INDIRECT VERIFICATION OF ELASTOMER HARDNESS TESTERS USING HARDNESS REFERENCE BLOCKS

A. Štibler[1], K. Herrmann[2]

[1] ZAG Ljubljana, Dimičeva 12, 1000 Ljubljana, Slovenia , E-mail:anton.stibler@zag.si
[2] PTB Braunschweig, Bundesallee 100, 38116 Braunschweig, Germany , E-mail:konrad.herrmann@ptb.de

Abstract − Hardness measurement methods for rubber and plastics are described in the standards ISO 7619-1, ISO 7619-2, ISO 868 and ISO 48. Rising requirements for the accuracy of this hardness measurement have led to the result that the users of rubber hardness testers and also metrology experts who deal with their calibration need a calibration method to check the overall function of the testers acknowledged by a standard. Therefore extensive experiments were conducted in a cooperation between the Physikalisch - Technische Bundesanstalt (PTB) in Braunschweig (Germany) and the ZAG Ljubljana (Slovenia). The results have been used as a basis for the elaboration of the relevant international standard ISO 18898 which was published in 2006. In this standard direct verification methods are laid down.

An indirect verification method using rubber hardness reference blocks, which is also applicable for the verification of hardness testers, is presented in this paper.

At present hardness reference blocks are available only for some of the hardness scales which are used for rubber materials. Suchwise, Shore A, IRHD N and IRHD M reference blocks are available in the market.
During the verification process the hardness tester is calibrated by using rubber hardness reference blocks. The evaluation of the repeatability and the error of the hardness testers including the uncertainty of measurement of the calibration results is given in this paper.

Keywords: Hardness; uncertainty; rubber; indirect verification

1. INTRODUCTION

Rubber and plastics (both groups of polymers) differ very considerably from other engineering materials. They are very deformable materials, and almost completely recoverable. Measurements of their physical properties, like hardness, thus differ remarkably from procedures used for other materials.

The principal properties requiring measurement are concerned with visco-elastic flow behaviour. This term covers plasticity, viscosity and other parameters.

Hardness is a property which shows the material´s resistance to the penetration of a specified indenter, which is forced into the material under specified conditions.

Commonly used hardness indentation measurement methods measure the degree of penetration into the material, which is inversely related to the hardness values. This means that for a hardness value of 0 total penetration is needed, whereas in the case of a maximum hardness value of 100 there is no penetration. Hardness depends on the modulus of elasticity (Young's modulus) of the material, the viscoelastic properties of the material, the thickness of the test piece, the geometry of the indenter, the pressure exerted, the rate of increase of pressure, and the time interval at which the hardness value is recorded.

A cone indenter (which can be truncated) or a ball indenter is used in standard hardness test methods.
The indenting force acting on the indenter during the test can be applied in two ways:

- by applying a varying (spring load) force…..Shore scales;
- by applying a constant (dead weight) force…IRHD scales.

In the case of a varying force the indenting force varies with the depth of indentation.

2. MEASUREMENT METHODS

Measurands for Shore A, IRHD N and IRHD M are presented in the figures below.
The dimensions are given in the standards ISO 7619-1, ISO 7619-2, ISO 868 and ISO 48.[1-4]

3. INDIRECT VERIFICATION

The indirect verification should be carried out at a temperature of (23 ±5) °C by means of calibrated reference blocks. If the verification is made outside of this temperature range, this shall be reported in the verification report.

3.1. Quality of the blocks
The test and bottom surface of the reference block shall be parallel, smooth and plane. The hardness shall be uniform all over the surface with repeatability of less or equal than 1,5 units for Shore A and 1,0 unit for IRHD N and IRHD M. At present elastomer reference blocks for the hardness scales Shore A, Shore D, IRHD N and IRHD M are on the market.

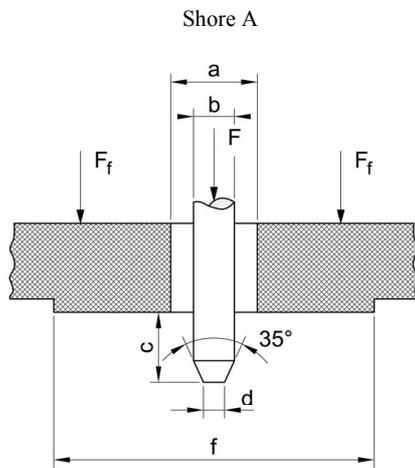Fig. 3 shows a set of elastomer blocks for the hardness scales Shore A and IRHD N.

Shore A

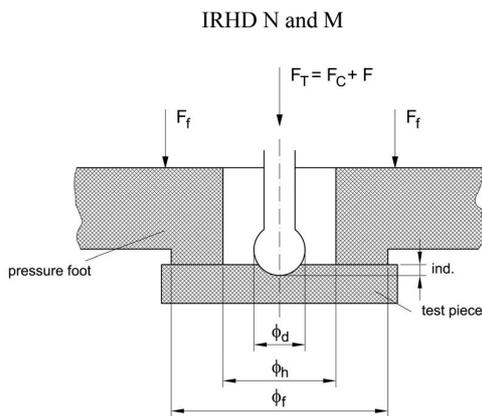Fig. 1. Measurands of indenter and pressure foot

IRHD N and M

Fig. 2. Measurands of indenter and pressure foot

Fig. 3. Set of elastomer hardness blocks for the hardness scales Shore A and IRHD N (courtesy Shore Co.)

Fig. 4 shows an elastomer hardness block for the hardness scale IRHD M, which is mounted in a steel socket.

Fig. 4. Elastomer hardness block for the hardness scale IRHD M (courtesy Bareiss Co.)

Fig. 5 shows a set of elastomer hardness blocks for the hardness scale IRHD M with designated measurement locations.
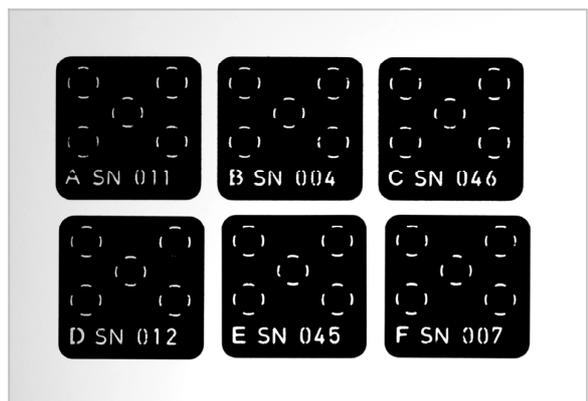

Fig. 5. Set of elastomer hardness blocks for the hardness scale IRHD M (courtesy Hildebrand Co.)

Finally, in Fig. 6 sets of elastomer hardness blocks for Shore A (rectangular form) and Shore D (disc form) are depicted.

Fig. 6. Sets of elastomer hardness blocks for Shore A (rectangular form) and Shore D (disc form) (courtesy Hildebrand Co.)

3.2. Procedure

Hardness measuring instruments shall be verified for each scale with at least three reference blocks. The blocks shall be selected from the hardness ranges given in Table 1. The hardness values of the blocks shall be chosen to approximate the limits of the intended use.

TABLE 1 – Hardness ranges of the blocks

Hardness scale	Hardness ranges
Shore A	25 to 95
IRHD N	25 to 95
IRHD M	35 to 85

On each reference block, five measurements uniformly distributed over the test surface shall be carried out. The test shall be made in accordance with ISO 7619-1. The hardness readings shall be taken to within 0.1 of a scale unit.

Before making these indentations at least one preliminary indentation shall be made to ensure that the instrument is working freely and that the reference block and the anvil are seated correctly. The result of this preliminary indentation shall be ignored.

For each reference block, let H_1, H_2,…H_5 be the values of the measured hardness arranged in increasing order of magnitude. The mean hardness value of the five measurements $\overline{H}$ is defined as follows:

$$\overline{H} = \frac{H_1 + H_2 + H_3 + H_4 + H_5}{5} \quad (1)$$

3.3. Repeatability

The repeatability r of the hardness testers under the particular verification conditions is calculated as:

$$r = H_5 - H_1 \quad (2)$$

The repeatability of the hardness tester is satisfactory when r fulfils the specification in Table 2.

3.4. Error

The error E of the hardness tester under the particular verification conditions is calculated by the following formula:

$$E = \overline{H} - H_{CRM}, \quad (3)$$

where H_{CRM} is the reported certified hardness value of the reference block

The error of the hardness tester shall not exceed the values given in Table 2.

TABLE 2 – Repeatability and error of the hardness tester

Hardness scale	Permissible repeatability r of the hardness tester Units of reading	Permissible error E of the hardness tester Units of reading
Shore A	3	± 4
IRHD N	2	± 3
IRHD M	2	± 6

4. UNCERTAINTY OF THE CALIBRATION RESULTS

4.1. Direct verification of the hardness testing instruments

The direct verification of the hardness tester according to ISO 18898 [5] comprises the verification of the test force, the depth measuring system and the indenter geometry.

4.1.1. Uncertainty of test force

The combined relative standard uncertainty of the test force calibration is calculated according to the following equation:

$$u_F = \sqrt{u_{FRS}^2 + u_{FHTM}^2} \quad (4)$$

With

u_{FRS} − relative uncertainty of measurement of the force transducer (from calibration certificate)

u_{FHTM} − relative standard uncertainty of the test force generated by the hardness tester

The last term is determined as follows:

$$u_{FHTM} = \frac{s_{Fi}}{\overline{F}\sqrt{n}} \quad (5)$$

with

s_{Fi} - standard deviation of the test force indication values for the i-th test force value

$\overline{F}$ - mean value of repeated test force measurements

n – number of repetitions of test force measurements

4.1.2. Uncertainty of depth measuring system

The combined relative standard uncertainty of the depth measuring system is calculated as follows:

$$u_L = \sqrt{u_{LRS}^2 + u_{ms}^2 + u_{LHTM}^2} \quad (6)$$

with

u_{LRS} − relative uncertainty of measurement of the depth calibration device (reference standard), from the calibration certificate for k = 1

u_{ms} − relative uncertainty of measurement due to the resolution of the measuring device

u_{LHTM} − relative standard uncertainty of measurement of the hardness tester

The last term is determined as follows:

$$u_{LHTM} = \frac{s_{Li}}{\overline{L}\sqrt{n}} \quad (7)$$

with

s_{Li} − standard deviation of the depth indication values for the i-th indication value of the depth calibration device

$\overline{L}$ - mean value of repeated depth measurements

n – number of repetitions of depth measurements

4.1.3. Uncertainty of indenter geometry

The indenters used for Shore A are a truncated cone, for Shore D a cone with a tip rounding and for IRHD balls. Accordingly the uncertainty of the indenter geometry must be determined in different ways. For the cone angle the uncertainty of the determination of the indenter geometry is calculated as follows:

$$u_\alpha = \sqrt{u_{\alpha RS}^2 + u_{\alpha IND}^2} \qquad (8)$$

with

u_α – uncertainty of the indenter cone angle

$u_{\alpha RS}$ – uncertainty of the angle standard (e.g. angle gauge) from the calibration certificate for k = 1

$u_{\alpha IND}$ – standard uncertainty of measurement of the angle measuring device

The last term is determined as follows:

$$u_{\alpha IND} = \frac{s_\alpha}{\bar{\alpha}\sqrt{n}} \qquad (9)$$

with

s_α – standard deviation of the angle measurements on the indenter with the angle measuring device

$\bar{\alpha}$ - mean value of repeated angle measurements

n – number of repetitions of angle measurements

The uncertainty calculation for ball-shaped indenters is carried out in an analogous way.

4.2. Indirect verification of the hardness testing instruments

By indirect verification with hardness reference blocks, the overall function of the hardness testing equipment is checked and the repeatability as well as the deviation of the hardneass testing instrument from the real hardness value are determined.

The uncertainty of measurement of the indirect verification of the hardness testing instrument follows from the equation:

$$u_{HTM} = \sqrt{u_{CRM}^2 + u_{CRM-D}^2 + u_{ms}^2 + u_H^2} \qquad (10)$$

Where

u_{CRM} is the calibration uncertainty of the hardness reference block according to the calibration certificate for k=1.

u_{CRM-D} is the hardness change of the hardness reference block since its last calibration due to drift

Triangular distribution type → standard uncertainty

$$u_{(xi)} = \sqrt{\frac{1}{6}.a_i^2} \qquad (11)$$

u_{ms} is the uncertainty due to the resolution of the hardness testing instrument

Rectangular distribution type → standard uncertainty

$$u_{(xi)} = \sqrt{\frac{1}{3}.a_i^2} \qquad (12)$$

u_H is the standard uncertainty of hardness testing instrument when measuring reference blocks

$$u_H = \frac{t.s_H}{\sqrt{n}} \qquad (13)$$

t....1,15...student factor

n = 5...number of measurements

standard deviation:

$$s_H = \frac{1}{n-1}\sqrt{\sum\left(H_i - \bar{H}\right)^2} \qquad (14)$$

Expanded uncertainty of the hardness measurements:

$$U_{HTM} = k . u_{HTM} \qquad (15)$$

k = 2....coverage factor

Criterion of acceptability

$$E = \bar{H} - H_{CRM} \qquad (16)$$

$E_{max} = |E| + U_{HTM} \leq$ permissible error given in Table 2

5. INTERVALS BETWEEN VERIFICATION

Direct and indirect verification shall be performed:

- at least once every 12 months;
- before measuring instrument has been set to work first time;
- after dismantling and reassembling if measurement values could be affected.

6. VERIFICATION REPORT / CALIBRATION CERTIFICATE

The verification report/calibration certificate with an accredited logo confirms that the laboratory, which has performed the verification, is competent.

The verification report shall include the following information:

a.) a reference to this part of ISO 18898;

b.) identification data for the hardness measuring instrument;

c.) standard means of verification (ref. blocks, force standard device, indentation standard device, etc.) with its calibration certificates data (Number, date of calibration, validity) as a proof of its traceability;

d.) verified hardness scale(s);

e.) verification temperature;

f.) result obtained;

g.) date of verification and reference to the verification institute;

h.) uncertainty of the verification result.

7. CONCLUSION

After the standard ISO 18898 on methods for the direct verification of elastomer hardness testers was put into force, here methods for the indirect verification of these hardness testers with rubber hardness blocks including quantitative requirements to repeatability and error limits of the hardness testers are proposed. Further, requirements to the calibration periods, methods for the determination of the measurement uncertainty and the content of the verification report are presented.

The indirect verification at least should be carried out once in a year. Depending on the frequency of use of the hardness tester, this indirect verification should be realized in shorter periods, even as a daily check.

The proposal contained in this paper should be considered in the framework of the standardization work.

REFERENCES

[1] ISO 7619-1: Rubber, vulcanized or thermoplastic – Determination of indentation hardness – Part 1: Durometer method (Shore hardness)

[2] ISO 7619-2: Rubber, vulcanized or thermoplastic – Determination of indentation hardness – Part 2: IRHD pocketmeter method

[3] ISO 868: Plastics and ebonite – Determination of indentation hardness by means of durometer (Shore hardness)

[4] ISO 48: Rubber, vulcanized or thermoplastic – Determination of hardness (hardness between 10 IRHD and 100 IRHD)

[5] ISO 18898: Rubber, Verification and calibration of hardness testers

Authors:
A. Stibler
ZAG Ljubljana, Dimiceva 12, 1000 Ljubljana, Slovenia
Tel: +386 1 2804 519
Fax: +386 1 2804 484
e-mail: Anton.Stibler@zag.si

Dr. K. Herrmann
Physikalisch-Technische Bundesanstalt Braunschweig und Berlin
Bundesallee 100, 38116 Braunschweig, Germany
Tel: +49 531 592 5140
Fax: +49 531 592 5105
e-mail: konrad.herrmann@ptb.de