"ZERO-POINT" IN THE EVALUATION OF MARTENS HARDNESS UNCERTAINTY

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Abstract
The Instrumented Indentation Test is based on simultaneous recording of force and indentation depth, obtained during test cycle. The force-depth curve, describing the indentation pattern, is typically formed by two parts having the “zero-point” in common, i.e. the first contact point between the indenter and the surface of test piece. The zero-point determination is a crucial aspect for Martens Hardness evaluation, so that relevant ISO standard suggests to estimate it by extrapolation of polynomial fitted functions.
In this paper a new model, based on a segmented function, is proposed. This approach implies the use of maximum likelihood estimator for parameters determination. The corresponding uncertainty is provided through the covariance matrix of the regression model.

Key words: Instrumented Indentation Test, Martens Hardness, Hardness Measurement, Uncertainty in Hardness Measurement, Hardness Standardization.

1 Introduction

Martens hardness is based on the simultaneous measurement of force and depth during the indentation, to get the test pattern, see figure 1. The measurement result is obtained by the identification of a mathematical model, which for a pyramidal indenter is a simple parabola. The identification exercise is less straightforward as it could seem. ISO/DIS 14577-1 Metallic materials -- Instrumented indentation test for hardness and materials parameters -- Part 1: Test method [1], proposes some measurement procedure, but practical drawbacks are not completely overcome, as shown by the uncertainty level in international comparisons [2]. One of the reasons for this situation can be explained by the very weak definition of the zero-point given both for force and depth measurement. Force zero condition seems to be well defined at any time before the contact of the indenter with the surface of the test piece, but looking carefully it is not so. The definition of force zero-point has, at least, two reasons of uncertainty: the first is the dynamic interactions and vibration effects, the second the instrumental noise. In fact, during the free movement of the indenter, inertial forces caused by the mass suspended on the force transducer are produced. The presence of instrumental noise is evident by itself.

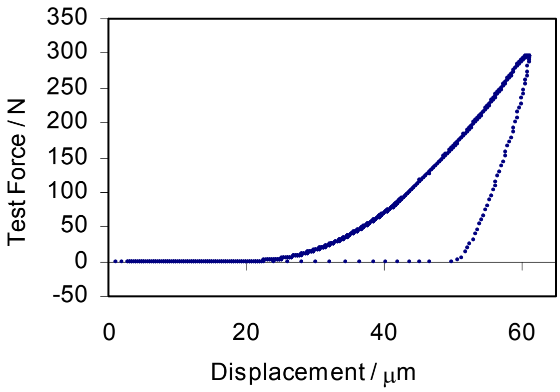

Figure 1: Force/Displacement characteristic curve obtained during an Instrumented Indentation Test (HM 300/54/550 = 6800 N/mm^2).

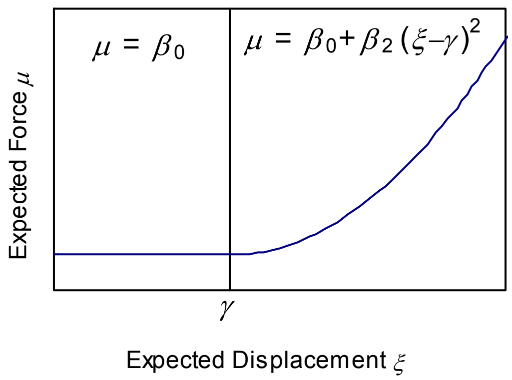

Figure 2: Force/Displacement Curve: segmented polynomial model used to fit the measured data in the neighbourhood of the zero-point (γ is its abscissa).

One of the methods proposed by the ISO Standard (method 2, clause 7.3), that is to establish force zero-point when the force signal shows an increase larger than 10^{-4} F_{max}, is questionable because the force signal noise is frequently larger than this threshold.The other method proposes an extrapolation of a fitted function applied to force and depth measurement values at least up to 10% of the maximum indentation depth. As it is well know, extrapolation is not considered a good practice and, in fact, in this specific case, it produces strong difficulties. As clearly shown by Menčik and Swain [3] the extrapolation procedure is not sufficiently robust, and, as shown by Ullner [4], it can frequently happen that different subsets of the same set of data give very different results, even, in some cases, imaginary numbers.

The problem of defining the origin for force and depth measurement is really important to get the Martens Hardness measurement with an acceptable uncertainty, as was underlined by Grau et al. [5] and, as said before, by Ullner [4], who gives some proposals to get a robust method for the precise determination of the contact point.

In this paper we consider the problem of avoiding both the use of extrapolation of the fitting curve and the application of regression to two separate arbitrarily bounded regions, frequently used to avoid to pollute the fitting of the parabolic part of the pattern with data obtained before of the contact point and vice versa.

Our proposal consists in adopting a segmented model for the Force/Depth Curve, such to cover both the parts before and after the contact point. Remembering the model proposed by the standard in appendix A.3:

$$h = m \cdot \sqrt{F} \tag{1}$$

where h is the indentation depth when the force F is applied and m the identification parameter, the two parts of the segmented model are a constant zero force path before the contact point and a parabolic increase of force after it:

$$\begin{cases} F = 0 & \text{for } h < 0 \\ F = \dfrac{h^2}{m} & \text{for } h \geq 0 \end{cases} \tag{2}$$

This model can be identified by statistical methods even taking into account the need of considering a free position of the contact point, that is an initial force signal F_0 and depth measurement value h_0, in such a way that also the relevant uncertainty of all the parameters determined can be expressed.

2 Statistical management of the Segmented Model

Data collected from the Instrumented Indentation Test in the neighbourhood of the zero-point can be fitted by a segmented polynomial curve, the Force/Displacement Curve (FDC), see figure 2. The first part of the FDC is an horizontal line representing the ”zero-load” or approach phase of the test, in which no force is applied on the indenter by the tested surface, but dynamic forces and a zero error of force measurement can be present. Let’s call it $\beta_0 = F_0$. As soon as contact is established, when the displacement transducer measures a position $\gamma = h_0$, a contact force is generated and, from that moment on, the FDC takes the shape of a second order polynomial [1].
Denoting with ξ the expected displacement of the indenter and μ the expected test force, the mathematical expression of the FDC is:

$$\begin{cases} \mu = \beta_0 & \text{for } \xi < \gamma \\ \mu = \beta_0 + \beta_2 (\xi - \gamma)^2 & \text{for } \xi \geq \gamma \end{cases} \tag{3}$$

where γ and β_0 were defined before as the abscissa of the zero-point and the expected force signal before contact, while β_2 is related to the parabolic phase of the curve and corresponds to the inverse of m of the model given by ISO standard. Let $\boldsymbol{\theta} = (\gamma, \beta_0, \beta_2)^T$ be the vector of the structural parameters of the FDC, the relationship (3) can be rewritten as: $\mu = f(\xi; \boldsymbol{\theta})$.
It is assumed that ξ and μ are measured with random errors, so that the random variables X and Y are observed instead; the relation is:

$$\begin{cases} X = \xi + \varepsilon_X \\ Y = \mu + \varepsilon_Y \end{cases} \tag{4}$$

where ε_X and ε_Y are model experimental errors.
Let $\mathbf{X} = (X_1, ..., X_n)^T$ be the vector of the n random variables representing the displacement measurements, $\boldsymbol{\xi} = (\xi_1, ..., \xi_n)^T$ be the vector of the corresponding expected values and $\boldsymbol{\varepsilon}_\mathbf{X} = (\varepsilon_{X_1}, ..., \varepsilon_{X_n})^T$ be the vector of relevant random errors. The same matrix notation can be used for the force and error vectors $\mathbf{Y}$, $\boldsymbol{\mu}$ and $\boldsymbol{\varepsilon}_\mathbf{Y}$.
The statistical measurement error model [6,7] may be defined as follows:

$$\mathbf{X} = \boldsymbol{\xi} + \boldsymbol{\varepsilon}_\mathbf{X}, \quad \mathbf{Y} = \boldsymbol{\mu} + \boldsymbol{\varepsilon}_\mathbf{Y}, \quad \boldsymbol{\mu} = \mathbf{f}(\boldsymbol{\xi}; \boldsymbol{\theta}) \tag{5}$$

2.1 Maximum Likelihood Estimators (MLEs)

In the calibration process of the force and displacement measuring instruments, systematic errors are compensated and instrumental uncertainties are evaluated. Therefore, the error random variables can be assumed independent and normally distributed, with zero average and known standard deviation, so that $\boldsymbol{\varepsilon} = (\boldsymbol{\varepsilon}_\mathbf{X}^T, \boldsymbol{\varepsilon}_\mathbf{Y}^T)^T \sim N(\mathbf{0}, \sigma^2 \mathbf{W})$.
The hypotheses introduced on the error distributions enable to write the log-likelihood function [8]:

$$\ell(\boldsymbol{\theta},\boldsymbol{\xi}) \propto -\frac{n}{2} log\,\sigma^2 - \frac{1}{2\sigma^2}\boldsymbol{\varepsilon}^T \mathbf{W}^{-1}\boldsymbol{\varepsilon} \qquad (6)$$

The Maximum Likelihood Estimators (MLEs) of the unknown parameter vectors, $\hat{\boldsymbol{\theta}}_n^{ML}$ e $\hat{\boldsymbol{\xi}}_n^{ML}$, are obtained maximizing the non-linear function (6). The problem can be solved by means of the Gauss-Newton's iterative method [8,9] based upon linear approximations obtained from the expansion of (6) in the Taylor series truncated at the first term. Furthermore, in order to achieve fast convergence and low computational time the Gauss-Newton method can be improved by means of the "trust region strategy" [10], in which the differences between the parameter values in two successive iterations are taken under control.
Moreover the iterative procedure of the Gauss-Newton's method requires to give the initial values of the unknown parameters, $\boldsymbol{\theta}^{(0)}$ e $\boldsymbol{\xi}^{(0)}$. The initial value of $\boldsymbol{\xi}$ is set using the observed displacements vector **x**, whereas the initial values of the structural parameters can be obtained relaxing the assumptions on the measurement errors. Neglecting the indentation measurement errors, the initial value of $\boldsymbol{\theta}$ is calculated maximizing the profile log-likelihood function [11] which is a conditional likelihood respect to the unknown parameter γ.

2.2 Uncertainty of the zero-point

Statistical properties of $\hat{\boldsymbol{\theta}}_n^{ML}$ have been analysed executing Monte Carlo simulations and acceptable results have been obtained. In fact, the estimators do not have large bias and experimental distributions follow the expected normal behaviour, see figures 3 and 4.
The standard uncertainty of the MLEs can be calculated from the covariance matrix [8,12]:

$$\mathbf{V} = \sigma^2\left(\mathbf{J}^T\boldsymbol{\psi}\mathbf{J}\right)^{-1} \qquad (7)$$

where $\mathbf{J} = \partial\boldsymbol{\mu}/\partial\boldsymbol{\theta}$, $\mathbf{M} = \partial\boldsymbol{\mu}/\partial\boldsymbol{\xi}$, $\boldsymbol{\psi} = \mathbf{C} - \mathbf{C}\mathbf{M}^T\left(\mathbf{A} + \mathbf{M}\mathbf{C}\mathbf{M}^T\right)^{-1}\mathbf{M}\mathbf{C}$ and $\mathbf{W}^{-1} = \begin{pmatrix} \mathbf{A} & \mathbf{0} \\ \mathbf{0} & \mathbf{C} \end{pmatrix}$.

In fact, the matrix **V** is an estimator of Var($\hat{\boldsymbol{\theta}}_n^{ML}$) and, although the analysis of simulation results shows that the variance estimators of the structural parameters are biased, the biases are lower than 5%. An estimate of the standard uncertainty of the zero-point is, therefore, the

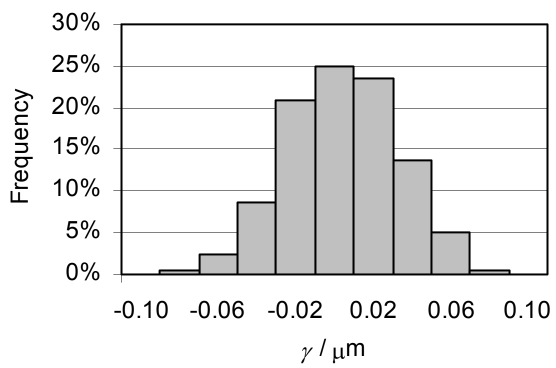

Figure 3: Histogram of the empirical distribution of $\hat{\gamma}_n^{ML}$ based on 1000 sample. The data for the Monte Carlo study have been generated using the model (5) with $\boldsymbol{\theta}$ =(0,0,0.1837)^T. Measurement errors have been considered independent and normally distributed with a standard uncertainty on the depth equal to 0.015 μm and a standard uncertainty on the force equal to 0.03 N.

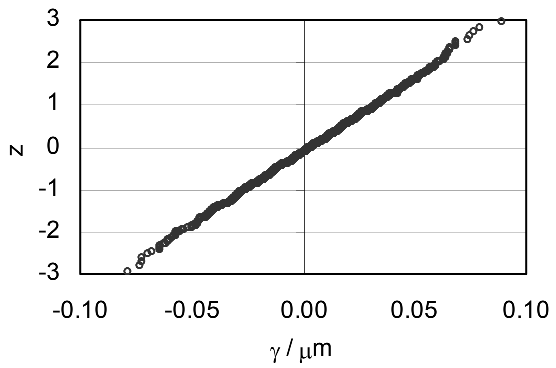

Figure 4: Normal probability plot of the empirical distribution of $\hat{\gamma}_n^{ML}$ based on 1000 sample. The data for the Monte Carlo study have been generated as reported for figure 3.

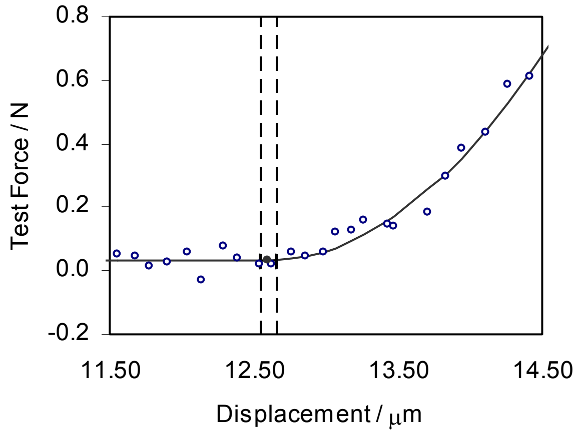

Figure 5: Experimental data (o) fitted by segmented regression curve (-). The maximum likelihood estimate of the abscissa of the zero-point is 12.60 μm and its 95% confidence interval is (12.54;12.66) μm (--).

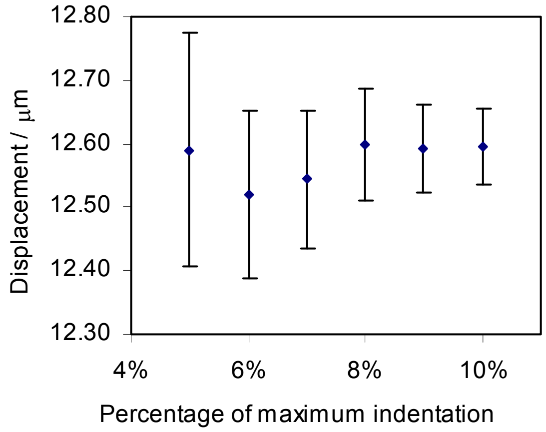

Figure 6: 95% confidence intervals of the abscissa of the zero-point calculated involving different percentages of maximum indentation depth. The uncertainty decreases with the increase of indentation depth used for the fitting.

square root of V_{11}. An 1-α confidence interval on the abscissa of the zero-point can be constructed remembering that the asymptotic distribution of $\hat{\gamma}_n^{ML}$ is a Gaussian random variable.

3 Experimental analysis

Some tests have been performed using the IMGC (Istituto di Metrologia Gustavo Colonnetti) Primary Hardness Standard Machine following the relevant ISO standard specifications [1]. Forces were generated by dead weights and continuously measured by a load cell with resolution of 10 mN. Displacements were measured using a laser interferometer system with a resolution of 0.01 μm. In order to identify the zero-point, 70 measured points around it were selected considering 10% of the maximum indentation depth. An automatic procedure has been implemented for solving the zero-point problem and the maximum likelihood estimates of the abscissa of the zero-point and of the structural parameters have been obtained using the Gauss-Newton's iterative method:

$$\hat{\gamma}_n^{ML} = 12.60\,\mu\text{m}$$
$$\hat{\beta}_{0n}^{ML} = 0.03\ \text{N}$$
$$\hat{\beta}_{2n}^{ML} = 0.0345\ \text{N}/\mu\text{m}^2$$

The estimate of the standard uncertainty of the abscissa of the zero-point obtained from the covariance matrix is 0.03 μm, so the 95% confidence interval results (12.54; 12.66) μm, see figure 5.

Conclusion

This paper describes a new model for determining the force-indentation curve parameters required in hardness Instrumented Indentation Test. The approach is based upon segmented regression method. Parameters are estimated as well as their uncertainties.

Application of segmented regression model enables to overcome some drawback present in traditional approaches. In fact the proposed method allows a completely automatic determination of the FDC parameters, avoiding the risk of imaginary results and any need to split the data in two arbitrarily separated regions. The algorithm, based upon a Gauss-Newton's iterative method, converges quickly to the MLEs, so that an estimate of the zero-point is always guaranteed. Uncertainty of FDC parameters, including zero-point abscissa, can be evaluated, so allowing the expression of Martens Hardness uncertainty, as required by relevant standard.
Additional advantages of this method are the fast convergence and the low computational time.

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