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

Part 6: Instrumented indentation test at elevated temperature

Matériaux métalliques — Essai d'indentation instrumenté pour les paramètres de dureté et de matériaux —

Partie 6: Essai d'indentation instrumenté à température élevée

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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 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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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.

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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 increase 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 requirement to measure the indent optically. Furthermore, by a variety of techniques, the instrumented indentation test is able to generate hardness and modulus values at different indentation depths within the indentation cycle.

This document has been prepared to enable the user to obtain hardness and other materials parameters using instrumented indentation testing at elevated temperature. The elastic and plastic properties of material components at elevated temperature are critical for determining the performance representative of in-service condition above ambient temperature. Typical applications include hard coatings, nuclear materials, welded materials, fuel cell materials, aerospace materials, etc. In-service properties of wear surfaces, cutting tool, high friction contacts are also significant applications.

This document covers the instrumented indentation testing systems with independent heating of both the indenter and test piece with feedback control and equipped with temperature sensing. This type of instrumented indentation testing system is suitable for testing materials of high or low thermal conductivity in air, in inert or reducing gaseous environment, and also in vacuum, which offers the potential for higher testing temperature. Active heating of the indenter tip allows independent adjustment of energy flow into the tip and test piece so that a condition of iso-thermal contact is achieved rather than just thermal equilibrium with constant (but unknown) thermal gradients. A temperature sensor mounted in an indenter body with a low heat capacity is sensitive to small heat flows and, therefore, can detect small temperature differences between test piece and tip.

The method described in this document can achieve low uncertainty in the temperature assigned to the indentation contact when testing materials with high thermal conductivity (uncertainty increases when testing low thermal conductivity materials). This is achieved by traceable calibration of the indenter tip temperature for a reproducible temperature offset between the contact temperature and indenter temperature sensor reading. A reproducible temperature offset is obtained by using a procedure to minimise (optimally to zero) the heat flow between the test piece and the indenter. This not only makes the temperature offset from indenter temperature sensor to indenter tip a reproducible amount, but also minimises the temperature gradient in the test piece (from the contact to the bulk of the test piece) that occurs when there is a heat flow through the contact. The minimum detectable heat flow of a system depends upon the heat capacity of the indenter and the resolution of the indenter temperature sensor. Any amount of heat flow causes a higher uncertainty in assigned contact temperature that is higher the lower the thermal conductivity of the test material is.

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

Part 6: Instrumented indentation test at elevated temperature

1 Scope

This document specifies the instrumented indentation method for testing at elevated temperature for determination of hardness and other materials parameters at temperatures above ambient. Elevated temperature testing is defined in this document to be when the test piece and indenter tip are heated above the ambient conditions of the instrument to a controlled and measured temperature; insulating shielding is used to enclose the hot zone to reduce heating effects so that the majority of the instrumented indentation testing machine is at ambient conditions.

This document is restricted to test machines that have been traceably calibrated and pass an indirect verification according to ISO 14577-2 when operating at elevated temperature to ensure that any effects on ambient sensors caused by the presence of a hot zone are accounted for. This document covers instrumented indentation testing at elevated temperatures in air, in inert or reducing gaseous environments, or in vacuum.

This document provides a method for instrumented indentation testing at elevated temperature with both the indenter tip and test piece actively heated, and with independent feedback control and temperature measurement of both the indenter tip and test piece.

This document provides a method for estimation of the uncertainty of the contact temperature. The uncertainty increases as the thermal conductivity of the test piece decreases. It is left to the user to decide if that uncertainty is fit for their purpose.

The test method in this document is not applicable to:

— instrumented indentation testing where there is no direct measurement of the temperature of the indenter tip body itself;

— instrumented indentation testing where above ambient temperatures are obtained by placing the entire instrument in a hot box to achieve iso-thermal heating of the whole system. These systems typically only achieve limited elevated temperature;

— instrumented indentation testing with active heating of the test piece but only passive heating of the indenter, e.g. by proximity to the hot test piece and thermal conduction through the indentation contact, hot gas, or any combination.

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*

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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 14577-5, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 5: Linear elastic dynamic instrumented indentation testing (DIIT)*

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, ISO 14577-4, ISO 14577-5 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 and designations

Symbols	Designations	Unit
$A_p(T)$	Projected area of contact under load of the indenter at distance h_c from the tip at elevated temperature	mm^2
a	Equivalent contact radius	mm
C_p	Heat capacity of the indenter assembly	J/K
$C_T(T)$	Total measured compliance of the contact at elevated temperature	nm/mN
$C_f(T)$	Instrument compliance at elevated temperature	nm/mN
$C_S(T)$	Compliance of the contact at elevated temperature after correction for machine compliance	nm/mN
$E(T)$	Young's modulus at elevated temperature	GPa
$E_r(T)$	Reduced modulus of the contact at elevated temperature	GPa
$E_i(T)$	Modulus of the indenter at elevated temperature	GPa
$E_{IT}(T)$	Indentation modulus at elevated temperature	GPa
$E^*(T)$	Plane strain indentation modulus at elevated temperature	GPa
$F_{max}(T)$	Maximum applied force at test temperature	N
h_c	Depth of the contact of the indenter with the test piece at $F_{max}(T)$	mm
$H_{IT}(T)$	Indentation hardness at elevated temperature	GPa
k	Thermal conductivity of the test piece	W/(m·k)
$\dot{Q}_{min}$	Minimum detectable heat flow between test piece and indenter tip	W
T	Temperature of the test	°C
T_c	Temperature at the contact point (this is the temperature assigned to the test results)	°C
T_d	Temperature at the displacement measuring device	°C
T_f	Temperature at the force measuring device	°C
T_{sh}	Temperature sensor reading for control of test piece heater	°C

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Table 1 *(continued)*

Symbols	Designations	Unit
T_{ih}	Temperature sensor reading at the position of the temperature sensor in the indenter body	°C
T_{ss}	Temperature sensor reading of the test piece surface	°C
T_{tip}	Temperature of the indenter at the tip	°C
ΔT_{ih}	The resolution of the indenter temperature sensor	°C
t_i	Integration period when the indenter and the test piece is in contact	s
$\nu_i(T)$	Poisson's ratio of the indenter at elevated temperature	–
$\nu_s(T)$	Poisson's ratio of the test piece at elevated temperature	–

4 Principle

4.1 General

Instrumented indentation tests conforming to ISO 14577-1, ISO 14577-4, or ISO 14577-5 are performed into a test piece heated to elevated temperature (i.e. significantly higher than ambient temperature) in a hot zone that is thermally insulated from the rest of the testing machine. The purpose of this method is to obtain the hardness and other material properties as defined in ISO 14577-1, ISO 14577-4, or ISO 14577-5 but at a measured temperature above the ambient conditions.

4.2 Instrument calibration and verification at elevated temperature

Although the majority of the testing machine is at ambient temperature, there are small heat leaks from the hot zone into the instrumentation or the instrument environment, which can shift the calibrations away from those obtained at room temperature. Therefore, the test machine shall be traceably calibrated and pass an indirect verification in accordance with ISO 14577-2 whilst operating at the required elevated test temperature.

4.3 Indenter material selection

The indenter material shall be carefully selected so that the indenter tip does not change during the test in a way that prevents the intended sample property measurements during the test. The indenter tip shall not deviate in shape or mechanical properties from its calibrated values and shall not chemically react with, or bond to, the test piece. In all cases, the elastic modulus and Poisson's ratio of the indenter tip (as a function of temperature) shall be used for analysis of the indentation response and the values used shall be recorded in the test report.

4.4 Test environment

The test environment shall be carefully selected and controlled so that it shall not degrade nor chemically alter (e.g. oxidise) the test piece surface in a way that prevents the intended sample property measurements during the test, so that the results returned will be from the material intended for testing and not the properties of the degraded material or oxide instead. As temperature increases, the reactivity of tip and test piece to oxygen increases. Therefore, the time taken to achieve thermal equilibrium and perform the tests should be minimised.

4.5 Indentation creep rate at elevated temperature

ISO 14577-1 places a maximum limit on the allowable indentation creep rate for a given indentation cycle. At elevated temperature, the indentation creep rate is typically higher than at room temperature. Suitable choice of force-displacement-time cycle shall be made to meet the requirements of ISO 14577-1 and should be optimised to minimise uncertainty in the measurement of the indentation stiffness and thermal drift. This is dealt with in more detail in the relevant sections of this document.

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