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NEW DEVELOPMENTS AND APPLICATIONS IN HARDNESS METROLOGY

Konrad Herrmann

Physikalisch-Technische Bundesanstalt Braunschweig and Berlin, Germany

Abstract – The overview on the development tendencies clarifies that the main developments refer to the increase of productivity and of the accuracy of measurements. With the development of new materials also new optimum hardness measuring methods are developed. Great efforts are undertaken to enlarge the information content of hardness measuring methods. In this connection great perspectives can be ascribed to the instrumented indentation test.

Keywords: Hardness metrology, measurement uncertainty, tendencies

1. INTRODUCTION

Hardness testing has a tremendous significance for quality assurance in industry. Although the most widely distributed hardness test methods for metals Rockwell, Brinell and Vickers already in the period between 1900 and 1925 were developed, just in the last ten years it has undergone a great many innovations. Here the increase of productivity and accuracy are top priority.

Hardness generally is defined as the resistance of a material against the intrusion of a harder material (the indenter). Hardness is a technical quantity and defined by the relationship $H = F/A$, where F is the test force and A a certain area under the indenter. For different hardness measuring methods A is defined in different ways. H in principle is a pressure, but it cannot be explained in a physical context. Moreover, the hardness measuring methods contain in their definition the geometrical specification of the indenter and a force-time-pattern. Therefore the hardness measurement results depend on the technical conditions ot the measurement, and this is the main reason that hardness is a technical quantity and does not belong to the SI units.

Hardness measurements are widely distributed in industry and research, because this measurement one gets information about mechanical properties of a material, not only of hardness, but also wear resistance and other correlated properties like lifetime and tensile strength. A very important point of view is that hardness measurements can be carried out very quickly, that the test cost are low and that the to be tested object practically will not be destroyed.

In practice, for different material groups several hardness measuring methods are applied. Fig. 1 shows their application ranges.

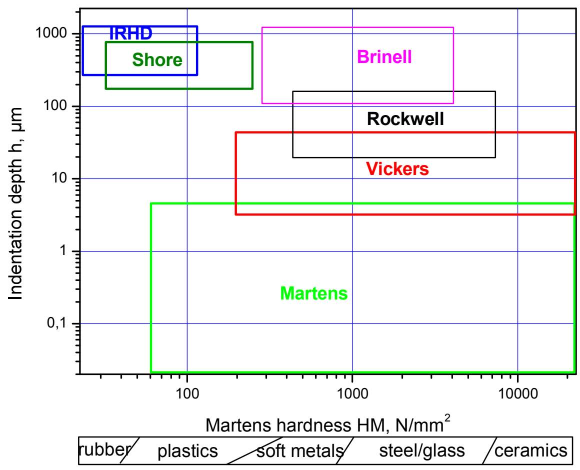

Fig. 1. Assignment of the most widely applied hardness measuring methods to material groups

In the following important development tendencies of hardness metrology will be discussed.

2. DEVELOPMENT TENDENCIES

2.1 Standardization, measurement uncertainty and traceability

Hardness laboratories are presently established in more than 20 National Metrology Institutes (NMI) worldwide taking care of the traceability of hardness measurements mainly in accredited calibration laboratories.

The general relationship between hardness definitions, NMIs, calibration laboratories and industry is depicted in Fig. 2.

The chain starts on the international level with the definitions of the various hardness scales in ISO standards. With standard hardness measurement machines on the national level primary hardness reference blocks for calibration laboratories are calibrated. The accuracy of the standard hardness measurement machines is achieved by direct calibrations on the highest level and checked by international comparisons.

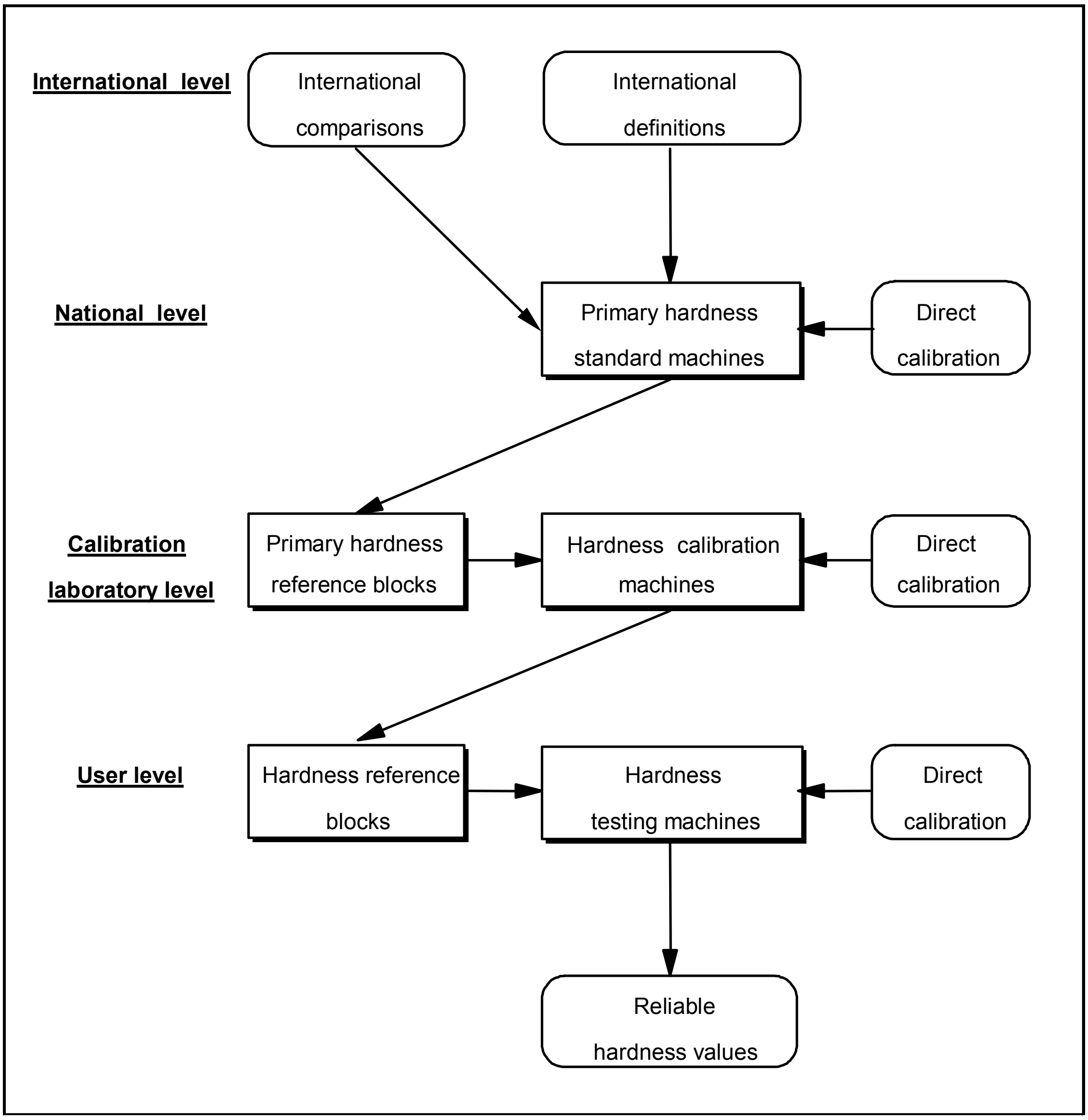

Fig. 2. Metrological chain of hardness measurements

The chain starts on the international level with the definitions of the various hardness scales in ISO standards. With standard hardness measurement machines on the national level primary hardness reference blocks for calibration laboratories are calibrated. The accuracy of the standard hardness measurement machines is achieved by direct calibrations on the highest level and checked by international comparisons.

The largest part of calibrations of hardness reference blocks is performed in calibration laboratories. For this serve hardness reference measurement machines, which are traced back by direct and indirect calibrations. The hardness reference blocks calibrated by them serve to check the hardness testing machines ot the users. Moreover the user periodically must carry out direct calibrations of the essential influence quantities, like test force, length measurement, indenter geometry and test cycle.

The recently revised ISO standards on hardness measuring methods Brinell, Vickers, Rockwell and Knoop now contain annexes with guidelines for the estimation of the uncertainty of hardness measurements. Because each of these ISO standards is structured in three parts, namely procedures for the test method, the calibration of the hardness testing machine and the calibration of hardness reference blocks, corresponding uncertainty guidelines for these three application fields had been developed. [1]

The guidelines enable the users of hardness measurements to calculate without extensive investigations the uncertainty with easily accessible information, i. e. data from the calibration certificate of the hardness reference block and the measurement results of the hardness reference block and of a sample on the hardness testing machine. On this basis the uncertainty of the hardness measuring method is determined as follows:

$$U = k\sqrt{u_E^2 + u_{CRM}^2 + u_H^2 + u_x^2 + u_{ms}^2} \qquad (1)$$

where:

u_E - maximum permissible deviation of the hardness testing machine

u_{CRM} - calibration uncertainty of the hardness reference block

u_x – stand. uncertainty when measuring the hardness reference block on the hardness testing machine

u_H - standard uncertainty when measuring the sample on the hardness testing machine

u_{ms} - standard uncertainty of the length measuring system's resolution in the hardness testing machine

Apart from the determination of the uncertainty of hardness measurements it is also necessary to establish the measurement capability of hardness testing machines, that is to say a tolerance which must be kept in a technological process is compared with the uncertainty of the used hardness testing machine. For this purpose in Germany the elaboration of a corresponding VDI/VDE standard is under way.[2] The measurement capability C_{gk} for the field of hardness is expressed as follows:

$$C_{gk} = 0.2 \cdot \frac{T}{4s_w + |b|} \qquad (2)$$

with

T – tolerance

s_w – standard deviation of repeatability

b - deviation of test machine

A test machine is considered as capable, if $C_{gk} \geq 1.33$.

At present the ISO standard on hardness conversion ISO 18265 is under revision. An important point of view for the revision is the fact that the conversion values were established under test conditions, which differ from the test conditions defined now in the standards. Namely the change of the indenter material from hardened steel to hardmetal for the Brinell and the Rockwell scales and the reduction of the test force duration time for the Rockwell scales from 30 s to (4±2) s exert an influence on the conversion values. Table 1 shows deviations of hardness conversion values due to differing parameter specifications.[3]

TABLE 1. Maximum deviations of hardness conversion values for non and low alloyed steels due to differing parameter specifications

Parameter	HB 5/750	HRC	HRB
Indenter material	40 HBW	-	2.3 HR
Duration time of test force	-	0.8 HR	2.4 HR

2.2. Contributions to the hardness definition

Especially the international comparisons have revealed that the definition uncertainty of the hardness scales according to the definitions in the above mentioned ISO hardness standards significantly restricts the further reduction of the measurement uncertainty. Therefore the Working Group on Hardness (WGH) of CCM has undertaken the task to redefine the hardness scales. Fig. 3 shows a proposal for the redefined Rockwell hardness measuring method.[4]

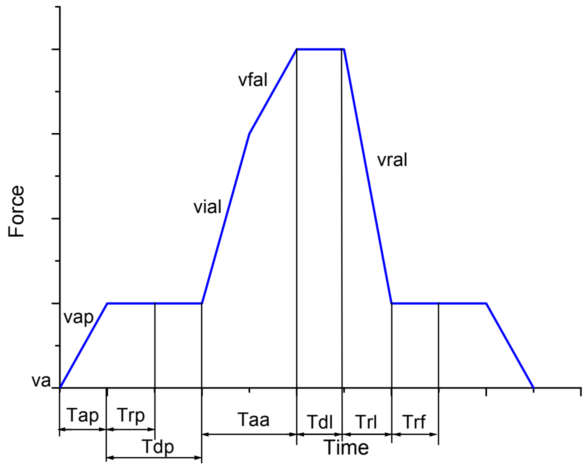

Fig. 3. Proposal for the redefinition of the Rockwell hardness measuring method

In contrast to ISO 6508-1 which at present prescribes the Rockwell hardness measuring method, for each section of the force-time-pattern the starting and the end point and the times when the depth values have to be taken are clearly defined. Moreover, in the section of the force application a reduction of the indenting velocity in the last part of the force increase is proposed which avoids an overshoot of the test force.

Recently a method for the calculation of the hardness of covalent and ionic crystals on the basis of the bond force S_{ij} between atoms was developed:[5]

$$S_{ij} = \sqrt{e_i e_j} / (d_{ij} n_{ij})$$
$$e_i = Z_i / R_i \qquad (3)$$

where:

Z_i - number of valence electrons of atom i

n_{ij} - number of bonds between neighbouring atoms i and j at shortest neighbouring distance d_{ij}

R_i - atom radius, in which is contained the valence electron charge Z_i

The hardness of an ideal monocrystal is proportional with the bond forces S_{ij} and the number of bonds in a unit cell of the crystal. In the simplest case of an element (for instance Si) the hardness delivers:

$$H[force / volume] = (C / \Omega) S_{ij} \qquad (4)$$

where:

C - proportionality constant, Ω - unit volume of the crystal

Investigation showed that the agreement of the theoretical hardness values by Simunek and Vackar with experimental data from Vickers and Knoop measurements is quite good.

This development shows that it is possible to calculate the hardness of ideal crystals on the basis of atom physics. This opens the possibility to establish reference materials in the range of high hardness, although the uncertainty of the such wise calculated hardness still must be investigated. For the practice it is important that on this basis the hardness of newly developed crystalline materials can be calculated on the basis of their crystal structure.

2.3. Further development of hardness measuring technique

The integration of closed-loop load-cell technology into hardness testing machines is the most beneficial improvement to conventional hardness testing machines in recent years. Compared with the so far used force application systems with calibrated weights or with hydraulic systems with valves this new technique enables universally to realize all conventional hardness scales Rockwell, Vickers and Brinell in one machine, moreover it overcomes the drawbacks of these traditional force application techniques. The computer controlled load-cell technology also enables to realize the force-time-pattern of the hardness measuring method automatically and with high precision. Further the load-cell technology allows to realize the instrumented indentation test with such hardness testing machines.[6]

In the field of the measurement of the indentation in the Rockwell hardness testing machines for the depth measurement ever more linescale systems with optical sensors are applied. The optical detector has the advantage that there is no mechanical contact between indenter and measuring sensor and as a consequence there is no friction.

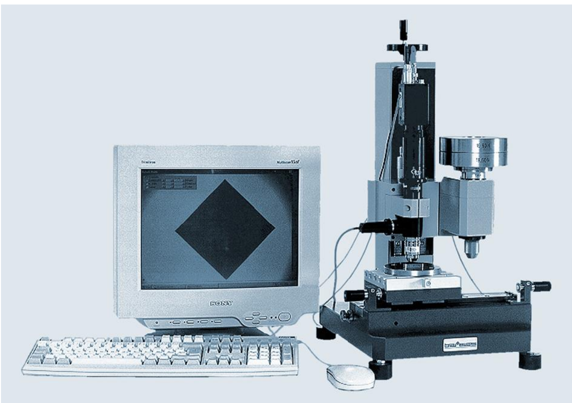

Fig. 4. Small force Vickers hardness tester with image analysis system (courtesy Bareiss Co.)

For the Vickers and Brinell methods optical image analysis on the basis of CCD sensor arrays have been introduced. These systems either display an enlarged view of the indent on a monitor, where the operator then must determine the edges of the indent, or the system is programmed to automatically measure the indent.

Both methods contribute to a considerable increase in the measurement productivity. Further they significantly reduce the operator fatigue and eye strain. At present many investigations are carried out in order to achieve a good agreement of measurement results obtained from conventional optical microscopes and from the new image analysis system. Fig. 4 presents a Vickers hardness measuring device with a CCD camera and a screen display for the indent measurement (force application by deadweights).

As the uncertainty of hardness measurements mainly depends on the properties and the geometry of the indenters, nowadays in the ISO hardness standards for ball indenters generally hardmetal balls are prescribed instead of steel balls which show larger deformation especially for harder to be tested materials. For the Rockwell and Vickers diamond indenters in the last years had been developed more accurate calibration methods of the indenter geometry. These calibration methods are based on interferometric and other optical principles and on the coordinate measurement with nanometric resolution.[7] In this way it was possible to reduce the calibration uncertainty of indenters.

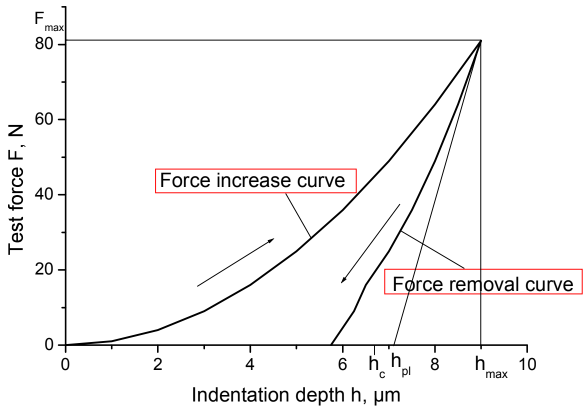

Fig. 5. Principle of the IIT

The automation of the indentation and measurement process is especially advantageous, where it is necessary to make multiple measurements, like on complicated samples or in the case of microhardness measurements.

2.4. Instrumented indentation test

At the instrumented indentation test (IIT) a synchronous measurement of test force F and indentation depth h is realized, where the indenter at first with a test force increasing until a chosen maximum value is pressed in the material; after this the test force will be reduced until the value zero. Fig. 5 shows this measurement procedure.

The figure clarifies that as result of the IIT two curves are obtained, from which a multitude of elastic and plastic material properties can be derived. The hysteresis between the force increasing and decreasing curve is

based on the fact that at the force increase occurs a plastic-elastic deformation and at the force decrease an elastic recovery.
For the IIT according to ISO 14577-1 the application ranges specified in table 1 are laid down.

TABLE 2. Application ranges of the IIT

Macro range	Micro range	Nano range
2 N ≤ F ≤ 30 kN	2 N > F; h > 0.2 µm	h ≤ 0.2 µm

From the IIT the elastic and plastic material properties specified in table 3 can be derived.

The high information content of the IIT especially in the micro and nano range is of high interest, because here often alternative measurement methods do not exist. The IIT is standardized in ISO 14577-1,-2,-3. [8] Peculiarities of the IIT on coatings are specified in ISO 14577-4.[9]

TABLE 3. Elastic and plastic material properties, which can be determined by IIT

Elastic resp. elastic-plastic properties	Plastic properties
Martens hardness HM	indentation hardness H_{IT}
Martens hardness, derived from the slope of the force-depth-curve HM_s	indentation creep C_{IT}
elastic indentation modulus E_{IT}	plastic deformation work W_{plast}
indentation relaxation R_{IT}	yield strength σ_y
elastic deformation work W_{elast}	
mechanical deformation work W_{total}	

The IIT in the nano range is mainly applied for the determination of the mechanical properties of thin layers of technical optics, architectural glasses, microelectronics devices, microsystem technique components and layers made of soft materials, like plastics and soft metals and finally soft materials, like biological tissue.

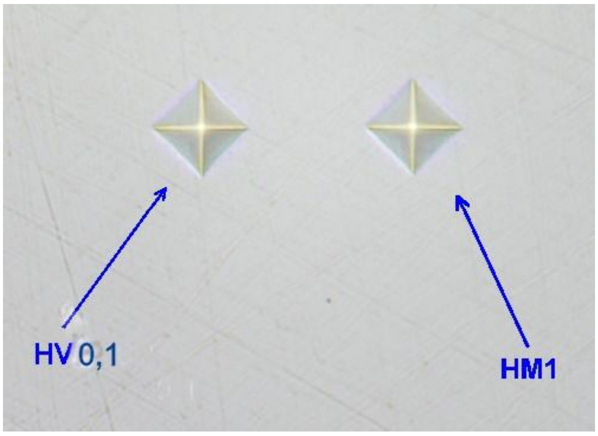

Fig. 6. Substitution of a MicroVickers by a Martens hardness measurement

The micro range of IIT advantageously is applied for the determination of mechanical properties of layers with a thickness > 2 µm and microelectronical and microsystem components. Applications are found in such industrial branches, like the tool manufacture and technical optics.

Recently, it is observed that the MicroVickers test according to ISO 6507-1 more and more is substituted by the IIT in the micro range, because the latter excels with a smaller uncertainty. Further the IIT is not limited to diagonal lengths of the MicroVickers indents of d ≥ 20 µm, so that indents can be realized which are even smaller than MicroVickers indents. Fig. 6 clarifies that the size of the HV0,1 indent is about the same as that of the HM1 indent. The diagonal length of the MicroVickers indent is 21 µm, the indentation depth of the Martens hardness indent is about 3 µm. The decisive difference between both indents consists in the fact that the measurement uncertainty of the MicroVickers measurement amounts to 4.6 %, but the uncertainty of the Martens hardness measurement only 2.3 % (values of PTB).

An interesting further development of the instrumented indentation test is the mapping of the hardness over an test area.[10] This is for instance especially useful if one wants to determine the distribution of material components in multi-component materials.
The main application of the IIT in the macro range is the determination of mechanical properties of various materials like metals, glasses, ceramics, plastics, paper and biological materials (bones and dental technique).
Recently, the determination of the hardening depth by IIT led to a remarkable rationalization effect as compared to the traditional method to perform between 10 and 30 MicroVickers indents on a carefully prepared cross section of the case hardened materials. At present investigations about the criteria to determine the hardening depth by IIT are carried out.[11]

At present, remarkable efforts are undertaken in order to derive the tensile parameters from the instrumented indentation test. There exists a high interest in the steel industry to use these possibilities, but this method is also extremely useful in all cases where it is not possible or convenient to manufacture tensile test specimen and perform the tensile test. In every case this application of the IIT will lead to a remarkable rationalization effect. Here especially the macro range is used, although some applications are found in the micro range. Mainly the following methods are used in order to derive the tensile parameters from the instrumented indentation test:

1) Conversion on the basis of statistical correlations
2) Expression of relationship between indentation and tensile parameters on the basis of neural network
3) Analytical expression of relationship between indentation and tensile parameters on the basis of dimensionless functions
4) Model of instrumented indentation test on the basis of Finite Element Method (FEM)

Fig. 7 shows the approach for the derivation of tensile test parameters from IIT based on FEM.

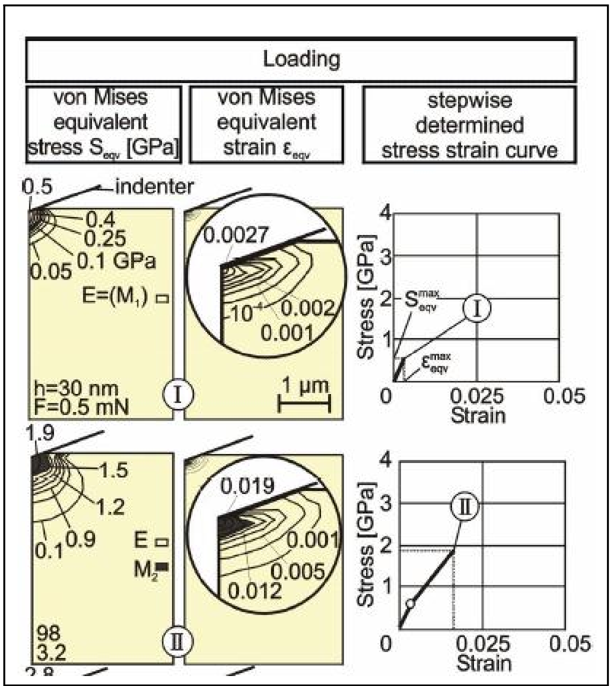

Fig. 7. Determination of stress-strain-curve by IIT and FEM [12]

Now a working group in the ISO TC 164/SC 3 “Hardness testing of metals” was founded in order to investigate the application ranges and limitations of the mentioned methods.

2.5. Portable and destruction-free hardness testers

For the hardness measurement of metals in industrial practice a huge amount of portable testers is used. Portable hardness testers are always applied in such cases, , if it is not possible to test the samples on stationary hardness testing machines, because the samples are too large and too heavy. Typical applications are hardness measurements for material identification, or in locations difficult to access, and in confined spaces.

Because at mobile hardness testing devices despite the large number of used devices up to now scarcely regulations about the definition of the test methods, their due realization and the calibration of the test devices exist, the German standardization group Material Testing DIN NMP 141 (Hardness Testing) in the last two years dedicated to the task to elaborate standards about Leeb hardness testing devices as well as mobile hardness testing devices with mechanical and electrical indentation depth measurement.

The Leeb hardness method belongs to the dynamic hardness test methods. The dynamic hardness differs essentially from the static hardness test methods. Whereas at the static hardness test methods the elastic deformation work plays a small role, at the dynamic hardness test always the Young’s modulus of the to be tested material has to be considered, because it significantly influences the rebound energy.

The hardness according to Leeb is defined as follows:

$$HL = \frac{v_R}{v_A} \cdot 1000 \qquad (5)$$

where:
v_R – rebound velocity
v_A – impact velocity

From equation (5) follows that the velocity of the impact body before and after the impact on the sample is measured and that from this the hardness value according to Leeb is calculated.

The standard draft DIN 50156 [13] about the Leeb hardness testing contains three parts:

- part 1: Test method
- part 2: Verification and calibration of the hardness testing devices
- part 3: Calibration of hardness reference blocks

Because the calibration of Leeb hardness reference blocks directly in Leeb hardness units enables a higher accuracy than their calibration in Vickers units and the following hardness conversion into Leeb hardness, in the PTB a Leeb hardness standard device, based on the principle of a down pipe was installed, which delivers traceability to calibration laboratories which calibrate Leeb hardness reference blocks.

The standard draft DIN 50157 [14] is related to the hardness testing with portable hardness testing devices, which are based on mechanical indentation depth measurement. The test method closely follows the Rockwell method, where the test forces are reduced, in order to fulfil the requirement of portability of the devices. The standard is structured into two parts:

1) Test method
2) Verification and calibration of hardness testing devices

The preliminary test force lies in a range between 10 N and 100 N und the total test force between 50 N and 1000 N. The hardness values, which are received with these devices, deliver the same values as stationary Rockwell hardness testing machines, but they are only obtained by corresponding calibration curves. As indenter a diamond cone with a cone angle of 120°, a cone frustum of diamond with a cone angle of 120° and a diameter of the frustum plateau of 0.064 mm and hardmetal balls with a diameter of 1,5875 mm are used. In a depth measurement range from 0 to 300 µm a hardness range from 10 to 70 HRC can be measured.

The mobile hardness testing with electrical indentation depth measurement is stipulated in the standard draft DIN 50158-1 and -2 [15]. A diamond indenter in the form of a cone or a pyramid provided with an electrically conductive layer manually is pressed with continually increasing force into the surface of a metal sample. The device registers the dependence of the electrical resistance on the increasing test force.

Recently in the framework of DIN drafts of standards on the UCI (UCI – Ultrasonic Contact Impedance) hardness testing were elaborated. Portable UCI hardness testers are widely distributed in industry. Again the standard with the designation DIN 50159 is structured into two parts [16]:

1) Test method

2) Verification and calibration of hardness testing devices

At the UCI method a vibration bar vibrating with ultrasonic frequency on whose lower end is a Vickers indenter is pressed with a defined test force into the sample (see Fig. 8). Its resonance frequency increases as soon as the indenter for the generation of the indent contacts the sample. The displacement of the resonance frequency Δf is determined under test force.

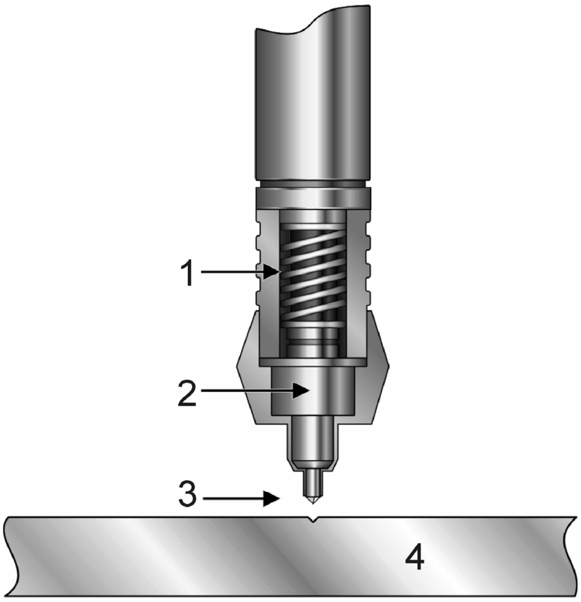

Fig. 8. Principle of the UCI hardness test method
1 – Test force applied by spring
2 – vibration bar
3 – Vickers indenter

By hardness reference blocks with a corresponding adjustment of the device the displacement of the resonance frequency Δf is assigned to the corresponding Vickers hardness. In the specification of the UCI method different test forces and requirements to the samples are laid down. The UCI hardness testers must be calibrated for each to be tested material. The standard foresees an indirect calibration of portable UCI hardness testers with hardness reference blocks.

A further newly developed, but not yet standardized method of portable hardness testing is the TIV method (Through Indenter Viewing) developed by GE Inspection Technologies Co.. This is an optical hardness test according to Vickers under test force.

An optical system including CCD camera views through the indenter. With this method the indenting process of the Vickers indenter into the sample material can be directly observed on a screen. When the test force is reached, the length of the diagonals will be determined and according to the definition of the Vickers hardness the hardness value will be calculated. Fig. 9 shows the set-up of the TIV device.

Fig. 9. Configuration of the portable TIV hardness tester consisting of handheld optical-mechanical Vickers tester and result screen (Courtesy GE Inspection Technologies)

Although at the beginning it was mentioned that hardness measuring methods practically do not destroy the sample, for many applications the indentations which are created by the hardness test cannot be tolerated. Therefore totally destruction-free hardness measurement methods are of high interest. Examples are the laserthermic, the ultrasonic and the eddy current measurement methods. These methods are based on a correlation between the mentioned physical phenomena and the hardness.

The KEMAG method which is standardised in [17] is based on a combination of various electromagnetic measurement methods. It is suited to measure the surface hardness and hardening depth of steel samples. In general, such measuring devices are calibrated with hardness reference blocks. The calibration is only valid for a chosen material and processing technology. Moreover, the dependence of the used physical effect on the distances from the material edges must be considered. Fig. 10 shows the device QUALIMAX according to the KEMAG method.

2.6. Elastomer hardness testing methods

Whereas in the field of hardness measurement of metals calibration methods for the hardness testing machines are applied already since decades, this is not the case for hardness measurements of rubber and plastics. Due to rising quality requirements in this field, a calibration instruction for IRHD and durometer rubber hardness testers was published as ISO 18898.[18]

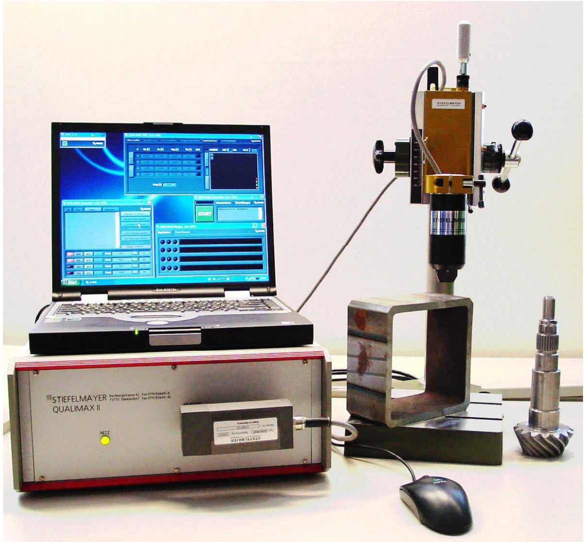

Fig. 10. View of the device QUALIMAX according to the KEMAG method (courtesy Stiefelmayer Co.)

It provides for the direct calibration of test force, indentation depth and indenter geometry. In Germany this service is offered by several calibration laboratories. For the daily check especially of portable rubber hardness testers rubber hardness test blocks are recommended. But contrary to hardness reference blocks made from metal which generally guarantee a stability of the calibrated hardness value for at least five years, rubber hardness test blocks have a stability of a calibrated hardness value of only six months. But this must not pose a problem, because if such blocks are used daily, they will be used up even in less than six months.

With the ever broader use of silicones especially in car industry and medicine the development of a hardness measuring method for silicones has become ever more urgent. The reason is that the measuring methods Shore A and Shore 00 which were used in the past for the hardness measurement of silicones do not possess sufficient capability to discriminate very low hardness values. Therefore a hardness measuring method for very soft elastomers like silicones with the designation Very Low Rubber Hardness (VLRH) was developed. At present the measuring method is being standardized in ISO. [19]

Because some rubber parts, like O-rings are produced in mass production, where for each O-ring a certificate is required, O-ring hardness test automats were developed and applied for quality assurance.

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