

Realization of Martens hardness method in macro range with high accuracy force and indentation depth

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ABSTRACT

TÜBİTAK UME Hardness Laboratory has been working on instrumentation in the field of hardness metrology since 2005 and three generations of hardness standardizing machines were developed since then to be used as reference (calibration/standardizing) machines in Türkiye. In former designs conventional hardness methods such as Rockwell, Brinell and Vickers scales were the main scope of the projects. In the final Project that was supported and funded by TÜBİTAK UME to develop three hardness standard machines to be used as national standards for the conventional hardness scales mentioned, the Instrumented Indentation Test (IIT) was also aimed at and some parameters like Martens hardness, creep, indentation hardness, (elastic and plastic) indentation work, etc. were also implemented onto the machines developed. It was a good occasion that the measurands in Rockwell hardness and IIT were the same, force and indentation depth besides time and this made it easier to realize the IIT on the same machine with a more suitable design to achieve the highest accuracy in terms of the measurands mentioned. In this paper the new design of the Rockwell-Brinell-Vickers hardness standard machine developed also for Martens hardness in macro range (3 kgf – 150 kgf) and preliminary Martens hardness measurements are explained.

Section: RESEARCH PAPER

Keywords: Hardness; Rockwell; Brinell; Vickers; Martens; indentation; instrumented indentation test

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1. INTRODUCTION

One of the main work areas at TÜBİTAK UME Hardness Laboratory has been instrumentation in hardness metrology field and as a result of the work achieved in this area primary hardness standard machines with dead-weight force application systems and laser interferometer depth measurement sensors have been designed and installed since 2005 in Türkiye for realization of the conventional hardness scales of Rockwell, Brinell and Vickers. With renewing the formerly established standards, much better systems working in the same manner were developed and installed with better metrological specifications, easier usage algorithm and much better design methodology [1], [2]. In the final design not only the conventional hardness scales were realized but also some parameters of IIT such as Martens hardness, indentation hardness, creep, indentation work (elastic,

plastic), etc. were all taken into consideration as it was a good occasion of their being based on the same measurands as the conventional hardness scale Rockwell; force, depth and time, providing us their realization with the highest possible accuracy.

In the conventional methods hardness for metallic materials is defined as the material resistance against plastic deformation and in these methods the size of indentation is measured during or after its realization to measure the size of the plastic deformation. In the IIT method, i.e. Martens hardness, material against plastic and elastic deformation is the main concern and the whole deformation under load is measured and the hardness value is calculated accordingly. In both types of hardness measurement methods, the depth of indentation against applied force is measured and the material reaction can be figured out by making use of the dead-weight force application system as source of force and laser interferometer optic systems as depth



Figure 1. Rockwell-Brinell-Vickers hardness standard machine of TÜBİTAK UME.

measurement sensor, both providing the highest accuracy measurement capability that will constitute the base of Martens hardness in this application and IIT in general.

The Rockwell-Brinell-Vickers hardness standard machine designed to realize the IIT too including Martens hardness is a special design made with the experience gained after successful implementation of previously developed hardness standardizing machines for TSE (Turkish Standards Institution) and TÜBİTAK UME Hardness Laboratory with better metrological properties, newer mechanical and software design, better repeatability and reproducibility in realization of conventional hardness scales regarding the relevant ISO hardness standards [3], [4], [5], [6], [7], [8]. Besides the conventional hardness scales realized in Rockwell, Brinell and Vickers methods, instrumented indentation test (IIT) was planned and the machine was designed such that IIT parameters such as Martens hardness, indentation hardness, creep and (elastic-plastic) indentation work, etc. be able to be measured with the highest accuracy in terms of force application and indentation depth measurement in accordance with ISO 14577-1 [9].

In this new design, as it was in the previous one, the force application system was considered to comprise mass stacks realizing force under the gravitational acceleration and a newly designed frame to transfer the realized force to the tip of the indenter. This time the mass stacks are also mentioned to be redesigned for easier calibration, mounting/demounting onto the machine and repair/replace, etc. The mechanical parts and machine body are completely renewed, aiming at more stringent, stable and reproducible construction. The dead-weight type Rockwell-Brinell-Vickers hardness standard machine (RBVHSM) designed and installed for realization of IIT too is given in Figure 1.

A laser interferometer optic system is integrated to the machine as an indentation depth measurement sensor. The depth measurement system is composed of laser source, linear interferometer and special design optics mounted on top of the indenter, connected to the indenter and realize the same displacement as the indenter. With this design it is aimed to figure out the path the indenter is passing inside the material during

realization of indentation for Martens hardness and other IIT parameters measurement.

The testing cycle is achieved via a high accurate force transducer to which the whole force application system is connected. The force application is sensed by measuring the force change on the force transducer instantaneously during load application. The indenter approach and indentation speeds are adjusted and controlled with the information taken from the laser interferometer system to be used to control the servo motor (M1) which will be used for realization of the indentation cycle and force application rate is measured via the change in the force transducer value in time. The RBVHSM is equipped with two motors more for selecting the masses related to each scale that provide the user to realize all measurements automatically.

2. THEORY AND MEASUREMENT METHOD

The measurement method of Rockwell, Brinell and Vickers hardness scales as conventional methods, are explained in detail in the relevant ISO hardness standards [3], [4], [5], [6], [7], [8]. In these scales the hardness measurement is mainly based on the material resistance against plastic deformation by realization of indentation and measurement of the size of the indentation such as depth, diameter and diagonal length, to be used for calculation of Rockwell, Brinell and Vickers hardness numbers, respectively. In all three methods the common approach is the measurement of the plastic deformation, the measurand indicating hardness value and realized under standardized conditions/components such as force applied, indenter and indentation cycle. According to its definition hardness measurement is realized in two steps; i) realization of deformation and ii) measurement of the resistance against this deformation. The general principle behind hardness measurement is the smaller size of indentation/deformation the harder the material or vice versa. The same procedure applies for Martens hardness with an important difference that is instead of plastic deformation elastic and plastic deformation is the main parameter to be measured and used for calculation of the hardness value. In a similar way the main components to be standardized for constituting Martens hardness scale are

- force application; possibly with the highest accuracy
- indentation cycle; possibly with the highest precision
- indenter; with the best geometrical properties
- indentation depth measurement; possibly with the highest accuracy.

In this study Martens hardness is realized in accordance with ISO 14577-1 [9].

The instrumented indentation test is realized with force (F) vs. indentation depth (h) cycle that also applies for Martens hardness is given in Figure 2. This is the general cycle used for extracting some other mechanical parameters of material such as creep, indentation work, indentation hardness, etc. with

- a : application of the test force,
- b : removal of the test force,
- c : tangent to the curve b at F_{\max} ,
- F : test force,
- F_{\max} : maximum test force,
- h : indentation depth under applied test force,
- h_{\max} : maximum indentation depth at F_{\max} ,
- h_p : permanent indentation depth after removal of the test force,
- h_r : point of intersection of the tangent c to the curve b at F_{\max} with the indentation depth-axis.

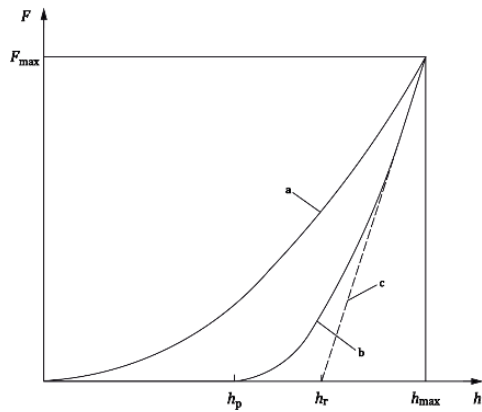


Figure 2. Instrumented Indentation Test (IIT) cycle.

In regard to the IIT that is applied according to the F vs. h cycle, one way to apply the force (F) vs. time (t) and the corresponding depth (h) vs. time (t) can be as in Figure 3. In these graphs the depth of indentation (h) is measured corresponding to the force applied (F) that can be a certain amount of force value as in this design corresponding to the conventional hardness scales' force values as it is a dead-weight hardness standard machine where

- a : application of the test force,
- b : maximum test force,
- c : removal of test force,
- d : test force = 0 N,
- e : indentation creep,
- f : recovery at zero test force.

In the IIT theory the material behaviour against penetration of a hard material under force is observed. In Martens hardness, in addition to the other metallic material conventional hardness scales (Rockwell) the total depth of indentation (corresponding to the elastic and plastic deformation) corresponding to the relevant force value is measured and the hardness value is calculated according to Equation (1) to Equation (3) that are given below.

$$HM = \frac{F}{A_s(h)} \quad (1)$$

$$A_s(h) = \frac{4 \cdot \sin \alpha}{\cos^2 \alpha} \cdot h^2 \quad (2)$$

$$= 26.43 \cdot h^2, \text{ for Vickers indenter}$$

$$HM = \frac{F}{26.43 \cdot h^2} \quad (3)$$

Once the applied force, indentation depth and time are measured simultaneously Martens hardness is calculated in accordance with Equations 1-3 and expressed as given in Figure 4.

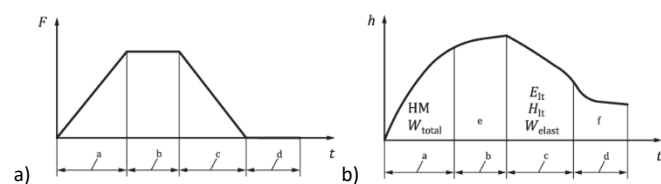


Figure 3. Martens hardness a) force and b) depth measurement cycles.

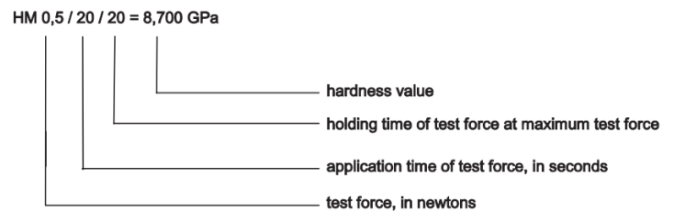


Figure 4. Designation of Martens hardness.

Since the total (plastic and elastic deformation) depth of indentation is the main concern of Martens hardness the zero point of all three measurands as well as their measurement accuracy are critical in the evaluation. The most affecting parameters can be considered as follows:

- zero point (start value) of force, depth and time,
- accuracy of force application and depth measurement,
- compliance of the machine [10],
- indenter geometry and penetration performance,
- interaction the sample and machine anvil,
- testing cycle such as time and velocity [11], [12].

With the design of the machine used in this application it is obvious that the accuracy of the three measurands is provided at the highest possible level with the smallest compliance error that can be resulted by the force application mechanism at such high force values. For determination of the zero points, high accuracy force measurement device (force transducer and indicator) and laser interferometer both with high data acquisition frequency are used and the zero points are determined with the highest precision and accuracy. Some other studies have shown force application velocity has significant effect on the measurements results [11], [12].

3. DESIGN OF THE MACHINE MECHANICS

The machine components used for realization of Rockwell, Brinell and Vickers Hardness scales as well as IIT, sensors used for measurement of force, depth and time for realization of measurements as well as to control all affecting parameters are designed to be with the highest accuracy and stability to come up with the most accurate outcome quantity hardness value. Not only metrological properties such as accuracy and stability but also the functionality, easiness to use and overcoming some important effects that are important for IIT are the main properties aimed at and the design was made accordingly.

There are two different methods used for detecting the zero-point in IIT which is used for also Martens hardness as explained in IIT method in the relevant standard ISO 14577-1 [9]; method 1: calculating the zero-point by extrapolation of a fitted function to the force vs. depth curve, method 2: the touch point determined from first increase of either the test force or contact stiffness. In this design of the machine both methods are possible but in the preliminary measurements of this application method 2 is used and its performance test results are given in Table 1.

3.1. Construction of Machine Body

In such applications where accuracy and stability in sub-micrometres are critical it is very important to have a rigid and sturdy body that will not affect the measurement results due to vibrations from neither the ambient nor the machine itself. To achieve a stable construction the machine body is designed to comprise the minimum number of parts with each having precise dimensions. All of the parts are produced with cast ironing with

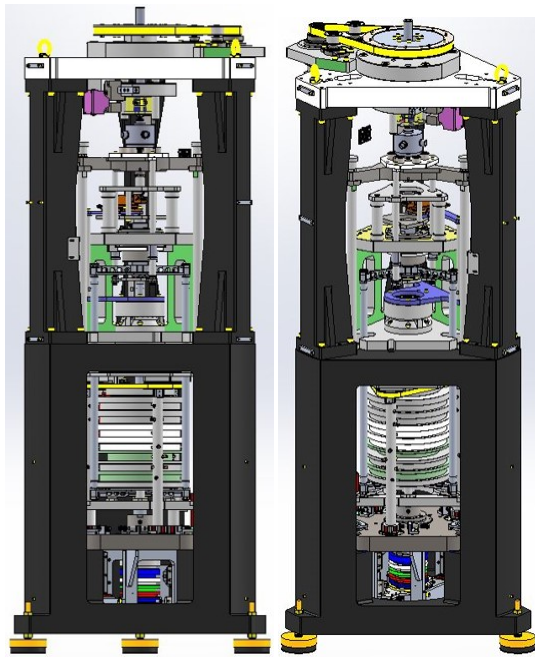


Figure 5. Rockwell-Brinell-Vickers hardness standard machine.

tight tolerances to achieve the requested quality. Besides production, mounting was a special application to fix every component in its best position to come up with a stable construction. The machine CAD is given in Figure 5.

3.2. Force Application System

As the methodology of force application was preferred to be dead weight system for better stability and highest accuracy, the force application system comprises mass stacks made up of stainless steel and a small frame to constitute the preload in Superficial Rockwell Hardness, HV3 in Vickers Hardness, 29.42 N force in Martens hardness and transfer the other loads to the tip of the indenter. The frame itself is adjusted to apply 3 kgf and the mass stacks are in additional order to constitute the other force values and scales. The other mass stacks are 12 kgf, 2×15 kgf, 8 kgf, 40 kgf (2×20) and 50 kgf (in three pieces) to constitute all Rockwell scales, Brinell scales up to 125 kgf and Vickers scales between HV3 – HV100, and in turn Martens hardness for the macro range (3 kgf – 150 kgf for the nominal values of the conventional hardness scales mentioned). The frame is guided by two air bearings at the two ends (Figure 6 a), both working with (4-6) bar air pressure to prevent its movement from any pendulum and rotational motions during load application that will improve the penetration repeatability, reproducibility and stability of the indenter. The whole assembly of the masses and the frame are held by another bigger frame that will provide us the two-steps force application that is needed in Rockwell scales and this bigger/outer frame is connected to a high accurate force measurement sensor to control the testing cycle.

The masses have high accuracy since they will be used in recalibration of the force sensor equipped onto the machine. The additional mass stacks that are also guided while seating on each other and the small frame constituting the preload for Superficial Rockwell scales are shown in Figure 6 a), force measurement device to which the whole mass stacks are mounted is given in Figure 6 b) and mounted mass stacks realizing force with high accuracy and repeatability is given in Figure 6 c).

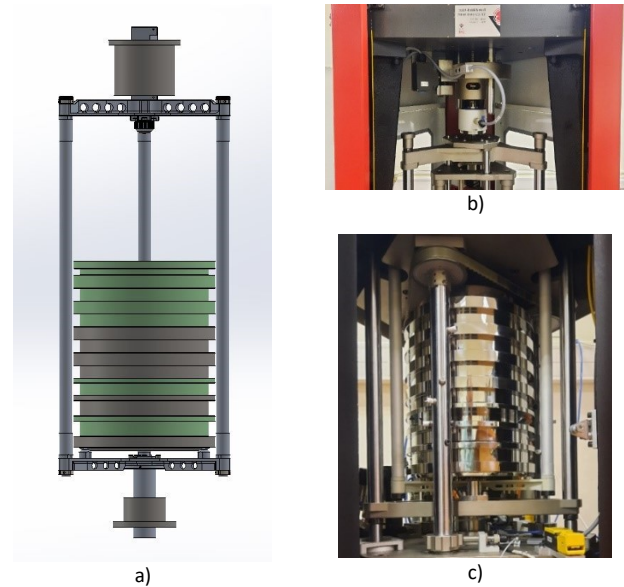


Figure 6. a) CAD of the force application system, b) force measurement device, c) mounted mass stacks of the Rockwell-Brinell-Vickers HSM.

3.3. Indentation Measurement System

For indentation depth measurement a high accurate and a contactless sensor, a laser interferometer system, is integrated onto the machine. The linear interferometer is placed on an adjustable plate and a corner cube is placed on and contacted to the indenter and fastened to the indenter. In this way the movement of the indenter is recorded by system software and depth vs. time graph is plotted.

3.4. Indentation Cycle

The testing cycle is realized with application of force with a predefined indenter approach speeds and force application rates with respect to time. To figure out the measurement cycle of the machine, Figure 6 b), is used and data from this device (F vs. t and F vs. h) is recorded and plotted. For the indenter speed measurement in all methods designed on this machine the laser system is used. The speed of the indenter as well as depth is measured by making use of the data taken from the laser interferometer. With the help of the speed information taken from the laser it is possible to characterize any test cycle for the material.

3.5. Automation/Control of the Machine

The machine is equipped with three servomotors, one force measurement sensor and one displacement measurement sensor as the main components for automation/control of the machine. The main servomotor (M1) is realizing the indentation by moving the force application system up-and-down, the secondary and tertiary servomotors (M2 and M3) are used to choose the relevant mass stacks to realize the relevant scale, the force transducer besides the measurement cycle is used to sense loading-unloading, parking etc. The machine is also equipped with enough limit switches, ambient conditions measurement sensors and air pressure control sensor for the air bearings.

4. PERFORMANCE

In this paper the design of the machine is explained in detail, both in terms of mechanical and automation with hardware and software regarding the measurement cycle and measurement

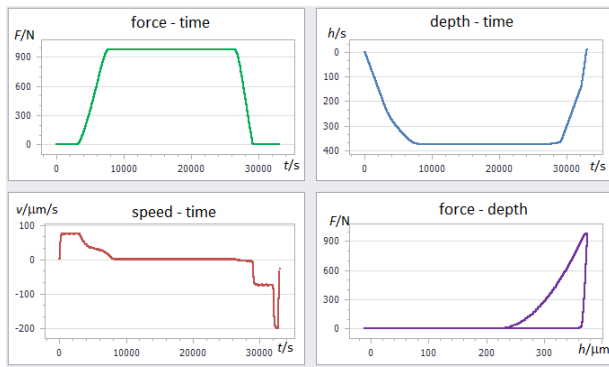


Figure 7. Measurement cycle realized for 100 kgf value.

principle. The most important property of this machine is the accuracy and stability in force application method and the design of the system to realize precise, repeatable and reproducible measurement cycle. Also accuracy in depth measurement, elimination of the compliance of the machine body as it is having a dead-weight force application capability is another contribution to accuracy not only in the conventional hardness measurement methods but also more important in IIT, i.e. Martens hardness.

4.1. Force and Time

The force uncertainty was managed with high accuracy of mass stacks and local gravitational acceleration that constitute force with an uncertainty value goes down to $2 \cdot 10^{-5}$ relative. The uncertainty for instantaneous of force measurement which is the uncertainty of force transducer goes down to $1 \cdot 10^{-4}$ relative. The time uncertainty in measurement cycle is observed to be less than 0.01 s, and realization of the cycle is less than 0.25 s uncertainty. The F vs. t , h vs. t , F vs. h together with the v vs. t (where v states for the speed of indenter) are all given in Figure 7.

Determination of zero-point, the reference point for Martens hardness measurement, in this application is realized with method 2 (the touch point determined from first increase of either the test force or contact stiffness by making use of high accuracy of force and depth of indentation measurement sensors). Zero-point determination data given in Table 1 is evaluated from Martens measurements made for performance test (Table 2 to Table 5). Five measurements were realized and the smallest values in terms of force and depth of indentation is recorded. Resolution of force measurement is 0.001 N (indicator resolution is 2000000 digit) and laser interferometer is 1 nm. The uncertainties are calculated with repeatability, reproducibility, resolution and approximate uncertainty of the measurement device. The zero-point values of the machine given in Table 1 is satisfactory compared with the requirements indicated in the ISO 14577-1 [9]. These results are for the preliminary Martens measurements and further studies are being performed for method 1 and for different testing cycles as well as hardness values. Measurement parameters in zero-point are as follows,

- F_t : touch force,
- h_t : touch depth,
- $h_{\text{max-mean}}$: mean value of the five h_{max} .

Table 1. Determination of zero point for different scales.

$F_{\text{max}} / \text{kgf}$	F_t / N	$h_t / \mu\text{m}$	U_{Ft} / N	$U_{ht} / \mu\text{m}$	$h_{\text{max-mean}}$	$U_{h_{\text{max-mean}}}$
3	0.127	0.528	0.006	0.030	24.676	0.164
20	0.385	1.523	0.033	0.216	67.434	0.360
100	0.863	2.490	0.176	0.384	153.396	0.430
150	0.810	1.374	0.161	0.327	187.548	0.328

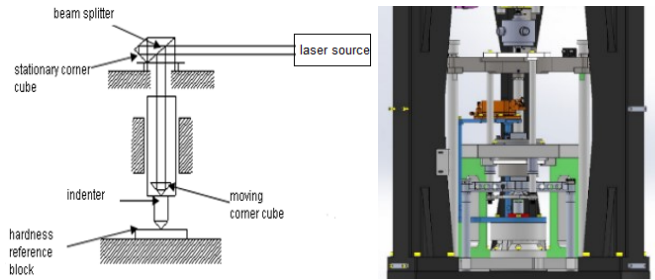


Figure 8. Laser interferometer system used for depth measurement.

4.2. Depth Measurement

In Rockwell scales as well as Martens Hardness depth measurement is realized with the laser with a resolution better than 10 nm and it is realized as schematically explained in Figure 8. The laser resolution is 1 nm but the stable value is (5 – 10) nm.

4.3. Indenter

IIT is constituted with different types of indenters made up of diamond or hard metal ball indenters. Martens hardness is mainly considered for diamond indenters of Vickers (square based pyramid) and Berkovich (triangular based pyramid). The scales realized in this machine are made with Vickers indenter that is used in Vickers hardness scales in accordance with ISO 6507-3 [8] as given in Figure 9.

Table 2. Martens hardness measurements with 3 kgf.

h / mm	$h_{\text{mean}} / \text{mm}$	MH / MPa	$MH_{\text{mean}} / \text{MPa}$	Rep / mm	$Rep / h \text{ in } \%$	Rep / MPa	$Rep / MH \text{ in } \%$	U_m / MPa
0.02471		1823.05						
0.02485		1802.57						
0.02457	0.02468	1843.89	1828.26	0.00016	0.65	23.76	1.30	24.64
0.02479		1811.31						
0.02446		1860.51						

Table 3. Martens hardness measurements with 20 kgf.

h / mm	$h_{\text{mean}} / \text{mm}$	MH / MPa	$MH_{\text{mean}} / \text{MPa}$	Rep / mm	$Rep / h \text{ in } \%$	Rep / MPa	$Rep / MH \text{ in } \%$	U_m / MPa
0.06728		1639.39						
0.06730		1638.41						
0.06774	0.06755	1617.20	1626.56	0.00026	0.38	12.31	0.76	16.29
0.06785		1611.96						
0.06756		1625.83						

Table 4. Martens hardness measurements with 100 kgf.

h / mm	$h_{\text{mean}} / \text{mm}$	MH / MPa	$MH_{\text{mean}} / \text{MPa}$	Rep / mm	$Rep / h \text{ in } \%$	Rep / MPa	$Rep / MH \text{ in } \%$	U_m / MPa
0.15296		1585.87						
0.15297		1585.66						
0.15355	0.15340	1573.71	1576.90	0.00042	0.27	8.67	0.55	11.85
0.15357		1573.30						
0.15393		1565.95						

Table 5. Martens hardness measurements with 150 kgf.

h / mm	$h_{\text{mean}} / \text{mm}$	MH / MPa	$MH_{\text{mean}} / \text{MPa}$	Rep / mm	$Rep / h \text{ in } \%$	Rep / MPa	$Rep / MH \text{ in } \%$	U_m / MPa
0.18744		1584.13						
0.18741		1584.64						
0.18715	0.18755	1589.04	1582.32	0.00032	0.17	5.42	0.34	7.81
0.18778		1578.40						
0.18796		1575.37						

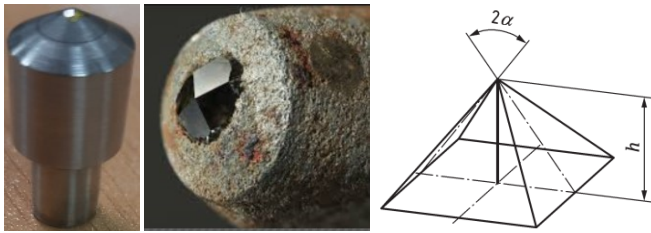


Figure 9. Vickers indenter used in measurement.

4.4. Martens Hardness Measurements

Martens hardness for the macro range (3 kgf – 150 kgf range) is realized in this machine and indentations realized under forces of 3 kgf, 20 kgf, 100 kgf and 150 kgf are measured. Mainly the repeatability of the machine together with a simple approach of uncertainty was evaluated for 150 HV100 Vickers hardness reference block with S/N 211-466 in terms of the percent of the indentation depth, b , and Martens hardness, HM , and uncertainty values in terms of MPa of HM . For every force value the same approach speeds and dwell times are used. In the uncertainty calculation the touch and maximum force and depth parameters were taken into consideration together with the uniformity of the reference block and repeatability of the machine. The results are considered to be significant particularly from repeatability point of view in terms of depth as well as Martens hardness measurement values. The results are given in Table 2 to Table 5.

In these measurements repeatability (R_{ep}) and uncertainty (U) values of indentation depth and Martens hardness (HM) are calculated as standard deviation of measurement values, repeatability of both parameters were calculated in terms of percent of the mean values and finally uncertainty of the measurement values. These values include effect of the machine body (shock, vibration), compliance, indenter, start point of force application and depth measurement, block and anvil interaction, homogeneity of the hardness reference block, repeatability of the measurement cycle (times, speeds, etc.). The preliminary results seem to be satisfactory in terms of repeatability and realization of the Martens hardness test cycle.

5. CONCLUSION

In this paper the design of the Rockwell-Brinell-Vickers hardness standard machine with dead-weight force application system and laser interferometer depth measurement is explained in detail, both in terms of mechanical parts and automation with hardware and software regarding realization of the conventional hardness scales as well as IIT, i.e. Martens Hardness. The most important properties of this machine are the high accuracy and stability in force application and depth of indentation measurement and the design of the system to realize precise, accurate and repeatable measurement cycle of Martens hardness with very good compliance.

The force uncertainty goes down to $2 \cdot 10^{-5}$ relative as it was provided with high accurate mass and local gravity values where ambient conditions were taken into consideration. The time uncertainty in measurement cycle is much less than 0.01 s for instantaneous force measurement and realization of the cycle with an uncertainty of 0.25 s. The force transducer used for realization of the cycle has metrological properties better than class 00 of ISO 376 [13] together with an indicator having 0.0005 accuracy class, carrier frequency of 225 Hz, 2,000,000 digit resolution and data acquisition of 1 kHz in the software of the

machine. All measurands (force, depth and time) are realized with the highest possible accuracy for realization of Martens hardness.

The preliminary performance of the machine in Martens hardness was evaluated for 4 different force levels of 3 kgf, 20 kgf, 100 kgf and 150 kgf by making use of a good quality of hardness reference block with 150 HV100 hardness scale and level. The repeatability of measurements including the effects of

- machine parameters such as
 - zero (start) values of force, depth and time,
 - compliance,
 - mechanical effects like vibration, shock, interaction of the block with the anvil, etc.,
- indenter, and,
- hardness reference block uniformity

was evaluated satisfactory, so it can be used for reference block calibration in Martens hardness scale in accordance with the relevant ISO hardness standard [14] for the whole force range of the machine that is 3 kgf – 150 kgf. As a near future planning the IIT as well as Martens hardness measurements will be extended to micro range with a new design of a reference system.

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