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CALIBRATION OF TEST DEVICE FOR MICROHARDNESS TESTING

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ABSTRACT
Proper calibration of the device is important to prevent potential sources of error, which ensures that the test results most effectively represent the value of the sample being tested.
In microhardness testing, the loading force does not exceed 1.96 N. The test surface requires a more complex metallographic preparation, and the smaller the injection force, the surface must be finer prepared. Precision measuring microscopes with an accuracy of ± 0,5μm are used to measure the size of the prints. Calibration of the Zwick Microhardness Testing Device of the Kemal Kapetanović Institute Metallographic Laboratory was performed for measuring ranges HV 0.3. The permissible deviation of the device including the measurement uncertainty shall meet the requirements of the standard for this area.

1. INTRODUCTION
Device calibration is one of the primary processes used to maintain the precision of the device, ie the process of configuring the device to give a sample result within an acceptable range. [1]
Understanding the calibration of devices and their proper use is an essential element during laboratory activities.
The primary goal of calibration, and therefore the measurement traceability of the device, is ensuring that the measurement is consistent within the manufacturer of the product and the customer who incorporates the product, that is, there must be a guarantee that the manufacturer and the customer use "the same measure" [1].

The purpose of micro-indentation hardness testing is to accurately calculate changes in hardness, intentional or accidental, in the test material. This test technique is known as microhardness testing; although the term is inappropriate because it gives the impression that these are very small values of the measured hardness, which is not the case.

For the measurement of microhardness are used loads from 0.09807N to 1.961 N, for microhardness measuring microstructural constituents of a material. The load time of the indenter is typically 10 to 15 seconds, but it can be longer for some materials [2].

2. DEVICE FOR MICROHARDNESS TESTING

When considering hardness as a measuring size, all devices for measuring hardness, according to the traceability hierarchy, can be classified into the following groups:
- hardness testers,
- reference blocks,
- primary reference blocks.

The purpose of hardness testing by micro-indentation is to accurately calculate changes in hardness, intentional or incidental, in the test material.
In microhardness testing, the loading force does not exceed 1.96 N. The test surface requires more complex metallographic preparation, and the smaller force indentation, the test surface must be finer prepared.
Precision measuring microscopes are used to measure the size of imprint, with a standard magnification of several hundred times, with an accuracy of ± 0.5μm. Microhardness measurement requires extensive experience and precision to achieve satisfactory measurement accuracy [2].

2.1. Testing devices for measuring microhardness

The basic components of each hardness tester are:
- a mechanism for producing adequate load force,
- a part for measuring the length of the imprints.

Therefore, the basic components of a device are considered to be the load and measurement systems that are most commonly connected to one device [2].
The basic elements of a microhardness tester are: stand, loading mechanism, an indentation.

Figure 1.Zwick devices for measuring microhardness testing

The load systems of microhardness measuring devices differ primarily in the manner in which the required load is achieved. Today, weight systems are the most widely used, and the transfer of load to the press is achieved through the direct action of the weights [2].
The Vickers microhardness indenter is a diamond quadrilateral pyramid. Different measuring microscopes are most commonly used to read the size of the indent.

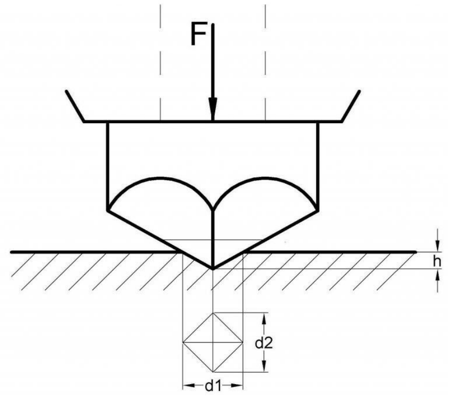

Figure 2: Schematic representation of the print

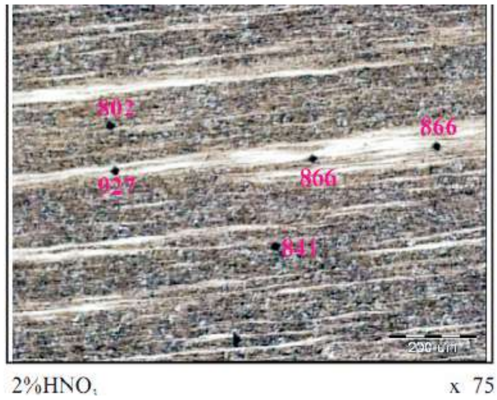

Figure 3. The appearance of the microhardness imprint on the steel sample

3. CALIBRATION OF DEVICE FOR MICROHARDNESS TESTING

Calibration is an operation that establishes a relationship between force values
(with associated uncertainties) shown by the test device and measured with one or more reference blocks [1].
Understanding the calibration of instruments and their proper use is an essential element in the overall laboratory activities. Proper calibration will ensure that the equipment stays within valid performance limits to accurately report test results.
Proper calibration of the device is important to prevent potential sources of error, which ensures that the test results most effectively represent the value of the tested sample [3].
Device calibration is one of the primary processes used to maintain the precision of the device, ie the process of configuring the device to give a sample result within an acceptable range. The Vickers hardness testers are calibrated according to the requirements of the standard; BAS EN ISO 6507-2:2018 [4].
Calibration standards for hardness testers require direct and indirect verification.
Direct device verification includes:
- calibration of the test force,
- verification of the indenters,
- calibration of the measuring system,
- verification of the test cycle.

Direct verification is required when installing a test fixture if the indirect verification period is longer than 14 months after service and repair of the fixture.Indirect verification is performed every 12 months with hardness reference blocks. Five indents shall be made on each hardness reference blocks selected in accordance with the requirements of the hardness test method.

3.1 Condition control of calibration items

Before approaching calibration, the calibration team shall check the calibration object in accordance with BAS EN ISO 6507-2:2018 [4]., and it shall be determined that the test force can be applied and removed without shock or vibration without affecting the reading .
In case the measuring system is part of the device it is necessary that:
- the change in test force does not affect the measurement when reading,

- change from removal of test force to measurement of hardness does not affect the reading,
- illumination does not affect reading,
- center of indent in the center of the field of view.

Laboratory has certificate of calibration of indenter and a certificate of the first calibration of the device. Calibration is performed with certified hardness blocks that have been calibrated according to the requirements of BAS EN ISO 6507-3:2018 [5]. whose calibration certificate has not expired. When the validity of the calibration certificate expires, the standard blocks are kept for the purpose of checking the technical correctness of the device, and new certified standard blocks are purchased for the calibration of the device. [3]

3.2 Performing a calibration

Indirect calibration is done with reference blocks in the temperature inerval (23 ± 5)° C, and if it is outside this temperature limit it must be recorded in the calibration certificate. Before checking the device, laboratory personel performs a measurement system check, ie a reference indent is measured on each reference blocks. The difference between the mean measured value and the certified mean value shall not exceed the maximum permissible error according to BAS EN ISO 6507-2:2018 [4]. The measured values, together with the calibration results, shall be recorded in the appropriate record and in the calibration certificate.

3.3 Determination of relative errors

The determination of the relative error of accuracy and repeatability shall be made in accordance with point 5 of BAS EN ISO 6507-2:2018 [4].. The obtained values of the relative errors of accuracy and repeatability shall be recorded in the records and in the calibration certificate together with the uncertainty of the results of the checks. For each reference block, the mean is determined by the readings of the indent:

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

repeatability:

$$r_{rel} = \frac{d_5 - d_1}{\bar{d}} x100, (\%)$$

and calibration device error:

$$E_{rel} = \frac{\bar{H} - H_c}{H_c} x100, (\%)$$

where Hc is the hardness of the reference blocks.

$$\bar{H} = \frac{H_1 + H_2 + H_3 + H_4 + ... + H_n}{n}$$

Repeatability: r = Hn-H1

Error: $b = \bar{H} - H_{CRM}$

Where H1, H2, H3, H4,…, Hn are the hardness values of all indents.
n-total number of indents
HCRM-certified reference blocks hardness

Therefore, we can conclude that proper and timely calibration of the device can increase productivity, optimize resources, ensure comparability and compatibility of the product and its acceptability anywhere in the world.

4. CONCLUSION

Proper calibration of the device is important to prevent potential sources of error, which ensures that the test results most effectively represent the value of the tested sample.
Proper calibration will ensure that the equipment remains within valid performance limits to accurately report test results.
In microhardness testing, the loading force does not exceed 1.96 N. The test surface requires more complex metallographic preparation, and the smaller the force indentation, the surface must be finer prepared. Precision measuring microscopes are used to measure the size of indent, with a standard magnification of several hundred times, with an accuracy of ± 0.5μm. Microhardness measurement requires extensive experience and precision to achieve satisfactory measurement accuracy.We can conclude that proper and timely calibration of the device can increase productivity, optimize resources, ensure comparability and compatibility of the product and its acceptability anywhere in the world, and its proper use is an essential element in the overall laboratory activity.

5. REFERENCES

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