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A criterion to identify sinking-in and piling-up in indentation of materials

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ABSTRACT

The instrumented indentation test is usually used to determine the mechanical properties of materials. Depending on the nature of the material, the way the matter flows under the indenter by piling-up or sinking-in affects the calculation of these mechanical properties. Consequently, corrections proposed by Oliver and Pharr and Loubet et al. should be done according to these two behaviors in addition to other corrections associated with the indenter tip defect as well as the compliance of the instrument. In this work we tested different materials having supposedly piling-up or sinking-in behavior: low-carbon steel, aluminum, brass, copper, beta tricalcium phosphate (β-TCP) bioceramic, rolled or sintered stainless steel and ceramic composite TiB_2–60% B_4C by using two types of indenter, i.e. Vickers and Berkovich ones. From the corrected load–indenter displacement curve, we showed that a criterion, defined as the ratio between the residual indentation depth and the maximum indentation depth reached at the maximum load, is able to identify the predominant deformation mode. For materials for which this ratio is higher than 0.83 piling-up prevails while it is sinking-in when it is lower than 0.83. When the ratio equals 0.83, the two modes of deformation should coexist since the calculations made using either correction of Oliver and Pharr or Loubet et al. give the same results. This novel way of considering the instrumented indentation measurements renders more accurate the determination of the hardness and the elastic modulus since the observation of the indent is then not required for identifying the deformation mode which affects the contact area calculation.

1. Introduction

During the indentation of a material by a very hard indenter, the matter may flow differently depending on the mechanical properties of the material, the nature and the shape of the indenter. Usually two distinct modes of deformation are considered: i) "Sinking-in" when the material is pulled down toward the tip of the indent and ii) "Piling-up" when the material is pushed away from the center of the indent. For classical indentation tests where the diagonal of the indent is measured optically, these modes of deformation have little effect on the hardness measurement since it is recognized that the diagonal length of the indent remains constant under the maximum loading and after the withdrawal of the indenter. For instrumented indentation tests, the mechanical properties, both hardness and elastic modulus, are calculated with a precise value of the contact area which is related to the contact depth. It is clear that whatever the two modes sinking-in and piling-up affect its calculation [1]. For example, Alcala et al. [2] mentioned that errors up to 30% can be introduced in the computation of the contact area if the deformation mode is not taken into account. Since the determination of true hardness and elastic modulus requires the knowledge of the contact depth as precisely as possible, numerous studies have been performed on the conditions of its determination and give the corrections that have to be applied in order to take into account the bluntness of the indenter tip [3], the frame compliance [4] and the two modes of deformation [5].

For these latter it was observed that for soft materials with low values of both hardness to elastic modulus ratio (H/E) and strain hardening exponent, n, to elastic modulus ratio (n/E), the piling-up mode predominates [6]. Similarly Cheng and Cheng [7], Xu and Rowcliffe [8], found that, for a given indenter, piling-up or sinking-in behavior are associated to the ratio of the yield stress Y to elastic modulus E. For high values of Y/E only sinking-in occurs while for small values piling-up or sinking-in may occur [9]. When E is not

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known, some information about the deformation mode can be drawn from the knowledge of the indentation depth h. A systematic study of piling-up and sinking-in modes as well as their influence on the determination of h has been performed by Giannakopoulos and Suresh [10] using finite-element simulations on elastic–plastic materials. It was found that the ratio of the residual depth h_f to the maximal depth of penetration h_{max} obtained directly from the load–displacement curve allows identifying the sinking-in or piling-up modes. For materials having $h_f/h_{max} > 0.875$ piling-up is likely to occur while it is sinking-in for $h_f/h_{max} < 0.875$. The lower limit $h_f/h_{max}=0$ corresponds to fully elastic deformation and the upper limit $h_f/h_{max}=1$ corresponds to rigid-plastic behavior. In the case of pyramidal indenter also, Alcala et al. [2] have rewritten the relation between the contact area and depth penetration in terms of a factor α which takes account of the surface deformation. It has been found that α is >1 for piling-up and <1 for sinking-in.

The methodologies described above are all of interest but they are based on measurements that have to be as precise as possible. Since these measurements are affected by experimental biases such as the indenter tip defect [3], and the compliance of the experimental set up, frame and sample dimensions and mounting [4], some corrections have to be done to the measurements according to these biases in order to obtain valid results [2–4]. The usual methods of calculation which take into account the surface deformation modes are those of Oliver and Pharr [11] for sinking-in and Loubet et al. [12] for piling-up. Although the effect can be considered as negligible for nano indentation measurements, a compliance correction is necessary in micro-indentation tests since the measured indentation depth is sensible to the sample mounting and the indentation testing conditions [13].

In this paper, a variety of materials that are likely to exhibit one or the other mode of deformation are studied. The frame compliance is determined for correcting the indentation depth. Two types of indenter, Berkovich and Vickers, are used to examine their effect on the deformation mode and, consequently, on the mechanical behavior. Corrected instrumented microindentation measurements are studied in order to confirm the literature assessments and to define a parameter able to identify the deformation mode of the different materials without any other observations of the indent or measurements than the values obtained from the standard instrumented indentation test. The elastic modulus determined by using Vickers and Berkovich indenters is afterwards compared and discussed after Oliver and Pharr [11] and Loubet et al. [12] corrections according to the deformation mode of the indent to validate the proposed methodology.

2. Theoretical background

2.1. Hardness and young modulus

During the two last decades, the instrumented indentation test (IIT) has been developed. It allows determining some more mechanical properties of materials than the sole conventional hardness, such as Young's modulus [11,14], the work-hardening coefficient [15–18] and the yield stress [19] as well as the fracture toughness [20]. From IIT measurements leading to a load (P)–indenter displacement (h) curve (Fig. 1), hardness, H, is defined as the ratio between the maximum load P_{max} and the projected contact area A_c:

$$H=\frac{P_{max}}{A_c} \tag{1}$$

By analyzing the unloading part of a load–depth curve obtained by instrumented indentation, Oliver and Pharr [11] used the following expression that relates the slope S at the origin of the unloading curve to the reduced modulus E_R:

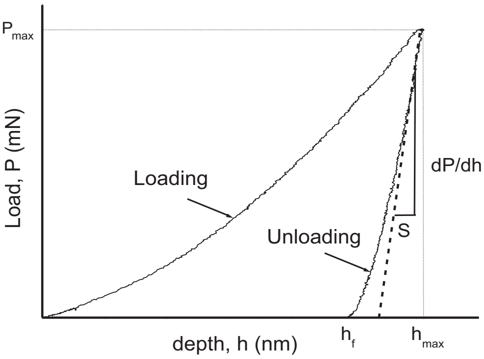

Fig. 1. Schematic load–indenter displacement curve obtained from instrumented indentation test using a Vickers indenter.

$$E_R=\frac{S}{2}\sqrt{\frac{\pi}{A_c}} \tag{2}$$

where E_R includes the material parameters of the indenter (E_i, ν_i) and of the investigated material (E, ν) in the relation:

$$\frac{1}{E_R}=\frac{1-\upsilon^2}{E}+\frac{1-\upsilon_i^2}{E_i} \tag{3}$$

The slope of the curve upon unloading is indicative of the stiffness S of the contact. This value generally is the inverse of the total compliance C_T which includes a contribution from both the compliance of the sample being tested and the load frame compliance of instrument. For the calculation of the slope at the origin of the unloading part of the IIT curve, Oliver and Pharr [11] suggested to fit the curve by a power law relating the indentation load P to the difference between the indentation depth h and the residual indentation depth:

$$P=B(h-h_f)^m \tag{4}$$

where B, m and h_f are values determined by a step by step best fit analysis. In practice, only data in the range 40–98% of the maximum load are used for the fitting.

2.2. The contact area

From Eqs. (1) and (2) it is seen that the projected contact area, A_c, is a key factor in the calculation of mechanical parameters. For a perfect geometry of the indenter, three sides for a pyramid Berkovich indenter and four sides for a pyramid Vickers indenter, the projected contact area is proportional to the square of the contact depth h_C by the following relation:

$$A_C=24.56\,h_c^2 \tag{5}$$

Depending on the deformation and mechanical properties of the tested material, we have mentioned that piling-up or sinking-in may occur during the indentation process. A schematic representation of the two modes is presented in Fig. 2 for a Vickers indenter.

It is clear that both modes of deformation render difficult a precise determination of the penetration depth and consequently of the contact area. Methods have been proposed by various authors. For sinking-in, Oliver and Pharr [11] expressed the contact depth h_C by a function of the maximum indentation depth, h_{max}, the maximum load, P_{max}, and the elastic unloading stiffness, S, as follows:

$$h_{Cs}=h_{max}-\frac{P_{max}}{S} \tag{6}$$