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Research paper

Experimental and computational issues for automated extraction of plasticity parameters from spherical indentation

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ABSTRACT

Software packages are being developed for automated extraction of plasticity parameters from indentation data (primarily load-displacement plots, although residual indent dimension data are also likely to be useful). Their design must be closely integrated with the associated experimental measurements. The procedure involves iterative FE simulation of the penetration of a spherical indenter into a sample, with automated convergence on a best-fit set of parameter values characterizing the yielding and work hardening response of the material (in a constitutive law). This paper outlines the main issues involved in optimization of experimental conditions and model formulation. Illustrative experimental data are presented from extruded rods of 5 metallic materials. Experimental issues include the dimensional scales of the indenter radius, R, and the depth of penetration, δ, with δ/R (the "penetration ratio") being of particular significance. A brief study is presented of the potentially conflicting requirements of deforming a volume large enough to represent the response of the bulk and having a value of δ/R that creates plastic strains in a range that will adequately capture the work hardening response. A key conclusion of this study is that a "mid-range" indentation facility is likely to be optimal, with a load capability of at least a few kN, able to create δ/R values up to ~40%, with R ~0.5–2 mm. Other experimental issues include displacement measurement techniques, calibration of machine compliance and the possibility of material anisotropy (due to crystallographic texture). Issues related to formulation of the FE model include specification of the domain and mesh, selection of the constitutive plasticity law and simulation of interfacial friction. The convergence algorithm used is also described.

1. Introduction

There has been increasing focus over the past decade or two on obtaining (true) stress-strain curves (well beyond the elastic limit) from outcomes of instrumented indentation experiments (mainly load-displacement plots, although residual indent shapes can also be used). Since these stress-strain curves are regarded as prime indicators of the plasticity characteristics of a material, and indentation is a much more versatile and convenient procedure than conventional uniaxial testing, this quest has a strong motivation. The approaches used fall into two main categories. Many studies (Taljat et al., 1998; Herbert et al., 2001; Basu et al., 2006; Kang et al., 2006; Pelletier 2006; Guelorget et al., 2007; Xu and Chen 2010; Hamada et al., 2012; Hausild et al., 2012; Pathak and Kalidindi 2015) have sought to identify analytical formulations that can be applied to the experimental data. This has obvious attractions, since such a formulation, even if involving relatively complex expressions and algorithms, would allow rapid extraction of the stress-strain curves via a well-defined path. Unfortunately, the stress and strain fields beneath an indenter, even one with a simple shape such as a sphere, are complex and change with penetration depth, making it very difficult to identify realistic analytical relationships. The prospects for this approach, certainly in terms of having a robust procedure that can be applied to a wide range of materials, are not promising.

The alternative approach (Dao et al., 2001; Bolzon et al., 2004; Bouzakis and Michailidis 2004; Bouzakis and Michailidis 2006; Pelletier 2006; Guelorget et al., 2007; Heinrich et al., 2009; Dean et al., 2010; Bobzin et al., 2013; Patel and Kalidindi 2016; Dean and Clyne 2017) is to use FEM modeling to (accurately) capture these evolving stress and strain fields, with the challenge then being to establish the stress-strain curve most closely consistent with measured indentation outcomes. This is a major challenge, but the approach is conceptually transparent, rigorous and simple (which cannot be said of the first type of methodology). However, its wide implementation has been inhibited by the need to carry out FEM modeling runs that are specific to each individual case, and also by uncertainties about how to converge on the

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"best fit" stress-strain curve and how to assess the confidence that can be placed in it.

For a material with a given (uniaxial) stress-strain curve, assumed to be applicable to deviatoric (von Mises) components of stress and strain for multi-axial situations, FEM can readily be used to predict the load-displacement plot (and residual indent dimensions). This can be done for any given indenter shape, provided that the important boundary conditions (potentially including the effects of friction between indenter and sample) can be established. However, the inverse problem of inferring the stress-strain relationship from such a load-displacement plot is much more challenging, with considerable scope for ambiguity, such as different stress-strain relationships giving effectively the same load-displacement plot.

For both types of approach, it has been recognized (Futakawa et al., 2001; Bucaille et al., 2003; Capehart and Cheng 2003; Chollacoop et al., 2003; Cheng and Cheng 2004; Ma et al., 2012) that there may be advantages in obtaining more comprehensive sets of experimental data. For example, doing repeat runs with indenters having different shapes has often been proposed, and indeed it is logical that this should be helpful, since the way that the stress-strain curve influences the indentation outcomes will be different with different indenter shapes. It has occasionally been suggested that simply using different indenter sizes may also be helpful, but this is unlikely to create benefits, since the stress and strain fields beneath an indenter are scale-independent. For example, the fields created by penetration of a sphere to a depth corresponding to, say, 10% of its radius are identical for radii of, say, 10 µm and 10 mm. The absolute value of the load at this point will be 10^6 greater for the latter case, while the penetration will be 10^3 greater, but the information being provided about the stress-strain response of the material is the same, provided the volume being interrogated is in both cases large enough to be representative of the bulk response.

The main requirement now, in order for procedures (and dedicated software packages) to become widely accepted and employed, is clear identification of the factors that affect sensitivities and efficient convergence on "correct" solutions for inferred properties. There are several key issues, concerning both experimental procedures and computational formulation. Some of these, including the development of algorithms for convergence on best fit parameter combinations, have been addressed by Isselin et al. (2006), while Karthik et al. (2012), among others (Giannakopoulos and Suresh 1999; Taljat and Pharr 2004), explored the influence of friction, concluding that it has a significant effect at penetration ratios above about 20%, particularly on the residual indent shape. Other workers (Sun et al., 1999; Ullner et al., 2010; Van Vliet, Prchlik et al. 2011) have drawn attention to the significance of machine compliance in the context of indentation load-displacement data. The present paper is aimed at examining all of the main issues in some detail, including the relationships between the experimental procedures and the numerical simulations. This is done using a wide range of experimental indentation data, illustrating how they are used in an automated way within software packages to obtain the values of parameters in constitutive stress-strain laws.

2. Experimental issues

2.1. Choice of indenter shape

There are several powerful motivations for using spherical indenters. One of these is that, since it is not a self-similar shape, the stress and strain fields change qualitatively as penetration takes place. Hence, the information being obtained over different depth ranges is analogous to carrying out separate tests with different indenter shapes (reducing the likelihood of different stress-strain curves giving very similar load-displacement plots). This point has been clarified previously (Dean and Clyne 2017).

There are also more practical motivations. One is that a sphere is much less prone to becoming damaged than are shapes having edges or points, and is also easier to specify and manufacture. Spheres (of WC-based cermets, with hardness and stiffness values high enough for most purposes), having diameters in the preferred range of about 1–4 mm (see Section 2.2 below), are cheap and readily obtained. There is also reduced risk with spheres of encountering the computational problems that are often associated with simulation of behavior in regions of high local curvature (edges or points).

Finally, at least with (approximately) isotropic materials, a spherical indenter allows the FEM modeling to be radially symmetric (2-D), which is not possible with many shaped indenters. The potential need for very large numbers of iterative FEM runs makes this a more significant issue than it would be under most other circumstances. All of the work described in this paper relates to use of spherical indenters.

2.2. Length scale effects

It is important, when the objective is to extract bulk properties, to indent on a suitable scale, while retaining the key advantages of being able to test small, flat samples, to carry out point-to-point mapping of properties etc. In particular, the volume being interrogated must have a (stress-strain) response that is representative of the bulk. It is on this *meso*-scale (such that indents are large enough for representative material response, but small enough to allow small samples and mapping) that this type of work needs to be focused.

The minimum indent size for representative response depends on microstructure, but in many cases it will require deformation of an assembly of grains - at least about a dozen and preferably more. Only when such an assembly is being deformed is it possible to capture the influence, not only of the crystallographic texture of the material, but also of the way that cooperative deformation of neighboring grains takes place. This is likely to be affected, not only by texture, but also by factors such as the ease of grain boundary sliding. Simply taking the average of the load-displacement responses from indents made in a large number of individual grains will not even approximately capture the bulk response. (The same arguments would apply to carrying out conventional uniaxial tests on a set of single crystal samples having orientations representative of the texture of a polycrystal.) A crude rule of thumb might be that, viewed on the free surface, the indent should straddle at least "several" grains. Of course, the corresponding minimum indent diameter might range from below 1 µm to above 1 mm, but it will certainly be small enough in most cases to offer the attractions outlined above.

Grain sizes of around 100 µm or more are, of course, common. In general, therefore, indent diameters should be at least a few hundred µm. This does require relatively large indenters (~ mm dimensions) and therefore large loads (~ hundreds of N, or even several kN), which may be beyond the range of some indentation systems (but perhaps below the commonly-used ranges of some conventional mechanical testing systems). However, systems in this "intermediate" load range are in general easier and cheaper to construct and use than either of the other two types of system. Moreover, a relatively coarse scale of indentation minimizes the problems associated with surface roughness, oxide films, contamination etc.

There is also a further issue, which relates to the indenter penetration depth, δ, as a ratio to the indenter radius, R. It might be imagined that, while the load needed to penetrate to a given δ/R, and the stresses in the material, would depend strongly on the material (hardness), the strains would not. In fact, this is not really true, since materials with different work hardening characteristics tend to exhibit significantly different plastic strain fields (for a given δ/R). Furthermore, even if the peak strain is, say, 40%, the indentation response will be considerably more sensitive to much lower strain regions of the stress-strain curve, in which most of the plastic deformation takes place. This issue is examined quantitatively in Section 5.4.1.