(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
13 December 2012 (13.12.2012)

WIPO | PCT

(10) International Publication Number
WO 2012/168404 A1

(51) International Patent Classification:
G01N 3/42 (2006.01)

(21) International Application Number:
PCT/EP2012/060856

(22) International Filing Date:
8 June 2012 (08.06.2012)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
MI2011A001049 10 June 2011 (10.06.2011) IT

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(81) Designated States *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

— *with international search report (Art. 21(3))*

WO 2012/168404 A1

(54) Title: INTEGRATED EXPERIMENTAL AND COMPUTATIONAL SYSTEM FOR THE NON- DESTRUCTIVE DETERMINATION OF THE MECHANICAL PROPERTIES OF MATERIALS IN SITU IN THE OIL, GAS AND PETROCHEMICAL INDUSTRY

(57) Abstract: This application discloses an experimental and computational system, with the relative method, for the non - destructive determination of parameters which quantify the mechanical, elastic and/or inelastic properties of structural materials or of tensional states in structural and plant components for structural diagnosis "in situ" preferably in industrial oil, gas and petrochemical fields, comprising:- an experimental subsystem containing: - a portable instrument suitable for indenting with a tip, the surface of said components for the production in situ of an imprint, on the surface of the structural or plant component; - a portable roughness meter suitable for revealing both the roughness of the surface and the geometry of the imprint. - a computational subsystem comprising a software suitable for generating functions which better represent the combination of points revealed with the roughness meter, a software for the simulation of mechanical phenomena involved in the production of said imprint, a software for the application of inverse analysis methods to the experimental data, and an auxiliary software with mathematical instruments suitable for accelerating the inverse analysis operations.

INTEGRATED EXPERIMENTAL AND COMPUTATIONAL SYSTEM FOR THE NON-DESTRUCTIVE DETERMINATION OF THE MECHANICAL PROPERTIES OF MATERIALS IN SITU IN THE OIL, GAS AND PETROCHEMICAL INDUSTRY

Description

The present invention relates to an integrated experimental and computational system for the non-destructive determination of parameters which quantify the mechanical, elastic and/or inelastic properties of structural materials or of tensional states in structural and plant components preferably in the industrial sector of the oil and gas and petrochemicals, for structural diagnosis "in situ" based on non-destructive tests and inverse analysis methods.

The non-destructive tests are effected with a portable instrument suitable for indenting for the production "in situ" of an imprint, i.e. a localized, non-destructive elasto-plastic deformation on the surface of said components.

The imprint, meaning the coordinates of a combination of points situated on the deformed surface of said imprint, after removal of the tip, and on the adjacent surface, non-deformed or having a negligible deformation, is revealed with an instrument suitable for measuring said coordinates.

The characterization of the materials by the identification of parameters in elasto-plastic

constitutive models is useful for the oil, gas and petrochemical industries, where there are pipelines and liquid and gas transportation lines, and also weldings between two tracts of a pipeline, "T"-joints, connections of the pipelines to valves, reactors or other components of an industrial plant.

A complete characterization system is developed "in situ" of the mechanical properties of metallic alloys and other materials for the structural diagnosis of pipelines and other components of industrial plants and transportation lines in operation, where a degradation of said properties may have been verified due to aging or the accidental running of lines or plants outside the safety regime, or when the mechanical properties of constituent materials must be verified at the end of the useful life envisaged by the design of the component, or simply due to lack of the original technical documentation.

The system is also applied for determining the local tensional state; in particular residual tensions and/or by external actions that may have been generated in the weldings present in the lines or pipelines or in other components of industrial plants.

There are various non-destructive characterization systems for these constituent materials of pipelines and/or weldings and/or other components of industrial plants.

In the non-destructive characterization system called ABI ("Automated Ball Indentation") or SSM

(Stress-Strain Microprobe) Systems (US-4852397), the load-depth curve of an indenter is used without exploiting the geometry of the imprint. This ABI method also carries out tests with a tip of the spherical indenter.

As it does not use Inverse Analysis methods, the ABI method must introduce empirical constants and experimental data into the equations, previously measured in the laboratory, obtained on alloys of the family of the alloy to be characterized "in situ"; furthermore, it does not provide any information on the possible anisotropy of the material, neither can it provide estimations of the tensor of residual tensions.

A second non-destructive characterization method uses the sole load-depth curve obtained by means of an indenter and the Inverse Analysis Method for determining the mechanical properties, without exploiting the geometry of the imprint. This method is frequently applied to the study of the mechanical response of thin films or coatings (See for example: T. Nakamura, Y. Gu, Identification of elastic-plastic anisotropic parameters using instrumented indentation and inverse analysis. Mechanics of Materials, vol. 39, pp. 340-356, 2007; A. E. Giannakopoulos, Determination of elastoplastic properties by instrumented sharp indentation. Scripta Materialia, vol. 40, pages 1191-1198, 1999).

An alternative method classified as "virtually non-destructive" is that called "Small Punch" ("Code of

practice on Small Punch technique for Metallic Materials", CEN document issued by CEN WS21, coordinated by V. Bicego, CESI, 2006). It requires the extraction of a thin test-sample from the outer surface of the piping, the preparation of flat samples starting from the plate and a punching test in the laboratory. The method uses only the load-shift curve of the punch and requires, for each mechanical property, the set-up of a "master curve" with measurements preliminarily effected on alloys of the family of the alloy to be characterized. It does not use computational simulations of the test nor inverse analyses. It cannot be effected in situ.

An integrated experimental and computational system has now been found together with the relative method for the mechanical characterization of materials and for determining tensional states for structural diagnoses "in situ". The system and method are based on non-destructive tests and on inverse analysis methods, which are capable of identifying from the geometry of the imprint alone, mechanical properties or tensions or also with simultaneous use, in simulations and calculations, of the geometry of the indentation and experimental data generated by portable instruments associated with the indenter (called "instrumented indenter"), i.e. the digitalized load-depth penetration curve of the indenter.

The complete system of the present invention does not require empirical constants, measurements prior to

the indentation test or data banks on the mechanical properties.

The system of the present invention can provide the desired information in "real time".

The system of the present invention can handle any geometry of the imprint or cavity produced by the tip of indenters or durometers, or any instrument capable of producing it.

In order to determine residual tensions in weldings, the system can also be applied immediately after the operation in the absence of degradation phenomena.

The system operates "in situ" on tubings, lines and functioning components and can also operate subsequently in the laboratory on representative samples taken on site.

The mechanical properties determined relate to the elastic, inelastic field and especially the plastic behaviour of the materials to be characterized. Among the parameters that can be identified with the system claimed in the present invention, the following can be mentioned: elastic moduli, ultimate tensile strength, yield point, strain hardening curve.

The experimental and computational system, object of the present invention, for the non-destructive determination of parameters which quantify the mechanical properties, elastic and/or inelastic, of structural materials or local tensional states, comprises:

- an experimental subsystem containing:
  - a portable instrument suitable for indenting with a tip having any geometry, preferably conical, spherical, pyramidal or cylindrical, more preferably conical, the surface of said components for the production "in situ" of an imprint, i.e. a localized, non-destructive elasto-plastic deformation, on the surface of the structural or plant component;
  - a portable roughness meter suitable for revealing both the roughness of the surface and the geometry of the imprint meaning the combination of coordinates of points situated on the deformed surface of said imprint, after removal of the tip and on the adjacent surface, non-deformed or having a negligible deformation;
- a computational subsystem comprising a software suitable for generating functions (surfaces and/or profiles) which better represent the combination of points revealed with the roughness meter, a software for the simulation of mechanical phenomena involved in the production of said imprint, a software for the application of the inverse analysis method to the experimental data, and an auxiliary software with mathematical instruments suitable for accelerating the inverse analysis operations, to enable the use of the software for effecting all the calculations "in situ" with a portable computer and get the desired results in "real time ".

The instrument suitable for the production "in situ" of an elasto-plastic deformation, non-destructive due to the small dimensions, on the surface of structural or plant components can preferably be selected from an instrumented portable indenter (i.e. capable of providing the penetration shift in relation to the force applied), a non-instrumented portable indenter, a non-instrumented portable durometer, a portable durometer with a spring for the generation of an imprint by means of dynamic impact.

The preferred tip of said portable instrument suitable for indenting is conical.

The instrument uses a manual or automatic movement plane X-Y for revealing the complete geometry of the imprint "in situ", generating a digitalized map consisting of a matrix of points or "nodes" in the plane X-Y and quotas in the axis Z.

The computational subsystem preferably consists of a software for the simulation of phenomena involved in indentation test and a software with various original modules for the application of suitable inverse analysis methods in "real time ".

One of the instruments for the production of the imprint and the instrument for revealing the geometry of the imprint can be possibly assembled in a single complete portable instrument to allow, by means of a manual or automatic movement, two consecutive operations, ensuring the positioning of the two instruments in the same axis perpendicular to the

surface of the structural or plant component (e.g. tubing, pipeline, etc.) to be characterized.

The functions of the instrument for the production of the imprint and the functions of the instrument for revealing the geometry of the imprint can be effected by a single multifunctional instrument.

The instruments for the almost-static production of the imprint and for revealing the geometry of the imprint, or the two instruments assembled in a single complete instrument, or the single instrument, can be fixed to the tubing or pipeline in any position along its circumference by means of magnets, metallic or polymeric bands or other fixing means to ensure the rigidity necessary for the test.

The instrument used in the experimental subsystem for revealing the geometry of the imprint (roughness meter) produces some improvements with respect to instruments cited in specific literature in the field of non-destructive tests.

As the results of the present invention greatly depend on the whole geometry of the imprint (when the indentation curve and geometry of the imprint are determined for using them as "input" data for the modeling) or strictly depend on the geometry of the imprint (when the geometry alone is used as "input" data for the modeling), an instrument having the following two characteristics has been selected as reference instrument:

I. The instrument must be of the "contact" type. In

this instrument there is not mathematical algorithms for the conversion of the electric signal into a distance or depth signal z but a proportionality constant between the two variables or possibly an initial calibration curve. The most reliable data can thus be provided for the modeling.

II: The instrument must determine only coordinates (x,y,z) of points situated on the surface of the imprint, and peripheral surfaces, and not profiles. This avoids the introduction of unknown algorithms, used by profilometers for providing a profile that can interpret the coordinates of a certain number of points.

In the case of the present invention with the use of a plane X,Y, the 3 coordinates (x,y,z) of a high number of points are determined "in situ" (in the order of nx10^4 or nx10^5 points). In this way, the original information which contains the description of the plasticity phenomenon is preserved. The mathematical instruments of the computational subsystem subsequently use different approaches for describing the surfaces or profiles which better describe the imprint and non-deformed peripheral surfaces. The number of profiles and selection of their position is extremely important in cases of the presence of anisotropy and/or residual tensions. It is convenient for these selections to be effected by the computational subsystem and not by the instrument during the measurement of the geometry of the imprint.

A secondary aspect for metallurgical studies but important for the reproducibility of the experiments is provided by the possibility of also determining the roughness of the walls of the imprint. In this way, the state of the tip of the indenter is controlled. As a "contact" instrument, the roughness meter proposed is the most suitable instrument for controlling the local roughness parameter.

A contact roughness meter such as that used in Example 1 of the present invention (Model SJ-400 Surface Roughness Tester produced by Mitutoyo, No. 99MBB093A5, Series No. 178) with "outputs" coordinate z and roughness Ra, Ry, Rz, Rq or Rdelta-c, satisfies the two points mentioned above and provides local roughness values.

Among other particular features of the roughness meter, its weight and dimensions are less or much lower than those of other instruments such as, for example, optical instruments, two decisive factors in some cases of components "in situ" with extremely reduced spaces available for the tests. Studies for controlling viscosity phenomena present in an indentation test can also be effected by means of roughness measurements.

Other improvements ascertained by the experimental comparison between the roughness meter and other instruments cited in literature for revealing the geometry of the imprint are listed below. In literature, it is stated in some cases that these instruments have actually been used for effecting

measurements, in other cases their use is only assumed.

The publication "Mechanical Characterization of Materials by Micro-Indentation and AFM Scanning" (G. Bolzon, M. Bocciarelli, E. J. Chiarullo, appearing in the collection Applied Scanning Probe Methods XII, B. Bhushan and H. Fuchs (Eds), Springer, 2009), considers the use of the Atomic Force Microscope (AFM) on a microscale and hypothesizes the use of the Scanning Force Microscope on a larger scale. The roughness meter proposed in the examples of the present invention enables the documentation of the geometry of the cavity having a depth of 10-800µm (typical depth values of the imprint range for example from 100 to 350 µm, an order of magnitude higher than the average dimension of the crystalline beads), whereas the above-mentioned microscopy is only capable of exploring a much narrower range of depth values which are not able to involve volumes representative of the mechanical properties of the material.

Laboratory contact profilometers and laser profilometers are mentioned in the publication "Proper Orthogonal Decomposition and Radial Basis Functions in Material Characterization Based on Instrumented Indentation" (V. Buljak, G. Maier, Engineering Structures, Vol. 33, pages 492-501, 2011). The weight and encumbrance of laboratory contact profilometers do not allow their use "in situ". As far as laser profilometers are concerned, this instrument does not allow a correct mapping of surfaces with a high slope.

Roughness meters do not have these disadvantages.

In the already cited publication "Assessment of Elastic-Plastic Material Parameters Comparatively by Three Procedures Based on Indentation Test and Inverse Analysis" (G. Bolzon, V. Buljak, G. Maier, B. Miller, Inverse Problems in Science & Engineering, Vol. 19, pages 815-837, 2011), the advantages of profilometry are generically listed. Instruments suitable for revealing the imprint based on optical microscopy inevitably generate abnormal signal peaks ("false"), which are not shown or are revealed only slightly by the roughness meter.

An alternative to the roughness meter to be used in the experimental subsystem can consist of an optical portable instrument for the topographical detection of the surface based on the focal variational principle as a method suitable for producing, by means of a high number of "pixels", a complete three-dimensional representation of the surfaces obtained by measuring the depth corresponding to the clearest image of each pixel. The survey of the geometry of the imprint can be effected in a single acquisition in the case of a visible field which is such as to represent the whole geometry of the imprint or by the superimposition of various acquisitions in the case of a visible field which is such as to represent partial areas of the geometry of the imprint itself.

The apparatus used in the case of Example 2 ("Portable Alicona Infinite Focus", Graz,

Austria) is capable of effecting the survey of the geometry of the imprint as topography, i.e. by determining only coordinates (x, y, z) of a very high number of points (in the order of nx10^5 or nx10^6 points).

The modeling of the computational subsystem subsequently uses the points for describing the type and convenient number of surfaces or profiles that can best describe the plasticity phenomenon.

Apart from the roughness meter which has never been used or proposed before for revealing the geometry of the imprint, the present invention proposes and uses a durometer in one of the examples, (Example 1, "Hardness Tester", Wolpert, for Durezza Rockwell HRC with a maximum load of 1500 N). So far, a durometer was an instrument which was not capable of determining mechanical properties of materials. Hardness values, in fact, do not represent an "input" datum for structural calculation codes. Through the determination of the geometry of the imprint and inverse analysis it has been possible to use the maximum load value only of the durometer without the necessity of availing of the whole indentation curve.

The method for the non-destructive determination of mechanical, elastic and/or inelastic properties of materials and/or local tensional states in structural and plant components, comprises the computational simulation of phenomena involved in indentation tests using suitable software, comparison of the results

between simulation and experiment to obtain the set of desired parameters by means of inverse analysis methods. This method uses experimental data relating to the geometry of an imprint obtained after said indentation tests.

The method comprises the following steps:

- production "in situ" on the surface of the material to be characterized, of an imprint or non-destructive elasto-plastic deformation by means of a suitable instrument, determining the maximum load or indentation curve (load-depth);
- detection "in situ" of the complete geometry of the imprint by means of a roughness meter for generating a map and defining it in digitalized form by means of the coordinates and quotas of points situated on the surface of the imprint and on the adjacent non-deformed surface;
- comparison of the results between simulation and experiment in order to obtain the set of desired parameters by means of inverse analysis methods.

Prior to the characterizations of said method, a preliminary step can be effected, consisting of suitable tribological treatment ("cleaning" or "lapping") of the surface to be indented.

The method claimed herein envisage that one can get the elastic-plastic properties of the material under test from the information on the geometry of the imprint and from the load-penetration depth curve returned by the instruments (contact surface roughness

tester, microscope for topographic measurements, instrumented indenter, not instrumented indenter, durometer) and from the software developed to support the integrated experimental and computational system.

The indenter can be static or dynamic (such as an impact durometer).

The method claimed herein also preferably envisages that the software developed to support the method of inverse analysis assess quantitatively the axisymmetric loss of the imprint, produced by an indenter with an axisymmetric tip, said loss generated by residual tensions and/or by the possible anisotropy of the material to obtain the value of the local tensions (residual and/or overall) and/or differences in the directional mechanical properties caused by the anisotropy. Through an analysis of the geometry of the imprint, the method claimed therefore allows the determination of the residual tensions in weldings of industrial plant components or tubings or in weldings in transportation lines of liquids and gas, and/or the determination of how the mechanical properties of the material vary in relation to the direction in the case of anisotropy.

The inverse analysis operations to be effected to obtain the set of desired parameters starting from the above experimental information are described for example in: G. Bolzon, G. Maier, M. Panico, International Journal of Solids and Structures, Vol. 41, pages 2957-2975, 2004; M. Bocciarelli, G. Bolzon,

G. Maier, Mechanics of Materials, Vol. 37, pages 855-868, 2005; M. Bocciarelli, G. Maier, Computational Materials Science, Vol. 39, pages 381-392, 2007.

The articles in scientific reviews indicated above are the result of preliminary theoretical studies aimed at developing the inverse analysis method described above, using "pseudo-experimental" data generated for optimizing computational operations.

Inverse analysis based on experimental data is preferably effected with an auxiliary software with original modules, which use mathematical instruments such as Proper Orthogonal Decomposition (POD) and Radial Basis Functions (RBF) or others, associated with artificial neural networks or genetic algorithms or classical optimization methods, in order to accelerate computational operations to allow all the calculations to be effected "in situ" with a portable computer, as illustrated for example in G. Bolzon, V. Buljak, G. Maier, B. Miller, Inverse Problems in Science & Engineering, Vol. 19, pages 815-837, 2011.

The same acceleration allows stochastic rather than deterministic approaches if the engineering problem requires this: Montecarlo, Bayes and Kalman filter methods suitable for providing estimates of parameters and their uncertainties (i.e. covariance matrix) require even ampler simulation sequences of the inverse analysis processes indicated above.

In the method proposed, these numerical laboratory developments are required "*una tantum*", as part of the

preparation of the experimental equipment, and are accumulated in a calculator with modest capacities, such as a portable device. This information is subsequently exploited by resorting to information reduction methods, for example based on "Proper Orthogonal Decomposition" (POD), and interpolation techniques, preferably by means of suitably selected "Radial Basis Functions" (RBF). The combination of POD plus RBF allows the response to a hypothetical test carried out on each material with characteristics within the range examined in the preparatory phase, to be obtained in real time.

The "modus operandi" described above, operating on pre-calculated "responses", is compared with traditional simulation for finite elements. Various comparative numerical validation operations show, for example, that typical calculation time ratios reach various orders of magnitude in favour of the new method, with discrepancies not greater than thousandths in terms of resulting tensions and shifts. These results are crucial for present practical purposes.

A detailed description of the mathematical instruments and operations used by the computational subsystem comprise:

- The value of the parameters of the material is determined by means of an inverse analysis procedure, which compares the data acquired by the instruments with the results of a numerical model of the

indentation or hardness test. The effective value of the parameters is defined through an optimization procedure, which minimizes the discrepancy between the experimental information and simulated response of the material.

- The simulation of the indentation test is normally effected by means of the finite element method, a technique which is effective but too costly for being able to be effected "in situ". The supporting software of the computational system therefore implements a reduced model, which analytically interpolates the results of a predefined number of numerical analyses, carried out in a preparatory phase in a numerical analysis laboratory. The model is made more efficient by the implementation of decomposition techniques which filter numerical disturbances not compatible with the dispersion of the experimental data.

The integrated experimental computational system thus obtained allows the value of the parameters to be defined and to compare, in real time, "in situ", the experimental result with that simulated by the suitably calibrated model. In this way, it is possible to immediately decide, "in situ", whether it is necessary or preferable to increase the number of tests in order to take the dispersion of the measurements into account and/or adjust the instrumental layout to take systematic errors into consideration.

The description of the procedures implemented in the computational subsystem shows that it is not at all

banal to reliably obtain the desired result in real time and that the use "in situ" of the experimental computational system does not consist of a simple operation to be effected only using portable instruments.

The experimental computational system therefore has the following combination of characteristics which distinguish it from any other apparently analogous system:

- The computational subsystem defines the value of the desired parameters in real time (in a few seconds), an aspect which allows measurements "in situ";
- The computational subsystem does not use any information a priori, not always available, on the characteristics of the material under examination;
- The experimental computational system allows to perform an immediate comparison between the experimental result and that of the computational sub-system with the calibrated model with the optimum value of the parameters, in order to evaluate the reliability of the result.

From the determination of the indentation curve and geometry of the imprint obtained in an area with residual tensions in a structural or plant component, it is possible to obtain the values of the components of the tensor of residual tensions preferably through an inverse analysis procedure, by combining the result of an instrumented indentation test with the simulation of the same, optimized through the definition of a

reduced analytical model. The entity of the residual tensions is deduced from a comparison with the results of a test carried out on a sample of reference material or in an area of the component, virtually free of residual tensions, which returns a modified indentation curve and a geometry of the imprint, through the minimization of the difference observed between measured quantities and simulated quantities.

Some examples are now provided in accordance with the invention, which should not be considered as limiting the scope of the invention itself.

Example 1

Determination of the mechanical properties of a metal or metal alloy on the basis of the geometry alone of the residual imprint.

The experimental-computational system was adopted for identifying characteristic parameters of the behaviour of a metal (electrolytic copper, in samples obtained starting from an extruded copper billet) on the basis of experimental information relating to the geometry alone of the residual imprint generated on the sample of a Rockwell HRC hardness test, with a maximum load of 1500 N.

The imprints were revealed with a portable Mitutoyo SJ-400 roughness meter with a spherical feeler having a radius of 0.002 mm and with a non-portable Zeiss-TSK Surfcom 1800D laboratory profilometer having a tip with a radius of 0.025 mm used as reference for controlling the functions

obtained by means of software starting from points revealed with the roughness meter.

The information collected during the experiment was represented in the experimental imprint (Depth [μm] in relation to the Radius [μm] shown in figure 1: this is the average of 8 measurements, calculated starting from the map of points with coordinates (x,y,z) determined with a plane (X,Y) and the roughness meter, along radiuses at 45° from each other starting from the indentation axis.

These data were used for identifying the parameters that characterize the mechanical behaviour of the material tested. Once the elastic modulus had been fixed at the value of E=80 GPa, the application of the inverse analysis methods returns the following parameters characteristic of the material:

- yield point: 273 MPa
- strain hardening index: 0.011.

The uniaxial behaviour curve "identified" corresponds to the above parameters (Tension[MPa] in relation to the Deformation [ε]) shown in figure 2, compared with the "experimental" curve resulting from the standard traction test for the same material and effected as reference.

In the comparison, it should be noted that the results of the traction tests are given in terms of tensions ad nominal deformations, whereas the result obtained from the inverse analysis procedures described in the previous sections is based on modeling of the

experiment in terms of "real" tensions and deformations, i.e. for "large" deformations. The distinction becomes practically relevant in mono-axial tests only in the ultimate localized striction phase, which is not reached in the test of Figure 1.

Example 2

Determination of the mechanical properties of a steel pipeline for transporting gas starting from the geometry of the imprint produced by indentation.

The experimental-computational system situated "in situ" on the piping, was adopted for identifying the parameters characteristic of the behaviour of a steel for piping starting from experimental information relating to the geometry alone of the imprint, visible in Figure 3, generated directly on the component by means of an instrumented indentation test, carried out with a maximum load of 2000 N. For the indentation, the system uses a portable AFFRI SR-HU09-P indenter. The complete three-dimensional representation of the surface, through the coordinates of a high number of pixels was obtained using a portable optical topographic detection instrument of the surface (Portable Alicona Infinite Focus).

The application of inverse analysis methods starting from data relating to the geometry alone of the imprint provides, once the elastic modulus has been fixed at a characteristic value of 205 GPa, the following nominal parameters of the material:

- yield point: 394 MPa

- maximum ultimate stress: 597 MPa
- strain hardening index: 21

The uniaxial behaviour curve corresponds to the above parameters (Stress [MPa] in relation to the Deformation [ε]) shown in Figure 4, compared with that resulting from the standard traction test for the same material.

In Figure 5 the geometry of the experimental imprint (Depth [μm] in relation to the Radius [μm]) from the complete three-dimensional representation of the surface is compared with that recalculated with the parameters identified.

Finally, Figure 6 shows a comparison between the recalculated indentation curve and that obtained experimentally (Displacement [μm], Force [N]). These experimental data were used only as control *a posteriori* and were not used for identifying the parameters.

The comparison between the various experimental curves and the curves calculated with the experimental-computational system, object of the present patent (Figures 4, 5 and 6) show a good concordance. In particular, the following characteristic parameters are determined from the standard mono-axial experimental traction curves used as reference:

- yield point: 401 MPa
- maximum stress: 594 MPa

The values calculated coincide with these reference

values within 1.7% for the yield point and within 0.5% for the maximum stress (i.e. for the ultimate tensile strength).

Example 3

Determination of the mechanical properties of a steel pipeline for transporting gas using information relating to both the load-depth penetration curve and the geometry of the imprint produced by indentation.

The experimental-computational system situated "in situ" on the piping, was adopted as in Example 2 for identifying the parameters characteristic of the behaviour of a steel.

The application of the inverse analysis methods provides, once the elastic modulus has been fixed at a characteristic value of 205 GPa, the following nominal parameters of the material:

- yield point: 395 MPa
- maximum ultimate stress: 595 MPa

The values calculated coincide with the reference values within 1.3% for the yield point and within 0.13% for the maximum stress (i.e. for the ultimate tensile strength).

Example 4

Determination of the residual tensions in a sample with calibrated residual tensions.

The sample consists of an aluminum cylinder having a thickness of 10 mm comprising a steel crown having a rectangular section and an internal diameter of 25 mm; the diameter of the aluminium cylinder is 25 mm + Δ mm.

For producing the sample, the aluminium insert was cooled in liquid nitrogen, the steel crown was heated to 300°C and the two parts were inserted into each other and brought to room temperature. The calibrated samples can have different Δ values (positive or negative; according to a certain diameter, Δ0°, and according to a perpendicular diameter, Δ90°).

In the present example, the aluminium cylinder (Al), was used, without residual tensions, and a sample (sample 1) with Δ0°= 0.1 mm and Δ90°= 0 mm.

The result of the application of the inverse method (indentation curve and geometry of the imprint identified, Id) taking into account the experimental or "nominal" information (only the geometry of the imprint or indentation curve and geometry of the imprint, Exp) is indicated in Figures 7 and 8. Figures 7 and 8 show the values of the residual tensions calculated (components Sigma x and Sigma y) in each situation.

CLAIMS

1. An experimental and computational system for the non-destructive determination of parameters which quantify the mechanical, elastic and/or inelastic properties of structural materials or of tensional states in structural and plant components for structural diagnosis "in situ" preferably in industrial oil, gas and petrochemical fields, comprising:

• an experimental subsystem containing:

- a portable instrument suitable for indenting with a tip, preferably conical, spherical, pyramidal or cylindrical, the surface of said components for the production "in situ" of an imprint, i.e. a localized, non-destructive elasto-plastic deformation, on the surface of the structural or plant component;

- a portable roughness meter suitable for revealing both the roughness of the surface and the geometry of the imprint meaning the combination of coordinates of points situated on the deformed surface of said imprint, after removal of the tip and on the adjacent surface, non-deformed or having a negligible deformation;

• a computational subsystem comprising a software suitable for generating functions (surfaces and/or profiles) which better represent the combination of points revealed with the roughness meter, a software for the simulation of mechanical phenomena involved in the production of said imprint, a software for the

application of the inverse analysis method to the experimental data, and an auxiliary software with mathematical instruments suitable for accelerating the inverse analysis operations, to enable the use of the software for effecting all the calculations "in situ" with a portable computer.

2. The system according to claim 1, wherein the instrument for the production "in situ" of an imprint or deformation on the surface of the structural or plant component is selected from a non-instrumented portable indenter, an instrumented indenter capable of providing the load-depth penetration curve, a non-instrumented portable durometer, a portable durometer with a spring for the generation of an imprint by means of dynamic impact.

3. The system according to claims 1 and 2, wherein the instrument uses conical tips.

4. The system according to claim 1, wherein one of the instruments for producing the imprint and the roughness meter are assembled in a single complete portable instrument to allow two consecutive operations.

5. The system according to claim 1, wherein the functions of the instrument for producing the imprint and the functions of the roughness meter are effected by means of a single multifunctional instrument.

6. An experimental and computational method for the non-destructive determination of mechanical, elastic and/or inelastic properties of elasto-plastic materials, comprising the computational simulation of

phenomena involved in indentation tests using suitable software, comparison of the results between simulation and experiment to obtain the set of desired parameters by means of inverse analysis methods using the information obtained by the geometry of the imprint, which means the combination of coordinates of points situated on the deformed surface of said imprint, after removal of the tip and on the adjacent surface, non-deformed or having a negligible deformation, obtained in said indentation tests, characterized in that it comprises:

- the production, "in situ" on the surface of the material to be characterized, of a non-destructive imprint or elasto-plastic deformation by means of a suitable instrument, determining the maximum load or indentation curve (load-depth);
- the detection "in situ" of both the roughness of the surface and the geometry of the imprint using a roughness meter;
- a computational subsystem comprising a software suitable for generating functions (surfaces and/or profiles) which better represent the combination of points revealed with the roughness meter;
- a comparison of the results obtained from the simulation and experiment in order to obtain the set of desired parameters by means of inverse analysis methods.

7. The experimental and computational method according

to claim 6, wherein, from the geometry of the imprint or, from the geometry of the imprint and load-depth penetration curve, provided by an instrumented indenter, it is possible to obtain the elasto-plastic mechanical properties of the material subjected to the test.

8. The experimental and computational method according to claim 6, wherein the axisymmetric loss of the imprint produced by an indenter with an axisymmetric tip is quantitatively evaluated, said loss generated by residual tensions and/or by the possible anisotropy of the material to obtain the value of the local tensions (residual and/or overall) and/or differences in the directional mechanical properties caused by the anisotropy.

9. The experimental and computational method according to claim 6, wherein, from the determination of the indentation curve and geometry of the imprint obtained in an area with residual tensions in a structural or plant component, it is possible to obtain the values of the tensor components of residual tensions, the determination of the residual tensions being effected through an inverse analysis procedure, by combining the result of an instrumented indentation test with the simulation of the same, optimized through the definition of a reduced analytical model, the entity of the stress in the presence of residual tensions being deduced from a comparison with the result of a test effected on a sample of reference material or in an

area of the component, virtually free of residual tensions, which returns a modified indentation curve and a geometry of the imprint, through minimization of the difference observed between measured and simulated quantities.

10. The system according to one of claims 1 to 5, wherein the portable roughness meter suitable for revealing both the roughness of the surface and the geometry of the imprint, is substituted by a portable optical topographic detection instrument of the surface based on the focal variational principle as a method suitable for producing, through a high number of pixels, a complete three-dimensional representation of the surfaces obtained by measuring the depth corresponding to the clearest image of each pixel in a single acquisition in the case of a visible field which is such as to represent the whole geometry of the imprint or by the superimposition of various acquisitions in the case of a visible field which is such as to represent partial areas of the geometry of the imprint.

11. The method according to one of claims 6 to 9, wherein the portable roughness meter suitable for revealing both the roughness of the surface and the geometry of the imprint, is substituted by a portable topographic optical detection instrument of the surface based on the focal variational principle as a method suitable for producing, through a high number of pixels, a complete three-dimensional representation of

the surfaces obtained by measuring the depth corresponding to the clearest image of each pixel in a single acquisition in the case of a visible field which is such as to represent the whole geometry of the imprint or by the superimposition of various acquisitions in the case of a visible field which is such as to represent partial areas of the geometry of the imprint.

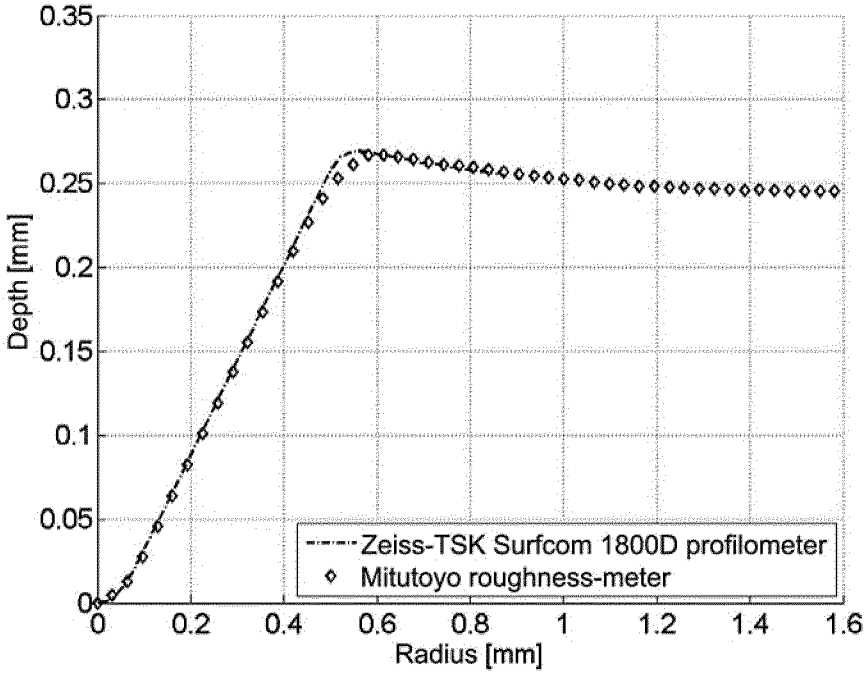


Figure 1: Calibration of the roughness meter - Comparison of the geometry of the imprint, average of 8 measurements, generated by the indenter on copper, obtained with a laboratory profilometer (Zeiss-TSK Surfcom 1800D as reference and a portable roughness-meter (Mitutoyo SJ-400)

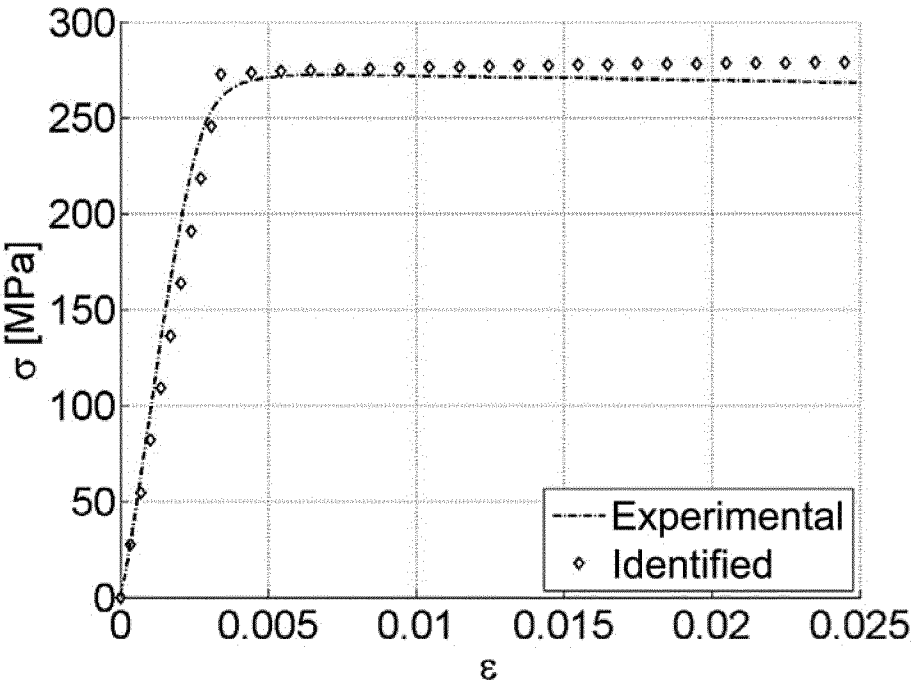


Figure 2: Constitutive bond identified (Curve Identified) and compared with the result of a mono-axial test (Experimental Curve)

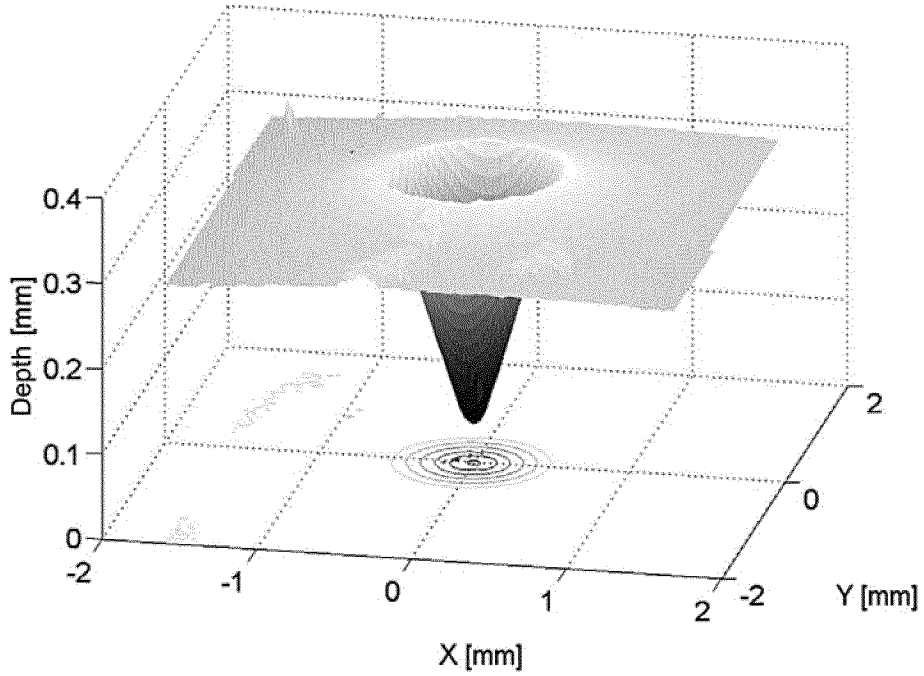


Figure 3: Three-dimensional reconstruction of the geometry of the imprint

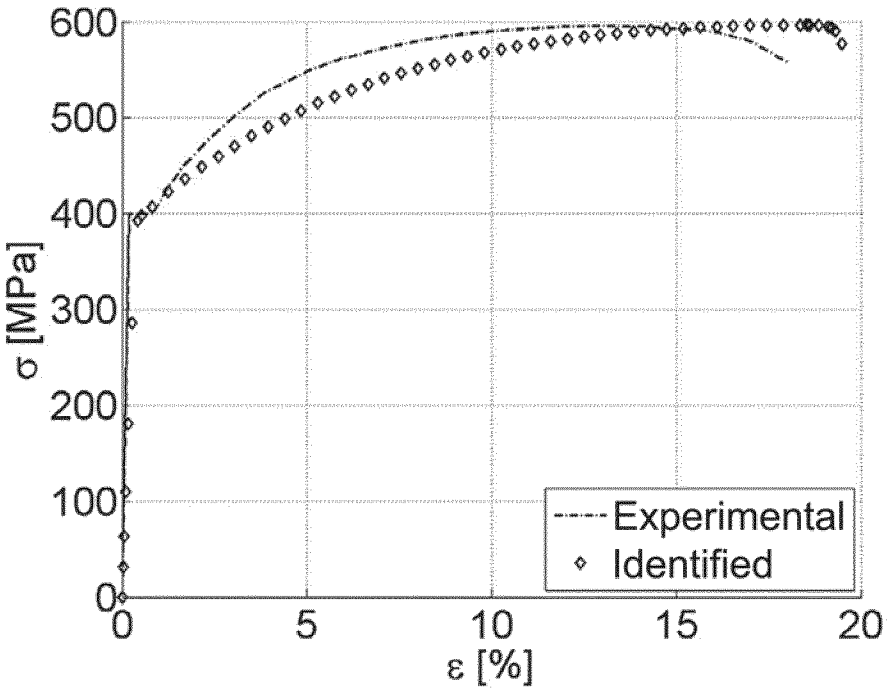


Figure 4: Constitutive bond identified (Curve Identified) for a pipeline steel and compared with the result of a mono-axial test (Experimental Curve)

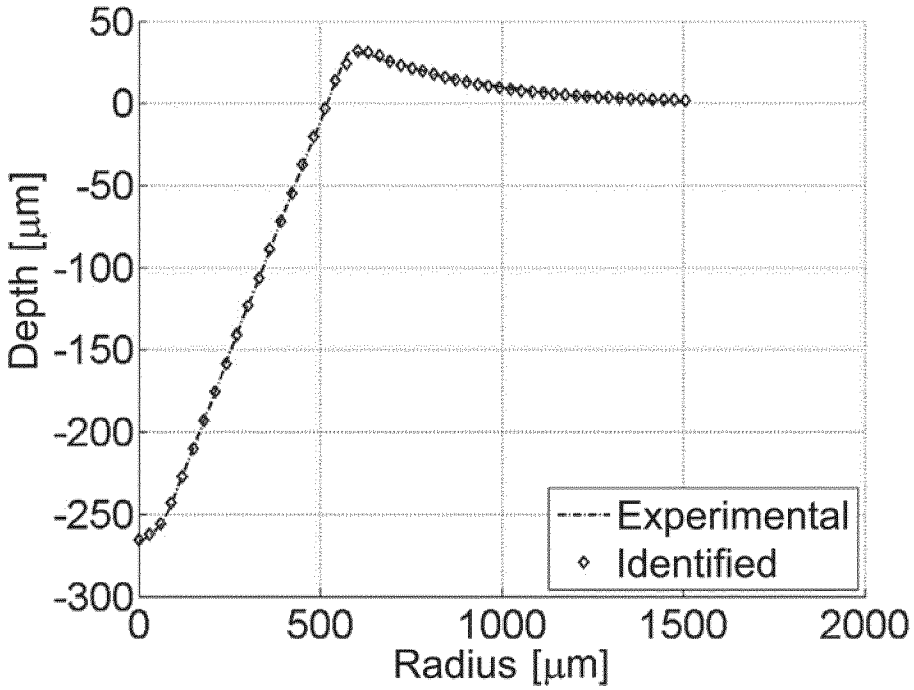


Figure 5: Geometry of the imprint and recalculated geometry with the parameters identified by the modelling.

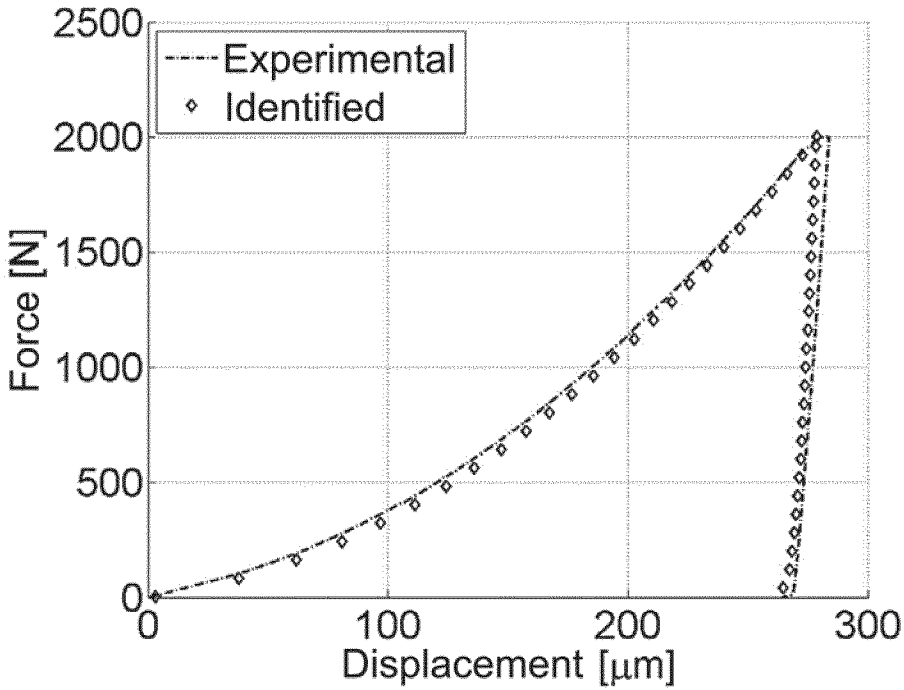


Figure 6: Experimental indentation curve (Experimental curve) and analogous curve recalculated with the parameters identified (Curve Identified) starting from the measurements relating to the imprint alone

Figure 7: Comparison between experimental data (experimental curve, Exp, shifted to the left) and results of the simulation (calculated curve, Id, shifted to the right) calibrated by using only the geometry of the imprint

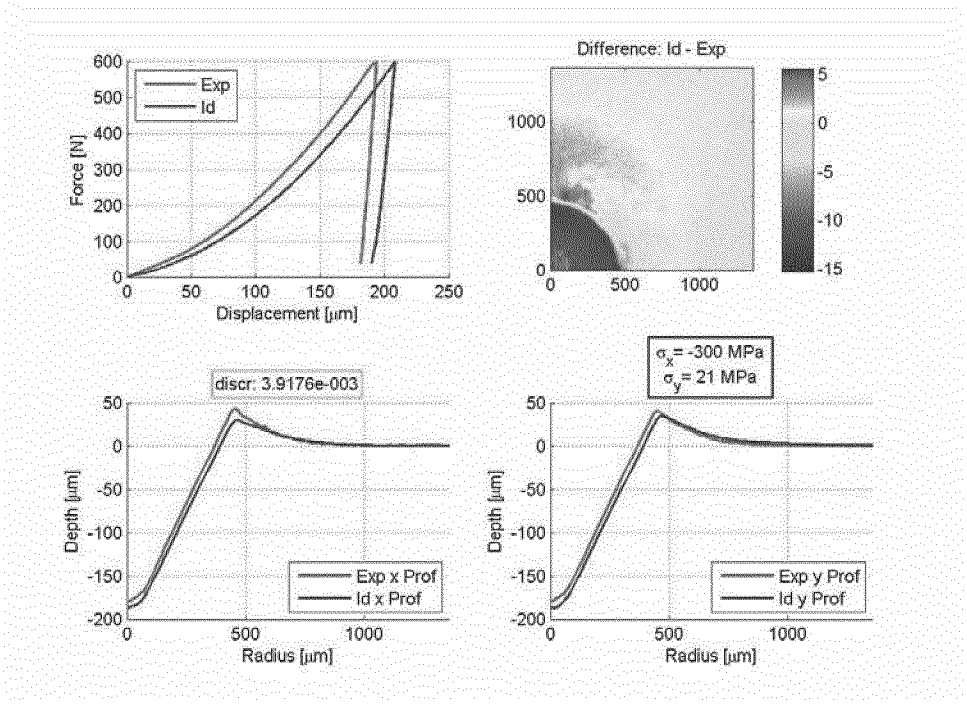
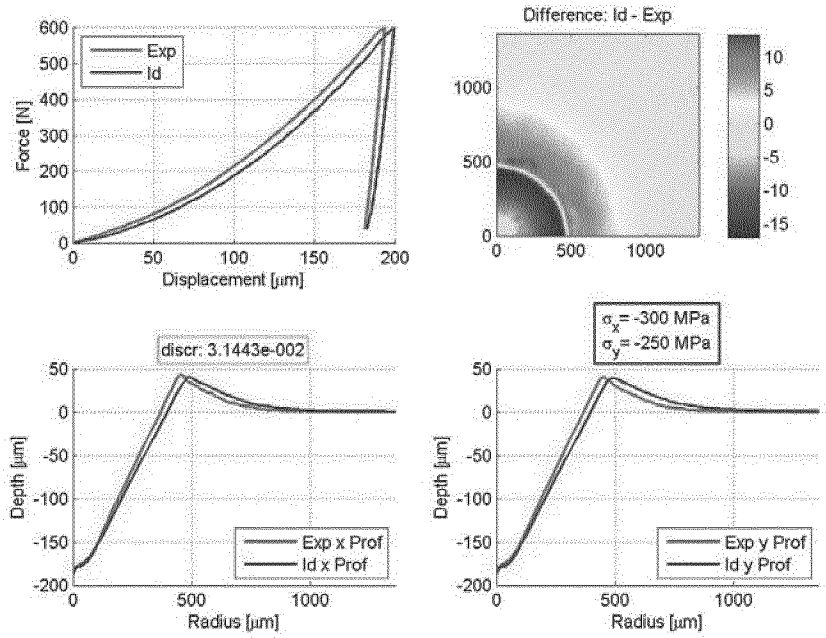


Figure 8: Comparison between experimental data (experimental curve, Exp, shifted to the left) and results of the simulation (calculated curve, Id, shifted to the right) calibrated by using the geometry of the imprint and the indentation curve.



INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2012/060856

A. CLASSIFICATION OF SUBJECT MATTER
INV. G01N3/42
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data, COMPENDEX

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	BULJAK V ET AL: "Proper Orthogonal Decomposition and Radial Basis Functions in material characterization based on instrumented indentation", ENGINEERING STRUCTURES, BUTTERWORTH, GB, vol. 33, no. 2, 1 February 2011 (2011-02-01), pages 492-501, XP027573028, ISSN: 0141-0296 [retrieved on 2010-12-23] cited in the application the whole document ----- -/--	1-11

[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

* Special categories of cited documents :

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Date of the actual completion of the international search	Date of mailing of the international search report
12 July 2012	23/07/2012

Name and mailing address of the ISA/	Authorized officer
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Form PCT/ISA/210 (second sheet) (April 2005)

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	G. BOLZON ET AL: "An indentation-based technique to determine in-depth residual stress profiles induced by surface treatment of metal components", FATIGUE & FRACTURE OF ENGINEERING MATERIALS & STRUCTURES, vol. 34, no. 2, 1 February 2011 (2011-02-01), pages 97-107, XP55032645, ISSN: 8756-758X, DOI: 10.1111/j.1460-2695.2010.01497.x the whole document	1-11
A	BOCCIARELLI ET AL: "Indentation and imprint mapping method for identification of residual stresses", COMPUTATIONAL MATERIALS SCIENCE, ELSEVIER, AMSTERDAM, NL, vol. 39, no. 2, 12 March 2007 (2007-03-12) , pages 381-392, XP005923494, ISSN: 0927-0256, DOI: 10.1016/J.COMMATSCI.2006.07.001 cited in the application the whole document	1-11
A	BELLEMARE S C ET AL: "A new method for evaluating the plastic properties of materials through instrumented frictional sliding tests", ACTA MATERIALIA, ELSEVIER, OXFORD, GB, vol. 58, no. 19, 1 November 2010 (2010-11-01), pages 6385-6392, XP027326689, ISSN: 1359-6454 [retrieved on 2010-10-01] abstract paragraph "2.3 Experimental materials and properties" paragraph "2.4 Experimental frictional test and parameters" paragraph "3. Results and discussion" paragraph "4. Concluding remarks"	1-11
A	WO 2010/003149 A1 (HYSITRON INC [US]; OH YUNJE [US]; SYED ASIF SYED AMANULA [US]; WARREN) 7 January 2010 (2010-01-07) abstract page 12, line 13 - page 13, line 8 page 23, line 24 - page 24, line 18 page 27, line 6 - page 28, line 10	1-11

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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	KR 2010 0049938 A (KOREA RES INST OF STANDARDS [KR]; UNIV CHUNG ANG IND [KR]) 13 May 2010 (2010-05-13) abstract figure 1 paragraphs [0003] - [0007] -----	1-11
A	NAKAMURA ET AL: "Identification of elastic-plastic anisotropic parameters using instrumented indentation and inverse analysis", MECHANICS OF MATERIALS, AMSTERDAM, NL, vol. 39, no. 4, 2 December 2006 (2006-12-02), pages 340-356, XP005788532, ISSN: 0167-6636, DOI: 10.1016/J.MECHMAT.2006.06.004 cited in the application the whole document -----	1-11

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/EP2012/060856

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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		WO 2010003149 A1	07-01-2010
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