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The NIMS toolbox has been developed to plot and to analyze (nano)indentation data (obtained with conical indenters) for bulk material or multilayer sample.

**With this Matlab toolbox, it is possible to:**

- plot and correct nanoindentation dataset with standard deviation;
- calculate the coefficient of the power law fit of the load-displacement curve;
- calculate the energy of the loading (area below the load-displacement curve);
- plot of the stiffness and the load/stiffness\(^2\) evolution;
- calculate the Young’s modulus and hardness of bulk materials;
- calculate the Young’s modulus and hardness of thin films on a substrate (for a bilayer or a multilayer sample (until 3 layers on a substrate));
- generate Python script of axisymmetrical FEM model for use in ABAQUS (conospherical indentation of multilayer sample).

Source code is hosted at Github.

Download the documentation as a pdf file.

Figure 1: *Screenshot of the main window of the NIMS toolbox.*
1.1 Getting started

First of all, download the source code of the Matlab toolbox.
Source code is hosted at Github.
To have more details about the use of the toolbox, please have a look to :

Getting_started.txt

1.1.1 How to use the GUI for indentation data analysis ?

First of all a GUI is a Graphical User Interface.

• Create or update your personal YAML config. file stored in the YAML folder

See here how to create / modify your YAML file…

• Run the following Matlab script :

demo.m

• Answer ‘y’ or ‘yes’ (or press ‘Enter’) to add path to the Matlab search paths, using this script:

path_management.m

• The following window opens:

• Import your (nano)indentation results, by clicking on the button ‘Select file’. Click here to have more details about valid format of data.

• A load-displacement curve is plotted (with a power law fit). The loading work is also given in the title of the plot.

• It is possible to plot the stiffness (raw data) without setting the GUI for Young’s modulus calculation.
Figure 1.1: Screenshot of the main window of the NIMS toolbox.

Figure 1.2: File selector.

Figure 1.3: Plot of the load-displacement curve after loading of data.
• Choose and set (if needed) the indenter used to obtain (nano)indentation data.
• Select the lowest and the highest depth values (optional).
• Set the CSM correction (Berkovich indenter only!) (optional).
• Set the number of layers of your sample (0 = only bulk material, 1/2/3 = 1 to 3 thin layers on a substrate) (see Figure 1.4).

![Figure 1.4: Convention use to define multilayer specimen.](image)

• Set the thickness, the Poisson’s coefficient and the Young’s modulus to each layer.
• Select the model to use for the contact displacement calculation and select the correction to apply.
• Select ‘Red. Young’s modulus(film+sub)’ or ‘Red. Young’s modulus(film)’ in order to plot the evolution of the reduced Young’s modulus (raw calculation) of the sample vs. the evolution of the reduced Young’s modulus (modeled) of the sample and/or of the thin film.
• Select the analytical bilayer or the multilayer model to use for the modelling of the reduced Young’s modulus of the top thin film.
• Press the button ‘SAVE’ and a YAML results file and a picture of the figure (.png format) are created and stored in the following folder.
• Press the button ‘FEM’ and generate a Python script to model nanoindentation of multilayer sample based on parameters used in the GUI for ABAQUS.

![Figure 1.5: Plot of the evolution of the Young’s modulus of the sample with the elastic multilayer model as a function of the indentation depth.](image)

1.1.2 Links

• Guidata on Matlab website.

1.1. Getting started
Figure 1.6: Plot of the evolution of the Young’s modulus of the film with the elastic multilayer model as a function of the indentation depth.

- Matlab GUI.

1.2 Configuration

1.2.1 What is a YAML File?

"YAML is a human friendly data serialization standard for all programming languages."

Visit the YAML website for more informations.

Visit the YAML code for Matlab.

You have to update the YAML configuration files in order to use correctly Matlab toolbox.

1.2.2 The YAML configuration files

Three YAML configuration files are used in the Matlab toolbox:

- `indenter_config.yaml` provides indenter’s properties (geometry and material).
- `data_config.yaml` provides a path on your computer to select easily your data.
- `numerics_config.yaml` provides numerical parameters used by the toolbox.

1.2.3 How to modify YAML configuration files?

Please find the 3 YAML configuration files in the YAML folder.

`indenter_config.yaml` can be used to change indenter’s properties:

- Write your Indenter_ID(s) (e.g.: Conical indenter, Berk_TB10161_091208, …);
- Write indenter’s properties (e.g.: Berk_TB10161_091208: [22.233, 437.603, 127.765, -417.878, -84.0989, 0]);
Figure 1.7: Screenshot of the YAML configuration file used for indenter’s properties.

Warning:

- For user-defined indenters, make the name of the indenter begins with the 4th first letters of the indenter name (e.g.: ‘Berk_130214’ for ‘Berkovich’).
- Do not remove standard indenters and standard materials!

**data_config.yaml** can be used to set the default absolute path for the folder where you store your indentation data. **numerics_config.yaml** can be used to change the numerical parameters used by the toolbox from their standard values.

Warning:

- Be careful to put a comma + a space between each data . . . ! (YAML convention)
- Use # in the beginning of the line to add comments.

It is also possible to edit and to load the different YAML configuration files, via the customized menu of the GUI.

Note: To open and to modify these YAML files, you can directly use Matlab or any code editor (e.g.: Notepad++).

### 1.3 Models for bulk material

The nanoindentation (or instrumented or depth sensing indentation) is a variety of indentation hardness tests applied to small volumes. During nanoindentation, an indenter is brought into contact with a sample and mechanically loaded.

**1.3. Models for bulk material**

The nanoindentation (or instrumented or depth sensing indentation) is a variety of indentation hardness tests applied to small volumes. During nanoindentation, an indenter is brought into contact with a sample and mechanically loaded.
The following parts give a short overview of models existing in the literature used for the extraction of mechanical properties of homogeneous bulk materials from indentation experiments with geometrically self-similar indenters (conical or sharp).

Please look at the ISO standard (ISO 14577 - 1 to 3), to perform nanoindentation tests on bulk material.


Some authors overviewed/reviewed already the nanoindentation technique:


1.3.1 Nanoindentation tests on bulk material

Conical and geometrically similar indenters

The geometric properties of conical and geometrically similar (Berkovich, Vickers, . . . ) indenters are well described in\textsuperscript{11}.

\textsuperscript{11} Fischer-Cripps A.C., “Nanoindentation” Springer 3rd Ed. (2011).
Table 1.1: Geometric properties of conical indenters.

<table>
<thead>
<tr>
<th>Indenter</th>
<th>Berkovich</th>
<th>Vickers</th>
<th>Cube-corner</th>
<th>Knoop</th>
<th>Conical (angle $\psi$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Shape</td>
<td>3-sided pyramid</td>
<td>4-sided pyramid</td>
<td>3-sided pyramid</td>
<td>4-sided pyramid</td>
<td>Conical (angle $\psi$)</td>
</tr>
<tr>
<td>Semi-angle from the apex</td>
<td>$65.3^\circ$</td>
<td>$68^\circ$</td>
<td>$35.2644^\circ$</td>
<td>$86.25^\circ / 65^\circ$</td>
<td>$\pi a_c^2$</td>
</tr>
<tr>
<td>Equivalent cone angle</td>
<td>$70.32^\circ$</td>
<td>$70.2996^\circ$</td>
<td>$42.28^\circ$</td>
<td>$77.64^\circ$</td>
<td>$\psi$</td>
</tr>
<tr>
<td>Projected Area</td>
<td>$24.56 h_c^2$</td>
<td>$24.504 h_c^2$</td>
<td>$2.5981 h_c^2$</td>
<td>$108.21 h_c^2$</td>
<td>$\pi a_c^2$</td>
</tr>
<tr>
<td>Volume-depth relation</td>
<td>$8.1873 h_c^3$</td>
<td>$8.1681 h_c^3$</td>
<td>$0.8657 h_c^3$</td>
<td>$-$</td>
<td>$-$</td>
</tr>
<tr>
<td>Projected area/face area</td>
<td>$0.908$</td>
<td>$0.927$</td>
<td>$0.5774$</td>
<td>$-$</td>
<td>$-$</td>
</tr>
<tr>
<td>Contact radius</td>
<td>$-$</td>
<td>$-$</td>
<td>$-$</td>
<td>$-$</td>
<td>$htan\psi$</td>
</tr>
</tbody>
</table>

Figure 1.8: a) Conical indenter (45°) and b) Berkovich indenter.

With $h_c$ the contact depth.

These indenters have self-similar geometries which implies a constant strain during indentation and similarity of the stress fields.

Note: Indenters are mainly in diamond. Diamond has a Young’s modulus of 1070GPa and a Poisson’s ratio of 0.07\(^{11}\).

Find here the Matlab function to plot the projected area of 3-sided pyramidal indenter as a function of indentation depth: `projectedArea_3sidePyramid.m`.

Find here the Matlab function to plot the the projected area of 4-sided pyramidal indenter as a function of indentation depth: `projectedArea_4sidePyramid.m`.

Practically, perfect conical indenters (no tip defect) don’t really exist and they are usually defined as cono-spherical indenters with a tip defect $h_{tip}$ (see following scheme)\(^{62}\).

The radius $R$ of the spherical part is calculated from the tip defect $h_{tip}$ and the cone angle $\alpha$, using the following equation. For Berkovich, Vickers and cube-corner indenters, the equivalent cone angle is used to set the cone angle.

$$ R = \frac{h_{tip}}{\sin(\alpha)} - 1 $$  \hspace{1cm} (1.1)

In case of a tip defect of 5nm for a Berkovich indenter, a tip radius of $R = 148$nm is calculated.

The transition depth $h_{trans}$ between the spherical and the conical parts of a cono-spherical indenter, is calculated from the following equation:

Figure 1.9: Scheme of a cono-spherical indenter.

\[ h_{\text{trans}} = R(1 - \sin(\alpha)) \]

Find here the Matlab function to calculate the tip radius: tipRadius.m.
Find here the Matlab function to calculate the transition depth: transitionDepth.m.
Find here the Matlab function to calculate the tip defect: tipDefect.m.

Load-Displacement curves

In this first part, only quasistatic (or monotonic) nanoindentation is considered, when a load is applied and removed to a sample. Parameters such as contact load \( F_c \) (in N) and depth of penetration (displacement) \( h_0 \) (in m) are continuously recorded at a rapid rate (normally 10 Hz) during loading and unloading steps of the indentation test. Usually, the depth resolution is around the nm-level and the load resolution is around nN-level.

Initial penetration

The first correction step in nanoindentation testing is the determination of the initial contact point between the indenter and the sample\(^\text{12}\).

\[ h = h_0 + h_i \]

With $h$ the corrected penetration and $h_0$ the recorded penetration.

See also: *Methodology to minimize displacement measurement uncertainties using dynamic nanoindentation testing*

A schematic of the load-displacement curve obtained from nanoindentation experiment after this first correction is given Figure 1.11.

The evolution of this curve depends on material properties of the sample and the indenter, and of the indenter's geometry.

The tangent (or the slope) of the part of the unloading curve at the maximum load gives access to the contact stiffness $S$ (in N/m):

$$S = \frac{dF_C}{dh} \quad (1.4)$$

![Figure 1.11: Schematic of indentation load-displacement curve.](image)

With $h_t$ the total penetration corrected of the frame compliance and $h_r$ the residual indentation or the plastic depth after unloading \(^{18}\).

$$h_r = h - F_cS \quad (1.5)$$

It is worth to mention that for quasistatic nanoindentation, the contact stiffness is a unique value obtained at the maximum load and at the maximum displacement. Nevertheless, it is possible to apply a multiple-point unload method, and then determine the contact stiffness for many indentation depths, as a function of the number of points defined by the user (see Figure 1.12)\(^{11}\).

![Figure 1.12: Schematic of indentation load-displacement curve with the multiple point unload method (here $n$ points).](image)

Finally, concerning the surface detection and initial penetration measurement, roughness surface sample and tilt angle between indenter and sample can have a great influence on indentation measurement\(^{25,60}\) and\(^{30}\). More information about the roughness effect can be found in the ‘Indentation Length Scales’ section of the TriDiMap documentation.

---


\(^{60}\) Xia Y. et al., “Effect of surface roughness in the determination of the mechanical properties of material using nanoindentation test” (2014).

Frame compliance

Before any analysis, it is important to correct raw data of the effect of the frame compliance. The frame compliance is defined by the deflections of the load frame instead of displacement into the studied material. This frame compliance \( C_f \) (in m/N) contributes to the measured indentation depth and to the contact stiffness\(^{12} \).

\[
\begin{align*}
    h_t &= h - F_c C_f \quad (1.6) \\
    S &= \left( \frac{dh}{dF_c} - C_f \right)^{-1} \quad (1.7)
\end{align*}
\]

To determine the frame compliance, it is required to plot \( \frac{dh}{dF_c} \) vs. the corrected total depth (\( 1/h_t \)) or the corrected plastic depth (\( 1/h_c \)) (see the following part “Indentation contact topography” for the definition of the plastic depth)\(^{9} \) and\(^{12} \). Then, a linear fit of this curve gives an intercept with the ordinate axis which is the frame compliance (see Figure 1.13).

![Figure 1.13: Schematic of the plot to determine the frame compliance.](image)

It is advised to perform indentation tests on a variety of bulk standard specimens (fused silica, silicon and sapphire provide a very good range), in order to estimate better the frame compliance.

Moreover, when the sample flexes or has heterogeneities (free edges, interfaces between regions of different properties...) nanoindentation measurements are affected by the structural compliance \( C_s \). Then, it is possible to correct experimental data of this artifact by following the experimental approach proposed by\(^{23} \).

Loading

Loubet et al. founded a good fit to the loading part of the load-displacement curve with a power-law relationship of the form\(^{33} \):

\[
F_c = Kh_t^n \quad (1.8)
\]

With \( K \) and \( n \) constants for a given material for a fixed indenter geometry.

It is possible to find in the literature sometimes the following equation to fit the loading curve:

\[
F_c = Kh_t^n + C_{\text{preload}} \quad (1.9)
\]

With \( C_{\text{preload}} \) a constant which is used to account a small preload prior indentation testing\(^{42} \).

---

9 Doerner M.F. and Nix W.D., “A method for interpreting the data from depth-sensing indentation instruments” (1986).
Using the load-displacement curves analysis performed by Loubet et al., Hainsworth et al. proposed the following relationship to describe loading curves\textsuperscript{20}:

\[ F_c = K h_t^2 \quad (1.10) \]

With \( K \) a constant function of material properties (Young’s modulus and hardness) and the indenter.

In the same time, Giannakopoulos and Larsson established parabolic relationships between the load and the indentation depth, for purely elastic indentation of bulk materials with ideally Berkovich indenter (1.11)\textsuperscript{29} and Vickers indenter (1.12)\textsuperscript{14}, by numerical studies.

\[ F_c = 2.1891 \left( 1 - 0.21\nu - 0.01\nu^2 - 0.41\nu^3 \right) \frac{E}{1 - \nu^2} h_t^2 \quad (1.11) \]

\[ F_c = 2.0746 \left( 1 - 0.1655\nu - 0.1737\nu^2 - 0.1862\nu^3 \right) \frac{E}{1 - \nu^2} h_t^2 \quad (1.12) \]

With \( \nu \) the Poisson’s ratio and \( E \) the Young’s modulus of the indented material.

Finally, it is first important to cite the work of Malzbender et al., who developed the relationship between the load and the indentation depth for elastoplastic materials, based on the knowledge of the Young’s modulus and the hardness values of the material\textsuperscript{38}. Then, It is worth to mention the model of Oyen et al., who described sharp indentation behavior of time-dependent materials\textsuperscript{47}.

**Unloading**

Pharr and Bolshakov founded that unloading curves were well described by the following power-law relationship\textsuperscript{29}:

\[ F_c = \alpha_u (h_t - h_r)^m \quad (1.13) \]

Where \( h_r \) is the final displacement after complete unloading, and \( \alpha_u \) and \( m \) are material constants. Many experiments performed by Pharr and Bolshakov leaded to an average value for \( m \) close to 1.5 for the Berkovich indenter.

**Loading rate**

The mechanical response of a material is function of the imposed indentation strain rate \( \dot{\epsilon} \) (in s\(^{-1}\))\textsuperscript{36}. Thus, it is meaningful to perform indentation tests with a constant indentation strain rate.

\[ \dot{\epsilon} = \frac{\dot{h}}{h} = \frac{1}{2} \frac{\dot{F}_c}{F_c} \quad (1.14) \]

\textsuperscript{38} Malzbender J. and de With G., “Indentation load–displacement curve, plastic deformation, and energy.” (2002).
Indentation contact topography

Indentation contact area is not usually estimated by experiments, given the fact that many possible errors can be induced during the post-mortem topography measurement (small size of residual indent, elastic or plastic recovery...), by atomic force microscopy (AFM) or scanning electron microscopy (SEM) for example. To extract contact area from AFM or SEM measurements, it is advised to use softwares with dedicated tools (e.g.: http://gwyddion.net/). Some tips are given in the following procedure\(^{10}\). A usual method is to calculate the angle between normal to the indent surface with the horizontal sample surface. When this angle is equal to the semi-angle of the apex of the indenter, the corresponding pixel can be used for the residual contact area. The total residual contact area is the sum of all pixels validating this angle condition. Here an example using an indenter with a full apex angle of 140° (see Figure 1.14).

![Figure 1.14: Schematic of residual cross sectionnal indentation contact topography : a) “pile-up” and b) “sink-in”.](image)

The best should be to estimate the contact area during the indentation test, by optical observation through a transparent indenter\(^{10,37}\) or a microscope in situ indentation test\(^{19}\).

For those reasons, contact area is most of the time estimated using contact depth evolution, given a geometrical relationship.

The indentation total depth is rarely equal to the indentation contact depth. Two kind of topographies can occur:

- the pile-up (indentation contact depth > indentation total depth) (see Figure 1.16 a and Figure 1.17);
- the sink-in (indentation contact depth < indentation total depth) (see Figure 1.16 b).

The flow of material below the indenter is function of mechanical properties of the material.

Pile-up occurs when work-hardening coefficient is low (< 0.3) or if the ratio yield stress over Young’s modulus is less than \(^{14,6}\) and \(^{8}\).

---

\(^{10}\) Mercier D., “AFM data analysis using Gwyddion software” (2018).


\(^{19}\) Guillonneau G. et al.,“Determination of the true projected contact area by in situ indentation testing.” (2019).


\(^{6}\) Cheng Y.T. and Cheng C.M., “Effects of ‘sinking in’ and ‘piling up’ on estimating the contact area under load in indentation.” (1998)

Three main models defining the depth of contact $h_c$ were developed to take into account this indentation contact topography.

Model of Doerner and Nix$^9$:

$$h_c = h_t - \frac{F_c}{S} \quad (1.15)$$

Model of Oliver and Pharr$^{45,49}$ and$^{46}$ in case of sink-in:

$$h_c = h_t - \epsilon \frac{F_c}{S} \quad (1.16)$$

Where $\epsilon$ is a function of the indenter’s geometry (0.72 for conical indenter, 0.75 for parabolics of revolution and 1 for a flat cylindrical punch). An expression of $\epsilon$ as a function of the power law exponent $m$ of the unloading curve fit has been proposed by Pharr et Bolshakov$^{49}$:

$$\epsilon = m \left( 1 - \frac{2\Gamma \left( \frac{m}{2(m-1)} \right)}{\sqrt{\pi} \Gamma \left( \frac{1}{2(m-1)} (m-1) \right)} \right)$$

With $\Gamma$ a Matlab function which interpolates the factorial function: gamma.

Find here the Matlab function to plot the $\epsilon$ function: epsilon Oliver Pharr.m.

0.72 should be most applicable for a Berkovich indenter, which is more like a cone than a paraboloid of revolution. But, Oliver and Pharr concluded after a large number of experiments that the best value for the Berkovich indenter is 0.75.


More recently, Merle et al. have found experimentally with indentation tests in fused silica, a value of 0.76 for $\epsilon$, which is in a good agreement with the literature for a paraboloid of revolution\textsuperscript{41}.

Model of Loubet et al.\textsuperscript{22,1} in case of pile-up:

$$h_c = \alpha \left( h_1 - \frac{F_c}{S} + h_0 \right) \tag{1.17}$$

Where $\alpha$ is a constant function of the indented material (usually around 1.2) and the tip-defect $h_0$.

Knowing the depth of contact, it is possible to determine the area of contact $A_c$ (in m$^2$) for a perfect conical indenter (with a semi-angle from the apex $\theta$):

$$A_c = \pi h_c^2 \tan^2 (\theta) \tag{1.18}$$

But, because conical indenters present imperfections and Berkovich or Vickers indenters are not perfectly conical, a general formula of the contact area has been established by Oliver and Pharr\textsuperscript{45,46}:

$$A_c = C_0 h_c^2 + \sum_{n=1}^{8} C_n h_c^{1/2n-1} \tag{1.19}$$

With the coefficients $C_0$ and $C_n$ obtained by curve fitting procedures, from nanoindentation experiments in fused silica (amorphous and isotropic material).

For a perfect Berkovich indenter $C_0$ is equal to 24.56 and for a perfect Vickers indenter $C_0$ is equal to 24.504 (see Table 1.1).

The second term of the area function $A_c$ describes a paraboloid of revolution, which approximates to a sphere at small penetration depths. A perfect sphere of radius $R$ is defined by the first two terms with $C_0 = -\pi$ and $C_1 = 2\pi R$. The first two terms also describe a hyperboloid of revolution, a very reasonable shape for a tip-rounded cone or pyramid that approaches a fixed angle at large distances from the tip.

In case of surface tilt, the area function can be corrected following different methods\textsuperscript{27} and\textsuperscript{24}.

\textsuperscript{41} Merle B. et al., “Experimental determination of the effective indenter shape and e-factor for nanoindentation by continuously measuring the unloading stiffness.” (2012).

\textsuperscript{22} Hochstetter G. et al., “Strain-rate effects on hardness of glassy polymers in the nanoscale range. Comparison between quasi-static and continuous stiffness measurements” (1999).

\textsuperscript{1} Bec S. et al., “Improvements in the indentation method with a surface force apparatus” (2006).

\textsuperscript{27} Kashani M.S. and Madhavan V., “The Effect of Surface Tilt on Nanoindentation Results” (2007).

\textsuperscript{24} Jakes J.E. and Stauffer D., “Contact area correction for surface tilt in pyramidal nanoindentation” (2021).
An equivalent contact radius $a_c$ (in m) is also defined based on the area function.

$$a_c = \sqrt{\frac{A_c}{\pi}} \quad (1.20)$$

One other way to express the function area is that suggested by Loubet et al.$^{32}$, which describes a pyramid with a small flat region on its tip, the so-called tip defect ($h_0$). This geometry is described by the addition of a constant to the first two terms in (1.19).

Find here the Matlab function to calculate the contact depth, the function area and the contact radius: `model_function_area.m`.

Recently, in the paper of Yetna N’jock M. et al.$^{61}$, a criterion was proposed to forecast the behaviour during indentation experiments, following Giannakopoulos and Suresh methodology$^{15}$. After analyzing either Vickers or Berkovich indentation tests on a wide range of materials, the following criterion is established $\Delta$:

$$\Delta = \frac{h'_r}{h'_t} \quad (1.21)$$

With $h'_r$ and $h'_t$ residual contact depth and maximum depth after applying a compliance correction. Three preponderant deformation modes are distinguished:

- $\Delta = 0.83$ no deformation mode is preponderant;
- $\Delta < 0.83$ implies sink-in formation;
- $\Delta > 0.83$ implies pile-up formation.

Giannakopoulos and Suresh founded a critical value for a similar criterion about 0.875$^{15}$.

### 1.3.2 Dynamic nanoindentation

The dynamic indentation is when a small dynamic oscillation (usually 2nm of amplitude) with a given frequency ($\omega$) (usually 45Hz) is imposed on the force (or displacement) signal. The amplitude of the displacement (or load) and the phase angle between the force and displacement signals ($\phi$) are measured using a frequency-specific amplifier$^{44}$ and$^{59}$. This technique allows to calculate the elastic stiffness and so the elastic properties continuously during the loading of the indenter$^{45}$, $^{31}$. This technique is named Continuous Stiffness Measurement (CSM) for Agilent - MTS nanoindenter and Dynamic Mechanical Analysis (DMA) (using the CMX control algorithms) for Hysitron nanoindenter.

With $m$ the mass of the indenter column, $C$ the harmonic contact damping in N.s/m, $C_s$ the system damping coefficient in N.s/m, $K_s$ the stiffness of the indenter support springs in N/m and $K_f$ the stiffness of the load frame in N/m. $C_\omega$ and $C_{\text{text}}\omega$ represent respectively the tip–sample damping factor and the system damping coefficient.

Values $m$, $C_s$, $K_s$ and $K_f$ are function of the equipment used and are determined during calibration process.

\[ S = \left[ \frac{F_c}{h_t} \cos \phi - (K_s - m\omega^2) - \frac{1}{K_I} \right]^{-1} \] (1.22)

\[ C\omega = \frac{F_c}{h_t} \sin \phi - C_s\omega \] (1.23)

This solution allows to determine the material properties as a continuous function of the indentation depth, but Pharr et al. have highlighted the influence of displacement oscillation on the basic measured quantities. According to the authors, “the sources of the measurement error have their origin in the relative stiffness of the contact and its relation to the displacements that can be recovered during the unloading portion of the oscillation”. Based on that, the authors proposed the following corrections to determine the actual load \( F_{c,\text{act}} \), the actual displacement \( h_{t,\text{act}} \) and the actual stiffness \( S_{\text{act}} \):

\[ F_{c,\text{act}} = F_c + \frac{\Delta F_c}{2} = F_c + \sqrt{2} \Delta F_{c,\text{rms}} \] (1.24)

\[ h_{t,\text{act}} = h_t + \frac{\Delta h_t}{2} = h_t + \sqrt{2} \Delta h_{t,\text{rms}} \] (1.25)

\[ S_{\text{act}} = \frac{1}{\sqrt{2\pi}} \left( \frac{1}{K} \right)^\frac{1}{m} \left[ 1 - \left( 1 - S \frac{2\sqrt{2} \Delta h_{t,\text{rms}}}{F_{c,\text{max}}} \right)^\frac{1}{m} \right] \frac{F_{c,\text{max}}}{\Delta h_{t,\text{rms}}} \] (1.26)

With \( K \) and \( m \) constants determined from unloading curves. These constants are related by the following equation:

\[ K = \left( \frac{2}{m\sqrt{\pi}} \right)^m \] (1.27)

Pharr and Bolshakov founded a value of 1.380 for \( m \) after many Berkovich indentation tests on a variety of materials. Thus, a value of 0.757 is used for the constant \( K \), using (1.27).

Find here the Matlab function to calculate the corrections to apply on depth, load and stiffness during dynamic nanoindentation: \texttt{CSM\_correction.m}.

Find here the Matlab function to calculate the constant \( K \) as a function of \( m \): \texttt{unload\_k\_m.m}.

---

\(^{50}\) Pharr G.M. et al., “Critical issues in making small-depth mechanical property measurements by nanoindentation with continuous stiffness measurement” (2009).
1.3.3 Extraction of elastic properties

Bulychev et al.\textsuperscript{3} and Shorshorov M. K. et al.\textsuperscript{54} were the first to determine the reduced Young’s modulus (or elastic modulus or storage modulus) of a material with the relationships established by Love\textsuperscript{34}, Galin\textsuperscript{13} and Sneddon\textsuperscript{53}, between the applied load and the displacement during an indentation test of an elastic material.

They proposed to express the reduced Young’s modulus $E^*$ (in GPa = N/m\textsuperscript{2}) as a function of the contact area and the contact stiffness:

$$E^* = \frac{1}{2} \sqrt{\frac{\pi}{A}} S$$

Then, Oliver and Pharr\textsuperscript{48,45} democratized this formulae after introducing a correction factor identified by King\textsuperscript{28}:

$$E^* = \frac{1}{2\beta} \sqrt{\frac{\pi}{A}} S$$

With $\beta$ a geometrical correction factor equal to:

- 1 for circular indenters (e.g.: conical and spherical indenter);
- 1.034 for three-sided pyramid indenters (e.g.: Berkovich indenter);
- 1.012 for four-sided pyramid indenters (e.g.: Vickers indenter).

Woirgard has demonstrated analytically that the exact value of $\beta$ for the perfectly sharp Berkovich indenter should be 1.062\textsuperscript{56}.

\textsuperscript{34} Love A.E.H., “Boussinesq’s problem for a rigid cone.” (1939).
\textsuperscript{56} Troyon M. and Lafaye S., “About the importance of introducing a correction factor in the Sneddon relationship for nanoindentation measurements” (2002).
Some authors proposed another correction factor function of the angle of the conical indenter and the Poisson’s ratio of the indented material\textsuperscript{21} and\textsuperscript{55}. For a conical indenter with an half-angle of $\gamma \leq 60^\circ$ (e.g.: Cube-Corner indenter), the analytical approximation is:

$$\beta = 1 + \frac{(1 - 2\nu)}{4 (1 - \nu) \tan \gamma}$$  \hspace{1cm} (1.30)

For a conical indenter with larger half-angle (e.g.: Berkovich indenter), the analytical approximation is:

$$\beta = \pi \frac{\pi/4 + 0.1548 \cot \gamma \frac{1-2\nu}{4(1-\nu)}}{\left[\pi/2 - 0.8311 \cot \gamma \frac{1-2\nu}{4(1-\nu)}\right]^2}$$  \hspace{1cm} (1.31)

With $\nu$ the Poisson’s ratio of the indented material.

Find here the Matlab function to plot the $\beta$ function of Hay et al.: \texttt{beta\_hay.m}.

![Figure 1.21: Plots of beta Hay: a) as a function of the half-angle of the conical indenter (for a Poisson’s ratio of 0.3), and b) as a function of the Poisson’s ratio for a Berkovich indenter.](image)

Knowing the material properties of the indenter, it is possible to calculate the reduced Young’s modulus $E'$ (in GPa = N/m$^2$) of the indented material.

$$\frac{1}{E'} = \frac{1}{E'} - \frac{1}{E_i}$$  \hspace{1cm} (1.32)

$$E = E' (1 - \nu^2)$$  \hspace{1cm} (1.33)

$$E_i = \frac{E_i}{(1 - \nu_i^2)}$$  \hspace{1cm} (1.34)

With $\nu$ the Poisson’s ratio of the indented material and $\nu_i$ the Poisson’s ratio of the material of the indenter.

\textbf{Note:} This method used to analyze indentation data is based on equations valid for isotropic homogeneous elastic solids.

Find here the Matlab function to calculate the Young’s modulus: \texttt{model\_elastic.m}.


\textsuperscript{55} Strader J.H. et al., “An experimental evaluation of the constant $b$ relating the contact stiffness to the contact area in nanoindentation.” (2006).
1.3.4 Extraction of viscoelastic properties

Viscoelasticity of a material can be characterized locally by nanoindentation using CSM or DMA techniques\(^{57,44}\) and\(^{59}\). This technique also called nano-DMA is a suitable technique for mechanical characterization of polymers and is complementary to traditional macroscale DMA and ThermoMechanical Analysis (TMA).

If dynamic nanoindentation is performed, a sinusoidal input is applied and the output signal is monitored. But, in case of a linear viscoelastic material, the output signal, which is still sinusoidal, can lag the input signal, and it is convenient to express the overall constitutive behavior in terms of the complex (shear) modulus \(E^*\) in GPa \(= N/m^2\) given by:

\[
E^* = \frac{\tau_A}{\gamma_A} = E'^* + iE''^* \tag{1.35}
\]

with \(\tau_A\) and \(\gamma_A\), respectively the shear-stress amplitude (in GPa = N/m²) and strain amplitude (dimensionless), with \(E'^*\) the reduced storage modulus and \(E''^*\) the reduced loss modulus (both in GPa = N/m²), and with \(i^2 = -1\). The storage modulus is in phase with the deformation and related to the elastic behavior. The storage modulus is defined using \(1.29\). Thus, the reduced loss modulus \(E''^*\) is the out-of-phase component and related to the viscous behavior. \(E^*\) is characteristic of internal damping (equal to a loss of energy due to internal friction) and defined by the following equation:

\[
E''^* = \frac{C\omega}{2} \sqrt{\frac{\pi}{A}} \tag{1.36}
\]

with \(C\omega\) the contact damping, given by \(1.23\).

It is convenient to calculate the loss tangent \((\tan(\delta))\) also called the loss factor \((\eta)\) or phase angle (dimensionless), which is defined by:

\[
\tan(\delta) = \eta = \frac{E'^*}{E''^*} = \frac{C\omega}{S} \tag{1.37}
\]

with \(\delta\) the phase-shift angle.

Figure 1.22: Vector diagram illustrating the relationship between complex shear modulus, storage modulus and loss modulus using the phase-shift angle. The elastic portion of the viscoelastic behavior is presented on the x-axis and the viscous portion on the y-axis.

Find here the Matlab function to calculate the loss modulus: loss_modulus.m.

Find here the Matlab function to calculate the loss tangent or loss factor: loss_tangent.m.
1.3.5 Extraction of plastic properties

The hardness $H$ (in GPa = N/m$^2$) of the material is defined according to Oliver and Pharr, by the following expression:

$$H = \frac{F_{c,\text{max}}}{A_c}$$ (1.38)

Find here the Matlab function to calculate the hardness: model_hardness.m.

1.3.6 Energy approach

Another way to access indentation data is the use of the energy $W_{\text{tot}}$ (in J = N/m) dissipated during the indentation. The elastic $W_e$ and plastic $W_p$ energies are respectively based on the integrals of the loading and unloading curves (see Figure 1.23)° and

$$W_{\text{tot}} = \int_{0}^{h_t} F_e \, dh$$ (1.39)

$$W_e = \int_{h_t}^{h_r} F_e \, dh$$ (1.40)

$$W_p = W_{\text{tot}} - W_e$$ (1.41)

The “trapz” Matlab function is used to calculate the area below the load-displacement curve: trapz.m.

1.3.7 Indentation recovery index

From the load-displacement curve, it is possible to define a parameter known as indentation recovery index ($\eta_i$). This index is defined by the ratio between recoverable or elastic deformation energy and the total deformation energy:

$$\eta_i = \frac{W_e}{W_{\text{tot}}} = \frac{W_{\text{tot}} - W_p}{W_{\text{tot}}} = \frac{h_t - h_r}{h_t}$$ (1.42)

Higher value of the indentation recovery index indicates greater capability to accommodate deformation during indentation test.

Find here the Matlab function to calculate the indentation recovery index: recovery_index.m.

---


1.3.8 Methodology to extract properties without the function area

The ratio of the irreversible work $W_{\text{tot}} - W_e$ to the total work $W_{\text{tot}}$, appears to be a unique function of the Young’s modulus and the hardness of the material, independent of the work-hardening behavior\(^7\).

\[
\frac{W_{\text{tot}} - W_e}{W_{\text{tot}}} = 1 - 5 \frac{H}{E^*} \quad (1.43)
\]

Then, combining the expression of the reduced Young’s modulus (1.29) with the expression of the hardness (1.38), leads to the following equation\(^26\) and\(^46\):

\[
\frac{\beta^2 4}{\pi} \frac{F_{c,\text{max}}}{S^2} = \frac{H}{E^*} \quad (1.44)
\]

The $\beta$ is initially not present in the equation given by\(^26\) or assumed to be equal to 1 in\(^46\).

These two last equations represent two independent relations that can be solved for $H$ and $E^*$ in a manner that does not directly involve the contact area.

The equation (1.44) is used as well to determine coefficients of the function area (1.19). Based on the assumption that the hardness and the Young’s modulus remain constant during indentation test in fused silica (isotropic material), the evolution of the ratio $\frac{F_{\text{max}}}{S^2}$ should stay constant as well in function of the indentation depth.

In 2012, Guillonneau et al. proposed a model to extract mechanical properties without using the indentation depth\(^16\) and\(^17\). The method is based on the detection of the second harmonic for dynamic indentation testing. This model is interesting especially for penetration depths in the range of 25 to 100nm, where the uncertainties related to the displacement measurement disturb a lot.

1.3.9 Methodology to minimize displacement measurement uncertainties using dynamic nanoindentation testing

Guillonneau et al. proposed a methodology to minimize displacement measurement uncertainties using dynamic nanoindentation\(^18\). The following equations are developed using respectively Loubet’s (1.17) and Oliver and Pharr’s models (1.16). Hardness and elastic modulus can be calculated independently of the indentation depth and the tip defect.

\[
h_c = \alpha \left( h - \frac{H\pi\tan(\theta)}{2E^*} h_c + h_0 \right) \quad (1.45)
\]

\[
\frac{dh_c}{dh} = \frac{\alpha}{1 + \frac{\alpha H\pi\tan(\theta)}{2E^*}} \quad (1.46)
\]

In this expression, $\frac{dh_c}{dh}$ can be determined by a simple linear fit of the plot $h_c = f(h)$. Thus, $\frac{H}{E^*}$ can be computed as:

\[
\frac{H}{E^*} = \frac{2}{\pi\tan(\theta)} \left( \frac{1}{\frac{dh_c}{dh}} - \frac{1}{\alpha} \right) \quad (1.47)
\]


\(^16\) Guillonneau G. et al.,“Extraction of mechanical properties with second harmonic detection for dynamic nanoindentation testing.” (2012).

\(^17\) Guillonneau G. et al.,“Determination of mechanical properties by nanoindentation independently of indentation depth measurement.” (2012).
Then, $E'^*$ can be calculated using (1.44) and $H$ knowing $E'^*$

$$E'^* = \frac{S^2}{2F_c \tan(\theta)} \left( \frac{1}{\frac{dh}{dx}} - \frac{1}{\alpha} \right)$$

and

$$H = \frac{4F_c E'^*}{\pi S^2}$$

(1.48) and (1.49) can be extended to the Oliver and Pharr’s contact model (1.16).

$$E'^* = \frac{S^2}{2\epsilon F_c \tan(\theta)} \left( \frac{1}{\frac{dh}{dx}} - \frac{1}{\alpha} \right)$$

and

$$H = \frac{4F_c E'^*}{\pi S^2}$$

(1.50) and (1.51)

Find here the Matlab function to calculate elastic modulus using Guillonneau’s methodology:
model_elasticModulus_Guillonneau.m.

Find here the Matlab function to calculate hardness using Guillonneau’s methodology:
model_hardness_Guillonneau.m.

### 1.3.10 Indentation Size Effect

The indentation size effect (ISE) is an hardness increase observed at small scales during nanoindentation tests, in other words materials will actually be harder at the small scale, than at the macro-scale. At macro-scale, materials hardness depends mainly on the level of statistically stored dislocations (SSD) which are created by homogeneous strain and are dependent upon the material and processing conditions. At micro-scale, hardness measurement is disturbed by the creation of geometrically necessary dislocations (GND), in addition to the dislocations statistically present, to maintain continuity within the material. As a conclusion, small indents have higher strain gradients relative to the size of the plastic zone and therefore have a higher measured hardness,$^{52, 43, 39}$ and$^{51}$.

![](image)

### 1.3.11 References

### 1.4 Models for thin films

The following parts give a short overview of models existing in the literature used for the extraction of mechanical properties of thin films deposited on a substrate from indentation experiments with conical indenters.

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Before everything, it is worth to mention the work performed by Jennett N.M. and Bushby A.J.\textsuperscript{26}, about nanoindentation test on coatings, during the European project INDICOAT (SMT4-CT98-2249).

Progress from this project help in the development of the ISO standard (ISO 14577 - 1-4). The ISO 14577 - 4 is dedicated to nanoindentation on coatings.


Some authors overviewed/reviewed already the nanoindentation technique applied to coatings:

- Fischer-Cripps, A.C., “Nanoindentation 3rd Ed.” (2011)

1.4.1 Nanoindentation tests on thin films

Composite reduced Young’s modulus and composite hardness

For indentation test on a coated specimen or on a multilayer sample (e.g.: thin films deposited on a substrate), the evolution of the Young’s modulus or the hardness calculated with models used for bulk materials, is function of the material properties and the thickness \( t \) (in m) of each underlying film (substrate included), and the properties and the geometry of the indenter.

Thus, the composite reduced Young’s modulus \( E' \) and the composite hardness \( H \) calculated with the models used for bulk materials, can generally be expressed as a combination of respectively the reduced Young’s moduli \( (E'_i) \) or the hardness \( (H_i) \) of each underlayer and respectively the reduced Young’s modulus \( (E'_s) \) or the hardness \( (H_s) \) of the substrate. The reduced Young’s moduli and the hardness are in GPa.

\[
E' = f(E'_{i\rightarrow N}, t_{i\rightarrow N}, E'_s) \tag{1.52}
\]
\[
H = f(H_{i\rightarrow N}, t_{i\rightarrow N}, H_s) \tag{1.53}
\]

Figure 1.24: *SEM cross-sectional observation of a multilayer sample.*

![SEM cross-sectional observation of a multilayer sample](image)

Figure 1.25: *Typical evolution of Young’s modulus and hardness for a coated specimen as a function of the normalized indentation depth.*

![Typical evolution of Young’s modulus and hardness](image)

With $i$ the indice of the layer and $N$ the total number of layers.

Finally, it is important to avoid confusion between indentation size effect and a decrease of hardness as a function of indentation depth observed in the case of a hard thin film deposited on a soft substrate. It might happen that both effects occur during indentation experiment, which complicates the analysis.

### Indentation contact topography

For nanoindentation tests on thin films, the contact topography is function of both thin film and substrate properties.

Figure 1.26: *Schematic depiction of a) “pile-up” and b) “sink-in” observed during thin film indentation.*

![Schematic depiction of a) “pile-up” and b) “sink-in”](image)

The Figure 1.26 a (“pile-up”) is typical of the case of a soft film on a hard substrate and the Figure 1.26 b (“sink-in”) of a hard film on a soft substrate. The pile-up can be emphasized in case of thin films, because of the material confinement by the substrate. To determine the depth of contact, the same models described for bulk material indentation are used, especially when the ratio of the indentation depth over the coating thickness remains low (<10%, see the rule proposed by Zhang F. et al., “Indentation of a hard film on a soft substrate: Strain gradient hardening effects” (2007).

by Bückle). But, some quantitative approaches are proposed in the literature to take into account both pile up and substrate effects in the material properties estimation of the coating.  

**Corrections to apply for thin film indentation**

During nanoindentation tests of thin film on substrate, the thickness of the film beneath the indenter is smaller than its original value, because of plastic flow during loading. The use of the original film thickness \( t \) in the regression model cause a systematic shift or distortion of the Young’s modulus curve. A correction proposed by Menčík et al. can be applied, assuming a rigid substrate and determining the effective thickness \( t_{\text{eff}} \) (in m) by

\[
\pi a^2 t_{\text{eff}} = \pi a^2 t - V
\]

With \( V \) the volume displaced by the indenter and approximated by \( \pi a^2 h_c/3 \), for a conical indenter and contact depths \( h_c \) smaller than the film thickness.

\[
t_{\text{eff}} = t - \frac{h_c}{3}
\]

Figure 1.27: Indentation penetration of a thin film on a sample.

Recently, Li et al. proposed to express the local thinning effect as:

\[
t_{\text{eff}} = t - \eta h_c
\]

With \( \eta \) a parameter depending on the mechanical properties of the film and the substrate, and on the geometry of the indenter. Preliminary finite element calculations show that \( \eta \) should be independent of indentation depth and that its value ranges from 0.3 to 0.7 for materials that do not work harden.

**Method of the tangent**

First of all, the easiest way to assess approximately the mechanical properties of the film is to use the tangential model. If one plots a tangent from the diagram’s zero point to the curves, then the tangential point is the Young’s modulus or the hardness of the film (see Figure 1.28).

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56 Zhou X. et al., “Investigation on methods for dealing with pile-up errors in evaluating the mechanical properties of thin metal films at sub-micron scale on hard substrates by nanoindentation technique.” (2008).


34 Li H. et al., “New methods of analyzing indentation experiments on very thin films” (2010).
1.4.2 Elastic properties of a thin film on a substrate

Several models to analyse indentation tests on bilayer sample and multilayer sample and to extract intrinsic material properties of the upper film (or the top coating) are detailed in the following part.

Some of the following models developed are detailed in the chapter 8 (“Nanoindentation of Thin Films”) of the book “Nanoindentation” written by A.C. Fischer-Cripps.\(^{17}\)

**Bückle (1961)**

Bückle proposed an empirical law (“rule of thumb”) to characterize thick coatings on a substrate.\(^5\) It is possible to estimate the Young’s modulus of the coating for indentation depth lower than 10 μm.

**Doerner and Nix (1986)**

The model of Doerner and Nix is detailed in many papers\(^{14,31,43}\) and\(^{48}\), and is described by the following equation:

\[
\frac{1}{E'} = \frac{1}{E_f'} + \left( \frac{1}{E_s'} - \frac{1}{E_f'} \right) e^{-\alpha(x)}
\]

(1.57)

With \(x = t/h_c\) and \(\alpha\) an empirically constant determined using the method of least squares.

The equation was modified by King\(^{31}\) with the replacement of \(t/h_c\) by \(t/\alpha_c\), and then by Saha and Nix\(^{48}\) with the replacement of \(t/h_c\) by \((t - h_c)/\alpha_c\).

An empirical formulae based on the model of Doerner and Nix was proposed by Chen et al. in 2004\(^{12}\):

\[
\frac{1}{E'} = \frac{1}{E_f'} \left[ 1 - e^{-\alpha(x)(2/3)} \right] + \frac{1}{E_s'} e^{-\alpha(x)(2/3)}
\]

(1.58)

With \(x = t/h_c\) and \(\alpha\) an empirically constant determined using the method of least squares.

Find here the Matlab function for the Doerner and Nix model\(^{14}\): `model_doerner_nix.m`.

Find here the Matlab function for the Doerner and Nix model modified by King\(^{31}\): `model_doerner_nix_king.m`.

\(^{17}\) Fischer-Cripps, A.C., “Nanoindentation 3rd Ed.” (2011)


\(^{14}\) Doerner M.F. and Nix W.D., “A method for interpreting the data from depth-sensing indentation instruments” (1986).


Find here the Matlab function for the Doerner and Nix model modified by Saha: model_doerner_nix_saha.m.
Find here the Matlab function function for the Doerner and Nix model modified by Chen: model_chen.m.

Gao et al. (1992)

The model of Gao is described by the following equation:

\[ E' = E'_s + \left( E'_t - E'_s \right) \phi_{Gaoo} (x) \]  
\[ \phi_{Gaoo} = \frac{2}{\pi} \arctan \left( \frac{1}{x} \right) + \frac{1}{2\pi (1 - \nu_c)} \left[ \left( 1 - 2\nu_c \right) \frac{1}{x} \ln \left( 1 + x^2 \right) - \frac{x}{1 + x^2} \right] \]  
\[ \nu_c = 1 + \left[ \frac{(1 - \nu_s) (1 - \nu_f)}{1 - (1 - \phi_{Gaoo}) \nu_f - \phi_{Gaoo} \nu_s} \right] \]  

With \( \nu_c \) the composite Poisson’s ratio, \( \nu_s \) the Poisson’s ratio of the substrate and \( \nu_f \) the Poisson’s ratio of the thin film.

\[ \phi_{Gaoo} = \frac{2}{\pi} \arctan \left( \frac{1}{x} \right) + \frac{1}{x\pi} \ln \left( 1 + x^2 \right) \]  

With \( x = a_c/t \).

Find here the Matlab function for the weighting function \( \phi_{Gaoo} \) : phi_gao_0.m.
Find here the Matlab function for the weighting function \( \phi_{Gaoo} \) : phi_gao_1.m.
Find here the Matlab function for \( \nu_c \) the composite Poisson’s ratio : composite_poissons_ratio.m.
Find here the Matlab function for the Gao et al. model : model_gao.m.

Menčík et al. (1997)

Menčík et al. proposed the following structures to express the combination of \( E'_t \) and \( E'_s \).

\[ E' = E'_s + \left( E'_t - E'_s \right) \phi (x) \]  
\[ E' = E'_s + \left( E'_t - E'_s \right) \psi (x) \]  

Where \( x \) is the ratio of the contact radius (\( a_c \)) or the contact depth (\( h_c \)), to the film thickness (\( t \)), and \( \phi \) and \( \psi \) are weight functions of the relative penetration \( x \). \( \phi \) is equal to 1 when \( x \) is equal to 0 and 0 when \( x \) is infinite.

Note: If the difference between Poisson’s ratio of the thin film and substrate is small, the values for uniaxial loading Young’s moduli, \( E, E_t, E_s \) can be used in previous equation.

---

Menčík et al. (linear model) (1997)

Menčík described the linear model by the following expression:

\[ E' = E'_f + \left( E'_s - E'_f \right) (x) \]  \hspace{1cm} (1.65)

With \( x = \frac{a_c}{t} \).

Find here the Matlab function for the Menčík et al. linear function: \texttt{model\_menick\_linear.m}.

Menčík et al. (exponential model) (1997)

Menčík described the exponential model by the following expression:

\[ E' = E'_s + \left( E'_f - E'_s \right) e^{-\alpha(x)} \]  \hspace{1cm} (1.66)

With \( x = \frac{a_c}{t} \) and \( \alpha \) is an empirically constant determined using the method of least squares.

Find here the Matlab function for the Menčík et al. exponential function: \texttt{model\_menick\_exponential.m}.

Menčík et al. (reciprocal exponential model) (1997)

Menčík described the reciprocal exponential model by the following expression:

\[ \frac{1}{E'} = \frac{1}{E'_s} + \frac{1}{E'_f} - \frac{1}{E'_s} \left( e^{-\alpha(x)} \right) \]  \hspace{1cm} (1.67)

With \( x = \frac{a_c}{t} \) and \( \alpha \) is an empirically constant determined using the method of least squares.

Find here the Matlab function for the Menčík et al. reciprocal exponential function: \texttt{model\_menick\_reciprocal\_exponential.m}.

Perriot et al. (2003)

The following system of equation describes the model developed by Perriot et al.:

\[ E' = E'_f + \frac{E'_s - E'_f}{1 + \left( \frac{km}{x} \right)^n} \]  \hspace{1cm} (1.68)

\[ \log(k_0) = -0.093 + 0.792 \log \left( \frac{E'_s}{E'_f} \right) + 0.05 \left[ \log \frac{E'_s}{E'_f} \right]^2 \]  \hspace{1cm} (1.69)

With \( x = \frac{a_c}{t} \), and \( k_0 \) and \( n \) are adjustable constants determined using the method of least squares.

Find here the Matlab function for the Perriot et al. model: \texttt{model\_perriot\_barthel.m}.

\footnote{Perriot A. and Barthel E., “Elastic contact to a coated half-space: Effective elastic modulus and real penetration” (2004).}
Jung et al. (2004)

Jung et al.\textsuperscript{29} have adapted for conical indentation of thin films, the simple empirical approach of Hu and Lawn\textsuperscript{23} developed initially for spherical indentation on bilayer structures. The following power-law relationship allows the evaluation of the Young’s modulus of a thin film deposited on a substrate from nanoindentation experiments:

\begin{equation}
E = E_s \left( \frac{E_f}{E_s} \right)^L
\end{equation}

with \( L \) is the exponent term described by a dimensionless function:

\begin{equation}
L = \frac{1}{1 + Ax^B}
\end{equation}

With \( x = h_c/t \) and where \( A \) and \( B \) are adjustable coefficients.

Jung et al. founded \( A = 3.76 \) and \( B = 1.38 \) after regression fits of (1.70) to different data sets. These coefficients are not universal and need to be “calibrated” with experimental data or with finite element data for specified material systems.

Finally, to be more consistent with other analytical models implemented in this toolbox, the model of Jung is modified by using the reduced form of the Young’s moduli:

\begin{equation}
E' = E'_s \left( \frac{E'_f}{E'_s} \right)^L
\end{equation}

Find here the Matlab function for the sigmoidal function used in the Jung’s model: sigmoidal_jung.m.

Find here the Matlab function for the Jung et al. model: model_jung.m.

Bec et al. (2006)

The elastic model of Bec et al. is based on indentation by a rigid cylindrical punch (radius \( a_c \)) of a homogeneous film deposited on a semi-infinite half space\textsuperscript{4}.

This system is modelled by two springs connected in series:

Find here the Matlab function for the Bec et al. model: model_bec.m.

Korsunsky and Constantinescu (2009)

Korsunsky and Constantinescu proposed a simple model response function to the analysis of indentation of elastic coated systems\textsuperscript{32} and\textsuperscript{33}. They expressed the reduced Young’s modulus in terms of a linear law of mixtures of the form:

\begin{equation}
E' = E'_1 + \frac{E'_2 - E'_1}{1 + \left( \frac{h}{b_0} \right) \eta}
\end{equation}


Figure 1.29: Schematic description of the bilayer model of Bec et al.

\[ K_f = \frac{\pi a}{2c} E'_f \]  
\[ K_s = 2aE'_s \]  
\[ K_z = 2aE'_z \]  
\[ \frac{1}{K_z} = \frac{1}{f_t(a_c)K_f} + \frac{1}{f_s(a_c)K_s} \]  
\[ f_t(a_c) = f_s(a_c) = 1 + \frac{2t}{\pi a_c} \]  
\[ \frac{1}{2aE'_c} = \frac{t}{(\pi a_c^2 + 2t a_c) E'_f} + \frac{1}{2 \left( a_c + \frac{2t}{\pi} \right) E'_s} \]  

Here \( E'_1, E'_2, \eta \) and \( \beta_0 \) are positive constants to be determined from fitting. It may be expected that for very shallow indentation \( (\beta_0 \ll 1) \), the corresponding parameter \( E'_1 \) ought to approach the Young’s modulus of the coating \( E'_f \). Similarly, one might also expect that for very deep indentation \( (\beta_0 \gg 1) \), the corresponding parameter \( E'_2 \) ought to approach the Young’s modulus of the substrate \( E'_s \). Values of \( \eta \) and \( \beta_0 \) depend on the indentation boundary condition, if the coating is defined as a freely sliding layer or as a perfectly bonded layer.

**Hay et al. (2011)**

The present model of Hay et al.\(^{21}\) is a development of the Song–Pharr model\(^{46,54}\), which is already inspired by the Gao model\(^{18}\).

\[ \frac{1}{\mu_c} = (1 - \phi_{Gao}) \frac{1}{\mu_s + F\phi_{Gao}\mu_t} + \phi_{Gao} \frac{1}{\mu_t} \]  

Where \( \mu_c \) (in GPa = N/m\(^2\)) is the composite shear modulus calculated from the composite Young’s modulus as:

\[ \mu_c = \frac{E}{2(1 + \nu_c)} \]  

Where \( \nu_c \) is the composite Poisson’s ratio given in Gao’s model.

---


Knowing $\mu_c$, it is possible to calculate $\mu_f$:

$$\mu_f = -B + \sqrt{B^2 - 4AC}$$

Where $A = F\phi_{Ga_o}$

$$B = \mu_s - (F\phi_{Ga_o}^2 - \phi_{Ga_o} + 1)\mu_c$$

With $F = 0.0626$, a constant obtained from finite element simulations.

$$C = -\phi_{Ga_o}\mu_c\mu_s$$

$$\mu_s = \frac{E_s}{2(1 + \nu_s)}$$

Finally, the Young’s modulus of the film is calculated from the shear modulus and Poisson’s ratio of the film:

$$E_f = 2\mu_f(1 + \nu_f)$$

Find here the Matlab function for the Hay et al. model: model_hay.m.

**Bull (2014)**

This model was originally proposed by Bull S.J. in 2011, in a paper about the mechanical characterization of ALD Alumina coatings\(^7\). Then, this simple method to determine the elastic modulus of a coating on a substrate using nanoindentation was developed in another paper in 2014\(^8\). This model is based on the load support of a truncated cone of material beneath the indenter.

$$E' = \frac{F_c}{2a_c(h_c + h_s)}$$  \hspace{1cm} (1.88)

$$h_c = \frac{F_c}{\pi E_t} \left[ \frac{1}{a_c \tan \alpha} - \frac{1}{a_c \tan \alpha + t_c \tan^2 \alpha} \right]$$  \hspace{1cm} (1.89)

$$h_s = \frac{F_c}{\pi E_s} \left[ \frac{1}{a_c \tan \alpha + t_c \tan^2 \alpha} - \frac{1}{a_c \tan \alpha + (t_c + t_s) \tan^2 \alpha} \right]$$  \hspace{1cm} (1.90)

Where $E_t$ and $E_s$ are the Young’s Modulus of the coating and substrate, $t_c$ and $t_s$ are the coating and substrate thickness, and $\alpha$ is the semi-angle of the cone material which supports the load. In fact, by assuming that the material thickness is very much greater than the contact radius, it is possible to replace in the previous equation $\tan \alpha$ by $2\pi = 32.48$. Finally, by assuming that the substrate is very much thicker than the coating ($t_s >> t_c$), the equation (1.88) can be rewritten:

\(^7\) Bull S.J., “Mechanical response of atomic layer deposition alumina coatings on stiff and compliant substrates” (2011).

\[
E' = \frac{1}{\frac{1}{E'_f} \left[ \frac{2t_f}{\pi a_c + 2t_f} \right] + \frac{1}{E'_s} \left[ \frac{\pi a_s}{\pi a_s + 2t_s} \right]},
\]  

(1.91)

This last equation from Bull S.J. is exactly the same as equation (1.78), proposed by Bec et al. in 2006, with a unique difference which is the use by Bull S.J. of the non reduced form of the Young’s moduli of the coating and the substrate.


Argatov I.I. and Sabina F.J. proposed an analytical model, based on the first-order asymptotic solution to the unilateral frictionless indentation problem. This model takes into account the effect of a compliant substrate in the small-scale indentation of thin coatings\(^1\).

### 1.4.3 Elastic properties of a thin film on a multilayer system

In 2008, Pailler-Mattei et al. proposed an extension of the Bec’s model to a bilayer system deposited on a substrate\(^{41}\). But more recently, Mercier et al. established a generalization of the Bec’s model to \(N + 1\) layers sample.

**Mercier et al. (2010)**

The elastic model of Mercier et al. for a multilayer sample on \(N + 1\) layers is an extension of the Bec et al. model\(^{37}\) and\(^{38}\).

\[
\frac{1}{2a_{c,0}E'} = \sum_{i=0}^{N} \left( \frac{t_i}{\pi a_{c,i}^{2} + 2t_i a_{c,i}} \right) E_{c,i}' + \frac{1}{2 \left( a_{c,N} + \frac{2t_N}{\pi} \right) E'_s},
\]  

(1.92)

\[
a_{c,i+1} = a_{c,i} + \frac{2t}{\pi}
\]  

(1.93)

With \(a_{c,0}\) equal to \(a_c\).

Thus, the Young’s modulus of the film can be calculated as:

\[
E'_{c,0} = \left[ \frac{\pi a_{c,0}^{2} + 2t_0 a_{c,0}}{t_0} \left[ \frac{1}{2a_{c,0}E'} - \left( \sum_{i=1}^{N} \left( \frac{t_i}{\pi a_{c,i}^{2} + 2t_i a_{c,i}} \right) E_{c,i}' + \frac{1}{2 \left( a_{c,N} + \frac{2t_N}{\pi} \right) E'_s} \right) \right] \right]^{-1}.
\]  

(1.94)

It is advised to perform nanoindentation tests on each layer of the multilayer sample, from the substrate up to the final stack of layers (see Figure 1.31). By successive iterations using the model of Mercier et al., values of Young’s modulus of each layer are extracted from the contact stiffness.

Find here the Matlab function for the Mercier et al. model: `model_multilayer_elastic.m`.

---


\(^{37}\) Mercier D. et al., “Young’s modulus measurement of a thin film from experimental nanoindentation performed on multilayer systems” (2010).

Puchi-Cabrera et al. (2015)

Puchi-Cabrera et al. proposed in 2015, a description of the composite elastic modulus of multilayer coated systems[^44], based on the physically-based concept advanced by Rahmoun et al.[^45]:

\[
\frac{1}{E} = \sum_{i=1}^{N} \frac{x_i^*}{E_i} + \frac{x_N^*}{E_s}
\]  

(1.95)

With \(x_i^*\) and \(x_N^*\), respectively the volume fraction of each layer and the corresponding volume fraction of the substrate material. In his paper, Puchi-Cabrera modified and extended from the bilayer to the multilayer specimen, the models of Doerner and Nix, Gao, Bec, Menčík, Perriot and Barthel, Antunes, Korsunsky and Constantinescu and Bull.

### 1.4.4 Plastic properties of a thin film on a substrate

Bückle (1961)

Bückle proposed an empirical law ("rule of thumb") to characterize thick coatings on a substrate[^5].

It is possible to estimate empirically the hardness of the coating for indentation depth lower than "40.

Because of the imperfections of the indenters, the roughness and the surface pollution, it is more meaningful to use this rule of the "40.

In the paper of Rother and Jehn[^47], a good agreement with Bückle’s “rule of thumb” is found for a range of indentation depths over coating thickness ratios between 1/10 (10%) and 1/7 (14.3%).

According to Veprek-Heijman M.G.J. and Veprek S., the Bückle’s rule is not valid in the case of superhard coatings the deformation of a softer substrate, such as steel\(^{53}\). To obtain correct measurements of the hardness of superhard coatings (H \(\geq 40\) GPa) their thickness must be sufficiently large (typically 5–7 \(\mu m\) or more on steel and about 3 \(\mu m\) on Si or cemented carbide). In the case of ultrahard coatings (H \(\geq 80\) GPa), the thickness should be even larger.

In parallel, Bückle proposed an expression of the composite hardness \(H\) in the case of a two-layer material with a weighted sum of the different layer hardnesses during indentation process\(^5\):

\[
H = aH_f + bH_s
\tag{1.96}
\]

With \(H_f\) the hardness of the film, \(H_s\) the hardness of the substrate and \(a + b = 1\). \(a\) varies from 1 when the hardness is not affected by the substrate, to 0 when the indentation depth is approaching the film thickness.

**Kao and Byrne (1981)**

Kao and Byrne proposed the following model to describe the evolution of the composite hardness as a function of the reciprocal indentation depth\(^{30}\), based on Bückle’s model\(^5\):

\[
H \approx H_s + 2k_1 t_i (H_f - H_s) \frac{1}{\ell h}
\tag{1.97}
\]

With \(k_1\) a weighting factor of about 9%, independent of material characteristics.

Find here the Matlab functions for the model of Kao: - model_kao.m. - model_kao_film.m.

**Jönsson and Hogmark (1984)**

Jönsson and Hogmark used a simple geometrical approach based on a area “law of mixtures” to separate the substrate and film contributions to the measured hardness from Vickers indentation\(^{27}\).

\[
H = \frac{A_f}{A} H_f + \frac{A_s}{A} H_s
\tag{1.98}
\]

With \(A_f\) the area on which the mean pressure \(H_f\) acts and \(A_s\) the area on which the mean pressure \(H_s\) acts. The total area \(A\) is the sum of \(A_f\) and \(A_s\) and the following expressions for the area ratios are given by Jönsson and Hogmark:

\[
\frac{A_f}{A} = 2C \frac{t}{d} - C^2 \frac{t^2}{d}
\tag{1.99}
\]

\[
\frac{A_s}{A} = 1 - \frac{A_f}{A}
\tag{1.100}
\]

With \(d\) the diagonal of the indent, \(t\) the film thickness and \(C\) a constant equal to 0.5 for hard coatings on very soft substrates (6.3 < \(\frac{H_f}{H_s}\) < 12.9) or to 1 when the coatings and substrate hardnesses are more similar (1.8 < \(\frac{H_f}{H_s}\) < 2.3).

\(^{53}\) Veprek-Heijman M.G.J. and Veprek S., “The deformation of the substrate during indentation into superhard coatings: Bückle’s rule revised” (2016).


**Burnett and Rickerby (1984)**

Burnett and Rickerby proposed afterwards a model based on a “volume law of mixtures” similar to Jönsson’s relation, considering the volumes of the plastic zones, $V_f$ and $V_s$, respectively in the film and in the substrate$^{9,10}$.

$$H = \frac{V_f}{V} H_f + \frac{V_s}{V} H_s$$  \hspace{1cm} (1.101)

With $V = V_f + V_s$.

Later, Iost and Bigot proposed to compare the predictions obtained using Jönsson-Hogmark and Burnett-Rickerby models and to understand why it is often reported that the Jönsson and Hogmark model does not hold for indentation prints less than the coating thickness. Methods were reviewed for calculating the composite hardness, and it was found that the simplifications made by the authors are not always valid. By taking in account all the terms of the equations of Jönsson and Hogmark, it was found that the relation between the hardness and the reciprocal length of the indentation print is not linear and depends on the ratio between the film thickness and the indentation print, as well as the variation of the hardness of the substrate and the film with the applied load. Comparison of the Burnett and Rickerby experimental data with the modified model led to very good agreement$^{24}$.

**Bhattacharya and Nix (1988)**

Bhattacharya and Nix proposed the following model from numerical simulations to extract the hardness of a coating$^3$:

$$H = H_s + (H_f - H_s) e^{-\alpha(x)^n}$$  \hspace{1cm} (1.102)

With $x = h/t$ and $\alpha$ and is an adjustable constants determined using the method of least squares. The variable $n$ is equal to 2 when the coating is harder than the substrate and to 1 when the substrate is harder than the coating.

In the original paper, $\alpha$ is equal to $\frac{H_f}{\sigma_f/\sqrt{E_f/E_s}}$, when the coating is harder than the substrate and to $\frac{\sigma_f}{\sigma_s E_f/E_s}$, when the substrate is harder than the coating.

Find here the Matlab functions for the model of Bhattacharya and Nix : model_bhattacharya.m.

**“Volume law of mixtures”**

Several authors developed composite hardness model to calculate hardness of a coating using a volume law of mixture:

- Sargent (1986)$^{49}$
- Chicot and Lesage (1995)$^{13}$
- He et al. (1996)$^{22}$
- Korsunsky et al. (1996)$^{32}$


1.4. Models for thin films
Based on the methodology proposed by Joslin and Oliver (1990)\textsuperscript{28} for a bulk material, extended to the coated system by Page et al.\textsuperscript{40}, Saha and Nix proposed to use the following equation, giving the evolution of the hardness as a function of indentation depth, even when pile-up occurs\textsuperscript{48}:

\[
H = \beta^2 \frac{4}{\pi} \frac{F_{c,\text{max}}}{S^2} \left( E^* \right)^2
\]  
\[
\frac{1}{E^*} = \frac{1}{E_i} + \frac{1}{E_f} + \left( \frac{1}{E_s} - \frac{1}{E_f} \right) e^{-\alpha(x)}
\]  

With \( x = (t - h_c)/a_c \) and \( E_f^* \) the reduced Young’s modulus of the indenter.

This model was reused later by Han et al.\textsuperscript{19} and\textsuperscript{20}.

\textbf{Note:} This model is valid only for the case of elastically inhomogeneous film/substrate systems.

\textbf{Chen (2004)}

In this paper, Chen et al. proposed a modified volume fraction (CZ) model, in which a conical deformation volume shape is assumed, but the conical tip angle is undetermined. The conical tip angle contains the influences of pile-up or sink-in since the total depth is used in the modified CZ model. Due to the introduction of the conical angle, the modified CZ model could describe both a soft film on a hard substrate system and a hard film on a soft substrate system\textsuperscript{12}.

\textbf{Iost (2005)}

More recently, Iost et al. compared 8 different models in term of robustness, taking into account the indentation size effect on film and substrate proposed in the literature to estimate the hardness of coatings from indentations where both coating and substrate interact\textsuperscript{25}.

\textbf{1.4.5 Plastic properties of a thin film on a multilayer system}

\textbf{Engel et al. (1992)}

Engel et al. proposed a simple method of interpreting the superficial (Vickers) hardness of multilayered specimen\textsuperscript{15}, by expanding the concept of Jönsson and Hogmark\textsuperscript{27}:

\textsuperscript{28} Joslin D.L. and Oliver W.C., “A new method for analyzing data from continuous depth-sensing microindentation tests” (1990).
\textsuperscript{19} Han S.M. et al., “Combinatorial studies of mechanical properties of Ti–Al thin films using nanoindentation” (2005).
\textsuperscript{15} Engel P.A. et al., “Interpretation of superficial hardness for multilayer platings” (1992).
With $N$ the number of layers deposited on the substrate, $A_{f,i}$ and $H_{f,i}$ respectively the flow pressure area and the hardness of an intermediate layer. According to the author, this model is applicable as long as the ratio of the film thickness $t_i$ over the indentation imprint size (i.e. the diagonal $d$ of square imprint in case of Vickers indentation) is less than 0.2, resulting in a parallel displacement of indenter, layers, and substrate.

**Rahmoun et al. (2009)**

The paper written by Ramhoun et al. is about the description of a multilayer model, developed to estimate coating hardness evolution in case of a multilayer specimen. After some simplification, the following equations are established to take into account the hardness variation to the applied load and the crumbling of the coating. This model, based on the Jönsson and Hogmark’s area law of mixtures, makes it possible to calculate the minimum in hardness value which depends on the hardness of the different phases, the thickness of the coatings and the compaction of the layers under the load transmitted by the Vickers indenter. From this model (which has some features in common with the Kao and Byrne approach), it can be shown that the substrate begins to influence the coating hardness at a critical penetration depth equal to the sum of interlayer and top coating thickness.

\[
H = \frac{A_f}{A} H_f + \frac{A_i}{A} H_i + \frac{A_s}{A} H_s \tag{1.107}
\]

\[
\frac{A_f}{A} = \frac{2t_f}{h} - \frac{t_i^2}{h^2} \tag{1.108}
\]

\[
\frac{A_i}{A} = \frac{2t_i}{h} - \frac{t_f^2}{h^2} - \frac{2t_i t_f}{h^2} \tag{1.109}
\]

\[
\frac{A_s}{A} = 1 - \frac{2(t_f + t_i)}{h} + \frac{(t_f + t_i)^2}{h^2} \tag{1.110}
\]

With $t_f$ the coating thickness, $t_i$ the interlayer thickness, $h$ the indentation depth, $A$ the total contact area, $A_f$, $A_i$, and $A_s$ respectively the coating, the interlayer and substrate area transmitting the mean contact pressure, and $H_f$, $H_i$ and $H_s$ respectively the coating, the interlayer and substrate hardness.

**Arrazat et al. (2010)**

For complex multilayer samples, Arrazat et al. proposed a model based on the volume law of mixture proposed by Sargent et al. In principle, the composite hardness is equal to the sum of intrinsic hardness of all materials plastically deformed, weighted by plastically deformed volume in the different layers.

1.4.6 References

1.5 Minimization process

1.5.1 Least mean squares

Experimental curves are fitted with models using the least mean squares method. The method of least squares is a standard approach in regression analysis and is based on the resolution of the following problem, by finding the coefficients $x$:

$$
\min_x \| F(x, x_{data})y_{data} \|_2^2 = \min_x \sum_i (F(x, x_{data_i})y_{data_i})^2
$$

(1.111)

With given input data $x_{data}$ and the observed output $y_{data}$.

- Matlab Optimization toolbox.
- Matlab nonlinear curve-fitting.
- Least squares method.
- Curve fitting.
- Mathematical optimization.

Fischer-Cripps A.C. wrote a detailed section about the non-linear least squares fitting in his book “Nanoindentation” (pp. 253-256)

1.5.2 Residuals

The residuals from a fitted model are the differences between the response data and the corresponding prediction of the response computed using the regression function. Mathematically, the definition of the residual for the $i$th observation in the data set is written:

$$
r_i = y_i - \hat{y}_i
$$

(1.112)

With $y_i$ denoting the $i$th response value and $\hat{y}_i$ the $i$th predicted response value.

If the model fits the data correctly, the residuals approximate the random errors.

- Matlab residual analysis.
- Error and residuals.
- How can I tell if a model fits my data?

1.6 Examples of nanoindentation data

Please look at the experimental procedure proposed by Jennett N. M. and Bushby A. J. to perform nanoindentation tests on bulk, coatings or multilayer systems, and to the ISO standard (ISO 14577 - 1 to 4).

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1.6.1 Type of data - Pre-Requirements

Only data from Continuous Stiffness Measurement (CSM), given as a function of the indentation depth are accepted in the NIMS toolbox (e.g.: CSM mode for Agilent - MTS nanoindenter or DMA - CMX algorithm for Hysitron nanoindenter).

You data must only have the loading part from the load-displacement curves of your (nano)indentation results. In the case of data saved in a ‘Sample’ or ‘Analyst Project’ sheet of a .xls file obtained with ‘Analyst’ (MTS software) (containing at least a ‘Hold Segment Type’ or a ‘END’ segment), the toolbox is able to consider only the loading part of your results.

Please, check if the surface detection is well done, especially if the substrate is compliant\(^2\) and\(^4\). For more explanations about the surface detection, look into the NIMS documentation.

It is advised to use average results from at least 10 indentation tests to avoid artefacts (e.g. pop-in, roughness, local impurities or dust on the sample’s surface...).

Note: To analyze pop-in distribution, the Matlab PopIn toolbox was developed. The Matlab code is available on GitHub with the documentation.

1.6.2 Agilent - MTS example files

- Both .txt or .xls files are accepted.
- 3 columns (Displacement / Load / Stiffness)
- 6 columns (Disp. / SD (Disp.) / Load / SD (Load.) / Stiff. / SD (Stiff.)) (SD for Standard Deviation)
- It is possible to get directly these type of average sheet in your .xls file, using the ‘Analyst’ Excel macro provided by MTS.
- MTS_0film_Si_CSM-2nm_noSD.txt
  - Data for a bulk Silicon sample.
  - Data obtained by Berkovich indentation with CSM mode (75Hz / amplitude 2nm) (no standard deviation).
- MTS_1film_SiO2_Si_CSM-2nm.xls
  - Data for a thin film of Silicon thermal oxide (500nm) on a bulk Silicon sample.
  - Data obtained by Berkovich indentation with CSM mode (75Hz / amplitude 2nm).
- MTS_1film_SiO2_Si_CSM-2nm_noSD.xls
  - Data for a thin film of Silicon thermal oxide (500nm) on a bulk Silicon sample.
  - Data obtained by Berkovich indentation with CSM mode (75Hz / amplitude 2nm) (no standard deviation).
- MTS_2films_Al_SiO2_Si_CSM-2nm.xls
  - Data for a thin film of PVD Aluminum (500nm) deposited on a bulk Silicon sample with a Silicon thermal oxide (500nm).
  - Data obtained by Berkovich indentation with CSM mode (75Hz / amplitude 2nm).


1.6. Examples of nanoindentation data
1.6.3 Hysitron example files

- Both .txt or .dat files are accepted.
- **Hysitron_dma.txt**
  - Data obtained by Berkovich indentation with DMA mode (205Hz / amplitude 0.65nm) - Courtesy of Dr. Igor Zlotnikov from Max Planck Institute of Colloids and Interfaces in Potsdam, Germany.

1.6.4 References

- Agilent website
- Hysitron website

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3 Mercier D. et al., “Young's modulus measurement of a thin film from experimental nanoindentation performed on multilayer systems” (2010).
1.7 FEM model

1.7.1 Finite Element Modelling (FEM) of conical indentation

The present model is a simulation of the conical nanoindentation process, using the FEM software ABAQUS.

The Matlab function used to generate a Python script for ABAQUS is: `python_abaqus`

The model is axisymmetric with a geometry dependent mesh and restricted boundaries conditions.

**Geometry of the (multilayer) sample**

Each layer of the sample is characterized by its thickness ($t_i$).

The thickness of the substrate ($t_{\text{sub}}$) is set as 2 times the highest thin film thickness.

The width ($w$) of the sample is calculated as a function of the substrate thickness or the indenter tip defect.

No delamination is allowed between thin films or between thin film and substrate.

**Note:** Dimensions are in nm.

![Geometry of the sample in the FEM model.](image)

**Figure 1.33:** Geometry of the sample in the FEM model.

**Geometry of the indenter**

The indenter is defined as a rigid cono-spherical indenter. A spherical part is defined at the apex of the conical indenter (see Figure 1.34).

![Scheme of a cono-spherical indenter.](image)

**Figure 1.34:** Scheme of a cono-spherical indenter.
The radius $R$ of the spherical part is calculated from the tip defect $h_{\text{tip}}$ and the cone angle $\alpha$, using the following equation. For Berkovich, Vickers and Cube-Corner indenters, the equivalent cone angle is used to set the cone angle.

$$ R = \frac{h_{\text{tip}}}{\sin(\alpha)} - 1 $$

(1.113)

In case of a perfect conical indenter ($h_{\text{tip}} = 0\text{nm}$), a tip defect of 1nm giving a radius of $R = 1.6\text{nm}$ is set into the Python file. Defining a cono-spherical tip avoids the geometrical singularity at the apex of the perfect conical indenter, which would imply an infinite stress at the contact interface. But, any non-zero positive value for the tip radius can be set into the GUI.

The transition depth $h_{\text{trans}}$ between the spherical and the conical parts of a cono-spherical indenter, is calculated from the following equation:

$$ h_{\text{trans}} = R(1 - \sin(\alpha)) $$

(1.114)

Find here the Matlab function to calculate the tip radius: `tipRadius.m`.
Find here the Matlab function to calculate the transition depth: `transitionDepth.m`.
Find here the Matlab function to calculate the tip defect: `tipDefect.m`.

**Mesh**

The multilayer sample is divided by default into solid elements with eight nodes and axisymmetric deformation element CAX8R is adopted.

It is possible to divide the sample into solid elements with four nodes and with axisymmetric deformation element CAX4R, by changing the value of the variable “linear_elements” in the Matlab function `python4abaqus` from 0 (quadratic elements) to 1 (linear elements).

**Note:**

- CAX4R: A 4-node bilinear axisymmetric quadrilateral, reduced integration, hourglass control.
- CAX3: A 3-node linear axisymmetric triangle.
- CAX8R: An 8-node biquadratic axisymmetric quadrilateral, reduced integration.
- CAX6M: A 6-node modified quadratic axisymmetric triangle.

Figure 1.35: Screenshot in Abaqus of the mesh example used in the FE model.
Material properties

For each layer of the multilayer sample, the material properties (Young’s modulus and Poisson’s ratio) are defined using the inputs given by the user from the GUI. Material properties are considered by default to be isotropic. The density is set by default to 1.0.

Note: Young’s moduli are in GPa.

Contact definition

The contact is defined by default frictionless for the tangential behavior and hard for the normal behavior. The external surface of the indenter is defined as the “master” region and the top surface of the (multilayer) sample is defined as the “slave” region.

Note: Usually, the effect of friction may be neglected when indenter tips with half-angle larger than 60° are used (e.g.: Berkovich, Vickers)\(^1\), \(^2\), \(^3\), and\(^5\).

Boundaries conditions

Nodes are constrained along the rotation axis from moving in the radial direction (\(x\)). The nodes on the bottom surface of the sample are constrained along the radial axis from moving in the radial (\(x\)) and vertical (\(z\)) directions (see Figure 1.36 and Figure 1.37).

Indentation process is simulated by imposing a vertical displacement to the rigid indenter along the (\(z\)) axis (see Figure 1.36 and Figure 1.37). A value of 200nm for the indentation depth is set by default.

Figure 1.36: Schematic of boundaries conditions used in the FE model.

Warning: Indentation displacement is given in nanometers and is negative.

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\(^1\) Atkins A.G. and Tabor D., “Plastic indentation in metals with cones” (1965).
1.7.2 Generation of the Python script for ABAQUS

After material properties are configured (Young’s moduli and Poisson’s ratios) and the model geometry is given (thickness for each thin films), a Python script for ABAQUS can be generated by pressing the ‘FEM’ button.

The python script is saved in the folder where your nanoindentation results are stored.

To generate the FEM model in ABAQUS, apply the following procedure:

- start ABAQUS
- select the folder containing input files : ‘File’ ==> ‘Set Work Directory...’
- select and run the Python file containing the FEM model (*.py) : ‘File’ ==> ‘Run Script’

**Note:** Dimensions are in nm and Young’s moduli are in GPa, implying that load is in nN.

1.7.3 Results of the FEM simulation

The following pictures were obtained for a multilayer Au/Ti/SiO2/Si.

![Screenshot of the Von Mises stress distribution at maximum load.](image)

1.7.4 References

- ABAQUS documentation
- Charleux L., “Abapy Documentation”


• Cai X. and Bangert H., “Hardness measurements of thin films-determining the critical ratio of depth to thickness using FEM.” (2005).
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• Pöhl F., “Determination of unique plastic properties from sharp indentation” (2019).


1.8 Links and References

1.8.1 Links

• Matlab GUI.
  • Visit the YAML website for more informations.
  • Visit the YAML code for Matlab.

1.8.2 Other interesting Matlab toolboxes about indentation

• STABiX / STABiX Documentation
  • PopIn / PopIn Documentation
1.8.3 Links about (nano)indentation

- SF2M - Groupe Indentation.
- Matlab Li’s code to determine elastic modulus and hardness of an ultra-thin film on a substrate using nanoindentation.

1.8.4 References

- Mercier D. et al., “Young’s modulus measurement of a thin film from experimental nanoindentation performed on multilayer systems” (2010).

(Nano)Indentation models (conical indenters)

• Hochstetter G. et al., “Strain-rate effects on hardness of glassy polymers in the nanoscale range. Comparison between quasi-static and continuous stiffness measurements” (1999).
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**Correction factors and Correction of experimental data**

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• Strader J.H. et al., “An experimental evaluation of the constant b relating the contact stiffness to the contact area in nanoindentation.” (2006).
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• Piccarolo S. et al., “Improving surface detection on nanoindentation of compliant materials” (2010).
• Jakes J.E. and Stauffer D., “Contact area correction for surface tilt in pyramidal nanoindentation” (2021).

Bilayer models to extract Young’s modulus of a thin film on a substrate (with conical indenters)

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• Bull S.J., “Mechanical response of atomic layer deposition alumina coatings on stiff and compliant substrates” (2011).
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Multilayer models to extract Young’s moduli of thin films on a multilayer sample (with conical indenters)

• Mercier D. et al., “Young’s modulus measurement of a thin film from experimental nanoindentation performed on multilayer systems” (2010).

Bilayer models to extract hardness of a thin film on a substrate (with conical indenters)

• Doerner M.F. et al., “Plastic properties of thin films on substrates as measured by submicron indentation hardness and substrate curvature techniques” (1986).
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Multilayer models to extract hardness of thin films on a multilayer sample (with conical indenters)

- Arrazat B. et al., “Nano indentation de couches dures ultra minces de ruthénium sur or” (2010).

Finite Element Modelling of conical indentation of thin film(s) on a substrate

- Charleux L., “Abapy Documentation”
- ABAQUS documentation
- Cai X. and Bangert H., “Hardness measurements of thin films-determining the critical ratio of depth to thickness using FEM.” (2005).
• Miller D.C. et al., “Thermo-mechanical properties of alumina films created using the atomic layer deposition technique.” (2010).
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• Pöhl F., “Determination of unique plastic properties from sharp indentation” (2019).

1.8.5 Softwares

• IndentAnalyser by ASMEC
• Elastica by ASMEC
• Softwares from SIOMEC
• Piuma dataviewer from Optics 11
• Punias
• Gwyddion
• Softwares from Nanovea
• Virtual nanoindenter
• Nanoindentation from Kibech S. et al.

1.8.6 Reviews / Overviews of nanoindentation technique

• Němeček J., “Nanoindentation in Material Science” (2012).
• Michailidis N. et al., “Nanoindentation” (2014).

1.8.7 Reviews / Overviews of nanoindentation technique applied to coatings


1.8.8 Books

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CHAPTER 2

Contact

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Keywords

Matlab toolbox ; nanoindentation ; conical indenter ; Young’s modulus ; hardness ; thin film ; multilayer system ; analytical model ; python script ; finite element modelling.