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Metallic materials — Measurement of mechanical properties by an instrumented indentation test — Indentation tensile properties

Matériaux métalliques — Mesure des caractéristiques mécaniques par un essai de pénétration instrumenté — Caractéristiques de traction par indentation

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Contents

Page

Foreword

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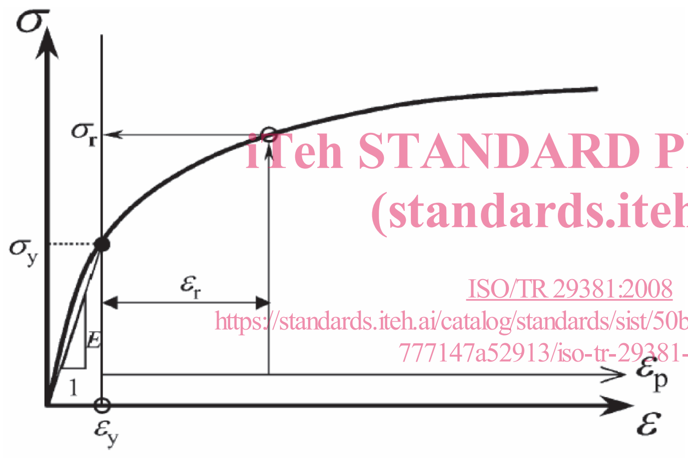
Introduction

0.1 General information for tensile properties

For centuries the elastic properties of materials have been described by Hooke's Law (*ca.* 1660) and the practical parameter of Young's modulus. This simple ratio of stress/strain is a practical, useful measure and, combined with a value for Poisson's ratio of a material (a measure of the dimensional change of a material in directions other than the principal axis in which it is being strained), it is possible to determine the stresses introduced by loading even quite complex structures. When the applied force is removed from an elastically deformed structure, it will recover completely. If, however, the stress in a material exceeds its yield point, then it will deform plastically and will retain a permanent deformation after the applied force is removed. The simplest description of the mechanical properties of the material is, therefore, a plot of stress vs. strain, from zero to the strain at which the material fails completely.



$$\sigma = \begin{cases} E\varepsilon & \varepsilon < \varepsilon_y \\ \sigma_y \left(1 + \dfrac{E}{\sigma_y}\varepsilon_p\right)^n & \varepsilon > \varepsilon_y \end{cases}$$

Key

E is Young's modulus

σ_y, ε_y are the yield point coordinates

ε_p is the nonlinear part of the total accumulated strain beyond ε_y

ε_r is the elasto-plastic strain induced by σ_r, the stress above the yield point

Figure 1 — Schematic of a typical true stress-strain curve for a work-hardening metal

Figure 1 shows just such a curve. From this curve, the key tensile properties of the material can be obtained.

— Young's modulus E is the gradient of the initial portion of the curve. It is also the gradient of the straight line along which elastic recovery occurs from any point along the curve.

— The deviation of the curve from a straight line marks the yield point, often described as the yield stress. A straight-line recovery, of gradient E, from any point at higher stress or strain than this point would no longer pass through the origin, i.e. plastic deformation will have occurred.

— The gradient of the curve after yielding is a measure of the work hardening of the material, i.e. elastic recovery occurs along a straight line, gradient E, and re-stressing the material also follows the same line such that further plastic deformation only begins once the previous maximum stress has been exceeded.

— The point at which the material fails completely marks two parameters of interest, one being the ultimate tensile stress (UTS); the other being the strain at failure.

These parameters form the key material specifications for any structural or functional design. It can be seen that the stress-strain curve is an essential "fingerprint" of the type of material. An elastic then perfectly plastic material will deform elastically up to the yield stress, and then it will continue to strain at constant stress until failure occurs at the strain-to-failure point. The yield stress is therefore also the UTS. A perfectly elastic, brittle material does not have a yield point, but exhibits a straight line (gradient of the Young's modulus) until it fails by fracture. A work-hardening material yields but is able to support increasing stresses as it strains to its UTS and maximum strain at failure point. The toughness of the material is often related to the area under the curve up to the failure point. This is a measure of the energy absorbed by the material before it fails. The tougher a material is, the more energy it absorbs before failure.

Beyond extraction of the key tensile properties described above, the whole stress-strain curve is highly desirable input for the design of structures and components, to ensure that they do not yield or fail in service. Computing power has become more available and so the use of software such as Finite Element Analysis (FEA) programs, which determine the stress and strain throughout structures by considering them as an array of connected small volumes of material, is increasingly common. For a purely elastic calculation, the input parameters of Young's modulus and Poisson's ratio are exactly the same as for an analytical stress analysis. However, if plasticity is to be considered, then a yield stress is required plus a description of the amount of plastic deformation that will occur at each stress above the yield point. This in effect requires input of the entire stress-strain curve.

Measurement of the tensile properties of a material is most commonly performed using a uniaxial tensile testing machine. A sample of material is clamped in the machine and the strain is induced by the application of an ever-increasing stress (stress and strain being measured by suitable means). The exact method has improved and evolved over time, but the general principle has remained the same for centuries. It is possible to obtain the Young's modulus of a material by other means, e.g. by using acoustic wave propagation [1], and materials property reference sources often quote elasticity values obtained by just this method [2], but tensile testing is the traditional method of choice for obtaining the yield stress and the plasticity part of the stress-strain curve.

The uniaxial tensile test has the benefit of making a measurement that is very similar to the final application in an easily understood way. However, it has a number of significant drawbacks.

— It has proved surprisingly difficult to reduce the test uncertainty below the 10 % level, although recent European projects have improved the identification and control of key uncertainties (EU project TENSTAND). Alignment in the instrument and the methods used to measure strain are key sources of uncertainty, as is the wide variety of algorithms used to obtain the tensile properties from the measured data.

— The material must be available in volumes large enough to be tested. Small-scale testing and micro-tensile testing are becoming possible but have additional uncertainties.

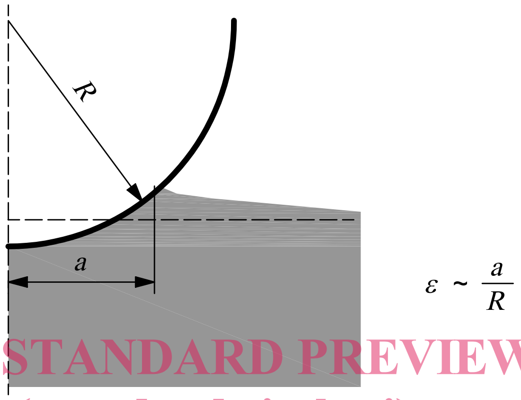
— It must be possible to machine the materials to a controlled geometry without damaging them or changing their properties (in particular their work-hardened state).

— The test is destructive and averaging includes uncertainties due to sample-to-sample inhomogeneity.

0.2 General information for indentation and tensile properties

The widespread use of FEA to simulate indentation force vs. displacement curves is ample evidence that there is a direct forward link from a stress-strain curve to the indentation response of a material. However, the increasing use of modelling and the attendant requirement to obtain the stress-strain curve as input to the models raises the question of whether it is possible to solve the inverse problem, i.e. obtain a stress-strain curve from the indentation response of a material. If this were possible, it would remove many of the drawbacks of tensile testing and revolutionize the availability of tensile property information. Nano-indentation is able to measure microscopic volumes of material, thus the tensile properties of materials that exist only as small particles or as surface treatments or coatings would become obtainable. Indentation testing can be made portable and thus non-destructive, *in situ*, on-site testing would become available, with relatively little (or no) sample preparation. Lifetime monitoring of real structures would become cheaper and easier without the need for witness specimens.

In 1951, Tabor [3] demonstrated empirically that there was clearly a relationship of some form between the hardness response and the relative strain imposed by indentation, since plots of mean indentation pressure vs. relative indentation size (the ratio of indent radius to indenter radius, a/R, see Figure 2) appeared to map onto stress-strain curves for many metals.



NOTE For a sphere, the strain induced by the indenter is proportional to a/R and is therefore a function of depth.

Figure 2 — Spherical indentation

The availability of instrumented indentation has made the collection of such information a simple matter. Indeed, there is a common instrumented indentation testing cycle, often called the ‘partial unloading’ method [4], which applies a progressively increasing force but stops at a series of steps where the force is partially removed to obtain the top part of the force-removal curve necessary to obtain the contact stiffness and contact depth (hence the contact radius, a) at that force. Progressively increasing and partially removing the force on an indenter in this way allows a wide range of indentation sizes to be applied in the same place. This makes it possible to make a truly local measurement of material response over a wide range of strains, which might then be repeated with relative ease to form a map of the mechanical properties of a material.

This Technical Report is intended to be a summary of the state of the art in deriving tensile properties from the indentation response of a material. Three approaches are described, and the key requirements, advantages and drawbacks are summarized in table form. The three methods are:

a) representative stress and strain,

b) inverse FEA methods, and

c) neural networks.

All three methods have been shown to “work”, in that they are able to obtain from indentation data a stress vs. strain relation that can be validated against tensile testing. However, more extensive intercomparison and sensitivity analyses are necessary to establish the robustness of each method’s ability to identify the unique, best solution to the problem.

The three methods described all start from the assumption that input of the correct stress-strain curve into a suitable FEA package will enable exact simulation of the observed indentation response. Therefore, in principle, the inverse method is a brute force simulation, using all possible combinations of input parameters until the best fit to the measured indentation response is found. Such an inverse method is the benchmark method, as it can unambiguously identify the globally best solution and, if convergence is not possible, can identify that fact and demonstrate where the lack of convergence lies. Surprisingly, the increasing availability of distributed computing networks makes this less unlikely than it might at first seem. It is clear, however, that any method that can economise on this amount of effort and obtain equivalent results (perhaps validated against selected distributed computing solutions) would be preferable. All of the methods described here are, in effect, different strategies for reducing the computing required by the user.

The representative stress vs. strain approach uses FEA to generate a one-off set of simulations, and uses empirical fitting to this set of results to derive general results. These relationships then place only a very low computational load on the user because no further FEA is required to obtain specific results. The best results are obtained by grouping materials with similar stress vs. strain relationships and generating a set of representative relationships for each group. Each group can be classified according to material hardening behaviour, e.g. as power-law hardening material or linear hardening material. For blind testing, these classifications can be made before testing by considering material factors such as magnetism and σ_y/E. The key user requirement is therefore to ensure that the correct sets of empirical relationships are used for the material being tested. This method is well suited to users of a small range of materials, or a range of similar materials, who wish to check or track the material properties over time.

The class of inverse methods in this Technical Report are distinguished by retaining the need for the user to perform some form of FEA simulation for each stress-strain curve obtained. A number of strategies for reducing the computational load by using other information obtainable from the indentation experiments are described. This method is the most flexible, in that it can obtain a result from an unknown material. It is therefore well suited to users who have FEA ability and need to be able to test any material without prior knowledge of what that material might be. This method typifies an approach wherein the objective is to find values for material parameters that minimize the variation between the experimental indentation data and the functional output by simulation. The values obtained by this method include uncertainties, and a proper calculation of these uncertainties must be considered.

In the final category, the method is a trained neural network. This can be thought of as a sophisticated method of encapsulating a large number of pre-generated FEA-derived solutions. The network is trained using a particular material response model until it has developed a function that enables it to predict a result for situations that fall between the exact solutions it has “learned”. This method therefore sits between the first two methods. The computational load to run a neural network is much lower than FEA, and is in many ways a “black box” to the user. It is able to deal with a wide range of unknown materials. However, it does have a limit to its abilities, which is defined by the size and quality of the input data and the extent and validity of the training process.

The objective of the methods described in this Technical Report is to derive a true stress-strain curve from indentation data measured experimentally and to obtain tensile properties such as yield strength, tensile strength and work-hardening exponent from the derived curve.

When using these methods to obtain tensile properties from indentation, it should be taken into account that indentation is, by comparison with bulk uniaxial tensile testing, predominantly a local property measurement. The mechanical response measured is from a very small volume of material close to the indentation site. This has the benefit of high spatial resolution testing and enables property mapping. For instance, its localized nature allows testing of the heat-affected zones of weldments, which cannot be tested destructively because of their irregular shape and small volume.

It has also the drawback that a local measurement is not always representative of the bulk-averaged response. Empirical observation indicates that indentation into metals creates a plastic deformation zone under the indenter that typically extends below the surface to about ten times the indentation depth. This is a practical limit to the region of validity of the information obtained, as beyond this depth the material is deformed only elastically. For example, if a case-hardened material, where the surface properties differ significantly from those of the bulk, were tested, the results would reflect the properties of the surface and not the bulk.

The methods in this Technical Report allow the derivation of a true stress-strain curve for the material tested by the application of particular models. Tensile properties, such as yield stress and ultimate tensile strength, are then inferred from these curves, but are not directly measured. Thus, the tensile properties obtained by the methods in this report are not intended to replace the requirement for destructive uniaxial tensile testing in the laboratory, where conditions make this possible. One of the greatest advantages of the instrumented indentation test (IIT) lies in non-destructive testing of in-service components in field applications where tensile testing is not available. Table 1 summarizes the characteristics of the indentation method and tensile testing.

Table 1 — Comparison of the main features of the tensile test and the instrumented indentation test (IIT)

	Tensile test	IIT
Properties characterized	Bulk (average)	Local (surface)
Testing nature	Destructive	Non-destructive
Sample preparation	Machining	Surface polishing
Potential examples	Laboratory (conventional) Large volume	In-field Small volume

Metallic materials — Measurement of mechanical properties by an instrumented indentation test — Indentation tensile properties

1 Scope

This Technical Report describes methods for evaluating tensile properties of metallic materials (true stress-strain curve and derived parameters) using an instrumented indentation test.

The ranges of application of instrumented indentation tests are in line with the classification of ISO 14577-1, but the range of force recommended is from 2 N to 3 kN.

This Technical Report includes the following three methods, all of which are sound in principle, are capable of practical use and are appropriate for the specified materials.

— Method 1: representative stress and strain;

— Method 2: inverse analysis by FEA;

— Method 3: neural networks.

In every method, tensile curves are derived from the experimentally measured indentation force-depth curve, from which indentation tensile properties are evaluated. The three methods described all need different user strategies and abilities to obtain the indentation tensile properties. The information required differs for each method and is described in detail in Clause 5.

The main assumption in the three methods is the absence of residual stress within the test piece. Existing residual stress can affect the estimation of indentation tensile properties. A procedure for evaluating residual stress using an instrumented indentation test is given for reference in Annex A.

2 Normative references

The following referenced documents are indispensable for the application 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 14577-1:2002, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 1: Test method*

ISO 14577-2:2002, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 2: Verification and calibration of testing machines*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
instrumented indentation test
IIT
test to appraise mechanical properties of a material by measuring test force and indentation depth when a test piece is indented with an indenter

NOTE See ISO 14577-1.

3.2
indentation tensile properties
mechanical properties of materials such as indentation yield strength, indentation tensile strength and indentation work-hardening exponent, obtained by analysing a true stress-strain curve determined by instrumented indentation testing

4 Symbols and designations

For the purposes of this Technical Report, the symbols and designations in Table 2 are used.

Table 2 — Symbols and designations in common

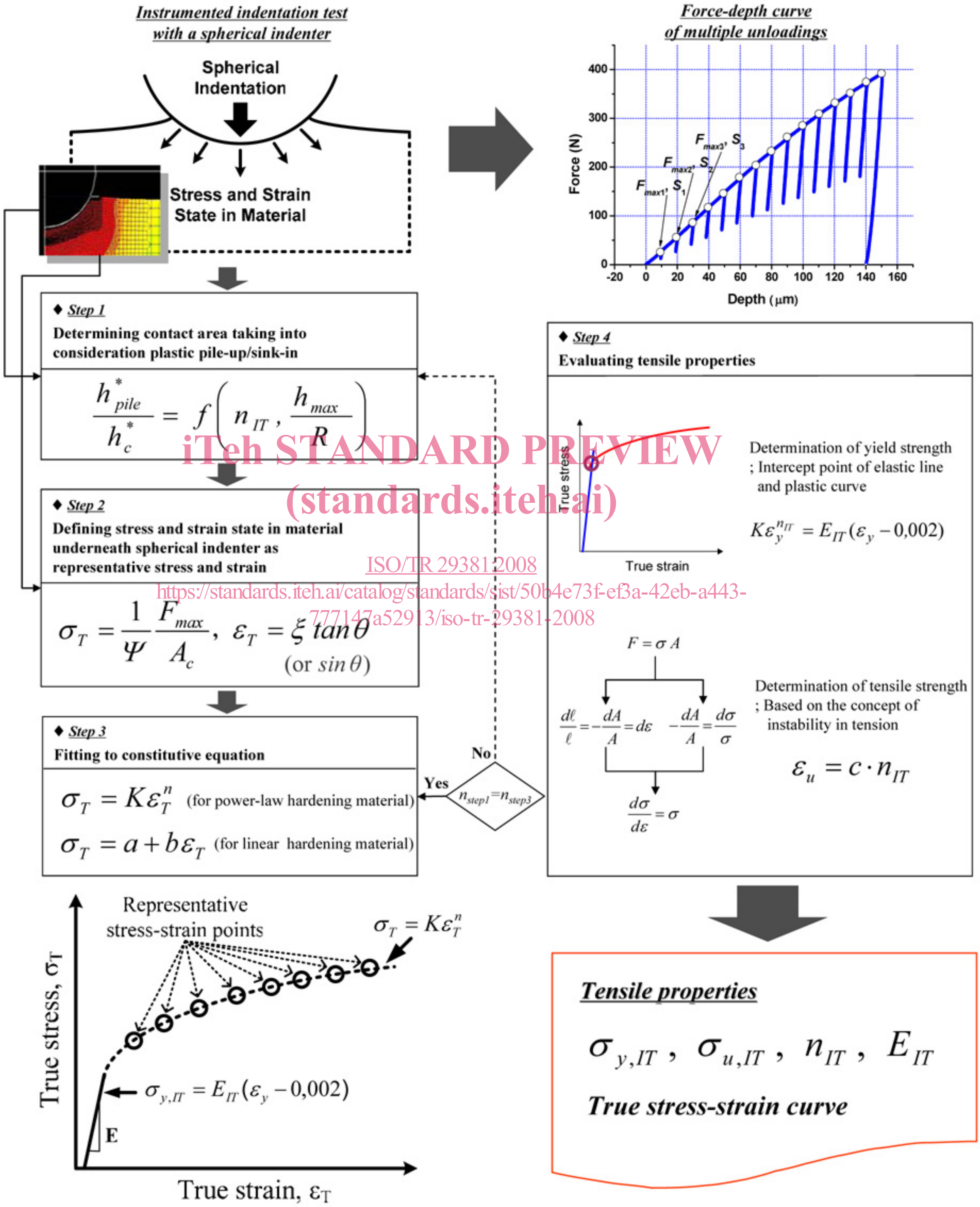
Symbol	Designation	Unit
F	Test force	N
F_{max}	Maximum test force	N
h	Indentation depth	mm
h_{max}	Maximum indentation depth at F_{max}	mm
h_c	Depth of contact of the indenter with the test piece at F_{max}	mm
h_p	Permanent indentation depth after removal of test piece at F_{max}	mm
R	Radius of spherical indenter	mm
$A_p(h_c)$	Projected area of the contact of the indenter at distance h_c from the tip	mm^2
H_{IT}	Indentation hardness	N/mm^2
E_{IT}	Indentation modulus (Young's modulus)	N/mm^2
$\sigma_{y,IT}$	Indentation yield strength	N/mm^2
$\sigma_{u,IT}$	Indentation tensile strength	N/mm^2
n_{IT}	Indentation work-hardening exponent	—
α	Angle, specific to the shape of the sharp indenter	°
σ_T	True stress	N/mm^2
ε_T	True strain	—
S	Stiffness (the slope of tangent to unloading curve at F_{max})	N/mm
NOTE 1 To avoid very long numbers, the use of multiples or sub-multiples of the units is permitted.		
NOTE 2 1 N/mm^2 = 1 MPa.		

5 Descriptions of the different methods

5.1 Method 1: Representative stress and strain

5.1.1 Principle (see Figure 3)

True stress-strain points on the tensile curve are obtained by defining the stress and strain states in a material at various indentation depths formed by a spherical indenter as representative stress-strain points. Indentation tensile properties can be evaluated by fitting the constitutive equation to the true stress-strain points.



Key

a, b, c correlated constants

n strain hardening exponent

Figure 3 — Principle of Method 1