MEASURING THE ELASTIC PROPERTIES OF ANISOTROPIC MATERIALS BY MEANS OF INDENTATION EXPERIMENTS

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ABSTRACT

The unloading process in an indentation experiment is usually modeled by considering the contact of a rigid punch with an elastically isotropic half space. Here we extend the analysis to elastically anisotropic solids. We review some of the basic formulae for describing the indentation of elastically anisotropic solids with axisymmetric indenters. We show how the indentation modulus can be calculated for arbitrary anisotropic solids and give results for solids with cubic crystal symmetry. We have calculated the contact stiffness for a flat triangular punch on a half space for various anisotropic materials. The indentation modulus for a triangular indenter is typically 5–6% higher than that for an axisymmetric indenter and varies only slightly with the orientation of the indenter in the plane of the indentation. We have conducted microindentation experiments to measure the indentation moduli of differently oriented surfaces of both cubic and hexagonal single crystals. For copper and B-brass, the (111) indentation moduli are approximately 10 and 25% larger than the (100) modulus. The (110) moduli are typically slightly smaller than the (111) moduli. The indentation modulus of zinc varies by as much as a factor of two, depending on the sample orientation. The hardnesses of the single crystals do not vary much with the orientation of the plane of indentation. For B-brass, the hardness of a (110) surface is only about 13% lower than the hardness of a (100) or (111) surface; for copper, the (110) hardness is 6% higher than for the other orientations. For zinc the maximum change in hardness with orientation is 20%.

1. INTRODUCTION

Indentation experiments have long been used to measure the hardness of materials. Interest in the indentation test as a means of measuring elastic properties has grown recently with the development of very low-load, depth-sensing indentation instruments. These instruments allow one to make indentations as shallow as a few nanometers. The microindentation test is, therefore, particularly well-suited for the in situ measurement of mechanical properties of small volumes of material. Examples include thin films used for microelectronic devices and for magnetic data storage, thin hard coatings, fine-grained ceramics and fibers and interfaces in composite materials.

In a typical indentation test, an indenter is driven into the material of interest at a constant loading or displacement rate until a certain load or depth has been reached. After a short hold period, the load is gradually removed from the indenter. During this process, both load and displacement are recorded continuously. The displacements recovered during unloading are elastic for most materials. Tabor (1948) and Stilwell...
and Tabor (1961) have shown that for spherical and conical indenters, the recovery of the indentation during unloading can be calculated from the theory of elasticity. Measurements of elastic moduli by means of microindentation are based on this observation. For these measurements, the unloading process is modeled by considering the contact of an indenter with an elastic half space (Doerner and Nix, 1986). It has been shown recently by Pharr et al. (1992) that for axisymmetric indenters and elastically isotropic half spaces, the contact compliance is independent of the shape of the indenter tip and is given by

$$ C = \frac{dh}{dL} = \frac{\sqrt{\pi}}{2} \frac{1}{E_i} \frac{1}{\sqrt{A}}, \quad (1) $$

where

$$ \frac{1}{E_i} = \left( \frac{1 - \nu^2}{E} \right)_{\text{indenter}} + \left( \frac{1 - \nu^2}{E} \right)_{\text{sample}} $$

and where $L$ and $h$ are the applied load and the indenter displacement, respectively. Here $A$ is the projected contact area between the indenter and the material.

The contact compliance can be measured as the reciprocal of the slope of the load–displacement curve for an indentation at any one point on the unloading curve (see Fig. 1). The indentation modulus, $E/(1 - \nu^3)$, can then be calculated from (1), provided one knows the contact area $A$. A number of models have been developed to determine this contact area from the unloading curve. The first and most widely used model was given by Doerner and Nix (1986). In this treatment, it is assumed that during initial unloading, the unloading process can be modeled as the contact of a rigid flat punch on an elastic half space. This means that, at least initially, the contact area between the indenter and the material remains constant and that the unloading curve is linear.

![Diagram](image)

**Fig. 1.** Typical load–displacement plot for an indentation.
The elastic deflection of the surface of the sample, $h_{el}$, can be found by extrapolating the tangent of the initial portion of the unloading curve to zero load (see Fig. 1). The depth, $h_c$, over which the indenter makes contact with the sample can be calculated as the difference of the maximum displacement, $h_{max}$, and the surface displacement, $h_{el}$, $h_c = h_{max} - h_{el}$. Given the exact geometry of the indenter, the contact area can be determined from $h_c$. In a more recent treatment by Oliver and Pharr (1992) the unloading process is modeled as the contact of a paraboloid of revolution with an elastic half space and $h_c$ is given by

$$h_c = h_{max} - \varepsilon \frac{L_{max}}{S_{max}}, \quad (2)$$

where $\varepsilon$ is equal to 3/4. If one assumes that the unloading process can be modeled as the contact of a cone with a flat elastic half space, $\varepsilon$ is approximately equal to 0.72.

The compliance one measures in an indentation experiment is not just the contact compliance, but rather the sum of the contact compliance and the machine compliance, i.e. the compliance of the sample support and that of the indenter column. Because the contact compliance varies linearly with $1/\sqrt{A}$ while the machine compliance is independent of $A$, the two contributions to the compliance can be separated by plotting the total compliance versus $1/\sqrt{A}$. The intercept in such a straight line plot is the machine compliance while the slope of the line is, according to (1), inversely proportional to the indentation modulus.

The indentation process is well understood for isotropic materials. Micro-indentation experiments, however, are often performed on single grains or on textured or single crystal epitaxial thin films. Until now little attention has been paid to the fact that these materials are elastically anisotropic. At most, it has been assumed that for anisotropic materials the elastic modulus given in (1) is some polycrystalline average of the elastic constants (Farthing et al., 1989) or corresponds to the elastic modulus in the direction of the indentation (Cammarata et al., 1990; Doerner, 1987). In this paper we first review some of the basic formulae that have been derived for the indentation of elastically anisotropic solids with axisymmetric indenters (Vlassak and Nix, 1993; Willis, 1966). We show how the indentation modulus can be calculated for arbitrary anisotropic solids and give results for solids with cubic crystal symmetry. Since the indenter has a triangular, rather than a circular, cross section in most microindentation experiments, we have also calculated the contact stiffness for a flat triangular punch and a half space for various anisotropic materials. To complete our investigation and to check the various theoretical predictions, we have measured the indentation moduli of differently oriented surfaces of both cubic and hexagonal single crystals using a Nanoindenter®.

2. THE INDENTATION MODULUS FOR AN ELASTICALLY ANISOTROPIC HALF SPACE

The indentation modulus for an isotropic material is defined by (1) and is equal to $E/(1 - \nu^2)$, independent of the shape of the indenter tip. For anisotropic materials it
is not \textit{a priori} clear how the indentation modulus should be related to the elastic constants. In order to define an indentation modulus for anisotropic materials, it is therefore necessary to derive an equation for the contact compliance in terms of the contact area $A$ and the elastic constants of the solid, similar to (1). It is obvious that for anisotropic materials the indentation modulus is some average of the elastic constants, but this average is not necessarily the same for different indenter tip shapes. In general, one can expect the indentation modulus for an anisotropic solid to depend on the shape of the indenter tip, even for axisymmetric indenters.

Formulae for the contact compliances for a flat punch and a paraboloid indenter on an anisotropic half space have been derived by Vlassak and Nix (1993). They have the same form as (1):

$$ C = \frac{dh}{dL} = \frac{\sqrt{\pi}}{2} \frac{1}{M} \frac{1}{\sqrt{A}} \quad (3) $$

where

$$ \frac{1}{M} = \left( \frac{1-v^2}{E} \right)_{\text{indenter}} + \left( \frac{1}{M_{\text{sample}}} \right). \quad (4) $$

Here $M$ is the indentation modulus of the material. The indentation modulus now depends on the indenter geometry and is given by

$$ M = \left( \frac{1}{2} \int_{\Omega_{\text{r}}} W(\xi) \, d\Omega \right)^{-1} \quad (5) $$

for a circular flat punch and by

$$ M = \left( \frac{1}{2} \int_{\Omega_{\text{r}}} W(\xi_1, \xi_2) \, d\Omega \right)^{-1} \quad (6) $$

for an axisymmetric paraboloid indenter. In these expressions $W(\xi_1, \xi_2)$ is the Fourier transform of the normal displacement of the surface of the half space under influence of a point load applied perpendicular to the surface. The integration is performed along a circle around the point load. For a paraboloid indenter the contact area has an elliptical shape and $\delta_1$ and $\delta_2$ are constants proportional to the main axes of the ellipse, such that $\delta_1 \cdot \delta_2 = 1$. The transform $W(\xi_1, \xi_2)$ is a complicated function of the elastic constants; an analytical expression is available only in some special cases, such as for transversely isotropic solids (Willis, 1966; Gladwell, 1980). With some effort, a numerical calculation of $W(\xi_1, \xi_2)$ is possible for solids with less symmetry (Vlassak and Nix, 1993). We will discuss a different approach to calculating the indentation modulus in a later paragraph.

Although (5) and (6) are not very useful for calculating the indentation modulus of a general anisotropic solid, they can be used to prove some interesting properties of the indentation modulus. If the material has a three or four-fold rotation axis perpendicular to the surface of the half space, the area of contact between the indenter and the half space is circular. In that case, $\delta_1 = \delta_2 = 1$ and (5) and (6) are identical.
The indentation modulus is then independent of whether one uses a flat punch or a paraboloid indenter. Thus, one can use either a flat punch or a paraboloid to model the contact between the indenter and the material; the same indentation modulus will be obtained, provided the correct contact area is used. The methods for calculating the contact area between indenter and material, developed for isotropic materials by Doerner and Nix (1986) and by Oliver and Pharr (1992), can still be used, with \( \varepsilon \) taking the values of 1 and 3/4, respectively (Vlassak and Nix, 1993). The \{100\} and \{111\} surfaces of solids with cubic symmetry are examples where the rotational symmetry simplifies the situation. If the half space has lower symmetry, the contact between the half space and a paraboloid indenter is elliptical rather than circular. The indentation modulus for a paraboloid indenter then differs from that for a flat punch. In order to calculate the indentation modulus for a paraboloid indenter, one has to first determine the constants \( \delta_1 \) and \( \delta_2 \). However, this is no easy task (Willis, 1966). Thus, in this study we use the flat punch model to calculate indentation moduli. This choice makes no difference for samples with high symmetry and only a small difference for other samples. In order to determine the contact area between the indenter and the sample, either the Doerner–Nix method or the Oliver–Pharr method can be used. The value of \( \varepsilon \) in the latter treatment is now given by

\[
\varepsilon = \frac{3}{2} \left[ 1 - \frac{\int_{|\xi|=1} W \left( \frac{\xi_1}{\delta_1}, \frac{\xi_2}{\delta_2} \right) \left( \frac{\xi_1}{\delta_1} \right)^2 \, ds}{\int_{|\xi|=1} W \left( \frac{\xi_1}{\delta_1}, \frac{\xi_2}{\delta_2} \right) \, ds} \right],
\]

(7)

Since for most anisotropic solids the values of \( \delta_1 \) and \( \delta_2 \) are close to one, \( \varepsilon \) is still approximately equal to 3/4.

As mentioned previously, the Fourier transform \( W(\xi_1, \xi_2) \) is not readily calculated for an arbitrary anisotropic solid. A more direct method for calculating the indentation modulus makes use of the displacement field of the surface of an anisotropic half space subjected to a unit point load applied at the surface. An expression for this field has been derived by Barnett and Lothe (1975) using a technique based on the Stroh formalism and is given in the Appendix for the convenience of the reader. Let \((x_1, x_2, x_3)\) be a coordinate system with the origin in the boundary of the half space, but with arbitrary orientation with respect to the half space. Assume a rigid, flat punch with a circular cross section of radius \( a \) is pressed against the half space imposing a constant displacement \( \delta \) under the punch. The center of the punch coincides with the origin of the coordinate system. Let \( L \) be the total load on the punch and \((\alpha_1, \alpha_2, \alpha_3)\) the direction cosines of the normal to the surface of the half space. It has been shown in our previous work (Vlassak and Nix, 1993) that the pressure distribution under the indenter is given by

\[
p = \frac{L}{2\pi a} \frac{1}{\sqrt{a^2 - |y|^2}} \quad |y| < a
\]

(8)

where \((y_1, y_2, y_3)\) is a position vector in the boundary of the half space. The displacement at \( y \) can then be written as
where \( y' \) is the vector \((y'_1, y'_2, y'_3)\) in the boundary of the half space and \( w(y) \) is the vertical displacement of the surface of the half space due to a unit point load applied at the origin and perpendicular to the boundary of the half space. Here \( S \) is the area over which the punch makes contact with the half space. Using the expression derived by Barnett and Lothe leads, after some manipulations (see Appendix), to the following expression for \( \delta \):

\[
\delta = \frac{L}{32\pi a} \int_0^{2\pi} \int_0^\pi x_m B_{im}^{-1} (\gamma) z_i \, d\gamma.
\]

where the repeated indices imply a summation over the repeated index from 1 to 3. In this equation, \( B_{im}^{-1} (\gamma) \) is a complicated function of the elastic constants and the angle \( \gamma \) between \( y \) and a fixed datum in the surface of the half space (see Appendix). Given the orientation of the half space and the elastic constants of the material, the integrand in (10) can be readily numerically integrated. The indentation modulus can then be calculated using

\[
M = \frac{\sqrt{\pi}}{2} \frac{1}{\sqrt{A}} \frac{L}{\delta} = 16\pi^2 \left( \int_0^{2\pi} \int_0^\pi x_m B_{im}^{-1} (\gamma) z_i \, d\gamma \right)^{-1}.
\]

Equations (5) and (11) were used by Vlassak and Nix (1993) to calculate the indentation modulus for the \{100\}, \{111\} and \{110\} surfaces of materials with cubic symmetry. The indentation modulus of the \( (hkl) \) surface of a single crystal can be written as the product of a correction factor \( \beta_{hkl} \) and the indentation modulus of an isotropic, randomly oriented, polycrystalline aggregate consisting of crystals with the same elastic properties as the single crystal. For cubic crystals, the correction factor \( \beta_{hkl} \) is a function of Poisson’s ratio in the cube directions and the anisotropy factor \( A \) of the crystal. Table 1 contains numerical data for \( \beta_{hkl} \) for the \{100\}, \{111\} and \{110\} surfaces of cubic crystals with various Poisson’s ratios and anisotropy factors. Indentation moduli for other Poisson’s ratios can be found by interpolation.

### 3. THE INDENTATION MODULUS FOR A TRIANGULAR INDUCTER

In most microindentation experiments the indenter is a three-sided pyramid, rather than an axisymmetric paraboloid or flat punch. This will, of course, affect the contact stiffness between the indenter and the sample. From the results of the previous section it is clear that the indentation modulus depends on the shape of the contact area between indenter and material. The contact area for a triangular pyramid is a triangle with slightly curved sides. If we neglect the curvature of the sides of this triangle, we can model the three-sided pyramid as a flat triangular punch. This assumption greatly simplifies the analysis and the loss of accuracy is expected to be small.
Table 1. Indentation moduli of solids with cubic crystal symmetry: correction factor $\beta_{(hkl)}$ as a function of the anisotropy factor $A$ and the Poisson’s ratio in the cube directions

$$M_{(hkl)} = \beta_{(hkl)} \left( \frac{E}{1 - v^2} \right)_{\text{anisotropic}},$$

where $\beta_{(hkl)} = a + c(A - A_0)^B$

<table>
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<tr>
<th>$v_{(100)}$</th>
<th>$a$</th>
<th>$c$</th>
<th>$A_0$</th>
<th>$B$</th>
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<td>1.409</td>
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<td>0.2367</td>
<td>$0.6 &lt; A &lt; 8$</td>
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<td>0.2604</td>
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<td>0.45</td>
<td>1.219</td>
<td>-0.219</td>
<td>0</td>
<td>0.2341</td>
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</tbody>
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<table>
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<td>0.230</td>
<td>-0.893</td>
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<tr>
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<td>0.234</td>
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<tr>
<td>0.35</td>
<td>0.724</td>
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</table>

In order to calculate the contact stiffness of a triangular punch on an anisotropic half space, we first have to determine the pressure distribution under the punch. Writing the penetration depth of the punch, $\delta$, in terms of the unknown pressure distribution leads to the following singular integral equation:

$$\delta = \delta(y) = \int_S p(y') w(y - y') \, dS,$$  \hspace{1cm} (12)

where $p(y')$ is the pressure distribution under the punch, $S$ is the triangular contact area, $y$ and $y'$ are vectors in the boundary of the half space and $w(y)$ is the normal component of the surface displacement due to a unit point load applied at the origin and perpendicular to the boundary of the half space. An expression for $w(y)$ has been derived by Barnett and Lothe (1975) and is given in the Appendix. An analytical solution for (12) is not available and the integral equation has to be solved numerically. In order to do this, the contact area $S$ is divided into a mesh of $n$ equilateral, triangular elements with $m$ nodes $(i,j)$ (see Fig. 2). Instead of requiring that (12) holds everywhere in $S$, we require only that the equation be satisfied at the nodes of the mesh. The displacement $\delta_{ij}$ at each node $(i,j)$ can be calculated by numerically integrating the integrand in (12). Using the nodes of the mesh as integration points, this leads to

$$\delta = \delta_{ij} = \frac{\sqrt{3}}{4n^2} \sum_k c_{kij} p_k w(y_{ij} - y_{ik}),$$  \hspace{1cm} (13)
where \( p_{ki} \) is the unknown pressure at node \((k, l)\), \( y_{ij} \) and \( y_{kl} \) are the position vectors of nodes \((i, j)\) and \((k, l)\), respectively, and \( e_{kl} \) is a weight factor equal to \(1/3\) for corner nodes, \(1\) for boundary nodes and \(2\) for internal nodes of the mesh. The integral equation (12) is thus reduced to a set of \( m \) simultaneous linear equations in the unknown pressures \( p_{ij} \), one for each node. This set of equations can be solved for the unknown pressures. Once the pressure distribution is known, the total load \( L \) on the punch is determined by integrating the pressure distribution over the contact area. The indentation modulus is then given by the left-hand side of (11).

A few complications arise when the integrand in (12) is numerically integrated. First, the surface displacement \( w(y - y') \) tends to infinity when \( y' \) approaches \( y \) and numerical integration is not feasible. This problem can be solved by averaging the angle dependent part of \( w(y - y') \) and assuming that the pressure varies linearly in the elements adjacent to the singularity. The integral can then be calculated analytically in these elements. The second difficulty results from the fact that the integral in (12) is a double integral and that a large number of elements are needed to obtain an accurate result. The load on the punch was calculated for various meshes and it was found to converge linearly with mesh size. This convergence is fairly slow. In order to limit computer memory use and computation time, the final result was obtained by extrapolating to zero mesh size. The solutions of integral equations of the form of (12) often have singularities near the boundaries of the integration domain. The pressure distribution under a flat, circular punch on an isotropic half space, for instance, tends to infinity at the edge of the punch (Sneddon, 1965). A second mesh was therefore used in which the elements near the boundary were only one half the size of the internal elements, but which was otherwise identical to the previous mesh.
The loads thus obtained are typically a few percent below those calculated with the other mesh, but extrapolation to zero mesh size yields the same load to within 0.1%.

We have used this method to evaluate the indentation modulus of an elastically isotropic material for a triangular indenter. The number of elements used in the calculation varied from 64 to 3600 and the indentation modulus was found to be

$$M_{iso} = 1.058 \frac{E}{1 - v^2}.$$  \hspace{1cm} (14)

The indentation modulus for a triangular punch is therefore 5.8% higher than the modulus for an axisymmetric punch. King (1987) has reported a value for the triangular punch which is only 3.4% higher. The difference between these values is probably due to the fact that in King's work rectangular rather than triangular elements were used and that the pressure near singularities was constant within each element. In any case, the indentation modulus of an isotropic solid does not vary much with indenter shape. This confirms a recent theoretical result by Gao and Wu (1993) that the contact stiffness of a flat punch on an isotropic half space is, to first order, independent of the cross-sectional shape of the punch. In addition to the modulus for isotropic materials, we have also calculated the indentation moduli of the \{100\}, \{110\} and \{111\} surfaces of a cubic crystal with an anisotropy factor of 5 and a Poisson's ratio in the cube directions of 0.3. The results of the calculations are shown in Fig. 3. The moduli have been normalized with the corresponding indentation modulus for a flat circular punch (see Table 1) and are shown as a function of the orientation of the triangular indenter in the surface of the half space. For anisotropic solids, one would expect the indentation modulus to depend on the orientation of the

![Fig. 3](image_url)
Fig. 4. The indentation modulus for a triangular indenter and a zinc surface parallel to the prismatic plane. The modulus has been normalized with the indentation modulus for a flat circular punch (130 GPa) and is plotted as a function of the orientation of the indenter in the surface of the half space. The abscissa is the angle between one of the sides of the indenter and the (2T10) direction.

In summary, the indentation modulus for a triangular indenter is typically a few percent larger than the modulus for axisymmetric indenters and varies slightly with the orientation of the indenter. In most practical applications, however, the experimental

<table>
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<th>Crystal structure</th>
<th>$c_{11}$</th>
<th>$c_{12}$</th>
<th>$c_{13}$</th>
<th>$c_{33}$</th>
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<td>204.4</td>
<td>-</td>
<td>-</td>
<td>160.8</td>
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<td>34.2</td>
<td>50.3</td>
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<td>38.3</td>
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Elastic constants are given in GPa.
† Taken from Simmons and Wong (1971).
‡ Interpolated between elastic constants from Lazarus (1949) and Artman and Thompson (1952).
scatter is larger than the amplitude of the variation and the orientation dependence of the indentation modulus can be neglected.

4. EXPERIMENTAL INVESTIGATION

4.1. Materials

The indentation moduli of several single crystals with various orientations were measured using nanoindentation. Crystals with both cubic and hexagonal crystal symmetry were tested. The materials with cubic symmetry had anisotropy factors ranging from 1 to 8.55 and included tungsten, aluminum, copper and β-brass, listed in order of increasing anisotropy factor. Zinc was tested as an example of a hexagonal crystal with large anisotropy. For the cubic materials, samples with surfaces parallel to the {100} and {111} planes were cut. For copper and β-brass, samples with the {110} orientation were also prepared. Two zinc crystals had surfaces parallel to the basal plane and the prismatic plane, respectively, and three zinc crystals had intermediate orientations. The orientations of the zinc samples are given in Table 4b. All the single crystals were oriented to within less than 1° of the desired orientation by means of Laue x-ray diffraction. The tungsten, aluminum and zinc samples were nominally pure while the copper samples contained 0.10 wt% Si. The composition of the β-brass crystal was 47.90 wt% zinc and 52.10 wt% copper. In order to minimize effects associated with segregation during crystal growth, the β-brass samples were cut from one crystal in such a way that the indentation sites in each sample came from locations in the single crystal less than 5 mm from each other and along the axis of the crystal. The samples were polished to a mirror finish using standard metallographic techniques (see Table 3).

4.2. Indentation procedure and analysis

All indentation experiments were performed using a Nanoindenter®, a high-resolution, depth-sensing indentation instrument, the description of which can be found elsewhere in the literature (Doerner and Nix, 1986; Doerner, 1987). Both applied load and displacement are continuously recorded during the experiments. The resolution of

<table>
<thead>
<tr>
<th>Table 3. Sample preparation for the single crystals</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>W</strong></td>
</tr>
<tr>
<td><strong>Al</strong></td>
</tr>
<tr>
<td><strong>Cu</strong></td>
</tr>
<tr>
<td><strong>β-brass</strong></td>
</tr>
<tr>
<td><strong>Zn</strong></td>
</tr>
</tbody>
</table>
load and displacement measurements are 0.2 nm and 0.25 μN, respectively. The indenter is a diamond Berkovich indenter, i.e. a three-sided pyramid with the same depth to area ratio as the Vickers indenter. In each of the samples, two or three indentation experiments were conducted, each consisting of 36 indentations with depths varying from 200 to 1000 nm. In zinc, indentations as shallow as 100 nm were made. A typical load–time history for one of the indentations is shown in Fig. 5. The velocity of the indenter upon loading was kept between 10 and 15 nm s\(^{-1}\). When the desired depth was reached, the load was held constant until the indenter velocity dropped below 0.1 nm s\(^{-1}\). This hold period was included in order to allow creep effects to diminish. Then the load was cycled twice, at a constant rate of loading and unloading, between the maximum load and 20% of the maximum load. The purpose of the cycling was to ensure that no reverse plasticity would occur during the final unloading. Finally the sample was fully unloaded. All measurements of the indentation modulus were made using data from this last unloading segment. The long hold period at 20% of the peak load was 140 s long and served to determine any drift in the displacement measurements that might occur due to thermal expansion of some components of the system. The thermal drift measured in this way was typically very small; the drift rate was of the order of 0.03 nm s\(^{-1}\) or less. It was assumed to be constant during the indentation experiments and was subtracted from the measured displacements for all materials except zinc. For most of the measurements, removing the thermal drift did not change the indentation moduli significantly. However, in zinc, even after two loading cycles, a considerable part of the displacement during the low-load hold period was due to time-dependent plastic deformation, so the drift rate could not be measured. For each indentation, the contact compliance at maximum load was determined by fitting a power law to the top 60% of the unloading data. The contact depth was evaluated using the Oliver Pharr method. The contact com-

![Typical load-time schematic for the indentation experiments](image)

Fig. 5. Load–time schematic for the indentations. The indenter velocity upon loading was between 10 and 15 nm s\(^{-1}\). The rest of the indentation is performed at a constant rate of loading or unloading, equal to the rate at maximum load during the first loading segment. All measurements of indentation moduli were made using data from the last unloading segment.
Indentation of anisotropic elastic materials

4.3. Experimental results and discussion

4.3.1. The indentation modulus. Some typical indentation curves for the {100} surfaces of the samples with cubic crystal symmetry are shown in Fig. 6. The experimental contact compliances for tungsten and β-brass are plotted in Figs 7 and 8 as a function of the inverse of the square root of the contact area for various sample orientations. According to (3), the data points should fall on a straight line, the slope of which is inversely proportional to the indentation modulus. The data for the {100} and {111} surfaces of tungsten fall on two parallel lines, indicating that the indentation moduli for these orientations are the same. The off-set between the two lines is due to a small difference in the compliances of the sample supports. The contact compliance of β-brass, on the other hand, shows a clear dependence on sample orientation. In order to better compare the experimental indentation moduli with the calculations, the indentation moduli of the cubic materials are all normalized with the {100} indentation modulus, i.e. we look at relative changes in the indentation modulus with orientation. The reason for this procedure is that the contact area between the indenter and the material cannot be determined very accurately and even a small error in contact area can systematically increase or decrease the measured indentation modulus. If the indentation moduli are normalized, however, they are not very sensitive to the contact area and any systematic error cancels out. Figure 9 shows the normalized indentation moduli of the cubic materials as a function of orientation. The solid lines are the normalized indentation moduli for tungsten, aluminum, copper and β-brass.

![Typical indentation plots for the {100}-surface of metals with cubic crystal symmetry](image)

Fig. 6. Typical load–displacement curves for the {100} surfaces of the samples with cubic crystal symmetry. The loading cycles have been omitted for clarity.
calculated for a flat, circular punch on a surface that first coincides with the (110) plane and then rotates about the [110] axis through the position of the (111) plane until it coincides with the (001) plane. The elastic constants used in the calculations are given in Table 2. The data points refer to the experimentally measured indentation moduli. The error bars correspond to plus or minus one standard deviation. The agreement between experimental values and theoretical calculations is very satisfactory and all deviations are well within experimental scatter. For copper and β
brass, the (111) indentation moduli are approximately 10 and 25% larger than the \{100\} modulus. The (110) moduli are typically slightly smaller than the (111) moduli. The zinc indentation modulus is plotted as a function of the orientation in Fig. 10. The modulus is normalized with the indentation modulus of the basal plane. The solid line corresponds to the normalized indentation modulus calculated for a surface that rotates about the \[10\overline{1}0\] axis; the dashed line is Young's modulus perpendicular to that surface.

Fig. 9. The normalized indentation moduli of the samples with cubic crystal symmetry as a function of orientation. The solid lines are the normalized indentation moduli for tungsten, aluminum, copper and \(\beta\) brass, calculated for a flat, circular punch on a surface that first coincides with the \{110\} plane and then rotates about the \{110\} axis through the position of the \{111\} plane until it coincides with the \{001\} plane. The error bars correspond to plus or minus one standard deviation.

Fig. 10. The zinc indentation modulus as a function of the orientation. The modulus is normalized with the indentation modulus of the basal plane. The solid line corresponds to the normalized indentation modulus calculated for a surface that rotates about the \[10\overline{1}0\] axis, the dashed line is Young's modulus perpendicular to that surface, also normalized by the indentation modulus of the basal plane.
Table 4a. *Theoretical and experimental values of the indentation moduli for the samples with cubic crystal symmetry. The theoretical values are for a flat, circular indenter.*

<table>
<thead>
<tr>
<th>Material</th>
<th>Anisotropy factor</th>
<th>$v_{100}$</th>
<th>$M_{100}$ (GPa)</th>
<th>$M_{110}$ (GPa)</th>
<th>$M_{111}$ (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>W</td>
<td>Theory</td>
<td>1.00</td>
<td>0.28</td>
<td>442</td>
<td>442</td>
</tr>
<tr>
<td></td>
<td>Experiment</td>
<td></td>
<td></td>
<td>439</td>
<td>438</td>
</tr>
<tr>
<td>Al</td>
<td>Theory</td>
<td>1.22</td>
<td>0.36</td>
<td>79</td>
<td>80</td>
</tr>
<tr>
<td></td>
<td>Experiment</td>
<td></td>
<td></td>
<td>77</td>
<td>79</td>
</tr>
<tr>
<td>Cu</td>
<td>Theory</td>
<td>2.21</td>
<td>0.40</td>
<td>129</td>
<td>138</td>
</tr>
<tr>
<td></td>
<td>Experiment</td>
<td></td>
<td></td>
<td>124</td>
<td>134</td>
</tr>
<tr>
<td>β-brass</td>
<td>Theory</td>
<td>8.55</td>
<td>0.40</td>
<td>95</td>
<td>112</td>
</tr>
<tr>
<td></td>
<td>Experiment</td>
<td></td>
<td></td>
<td>104</td>
<td>121</td>
</tr>
</tbody>
</table>

Table 4b. *Theoretical and experimental values of the indentation moduli for the zinc single crystals. The theoretical values are for a flat, circular indenter. The angle is the dihedral angle between the surface of the sample and the basal plane.*

<table>
<thead>
<tr>
<th>Dihedral angle (deg)†</th>
<th>Theoretical indentation modulus (GPa)</th>
<th>Experimental indentation modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td>68</td>
<td>60</td>
</tr>
<tr>
<td>23.5</td>
<td>74</td>
<td>65</td>
</tr>
<tr>
<td>43.4</td>
<td>92</td>
<td>79</td>
</tr>
<tr>
<td>67.0</td>
<td>117</td>
<td>98</td>
</tr>
<tr>
<td>88.0</td>
<td>130</td>
<td>122</td>
</tr>
</tbody>
</table>

† 0° and 90° correspond to the basal plane and the prismatic plane, respectively.

to the surface, also normalized by the indentation modulus of the basal plane. The experimental indentation moduli agree very well with the calculated values of the indentation modulus, but differ significantly from Young's modulus. The indentation modulus of zinc varies by as much as a factor of two depending on the orientation of the surface.

The absolute values of the experimental indentation moduli are given in Table 4, along with the values calculated for a flat circular indenter. The experimental moduli agree well with the theoretical predictions for tungsten, aluminum and copper; they are slightly high for β-brass and slightly too low for zinc. These discrepancies can be attributed to a number of causes. For zinc, some time-dependent plasticity took place during the final unloading segments of the indentations. This does not seem to affect the relative indentation moduli, but could easily introduce a systematic error in the absolute values. The presence of a thin oxide layer on the surface of the samples could also account for some of the difference. As discussed by Laursen and Simo (1992), the contact area between the indenter and the material used in the Oliver-Pharr model is smaller than the actual contact area, so that contact areas are typically...
overestimated. As a result, by using the Oliver–Pharr method one tends to underestimate the actual indentation moduli. Because of the uncertainty in the absolute values of the indentation moduli, it is difficult to determine whether the triangular shape of the Berkovich indenter has a significant effect on the indentation modulus. For the ideal case in which the contact area is a perfect triangle, the theoretical values of the indentation moduli in Table 4 would have to be increased by approximately 6%, bringing the values for β-brass into better agreement. Agreement for the other materials would get worse, however. In practice, one would expect the indentation modulus for a Berkovich indenter to lie between that for a circular and a triangular indenter, since the contact area for a pyramidal indenter has the shape of a triangle with slightly convex sides. Plastic flow and pile-up of material during the indentation can increase the curvature of the sides of the contact triangle and further diminish the effect of the indenter geometry.

4.3.2. Hardness of single crystals. In addition to indentation moduli, the indentation curves also allow one to obtain hardnesses. Figures 11 and 12 show the hardnesses of the cubic crystals and the zinc crystals, respectively, for various orientations, for an indentation depth of 1000 nm. In spite of the large anisotropy of the yield stress of single crystals, the hardness does not vary much with the orientation of the plane of the indentation. For β-brass, for instance, the hardness of a {110} surface is only about 13% lower than the hardness of a {100} or {111} surface. Even for zinc, in which slip on the basal plane is much easier than on the other slip systems, the maximum change in hardness is only 20%. This is a result of the fact that the plastic strain field in indentation is very complicated and in order to accommodate the imposed deformation, multiple slip systems are active at the same time. The same slip systems may be operating for differently oriented samples and the hardness is averaged over these slip systems. For hexagonal close-packed metals, it has been observed that twinning is an important mode of deformation during indentation of the basal plane, but not of the prismatic plane (Partridge and Roberts, 1963). At least some of the

Fig. 11. Hardnesses of the samples with cubic crystal symmetry as a function of orientation. The hardnesses are calculated for an indentation depth of 1000 nm.
variation in the hardness of the zinc single crystals can be attributed to this fact. Variations of the hardness with indenter orientation in the plane of the indentation were found to be of the same order as the scatter in the data. Given the threefold symmetry of the Berkovich indenter, one would indeed expect the hardness to vary only slightly with the in-plane orientation of the indenter. This behavior is very different from that of the Knoop hardness, which depends strongly on the crystallographic orientation of the long axis of the Knoop indenter in the plane of the indentation (Garfinkle and Garlick, 1968; Brookes et al., 1971).

For many metals, the hardness is approximately three times the yield stress. This is not observed for single crystals where the hardness is mainly determined by the strain-hardening behavior and is of the order of 1% of the shear modulus (Gerk, 1977). Even though it is not clear which shear modulus should be used for anisotropic solids, the hardnesses in this study seem to follow this correlation. They are much higher than would be expected on the basis of the yield stress. Figures 13 and 14 show two optical micrographs of indentations in surfaces parallel to the basal plane and the prismatic plane of a zinc single crystal. The shapes of the indentations are clearly different for these orientations; the indentations in the basal plane are much more symmetric and show significant pile-up along the sides of the indentations. By contrast, the indentations in the prismatic plane are clearly elongated and pile-up is most prevalent in the corners nearest the [0001] direction. This indicates that in addition to a difference in plastic deformation, there is also a noticeable difference in the elastic strain field. However, this does not seem to influence the indentation modulus significantly.

5. CONCLUDING REMARKS

In this paper, we have reviewed some of the basic formulae describing the indentation of elastically anisotropic solids with axisymmetric indenters. We have shown
Fig. 13. Optical micrographs of indentations in a zinc single crystal. The surface of the sample is parallel to the basal plane.

Fig. 14. Optical micrographs of indentations in a zinc single crystal. The surface of the sample is parallel to the prismatic plane.
how the indentation modulus can be calculated for arbitrary anisotropic solids. The indentation modulus is typically very different from Young’s modulus in the direction of the indentation and is best approximated by a weighted average of the elastic constants given by (5) or (11). Since the indenter has a triangular, rather than a circular, cross section in most microindentation experiments, we have calculated the contact stiffness of a flat triangular punch on a half space for various anisotropic materials. The indentation modulus for a triangular indenter is typically 5–6% higher than that for an axisymmetric indenter and varies only slightly with the orientation of the indenter in the plane of the indentation.

We have conducted microindentation experiments to measure the indentation moduli of differently oriented surfaces of both cubic and hexagonal single crystals. The indentation moduli are predicted very satisfactorily by (5). For copper and β-brass, the (111) indentation moduli are approximately 10 and 25% larger than the {100} modulus. The (110) moduli are typically slightly smaller than the (111) moduli. The indentation modulus of zinc varies by as much as a factor of two, depending on the sample orientation. In spite of the large anisotropy of the yield stress of single crystals, the hardness of the single crystals does not vary much with the orientation of the plane of indentation. For β-brass, the hardness of a {110} surface is only approximately 13% lower than the hardness of a {100} or {111} surface. For zinc the maximum change in hardness with orientation is 20%.

ACKNOWLEDGEMENTS

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REFERENCES

APPENDIX

In this Appendix we give the expression derived by Barnett and Lothe (1975) for the displacement field of the boundary of an anisotropic elastic half space subjected to a unit point load applied at the boundary and derive an equation for the displacement under a flat circular punch using this expression and (9).

Consider a coordinate system \((x_1, x_2, x_3)\). Let \(t\) be a unit vector in an infinite anisotropic medium and \(m\) and \(n\) two orthogonal unit vectors perpendicular to \(t\), so that \((m, n, t)\) form a right-hand Cartesian system. Let \(\varphi\) be the angle between \(m\) and some fixed datum in the plane perpendicular to \(t\). Repeated indices imply a summation over the repeated index from 1 to 3.

We define the matrices \(B\) and \(S\) by

\[
B_{ij}(t) = B_{ij}(t) = \frac{1}{8\pi^2} \int_0^{2\pi} \left\{ (mm)_{ij} - (mn)_{ij} (mm)^{-1} (mn)_{ij} \right\} d\varphi \\
S_{ij}(t) = -\frac{1}{2\pi} \int_0^{2\pi} (nn)^{-1} (mn)_{ij} d\varphi.
\]  

In (A1) and (A2) the matrices \((ab)\) are given by
where the $C_{ijkl}$ denote the elastic stiffnesses of the anisotropic material. $B$ and $S$ depend only on the elastic constants of the material and the direction $t$. $B$ is symmetric and positive definite (Barnett and Lothe, 1975) and $SB^{-1}$ is antisymmetric [this can be seen after some straightforward manipulations of (3.24) in the work by Lothe and Barnett (1976)].

Consider a half space with its boundary through the origin of the coordinate system. The orientation of the boundary of the half space is arbitrary with respect to the coordinate system and is given by the direction cosines $(a_1, a_2, a_3)$ of the normal to the boundary. The displacement in the $x_n$ direction at a point $q$ in the half space boundary due to a unit point load applied in the $x_m$ direction at the origin of the coordinate system is then given by (Barnett and Lothe, 1975)

$$u_{km}(y) = \frac{1}{8\pi^2|y|} \left[ B_{km}^{-1} \left( \frac{y}{|y|} \right) + \frac{1}{\pi} \int_0^\infty B_{ij}^{-1}(t)S_{mj}(t) \sin \left( \frac{\gamma - \gamma_0}{\pi} \right) d\gamma \right].$$

where $P$ indicates principal value, $y$ is the position vector of $q$ and $t$ lies in the half space boundary. $\gamma$ and $\gamma_0$ are the angles between some fixed datum in the half space boundary and $t$ and $y$, respectively. If the concentrated unit load is perpendicular to the boundary of the half space, the displacement $w(y)$ in the direction of the load is given by

$$w(y) = \int_0^\infty \left[ \frac{1}{8\pi^2|y|} B_{km}^{-1} \left( \frac{y}{|y|} \right) \right] d\gamma = \frac{1}{8\pi^2|y|} \left[ \frac{1}{\pi} \int_0^\infty B_{ij}^{-1}(t)S_{mj}(t) \sin \left( \frac{\gamma - \gamma_0}{\pi} \right) d\gamma \right].$$

Since $SB^{-1}$ is antisymmetric, the last term in (A5) vanishes and (A5) reduces to

$$w(y) = \frac{1}{8\pi^2|y|} \left[ \frac{1}{\pi} \int_0^\infty B_{ij}^{-1}(t)S_{mj}(t) \sin \left( \frac{\gamma - \gamma_0}{\pi} \right) d\gamma \right].$$

Substituting (A6) into (9) and taking $y$ equal to zero lead, after transformation to polar coordinates $(\rho, \gamma)$, to

$$\delta = \frac{L}{16\pi^2 a} \int_0^{2\pi} \frac{\rho \sqrt{a^2 - \rho^2}}{\rho} d\gamma d\rho,$$

where $\rho$ is equal to $|y|$ and $\gamma$ is the angle between $y$ and some fixed datum in the half space boundary. The deflection under the flat circular punch is finally given by

$$\delta = \frac{L}{32\pi^2 a} \int_0^{2\pi} \frac{\rho \sqrt{a^2 - \rho^2}}{\rho} d\gamma d\rho.$$