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A method for measuring the contact area in instrumented indentation testing by tip scanning probe microscopy imaging

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Abstract

The determination of the contact area is a key step in deriving mechanical properties such as hardness or an elastic modulus by instrumented indentation testing. Two families of procedures are dedicated to extracting this area: on the one hand, post-mortem measurements that require residual imprint imaging, and on the other hand, direct methods that only rely on the load vs. penetration depth curve. With the development of built-in scanning probe microscopy imaging capabilities such as atomic force microscopy and indentation tip scanning probe microscopy, last-generation indentation devices have made systematic residual imprint imaging much faster and more reliable. In this paper, a new post-mortem method is introduced and further compared to three existing classical direct methods by means of a numerical and experimental benchmark covering a large range of materials. It is shown that the new method systematically leads to lower error levels regardless of the type of material. The pros and cons of the new method vs. direct methods are also discussed, demonstrating its efficiency in easily extracting mechanical properties with enhanced confidence.

Keywords: Nanoindentation; Atomic force microscopy; Hardness; Elastic behavior; Finite-element analysis

1. Introduction

Over the last two decades, the instrumented indentation technique (IIT) has become widely used to probe the mechanical properties of samples of virtually any size or nature. However, the intrinsic heterogeneity of the mechanical fields underneath the indenter prevents the determination of any straightforward relationships between the measured load vs. displacement curve and any expected mechanical properties as would be the case for tensile testing. Many models have been published in the literature in order to enable the measurement of properties such as elastic modulus, hardness or various plastic properties. Despite their diversity, most of these models rely heavily on the accurate measurement of the projected contact area between the indenter and the sample's surface. Existing methods dedicated to estimating the true contact area can be classified into two subcategories: direct methods, which rely on the sole load vs. displacement curve [1–3], and post-mortem methods, which use additional data extracted from the residual imprint left on the sample's surface. For example, Vickers, Brinell and Knoop hardness scales rely on post-mortem measurements of the geometric size of the residual imprint. However, in the case of Vickers hardness,

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the contact area is only estimated through the diagonals of the imprint; the possible effect of piling-up or sinking-in is then neglected. Other post-mortem methods use indent cross-sections to estimate the projected contact area [4,5]. In the 1990s, the development of nanoindentation led to a growing interest in direct methods because they do not require time-consuming post-mortem measurement of micrometer- or even nanometer-scale imprints, typically using atomic force microscopy (AFM) or scanning electron microscopy (SEM). The uncertainty level with direct measurements remains high, mainly because of the difficulty of predicting the occurrence of piling-up and sinking-in. Oliver and Pharr considered this issue as one of the “holy grails” in IIT [2]. Recent developments in scanning probe microscopy (SPM) using indentation tips (ITSPM) have attracted new interest in post-mortem measurements. Indeed, ITSPM allows systematic imprint imaging without manipulating the sample or facing repositioning issues to determine the imprint to be imaged. Nevertheless ITSPM imaging suffers from drawbacks when compared to AFM: it is slower, and it uses a blunter tip associated with a much wider pyramidal geometry and a higher force applied to the surface while scanning. While the latter may damage delicate material surfaces, the former will introduce artifacts. Nonetheless, these artifacts will not affect the present method. In addition, ITSPM only allows for contact mode imaging; non-contact or intermittent contact modes are not possible. As a consequence, only the techniques based on height images can be used with ITSPM and there is a need for new methods as very recently reviewed by Marteau et al. [6]. This paper introduces a new post-mortem procedure that relies only on the height image and is therefore valid for most types of SPM images, including ITSPM. In this paper, a benchmark based on both numerical indentation tests as well as experimental indentation tests on properly chosen materials to span all possible behaviors is first introduced. Then, the existing direct methods are reviewed and a complete description of the proposed method is given. These methods are then confronted using the above-mentioned benchmark and the results are finally discussed.

2. Numerical and experimental benchmark

A typical instrumented indentation test features a loading step where the load P is increased up to a maximum value P_{max}, then held constant in order to detect creep, and finally decreased during the unloading step until contact is lost between the indenter and the sample. A residual imprint is left on the initially flat surface of the sample. During the test, the load P as well as the penetration of the indenter into the surface of the sample h is continuously recorded and can be plotted as shown in Fig. 1. For most materials, the unloading step can be cycled with only minor hysteresis; it is then assumed that only elastic strains develop in the sample. As a consequence, the initial slope S of the unloading step is called the elastic contact stiffness.

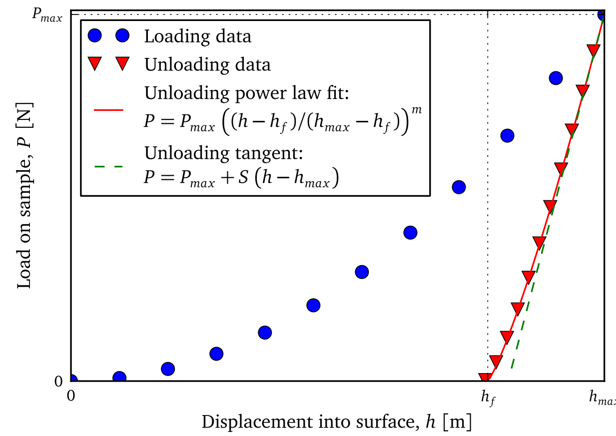

Fig. 1. Typical sharp indentation load on sample vs. displacement into the surface curve. The test is split into a loading step and an unloading step. The experimental curve generally also includes an holding step which is not represented in this case. The contact stiffness S is the unloading step’s initial slope. However, the direct determination of S via the upper part of the step is unreliable as it uses only a small part of the curve. For increased accuracy, the whole step is systematically fitted by a power-law function which is used to compute back the contact stiffness S as initially recommended in Ref. [2].

Useful data can potentially be extracted from both the load vs. displacement curve and the residual imprint. The contact area A_c is defined as the projection of the contact zone between the indenter and the sample at maximum load on the plane of the initially flat surface of the sample.

2.1. Numerical approach

Finite-element modeling (FEM) simulations are performed using a two-dimensional axisymmetrical model represented in Fig. 2. The sample is meshed with 3316 four-noded quadrilateral elements. The indenter is considered as a rigid cone exhibiting an half-angle $\Psi = 70.29°$ to match the theoretical area function of the Vickers and modified Berkovich indenters [7]. The displacement of the indenter h is controlled and the force P is recorded. The dimensions of the mesh are chosen to minimize the effect of the far-field boundary conditions. The typical ratio of the maximum contact radius and the sample size is about 2×10^3. The problem is solved using the commercial software ABAQUS (version 6.11, 3ds.com). The numerical model is compared to the elastic solution from Ref. [8] (see [9,10]) using a blunt conical indenter ($\Psi = 89.5°$) to respect the purely axial contact pressure hypothesis used in the elastic solution. The relative error is computed from the load vs. penetration curve and is below 0.1%. Pre-processing, post-processing and data-storage tasks are performed using a dedicated framework based on the open source programming language Python 2.7 [11–13] and the database engine SQLite 3.7 [14]. The indented material is assumed to be isotropic, linearly elastic. The Poisson’s ratio ν has a fixed value of 0.3 and the Young’s modulus is referred to as E. The contact between the indenter and