Elastic modulus determination from depth sensing indentation testing

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Depth sensing indentation (DSI) testing is generally considered as a simple method for the determination of the Young's modulus of materials [1–5]. Methods found in the literature for the calculation of Young's modulus from indentation tests are based in most cases on the theoretical solution of the Boussinesq problem given by Sneddon [6] who determined the load–indentation depth functions for various indenters in an elastic half space. The most frequently used method of Young's modulus determination was developed by Oliver and Pharr [3]. Using Sneddon's results, they elaborated an iterative procedure for the determination of the reduced Young's modulus, E_r ($E_r = (E/1 - v^2)$, where E is the Young's modulus and v is the Poisson's ratio). The uncertainty of both parameters is relatively high and the procedure is complicated.

This letter is a continuation of a recently published work [7] in which a semi-empirical formula is proposed for the determination of the Young's modulus of materials from depth sensing Vickers indentation testing. In the following, on the basis of our previous results, this formula is verified by calculating the elastic energy associated with the stress fields building up beneath the indenter during Vickers indentation.

Our indentation measurements were carried out in the macrohardness region ($P_m = 100$ N) on the following materials: metals (steel, 99.999% pure Al, Cu, Mg, Ni), soda lime silica glass, sodium chloride, plastic (polypropylene), apatite–mullite glass–ceramic, silicon, titanium oxide ceramic, tetragonal zirconia polycrystalline ceramic containing 10 mol% CeO_2 (Ce TZP) and S_3N_4 ceramics with three different compositions (x_1 wt%Si_3N_4 x_2 wt%AlN x_3 wt%Al_2O_3 x_4 wt%Y_2O_3; for material A: $x_1 = 90$, $x_2 = 0$, $x_3 = 4$, $x_4 = 6$; for material B: $x_1 = 87$, $x_2 = 4$, $x_3 = 4$, $x_4 = 5$; for material C: $x_1 = 90.9$, $x_2 = 0$, $x_3 = 3$, $x_4 = 6.1$) sintered to different densities.

During the loading period of the test the Vickers pyramid penetrates the sample at constant velocity and the same velocity is applied in the unloading period when the pyramid moves backwards. The load–penetration depth function can be described by quadratic polynomials:

$$P = c_2 h + c_3 h^2 \tag{1}$$

$$P = c_2^*(h - h_0) + c_3^*(h - h_0)^2 \tag{2}$$

both in the loading and in the unloading periods, respectively, where P is the load, h is the penetration depth, h_0 is the residual indentation depth after removing the punch and c_2, c_3, c_2^*, c_3^* are fitting parameters. The total indentation work (W_t) performed during loading and the elastic work (W_e) regained during unloading can be calculated by the integration of Equation 1 and 2, respectively (Fig. 1).

It was shown in our previous paper [7] that despite the linear terms appearing in Equations 1 and 2, for all of the materials investigated and in a wide load range, the following relationships are valid with good accuracy:

$$W_t = \frac{1}{3(c_3)^{1/2}} P_m^{3/2} \tag{3}$$

$$W_e = \frac{1}{3(c_3^*)^{1/2}} P_m^{3/2} \tag{4}$$

$$\frac{W_e}{W_t} = \left(\frac{c_3}{c_3^*}\right)^{1/2} \tag{5}$$

According to our previous results, a semi-empirical relationship has been found between the parameters of the load–depth curves and the Young's modulus

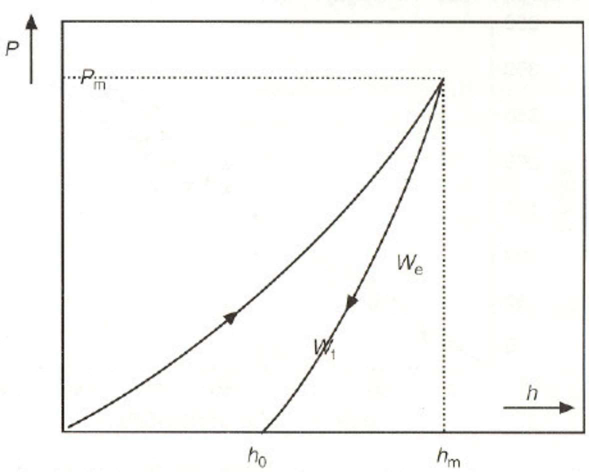

Figure 1 Schematic diagram of an indentation cycle.

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of the material investigated:

$$E = 0.71(1 - v^2)c_3 \frac{W_t}{W_e} \tag{6}$$

Fig. 2 shows that the E values determined from DSI measurements with the help of Equation 6 agree well within the experimental errors with the Young's moduli measured by four-point bending tests. There are several models in the literature for describing the elastic stress field in materials around axisymmetric indenters. The most widely used one was developed by Yoffe [8]. This model is a superposed combination of the Boussinesq field and the "blister" field. In spherical polar coordinates (r, θ, ϕ) the elastic stress components are given by:

$$\left.\begin{aligned}
\sigma_{rr} &= \frac{P}{2\pi r^2}[1 - 2v - 2(2 - v)\cos\theta] \\
&\quad + \frac{B}{r^3}4[(5 - v)\cos^2\theta - (2 - v)] \\
\sigma_{\theta\theta} &= \frac{P}{2\pi r^2}\frac{(1 - 2v)\cos^2\theta}{(1 + \cos\theta)} + \frac{-B}{r^3}2(1 - 2v)\cos^2\theta \\
\sigma_{\phi\phi} &= \frac{P(1 - 2v)}{2\pi r^2}\left[\cos\theta - \frac{1}{1 + \cos\theta}\right] \\
&\quad + \frac{B}{r^3}2(1 - 2v)(2 - 3\cos^2\theta) \\
\tau_{r\theta} &= \frac{P(1 - 2v)}{2\pi r^2}\frac{\sin\theta\cos\theta}{(1 + \cos\theta)} \\
&\quad + \frac{B}{r^3}4(1 + v)\sin\theta\cos\theta
\end{aligned}\right\} \tag{7}$$

where B is a constant, a measure of the strength of the "blister" field. As a consequence of the high stresses at the tip of the sharp indenter, a plastic zone develops under the punch where Equations 7 are not valid. The shape of this zone changes with the mechanical properties of the material investigated and also with the type of the indenter applied but it is roughly hemispherical, with a diameter

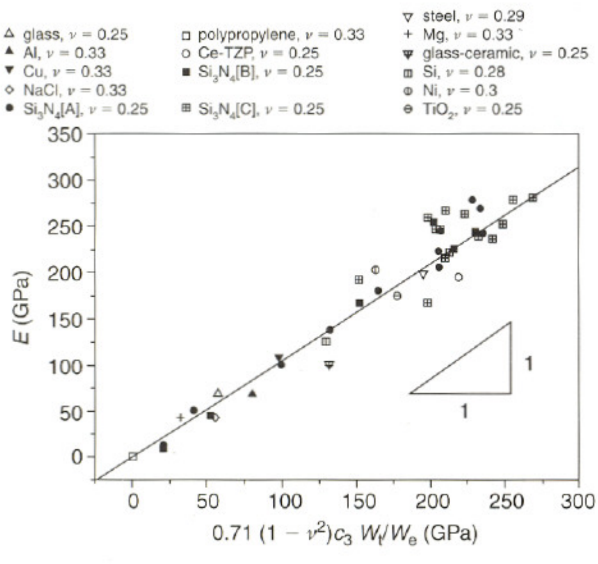

Figure 2 Relationship between Young's moduli determined by four-point bending test and those calculated on the basis of Equation 6.

equal to that of the contact impression [8–11]. In the following the plastic zone is considered as a hemispherical hydrostatic core with a radius of a, the half diagonal of the Vickers pattern. In this hydrostatic core the pressure is taken as the applied mean contact pressure beneath the pyramid:

$$H = \frac{P_m}{2a^2} \tag{8}$$

where P_m is the maximum applied load during the indentation cycle.

The normal (σ_{rr}) and shear ($\tau_{r\theta}$) components of the stress field must be continuous at the zone boundary, and B can be determined in Equation 7 from these conditions. A single value of B can not satisfy these conditions exactly; however, $0.019\,Ha^3$ is a good approximation for the value of parameter B because the difference between the stress components calculated for both sides of the interface of the hemispherical zone is minimum for this value.

The elastic strain energy can be calculated with the help of the above stress field. In the hydrostatic zone the energy per unit volume is:

$$w_e^c = (1 - 2v)\cdot\frac{3H^2}{2E} \tag{9}$$

if the variation of the elastic modulus due to compaction can be neglected. The volume of the zone can be obtained by subtracting the volume of the indenter tip from that of the hemisphere with radius a:

$$V = 1.9 \cdot a^3 \tag{10}$$

Using Equations 9 and 10 the elastic energy in the hydrostatic core can be given by:

$$W_e^c = 2.85(1 - 2v)\cdot\frac{H^2a^3}{E} \tag{11}$$

The strain energy of the surrounding region ($r > a$) can be obtained as follows:

$$W_e^m = -\tfrac{1}{2}\int(u_r\sigma_{rr} + u_\theta\tau_{r\theta})dS \tag{12}$$

where u_r and u_θ are the elastic displacements, and S is the hemispherical surface of radius a. Taking into account that the "blister" components of the stress field are retained after unloading, the elastic energy of the surrounding region can be written as:

$$W_e^m = f(v)\cdot\frac{H^2a^3}{E} \tag{13}$$

where $f(v)$ depends on Poisson's ratio (v) and $f(v) = 0.81$, 0.815 or 0.82 as $v = 0.25$, 0.29 or 0.33.

Adding the energy of Equation 11 to Equation 13, the elastic energy regained during unloading (W_e) is obtained as:

$$W_e = g(v)\cdot\frac{H^2a^3}{E} \tag{14}$$

where $g(v) = 2.24$, 2.01 or 1.77 as $v = 0.25$, 0.29 or 0.33.

Using Equations 3 and 8 the elastic energy can be expressed with E, H and the parameters of the indentation curves:

$$W_{\mathrm{e}} = 1.06 \cdot g(v) \frac{W_{\mathrm{t}} c_3^{1/2} H^{1/2}}{E} \quad (15)$$

It can be shown (see the Appendix) that the mean contact pressure, H, can be expressed in the following form:

$$H = \alpha_1 c_3 \frac{1}{\left((k)^{1/2} - \dfrac{\varepsilon}{2}\dfrac{W_{\mathrm{e}}}{W_{\mathrm{t}}}\right)^2} \quad (16)$$

For the meaning of k and ε see the Appendix.

Rearranging Equation 15 and using Equation 16, the following relationship is obtained for Young's modulus:

$$E = 0.214 g(v) \frac{1}{(k)^{1/2} - \dfrac{\varepsilon}{2}\dfrac{W_{\mathrm{e}}}{W_{\mathrm{t}}}} c_3 \frac{W_{\mathrm{t}}}{W_{\mathrm{e}}} \quad (17)$$

Fig. 3 shows that the Young's moduli determined from our previous semi-empirical formula (Equation 6) agree well with those obtained from the elastic energy (Equation 17) for all the materials investigated. Consequently, the theoretical considerations described above verify the application of the formula of Equation 6 for the determination of the Young's modulus. This formula is simpler than Equation 17 determined on the basis of energetical considerations.

Appendix

According to Oliver and Pharr [3] the mean contact pressure (H) can be given by the following expression:

$$H = \alpha_1 \frac{P_{\mathrm{m}}}{h_{\mathrm{c}}^2} \quad (\mathrm{A1})$$

where h_{c} is the contact depth at the maximum load and $\alpha_1 = 0.0408$.

Using Sneddon's elastic theory [6] and the empirical results of Oliver and Pharr [3], the contact depth can be obtained as:

$$h_{\mathrm{c}} = h_{\mathrm{m}} - \varepsilon \frac{P_{\mathrm{m}}}{S} \quad (\mathrm{A2})$$

where S is the slope of the initial part of the unloading curve and $\varepsilon = 0.75$ for the case of Vickers indenter. In another work [12] it has been shown that the mean contact pressure can be expressed with the indentation parameters in the following way.

According to Equation 2, S can be given as:

$$S = \left.\frac{\mathrm{d}P}{\mathrm{d}h}\right|_{h_{\max}} = c^*{}_2 + 2c^*{}_3(h_{\mathrm{m}} - h_0). \quad (\mathrm{A3})$$

The second term in Equation A3 may be expressed as a fraction of S:

$$\bar{k}S = 2c^*{}_3(h_{\mathrm{m}} - h_0) \quad (\mathrm{A4})$$

and, similarly, the quadratic term of the load–depth function of the unloading curve as a fraction of the maximum load:

$$k^* P_{\mathrm{m}} = c^*{}_3(h_{\mathrm{m}} - h_0)^2 \quad (\mathrm{A5})$$

With Equations A4 and A5, P_{m}/S can be given as:

$$\frac{P_{\mathrm{m}}}{S} = \frac{k^*}{\bar{k}} \frac{h_{\mathrm{m}} - h_0}{2} \quad (\mathrm{A6})$$

From Equations 2 and A2–A6, h_{c} can be given in the following form:

$$h_{\mathrm{c}} = h_{\mathrm{m}} - \frac{2}{1 + k^*} \frac{\varepsilon}{2} (h_{\mathrm{m}} - h_0) \quad (\mathrm{A7})$$

To simplify Equation A7 the quadratic term in Equation 1 is expressed as a fraction of the maximum load:

$$kP_{\mathrm{m}} = c_3 h_{\mathrm{m}}{}^2 \quad (\mathrm{A8})$$

With the help of Equations A5, A7 and A8, the H mean contact pressure in Equation A1 can be expressed as follows:

$$H = \alpha_1 \frac{c_3 h_{\mathrm{m}}{}^2}{k\left(1 - \dfrac{\varepsilon}{2}\dfrac{2(k^*)^{1/2}}{1 + k^*}\dfrac{1}{(k)^{1/2}}\left(\dfrac{c_3}{c_3{}^*}\right)^{1/2}\right)^2 \cdot h_{\mathrm{m}}{}^2}$$

$$= \alpha_1 c_3 \frac{1}{\left((\mathrm{k})^{1/2} - \dfrac{2(k^*)^{1/2}}{1 + k^*}\dfrac{\varepsilon}{2}\left(\dfrac{c_3}{c_3{}^*}\right)^{1/2}\right)^2} \quad (\mathrm{A9})$$

According to our measurements for the different materials and loadings investigated, k and k^* vary between 0.64 and 1. If $k^* \geqslant 0.64$ then $(2(\mathrm{k}^*)^{1/2}/1 + k^*) \geqslant 0.98$, therefore this quantity can be taken as 1. Using Equation 5 the following relationship is obtained for H:

$$H = \alpha_1 c_3 \frac{1}{\left((\mathrm{k})^{1/2} - \dfrac{\varepsilon}{2}\dfrac{W_{\mathrm{e}}}{W_{\mathrm{t}}}\right)^2} \quad (\mathrm{A10})$$

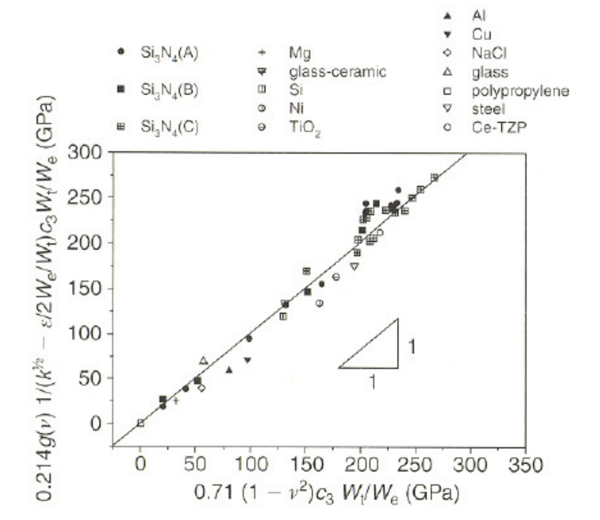

Figure 3 The Young's moduli calculated from Equation 6 compared to the moduli determined on the basis of Equation 17.

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