International Journal of Mechanical Sciences 123 (2017) 162–176

Contents lists available at ScienceDirect

International Journal of Mechanical Sciences

journal homepage: www.elsevier.com/locate/ijmecsci

A robust inverse analysis method to estimate the local tensile properties of heterogeneous materials from nano-indentation data

Damaso M. De Bono[a,b,*], Tyler London[a], Mark Baker[b], Mark J. Whiting[b]

[a] *TWI Technology Centre, Ferrous Road, Riverside Park, Middlesbrough TS2 1DJ, UK*
[b] *Faculty of Engineering and Physical Sciences, University of Surrey, Guildford, Surrey GU2 7XH, UK*

ARTICLE INFO

Keywords:
Inverse analysis
Nano-indentation
FEA
Objective function
Structural steel
Tensile properties
Multi-phase material
Composite material
Elastic-plastic constitutive behaviour

ABSTRACT

Most current analysis of nano-indentation test data assumes the sample to behave as an isotropic, homogeneous body. In practice, engineering materials such as structural steels, titanium alloys and high strength aluminium alloys are multi-phase metals with microstructural length scales that can be the same order of magnitude as the maximum achievable nano-indentation depth. This heterogeneity results in considerable scatter in the indentation load-displacement traces and complicates inverse analysis of this data. To address this problem, an improved and optimised inverse analysis procedure to estimate bulk tensile properties of heterogeneous materials using a new 'multi-objective' function has been developed which considers nano-indentation data obtained from several indentation sites. The technique was applied to S355 structural steel bulk samples as well as an autogenously electron beam welded sample where there is a local variation of material properties. Using the new inverse analysis approach on the S355 bulk material resulted in an error within 3% of the experimental yield strength and strain hardening exponent data, which compares to an approximate 9% error in the yield strength and an 8% error in the strain hardening exponent using a more conventional approach to the inverse analysis method. Applying the new method to indentation data from different regions of an S355 steel weld and using this data as an input into an FE model of the cross-weld, tensile data from the FE model resulted matching the experimentally measured properties to within 5%, confirming the efficacy of the new inverse analysis approach.

1. Introduction

The inverse analysis of nano-indentation data has attracted increasing interest in the scientific community because of its potential to predict and measure elastic-plastic properties in local areas for different material applications, from coatings to welds, which would be difficult to test otherwise using more standard testing methodologies [1–9].

The inverse indentation problem aims to identify the unknown tensile properties of a material from only the load-depth trace obtained from experimental indentation testing. There are three main inverse analysis techniques that can be employed to extract tensile properties of materials from instrumented indentation experimental data: the representative stress-strain method [10–17], iterative FEA [1–5,7,9], and artificial neural networks [18–20]. This paper is concerned only with the inverse analysis technique by iterative FE simulations. For this approach, in order to approximately solve the inverse problem for a given material, finite element models of the experimental set up are analysed. Different sets of elastic-plastic material properties (e.g. Young's modulus, yield strength, strain hardening exponent) are used in the simulations until the simulated load-depth curve matches the experimentally measured load-depth curve. The combination of elastic-plastic material properties used in the FE model that result in the simulated load-depth curve matching the experimental curve are assumed to be the elastic-plastic properties of the material being investigated.

Inverse analysis by iterative FE simulations requires two main assumptions. The first assumption is that the model is sufficiently accurate and representative of the real experiment. This means that if

---

Abbreviations: D, Characteristic material length scale (eg grain size); E, Young's modulus (typically in MPa); EB, Electron beam; EBW, Electron beam welding; E_r, Reduced modulus; FEA, FE, Finite element analysis, finite element; H, Indentation depth; H, Indentation hardness; HAZ, Heat affected zone; LD, Longitudinal direction; M, Strain hardening exponent in Holloman's stress-strain constitutive law; n_{exp}, Number of experimental measurements; P_{exp}^{avj}, Experimental averaged load; P_{exp}, Experimental indentation load; P_{sim}, Simulation indentation load; TD, Transverse direction; TTD, Through thickness direction; σ, True stress; σ_y, Yield stress in Holloman's stress-strain constitutive law; σ_y^{inv}, Optimal inverse analysis solution for the yield strength; ε, True (logarithmic) strain; Φ, Least square error; m, Strain hardening exponent; m^{inv}, Optimal inverse analysis solution for the strain hardening exponent

* Corresponding author.

E-mail addresses: damaso.debono@twi.co.uk (D.M. De Bono), tyler.london@twi.co.uk (T. London), m.baker@surrey.ac.uk (M. Baker), m.whiting@surrey.ac.uk (M.J. Whiting).

http://dx.doi.org/10.1016/j.ijmecsci.2017.02.006
Received 24 October 2016; Received in revised form 30 January 2017; Accepted 6 February 2017

the stress-strain curve corresponding to the indented material is used as the input in the FE model, then the corresponding simulation of the indentation testing will produce a load-depth curve that very nearly replicates the experimentally measured load-depth curve. The second assumption concerns uniqueness. Specifically, the inverse analysis problem assumes that there is only one set of elastic-plastic parameters for which the simulation produces a load-depth curve that replicates the experimental load-depth curve. If this is not the case, then it would be possible for materials with two different stress-strain curves to generate the same load-depth trace. As result, if this was true it would not be possible to uniquely identify the tensile behaviour of the indented material through inverse analysis. The issue of uniqueness has proved to be a non-trivial subject and it has been studied by several authors [21–26].

Most materials relevant to many industrial applications (energy, civil, oil and gas, transport, etc) are highly heterogeneous and multiphase, this heterogeneity extending from the nano- to macro- scale. In these cases, it is crucial to ensure that the experimental indentation data used in the inverse analysis process are representative of the material bulk response.

When indentation volumes and microstructural volumes are of the same order, this can often undermine the potential of using indentation to measure bulk mechanical properties of the material. Most indentation solutions are based on the self-similarity approach, derived from the infinite half-space model and that model assumes spatially uniform mechanical properties [27]. As a consequence, the properties extracted from indentation data are ultimately averaged quantities characteristic of a material length scale, which is defined by the indentation depth (h) or the indentation radius (a). Based on these considerations, if the microstructural length of the material (D) is of the order of the indentation depth (h), the classical tools of continuum indentation analysis would not apply. Several authors [27–31] have investigated the influence of microstructure heterogeneities on the indentation response. Statistical nano-indentation techniques were generally used during the course of these studies, where large grids of nano-indentations were undertaken and measured. This approach enabled sampling a large area of the material, providing a significant amount of experimental data that can be analysed by statistical means.

If the material heterogeneity is characterised by a length scale (D) and if the indentation depth (h) is much smaller than the characteristic size of the heterogeneity ($h \ll D$), then a single indentation will generate data that is representative of the individual phase response. Conversely, if the maximum indentation depth is much larger than the characteristic size of the microstructure characteristic length, $h \gg D$, the test data will be representative of the composite response of the material. The 1/10 Buckle's rule-of-thumb is a reference criterion for all the investigations in this field. Based on this rule, in order to measure the properties of the individual phase the indentation depth should be at most 1/10 of the characteristic size of the microstructure ($h < 0.1D$). At higher indentation depths, $h > 0.1D$, the individual microstructural heterogeneities start to interfere with themselves in the indentation response, ultimately generating an averaged homogenised (bulk) response of the material [31] (Fig. 1).

Due to constraints in the achievable maximum load and maximum depth sampled in commercial nano/micro-indentation instruments, the influence of microstructural characteristic lengths in the indentation response is almost inevitable. This results in a significant variability of the experimentally measured load-depth curves, ultimately raising concerns over the validity of using experimental load-depth curves during the inverse analysis process. In this case, several authors aiming to characterise composite microstructure materials [1–4,6–8,32] overcame the variability exhibited in the experimental load-depth curves by using the conventional approach of selecting a representative experimental curve (e.g. the average load-depth curve) and determining the least squares error with respect to the simulated curves. Whilst this approach can be effective for materials that exhibit little variability, it can be an additional source of errors introduced in the calculation of the inverse analysis parameters of the material when the load-depth curves exhibit scatter. The study undertaken and described in this paper aims to develop and validate a more robust methodological approach for inverse analysis of experimental load-depth nano-indentation data measured from heterogeneous materials. This was achieved through the definition of a new weighted averaging approach that is able to handle the variable indentation response of the material depending on the indentation site. The new methodology was validated by determining the elastic-plastic constitutive behaviour of S355 structural steel samples as well as an autogenously electron beam welded sample.

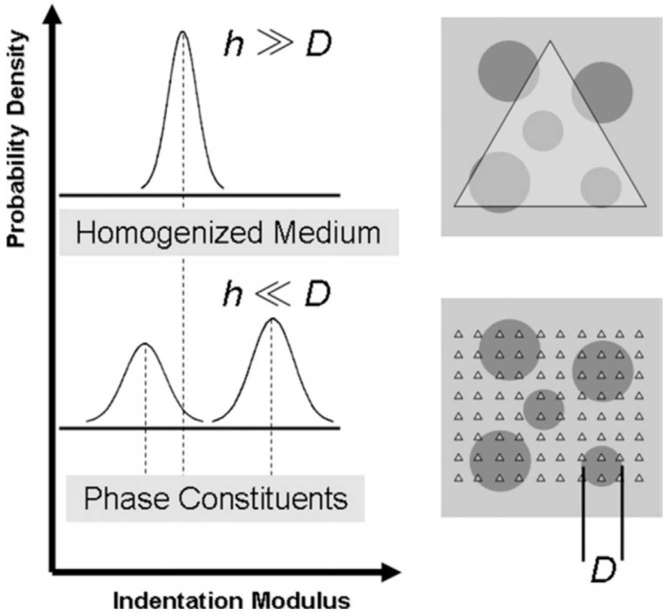

Fig. 1. Schematic diagram of the grid indentation technique applied to heterogeneous materials (adapted from [27]).

2. Method and approach

2.1. Experimental test programme

2.1.1. Material

The material chosen for the study was structural steel S355. The composition for this grade of steel is reported in Table 1.

S355 is a low carbon steel widely used in the construction, maintenance and manufacturing industries and suitable for numerous general engineering and structural applications.

The inverse analysis technique was first validated by considering only the parent material of the steel. Successively, a second phase of the validation process comprised applying the inverse analysis technique to investigate the tensile properties of a weld generated by butt welding two S355 plates together using electron beam technology (Fig. 2).

For the first stage of the validation, three cross-sections were produced that were aligned with the three principal directions of the plate, as represented in Fig. 2: longitudinal direction (LD), transverse direction (TD) and through thickness direction (TTD). The objective was to investigate potential differences in anisotropy of the microstructure that need to be taken into account.

Three metallographic specimens were prepared in the three directions of the plate. The specimens were polished through standard polishing techniques to a 1/4 μm finish. Reflective light microscopy micrographs of the cross-sections in all three directions were generated and these are shown in Fig. 3. The micrographs show that the microstructure is isotropically consistent. Ferrite grains with a small volume fraction of pearlite nodules are present. The other dominant microstructural feature is upper bainite, in which the dominant phase is