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Calibration of Vickers Hardness Test Equipment Using Standard Block (Case study in PT. Tenaris SPIJ, Cilegon)

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ABSTRACT

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A calibration is an activity to determine the conventional validity of the appointment value of measuring instruments and measuring materials by comparing them to standard measurements that are traceable to national and international standards for measurement units and or international and certified reference materials. The calibration is generally a process of adjusting the output or indication of a measurement device to match the magnitude of the standard used in certain accuracy. The purpose of this paper is to find out the vickers hardness test calibration whether the machine running normally or there is a problem with the device. A case study was conducted in PT. Tenaris SPIJ, Cilegon, Indonesia. A vickers hardness testing in the case study has its own reference standard, namely ISO 6507-2: 2018 Metal material - Hardness Test Verification and Calibration of the test machine. The vickers hardness test equipment is a machine to conduct tests using a diamond pyramid with a plane angle of 136°, by means of an emphasis by an indentor that results in a trace or indentation on the surface of the test object. The vickers hardness testing is abbreviated as HV or HVN and also known as Diamond Pyramid Hardness Test (DPH). Result of calibration in the case study was found the repeatability value on vickers hardness testing machine of 32 HV 10 and the error value of $\pm 3\%$.

KEY WORDS: Calibration, Vickers hardness test, Standard block

1.0 INTRODUCTION

A calibration is a series of activities that make up the relationship between the value indicated by the measuring instrument or measurement system or the value represented by the measuring material, which known values relating to the amount measured under certain conditions [1]. Calibration is very important in the industrial world due to its function can determine the condition of the goods whether suitable or not for using [2]. The calibration may employ a certain standards such as national and international standards [1]. After the calibration is done the equipment awarded a calibration certificate.

A calibration should be performed using the latest reference standards. Because the latest reference standards are revised to ensure the truth of the previous standards [3]. A crucial aspect to minimize the inaccurate measurements and knowing the value of measurement uncertainty to be correction in instrumentation design [4,5]. The instrument calibration is one of quality control to checks performing of measurement uncertainty to be correction.

In this paper is conducted a calibration of a vickers hardness test equipment. A vickers hardness testing is a machine used to conduct tests that use a diamond pyramid with an angle of 136 degrees [6]. The vickers hardness testing machines must be calibrated every year [1]. The purpose of vickers hardness test calibration is to ensure the equipment running normal condition. The vickers hardness testing in this paper has employed the standard reference, namely ISO 6507-2: 2018 Metal material -Hardness Test Verification and Calibration of the test machine [6,7]. The reference standard on the Vickers hardness testing machine is a standard issued in 2018 [6,7], in the year before 2018, on the Vickers hardness calibration machine has 2 reference standards namely ISO 6507-2: 2005 [8] Metal Material-Hardness Test Vickers Hardness Test Verification. and PC-310-01: 2006 General Guidelines for Presentation of Calculation of Measurement Uncertainty [9].



1.1 Hardness Test

Hardness is one of the mechanical properties of a material. The hardness of a material must be known especially for material that in its use will experience friction (friction force) and plastic deformation [6]. Plastic deformation itself is the state of the material when the material is given with force then the micro structure of the material can no longer return to its original form, meaning that the material cannot be reshaped. In short, violence is defined as the ability of a material to withstand the burden of identification or penetration (emphasis).

Hardness Test is the most effective test to test the hardness of a material, because with this test we can easily find out the description of the mechanical properties of a material. Several factors can influence hardness testing such as [10-16]: (1) instrument factors i.e. load, indentation, indenter; (2) material: heterogeneity of the microstructure, quality of specimen preparation, specimen surface, type of material, material treatment, shape of material; (3) measurement procedure and operators; (4) environment: temperature, humidity, vibrations etc. Although the measurement is only done in certain areas, the value of harness test is valid enough to express the strength of a material. By conducting a hardness test, the material can be easily classified as ductile or brittle material.

In general, hardness testing uses 4 types of harness methods, namely [6,7]:

1. Brinnel (HB / BHN)

Hardness testing using the Brinnel method aims to determine the hardness of a material in the form of material resistance to the ball (identifier) which is stressed on the surface of the test material (specimen). Ideally, the Brinnel test is intended for materials that have rough surfaces with strength tests ranging from 500-3000 kgf. Identifiers (steel balls) are usually hardened and made or made from Tungsten Carbide.

2. Rockwell (HR / RHN)

Hardness testing by the Rockwell method aims to determine the hardness of a material in the form of material resistance to the identifier in the form of a steel ball or diamond cone that is stressed on the surface of the test material.

- Micro Hardness (Knoop hardness) Microhardness test or Knoop Hardness test is a suitable test for testing materials with low hardness values. Knoop is usually used to measure fragile materials such as ceramics.
- 4. Vickers (HV / VHN)

Hardness testing using the Vickers method aims to determine the hardness of a material in the durability of the material against a diamond identifier that is quite small and has a geometric shape in the shape of a pyramid. The burden imposed is also quite small compared to Rockwell and Brinell tests, which is between 1 to 1000 grams. Vickers (HV) hardness level is defined as the quotient (coefficient) of the test load (F) with the compressed (jumping) surface area of the identifier (diagonal) multiplied by sin (136°/ 2).

1.2 Vickers Hardness Testing Machine

Vickers Hardness Test Machine is a machine used to conduct tests using a diamond pyramid with a field angle of 136° , by means of an emphasis by an identifier that produces a trace or indentation on the surface of the test object. Hardness testing machines are often needed in technical activities to determine the mechanical properties of a material [6,7].

Vickers hardness level is defined as the load divided by the surface area of the curve. In practice, this area is calculated from microscopic measurements of the diagonal trace length. The vickers hardness testing uses only one type of identification, pyramid-shaped identity that can be used to test almost all types of metals ranging from soft to hard. Vickers hardness testing is usually abbreviated as HV or HVN and also known as Diamond Pyramid Hardess Test (DPH). In general there are 3 types of trace shapes (indentations) produced by the emphasis of the indenter, namely the perfect square shape, the pillow shape and the trace shape of the barrel [7].



Figure 1: Form of penetration traces [7]

1.3 Vickers Hardness Testing Machine Components

Vickers hardness testing machine has components to support its operating system, where each component has a different important role and function. The components in the vickers hardness testing machine is depicted in Figure 2 [7].



Figure 2: Vickers hardness testing machine components [7]

1.4 Calibration

According to ISO / IEC Guide 17025: 2005 [8] and Vocabulary of International Metrology (VIM) Calibration is a series of activities that form the relationship between the values indicated by the measuring instrument or measurement system or the value represented by the measuring material, with values known to be related to the amount which is measured under certain conditions. In other words calibration is an activity to determine the conventional validity of the value of the appointment of the measuring instrument and measure the material by comparing it with standard measurements that can be tracked (treaceable) with



national and international standards for measurement units and or international and certified reference materials. Calibration is generally the process of adjusting the output or indication of a measurement device to fit the magnitude of the standard used in a certain accuracy. For example, a thermometer can be calibrated so that error indications or corrections can be determined and adjusted through a calibration constant, so that the thermometer shows the actual temperature in Celsius at certain points on the scale.

In several countries, including Indonesia, has a national metrology institute (National Metrology Institute). In Indonesia there is a research center for Instrumentation and Metrology Calibration (Puslit KIM LIPI) which has the highest measurement standards (in SI and its derivative units) which will be used as a reference for calibrated devices [6]. KIM LIPI Puslit also supports metrological infrastructure in one country and often other countries by setting high or international standard measurement chains with the tools used. The calibration results must be accompanied by a "traceable uncertainty" statement to determine the level of confidence that is carefully evaluated with uncertainty analysis [7]. The role of calibration in industrial activities is one of the quality assurance benchmarks of a product, so that all measuring instruments (instrumentation) and measurement materials really need to be calibrated, according to the requirements of applicable standards or technical specifications [1,7].

1.5 Force

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Force is any interaction that can cause a mass object to change in motion, both in the form of direction and geometric construction. Force can also be said as a pull or push made by an object against another object. Force is a vector quantity that has a quantity, capture point, and line of force. In other words, force can cause objects with a certain mass to change their speed (including moving from a stationary position), or accelerating or changing shape. Force has magnitude and direction, so it is a vector quantity. The SI unit used to measure the force is Newton (denoted by N) own style is symbolized by the symbol F [8].

1.6 Vickers Hardness Testing Machine Calibration

Basically, hardness testing machine calibration is the same as other machine calibration. The difference is the standard used to make the tools used differently. The purpose of the hardness testing machine is to calibrate to compare the size of the measured instrument with the value of the measured measuring instrument. The requirements to calibrate the vickers hardness testing machine are as follows [7]:

- a. Make sure the test machine is functioning properly.
- b. Make sure the room temperature is well controlled.
- c. For penetrators used a diamond pyramid with an angle of 136 ± 0.50 C.
- d. Make sure the base to place the standard block in the water level position using the water fitting.
- e. Make sure the standard blocks are suitable, clean and have a stable temperature.
- f. Determination of resolution of hardness testing machines. Analog scale

If the hardness measurement system uses an analog scale, for example dial dial, micrometer then the resolution (r) must be determined as follows:

- a) If the scale interval is greater than 1.25 mm then the resolution (r) is estimated at 1/10 of the scale interval.
- b) If the interval is smaller than 1.25 mm, then the resolution (r) is estimated to be 1/5 of the scale.
- Digital scale
 - If the hardness measurement system uses a digital scale then the resolution (r) is estimated based on half of the numbers that change, but not more than one digit change.

2.0 EXPERIMENTAL METHOD

This research was conducted at PT. Tenaris Seamless Pipe Indonesia Jaya Cilegon, and before performing the calibration, it is better to understand the methods before carrying out the calibration process, as for the reference method or standard in the calibration process using ISO 6507-2: 2018 Metallic materials-Hardness Test-Verification and Calibration of testing machines [7]. The standard is the latest standard in carrying out the process of hardness testing machine calibration, and when you want to do the calibration should use the latest standards.

- The tools and materials used before calibration are as follows:
- Vickers hardness testing machine to be calibrated
- Standard Blocks
- Water pas.
- Thermometer, 0 to 500 C or greater, resolution: ± 1.00 C
- Stopwatch, 1 digit resolution = 1/100 second
- Worksheets

After preparing the tools and materials, the procedure for calibrating the vickers hardness testing machine is as follows:

- 1. Prepare the test machine in working condition.
- 2. Press the power button to start the engine
- 3. Place the standard block on the base of the solid hardness testing machine, the surface of the standard block to be penetrated in a flat position and perpendicular to the axis of penetration according to the instrument user manual.
- 4. Bring the penetrator close to touch the surface of a standard block, apply the same force as F, without shock or vibration, the time required to reach the initial (minor) force F is between 2 to 8 seconds.



Figure 3: Standard blocks



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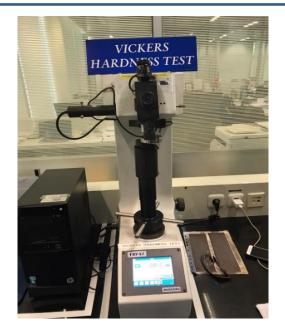


Figure 4: Prepare vickers hardness testing machine

- 5. The strength of time F is not less than 10 seconds and not more than 15 seconds.
- 6. Remove the force F and measure the center (diagonal) lines d11 and d12 from the penetration of the diamond pyramid.
- 7. Record the Vickers hardness value for the appropriate center line of penetration and calculate the hardness value using the equation [7,10]:

$$HV = 0.102 \frac{2F\sin(\alpha - 2)}{d^2}$$
$$= 0.102 \frac{1.8544 F}{d^2}$$

Note: α = angle between the pyramid of diamonds 1360 F = penetration force (N)

d = average diagonal distance (d11 + d12)/2

- 8. Do at least (minimum) twice the initial hardness measurement with a standard block to ensure that the machine is working properly, penetrator and grounding are in the correct position. The results of starting measurements can be ignored.
- 9. Penetrate five times for each standard block.
- 10. The distance between the two centers of penetration is a minimum of 4 times the diameter of the penetration mark but must not be less than 2 mm. The distance between the center of penetration with the side of a standard block is not less than 2 times the diameter.

2.1 Data Collection Method

The following is a method of data retrieval carried out by the author at PT. Tenaris Seamless Pipe Indonesia Jaya Cilegon as follows:

• Literature Study, by preparing theories used in the formulation of problems and working on mathematical calculations.

- Observation, by directly observing the research objects to be analyzed.
- Interview, with discussion and question and answer directly to the field supervisor and staff and employees.

2.2 Scope of work

This method explains the vickers hardness testing machine with an indirect method using a standard block. This method is also used to check between using vickers hardness calibration machines.

2.3 Reference Standard

The standard of reference was employed in this paper:

- ISO 6507-2: 2005 Metallic Materials Hardness Test Verification of Vickers Hardness Test [8].
- PC-310-01: 2006 General Guidelines for Presentation of Calculation of Measurement Uncertainty [9].
- ISO 6507-2: 2018 Metallic Materials Hardness Test Verification and Calibration of testing machines [7].

3.4 Calibration Preparation

Preparations that must be made before performing the calibration were as follows:

- 1. Make sure the test machine is in good working condition and calibration is carried out under controlled room temperature conditions, with a temperature of 23 ± 50 C. If the calibration is carried out outside the specified temperature range, it must be recorded in the calibration report.
- 2. The nominal force used in the Vickers hardness test can be seen in Table 1.

SYMBOL OF VIOLENCE	NOMINAL TEST STYLE, F					
	Ν					
HV 0,2	1,961					
HV 0,3	2,942					
HV 0,5	4,903					
HV 1	9,807					
HV 2	19,61					
HV 2,5	24,52					
HV 3	29,42					
HV 5	49,03					
HV 10	98,07					
HV 20	196,1					
HV 30	294,2					
HV 50	490,3					
HV 100	980,7					

Table 1: Nominal force hardness test [7]

- 3. For penetrators used a diamond pyramid with an angle of 136 \pm 0.50 C.
- 4. If the machine can work with many test styles, then for the two calibration test styles chosen, one of the test styles is the force often used for testing. For each strength test, two different standard blocks are used with the following measurement areas:

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- 225 HV
- 400 to 600 HV
- 700 HV
- If the machine only works with one test force, calibration is recommended using three standard blocks with a hardness value.
- 6. In special use, the hardness testing machine can be calibrated to a hardness value close to the hardness test value.
- 7. Make sure the foundation to put a standard block in the position of the water level using water installation.
- 8. Prepare the appropriate standard hardness block, clean it from dirt and grease and the condition is \pm 30 minutes at ambient temperature so that it reaches a stable temperature.
- 9. Install the appropriate penetrator on the hardness testing machine. The penetrator axis is in line with the appropriate load axis.
- 10. Prepare the hardness testing machine calibration worksheet in accordance with point 9.
- 11. Record hardness testing machines, standard blocks, and room temperature during calibration.

3.0 RESULT AND DISCUSSION

3.1 Repeatability

For each standard block value d1, d2, d3, d4 and d5 are obtained from an average of 2 diagonals; the posts are sorted from minimum to maximum. The repetition of the test machine is: d5 d1.

 ✓ Example of observational data using vickers NO standard hardness block. EP 9931358 Hardness value 526 HV 10. Known: 5 observational data: d1 = 0.188 mm, H1 = 525 HV 10 d2 = 0.189 mm, H2 = 519 HV 10

d3 = 0.188 mm, H3 = 525 HV 10 d4 = 0.188 mm, H4 = 535 HV 10 d5 = 0.188 mm, H5 = 525 HV 10

$$d = \frac{d1 + d2 + d3 + d4 + d5}{5}$$

= 0,1882 mm, then the value of H = 524 HV 10

 ✓ Calculate Repeatability Repeatability = H maks - H min = 525 - 519 = 6 HV 10

In percent
$$= \frac{a}{H \text{ std}} \times 100\%$$
$$= \frac{6}{526} \times 100\%$$
$$= 1.14\%$$

Terms > 400 HV 10 =
$$0.03 \text{ x d x 2}$$
 (Table 2)
= 0.03 x H std x 2
= 0.03 x 526 x 2
= 32 HV 10

✓ The repeatability of the calibrated hardness testing machine is depicted in Table 2.

Table 2: Repeatability	maximum hardness	s testing machine
------------------------	------------------	-------------------

	1	2			0					
Standard	Repeatability Maximum Hardness Testing Machine									
Block	HV 5	HV 0,2		HV 0,2	HV 5					
Violence	to	to	<hv 0,2<="" td=""><td>to</td><td>to</td></hv>	to	to					
violence	HV 100	<h5< td=""><td></td><td><hv 5<="" td=""><td colspan="2">HV 100</td></hv></td></h5<>		<hv 5<="" td=""><td colspan="2">HV 100</td></hv>	HV 100					
< 225 HV	0,03 d	0,06 d	0,06 d	12 at 100	6 at 100					
				24 at 200	12 at 200					
> 225 HV	0,02 d	0,04 d	0,05 d	20 at 250	10 at 250					
				28 at 350	14 at 350					
				48 at 600	24 at 600					
				60 at 750	30 at 750					

Repeat = $(a1 \times b1) \times 2$

where 1 = Terms repeatability

b2 = Standard block hardness value

3.2 Error

The error of the hardness testing machine is $\overline{H} - H$

$$\bar{H} = \frac{H1 + H2 + H3 + H4 + H5}{r}$$
(1)

Where:

- Hi = until H5 is the hardness value corresponding to d1 to d5
- \overline{H} = hardness value of a standard block
- d = diagonal results of penetration
- ✓ Example Calculation of error Known : $\overline{H} = 524 \text{ HV } 10$ H = 526 HV 10

$$\begin{array}{l} \text{Error} = \overline{H} - \text{H std} \\ \text{Error} = 524 - 526 = -2 \text{ HV } 10 \end{array}$$

(2)

In Percent =
$$\frac{B}{H \text{ std}} \times 100 \%$$
$$= \frac{-2}{526} \times 100 \%$$
$$= 0.38 \%$$

Terms of error $= \pm 3 \%$

The maximum error of the vickers hardness testing machine expressed in percent against the standard block hardness value can be seen in Table 3.

3.3 Measurement Uncertainty

For calculation of measurement uncertainty see the general guidelines for testing the uncertainty method PC-310-01-1997.



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Table 3: Maximum error of vickers hardness testing mach	ine
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Violent	Th	ie max	imum	allowa	able ha	ardnes	s erro	is ex	presse	d in p	ercent	hardr	ess of	standa	rd HV b	locks
symbol	50	100	150	200	250	300	350	400	450	500	600	700	800	900	1000	1500
HV 0,01																
HV 0,015	10															
HV 0,02	8															
HV 0,025	8	10														
HV 0,05	6	8	9	10												
HV 0,1	5	6	7	8	8	9	10	10	11							
HV 0,2		4		6		8		9		10	11	11	12	12		
HV 0,3		4		5		6		7		8	9	10	10	11	11	
HV 0,5		3		5		5		6		6	7	7	8	8	9	11
HV 1		3		4		4		4		5	5	5	б	6	6	8
HV 2		3		3		3		4		4	4	4	4	5	5	6
HV 3		3		3		3		3		3	4	4	4	4	4	5
HV 5		3		3		3		3		3	3	3	3	3	4	4
HV 10		3		3		3		3		3	3	3	3	3	3	3
HV 20		3		3		3		3		3	3	3	3	3	3	3
HV 30		3		3		2		2		2	2	2	2	2	2	2
HV 50		3		3		2		2		2	2	2	2	2	2	2
HV 100				3		2		2		2	2	2	2	2	2	2

Example of calculating calibration uncertainty on a calibrated device as following:

- 1. Analysis Type A (µa)
 - Obtained 5 observational data and calculate the standard deviation (σ) using the equation:

$$\sigma = \frac{\sqrt{\Sigma (\text{Hi} - \overline{H})^2}}{n-1}$$
where: Hi = hardness value to -1
 \overline{H} = average hardness value
N = amount of data (3)

$$\sigma = \frac{\sqrt{(525-524)^2 + (519-524)^2 + (525-524)^2 + (525-524)^2 + (525-524)^2}}{5-1}$$

$$= 2.69HV10$$
then:

$$\mu a = \frac{C}{\sqrt{n}}$$

$$= 1.20 \text{ HV } 10$$
In Percent = $\frac{D}{H \text{ std}} \text{ x100 } \% = 0.228\%$
(4)

- 2. Analysis Type B (µb)
- a. On a standard calibration / block tool (μ b1) In the certificate stated U95 (CL, k=2) = ± 1.0 % Then:

$$\mu b1 = \frac{E}{Coverage Factor (k)}$$

In Percent = 0.50 %

b. On the hardness testing machine indicator (µb2) Readibility: 0,001 unit Distribution Rectangular (a) = ± 0,001 unit Uncertainty of hardness testing machines $\mu b2 = \frac{a}{\text{Scale of measure}}$ = 0,0005 unit

In Percent =
$$\frac{F}{\text{Scale of measure}} \times 100\%$$

= 0.025%

c. On the influence of temperature (temperature changes) (μ b3) After the stabilization process (conditioning) the temperature difference between the standard block and the hardness testing machine is in the range \pm 0.2°C and the average value of the coefficient of thermal expansion of the standard block is 11.5 x 10⁻⁶ ° C⁻¹. Then:

$$\mu b3 = \frac{\pm 0.2}{\sqrt{3}} = 1.15^{\circ} C$$

With the sensitivity coefficient obtained from the differential mathematical models = $1.7 \ \mu m^{\circ} C^{-1}$ Then $\mu b3 = 11.5 \ x \ 10^{-6} \circ C^{-1} x \ 1.15 \circ C \ x \ 100\% = 0.00132 \ \%$

- $1100 \ \mu 05 = 11.5 \ \kappa 10 \quad C \quad \kappa 11.15 \quad C \quad \kappa 100 \ \kappa = 0.00152 \ \kappa$
- 3. Uncertainty obtained from the above calculation is made in Table 4.

Type Uncertainty A (%)	Type Uncertainty B (%)
	1 = 0,50
1 = 0,22814	2 = 0,025
	3 = 0,00132

a. Uncertainty combined μc

$$\mu c = \sqrt{(\text{Tiper A})^2 = (\text{Tipe B})^2}$$

= $\sqrt{(0.22814)^2 + [(0.50)^2 + (0.025)^2 + (0.00132)^2]}$
= 0.50063%

b. Uncertainty expanded U95

In this method the coverage factor value (k) is assumed to be 2.0, for a 95% confidence level approach with intervals y-u to y+u or y±u, then the uncertainty is expanded U95 = k . μ c. U95 = 2.0 x 0.50063 = 1.00125 %

c. Writing on the report The writing on the report is rounded to two decimal places, then U95 = 1.00%

4.0 CONCLUSION

(5)

The conclusion is obtained to calibration of vickers hardness test equipment using standard block in *Balai Besar dan Barang Teknik (B4T)*: the reference standard used in calibrating vickers hardness testing machines is ISO 6507-2: 2005, PC-310-01: 2006, and ISO 6507-2: 2018. The result of repeatability value on vickers hardness testing machine is 6 HV 10, in percent of 1.14 and terms repeatebality of > 400 HV 10 = 32 HV 10. The error value on vickers hardness testing machine is minus 2 HV 10, in percent of 0.38% and term of errors is ± 3 .

The measurement uncertainty value has several analyzes namely: analysis type A (μ a), σ = 2.69 HV 10, μ a = 1.20 HV 10, in percent = 0.228 %. Analysis type B (μ b) consists of on a standard calibration / block tool (μ b1) is in percent = 0.50 %. On the hardness testing machine indicator (μ b2) is 0.0005 unit=



0.025%. On the influence of temperature (temperature changes) (μ b3) is 1.15°C. The uncertainty obtained is μ c = 0.50063% and uncertainty expanded U95 is 1.00%.

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