TECHNICAL SPECIFICATION

ISO/TS 19096

First edition 2023-11

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





COPYRIGHT PROTECTED DOCUMENT

© ISO 2023

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office CP 401 • Ch. de Blandonnet 8 CH-1214 Vernier, Geneva Phone: +41 22 749 01 11 Email: copyright@iso.org Website: www.iso.org

Published in Switzerland

Contents		Page
Fore	word	iv
Introduction		v
1	Scope	1
2	Normative references	1
3	Terms and definitions	1
4	Symbols and designations	1
5	Principle	2
	5.1 Shift of force/indentation depth curve by stress change	
6	Testing machine	3
7	Test piece	3
8	Procedure	4
9	Calculation of stress change 9.1 Force and projected area calculation at each state 9.2 Force difference 9.3 Projected area 9.4 Calculation of average stress change	5 6
10	Uncertainty of the results	6
11	Test report	7
Anne	ex A (normative) Procedure for hardness uniformity verification	8
Anne	ex B (normative) Combining with stress relief method	9
Anne	ex C (informative) Determination of stress change ratio using Knoop indenter	11
	ex D (informative) Verification of instrumented indentation test residual stress measurement method by bending specimen	
Anne	ex E (informative) Comparison with hole-drilling and saw-cutting methods	15
	ography	

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).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

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*.

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

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 $h_{\rm max}$): 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 $h_{\rm max}$ 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 <u>Clause 8</u>. 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 $L_{\rm max}$): 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 ($h_{\rm max}$) 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 $\underline{Annex\ A}$ 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 $\underline{Annex\ B}$. 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.