International Standard

ISO 14577-2

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

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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 document 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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This document was prepared by Technical Committee ISO/TC 164, *Mechanical testing of metals*, Subcommittee SC 3, *Hardness testing,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 459/SC1 *Test methods for steel (other than chemical analysis)*,in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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

The main changes are as follows:

— New procedure for indirect verification.

— New method 6 for machine compliance calibration.

— Replacement of tungsten carbide balls by balls in general.

A list of all parts in the ISO 14577 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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)[2] 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.

Part 2 of ISO 14577 specifies the methods of verification and calibration of instrumented indentation testing machines.

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

Part 2: Verification and calibration of testing machines

1 Scope

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

It specifies a direct verification method for verifying and calibrating the main functions of the testing machine and an indirect verification method suitable for the determination of the repeatability of the testing machine.

The methods in ISO 14577 are applicable to all systems that comply with the requirements of this part of ISO 14577.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. 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 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 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 General conditions

4.1 Requirements

There is the requirement that an indirect verification method be used in addition to the direct method and for the periodic routine checking of the testing machine in service.

4.2 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 4.3 to 4.5 are met.

4.3 Functional installation

The testing machine shall be configured to operate in conformity with and shall be installed in an environment that meets the requirements of this document, ISO 14577-1, and, where applicable, ISO 14577-3. The testing machine shall be protected from vibrations that would significantly affect the test results. For testing in the micro and nano ranges, the testing machine shall also be protected from air currents and temperature fluctuations (see ISO 14577-1:2026, 8.1).

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 Certified Reference Material (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 as calculated in ISO 14577-1:2026, Clause 4, Clause 9 and Annex H.

4.4 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).

4.5 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.

5 Direct verification and calibration

5.1 General

5.1.1 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.

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

5.1.3 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 contact depth is less than 6 µm, and

f) verification of the test cycle.

5.2 Calibration of the test force

5.2.1 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 performed at least three times. The average of all the measured values at each force point shall be used as the calibration value for the machine. The difference between the force value from the testing machine and the force value from the calibration device shall not exceed half of the tolerances given in Table 1.

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

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

b) balancing against a force with an uncertainty ≤ 0,2 % applied by means of calibrated masses with mechanical advantage;

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

For each individual measured point used to calculate the calibration value, 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.

5.3 Calibration of the displacement measuring device

5.3.1 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.

5.3.2 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 traceable to SI. 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 performed 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.