

IMEKO 2010 TC3, TC5 and TC22 Conferences  
Metrology in Modern Context  
November 22–25, 2010, Pattaya, Chonburi, Thailand

## SOME PROBLEMS USING REFERENCE RUBBER BLOCKS AS A STANDARD

*Anton Stibler*<sup>1</sup>, *Febo Menelao*<sup>2</sup>, *Konrad Herrmann*<sup>2</sup>

<sup>1</sup>ZAG-Slovenian National Building and Civil Engineering Institute, Ljubljana, Slovenija, anton.stibler@zag.si

<sup>2</sup>PTB-Physikalisch-Technische Bundesanstalt. Braunschweig, Germany

**Abstract** – Reference blocks can be used for the verification of hardness testers of rubber in the same way as in the case of hardness measurement of metals. When using hardness reference blocks for verification the so called indirect verification method is applied. This method is not yet part of the standard ISO 18898: Rubber, Verification and Calibration of Hardness Testers, but there is a high chance that this method will be a mandatory part of this standard in near future.

This paper deals with some problems when using rubber hardness blocks as a standard for the verification of hardness testers. Mechanical properties of rubber material are relatively unstable compared with those of metals but on the other hand rubber is a very deformable material and almost completely recoverable. This fact and results obtained during practical investigations lead us to the conclusion that even several indentations performed into the same point of the surface of the blocks can give us repeatable and therefore reliable hardness values. This is not the case in hardness measurement of metals where each indentation causes total destruction of the material under and around the indenter.

**Keywords:** Rubber hardness testers, rubber hardness blocks, calibrations

### 1. INTRODUCTION

In the field of metal hardness the use of hardness reference blocks for the indirect verification and calibration of hardness testing machines is a very convenient approach because the method is very fast and the hardness reference blocks are relatively cheap. At the same time hardness reference blocks made from metals guarantee a high quality which was developed in the course of more than 50 years. On the other hand, in the field of rubber hardness so far only test blocks made from rubber and plastics exist which are used for the daily check of corresponding hardness testers. The decisive reason for this restricted use is the limited stability of the blocks over time and the larger local inhomogeneity of rubber blocks as compared with metal blocks. But because recently rubber hardness blocks with improved quality are available on the market, the use of such blocks for the verification and calibration of rubber hardness testers seems to be realistic.

For the verification and calibration of rubber hardness testers the ISO 18898 describes direct calibration methods of force, indentation depth and time, but it does not contain indirect calibration methods using rubber hardness blocks.

The aim of this paper is to clarify the possibility to use rubber and plastics blocks for the verification and calibration of hardness testers, to present results of the investigation of the stability and uniformity of the blocks and ways for the reduction of measurement deviations of rubber hardness testers by periodic calibrations with rubber hardness reference blocks.

### 2. STABILITY OF HARDNESS OF RUBBER BLOCKS

In general stability of rubber hardness blocks depends on time (aging effect), conditions under which they are stored and possible changes of the material due to the use of the blocks.

#### 2.1. Aging effect

The limited time stability of rubber hardness blocks is due to chemical changes of the block material. For instance the softeners (oils), which are not chemically bonded to rubber molecules, migrate by diffusion to the rubber surface, where they evaporate. So, the gradual time loss of softeners causes rubber hardness to increase.

Several series of measurement – recalibrations have been carried out since 2000 (1994) to establish changes in hardness of rubber blocks over the time. Three sets of Shore A, three sets of IRHD N and two sets of IRHD M reference blocks were used. The sets were made by the following producers: SHORE Instruments, HAMPDEN EQUIPMENT LTD, RAPRA and WALLACE. The sets of blocks were stored and kept between each recalibration in Laboratory for Metrology in Ljubljana under normal temperature condition between 19 °C and 25 °C and humidity between 30 % and 70 %.

Recalibrations were carried out on reference standards in the PTB (Physikalisch-Technische Bundesanstalt) in Braunschweig where rubber hardness reference standards are set up. The rubber hardness standard device is of the type U7294A made by Bareiss Co. in Oberdischingen (Germany). On this device corresponding measuring heads for the above mentioned rubber hardness scales were used.

Figures 1 and 2 show the typical hardness change of a sets of Shore A and IRHD N rubber hardness blocks over a period of ten years. From the slope of the curves it can be seen that the hardness slowly increases over the time.

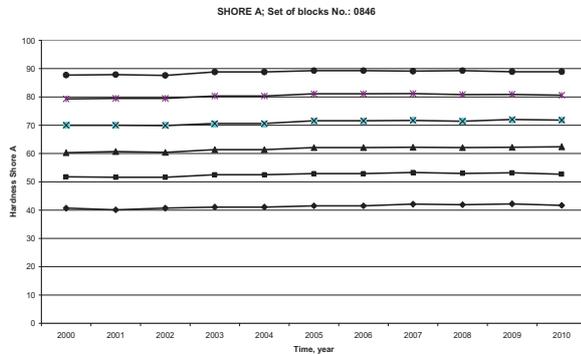


Fig. 1. Hardness change of Shore A rubber hardness blocks over time.

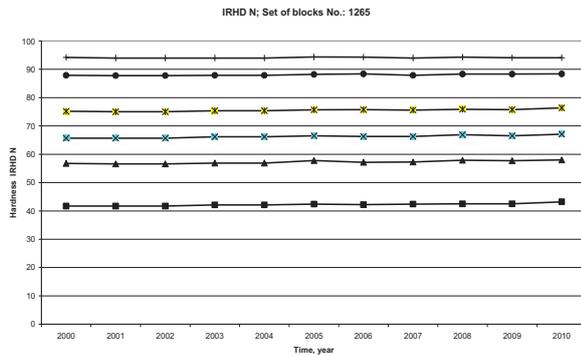


Fig. 2. Hardness change of IRHD N rubber hardness blocks over time.

Investigation results show a maximum yearly hardness change for the IRHD N sets of the reference blocks of 0,2 IRHD to 0,4 IRHD units. For Shore A sets of reference blocks this change is higher and reaches an amount of 0,3 Shore to 0,6 Shore units.

**2.2. Conditions of storage**

Stability of rubber blocks is affected by the conditions under which the blocks are stored. High temperature may cause a drop of hardness of the blocks but a normal condition between 10 °C and 30 °C and a relative humidity below 80 % are not critical. Results shown on Fig. 1 and 2 confirm this statement.

**2.3. Usage**

How do previously made indentations and the daily use of the blocks affect measured hardness?

At first we tried to find out whether indentation causes any plastic deformation as this is the case when measuring hardness of metals. To investigate this several series of indentations in one defined place were carried out on four different blocks. Results are shown on figure 3.

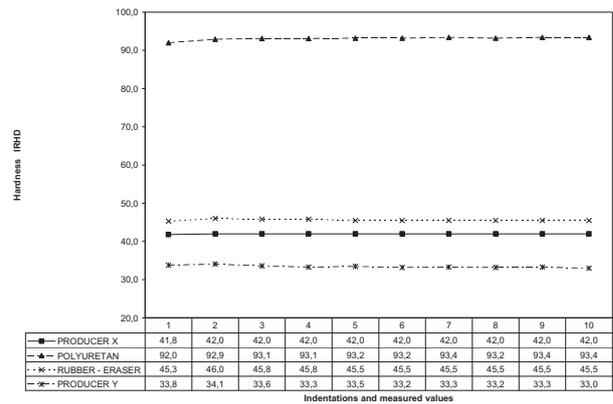


Fig. 3 One series of hardness measurement at one defined place on various materials and reference blocks.

Results for the block of producer x show excellent agreement after the first indentation is carried out while others show some relatively small variations after the second indentation which could be assigned to the material and its viscoelastic behaviour.

To find out how daily use of the blocks affects measured hardness a large number of indentations evenly spread on the surfaces of two sets of the blocks were carried out. This experiment is presented in the following items:

**2.3.1. Blocks**

The rubber test blocks had a size of 70 mm diameter and 6 mm thickness and were manufactured by Evonik Co. in Huerth (Germany). Fig. 4 shows the two sets, each for Shore A and IRHD-N.



Fig. 4 View of the two sets of rubber hardness test blocks for Shore A and IRHD-N

**2.3.2. Procedure**

In 15 days in Februari 2010 on each rubber test block 30 indentations were made each day. This result in 450 indentations per block after the whole investigation period. The measurements were performed with the rubber hardness tester U 7294 from Bareiss Co. in Oberdischingen (Germany). Indentations were evenly distributed on the surface and carried out in accordance with ISO 7619-1 and ISO 48. From the 30 measurements on each block the

average  $\bar{x}$ , the standard deviation  $s$  and the variation coefficient  $V = s/\bar{x}$  were calculated.

Further, at the begin of each week 10 measurements were made where the average of these measurements was used as the calibration value of the rubber block. For each day of the investigation the deviation of the indicated value  $\Delta H$  from the calibration value was determined by

$$\Delta H = H - H_C$$

where

H – indicated hardness value

H<sub>C</sub> – calibration value

### 2.3.3. Results

#### Change of hardness

Change of hardness in dependence on the number of indentations is depicted in Fig. 5, 6 and 7.

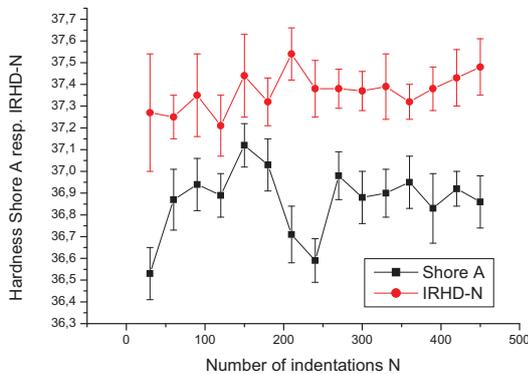


Fig. 5 Comparison of the change of hardness and the standard deviation (1 s) of the hardness values over the number of indentations for 40 Shore A and 40 IRHD-N

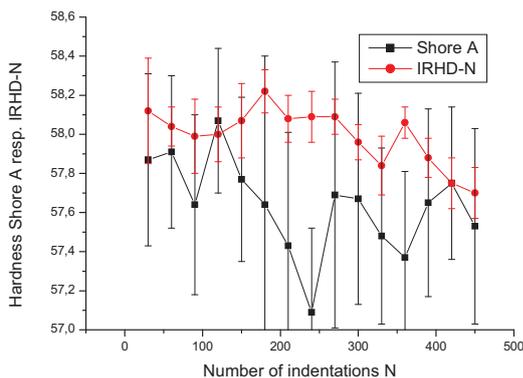


Fig. 6 Comparison of the change of hardness and the standard deviation (1 s) of the hardness values over the number of indentations for 60 Shore A and 60 IRHD-N

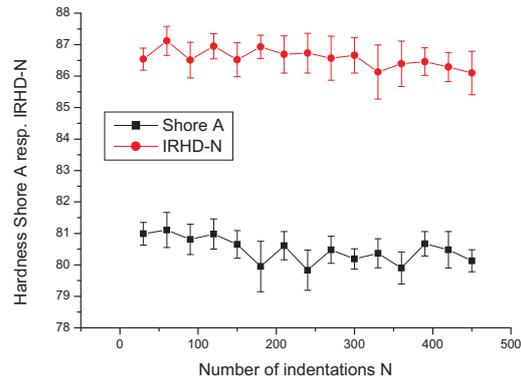


Fig. 7 Comparison of the change of hardness and the standard deviation (1 s) of the hardness values over the number of indentations for 80 Shore A and 86 IRHD-N

The figures clarify that the blocks for 40 Shore A and 40 IRHD-N have a slight increase in hardness, whereas the remaining blocks show a slight decrease in hardness. Generally, the variation of the hardness values at the method Shore A is larger than that for the method IRHD-N. The reason for the better performance of the method IRHD is that IRHD uses a ball indenter instead of the truncated cone for the method Shore A which produces high stress peaks at the edge of the cone. Further, the force duration time at IRHD-N is 30 s against 3 s at Shore A. After 30 s the creep of the material is much less than after 3 s. But, in summary the hardness change is not larger than 0,5 IRHD-N and 0,5 Shore A for all blocks after 450 indentations.

#### Change of variation coefficient

The change of the variation coefficient in dependence on the number of indentations is depicted in the Fig. 8 and 9.

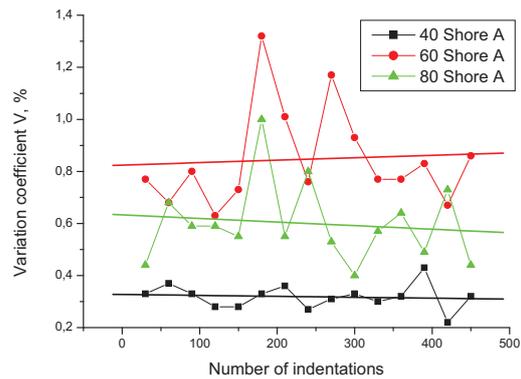


Fig. 8 Change of the variation coefficient of Shore A hardness values in dependence on the number of indentations

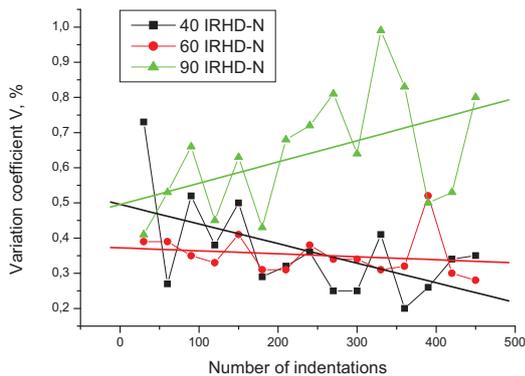


Fig. 9 Change of the variation coefficient of IRHD-N hardness values in dependence on the number of indentations

Generally, the variation coefficient increases with increasing hardness. Whereas for the method Shore A no dependence on the number of indentations can be seen, for the method IRHD-N there is a slight decrease of the variation coefficient at 40 IRHD-N, no change at 60 IRHD-N and an increase at 86 IRHD-N.

#### *Change of hardness deviation*

Change of the hardness deviation in dependence on the number of indentations can be seen in the Fig. 10 and 11.

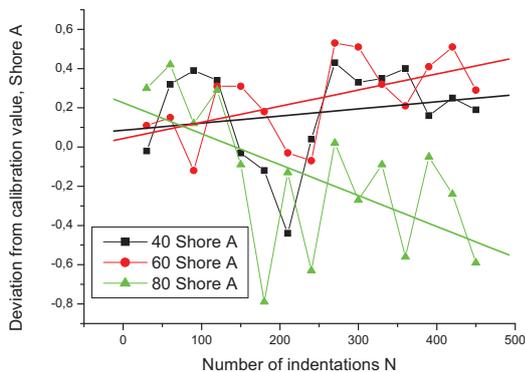


Fig. 10 Change of the hardness deviation from the calibration value for Shore A hardness in dependence on the number of indentations

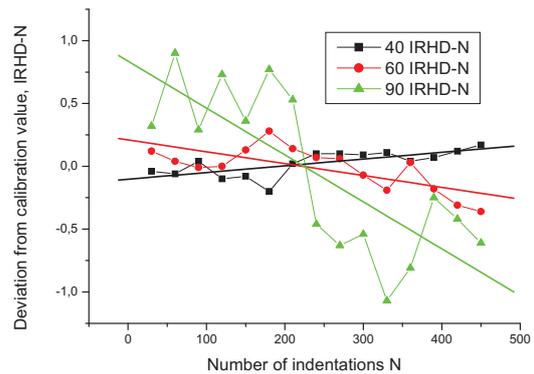


Fig. 11 Change of the hardness deviation from the calibration value for IRHD-N hardness in dependence on the number of indentations

The figures clarify that the hardness deviations for 40 Shore A and 60 Shore A resp. IRHD-N are somewhat smaller than for 80 Shore A resp. 86 IRHD-N. In absolute numbers the hardness deviation from the calibration value is not larger than 0,5 Shore A resp. IRHD-N for 40 Shore A and 60 Shore A resp. IRHD-N. For 80 Shore A a maximum hardness deviation of 0,8 Shore A and for 86 IRHD-N a maximum hardness deviation of 1,1 IRHD-N was observed.

### 3. CONCLUSIONS

On the basis of the presented results we can conclude that hardness measurement of elastomers is a repeatable process. This conclusion is in contrary to the hardness measurement of metals where the measurement is not repeatable.

Results obtained with repeated measurements in one defined place are in good agreement specially after the second initial indentation.

Results obtained with 450 indentations evenly spread on the surface of the two sets of blocks confirm that blocks can be used for the daily quality assurance of rubber hardness testing.

### REFERENCES

- [1] ISO 7619-1: Rubber, vulcanized or thermo-plastic, Determination of indentation hardness, Part 1: Durometer method (Shore hardness)
- [2] ISO 868: Plastics and ebonite - Determination of indentation hardness by means of a durometer (Shore hardness)
- [3] ISO 48: Rubber, vulcanized or thermoplastic - Determination of hardness (hardness between 10 IRHD and 100 IRHD)
- [4] ISO 18898: Rubber - Calibration and verification of hardness testers.
- [5] R. Brown, Physical Testing of Rubber; Chapman & Hall, 1996
- [6] A. Štibler, K. Herrmann, Z. Šušterič: Long-term stability of rubber hardness reference blocks; HARDMEKO 2004, Novemb.11-12, 2004, Washington, D.C., USA
- [7] A. Štibler, K. Herrmann, Calibration of Hardness Testing Instruments for Elastomers and Research Work in Recent Years; Herteprüfung 2006, VDI-Berichte, Berlin, Nov. 20-21, 2006.