SLOVENSKI STANDARD
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Kovinski materiali - Instrumentirano vtiskanje pri preskušanju trdote in drugih lastnosti materialov - 2. del: Overjanje in kalibriranje preskuševalnih strojev (ISO/DIS 14577-2:2024)

Metallic materials - Instrumented indentation test for hardness and materials parameters - Part 2: Verification and calibration of testing machines (ISO/DIS 14577-2:2024)

Metallische Werkstoffe - Instrumentierte Eindringprüfung zur Bestimmung der Härte und anderer Werkstoffparameter - Teil 2: Überprüfung und Kalibrierung der Prüfmaschinen (ISO/DIS 14577-2:2024)

Matériaux métalliques - Essai de pénétration instrumenté pour la détermination de la dureté et de paramètres des matériaux - Partie 2: Vérification et étalonnage des machines d'essai (ISO/DIS 14577-2:2024)

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Metallic materials — Instrumented indentation test for hardness and materials parameters —

Part 2: **Verification and calibration of testing machines**

Matériaux métalliques — Essai de pénétration instrumenté pour la détermination de la dureté et de paramètres des matériaux —

Partie 2: Vérification et étalonnage des machines d'essai

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 164, *Mechanical testing of metals*, Subcommittee SC 3, *Hardness testing*.

This third edition cancels and replaces the second edition (ISO 14577-2:2015), which has been technically revised.

ISO 14577 consists of the following parts, under the general title *Metallic materials — Instrumented indentation test for hardness and materials parameters*:

— *Part 1: Test method*

— *Part 2: Verification and calibration of testing machines*

— *Part 3: Calibration of reference blocks*

— *Part 4: Test method for metallic and non-metallic coatings*

— *Part 5: Linear elastic dynamic instrumented indentation testing (DIIT)*

Introduction

Hardness has typically been defined as the resistance of a material to permanent penetration by another harder material. The results obtained when performing Rockwell, Vickers, and Brinell tests are determined after the test force has been removed. Therefore, the effect of elastic deformation under the indenter has been ignored.

ISO 14577 (all parts) has been prepared to enable the user to evaluate the indentation of materials by considering both the force and displacement during plastic and elastic deformation. By monitoring the complete cycle of increasing and removal of the test force, hardness values equivalent to traditional hardness values can be determined. More significantly, additional properties of the material, such as its indentation modulus and elasto-plastic hardness, can also be determined. All these values can be calculated without the need to measure the indent optically. Furthermore, by a variety of techniques, the instrumented indentation test allows to record hardness and modulus depth profiles within a, probably complex, indentation cycle.

ISO 14577 (all parts) has been written to allow a wide variety of post test data analysis.

Metallic materials — Instrumented indentation test for hardness and materials parameters —

Part 2:
Verification and calibration of testing machines

1 Scope

This part of ISO 14577 specifies the method of verification and calibration of testing machines for carrying out the instrumented indentation test in accordance with ISO 14577-1.

It describes a direct verification method for checking the main functions of the testing machine and an indirect verification method suitable for the determination of the repeatability of the testing machine. There is a requirement that the indirect method be used in addition to the direct method and for the periodic routine checking of the testing machine in service.

It is a requirement that the indirect method of verification of the testing machine be carried out independently for each test method.

This part of ISO 14577 is also applicable for transportable testing machines.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 376, *Metallic materials — Calibration of force-proving instruments used for the verification of uniaxial testing machines*

ISO 3878, *Hardmetals — Vickers hardness test*

ISO 14577-1, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 1: Test method*

ISO 14577-3, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 3: Calibration of reference blocks*

3 General conditions

3.1 Preparation

The machine shall be designed in such a way that it can be verified.

Before verification and calibration of the testing machine, it shall be checked to ensure that the conditions laid down in 3.2 to 3.4 are met.

3.2 Functional installation

The testing machine shall be configured to operate in compliance with and shall be installed in an environment that meets the requirements of this part of ISO 14577, ISO 14577-1, and, where applicable, ISO 14577-3. The testing machine shall be protected from vibrations. For testing in the micro and nano ranges, the testing machine shall also be protected from air currents and temperature fluctuations (see 7.1. in ISO 14577-1).

The influence of environment on the data, i.e. the noise floor, shall be estimated by performing a low force (e.g. equivalent to the usual initial contact force) indentation on a CRM and analysing the displacement over time. The force variability is the indent stiffness (obtained from force removal curve) multiplied by the standard deviation of the displacement once any background drift in mean displacement has been subtracted. These uncertainties shall then be included in the total combined uncertainty tests as calculated in Clause 8 and Annex H in ISO 14577-1, clause 4.

3.3 Indenter

In order to get repeatable measurements of the force/indentation depth data set, the indenter holder shall be firmly mounted into the testing machine.

The indenter holder should be designed in such a way that its contribution to the overall compliance is minimized (see Annex A).

3.4 Application of the test force

The test force shall be applied and removed without shock or vibration that can significantly affect the test results. It shall be possible to verify the process of increasing, holding, and removal of the test force.

4 Direct verification and calibration

4.1 General

4.1.1 Direct verification and calibration shall be carried out at the temperature of use, which is typically held at a stable value over the time of measurement in the range 10 °C to 35 °C, but preferably in the range (23 ± 5) °C. If a range of operating temperatures is required, then direct calibration and verification should be carried out at suitable points over that temperature range to determine the calibration validity as a function of temperature. If necessary, a calibration correction function or a set of calibrations valid at specific operating temperatures can be determined.

4.1.2 The instruments used for direct calibration and verification shall be traceable to National Standards as far as available.

4.1.3 Direct verification and calibration involves

a) calibration of the test force,

b) calibration of the displacement measuring device,

c) verification and calibration of the machine compliance,

d) verification of the indenter,

e) calibration and verification of the indenter area function, if the indentation depth is less than 6 µm, and

f) verification of the test cycle.

4.2 Calibration of the test force

4.2.1 Each range of force used shall be calibrated over the whole force range for both application and removal of the test force. A minimum of 16 evenly distributed points in the test force range shall be calibrated, i.e. 16 during application and 16 during removal of the test force. The procedure shall be repeated at least three times and the average calibration value shall be used. The maximum difference in calibration values shall not exceed half of the tolerances given in Table 1.

4.2.2 The test force shall be measured by a traceable method, for example, the following:

a) measuring by means of an elastic proving-device in accordance with class 1, or better of, ISO 376;

b) balancing against a force, accurate to within ±0,2 % applied by means of calibrated masses with mechanical advantage;

c) electronic balance with a suitable accuracy of 0,1 % of the minimum calibrated test force or 10 µg (0,1 µN) for the nano range.

For each measured point used for calibration, the difference between the measured and the nominal test force shall be within the tolerances given in Table 1.

Table 1 — Tolerances for test forces

Range of the test force *F* N	**Tolerances** %
$F \geq 2$	1,0
$0{,}001 \leq F < 2$	1,0
$F < 0{,}001$	2,5[a]
[a] For the nano range, a tolerance of 1 % is strongly recommended.	

4.3 Calibration of the displacement measuring device

4.3.1 The resolution required for the displacement measuring device of the testing machine depends on the size of the smallest indentation depth being measured. For the micro range, this value is by definition h = 0,2 µm; for the macro range it is typically larger than 2 µm.

The scale of the displacement measuring device shall be graduated to permit a resolution of indentation depth measurement in accordance with Table 2.

4.3.2 The displacement measuring device of the testing machine shall be calibrated for every range used by means of a suitable method and a corresponding system. The device shall be calibrated at a minimum of 16 points in each direction evenly distributed throughout its indentation displacement range. The procedure shall be repeated three times.

Some testing machines have a long-stroke displacement measuring device where the location of the indentation range of the displacement measuring device varies to suit the sample. For these types of machines, it shall be verified that the calibration is valid in accordance with table 2 for all of the used measurement positions in the travel range.

The following methods are recommended for the measurement of the relative displacement of the indenter: laser interference method, inductive method, capacitive method, and piezotranslator method.

For each measured point used for calibration, the difference between the measured and the nominal displacement shall be within the tolerances given in Table 2.