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Use of spherical indentation data changes to materials characterization based on a new multiple cyclic loading protocol

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Abstract

An interpretation of spherical indentation experiments and the determination of hardening law parameters is the challenge in this paper. A new protocol based on multiple cyclic loading allowed models describing the indentation data changes to be determined from a numerical study. Inversion of these models, determined for two constitutive equations with isotropic hardening, combined with a sensitivity study enabled the study of the uniqueness of the solution. It is shown that although the new protocol is not necessary to determine the two Hollomon equation parameters, it is necessary to determine accurate values of the three Ludwig equation parameters.

From an experimental study, it was shown that methods based on a "reverse" analysis can be used in order to have an evaluation of the mechanical properties in a first-order approximation.

Keywords: Spherical indentation; Materials characterization; Multiple cyclic loading

1. Introduction

An instrumented indentation test consists in measuring, simultaneously, the indentation load P and the indentation depth h during the penetration of an indenter into a sample. This test can be used in order to determine some parameters of the work-hardening behavior law of the tested sample. The more commonly used is the Hollomon isotropic work-hardening law defined by the following equation:

$$\sigma = \sigma_y^{1-n} E^n \varepsilon^n \tag{1}$$

This relationship gives, in the case of a monotonic solicitation in the elasto-plastic regime, the material flow stress. E, σ_y, n and ε, are, respectively the Young modulus, the yield stress, the work-hardening exponent and the strain.

The first identified parameter, from an indentation test, is the reduced Young modulus E^* defined by $E^* = [(1-\nu_i^2)/E_i + (1-\nu_s^2)/E_s]^{-1}$, where ν is the Poisson ratio, s stands for "sample," whereas i stands for "indenter". The determination of E^* is based on the Hertz elastic contacts theory [1]. Sneddon [2] proposed a general solution to the problem of an elastic sample indented by any shape of indenter. In the case of spherical indentation, the contact stiffness is defined as follows:

$$S = \frac{dF}{dh} = 2E^* a \tag{2}$$

where a is the contact radius. When the elastic regime is over, Eq. (2) can still be applied at the beginning of the unloading [3–5]. Following this, Doerner and Nix [6], Loubet et al. [7] and Oliver and Pharr [8] proposed to deduce E^* from an indentation test using relationship (2). These methods can be distinguished from one another by the way the contact radius is evaluated. Lastly, Hay and Wolff [9] proposed a correction for the application of the Hertz theory by introducing a factor in order to take into account the radial displacements of material under the indenter.

Concerning the non-elastic behavior, many methods have been proposed to deduce the mechanical parameters, usually deduced from a tensile test, from an indentation test. These methods can be distinguished from one another by the indenter they use (Vickers, sharp, spherical, etc.) and the data they use. Moreover, two kinds of methods can be used to extract mechanical properties from the indentation test. The first is based on the inversion of models established from a numerical study and is called a "reverse" analysis. The second is based on an "inverse"

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analysis. The greater part of established methods are based on a “reverse” analysis, however, Nakamura et al. [10] proposed the determination of the properties of graded materials by using an “inverse” analysis protocol in spherical indentation. We can also quote Bolzon et al. [11] who applied “inverse” analysis to conical indentation. Most of the “reverse” analysis methods are based on the modelling of the representative strain introduced by Tabor [12]. Dao et al. [13] proposed to extract the Eq. (1) parameters from a unique sharp indentation. They also showed that the determined parameters are very sensitive to small variations in the indentation data. This work has been followed by Chollacoop et al. [14] who applied the previous method with two sharp indenters with different apex angles. Then, they showed an improvement in the determination of the material properties with two indenters. Moreover, the use of two sharp indenters significantly decreased the sensitivity of the predicted parameters to the perturbations in the indentation data. Bucaille et al. [15] also proposed using different sharp indenters in order to solve the problem.

The uniqueness of the solution has been fully studied by Cheng and Cheng [16], who demonstrated that in the case of sharp indentation, several sets of Eq. (1) parameters can lead to the same $P(h)$ curve. However, they demonstrated that this problem does not occur in spherical indentation. Indeed, the method of Dao et al. [13] has been extended to the spherical indentation by Cao and Lu [17] and led to a unique determination of the Eq. (1) parameters. We can also quote Beghini et al. [18] who determined Eq. (1) parameters from the inversion of a $P(h)$ curve model in spherical indentation.

This paper deals with a study of the uniqueness of the solution in the case of spherical indentation. Moreover, we propose an experimental method based on a “reverse” analysis to evaluate the parameters of two constitutive equations by using a unique indenter and a new multiple cyclic loading protocol.

2. Data deduced from an indentation test

Depending on the kind of loading, different data can be deduced from an indentation test (see Fig. 1, the curves of which are deduced from numerical results). If the test consists of a unique loading, the $P(h)$ curve and the total energy $W_t(h)$ changes can be deduced. If the test consists of a loading and an unloading cycle, in addition to $P(h)$ and $W_t(h)$, both the elastic $W_e(h_{max})$ and plastic $W_p(h_{max})$ energies at the end of the loading can be deduced. Moreover, we can deduce the contact stiffness $S(h_{max})$ (defined in Eq. (2)) at the maximal load. Finally, if the test consists of n loading, unloading and reloading cycles, in addition to the previous data we can deduce n points of $W_e(h_{(max)i})$, $W_p(h_{(max)i})$ and $S(h_{(max)i})$ changes. Moreover, from the energies, we can deduce the energy ratio changes as $W_p/W_t(h_{(max)i})$, $W_e/W_t(h_{(max)i})$ and $W_e/W_p\ (h_{(max)i})$.

Among the three energies, only two are independent. Indeed, they are linked by

$$W_p = W_t - W_e \quad (3)$$

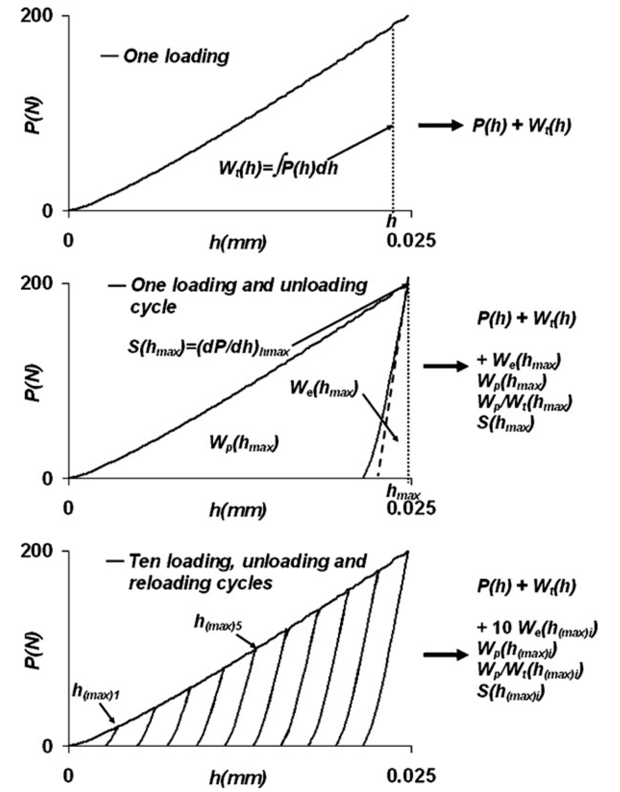

Fig. 1. Different kinds of loading cycles lead to different indentation data.

Moreover, only one energy ratio is sufficient in order to deduce the two others. Indeed, they are linked by

$$\frac{W_p}{W_t} = 1 - \frac{W_e}{W_t} = \left(1 + \frac{W_e}{W_p}\right)^{-1} \quad (4)$$

This is the reason why, for the three energies, we propose to study both the elastic and total energies. Moreover, although the W_p/W_t ratio is linked to the two studied energies, we propose to study it in order to limit the errors of modelling. Concerning the contact stiffness S, because it is obtained by derivating the $P(h)$ curve, it is very sensitive to oscillations of the $P(h)$ curve. For this reason this data is not studied in the following numerical study. Thus, this study concerns the $P(h)$, W_e, W_t and W_p/W_t changes during a spherical indentation test which are deduced from 10 loading, unloading and reloading cycles.

3. Numerical study

3.1. Presentation

A numerical study was conducted in axi-symetric mode with the finite element (FE) code Cast3M. Fig. 2 shows the FE model where the axi-symetric boundary conditions are imposed on the axi-symetric axis.

The sample is modelled by a bulk divided into several areas. In the contact area, the mesh is made of 8-node elements (named QUA8 in the FE code) with quadratic interpolation. In this area, the size of the elements is less than 4 μm in order to obtain $P(h)$ curves with the lowest oscillations. The rest of the sample is