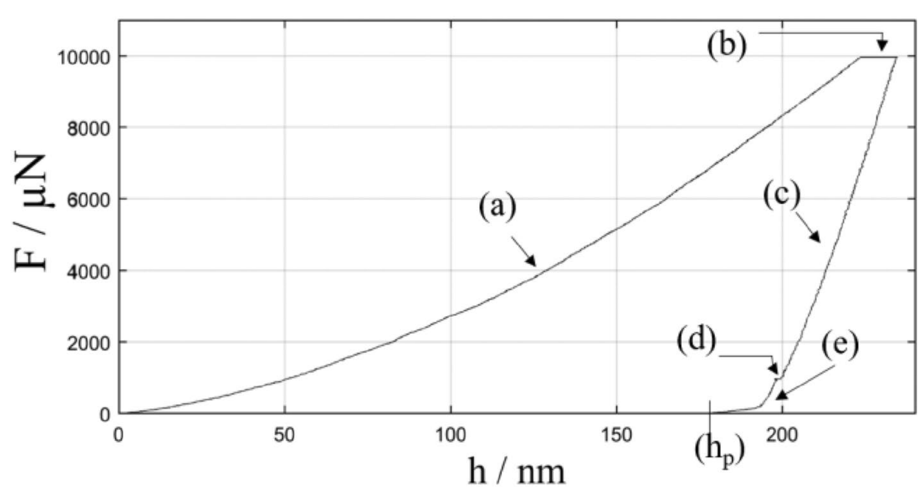

Fig. 1 Example of indentation curve (a) loading curve, (b) holding at maximum load necessary for creep compensation, (c) unloading curve, (d) non-standard holding at 10% of maximum load to compensate for thermal drift, (e) final unloading and the residual indentation h_p

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

It is relevant to recall that to remove systematic contribution due to machine compliance and indenter deviation from ideal geometry; the measured h and contact stiffness, S_m, which results from the application of methods on raw data, shall be corrected. Correction is discussed elsewhere and evaluates the S and the corrected indenter displacement, h_c [4, 11]; if such correction is not applied, the resulting measured contact stiffness includes both the stiffness of the specimen and of the testing equipment.

2.1 State of the Art

In order to evaluate the (measured) contact stiffness, the literature and standards require the unloading curve to be fitted according to a pre-defined mathematical model, which has to be differentiated and computed in the point corresponding to the onset of unloading. Four models have been proposed, which are the linear extrapolation (LE) [12], the power law method (PL) [13, 14], the sinus (SN) [6] and the logarithmic (LN) [7] model.

The models have been defined catering for different solutions of the Boussinesq's problem, e.g. solution of the stress–displacement field generated by a concentrated load applied normally to the surface of an elastic half-space, which models the indentation. In fact, indentation with punches of arbitrary geometry can be reduced to Boussinesq's problem [15].

LE has been defined by Doerner and Nix [12] considering that indentation at least in the neighbourhood of unloading onset can be well approximated by the Hertzian solution, i.e. flat punch geometry, which implies that the contact area is constant and entails that unloading curve can be modelled by linear function.

However, Oliver and Pharr [13, 14] observed that unloading curve is far from being linear, and therefore, according to general Sneddon's solution of Boussinesq's problem [15], see Eq. (3), they suggested adopting a nonlinear fitting with a power law (PL) relationship, see Eq. (4). In Eqs. (3) and (4), β and B are material parameters, h_p is the residual indentation depth (Fig. 1) and m depends on indenter geometry (e.g. it is equal to two in the case of conical indenter). However, both LE and PL present shortcomings. In fact, the former tends to evaluate the secant rather than the derivative of the unloading curve, which results in underestimating the contact stiffness, despite being associated to small measurement uncertainty. The latter, on the other hand, due to the presence of the residual indentation depth parameter, whose evaluation is highly uncertain, provides results which are unsatisfactory from the measurement uncertainty perspective.

Therefore, improvements in LE methods have been recently defined, in order to cater for experimental curvature [6, 7]. They require the unloading curve to be nonlinearly fitted according to sinus or logarithmic models, see Eqs. (5) and (6), where $h_{\max}$ is the maximum indentation depth (Fig. 1) and k_X and k_Y are fitting parameters which account for sample material and indenter geometry.

$$F = \beta h^m \tag{3}$$

$$F = B(h - h_p)^m \tag{4}$$

$$F = F_{\max} - \frac{1}{k_Y}\sin(k_X(h_{\max} - h)) \tag{5}$$

$$F = F_{\max} - \frac{1}{k_Y}\ln(k_X(h_{\max} - h)) \tag{6}$$

2.2 Proposed Methodologies

However, when compatibility of the models for S evaluation is addressed, critical condition is highlighted that prevents from concluding on an absolute preference of a model with respect to the others [7]. In fact, independently from the adopted mathematical model, the procedures proposed in the literature present an inherent criticality due to the parameter to be computed. Considering the definition of the contact stiffness, its evaluation requires the interpolated model to be differentiated. However, even though regression minimises the sum of squared residuals, it does not guarantee any properties of the derivative. Furthermore, the literature [7] demonstrated that residuals of fitting operation are characterised by a trend which limits adequateness of fitting. Thus, authors suggest considering direct derivative evaluation to provide a metrological evaluation consistent with the definition of the parameter.

Therefore, the derivative of the unloading force–displacement, $F(h)$, curve at the start of unloading should be evaluated. However, this is not a trivial issue because of the sensitivity of the derivative computation to spikes and measurement noise of the signal to be differentiated. The literature has faced this issue and introduced several solutions that have been later developed to solve differential equation [16]. Algorithms are in general based on the requirement for smoothing local disturbances, which may be due to measurement noise. This is usually achieved by average weighting incremental differences evaluated on different interval widths in the neighbourhood of the studied point.

In this work, the algorithm proposed by Fornberg [17] has been adopted. It computes the derivative of a signal, f', as a function of the signal itself, f, as stated in Eq. (7.1) by means of weights calculation.

$$\left.\frac{\mathrm{d}^M f}{\mathrm{d}^M x}\right|_{x_0} \approx \sum_{\nu=0}^{N} \delta_{N,\nu}^{M} f_{(\alpha_\nu)} \tag{7.1}$$

$$\delta^m_{n,\nu} = \frac{(\alpha_n - x_0)\delta^m_{n-1,\nu} - \delta^{m-1}_{n-1,\nu}}{\alpha_n - \alpha_\nu} \quad (7.2)$$

The algorithm requires the specification of the derivative order M, which in this case has been set to 1, and a precision order N, in this case set to 20, that define the node vector α in which the function is computed. The algorithm computes the weights as function of m, yielding from zero to M, and n, from m to N, and α_ν, see Eq. (7.2).

However, in order to reduce measurement uncertainty, a devoted procedure (named $M1$) has been applied. First, in order to reduce spikes, i.e. outliers, and measurement noise, force and displacement signals have been filtered by means of a mobile average filter. Second, to provide reasonable uncertainty, the trend of the derivative was evaluated by applying Fornberg's algorithm to the filtered signal in a suitably long interval. Finally, S has been evaluated by computing, at the onset of unloading, the fitting curve of the derivative trend obtained by means of a nonlinear regression with a power law model, see Eq. (8), with a proper change in reference system set in $h_{\max}$ to reduce uncertainty. This choice depends on the fact that because $h_{\max}$ is defined at the intercept between a plateau (the hold phase of the indentation curve) and a power law curve in a point with derivative different from zero (onset of unloading), it yields to a less uncertain evaluation with respect to h_p, which is instead at the intercept of the x-axis and a point with almost null derivative of the unloading curve [6, 7].

$$\frac{\partial F}{\partial h} = \beta m h^{m-1} = \beta m (h_{\max} - H)^{m-1} \quad (8)$$

This procedure is necessary to cope with the low number of available points in the neighbourhood of the start of unloading, and to provide the evaluation with an uncertain assessment. $M1$ is applied to the unloading curve portion ranging from 98 to 20% of maximum applied force in line with the standard application of PL [4, 5].

However, numerical evaluation of the derivative is severely affected by random measurement errors, which make high measurement uncertainty to be expected. Therefore, alternative approaches have been investigated to overcome this issue. They are based on the robustness of the secant evaluation of the $F(h)$ unloading curve. These approaches can be exploited to evaluate the derivative trend to extrapolate the contact stiffness by means of linear regression, according to Sneddon [15], by considering secant, d_i, at different positions. In particular, two further methodologies have been considered.

The first (named $M2$) evaluates the secant at different positions as the slope of the regression line of a portion, or window, of unloading curve that yields from the start of unloading to an increasing distance from it.

Table 1 Window width and centre adopted for $M2$ and $M3$ expressed with respect to unloading curve length and from start of unloading, respectively

$M2$			$M3$		
i	Window width (%)	Window centre (%)	i	Window width (%)	Window centre (%)
1	5	2.5			
2	10	5	1	10	5
3	15	7.5	2	10	7.5
4	20	10	3	10	10
5	25	12.5	4	10	12.5
6	30	15	5	10	15

On the other hand, the second (named $M3$) evaluates the regression line on portions, or window, of the unloading curve of the same width, expressed as number of considered points, but centred on different locations.

Once the secants have been computed, they require to be fitted; however, uncertainty associated with the secant evaluation at different positions has to be accounted for properly. In fact, standard deviation of the slope of the regression line, $s(d_i)$, is related to the number of fitted points. Moreover, when considering secant evaluation nearby the start of unloading, greater uncertainty has to be expected due to the noisier signal that is generated in this transient operating condition of the force–displacement transducers. Therefore, to introduce uncertainty effect in the extrapolation of derivative trend, linear fitting is applied to a point cloud built as follows: at the different locations at which secants are computed, a set of one hundred points extracted from a normal distribution $N(d_i, s(d_i))$ is considered. The assumption of normal distribution is supported by the preliminary application of mobile average filter to the measured force and displacement signals devoted to eliminating measurement noise and outliers and the verification of absence of significant systematic components affecting the measurements, and hence slope.

Also in this case, to be consistent with linear derivative approximation, only the initial part of unloading is accounted for by properly choosing the width and position of considered unloading curve portion, which are summarised in Table 1.

2.3 Experimental Set-up

The three methodologies introduced in Sect. 2.2 have been applied to nano-indentations performed on both a reference material, i.e. fused silica (Young's modulus of (73.3 ± 0.6) GPa), and a high-alloyed bearing steel, i.e. Ferrium® C61 (Young's modulus of (205 ± 2) GPa), at two different load levels, 10 and 5 mN. Above-mentioned Young's modulus

Fig. 2 The Hysitron TI 950 indentation platform exploited to perform the indentations

values and the relevant uncertainties were obtained by means of resonance frequency method. Indentations were performed at the Istituto Italiano di Tecnologia (IIT) and at Oklahoma State University (OSU) with two different testing machine platforms manufactured by Hysitron, i.e. two Triboindenter TI 950 (Fig. 2). The adoption of two nominally equal instruments to indent different calibrated specimens of same standard material is aimed to test the generality of results. Indentation performed on reference material and steel was repeated ten times in order to cater for the reproducibility. Data were processed by the authors' implementation in *MATLAB*.

3 Results and Discussion

Comparison of the methodologies that have been already presented in the literature, i.e. SN and LN, or accepted in reference standard, i.e. PL and LE, with the three introduced in the present work will be presented in terms of both the measured contact stiffness and indentation modulus evaluation. Moreover, expanded uncertainty, evaluated consistently with GUM [18], with proper uncertainty propagation, will be assessed to provide results with a metrological consistent framework.

Because frame compliance requires calibration which entails the evaluation of contact stiffness, results will be provided in terms of measured contact stiffness, rather than contact stiffness, to avoid any indirect contribution from calibration of the testing equipment.

First of all, adequateness of the mobile average filter has to be investigated. Therefore, normality of the distribution of both force and displacement residuals was investigated by performing a Chi-squared test with a risk of error of first kind (conventionally set to 5%). The test cannot reject null hypothesis of normal distribution of the residuals, since both force and displacement residuals appear linear when considering their normal probability plot (NPP), as Fig. 3 shows.

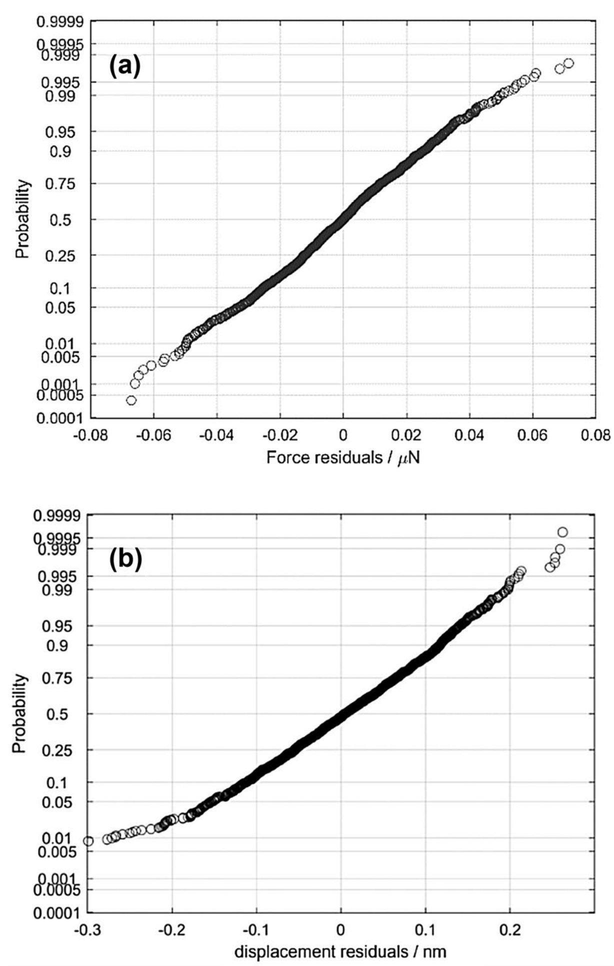

Fig. 3 NPP of mobile average filter residuals of **a** force and **b** displacement. Sample indentation performed on fused silica

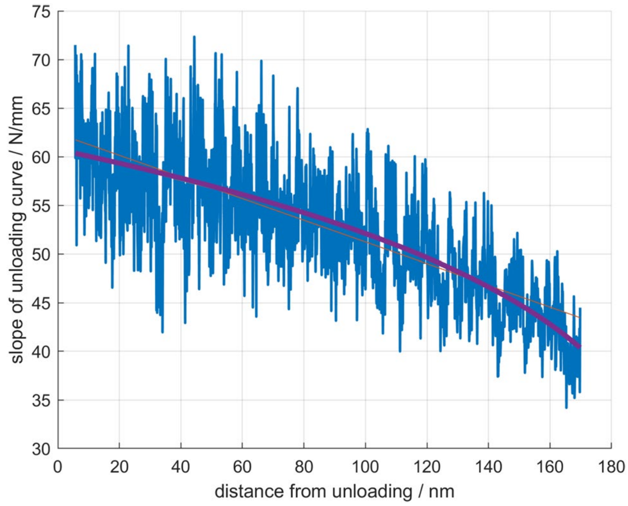

Fig. 4 Results of the application of $M1$ method to sample indentation on fused silica at 10 mN. Blue: slope of the unloading curve as a function of distance from onset of unloading. Orange: linear interpolation of the unloading curve slope, notice unsatisfactory fitting. Purple: power law interpolation of the unloading curve slope

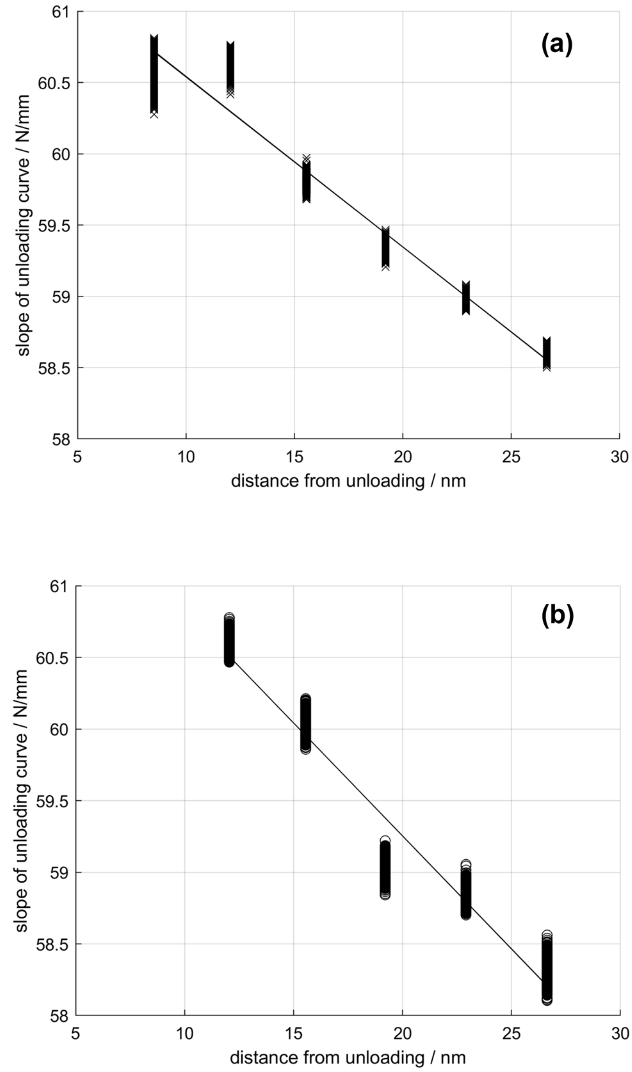

Fig. 5 Results of the application of **a** $M2$ and **b** $M3$ methods to sample indentation on fused silica at 10 mN. Slope of the unloading curve as a function of distance from onset of unloading is shown

Figures 4 and 5 show results of the application of the three proposed methodologies for the derivative direct evaluation. Although Sneddon's solution of contact between a flat surface and a conical indenter [15] represents a reliable first approximation for elasto-plastic regime, the power law regression (purple curve in Fig. 4) has been applied to $M1$ because relevant curvature can be highlighted in the derivative (blue curve in Fig. 4), which hinders from the adoption of a linear model (orange curve in Fig. 4). Fitting with a linear model could be a viable solution considering only the first portion of the curve, i.e. up to 80% F_{max}, to be consistent with Sneddon's theory; however, preliminary studies demonstrated high sensitivity of this approach to local disturbances in the derivative, which led to prefer a power law model to provide suitable robustness. Furthermore, the adoption of such nonlinear fitting is compliant to experimental evidences of Oliver and Pharr [14]. In fact, they highlighted that the actual condition of elasto-plastic contact, met at the onset of unloading, introduces a deviation from Sneddon's theoretical quadratic dependence of force on displacement, which results in a trend of the curve that lies between the linear and the quadratic.

On the other hand, differently from $M1$, linear fitting has been adopted for $M2$ and $M3$ because they consider shorter portion of unloading, but high sensitivity to local fluctuation is shown, see Fig. 5a, b, respectively, which often results in inappropriateness of power law model.

In the following, results are presented in terms of measured contact stiffness and indentation modulus. Figures 6 and 7 show results related to fused silica obtained at the OSU and IIT, respectively. As far as S is concerned, LE and SN provide a relative underestimation, consistent with their definition that evaluates the secant rather than the tangent to the unloading curve. On the other hand, contact stiffness assessed by LN systematically is higher. PL provides S estimation with a higher uncertainty, mostly due to h_p evaluation. This estimation lies between the other literature methodologies, but it is weakly compatible with them.

As far as proposed methodologies are concerned, $M1$ generally provides S estimation similar to PL method with a lower measurement uncertainty, if data are not affected by significant noise. These disturbances demonstrate to lead to evaluation systematically different, such as in the case of indentations of fused silica performed at the Istituto Italiano di Tecnologia at a maximum load of 10 mN.

$M2$ and $M3$ results tend to be compatible with the formerly proposed methods. However, due to their definition,

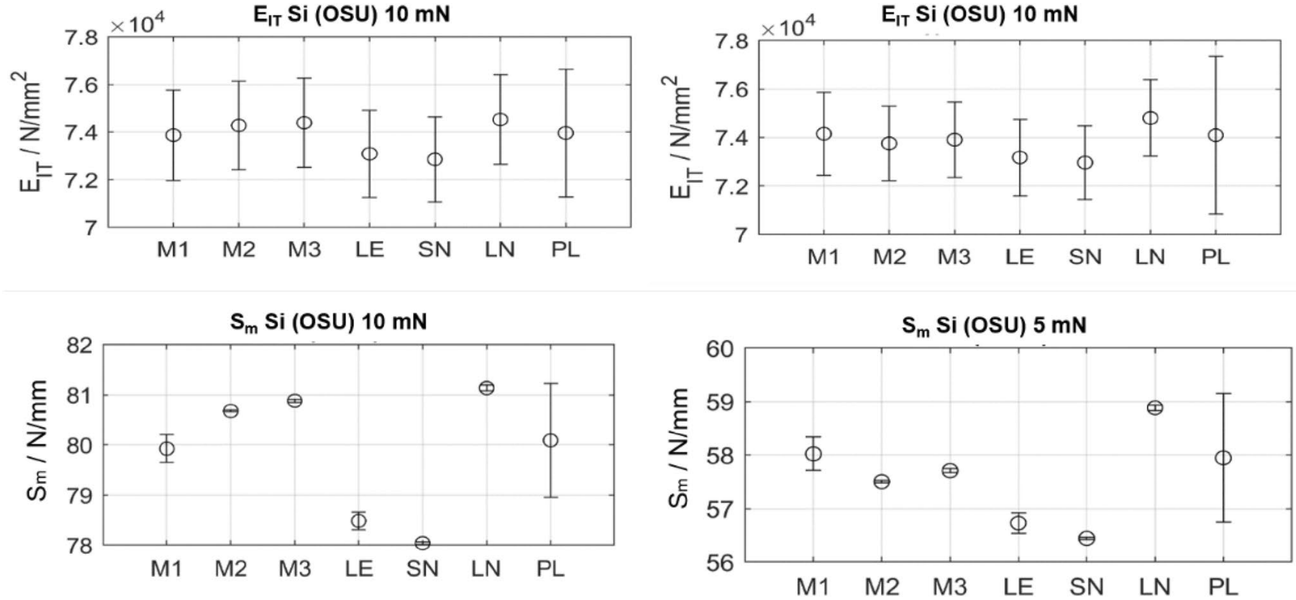

Fig. 6 Indentation modulus and contact stiffness of fused silica indented at 10 and 5 mN at the Oklahoma State University

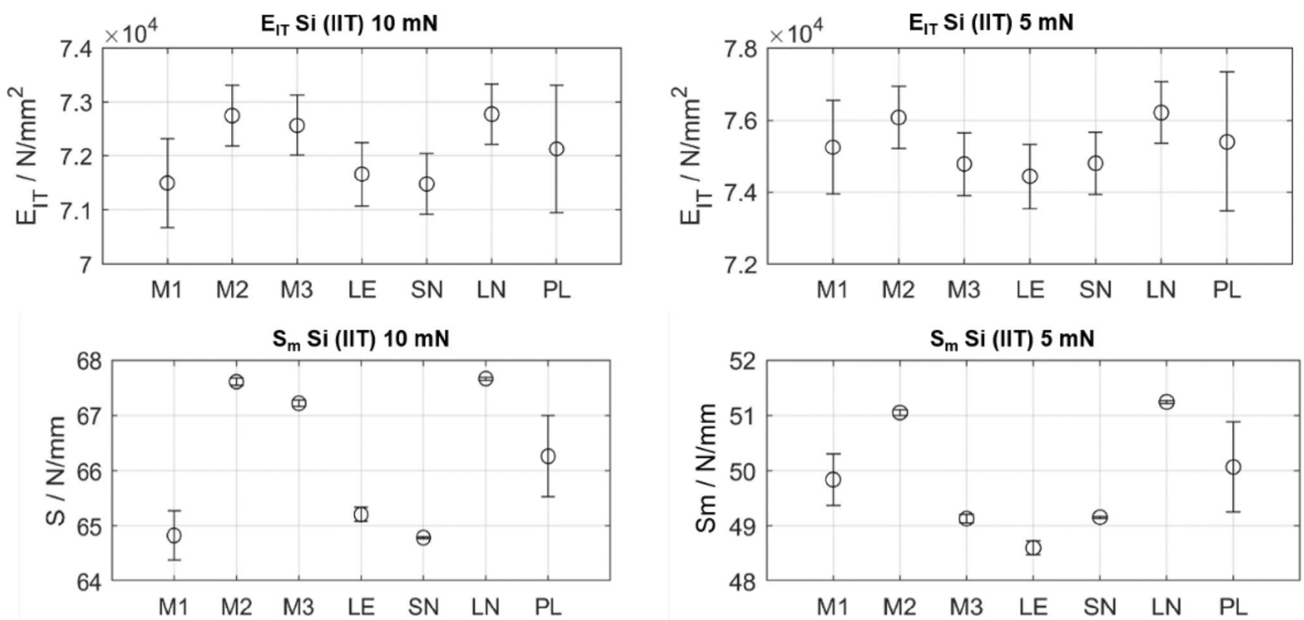

Fig. 7 Indentation modulus and contact stiffness of fused silica indented at 10 and 5 mN at the Istituto Italiano di Tecnologia

which caters for limited unloading curve portion, differently from $M1$, when significant noise is present, disturbances introduce a component which is strongly smoothed by the method, thus never providing discordant values as in $M1$. On the other hand, $M2$ and $M3$ definitions produce fluctuation of the results, which hampers from concluding on their general behaviour and robustness.

Since results on reference material were consistent between different testing machines, a Ferrium® C61 was tested. Also in this case, methods provide S estimation, whose relative trend is in line with indentations performed on fused silica, see Figure 8; in particular, tests performed at 5 mN offer a further example of $M1$ sensitivity to measurement noise. (In fact, related results are not shown for scale issues.)

Given the mathematical relationship between indentation modulus E_{IT} and contact stiffness S (see Eq. 1), estimation of E_{IT} allows similar observations to be drawn, but due to uncertainty propagation, differences are less evident.

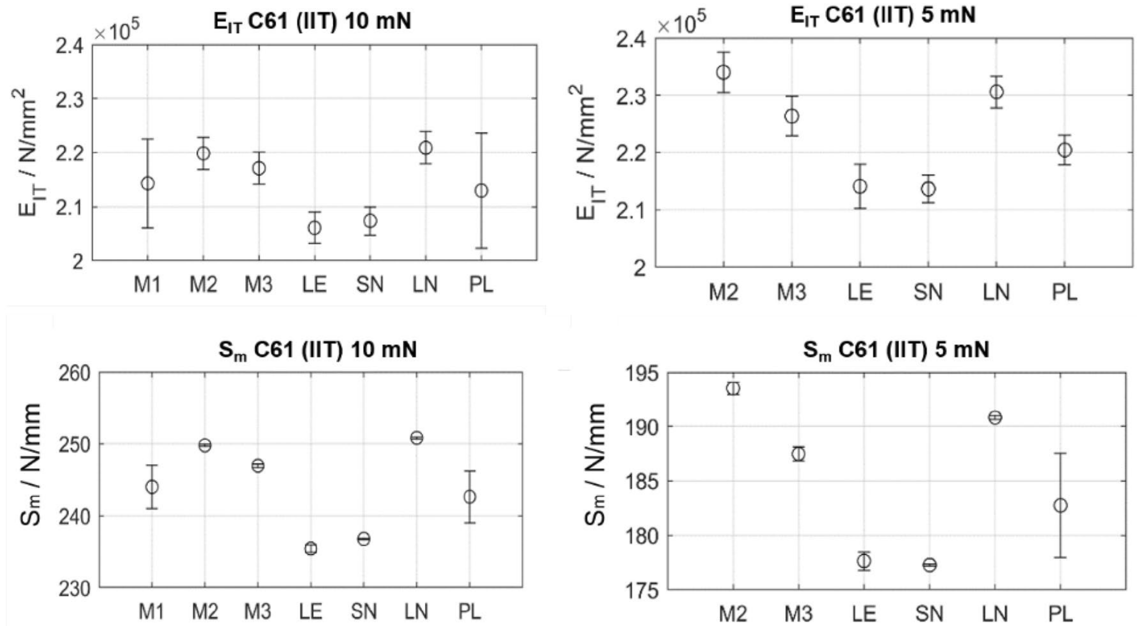

Fig. 8 Indentation modulus and contact stiffness of Ferrium® C61 indented at 10 and 5 mN at the Istituto Italiano di Tecnologia

4 Conclusions

Instrumented indentation test is a powerful mechanical characterisation technique which, being semi- (or non)-destructive and allowing for evaluating hardness, elastic and flow properties and characterising the microstructure of materials, has industrial relevance also in the perspective of product and process quality control.

However, instrumented indentation test standard methods present shortcomings, which results in bias and high measurement uncertainty that affect the results. In particular, the most affecting factors have been demonstrated to be the contact stiffness and the testing equipment calibration.

The present work addressed the evaluation of the contact stiffness in instrumented indentation test by proposing a direct evaluation of the unloading curve derivative, to provide a metrological more robust framework. Three proposed methodologies were compared with the four formerly introduced in the literature.

The first one ($M1$) is based on a finite difference algorithm, while the other ($M2$ and $M3$) exploits the local robustness of secant evaluation.

Experimental results relative to tests performed on different materials and by different testing machines are consistent with each other, thus independent from those factors and from maximum test load. In particular, the proposed methodology based on Fornberg's algorithm ($M1$) appears to be promising. In fact, it improves estimation obtained from power law method (PL) by providing results with a smaller measurement uncertainty, when noise is not significant, which, on the other hand, hinders a robust application of the other two proposed methods.

Future work will be aimed at improving robustness of $M1$ considering application on extended load scales and more material and indenter geometries; furthermore, to provide proper metrological framework, calibration of the testing equipment shall be addressed.

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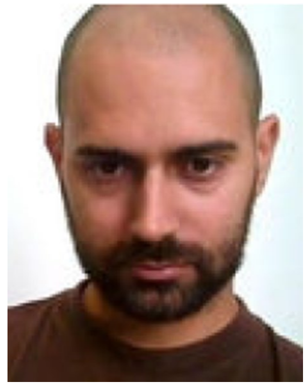

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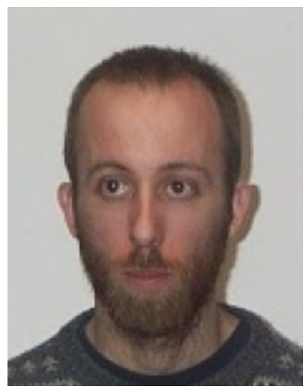

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