INTERNATIONAL STANDARD

ISO 14577-5

First edition
2022-10

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

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

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

Partie 5: Essai d'indentation élastique linéaire dynamique instrumenté (DIIT)

Reference number
ISO 14577-5:2022(E)

Contents

Page

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 164, *Mechanical testing of metals*, Subcommittee SC 3, *Hardness testing*.

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) 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.

This document has been prepared to enable the user to make reliable measurements of indentation hardness and indentation modulus, when dynamic indentation test techniques are used to improve instrumented indentation test techniques.

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

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

1 Scope

This document specifies the method of linear elastic dynamic instrumented indentation test for determination of indentation hardness and indentation modulus of materials showing elastic-plastic behaviour when oscillatory force or displacement is applied to the indenter while the load or displacement is held constant at a prescribed target value or while the indenter is continuously loaded to a prescribed target load or target depth.

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

ISO 14577-2, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 2: Verification and calibration of testing machines*

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

ISO 14577-4, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 4: Test method for metallic and non-metallic coatings*

ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

3 Terms, definitions and symbols

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 14577-1, ISO 14577-2, ISO 14577-3 and ISO 14577-4 apply.

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/

3.2 Symbols

Table 1 — Symbols

Symbols	Designations	Unit
$A_p(h_{dc})$	Projected area of contact of the indenter at distance h_{dc} from the tip	mm^2
D	Damping coefficient of the measurement head	mN·s/nm
E_{rd}	Dynamic reduced modulus of the contact	GPa
f	Frequency of oscillation	s^{-1}
$\bar{F}(\bar{h})$	Mean test force at the mean indentation depth (equivalent to the applied force without oscillation)	mN
$F_d(t)$	Instantaneous value of the oscillating force	mN
F_{d0}	Amplitude of force oscillation	mN
$\bar{h}$	Mean indentation depth under mean applied force (equivalent to the indentation depth without oscillation)	mm
$h_d(t)$	Instantaneous value of the oscillating displacement	nm
h_{d0}	Amplitude of displacement oscillation	nm
h_{dc}	Depth of the contact of the indenter with the test piece at $\bar{F}(\bar{h})+F_{d0}$	mm
H_d	Dynamic indentation hardness	GPa
k_s	Dynamic stiffness of the indenter shaft supporting springs	mN/nm
m	Oscillating mass (indenter and shaft)	g
S_d	Dynamic contact stiffness [dynamic stiffness of the contact at $\bar{F}(\bar{h})+F_{d0}$]	mN/nm
ϕ	Phase angle between force and displacement oscillation	°
ω	Angular frequency of oscillation ($\omega = 2\pi f$)	s^{-1}

4 Principle

A harmonic force or displacement oscillation with a known angular frequency, ω, and oscillation amplitude, is applied to the indenter being in contact with the sample during loading and/or any holding period. Commonly the force, $F_d(t)$ Formula (1), is controlled while the resulting displacement, $h_d(t)$ Formula (2), is measured. The transfer function, F_{d0}/h_{d0}, can be determined simultaneously during the test or post-test.

$$F_d(t) = F_{d0}\sin(\omega t) \quad (1)$$

$$h_d(t) = h_{d0}\sin(\omega t) \quad (2)$$

To calculate the dynamic contact stiffness, S_d, the dynamic behaviour of the sample and the actuator is modelled by a simple harmonic oscillator moving in one direction (Figure 1). This model describes only pure elastic material behaviour. The dynamic characteristics of the actuator, dynamic stiffness of the indenter shaft supporting springs, k_s, instrumented damping coefficient as functions of the frequency, f, and the oscillating mass, m, of the actuator (indenter plus shaft), must be known and are assumed to be constant. The mass of the sample volume influenced by oscillation must be negligible in comparison to the moving mass of the instrument.

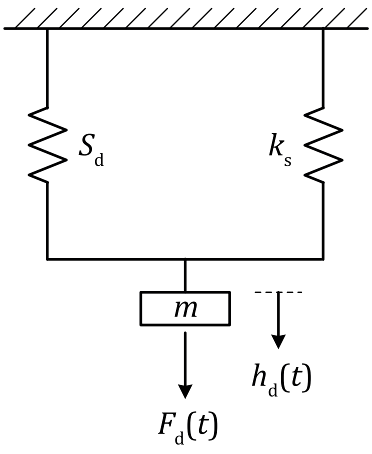

Figure 1 — Dynamic model of the indentation system including indenter–sample contact

Applying this model, the magnitude of the transfer function, F_{d0}/h_{d0}, is given by Formula (3).

$$\frac{F_{d0}}{h_{d0}} = S_d + \left(k_s - m\omega^2\right) \tag{3}$$

If the value of the transfer function is known, the dynamic contact stiffness, S_d, can be calculated using Formula (4).

$$S_d = \left|\frac{F_{d0}}{h_{d0}}\right| - \left(k_s - m\omega^2\right) \tag{4}$$

5 Testing machine

5.1 The testing machine shall be able to drive the actuator with a prescribed frequency and amplitude of oscillatory force or displacement with simultaneously measuring the resulting dynamic displacement or force. This may be done directly from the measured oscillation or by using a phase-lock amplifier. During a period of stiffness measurement, the frequency shall be constant.

5.2 The testing machine shall be calibrated according to ISO 14577-2 and shall be calibrated for the measurement of dynamic force, dynamic displacement and frequency.

For the direct calibration of the measurement of dynamic displacement, an interferometer working with an acquisition rate at least 50 times larger than the oscillation frequency can be used. For the direct calibration of the measurement of the dynamic force, a cantilever with accurately calibrated stiffness can be used. For daily verification of the testing machine reference blocks with certified modulus measured according to this document can be used.

If dynamic displacement and force are measured using a phase-lock amplifier, the measurement of dynamic displacement and dynamic force can be calibrated by comparison of the values (e.g. amplitude and frequency) extracted from the dynamic and static signals measured with an acquisition rate at least 50 times larger than the oscillation frequency.

Using a phase-lock amplifier, usually root mean square (rms) values for dynamic displacement and force are measured. The amplitudes of force oscillation and displacement oscillation defined in Table 1 are peak-to-mean (ptm) amplitudes. The ptm amplitude equals $\sqrt{2}$ times the rms amplitude. If the rms amplitudes for dynamic displacement and force are measured, they shall be converted to the ptm amplitudes.

5.3 The testing machine shall be configured in a manner that compensates for additional phase differences induced by the instrument's electronics that process both the load and displacement signals. If the testing machine can account for the instrument's collective contributions to the measured phase difference, the testing machine can be validated by measuring phase during stiff contact with a linear elastic material. Potential linear elastic materials are BK7 glass, fused silica and sapphire, which are assumed to exhibit no measurable damping, because at room temperature they are simple elastic solids.

5.4 The dynamic characteristics of the actuator, dynamic stiffness of the indenter shaft supporting springs, k_s, instrumented dynamic damping coefficient, D, and the oscillating mass, m, of the actuator (indenter plus shaft), must be known. These values can be estimated from measurements, which are performed with the indenter column oscillating in free space (Annex A).

5.5 The testing machine shall maintain its calibration over the testing machine's operating temperature range.

6 Procedure of data evaluation and determination of materials parameter

6.1 General

Data evaluation and determination of materials parameter is only possible if the amplitude of displacement oscillation is smaller than the amount of elastic recovery during complete unloading. Otherwise the surface contact of the indenter is lost. A calculation of the depth below which surface contact is lost can be found in Reference [1].

The dynamic contact depth, h_{dc}, at $\bar{F}(\bar{h})+F_{d0}$ can be calculated using the dynamic contact stiffness, S_d, estimated for this indentation depth by Formula (5).

$$h_{dc}=(\bar{h}+h_{d0})-\varepsilon\frac{\bar{F}(\bar{h})+F_{d0}}{S_d} \tag{5}$$

A variable epsilon correction shall be calculated and used if an unloading segment is available (Annex A, ISO 14577-1) otherwise a constant epsilon value can be used in depending on the indenter geometry.

NOTE Because a correct ε value can only be estimated from the final unloading it is assumed that this ε is valid for all indentation depths. This leads especially for small indentation depth to an additional error in the estimation of the area function.

6.2 Determination of dynamic indentation hardness, H_d

The dynamic indentation hardness, H_d, for a given dynamic contact depth, h_{dc}, and force, $\bar{F}(\bar{h})$, can be calculated using Formula (6). It is assumed that the contact area is constant during the time of measurement of the dynamic contact stiffness.

$$H_d=\frac{\bar{F}(\bar{h})+F_{d0}}{A_p(h_{dc})} \tag{6}$$

6.3 Designation of dynamic indentation hardness, H_d

EXAMPLE

H_d 1/75 = 8,4 GPa

- 8,4 — Dynamic indentation hardness value, in GPa
- 75 — Frequency of oscillation, in Hz
- 1 — Amplitude of displacement oscillation, in nm

6.4 Determination of dynamic reduced modulus, E_{rd}

The dynamic reduced modulus, E_{rd}, for a given dynamic contact depth, h_{dc}, can be calculated using Formula (7).

$$E_{rd} = \frac{\sqrt{\pi}}{2\sqrt{A_p(h_{dc})}} S_d \tag{7}$$

6.5 Designation of dynamic reduced modulus, E_{rd}

EXAMPLE

E_{rd} 1/75 = 68 GPa

- 68 GPa — Dynamic reduced modulus value, in GPa
- 75 — Frequency of oscillation, in Hz
- 1 — Amplitude of displacement oscillation, in nm

7 Test procedure

7.1 During the test, an oscillatory force or displacement is applied to the indenter while the load or displacement is held constant at a prescribed target value or while the indenter is continuously loaded to a prescribed target load or target depth.

7.2 The requirements for the loading procedure are described in ISO 14577-1.

7.3 Experiments are usually conducted using one of three different loading profiles, i.e.

a) Stepwise loading: the load or displacement is increased to a prescribed target value and is held for a period and then is increased to another prescribed target value and is held for another period and repeated. The dynamic contact stiffness is measured while the load or displacement is held constant at prescribed target values.

b) Constant strain-rate loading: the load or displacement is exponentially increased to a prescribed target value at a constant indentation strain rate, $(\mathrm{d}\bar{h}/\mathrm{d}t)/\bar{h}$. The dynamic contact stiffness is continuously measured while the indenter is exponentially loaded to a prescribed load or depth limit.

c) Linear loading: the load or displacement is linearly increased to a prescribed target value at a constant loading rate, $\mathrm{d}\bar{F}/\mathrm{d}t$. The dynamic contact stiffness is continuously measured while the indenter is linearly loaded to a prescribed load or depth limit.

7.4 All geometries of indenters described in ISO 14577-1 other than flat punches can be used.

7.5 For data evaluation and determination of materials parameter it is assumed, that the contact area is constant during the time of measurement of dynamic contact stiffness, given by the time constant of measurement. If the oscillatory force or displacement is applied to the indenter while the indenter is continuously loaded, the dynamic contact stiffness and therefore the contact area will not be constant. The actual change of contact area depends on amplitude and frequency of applied oscillation, loading rate, time constant of measurement and on the modulus to hardness ratio, E/H, of the material under investigation. A detailed discussion of the influence of these parameters can be found in Reference [2]. In general, it is recommended to use the highest permissible frequency of testing machine given by the manufacturer and to keep the harmonic amplitude small enough to keep the oscillation substantially within the regime of linear elasticity and the loading rate low. Optimal testing conditions can be found by comparison of the dynamic and static values measured for the same sample.

7.6 The amplitude of oscillation should be taken in a way that effects on the overall forces and displacement can be neglected. For most materials a displacement amplitude between 1 nm and 5 nm is recommended. For materials with modulus to hardness ratios above about 50, the oscillation can influence the overall loads and displacement already at amplitudes less than 5 nm. The measured values for force and displacement should be corrected for such an influence[2]. For systems that impose a force oscillation this can be accomplished by limiting the amplitude of the oscillating force relative to the mean applied force.

7.7 The maximum oscillation frequency is limited by resonance frequency of the system and by time constant and data logging frequency of measurement of dynamic contact stiffness. The minimum oscillation frequency depends on the material under investigation and must be high enough to avoid any additional creep during the measurement of dynamic contact stiffness. Frequencies between 1 Hz and 300 Hz are usual. Even if there is no damping in the system or the sample, using test frequencies near or above the resonant frequency of the system, can generate high phase angles. Additional steps must be taken to accurately calculate the contact stiffness if experiment goes through any resonance.

7.8 The phase angle (after exclusion of the offset caused by the measurement head and electronics) is an indicator of any viscous properties or ongoing plasticity of the material being tested. A phase angle of less than 5° is acceptable.

a) For stepwise loading, the oscillatory force or displacement is applied to the indenter while the load or displacement is held constant. This reduces any contribution to the phase angle due to plastic deformation. The phase angle is usually close to zero when testing elastic-plastic materials. If the phase angle exceeds 5°, it is recommended to check whether obvious creep occurs during the holding period. If the phase angle exceeds 5° due to creep, it is recommended to increase the oscillation frequency, *f*, as much as possible.

b) For constant strain-rate loading, the oscillatory force or displacement is applied to the indenter while the load or displacement is exponentially increased to a prescribed target value. The phase angle usually increases with the increasing of indentation depth while testing materials with high modulus to hardness ratio. When the phase angle exceeds 5°, it is recommended to increase the oscillation frequency, *f*, as much as possible and appropriately decrease the indentation strain rate, $\left(\mathrm{d}\bar{F}/\mathrm{d}t\right)/\bar{F}$, to keep the loading parameter, $\left(\left(\mathrm{d}\bar{F}/\mathrm{d}t\right)/\bar{F}\right)/\left(\left(F_{\mathrm{d}0}/\bar{F}\right)\cdot f\right)$, low to reduce the phase angle[2]. If oscillatory displacement is applied, it is not recommended to increase the displacement oscillation amplitude because large displacement oscillation amplitude will cause loss of indenter-sample contact at small indentation depth[1]. If oscillatory force is applied, it is recommended to use a constant dynamic force fraction, $F_{\mathrm{d}0}/\bar{F}$ [3].

c) For linear loading, the oscillatory force or displacement is applied to the indenter while the load or displacement is linearly increased to a prescribed target value. When the phase angle exceeds 5°, it is recommended to increase the oscillation frequency, *f*, as much as possible and appropriately decrease the loading rate, $\mathrm{d}\bar{F}/\mathrm{d}t$, to keep the loading parameter, $\left(\mathrm{d}\bar{F}/\mathrm{d}t\right)/\left(F_{\mathrm{d}0}\cdot f\right)$, low to reduce the phase angle. If oscillatory displacement is applied, it is not recommended to increase the displacement oscillation amplitude because large displacement oscillation amplitude will cause loss of indenter-sample contact at small indentation depth.

8 Uncertainty of results

Beyond the sources of experimental error described in ISO 14577-1 transfer function is also potentially affected by calibration, measurement time constants, phase differences created by the instrument's electronics, the actuator's dynamics and the instrument's stiffness.

A complete evaluation of the uncertainty shall be carried out in accordance with ISO/IEC GUM. A detailed description of one example of evaluation of uncertainty is given in Annex B.

9 Test report

The test report shall include the following information:

a) reference to this document, i.e. ISO 14577-5:2022;

b) all details necessary for identifying the test piece;

c) material and shape of the indenter and, where used, the detailed area function of the indenter;

d) testing cycle (control method and full description of the cycle profile); this should include:

   1) rates and times of force or displacement application;

   2) position and length of hold points;

   3) data logging frequency or number of points logged for each section of the test cycle;

e) dynamic settings:

   1) frequency of oscillation;

   2) target amplitude of controlled signal (force or displacement);

f) date and time of the last estimation of dynamic stiffness of the indenter shaft supporting springs, k_s, and oscillating mass of the actuator (actual values if available);

g) date and time of test;

h) environmental conditions of the test: temperature, humidity and air pressure;

i) number of tests, the result obtained with the total expanded uncertainty;

j) all operations not specified by this document, or regarded as optional;

k) details of any occurrence which may have affected the results.

Annex A
(informative)

Estimation of dynamic behaviour of the actuator

If the instrument actuator is modelled by a single degree of freedom simple harmonic oscillator, dynamic stiffness of the indenter shaft supporting springs, k_s, and oscillating mass, m, and damping coefficient, D, of the oscillator can be determined by fitting these values to the transfer function obtained as function of frequency by oscillating the indenter in air using Formula (A.1) and Formula (A.2). The resonance frequency of the actuator, f_r, can be calculated using Formula (A.3).

$$m = \frac{k_s - \left| \frac{F_{d0}}{h_{d0}} \right| \cos\phi}{(2\pi f)^2} \quad \text{(A.1)}$$

$$D = \frac{\left| \frac{F_{d0}}{h_{d0}} \right| \sin\phi}{2\pi f} \quad \text{(A.2)}$$

$$f_r = \frac{1}{2\pi} \sqrt{\frac{k_s}{m} - \left(\frac{D}{2m} \right)^2} \quad \text{(A.3)}$$

If the actuator is not free from external modes of vibration or the oscillating volume of material in the contact adds a significant amount of mass to the system this model is not applicable.

Because the estimated values can depend on the surrounding atmosphere (temperature, air pressure and humidity) it is recommended to estimate the dynamic characteristics just before every dynamic test unless it can be demonstrated that this dependence is negligible for the instrument being used.

In case of testing machines applying the force directly to the shaft with the tip, it is important to keep the indenter at a position which corresponds to the future position of the sample surface under investigation and to realize the same bending of the supporting springs of the indenter shaft as in the indentation test.