FUNDAMENTAL RELATIONS FOR FRICTIONAL AND ADHESIVE NANOINDENTATION TESTS

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Summary Fundamental relations for depth-sensing nanoindentation are derived for indenters of various shapes and for various boundary conditions within the contact region. In particular, it is shown that some uncertainties in nanoindentation measurements, which are sometimes attributed to properties of the material, can be explained and quantitatively described by properly accounting for geometric deviation of the indenter tip from its nominal geometry.

Indentation testing is widely used for analysis and estimations of mechanical properties of materials. Evidently, the load-displacement diagram at loading reflects both elastic and plastic deformations of the material, while the unloading is taking place elastically. The Bulychev-Alekhin-Shorshorov (BASh) equation for the stiffness S of the upper portion of the load-displacement curve at unloading is the following [4]:

$$S = \frac{dP}{dh} = \frac{2\sqrt{A}}{\sqrt{\pi}} E^*. \tag{1}$$

Here a is the radius of the contact region, A is the contact area ($A = \pi a^2$), P is the external load, h is the indentation depth of the indenter tip, and E^* is the reduced Young's modulus. Thus, nanoindentation techniques provide a unique opportunity to obtain mechanical properties of materials of very small volumes. The BASh relation is an example of fundamental relations which can be obtained from the analysis of frictionless contact problems.

The estimations of the thin film mechanical properties can be affected by various factors. It is widely accepted that the most significant source of uncertainty in nanoindentation measurement is the deviation of the indenter tip from nominal geometry. First, we consider the loading branch. It is assumed that the stress-strain behavior of materials can be described as a homogeneous (power-law) relation with the work-hardening exponent κ. It is argued that for shallow indentation where the tip bluntness is on the same order as the indentation depth, the indenter shapes can be well approximated by non-axisymmetric monomial functions of radius. However, if the stress-strain relation of the coating is $\sigma \sim \epsilon^\kappa$ and the indenter shape is described by a homogeneous function of degree d then the problem is self-similar. Let P_1, A_1, l_1 and h_1 be respectively some initial load, the corresponding contact area, the characteristic size of the contact region and the displacement. Using similarity rescaling formulae for 3D frictional contact problems [1], the fundamental relations are derived for depth of indentation, size of the contact region, load, hardness, and contact area, which are valid for both linear and non-linear, isotropic and anisotropic materials [2]:

$$\frac{l}{l_1} = \left(\frac{P}{P_1}\right)^{\frac{1}{2+\kappa(d-1)}}, \quad \frac{h}{h_1} = \left(\frac{P}{P_1}\right)^{\frac{d}{2+\kappa(d-1)}} \tag{2}$$

and the rescaling formula for the contact area is

$$\frac{A}{A_1} = \left(\frac{h}{h_1}\right)^{\frac{2}{d}}. \tag{3}$$

The formulae depend on the material hardening exponent κ and the degree of the monomial function of the shape d. However, it follows from (3) that $h \sim A^{d/2}$ independently of the work hardening exponent κ. Hence, one can calibrate the indenter tip from area-displacement curve. For example, one can obtain that the indenter shape used by Doerner and Nix [5] for $h \leq 90$nm can be described as a monomial function of degree $d = 1.44$. The formulae (2) and (3) are especially important for shallow indentation (usually less than 100 nm) where the tip bluntness is on the same order as the indentation depth. Further, the hardness (the average pressure) is the following function of the depth of indentation

$$\frac{H}{H_1} = \left(\frac{h}{h_1}\right)^{\frac{\kappa(d-1)}{d}}.$$

Hence, H is constant only for $d = 1$. Thus, the non-ideal indenter geometries can affect the interpretation of the experimental results, in particular the apparent hardness.

On the unloading branch, it is assumed that the materials are isotropic and linear elastic. The authors developed the Mossakovskii approach to the adhesive problem [7] and derived a relation that for evaluation of elastic modulus of materials by the slope of the unloading branch assuming adhesive (no-slip) contact [3]:

$$S = \frac{dP}{dh} = C\frac{2E}{1-\nu^2}\frac{\sqrt{A}}{\sqrt{\pi}}. \tag{4}$$

Thus, the BASh relation (1) should be corrected by the factor C in the case of frictional contact, where in the case of adhesive (no-slip) contact

$$C = \frac{(1-\nu)\ln(3-4\nu)}{1-2\nu}. \tag{5}$$

This factor decreases from $C = \ln 3 = 1.0986$ at $\nu = 0$ and takes its minimum $C = 1$ at $\nu = 0.5$. Taking into account that full adhesion preventing any slip within the contact region is not the case for real physical contact and there is some frictional slip at the edge of the contact region, one can conclude that the values of the correction factor C in (4) cannot exceed the upper bound (5). The relation is analogous to the BASh relation and similarly to the frictionless analysis [8], the obtained relation is independent of the geometry of the indenter.

Considering frictionless contact between an isotropic, linear elastic material and an arbitrary axisymmetric indenter of a monomial shape

$$f(\rho) = B_d \rho^d, \tag{6}$$

Galin [6] derived the following formulae

$$P = \frac{E}{1-\nu^2} B_d \frac{d^2}{d+1} 2^{d-1} \frac{[\Gamma(d/2)]^2}{\Gamma(d)} a^{d+1}, \quad h = B_d d 2^{d-2} \frac{[\Gamma(d/2)]^2}{\Gamma(d)} a^d. \tag{7}$$

Using (7), he established the following relation between the force P and the displacement h

$$P = \frac{E}{1-\nu^2} \left[B_d^{-\frac{1}{d}} 2^{2/d} d^{\frac{d-1}{d}} \frac{1}{d+1} [\Gamma(d/2)]^{-\frac{2}{d}} [\Gamma(d)]^{\frac{1}{d}} \right] h^{\frac{d+1}{d}}. \tag{8}$$

Developing the Mossakovskii approach [7], we obtained the following exact relations among the force P, the contact radius a and the displacement h:

$$P = \frac{E\ln(3-4\nu)}{(1+\nu)(1-2\nu)} B_d \frac{d}{d+1} 2^{d-1} \frac{[\Gamma(d/2)]^2}{\Gamma(d)} \frac{1}{I^*(d)} a^{d+1}, \quad h = B_d 2^{d-2} \frac{[\Gamma(d/2)]^2}{\Gamma(d)} \frac{1}{I^*(d)} a^d. \tag{9}$$

Using (9), one can establish the following relation between the force P and the displacement h for a monomial punch in the case of adhesive contact

$$P = \frac{E\ln(3-4\nu)}{(1+\nu)(1-2\nu)} \frac{d}{d+1} \left[\frac{4I^*(d)}{B_d} \frac{\Gamma(d)}{[\Gamma(d/2)]^2} \right]^{1/d} h^{\frac{d+1}{d}}, \quad \beta = \frac{1}{2\pi}\ln(3-4\nu), \quad I^*(d) = \int_0^1 t^{d-1} \cos\left(\beta \ln\frac{1-t}{1+t}\right) dt. \tag{10}$$

Here $\Gamma(d)$ is the Euler gamma function. In the case $\nu = 0.5$, the formulae (9) and (10) are identical with the corresponding formulae (7) and (8) obtained for frictionless contact. From (9) and (10), one can obtain the Mossakovskii solution [7] for a sphere ($d = 2$) and the Spence solution [9] for a cone ($d = 1$).

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