POLITECNICO DI TORINO

MASTER'S DEGREE THESIS

TESI DI LAUREA MAGISTRALE

Metrological characterisation of a new primary Micro-Hardness machine

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1. INTRODUCTION

1.1 INRIM

Figure 1 – INRiM Logo

The National Institute of Metrological Research (INRiM) is a national public organization established in 2006. INRiM carries out and promotes research in the field of metrology, develops the most advanced samples and measurement methods and related technologies both hardware and software, through which it performs the functions of a primary metrological institute. They follow all the standards and agreements concurrent with their fellow institutes located all over the world. They are also delegated to produce and maintains units of measurement. Traceability is a key for them, and they strive to maintain the traceability of all the measurements to keep all units in check. [1]

INRiM has a high position in the European metrological institutes: since its ideally located near the National Research System, it's having the requirement to continually compete with other metrological institutes and push to maintain uniformity all around the world as its fundamental goal through rigorous scientific research and adapting scientific breakthroughs in all related areas of national measurement standards and related Calibration and Measurement Capabilities (CMC).

The history of INRiM actually started in the year 2016, when it took its new form and activities that were previously undertaken by IEN and the Gustavo Colonnetti Institute of Metrology, which are some of the oldest institutes in the field founded in Torino.[2]

1.2 ISO

Figure 2 - ISO Logo

Delegates from 25 nations, representing the ISA and UNSCC, met in London in October 1946 and decided to collaborate to form the International Organization for Standardization. The company started conducting business formally on February 23, 1947. It is a non-governmental organization whose mission is to create global standards across all industries, with the exception of electrical and electronic engineering. With its main office in Geneva, Switzerland, it now has 167 members in total. The ISO's three official languages are English, French, and Russian. [2]

The International Federation of the National Standardizing Associations (ISA), which had a heavy emphasis on mechanical engineering, was the organization that started it all back in 1926. After the war, the newly established United Nations Standards Coordinating Committee (UNSCC) contacted the ISA with a request to establish a new international standards organization. The ISA had been suspended in 1942 due to World War II.

Different national standards bodies are members of the International Organization for Standardization, an impartial organization. Members that now represent ISO in their nation will only have one member as of 2022.

Technical committees' primary responsibility is to create International Standards. Technical committees' adopted drafts of international standards are distributed to member bodies for voting. An international standard can only be published with the consent of at least 75% of the member bodies voting.
Over 24,261 standards have been created by ISO, ranging from those for manufactured goods and technology to those for food safety, agriculture, and healthcare. There are 804 technical committees and subcommittees within ISO that work on developing standards.

Each year, members gather at a General Assembly to talk about ISO's strategic goals. The organization's central secretariat, which is situated in Geneva, manages operations.
More than 250 technical committees create the ISO standards, and they are under the control of the technical management board.

1.3 HARDNESS

1.3.1 WHAT IS THE DEFINITION OF HARDNESS TESTING?

Hardness testing is defined as "a test to measure the resistance of a substance to permanent distortion by penetration of another harder material." Hardness, on the other hand, is not a basic feature of a material. As a result, while generating hardness test findings, always examine the quantitative value in relation to: The indenter's supplied load, A distinct loading time profile and a distinct load duration [3]

Hardness testing is a non-destructive test procedure that includes applying a steady force to a metal surface using a rounded or pointed item under controlled conditions to form an indentation. This is then measured to establish the material's hardness.

The use of hardness testing allows you to analyze the qualities of a material, such as strength, ductility, and wear resistance, and so determine if a material or material treatment is appropriate for the purpose you require.

Hardness testing, like tensile testing, is a useful predictor of mechanical qualities inside materials. It indicates a material's capacity to resist indentation and, hence, strength, wear resistance, and toughness.
The method entails applying a steady load to a rounded or pointed indenter in order to generate an indentation in the material surface. The depth of penetration is then tested to determine hardness. Hardness affects a wide range of physical characteristics, such as how much the metal will wear, scratch, or withstand stress.

There are various hardness test techniques available, each with their own set of criteria and benefits, but selecting the proper hardness test is typically dictated by the size and geometry of the sample, the area to be evaluated, and the simplicity of application.

1.3.2 HOW DO HARDNESS TESTS WORK?

A hardness test is normally conducted by pressing a precisely dimensioned and loaded item (indenter) into the surface of the material being tested. The hardness is assessed by measuring the depth of indenter penetration or the size of the indenter impression.
Rockwell, Instrumented Indentation Testing, and Ball Indentation are hardness tests that assess the depth of indenter penetration. Vickers, Knoop, and Brinell hardness tests are used to determine the size of the imprint created by the indenter.

1.3.3 SELECTING THE BEST HARDNESS TEST METHOD

The hardness test you pick should be dictated by the microstructure of the material you are testing, such as its homogeneity, as well as the kind of material, the size of the component, and its condition.

The material under the indent in all hardness tests should be representative of the whole microstructure (unless you attempting to ascertain the different constituents in the microstructure). As a result, if a microstructure is highly coarse and heterogeneous, a bigger imprint is required than for a homogeneous material. [4]

There are four basic hardness tests, each with its own set of advantages and disadvantages. There are many standards for these tests that specify the techniques and implementation of the hardness test.

Important factors to consider when choosing a hardness test technique include:

- The type of material to be hardness tested
- Whether compliance with a standard is required
- The approximate hardness of the material
- The homogeneity/heterogeneity of the material
- The size of the part
- Whether mounting is necessary
- The number of samples to be tested
- The required accuracy of the result

The different types of hardness test that are followed are as follows:

- Rockwell hardness testing
- Brinell hardness testing
- Knoop hardness testing
- Vickers hardness testing

1.4 DESIGNATION OF HARDNESS NUMBER

Vickers hardness, HV, is designated as shown

252 HV 25/20

252 – Vickers hardness value
HV – Hardness symbol
25 – Approximate test force value in kgf
20 – Duration of test for (Only to be mentioned when the time duration of force application isn't with the test range of 10 to 15 seconds) [5]

Here kgf is kilograms of force where 1 kgf = 9.80665 N

1.5 HOW TO ENSURE ACCURACY AND REPEATABILITY IN HARDNESS TESTING

The proper implementation of hardness testing necessitates careful planning and execution. However, after you've mastered the fundamentals, most hardness tests are accurate and repeatable.

Several factors impact the outcome of hardness testing. As a general rule, the lower the load used in the hardness test, the more elements that must be regulated to guarantee an appropriate hardness test conclusion.

Here are a few of the most critical elements to consider while doing a hardness test to get an accurate result. Light, filth, vibrations, temperature, and humidity should all be kept under control. The tester and stage should be clamped or held in a holder or anvil, and the sample should be clamped or held in a holder or anvil. The indenter should be perpendicular to the surface being examined. [6]

When utilizing Vickers, Knoop, or Brinell, the illumination parameters should be kept consistent during the test. When changing the indenter or objective lens, the tester should be recalibrated/verified.

1.6 SURFACE PREPARATION REQUIREMENTS FOR HARDNESS TESTING

The surface of metallic or other materials must be prepared before hardness testing. The needed surface condition is determined by the kind of test and load. In general, the quality of surface preparation has a direct influence on the hardness test result, therefore before choosing an inferior surface preparation, evaluate the trade-off between surface quality and test result fluctuation.

When it comes to microvickers testing, there are unique protocols in place. Micro hardness testing necessitates a polished or electropolished surface due to the smaller stresses applied during hardness testing. The borders/corners of an optically assessed impression must be clearly apparent. This procedure can be carried out physically, chemically, or electrochemically. Its also important to keep the effects of temperature in mind.

1.7 DEFORMATIONS

Deformations can be introduced via cutting and grinding. Depending on the hardness test load, they must be eliminated by polishing down to 6.0, 3.0, or 1.0 μm. For minor loads, the surface must be fully free of deformations, and the specimens must be polished with oxide or electrolytic polishing to achieve this. You should also keep in mind that soft and/or ductile materials are more sensitive (i.e., for HV less than 120-150). [3]

1.8 INDENT SPACING

The indentation will distort the surrounding material and change its characteristics during hardness testing. The guidelines provide a specified distance between repeated indentations to minimize misunderstanding of perceived hardness.
Steel, copper, and copper alloys require at least three diagonal widths between indents.
In the case of lead, zinc, aluminium, and tin, the distance between indents must be at least six diagonal widths. [7]

1.9 TRACEABILITY

Traceability refers to a standard's value when it can be linked to specified references (national or international standards) via an unbroken chain of comparisons, all with declared uncertainties (ISO). To put it simply, measurement traceability is a means of guaranteeing that a measurement takes into account all uncertainties and is an accurate depiction of the thing being measured. This approach is based on testing a measurement against a higher calibration reference standard. One widespread misperception about traceability is that the measuring equipment is traceable; however, only the measurement result or standard value is traceable. [8]

1.9.1 WHY IS MEASUREMENT TRACEABILITY IMPORTANT?

Simply defined, calibration to a traceable standard certifies the accuracy of a sensor as well as any related uncertainties. Measurement Traceability is critical for assuring the accuracy of a sensor to both a consumer and a manufacturer for a specific procedure. Establishing that a sensor's accuracy can be traced to a higher standard provides manufacturers with credibility, certifying the correctness of their data to their consumers. Traceability assures that a manufactured item or calibration is accurate and meets the application's standards.

Prior to usage, all equipment used for assessment, including those for subsidiary measures that affect the validity of the results, must be calibrated. The laboratory must have a well-established calibration program and technique.

The measurement standards and measuring instruments are traceable to the International System of Units (SI) through an unbroken chain of calibrations or comparisons linking them to relevant primary standards of the SI units of measurement.

Traceability of findings must be ensured when employing external calibration services by obtaining calibration services from laboratories that can demonstrate competency, measuring capacity, and traceability.

Metrological Traceability is commonly described as "the property of a measurement result by which the result can be traced to a national or worldwide reference standard through a recorded, unbroken chain of calibrations, each contributing to the measurement uncertainty of the measurement."

Traceability means that the outcome of each measurement can be traced back to a calibrated measurement standard with an associated measurement uncertainty.

The term "unbroken chain" refers to the need that the measurement standard be calibrated by a highly accurate measurement standard, and so on until the measurement or value of a standard can be traced back to national or worldwide reference standards. This 'Calibration Hierarchy' between measurement standards must be recorded, usually with a Calibration Certificate. [9]

1.9.2 TRACEABILITY PYRAMID

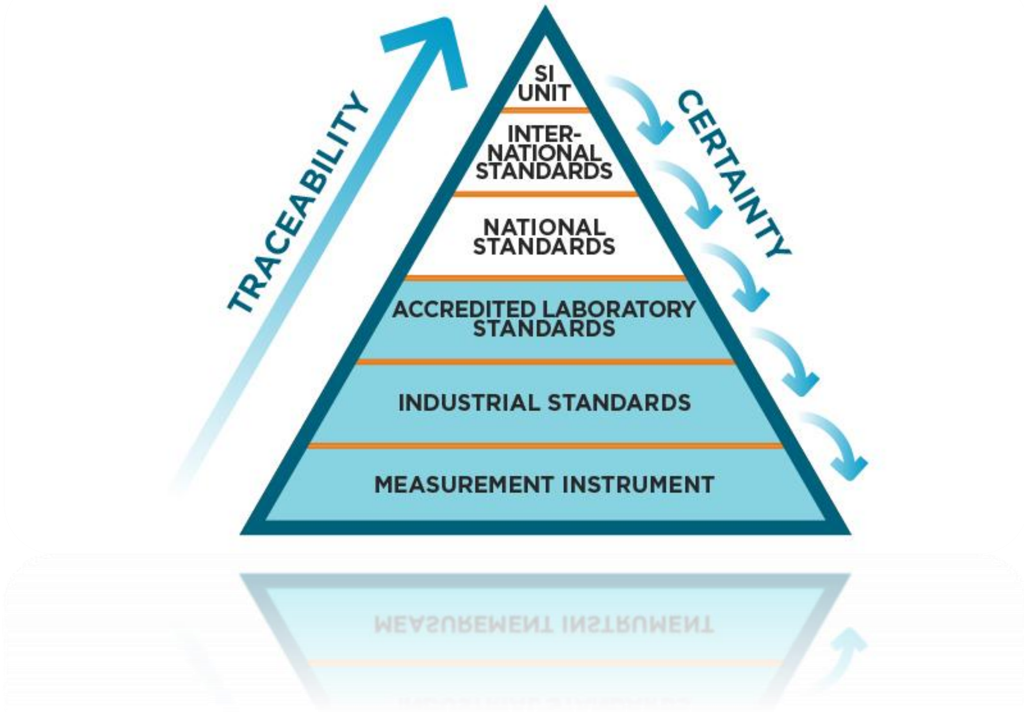


Figure 3 - Traceability Pyramid

The levels in between contain external calibration laboratories or National Metrology Institutes that check their standards directly with worldwide reference standards to ensure the maximum accuracy of their calibrations.

The apex of the pyramid has the best accuracy and the lowest measurement uncertainty. As the measurement errors are compounded, each level descending from the top of the pyramid loses a certain degree of precision. [10]

1.9.3 MISCONCEPTIONS OF MEASUREMENT TRACEABILITY

Only the measured value or the designated reference value of a standard are subject to measurement traceability. One widespread mistake is that the measuring standard is traceable in and of itself. Only the result measured by the standards may be traced, not the measurement standard. Similarly, measurement traceability cannot be attributed to any single calibration report, national or international standards laboratory, or technique.

Another common fallacy is that a traceable measurement may be utilized for any purpose. Measurement tracing assures that a measurement accurately represents the value being measured. Individual specifications and uncertainty levels for each measurement must be verified to verify they are appropriate for the intended purpose. [11]

1.9.4 TRACEABILITY REQUIREMENTS

Traceability of measurements is a critical necessity for every company. Measurement data may be used to make vital decisions that have a substantial influence on people's health and safety, such as in the aviation or medical industries. Some of the stated flaws in regulatory audits is errors or omissions in traceability documentation. To guarantee that the standards for Measurement Record keeping are developed and maintained, a business must have an internal measurement accountability system in place. In order for a measurement in your business to be considered traceable, it must:

- Schedule Regular Calibrations - Each measuring standard in an organization must be calibrated at regular intervals. If the calibration for any standard in the traceability chain expires, the traceability is broken.

- Use Competent Laboratories – The calibration of measurement standards needs to be conducted by an accredited calibration laboratory or by a National Metrology Institute. This ensures that the laboratory is competent to perform the calibrations and properly calculate the measurement uncertainty values.

- It is critical that the calibration certificates for equipment that are sent to an external calibration laboratory are reviewed to ensure that the proper information is included on the certificate. The following items must be included on the calibration certificate to state that the measurement value is traceable:

- Reference Standards Used – The calibration certificate must list the reference standards that the external calibration laboratory used in the calibration. Those standards provide the link to the chain of comparisons that establishes a connection to the national or international reference standards.

- Documented Measurement Uncertainty – Every quantitative measurement needs to include the measurement uncertainty value. If the uncertainty information is missing from the calibration certificate, you cannot claim that the measurement is traceable.

- Documented Measurement Procedure – The calibration certificate must list the procedure that was used for the calibration. The calibration must be completed according to a written procedure that is a part of the external calibration laboratory's quality system.

  [12] [13]

1.10 CCM - WORKING GROUP ON HARDNESS

Figure 4 - CCM BIPM Logo

In 1999, at the 88th Session of the International Committee of Weights and Measures (CIPM), Dr Kozo lizuka, President of the Consultative Committee for Mass and Related Quantities (CM), stated "Although the definition of hardness scales is certainly conventional in the sense of the use of arbitrarily chosen formula, the testing method is defined by a combination of physical quantities expressed by SI units; the standard of hardness is established and maintained in most of NMls and the traceability to the standard of MIs is demanded in industry and elsewhere." The subsequent discussions led to the realization that hardness standards should be included in the key comparison database (KCDB) for the mutual recognition arrangement (MRA) and thus, a full Working Group on Hardness (CCM-WGH) was established in the framework of the CCM. [13]

Influence parameters may be explored, and global hardness testing standards can be revised, recommended, and accepted for usage with NMI to remove measurement inconsistencies at the highest levels on a national scale. Because global agreement is necessary, the CCM-WGH collaborates closely with ISO/TC 164/SC 3 to ensure proper dissemination of the hardness scales. The hardness test parameters in the CCM-WGH standards are provided with precise values, rather than ranges of permitted limits, as this test method stipulates. As appropriate, the stated values of the CCM-WGH definitions have been recognized as the values to be used in this document.

1.11 KOHLER ILLUMINATION

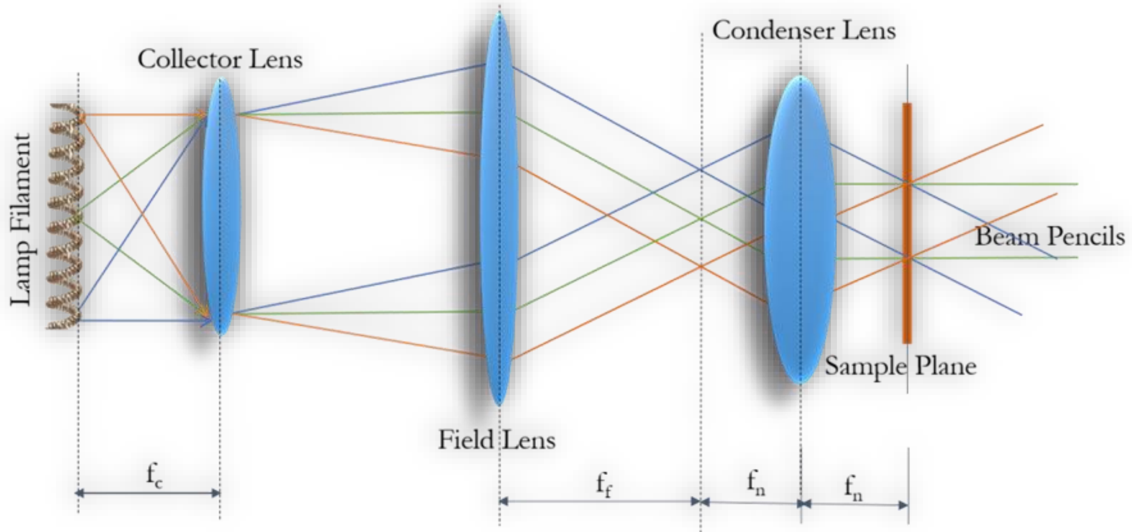


Figure 5 - Kohler system Internals

Köhler illumination is a method of specimen lighting used in transmitted and reflected light optical microscopy (trans- and epi-illuminated). Köhler lighting guarantees that an image of the illumination source (for example, a halogen lamp filament) is not evident in the final image. In current scientific light microscopy, Köhler illumination is the most often used approach for sample lighting. It necessitates the use of extra optical components, which are more costly and may not be present in simpler light microscopes. [14]

1.12 NORMATIVE REFERENCE

- ISO 376:2011(E) Calibration of force proving instruments used for the verification of uniaxial testing machines
- ISO 6507 – 1:2018(E) Metallic materials – Vickers Hardness test: Part 1 – Test method
- ISO 6507 – 2:2018(E) Metallic materials – Vickers Hardness test: Part 2 – Verification and calibration of testing machines
- ISO 6507 – 3:2018(E) Metallic materials – Vickers Hardness test: Part 3 – calibration of reference blocks
- ISO 4287
- ISO 1463
- JCGM100:2008

2. EXPERIMENTATION

2.1 CALIBRATION OF THE TRANSDUCER (20N)

This is done in keeping with the ISO 376 standard
METALLIC MATERIALS CALIBRATION OF FORCE PROVING INSTRUMENTS USED FOR THE VERIFICATION OF UNIAXIAL TESTING MACHINES [15]

2.1.1 INTRODUCTION

The ISO 164/SC 1 group that works with the uncertainty of force-proving instruments has developed these procedures.

This method allows the calibration to be performed in a couple of ways:

- With reversible measurement for force-proving instruments which will be used with ascending and descending forces.
- The other methods will be where we would not make use of reversible measurement for force-proving instruments, thus we will only use it with increasing forces.

We will be making use of the first method thus there will be no requirement for the creep test to be performed. [15]

2.1.2 SCOPE

This International Standard Organization describes us the process for calibrating force transducer that we will be using for uniaxial loads in the increasing and decreasing manner. This Standard will also apply to force transducers in which the force is determined by taking into account the Elastic deformations of the loaded part as well.

The following document serves as the normative reference for this document: ISO/IEC 17025:2005

Calibration with a specific force will be applied to the force transducer and the results from the indicating instrument connected to the transducer will be noted. The force transducer will have a ball joint in the centre so as to ensure that axial force is always axial, whether in compression or tension. [9]

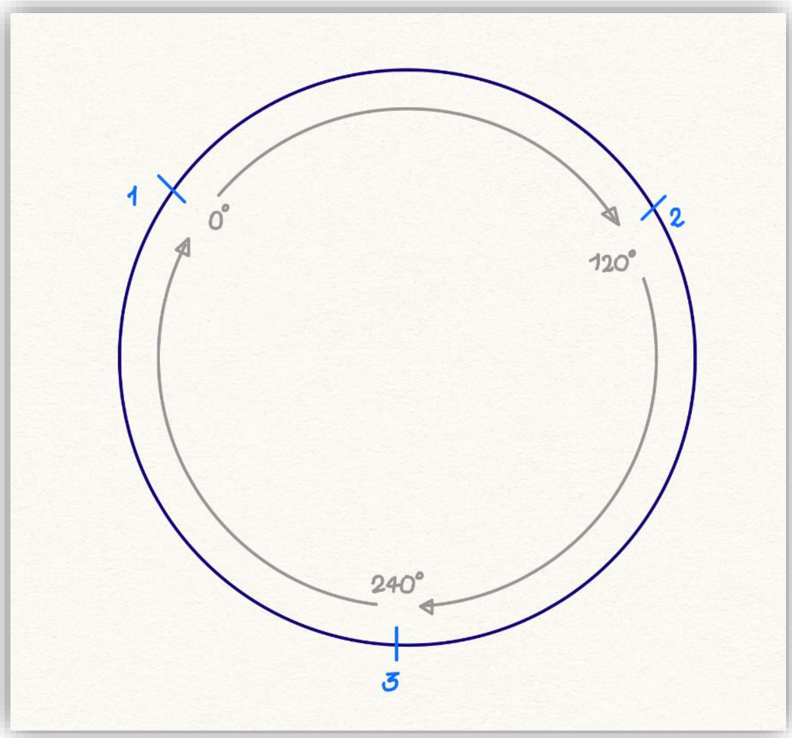
The minimum force must be calculated as F_f*0.02 = Approximately 41 grams or .40 N

2.1.3 CALIBRATION OF THE INSTRUMENT

2.1.3.1 PROCEDURE TO CALIBRATE

The procedure we will follow is the ISO 376:2011 (E) which is the standard for the force calibration of (Uniaxial testing machines)

Also, the preliminary test, overload test, verification related to the force application and variable voltage test must be carried out in order to run the test smoothly with minimal uncertainties.
We can also make an interpolation curve since the number of forces that we are using would be more than eight. [15]



[16]

Figure 6 - Positions for the Force Transducer

2.1.3.2 PRELOADING

Before the loads for the calibrations are put into the apparatus, in each direction (tension or compression), the all the loadings must be put on the load housing three times this is called as the preload test. The time required to apply these loads must be between 60 seconds and 90 seconds.

I. Load the transducer with the maximum load (20N) in our case
II. Leave the set up untouched for 90 seconds
III. Release or remove the weights
IV. Repeat the procedure three times with the same weights
V. Tabulate the recorded data
VI. As the direction is changed (Rotated) the procedure must be repeated.

Figure 7 - 20N Force Transducer

2.1.3.3 MEASUREMENT PROCEDURE

The transduced will be calibrated by applying two sets of calibration forces in an increasing/decreasing manner, with increasing values initially and then lowering values. We must rotate the transducer on its axis to positions equally dispersed throughout 360° (i.e., 0°, 120°, 240°) between each test cycle.

I. Perform the preload testing
II. Test for creep after the preload testing
III. Load a force of 2 N and wait for 30 seconds then tabulate the acquired data.
IV. Incrementally add 2 N of force (In the form of weights) and perform the test
V. Repeat till you reach 20 N of total force and reverse the process.
VI. Remove the weights (2 N every iteration) and tabulate the acquired data
VII. Repeat the procedure till you reach a zero force.
VIII. Once this series of loads have been measured rotate the transducer being measure by 120 degrees along the same axis.
IX. Wait for 3 minutes
X. Repeat from steps 3 to 8 and rotate the transducer again for a total of 240 degrees from the initial position.
XI. Repeat from 3 to 8 again and tabulate the data for calculations.

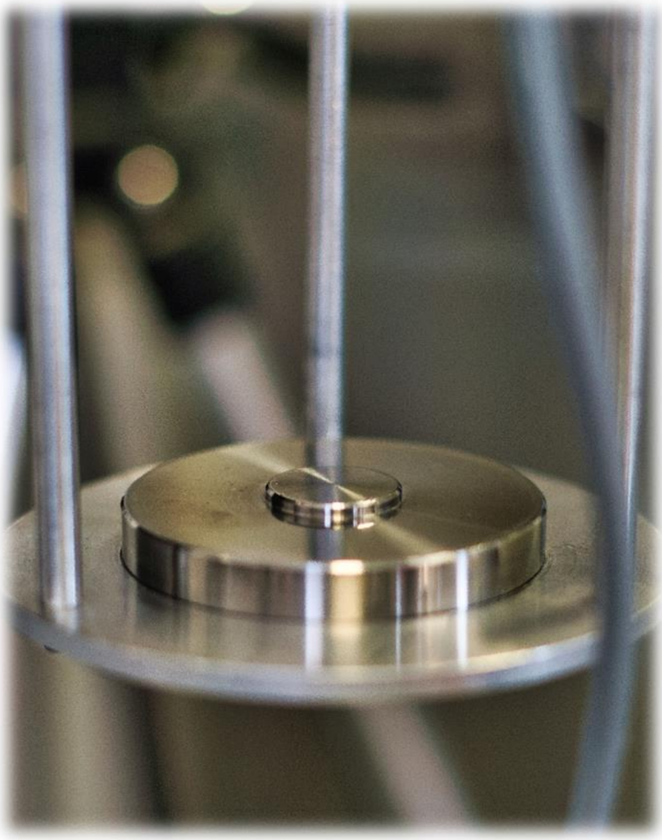


Figure 9 - Single Load

Figure 8 - Total Load

We have 10 forces that are distributed evenly over the range of the calibration. The interpolation curve is determined from the average values of the deflections with rotation,
This procedure determines a combined value of hysteresis of the device and of the calibration machine.

2.1.3.4 CREEP TEST

The transducer is loaded in an incremental direction. We then wait for 30 seconds and take the reading and wait for another 300 seconds to take a second reading and check for the difference and apply the formula to account for the creep characteristics. [16]

If creep test is performed. After preloading we follow the calibration procedure. After which, the calibration certificate will provide the following information:

I. When the creep measurement is performed (after preloading, after the last measurement series, etc.)
II. The time period for which the force was applied before the removal.
III. The method of creep measurement (creep at maximum force or after force removal).

2.1.3.5 PRECAUTIONS

I. The values linked to zero force were recorded after a minimum of 30 seconds after the force was withdrawn.
II. There was a 3-minute pause between measurements. The zero signal was recorded before beginning the calibration of the electrical force-proving equipment.

III. The time interval between successive loads was kept as uniform as possible, and no reading were taken within 30 s of the start of the force change to make sure that the setup is stable.

IV. The calibration procedure was performed at a stable temperature to within ±1 °C. This temperature was in the given range of 18 °C to 28 °C. Adequate time was allowed for the force transducer to attain a stable temperature.

V. When it is known that the force transducer is not temperature-compensated, care should be taken to ensure that temperature variations do not affect the calibration which was not the case in our experimental setup.

VI. Precautions were taken to prevent the instrument from experiencing forces greater than the maximum calibration force.

VII. Instruments classified for specific forces were used only for these forces. [15]

2.1.3.6 DETERMINATION OF DEFLECTION

The difference between a reading taken while under force and one taken while not under force is referred to as a deflection. This definition of deflection encompasses both output readings in length units and electrical units. [15]

Figure 10 AEP MP 10 PLUS Force Transducer signal reader

Figure 11 - Polytec OFV Sensor Head

2.1.3.7 ASSESSMENT OF THE FORCE TRANSDUCER

I. The following equation is used to compute relative reproducibility and repeatability errors for all calibration forces when the force-proving equipment is rotating: [15]

$$b = \left|\frac{X_{max} - X_{min}}{X_r}\right| \times 100$$

Equation 1

where the value of $X_r = \frac{X_1 + X_2 + X_3}{3}$

Equation 2

II. Relative interpolation error,
The deflection $X\ r$ as a function of the calibrating force is used to calculate the error

$$f_c = \frac{X_r - X_a}{X_a} \times 100$$

Equation 3

III. Relative reversibility error,
At each calibration, the relative reversibility error is calculated by performing a verification with rising forces and then with decreasing forces.

$$v = \left|\frac{X_3 - X_2}{X_2}\right| \times 100$$

Equation 4

IV. Relative creep error,
Here we calculate the difference in outputs i_{30} obtained at 30 seconds and i_{300} obtained 300 seconds after the application of the maximum calibration force and we express this difference as a percentage of maximum deflection:

$$c = \left|\frac{i_{300} - i_{30}}{X_N}\right| \times 100$$

Equation 5

2.1.4 CLASSIFICATION & CERTIFICATION

2.1.4.1 CATEGORISATION OF THE FORCE TRANSDUCER

According to the ISO 376:2011 (E) standards, Instruments for proving force fall into four kinds. All force testing devices must adhere to the requirement that the instrument cover at least the range. 50% to 100% of the loads that are being measures i.e., F_n. [15]

For our scenario we come under:

Case D: For instruments classified for interpolation and incremental and or decremental loading, the criteria which shall be considered are:

- I. The relative reproducibility and repeatability errors.
- II. The relative interpolation errors.
- III. The relative reversibility errors.
- IV. Although Creep is not a requirement, we have considered it as well.

CLASS	RELATIVE ERROR OF THE FORCE-PROVING INSTRUMENT %						EXPANDED UNCERTAINTY OF APPLIED CALIBRATION FORCE (95 % LEVEL OF CONFIDENCE) %
	b	b'	f_c	f_0	v	c	
00	0.05	0.025	±0.025	±0.012	0.07	0.025	±0.01
0.5	0.10	0.05	±0.05	±0.025	0.15	0.05	±0.02
1	0.20	0.10	±0.10	±0.050	0.30	0.10	±0.05
2	0.40	0.20	±0.20	±0.10	0.50	0.20	±0.10

Table 1

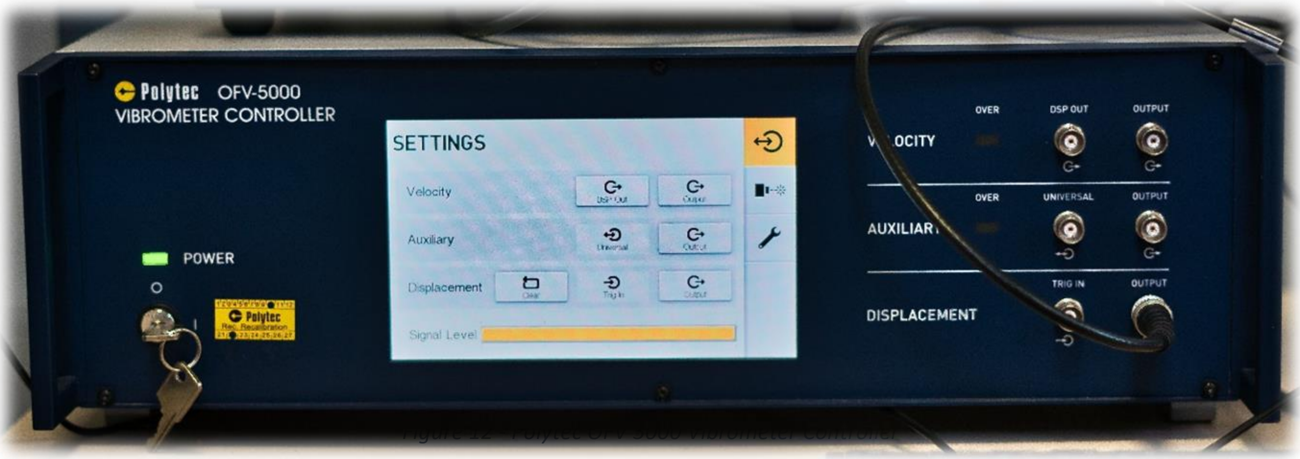


Figure 12 - Polytec OFV 5000 Vibrometer Controller

2.1.4.2 CERTIFICATION AND VALIDITY

The calibration authority will create a certificate after the force-transducer equipment meets the requirements of the International Standard, in accordance with ISO/IEC 17025, stating the following information: [9] [15]

I. We made use of a 20 Newtons Force transducer with a resolution of 2 mv/V that was made in Germany

II. The mode of force application included both tension and compression.

III. The instrument is in accordance with the requirements of preliminary tests.

IV. Our transducer falls under the class 1 (as shown from the results below) and the range (or forces) of validity is from 0 to 20 N. (Refer table 1 above)

V. The loading direction was incremental and decremental.

VI. The date of the calibration was on the 10th of December 2021.

VII. The temperature of 23°c was noted in the room on the day of the calibration.

VIII. The relative creep error is as given below.

Figure 13 - Setup for ISO 376

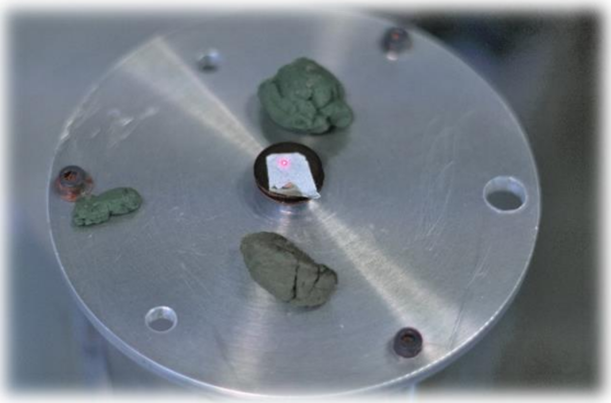


Figure 14 - Laser with reflective paper for accurate readings on the transducer balance plate for uniaxial loading

The instrument's verification and certification are only good for a maximum of 26 months, and it needs to be recalibrated if it experiences overload that is more than the test overload or needs to be repaired.

MEASUREMENT	SYMBOL	VALUE	UNITS
AVERAGE VALUE OF DEFLECTIONS	X_r	1.998	mV/V
MINIMUM DEFLECTIONS	X_{min}	0.200	mV/V
MAXIMUM DEFLECTIONS	X_{max}	0.198	mV/V
RELATIVE REPRODUCIBILITY ERROR WITH ROTATION	b	0.0953	%
RELATIVE INTERPOLATION ERROR	f_c	-0.022	%
RELATIVE REVERSIBILITY ERROR	v	0.094	%
RELATIVE CREEP ERROR	c	0.131	%

Table 2

We can determine that we fall under class 1 of transducers based on the values that were obtained from the test and tabulated in table 2. Keeping this in check we can proceed to determine the uncertainties of the next standard which is ISO 6507-1 2018.

2.2 METALLIC MATERIALS – VICKERS HARDNESS TEST PART 1: TEST METHOD

This is done according to the ISO 6507-1:2018 (E) standard [17]

2.2.1 SCOPE

The three test ranges for the Vickers hardness test method for metallic materials and cemented carbides, including hard metals, are listed below.

Ranges of test force, *F* N	**Hardness symbol**	**Designation**
$F \geq 49.03$	≥HV 5	Vickers hardness test
$1.961 \leq F < 49.03$	HV 0.2 to <HV 5	Low-force Vickers hardness test
$0.009\,807 \leq F < 1.961$	HV 0.001 to <HV 0.2	Vickers microhardness test

Table 3

Smaller indentations are not covered by this standard; hence the indentation of the diagonals must be between 0.020 mm and 1.400 mm. Due to the imprecise tip geometry and inadequate optical measurement equipment, there would be significant uncertainties. Recently, this test approach has also been used to organic and metallic coatings with minimum thicknesses of 0.030 mm.

2.2.2 INDENTER AND INDENTATION

A diamond indenter is used which is shaped like a right pyramid with a square base at a specified angle between opposite faces at the vertex, which is forced onto the surface of the test piece followed by the measurement of the diagonals formed by the indentations after removal of the test force.

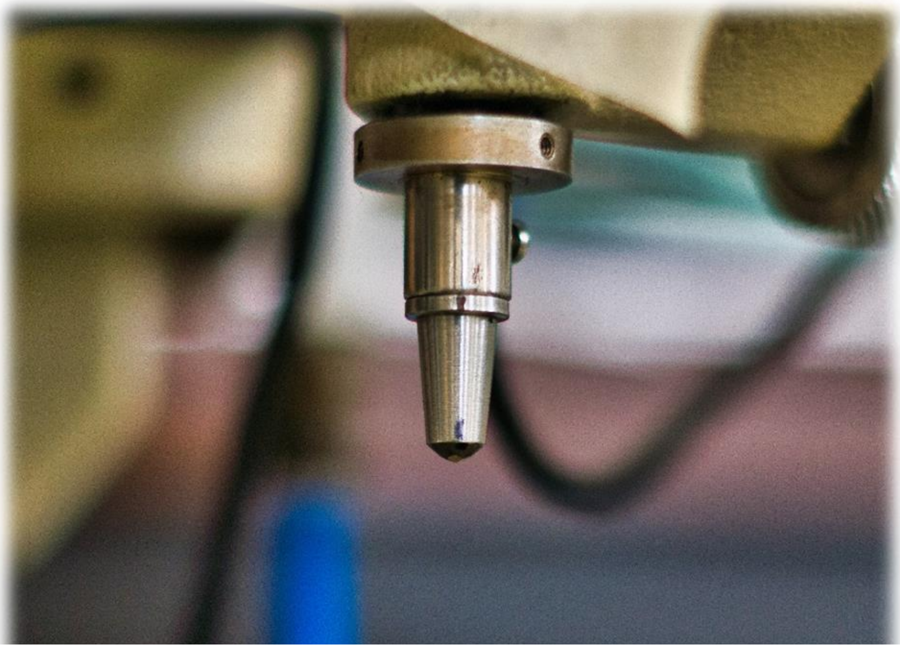


Figure 15 - Diamond Indenter

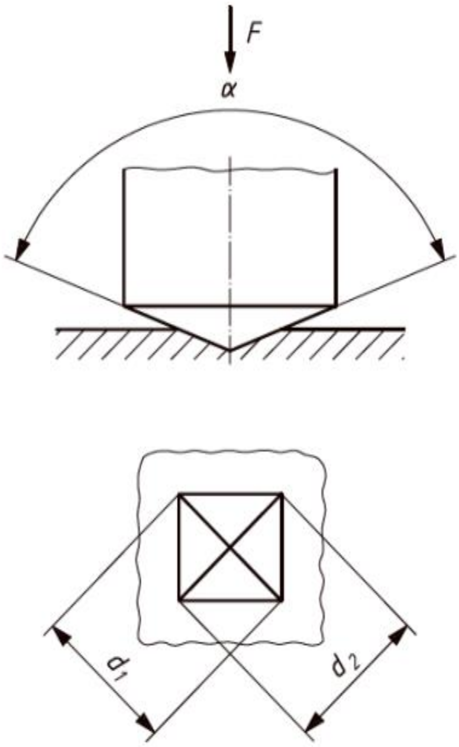


Figure 16 - Representation of indentation and its parameters

The Vickers hardness value is proportional to the quotient obtained by dividing the test force by the area of the sloped surface of indentation, which forms the pyramid. Where the diagonals are represented by d_1 and d_2. [17]

Where the formula for Hardness value HV is as follows:

$$= \frac{1}{g_n} \times \frac{F}{d^2 \Big/ \left(2\sin\frac{\alpha}{2}\right)}$$

Equation 6

Which for a nominal angle of 136° will be approximately equal to:

$$\approx 0.1891 \times \frac{F}{d^2}$$

Equation 7

The diamond indenter must be in accordance with the shape and size specified in ISO 6507 – 2 [18]

2.2.3 TESTING AND MEASURING SYSTEMS

According to ISO 6507-2, the testing apparatus must be able to replicate the predefined test forces within the allowed range.

The diagonal measurement system would also be covered by the ISO standard. There should be enough magnifications to allow the diagonal to be increased to more than 25% but less than 75% of the largest optical field of vision. Near the field of view's edge, many objective lenses become nonlinear. [18]

DIAGONAL LENGTH, d mm	RESOLUTION OF THE MEASURING SYSTEM
$0.020 \leq D < 0.080$	0.000 4 mm
$0.080 \leq D \leq 1.400$	0.5 % of *d*

Table 4

A diagonal measuring system that uses a camera for measurement can utilize the entire camera's field of view as long as it is made with the optical system's field of view limits in mind. The size of the smallest depression to be measured determines the resolution required of the diagonal measuring system, which must be in agreement with values given in the table.

2.2.3.1 TEST PIECE

A There won't be any adjustment tables utilized for the variables to be applied for tests that are conducted on curved surfaces because all of the surfaces we use will be flat. Additionally, there won't be any use of test pieces with odd forms or unstable surfaces, therefore the need for specific supports is also gone.

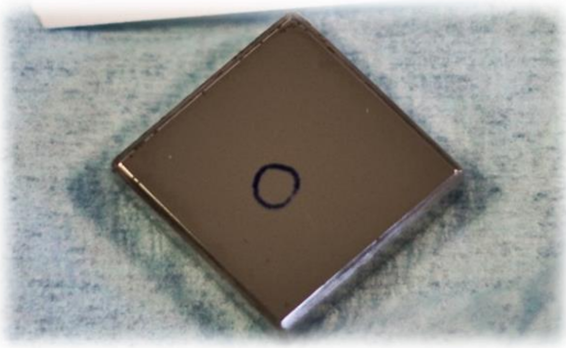


Figure 18 - 473 HV Test Piece

Figure 17 - 260 HV Test Piece

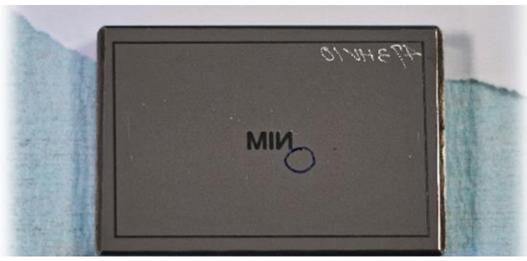


Figure 19 - 780 HV Test Piece

2.2.3.2 TEST SURFACE

Unless otherwise indicated in product standards, the test must be performed on a surface that is even, smooth, free of oxide scale, foreign objects, and any lubricants. The surface's quality must enable precise measurement of the indentation's diagonal length. Hard-metal samples must have a layer that has been removed from the surface that is at least 0.2 mm thick. [17]

2.2.3.3 PREPARATION

Surface preparation must be done in a way that prevents damage to the surface or changes in surface hardness brought on by excessive heating or cold working. Vickers micro-hardness indentations have a very little depth, hence extra care must be given during preparing. It is advised to employ a polishing or electropolishing technique that is appropriate for the material being measured.

2.2.3.4 THICKNESS

At least 1.5 times the diagonal length of the indentation must be covered by the thickness of the test piece or layer being tested. After the test, there must be no evidence of deformation at the back of the test item. A hard metal test piece must have a minimum thickness of 1 mm. About 1/7 of the diagonal length is the indentation's depth (0.143 d). [19]

2.2.4 CHAIN OF CALIBRATIONS

To prove that the testing machine is appropriate for use with this experiment, ISO 6507-2 specifies a set of calibration and verification procedures. The calibration methods include hardness tests on a variety of reference blocks as well as direct measurements of the test forces, indenter shape, indentation measuring equipment, and other elements that affect the machine's performance. The results of each of these calibration measures must fall within predetermined ranges in order for the equipment to pass verification. In the past, the testing machine's calibration and verification via reference block measurements has been referred to as indirect verification and the calibration and verification of the machine's components as direct verification. [20]

Both the technique necessary to calibrate the reference blocks used in the testing machine's indirect verification and the necessary calibration and verification procedures for the equipment used to calibrate these blocks are specified in ISO 6507-3. It is clear that there can be either an "unbroken chain of calibrations" or a "indirect verification path" when attempting to offer measurement traceability to the testing apparatus.

Direct Verification criteria describe measurements of specific testing machine components. Traceability of each of these measurements to the International System of units (SI) is provided by calibration chains, typically as implemented by the National Metrology Institute (NMI). [20]

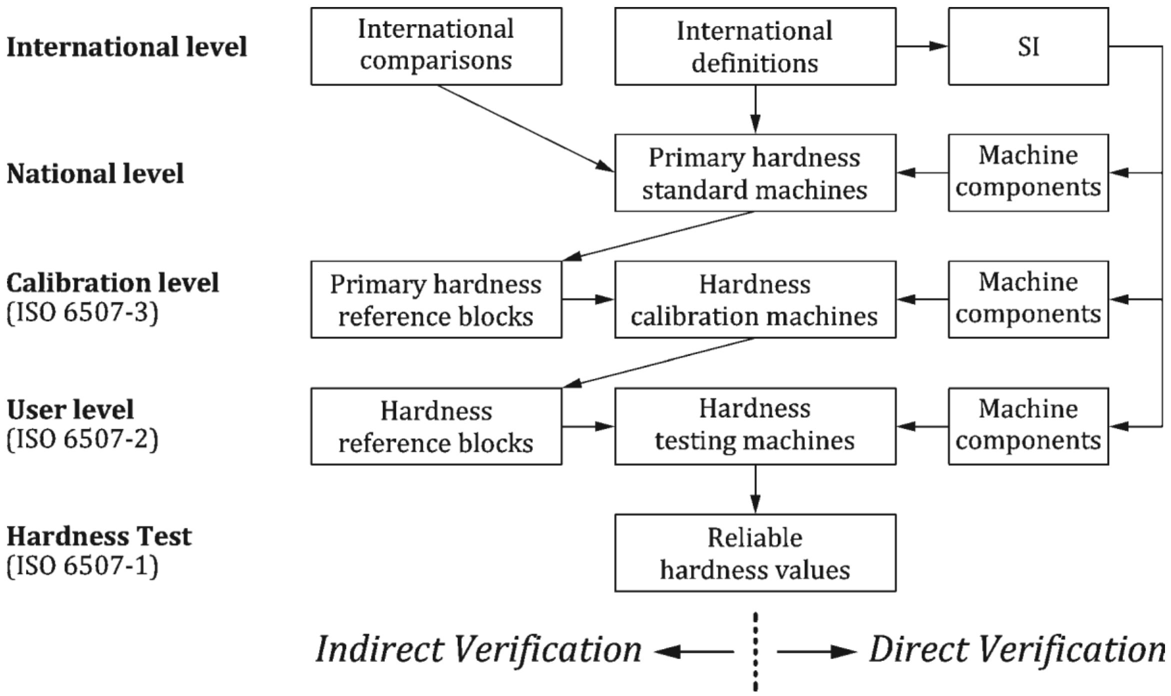


Figure 20 - Traceability levels for verification

A testing device's possible traceability path is formed by these calibration chains. The Figure shows a traceability path that involves the calibration of reference blocks and the following Indirect Verification of Vickers hardness machines for each level in the calibration hierarchy—national, calibration, and user—through a single calibration chain. Primary reference blocks are calibrated by a primary standard machine (at the national level) before being used to calibrate a calibration device. This device calibrates the reference blocks that are afterwards used to calibrate testing machines (user levers). [21]

2.2.5 VICKERS HARDNESS REFERENCE

A reference to which traceability is claimed is the other prerequisite for attaining traceability. Vickers hardness is an ordinal quantity that depends on a specific test procedure rather than being a basic attribute of a material. Ideally, an internationally accepted definition of this procedure that includes the values of all test parameters would serve as the gold standard for Vickers hardness measurements. The realization or fulfilment of this specification by a laboratory would therefore serve as the basis for hardness traceability, with the accuracy of this realization being reflected in the laboratory's measurement uncertainty and verified by cross-national comparisons. The CM Working Group on Hardness (CCM-WGH) would create the globally accepted definition, and NMIs that standardize Vickers hardness would implement it. [22]

2.2.6 VICKERS HARDNESS MEASUREMENT TRACEABILITY

2.2.6.1 GENERAL

Vickers hardness measurement experience spanning decades has shown that it is most practical to obtain traceability and assess measurement uncertainty for the lower levels of the calibration hierarchy based primarily on the indirect verification calibration chain; however, proper traceability of the individual machine component quantity values is also crucial. Vickers hardness measurements used in industry have proven to be adequate for this traceability method.

2.2.6.2 CALIBRATION LEVEL TRACEABILITY

The Indirect Verification calibration chain using primary reference blocks that have been calibrated at the National NMI level provides the best opportunity for measurement traceability. The path that should be taken to determine measurement uncertainty is also this one. To make sure that offsetting errors are not considerable, the stated calibration machine components should also be calibrated often. The Vickers scale's CCM-WGH definition should be realized by the NM, or in the absence of a CCM-WGH definition, the MI's realization of its own preferred definition, for hardness traceability. The reference to which traceability is claimed may need to be to the calibration laboratory's realization of the Vickers scale definition based on an international test method, such as that defined by this standard, if the NMI does not offer calibrated reference blocks or conduct comparison measurements with a calibration laboratory and it is not practical to use reference blocks of another NMI. In this situation, the measurement traceability of the calibration laboratory may be attained either through the Direct Verification path proven by intercomparisons or through the Indirect Verification approach employing consensus reference block standards. [21]

2.2.6.2 USER LEVEL TRACEABILITY

The Indirect Verification calibration chain, which makes use of reference blocks certified at the national or calibration level, is the most effective way to ensure measurement traceability. This is the most feasible approach, and it should be used to determine measurement uncertainty as with calibration level traceability. In order to make sure that offsetting mistakes are not severe, it is also preferable for the components of the hardness machine to undergo periodic Direct Verification. Even though this document's minimal requirement is that these measurements be taken whenever the hardness machine is built or serviced, this is not standard industrial practice. [21]

2.2.7 TEST PROCEDURE

2.2.7.1 TEST TEMPERATURE

The test is normally carried out at ambient temperature within the limits of 10 °C to 35 °C. If the test is carried out at a temperature outside this range, it shall be noted in the test report. Tests carried out under controlled conditions shall be made at a temperature of (23 $\pm$ 5) °C.

2.2.7.2 TEST FORCE

The test forces given in Table 4 are typical. Other test forces may be used including greater than 980.7 N, but not less than 0.009 807 N. Test forces shall be chosen such that result in indentations with diagonals greater than 0.020 mm.

2.2.7.3 PERIODIC VERIFICATION

For each test force utilized, the periodic verification specified in Annex C must be carried out no later than one week before to use, albeit it is advised to execute it the day of use. Every time the test force is modified, the periodic verification is advised. Every time the indenter is changed, the periodic verification is required.

2.2.7.4 TEST PIECE SUPPORT AND ORIENTATION

The test component needs to be set up on a strong support. The surfaces of the supports must be spotless and free of foreign objects (scale, oil, dirt, etc.). It is crucial that the test piece rests securely on the support to prevent any movement throughout the test that could alter the outcome.
There might be a difference in length between the two diagonals of the indentation for anisotropic materials. Therefore, the indentation should be created whenever possible so that the diagonals are orientated in plane at roughly a 45-degree angle to the direction of cold working. Limits for the variations in the lengths of the two diagonals may be stated in the product specification.

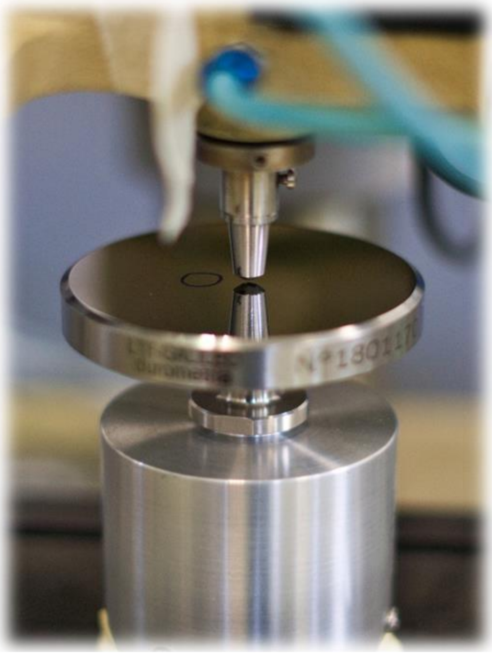


Figure 21 - Microvickers Test in process on 260 HV

2.2.7.5 FOCUS ON TEST SURFACE

Focusing is required for the diagonal measuring system microscope in order to see the specimen surface and the intended test site.

2.2.7.6 TEST FORCE APPLICATION

The test force must be applied in a direction perpendicular to the test surface while the indenter is in contact with it, without shock, vibration, or overload, until the applied force reaches the required level. It takes 7 (+1 to -5) seconds from the moment the force is first applied until the full test force is reached. [17]

For the Vickers hardness range and low-force Vickers hardness range tests, the indenter shall contact the test piece at a velocity of 0.2 mm/s. For micro-hardness tests, the indenter shall contact the test piece at a velocity of ≤ 0.070 mm/s.
The duration of the test force shall be 14 (+1 to -4) seconds except for tests on materials whose time-dependent properties would make this an unsuitable range.
Check annex for the tabulated timetables. [17]

2.2.7.7 PREVENTION OF THE EFFECT OF SHOCK OR VIBRATION

Throughout the test, the testing machine shall be protected from shock or vibration.

2.2.7.8 MINIMUM DISTANCE BETWEEN ADJACENT INDENTATIONS

The smallest possible distance between two adjacent indentations, the smallest possible distance between an indentation and the test piece's edge, and the smallest possible distance between an indentation and the coating/substrate interface, as illustrated. [5]

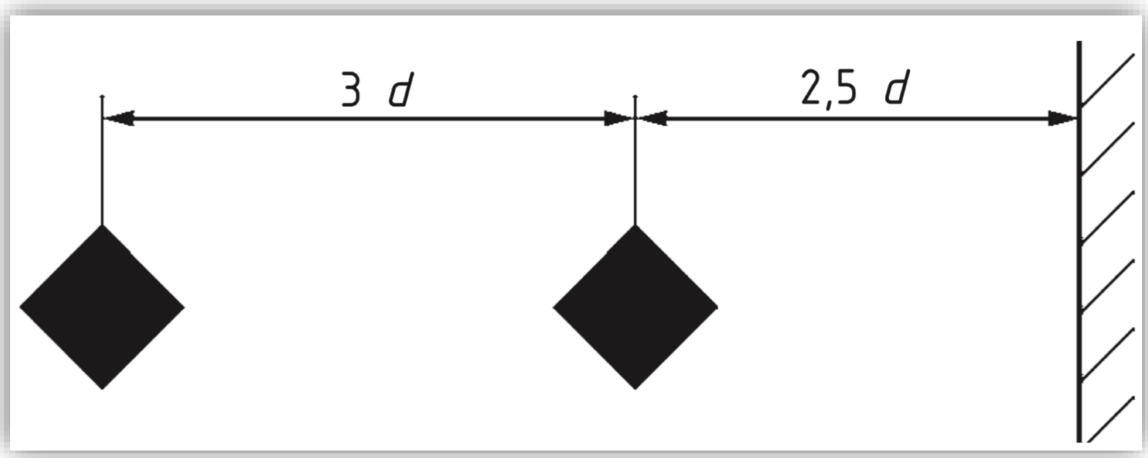


Figure 22 - Indent spacing details

The distance between the centre of any indentation and the edge of the test piece, and the distance between the centre of any indentation and the coating/substrate interface shall be at least 2,5 times the mean diagonal length of the indentation in the case of steel, copper and copper alloys and at least three times the mean diagonal length of the indentation in the case of light metals, lead and tin and their alloys. The distance between the centres of two adjacent indentations shall be at least three times the mean diagonal length of the indentation in the case of steel, copper and copper alloys and at least six times the mean diagonal length in the case of light metals, lead and tin and their alloys. If two adjacent indentations differ in size, the spacing shall be based on the mean diagonal length of the larger indentation. [5]

Check appendix for the tables with each indentation values listed

2.2.7.9 MEASUREMENT OF THE DIAGONAL LENGTH

Measure the lengths of the two diagonals. The Vickers hardness will be determined by taking the arithmetical mean of the two readings. In the microscope's field of view, the indentation's perimeter must be precisely determined for every test.

The best magnifications are those that allow the diagonal to be extended to more than 25% but less than 75% of the maximal optical field of view. The difference in diagonal length for flat surfaces in the micro-Vickers test should not be larger than 5%; otherwise, this must be expressly indicated in the report. [17]

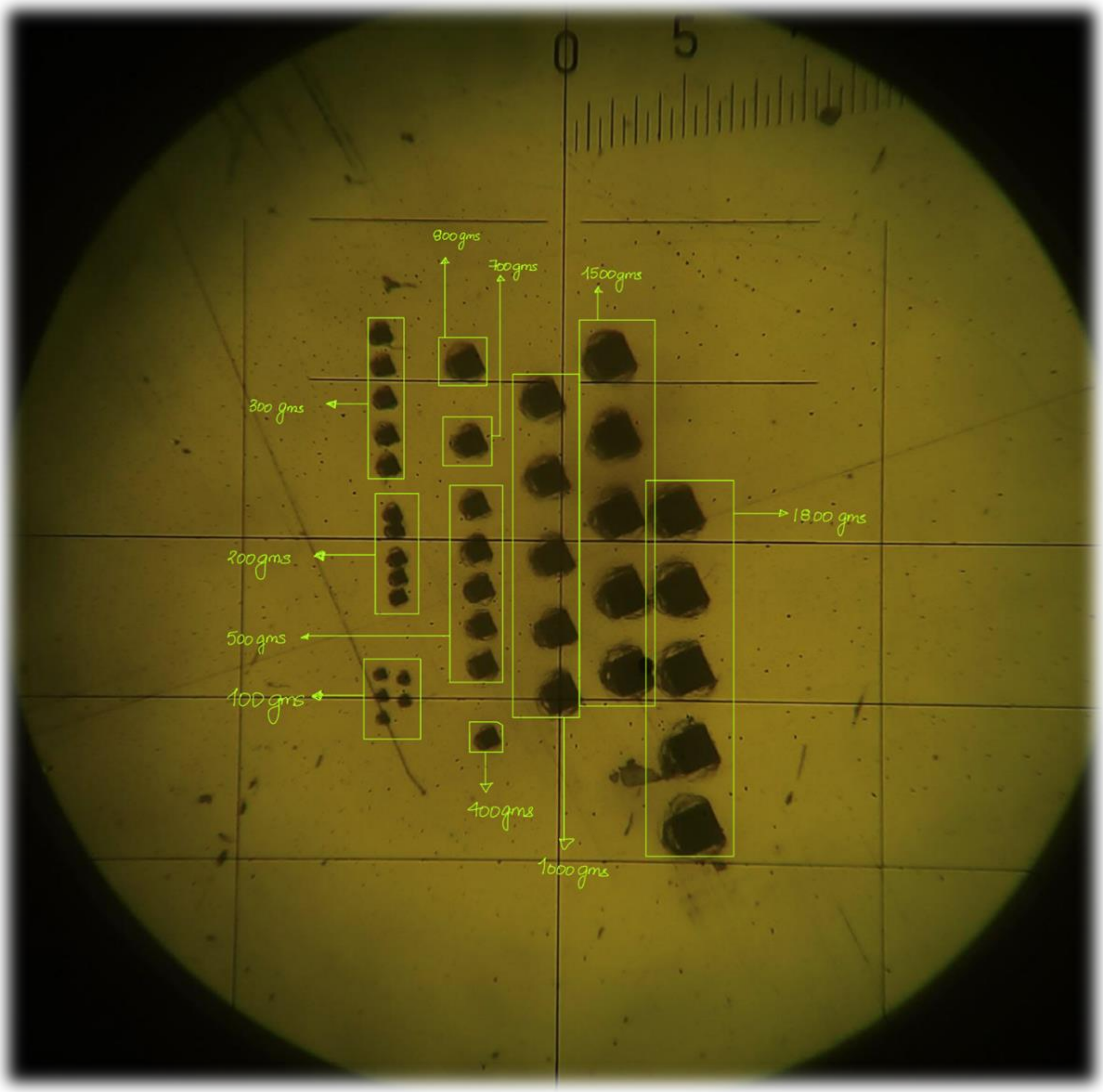


Figure 23 - Examples of Various indentations to understand the different scales with which forces effect a test piece

2.2.7.10 CALCULATION OF HARDNESS VALUE

Calculate the Vickers hardness value using the formula given previously by equation 7

Where alpha is the mean angle between the opposite faces at the vertex of the pyramid indenter
F is the test force in newtons
Mean diameter of the diagonals d is in mm
g_n is = 9,80665 $m\backslash s^2$

2.2.8 UNCERTAINTY

The method for calculating uncertainty described here solely takes into account uncertainties related to the hardness testing machine's overall measurement performance in relation to the hardness reference blocks. These performance uncertainties are a reflection of how all the individual uncertainties work together (indirect verification). This strategy places a premium on the individual machine parts operating within the tolerances. This approach should be used for a maximum of a year following the successful completion of a direct verification.

See Image above for the four-level metrological chain structure required to define and disseminate hardness scales. The chain begins at the international level, when international intercomparisons are conducted using international definitions of the various hardness measures. Primary hardness reference blocks are "produced" by a number of primary hardness standard machines at the national level for calibration laboratories. Naturally, these machines should have the highest level of precision for direct calibration and verification [23]

2.2.8.1 GENERAL PROCEDURE

The procedure calculates a combined uncertainty, u_H, by the Root-Squared-Sum-Method (RSS) out of the different sources given. [17]

$$u_H = t \times s_H$$

Equation 8

Where $t = 1.14$ for an $n = 5$

The expanded uncertainty, U_H, is derived from u_H by multiplying with the coverage factor k = 2.

The bias, b, of a hardness testing machine which is derived from the difference
Between:

I. The certified calibration value of the hardness reference block used, and
II. the mean hardness value of the five indentations made in this block during calibration of the hardness testing machine (see ISO 6507-2) can be implemented in different ways into the determination of uncertainty.

There are two methods to determine the uncertainty of hardness measurements:

I. Method M1 accounts for the systematic bias of the hardness machine in two different ways. In one approach, the uncertainty contribution from the systematic bias is added arithmetically to this value. In the other approach, a correction is made to the measurement result to compensate for the systematic bias.
II. Method M2 allows the determination of uncertainty without having to consider the magnitude of the systematic bias. [17]

2.2.8.1 *PROCEDURE FOR CALCULATING UNCERTAINTY: HARDNESS MEASUREMENT VALUES*

2.2.8.1.1 PROCEDURE WITH BIAS (METHOD M1)

The measurement bias, b, of the hardness testing machine can be expected to be a systematic effect. In JCGM 100:2008, it is recommended that a correction be used to compensate for systematic effects, and this is the basis of M1. The result of using this method is that either all determined hardness must be reduced by the ‘b’ or the uncertainty has to be increased by ‘b’. [17]

Th combined expanded measurement uncertainty for a single hardness measurement is calculated using the formula:

$$U_{M1} = k \times \sqrt{u_H^2 + 2 \times u_{ms}^2 + u_{HTM}^2}$$

Equation 9

Where

u_H contributes to uncertainty due to lack of repeatability for the measurements in the hardness testing machine

u_{ms} contributes to uncertainty due to resolution for the measurements in the hardness testing machine. The length measuring indications and the optical resolution of the microscope both have been considered. Since we have two diagonals, and both will be independently measured this parameter will be multiplied twice.

u_{HTM} The is the contribution of the standard uncertainty of the bias of the system that is related to b. Its formula is as follows:

$$u_{HTM} = \sqrt{u_{CRM}^2 + 2 \times u_{ms}^2 + u_{HCRM}^2}$$

Equation 10

u_{CRM} It's the measurements uncertainty because of the discrepancies in the calibration uncertainty for k = 1

u_{HCRM} It's the uncertainty contribution due to the lack of measurement repeatability of the hardness testing machine. Another contributing factor is the non-uniformity of the CRM, its calculated as the standard deviation of the mean hardness when measuring the CRM

u_{ms} It's the uncertainty measurement due to the resolution of the hardness testing machine, when we measure the CRM.

2.2.8.1.2 PROCEDURE WITHOUT BIAS (METHOD M2)

Technique M2 can be used in place of method M1 in some situations. Only hardness testing devices that have successfully undergone an indirect verification in line with ISO 6507-2, utilizing the value$|b| + U_{HTM}$, rather than relying solely on the bias value, b, to assess compliance with the bias's maximum allowable deviation. One component of the uncertainty, u_E, is defined in method M2 using the maximum permissible bias, b, as described in ISO 6507-2 (the positive amount by which the machine's reading is permitted to deviate from the value of the reference block). Regarding the bias limit, no compensation is applied for the hardness values. U has determined the following [17]:

$$U_{M2} = k \times \sqrt{u_H^2 + 2 \times u_{ms}^2 + u_E^2}$$

Equation 11

u_E It's the uncertainty measurement contribution due to the maximum permissible deviation of the bias which is given by:

$$u_E = b_E/\sqrt{3}$$

Equation 12

Where

b_E is the maximum permissible deviation of the bias based on the test done in ISO 6507-2

The combined expanded measurement uncertainty for a single future hardness measurement is calculated according to the Formula:

$$X = X \pm U_{M2}$$

Equation 13

2.2.8.1.3 UNCERTAINTY OF THE RESULTS

According to JCGM100:2008[4], a thorough examination of the uncertainty should be conducted. Quantifying each identifiable contributor to the uncertainty may not always be practicable. In this instance, the statistical analysis of several indentations made into the test piece can be used to estimate the type A standard uncertainty. If type A and type B standard uncertainties are combined, care should be taken to ensure that the contributions are not counted more than once.

The Joint Committee for Guides in Metrology's GUM series, which is a more accurate assessment taking into account all the uncertainties and resolution of the systems utilized, can also be specifically referred to for the formulation of uncertainty. We will also be using this series. [24]

3. REFERENCE EXPERIMENTS

3.1 METALLIC MATERIALS – VICKERS HARDNESS TEST PART 2: VERIFICATION AND CALIBRATION OF TESTING MACHINES

This is done according to the ISO 6507-2:2018 (E) standard

3.1.1 SCOPE

The procedure for verifying and calibrating testing devices and diagonal measuring systems to establish Vickers hardness is laid out in the aforementioned paper.

The testing device, indenter, and diagonal length measuring system are all subject to a direct procedure for calibration and verification. Also mentioned is an indirect verification technique that makes use of reference blocks.
Since the machines have already been tested and confirmed in accordance with the standard with validity, we will just be making a broad reference to this standard to explain the procedure used to verify machines. [18]

3.1.2 GENERAL CONDITIONS

The Vickers hardness testing device must be examined to make sure it is properly set up in accordance with the manufacturer's instructions before it can be confirmed. Particular things to look for in this situation include:

- The plunger holding the diamond indenter must be capable of sliding in its guide without any friction or excessive side play.
- The diamond indenter-holder is firmly mounted in the plunger clamping assembly.
- The test force can be applied and removed without any effects of shock, vibration, or overshoot and such that the readings are not influenced.
- Regarding the diagonal measuring system [18]:

  I. If integral with the machine, the change from removing the test force to measuring mode does not influence the readings (Not in our case, they are separate).
  II. The illumination system of the microscope produces lighting that is uniform to observe the whole field with good amount of contrast between the indentation and the surrounding surface so that the boundary can be defined clearly.
  III. The centre of the indentation must be in the centre of the field of view.

3.1.3 DIRECT VERIFICATION

3.1.2.1 GENERAL

Direct verification involves:

I. Calibration of the test force.
II. Verification of the indenter.
III. Calibration and verification of the diagonal measuring system.
IV. Verification of the testing cycle.

Direct verification should be carried out at a temperature of (23 ± 5) ° C would be reported if it wasn't in this range. The instruments used for calibration and verification must at the very least be traceable to national standards.

3.1.2.2 CALIBRATION OF THE TEST FORCE

Measurement is required for all applied forces made while the testing device is operating. Anytime the indenter must be examined at a minimum of three distinct locations within the test range.

The test force will be measured by one of the methods:

I. By means of an elastic proving device in accordance with ISO 376, class 1 or better (Which we followed).
II. Another by balancing against a force, accurate to $\pm 0.2\%$, applied by means of calibrated masses or another method with an equivalent accuracy.

It must be demonstrated that the output of the force-proving device does not vary by more than 0.2% in the period of 1s to 30 s following a change in force.

Three readings must be taken for each of the test force, F, at each position of the plunger. All readings shall be within the maximum permissible percent relative error, ΔF_{rel}, as shown below (insert table number).

The ΔF_{rel}, is the percentage relative error of each measurement of the force, F, which is calculated according to the Formula (Number of the formula [18]):

$$\Delta F_{rel} = 100 \times \frac{F - F_{RS}}{F_{RS}}$$

Equation 14

Where

F it's the measured test force

F_{RS} it's the nominal test force

This was assumed to be within the limits of the standards.

RANGES OF THE NOMINAL TEST FORCE, F_{RS} N	**MAXIMUM PERMISSIBLE RELATIVE ERROR, ΔF_{rel} %F**
0.009 807 ≤ F_{RS} < 0.098 07	± 2
0.098 07 ≤ F_{RS} < 1.961	± 1.5
F_{RS} ≥ 1.961	± 1

Table 5

3.1.2.3 VERIFICATION OF THE INDENTER

A square-based diamond pyramid was used, and all its four faces were polished to make it free from any surface defects.

The verification of the indenter shape can be done in a couple of ways, direct measurement, or optical measurement. The device used for the verification must have a maximum expanded uncertainty of no more than 0.07°. The angles measured between the opposite faces of the vertex of the diamond pyramid indenter must be within the range 136° $\pm$ 0.5°.

The four faces should cross in the center, but there is typically a line known as the junction between opposing faces, denoted by the letter "a." Directly measuring the indenter's tip or measuring the impression in an indentation of the tip must be done to estimate the length of the line of junction. Table provides the maximum permitted length of the line of connection between opposing faces. [25]

RANGES OF THE NOMINAL TEST FORCE, F_{RS} N	**MAXIMUM PERMISSIBLE LENGTH OF THE JUNCTION, a mm**
0.009 807 ≤ F_{RS} < 0.098 07	0.0005
0.098 07 ≤ F_{RS} < 1.961	0.001
F_{RS} ≥ 1.961	0.002

Table 6

we would fall in the range where the maximum permissible junction length would be 0.001 mm and 0.002 mm base on the test forces that we are using.

CALIBRATION AND VERIFICATION OF THE DIAGONAL MEASURING SYSTEM

For each magnification level, the method for measuring the indentation's diagonal must be confirmed. Two separate scales must be calibrated in both orientations, or for both scales, when they are utilized on two perpendicular axes. A calibrated, stage-operated micrometer must be used for measurements. [18]

The following must be given as the maximum enlarged uncertainty of the line interval distances on the stage micrometer:

MEASUREMENT PARAMETERS	CALIBRATION AND VERIFICATION REQUIREMENTS
Maximum expanded uncertainty of the distances between the line intervals on the micrometer	Greater of 0.000 4 mm or 0.2 %
Maximum permissible error of the measurements of the micrometer intervals	Greater of 0.000 8 mm or 1.0 % of the length measured

Table 7

Each functional test force range must be covered by measurements taken at least four regularly spaced intervals, centrally planned. Each of the uniformly spaced intervals will need the taking of three measurements.

VERIFICATION OF THE TESTING CYCLE

The maximum extended uncertainty of the timing apparatus utilized for the test cycle should be 1 s or less. The timing values obtained must be within the parameters specified in ISO 6507-1 for the testing cycle. *[17]*

UNCERTAINTY OF CALIBRATION/VERIFICATION (Direct)

CALIBRATION OF THE TEST FORCE

The combined relative standard uncertainty of the test force calibration is calculated according to Formula [18]:

$$u_F = \sqrt{u_{FRS}^2 + u_{FHTM}^2}$$

Equation 15

Where

u_{FRS} It's the relative uncertainty for the measurements from the force transducer (we get this from the calibration certificates)

u_{FHTM} It's the relative standard uncertainty of the test force from the testing machine for hardness.

The uncertainty of measurement of the force transducer, is indicated in the corresponding calibration certificates. The quantities influenced, like:

a. Dependence on the temperature,
b. long-term stability, and
c. interpolation for the deviation,

For the hardness testing machines, rotational position in relation to the central axis of force application for the indenter must also be considered, depending on the design of the force transducer.

CALIBRATION OF DIAGONAL MEASURING SYSTEM

The combined relative standard uncertainty of the reference instrument for the diagonal measuring system is calculated according to the formula [18]:

$$u_L = \sqrt{u_{LRS}^2 + 2 \times u_{ms}^2 + u_{LHTM}^2}$$

Equation 16

Where

u_{LRS} It's the uncertainty of the measurements for the micrometer (stage) for k = 1.

u_{ms} It's the uncertainty of the measurements for the resolutions of the diagonal measurement system

u_{LHTM} It's the uncertainty of the measurements of the diagonal measuring system of the testing machine for hardness

The measuring microscope's optical resolution and measurement-indicating device must be taken into account. In the calculation for the stage micrometer, the total resolution of the measuring equipment will be taken into account twice. [18]

VERIFICATION OF THE TEST CYCLE

When we measure with a usual time-measuring device (stopwatch), the uncertainty of measurement can be indicated as 0.1 s. Thus, an estimation of the uncertainty of measurement would not be necessary.

INDIRECT VERIFICATION

GENERAL

Indirect verification shall be conducted in accordance with the schedule given below

REQUIREMENTS OF VERIFICATION	FORCE	DIAGONAL MEASURING SYSTEM	TEST CYCLE	INDENTER
Before setting to work first time	x	x	x	x
After dismantling and reassembling, if force, diagonal measuring system or test cycle are affected.	x	x	x	
Failure of indirect verification	x	x	x	
Indirect verification > 13 months ago	x	x	x	

Table 8

It should be noted that the indenter needs to be examined right away after two years of use.

The use of reference blocks calibrated in accordance with ISO 6507-3 allows for the indirect evaluation of the testing device's overall performance.

Indirect verification must be carried within the temperature range of (23 $\pm$ 5) °C. If the test is conducted outside of the specified temperature range, it must be disclosed in the verification report. The calibration and verification instruments employed must be able to be tracked back to national standards. [18]

TEST FORCE AND HARDNESS LEVELS

Testing against reference blocks that have already been calibrated in accordance with ISO 6507-3 is required to verify the testing apparatuses. Utilizing the identical test forces that the machine would employ during testing, the blocks must be calibrated. At least two reference blocks must be chosen from the hardness ranges listed for each test force that the machine will be checked for when certifying it for multiple test forces. [20]

When verifying testing machines using only one test force, three reference blocks shall be used, one from each of the three hardness ranges specified below.

The hardness ranges should be chosen, when possible, to replicate the hardness levels most tested when using the specific test forces.

I. <250 HV
II. 400 HV to 600 HV
III. >700 HV

Having said that we have made use of three reference blocks from each range for each test force so that all possible ranges, iterations and combinations of test force and hardness are covered.

MEASUREMENT OF REFERENCE INDENTATION

On each reference block, one reference indentation from the current calibration needs to be measured. The discrepancy between the measured mean value and the certified diagonal length's mean value for each indentation shall not be larger than 0.001 mm, or 1.25 percent of the indentation's reference length. This test can also be performed on an indentation of the same size and hardness in a different reference block. *[20]*

NUMBER OF INDENTATIONS

We mark five indentations that must be made and measured on each reference block. It is necessary to conduct the test in line with ISO 6507-1. The blocks' calibrated surfaces alone must be used for testing.

VERIFICATION RESULT

For each reference block, let $H_1, H_2, H_3, H_{4,} H_5$ be the measured hardness arranged in increasing order of magnitude corresponding to the measured diagonals, $d_1, d_2, d_3, d_{4,} d_5$, in decreasing order of magnitude. The mean hardness value, H, and the mean diagonal length, d, is calculated according to Formula below.

$$H = \frac{H_1 + H_2 + H_3 + H_4 + H_5}{5}$$

Equation 17

$$d = \frac{d_1 + d_2 + d_3 + d_4 + d_5}{5}$$

Equation 18

These equations were used to calculate the uncertainties and other parameters that lead to the calculation of uncertainties [20]

REPEATABILITY

Every testing machine has a relative repeatability, r_{rel} which is expressed as a percentage of H is calculated according to Formula:

$$r_{rel} = 100 \times \frac{H_5 - H_1}{H}$$

Equation 19

The repeatability of the testing machine is satisfactory if $(d_1 - d_5)$ ≤ 0.001 mm. If $(d_1 - d_5)$ > 0.001 mm, the testing machine is satisfactory if r_{rel} is less than or equal to the percentages indicated in Table.

VICKERS HARDNESS OF THE REFERENCE BLOCK	MAXIMUM PERMISSIBLE RELATIVE HV REPEATABILITY OF THE TESTING MACHINE, r_{rel} % HV		
	HV 5 to HV 100	HV 0.2 to < HV 5	< HV 0.2
HV ≤250	6.0	12.0	18.0
HV >250	4.0	8.0	12.0

Table 9

Here we must note that the materials with a lower hardness exhibit higher repeatability values in comparison to material with a higher hardness value. [20]

BIAS

The bias is represented by the symbol, b, for testing machine under verification conditions the bias is calculated according to the as follows:

$$b = H - H_{CRM}$$

Equation 20

where

H_{CRM} Is the certified hardness of the reference block used.

The percentage bias, b_{rel} which we tabulated in the ISO 6507-1: 2018 above is calculated according to the formula [20]:

$$b_{rel} = 100 \times \frac{H - H_{CRM}}{H}$$

Equation 21

The maximum positive or negative bias of the testing machine, expressed as a percentage of the specified hardness of the reference block, shall not exceed the values give here [18]:

MEAN DIAGONAL LENGTH, *d* mm	**MAXIMUM PERMISSIBLE PERCENT HV BIAS, b_{rel}, OF THE TESTING MACHINE ± %HV**
$0.02 \le d < 0.14$	$0.21/d + 1.5$
$0.14 \le d \le 1.4$	3

Table 10

The calculated tables are in the annex

UNCERTAINTY OF CALIBRATION / VERIFICATION (Indirect)

Through indirect verification using hardness reference blocks, the machine's general functionality is examined, and metrics like repeatability and the hardness testing machine's departure from the actual hardness value are computed.

The following formula is used to compute the measurement uncertainty for the hardness indirect verification testing machine [18]:

$$u_{HTM} = \sqrt{u_{CRM}^2 + u_{CRM-D}^2 + u_H^2 + 2 \times u_{ms}^2}$$

Equation 22

Where,

u_{CRM}	It's the calibration uncertainty for the reference block with k = 1
u_{CRM-D}	It's the change in hardness of the reference block since the last time it was measured which happens due to drift.
u_{HCRM}	It's the standard uncertainty when measuring the CRM with the hardness testing machine.
u_{ms}	It's the uncertainty of the diagonal measuring system.

INTERVALS BETWEEN VERIFICATIONS

Direct verifications shall be performed according to the schedule given in the Table. It is recommended that direct verifications be performed every 12 months.

Indirect verification shall be performed at least once every 12 months and after a direct verification has been performed. [18]

VERIFICATION REPORT / CALIBRATION CERTIFICATE

VICKERS TESTING MACHINE

In our case, the test equipment and indenter were already calibrated by INRiM. However, there are several safety measures that must be followed when working with the equipment. Experience has taught us that many indenters can quickly develop defects after being used. Small fissures, pits, or other surface imperfections are the cause of this. Many indenters can be recovered by regrinding if such flaws can be identified beforehand. Otherwise, any surface flaw could quickly get worse and render the indenter useless [18].

Therefore,

I. The condition of indenters must be checked by visually monitoring the aspect of indentation on a reference block, every day the testing machine is used.
II. A verified indenter is no longer valid when it shows signs of any defects.

III. Regrinding or repairing indenters must meet all the requirements of the standards if it's meant to be used again for testing.

METALLIC MATERIALS – VICKERS HARDNESS TEST PART 3: CALIBRATION OF REFERENCE BLOCKS

SCOPE

This is a method that specifies the calibration of reference blocks used for the indirect verification of Vickers hardness testing of machines as specified in ISO 6507 – 2:2018

It is important to note that this method is only for indentation for diagonals > 0.020 mm. [20]

MANUFACTURE OF REFERENCE BLOCKS

GENERAL

The test block must be specifically created to serve as a hardness reference block using a manufacturing process that will give it the necessary characteristics like homogeneity, structural stability, surface hardness uniformity, and time dependent stability in hardness so that it varies as little as possible over time.

THICKNESS

The thickness of the reference block must not be less than 5 mm. (All our test blocks are over 10mm in thickness) [20]

TEST SURFACE AREA

The test surface area of the reference block shall not exceed 40 cm^2. (All our test blocks are well within that limit) [20]

MAGNETISM

Any magnetism must be absent from the reference blocks. The blocks, if made of steel, should have been demagnetized at the end of the production process or before calibration, it is advised that the maker make sure.

FLATNESS AND PARALLELISM

The test piece's and the support surfaces' maximum flatness deviation must exceed 0.005 mm. In a piece of 50 mm, the maximum parallelism error cannot be greater than 0.010 mm. [20]

SURFACE ROUGHNESS

The test piece surface must be free from scratches and defects that can interfere with the measurement of the indentations. The test surface roughness, which is represented by the symbol,

R_a, must not exceed 0.05 μm. The bottom surface on which the piece is supported must also be finely ground for a smooth finish. [20]

PREVENTION OF THE REGRIND OF THE TEST SURFACE

To verify that no material has been removed from the reference block after testing, its thickness when calibrating must be noted nearest 0.01 mm, or an identifying mark must be made on the test surface of the block. The thickness values are listed in the Annex

CALIBRATION MACHINE

GENERAL

This standard must adhere to the general standards given in ISO 6507-2, as well as the calibration machine's requirements, which are detailed in the steps below

DIRECT VERIFICATION

The calibrating device must be verified directly at intervals not more than 12 months.
Direct verification includes [20]:

I. Verification of the test forces,
II. Verification of the (diamond) indenter,
III. Calibration and verification of the measuring system for the diagonals, and
IV. Verification of the test cycle or, the force vs time behaviour of the system.

TRACEABILITY OF VERIFICATION INSTRUMENTS

The instruments used for verification and calibration must be traceable to the national standards.

TEST FORCE

Every test force must be verified at three different positions of the plunger, which are spaced at equal increments covering the limits of travel that would be used during testing. At each position, the force must be measured three times using an elastic measuring device according to ISO 376, class 0.5 or better, we can also use another device which would have the same accuracy levels or better. Every measurement must agree with the nominal values of test forces to within $\pm$0.2 % for normal hardness, to within $\pm$0.3 % for low-force hardness and to within $\pm$0.5 % for micro-hardness. [15]

INDENTER

The indenter must comply with ISO 6507-2 and must satisfy the following [20]:

I. The four surfaces of the square-shaped diamond pyramid must be polished to a high degree, to make it free from surface defects and flat within 0.0003 mm.
II. The angle measured between the opposite faces of the diamond pyramid must be 136°$\pm$0.1°.
III. The axis of the diamond pyramid and the axis of the indenter-holder must be coincident and the difference between the axis shall be less than 0.3°.

IV. The point of the diamond indenter must be checked with a high-power microscope or with an interference microscope to be more precise. The junction must be checked to be within the parameters as mentioned in the table

RANGES OF THE TEST FORCE, **N**	**MAXIMUM PERMISSIBLE LENGTH OF THE JUNCTION, a** **mm**
49.03 ≤ F	0.001
1.961 ≤ F < 49.03	0.001
F ≥ 0.009807	0.002

Table 11

A valid calibration certificate must be obtained which should confirms the geometrical deviations of the indenter.

DIAGONAL MEASURING SYSTEM

The scale of the diagonal measuring system shall be graduated to permit estimation of the diagonals of the indentation in accordance with the table given.

DIAGONAL LENGTH, *d* mm	**RESOLUTION OF THE DIAGONAL MEASURING SYSTEM**	**MAXIMUM PERMISSIBLE ERROR**
$D \leq 0.060$	0.000 15 mm	±0.000 3 mm
$0.060 < D \leq 0.200$	0.25 % of d	±0.5 % of d
$D > 0.200$	0.000 5 mm	±0.001 mm

Table 12

Measurements on an accurate stage micrometer must be taken in order to verify the system of measuring the diagonal of the indentation for each magnification and for each included line scale (where applicable). Each working range must be covered by measurements at five (Minimum) evenly spaced intervals that are located in the center of the field of view.

The stage micrometer's minimum count must have a distance between intervals (expanded uncertainty) maximum value of 0.0002 mm or 0.04 percent of the value, whichever is greater.

There must be three measurements taken at each of the evenly spaced periods. For each measurement in each interval, the maximum permitted error cannot exceed the values listed in the Table. Same table as above [26]

CALIBRATION PROCEDURE

The blocks used for reference must be calibrated, at a temperature of (23 ± 5) °C, using the general procedure specified in ISO 6507-1.

During calibration, the thermal drift must not exceed 1 °C.

The time from the initial application of force until the full test force is reached and the approach velocity of the indenter must meet the standard time requirements as given in the table.

The duration of application of the test force must be 14^{+1}_{-1} s.

For micro hardness testing, (0.009807 N ≤ F < 1.961 N), the maximum vibrational acceleration reaching the calibration machine must not exceed 0.005 g_n (g_n equals the standard acceleration of gravity: g_n = 9.806 65 m/s^2). [20]

RANGES OF TEST FORCE, *F* N	TIME FOR APPLICATION OF THE TEST FORCE s	APPROACH VELOCITY OF THE INDENTER mm/s
$F < 1.961$	7^{+1}_{-1}	0.05 to 0.2
$1.961 \le F < 49.03$	7^{+1}_{-1}	0.05 to 0.2
$F \ge 49.03$	7^{+1}_{-1}	0.015 to 0.07

Table 13

NUMBER OF INDENTATIONS

A minimum of five indentations, evenly spaced across the test surface, must be made on each reference block. There must be at least one indentation designated as a reference indentation.

More than five indentations should be made in order to reduce measurement uncertainty for Micro-Vickers tests. It is advised to indent the reference block five times, at intervals of 10, 15, or 25. The accuracy is also increased in this manner.

UNIFORMITY OF HARDNESS

RELATIVE NON-UNIFORMITY

For each reference block, let $H_1, H_2, \dots, H_n$ be the n measured hardness values arranged in increasing order of magnitude corresponding to the measured diagonals $d_1, d_2, \dots, d_n$ in decreasing order of magnitude. The average hardness, H, is calculated according to Formula:

$$H = \frac{H_1 + H_2 \dots + H_n}{5}$$

Equation 23

The relative non-uniformity, r_{rel}, expressed as a percentage of H, is calculated according to Formula:

$$r_{rel} = 100 \times \frac{H_n - H_1}{H}$$

Equation 24

The maximum permissible value of non-uniformity, r_{rel}, of a reference block is given in Tables

We only be taking the table where n = 5 since we have made that same number of indentations. [20]

HARDNESS OF BLOCK	MAXIMUM PERMISSIBLE VALUE OF NON-UNIFORMITY, R_{REL}, %		
	<HV 0,2	HV 0,2 to <HV 5	HV 5 to HV 100
≤250 HV	8.0 or $d_1 - d_n$ = 0.001 mm	6.0	4.0
>250 HV		4.0	2.0

Table 14

UNCERTAINTY OF MEASUREMENT OF HARDNESS REFERENCE BLOCKS

The determination of the uncertainty of measurement bias of hardness calibration machine for hardness reference block measurement is given as:

$$u_{CM} = \sqrt{u_{CRM-P}^2 + u_{xCRM-P}^2 + u_{CRM-D}^2 + 2 \times u_{ms}^2}$$

Equation 25

Where

u_{CRM-P} It's the calibration uncertainty of the reference block for primary hardness with k = 1

u_{xCRM-P} It's the standard uncertainty of hardness calibration machine for the measurements of the CRM

u_{CRM-D} It's the standard uncertainty due to the hardness change since its last calibration due to drift

u_{ms} It's the uncertainty of the diagonal measuring system.

and the uncertainty of the measurement value is calculated according to Formula:

$$u_{CRM} = \sqrt{u_{CM}^2 + u_{xCRM}^2 + 2 \times u_{ms}^2}$$

Equation 26

Where

u_{CM} It's the uncertainty of measurement bias of the hardness calibration machine.

u_{xCRM} It's the standard uncertainty of hardness measurement of hardness reference blocks with the hardness calibration machines.

u_{ms} It's the uncertainty due to the resolution of the hardness calibration machine. [20]

MARKING

Every reference block must be marked with the following information:

- The arithmetic mean of the hardness values that were calculated in the calibration test,
- The name of the supplier or manufacturer and their logo when needed.
- The serial number.

- The name or mark of the agency that is calibrating the test piece.
- Identifying mark on the test surface or the thickness of the test piece.
- The year of calibration to be included when not mentioned in the serial number.

All markings must be placed on the test surface or on the side of the block. Marks put on the side of the block must be up when the test surface is facing up. [20]

CALIBRATION CERTIFICATE

All delivered reference blocks must be accompanied with a document giving the following information:

- A reference to ISO 6507-3:2018
- The serial number printed or marked on the block.
- The date of calibration.
- The arithmetic-mean of the hardness values in the correct format as defined in ISO 6507-1 and the value that characterizes the non-uniformity of the test piece.
- Information about the diagonal and location of the indentation. [20]

VALIDITY

Only the scale for which it is calibrated will the reference block be appropriate. There will be a 5-year validity limit on the calibration. However, it is important to remember that the calibration validity will be lowered to two to three years for alloys made of aluminium and copper.

4. CONCLUSION

TEST REPORT

The test report includes the following data [17]:

i. A reference to this document, i.e., ISO 6507-1:2018 which can also be seen in the reference section.
ii. The test pieces used were three with the following hardness values that were know before the test as follows:
    a. 260 HV with identification code: 1801170 (Circular in shape)
    b. 470 HV with identification code: 473HV10 (Rectangular in shape)
    c. 700/800 HV with identification code: G186-51 (Square shaped)
iii. The date of the test was the 15th of March 2022.
iv. The hardness result obtained in a few methods, HV are as follows:

Based on the calculations for the periodic verification of the testing machine the percentage bias was calculated as follows:

$$b_{rel} = 100 \times \frac{H - H_{CRM}}{H_{CRM}}$$

Equation 27

where

H It's the hardness with respect to the measurement taken

H_{CRM} It's the certified hardness value of the reference block used.

MEAN DIAGONAL LENGTH, D MM	MAXIMUM PERMISSIBLE PERCENT HV BIAS, B_{REL} OF THE TESTING MACHINE ± %HV
$0.02 \le D < 0.14$	$0.21/d + 1.5$
$0.14 \le D \le 1.400$	3

Table 15

and the Maximum permissible bias is calculated and categorised according to the following table:

The standard uncertainty due to b_e is given by:

$$u_E = b_e/\sqrt{3}$$

Equation 28

Which leads to the following table with the calculated values as:

CRM HARDNESS VALUE	HARDNESS NUMBER	b_{rel} %	MAX b_{rel} LIMIT (b_e) %	WITHIN LIMITS YES/NO
260	233	-10.5	12.02	YES
260	256	-1.66	3.96	YES
260	259	-0.55	3	YES
473	452	-4.46	16.16	YES
473	479	1.17	4.87	YES
473	482	1.94	3.45	YES
780	789	1.20	20.88	YES
780	790	1.29	5.83	YES
780	783	0.35	3.99	YES

Table 16

The uncertainty that we will have considered of the two methods is the second one i.e., M2 thus the formula we will follow is equation 11.

Its parameters are calculated as follows:

$$\delta_{ms} = \sqrt{\delta_{OR}^2 + \delta_{IR}^2}$$

Equation 29

Where,

δ_{OR} It's the resolution of the lens i.e., objective of the microscope = 0.0005 mm

δ_{IR} It's the resolution of the measuring system display indicator = 0.0001 mm

Therefore,

$$\delta_{ms} = 0.00051\ mm$$

Figure 24 - Lenses used for the measurement of the diagonals

Since we now have the value of δ_{ms} we can calculate the standard uncertainty due to the resolution of the hardness value indicating display that we are using as follows:

$$u_{ms} = -\frac{2x}{d} \times \frac{\delta_{ms}}{2\sqrt{3}}$$

Equation 30

we already know from u_H

thus, the standard deviations of the repeatability measurements are calculated as follows:

$$s_H = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}(H_i - H)^2}$$

Equation 31

Then based in the S_H value the u_H is calculated as follows:

$$u_H = t \times s_H$$

Equation 32

We can then tabulate the parameters of M2 as follows:

Designation	Source of Uncertainty	Value
233 HV 0.05	u_E	16.15
	s_H	12.86
	u_H	6.55
	u_{ms}	-3.43
256 HV 1	u_E	5.85
	s_H	5.58
	u_H	2.84
	u_{ms}	-0.88
259 HV 3	u_E	4.47
	s_H	5.81
	u_H	2.96
	u_{ms}	-0.51
452 HV 0.05	u_E	42.17
	s_H	10.12
	u_H	5.16
	u_{ms}	-9.29
479 HV 1	u_E	13.46
	s_H	4.75
	u_H	4.75

	u_{ms}	-2.26
482 HV 3	u_E	9.62
	s_H	5.39
	u_H	5.39
	u_{ms}	-1.32
789 HV 0.05	u_E	95.18
	s_H	35.06
	u_H	17.87
	u_{ms}	-21.45
790 HV 1	u_E	26.62
	s_H	19.50
	u_H	9.94
	u_{ms}	-4.80
783 HV 3	u_E	18.04
	s_H	16.05
	u_H	8.18
	u_{ms}	-4.80

Table 17

There is another parameter called as the Coefficient of Variation that we will not make use of in these calculations its formulated as follows:

$$C_V = \frac{100 \times s}{H}$$

Equation 33

Where

s It's the standard deviation

H It´s the average of the Hardness values that were measured.

Please note while the u_H was being calculated an additional term $\sqrt{n}$ was divided with the resulting value, this was done to account for the non-uniformity of the test pieces.

Now that we have all the parameters for the uncertainty, we can calculate the expanded uncertainty according to the M2 method as given by the formula that is equation 11

MEASURE DESIGNATION	EXPANDED MEASUREMENT UNCERTAINTY U_{M2}	FINAL MEASUREMENT RESULT $X = x \pm U_{M2}$
233 HV 0.05	36.19	(233$\pm$36) HV 0.05
256 HV 1	13.26	(256$\pm$13) HV 1
259 HV 3	10.84	(259$\pm$11) HV 3
452 HV 0.05	88.94	(452$\pm$89) HV 0.05
479 HV 1	28.11	(479$\pm$28) HV 1
482 HV 3	20.36	(482$\pm$20) HV 3
789 HV 0.05	202.97	(789$\pm$203) HV 0.05
790 HV 1	58.44	(790$\pm$58) HV 1
783 HV 3	40.37	(783$\pm$40) HV 3

Table 18

We now have the expanded uncertainties associated to the measurements made and we will comment on the results below before that we will compare our results with another method of calculation to solidify our results making a reference to the (GUM) JCMG 100:2008 which will make the calculations under the same ISO 6507 – 1 standard but include additional term to account for other uncertainties and present us with a more accurate uncertainty evaluation.

Note the forces of 29.4 N were made on a different testing machine with its picture below and the forces of .49 N and 9.8 N on the micro durometer.

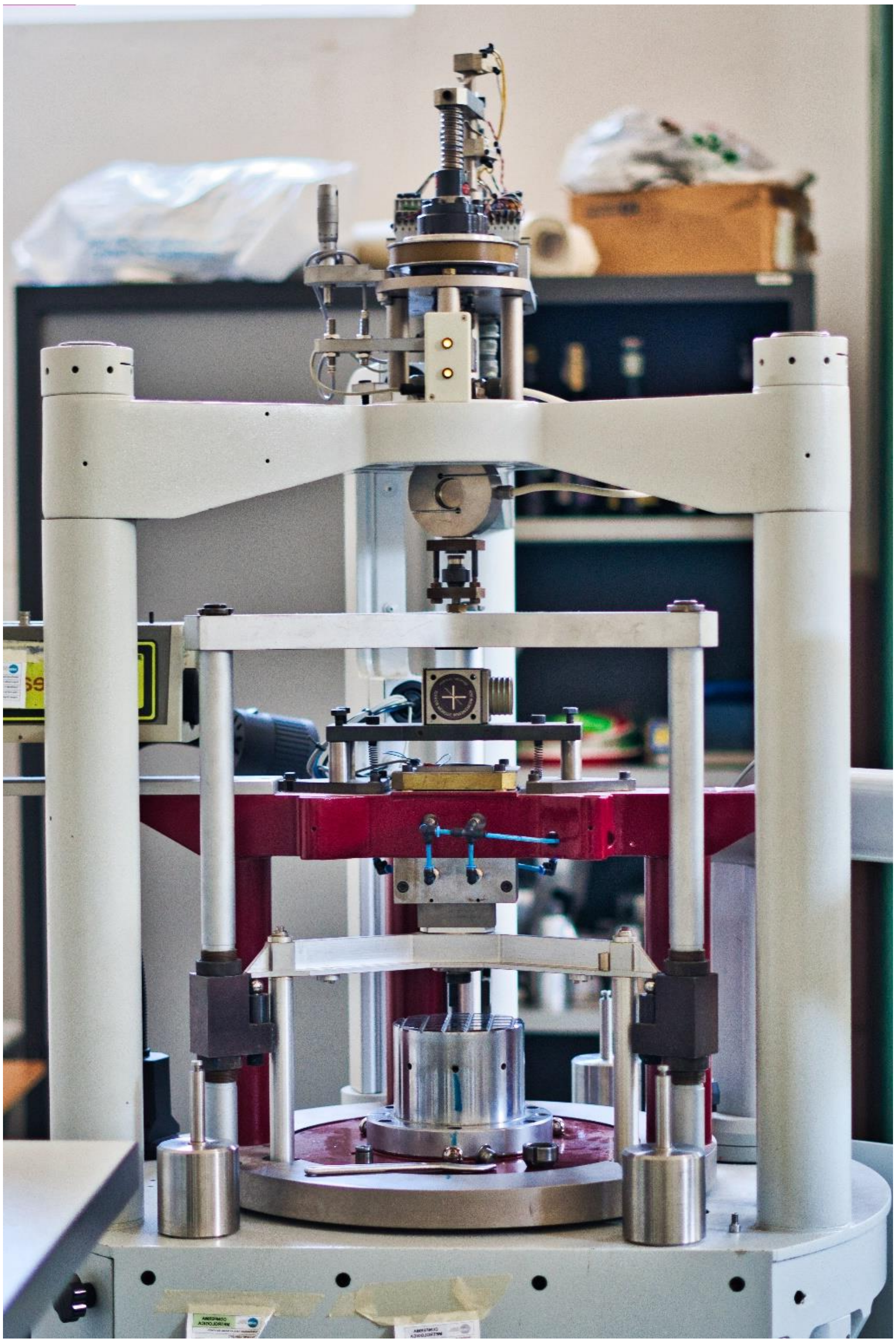


Figure 25 - Rockwell hardness testing machines which has a minimum test force of 29.4 N

ISO 6507-1:2018 EQ N.O – HARDNESS VALUE HV	CRM VALUE HV	EXPANDED MEASUREMENT UNCERTAINTY U_{M2} %	FORCE N	MACHINE USED FOR TEST PROCEDURE
233	260	15.55	.49 (HV 0.05)	Officine Galileo LTF Microdurometer
256	260	5.18	9.8 (HV 1)	Officine Galileo LTF Microdurometer
259	260	4.19	29.4 (HV 3)	Officine Galileo LTF Rockwell
452	473	19.68	.49 (HV 0.05)	Officine Galileo LTF Microdurometer
479	473	5.87	9.8 (HV 1)	Officine Galileo LTF Microdurometer
482	473	4.22	29.4 (HV 3)	Officine Galileo LTF Rockwell
789	780	25.71	.49 (HV 0.05)	Officine Galileo LTF Microdurometer
790	780	7.39	9.8 (HV 1)	Officine Galileo LTF Microdurometer
783	780	5.16	29.4 (HV 3)	Officine Galileo LTF Rockwell

Table 19

A more accurate benchmark test procedure was introduced at this stage to ensure that the values obtained were adhering to the standards. This was done in accordance with the JCGM 100:2008 (GUM) ISO 6507-1:2018 procedures with the parameters selected as given [24]:

Repeatability: maximum relative difference within the range of $\pm\ \mathbf{1}\%$

Corresponding expanded uncertainty = 0.1 µm

Risk of error of = 5%

Repeatability (standard deviation) = 0.12 µm

Resolution = 1 µm

Confidence level applied was 95 %

ISO 6507-1:2018 IN ACCORDANCE WITH JCGM 100:2008 HV	CRM VALUE HV	EXPANDED UNCERTAINTY %	FORCE N	PERCENTAGE DELTA BETWEEN THE TWO EXPANDED UNCERTAINTIES RESULTS OBTAINED %
240	260	3.47	.49 (HV 0.05)	12.08
257	260	1.40	9.8 (HV 1)	3.78
260	260	1.25	29.4 (HV 3)	2.94
471	473	4.65	.49 (HV 0.05)	15.03
483	473	1.56	9.8 (HV 1)	4.31
485	473	1.32	29.4 (HV 3)	2.90
835	780	6.20	.49 (HV 0.05)	19.51
800	780	1.78	9.8 (HV 1)	5.61
783	780	1.40	29.4 (HV 3)	3.75

Table 20

For the calculations in the above table the Mathematical model followed was as follows [24]:

$$HV = \frac{F sin\left(\frac{\alpha}{2}\right)}{g_n \frac{\left[\frac{d_1 + d_2}{2}\right]^2 - t^2}{2}}$$

Equation 34

A point to be noted here is that the coverage factor K was kept constant at K = 2.

We can thus conclude that the achieved values are off by an average of 1.689 % for the expanded uncertainties obtained from the ISO 6507-1:2018 In accordance with JCGM 100:2008 (GUM) and by the M2 method for uncertainties.

THE NEW PRIMARY HARDNESS TESTING MACHINE (MEASUREMENTS ONLY)

Figure 26 - New MicroVickers testing system with Integrated Diagonal measurement system

It gives out automated values of the diagonal lengths and corresponding hardness values along with the standard deviations.
The results obtained from the measurements are as follows:

DESIGNATION	D	STD DEV	STD DEV %	HV	STD DEV	STD DEV %
780 HV0.05	11.204	0.069	0.612	**744.5**	9.055	1.216
780 HV1	48.73	0.035	0.071	**780.9**	1.107	0.142
780 HV3	84.659	0.04	0.047	**776.1**	0.734	0.095
473 HV0.05	14.4638	0.176	1.204	**462**	10.479	2.408
473 HV1	62.556	0.127	0.203	**478**	0.097	0.406
473 HV3	108.008	0.379	0.351	**476.9**	3.3	0.692
260 HV0.05	19.728	0.264	1.34	**240.5**	6.318	2.627
260 HV1	86.077	0.346	0.102	**252**	0.102	0.811
260 HV3	147.343	0.615	0.418	**256.3**	2.128	0.83

Table 21

Previously we were able to conclude that the measurement calculations of uncertainty were more accurate and precise when following the JCGM thus we will proceed to use that method to characterize our calculations with the following results:

DESIGNATION	EXPANDED UNCERTAINTY HV	EXPANDED UNCERTAINTY %
780 HV0.05	44.60	5.72%
780 HV1	13.47	1.70%
780 HV3	10.71	1.37%
473 HV0.05	23.18	5.02%
473 HV1	7.49	1.57%
473 HV3	7.07	1.47%
260 HV0.05	10.46	4.26%
260 HV1	3.99	1.58%
260 HV3	3.85	1.50%

Table 22

Now that we have all the values in places, we can check for the normalized error vales obtained [27]:

New Measurements	Expanded uncertainty (New)	Old Measurements	Expanded uncertainty (Old)	Normalized error E_n
744.5	44.6	835	51.79	**1.32**
780.9	13.47	800	14.21	**0.97**
776.1	10.71	783	10.86	**0.45**
462	23.18	471	21.94	**0.28**
478	7.49	483	7.56	**0.47**
476.9	7.07	485	6.38	**0.85**
240.5	10.46	240	8.32	**-0.037**
252	3.99	257	3.6	**0.93**
256.3	3.85	260	3.25	**0.73**

Table 23

v. We can note that the uncertainties are high in the case of the lowest force that we used of .49 Newtons, this could be due to various factors like the resolution of the optical system, calibration of the transducers for low forces, but most importantly because the value of the diagonals is below the given threshold of 20 Micrometres which causes the uncertainties to increase and thus result in a higher normalized error in one case.

vi. The temperature of the test was within the ambient range.

vii. Multiple methods were assessed to conduct the final analysis of the results

   a. Method M2 for Expanded Uncertainty

   b. ISO 6507-1:2018 In accordance with JCGM 100:2008 (GUM) [24]

While following all the procedures according to the ISO standards we can understand that:

i. JCGM 100:2008 (GUM) is a more accurate method for the uncertainty estimation.

ii. The nominal forces of .49 N and 9.8 N were applied on the old Microdurometer and the uncertainties were higher in comparison to the Rockwell Hardness testing machine on which the nominal force of 29.4 N was applied with the same indenter, signifying that it has better repeatability and reproducibility.

iii. The Latest Microdurometer has by far the fastest measurement times and in practice the systematic errors will be reduced when considered since the whole system of measurement and indentation is in one unit and automated to a high degree of precision.

iv. Based on the results obtained The Latest Microdurometers measurement system can be put into practice since the output from the system is highly precise and accurate. While it is to be noted that for indentations smaller than 20 micrometers the necessary changes will have to be made to the optical measuring system (Lens change)

5. ANNEX

DESIGNATION	d_1	d_2	σ_H	σ_V
260 HV 1	85.28	86.46	0.04	0.02
260 HV 1	85.67	86.12	0.06	0.07
260 HV 1	84.08	84.41	0.06	0.05
260 HV 1	85.41	85.9	0.08	0.07
260 HV 1	83.77	84.28	0.05	0.11

Table 24

DESIGNATION	d_1	d_2	σ_H	σ_V
260 HV 3	149.12	148.45	0.15	0.1
260 HV 3	147.44	147.15	0.15	0.12
260 HV 3	147.13	145.64	0.16	0.12
260 HV 3	146.68	146.26	0.12	0.16
260 HV 3	145.46	143.06	0.21	0.95

Table 25

DESIGNATION	d_1	d_2	σ_H	σ_V
260 HV 0.05	20.3	19.85	0.05	0.04
260 HV 0.05	18.95	19.22	0.04	0.04
260 HV 0.05	21.07	19.75	0.06	0.03
260 HV 0.05	20.23	19.52	0.05	0.04
260 HV 0.05	19.69	20.98	0.05	0.05

Table 26

DESIGNATION	d_1	d_2	σ_H	σ_V
470 HV 0.05	14.36	14.05	0.04	0.05
470 HV 0.05	14.84	14.3	0.04	0.04
470 HV 0.05	14.05	14.28	0.06	0.03
470 HV 0.05	14.07	14.69	0.04	0.09
470 HV 0.05	14.35	14.21	0.04	0.04

Table 27

DESIGNATION	d_1	d_2	σ_H	σ_V
470 HV 1	61.89	62.31	0.04	0.03
470 HV 1	62.44	62.63	0.04	0.03
470 HV 1	61.67	62.19	0.04	0.03
470 HV 1	61.74	62.24	0.04	0.08
470 HV 1	62.78	62.39	0.05	0.03

Table 28

DESIGNATION	d_1	d_2	σ_H	σ_V
470 HV 3	108.19	108.5	0.15	0.12
470 HV 3	107.66	107.15	0.15	0.13
470 HV 3	106.8	107	0.13	0.15
470 HV 3	107.64	106.03	0.17	0.18
470 HV 3	106.93	107.87	0.17	0.16

Table 29

DESIGNATION	d_1	d_2	σ_H	σ_V
780 HV 0.05	10.56	10.49	0.08	0.09
780 HV 0.05	10.99	11.3	0.01	0.05
780 HV 0.05	10.79	11.05	0.07	0.08
780 HV 0.05	10.75	10.6	0.11	0.15
780 HV 0.05	11.36	10.45	0.09	0.15

Table 30

DESIGNATION	d_1	d_2	σ_H	σ_V
780 HV 1	48.32	48.8	0.04	0.09
780 HV 1	47.98	48.31	0.04	0.04
780 HV 1	47.75	48.13	0.04	0.04
780 HV 1	47.81	48.33	0.04	0.04
780 HV 1	49.36	49.51	0.06	0.05

Table 31

DESIGNATION	d_1	d_2	σ_H	σ_V
780 HV 3	84.59	83.78	0.11	0.17
780 HV 3	84.69	84.8	0.1	0.13
780 HV 3	83.4	82.28	0.53	0.14
780 HV 3	85.17	84.74	0.12	0.11
780 HV 3	84.52	84.79	0.11	0.13

Table 32

TIMETABLES

DESIGNATION	TIME TAKE TO REACH MAX FORCE	TIME PERIOD OF FORCE APPLICATION
260 HV 0.05	5.5	14.3
260 HV 0.05	5.4	13.8
260 HV 0.05	5.4	13.1
260 HV 0.05	5.5	13
260 HV 0.05	5.1	13.4

Table 33

DESIGNATION	TIME TAKE TO REACH MAX FORCE	TIME PERIOD OF FORCE APPLICATION
260 HV 1	12.9	12.2
260 HV 1	12.7	12.3
260 HV 1	12.1	13.1
260 HV 1	12.4	12.7
260 HV 1	11.9	13.6

Table 34

DESIGNATION	TIME TAKE TO REACH MAX FORCE	TIME PERIOD OF FORCE APPLICATION
260 HV 3	7.06	14.25
260 HV 3	6.69	14.19
260 HV 3	7.06	14.19
260 HV 3	7.06	14.31
260 HV 3	5.56	14.25

Table 35

DESIGNATION	TIME TAKE TO REACH MAX FORCE	TIME PERIOD OF FORCE APPLICATION
470 HV 0.05	4.6	13
470 HV 0.05	5.2	13.6
470 HV 0.05	4.9	12.9
470 HV 0.05	5.1	13.4
470 HV 0.05	4.9	13.4

Table 36

DESIGNATION	TIME TAKE TO REACH MAX FORCE	TIME PERIOD OF FORCE APPLICATION
470 HV 1	10.9	13.2
470 HV 1	10.6	14.5
470 HV 1	10.8	12.9
470 HV 1	10.7	13.9
470 HV 1	10.7	13.2

Table 37

DESIGNATION	TIME TAKE TO REACH MAX FORCE	TIME PERIOD OF FORCE APPLICATION
470 HV 3	6.88	14.31
470 HV 3	6.5	14.25
470 HV 3	6.94	14.31
470 HV 3	7.06	14.31
470 HV 3	6.5	14.19

Table 38

DESIGNATION	TIME TAKE TO REACH MAX FORCE	TIME PERIOD OF FORCE APPLICATION
780 HV 0.05	5.9	15
780 HV 0.05	7.1	12.3
780 HV 0.05	6.7	12.3
780 HV 0.05	6.6	12.3
780 HV 0.05	7.1	13.1

Table 39

DESIGNATION	TIME TAKE TO REACH MAX FORCE	TIME PERIOD OF FORCE APPLICATION
780 HV 1	12.6	12.5
780 HV 1	12.1	12.8
780 HV 1	12.3	12.2
780 HV 1	12.3	12.9
780 HV 1	12.2	12.7

Table 40

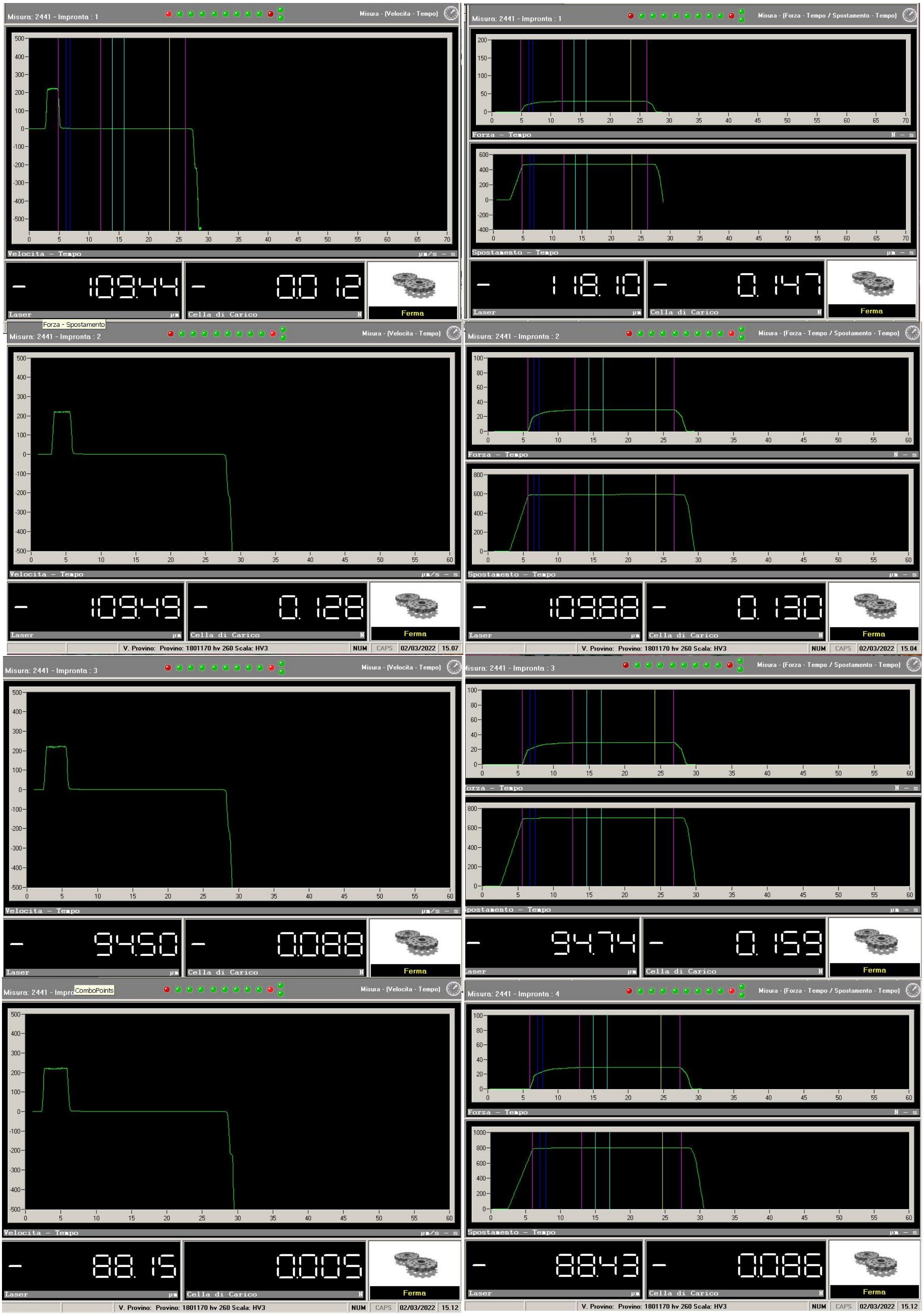
DESIGNATION	TIME TAKE TO REACH MAX FORCE	TIME PERIOD OF FORCE APPLICATION
780 HV 3	7	14.31
780 HV 3	7.06	14.06
780 HV 3	6.44	14.25
780 HV 3	6.63	14.12
780 HV 3	6.88	14.25

Table 41

DESIGNATION	THICKNESS OF THE TEST PIECE mm
260 HV	10.30
470 HV	11.46
780 HV	10.46

Table 42

Figure 27 - Usage of Handheld digital Micrometer calipers for testing the thickness of the test pieces

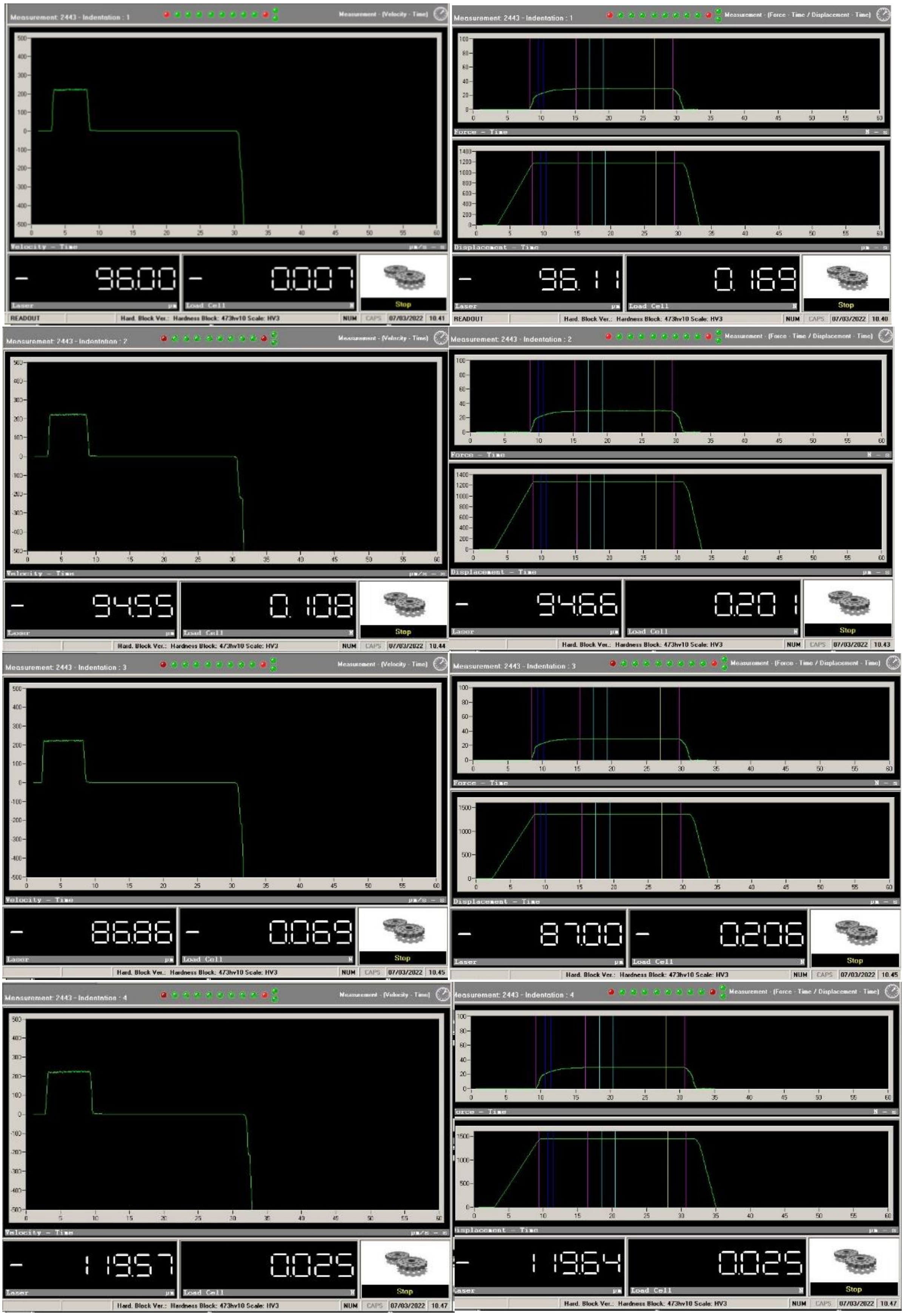

Misura: 2441 - Impronta : 1
Misura - (Velocita - Tempo)
Velocita - Tempo
µm/s - s
Laser
- 109.44
µm
Cella di Carico
- 0.0 12
N
Ferma
Misura: 2441 - Impronta : 1
Misura - (Forza - Tempo / Spostamento - Tempo)
Forza - Tempo
N - s
Spostamento - Tempo
µm - s
Laser
- 1 18. 10
µm
Cella di Carico
- 0. 147
N
Ferma
Forza - Spostamento
Misura: 2441 - Impronta : 2
Misura - (Velocita - Tempo)
Velocita - Tempo
µm/s - s
Laser
- 109.49
µm
Cella di Carico
- 0. 128
N
Ferma
V. Provino: Provino: 1801170 hv 260 Scala: HV3
NUM
CAPS
02/03/2022
15.07
Misura: 2441 - Impronta : 2
Misura - (Forza - Tempo / Spostamento - Tempo)
Forza - Tempo
N - s
Spostamento - Tempo
µm - s
Laser
- 109.88
µm
Cella di Carico
- 0. 130
N
Ferma
V. Provino: Provino: 1801170 hv 260 Scala: HV3
NUM
CAPS
02/03/2022
15.04
Misura: 2441 - Impronta : 3
Misura - (Velocita - Tempo)
Velocita - Tempo
µm/s - s
Laser
- 94.50
µm
Cella di Carico
- 0.088
N
Ferma
Misura: 2441 - Impronta : 3
Misura - (Forza - Tempo / Spostamento - Tempo)
Forza - Tempo
N - s
Spostamento - Tempo
µm - s
Laser
- 94.74
µm
Cella di Carico
- 0. 159
N
Ferma
Misura: 2441 - Impro
ComboPoints
Misura - (Velocita - Tempo)
Velocita - Tempo
µm/s - s
Laser
- 88. 15
µm
Cella di Carico
- 0.005
N
Ferma
V. Provino: Provino: 1801170 hv 260 Scala: HV3
NUM
CAPS
02/03/2022
15.12
Misura: 2441 - Impronta : 4
Misura - (Forza - Tempo / Spostamento - Tempo)
Forza - Tempo
N - s
Spostamento - Tempo
µm - s
Laser
- 88.43
µm
Cella di Carico
- 0.086
N
Ferma
V. Provino: Provino: 1801170 hv 260 Scala: HV3
NUM
CAPS
02/03/2022
15.12

Impronta	Posizione	X	Ta	Tdl	Va	Vfal
1	1 Manuale	☐	7,06	14,25	201,3	1,1
2	2 Manuale	☐	6,69	14,19	205,1	1,0
3	3 Manuale	☐	7,06	14,19	209,1	1,2
4	4 Manuale	☐	7,06	14,31	209,8	1,1
5	5 Manuale	☐	5,56	14,25	199,0	0,9

Legenda

Ta Tempo appl. carico
Tdl Tempo mantenimento carico
Va Velocità di avvicinamento
Vfal Veloc. finale di applicaz. carico

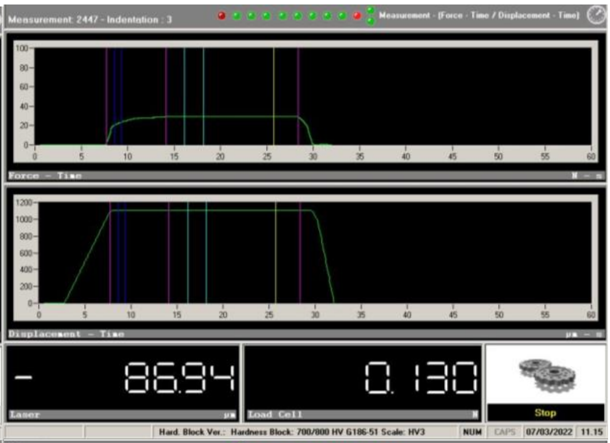
Figure 28 - Graphs along with tabulated values for the test procedure according to ISO 6507-1 for 260 HV

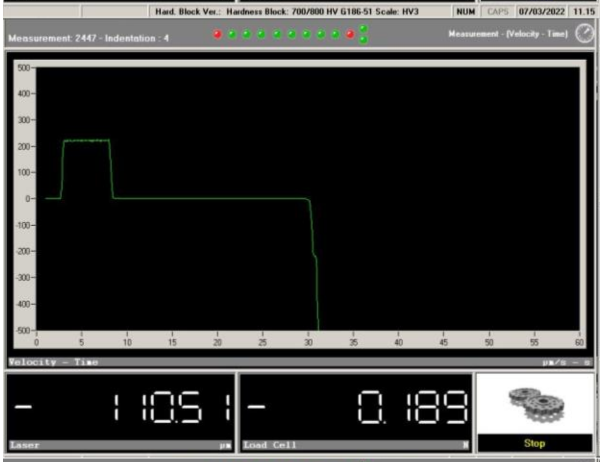

Measurement: 2443 - Indentation : 1
Measurement - (Velocity - Time)
Velocity - Time
Laser
96.00
Load Cell
0.007
Stop
Measurement - (Force - Time / Displacement - Time)
Force - Time
Displacement - Time
96.11
0.169
Hard. Block Ver.: Hardness Block: 473hv10 Scale: HV3
07/03/2022
Measurement: 2443 - Indentation : 2
94.55
0.108
94.66
0.201
Measurement: 2443 - Indentation : 3
86.86
0.069
87.00
0.206
Measurement: 2443 - Indentation : 4
119.57
0.025
119.64
0.025

Indentation	Position	X	Ta	Tdl	Va	Vfal
1	1 Manual	☐	6,88	14,31	214,9	1,0
2	2 Manual	☐	6,50	14,25	215,5	0,9
3	3 Manual	☐	6,94	14,31	214,9	1,0
4	4 Manual	☐	7,06	14,31	216,2	1,0
5	5 Manual	☐	6,50	14,19	216,5	0,9

Figure 29 - Graphs along with tabulated values for the test procedure according to ISO 6507-1 for 470 HV

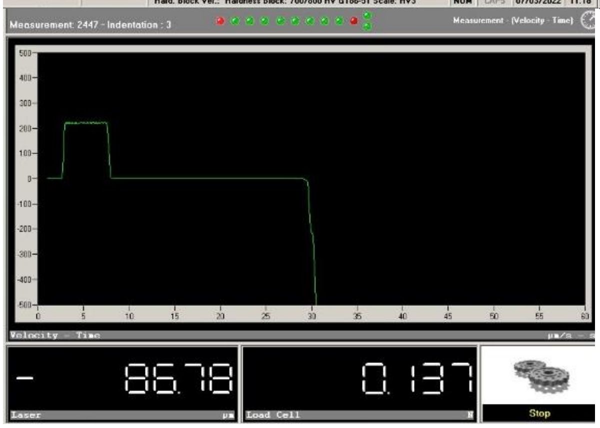

Measurement: 2447 - Indentation : 3
Measurement - (Velocity - Time)
Velocity - Time
86.78
0.137
Laser
Load Cell
Stop
Hard. Block Ver.: Hardness Block: 700/800 HV G186-51 Scale: HV3
NUM
CAPS
07/03/2022
11.15

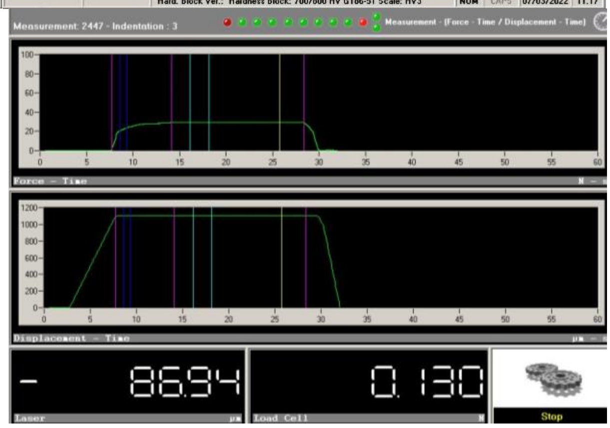

Measurement: 2447 - Indentation : 3
Measurement - (Force - Time / Displacement - Time)
Force - Time
Displacement - Time
86.94
0.130
Laser
Load Cell
Stop
Hard. Block Ver.: Hardness Block: 700/800 HV G186-51 Scale: HV3
NUM
CAPS
07/03/2022
11.15

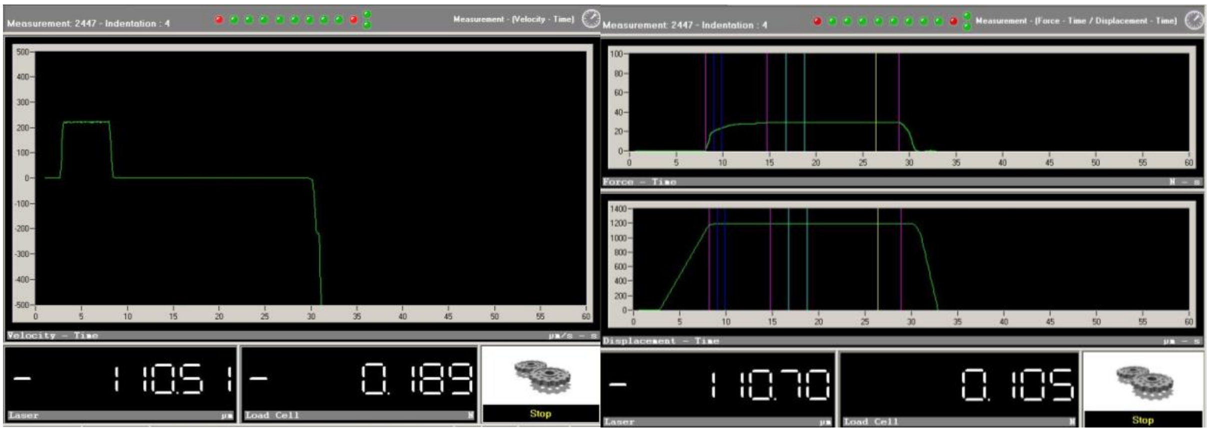

Measurement: 2447 - Indentation : 4
Measurement - (Velocity - Time)
Velocity - Time
110.51
0.189
Laser
Load Cell
Stop

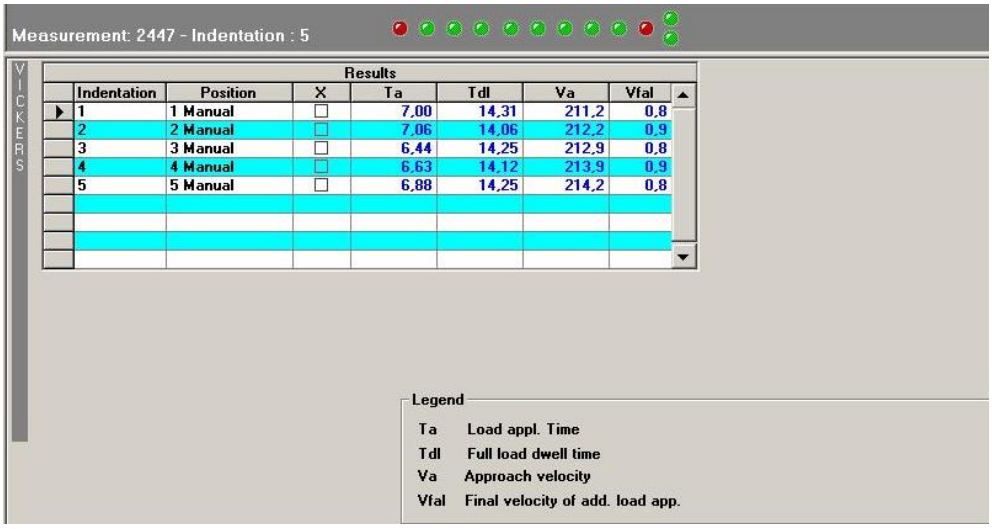

Measurement: 2447 - Indentation : 5
Measurement - (Velocity - Time)
Velocity - Time
98.73
0.074
Laser
Load Cell
Stop
Hard. Block Ver.: Hardness Block: 700/800 HV G186-51 Scale: HV3
NUM
CAPS
07/03/2022
11.18


Measurement: 2447 - Indentation : 5
Measurement - (Force - Time / Displacement - Time)
Force - Time
Displacement - Time
98.99
0.056
Laser
Load Cell
Stop
Hard. Block Ver.: Hardness Block: 700/800 HV G186-51 Scale: HV3
NUM
CAPS
07/03/2022
11.17


Measurement: 2447 - Indentation : 3
Measurement - (Velocity - Time)
Velocity - Time
86.78
0.137
Laser
Load Cell
Stop


Measurement: 2447 - Indentation : 3
Measurement - (Force - Time / Displacement - Time)
Force - Time
Displacement - Time
86.94
0.130
Laser
Load Cell
Stop





Indentation	Position	X	Ta	Tdl	Va	Vfal
1	1 Manual	☐	7,00	14,31	211,2	0,8
2	2 Manual	☐	7,06	14,06	212,2	0,9
3	3 Manual	☐	6,44	14,25	212,9	0,8
4	4 Manual	☐	6,63	14,12	213,9	0,9
5	5 Manual	☐	6,88	14,25	214,2	0,8

Legend

Ta	Load appl. Time
Tdl	Full load dwell time
Va	Approach velocity
Vfal	Final velocity of add. load app.

Figure 30 - Graphs along with tabulated values for the test procedure according to ISO 6507-1 for 780 HV

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