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Plastics — Instrumented micro-indentation test for hardness measurement

Plastiques — Essai instrumenté de micro-indentation pour le mesurage de la dureté

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Foreword

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical behaviour*.

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

This document was developed in response to a demand to define an instrumented indentation hardness test method in the micro scale for quality control of plastics materials and material selection. The test conditions are defined to ensure reproducibility and comparability of test results.

The Rockwell hardness method (ISO 2039-2) and the ball indentation method (ISO 2039-1) have been formulated for determining the indentation hardness of plastics. These test methods use different scales depending on the hardness of the material and it is difficult to compare hardness values of materials when they are measured on different scales.

The Vickers hardness test method (ISO 6507-1), used for metallic materials, is a method that does not use different scales. The Vickers hardness is calculated as the ratio of indentation load and the residual area of contact of the indenter. However, when this method is applied to plastics materials, it is difficult to measure the residual area of contact of the indenter because the edge of the indentation cannot be specified.

The instrumented indentation hardness test method (ISO 14577-1) is intended for hardness measurement in the range from nano-indentation to macro-indentation. In this method, since the contact area of the indenter is directly determined from the indentation depth, the above-mentioned problem is solved. It is therefore possible to determine the indentation hardness of the plastic materials.

In this document, in order to avoid errors due to detection of initial contact, a test force that can result in sufficient indentation depth within the range of micro-indentation is specified. For example, the range of indentation depth under this condition, which is from 10 µm to 50 µm in the case of the four types of materials described in this document, also corresponds to the dimension (scale) of the diagonal length range of 0,020 mm to 1,400 mm specified by the Vickers hardness test method (ISO 6507-1). In this document, the measurement time of a single condition is specified similarly to the Rockwell hardness method (ISO 2039-2).

Plastics — Instrumented micro-indentation test for hardness measurement

1 Scope

This document specifies a hardness test method for plastics using instrumented indentation in the micro scale with one clearly defined test condition to ensure reproducibility and comparability of test results.

The test method is selectively suitable for use with the following materials:

— moulding, extrusion and cast thermoplastic materials; rigid and semi-rigid thermoplastics sheets;

— rigid and semi-rigid thermosetting moulding materials; thermosetting sheets.

This test method could also be utilized for nanometric filled system, considering the fillers are distributed uniformly in the polymer matrix.

This test method allows dumbbell type specimen, strip type specimen, platens and specimen cut from any finished parts or products.

This test method is useful for quality control, material selection, and screening of new formulations.

NOTE This document does not aim to describe all scientific or technical aspects of microhardness testing on plastics in general.

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 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 2602, *Statistical interpretation of test results — Estimation of the mean — Confidence interval*

ISO 4287, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

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

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

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

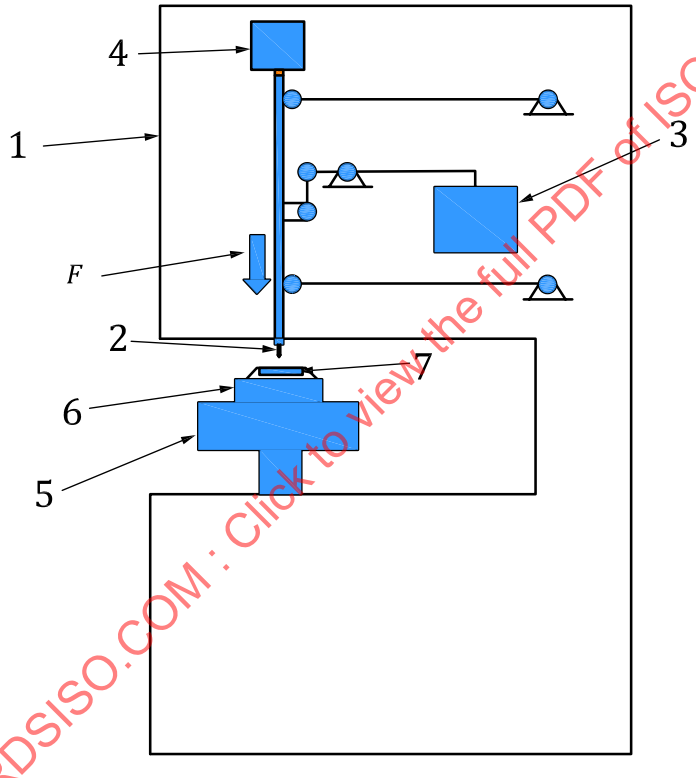
4 Principle

The original Berkovich indenter is forced under a specified load into the surface of the test specimen. The depth of indentation is measured under load. Indentation hardness H_{IT} is calculated from F_{max} and A_p (see 10.1).

5 Apparatus

5.1 General

The testing apparatus comprises of a rigid frame, a stage, a test specimen holder, an indenter, a loading unit, and a displacement measuring device. The apparatus is required to measure and record the applied force, the indentation depth, and the time throughout the testing cycle. The apparatus shall have the capability of compensating for the machine compliance and of utilizing the appropriate indenter area function in accordance with ISO 14577-1. The general design is as shown in Figure 1.



Key

1	machine frame	5	stage
2	indenter	6	test specimen holder
3	loading unit	7	test specimen
4	displacement measuring device		
F	test force		

Figure 1 — Example of testing apparatus

5.2 Indenter

The testing apparatus shall be equipped with an original Berkovich indenter in accordance with ISO 14577-1. The indenter should be calibrated according to ISO 14577-2.

5.3 Displacement measurement device

The displacement measuring device measures the indentation displacement directly or measures the displacement reference with touch point of the presser foot to surface of the test specimen. The maximum permissible error of the displacement is ± 1 % of indentation depth, when the displacement is more than 6 µm.

5.4 Loading unit and force measuring device

The loading unit applies predetermined test forces. The loading unit and the force measuring device shall satisfy the following conditions.

a) The loading unit is able to apply 500 mN.

b) The repeatability of the test force is ±1,5 %.

c) The tolerance of the force application time is ±10 %.

5.5 Periodic checks of test apparatus

Periodic checks of the test apparatus using a control chart shall be conducted.

NOTE Some reference materials which hardness value is close to that of plastics, for example certified reference material for instrumented indentation test made from plastics or a hardness reference plate for macro Vickers hardness (preferably 30 HV, see ISO 6507-1 and ISO 6507-3 for details), can be used for this purpose.

6 Test specimen

6.1 Test specimens should be prepared by the appropriate methods described in ISO 294-1, ISO 294-2, ISO 294-3 and ISO 20753.

6.2 The test specimen thickness shall be at least 40 times the h_{max} value.

NOTE The minimum requirement of the test specimen thickness differs owing to the test specimen hardness. For example, if the hardness of the test specimen is about H_{IT} 0,5/30/40/30 = 95 N/mm^2, the indentation depth of F_{max} is about 16 µm, the minimum thickness of the test specimen is about 640 µm. If the hardness of the test specimen is about H_{IT} 0,5/30/40/30 = 225 N/mm^2, the indentation depth of F_{max} is about 11 µm, the minimum thickness of the test specimen is about 440 µm.

6.3 The test specimen shall not be twisted and shall have parallel surfaces for testing.

6.4 The surface roughness *Ra* (as defined in ISO 4287) shall be satisfied by $Ra \leq h_{max}/20$.

NOTE Any curvature in the surface can influence test result.

6.5 It is important to specify the position and orientation of the test specimen, when the test specimen is prepared by machining.

6.6 The test specimen which is cut from any finished parts or products shall be prepared with the cutting size to minimize cutting effect on the test results (see 8.7).

7 Conditioning

Unless other environmental conditions have been agreed between the interested parties or required by the specification standard of the material, the preferred conditions are (23 ± 2) °C and (50 ± 10) % relative humidity, as defined in ISO 291.

8 Procedure

8.1 Conduct the test in the same standard atmosphere as that used for conditioning the test specimen, unless otherwise agreed upon by the interested parties.

8.2 Throughout the test, protect the testing apparatus from shock, vibration and air movements that can significantly influence the test result.

8.3 The test specimen shall either be placed on a support that is rigid in the direction of indentation or fixed in a suitable test specimen holder. The contact surfaces between test specimen, support and test specimen holder shall be free from extraneous matter which might increase the compliance (reduce the stiffness) of the test specimen support.

When the test specimen is fixed by holding from upper side of the test specimen, the fixing condition shall satisfy the following:

a) the stress which is applied for fixing the test specimen shall be smaller than that of indenter;

b) indentation shall be at least four times their indentation diameter away from the edge of the test specimen holder where touches to the test specimen directly.

When the test specimen is fixed by adhesion, hot melt adhesive shall not be used because heat treatment influences the test specimen.

8.4 The surface of the test specimen shall be free of dust, dirt and grease. So, the test points shall be confirmed by using a microscope before testing.

8.5 The zero point for the measurement of the force/indentation depth curve is the touch point determined during the first increase of either the test force or the contact stiffness.

8.6 The testing cycle is force-controlled. The controlled parameters can vary either continuously or step by step. To avoid the influence of time factor, the force application time, force removal time and holding time at maximum force are fixed as follows:

a) maximum force shall be 500 mN;

b) force application and force removal time shall be 30 s;

c) holding time at maximum force shall be 40 s.

NOTE It is possible to use the displacement rate controlled if the force application and force removal time is 30 s. The results of force control and that of displacement rate control can be different.

A full description of all parts of the testing cycle shall be stated in the test report.

8.7 It is important that the test results are not affected by the presence of an interface, free surface or by any plastic deformation introduced by a previous indentation in a series.

The effect of any of these depends on the indenter geometry and the materials properties of the test specimen. Indentations shall be at least four times their indentation diameter away from interfaces of free surfaces and the minimum distance between indentations shall be at least four times the largest

indentation diameter. When the test specimen is cut from any finished parts or products, the cutting edge should be over 10 mm away from the indentation point.

NOTE The minimum distance between indentations or between indentation and free surfaces of test specimen differs owing to the test specimen hardness. For example, if the hardness of the test specimen is about H_{IT} 0,5/30/40/30 = 95 N/mm^2, the indentation depth of F_{max} is about 16 µm, the minimum distance between indentations or between indentation and free surfaces of test specimen is about 480 µm. If the hardness of the test specimen is about H_{IT} 0,5/30/40/30 = 225 N/mm^2, the indentation depth of F_{max} is about 11 µm, the minimum distance between indentations or between indentation and free surfaces of test specimen is about 330 µm.

9 Number of test specimens

At least five hardness tests should be made on an indentation area in the test specimen.

Calculate the arithmetic means of the test results and, if required, the standard deviations and the 95 % confidence intervals of the mean values in accordance with the procedure given in ISO 2602.

The number of measurements may be more than five if greater precision of the mean value is required.

More information on precision can be found in Annex A.

10 Expression of results

10.1 The indentation hardness is calculated using Formulae (1) to (3):

$$H_{IT} = \frac{F_{max}}{A_p} \tag{1}$$

$$A_p = 23,96 \times h_c{}^2 \tag{2}$$

$$h_c = h_{max} - \frac{3}{4} \times (h_{max} - h_r) \tag{3}$$

where

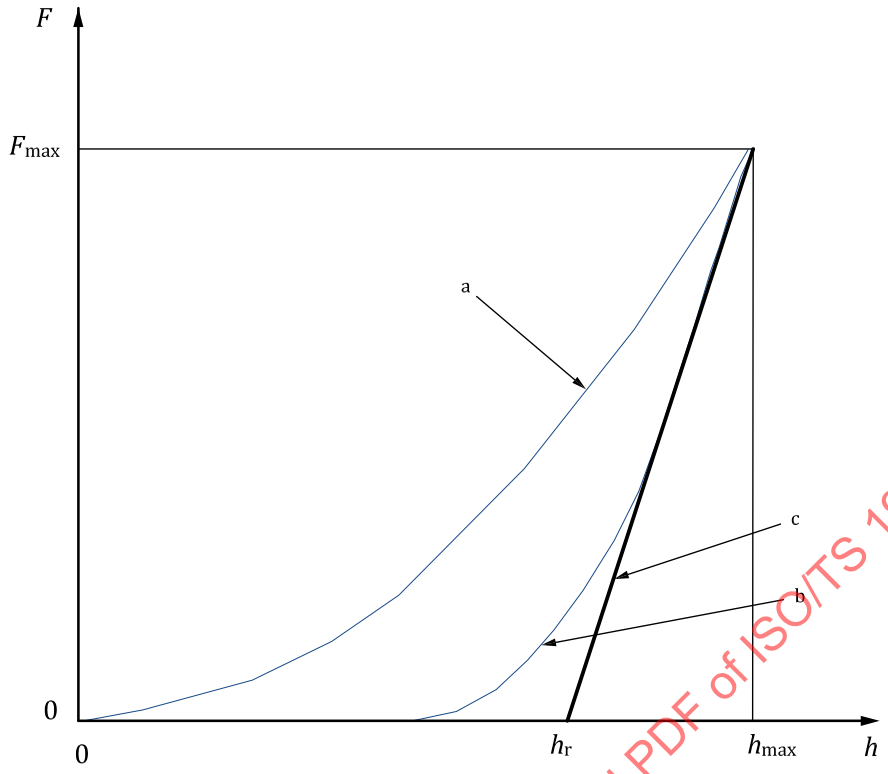
F_{max} is the maximum force applied to the test specimen by means of the indenter, in newtons (N);

A_p is the projected area of contact of the original Berkovich indenter at distance h_c from the tip, in square millimetres (mm^2);

h_c is the depth of the contact of the original Berkovich indenter with the test specimen at maximum test force F_{max}, in millimetres (mm);

h_r is the point of intersection of the tangent to curve b at F_{max} with the indentation depth-axis, in millimetres (mm) (see Figure 2);

h_{max} is the maximum indentation depth at F_{max}, in millimetres (mm).



Key

F force

h indentation depth

a Application of the test force.

b Removal of the test force.

c Tangent curve to b at F_{max}.

Figure 2 — Force/indentation depth curve

10.2 Express the indentation hardness as follows:

H_{IT} 0,5/30/40/30 = 146 N/mm^2 under the test conditions in 8.6.

10.3 Report the individual data, their mean value and the standard deviation.

11 Test report

The test report should include the following information:

a) a reference to this document, i.e. ISO/TS 19278:2019;

b) details of the test specimen:

1) a full description of the test specimen and its origin;

2) the location of the indentation on the test specimen;

