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Statistical analysis of the Vickers hardness

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Abstract

The statistical dispersion in Vickers hardness measurements is discussed in order to check the reliability of hardness measurements. Several indentations were made under the same nominal conditions on a hardness standard. This was repeated for different loads. The distribution of diagonal lengths of indentation prints is found to be of Gaussian type. From the distribution function of the indentation lengths, the probability density function of the hardness is derived for the general case of several indentation measurements. The mean value and variance of hardness are compared with results based on Gaussian statistics. From this comparison, it follows that statistical analysis relying upon Gaussian distributions can be carried out within a given confidence level which depends on the number of indentations. A method is presented to calculate the number of indentations needed to achieve a certain level of accuracy.

Keywords: Vickers hardness; Gaussian statistics; Probability density; Measurement accuracy

1. Introduction

The Vickers indentation test is a common method used to characterize the hardness of materials. These experiments are simple to perform, need a small quantity of material, are generally non-destructive and can be repeated many times. As all mechanical tests, they are subject to large statistical fluctuations, which are important to be evaluated. The probability distribution function (PDF) of the Vickers hardness was reported in the literature to correspond to a Gaussian (or Normal law) [1], a log normal [2] or a Weibull distribution [3,4]. The objective of this work is to establish the statistical distribution function of a set of hardness values composed of n indentations and to evaluate the accuracy of the result. In this manner, one should be able to design a measurement for any desired confidence level. It also becomes possible to distinguish between the precision of the result and a hypothetical heterogeneity of the sample. The indentation experiment is described in Section 2, the statistical analysis of Vickers diagonal lengths is presented in Section 3. The hardness PDF is

calculated by statistical considerations. Its mean, variance and confidence intervals are derived. The errors made by approximating the hardness probability by a normal law are calculated and lead to the determination of an interval of confidence. The consequences of these results are discussed in Section 4 and an experimental method is proposed which permits the hardness to be determined within a given accuracy.

2. Statistical analysis of indentation measurements

2.1. Experimental procedure

Experimental indentation tests were performed with a Vickers microhardness tester Leitz Miniload. The loads were 0.0981, 0.491, 0.981, 1.96, 4.91, and 9.81 N. They were applied with a dwell time of 10 s and maintained in contact with the sample surface for another 10 s. The tested material was a hardness standard, whose surface was carefully electropolished to avoid compressive surface stresses. Thirty indentations were performed at each load and both diagonal lengths of each indentation were optically measured in the hardness apparatus with a magnification of 400, or using a

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Lasertec confocal microscope to obtain a better precision. The results are reported in Table 1. The hardness is calculated with the equation

$$H_v = \frac{kP}{d^2}, \quad (1)$$

where k a constant depending on the indenter geometry and on the units chosen (here $k = 1.89 \times 10^5$), P is the applied force (in N) and d the diagonal length (in μm) of the indentation. As commonly reported in the literature, the units of the Vickers hardness are omitted and correspond to kgf mm^{-2} ($1 \text{ kgf mm}^{-2} = 9.81 \text{ MPa}$).

2.2. Results

2.2.1. Test of Normal Law for the indentation diagonal

The statistical frequency of the indentation length was investigated in order to verify the validity of Bückle's assumption [5] of a Gaussian distribution. The histograms as well as the fitted Gaussian PDF are shown in Fig. 1 (least-squares fitting). Indentation prints measured by optical and by confocal microscopy are presented. The data were investigated by means of five tests: Kolmogorov–Smirnov, Shapiro, Pearson χ^2 , Lilliefors and Henry [6]. The results are presented in Table 2. For all the tests investigated, the normal law assumption is followed at the 95% confidence level, except for the indentation set done with a load $P = 0.49 \text{ N}$ and recorded by confocal microscopy. Therefore, the Gaussian distribution is considered to be a good representation of the PDF of the indentation diagonal length.

Table 1
Average diagonal length and standard deviation from a set of indentation prints with different applied loads recorded by the optical device of the hardness tester (a) and with the confocal microscope (b)

Load (N)	Number of indentations	Average length μ_d (μm)	σ_d (μm)
<i>(a)</i>			
0.491	30	10.16	0.49
0.981	30	14.56	0.29
1.96	44	21.03	0.62
4.91	30	33.45	0.33
9.81	30	47.72	0.29
<i>(b)</i>			
0.0981	30	4.21	0.27
0.491	30	9.90	0.28
0.981	30	14.43	0.33
1.96	44	20.75	0.25
4.91	30	33.42	0.22
9.81	30	48.12	0.25

This first result is interpreted by considering that the variability of the diagonal length is the additive combination of random variables such as: uncertainty on the applied load, errors on the indentation print measurement, vibrations, lack of perpendicular alignment of the indenter relative to the surface, surface stresses, precipitates or grain boundaries, orientation of anisotropic material, experimenter's errors, etc. [5]. Owing to the Central Limit Theorem, as the number of random variables increases, the shape of the indentation length PDF approaches normal shape, even if the distributions of the variables listed before are not following a normal distribution.

2.2.2. Tests of homogeneity of variances

Variances of indentation diagonal lengths are homogenous if the magnitudes of fluctuations are independent of the average value. This is equivalent to stating that for a given hardness the variance of diagonal lengths is not affected by a change of the applied load. To test this assumption, the test of Bartlett, Cochran and Levene [7] was used. Results are shown in Table 3. The variances are found to be homogeneous when recorded by confocal microscopy. Measurements obtained by the optical device of the hardness tester are not homogeneous when considered as a whole. Indeed, the subset of measurements taken with applied loads of 4.90 and 9.81 N are consistent with the variance homogeneity and lead to variances comparable to those measured by confocal microscopy. This discrepancy concerns measurements of small applied loads, which are more likely to be affected by experimental errors. Thus, the standard deviation of the indentation print is considered to be constant and equal to $0.27 \mu\text{m}$. This might imply that the same deformation mechanisms are involved in this range of loads.

3. Statistical analysis of the hardness

3.1. Probability density function of the hardness

Following the results of the preceding section, the Vickers hardness PDF can be calculated using the knowledge of the diagonal length PDF $g(d)$ which is a Gaussian distribution parameterized by its mean μ_d and its variance σ_d^2 .

$$g(d) = \frac{1}{\sqrt{2\pi}\sigma_d} \exp\left[-\frac{1}{2}\left(\frac{d-\mu_d}{\sigma_d}\right)^2\right] \quad (2)$$

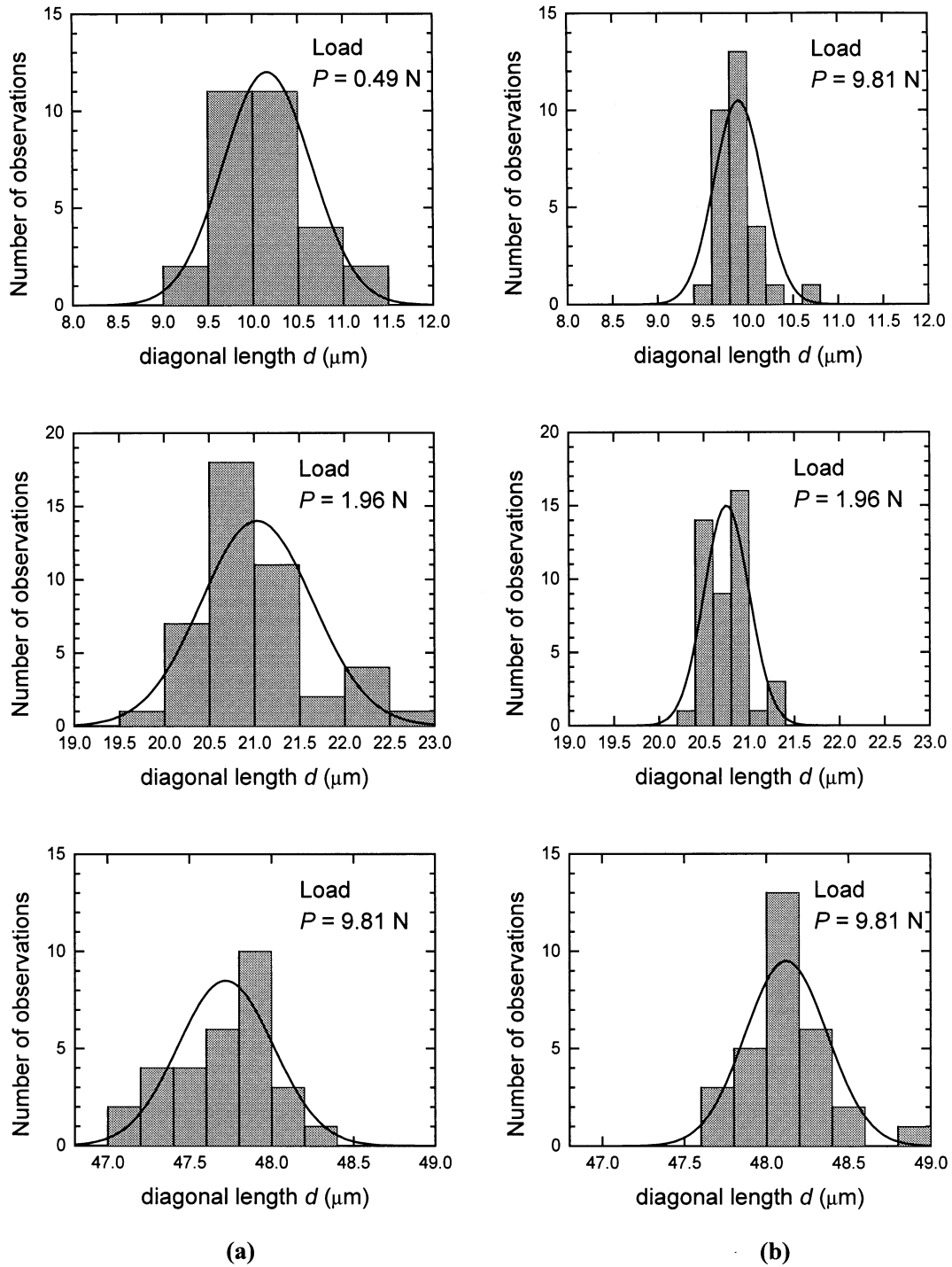


Fig. 1. Histograms of the diagonal lengths recorded by the optical device (a) and with the confocal microscope (b).

The Vickers hardness PDF $f(H_V)$ is found by asserting the conservation of the probability density when expressed by the diagonal length or by the corresponding hardness

$$f(H_V) dH_V = g(d) dd. \quad (3)$$

Expressing the right-hand side explicitly in term of the hardness leads to

$$f(H_V) = \frac{\sqrt{kP}}{2\sqrt{2\pi}\sigma_d} H_V^{-3/2} \exp\left[-\frac{1}{2}\left(\frac{\sqrt{\frac{kP}{H_V}} - \mu_d}{\sigma_d}\right)^2\right]. \quad (4)$$

Table 2

Result of non-parametric tests [6] at the 95% confidence level recorded with the optical device of the hardness tester (a) and by confocal microscopy (b)

Load (N)	Number of indentations	Kolmogorovs. Smirnov	Shapiro-Wilk	Lilliefors	Pearson (χ^2)
<i>(a)</i>					
0.491	30	Accepted	Accepted	Accepted	Accepted
0.981	30	Accepted	Accepted	Accepted	Accepted
1.96	44	Accepted	Accepted	Accepted	Accepted
4.91	30	Accepted	Accepted	Accepted	Accepted
9.81	30	Accepted	Accepted	Accepted	Accepted
<i>(b)</i>					
0.0981	30	Accepted	Accepted	Accepted	Accepted
0.491	30	Accepted	Accepted	Accepted	Accepted
0.981	30	Accepted	Accepted	Accepted	Accepted
1.96	44	Accepted	Accepted	Accepted	Accepted
4.91	30	Accepted	Accepted	Accepted	Accepted
9.81	30	Accepted	Accepted	Accepted	Accepted

If a set of n indentations is considered, according to Gaussian statistics, the average diagonal length remains unchanged and the variance decreases by a factor n .

$$\sigma_{d,n}^2 = \frac{\sigma_d^2}{n} \quad (5)$$

Thus, the hardness PDF can be written as

$$f_n(H_V) = \frac{\sqrt{kP}}{2\sqrt{2\pi} \frac{\sigma_d}{\sqrt{n}}} H_V^{-3/2} \exp \left[-\frac{1}{2} \left(\frac{\sqrt{\frac{kP}{H_V}} - \mu_d}{\frac{\sigma_d}{\sqrt{n}}} \right)^2 \right]. \quad (6)$$

Hardness PDFs are not Gaussian distributions. This is seen in Fig. 2, where they are represented for several mean indentation lengths with an applied load of 0.049 N and a standard deviation $\sigma_d = 0.27 \mu\text{m}$. They reveal a finite skewness, which is less pronounced when the average diagonal length increases. Calculations of confidence intervals and other statistical estimates are usually based on the assumption of an underlying normal distribution (especially, the symmetry of the PDF is of great importance). Here, this hypothesis does

Table 3

Study of the homogeneity of variances with the tests from Bartlett, Cochran and Levene [7]^a

Test	Confocal Microscopy	Optical Microscopy
Bartlett	Accepted 0.3	Rejected 0.00001
Cochran	Accepted 0.11	Rejected 0.00002
Levene	Accepted 0.52	Rejected 0.008

^a For result > 0.05 , the assumption of homogeneity of the variances is accepted at the 95% confidence level.

not hold. However, as will be shown in the next section, characteristic parameters of the distribution tend to those of the Gaussian PDF when the standard deviation becomes smaller than the mean diagonal length.

3.2. Mode, mean, variance and interval of confidence

Although the hardness PDF contains the full statistical information, its reduction to a few characteristic parameters is sufficient for an error analysis (see Appendices A, B, C and D for a complete derivation).

The mode of the hardness PDF $H_{V,\text{mode}}$ is the most probable hardness. It corresponds to the maximum of the hardness PDF.

$$H_{V,\text{mode}} = \frac{kP}{\mu_d^2} 4 \left[1 + \sqrt{1 + 12 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^2} \right]^{-2}. \quad (7)$$

Although this result is exact, it is useful to rewrite it as a Taylor expansion in powers of $\sigma_{d,n}/\mu_d$,

$$H_{V,\text{mode}} = \frac{kP}{\mu_d^2} \left[1 - 6 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^2 + 45 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^4 + \dots \right] \quad (8)$$

The mean hardness μ_{HV} is the first moment of the hardness PDF. It can be approximated by a Taylor expansion,

$$\mu_{HV} = \frac{kP}{\mu_d^2} \left[1 + 3 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^2 + 15 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^4 + \dots \right] \quad (9)$$

An analogous derivation gives the variance

$$\sigma_{HV}^2 = \left(\frac{kP}{\mu_d^2} \right)^2 \left[4 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^2 + 57 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^4 + \dots \right] \quad (10)$$

As a consequence of the homogeneity of variances, for two measurements on the same sample only differing by the applied load, the measurement with the bigger load will be more precise because the average diagonal length will increase while the variance remains constant.

The confidence interval corresponding to a given confidence level is defined by the range $[H_{V,\text{min}}, H_{V,\text{max}}]$

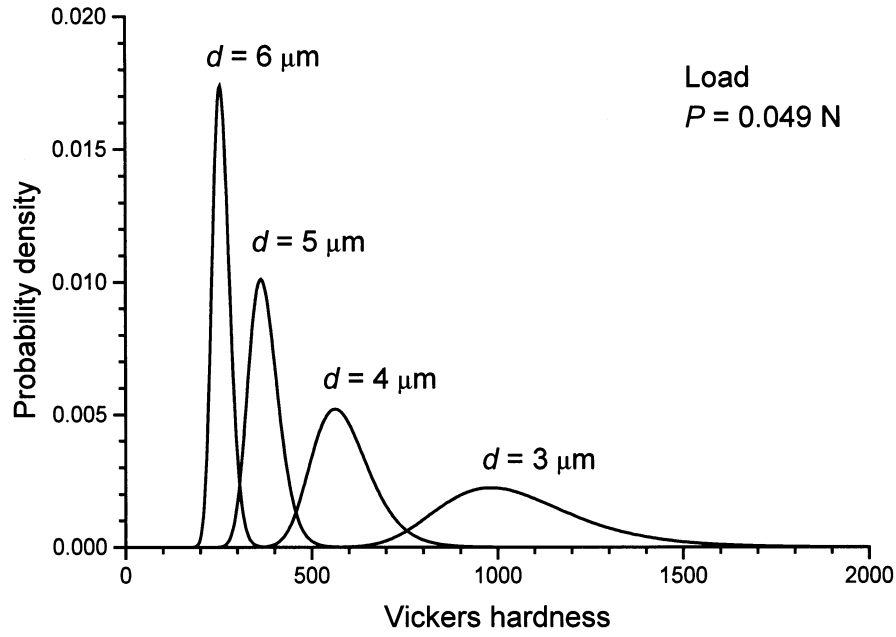


Fig. 2. Hardness PDF for indentation prints of 3, 4, 5, and 6 μm as given by Eq. (6), for a load $P = 0.049\text{ N}$ and $\sigma_d = 0.27\text{ }\mu\text{m}$.

which contains the fraction α of the statistical events. Due to the conservation of the probability density, this range is indeed the image of the interval $[d_{\min}, d_{\max}]$ by means of the function of Eq. (1) that corresponds to the same confidence level.

$$d_{\max}^{\min} = \mu_d \mp u_\alpha \sigma_{d,n}, \quad (11)$$

where u_α is the reduced value defining the α -confidence interval in Gaussian statistics ($u_\alpha = 1.96, 2.33,$ and 2.56 for $\alpha = 95, 98,$ and 99% respectively). The boundaries of the hardness interval are

$$H_{V_{\max}}^{\min} = \frac{kP}{\mu_d^2} \left(1 \pm u_\alpha \frac{\sigma_{d,n}}{\mu_d} \right)^{-2}. \quad (12)$$

This interval is centered on the hardness

$$H_{V_{\text{center}}} = \frac{kP}{\mu_d^2} \left[1 + 3 \left(u_\alpha \frac{\sigma_{d,n}}{\mu_d} \right)^2 + 5 \left(u_\alpha \frac{\sigma_{d,n}}{\mu_d} \right)^4 + \dots \right] \quad (13)$$

its width is

$$\Delta H_V = \frac{kP}{\mu_d^2} \left[4 \left(u_\alpha \frac{\sigma_{d,n}}{\mu_d} \right) + 8 \left(u_\alpha \frac{\sigma_{d,n}}{\mu_d} \right)^3 + 12 \left(u_\alpha \frac{\sigma_{d,n}}{\mu_d} \right)^5 + \dots \right]. \quad (14)$$

3.3. Gaussian approximation

The mean hardness, its variance and an α -confidence interval could be calculated from the hardness PDF (Eq. (6)) as function of the relative spread in diagonal

length $\frac{\sigma_d}{\sqrt{n}\mu_d}$. In practice, this factor is sufficiently small to be able to cut every Taylor expansion after the first term of non-vanishing power. Furthermore, as the number of indentations increases, this factor tends to zero. This implies that the mean hardness corresponds to the mean diagonal length and that the hardness variance vanishes as the variance in diagonal length does.

On the other hand, it is convenient to approximate the hardness PDF by a Gaussian distribution, so that the hardness random variable becomes easier to handle. The best fitted Gaussian PDF is exactly the one, which is parameterized by the mean hardness and the hardness variance found earlier. Therefore, if the number of indentations increases, the Gaussian approximation becomes more valid.

In order to discuss the error made by accepting the approximation, the interval of confidence is calculated for a confidence level α and both intervals are compared with each other. The boundaries are

$$H_V^{\text{Gauss}\max} = \mu_{HV} \mp u_\alpha \sigma_{HV}. \quad (15)$$

The interval is centered on the mean hardness μ_{HV} and has a width of

$$\Delta H_V^{\text{Gauss}} = \frac{kP}{\mu_d^2} u_\alpha \left[4 \left(\frac{\sigma_{d,n}}{\mu_d} \right) + \frac{57}{2} \left(\frac{\sigma_{d,n}}{\mu_d} \right)^3 + \dots \right]. \quad (16)$$

It is noticeable that first-order terms of the interval widths of the hardness PDF and its approximation are equal. For higher-order terms, this is no longer the case. On the other hand, intervals of confidence are

centered on different values. Comparison of Eq. (13) with Eq. (9) shows the existing bias, which can reach up to several percent of the hardness. However, the shift of the center is always smaller than half the width of the interval of confidence (for sufficiently large values of confidence level).

Thus, the interval of confidence of the Gaussian PDF was obtained. The error made by the approximation occurs mainly in the determination of the interval center rather than in its width.

4. Number of indentation prints

In practical situations, the accuracy of a hardness measurement consisting of n indentations is of great importance. It is also very convenient to be able to predict the number of indentations required for a certain precision. The method presented here extracts these values from the experiment itself. Therefore, a given set of diagonal lengths $\{d_i\}_{i=1}^n$ has first to be recorded (say $n=5$). From this, the average diagonal length and variance can be calculated.

$$\mu_d = \frac{1}{n} \sum_{i=1}^n d_i \quad (17)$$

$$\sigma_d^2 = \frac{1}{n-1} \sum_{i=1}^n (d_i - \mu_d)^2 \quad (18)$$

Strictly speaking, these values are only estimates of the average and variance. Thus, some precautions have to be taken. It has to be checked that these values are good estimates, which means, they should be approximately constant as n increases. Especially, the average diagonal length calculated from a measurement subset should remain within the interval defined by the standard deviation σ_d .

Using Eqs. (5), (9) and (16), the interval of confidence can be calculated for a given confidence level within the Gaussian approximation. The number \tilde{n} of indentations required to attain the accuracy level α is found by inverting these equations.

$$\tilde{n} = \left(\frac{2u_\alpha}{(1-\alpha)} \frac{\sigma_d}{\mu_d} \right)^2 \quad (19)$$

Table 4
Number of indentations required to reach a 95, 98, and 99% confidence level for some value of the ratio σ_d/μ_d as calculated by Eq. (19).

S.D. $\frac{\sigma_d}{\mu_d}$	$\tilde{n}_{95\%}$	$\tilde{n}_{98\%}$	$\tilde{n}_{99\%}$
0.005	1	2	7
0.010	1	6	27
0.020	3	22	105
0.050	16	136	656

Calculated numbers of indentations required to reach a confidence level of 95, 98, and 99% are shown in Table 4. The precision α enters in two different ways into the calculation. First, in the sense of a signal-to-noise ratio, it defines the maximum acceptable spread of the hardness. This has the strongest influence on \tilde{n} through the $(1-\alpha)$ term in the denominator, which is much smaller than one. Secondly, it defines the width of the interval through the value of u_α . This term shows a weaker α -dependency.

In practice, after having recorded the estimated number of indentations \tilde{n} , the procedure should be re-iterated. Using the larger set of diagonal lengths, the average and variance have to be calculated once again, because they provide better estimates than the former one. Then, the accuracy of the result can be checked and the procedure ends, when the final precision has been reached.

5. Conclusion

Some authors have shown that the hardness should obey a Gaussian distribution [1], but, in conflict with the hypothesis of a normal law, they observed a variation of the skewness with increasing load. Weibull distributions were also reported in the literature [3,4]. They have the advantage of being able to represent the asymmetry of the distributions, but the determination of the Weibull parameters is difficult. Furthermore, Weibull statistics is generally used to account for fatigue phenomena and has no clear physical meaning when applied to hardness tests. The log normal distribution [2] is justified by the Kolmogorov hypothesis [8], which states that fluctuations are due to multiplicative combinations of elementary sources of noise. However, there is no evidence for the hardness to behave as such. In this paper, it has been shown that indentation lengths rather than the hardness follow a Gaussian distribution. Thus, the hardness distribution was derived. It is an asymmetric distribution, where Gaussian statistics is only valid as an approximation. The required number of indentations to achieve a given accuracy is directly proportional to the square of the spread in diagonal length and inversely proportional to the square of the desired signal-to-noise ratio. A quantitative estimate of the measurement spread can be achieved and, as an application, heterogeneous specimens can be studied with this method. The number of indentations should be sufficiently large to ensure that a Gaussian distribution could approximate the hardness. The indentation size effect, which is the observed variation of the hardness when the applied load is changed, can also be studied using these results.

Appendix A

A.1. Mode of the hardness

The zero of the first derivative of the hardness PDF Eq. (6) leads to the mode of the hardness.

$$H_{V,\text{mode}} = \frac{kP}{\mu_d^2} 4 \left[1 + \sqrt{1 + 12 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^2} \right]^{-2} \quad (\text{A1.1})$$

Appendix B

B.1. Mean of the hardness

The average hardness is the first moment of the hardness PDF as given by Eq. (6).

$$\mu_{\text{HV}} = \int_0^{\infty} H_V f_n(H_V) dH_V \quad (\text{A2.1})$$

This was integrated after substitution of the integration variable.

$$x = \frac{\sqrt{\frac{kP}{H_V}} - \mu_d}{\sigma_{d,n}} \quad (\text{A2.2})$$

$$\mu_{\text{HV}} = \frac{1}{\sqrt{2\pi}} \int_{-\infty}^{+\infty} \frac{kP}{\mu_d^2} \frac{1}{\left(1 + \frac{\sigma_{d,n}}{\mu_d} x\right)^2} e^{-\frac{1}{2}x^2} dx \quad (\text{A2.3})$$

The Taylor expansion of the denominator gives the following expression.

$$\mu_{\text{HV}} = \frac{kP}{\mu_d^2} \frac{1}{\sqrt{2\pi}} \int_{-\infty}^{+\infty} \left[1 - 2 \left(\frac{\sigma_{d,n}}{\mu_d} \right) x + 3 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^2 x^2 - 4 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^3 x^3 + 5 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^4 x^4 + \dots \right] e^{-\frac{1}{2}x^2} dx \quad (\text{A2.4})$$

The odd terms give no contribution to the sum because of the even symmetry of the integration range. The even terms are recursively integrated.

$$\frac{1}{\sqrt{2\pi}} \int_{-\infty}^{+\infty} x^{2n} e^{-\frac{1}{2}x^2} dx = (2n-1) \cdot (2n-3) \cdot \dots \cdot 5 \cdot 3 \cdot 1 \quad (\text{A2.5})$$

From this, the mean hardness follows.

$$\mu_{\text{HV}} = \frac{kP}{\mu_d^2} \left[1 + 3 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^2 + 15 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^4 + 105 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^6 + \dots \right] \quad (\text{A2.6})$$

Appendix C

C.1. Hardness variance

The variance of the hardness is the second moment of the hardness PDF.

$$\sigma_{\text{HV}}^2 = \int_0^{\infty} (H_V - \mu_{\text{HV}})^2 f_n(H_V) dH_V \quad (\text{A3.1})$$

The same integration way was followed. First, the integration variable was substituted.

$$\mu_{\text{HV}}^2 = \left(\frac{kP}{\mu_d^2} \right)^2 \frac{1}{\sqrt{2\pi}} \int_{-\infty}^{+\infty} \left\{ \left(1 + \frac{\sigma_{d,n}}{\mu_d} x \right)^{-2} - \left[1 + 3 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^2 + 15 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^4 + 105 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^6 \right]^2 \right\} e^{-\frac{1}{2}x^2} dx \quad (\text{A3.2})$$

Then, the expression was developed and the even power terms were retained.

$$\sigma_{\text{HV}}^2 = \left(\frac{kP}{\mu_d^2} \right)^2 \frac{1}{\sqrt{2\pi}} \int_{-\infty}^{+\infty} \left[\left(\frac{\sigma_{d,n}}{\mu_d} \right)^2 x^2 - 18 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^4 x^2 - 90 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^6 x^2 + 25 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^4 x^4 - 30 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^6 x^4 + 24 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^6 x^6 + \dots \right] e^{-\frac{1}{2}x^2} dx \quad (\text{A3.3})$$

From which the hardness variance follows.

$$\sigma_{\text{HV}}^2 = \left(\frac{kP}{\mu_d^2} \right)^2 \left[4 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^2 + 57 \left(\frac{\sigma_{d,n}}{\mu_d} \right)^4 + \dots \right] \quad (\text{A3.4})$$

Appendix D

D.1. Number of indentations

The minimum number of indentation is found by asserting that the spread of the hardness should be smaller than $(1 - \alpha)$, which is complementary to the confidence level.

$$\frac{2u_\alpha \sigma_{d,n}}{\mu_d} \leq (1 - \alpha) \quad (\text{A4.1})$$

$$n \geq \left(\frac{2u_\alpha \sigma_d}{(1 - \alpha) \mu_d} \right)^2 \quad (\text{A4.2})$$

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