TECHNICAL REPORT

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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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Foreword

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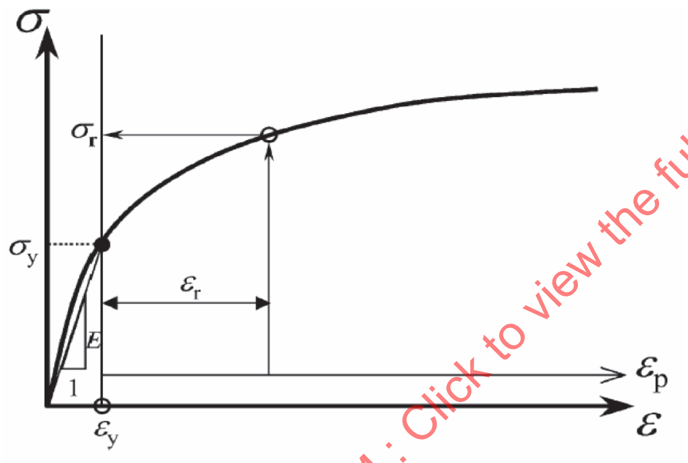
ISO/TR 29381 was prepared by Technical Committee ISO/TC 164, *Mechanical testing of metals*, Subcommittee SC 3, *Hardness testing*.

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.

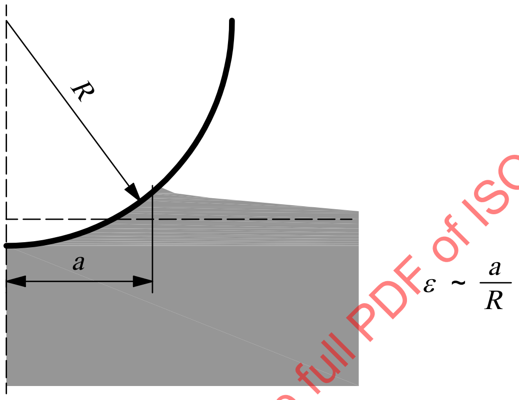
— It must be possible to machine the materials to a controlled geometry without damaging them or changing their properties (in particular their work-hardened state).

— 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

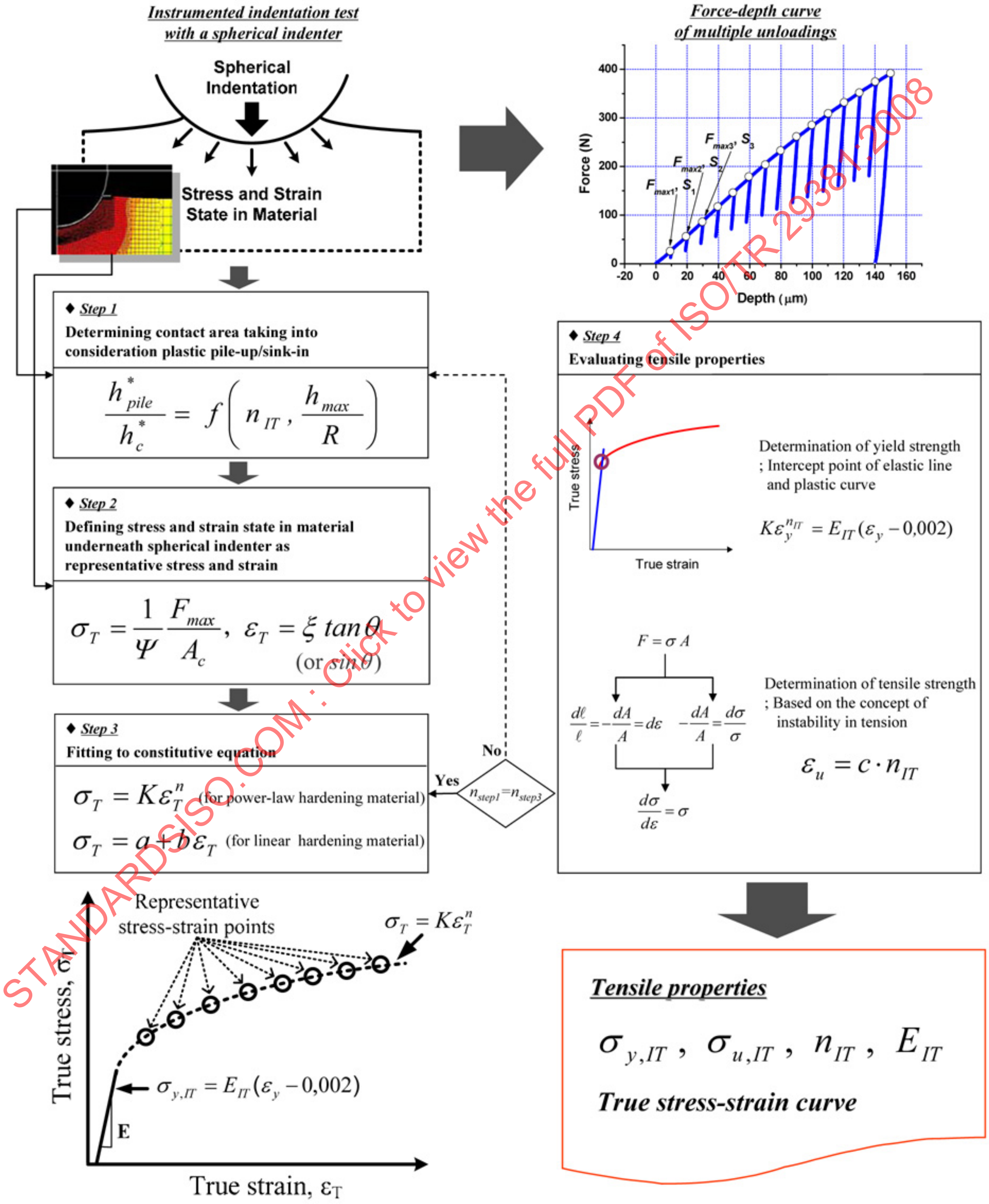
Symbol	Designation	Unit
F	Test force	N
F_{max}	Maximum test force	N
h	Indentation depth	mm
h_{max}	Maximum indentation depth at F_{max}	mm
h_c	Depth of contact of the indenter with the test piece at F_{max}	mm
h_p	Permanent indentation depth after removal of test piece at F_{max}	mm
R	Radius of spherical 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

5.1.2 Symbols and designations for Method 1

See Table 3.

Table 3 — Symbols and designations for Method 1

Symbol	Designation	Unit
h^*_{pile}	Height of plastic pile-up	mm
h^*_c	Elastic contact depth	mm
A_c	Projected area of the contact of the indenter at distance h_c correlated to h^*_{pile} and h^*_c from the tip	mm^2
Ψ	Plastic constraint factor	—
ξ	Proportional constant of true strain and tanθ	—
θ	Half of contact angle	°
K	Strength coefficient	—
ε_u	True uniform strain	—
NOTE For power-law hardening materials, $\Psi = 3$ and $\xi = 0{,}14$ are useful values.		

5.1.3 Testing machine

The testing machine should be

- capable of measuring and reporting applied force, indentation depth and time throughout the testing cycle;
- capable of applying predetermined test forces within the required scope of the test;
- capable of applying test forces of multiple unloading;
- protected from shock and vibration, air movements and variations in temperature that can significantly influence the test result.

The indenter used should be a spherical indenter made of polished tungsten carbide or a material having greater stiffness than that of the test piece. The user is referred to ISO 14577-2 for further specifications.

Further information on machine compliance, utilizing the appropriate indenter area function, indenter shapes and temperature calibration, are found in Clause 5 of ISO 14577-1:2002;

5.1.4 Test piece

See Clause 6 of ISO 14577-1:2002 for a description of the test piece.

5.1.5 Procedure for obtaining force-depth curve

The user is referred to specifications in Clause 7 of ISO 14577-1:2002.

In order to avoid significant increase in testing machine compliance, firm support or fixing of the test piece is recommended.

The indentations should be positioned at least three times their indentation diameter from interfaces or free surfaces; the distance between indentations should be at least five times the largest indentation diameter.

Partial repetition of the force-removal procedure and acquisition of indentation parameters such as F_{max}, h_{max} and S from each partial unloading is carried out to obtain the data necessary to generate the force-depth curve.

The testing cycle should be either indentation depth-controlled or force-controlled, with a full description of all parts of the testing cycle given in the test report, including:

a) the nature of the control (i.e. force or displacement control);

b) the displacement (or force) application/removal rate;

c) the length and position of each hold period;

d) the diameter of the spherical indenter;

e) the maximum indentation depth ratio or maximum displacement or maximum test force;

f) the force-removal ratio;

g) the number of force-removal curves;

h) the data-logging frequency (or number of data points).

See Figure 4 for a typical force-depth curve obtained with Method 1.

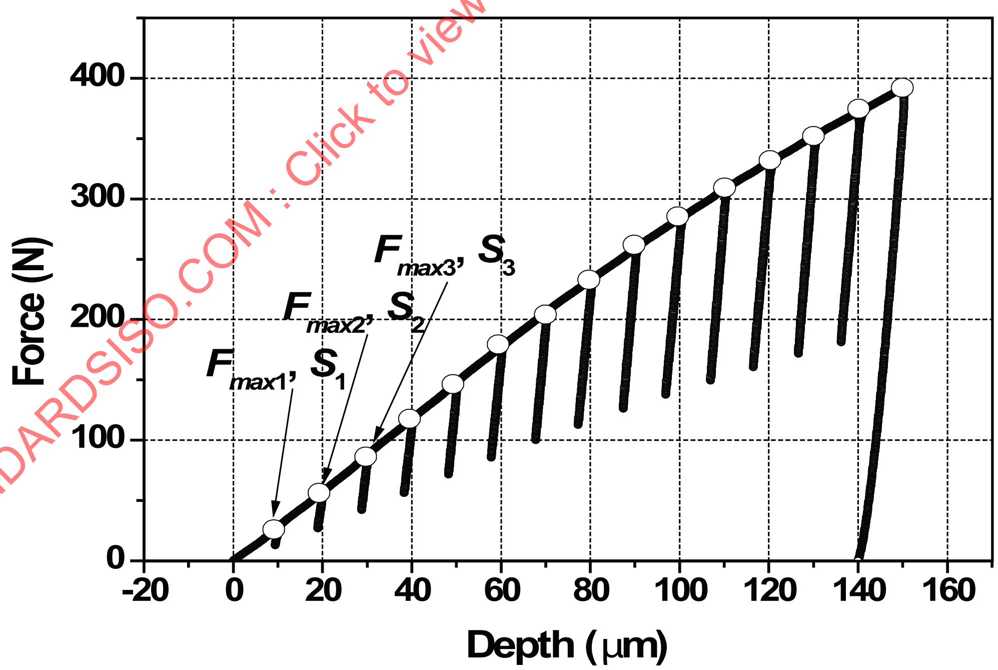
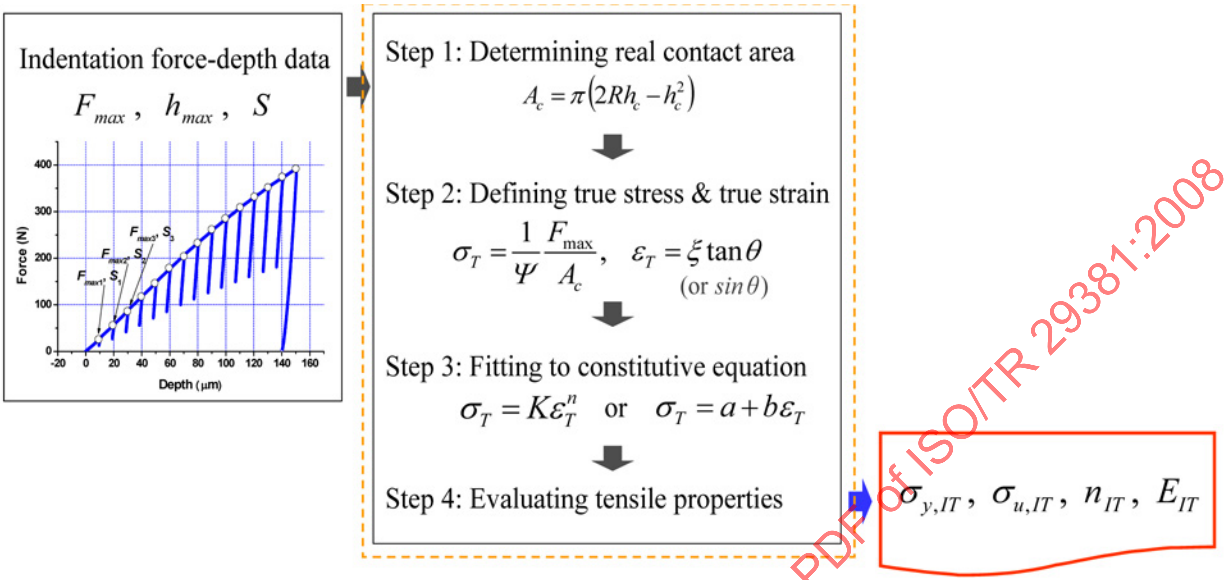


Figure 4 — Example of force-depth curve

5.1.6 Data analysis

See Figure 5.



Key

a, b constants

Figure 5 — Steps for data analysis

5.1.6.1 Step 1 — Determining real contact area

Carry out the following procedures to determine the real contact area:

a) determine the plastic pile-up/sink-in height, h_{pile}^*, by predetermined quadratic equation for n_{IT} and h_{max}/R;

b) determine the real contact area, A_{c}, from the real contact depth h_{c} correlated to h_{pile}^* and h_{c}^*.

5.1.6.2 Step 2 — Defining true stress/strain

Carry out the following procedures to define true stress/strain:

a) define the true strain from the estimated contact angle θ,

b) define the true stress from the F_{max} obtained in the force-depth curve and the A_{c} determined in 5.1.6.1 b).

5.1.6.3 Steps 3 and 4 — Evaluating indentation tensile properties

Carry out the following procedures to evaluate indentation tensile properties:

a) fit the true stress-strain points to a relevant constitutive equation such as $\sigma_{\text{T}} = K\varepsilon_{\text{T}}^n$ for power-law hardening materials or $\sigma_{\text{T}} = a + b\varepsilon_{\text{T}}$ for linear hardening materials;

b) determine the elastic modulus from the contact stiffness;

c) determine the yield strength from the intercept point of the linear curve with the elastic modulus slope and the plastic flow;

d) determine the tensile strength based on the concept of instability in tension.

5.1.7 Experimental evidence

See Figures 6, 7 and 8. For additional publications and patents concerning Method 1, see references [5] to [9].

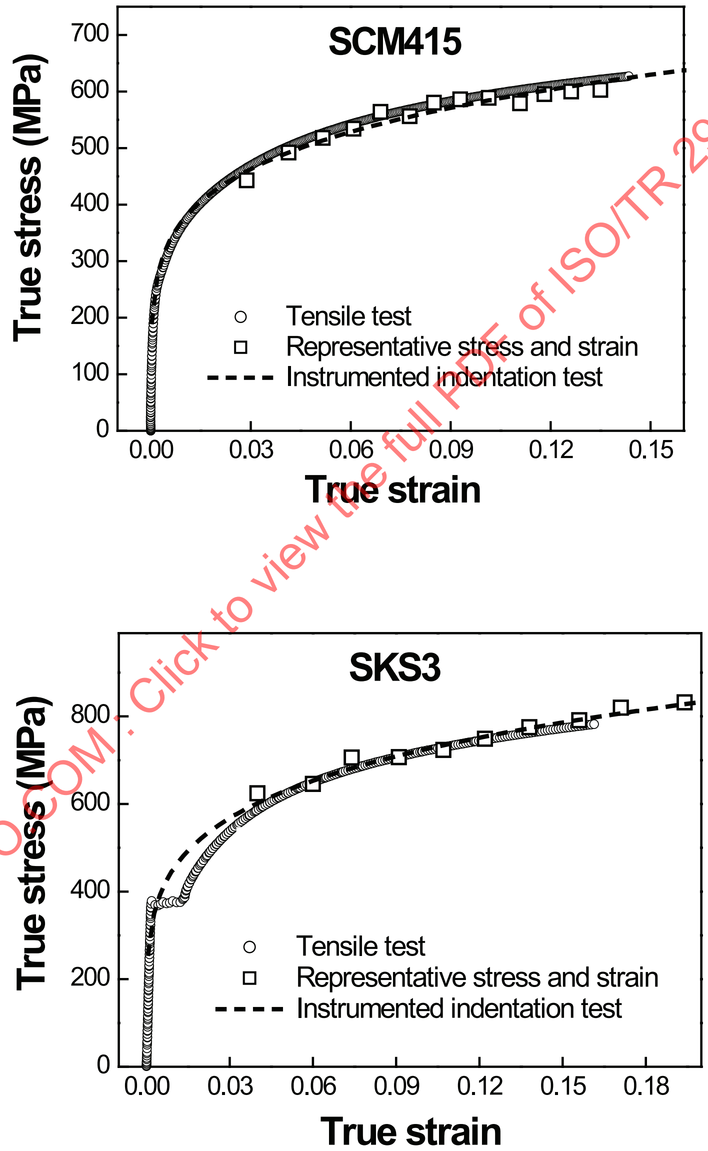
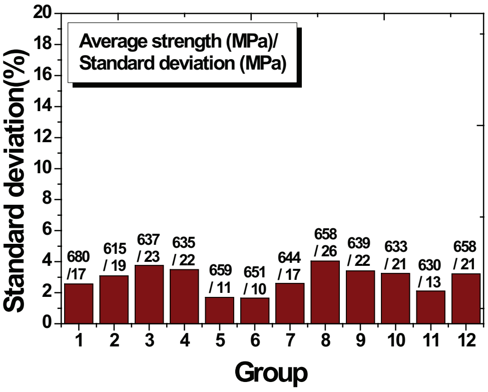
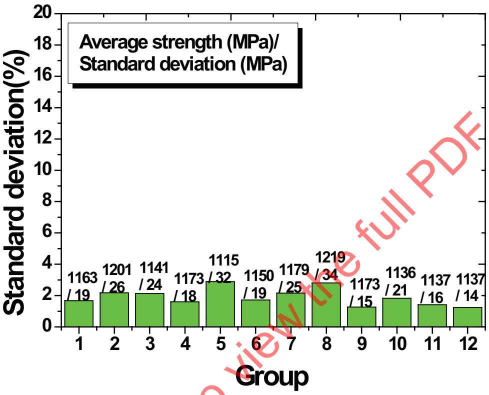


Figure 6 — Examples for data analysis
(see References [5], [6])



a) Yield strength



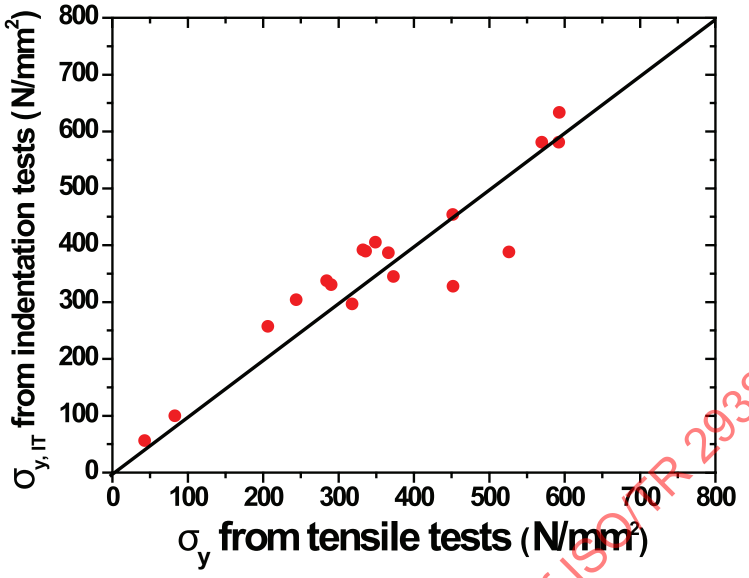
b) Tensile strength

Key

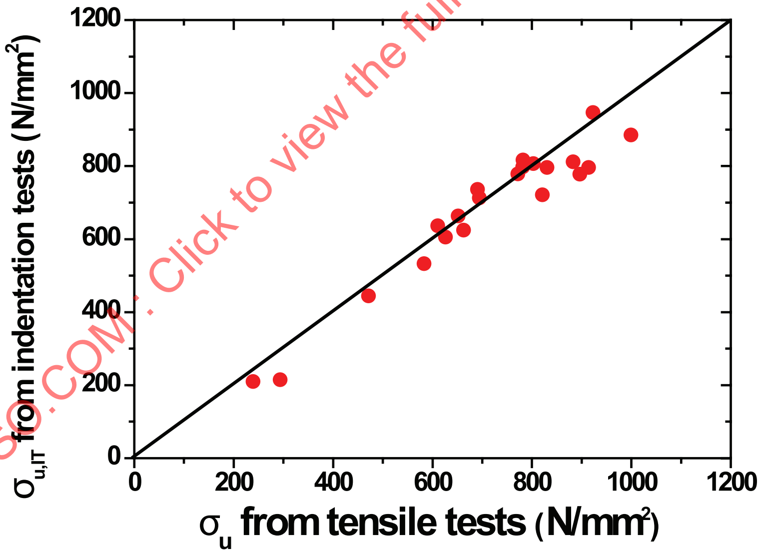
1 Korea Gas Corporation
2 Agency of Technology & Standards
3 Korea Power Engineering Co., Inc.
4 Korea Institute of Science & Technology
5 Korea Institute of Machinery & Material
6 Seoul National University
7 Cho-sun University
8 Daewoo Electronics
9 Doosan Heavy Industries & Construction
10 Korea Gas Safety Corporation
11 Hyundai Mobis
12 POSCO

NOTE The average standard deviation of the results returned from the different laboratories was 2,90 % in yield strength and 1,90 % in tensile strength. Participation groups (total 12 groups, 5 repeat experiments per group).

Figure 7 — Results from a round-robin test using machines and indenters of a single type



a) Yield strength



b) Tensile strength

Figure 8 — Comparison with tensile tests for over 20 metallic materials

5.1.8 Relative advantages and disadvantages of Method 1

a) Advantages:

- deterministic algorithm;
- no need for computational simulations;
- sufficient experimental verification data;
- completion of national round-robin test.

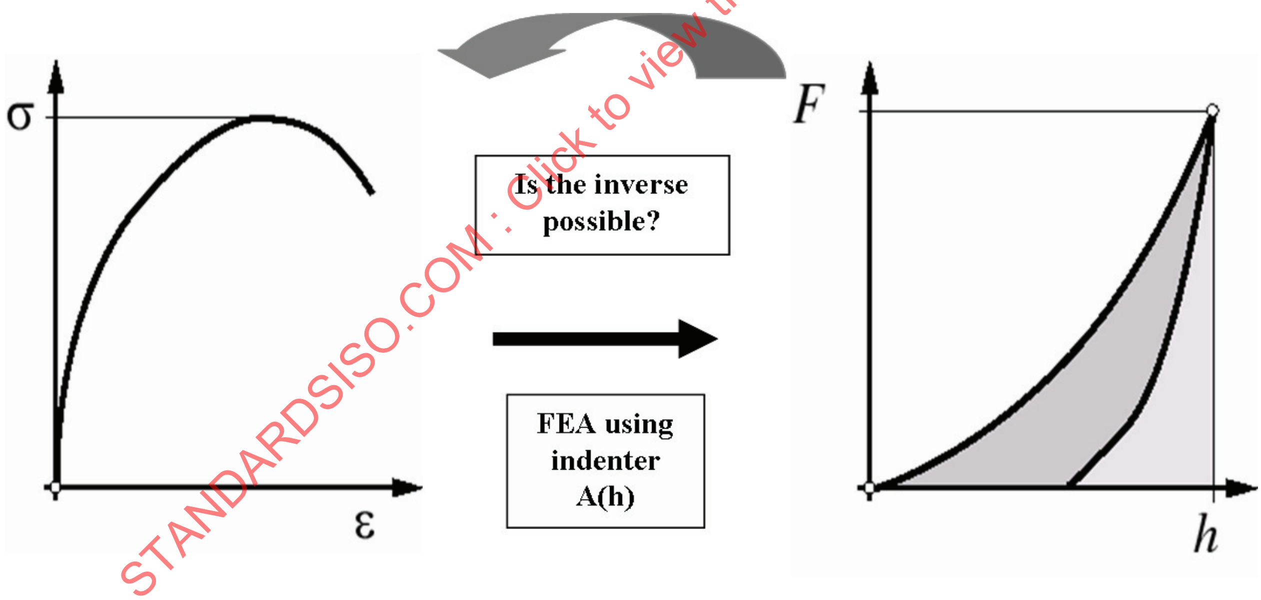
b) Disadvantages:

- rigid algorithm;
- numerical estimations of empirical parameters.

5.2 Method 2: Inverse analysis by FEA

5.2.1 Principle

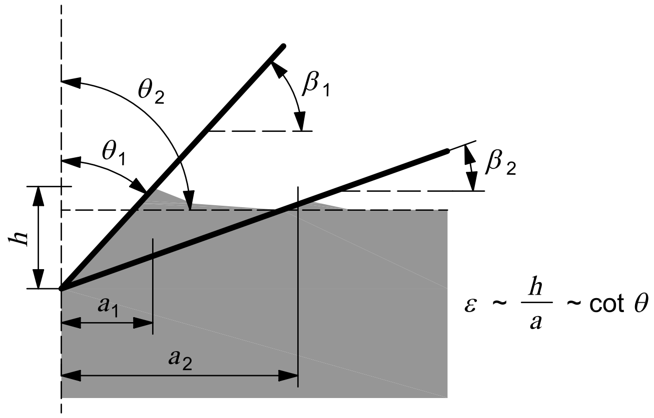
Over many years, researchers have used Finite Element Analysis (FEA) to simulate indentation responses. However, since indentation testing is often much less destructive and much more convenient to perform (even in the field), there has been an increasing interest in the inverse problem, i.e. the determination of the stress-strain curve from the indentation response. (See Figure 9.)



NOTE It is routine to go from the stress-strain curve to indentation response using Finite Element Analysis.

Figure 9 — Description of inverse analysis

There have been a number of approaches to obtaining the stress-strain curve by indentation and researchers have disagreed about whether it is possible to obtain a unique stress-strain curve from a series of indentation responses, since, in principle, there are an infinite number of routes to the same stress-strain point.



NOTE For a conical indenter, the strain is proportional to the tangent of the cone angle and thus is constant with indentation depth.

Figure 10 — Conical indentation

Some have claimed that, if only a short-range optimization is performed, it is possible to have practical solutions to the problem. An example of this is the procedure developed by Bouzakis *et al.* [11] whereby the force-increasing curve of a spherical indentation is used to represent the case of monotonically increasing strain. Each point of the loading curve is simulated sequentially (presuming a first guess of the elastic properties of the material) using the history of the previous point and adding to the input stress-strain curve of the FEA code by making the assumption that only a change in the tangent modulus of the stress-strain input curve is required to generate the next strain point in the FEA input data. The FEA solution is then iteratively calculated until a match with the experimental force-displacement point is found. In this way, the infinity of possible routes to the individual point in the force-displacement curve is collapsed onto the previously determined input data and it is only the route to the next (higher strain) point that is allowed to vary. Thus a series of local optimizations can be performed without worrying that a better global optimum has been missed. Indeed, such an approach can benefit from constrained optimization routines if additional information, such as the Young's modulus and yield stress, can be estimated.

There are therefore two distinct phases in obtaining the indentation data necessary to derive the stress-strain relationship for a material:

— obtain indentation stress at a range of different effective strain values;

— calculate the stress-strain relationship necessary to enable optimum simulation of the experimental data.

5.2.2 Symbols and designations for Method 2

See Table 4.

Table 4 — Symbols and designations for Method 2

Symbol	Designation	Unit
θ	Angle, specific to the shape of the sharp indenter	°
i	Number of digitalized force-depth pairs during loading	—
j	Number of produced tangent moduli	—
d_F	Deviation tolerance	%
M	Tangent modulus	N/mm^2
M_1	Elastic modulus E_{IT}	N/mm^2
S_i	von Mises stress	N/mm^2
ε_i	von Mises strain	—

5.2.3 Testing machine

The testing machine should be

- capable of measuring and reporting applied force, indentation depth and time throughout the testing cycle;
- capable of applying predetermined test forces within the required scope;
- capable of measuring the surface profile of the residual impression;
- protected from shock and vibration, air movements and variations in temperature that can significantly influence the test result.

Indenters of various shapes and angles may be used, such as spherical, conical, Vickers and Berkovich (specified in ISO 14577-2). For spherical indenters, only those of large indenter radii ($R > 200$ µm) are recommended, unless indentation size effect corrections are made.

Further information on machine compliance, utilizing the appropriate indenter area function, indenter shapes and temperature calibration, are found in Clause 5 of ISO 14577-1:2002;

5.2.4 Test piece

See Clause 6 of ISO 14577-2:2002 for a description of the test piece.

5.2.5 Data capture procedure

5.2.5.1 Multiple cones

It is possible to obtain indentation data using a range of different conical indenters, where the effective strain of the indentation is related to the angle of the cone. The advantage here is that data are obtained by performing a series of single, simple indentation cycles. The disadvantage is that the indenter has to be changed between each experiment, and it is not possible to obtain all the information from a single point. Thus any inhomogeneity in the sample will distort the obtained stress-strain relationship. It is also critical to have a large number of certified indenters, so that the cone angles are well calibrated.

5.2.5.2 Spherical indentation

A more practical method is to use a spherical indenter. A sphere has the advantage that the effective strain changes as a function of indentation depth. Thus a range of indentation strains can be obtained, without changing the indenter, by performing indentations with progressively increasing force at the same spot. This method is well established in the literature. A typical procedure is to increase to a temporary maximum force, remove a fraction of the force quickly, to obtain either a force-decreasing curve or a single reduced-force point, and then increase the force again to a higher maximum value before repeating the force-removal procedure. In this way a large number of indentation maximum forces, contact depths and contact stiffnesses can be obtained from a single indentation site. It is only necessary to remove the force partially, as it is the information near to the local F_{max} that is required to define the contact response. The force is removed quickly to reduce the problems associated with indentation creep rates distorting the measured contact stiffness. Depending on the data acquisition rate of the instrumented indentation instrument, there may be a sufficient number of points in the partial force-removal curve to be analysed using the ISO 14577 method to obtain the contact stiffness and contact area. Sometimes, however, a step reduction in force is used from which only a single point on the force removal curve can be obtained. This is used to obtain the same parameters, but must by necessity assume the functional form of the unmeasured force removal curve.

5.2.6 Computational simulations (see Figure 11)

There is little difference in the form of the FEA analysis necessary to simulate the indentation curves for either cones or spheres. The independent parameters governing the force-increasing curve are: Young's modulus, yield stress, Poisson's ratio, work-hardening coefficient, and indenter geometry. In principle, these can be directly varied as inputs to the FEA model, and the least-squares error from the output to the force-displacement curve of the indentation experiment determined, fed back into the iterative process and minimized. Some workers [10,12,13] have used dimensional analysis to identify a series of dimensionless parameters which are combinations of these parameters and can be used as FEA variables instead. The literature examples quoted are generally for self-similar indenter geometries, such as a cone. The advantage here is that a large area of parameter space can be simulated in advance, allowing rapid optimization of solutions to any particular experimental curve.

To carry out Method 2 (inverse analysis by FEA):

a) Create and validate FEA model to generate simulated force-displacement curves.

b) Implement the model using direct parameters or generalized dimensionless functions.

c) Construct an inverse or iteration algorithm with the chosen parameters (e.g. dimensionless functions). It is necessary to decide which figure is best to use to determine the goodness of fit of the simulation to the experiment.

d) Run the iteration to find the stress and strain values for each indentation.

e) For cones, combine the results from the range of cone indenters used to provide a series of points on a single true stress-strain curve.

f) For a sphere, compare the FEA simulation and the results of experiments using only one indenter geometry (sphere) at different indentation depths.

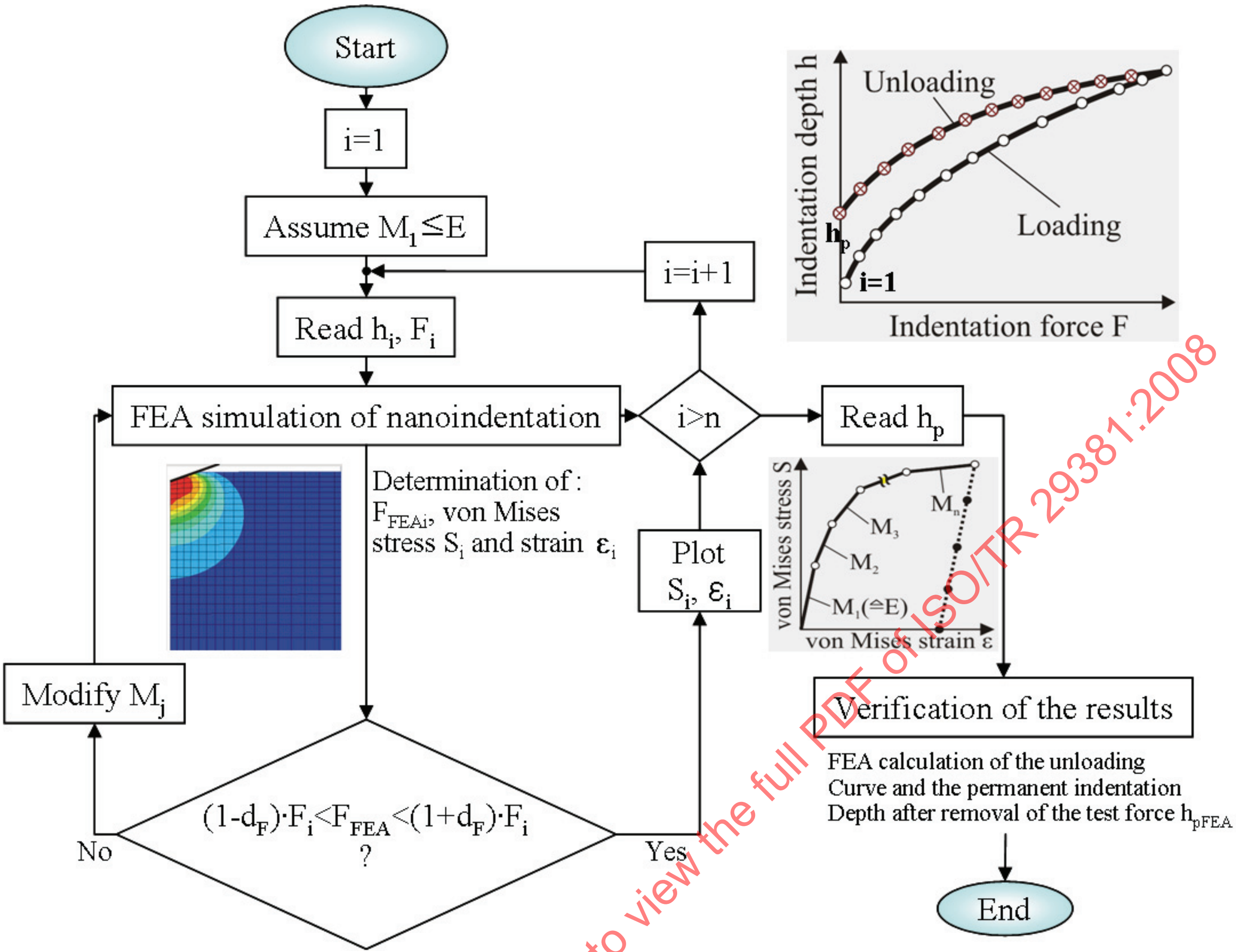


Figure 11 — Computational simulations

5.2.7 Data analysis

Several different approaches to the data analysis can be used.

a) Bouzakis method of progressive inverse modelling [11]

This works best if there is some *a priori* knowledge of the material properties, e.g. Young's modulus. Alternatively, if a large-radius sphere is used, then a large part of the indentation response may be elastic, allowing the initial elastic modulus and yield point to be obtained. Variations of this method make assumptions about the relationship between penetration depth and actual contact area.

b) Parametric approach to FEA inverse modelling [12,13]

This approach uses the mean indentation pressure, the indentation plain-strain modulus, the indenter area function and shape, and the coefficient of the fit to the force-increasing curve of the indentation data as input parameters. The algorithm then determines the representative strain for which all plots of the parameters normalized to the indentation pressure coincide on a single curve.

c) Generalized optimization method

This method can take not only the experimental indentation data but also other information, such as a fit to the surface profile of the indentations. In indentation systems that have in-built imaging capabilities, this is readily available, but it does slow down the data acquisition rate considerably. Also, if relocation of the indentation is required, this will tend to make repeated indentation at the same site impossible, thereby ruining the mapping capability of the technique. An analysis flow diagram is given in Figure 12 below. The FEA run time in this approach can be reduced by the usual practices of using a variable-density 2-D axisymmetric mesh with infinite elements to eliminate boundary effects.

d) Distributed computing approach

This is a brute force method that simply calculates all possible parameter sets and determines the global optimum solution. There are issues about software licenses for the very many processors, but the distributed computing approach is the most likely to obtain an easily validated optimum result. It is also likely to be the most effective at demonstrating the problem, should convergence not be achieved. Since this computes all possible solutions, it could in principle be compatible with the more generalized optimization approach, especially if the other input data can be directly compared with existing output from the FEA simulations.

e) Genetic programming

This emerging technique uses an adaptive algorithm to optimise the relationship between the data and the model. This method should be better than prescribing a particular functional form to a fit, as it allows the functional form also to be optimized. It is likely, therefore, that the optimized functional form of the model is one that has real physical meaning. Depending on the complexity of implementation, this approach may be the most adaptable to different material types and more efficient than the distributed computing approach, which has a fixed number of computations to do and cannot obtain an advantage from a good first guess. There are some similarities to neural networks in this approach, but the output is, at least in principle, more accessible to verification than a neural network.

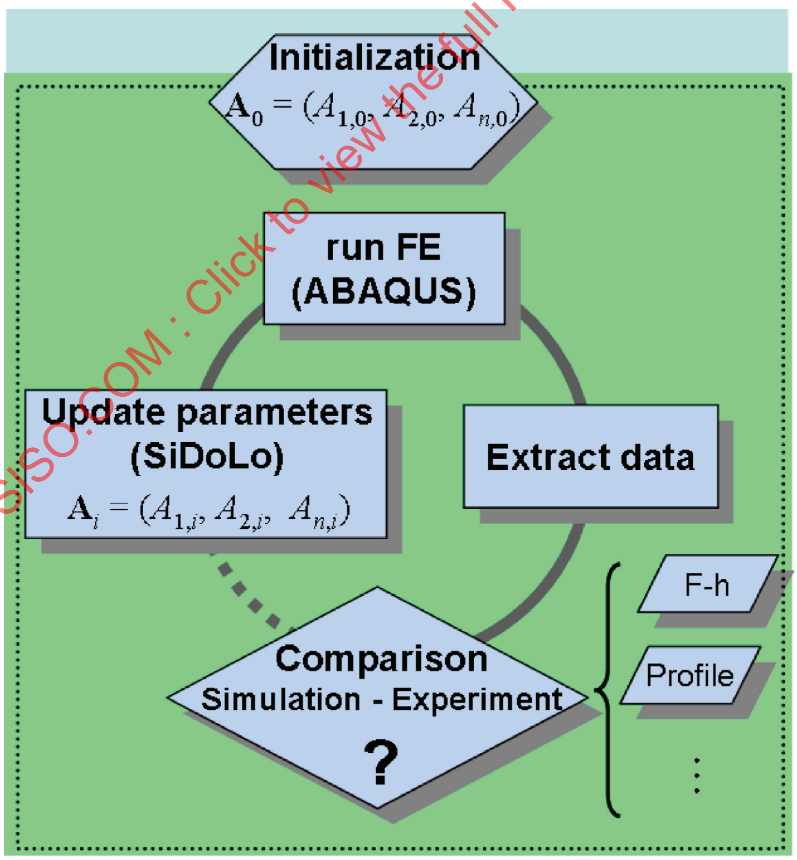
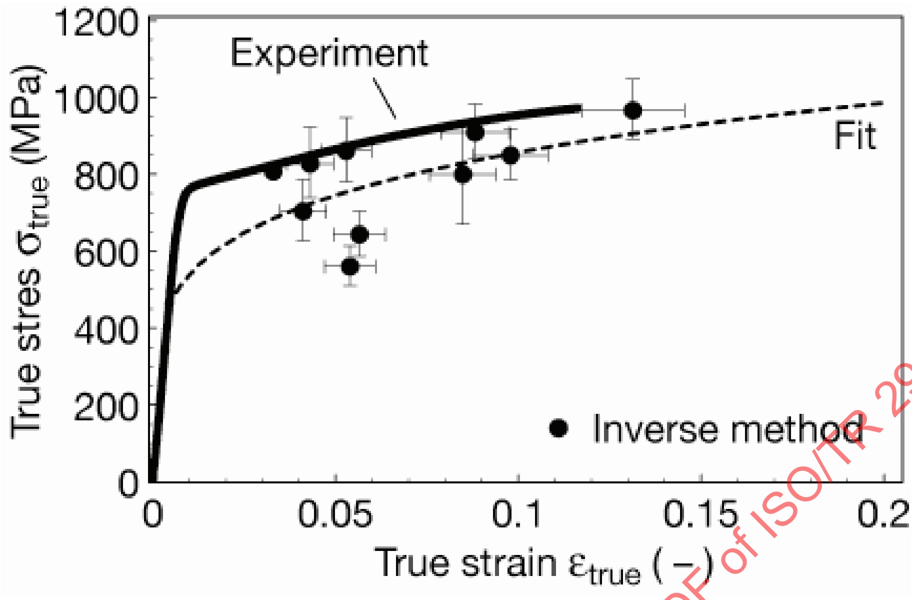


Figure 12 — Example of an optimization flowchart for determining stress-strain relationships from a range of input data

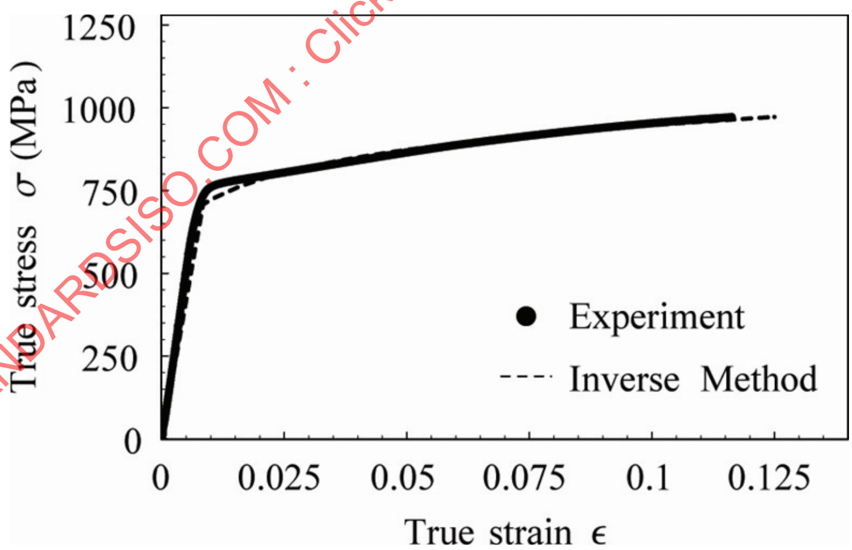
5.2.8 Experimental evidence

See Figure 13. For additional publications and patents concerning Method 2, see references [10] to [15].



NOTE CuCo$_2$Be indented with a series of conical indenters with different cone angles 38,6°, 47,0°, 50,0°, 51,0°, 61,7° and Vickers (70,3°) is compared with that from uniaxial tensile testing using a Zwick Universal testing machine. (Subsequent analysis showed that the two low-stress outliers were from damaged indenters.) Ignoring these brings the fit much closer to experiment.

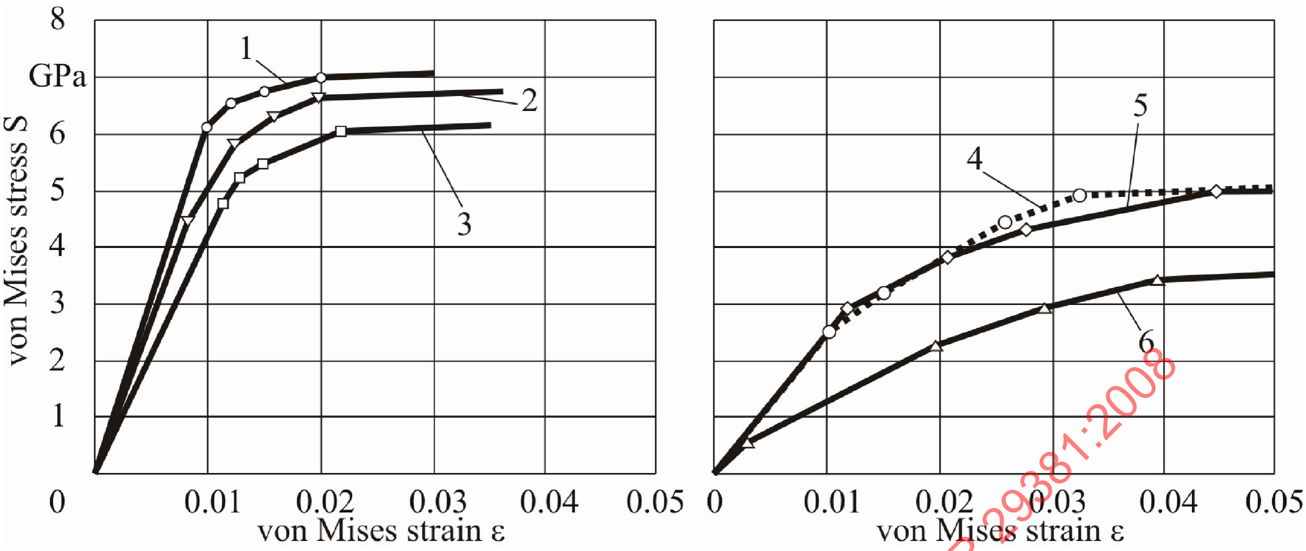
a) CuCo$_2$Be indented with a series of conical indenters



NOTE CuCo$_2$Be indented with a WC sphere (R = 0,5 mm) using a Zwick Universal testing machine.

b) CuCo$_2$Be indented with a WC sphere

Figure 13 (*continued*)



Key

1 $(Ti_{46}Al_{54})N$ PVD coating, $t = 2$ µm

2 Cemented carbide K05 – K20

3 Ceramic Al_2O_3 – TiC (24 % Al; 67 % O; 7,5 % Ti; 1,8 % C)

4 High speed steel (S-6-5-2)

5 Hardened steel (1 % C; 0,5 % Si; 1,3 % Cr; 0,3 % Mn)

6 Si (100)

c) Comparison curves

Figure 13 — Experimental evidence

5.2.9 Relative advantages and disadvantages

a) Advantages:

— application of any indenter geometry;

— flexible algorithm (implementation of new materials model).

b) Disadvantages:

— expertise in FEA;

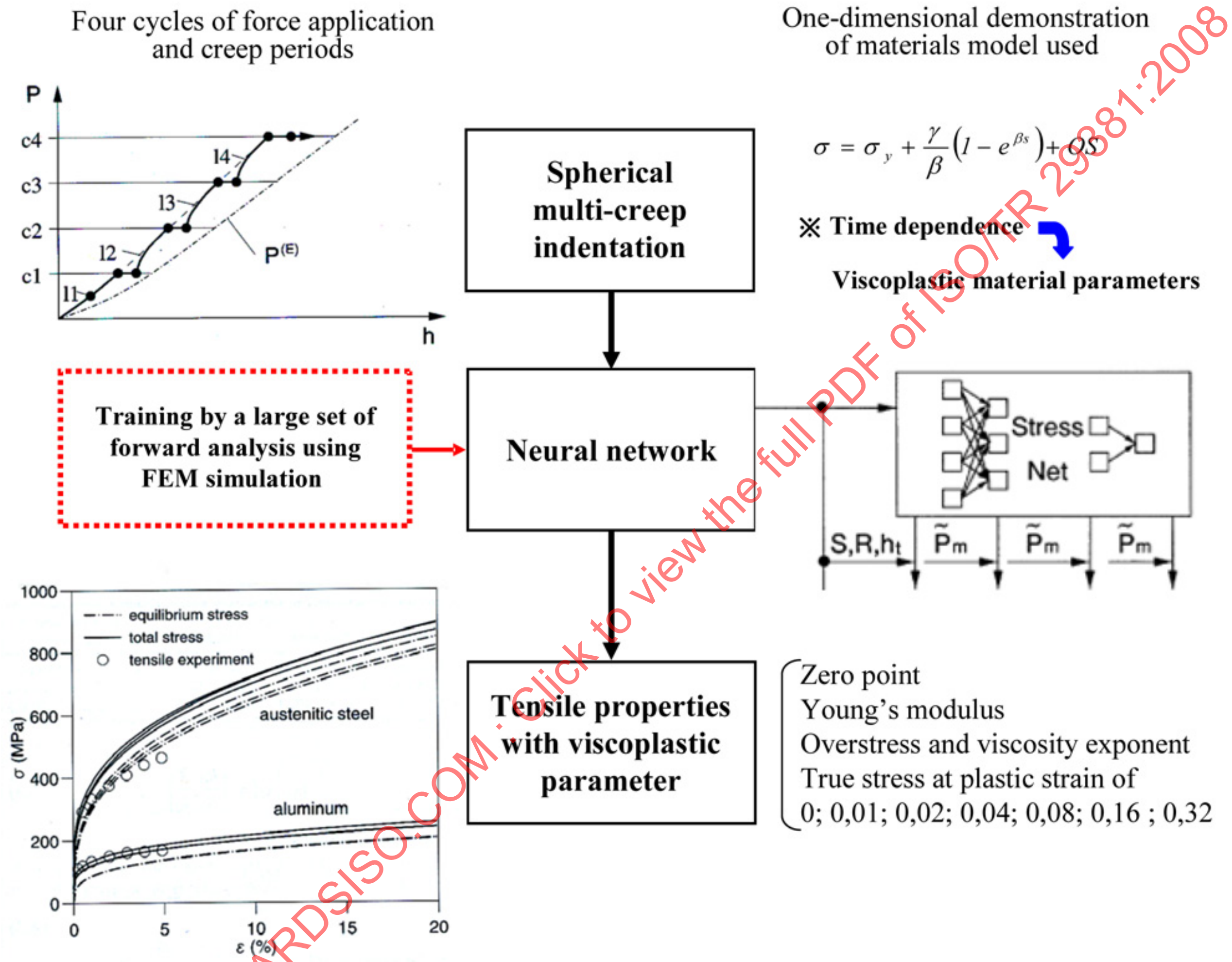
— need for special software (FEA code, optimization program);

— indispensable determination of starting parameters.

5.3 Method 3: Neural networks

5.3.1 Principle

The principle of Method 3 is illustrated in Figure 14. In the example, using the viscoplastic materials model and four cycles of force application, including creep periods, the spherical indentation response was simulated by FEA. A randomly chosen set of a pair of input (force-depth data) and output (materials parameter) vectors was used for training the neural networks. In this manner, the given trained neural network is able to learn an approximate relation between input and output data.



Key

P force

$P^{(E)}$ equilibrium response

$\tilde{p}_m$ mean pressure under contact

Figure 14 — Principle of Method 3

5.3.2 Symbols and designations for Method 3

See Table 5.

Table 5 — Symbols and designations for Method 3

Symbol	Designation	Unit
η	Viscosity parameter	$(N/mm^2)^m \cdot s$
m	Viscosity exponent	—
C	Elasticity tensor	—
F_0, h_0, t_0	Values at the end of the last loading segment	N, µm, s
k	Isotropic hardening	N/mm^2
t	Time	s
β	Work-hardening parameter	—
Σ	Maximum equilibrium stress	N/mm^2
γ	Initial slope of work hardening	N/mm^2
OS	Overstress	N/mm^2
s	Accumulated inelastic strain	—
ε_e	Elastic strain	—
ε_i	Inelastic strain	—
$\dot{\varepsilon}_i$	von Mises strain	s^{-1}

5.3.3 Testing machine

The testing machine should

— be capable of measuring and reporting applied force, indentation depth and time throughout the testing cycle;

— be capable of applying predetermined test forces within the required scope;

— ensure that the force plateau is approached in 2 % of the force application time without over-force;

— ensure that the force increase is linear up to 99 % of that force plateau;

— ensure that relative fluctuations of the force during the period of creep do not exceed 2 ‰.

The indenter used should have spherical geometry up to a depth of 0,12 R with a profile shape tolerance of 4×10^{-3} R. The user is referred to ISO 14577-2:2002 for further specifications.

Further information on machine compliance, utilizing the appropriate indenter area function, indenter shapes and temperature calibration are found in Clause 5 of ISO 14577-1:2002.

5.3.4 Test piece

See Clause 6 of ISO 14577-2:2002 for a description of the test piece.

5.3.5 Computational simulations (see Figure 15)

To carry out Method 3 (use of neural networks), the following approach is used for computational simulations:

a) construction and training of neural networks through a large set of multi-creep indentation tests and FEA simulations;

b) verification of neural networks by the latest set of FEA that is different from the training set.

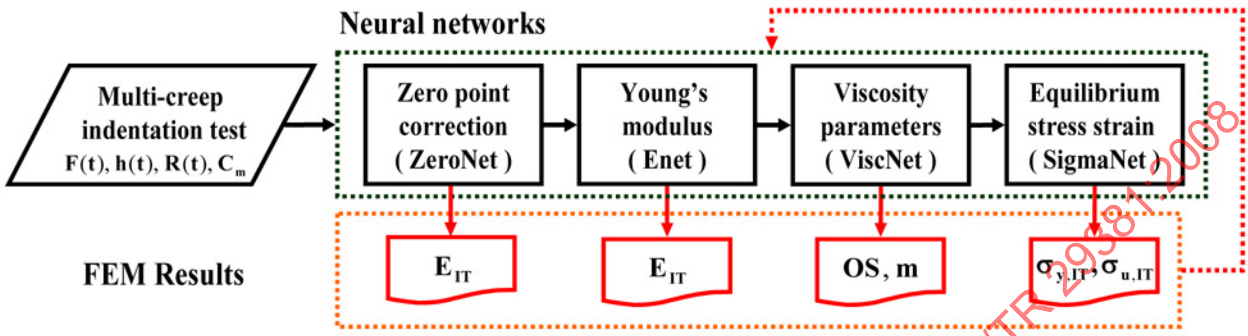


Figure 15 — Computational simulations

5.3.6 Procedure to obtain force-depth curve

The user is referred to specifications in Clause 7 of ISO 14577-1:2002.

The following steps are carried out to obtain the force-depth curve:

a) execution of multi-creep indentation test (duration at F_0 of 600 s and at 0,25 F_0, 0,5 F_0, 0,75 F_0 of 100 s);

b) determination of the maximum force, F_0 (corresponding to h_0 = 8 % to 12 % of indenter radius R);

c) determination of the time, t_0 (force increase without periods of creep, from 10 s to 60 s);

d) determination of the force-removal rate, equal to the force-application rate F_0/t_0;

e) zero point of the indentation depth determined from the data points after contact;

f) determination of deviation of the zero point (which should be $< 5 \times 10^{-3}\ R$).

An example of a force-depth curve is given in Figure 16.

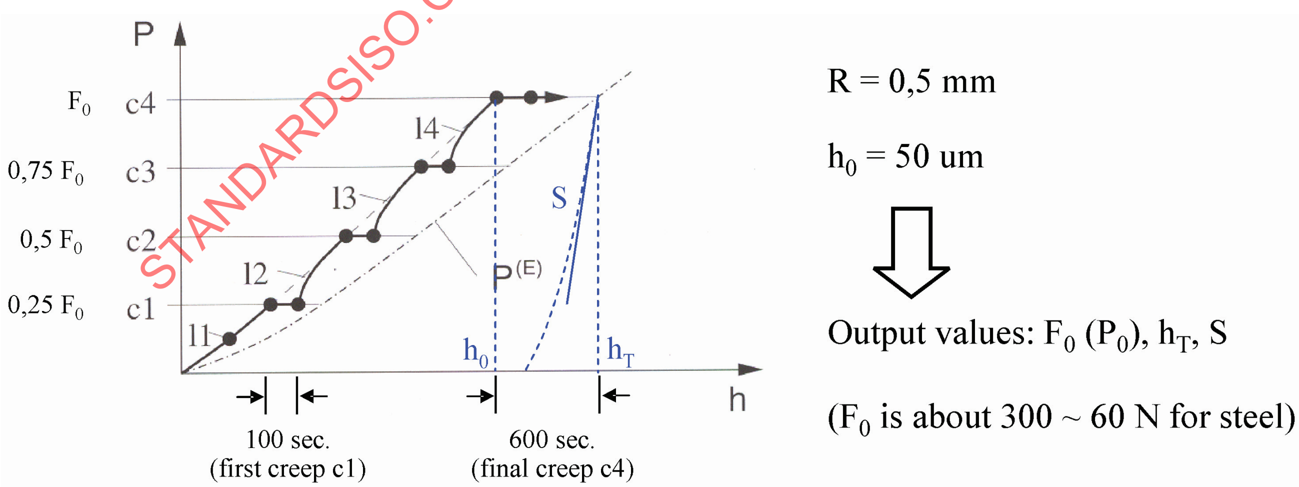


Figure 16 — Example of force-depth curve

5.3.7 Data analysis by trained neural networks

The system of the trained neural networks is a black box. The real structure of the system is given in Figure 17.

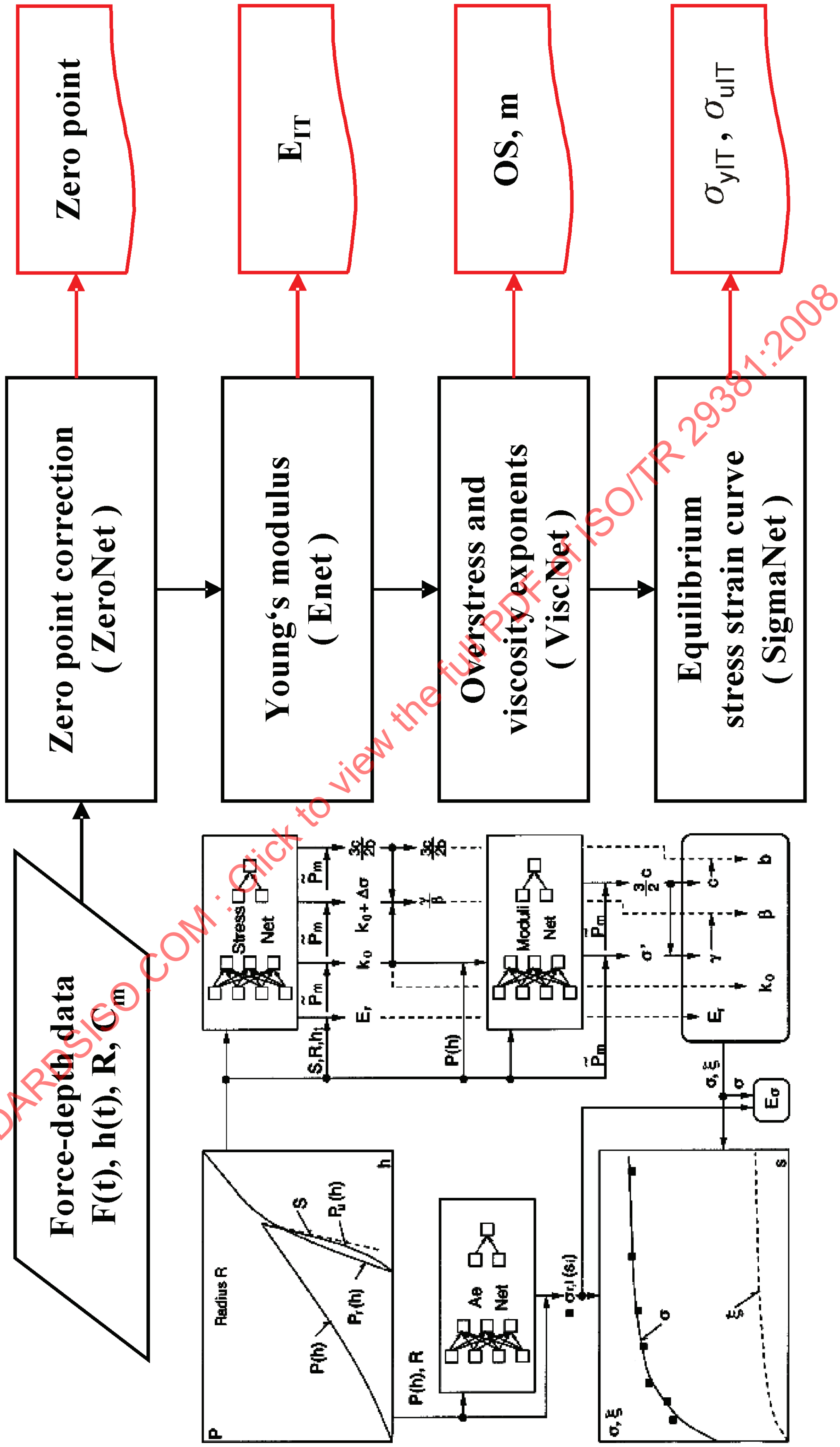


Figure 17 — Flow chart for system of trained neural networks

5.3.8 Experimental evidence

See Figure 18. For additional publications and patents concerning Method 3, see references [16] to [18].

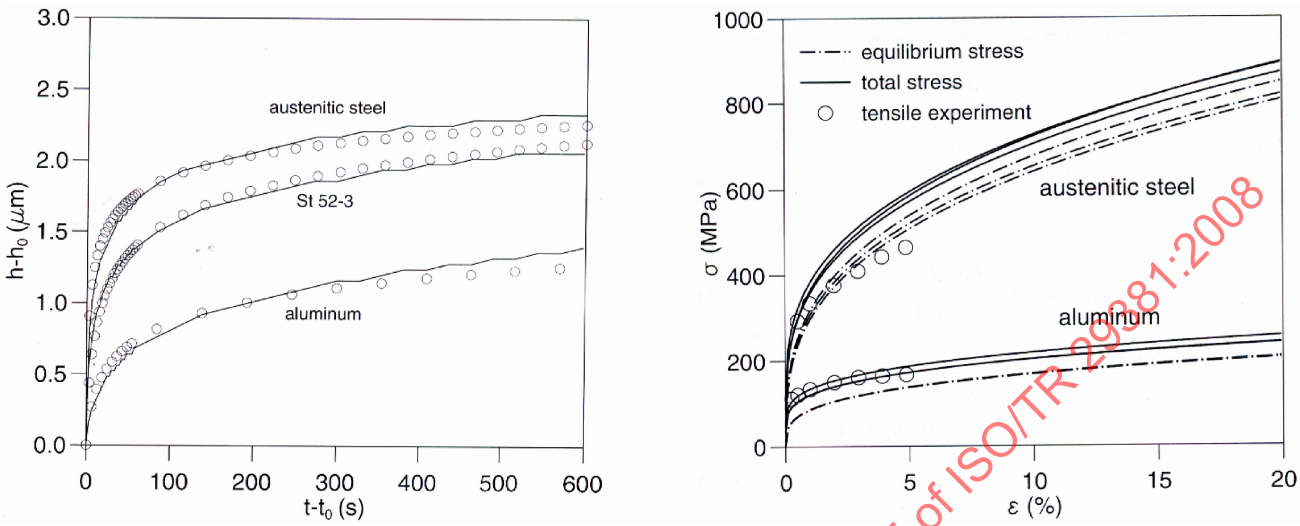


Figure 18 — Experimental evidence

5.3.9 Relative advantages and disadvantages

a) Advantages:

— consideration of time dependency (viscoplastic properties);

— uniqueness of solution for many types of materials;

— stress-strain curve obtained is robust against experimental error.

b) Disadvantages:

— need for special software (procedure for the trained neural networks);

— time-consuming test cycle;

— results for viscosity parameters are not as stable as the stress-strain curve results.

