

1st International Workshop on Plasticity, Damage and Fracture of Engineering Materials

## Mechanical characterization of metals by small sampling size

Gabriella Bolzon<sup>a\*</sup> and Barbara Rivolta<sup>b</sup>

<sup>a</sup> *Department of Civil and Environmental Engineering, Politecnico di Milano, 20133 Milano, Italy*

<sup>b</sup> *Department of Mechanical Engineering, Politecnico di Milano, 20158 Milano, Italy*

---

### Abstract

Classical plasticity models permit to simulate accurately the macroscopic response of several metals. This ability supports the development of indirect material characterization methodologies based on non-destructive testing, potentially applied for the fast integrity assessment of structural components in operating conditions. In this context, reducing the sampling size mitigates the invasiveness of the experiments and improves the manageability of portable equipment, although the representativeness of the information collected from small material volumes may become an issue.

This contribution focuses on the equivalence between the mechanical properties that can be inferred from metal sampling of typical dimensions of hundreds or dozens microns, corresponding to indentation tests carried out at maximum load differing by one order of magnitude. This topic is addressed by the comparison of the results gathered from experimental and numerical analyses of pipeline steel.

© 2019 The Authors. Published by Elsevier B.V.

This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>)

Peer-review under responsibility of the 1st International Workshop on Plasticity, Damage and Fracture of Engineering Materials organizers

**Keywords:** Mechanical characterization; metals; non-destructive testing; inverse analysis; sampling size.

---

---

\* Corresponding author. Tel.: +39-02-2399-4319; fax: +39-02-2399-4300.

E-mail address: [gabriella.bolzon@polimi.it](mailto:gabriella.bolzon@polimi.it)

## 1. Introduction

Instrumented (depth-sensing) indentation represents a popular mechanical characterization approach based on quick, flexible and (almost) non-destructive testing. In fact, the maximum applied load and the geometry of the indenter tip can be defined on the basis of the material or structural component to be sampled. Thus, applications concern many situations and span different scales, as for instance shown by Bolzon et al. (2008), Bolzon et al. (2010), Palacio and Bhushan (2013), Bolzon et al. (2014), Arzate-Vázquez et al. (2015), Broitman (2016), and by the references listed in these contributions.

The output of indentation tests can be interpreted with the aid of numerical simulations of the experiments, where classical plasticity models reproduce accurately the macroscopic response of several materials, in particular metals, under multiaxial stress-states. Combined experimental-computational tools foster the development of indirect material characterization procedures apt to perform the integrity assessment of structural components on-site, in operating conditions.

An extensive validation study performed by Bolzon et al. (2012) considered indentation tests at 1-2 kN maximum force, conforming to EN ISO 6508:2005 Standards for Rockwell hardness evaluations. The results showed that accurate values of the main mechanical characteristics (elastic modulus, initial yield limit, hardening coefficient, overall strength) of metals are recovered by this methodology when the data describing the geometry of the residual deformation left on metal surfaces are exploited. This information improves the effectiveness and the reliability of the identification procedure, as assessed by Bolzon et al. (2011).

The present contribution focuses on the possibility of reducing the load levels formerly considered. This provision shall attenuate further the invasiveness of the test and improve the manageability of the portable instruments used for the diagnostic analysis of structural components, although the representativeness of the information collected from small material volumes may represent an issue.

The possible equivalence between the material properties that can be inferred from indentation tests carried out at a maximum load of the order of thousands or hundreds N is therefore investigated. This issue is addressed by the comparison of the data gathered from laboratory tests and numerical analyses of pipeline steel.

The present results complement those provided by Bolzon et al. (2018), concerning the evolution of the bulk material properties of steels due to aging in demanding working conditions.

## 2. Experimental and numerical study

The present study considers a sample of pipeline steel, initially tested and characterized according to the methodology proposed by Bolzon et al. (2012). The procedure consists of the following steps.

- A spherical-conical Rockwell indenter tip is pressed against the polished material surface until 1.5 kN reaction force is achieved.
- The load is progressively released and the tool is eventually removed.
- A precise (multi-focal) microscope provides an accurate three-dimensional mapping of the permanent impression produced on the surface of the material sample.
- The data describing the load vs penetration curves and the geometrical profile of the residual imprint constitute the input of an inverse analysis procedure based on the comparison between the measurements and the results of a simulation model of the test.
- The material characteristics are sequentially updated in order to achieve an optimum value, which minimizes the discrepancy between the experimental and the computational output.
- The reliability of the final estimates is checked through the comparison between the actual and the recalculated system response.

In the present application, the material is assumed to be homogeneous and isotropic, with an initial linear elastic response characterized by the modulus of elasticity  $E$  and by the lateral contraction ratio  $\nu$ . The classical Huber-Hencky-von Mises (HHM) constitutive law and an associative flow rule describe the elastic domain and its evolution

beyond the initial yield limit  $\sigma_y$ . Irreversible deformation induces isotropic hardening, described by the exponential rule:

$$\sigma(\varepsilon^p) = \sigma_y \left( 1 + \frac{E\varepsilon^p}{\sigma_y} \right)^n \quad (1)$$

Relation (1) defines the uniaxial response of the material in the inelastic range, in terms of the stress value  $\sigma$  as a function of the plastic strain  $\varepsilon^p$ . Multiaxial stress states produced by indentation are described in the same way, replacing the stress  $\sigma$  and the plastic strain  $\varepsilon^p$  by the corresponding equivalent HHM quantities.

The parameters to be identified by the inverse analysis procedure outlined above are:  $E$ ,  $\sigma_y$  and  $n$ , while  $\nu$  is fixed to the typical value 0.3 for steel. In fact, indentation results are little sensitive to the lateral contraction ratio (Bolzon et al., 2004).

The experiment can be reproduced in the finite element framework, accounting for both material and geometrical non-linearities, as well as for the interaction between the sample surface and the indentation tool. The popular commercial code Abaqus (2016) is used in the present context.

The geometry of the Rockwell tip, shown in Fig. 1, conforms to the specifications provided by Standards. This axis-symmetric solid, made of diamond, is schematized as a rigid body in the simulation model of the test. The interaction of the tip with the material surface obeys Coulomb's relationship with friction coefficient 0.15, suggested by the former experience.

The discretized regions, represented in Fig. 1, have a different radius (1500  $\mu\text{m}$  and 4000  $\mu\text{m}$ , respectively) in order to make the (fixed) boundary conditions almost irrelevant. The meshes are refined under the indenter tip to reproduce the system response accurately. In the contact zone, the typical finite element size is 4  $\mu\text{m}$ . Then, the distance among the nodes placed in the top radius is increased progressively, as shown in the figure.

The numerical analyses performed with these models are rather time consuming. Therefore, an analytical surrogate formulated and trained as shown by Bolzon and Talassi (2012), replaces the original computations in the parameter calibration process. This provision permits to reproduce the experimental output in almost real time.

The parameter set identified in the present application corresponds to:  $E = 205 \text{ GPa}$ ,  $\sigma_y = 318 \text{ MPa}$  and  $n = 0.157$ . These values provide a good matching between the performed measurements and the numerical results.

The thus calibrated model permits to simulate also the test at 200 N maximum force, which represents a rather typical load level for the devices at present available on the market.

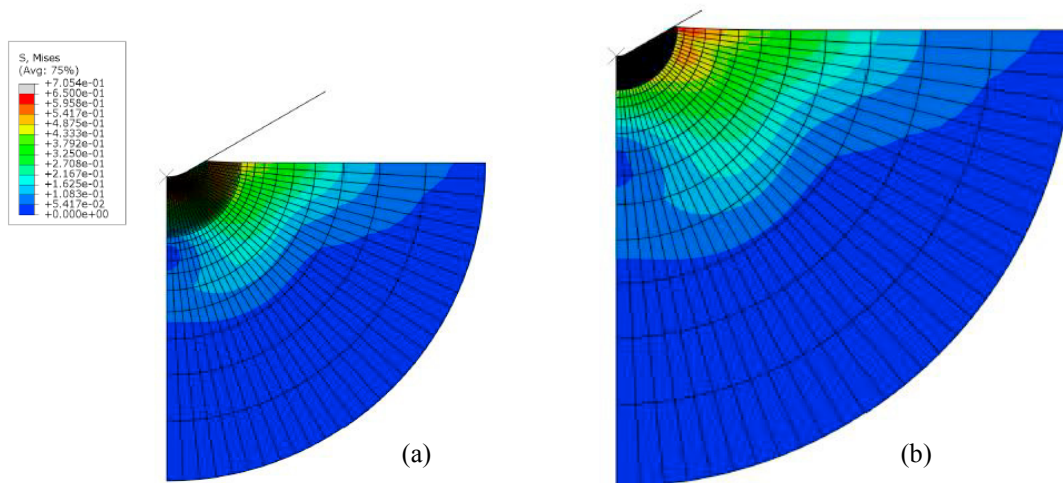


Fig. 1. The material deformation and the equivalent stress distribution under the indenter tip for the reaction force: (a) 200 N; (b) 1.5 kN. The two images are on a different scale.

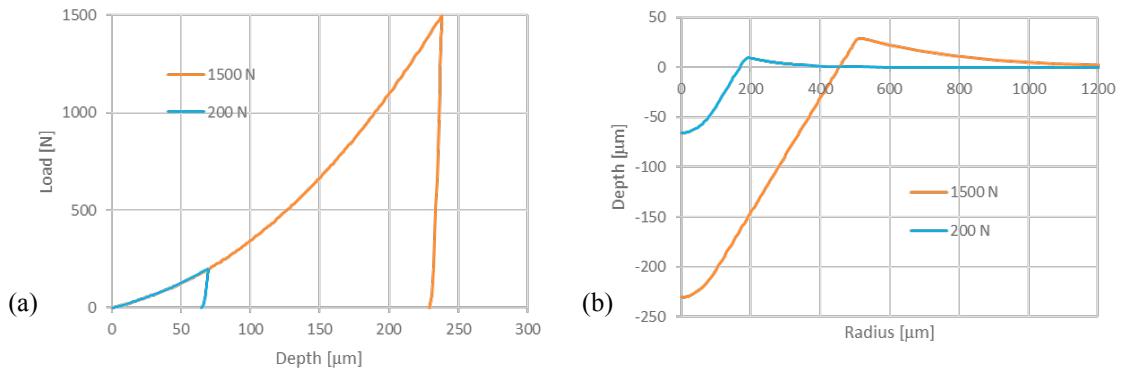


Fig. 2. (a) Indentation (load-penetration depth) curves; (b) profile of the axis-symmetric residual imprint.

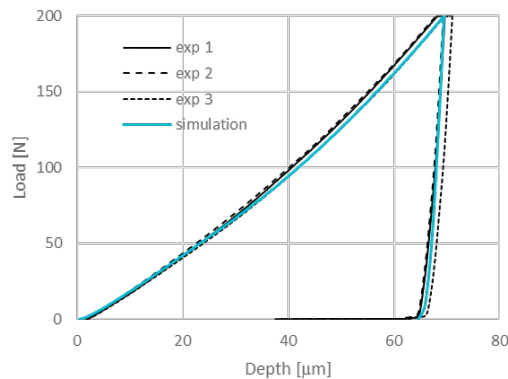


Fig. 3. Indentation (load-penetration depth) curves at 200 N maximum load: experimental vs numerical results.

The graphs in Fig. 2 compare the main results obtained from the relevant finite element analyses. In particular, Fig. 2(b) evidences the different material volume involved by indentation at the different load levels considered in this contribution.

Fig. 3 contrasts the numerical load vs penetration depth curves, represented in Fig. 1(a), to the corresponding experimental result obtained at 200 N maximum load. The agreement is remarkably good.

### 3. Discussion

The inverse analysis tool developed by Bolzon et al. (2012) for the calibration of the main mechanical parameters of metal components is based on indentation tests carried out at a few kN maximum force. This load level is consistent with the requirements of Standards for structural applications, and produces results highly representative of the bulk material properties. In fact, several thousands of metal grains are involved in the test at this scale, as shown by Bolzon et al. (2014).

The results obtained in the present investigation suggest that the volume interested by Rockwell indentation at 200 N maximum force is almost equivalent, in terms of macroscopic material response, to that involved by much higher loads. The representativeness of this small sampling is confirmed also by the limited dispersion of the experimental curves, for instance reported in Fig. 2, which correspond to distinct but nominally identical positions on the sample surface. The estimated number of metal grains contained in the residual imprint for this load level is about 2000 for an average grain size 10 μm. This number reduces to 250 for an average grain size 20 μm. However, the equivalent stress distribution visualized in Fig. 1 suggests that the material volume involved with the test is much larger than the permanent impression produced by indentation.

In the present study, the indentation tests were performed in laboratory, on a surface accurately lapped and cleaned. Thus, the influence of the surface roughness on the output of the indentation test was almost ruled out. However, the effect of friction and of other noise sources is expected to be marginal also in on-site applications.

#### 4. Closing remarks

Instrumented (depth-sensing) indentation represents an effective non-destructive approach to the mechanical characterization of materials, which can be potentially applied for the safety assessment of structural components in operation. A significant reduction of the maximum load facilitates the development of the relevant tools, with a potential gain particularly relevant for portable equipment. The results of the present investigation show that this task is feasible, at least in the case of metals.

One possible alternative, suggested by the experience gained so far, considers the imprint geometry as the only source of information for material characterization purposes. The relevant methodology proved to be reliable at 1-2 kN maximum load. The accuracy of the portable instruments at present available on the market for the mapping of the smaller imprints produced at 200 N, as considered in the present study, is at present under evaluation.

#### Acknowledgements

This study has been carried out within the NATO SPS project G5055 “Development of Novel Methods for the Prevention of Pipeline Failures with Security Implications”. NATO financial support is gratefully acknowledged. The finite element analyses have been performed by Greta Cornaggia. The material characterization based on indentation tests at 1.5 kN maximum load has been carried out at the laboratories of the Company EniProgetti in Venezia-Marghera (Italy). This collaboration is also gratefully recognized.

#### References

- Abaqus, 2016. Dassault Systèmes Simulia Corp. Paris/Boston.
- Arzate-Vázquez I., Chanona-Pérez J., Rodríguez-Castro G., Fuerte-Hernández A., Méndez-Méndez J., Gutiérrez-López G., 2015. Indentation technique: overview and applications in food science. In: Hernández-Sánchez H., Gutiérrez-López G. (Eds.), *Food Nanoscience and Nanotechnology*, Springer, pp. 81–98.
- Bolzon, G., Bocciarelli, M., Chiarullo, E.J., 2008. Mechanical characterization of materials by micro-indentation and AFM scanning. In: Bhushan, B., Fuchs, H. (Eds.), *Applied Scanning Probe Methods XII Characterization*. Springer-Verlag, Heidelberg, pp. 85–120.
- Bolzon, G., Buljak, V., Maier, G., Miller, B., 2011. Assessment of elastic-plastic material parameters comparatively by three procedures based on indentation test and inverse analysis. *Inverse Problems in Science and Engineering* 19, 815–837.
- Bolzon, G., Chiarullo E.J., Egizabal, P., Estournes, C., 2010. Constitutive modelling and mechanical characterization of aluminium based metal matrix composites produced by spark plasma sintering. *Mechanics of Materials* 42, 548–558.
- Bolzon, G., Gabetta, G., Molinas, B.J., 2014. Integrity assessment of pipeline systems by an enhanced indentation technique. *ASCE Journal of Pipeline Systems Engineering and Practice* 04014010, 1–7.
- Bolzon, G., Maier, G., Panico, M., 2004. Material model calibration by indentation, imprint mapping and inverse analysis. *International Journal of Solids and Structures* 41, 2957–2975.
- Bolzon, G., Molinas, B., Talassi, M., 2012. Mechanical characterisation of metals by indentation tests: an experimental verification study for on-site applications. *Strain* 48, 517–527.
- Bolzon, G., Rivolta, B., Nykyforchyn, H., Zvirko, O., 2018. Mechanical analysis at different scales of gas pipelines. *Engineering Failure Analysis* 90, 434–439.
- Bolzon G., Talassi, M., 2012. Model reduction techniques in computational materials mechanics. In: Zavarise G., Boso D.P. (Ed.s). *Bytes and science*. CIMNE, Barcelona, pp.131–143.
- Brotman E., 2016. Indentation hardness measurements at macro-, micro-, and nanoscale: a critical overview. *Tribology Letters* 65(23), 1–18.
- Palacio M.L.B., Bhushan B., 2013. Depth-sensing indentation of nanomaterials and nanostructures. *Materials Characterization* 78, 1–20.
- EN ISO 6508, 2005. Metallic materials – Rockwell hardness test.