## Contents

1 Contents

1.1 Getting started

1.1.1 How to use the GUI

1.1.2 Main GUI for the indentation mapping

1.1.3 Second GUI for the maps correlation (ICBTI)

1.1.4 Links

1.2 (Nano-)Indentation mapping

1.2.1 Indentation grid technique

1.2.2 Indentation length scales

1.2.3 Interpolation step

1.2.4 Smoothing step

1.2.5 Visualization of maps

1.2.6 Overlay

1.2.7 References

1.3 Mapping options

1.3.1 Crop

1.3.2 Colormap

1.3.3 Colorbar

1.3.4 Markers / Grid

1.3.5 References

1.4 Nanoindentation tomography

1.4.1 3D / 4D map

1.4.2 Other 3D view

1.4.3 References

1.5 Statistical analysis

1.5.1 Probability density function (PDF)

1.5.2 Cumulative density function (CDF)

1.5.3 References

1.6 Elastic modulus vs Hardness plot

1.6.1 E-H map sectorization

1.6.2 Clustering with Gaussian Mixture Models

1.6.3 Ashby map

1.6.4 References

1.7 Image correlation

1.7.1 Loading and analyzing/ comparing binary maps

1.7.2 Image Correlation Based Targeted Indentation (ICBTI)
# 1.7.3 References

---

# 1.8 Examples of nanoindentation data

## 1.8.1 File examples from ‘MTS/Agilent’

---

## 1.8.2 File example from ‘Hysitron’

---

## 1.8.3 File example from ‘ASMEC’

---

## 1.8.4 File example after crop and interpolation steps

---

## 1.8.5 New results file

---

# 1.9 Links and References

## 1.9.1 Matlab links

---

## 1.9.2 Other interesting Matlab toolboxes about indentation

---

## 1.9.3 Links about (nano)indentation

---

## 1.9.4 Indentation maps and statistical analysis in the literature

---

## 1.9.5 Colormap

---

## 1.9.6 Useful softwares / toolboxes for mapping

---

# 1.10 Author

---

# 1.11 Reference papers

---

# 1.12 Keywords

---

# 1.13 Contributors

---

# 1.14 Acknowledgements

---

# 1.15 References

---
Determination of the mechanical properties of each individual phase in the case of a multiphase, heterogeneous or composite material can be achieved using the grid nanoindentation technique\textsuperscript{1}, \textsuperscript{2} and\textsuperscript{3}.

The TriDiMap toolbox has been developed to plot, to map and to analyze (nano)indentation dataset.

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

- to map (in 2D or 3D), to interpolate and to smooth indentation map;
- to plot elastic modulus vs hardness values;
- to plot and to fit probability density functions;
- to plot and to fit cumulative density functions;
- to extract statistical values (mean, min, max with standard deviations) of mechanical properties and fractions for each phase;
- to correct mechanical map using image correlation with microstructural map;
- to correlate/compare mechanical map with microstructural map;
- to plot 4D mechanical property map (indentation tomography).

Source code is hosted at Github.

Download source code as a .zip file.

Download the documentation as a pdf file.

---

**Figure 1: Screenshot of the TriDiMap toolbox**

\textsuperscript{1} Constantinides G. et al., “Grid indentation analysis of composite microstructure and mechanics: Principles and validation” (2006).
\textsuperscript{3} Randall N.X. et al., “Nanoindentation analysis as a two-dimensional tool for mapping the mechanical properties of complex surfaces” (2009).
1.1 Getting started

First of all, download the source code of the Matlab toolbox. Source code is hosted at Github.

1.1.1 How to use the GUI

First of all a GUI is a Graphical User Interface.

- Run the following Matlab script:
  ```matlab
demo.m
  ```

- Answer ‘y’ or ‘yes’ (or press ‘Enter’) to add path to the Matlab search paths, using this script:
  ```matlab
  path_management.m
  ```

- The following window opens:

1.1.2 Main GUI for the indentation mapping

The following window opens for indentation mapping:

- 1) Initial settings and indentation data loading:
  - Set your type of equipment and units for length and mechanical property. Set ‘Agilent’ in case of .xls file generated using the TriDiMap GUI.
  - Set the number of indents along the X and the Y directions.
  - Import your (nano)indentation results, by clicking on the button ‘Select file’. Click here to have more details about valid format of data.
Figure 1.1: Screenshot of the main window of the TriDiMap toolbox

Figure 1.2: Screenshot of the detailed main window of the TriDiMap toolbox
– Distance between indents (along X and Y axis) should be automatically calculated, but it is sometimes wrong. In this case, the user has to set carefully (i.e. checking the length unit, it is in micron by default) by himself these 2 distances.

- **2) Plot settings:**
  - Set your type of equipment and units for length and mechanical property. Set ‘Agilent’ in case of .xls file generated using the TriDiMap GUI.
  - Hardness map is plotted by default.
  - **It is possible to modify (to crop, to smooth, to set other colorbar…) this map and to plot other 2D or 3D maps, and to:**
    * 2D map
    * 3D / 4D map
    * Statistical analysis
    * Hardness vs Elastic modulus map
    * Image correlation
- **3) Average, minimum and maximum values are calculated.**
- **4) Plot options:**
  - Set the colormap
  - Set the colorbar (number of steps, log scale, normalization…)
  - Add markers, min/max values…
- **5) Get values from the map by clicking on a pixel.**
- **6) Buttons to save data (screenshots, .xls file…), to open the 2nd GUI (maps correlation) or to quit…**

Check the different file format possible to import in the TriDiMap toolbox: **File formats**

![Figure 1.3: Plot of the hardness map after loading of data](image)

**Note:** X-Y axis is given on the top right of the GUI as a reference, to indicate the user how maps are read and plotted…
Warning: Only square or rectangular indentation grids can be loaded into the Matlab toolbox.

1.1.3 Second GUI for the maps correlation (ICBTI)

A second window opens for maps correlation, when user click on the ‘BINARIZATION’:

![Screenshot of the second window of the TriDiMap toolbox](image1.png)

Figure 1.4: Screenshot of the second window of the TriDiMap toolbox

It is always possible to go back to the main GUI, by clicking on the ‘MAPPING’ button.

![Screenshot of the detailed second window of the TriDiMap toolbox](image2.png)

Figure 1.5: Screenshot of the detailed second window of the TriDiMap toolbox

- 1) Initial settings and indentation data loading (mechanical maps):
  - Set your type of equipment and units for length and mechanical property. Set ‘Agilent’ in case of .xls file generated using the TriDiMap GUI.
  - Set the number of indents along the X and the Y directions.
  - Import your (nano)indentation results, by clicking on the button ‘Select file’. Click here to have more details about valid format of data.
  - Distance between indents (along X and Y axis) should be automatically calculated, but it is sometimes wrong. In this case, the user has to set carefully (i.e. checking the length unit, it is in micron by default) by himself these 2 distances.

- 2) Threshold definition to separate soft phase from hard/stiff phase.

- 3) Microstructural map loading.
1.2 (Nano-)Indentation mapping

1.2.1 Indentation grid technique

Mechanical property mapping are generated by plotting (nano-)indentation results obtained using the grid indentation technique. The nanoindentation grid technique is well described in the paper written by Constantinides et al., but also in the following references and.

Position of each (nano-)indentation test or distance between indents has to be known or recorded, in order to plot a consistent mechanical property map representative of the experiment.

An indentation map is represented mathematically using a matrix of N columns by M rows. If positions of indentation tests are not known, it is required to know the pattern of indentation tests (line by line, snake shape, diagonals...). An example of a pattern is given here.

An example of 2D generated hardness map is given below. By default, a pixelized map is plotted. Each pixel represents an indentation test and the color of a pixel corresponds to the the intensity of the calculated mechanical property. In the given following screenshot, the white pixels corresponds to tests, which failed (NaN = Not a numeric). Indentation failure corresponds to traditional artifacts of indentation testing (error on surface detection because of contamination/roughness/topography effects, error with the thermal drift correction, etc...) A ratio of failed tests over total number of indentation tests is given on the left part of the GUI, to estimate the experimental validity of the indentation tests. For example, more than 20% of failed tests start to be problematic for the results analysis... But, this is an empirical statement, which depends on the NaN pixels distribution over the map. To remove these empty pixels, it is possible to tick a checkbox into the settings on the GUI, and a mechanical property value is attributed to the empty pixel, by doing a simple averaging of the surrounding pixels (8 in total).

1.2.2 Indentation length scales

According to Constantinides et al., the indentation depth h should be at most 1/10 of the characteristic size of the microstructure D (e.g.: particle size in a matrix, grain or void diameter...), in order to apply continuum indentation analysis to heterogeneous systems and to access phase properties. This rule refers to the well-known 10% rule of thumb proposed by Bückle, which is a rough first estimation. In cases where the contrast between the mechanical properties of the two phases becomes significant (e.g.: ratio between the elastic moduli lower than 0.2 or higher than 5), the method is not really reliable anymore and special care should be taken in the interpretation of the indentation results.

---

3 Randall N.X. et al., “Nanoindentation analysis as a two-dimensional tool for mapping the mechanical properties of complex surfaces” (2009).
Figure 1.6: Raw 2D hardness map obtained from a 25x25 indentation grid

Figure 1.7: 2D hardness map obtained from a 25x25 indentation grid with (left) and without (right) failed indentation.
Moreover, the indentation depth should be at least 3 times the mean square deviation of surface roughness\(^2\).

![Figure 1.8: Schematic of the grid indentation technique for heterogeneous materials](image)

Once the maximum indentation depth is defined following this first rule, it is required to well define the distance between each indents. To avoid overlap of indents, the distance \(d\) between 2 indents has to be higher than the plastic radius \(R_p\) below 1 indent. Usually, the plastic radius in metals is between 3 and 6 times the contact radius \(a_c\), between the indenter and the sample surface. And finally, the contact radius is roughly estimated to be 3.5 times the indentation depth \(h\) in the case of Berkovich indentation and 0.7 times the indentation depth in case of cube-corner indentation.

Thus, indentation step \(d\) can be defined by the following rule of thumb in case of Berkovich indentation:

\[
d > R_p = 10.5x \text{ to } 21x \ h
\]

(1.1)

And in case of cube-corner indentation:

\[
d > R_p = 2.1x \text{ to } 4.2x \ h
\]

(1.2)

![Figure 1.9: Cross-sectional scheme of 2 indents, with the definition of geometrical parameters](image)

More recently, it has been demonstrated that a minimum indent spacing of 10 times the indentation depth was sufficient to obtain insignificant hardness deviation for different bulk materials and coatings tested with a Berkovich indenter. And this result has been generalized for other indenter geometries (spherical and Vickers tips), ans it was found that a minimum indent spacing of 1.5 times the indent contact lateral dimension is enough to get accurate results\(^6\).

---

\(^2\) Michalek J. et al., “Application of Nanoindentation and 2D and 3D Imaging to Characterise Selected Features of the Internal Microstructure of Spun Concrete” (2019).

\(^6\) Sudharshan Phani P. and Oliver W. C., “A critical assessment of the effect of indentation spacing on the measurement of hardness and modulus using instrumented indentation testing” (2019).
1.2.3 Interpolation step

The Matlab function used to interpolate linearly the indentation maps is: interp2.m

The process of interpolation does not modify the raw data intensity values, but increase the number of pixels, by a given factor of x2, x4, x8 or x16 (default values, which can be modified). For example, a map of 25x25 linearly interpolated by a factor of x2, becomes a map of 49x49 pixels. After interpolation, it is possible to create a new .xls file (with interpolated dataset), by pressing the ‘XLS’ button at the bottom of the GUI.

![Figure 1.10: Process of interpolation step](image)

1.2.4 Smoothing step

The Matlab third party code used to smooth the indentation maps is: smooth2a.m

This smooth operation of a 2D matrix is based on a mean filter over a user-defined rectangle. The smoothing step is a solution to apply to smooth sharp peaks or sharp valleys on the mechanical topography. Sharpness can arises when there is a large difference in term of intensity between 1 pixel and its surrounding neighbors (e.g. surface effects, or hard particle on a soft matrix, etc...).

![Figure 1.11: Process of smoothing step](image)

Smoothing process is a modification (clipping of the signal) of the raw values and an error map can be generated, by simply calculating the difference between the non smoothed map (raw map) and the smoothed map.

Linear interpolation and smoothing operations are sometimes applied on the raw dataset in order to lessen pixelization effect and noise from the measurement, to get cleaner and more readable maps.

The Matlab function used to interpolate and to smooth the indentation maps using TriDiMap is: TriDiMap_interpolation_smoothing.m
1.2.5 Visualization of maps

It is possible to plot similar 2D or 3D other maps using different functions of Matlab:

- surf.m
- surfc.m
- waterfall.m
- contour.m
- meshz.m
- bar.m

**Warning:** Indentation experiments are very sensitive to environmental effects and thermal drift. Usually, performing such indentation maps can be time-consuming.

1.2.6 Overlay

To perform an overlay, the best is to save the mechanical map, using the ‘SAVE’ button (at the bottom of the GUI). An image of the map only is saved (no axis, no colorbar...) into the same folder, where data were loaded from. Then, using Powerpoint for example, it is possible to draw a rectangular shape onto the microstructural map and to fill this rectangular shape using the saved mechanical map. Then, transparency effect has to be applied (60%-70% for example) in order to see both maps.

**Note:** It is better if micrographs are obtained before and after indentation experiments, respectively to have a nice overlay (without residual imprints) and to help for overlaying.

1.2.7 References

1.3 Mapping options

1.3.1 Crop

Maps can be cropped to remove some useless part of the map (e.g. bakelite around the specimen, oxidized or contaminated surface...) or to avoid some artifact of measurements (e.g. surface effects, roughness or slope on the surface...). This option is really useful, especially to clean dataset for further statistical analysis. After cropping, it is possible to create a new.xls file (with cropped dataset), by pressing the ‘XLS’ button at the bottom of the GUI.
Figure 1.13: Overlay of hardness (on the left) and modulus of elasticity (on the right) maps with the optical microscopic observation of indentation grid (with 70% transparency)

### 1.3.2 Colormap

It is possible to set different colormap for the mapping into the GUI. The default ‘Jet’ colormap from Matlab\(^1\) is not divergent and obscures real patterns. Thus, it is sometimes difficult to distinguish the default line color styles. Many authors proposed to use divergent colormaps instead of default Matlab colormaps\(^2\),\(^3\),\(^4\).

The Matlab function used to generate a divergent colormap is: diverging_map.m

The colormap can be flipped for aesthetic reason.

Figure 1.14: Maps with non divergent colormap (on the left) and with divergent colormap (on the right)

### 1.3.3 Colorbar

The limits (minimum and maximum) and the number of steps of the colorbar can be defined automatically or can be set by hand. A logarithmic colorbar can be applied to emphasize gradient on a map when there is a large difference in term of mechanical property values between 2 phases.

---

1. Matlab Colormap.
2. Eddins S., “Divergent colormaps”.
4. MyCarta, “A good divergent color palette for Matlab”. 

---
1.3.4 Markers / Grid

Simple grid (corresponding to a regular pattern) or markers grid showing indent positions can be plotted on an indentation map, in order to help for the overlay of the mechanical map onto the microstructural map.

1.3.5 References

1.4 Nanoindentation tomography

1.4.1 3D / 4D map

Instrumented indentation technique provides usually contact stiffness, indenter displacement, and applied load as a function of position (horizontal and vertical), within a volume of tested material by using the dynamic mode (i.e. continuous stiffness measurement or CSM). From such experiments, it is possible to obtain precise and accurate mechanical property gradients\(^1\) or tomograms\(^2\) (i.e., volumetric information). At each indentation depth, corresponds a mechanical property map (i.e. a slice). By stacking each map or slice on each other, it is possible to generate a 3D representation of the indentation experiment. In the literature, such maps are sometimes described as 4D map\(^2\), with the first and the second dimensions as the indentation position, the 3rd dimension as the indentation depth and the 4th dimension as the mechanical property value.

---

\(^1\) Tromas C. et al., “Hardness and elastic modulus gradients in plasma-nitrided 316L polycrystalline stainless steel investigated by nanoindentation tomography” (2012).

Some options are available for the graphical rendering of the tomographic visualization. For example, it is possible to modify the “Z axis ratio” value, to increase or decrease the vertical over the horizontal scale ratio. Higher is this ratio and lower is the vertical expansion. Another interesting option is the transparency effect. It is possible to make the tomogram fully opaque (variable set to 1) or fully transparent (variable set to 0).

The Matlab function used to generate 3D or 4D maps is: slice.m

**Warning:** It is not a 3D reconstruction, thus no need to align, to rotate or to distort the different slices. There is no misalignment or local disorientation, only if the indentation direction is not perpendicular to the sample surface.

![Mapping of Hardness (GPa)](image)

**Figure 1.17:** 4D mapping of sample hardness obtained from indentation tests (25x25 pixels x25 slices)

**Note:** To generate such 4D map, it is required the first time to load raw *.xls files. Given the fact, that the imported data is saved as *.mat into the same folder, it is possible the next time to reload only this *.mat file to save some loading time…

Example of a .mat file: MTS_example1_25x25.xls_3DSlice.mat

### 1.4.2 Other 3D view

Using the TriDiMap toolbox and ‘Powerpoint’, the following figure can be obtained by doing a combination of surface and in-depth indentation maps.
1.4.3 References

1.5 Statistical analysis

For statistical analysis of the mechanical property values distribution, it is possible to use histograms or to use a cumulative distribution.

1.5.1 Probability density function (PDF)

Using histograms is really interesting for the visualization point of view with a common pattern, like the bell–shaped
such a curve is known as the “normal distribution” or the “Gaussian distribution”. For an homogeneous material, only 1 peak is expected and for an heterogeneous material, a peak per phase can be expected. In this last case, intermediate peaks between phase peaks can be observed when interfaces are not spatially negligible. The drawback of this plot is the user-dependence of the bin size and thus of the distribution shape (i.e. peak intensity). Indeed, the shape of the distributions is bin size dependent, while this bin size (e.g. 0.5GPa or 3GPa) is defined arbitrary by the user. This issue is well known in the literature and is a little bit discussed in this presentation\(^8\) and this paper\(^9\). The shape of the distributions is also bin step dependent. In other words, if the the histogram starts from an odd or an even value, the distribution is different.

![Distribution Schematics](image)

**Figure 1.20:** Example of different fitted histogram distribution schematized as a function of the specimen

![Histogram Distribution](image)

**Figure 1.21:** Effect of the bin size definition on the distribution shape

The next step is to fit this distribution using a probability density function. Such mathematical approach is very well

\(^8\) Mercier D. et al., “Combined techniques for the characterization of an electrodeposited Nickel coating with embedded SiC particles: from microstructure to local mechanical properties” (2016).

Figure 1.22: **Effect of the bin step on the distribution shape: the histogram start from 0GPa on the left and from 1GPa on the right, using the same bin size of 2GPa.**

defined in the literature\(^2\),\(^6\) and\(^7\). It is worth to note that the result obtain after deconvolution (average values and standard deviations for each peak) is dependent of the bin size. A solution to avoid user definition of the bin size is to use the Freedman–Diaconis rule of thumb, which gives an estimation of the bin size after calculation the interquartile (IQR) range of the data\(^1\). To activate this option, check the box for ‘Auto Bin Size’ on the GUI.

\[
\text{Bin Size} = \frac{2 \times IQR}{n^{\frac{1}{3}}}
\]  

With \(n\) is the number of observations in the sample.

The Matlab function used to calculate the interquartile range of the data is: `iqrVal.m`

Another rule of thumb is to use between 6 and 20 bins in a histogram.

The Matlab function used to plot the distribution of mechanical property values using histogram is: `pdfGaussian.m`

The Matlab function used to fit using a probability density function and to process the deconvolution is: `TriDiMap_runDeconvolution.m`

This last function has been extensively inspired by the work of Němeček J. et al.\(^3\),\(^4\) and\(^5\).

An example of fitting and deconvolution process is given in the following figure, followed by another example of decomposition process leading to phase mapping and plot of corresponding cumulative distributions.

**Note:** Effect of the interphase can be considered and could be implemented in this toolbox\(^10\).

**Note:** The choice of the bin size could be defined as an automatic calculation, based on the number of phases, the number of data and the minimum of peak intensity…

When data are noisy due to experimental artefacts (e.g. surface contamination, interfaces effect or interesting phase inside the sample…) for example, with very high or very low mechanical property values, it is always possible to cut the signal, by setting manually (on the GUI) the extrema. This operation can be seen as an arbitrary cleaning, but careful with a fitting process, which gives different mean values, given peak shapes or peak number modification.

\(^7\) Haušild P. et al., “Determination of the individual phase properties from the measured grid indentation data” (2016).
\(^3\) Němeček J., “Probability density function 1.0” (2010).
\(^4\) Němeček J., “Probability density function 2.1” (2010).
Figure 1.23: Histograms of hardness values with Gaussian PDF after fitting and deconvolution step

Note: Sometimes the fit does not converge, just restart the fitting process... or change a little bit the bin size.

1.5.2 Cumulative density function (CDF)

The cumulative distribution of mechanical property is much better than an histogram plot (no bin size dependency). But, it is much more difficult to decompose and in this toolbox, only Gaussian and Weibull fitting are proposed, which is only interesting for an homogeneous material. The Weibull function is from the PopIn toolbox\textsuperscript{11}.

The Matlab function used to plot the cumulative distribution of mechanical property values is: \texttt{cdfGaussian.m}
The Matlab function used to fit the cumulative distribution with a Weibull function is: \texttt{TriDiMap_Weibull_cdf.m}

1.5.3 References

1.6 Elastic modulus vs Hardness plot

Another way to visualize the distribution of mechanical property results is to plot for example the elastic modulus (E) values vs the hardness (H) values. Such a plot leads sometimes to the observation of families of points and the definition of “sectors” or “bubbles”, each one corresponding to a single phase (e.g. soft matrix vs hard and stiff particles).

The correlation between elastic and plastic properties has been extensively studied in the literature\textsuperscript{5,2,11, and7}.

\textsuperscript{11}Mercier D.et al., “PopIn documentation”.
Figure 1.24: *Phase map and cumulative distributions of each phase resulting from decomposition step*
Figure 1.25: Example of manually saturated indentation data, with a comparison between automatic maximum and a maximum set to 8GPa
Figure 1.26: Cumulative distribution of elastic modulus values with Gaussian and Weibull fitting

Figure 1.27: Cumulative distribution of elastic modulus values with cumulative distributions for each different phase

1.6. Elastic modulus vs Hardness plot
Elastic modulus is an intrinsic material property and hardness is an engineering property, which can be related to yield strength for some materials.

1.6.1 E-H map sectorization

As a first analysis of such a plot, sectors can be defined by giving an average value of elastic modulus and an average of hardness value, separating respectively by an horizontal line and a vertical line the different bubbles of points. Each sector is defined by a unique color.

Finally, average values of mechanical properties are given for each sectors directly into the graph, and a 4 color-coded map corresponding to this plot can be generated (see 2nd figure).

Figure 1.28: Example of sectorized elastic modulus vs hardness plot

Figure 1.29: Sectorized elastic modulus vs hardness plot with mean values and corresponding mechanical map
1.6.2 Clustering with Gaussian Mixture Models

The Gaussian mixture Models (GMM) are often used for data clustering\textsuperscript{12}. This method is well described in the Matlab documentation\textsuperscript{8,9} and\textsuperscript{10} but also in the literature\textsuperscript{4}.

This method is powerful to separate contribution of 2 or 3 phases (especially in the case of a soft metallic matrix with hard ceramic particles) in the cloud of experimental points\textsuperscript{6}. Average mechanical property values can also be extracted using this method and a 2 or 3 color map can be obtained too.

The Matlab third party code used to define clusters GMM is: GMMClustering.m

![Figure 1.30: Elastic modulus vs hardness plot with clusters of points obtained with GMM](image)

1.6.3 Ashby map

Such plot could be seen as a conventional Ashby map\textsuperscript{1}, with materials families. An example of a typical Ashby map is given afterwards, using the CES Selector 2018 software\textsuperscript{3}. At some point, it is possible to add material reference (bulk, homogenuous, monophasic, . . . ) values on the E-H map, in order to compare experimental data with data from the literature.

1.6.4 References

1.7 Image correlation

Coupling statistical indentation and microscopy observation can be a good solution to evaluate micromechanical properties of materials\textsuperscript{1}.


\textsuperscript{8} Mathworks - Gaussian Mixture Models

\textsuperscript{9} Mathworks - Cluster

\textsuperscript{10} Mathworks - Cluster Using Gaussian Mixture Models


\textsuperscript{6} Hu C., “Nanoindentation as a tool to measure and map mechanical properties of hardened cement pastes” (2005).


\textsuperscript{3} CES Selector 2018

\textsuperscript{1} Hillouin B. et al., “Coupling statistical indentation and microscopy to evaluate micromechanical properties of materials: Application to viscoelastic behavior of irradiated mortars” (2018).
1.7.1 Loading and analyzing/comparing binary maps

First, mechanical maps generated using indentation experiments have to be loaded. These maps are automatically binarized, applying a threshold (mean hardness and mean elastic modulus values by default). These thresholds can be modified or optimized later by comparing phase ratio determined from the microstructural map. Afterwards, a binarized microstructural map has to be loaded, with the same resolution as the mechanical maps (e.g. 25x25 pixels in the following examples). A solution to avoid pixelized maps is to apply linear interpolation on the mechanical map and thus increasing the mechanical maps resolution.

To obtain a pixelized microstructural map, a solution is to use the software ImageJ (or Fiji). Once an image of the indentation grid is obtained by microscopy with enough quality (good contrast between phases, enough resolution...), load it in the image analysis software. Crop the image around the indentation grid and let the minimum of space around the matrix of imprints. Adjust the contrast/brightness of the image to emphasize the different phases in your sample. Then, adjust the number of pixels horizontally and vertically, before to save it as a .png microstructural map.

Examples of microstructural maps: data_image

Note: It is much better to obtain the microstructural image before the indentation experiments. Otherwise, residual indents affect the image quality and thus some additional operations of cleaning are required (e.g. despeckle, FFT, ...).

1.7.2 Image Correlation Based Targeted Indentation (ICBTI)

Phase fractions and mechanical property values can be extracted from grid nanoindentation experiments using innovative and quantitative statistical treatments of datasets, with image correlation based targeted indentation (ICBTI).
1.7. Image correlation

Figure 1.32: Example of image processing of the microstructural map

Figure 1.33: Visualization of binarized mechanical maps

Figure 1.34: Comparison between binarized microstructural map and mechanical maps
TriDiMap Documentation, Release 3.0.0

Figure 1.35: Phase ratio optimized comparison between binarized microstructural map and mechanical maps

analysis. The concept of the ICBTI is explained in different papers\(^3\),\(^4\) and\(^5\).

The microstructural map thresholding is based on greyscale distribution of the micrograph, while for the mechanical property maps thresholding is based on the phase distribution. For mechanical property map, threshold values are obtained by finding the intermediate value between matrix and particle peaks, while respecting phase ratios obtained from microscopical image analysis. After thresholding and binarization operations, the difference between binarized mechanical property and microstructural maps is computed and plotted so as to create correlation maps. This procedure is described graphically in the following figures. Mechanical property–microstructure correlation maps allow assessing the match between indents and the probed phase in order to select indents representative for each single phase. Then, the intersection between these two mechanical properties–microstructure correlation maps gives a new correlation map without measurements artefacts. Such abnormal measurements may come from the presence of a defect (void, crack, surface pollution, etc.) or from the detection of an underlying or very small particle.

Cleaned E-H plots are generated by clicking on the button ‘E vs H’ at the bottom left of the GUI. Correlation maps are obtained by clicking on the button ‘Corr. Map’ at the bottom left of the GUI.

For the cleaned E-H plot, it is asked to add material reference (bulk, homogeneous, monophasic, . . . ), in order to compare experimental data with reference data.

1.7.3 References

1.8 Examples of nanoindentation data

**Warning:** Only square or rectangular indentation grids can be loaded into the Matlab toolbox.

**Warning:** Only .xls or .xlsx file can be loaded into the Matlab toolbox. Or Matlab code has to be updated. See the section ‘New results file’ below.

\(^3\) Mercier D. et al., “Mechanical characterization by nanoindentation of an electroplated nickel matrix composite coating” (2017).


\(^5\) Mercier D. et al., “Mechanical properties mapping of cast bimetallic work roll shell material by nanoindentation” (2018).
Figure 1.36: Principle of the ICBTI analysis, for a Ni-SiC sample

Figure 1.37: Principle of the ICBTI analysis, for a steel with carbides (flower) sample

1.8. Examples of nanoindentation data
Figure 1.38: Scheme of different indentation tests (cross-sectional view) and mathematical criteria for discrimination between correct measurements and artefacts in the correlation process during the ICTBI analysis.

Figure 1.39: Cleaned E-H plot.
Note: For 2D maps, only a sheet with average values at a given indentation depth is enough. But for a 3D mapping, sheet with mechanical properties as a function of indentation depth are required.

1.8.1 File examples from ‘MTS/Agilent’

For a 2D map: MTS_example1_25x25.xls
For a 3D map: MTS_example3_5x5_testSlice.xlsx

1.8.2 File example from ‘Hysitron’

For a 2D map: Hysitron_example_10x10.xlsx

1.8.3 File example from ‘ASMEC’

For a 2D map: ASMEC_example_20x11.xlsx

1.8.4 File example after crop and interpolation steps

For a 2D map: MTS_example1_25x25.xls_interp_81x81.xls Don’t forget to set ‘Agilent’ for the equipment, when you load an .xls file generated using the TriDiMap GUI.

1.8.5 New results file

In case of other or new experimental results file, the most important is to know the pattern of the indentation grid. For example, the following figure shows the pattern of a typical indentation grid obtained using the software (e.g. Testworks...) of MTS/Agilent.

The Matlab function to modify to load specific indentation results file is: TriDiMap_loadingAllTests.m

Contact me, in case of special data file format.

1.9 Links and References

1.9.1 Matlab links

- Matlab GUI.
- Visit the YAML website for more informations.
- Visit the YAML code for Matlab.
1.9.2 Other interesting Matlab toolboxes about indentation

- STABiX / STABiX Documentation
- NIMS / NIMS Documentation
- PopIn / PopIn Documentation

1.9.3 Links about (nano)indentation

- SF2M - Groupe Indentation.

1.9.4 Indentation maps and statistical analysis in the literature

• Randall N.X. et al., “Nanoindentation analysis as a two-dimensional tool for mapping the mechanical properties of complex surfaces” (2009).
• Němeček J. et al., “Nanoindentation Based Analysis of Heterogeneous Structural Materials ” (2012).
• Rettler E. et al., “Mapping the mechanical properties of biomaterials on different length scales: depth-sensing indentation and AFM based nanoindentation” (2013).
• Amanieu H.Y. et al., “Mechanical property measurements of heterogeneous materials by selective nanoindentation: Application to LiMn2O4 cathode” (2014).
• Šavija B. et al., “Corrosion induced cover cracking studied by X-ray computed tomography, nanoindentation, and energy dispersive X-ray spectrometry (EDS)” (2014).
• Hu C., “Nanoindentation as a tool to measure and map mechanical properties of hardened cement pastes”, MRS Communication (2015).
• Vasconcelos, L.S.D. et al., “Grid indentation analysis of mechanical properties of composite electrodes in Li-ion batteries” (2016).
• Haušild P. et al., “Determination of the individual phase properties from the measured grid indentation data” (2016).
• Mercier D. et al., “Mechanical properties mapping of cast bimetallic work roll shell material by nanoindentation” (2018).
• Bruker/Hysitron, “Application Note #1511, Hardness Mapping of a DP980 Steel Sample” (2018).
• Hillouin B. et al., “Coupling statistical indentation and microscopy to evaluate micromechanical properties of materials: Application to viscoelastic behavior of irradiated mortars” (2018).
• Kariem H., “The viscoelastic behaviour of material phases in red clay identified by means of grid nanoindentation” (2019).
• Besharatloo H. et al., “Small-scale mechanical properties of constitutive phases within a polycrystalline cubic boron nitride composite” (2019).
• Sudharshan Phani P. and Oliver W. C., “A critical assessment of the effect of indentation spacing on the measurement of hardness and modulus using instrumented indentation testing” (2019).
• Gaillard Y. and Amiot F., “Grid nano-indentation as full-field measurements” (2020).
• Zeng F et al., “Micromechanical Characterization of Ce0.8Gd0.2O2-δ–FeCo2O4 Dual Phase Oxygen Transport Membranes” (2020).

1.9.5 Colormap

• Matlab Colormap.
• Eddins S., “Divergent colormaps”. 

32 Chapter 1. Contents
• Moreland K., “Diverging Color Maps for Scientific Visualization”.
• MyCarta, “A good divergent color palette for Matlab”.

1.9.6 Useful softwares / toolboxes for mapping

• Němeček J., “Probability density function 1.0” (2010).
• Němeček J., “Probability density function 2.1” (2010).
• Němeček J., “Deconvolution algorithm 3.0” (2010).
• ImageJ
• Bruker/Hysitron - XPM module

1.10 Author

Author David Mercier [1]
[1] CRM Group, Avenue du Bois Saint-Jean 21, B27 – Quartier Polytech 4, 4000 Liège, Belgium

1.11 Reference papers

• Mercier D. et al., “Mechanical properties mapping of cast bimetallic work roll shell material by nanoindentation” (2018).

1.12 Keywords

Matlab toolbox; nanoindentation; mapping; grid; 2D; 3D; mechanical properties; probability density function; deconvolution; multimodal Gaussian fit; cumulative density function; image correlation.

1.13 Contributors

• Pierre Huyghes (ULB, Bruxelles) and Antoine Hilhorst (UCL, Louvain-La-Neuve) contributed to the Matlab code.
• Franck Nozahic (OCAS, Zwijnaarde) contributed to the documentation.
1.14 Acknowledgements

The author is grateful to Dr. Jiri Nemecek from Czech Technical University, Czech Republic (Prague) and to Dr. Nicholas Randall from Anton Paar, for discussions and many advices about nanoindentation mapping.

The author is grateful to Jean-François Vanhumbeeck and Xavier Vanden Eynde (CRM Group, Belgium (Liège)), for fruitful discussion and co-authoring of reference papers.

The author is grateful to Debora Rosseel, Franck Nozahic and Mélanie Gauvin (OCAS, Belgium (Zwijnaarde)), for providing example files and lots of fruitful feedbacks/remarks.

1.15 References