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Indenter Shape Characterization for the Nanoindentation Measurement of Nanostructured and Other Types of Materials

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Abstract

A review and comparison of different techniques for indenter shape characterization have been carried out. The techniques considered in this study are: a series of indentations in a reference material with known mechanical properties, a direct AFM observation of indenter apex and SPM imaging of calibration gratings supported with laser interferometer. All of the described techniques are analyzed in terms of the factors affecting the indenter shape characterization and the efficiency of obtaining the resulting indenter area function.

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1. Introduction

Pushing a hard tip of a known shape (an indenter) into the material is the most common method for the material hardness measurement. Depending on the measurement technique, the indenters of various shapes can be applied. In macroscopic tests the hardness value is determined over the residual imprint size or the indenter penetration depth

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into the surface.

However, this method has some shortcomings. Firstly, according to the indentation method in order to get hardness, one has to determine the residual imprint area. Traditionally, that was done by optic measurement, which may not give enough resolution for low-depth indentation in nano-range. On the other hand, determination of this area by means of atomic force or electron microscopy takes a lot of time. Secondly, this method gives no information about the elastic modulus of the sample.

These drawbacks were overcame in the the instrumented indentation testing (IIT),which is also often referred to as nanoindentation. Has been increasingly applied to determine the mechanical properties of materials at the submicron and nanometer length scales. The basics of this method were developed in 1960-70 in the USSR [1, 2]. Over the next several decades this techniques was intensively developed in terms of new equipment and mathematical models describing the interaction between the indenter and the material during the indentation [3-5]. The paper of Oliver and Farr [6], proposing the most consistent analysis, made the technique a widespread one. Since year 2002 instrumented indentation test is a basis for the international standard for mechanical testing ISO 14577 [7].

Unlike the traditional methods in nanoindentation hardness value is estimated by a mathematical processing of the applied load dependency on the indenter penetration depth. The key requirement of this method is that the shape of the indenter pushed in the material should be precisely characterized. The indenter shape is defined by the so-called area function *A(h)* which represents the dependence of the cross-sectional (projected) area A_p or surface area A_s on a distance *h* along the indenter axis.

Even though nanoindentaion method is developed, its application meets its own source of errors. One or even major of these problems is determination of the indentor shape are [8] that is why searching the way for the most accurate and rapid tip shape determination is one of the main tasks in further improvement of metrological assurance of instrumented indentation test.

In the present experimental study the comparison of different techniques for indenter shape characterization is presented and analyzed.

2. Hardness testing method and instrument

Instrumented indentation test consists in uniaxial pushing the solid indenter (typically, the diamond pyramid) into the sample surface until the required maximum load or penetration depth is reached with subsequent pulling it backwards. During this test, the load value and the corresponding indenter displacement are recorded. The resulting functional dependency *P(h)* is called a loading/unloading curve. All calculations of mechanical properties are made using this *P(h)* graph. The sample hardness *H* is determined by the following solution [6, 9]:

$$H = \frac{P_{\max}}{A_c} \quad (1)$$

Here A_c is an- indentation contact area at a maximum applied load P_{max}. Typically contact area is taken from so called area function which relates area value and contact depth specific for the particular indentation test:

$$A_c = f(h_c) \quad (2)$$

The ISO 14577 standard [7] refers to different hardness values, depending on which area A_c is used in equation (1). For calculation of indentation hardness the projected are (A_p) is used, while for calculation of Martens hardness the surface area (A_s) is used.

Nanoindentation experiments and SPM imaging results presented in this paper were obtained using NanoScan-3D scanning nanohardness tester [10, 11]. This instrument uses a patented sensor which can operate in two modes: a dynamic resonant mode and a static bending mode. Dynamic mode is used for SPM imaging of surface topography and static mode is used for indentation, scratch and wears testing. In the presented experiments