

## AN APPLICATION OF THE ASYMPTOTIC THEORY TO A THRESHOLD MODEL FOR THE ESTIMATE OF MARTENS HARDNESS

G. Vicario, G. Barbato, G. Brondino

## 1. INTRODUCTION

Hardness measurements are widely carried out in industry, since traditional hardness scales, such as the Brinell, Rockwell, Vickers and Knoop offer good performances at a reasonable cost. In all of them an indenter (spherical, conical with a spherical tip, pyramidal) is pressed against the flat surface under test with a given force (figure 1), and either the depth or the width of the indentation is measured; the larger the indentation, the lower the hardness is. The measurement process is simple and straightforward, and results are very useful even when a single reading is obtained for force and indentation. Indeed, the close connection between the hardness number and the tensile strength often permits to substitute a much cheaper and faster hardness test for an expensive tensile test. The limitations of traditional hardness methods in getting information from a single force have been got over by introduction of the "Instrumented Indentation Test" (ISO/DIS-14577-1:2000), whereby a continuous record of both applied force and indentation depth is used. The part of this new method closer to a hardness number is called "Martens Hardness".

In a first approximation, hardness is directly proportional to the ratio between the applied force and the indentation surface, that is the average pressure, as proposed by Brinell and confirmed also for other indenters, for example, those pyramid shaped; the force vs. depth pattern should therefore follow a parabolic law evolution. The indenter, usually a diamond pyramid with a base either square (Vickers type) or triangular (Berkovich type), is pressed into the surface under test; the force signal should be constant, and almost zero, until the indenter contacts the sample surface. From that moment onward, the force should increase, versus the indentation depth, with a parabolic law.

As instruments used to measure force and indentation depth are affected by systematic and random errors, results deviate from the theoretical pattern (figure 2). A force measuring transducer is usually affected by a systematic *zero error* (therefore when no force is applied the relevant signal must be considered) and random errors, normally distributed around the *zero error*, with an almost constant standard deviation. As regards the parabolic part, systematic errors of both

force and indentation depth measurements can be considered negligible; however, owing to random errors, measurement signals can be considered normally distributed, the force signal with a slowly increasing standard deviation and the indentation signal with an almost constant standard deviation.

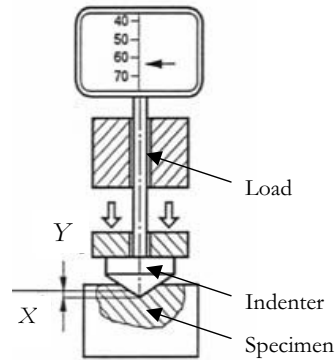


Figure 1 – Scheme of a hardness measuring machine.

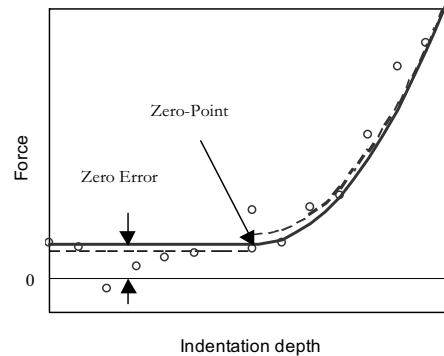


Figure 2 – Theoretical pattern (-), experimental data (o) and regression patterns of the constant and parabolic parts (--); in this case the two parts do not intersect.

The International Standard Organization (ISO/DIS-14577-1:2000, p. 7) requires to estimate the position of the *zero-point*, that is the first contact point of the indenter with the test piece surface, and its uncertainty. Usually, the ISO requirement is met by determining the *zero error* of the force measuring instrument through averaging the measurement results obtained before contact of the indenter with the sample surface, and therefore by determining the regression parabola by means of the data obtained after contact, corrected for *zero error*. The indentation *zero-point* is then determined as the intersection of the initial horizontal line with the parabolic curve which follows (Mencik and Swain, 1994; Ullner and Quinn, 1997).

This method has, evidently, some drawbacks:

a. owing to the data pattern around the contact point, that region cannot easily be split into two sets, one being attributed to the constant part and the other to the parabolic part; an arbitrary part of data, the more important being the nearer to the contact point to be determined, may therefore arbitrarily be excluded from regression calculation;

b. the point to be determined at the end of the constant part, coincides with the apex of the parabola; since both lines have there the same tangent, identification of the abscissa of that point entails an ill-conditioned problem;

c. as shown by Ullner (2000), in some cases the routine for identification of the point under consideration fails, as no intersection can be found between them (figure 2).

To find a solution to these problems, in the next section we propose to adopt a single segmented model, constant in its first part and parabolic in the second one.

## 2. DESCRIPTION OF THE MODEL

Data collected from the “Instrumented Indentation Test” can be fitted by a segmented curve, the Force/Depth Curve (FDC), as figure 3 shows. The first part of the FDC is a horizontal line representing the *zero-load* or approach phase of the test, in which no force is applied by the indenter to the specimen.

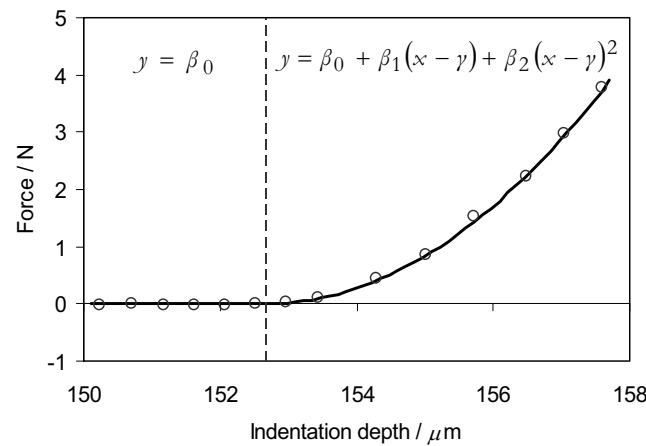


Figure 3 – Force/Depth Curve, showing applied force  $y$  plotted against indentation depth  $x$ . A segmented curve is fitted to measurement data obtained during the “Instrumented Indentation Test”. Abscissa of the *zero-point* ( $\gamma$ ), corresponding to initial contact of the indenter with the specimen, is the unknown parameter of interest.

As soon as contact is established, a force is generated and from that moment the FDC takes the form of a second order polynomial (Grau *et al.*, 1994); the contact point between the horizontal line and the parabola is the *zero-point*. In accordance with continuity considerations, the model of the “Instrumented Indentation Test” may be written:

$$\begin{cases} y = \beta_0 & \text{for } x < \gamma \\ y = \beta_0 + \beta_1(x - \gamma) + \beta_2(x - \gamma)^2 & \text{for } x \geq \gamma \end{cases} \quad (1)$$

where  $x$  is the indentation depth and  $y$  the applied force. The parameters  $\beta_j$  ( $j=0,1,2$ ) are related to the FDC, the threshold parameter  $\gamma$  is the abscissa of the *zero-point*.

Assumption of FDC continuity and of its first derivative appears to be reasonable. Therefore model (1) becomes:

$$y = \beta_0 + \beta_2 \left( [x - \gamma]^+ \right)^2 \quad (2)$$

where  $[x - \gamma]^+ = \max(0, x - \gamma)$  is the positive part of  $x - \gamma$  (Gallant and Fuller, 1973).

Values of variables  $x$  and  $y$  at the measurement points are not observable. The values of the two random variables  $X_i$  and  $Y_i$ , instead, are observable:

$$\begin{cases} X_i = x_i + \varepsilon_{X_i} \\ Y_i = y_i + \varepsilon_{Y_i} \end{cases} \quad \text{con } i = 1, \dots, n; \quad (3)$$

where  $\varepsilon_{X_i}$  and  $\varepsilon_{Y_i}$  are the model experimental errors, unavoidable in both force and indentation depth measurements. By substituting (3) into (2), one obtains:

$$Y = \beta_0 + \beta_2 \left( [X - \varepsilon_X - \gamma]^+ \right)^2 + \varepsilon_Y. \quad (4)$$

Whereas (2) is defined as a *functional relation* between the two mathematical variables  $x$  and  $y$ , equation (4) is a *structural relation* between the observable random variables  $X$  and  $Y$  (Kendall and Stuart, 1973; Fuller, 1987).

Even if the only parameter of interest is  $\gamma$ , the remaining nuisance parameters in (4) can be split into structural and incidental. Structural nuisance parameters  $\beta_0$  and  $\beta_2$ , common to all observations, are related to the form of the FDC. Incidental nuisance parameters are specific to individual observations and to the structural relationship of model (4). There are as many incidental parameters as the observed values, and they are the actual values of indentation depth. Therefore if there are  $n$  pairs of measurement data, there are  $n+3$  unknown parameters to be estimated (table 1).

TABLE 1  
*Classification of the parameters*

OF INTEREST	PARAMETERS	
	NUISANCE	
	Structural	Incidental
$\gamma$	$\beta_0 \quad \beta_2$	$x_i$ with $i=1, \dots, n$

Model (4) is an example of a change-point problem, whose behaviour depends on the unknown contact point parameter  $\gamma$ . In practice, since  $\gamma$  can be considered a threshold parameter, model (4) can also be termed a threshold model.

For the sake of concision let us resort to matrix notations. Let  $\mathbf{x}=(x_1, \dots, x_n)^T \in \mathbb{R}^n$  be the vector of  $n$  actual values of the mathematical variable  $x$ ;  $\mathbf{X}=(X_1, \dots, X_n)^T \in \mathbb{R}^n$  be the vector of the corresponding random variables. The same notation can be used for the dependent variables  $\mathbf{y}=(y_1, \dots, y_n)^T \in \mathbb{R}^n$ ,  $\mathbf{Y}=(Y_1, \dots, Y_n)^T \in \mathbb{R}^n$  and  $y_i = \beta_0 + \beta_2 \left( [x_i - \gamma]^+ \right)^2$ , with  $i = 1, 2, \dots, n$ . According to the previous notation, the vector of the unknown parameters is:

$$\theta = \left( \gamma, \beta^T, \mathbf{x}^T \right)^T \in \mathbb{R}^{n+3}$$

where  $\beta = (\beta_0, \beta_2)^T \in \mathbb{R}^2$  is the vector of the structural nuisance parameters. Consequently, the relation (2) between mathematical variables can be expressed in matrix form:

$$\mathbf{y} = \mathbf{A}(\gamma, \mathbf{x})\beta \tag{5}$$

where  $\mathbf{A}(\gamma, \mathbf{x}) = (\mathbf{1}_n \quad \Delta^2 \mathbf{1}_n) \in \mathbb{R}^{n,2}$ ,  $\Delta = \text{diag}\{[x_i - \gamma]^+\} \in \mathbb{R}^{n,n}$  and  $\mathbf{1}_n = (1, \dots, 1)^T$ .

### 3. MAXIMUM LIKELIHOOD ESTIMATORS

In the calibration process of force and indentation measuring instruments systematic errors are compensated for and instrumental uncertainties are evaluated. Therefore, error random variables can be assumed independent and normally distributed, with zero average and known standard deviation. The performance of an indentation measuring instrument is assumed to be constant over its range; therefore indentation depth errors  $\varepsilon_{X_i}$  have common standard deviation  $\sigma_{\varepsilon_X}$ . On the contrary, errors  $\varepsilon_{Y_i}$  in the measured force as a rule increase with  $y$ . Such dependence is compatible with a linear model  $\sigma_{\varepsilon_{Y_i}} = a + bY_i$ , with  $i = 1, 2, \dots, n$ , where  $a$  represents the absolute component and  $b$  takes account of the effect of force. These assumptions on measurement errors can be summarized as:

$$\varepsilon \sim N\left(\mathbf{0}_{2n}, \sigma_{\varepsilon_X}^2 (\mathbf{W}^T \mathbf{W})^{-1}\right) \in \mathbb{R}^{2n} \tag{6}$$

where  $\varepsilon = (\varepsilon_X^T, \varepsilon_Y^T) \in \mathbb{R}^{2n}$ ,  $\varepsilon_X$  and  $\varepsilon_Y$  are, respectively, the error vectors of indentation and force determinations. The weighted matrix  $\mathbf{W}$  is a block diagonal matrix:

$$\mathbf{W} = \begin{pmatrix} \mathbf{I}_n & \mathbf{0}_n \\ \mathbf{0}_n & \mathbf{D}_n \end{pmatrix}$$

with  $\mathbf{I}_n$  being the identity matrix and  $\mathbf{D}_n = \text{diag}\{\sigma_{\varepsilon_X} / \sigma_{\varepsilon_{Y_i}}\}$ .

The hypothesis introduced on error distribution enable the log-likelihood function to be written as:

$$\ell(\theta) \propto -\frac{1}{2\sigma_{\varepsilon_X}^2} \left\{ \begin{bmatrix} \mathbf{X} - \mathbf{x} \\ \mathbf{Y} - \mathbf{y} \end{bmatrix}^T \mathbf{W}^T \mathbf{W} \begin{bmatrix} \mathbf{X} - \mathbf{x} \\ \mathbf{Y} - \mathbf{y} \end{bmatrix} \right\} = -\frac{1}{2\sigma_{\varepsilon_X}^2} \|\mathbf{g}(\theta)\|_2^2 \tag{7}$$

where  $\mathbf{g}(\theta) = \mathbf{W} \begin{pmatrix} \mathbf{X} - \mathbf{x} \\ \mathbf{Y} - \mathbf{A}(\gamma, \mathbf{x})\beta \end{pmatrix} \in \mathbb{R}^{2n}$ .

The Maximum Likelihood Estimator (MLE) of the unknown parameter vector  $\theta$  is obtained by maximizing (7) or, equivalently,  $-\frac{1}{2}\|\mathbf{g}(\theta)\|_2^2$ . This is a non-linear problem which can be solved by means of the Gauss-Newton iterative method, since vector  $\mathbf{g}(\theta)$  is non-linear in  $\theta$ . The solution  $\theta^{(k)}$  at the  $k$ -th step of the iteration method is achieved by solution of the linearised form obtained from expansion of  $\mathbf{g}(\theta)$  in the Taylor series truncated at the first term. Then, the linearised problem at the  $k$ -th iteration is:

$$\max_{\theta} -\frac{1}{2}\|\mathbf{g}^{(k)} + \mathbf{J}^{(k)}(\theta^{(k+1)} - \theta^{(k)})\|_2^2 \quad (8)$$

where  $\mathbf{g}^{(k)} = \mathbf{g}(\theta^{(k)})$  and  $\mathbf{J}^{(k)} = \mathbf{J}(\theta^{(k)})$  is the Jacobian of  $\mathbf{g}(\theta)$ . If  $\mathbf{J}^{(k)}$  has a full rank, the solution of (8) is:

$$\theta^{(k+1)} = \theta^{(k)} - \left[\mathbf{J}^{(k)T} \mathbf{J}^{(k)}\right]^{-1} \mathbf{J}^{(k)T} \mathbf{g}^{(k)}. \quad (9)$$

Otherwise it is necessary before that, to resort to a QR decomposition of  $\mathbf{J}^{(k)}$ . In the *zero-point* problem the Gauss-Newton iterative method is not ill-conditioned and quickly converges to  $\hat{\theta}$ . If  $\hat{\theta}$  is a local maximum, then it is the MLE. Unfortunately,  $\ell(\theta)$  is not twice continuously differentiable because its second derivatives are discontinuous in  $\mathbf{x}$  and  $\gamma$ . Nevertheless, if there is a neighbourhood of  $\hat{\theta}$  exists in which  $\ell(\theta)$  is twice continuously differentiable and the Hessian matrix in it is negative definite, then  $\hat{\theta}$  is the MLE. The use of a trust region strategy (Dennis and Schnabel, 1983) can improve the Gauss-Newton method. In such case, formula (8) is solved by checking the step length by means of the trust region  $\|\theta^{(k+1)} - \theta^{(k)}\|_2 \leq r$ , where  $r$  is the trust region radius.

In order to achieve fast convergence and low computational time, the initial value of the iterative requires careful selection. Moreover, practical reasons suggest that the choice of an initial value  $\theta^{(0)}$  should not be left to the user alone and that an automatic estimation procedure should be adopted. The proposed suggestion is based upon the use of profile likelihood (Barnforff-Nielsen and Cox, 1994) to estimate the initial values both of the *zero-point* and of the structural nuisance parameters, relaxing the assumptions on measurement errors. In fact, if errors in indentation measurements can be neglected, then  $\varepsilon_{\mathbf{x}}$  is the null vector and the unknown parameters are  $\theta_s = (\gamma, \beta^T)^T$ . Besides, if the relative component of force uncertainty,  $b$ , is assumed not to be significant, then force measurements errors are homoscedastics and  $\varepsilon_{\mathbf{y}} \sim N(\mathbf{0}_n, \sigma_{\varepsilon_{\mathbf{y}}}^2 \mathbf{I}_n)$ . Under less strict assumptions, the structural relationship (4) becomes a classic non-linear regression model:

$$\mathbf{Y} = \mathbf{A}(\gamma)\beta + \varepsilon_{\mathbf{Y}}. \tag{10}$$

The vector  $\theta_s$  of unknown parameters is made up of two parts, one is the parameter of interest  $\gamma$ , the other is the vector of structural nuisance parameters  $\beta$ . The parameter  $\gamma$  may take values in  $(X_{\min}, X_{\max})$ , where  $X_{\min}$  and  $X_{\max}$  are, respectively, the minimum and the maximum measured depth; over this interval the profile log-likelihood function (figure 4) of the model is:

$$\ell_p(\gamma) = \max_{\theta_s|\gamma} \ell(\theta_s) \propto -\frac{1}{2\sigma_{\varepsilon_Y}^2} \left\| \mathbf{Y} - \mathbf{A}(\gamma)\hat{\beta}_\gamma \right\|_2^2 \tag{11}$$

where  $\hat{\beta}_\gamma$  denotes the maximum likelihood estimate of  $\beta$  for a given value of  $\gamma$ .

If  $\gamma$  is fixed, equation (10) becomes a polynomial regression model and  $\hat{\beta}_\gamma$  is obtained from the well-known formula:

$$\hat{\beta}_\gamma = [\mathbf{A}(\gamma)^T \mathbf{A}(\gamma)]^{-1} \mathbf{A}(\gamma)^T \mathbf{Y}.$$

The profile likelihood estimate of the *zero-point*,  $\hat{\gamma}_p$ , can be obtained by maximizing the profile log-likelihood evaluated at a finite number of points  $\gamma_q$ , for  $q=1,2,\dots,m$ .

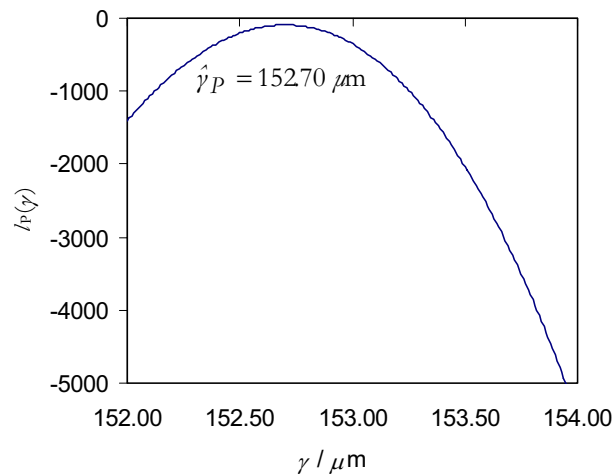


Figure 4 – Graph of the Profile log-Likelihood Function:  $\ell_p(\gamma)$  is plotted against the parameter of interest  $\gamma$ .

Consequently,  $\hat{\gamma}_p$  and  $\hat{\beta}_{\hat{\gamma}_p}$  are the estimates of the unknown parameters of model (10) and they can be used as start values of  $\theta$  in the Gauss-Newton iterative method together with the observed vector  $\mathbf{X}$  used for setting the start value of

x. The algorithm for the automation calculus of the initial value of  $\theta$  consists in the following steps:

- Step 1            Fix  $\gamma_q \in (X_{\min}, X_{\max})$  ( $q = 1, 2, \dots, m$ );  
 Step 2            For  $q$  from 1 to  $m$ :  
                   compute  $\hat{\beta}_{\gamma_q}$ ,  
                   evaluate  $\ell_p(\gamma_q)$ ;  
 Step 3            Estimate  $\gamma$  from  $\ell_p(\hat{\gamma}_p) = \max \ell_p(\gamma_q)$ ;  
 Step 4            Start the Gauss-Newton iterative method with use of the initial unknown parameter:

$$\theta^{(0)} = \begin{pmatrix} \hat{\gamma}_p \\ \hat{\beta}_{\hat{\gamma}_p} \\ \mathbf{X} \end{pmatrix}.$$

By substitution of  $\theta^{(0)}$  in (9), the starting value of the Gauss-Newton iterative method, the maximum likelihood estimate of  $\theta$  is obtained.

Asymptotically, the variance-covariance matrix of the MLEs may be estimated by:

$$\hat{\mathbf{V}} = \sigma_{\varepsilon_x}^2 \left( \mathbf{J}(\hat{\theta})^T \mathbf{J}(\hat{\theta}) \right)^{-1}. \quad (12)$$

It is well-known (Seber and Wild, 1989) that MLEs of structural parameters are not necessarily consistent, when incidental parameters exist. Unfortunately the presence of incidental parameters doesn't guarantee that MLEs are unbiased. Nevertheless, Monte Carlo simulations (see section 4) showed that such a deviation is less than 5% and therefore compatible with the usual metrology requirements of uncertainty expression.

If vector  $\mathbf{g}$  in (7) is adequately approximated by a linear function in the neighbourhood of  $\hat{\theta}$ , a confidence interval for  $\hat{\theta}$  (see section 5) can be evaluated from  $\hat{\mathbf{V}}$ . In the *zero-point* problem, the validity of the linear assumption is confirmed by the computation of the parameter-effects curvature and intrinsic curvature indices, respectively  $K_{\max}^T$  and  $K_{\max}^N$ , according to Bates and Watts (1988):

$$K_{\max}^T = \max_{\mathbf{u} \in \Theta} \frac{\|\mathbf{u}^T \mathbf{J}_{\bullet}^T(\hat{\theta}) \mathbf{u}\|}{\|\mathbf{J}(\hat{\theta}) \mathbf{u}\|^2}, \quad K_{\max}^N = \max_{\mathbf{u} \in \Theta} \frac{\|\mathbf{u}^T \mathbf{J}_{\bullet}^N(\hat{\theta}) \mathbf{u}\|}{\|\mathbf{J}(\hat{\theta}) \mathbf{u}\|^2} \quad (13)$$

where  $\mathbf{J}_{\bullet}^T(\hat{\theta})$  and  $\mathbf{J}_{\bullet}^N(\hat{\theta})$  are the tangential and normal components of the first derivative of  $\mathbf{J}$  evaluated at point  $\hat{\theta}$ .  $\mathbf{u}$  is any versor in the parameter space  $\Theta$ .



4. COMPUTER SIMULATION

In order to investigate the properties of  $\hat{\theta}$ , we have resorted to the Monte Carlo method. The data for the Monte Carlo study were generated by means of the model (2) with  $\gamma = 0$ ,  $\beta_0 = 0$ ,  $\beta_2 = 0.145$  and a sample size of 60. Indentation measurement errors were generated as a random sample from normal distributions having zero average and  $\sigma_{\varepsilon_X} = 0.02 \mu\text{m}$ , whereas force measurement errors having zero average and  $\sigma_{\varepsilon_{Y_i}} = (0.04+0.001 Y_i) \text{ N}$ . For this parameter set,  $10^4$  samples were generated and for each sample the ML estimates of the structural parameters and of the variance-covariance matrix were evaluated. Results of estimations and statistical indices from these simulations are summarized in table 2 and 3. The estimators do not exhibit large bias and their empirical distributions are not far from normal distributions. We obtained similar results from simulations with different sets of parameters and different sample sizes.

TABLE 2

*Monte Carlo indices of MLEs of structural parameters based on  $10^4$  samples. The samples were generated using model (2) with  $\gamma=0$ ,  $\beta_0 = 0$  and  $\beta_2 = 0.145$*

Estimator	Mean	Variance	Percentile 25%	Percentile 75%
$\hat{\gamma}$	0.0005	$4.26 \times 10^{-4}$	-0.0138	0.0147
$\hat{\beta}_0$	0.0000	$5.86 \times 10^{-5}$	-0.0052	0.0051
$\hat{\beta}_2$	0.1450	$3.11 \times 10^{-7}$	0.1446	0.1454

TABLE 3

*Monte Carlo indices of the variance-covariance matrix  $\hat{\nu}$  based on  $10^4$  samples. The interest is focused on the upper-left  $3 \times 3$  part of  $\hat{\nu}$ , that is the variance-covariance matrix for the structural model parameters  $\gamma$  and  $\beta$*

Variance / Correlation	Mean	Variance
$s_{\hat{\gamma}}^2$	$4.35 \times 10^{-4}$	$1.35 \times 10^{-12}$
$s_{\hat{\beta}_0}^2$	$5.90 \times 10^{-5}$	$4.11 \times 10^{-15}$
$s_{\hat{\beta}_2}^2$	$3.12 \times 10^{-7}$	$8.84 \times 10^{-18}$
$r_{\hat{\gamma}, \hat{\beta}_0}$	0.39	$7.74 \times 10^{-7}$
$r_{\hat{\gamma}, \hat{\beta}_2}$	0.96	$3.75 \times 10^{-9}$
$r_{\hat{\beta}_0, \hat{\beta}_2}$	0.27	$6.10 \times 10^{-7}$

## 5. CASE STUDY

We implemented an automatic procedure to solve the *zero-point* problem, and used it to analyse the data recording for the 2nd International Conference of the European Society for Precision Engineering and Nanotechnology (EUSPEN). We performed some tests with the Primary Hardness Standard Machine of IMGC (Istituto di Metrologia Gustavo Colonnetti) by following the relevant ISO standard specifications (ISO/DIS-14577-1:2000). Forces were generated by dead weights and measured by a load cell having 10 mN resolution and an uncertainty of  $(40+0.001Y_i)$  mN. Displacements were measured with a laser interferometer system having  $0.01 \mu\text{m}$  resolution and  $0.02 \mu\text{m}$  uncertainty. In order to identify the *zero-point*, a number of 60 measurement points around it were selected. By solution of the simplified model (10) by means of the above algorithm, we computed the initial values of the parameters:

$$\begin{aligned}\gamma^{(0)} &= 152.70 \mu\text{m}; \\ \beta_0^{(0)} &= 0.01 \text{ N}; \\ \beta_2^{(0)} &= 0.1542 \text{ N } \mu\text{m}^{-2}.\end{aligned}$$

The ML estimates of the *zero-point* and of the structural parameters have been obtained by means of the Gauss-Newton iterative method:

$$\begin{aligned}\hat{\gamma} &= 152.66 \mu\text{m}; \\ \hat{\beta}_0 &= 0.00 \text{ N}; \\ \hat{\beta}_2 &= 0.1531 \text{ N } \mu\text{m}^{-2}.\end{aligned}$$

It must be noted that  $\hat{\beta}_0$  is an estimate of the systematic *zero error* of the force measuring transducer, negligible in this case, and  $\hat{\beta}_2$  is proportional to the Martens Hardness, an item of the information on the mechanical characteristics of the tested material given by “Instrumented Indentation Test”.

In order to prove the linear approximation, the two curvature indices of (13) have been calculated:  $K_{\max}^T = 4.35 \times 10^{-3}$  and  $K_{\max}^N = 1.35 \times 10^{-4}$ . These results are compatible with the upper bound value suggested Bates and Watts (1988, p. 242) and linear approximation is reasonable. The variance-covariance matrix estimate is:

$$\hat{\mathbf{V}} = \begin{pmatrix} 4.7 \times 10^{-4} & 7.7 \times 10^{-5} & 1.3 \times 10^{-5} \\ 7.7 \times 10^{-5} & 7.8 \times 10^{-5} & 1.5 \times 10^{-6} \\ 1.3 \times 10^{-5} & 1.5 \times 10^{-6} & 4.0 \times 10^{-7} \end{pmatrix}.$$

The estimate of variance of the abscissa of the *zero-point* is  $4.7 \times 10^{-4}$ , so that the confidence interval at 95% results  $(152.62, 152.70) \mu\text{m}$ .

## 6. CONCLUSIONS

Difficulties left unsolved by the traditional separate evaluation of the two parts of FDC are overcome by resorting to a single segmented model describing FDC, and by adopting an appropriate statistical estimation methodology. In the threshold model the abscissa of the *zero-point* is one of the estimated parameters. In the approach we propose, the solution is consequently always guaranteed, since the ill-conditioned matrix, associated with the intersection of the two separate curves, need not be considered. Moreover, with the proposed error-in-variables model the measurement uncertainty of the depth measuring instrument can also be considered. The maximum likelihood method does not give rise to computational problems and it converges quickly to MLEs even if the *zero-point* problem is non-linear. Evaluation of the associated variances-covariance matrix, as required by the relevant standards to express the Hardness Martens uncertainty, is also made possible. Properties of the MLEs fully satisfy, therefore, the requirements of the hardness measurement process. Finally, the method proposed can be implemented in an automatic procedure, where no a priori information is required besides that concerning the uncertainties of the measuring instruments.

*Dipartimento di Matematica  
Politecnico di Torino*

GRAZIA VICARIO

*Dipartimento di Sistemi di Produzione ed Economia dell'Azienda  
Politecnico di Torino*

GIULIO BARBATO  
GABRIELE BRONDINO

## REFERENCES

- D.M. BATES, D.G. WATTS, (1988), *Nonlinear regression analysis and its applications*, John Wiley & Sons, New York.
- O.E. BARNDORFF-NIELSEN, D.R. COX, (1994), *Inference and Asymptotics*, Chapman & Hall, London.
- J.E. DENNIS, R.B. SCHNABEL, (1983), *Numerical Methods for Unconstrained Optimization and Nonlinear Equation*, Prentice-Hall, Englewood Cliffs.
- W.A. FULLER, (1987), *Measurement Error Models*, John Wiley & Sons, New York.
- A.R. GALLANT, W.A. FULLER, (1973), *Fitting segmented polynomial regression models whose join points have to be estimated*, "Journal of the American Statistical Association", 68, 341, pp. 144-147.
- P. GRAU, G. BERG, W. FRANZEL, H. MEINHARD, (1994), *Recording hardness testing. Problems of measurement at small indentation depth*, "Phys. Stat. Sol.", 146, pp. 537-548.
- M.G. KENDALL, A. STUART, (1973), *The advanced Theory of Statistics*, Vol. 2, C. Griffin & Company Limited, London.
- J. MENCIK, M.V. SWAIN, (1994), *Micro-Indentation Test with Pointed Indenters*, "Materials Forum", 18, pp. 227-288.
- ISO/DIS-14577-1, (2000), *Metallic materials - Instrumented indentation test for hardness and materials parameters - Part 1: Test method*.
- G.A.F. SEBER, C.J. WILD, (1989), *Nonlinear Regression*, John Wiley & Sons, New York.

- C. ULLNER, (2000), *Requirement of a robust method for the precise determination of the contact point in the depth sensing hardness test*, "Measurement", 27, pp. 43-51.
- C. ULLNER, G.D. QUINN, (1997), *Round Robin on Recording Hardness*, Technical report, VAMAS Technical Working Area 3.

#### RIASSUNTO

##### *Un'applicazione della teoria asintotica ad un modello soglia per la stima della durezza Martens*

Nel campo della metrologia meccanica un ruolo significativo è ricoperto dalle misure di durezza da sempre utilizzate per testare le caratteristiche dei materiali nei processi produttivi industriali. Un nuovo metodo di misura, detto durezza Martens, trae le informazioni dai dati dell'evoluzione di forza e spostamento durante l'intero ciclo di prova. La Curva Forza/Profondità che descrive la prova è formata da due parti unite nel cosiddetto *zero-point*. Viene proposto un modello di regressione a tratti basato sull'introduzione di un parametro soglia al fine di stimare l'ascissa dello *zero-point*. Il problema è particolarmente complesso, in quanto il legame tra le variabili forza e profondità è di tipo strutturale e la numerosità dei parametri di disturbo insegue quella delle misure effettuate. Una stima dei parametri incogniti del modello viene fornita mediante la teoria asintotica basata sulla verosimiglianza. Simulazioni effettuate con metodi Monte Carlo permettono di analizzare le proprietà degli stimatori al variare delle ipotesi introdotte sugli errori di misura e contribuiscono alla definizione delle condizioni di applicabilità del metodo proposto.

#### SUMMARY

##### *An application of the asymptotic theory to a threshold model for the estimate of Martens Hardness*

Hardness measurements have a significant role in mechanical metrology, as they are frequently used to characterise materials properties relevant to industrial processes. A recently introduced method, called Martens Hardness, is based on force and indentation records obtained during a test cycle; the Force/Depth Curve, which describes the indentation pattern, is typically formed by two parts having a *zero-point* in common. A segmented regression model is proposed in this paper, based on the introduction of a threshold parameter in order to estimate the unknown *zero-point*. The problem is not trivial, since the relationship between observed force and indentation depth is structural and, moreover, the number of nuisance parameters grows with the number of measured data. The asymptotic likelihood theory leads to an estimate of the unknown parameters of the model. Monte Carlo simulations are resorted to in order to analyse the properties of estimators under different hypotheses about measurement errors, and to establish the applicability conditions of the method proposed.