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

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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 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.

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, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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

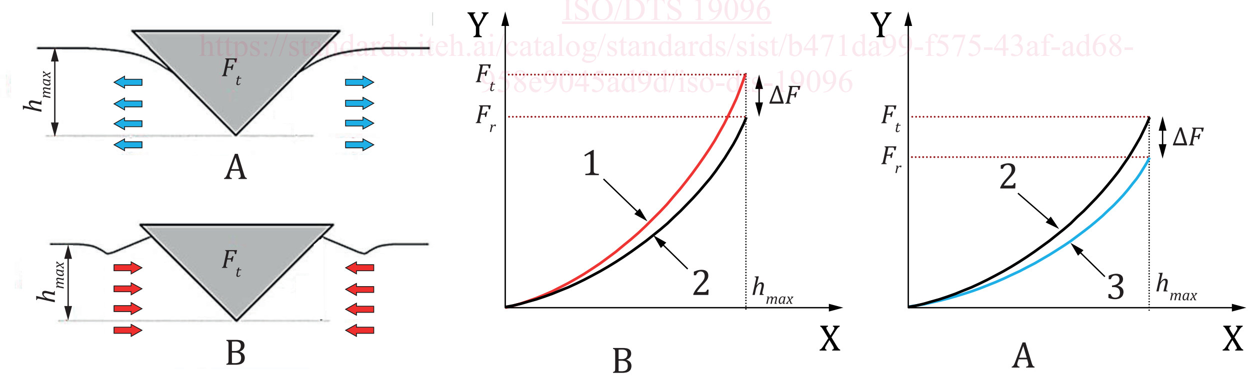
Table 1 *(continued)*

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

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

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). 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), $\Delta\sigma'_{\mathrm{z}}$, can influence the force/indentation depth curve when the indentation test is performed along the z direction. $\Delta\sigma'_{\mathrm{z}}$ 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]:

$$\Delta\sigma'_{\mathrm{z}} = \frac{(1+p)}{3}\,\Delta\sigma = \frac{(F_{\mathrm{r}} - F_{\mathrm{t}})}{A} \tag{5.1}$$

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 $p\,\Delta\sigma$:

$$\Delta\sigma = \frac{3}{(1+p)}\frac{(F_{\mathrm{r}} - F_{\mathrm{t}})}{A} \tag{5.2}$$

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.

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.

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

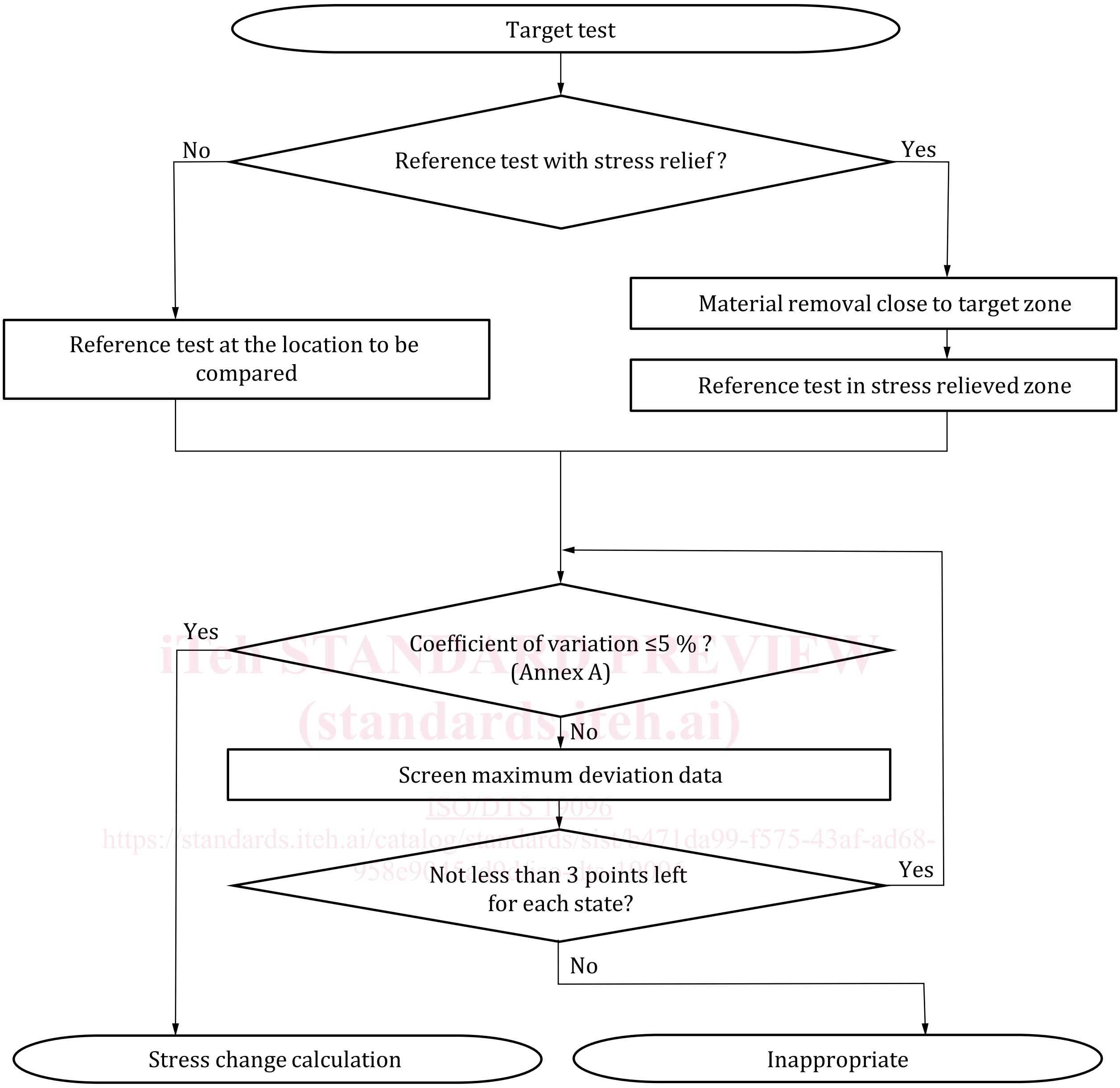


Figure 2 — Flow chart for selection of test procedure to measure stress change

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:

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