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Metallic materials — Instrumented indentation test for hardness and materials parameters — Evaluation of stress change using indentation force differences

Matériaux métalliques — Essai d'indentation instrumenté pour les paramètres de dureté et de matériaux — Évaluation de la variation de contrainte en utilisant les différences de force d'indentation

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Foreword

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

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Introduction

Residual stress is defined as the "locked-in" stress that exists in materials and structures independent of the presence of any external loads. The mechanisms that create residual stress are diverse and include non-uniform plastic deformation, surface modification and thermal gradients.

Numerous techniques have been developed for evaluating residual stress, each with their own merits and drawbacks. Physical methods such as X-ray diffraction (XRD) and neutron diffraction are non-destructive tests based on measuring lattice parameters, and thus they are restricted to crystalline materials; in addition, they are sensitive to microstructure and to the test environment.

On the other hand, destructive methods such as hole drilling and sectioning method let us quantify the residual stress mechanically and require no reference sample. However, these methods cannot avoid destruction of the sample and require a strain gauge attachment. Then the observed change in strain must be converted to the stress.

The results of these methods for determining residual stress can differ because the residual stress sensing depth and area in each method are different. The hole-drilling measures the amount of strain relaxation caused by the removal of the hole material. The spatial resolution of the method is approximately the size of the hole (typically 2 mm diameter). In case of XRD, the smaller size of irradiated area requires a longer measurement time. The indentation method requires less precise surface preparation than XRD because it obtains a direct response from the material, and strain gauges are unnecessary. It takes less than 30 s to measure one point and has high in-field applicability. This document, using a semi-destructive method for measuring stress change, makes it unnecessary to machine samples from in-service components or manufactured products exhibiting internal or external stress changes.

Residual stress is not a material property but a state of stress. In general, it has been observed that when a material is subject to stress change, its indentation curve is shifted upward or downward compared to the initial indentation curve, because the stress change makes indentation easier (relatively tensile) and more difficult (relatively compressive). In a constant depth test (fixed h_{max}): an increase in compressive stress squeezes the material around the indenter and hence a greater load is needed to reach to the same indentation depth than in the initial stress state. On the other hand, an increase in tensile stress releases the material and a smaller load is necessary to keep the same indentation depth than in the initial stress state. In fact, a smaller load/larger load is required at constant h_{max} from initial surface. It seems as if an imaginary (virtual) force works in the same/opposite direction as/to the indenting direction.

To quantify the effect of stress on indentation behaviour, the deviatoric stress concept along the indenting direction is proposed in this document. The method for calculation of the average stress change is given in Clause 8. The described procedure can be applied only when the observed change in force-displacement curves is a result of stress change. The proposed method measures the near-surface stress change in the direction parallel to the test surface.

Similarly, in a constant load test (fixed L_{max}): compressive stress change makes indentation difficult and hence the indentation depth becomes shallow. Tensile stress change makes indentation easy and the indentation depth becomes deeper. Thus, in the elastic modulus approach, the sign (mode) of stress change can be determined by using this constant-load test as is similar to the above constant-depth (h_{max}) test in this proposal.

The material for the reference and target states should be selected so as to maintain identical chemical composition with relatively little change of mechanical properties to the target material. This test method is limited to examinations that conform to the conditions given in Annex A. Annex A provides a procedure to achieve satisfactory results by sorting out locally hardening test points. The test point showing the largest deviation is reasonably considered as being from a locally severely changed region. The test point showing the greatest deviation from the average value should be screened out and this process be repeated with remaining test points until the criterion is met. Nevertheless, it is

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recommended to carefully control the factors between the target and reference states, such as chemical composition, grain size, dislocation density and texture, which can cause errors in measurements.

If the condition given in Annex A is not satisfied, destructive stress relief methods by electrical discharge machining or focused ion beam can be combined to obtain the reference state (stress-free state) without changing material properties following Annex B. The stress change from this document can be converted to the residual stress of the target state by considering the stress value of the reference state measured by other methods, such as X-ray diffraction and hole-drilling method.

This document proposes a method to measure the average stress change between reference and target states. Residual stress caused by non-uniform plastic forming and heat treatment usually shows stress components of the same sign in the region requiring stress evaluation. Therefore, there is high demand for the proposed method in many fields. Additionally, if the user wants to resolve stress components, Annex C in the draft can be utilized. The average stress change measured by this method is change of half the first invariant of stress tensor because the stress normal to the test surface is zero. In other words, the average normal stress change is always constant, even if the coordinate system is rotated on the surface.

The method proposed in the draft has been applied and verified for many different materials and conditions, and extensive evidence shows that it is both reasonable and useful, as shown in Annex D and E. The purpose of this item is to measure the stress change between reference and target states. As proposed in this draft, the relative stress change can be quantitatively determined and whether the stress change involved is tensile (indentation curve down) or compressive (indentation curve up) compared to the reference (initial) state. Thus, if the state of initial stress is known, it is possible to determine the magnitude and sign of the altered stress state as well.

Some materials show the sensitivity of indentation force to residual stress, which results the force difference greater in a tensile stress state than a compressive stress state, although the difference is in general not large. Even for materials showing different sensitivity of peak load in compressive vs. tensile stress, the load difference is a monotone function of stress change, so that the region of maximum stress can be identified. Furthermore, for many materials, the load difference sensitivity does not significantly violate the fundamental concept.

This document has been prepared to provide useful guidelines on how to extract a two-dimensional representation of the entire 3D residual stress state by means of local size-controllable indentations over the component surface. The testing surface may be indented in one-dimensional or two-dimensional indentation arrays for a more reliable evaluation of the entire bulk residual stress state. The stress state detected by the proposed methodology provides accurate measurement of the plane stress residual stress state from the near-surface region in the component.

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Metallic materials — Instrumented indentation test for hardness and materials parameters — Evaluation of stress change using indentation force differences

1 Scope

This document specifies the method of instrumented indentation test for evaluation of stress change between reference and target states using indentation force differences.

This document primarily applies to measuring the stress change in a specific location and the stress difference between different locations.

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 14577-1:2015, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 1: Test method*

ISO 14577-2:2015, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 2: Verification and calibration of testing machines*

ISO/IEC Guide 98-3:2008, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

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 Symbols and designations

For the purpose of this document, the symbols and designations in Table 1 apply.

Table 1 — Symbols and designations

Symbol	Designation	Unit
A	Average projected area of reference and target states	mm^2
A_r	Projected area of reference state	mm^2
A_t	Projected area of target state	mm^2
ΔF	Force difference from target curve to reference curve at maximum indentation displacement	N
F_{max}	Maximum test force	N
F_r	Maximum test force on reference state	N
F_t	Maximum test force on target state	N

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Table 1 *(continued)*

Symbol	Designation	Unit
h	Indentation depth	mm
h_{max}	Maximum indentation depth (should be the same for target and reference state)	mm
p	Ratio of stress changes along one direction to that along the normal direction	-
r	Reference state (used as subscript)	-
t	Target state (used as subscript)	-
$\Delta\sigma_{avg}$	Average stress change of surface stress change components.	MPa
$\Delta\sigma$	Stress change from reference state to target state along a direction perpendicular to indenting direction	MPa
$\Delta\sigma'$	Shear deviatoric stress component of stress change from reference state to target state	MPa
$\Delta\sigma'_z$	z component of shear deviatoric stress of stress change from reference state to target state	MPa

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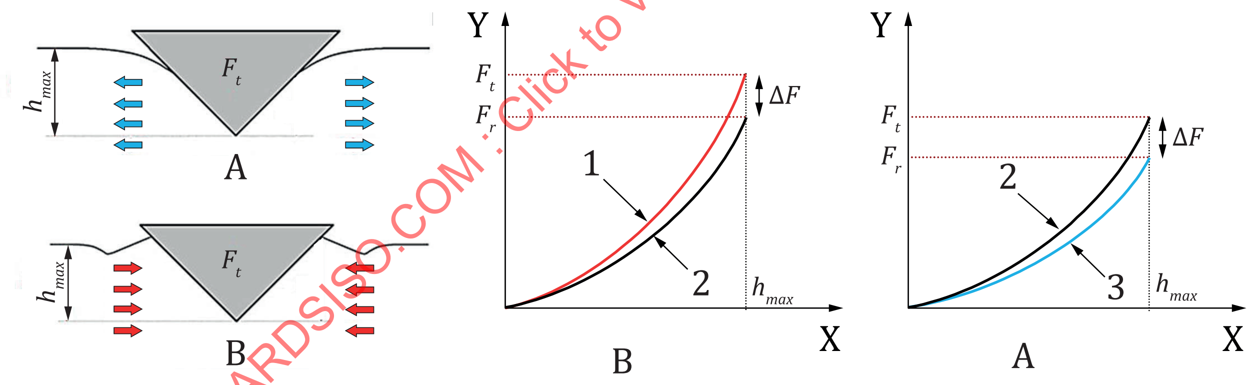
5 Principle

5.1 Shift of force/indentation depth curve by stress change

The stress change in the same material between two different states creates a shift in the force/indentation depth curve (see Figure 1). A stress increase to be in a relatively tensile state makes indentation easier because the material around the indenter is relaxed. Thus, the indentation force required to reach a given depth in a relatively tensile stress state is lower than that in the initial stress state. In a relatively compressive stress state, the reverse is true. Therefore, the stress change can be evaluated by measuring the indentation force difference at maximum indentation depth $(F_r - F_t) = \Delta F$ between the reference and target states.



Key

X indentation depth
Y force
A tensile stress
B compressive stress
1 compressive
2 reference
3 tensile

Figure 1 — Change in morphology and force/indentation depth curve with stress change

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5.2 Derivation of stress change from force difference

The stress change in one direction can be expressed as $\Delta\sigma$; the stress normal to $\Delta\sigma$ on the surface can be expressed as $p\,\Delta\sigma$ (p is the stress ratio change). Since the stress change normal to the surface (along the indentation test direction) is taken as zero, only surface biaxial stresses affect the shape of the force/indentation depth curve. The biaxial stress change can be divided into a hydrostatic stress term and a shear deviatoric stress term. The only shear deviatoric stress component ($\Delta\sigma'$) applied along the indentation test direction (z), $\Delta\sigma'_z$, can influence the force/indentation depth curve when the indentation test is performed along the z direction. $\Delta\sigma'_z$ can be related to the force difference as in Formula (5.1). This formula reflects the fact that the shear deviatoric stress along the indentation test direction is directly related to the indentation stress change (indentation force difference divided by projected area) [1],[2]:

$$\Delta\sigma'_z = \frac{(1+p)}{3}\,\Delta\sigma = \frac{(F_r - F_t)}{A} \tag{5.1}$$

From Formula (5.1), the stress change; $\Delta\sigma$ can be expressed as in Formula (5.2), and the other stress normal to $\Delta\sigma$ can be determined as $p\,\Delta\sigma$:

$$\Delta\sigma = \frac{3}{(1+p)}\,\frac{(F_r - F_t)}{A} \tag{5.2}$$

6 Testing machine

6.1 The testing machine shall have the capability of applying predetermined test forces or displacements within the required scope and shall fulfil the requirements of ISO 14577-2.

6.2 The testing machine shall have the capability of measuring and reporting applied force, indentation displacement and time throughout the testing cycle.

6.3 The testing machine shall have the capability of compensating for the machine compliance (see ISO 14577-1:2015 Annex C and ISO 14577-2:2015, 4.5).

6.4 A self-similar sharp indenter (e.g. Vickers pyramid and Berkovich pyramid) following ISO 14577-1:2015, Clause 4, should be used for the measurement.

6.5 The testing machine shall operate at a temperature within the permissible range specified in ISO 14577-1:2015 7.1 and shall maintain its calibration within the limits specified in ISO 14577-2:2015, Clause 4.

6.6 The testing machine shall be calibrated following the procedures detailed in ISO 14577-2:2015, Annex D and the use of a reference block (see ISO 14577-3) that shall be isotropic and homogeneous. The repeatability of the testing machine shall be below 3,3 % of the coefficient of variation by using a reference block with maximum permissible coefficient of variation below 3 %.

7 Test piece

7.1 The test piece shall fulfil the requirements of ISO 14577-1:2015, Clause 6.

7.2 The preparation of the test piece shall be carried out in such a way that any alteration of the surface hardness and/or residual stress is minimized.

7.3 The thickness of the test piece shall be known or measured and its tolerance shall be specified.

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8 Procedure

8.1 The test shall be in controlled conditions that fulfil the requirements of ISO 14577-1:2015, Clause 7.

8.2 To measure a stress change in the same material, the force/indentation depth curve obtained in the target state (target force/indentation depth curve) and that obtained in the reference state (reference force/indentation depth curve) at the same maximum displacement are required (see Figure 1). The flow chart showing the overall test procedure is seen in Figure 2.

8.3 Each state includes a minimum of six force/indentation depth curves. The procedure requires a minimum of six indents which requires different sized array depending on the size of indents. The stress comparison is with the stress average over this array size.

8.4 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 piece. Indentations shall be at least three times their indentation diameter away from interfaces or free surfaces and the minimum distance between indentations shall be at least five times the largest indentation diameter.

The indentation diameter is the in-plane diameter at the surface of the test piece of the circular impression of an indent created by a conical indenter. For non-circular impressions, the indentation diameter is the diameter of the smallest circle capable of enclosing the indentation. Occasional cracking can occur at the corners of the indentation. When this occurs, the indentation curve with crack shall be excluded from the calculation of stress change. If sufficient data are not obtained due to cracking, the maximum displacement shall be lowered and the material shall be retested.

The minimum distances specified are best applicable to ceramic materials and metals such as iron and its alloys. For other materials, it is recommended that separations of at least 10 indentation diameters be used.

If in doubt, it is recommended that the values from the first indentation are compared with those from subsequent indentations in a series. If there is a significant difference, the indentations can be too close and the distance should be increased. A factor of two increases in separation is suggested.

It can be desirable to measure thin coatings in cross-section (e.g. to avoid problems due to surface roughness). In this case, there cannot be enough coating thickness to meet the minimum spacing requirements as specified above. Smaller spacing can be used if there is experimental evidence that this does not significantly influence the force/indentation depth/time data sets with respect to correctly spaced indentations on similar test pieces with thicker coatings. Note that the currently proposed method assumes that there is no component of stress in the out of plane direction and cannot calculate the stress for this situation.

8.5 The obtained data set shall conform to the criterion in Annex A.

Annex B shall be applied, if the criterion in Annex A is not satisfied or if there is no appropriate location to be chosen as test region for the reference state.

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8.7 The indented projected area shall be observed directly by suitable means.

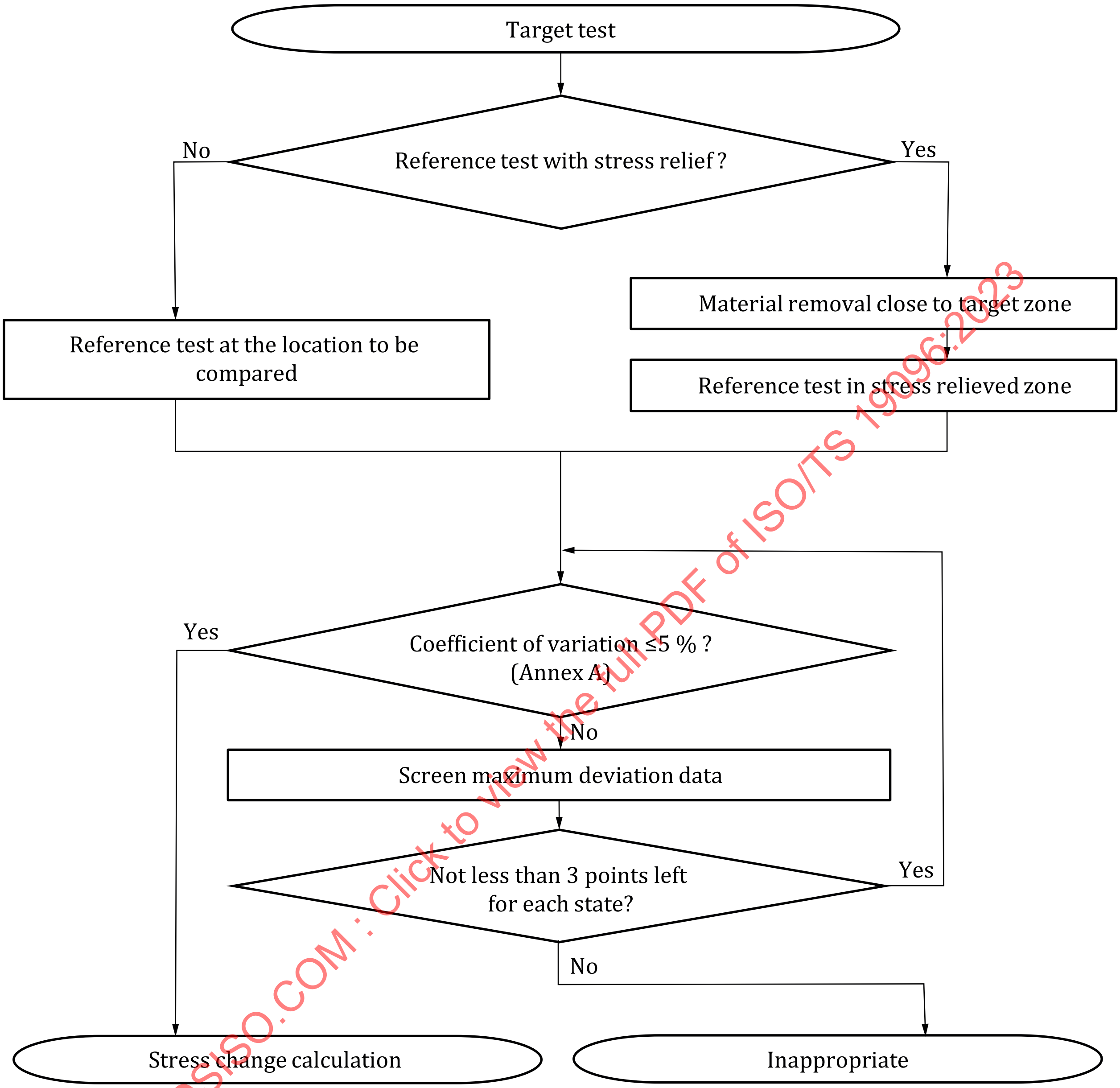

Figure 2 — Flow chart for selection of test procedure to measure stress change

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9 Calculation of stress change

9.1 Force and projected area calculation at each state

The force and projected area in each state can be calculated with the data set satisfying Annex A as in the following formulas:

$$F_r = \frac{\sum_{i=1}^{n} F_{r,i}}{n} \tag{9.1}$$

$$F_t = \frac{\sum_{i=1}^{n} F_{t,i}}{n} \tag{9.2}$$

$$A_r = \frac{\sum_{i=1}^{n} A_{r,i}}{n} \tag{9.3}$$

$$A_t = \frac{\sum_{i=1}^{n} A_{t,i}}{n} \tag{9.4}$$

9.2 Force difference

From the obtained target force/indentation depth curves and reference force/indentation depth curves, the force difference between two state is calculated by subtracting average maximum force of target state from average maximum force of reference state (see Figure 1) as in following formula:

$$\Delta F = F_r - F_t \tag{9.5}$$

9.3 Projected area

The area for the reference and target states will be averaged to calculate stress change as given in Formula (9.6).

$$A = \frac{A_r + A_t}{2} \tag{9.6}$$

NOTE If the reference sample is in a stress-free state, A_r can be used directly as A in Formula (9.6).

9.4 Calculation of average stress change

The calculation of average stress change is expressed by Formula (9.7) using Formula (5.2).

$$\Delta\sigma_{avg} = \frac{(\Delta\sigma + p\Delta\sigma)}{2} = \frac{3}{2}\frac{(F_r - F_t)}{A} = \frac{3}{2}\frac{(\Delta F)}{A} \tag{9.7}$$

The determination of stress change ratio (p) using a Knoop indenter is described in Annex C.

10 Uncertainty of the results

A complete evaluation of the uncertainty shall be carried out in accordance with ISO/IEC Guide 98-3. A detailed description of two methods of evaluation of uncertainty is given in ISO 14577-1:2015, Annex H.

This approach calculates a combined uncertainty from individual contributions. These may be grouped into random and systematic uncertainties. Individual parameters can contribute one or both types of uncertainty to the total measurement uncertainty. Thus, the uncertainty in measured displacement can have a random component due to the resolution of the scale used and vibrational noise, etc., plus a systematic component due to the displacement sensor calibration uncertainty. The following sources of uncertainty shall be considered:

— zero point assignment;

— measurement of force and displacement (including effects of ambient vibrations and magnetic field strength changes;

— fitting of the force-removal curve;

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— thermal drift rate;

— contact area due to surface roughness;

— force, displacement;

— testing machine compliance;

— calibration drift due to uncertainty in temperature of testing machine and time since last calibration;

— tilt of test surface.

It is not always possible to quantify all the identified contributions to the random uncertainty. In this case, an estimate of standard uncertainty may be obtained from the statistical analysis of repeated indentations into the test material. Care should be taken that systematic standard uncertainties that can contribute to the random standard uncertainty are not counted twice (see ISO/IEC Guide 98-3:2008, Clause 4).

11 Test report

The test report shall include the following information:

a) reference to this document, i.e. ISO/TS 19096:2023;

b) all details necessary for identifying the test piece;

   1) target test,

   2) reference test,

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) set point values,

   2) rates and times of force or displacement,

   3) position and length of hold points, and

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

e) result obtained, the total expanded uncertainty and the number of tests;

f) method and functional form of any fit used for the determination of the zero-point;

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

h) details of any occurrence that can have affected the results;

i) temperature of the test;

j) date and time of test;

k) analysis methods;

l) if required, all agreed additional information including determined values from the measured force/indentation depth curve and detailed information about the uncertainty budget.

Test report should describe the location of the indentation on the test piece and the unique identifier of the instrument or the specific instrument configuration used to perform the test.

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Annex A
(normative)

Procedure for hardness uniformity verification

A.1 General

The scatter in hardness should be kept to a minimum and uniformity shall be confirmed for reliable evaluation of stress change by the criterion given in A.2. Each state includes a minimum of six force/indentation depth curves. The hardness of each test point shall be determined as F_{max} / A_p, where A_p is the measured projected area.

A.2 Hardness uniformity verification

A.2.1 The arithmetic mean value, $\bar{H}$, is calculated from the n values H_1, ..., H_n, as given by Formula (A.1):

$$\bar{H} = \frac{H_1 + \cdots + H_n}{n} \tag{A.1}$$

where H_i is the hardness value of the i th test point.

As a measure of the scatter, the experimental standard deviation, $s(H)$, is calculated as given by Formula (A.2):

$$s(H) = \sqrt{\frac{\sum_{i=1}^{n} (H_i - \bar{H})^2}{n}} \tag{A.2}$$

The relative scatter of the measured hardness values is the coefficient of variation or relative experimental standard deviation, V, expressed as a percentage, as given by Formula (A.3):

$$V = \frac{s(H)}{\bar{H}} \times 100 \tag{A.3}$$

A.2.2 The maximum permissible coefficient of variation calculated with the data set including reference and target points is 5 %. If this criterion is not satisfied, the measured results may possibly differ from the actual stress distribution.

A.2.3 When the coefficient of variation or relative experimental standard deviation calculated from A.2.1 does not satisfy the criterion in A.2.2, the test point showing the greatest deviation from the average value should be screened out and A.2.1 shall be repeated with remaining test points. The remaining test points of each state shall be not less than three after screening.

A.2.4 If the coefficient of variation fails to satisfy A.2.2, there is considered to be a large scatter in hardness, which is not appropriate for calculating the stress change by force difference between the obtained curves. As an alternative, the reference state in combination with stress relief method can be obtained following Annex B.

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Annex B
(normative)

Combining with stress relief method

B.1 General

Annex B gives information on obtaining the reference state in combination with a stress relief method by electrical discharge machining and focused ion beam, as shown in Figure B.1, in cases that do not satisfy the criterion in Annex A or if there is no appropriate location to be chosen as test region for the reference state. This reference state can be considered where the stress in the direction perpendicular to the cutting plane is relieved.

Measuring hardness by cutting near the free surface can result in substantial error if Annex B is applied for soft materials such as polymers. In this case, the structural compliance arising from a free edge shall be considered.[3]

B.2 Procedure

B.2.1 The stress relief method shall be determined in consideration of minimizing the change in mechanical properties and scale of indentation displacement. For macro and micro scales, electrical discharge machining is recommended, and for nano scale, focused ion beam is recommended.

B.2.2 The material removal for stress relief shall be performed at a location close to the target zone. The depth of cutting plane shall be greater than ten times the maximum indentation depth. The length of the cutting line shall be determined by considering the expected reference test numbers.

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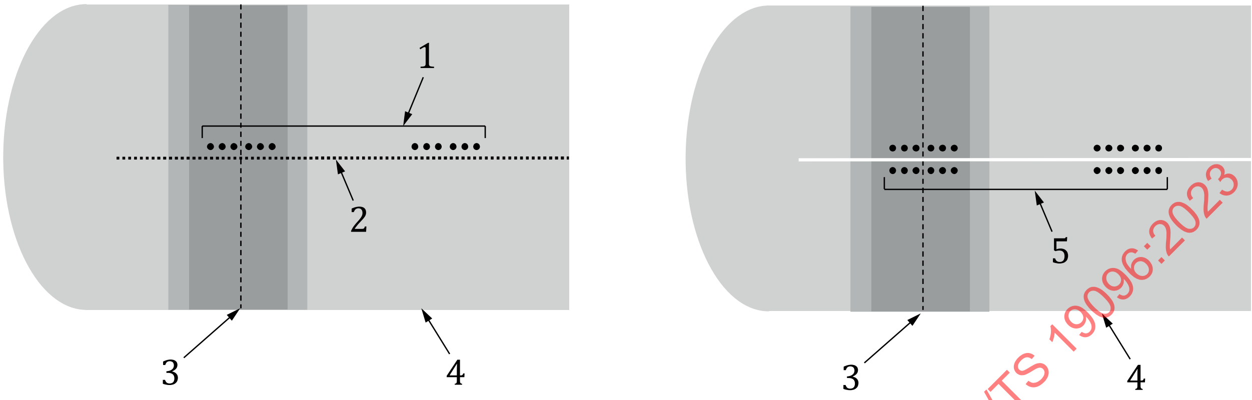
B.2.3 The reference state indentation tests should be along the length of the cutting line. The reference state indentation tests should be three times their diagonal or diameter away from the cutting line. The test for the reference state should include a minimum of six force/indentation depth curves. The tests shall be under controlled conditions that fulfil the requirements of Clause 7 and ISO 14577-1:2015, Clause 7.



Key

1 target test before stress relief

2 line to be cut

3 weldment

4 base metal

5 reference test after stress relief

Figure B.1 — Indentation test combined with stress relief method

B.3 Hardness uniformity verification

The uniformity in hardness of each state including reference and target points conforms to the criterion given in Annex A.

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Annex C
(informative)

Determination of stress change ratio using Knoop indenter

C.1 General

The Knoop indenter is an orthogonal pyramid with a lozenge base and two different apex angles as shown in Figure C.1. The general requirements for the Knoop indenter shall follow ISO 4545-1:2017.

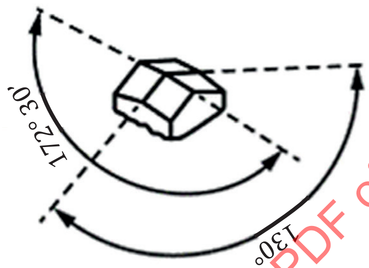

Figure C.1 — Shape of Knoop indenter

As the Knoop indenter has two semi-apex angles, force/indentation depth curves in each direction show different force differences (ΔF_x and ΔF_y) by the stress change from reference state to target state along the x-axis and y-axis, respectively ($\Delta\sigma_x$ and $\Delta\sigma_y$), as shown in Figure C.2.

For the calculation of the stress change ratio (p), indentation tests using a Knoop indenter in two different directions normal to each other are required. The two test locations shall be adjacent to minimize the differences from properties and stress states.

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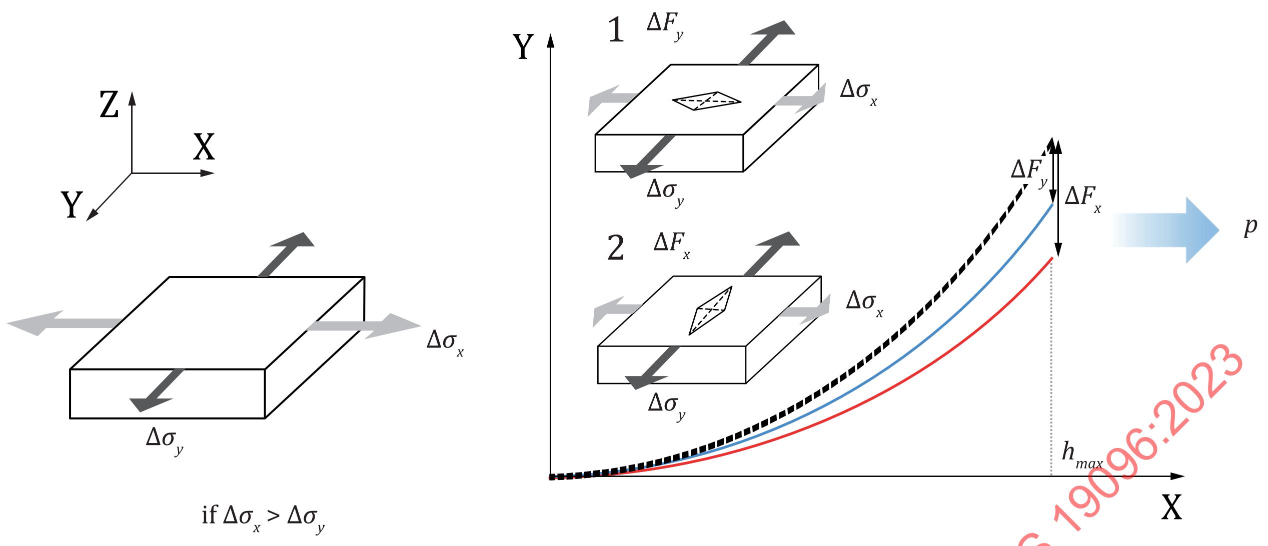

Key

X indentation depth

Y force

Figure C.2 — Change in force/indentation depth curve with non-equibiaxial stress change using Knoop indenter

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C.2 Stress change ratio determination using Knoop indenter

C.2.1 General

The testing machine, test piece and procedures shall fulfil the requirements of Clauses 5 to 7.

C.2.2 Force difference

From the obtained target force/indentation depth curves and reference force/indentation depth curves, the force differences for two directions between target and reference curves can be measured at the maximum indentation depth (Figure C.2).

C.2.3 Calculation of stress change ratio

The calculation of stress change ratio is expressed as in Formula (C.1), where the ratio of conversion factor ratio ($\frac{\alpha_{||}}{\alpha_{\perp}}$) for a Knoop indenter is 0,34. The conversion factors are the correlation between the indentation force difference and stress change component perpendicular ($\alpha_{\perp}$) or parallel ($\alpha_{||}$) to the long diagonal of the Knoop indenter[4].

$$p = \frac{\frac{\Delta F_x}{\Delta F_y} + \frac{\alpha_{||}}{\alpha_{\perp}}}{1 - \frac{\alpha_{||}}{\alpha_{\perp}} \frac{\Delta F_x}{\Delta F_y}} \quad \text{(C.1)}$$

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Annex D
(informative)

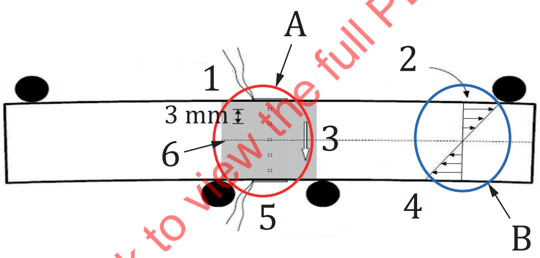
Verification of instrumented indentation test residual stress measurement method by bending specimen

D.1 General

This annex gives information on comparing the known bending stress with the stress measured by instrumented indentation test described in the main body of this document.

D.2 Known stress using four-point bending jig

A known stress was intentionally induced on a bending specimen (SA-508 Gr. 1A) in the range of elastic deformation (elastic bending stress was measured accurately by strain gauge) as shown in Figure D.1. The dimensions of the bending specimen are 25 mm × 30 mm × 200 mm. The specimen was heat treated for stress relief[5].



Key

A indentation on bending state
B elastic stress field by bending
1 tensile face (outer span)
2 bending stress profile
3 indentation array
4 compressive face (inner span)
5 strain gage
6 neutral line

Figure D.1 — Four-point bending jig for applying stress

D.3 Results of comparison between known bending stress and instrumented indentation test measurement

Instrumented indentation tests for reference state and target state were performed before and after applying elastic stress, respectively, on the centre line of the specimen. The test procedure and calculation of stress changes followed Clauses 5 to 8 of this document. Figure D.2 demonstrates one-to-one matching within small constant scatter (less than 50 MPa) over the whole stress range.

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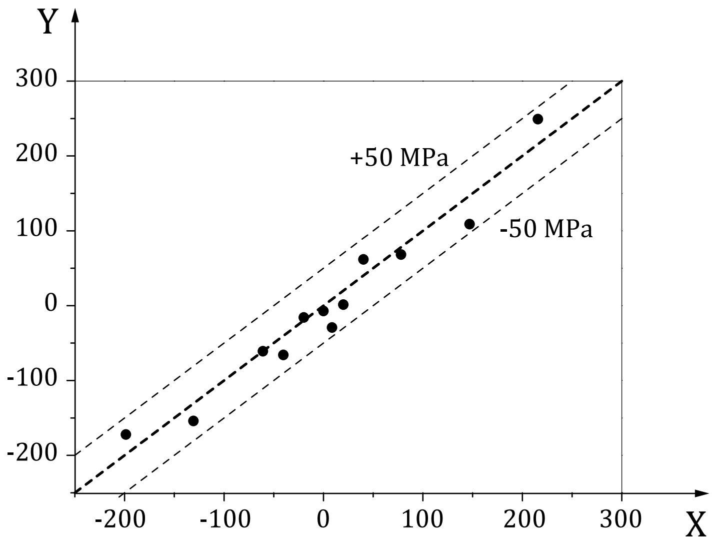

Key

X applied stress by bending (MPa)

Y analysed stress by indentation (MPa)

Figure D.2 — Comparison with known bending stress using instrumented indentation test with strain gauge

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