XVII IMEKO World Congress
Metrology in the 3rd Millennium
June 22−27, 2003, Dubrovnik, Croatia

PROPOSAL FOR A PRACTICAL PROCEDURE FOR THE EXPRESSION OF UNCERTAINTY IN HARDNESS MEASUREMENT

*Giulio Barbato**, *Alessandro Germak°, Samuel Low^*

*Dipartimento di Sistemi di Produzione ed Economia dell'Azienda - Politecnico di Torino, Torino, Italy
° CNR, Istituto di metrologia "G. Colonnetti" – Torino- Italy
^ National Institute of Standards and Technology - NIST, Gaithersburg, USA

Abstract − Hardness measurement is widely used in industrial applications for quality control and acceptance testing of products because it is fast, inexpensive and relatively non-destructive. Uncertainty evaluation is complicated because calibration procedures require the use of direct and indirect verification tests, but the effects on the measurand itself due to test parameters variations are difficult to predict, such as the force-time pattern, inelastic performances of Rockwell indenters and the numerical aperture for Brinell and Vickers indentations measurement. This paper starts by accepting as a matter of fact that standard specifications have demonstrated acceptable performance, indicating that their application is correct for most of the materials generally used. Accepting this premise, the next step was to try to translate the practice, confirmed by many years of experience, into the new language of uncertainty, strongly required by quality documents.

Keywords: Hardness, Uncertainty, Calibration.

1. INTRODUCTION

Hardness measurements have to guarantee to produce results within a reasonable difference between the different parties (producer, customer and, if present, an independent third party). Moreover, this condition should be verified not only for materials of common use such as steel, but also for different metallic materials frequently used today. The problem to face, therefore, can be considered as split in two:

- guarantee the measurement compatibility when common metallic materials, similar to that of the reference blocks, are used;
- guarantee the measurement compatibility when metallic materials different from that of the reference blocks are used.

The usual practice of indirect verification of hardness testers is sufficient for the first circumstance: when two different hardness testers give compatible results on hardness reference blocks, it is likely that the results obtained on test pieces of similar materials should be compatible. This is confirmed by common practice. However, even if the indirect verification has the merit to take into account the effects of the whole measurement procedure, having compatible results on hardness reference blocks is not sufficient to assure that the two testers will give compatible results on materials different from that of the test blocks used. In fact, it certainly could be possible to have a tester that uses lower forces and a different indentation measurement scale which are matched in such a way to obtain results well in line with the values of specific reference blocks. This is a common occurrence, for instance, with hand operated testers. It is certainly evident that measurement compatibility when testing the material of reference blocks, will not automatically apply to any different material. This gives reason to also require that a direct verification be performed, aimed to assure that the measurement procedure of the hardness tester represents, within given tolerances, the principal features of the hardness scale used.

The solution, therefore, is not so simple, but it should not be overcomplicated, as industrial practice requires an uncertainty evaluation that can be applied by any normal workshop that uses hardness measurements for quality assessments. To address the two sides of the problem from a practical point of view, the common practice used up to now of using both indirect verification (performed by reference hardness blocks) and direct verification (performed by direct measurement of force, length and time) has been shown to be acceptable both for the practical application and the general guarantee of acceptable compatibility of results between the three parties involved. Therefore, even if we know that this is an approximate solution of the problem, we shall accept the traditional method that requires a direct verification, to be sure that the hardness tester examined operates in line with the standard procedure, and an indirect verification, to take into account little, non-measurable causes of variations, as the load-time pattern, indenter micro-geometry and hysteresis, etc. Being that the direct measurement is only an evaluation of conformity of the procedure, uncertainty could be assessed on the basis of indirect measurement only, at least when a well defined starting point is available. The problem of the starting point is the international or national hardness standard references, for which national and international metrology organizations are working.

A reasonable path to follow is to try to translate the practice confirmed by many years of experience into the new language of uncertainty. The traditional practice, in fact, is a completely conventional one. It asks only to check that a series of given parameters, direct, as forces, indentation dimension measurement, indenter geometry etc., or indirect, as the values measured on reference blocks, are within given tolerances. This is easy to accomplish, but it does not provide a value of the uncertainty produced by that amount of tolerances, and therefore does not inform the end user of the important contribution of uncertainty produced by the measurement system. The uncertainty contribution of the measurement system must be taken into account, together with the uncertainty contributions given by the specific measurand, to evaluate the uncertainty of his measurement results. This is also in line with the varying needs of end users, which we have divided into three different levels:

- the simple need for a typical value of uncertainty corresponding to the general tolerances given by the standard specifications;
- the need to evaluate the uncertainty corresponding to the measurement obtained during the last indirect verification, when a correction for the measurement bias is not applied;
- the need to evaluate the uncertainty corresponding to the measurement obtained during the last indirect verification, when a correction for the measurement bias is applied.

In our opinion, these three levels cover the normal practice for:

- users that are satisfied to have a declaration of conformity, because they can accept higher levels of uncertainty;
- users that need smaller uncertainties, therefore chose very good hardness reference blocks, require a calibration certificate complete with measurement results and the expression of calibration uncertainty, but will not charge their measurement procedure with the correction of the bias;
- users that need the best result and will apply the bias correction to their measurements.

The use of the Guide to the Expression of Uncertainty in Measurement (GUM) [1] allows one to obtain a practical solution, but it is clear that some effects connected with the measurement of less common metallic materials have not been investigated sufficiently to ignore the possibility that some specific effects may be larger than the evaluated uncertainty. Nevertheless, one should remember that the prescriptions given by standard documents used for many years have demonstrated acceptable performance, indicating that their application is correct for most of the materials generally used.

In conclusion, we consider that, when the tolerances on direct measurement are correctly applied, the contributions of uncertainty due to the effects of the material tested are negligible. Nevertheless, for materials that exhibit large effects due to indentation velocity (e.g. for materials with high sensitivity to strain-rate) and dwell time (e.g. for materials with high creep), a caution should be given to follow a specific prescribed testing procedure. The uncertainty contributions remaining are, therefore, that due to the metrological chain of indirect verifications, that is due to the combination of:

- the uncertainty of metrological primary hardness reference blocks calibrated by National Metrology Institutes;
- the uncertainty of the calibration of hardness calibration machines of producers of commercial hardness reference blocks;
- the uncertainty of the calibration of commercial hardness reference blocks;
- the uncertainty of the calibration of hardness testers; and
- the uncertainty of a hardness measurement of the end user.

2. THE STARTING POINT FOR UNCERTAINTY EVALUATION

The starting point is the uncertainty of metrological primary hardness reference blocks calibrated by National Metrology Institutes. As stated previously, the starting point is very important, because the effects of tolerances of the hardness scale definitions frequently produce a very important uncertainty contribution [2]. Just as the common practice showed that direct measurements are not sufficient to assure compatibility, the scale definition based only on tolerances is also inadequate. Past experience has shown large differences even between National Metrology Institutes [3], which have been reduced by the extensive work of international comparisons [4, 5, 6, 7, 8] and refinement of the measurement procedure. One can say, therefore, that the uncertainty of the starting point depends on the framework considered:

- a completely general framework of the group of metrological laboratories that have established hardness scales in agreement with the parameter tolerances defined by international standard documents;
- a specific framework of groups of metrological laboratories that work to establish harmonization though comparisons and refinement of measurement procedures.

An example of the effectiveness of these two types of frameworks can be seen very well by comparing the Rockwell C hardness uncertainty band of about ±0,9 HRC, obtained in the Organisation Internationale de Métrologie Légale (OIML) comparison [3], with that of less than ±0,3 HRC obtained within the framework of the European HRC scale [7, 8]. Even better results are expected from the ongoing harmonization work within the framework of the Comité International des Poids et Mesures - Consultative Committee for Mass and Related Quantities (CIPM-CCM) [9]. It is easy to understand that the uncertainty band that covers the compatibility results among all the metrology institutes of the world is larger than the uncertainty band that covers the results of a well-acquainted group of Metrology Institutes. Because of these differences, it is very important that the uncertainty declared for the starting point be well defined, that is, whether it represents the coverage band that contains all possible parameter ranges within the standard definition of the scale, or the coverage band referring to a well defined agreement between a group of metrological laboratories, or the coverage band of a single metrological laboratory that represents only its stability and reproducibility.

In any case, the information obtained from the calibration certificate of the hardness reference block is the hardness value of the block, H_1, an expanded uncertainty, U_1, together with its confidence level and the number of degrees of freedom (if the confidence level and the degrees of freedom are not given, usually it means that a coverage factor k=2 has been used). Using this information it is easy to evaluate the relevant standard uncertainty u_1. One should pay attention to the fact that u_1 is the standard uncertainty of the hardness value of the hardness reference block, and therefore is frequently the uncertainty of the average of the measurements made on the block itself.

3. THE METROLOGICAL CHAIN

The typical metrological chain can be described as follows:

- National Metrology Institutes produce primary hardness reference blocks (that is, characterise primary hardness reference blocks by determining their hardness value H_{1m1}[1]);
- industrial producers calibrate their hardness calibration machines by means of primary hardness reference blocks (that is, characterise each hardness calibration machine measuring the value of hardness H_{1m2} and determining the bias of the hardness calibration machine $B_{1,2} = H_{1m2} - H_{1m1}$)
- industrial producers calibrate their production of hardness reference blocks by means of their hardness calibration machines (that is, characterise hardness reference blocks determining their hardness value H_{2m2});
- calibration laboratories or end users calibrate hardness testers by means of hardness reference blocks (that is, characterise each hardness tester measuring the value of hardness H_{2m3} and determining the bias of the hardness testers $B_{2,3} = H_{2m3} - H_{2m2}$);
- the end user uses hardness testers to make measurements H_{3m3} on test pieces.

As one can observe, after the starting point H_{1m1}, which is, as stated before, the responsibility of National Metrology Laboratories to give the relevant value of the standard uncertainty u_{1m1}, the subsequent steps can be grouped into two categories: one representing the calibration of a hardness measurement system (hardness calibration machine or hardness tester), and the second, the use of the hardness measurement system to perform measurements on a piece of material (hardness reference block or test piece). We can focus our attention on the first double step; the second being similar in principle.

3.1. Calibration of an Hardness Calibration Machine

As a first approximation, let us suppose that the measurements of the whole metrological chain are taken in a sufficiently short time (that is, no variation in time) and that all the measurements are made on sufficiently new blocks (that is, no effect of previous indentations).

In this case, we have two main contributions to the uncertainty:

- the first contribution is the standard uncertainty of the primary hardness reference block u_{1m1}, given by the certification of the block;
- the second contribution u_{1m2} is that of the determination of H_{1m2} using the hardness calibration machine. This determination is usually the average of five measurements, and suffers primarily from the non-uniformity of the primary hardness reference block and the non-repeatability of the hardness calibration machine.

These two effects are so connected that it is very difficult and nearly useless to separate them. The joint effect can be evaluated by the standard deviation s_{1m2} of the five measurement results. Attention shall be given to the fact that H_{1m2} is the mean value of five measurements; therefore its standard uncertainty is given by:

$$u_{1m2} = \frac{s_{1m2}}{\sqrt{5}} . \tag{1}$$

Notice that the effect of resolution is taken into account by this calculation; therefore it is not necessary to evaluate it separately. Nevertheless, in the very rare situation of a resolution larger than the uniformity and repeatability effects, clearly indicated by no variation or of a single digit variation of the measurement results, it is useful to consider separately the uncertainty contribution due to resolution r_2 taken as type B contribution:

$$u_{m2r} = \frac{r_2}{\sqrt{12}} . \tag{2}$$

Combining these uncertainty contributions allows one to evaluate the uncertainty in the measurement difference between the primary hardness standardizing machine and the hardness calibration machine, which is estimated by the bias value:

$$B_{1,2} = H_{1m2} - H_{1m1} . \tag{3}$$

The relevant standard uncertainty is given by:

$$u_{B_{1,2}} = \sqrt{u_{1m1}^2 + u_{1m2}^2 + u_{m2r}^2} . \tag{4}$$

[1] Explanation of the symbols used in subscript: the first 1, indicates that the block is a first level type (standard block); m1 indicates that a machine of the first level (standard machine) was used. Therefore, for example, when the measurement on standardising blocks (level 2) are made with a hardness tester (level 3) we'll use the symbol H_{2m3} and so on.

Note that if the hardness value H_{1m1} of the primary hardness reference block is significantly dependent on the time passed since its calibration or on the number of previously made indentations; their contributions of uncertainty should be added. Since these effects are dependent on the material of the block, they should be defined by the authority that issued the block certificate, which can evaluate these effects and define the conditions to be observed to keep them negligible.

A more important and frequently non-negligible problem is interpolation of the measurement bias, as usually the bias of the hardness calibration machine is determined on three hardness levels, while hardness reference blocks are calibrated at many more hardness levels, so that an interpolation is required. One solution is to characterise the hardness calibration machine by defining, for instance with the least squares method, a typical model of the bias $B_{m2}(H)$ with its relevant standard uncertainty $u_{Bm2}(H)$ which are both functions of the hardness level H.

The standard uncertainty of H_2 should, therefore, be estimated by:

$$u_{H_2} = \sqrt{u_{1m1}^2 + u_{1m2}^2 + u_{m2r}^2 + u_{Bm2}^2(H)}\,. \quad (5)$$

3.2. Calibration of a Hardness Reference Block

As a first approximation, let us examine the case when the hardness of the hardness reference block H_{2m2} is nearly equal to that of the primary hardness reference block H_{1m1} (that is, no interpolation error). The calibration is accomplished by making five hardness measurements with the hardness calibration machine on the hardness reference block. In principle, the scheme is analogous to the previous step of calibrating the hardness calibration machine: five measurements are made on a hardness reference block. Taking the average H_{2m2} of the five calibration measurements and correcting it with the bias $B_{1,2}$, one obtains an estimate of the hardness H_2 of the hardness reference block, as

$$H_2 = H_{2m2} - B_{1,2}\,. \quad (6)$$

Again we have two main contributions to the uncertainty of H_2, that of H_{2m2} that can be evaluated by the standard deviation of s_{2m2} of the five measurement results and calculated by the formula:

$$u_{2m2} = \frac{s_{2m2}}{\sqrt{5}} \quad (7)$$

and that of the bias $B_{1,2}$ already evaluated by $u_{B_{1,2}}$.

Notice that effects of variations of testing parameters over time, for example, change of temperature, machine operator and other environmental conditions, can significantly affect the uncertainty. For this reason an evaluation of the reproducibility instead of repeatability should be done.

An additional, frequently negligible, component is that due to the resolution of the Hardness Calibration Machine u_{m2r}.

The standard uncertainty of H_2 should be, therefore, estimated by:

$$u_{H_2} = \sqrt{u_{2m2}^2 + u_{B_{1,2}}^2 + u_{m2r}^2} = \sqrt{u_{2m2}^2 + u_{1m1}^2 + u_{1m2}^2 + 2u_{m2r}^2 + u_{Bm2}^2(H)}\,. \quad (8)$$

3.3. Calibration of a hardness tester

The calibration of a hardness tester is very similar to the calibration of a hardness calibration machine. Hardness reference blocks are used to determine the bias error of the tester $B_{1,3}$ by comparing the certified value H_2 of the block with the average H_{3m2} of five values measured by the tester on the hardness reference block. Using the same symbols as before, but changing the index from 1 to 2, because a hardness reference block is used, and from m2 to m3, because a hardness tester is used:

$$B_{1,3} = H_{2m3} - H_2 \quad (9)$$

and the relevant uncertainty is given by:

$$u_{B_{1,3}} = \sqrt{u_{H_2}^2 + u_{2m3}^2 + u_{m3r}^2}\,. \quad (10)$$

The problem of interpolation could also be important for the hardness tester. The hardness tester, too, can be characterised by defining, with the least squares method, a typical model of the bias $B_{m3}(H)$ with its relevant standard uncertainty $u_{Bm3}(H)$ which are both functions of the hardness level H.

The standard uncertainty of $B_{1,3}$ should be, therefore, estimated by:

$$u_{B_{1,3}} = \sqrt{u_{H_2}^2 + u_{2m3}^2 + u_{m3r}^2 + u_{Bm3}^2(H)}\,. \quad (11)$$

3.4. Measurement on a test piece

The uncertainty contributions for measurements on a test piece are the same as for the calibration of a hardness reference block, that is, the uncertainty on the bias of the hardness tester used and the uncertainty due to non-repeatability of the tester and non-uniformity of the test piece. Notice that the non-repeatability of measurements on a test piece, which can have any shape and elastic characteristic, can be very different from the repeatability as evaluated on a hardness reference block. This means that a single measurement on a test piece is not sufficient to estimate this component of uncertainty, which can only be evaluated using the standard deviation of multiple results. Care should be taken on the meaning of the requested results. An example can help. Let's consider that hardness measurements are made to determine the correct parameters of a heat treatment. In this case, one has a well defined target to be compared with the mean hardness of the treated material. The significant value is the mean value, and it could be correct to use the standard deviation of the mean value as an index of the possible variation of the process, therefore the non-repeatability and non-uniformity may be estimated by the standard uncertainty:

$$u_{3m3} = \frac{s_{3m3}}{\sqrt{n}} \qquad (12)$$

where n is the number of data used to calculate the mean.

Contrarily, in the case that the hardness evaluation is made for a safety check on the proof load of the material tested, it is no longer the mean value that is of interest, but the values of every single piece, which can break under excessive load. In this case, the non-repeatability and non-uniformity contribution u_{3m3} is given by the standard deviation s_{3m3} itself. Again, as observed in 3.2, reproducibility instead of repeatability should be considered. Correcting the average hardness value of each piece H_{3m3} with the bias $B_{m3}(H)$, one obtains an estimate of the hardness H_3 of:

$$H_3 = H_{3m3} - B_{m3}(H), \qquad (13)$$

and the relevant uncertainty can be expressed as:

$$u_{H_3} = \sqrt{u_{3m3}^2 + u_{B_{1,3}}^2 + u_{m3r}^2} = \sqrt{u_{3m3}^2 + u_{2m2}^2 + u_{2m3}^2 + 2u_{m3r}^2 + u_{Bm3}^2(H)}. \qquad (14)$$

The expanded uncertainty, usually having a small number ν of degrees of freedom, shall be calculated using a coverage factor corresponding to the Student-t distribution for a confidence level of 95%.

4. USEFUL APPROXIMATIONS OF THE UNCERTAINTY VALUE

The described evaluation of uncertainty corresponds to the correct practice prescribed by the GUM to correct measurement results with the bias error. This practice is usually applied in the calibration of hardness reference blocks, but is frequently not applied by the end user. Let's, therefore, examine two levels of approximation, the first one for the user that is willing to accept a large uncertainty without performing the calculations described above, and a second one for the user that accepts to evaluate uncertainty but cannot apply the bias correction.

4.1. Simpler approximation but larger uncertainty

This approximation is, practically, a translation of the usual conventional method of tolerances in standard documents into the language of uncertainty. It is based on type B uncertainty contributions.

Let's examine the single factors:

- u_{2m2} is defined by a maximum range $2T_{2m2}$ in the document describing the calibration of Hardness Reference Blocks
- u_{2m3} is defined by a maximum range $2T_{2m3}$ in the document describing the calibration of hardness testers
- u_{m3r} is defined by the resolution r required in the document describing the calibration of hardness testers
- u_{Bm3} is defined by a tolerance T_{Bm3} in the document describing the calibration of hardness testers

Most of the contributions of uncertainty can, therefore, be calculate as type B contributions. One shall add information on the maximum variation range, defined as $2T_{2m2}$, expected for the non-uniformity of test pieces, to complete the data of the calculation. It can be useful to evaluate two factors, the first dependent only on the tolerances of the standard document, which can be calculated once for all applications (and which should be put on the standard document itself):

$$\left(u_{H_3}\right)_1 = \sqrt{\frac{T_{2m2}^2 + T_{2m3}^2 + 2r_{m3}^2 + T_{Bm3}^2}{3}} \qquad (15)$$

and the second strongly depending on the material tested

$$\left(u_{H_3}\right)_2 = \sqrt{\frac{T_{3m3}^2}{3}}. \qquad (16)$$

Therefore the total uncertainty is given by:

$$u_{H_3} \approx \sqrt{\left(u_{H_3}\right)_1^2 + \frac{T_{3m3}^2}{3}} \qquad (17)$$

and, being that the contributions are mainly type B, the extended uncertainty at 95% confidence level is given by:

$$U_{H_3} \approx 2u_{H_3}. \qquad (18)$$

4.2. Approximation when bias correction is neglected

The case of neglecting bias correction is common, so the GUM proposes specific approximations in appendix F.2.4.5. The simpler approximation consists of adding the maximum expanded uncertainty U_{max}, calculated assuming that the bias is identically zero, to the maximum absolute value of the bias B_{max}. For calculating the expanded uncertainty, an evaluation of the degrees of freedom should be done, and the relevant Student-t distribution used as coverage factor. The approximated extended uncertainty, in this case, is:

$$U_{H_3} \approx \left(t\sqrt{u_{3m3}^2 + u_{2m2}^2 + u_{2m3}^2 + 2u_{m3r}^2}\right)_{max} + B_{max}. \qquad (19)$$

5. CONCLUSIONS

The uncertainty evaluation for hardness measurement can be greatly simplified by the assumption that measurements on materials different from those used for hardness reference blocks do not produce unexpected effects. This assumption shall be verified when very special materials are used, but, in common practice, can be accepted, as confirmed by many years of satisfactory experience with the present standard methods. The calculations are traditional and can become very simple by accepting significant approximations and a larger uncertainty band. In any case, however, care should be taken on two factors, the effect of the age of reference blocks and the effect of the number of indentations present on the block. These factors can be significant and it should be the responsibility of the block producer to declare these effects or to provide appropriate restrictions on the block usage in the certificate. It is also important that a declaration of the framework of validity of the uncertainty assessment be given in the certificates of primary hardness reference blocks and hardness reference blocks, to allow the user to understand in which conditions his uncertainty calculations will assure result compatibility.

REFERENCES

[1] GUM: "Guide to the Expression of Uncertainty in Measurement", *BIPM, IEC, ISCC, ISO, IUPAC, IUPAP, OIML, ISO*, 1993.

[2] G. Barbato, F. Petik, Metrological involvement in the definition and dissemination of hardness scales, *XIII IMEKO World Congress*, 761-766, 1994.

[3] OIML SP 19/SR 4, « Compe-rendu de la comparaison internationale des échelles de dureté », Prague, 1984.

[4] H. Stute; Final Report, Ref. Contract no 297/1/4/31/77/8 BCR D. *Staatliches Materialprüfungsamt Nordrhein-Westfalen*, Dortmund, Germany, 1980.

[5] J. Wood, "Intercomparison of Hardness Measurement for the Community Bureau of Reference (BCR)", *National Physical Laboratory*, Teddington, United Kingdom, 1986.

[6] G. Peggs, "Synthesis Report on the Intercomparison of Hardness Measurements for the Community Bureau of reference (BCR)", *National Physical Laboratory*, ISSN 0309-3069, Teddington, United Kingdom, 1988.

[7] D. Gould, C. Helmrath, "The development of a unified HRC hardness scale", *VDI Berichte 804*, Düsseldorf, 316-324, 1989.

[8] G. Barbato, S. Desogus, A. Germak, "Comments on the results obtained in the comparison of the HRC scales maintained at ETCA, IMGC and MPA NRW", *VDI Berichte n. 1194*, November 1995, pp. 11-17.

[9] T. Polzin, D. Schwenk, "World-Wide Unified Scales for the Rockwell Hardness Test with Conical Indenters – Possibilities to Achieve a World Wide Scale", *VDI/VDE-GMA, IMEKO TC3/TC5/TC20 Joint International Conference, 24-26 Sept. 2002, VDI-Berichte 1685,* Tagung Celle, Germany, 2002: pp. 493-498

Authors:
Professor Giulio Barbato, Dipartimento di Sistemi di Produzione ed Economia dell'Azienda, Politecnico di Torino, Corso Duca degli Abruzzi 24, 10129, Torino, Italy, phone: +39 011 5647285, fax: +39 011 5647299, giulio.barbato@polito.it.
Alessandro Germak, Istituto di Metrologia "G. Colonnetti" (IMGC-CNR), strada delle cacce 73, I-10135 Torino, Italy, Tel: +39.011.3977.367, Fax: +39.011.3977.426, E-mail: A.Germak@imgc.cnr.it
Samuel R. Low, National Institute of Standards and Technology (NIST), 100 Bureau Drive, Stop 8553, Gaithersburg, MD 20899-8553 U.S.A., Tel: +1.301.975.5709, Fax: +1.301.975.4553, e-mail: samuel.low@nist.gov