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Measurement of elastic modulus by instrumented indentation in the macro-range: Uncertainty evaluation

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ABSTRACT

Elastic modulus is one of the most important parameters evaluated by instrumented indentation test. This paper refers to its measurement in the macro range, where, missing adequate reference materials, traceability proposed by ISO 14577-1 cannot be applied. In this condition, the uncertainty evaluation shall be performed by propagating the uncertainties of the quantities involved in the measurement procedure. This paper describes an experiment, designed in order to separate the possible effects due to the instrument (mainly elasticity effects) and the measured bulk stainless steel test block (mainly non-uniformity of the material). A careful use of design of experiments and other statistical methods allow to clean data from systematic effects and outliers. Thereafter, the application of GUM (Guide to the expression of Uncertainty in Measurement, JCGM 100) allows the expression of uncertainties of the measurement methods proposed by ISO 14577-1 and of a further method here proposed to avoid some disadvantages of the recommended ones.

1. Introduction

Conventional hardness tests involve many characteristics of the material tested, including the elastic modulus, but it is not possible to separate their effect and evaluate the value of single characteristics. To overcome this limit, the instrumented indentation test was developed. Continuous recording of forces and indenter displacements during a controlled loading-holding-unloading indentation cycle gives sufficient information for estimating the value of the elastic modulus, obtained from the resulting indentation curve post-processed according to ISO 14577-1 [1]. The quantities measured at the same time are, therefore, the applied force F and the indentation depth h. Force measurement is very accurate: force transducers have typical uncertainties of less than 0.5% of the applied force, with a zero error nearly negligible, but indentation depth is strongly affected by the measurement device. In fact, force and length measurements shall have a common path, at least the indenter and indenter holder, contributing to the measured displacement with elastic or inelastic effects produced by the applied force: this is an error in the measurement of the indentation depth. Measurement uncertainty shall consider this factor, in addition to the instrumental factors involved by usual force and displacement measurements. This factor is so important that ISO 14577-4 [2] presents a correction procedure. An alternative correction procedure, proposed for correcting the same effect in Rockwell hardness measurements [3], is preliminarily discussed in this paper.

2. Measurement procedure

2.1. General background

Traditional hardness tests are based on a measurement of the effect produced applying a force to the surface of the material under test through a very hard body, the indenter, made of diamond or tungsten carbide. The hardness evaluation is made when the plastic deformation is completed, by measuring the indentation produced, therefore giving a single evaluation involving all the effects of the plastic deformation produced. Instrumented indentation test (IIT), on the contrary, is based on a large number of simultaneous measurements of force and length, i.e. the force applied by the indenter to the material under test and the corresponding indentation depth, ideally at any instant of the loading and unloading cycle. The resulting indentation curves (ICs) are post-processed, according to ISO 14577-1, to extract indentation parameters such as the equivalent conventional (frequently Vickers) hardness, the indentation modulus and the Martens hardness.

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The standard ISO 14577-1 defines three distinct application ranges according the maximum depth of indentation h_{max} and the maximum applied force F_{max}:

- macro: $2\ \mathrm{N} \le F_{max} \le 30\ \mathrm{kN}$
- micro: $F_{max} < 2\ \mathrm{N}$, $h_{max} > 0.2\ \mu\mathrm{m}$
- nano: $h_{max} \le 0.2\ \mu\mathrm{m}$

In this paper, we consider the macro-range where, due to the high imposed forces, the indentation size is usually larger than the microstructural size, so assuring that the indentation curves are representative of the bulk mechanical behavior of the sample material, even if a nonhomogeneous phase distribution in the sample can still alter the measurement. This loading range is helpful to gain a global mechanical response from high strength bulk materials and thick coatings. Indeed, the scope of our investigation is not limited to bulk materials but may be widened to layers, multilayers and coated material if the influence of the substrate on the elastic measurements and the thickness effect are properly evaluated [4–8]. The macro-range overcomes also the indentation size effect (ISE) typical in nano/micro-IITs, i.e. hardness measurements that increase or decrease with the indentation residual size [9]. Even if ISO 14577-1 was published more than ten years ago, the macro-range has not yet an adequate traceability to the national standards through reference hardness blocks, therefore the uncertainty shall be evaluated considering all the steps of the measurement procedure (called in the field of hardness measurements “Direct method”). This is more complicated than evaluating the comparison with reference blocks (called “Indirect method).

For the specific measurement of Young's modulus, many methods are currently used: e.g., tensile test or impulse excitation of vibration [10]. Young's modulus is an intrinsic property of the material, strongly related to the strength of the chemical bonds between atoms by the Condon-Morse model [11]. The indentation modulus E_{IT}, which represents a quite close estimate of Young's modulus [12], is obtained with the instrumented indentation test.

ISO 14577-1 describes the procedure for E_{IT} measurement using the tabulated elastic modulus E_i and Poisson's ratio ν_i of the indenter, Poisson's ratio ν_s of the hardness block (considered isotropic and homogenous) and by introducing a reduced modulus E_r:

$$E_{IT} = \frac{1-\nu_s^2}{\frac{1}{E_r} - \frac{1-\nu_i^2}{E_i}} \tag{1}$$

The force–displacement curve, determined in the indentation test, is used for calculating E_r from the slope S of the line tangent to the unloading curve at maximum depth (called contact stiffness) and the contact area A_p between indenter and tested material:

$$E_r = \frac{S\sqrt{\pi}}{2\sqrt{A_p}} \tag{2}$$

Therefore

$$E_{IT} = \frac{1-\nu_s^2}{\frac{2\sqrt{A_p}}{S\sqrt{\pi}} - \frac{1-\nu_i^2}{E_i}} \tag{3}$$

The contact stiffness S and the contact area A_p are evaluated using the values of force F and indentation depth h determined by data of the unloading cycle.

Schematic representations of the force–displacement curve of an instrumented indentation test and of the cross section of the residual indentation are provided in Figs. 1 and 2.

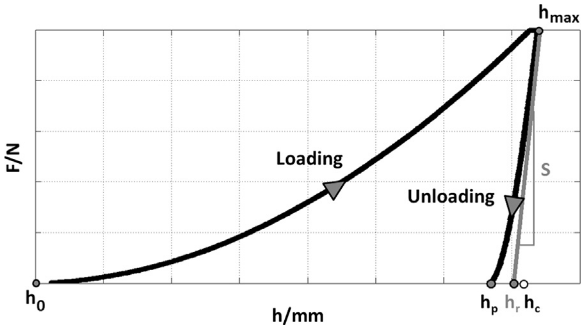

Fig. 1. Force–displacement curve of an instrumented indentation test (IIT).

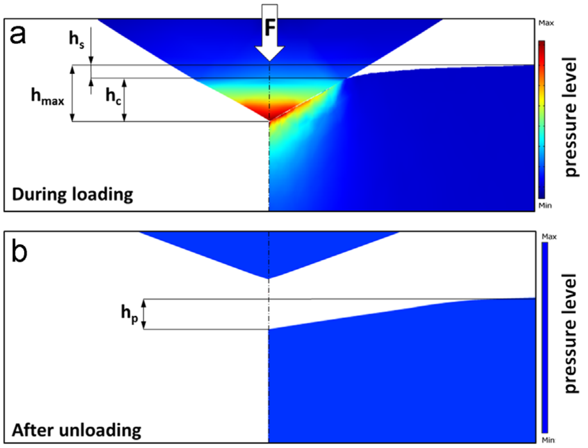

Fig. 2. Cross section of an indentation.

2.2. *Force measurement*

Force measurement is generally performed by a force transducer directly connected to the indenter holder, thus avoiding any possible error due to friction in the moving mechanism. Given the small dimensions and the close dimensional tolerances of indenters and indenter holders, errors due to misalignment and relevant side components of the force are negligible. Moreover, in our case, a force calibration of the hardness instrument by comparison with a reference force transducer was done.

2.3. *Indentation depth measurement*

Indentation depth h is measured by a displacement transducer, which cannot be installed between the tip of the indenter and the surface of the material under test. In some hardness measuring instruments, the full frame is involved in the relevant length measurement, thus a large part of the hardness measuring instrument has at the same time the task of transferring the applied force and the produced indentation depth, but in this case the deformation of this part of the instrument is included in the measure of the indentation depth, entailing a significant error. In our case, the displacement transducer measures the indentation depth between the surface of the material tested (given by the reference ratchet) and the indenter tip (given by the indenter holder), thus reducing to a minimum the path common to force and displacement. A schematic description is provided in Fig. 3.

Even in this case the effect of deformation of the common path shall be corrected, carefully considering the effect of the compliance of the common path, usually called frame compliance C_f. The effect of C_f is an increase of the measured indentation depth and, consequently, a decrease of S (Fig. 1), thus leading to an underestimation of the indentation modulus. Since this effect is