

Measure what is measurable and make measurable that which is not.

Galileo Galilei (1564-1642)

# Software Manual

# Indentation

Indentation Software Version 7

# Software Manual

# **Indentation**

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# 1 ABBREVIATIONS & SYMBOLS

Physical quantity symbols are according to the ISO 14577 standard.

Abbreviations Symbols	Designations	Units
ADO	adjust depth offset	
AFM	Atomic Force Microscope	
A <sub>p</sub>	projected contact area	m <sup>2</sup>
Снм	creep Martens hardness	%
C <sub>IT</sub>	indentation creep	%
COS	Conscan confocal (observation head)	
e.g.	for example	
E*	plane strain modulus	Pa
$E_{IT}$	indentation modulus	Pa
ε (Epsilon)	geometric constant	
E <sub>r</sub>	reduced modulus	Pa
F	test force	N
F <sub>max</sub>	maximum test force	N
h	indentation depth under applied test force	m
h <sub>c</sub>	contact depth of the indenter with the sample at $F_{\text{max}}$	m
H <sub>IT</sub>	indentation hardness	Pa
HM	Martens hardness	Pa
h <sub>max</sub>	maximum indentation depth	m
hp	permanent indentation depth	m
h <sub>r</sub>	tangent indentation depth	m
HV	Vickers hardness	Vickers
$HV_{IT}$	Vickers hardness calculated from H <sub>IT</sub>	Vickers
.INI/.ini file	initialization file	
m	power law exponent	
МСТ	Micro Combi Tester (measurement head) (applicable for MCT 1 <sup>st</sup> generation <b>or</b> MCT <sup>3</sup> new generation)	
MHT	Microindentation Tester (measurement head) (applicable for MHT 1 <sup>st</sup> generation <b>or</b> MHT <sup>3</sup> new generation)	
min	minutes	
min.	minimum	
NHT	Nanoindentation Tester (measurement head) (applicable for NHT <sup>2</sup> 2 <sup>nd</sup> generation or NHT <sup>3</sup> new generation)	
ν (Nu)	Poisson's ratio	
RIT	indentation relaxation	%
O&P	Oliver & Pharr	
S	contact stiffness	N/m

	ec.	seconds			
ST	eP 4	Surface Testing Platform dedicated for 4 units			
		High Vacuum S	Surface Testing Pl	atform dedicated for 5	
STeP	<sup>9</sup> 5 HV	units			
		(primary & sec	condary vacuum p	umps)	
		Vacuum Surfa	ce Testing Platforr	n dedicated for 5	
STel	P 5 V	units			
		(primary vacui	um pump only)		
STe	eP 6		g Platform dedicat	ted for 6 units	
T	TX	Table Top			
		Ultra Nanoindentation Tester			
		(measurement head)			
UN	NHT	(applicable for UNHT 1 <sup>st</sup> generation			
		or UNHT <sup>3</sup> new generation			
or UNHT <sup>3</sup> HTV High Temperature new generation)		e new generation)			
Anton Paar BioindenterTM		general,			
UNH	IT Bio	(applicable for UNHT <sup>3</sup> new generation)			
ONTH DIO		(measurement head)			
	optical Video microscope				
V	ID	(observation head)			
Welast elastic reverse deformation work of indentation		of indentation	N.m		
				N.m	
•				N.m	
	Wtotal total mechanical work of indentation				
η	IT	elastic part of indentation work 9		%	
i	1 N/m	$m^2 = 1 MPa$	1 MPa $1 J = 1 N.m$ 1 Vickers = 1 kgf/mm <sup>2</sup>		mm²

# 2 INTRODUCTION

### 2.1 ABOUT INDENTATION SOFTWARE



By choosing an Indentation Tester from the complete range offered by Anton Paar, the user benefits from all advantages of our powerful software: an easy to use window interface including numerous indentation measurement types, analysis methods, image management and advanced reporting functions.

The user interface of the *Indentation Software* (for **Windows**® **7 and 10**) provided by Anton Paar is described in this manual.

This manual concerns the following indentation instruments of Anton Paar:

```
STEP -

MHT

NHT

UNHT or UNHT HTV if ☑ High Temperature is checked in chap. 3.1.2.1, p. 14

UNHT Bio

TTX-

NHT

UNHT

UNHT

UNHT

UNHT
```

Some instructions can be specific to one instrument/measurement head; in that case it will be specified.

### 2.2 ENCLOSED CONTENT

This manual explains how to optimize the *Indentation Software* from Anton Paar. For information concerning the instrument, refer to its separate user manual.

### 2.3 How to use this manual?

- Software is preinstalled, refer to the *Common Scratch & Indentation* software manual chap. Starting of software, which explains how to use the *Indentation Software*.
- Software is not installed, refer to the Common Scratch & Indentation software manual - chapters About installation and Hardware configuration, which guides the user through the installation and configuration processes.
- Some Indentation Software features are common with Scratch Software.

  Therefore if the information is not described in this manual, refer to the Common Scratch & Indentation software manual.

For information concerning the instruments, refer to each corresponding \*measurement head\* user manual.

For information concerning the *Video Software*, refer to the *Video software* manual.

- 1 In this manual:
  - The images may differ from the actual product.
  - "acquisition system" means dedicated computer with acquisition card and software.
  - "STeP" means STeP 4, STeP 5 HV, STeP 5 V and STeP 6.
  - "STeP 5" means STeP 5 HV and STeP 5 V.
  - If not specified, generally "measurement(s)" means indentation measurement(s) → for single measurement or for matrix of measurements.
- for <u>Troubleshooting</u> see chap. 6, p. 173.

# 3 MANAGING THE INSTRUMENT

# 3.1 HARDWARE CONFIGURATION

Refer to the **Common Scratch & Indentation software manual chap. Hardware configuration**.

#### 3.1.1 INSTRUMENTS TABS

(i) Common tabs/functions for the Scratch and Indentation instruments are described in the *Common Scratch & Indentation software manual - Hardware configuration -* under each chapter about *tab*.

### 3.1.2 MY CONFIGURATION TAB



### 3.1.2.1 UNHT

Configuration  ☑ Hardware generator	This inactive (grayed out) box is automatically checked to activate the (standard) Sinus hardware present on the UNHT.		
☑ High Temperature	This box should be checked if the instrument is a STeP 5 HV - UNHT HTV.		

# 3.1.2.2 NHT

This box should be checked to activate the **optional** Sinus hardware present on the NHT; the corresponding software features become active (no longer grayed out).

If this option is not present, this box should be unchecked.

### 3.1.2.3 MHT

This box should be always unchecked with MHT.

#### 3.1.3 'RANGES' TAB(S)



**Refer** to the **Common Scratch & Indentation software manual** and carefully read - chap. 'Ranges' tab(s).

#### 3.1.3.1 **UNHT** range tabs

UNHT has 3 'Ranges' tabs.

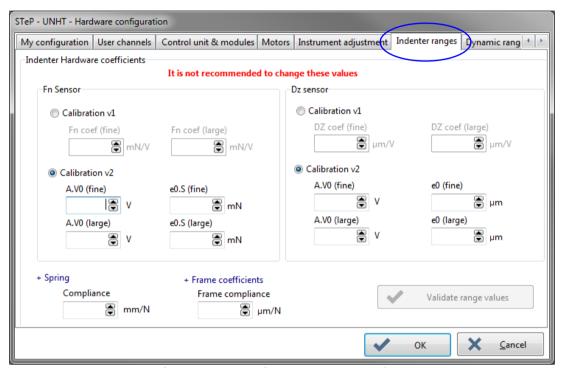


Fig.1 UNHT Indenter ranges tab

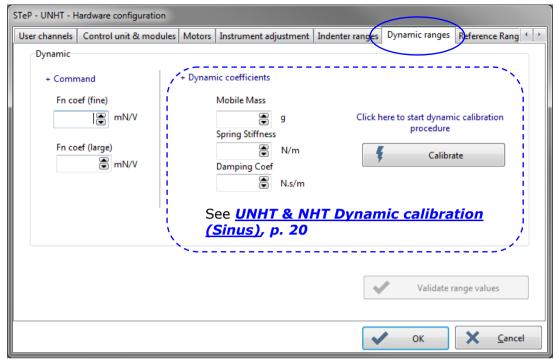


Fig.2 UNHT Dynamic ranges tab

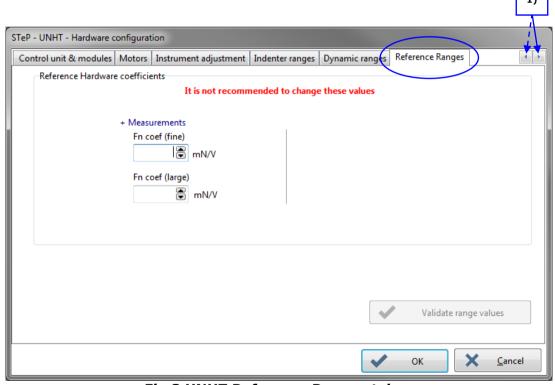


Fig.3 UNHT Reference Ranges tab

<sup>1)</sup> select (2 times max) the right cursor to display (fully) the 2 last tabs or select the left cursor to display (again) the 2 first tabs

# 3.1.3.2 NHT range tabs

NHT has 2 'Ranges' tabs.

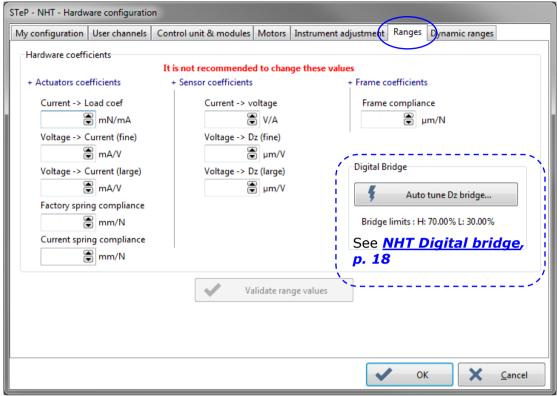


Fig.4 NHT Ranges tab

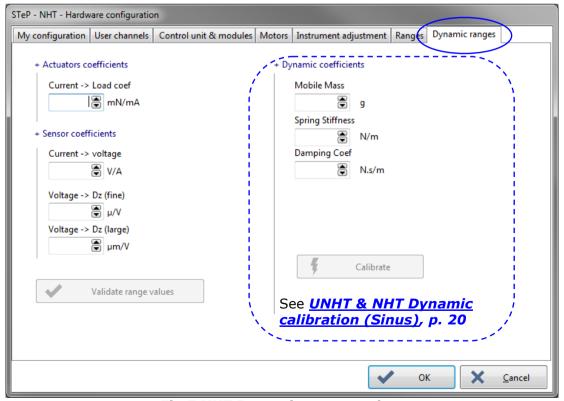


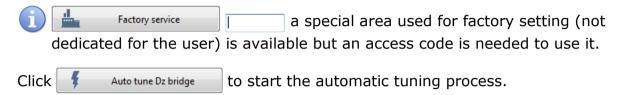
Fig.5 NHT Dynamic ranges tab

#### **NHT Digital bridge**

To properly use the instrument, the auto tuning of the Dz sensor bridge should be performed once during the instrument (control unit) installation. However this tuning can be performed again if needed. The procedure is an automatic tuning of the Dz bridge (electronic adjustment process).



The Dz Bridge tuning window appears Fig. 6 below.



The tuning is processing: the software scans (progression bar % increases) the entire Dz sensor bridge and adjusts the limits for a quicker detection.

The *previous limits* saved in the software are displayed.

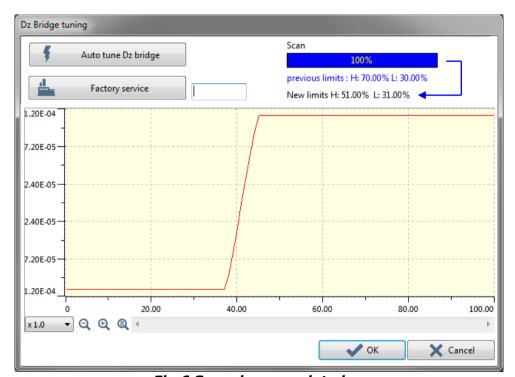
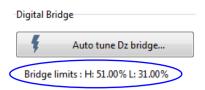


Fig.6 Scanning completed

End of the tuning: the scanning is completed (100 %) and the *New limits* calculated by the software are displayed.

Click overwrite the previous ones) in the software.

# In the "Ranges" tab Fig.4, p. 17,



the new Dz bridge limits appear.

If the tuning is performed again (after the first time), the *New limits* should be the same or almost the same as the *previous limits* - only a few % of difference should be displayed.

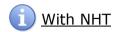
previous limits : H: 51.00% L: 31.00% New limits H: 51.00% L: 31.00%

in *Fig.*6, p. 18

### **UNHT & NHT Dynamic calibration (Sinus)**

The *Dynamic coefficients* fields and "*Calibrate*" button are located in the "*Dynamic ranges*" tab of the UNHT <u>Fig. 2</u>, p. 16 and NHT <u>Fig. 5</u>, p. 17.

The dynamic calibration is only available with the **optional** Sinus mode.



If \_\_\_\_\_\_\_ is inactive (grayed out), it means the **optional** Sinus hardware is not present (see *chap. 3.1.2.2 NHT*, *p. 14*) and this calibration cannot be performed.

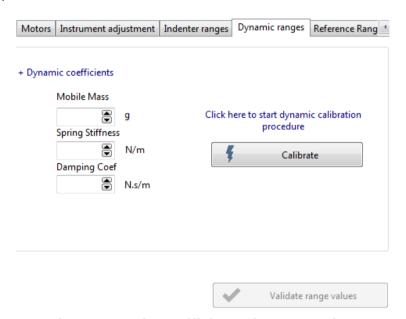


Fig.7 Dynamic coefficients (e.g. UNHT)

Before running any Sinus measurements, including the indenter Sinus calibration, this dynamic calibration should be performed:

- At the instrument installation.
- Each time a new indenter is used.

First it is recommended that an ADO has been **successfully** ( $\Re$ ) performed; see **chap.** 4.4 <u>Adjusting the depth offset (ADO)</u>, **p.** 56.

Then click Calibrate to open the following UNHT or NHT Head Dynamic Calibration window Fig. 8, p. 21.

If the procedure was already performed once before, the window contains the curves and values of the previous calibration.

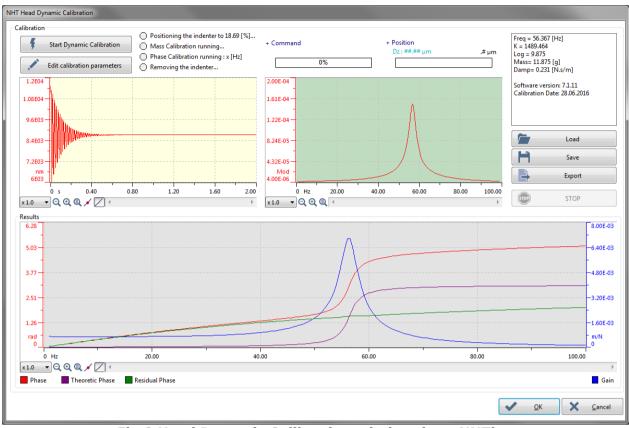
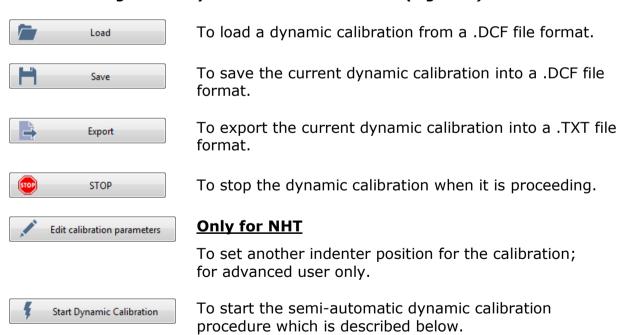
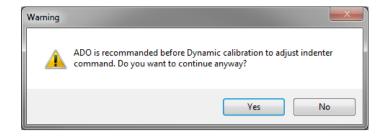
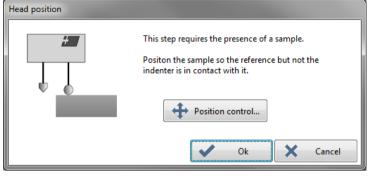


Fig.8 Head Dynamic Calibration window (e.g. NHT)





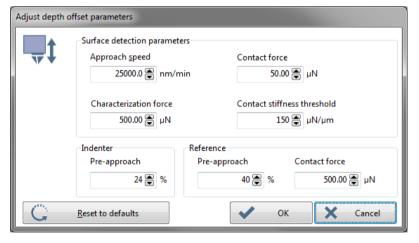
If no ADO or non-successful ADO has been performed, click Yes; read chap. 4.4 Adjusting the depth offset (ADO), p. 56 and then come back below.





Click Position control... to move the **edge** of the sample under the reference and so that the indenter **cannot** touch the sample (UNHT reference, NHT reference ring), as shown on the corresponding image.





# **For UNHT**

This additional ADO parameters window with the same previous successful ADO parameters appears and is used to approach the UNHT reference to the sample surface and also to preapproach the indenter.

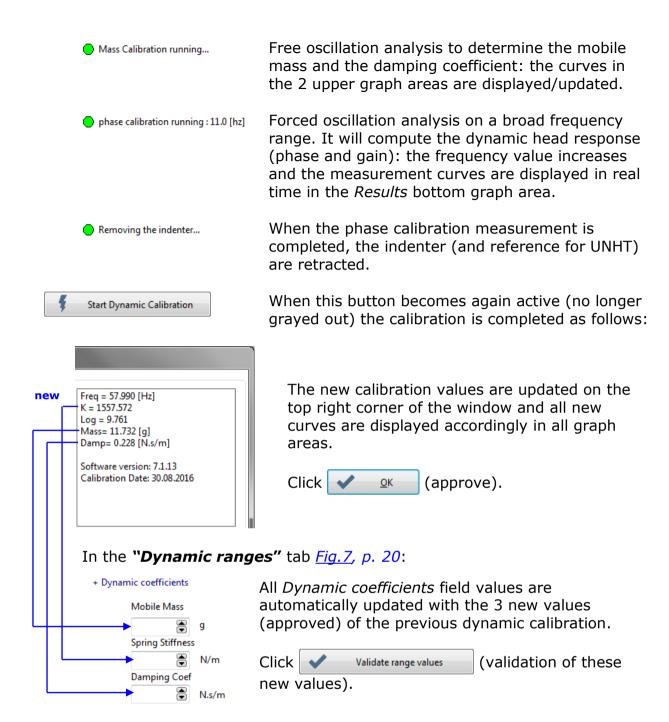


Then in the *Head Dynamic Calibration* window <u>Fig.8</u>, p. 21:

The 4 following circle status (on window top left side) blinks green one after the other (from top to bottom); wait.

Positioning the indenter...

(The reference approaches for UNHT and) the indenter pre-approaches.



# 3.1.3.3 MHT range tab

MHT has only one "Ranges" tab.

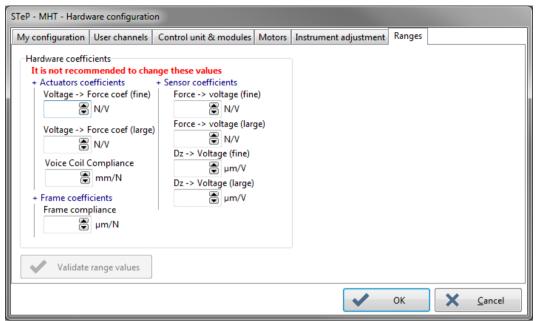
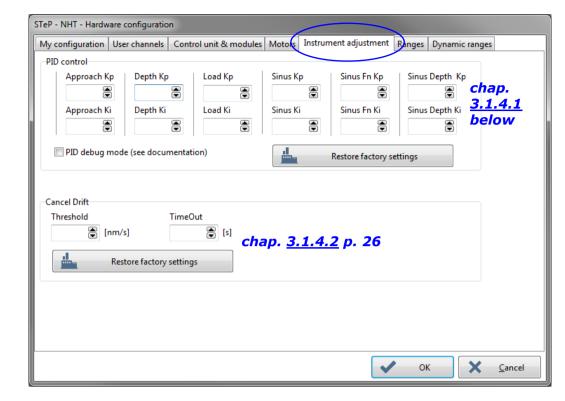
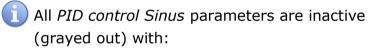


Fig.9 MHT Ranges tab

# 3.1.4 INSTRUMENT ADJUSTMENT TAB



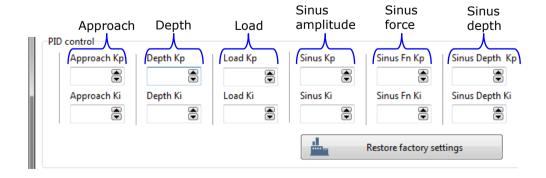




- MHT
- NHT, if Hardware generator is unchecked;
   chap. 3.1.2.2 NHT, p. 14

# 3.1.4.1 PID control parameters

It is not recommended to modify the factory parameter field values, which guarantee a good depth or load control during the indentation measurement on the majority of materials. If necessary, click Restore factory settings to restore these factory parameter values.



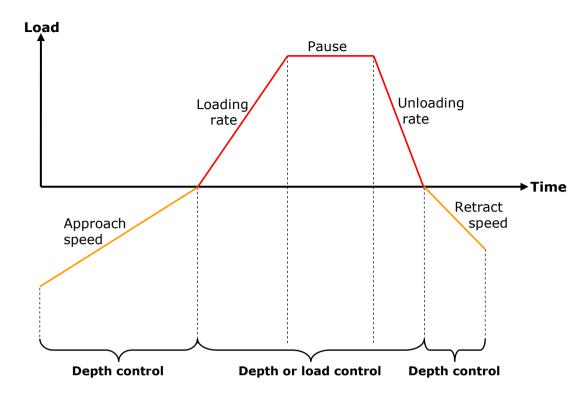
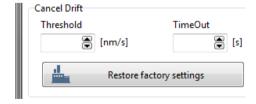


Fig.10 Control loop schematic description

 In measurement cycle the approach phase and retract phase are depth controlled.

# 3.1.4.2 Stabilization (cancel drift)

During the automatic ADO process; from *chap.* <u>4.4.1</u>, *p.* <u>57</u> to <u>4.4.3</u>, *p.* <u>70</u> and during the indentation measurement process; *chap.* <u>4.7</u>, *p.* <u>122</u>, a *Stabilization* step  $\blacksquare$  Stabilization 30 [s] (in *Status* area) is performed to wait for an acceptable thermal drift before performing an indentation.



The stabilization ends:

When the thermal drift is under the set *Threshold* parameter field value,

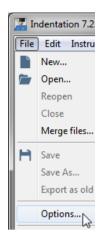
#### OR

after the elapsed set *TimeOut* parameter field value.

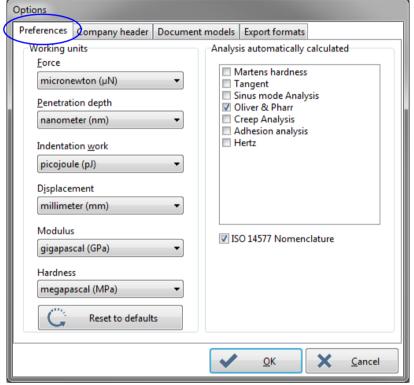
Click Restore factory settings to restore the factory settings.

# 3.2 OPTIONS/PREFERENCES (ISO NOMENCLATURE)

# To display ISO 14577 nomenclature



Select File/Options... from the menu bar.

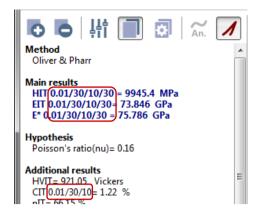






The feature below is applicable with *Marten hardness*, *Tangent* and e.g. *Oliver* & *Pharr* analysis methods; see *chap.* 5.4, p. 144.

Check ☑ ISO 14577 Nomenclature to display the analysis results in ISO 14577 format *Fig. 12*, *p. 28*.



In the analysis result area (see <u>Fig.62</u>, p. 130) the **Main** and **Additional** results are displayed in ISO 14577 format.

E.g. HIT 0.01/30/10/30;

see the details of the nomenclature in accordance with the **ISO 14577-1:2002** standard.

Fig.12 ISO 14577 nomenclature

### See also:

- chap. 1 Abbreviations & symbols, p. 10
- chap. 7 Software formulas, p. 176



■ ISO 14577 Nomenclature unchecked in Fig. 11, p. 27 is the default format

Main results HIT= 9945.4 MPa EIT= 73.846 GPa E\*= 75.786 GPa

### 3.3 CALIBRATION WINDOW & TABS

Indentation 7.2.5

File Edit Instrument Adminis

Adjust depth offs

Start a new inden

Position control...

Indenters...

Control indenters

Configure sample

Select *Instrument/Calibrations...* from the menu bar.

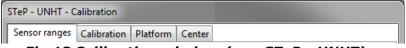


Fig.13 Calibration window (e.g. STeP - UNHT)

This window contains several tabs (depending on instrument) which allow configuring several calibration features:

<u>Tab</u>	for	chap.	
"Sensor ranges"	UNHT/UNHT Bio	<u>3.3.1</u>	below
"Calibration"	All instruments	<u>3.3.2</u> ,	p. 30
"Platform"	All, except TTX-NHT	<i>3.3.3</i> ,	p. 31
"Center"	All instruments	<u>3.3.4</u> ,	p. 33

# 3.3.1 Sensor ranges tab (UNHT/UNHT Bio)

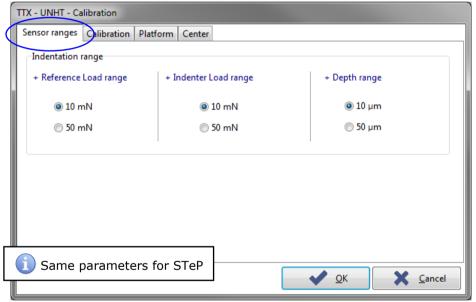


Fig.14 Sensor ranges tab (e.g. TTX-UNHT with fine ranges)



This tab is also available in the UNHT and UNHT Bio *Preferences* windows; for the detailed description see respectively *chap.* <u>4.6.5.3</u>, *p.* 115 and *chap.* <u>4.6.5.4</u>, *p.* 115

# 3.3.2 CALIBRATION TAB (DISTANCE)

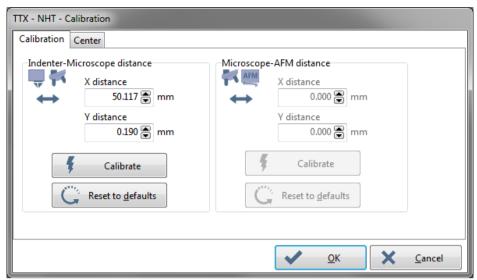


Fig.15 Distance calibration tab (e.g. TTX-NHT)

The distances<sup>1)</sup> between,

- the indenter and microscope (VID)
- the indenter and AFM<sup>2)</sup>

should be calibrated:

Click Calibrate to (re-)start the corresponding calibration procedure and refer to the **Common Scratch and Indentation software manual** - **chap. Distance calibration** for the detailed procedures.

Each active calibration should be re-performed whenever the user changes the indenter, in order to accurately localize the region of the indentation in the *Video* window/AFM<sup>2)</sup> area.

<sup>1)</sup> in case of manual Y table, the *Y distance* fields are inactive (grayed out)

<sup>&</sup>lt;sup>2)</sup> calibration with AFM is active (no longer grayed out) if the **optional** AFM is mounted only on the STeP instrument

#### 3.3.3 PLATFORM TAB (NOT FOR TTX-NHT)

This tab is not available with TTX-NHT (as it does not have a motorized Z table).

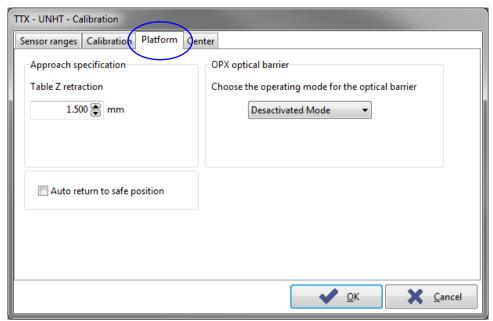
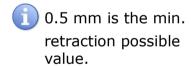


Fig.16 Platform tab (e.g. TTX-UNHT)

#### **Table Z retraction**

Table Z retraction 1.500 🖀 mm initial value



After each time that the motorized Z table is moved up (raised): e.g. for the focus adjustment and then the position control is closed, or after a measurement..., the table automatically moves back down (lowers) to the retraction value set in this field = rest position.



Beware of any collision.

This parameter can be adjusted according to the height of sample used. If the sample is not flat and/or tilted, set enough margins in order that the sample does **not** enter in collision with the indenter during lateral displacements, e.g. between each indentation in a matrix.

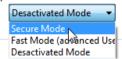
#### Safe position

Auto return to safe position

Check this box to automatically return to the safe position at the end of each measurement: the motorized tables/sample move to the extreme right front position; same position as clicking  $\triangleq$  on the main toolbar.

### **Optical barrier**

Choose the operating mode for the optical



Choose one of the 3 following security modes for the optical barrier.

**Secure Mode**: if the optical barrier is interrupted, the current measurement, optical analysis or positioning is stopped. To perform a matrix of measurements with this mode, <u>Table Z retraction</u>, p. 31 should be set at least to 7 mm, in order to not interrupt the optical barrier.

When the user is sure that there is no risk of a collision during all indentation matrix of measurements and additionally to save time, the following other operating modes can be selected. In these cases a lower value

can be set in **Table Z retraction**, p. 31 but 1



beware of any collision:

**Fast Mode (advanced User)**: is similar to Secure Mode above, **except** the barrier is deactivated during measurements and optical analyses.

Deactivated Mode: the optical barrier is deactivated all the time.

### 3.3.4 CENTER TAB

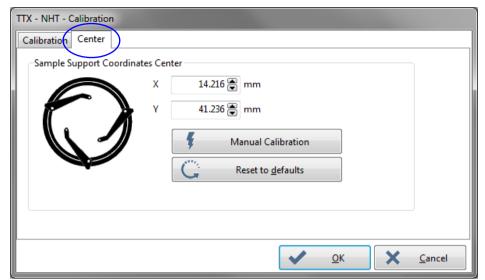


Fig.17 Center tab (e.g. TTX-NHT)

For the description details, refer to the **Common Scratch & Indentation software manual** - **chap. Managing the instrument** - **Distance calibration** - **center calibration**.

- In case of a manual Y table, the Y field is inactive (grayed out); the Y table should be manually moved.
- 1 X and Y field values shown in <u>Fig.17</u> above are only an example; a calibration should have been performed for each available instrument (different field values).

# 3.4 Managing indenters

The indenter which is mounted on the measurement head should be created and/or selected.



Select *Instrument/Indenters...* from the menu bar.

# **Edit indenters**

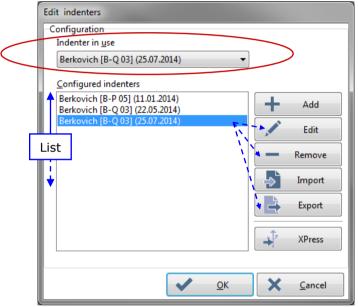
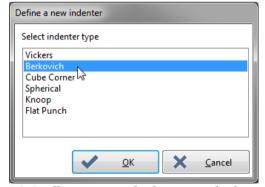


Fig.18 Edit indenter windows



To create a new indenter in the  $\underline{C}$  on figured indenters list.



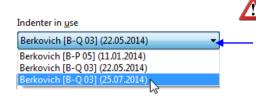
Select (double click) the type of the indenter which is used; see *chap. 3.4.1 Indenter properties window, p. 37*.

Fig.19 Define a new indenter window



To modify the selected (highlighted) indenter in the <u>Configured indenters</u> list; see <u>chap. 3.4.1 <u>Indenter</u> <u>properties window</u>, p. 37.</u>

### Before running a measurement



If there are several <u>Configured indenters</u> in the list, do **not** forget to select the <u>Indenter in use</u> with the last calibration date if available (the <u>Calibration date</u> chap. 3.4.3, p. 47 is displayed next to the indenter serial number).

- To keep the old indenter calibration, create a new indenter recommended.
- To overwrite an indenter calibration, edit the existing indenter; see *chap. 3.4.2 Indenter calibration*, *p. 38*.

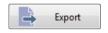


To remove the selected (highlighted) indenter from the *Configured indenters* list.



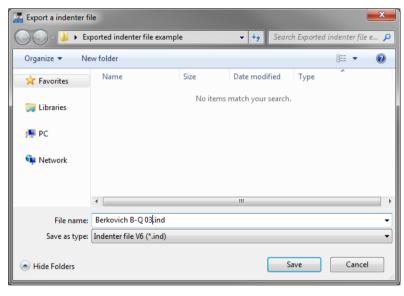
Not applicable.

#### Import/export

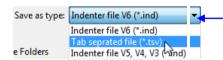


To export the selected (highlighted) indenter in the *Configured indenters* list (saved as an indenter file).

#### This window allows:



- Choosing a location where to save the indenter file.
- Modifying the default File name: corresponding to the selected indenter.
- Changing of the default V6 .IND file format.



Then click Save



To import an indenter (which was exported) in the <u>Configured indenters</u> list.

A similar as for the export above, a window allows choosing a location where to open an indenter file with .IND format.

Then select the file and click open or double click on the file to open it.

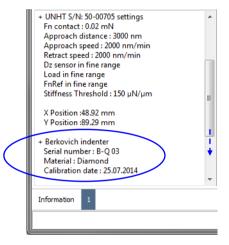
#### Indenter file (.IND) info

The exported/imported indenter .IND file format includes only the result of the calibration: only the **average point** (of the 5 indents kept or excluded from the .MIT file) **for each load**. Therefore it should not be confuse with the indenter calibration .MIT file format which includes all indentation measurements.

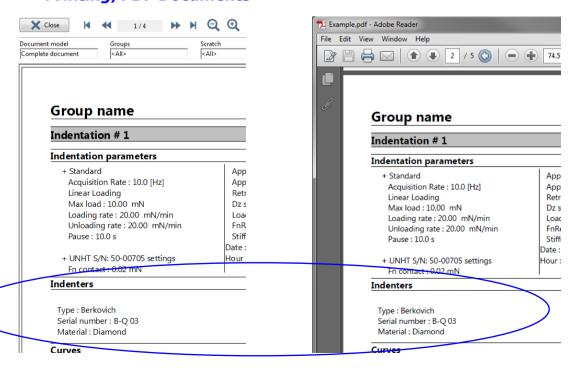
#### **Indenter in results**

Later, after each measurement, the indenter in use appears:

on the bottom left side of the analysis results window; see from chap. 5.11
 Managing the indenter in the result analysis, p. 164.



- in the printed/PDF report (from page 2 and depending on the report option configuration); refer to the Common Scratch & Indentation software manual - chapters:
  - Customizing options (Document model tabs)
  - Printing/PDF Documents



### 3.4.1 INDENTER PROPERTIES WINDOW

+ Add or fig. 18, p. 34.

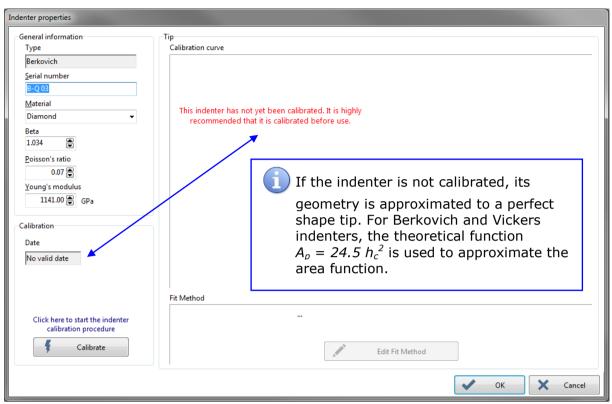
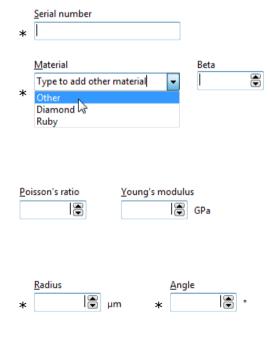


Fig. 20 Indenter properties window with no calibration/date



This field **should** be filled in.

**Adapt** (set) the material and values of **all** other fields which will be used for the measurement analysis results.

- The initial **Beta** value corresponds to the indenter type selected in *Define a* new indenter window <u>Fig. 19</u>, p. 34.
- The initial **Poisson's ratio** + **Young's modulus** values correspond to the diamond material.

If Spherical or Flat Punch indenter type is selected in *Define a new indenter* window *Fig. 19, p. 34* these 2 additional fields appear.

<sup>\*</sup> indicated on the indenter provided certificate



To (re)calibrate the indenter; see the following *chap. 3.4.2 Indenter calibration , p. 38*.

# 3.4.2 INDENTER CALIBRATION (QUASISTATIC/SINUS)

Galibrate is clicked in the *Indenter properties* window <u>Fig.20</u>, p. 37. The calibration procedure starts.

# 3.4.2.1 Measuring a matrix of indentations

- Use a certified Fused silica sample with UNHT and NHT, or a certified BK7 sample with MHT.
- Sinus calibration is not available ( Measuring a new matrix of sinus indentation inactive/grayed out) for MHT and UNHT Bio, and NHT without the **optional** sinus mode; see *chap. 3.1.2.2* NHT, p. 14.

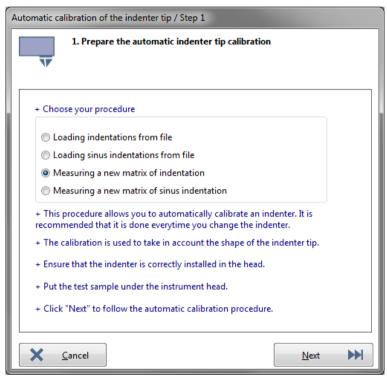


Fig.21 Choose a calibration procedure

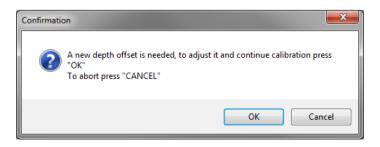
Select 

Measuring a new matrix of indentation for a quasistatic calibration 

Measuring a new matrix of sinus indentation for a Sinus calibration and click

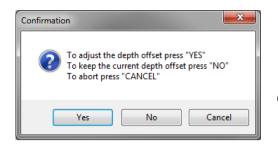
Mext

If the ADO is **not** yet successfully ( ) performed:



Click ok to start a new ADO - for MHT in **fine** Dz range; see **chap. 4.4 Adjusting the depth offset (ADO)**, **p. 56**.

# Otherwise if an ADO has already been **successfully** ( ) performed:



Click № to keep the current ADO (the sample has already been moved with a minimum displacement from the ADO indent → similar sample topography).

### **OR**

Click Yes to start a new ADO (sample has not been moved or has been moved too much from the current ADO indent → the sample topography may vary); see *chap. 4.4*Adjusting the depth offset (ADO), p. 56.

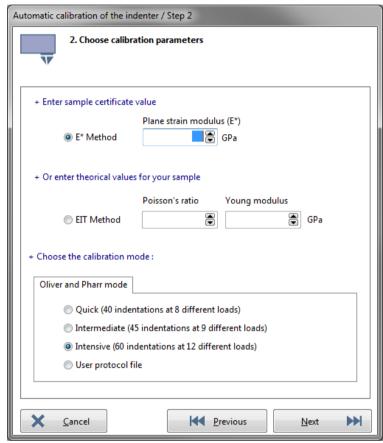
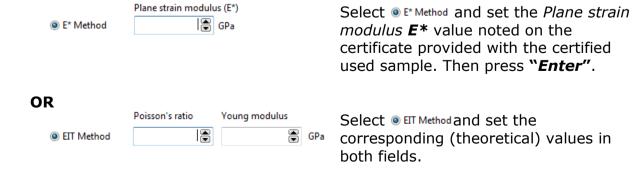
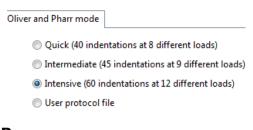
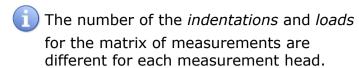


Fig.22 Calibration parameters (e.g. quasistatic UNHT)

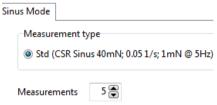




Select a quasistatic calibration mode.



OR

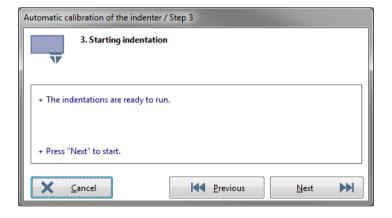


E.g. Sinus for UNHT

The Sinus mode is selected, set Measurements which is the number of indentations for the matrix single row advised value is 5.

Automatic Constant Strain Rate
(CSR Sinus mN), load (mN) and
frequency (Hz) values (to perform later
each calibration measurement) are
different for NHT.



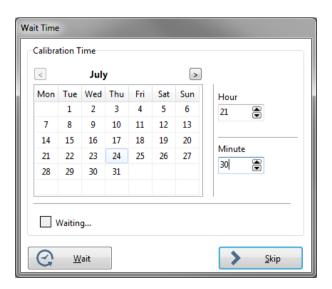


### **Wait time**

<u>N</u>ext

1

Click



### Set a delayed starting



If necessary, to select another measurement date (than today) in the calendar. If necessary, click  $\triangleright$  to go to the next month (and then click  $\triangleleft$  to come back).

To set the hour and minutes of the selected date when the matrix of measurements should start if the below button is clicked.

To wait for the date and time previously set (above), before starting the automatic matrix of measurements:

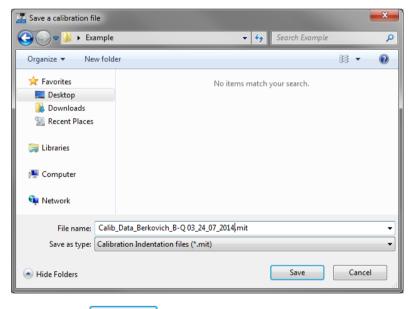
The remaining waiting time until the first measurement starts is displayed Waiting .. 4 H 27 min 25 sec

# OR

# Direct starting



Then the *Indentation running...* window (same as <u>Fig. 58</u>, p. 122) appears; wait for all automatic matrix of measurements are performed. Once the last measurement is completed, the following window appears and allows:



- Choosing a location where to save the calibration file.
- Modification of the default *File name*: calibrated indenter with the date when the 1<sup>st</sup> calibration measurement has been **started**. The default file format is .MIT

Then click Save

Then, the following window appears.

### **Contact area determination**

E.g. with **quasistatic**, they are batches of 5 indent curves measured at different indentation loads: each 5 indents are performed at the same load. For all these indents/curves, e.g. 12 loads x 5 indents = 60 indents/curves, first the contact points should be refined if necessary and then only the relevant indents should be kept  $\rightarrow$  the bad indents should be excluded from the calibration results.

With **Sinus** it is similar, **exception**: the number of indents measured depends on the previous setting (e.g. 5 indents advised) and there are no loads to select e.g. = totally 5 indents/curves.

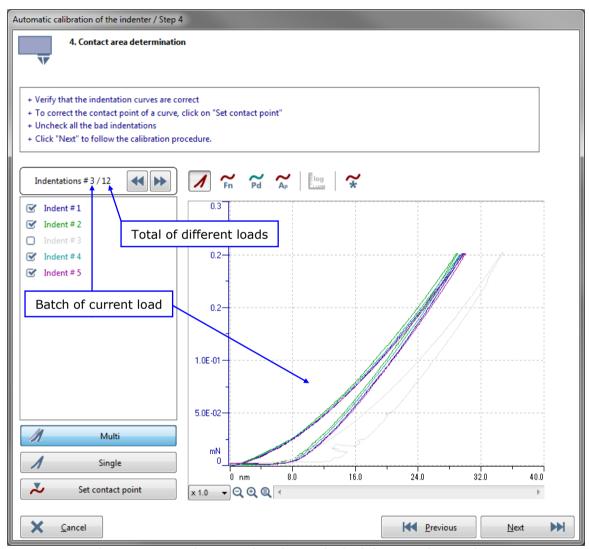
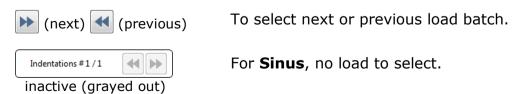


Fig.23 E.g. Indent #3/12 is excluded (non-relevant)





To show **all** curves of the selected load batch.

Place the mouse cursor on one indent of the batch to highlight the corresponding curve.



To show **one** curve of the selected load batch.

Place the mouse cursor on one indent of the batch to display it.

# **Procedure**



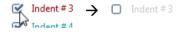
To verify and refine if necessary the contact point for



all indent curves of the current load batch; see chapters 5.1.1.1 Contact point features, p. 133 and 5.1.1.2 Verifying/refining the contact point, p. 135.

Then,

### **Keep only the relevant indents**



e.g. see *Fig.23*, p. 42

Uncheck to exclude (from the calibration results; see *Validation of the results*, *p. 45*.) each non-relevant indent/bad curve, which becomes gray.

If necessary, check it again to re-include it (relevant).

With **quasistatic** mode, repeat the **Procedure above** for **each** load ( e.g. 12 loads , so totally 60 (5x 12) contact points and curves should be verified).

To display the indent curves of the current load ( ) on the graph



F vs. h (combined curves), OR



**F** vs. **time** (force curves), **OR** 

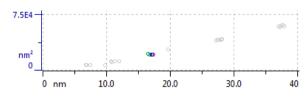


h vs. time (depth curves)

### OR



To display the projected area vs. contact depth:



Only with **quasistatic**, the indents of **all** loads are represented with circles on the graph. Only the relevant indents are displayed in color.



To display graph logarithm scales (selected).



To display only the relevant indents of the **current** load; see Keep only the relevant indents, p. 43:

The non-relevant ( Indent#3 unchecked) indents in gray are hidden from the graph.

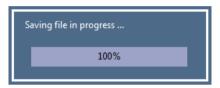


To zoom the indent curves on the graph; for the descriptions, refer to the Common Scratch & Indentation software manual chap. Features on graph areas - Zoom

When all contact points and curves are verified, click



igwedge Any modifications (contact points and exclusion of non-relevant indents/bad curves) are automatically **saved** on the current calibration measurement file .MIT (which were saved after the matrix of measurements or loaded).



The following window appears.

### Validation of the results

According to the previous <u>Contact area determination</u>, from **p. 42**, the red curve fits on the crosses displayed on the graph of the window <u>Fig. 24</u> <u>below</u>:

E.g. with a quasistatic calibration, each cross corresponds to the average of the relevant (kept) indents for each load (e.g. 12 loads = 12 crosses).

With a sinus calibration, each cross corresponds to the average of the relevant (kept) indents of the sinus analysis at the corresponding contact depth (more crosses).

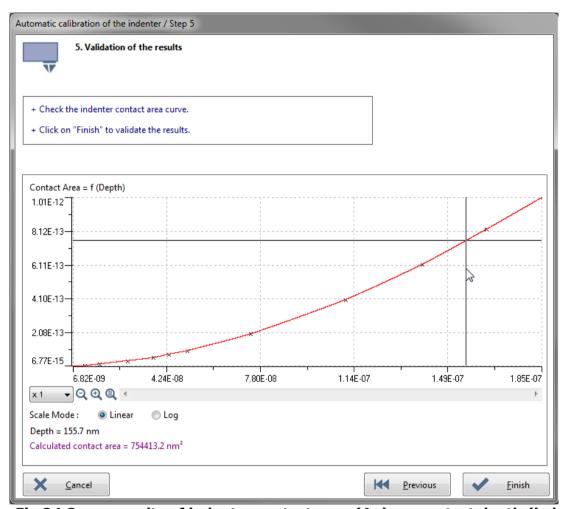


Fig.24 Curve results of indenter contact area (Ap) vs. contact depth (hc)

Select © Log to display graph logarithm scales **OR** select (initial) © Linear to display graph linear scales.

Verify the curve and if necessary click Previous and modify the <u>Contact area</u> <u>determination</u>, p. 42.

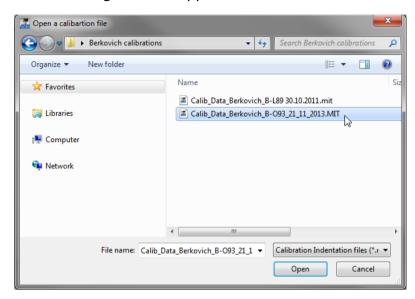
When the curve is suitable, click and see:

- chap. 3.4.3 Calibration date, p. 47.
- chap.3.4.4 Fit methods Ap(hc) of the calibration, p. 48.

# 3.4.2.2 Loading indentations from file

This procedure is similar than for <u>Measuring a matrix of indentations</u> described in *chap. 3.4.2.1, p. 38*, exceptions:

- There are no parts related to perform measurements.
- Select © Loading indentations from file **OR** © Loading sinus indentations from file in <u>Fig. 21</u>, p. 38 (chose a calibration procedure in the <u>Step 1</u> window).
- The following window appears and allows:



- Choosing a location where to open a calibration file with .MIT format.
- Then select the file and click Open or double click on the file to open it.

### 3.4.3 CALIBRATION DATE

Linish is clicked after a calibration in <u>Fig. 24</u>, p. 45 or ledit is clicked in the <u>Edit indenter</u> windows <u>Fig. 18</u>, p. 34 for an indenter already calibrated.

The Curve results of indenter contact area (Ap) vs. contact depth (hc) <u>Fig.24</u>, p. 45 is reported on the graph of the window below.

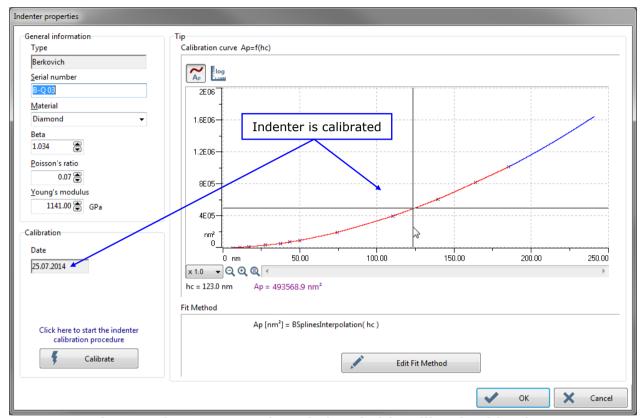


Fig.25 Indenter properties window (with calibration/date)



The displayed indenter calibration date **always** corresponds to the date when the last calibration measurement (weakest force) of the matrix was **ended**.

This date is also displayed next to the serial number of the indenter in the *Edit indenter* windows *Fig.18*, *p. 34*;

The date format depends on the acquisition system setting; e.g. could be with this different format 25/07/2014.

# 3.4.4 FIT METHODS $A_p(h_c)$ OF THE CALIBRATION

Linish is clicked after a calibration in <u>Fig. 24</u>, p. 45 or ledit is clicked in the <u>Edit indenter</u> windows <u>Fig. 18</u>, p. 34 for an indenter already calibrated.

The Curve results of indenter contact area (Ap) vs. contact depth from <u>Fig.24</u>, p. 45 is reported on the graph of the window below.

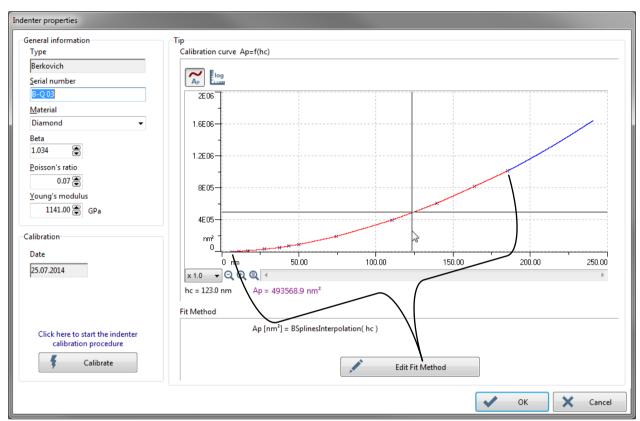


Fig.26 Indenter properties window Ap(hc) curve

# Graph scale display modes

Also applicable in the following *Edit Fit Method* window *Fig.27*, p. 49.



To display graph linear scales, in order to focus on the highest indentation depths: **red** and **blue** part of the curve, e.g. in <u>Fig.26</u> above.

### OR



To display graph logarithm scales, in order to focus on the lowest indentation depths: **green** and **red** parts of the curve, e.g. in the *Fig.27*, p. 49.

## Part of the curve in blue (Fig. 26 above)

Theoretical function  $(A_p=24.5~h_c^{~2}$  for Berkovich and Vickers indenters,  $A_p=\pi h_c(2R-h)$  for a sphere) with a shift in Y ( $\Delta Y$ ) to ensure the continuity on the **last cross** (highest depth) of the calibration.

# Part of the curve in green (Fig.27 below)

For Oliver & Pharr fit methods: sphere equation passing by the 1<sup>st</sup> cross (lowest depth) of the calibration.

For BSpline: spline equation passing by (0;0) and having the same tangent in (0;0) than the sphere equation passing by the  $1^{st}$  cross (lowest depth) of the calibration.

# Part of the curve in red (Fig.26, p. 48 and Fig.27 below)

Click to open the following *Edit Fit Method* window, which allows selection of a method to compute a best fit for the contact area function.

The default fit method is the OBSpline Interpolation. If necessary select another fit method either a OLinear Interpolation, OCO OLIVER & Pharr Or a OPOlynome fit.

For Oliver and Pharr or Polynomial fit methods, the number of the computed coefficients should be adapted in the corresponding field, respectively

Fractional terms: 
Or Degree:

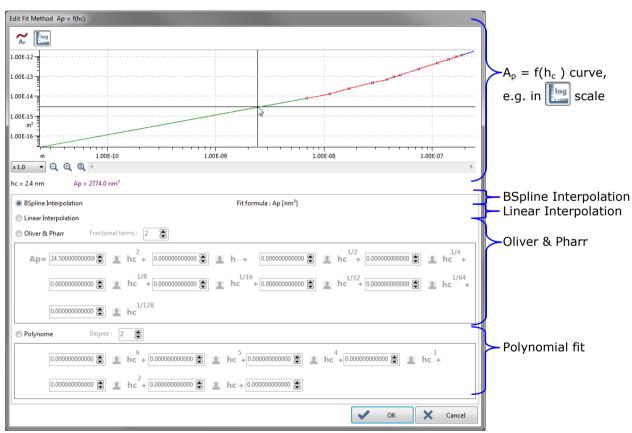


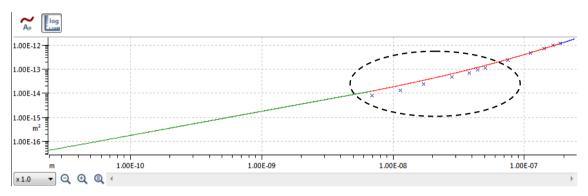
Fig.27 Edit Fit Method window

The following example describes the way to find the best fit with *Oliver & Pharr* fit method. In an ideal case the function should be a line passing by each cross (e.g. 12 crosses).

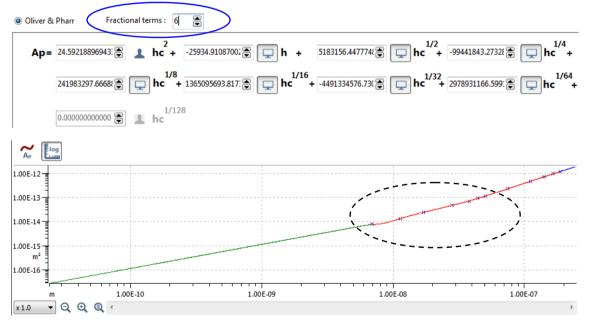
The first term  $h_c^2$ , the lead term, is by default to 24.5 (coefficient for a Berkovich or Vickers indenter).



If a low Fractional terms: (e.g. 0) is used, the fit obtained is not good for some of the lowest indentation depths.



# E.g. 6 fractional terms gives the best fit.



For Oliver and Pharr or Polynomial fit methods, each parameter for the  $A_p$  fit can be manually set by clicking  $\square$ : e.g.  $hc^{1/8}$  + 1357806293|

(Re)click \_ for each filed which should be automatically (re)calculated by the software.

#### 3.5 **UNHT** APPROACH MONITOR WINDOW (.INI FILE MANAGEMENT)



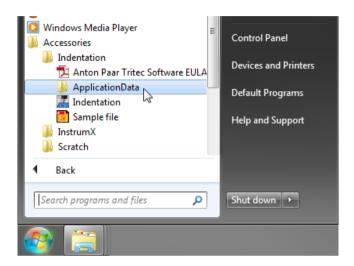
 $oldsymbol{\Lambda}$  Be careful, bad modifications in the UNHT.INI file can have a direct influence on the UNHT behavior/measurements. Therefore in the file Notepad, edit only the lines (highlighted in gray) which are described below and exactly as they are typed (spelling is considered/case sensitive).

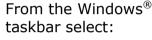


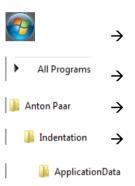
";" in front of each single line of the INI file allows comments.

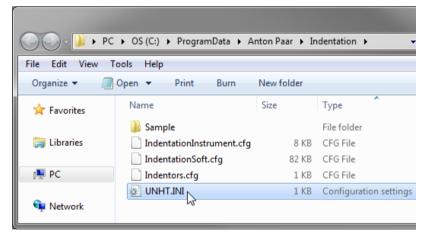
### **INI** file activation

To activate the INI file management directly from the software.



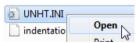






To open the UNHT.INI file in the Windows® Notepad:

- Right click on it and select open in the context menu



- Double click on it.



In the Notepad, at the section [Extra], edit ReloadIni to = 1:

> [Extra] ReloadIni = 1

Save the file and close the Notepad.

Then restart the *Indentation Software* (this should be done only once):

Select *File/Exit* from the menu bar or click on the right top of the main window and start again the software by double clicking on the acquisition system desktop.

Now the .INI file appears in the Instrument menu bar.

### **UNHT Approach Monitor window**

To hide or display later (during ADO process, measurements...) the *UNHT Approach Monitor* window, see the following descriptions.

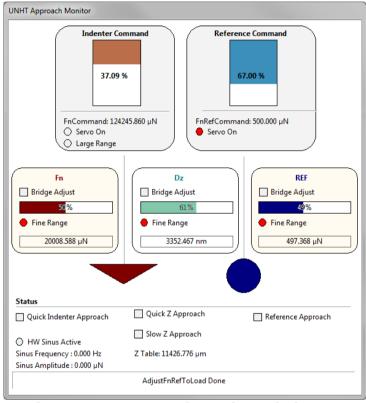
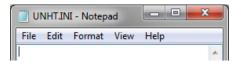


Fig. 28 UNHT Approach Monitor window



Thanks to the **INI file activation**, **p. 51**:

Select *Instrument/*.INI file/Edit.INI file from the menu bar to open the Notepad .INI file.



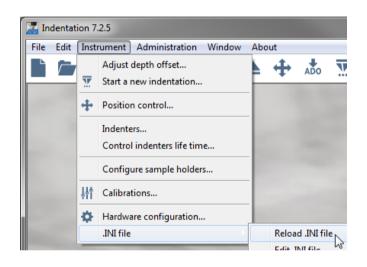
The *UNHT Approach Monitor* window *Fig.28*, *p. 52* can be **deactivated** (will be hidden):

```
In the Notepad, at the section
[Monitoring], edit Show to = 0:
    [Monitoring]
    Show = 0
```

The UNHT Approach Monitor window <u>Fig. 28</u>, p. 52 can be **(re) activated** (will be displayed):

```
In the Notepad, at the section
[Monitoring], edit Show to = 1:
    [Monitoring]
    Show = 1
```

Save the file and close the Notepad.



Do not forget to then select **Instrument/.INI file/ Reload .INI file**. to activate the last .INI file modifications.

(there is no need to restart the software)

#### TAKING A NEW MEASUREMENT 4

#### 4.1 INTRODUCTION

For detailed manipulations and instructions, refer to each corresponding \*Measurement head\* user manual.

#### 4.1.1 **WARNING**



Mhen installing the sample in a sample holder AND when moving the sample/(motorized) tables from the video microscope to the measurement head and vice versa, avoid any collision with:

- the measurement head, especially with the indenter/ reference (UNHT)/reference ring (NHT)/reference fork (MHT)
- the video microscope optical objectives

#### 4.1.2 **NOTES FOR GOOD MEASUREMENTS**

For a maximum accuracy, the sample surface should be always perpendicular to the indenter axis (sample surface leveled). For some specific applications, a special sample holder is required to fulfill this condition.

Each sample should be firmly fixed (clamped, glued for UNHT Bio...) into the sample holder  $\rightarrow$  the sample should not slide when it is indented and also when the (motorized) tables move.

Each sample and sample holder support surfaces should be clean and dust-free. Before performing a measurement (ADO included), the chosen sample surface area should be free of previous indents.

The indenter should be clean and not too much worn out.

The sample temperature should remain stable during measurement.

#### 4.1.3 **GENERALITY**

A new indentation measurement or measurements (matrix of indentations) involve a succession of logical steps, such as:

- 1. Installing the sample
- 2. Choosing the sample area for the measurement(s)
- 3. Adjusting the depth offset (ADO)
- 4. Setting up the configuration/parameters for the measurement(s)
- 5. Running the measurement(s)
- 6. Analysis of the measurement results

Each of these steps is described in the following chapters.

#### 4.2 **INSTALLING THE SAMPLE**

- 1. To move the motorized tables to safe position (max. right front table position), click  $\triangleq$  on the toolbar.
- 2. Install firmly the sample into the sample holder.

#### 4.3 CHOOSING THE INDENTATION AREA

For all information about the *Position control* window features (management of the indenter/video microscope areas, different methods to move with different speed...), refer to the Common Scratch & Indentation software manual chap. Managing the instrument - Control of the sample position.



always verify the sample height before moving.

- 1. To choose the sample measurement area, click + on the toolbar. In the *Position control* window which appears:
- 2. To move the sample/motorized tables under the video microscope click The microscope zone should be activated (under microscope icon), Open video

For the video microscope module with STeP 4/6: to lower the module (if not already at the lowest position) click .

3. To adjust the focus of the sample surface on the Video screen, move the motorized Z table: click to raise / to lower.

With a TTX-NHT, use the coarse and fine focusing thumbwheels.

4. To move the sample/motorized X-Y tables in order to choose the measurement area on the Video screen (under the middle of the crosshair), click

With a TTX, the non-motorized table(s) should be manually move.

When exiting the *Position control* window, the motorized table automatically retracts.

Then the sample will be automatically moved under the indenter, at the chosen measurement area, before starting the ADO procedure  $(\frac{1}{1000})$  and then before starting the measurement procedure  $(\overline{\mathbf{v}})$ .

# 4.4 ADJUSTING THE DEPTH OFFSET (ADO)

The ADO procedure is carried out to correctly setup the depth sensor measurement range for the following indentation calibration/measurement(s):

- Because of the unpredictable sample surface topography.
- To optimize the duration of the measurement(s).

This procedure should be performed:

- If the sample surface topography may vary between the indentation measurements.
- If the indentation measurements are performed on multi-sample where the surface topography varies.
- After changing the sample.
- After changing the indenter.
- Only for UNHT, after changing the reference.
- After restarting the software.

Before performing this procedure, the current position (measurement area) on the sample surface should be free of previous indents and clean.

# Manual ADO procedure starting



Select *Instrument/Adjust depth offset...* from the menu bar or click  $\stackrel{*}{\not{\sim}}$  /  $\stackrel{*}{\not{\sim}}$  on the toolbar to start the following ADO step by step procedure windows:

- **1**<sup>st</sup> Adjust depth offset parameters window
- 2<sup>nd</sup> Adjust depth offset in progress window for the automatic (semi-automatic for MHT without electronic bridge) process (this window includes several sequence steps) an indentation is performed.
- **3<sup>rd</sup>** Sample displacement after ADO window

## Automatic ADO procedure starting

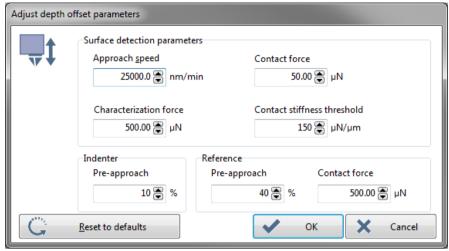
The procedure can be automatically started from some calibration/measurement processes **if** the ADO has never been performed or if the last ADO is not successful ( $\frac{1}{100}$ ).

The **1**<sup>st</sup> and **2**<sup>nd</sup> windows described above are different depending on the type of the measurement head which is used. All of them are described in the 3 following chapters.

At the end of the procedure, if the ADO has been **successfully** performed, a green disk with a tick appears on the ado icon  $\Re$  (main toolbar). If not,  $\Re$  appears and the procedure should be performed again until it is successful.

# 4.4.1 UNHT/UNHT BIO ADO

The  $\mathbf{1}^{st}$  procedure window allows setting of the parameters which will be used to perform the ADO automatic process ( $\mathbf{2}^{nd}$  window).

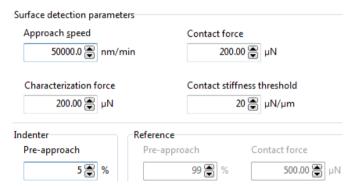


E.g. parameters shown on *Fig.29* are the defaults values which are normally suitable to perform a successful ADO with the UNHT.

Otherwise set other field values according to the application.

Fig.29 UNHT ADO parameters window

There is no reference with UNHT Bio, therefore the corresponding field are inactive (grayed out).



E.g. parameter values shown on <u>Fig.30</u> are normally suitable to perform a successful ADO with the UNHT Bio.

Otherwise set other field values according to the application.

Fig.30 UNHT Bio ADO parameters window



To reset all fields with the default values.

# Indenter Pre-approach and Reference Pre-approach

 $100\ \%$  is the rest position corresponding to the fully extended (lowest) position of each actuator displacement.

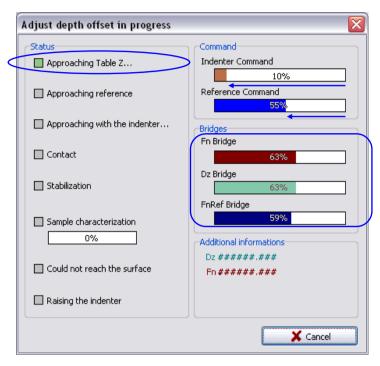
Rest position for the motorized Z table is the retracted (lowest) position; see *Table Z retraction*, *p. 31*.

For **Contact stiffness threshold** details, see identical information as described in **chap.** 4.6.5.8 (indentation measurement parameters), **p. 119**.

Click or to start the following automatic ADO process.

The  $2^{nd}$  procedure window is performing the automatic ADO process (steps). If necessary, click  $\times$  Cancel to stop the process and cancel the ADO. Otherwise wait until the  $3^{rd}$  procedure window appears.

1 Steps concerning the reference are not applicable with UNHT Bio.



With **STeP 4/6**, the motorized head module approaches (moves down).

Then the indenter and reference retract (move up) to the previously set values:



except +15 % for the reference.

Then Fn, Ref and Dz sensor bridges are automatically adjusted.

And then the motorized Z table slowly approaches (moves up).

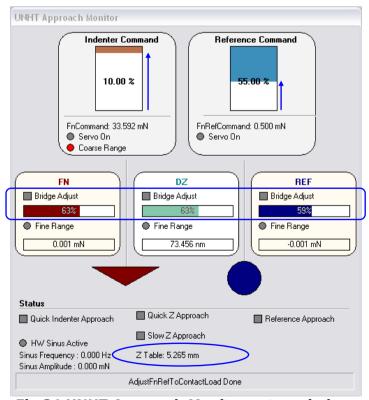


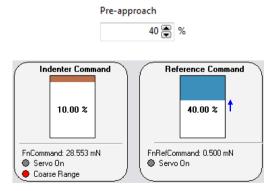
Fig.31 UNHT Approach Monitor extra window

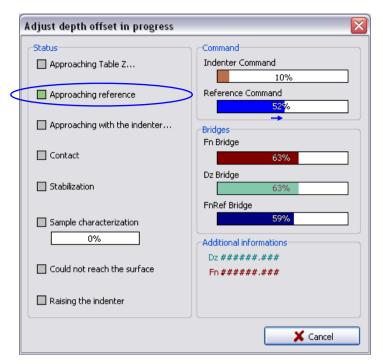
The extra UNHT Approach

Monitor window Fig.31 can be hidden or displayed with the UNHT.INI file; see chap. 3.5, p. 51.

When the sample surface contacts the reference, the motorized Z table stops and the reference actuator is retracted (moves up) by **15** % → return to the previously set value:

Reference

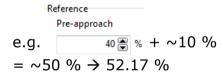


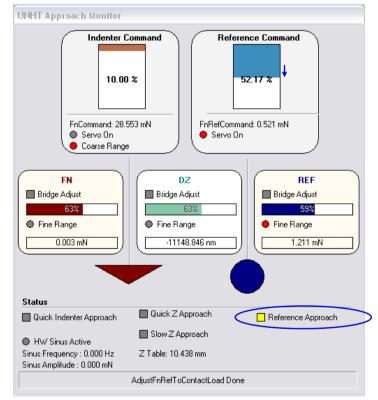


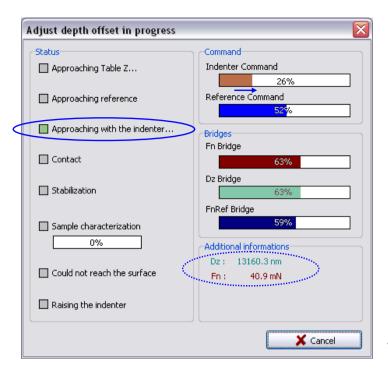
The reference approaches (moves down) until it reaches the previously set value:

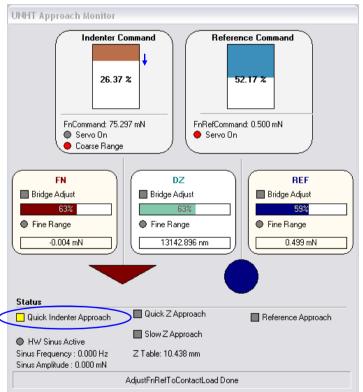


The reference touches
~10 % above the previously set position:







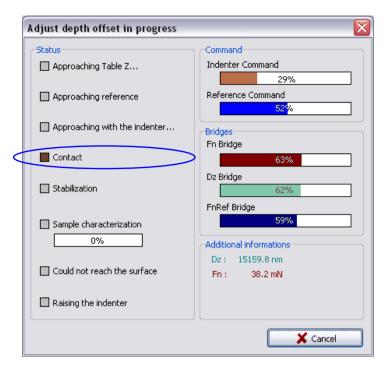


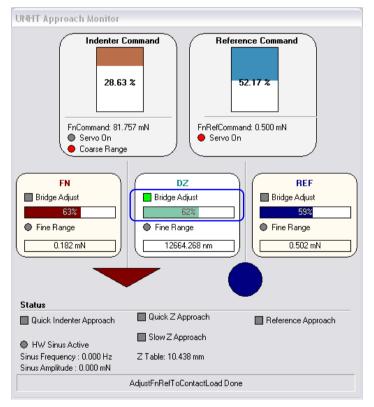
The indenter approaches (moves down) with the previously set:

Approach <u>s</u>peed

25000.0 nm/min

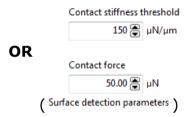
until ...



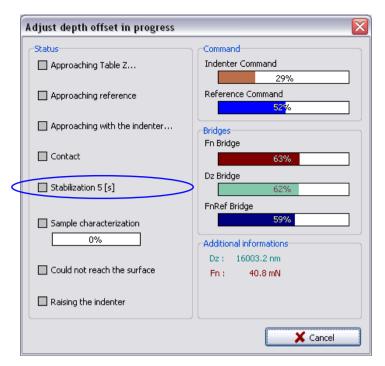


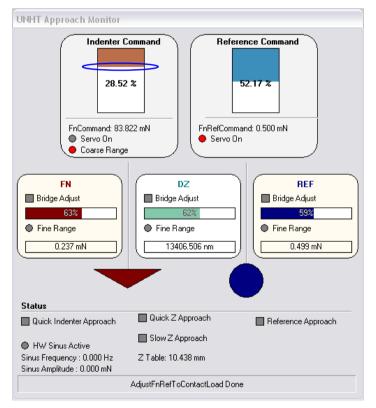
... it contacts the sample surface:

The indenter has either reached the previously set:



Then the Dz sensor bridge is adjusted.

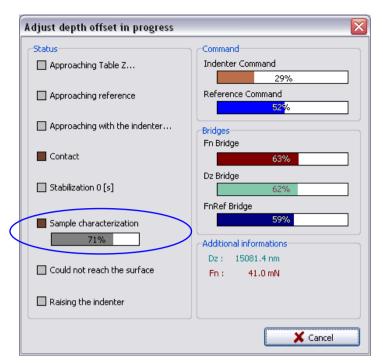


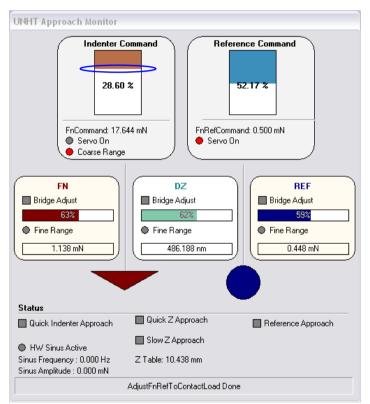


(indenter is still in contact)

Wait for the stabilization; see chap. 3.1.4.2 <u>Stabilization</u> (cancel drift), p. 26.

The indenter position slightly varies.

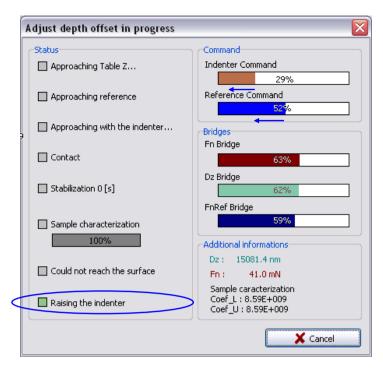




An indentation with the previously set:



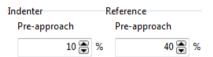
is performed to evaluate the sample mechanical properties; the indenter position slightly varies.



UNHT Approach Monitor Indenter Command Reference Command 10.00 % 40.00 % FnCommand: 57.545 mN FnRefCommand: 0.500 mN Servo On Coarse Range FN DΖ REF Bridge Adjust Bridge Adjust Bridge Adjust Fine Range Fine Range Fine Range 0.474 mN -0.048 mN -3774.827 nm Status Quick Z Approach Quick Indenter Approach Reference Approach Slow Z Approach HW Sinus Active Sinus Frequency: 0.000 Hz Z Table: 10.438 mm Sinus Amplitude: 0.000 mN AdjustFnRefToContactLoad Done

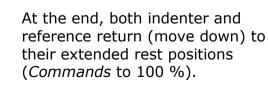
The ADO process is completed.

The indenter and reference return/retract (move up) to the previously set values:



Then the motorized Z table retracts (moves down) to its rest position.

And then with **STeP 4/6**, the motorized head module retracts (moves up).

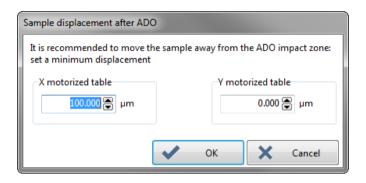


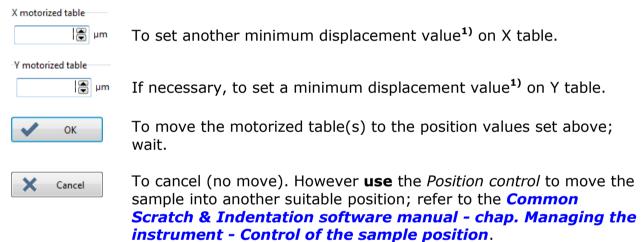
### Sample displacement after ADO

After the ADO process, the 3<sup>rd</sup> procedure window allows setting of a minimum displacement to move the sample.



 $\bigwedge$  To **avoid** performing the following calibration/measurement indent inside the ADO indent (current position), the sample/motorized table(s) position should be shifted of a minimum displacement<sup>1)</sup> (otherwise the sample topography may vary).



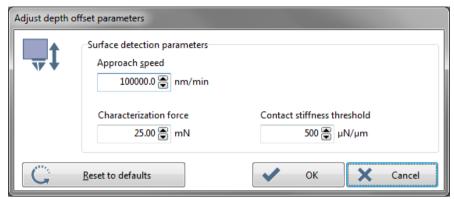


If the ADO procedure has been **successfully** performed, a green disk with a tick appears on  $\Re$  (main toolbar).

<sup>1)</sup> it depends on the current sample material: approx. 20 x  $h_m$ 

### 4.4.2 NHT ADO

The  $\mathbf{1}^{st}$  procedure window allows setting of the parameters which will be used to perform the ADO automatic process ( $\mathbf{2}^{nd}$  window).



E.g. parameters shown on <u>Fig.32</u> are the default values which are normally suitable to perform a successful ADO.

Otherwise set other field values according to the application.

Fig.32 NHT ADO parameters window



To reset all fields with the default values.

For **Contact stiffness threshold** details, see identical information as described in **chap.** <u>4.6.5.8</u> (indentation measurement parameters), **p. 119**.

Click to start the following automatic ADO process.

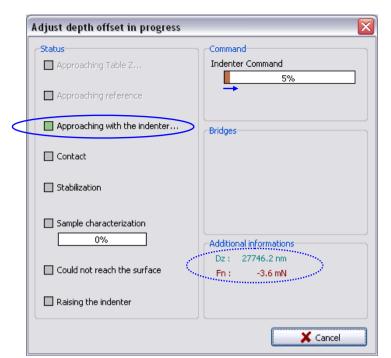
The  $2^{nd}$  procedure window is performing the automatic ADO process (steps). If necessary, click  $\times$  Cancel to stop the process and cancel the ADO. Otherwise wait until the  $3^{rd}$  procedure window appears.



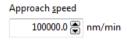
With **STeP 4/6** and **TTX**, the motorized head module approaches (moves down).

Then with **STeP 4/6**, the Z motorized table approaches (moves up) until the sample contacts the reference ring.

And then the indenter preapproaches (moves down) and the Dz sensor bridge is automatically adjusted a 1<sup>st</sup> time.



The indenter approaches (moves down) with the previously set:



until ...

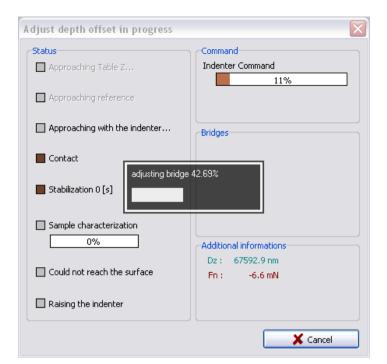


... it contacts the sample:

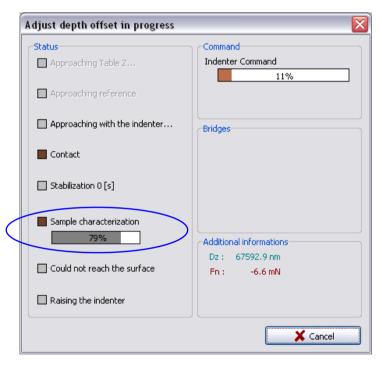
The indenter has reached the previously set:



Then wait for the stabilization; see *chap. 3.1.4.2* <u>Stabilization</u> (cancel drift), p. 26.



The Dz sensor bridge is automatically adjusted a 2<sup>nd</sup> time.



An indentation with the previously set:



is performed to evaluate the sample mechanical properties.



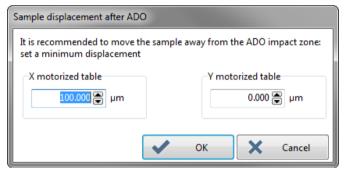
The ADO process is completed.

The indenter is retracted (rises) until top position (0 %).

With STeP 4/6 and TTX, the motorized head module retracts (moves up).

Then the STeP 4/6 Z motorized table retracts (moves down).

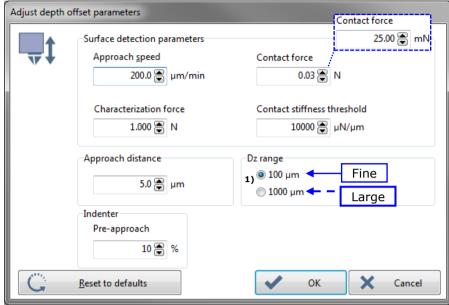
Read Sample displacement after ADO, p. 65 (same description as for the UNHT)



If the ADO procedure has been **successfully** performed, a green disk with a tick appears on  $\Re$  (main toolbar).

### 4.4.3 MHT ADO

The  $\mathbf{1}^{st}$  procedure window allows setting of the parameters which will be used to perform the ADO semi-automatic process ( $\mathbf{2}^{nd}$  window).



E.g. parameters shown on *Fig.33* are the default values which are normally suitable to perform a successful ADO.

Otherwise set other field values according to the application.

1) Dz range values can be different for old MCT versions.

Fig.33 MHT ADO parameters window



To reset all fields with the default values.

### Dz range



If the ADO procedure is started from the indenter calibration

( Calibrate chap. 3.4.1 <u>Indenter properties window</u>, p. 37), the fine Dz range must be selected (e.g. 100 µm).

Otherwise select a suitable *Dz range* according to the sample used/application; it is recommended to use the fine *Dz range* but if the depth saturates, then select the large *Dz range*.



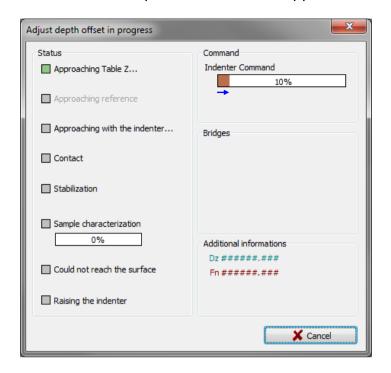
The measurement preferences **Depth range** which will be selected later in **chap. 4.6.5.6**, **p. 117 should** be the same as the ADO **Dz range** selected for the current successful ADO (fine: e.g. 100 µm). Otherwise an error message will appear; see **chap. 6.2**, **p. 173**.

The **Indenter Pre-approach** parameter moves down the indenter out of its rest position (top end stop 0 %) before the first approach step. The minimum and recommended value for this parameter is 10 %.

For **Contact stiffness threshold** details, see identical information as described in **chap.** 4.6.5.8 (indentation measurement parameters), **p. 119**.

Click to start the following semi-automatic ADO process.

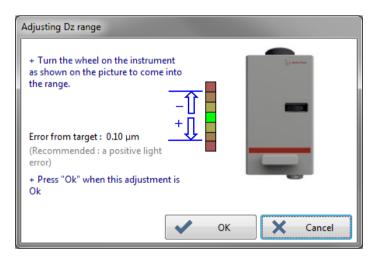
The  $2^{nd}$  procedure window is performing the semi-automatic ADO process (steps). If necessary, click  $\times$  Cancel to stop the process and cancel the ADO. Otherwise wait until the  $3^{rd}$  procedure window appears.

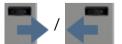


The indenter pre-approach (moves down) to the previously set value:









1) the rotation direction (an arrow) is displayed on this extra *Adjusting Dz range* window

Then click ✓ OK .

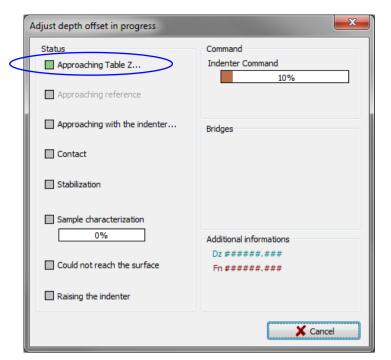
## Dz range adjustment

Turn<sup>1)</sup> the wheel to adjust the previously set:

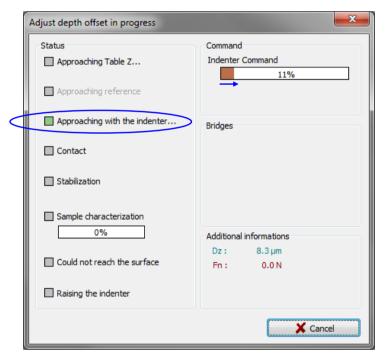


for the following indentation calibration or measurement(s) which will be performed later, after this ADO procedure:

With the special tool long pin (or with the old centering tool, or a small Allen key), turn<sup>1)</sup> the wheel to adjust the Dz vertical cursor preferably into the central green position or into any yellow (down: positive error from target/up: negative error from target).



Then the Z motorized table approaches (moves up) until the sample contacts the reference fork.

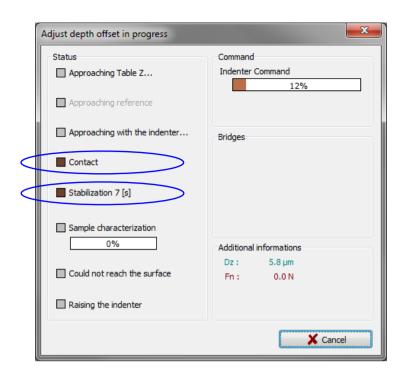


The indenter approaches (moves down) with the previously set:

Approach <u>s</u>peed

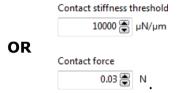
200.0 μm/min

until ...



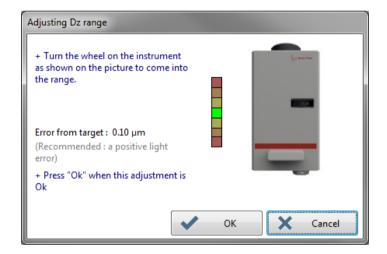
... it contacts the sample:

The indenter has either reached the previously set:

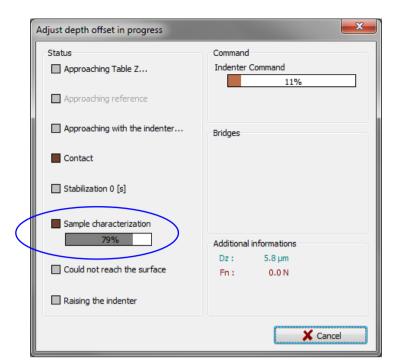


Then wait for the stabilization; see *chap. 3.1.4.2* <u>Stabilization</u> (cancel drift), p. 26.





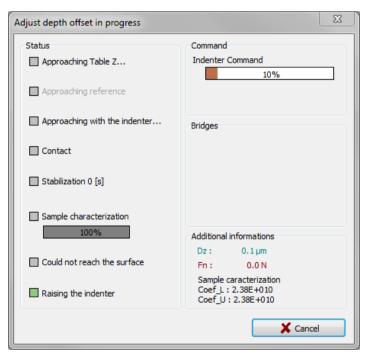
Same information as described in <u>Dz range adjustment</u>, p. 71 (for the other Dz range).



An indentation with the specified characterization force



is performed to evaluate the sample mechanical properties.



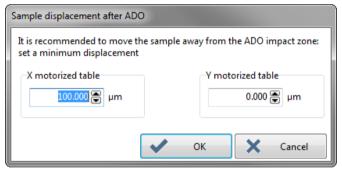
The ADO process is completed.

The Z motorized table retracts (moves down).



The indenter is retracted (rises) until top position (0 %).

Read Sample displacement after ADO, p. 65 (same description as for UNHT).



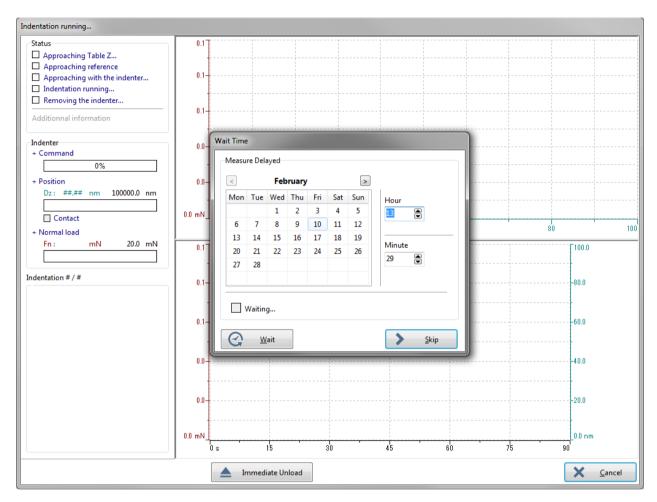
If the ADO procedure has been **successfully** performed, a green disk with a tick appears on  $\Re$  (main toolbar).

# 4.5 DELAYING THE MEASUREMENT(S)

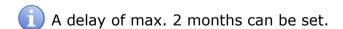
A delay for any measurement can be set and activated.

Click  $\bigcirc$  on the toolbar to enable it  $\bigcirc$  (re-click to disable).

When is enabled: Before any measurement is started<sup>1)</sup>, the *Wait time* window appears.

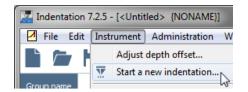


See the same information than described in <u>Wait time</u>, p. 40 (for the indenter calibration matrix of measurements), **exception**: it is applicable for a single measurement or for a matrix of measurements.



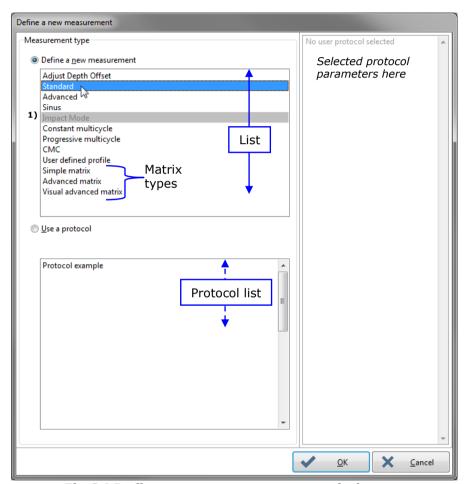
<sup>1)</sup> see chap. 4.6.4 Measurement type parameters, p. 81

## 4.6 SELECTING THE MEASUREMENT TYPE & SETTING ITS PARAMETERS



Select Instrument/Start a new indentation... from the menu bar or click  $\overline{\mathbf{y}}$  on the toolbar.

The window below allows selection of a new measurement type (single indentation measurement or **matrix** of several indentation measurements).



In the list, select an active measurement type (double click).

## **OR**

Select a protocol in the protocol list; see *chap.* <u>4.6.6</u>, *p.* 121 (how to save a protocol).

(grayed out)
measurement
type cannot be
selected as it is
not applicable
for the current
measurement
head

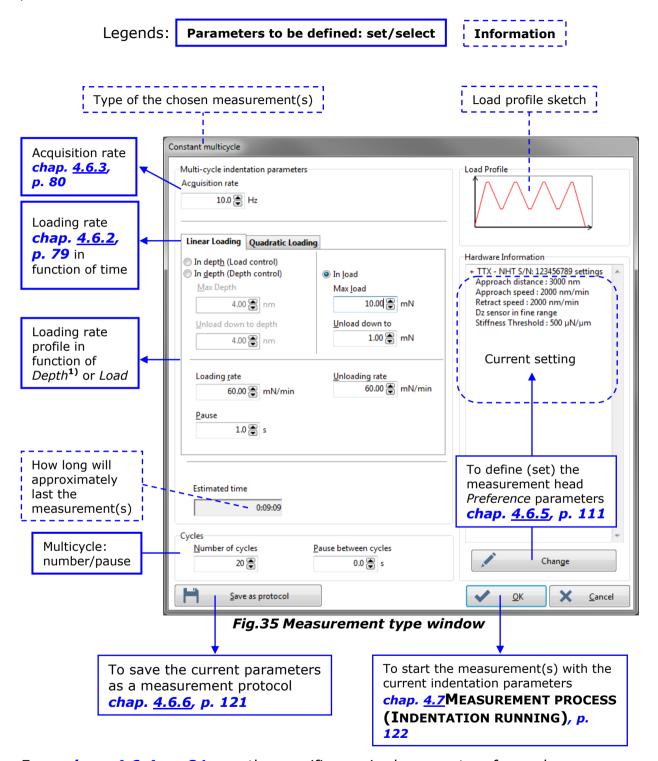
Fig.34 Define a new measurement window

Then the corresponding measurement type window will appear and allow setting the required parameters before starting the measurement(s); see the following chapters for:

- the main **description** of a measurement type window **chap. 4.6.1**, **p. 78**
- the common **loading rate** parameter description **chap.** <u>4.6.2</u>, **p. 79**
- the common acquisition rate parameter description chap. 4.6.3, p. 80
- all required parameters for each measurement type window; see from chap. 4.6.4, p. 81
- the preference parameters dedicated for each measurement head;
   see from chap. 4.6.5, p. 111

## 4.6.1 MAIN DESCRIPTION OF A MEASUREMENT TYPE WINDOW

A measurement type is selected in the *Define a new measurement* window <u>Fig.34</u>, p. 77.



From *chap.* <u>4.6.4</u>, *p.* **81**, see the specific required parameters for each measurement type window.

<sup>1)</sup> with Load or Depth control

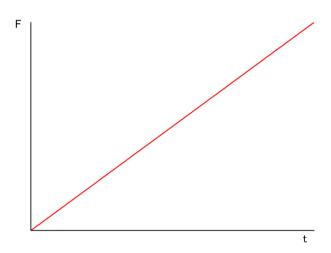
## 4.6.2 LOADING PROFILES

The measurement(s) will be performed with one of the loading profiles described below. Depending on the selected measurement type (in *Define a new measurement* window *Fig.34*, p. 77), several loading profiles are proposed (one can be selected).

# 4.6.2.1 Linear loading/Constant time loading

The linear loading or constant time loading applies the indenter force following the equation below:

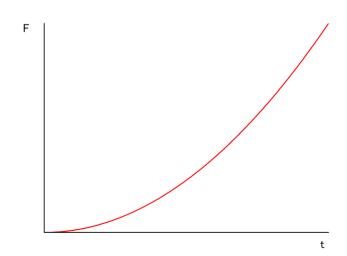
$$F = k \cdot t$$



# 4.6.2.2 Quadratic loading

The quadratic loading applies the indenter force following the equation below:

$$F = k \cdot t^2$$

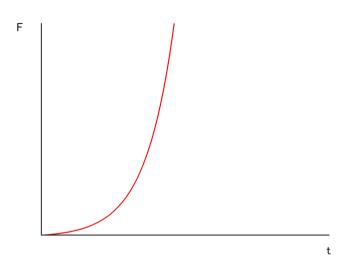


# 4.6.2.3 Constants strain rate (CSR)

This profile is recommended for materials having viscoelastic properties.

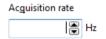
The constant strain rate (CSR) applies the indenter force following the equation below:

$$\frac{dP}{dt} \cdot \frac{1}{P} = cte$$

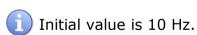


# 4.6.3 COMMON ACQUISITION RATE PARAMETER

Common parameter to all measurement types; see from chap. 4.6.4, p. 81.



To set the acquisition rate. The value corresponds to the number of data point recorded per second during the measurement(s).



Min. value is 1 Hz and max. value is 400 Hz

## However, for:

- Standard measurement type chap. 4.6.4.1, p. 81, there is no field to set, the value is fixed to 10 Hz
- Sinus measurement type **chap. 4.6.4.3**, **p. 85**, there is no field to set, the value is automatically calculated (20x the Sinus frequency value)
- User defined profile measurement type **chap.** 4.6.4.7, **p. 95**, it depends on the load profile segments which are created.

## 4.6.4 MEASUREMENT TYPE PARAMETERS

A measurement type is selected in the *Define a new measurement* window <u>Fig.34</u>, p. 77.

See the following sections which show all windows of each measurement type and describe the corresponding parameters which should be set before starting the measurement(s).

## 4.6.4.1 Standard

The *Standard* measurement type is the simplest **single** indentation measurement, performed in **1 cycle** of automatic linear loading/unloading *chap. 4.6.2.1, p. 79*.

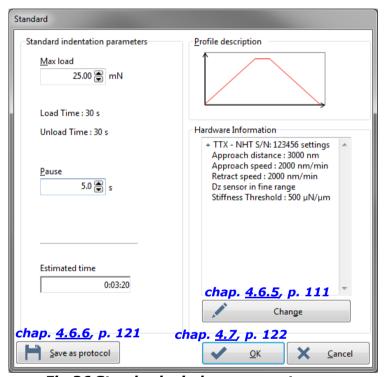


Fig.36 Standard window parameters

About the acquisition rate, see *chap.* 4.6.3, *p.* 80.

<u>M</u> ax load   → mN	To set the maximum load.
Load Time: 30 s	A linear loading rate will be computed to reach the maximum load (set above) within 30 sec.
Pause	To set a pause at the maximum load before unloading.
1 👿 3	Set the value to 0 for "no pause".
Unload Time: 30 s	After the pause, a linear unloading rate will be computed to fully unload within 30 sec.

#### 4.6.4.2 Advanced

The *Advanced* measurement type is a **single** indentation measurement, performed in **1 cycle** of loading/unloading defined by the user.

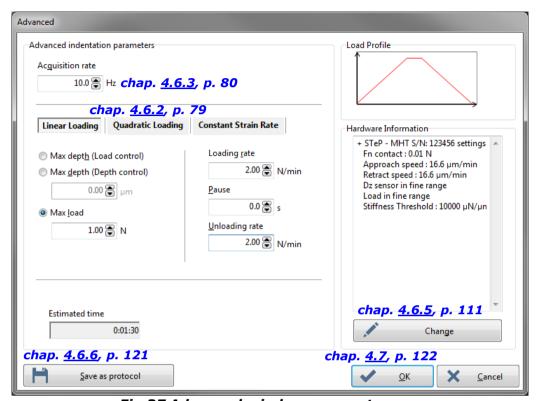


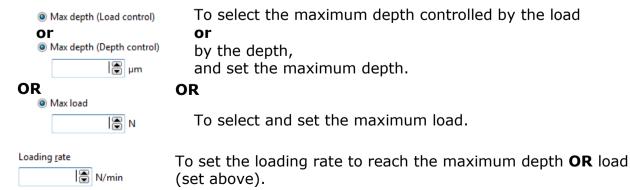
Fig.37 Advanced window parameters

Linear Loading | Quadratic Loading | Constant Strain Rate | To select a loading profile. The corresponding set of parameters is described in the following sections:

<u>Linear loading</u> below <u>Quadratic loading</u> p. 83 <u>Constant strain rate (CSR)</u> p. 84

#### **Linear loading**

See *Fig.37* above.



#### Hazardous result

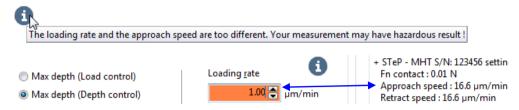


with Max depth (Depth control) selected, if Approach speed: value in Preferences window

Loading rate

N/min

(see chap. 4.6.5, p. 111) is at least 4 times faster than value, the measurement **may have** hazardous results.



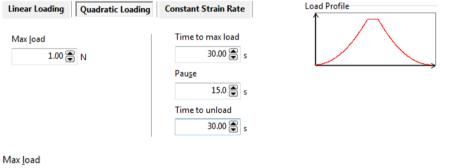
Pause S 🗬 To set a pause at the maximum load **OR** depth (set above) before unloading.

Set the value to 0 for "no pause".

Unloading rate N/min

To set the unloading rate to fully unload after the pause (set above).

## **Quadratic loading**



To set the maximum load. I∰ N

To set the time to reach the maximum load (set above). 

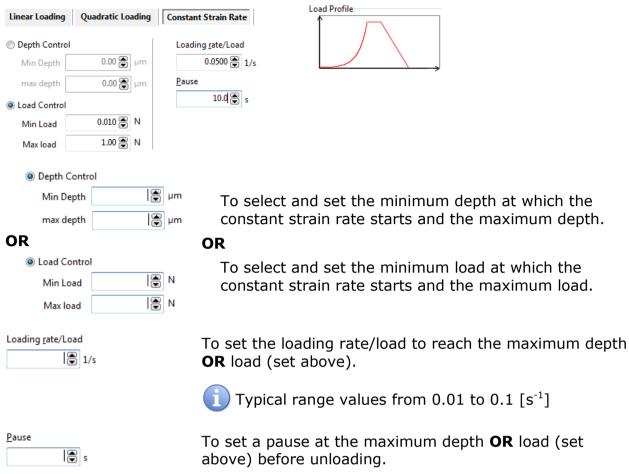
Pause S S

To set a pause at the maximum load (set above) before unloading. Set the value to 0 for "no pause".

Time to unload 

To set the time to fully unload after the pause (set above).

## Constant strain rate (CSR)



Set the value to 0 for "no pause".

A linear unloading rate to fully unload will be automatically performed after the pause (set above).

#### 4.6.4.3 Sinus

Sinus measurement type is not available (Sinus inactive/grayed out) in the list of <u>Fig.34</u>, p. 77 for MHT, UNHT Bio, and NHT without the **optional** Sinus mode (see **chap. 3.1.2.2 NHT**, **p. 14**).

The *Sinus* measurement type is a **single** indentation measurement, performed in **1 cycle** of loading/unloading defined by the user. A sine wave is added during the loading, and if necessary during the pause/unloading. This measurement type allows obtaining a depth related analysis; see *chap.* **5.7.1**, *p.* **153**.

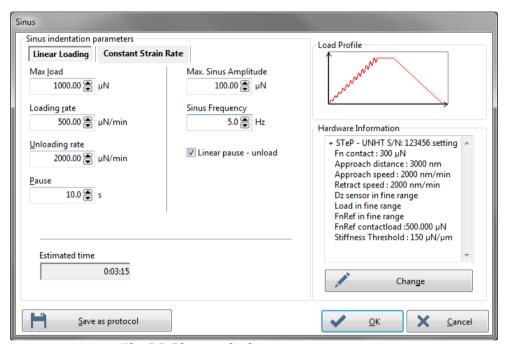


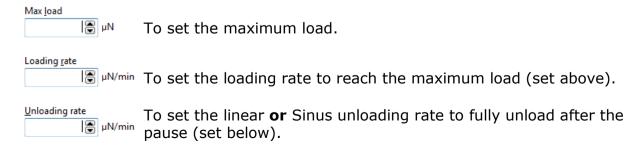
Fig.38 Sinus window parameters

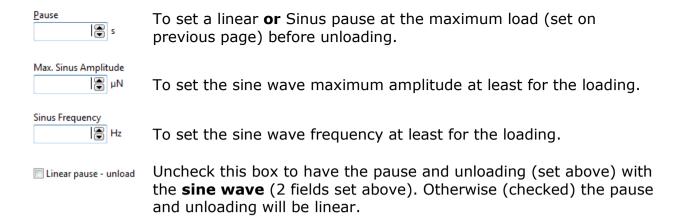
Linear Loading | Constant Strain Rate | To select a loading profile. The corresponding set of parameters is described in the following sections:

<u>Linear loading</u> below <u>Constant strain rate (CSR)</u> p. 86

## **Linear loading**

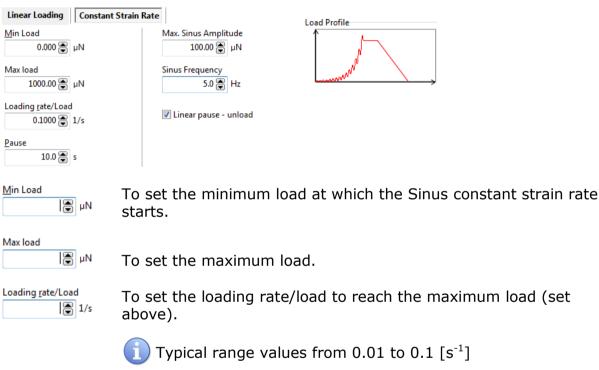
See *Fig.38* above.





## **Constant strain rate (CSR)**

the pause (set below).



An automatic linear **or** Sinus unloading g rate will be performed to fully unload after

To set a pause at the maximum load (set above) before unloading.

Max. Sinus Amplitude

Sinus Frequency

Hz

Linear pause - unload

## 4.6.4.4 Constant multicycle

The *Constant multicycle* measurement type is a **single** indentation measurement, performed in **several cycles** of loading/unloading defined by the user. Maximum and minimum depths or loads are the same for all measurement cycles.

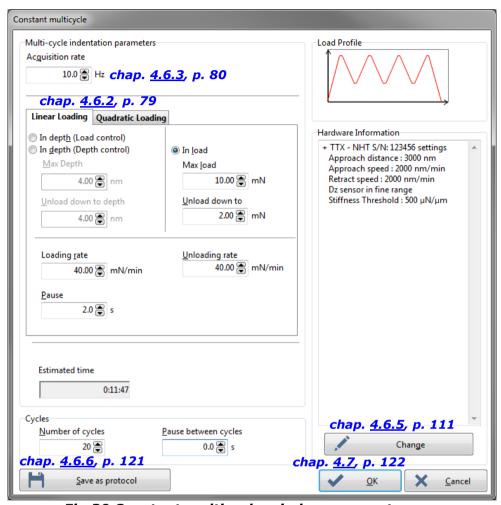


Fig.39 Constant multicycle window parameters

To select a loading profile. The corresponding set of parameters, the **same** for all cycles, is described in the following sections:

<u>Linear loading</u> p. 88 <u>Quadratic loading</u> p. 89

## **Cycles**

Number of cycles

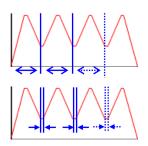
To set the number of the cycles.

Min. value = 2

Pause between cycles

To set a pause between all cycles (set above).

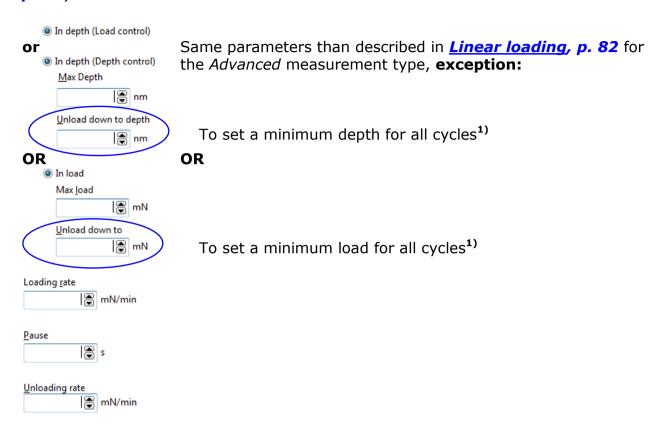
Set the value to 0 for "no pause".



## **Linear loading**

See Fig. 39, p. 87.

The **same** parameters below will be applied for all cycle repetitions (set in *Cycles*, p. 87).



<sup>1)</sup> except for the last cycle which will be fully unloaded

# **Quadratic loading**

Linear Loading Quadratic Loading		Load Profile
Max load  10.00 → mN	Time to max load  15.00 🖝 s	
Unload down to 2.00 ⊕ mN	Pause  2.0  s  Time to unload  15.00  s	

The **same** parameters below will be applied for all cycle repetitions (set in <u>Cycles</u>, **p. 87**).

Max <u>l</u> oad  ⊕ mN	Same parameters than described in <i>Quadratic loading</i> , <i>p. 83</i> for the <i>Advanced</i> measurement type, <b>exception</b> :
Unload down to  □ mN	To set a minimum load for all cycles.
Time to max load	
Pau <u>s</u> e	
Time to unload	

<sup>1)</sup> **except** for the last cycle which will be fully unloaded

## 4.6.4.5 Progressive multicycle

The *Progressive multicycle* measurement type is a **single** indentation measurement, performed in **several cycles** of loading/unloading defined by the user. The only difference with the *Constant multicycle* measurement type is that the **maximum** depths or loads increase for each measurement cycle (the minimum depths or loads remain the same for all measurement cycles).

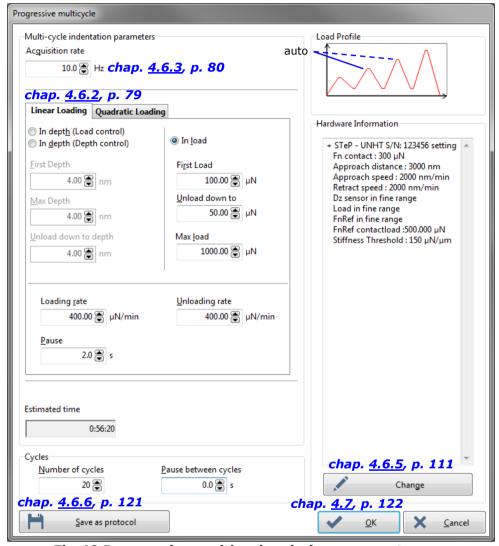


Fig.40 Progressive multicycle window parameters

Linear Loading Quadratic Loading To select a loading profile. The corresponding set of parameters is described in the following sections:

<u>Linear loading</u> p. 91 <u>Quadratic loading</u> p. 92

#### **Cycles**

Number of cycles

Pause between cycles

Same parameters than described in <u>Cycles</u>, p. 87 of the Constant multicycle measurement type.

## **Linear loading**

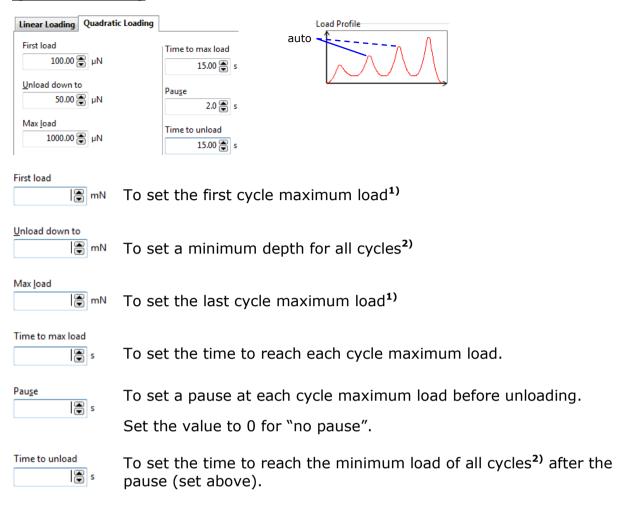
See *Fig.40*, p. 90.

In depth (Load control)	To select the depth mode controlled by the load <b>or</b>
In depth (Depth control)	by the depth and
First Depth	To set the first cycle maximum depth <sup>1)</sup>
Max Depth	To set the last cycle maximum depth <sup>1)</sup>
Unload down to depth	To set a minimum depth for all cycles <sup>2)</sup>
OR	OR To select the load mode and
Fi <u>r</u> st Load	To set the first cycle maximum load <sup>1)</sup>
<u>U</u> nload down to    → mN	To set a minimum load for all cycles <sup>2)</sup>
Max <u>l</u> oad   ♠ mN	To set the last cycle maximum load <sup>1)</sup>
Loading <u>r</u> ate	To set the loading rate to reach each cycle maximum depth <b>OR</b> load; see the same information than described in <b>Hazardous result</b> , <b>p. 83</b> of <b>Advanced</b> measurement type.
<u>P</u> ause	To set a pause at each cycle maximum depth <b>OR</b> load before unloading.
	Set the value to 0 for "no pause".
Unloading rate    → mN/min	To set the unloading rate to reach the minimum depth <b>OR</b> load of all cycles <sup>2)</sup> after the pause (set above).

between the first and the last maximum depth/load and depending on the number of cycles, the software will compute a linear increment for each intermediate maximum depth/load

<sup>2)</sup> exception: the last cycle will be fully unloaded

## **Quadratic loading**



between the first and the last maximum depth/load and depending on the number of cycles, the software will compute a linear increment for each intermediate maximum depth/load

<sup>2)</sup> exception: the last cycle will be fully unloaded

## 4.6.4.6 Continuous multicycle (CMC)

The *CMC* measurement type is a single indentation measurement, performed in **several** cycles of loading/unloading defined by the user. The differences with the *Progressive multicycle* measurement type are:

- There is only a loading profile: constant time loading/unloading.
- It is only load controlled (no depth control).
- The minimum load increases for each measurement cycle (as well as the maximum load).

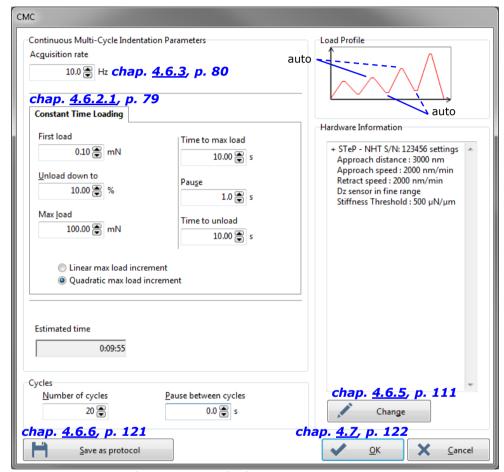


Fig.41 CMC window parameters

The default **Constant time loading** set of parameters is described in the following section, **p. 94**.

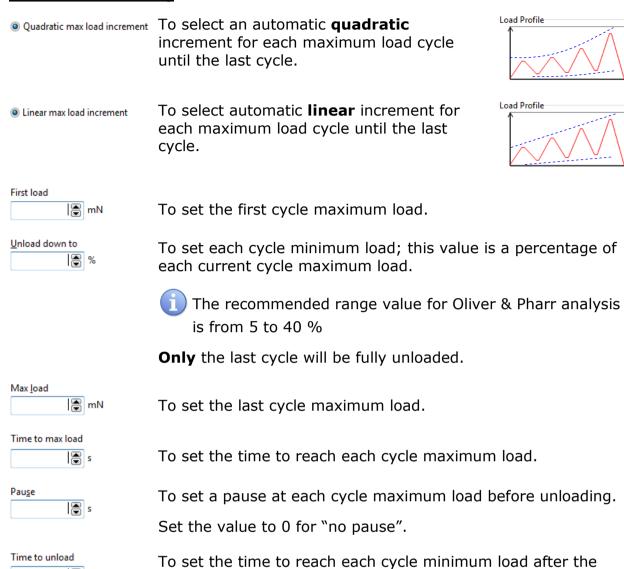
## **Cycles**



Same parameters than described in <u>Cycles</u>, p. 87 of the Constant multicycle measurement type.

## **Constant time loading**

S



pause (set above).

## 4.6.4.7 User defined profile

The *User defined profile* measurement type is a **single** indentation measurement, performed in **several segments** fully defined by the user. Each segment can be different from one to another.

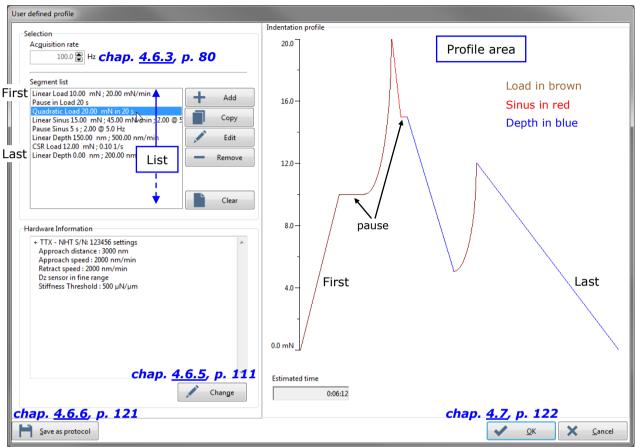
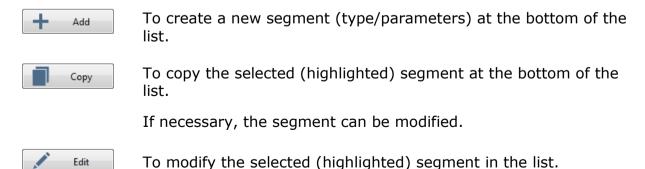
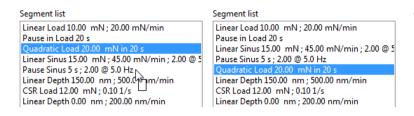


Fig.42 User defined profile window parameters

There are load profile or pause segment types. Each segment in the list from top to bottom is respectively displayed from the left to the right in the profile area and corresponds to the order/shape of the measurement.

The 3 following buttons open the Edit segment window Fig. 43, p. 96.





To change the order of any segment, select it (highlighted), drag it and drop it elsewhere in the list; the shape changes accordingly in the profile area.

\* To remove the selected (highlighted) segment from the list.

\* To clear all segments from the list.

\* click Yes to confirm the removal/clearing **or** click No to cancel

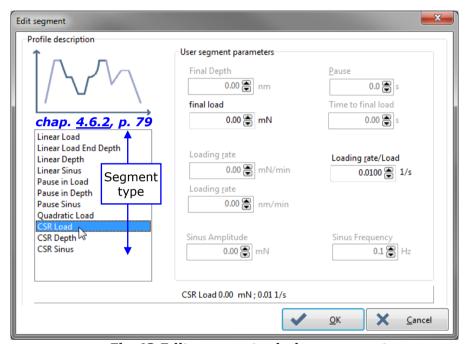


Fig.43 Edit segment window parameters

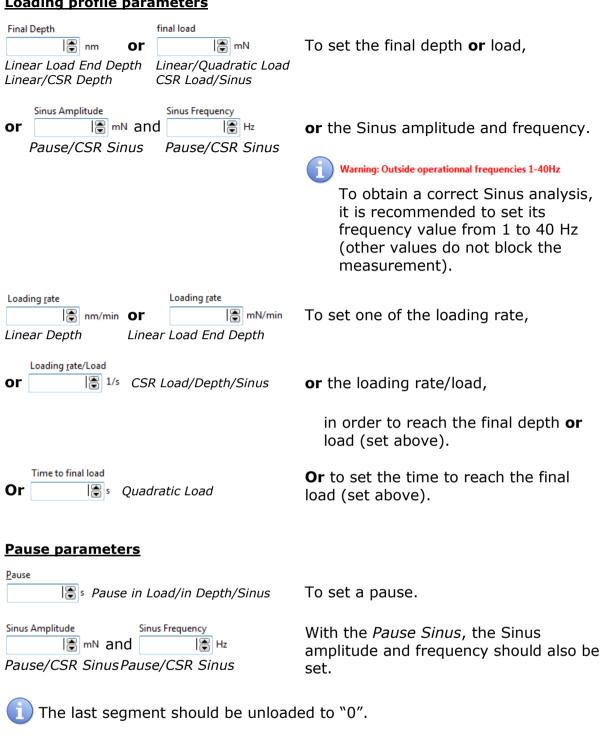
Select a segment type: a loading profile **or** a pause (becomes highlighted).

The corresponding parameter(s) are active and should be set; remaining parameters are inactive (grayed out).

The corresponding set of parameters is described in the following sections:

<u>Loading profile parameters</u> p. 97
<u>Pause parameters</u> p. 97

## Loading profile parameters



to valid the current segment settings in the list *Fig.42*, *p.* 95. Click

An Information window message appears in case of a parameter in not properly set.

## 4.6.4.8 Simple matrix

The *Simple matrix* measurement type performs a **matrix** of **identical** indentation measurements with a (same) **defined** X & Y spacing.

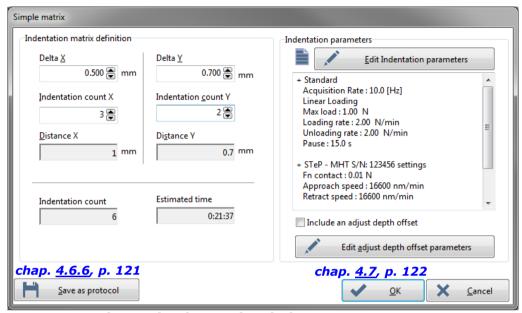
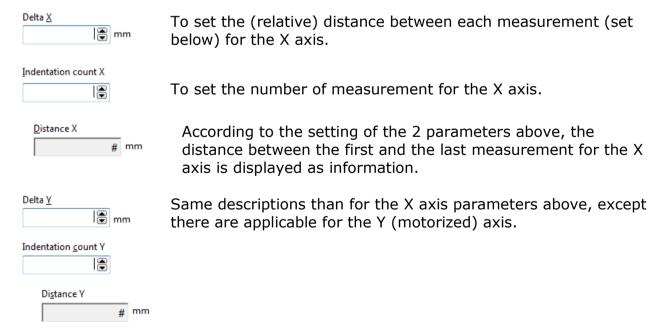


Fig.44 Simple matrix window parameters

For certain instrument, if the Y table is manual the corresponding fields are inactive (grayed out).

The matrix of measurements will start from the current position of the tables.

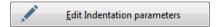
#### **Matrix parameters**



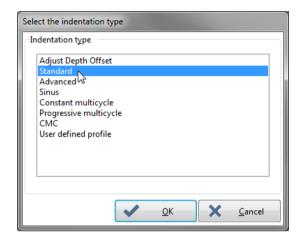


The total number of the measurements which will be performed is displayed as information.



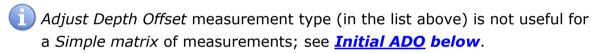


To select one of the following measurement types, the same one will be used to perform the **whole** matrix of measurements.



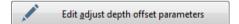
Select (double click) the desired measurement type. The corresponding parameters should be set as same as described in the following *chapters*:

Standard	4.6.4.1, p. 81
Advanced	4.6.4.2, p. 82
Sinus	4.6.4.3, p. 85
Constant multicycle	4.6.4.4, p. 87
Progressive multicycle	4.6.4.5, p. 90
CMC	4.6.4.6, p. 93
User define profile	4.6.4.7, p. 95



#### **Initial ADO**

Include an adjust depth offset



If necessary, check this box to **include** an (automatic) ADO before performing the matrix of measurements.

To set the parameters for the **included** ADO (if box is checked above); the same ADO window parameters as described for the corresponding head below should be set:

UNHT ADO <u>Fig.29</u>, p. 57 UNHT Bio ADO <u>Fig.30</u>, p. 57 NHT ADO <u>Fig.32</u>, p. 66 MHT ADO <u>Fig.33</u>, p. 70

The ADO procedure will automatically begin once the matrix of measurements will be started ( v v v s clicked in *Fig.44*, *p.* 98).

#### 4.6.4.9 Advanced matrix

The *Advanced matrix* measurement type performs a list of indentation/ADO measurements. For each measurement definition which composes the matrix, a **different** position can be defined and a **different** measurement type (with its specific parameters) can be selected.

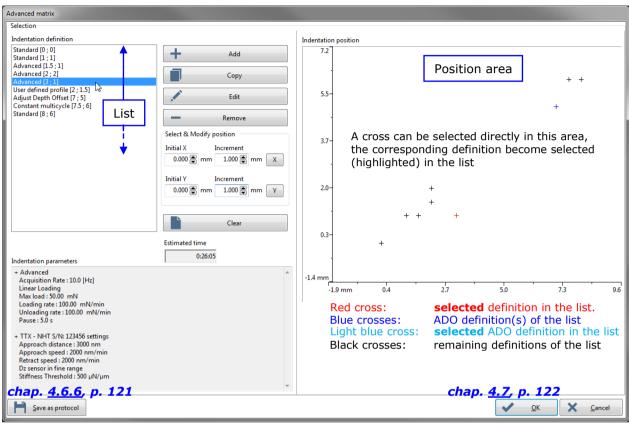
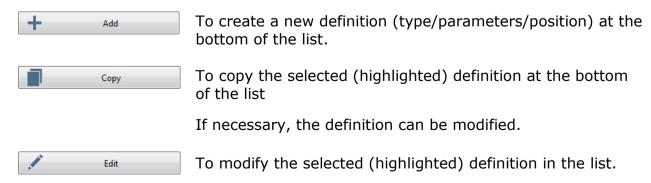


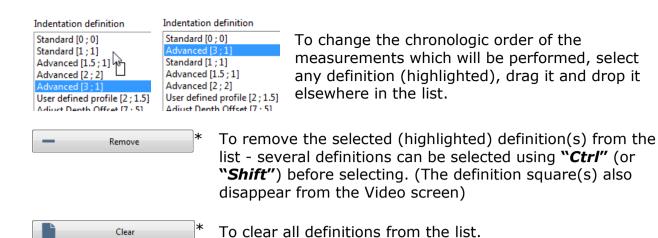
Fig.45 Advanced matrix window parameters

The corresponding position of each measurement definition from the list is represented with a cross in the position area. Each measurement definition will be performed in the chronological order of the list (from top to bottom).

## Parameters to create and modify each new selected measurement definition

The 3 following buttons open the *Indentation position* window *Fig.46*, p. 101.





\* click Yes to confirm the removal/clearing **or** click No to cancel

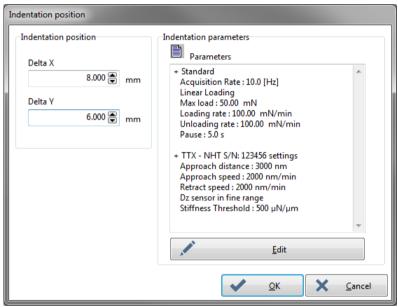
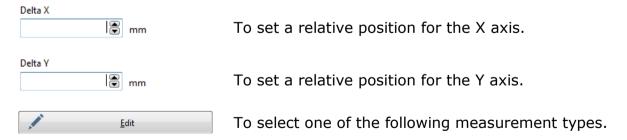
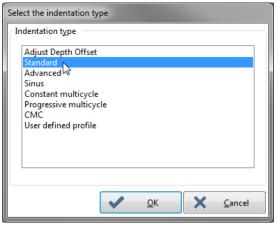


Fig.46 Indentation position window parameters

For each current (selected) definition:





(ADO or indentation measurement)

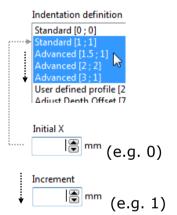
Select (double click) the desired measurement type. The corresponding parameters should be set as same as described in the following *chapters*:

Standard	<u>4.6.4.1</u> , p. 81
Advanced	<u>4.6.4.2</u> , p. 82
Sinus	4.6.4.3, p. 85
Constant multicycle	<u>4.6.4.4</u> , p. 87
Progressive multicycle	<u>4.6.4.5</u> , p. 90
CMC	<u>4.6.4.6</u> , p. 93
User define profile	<u>4.6.4.7</u> , p. 95

Adjust Depth Offset, see chap. 4.6.4.11 Multi-ADO for advanced & visual advanced matrix, p. 110

## To modify positions for several measurement definitions (regular positions)

The following features can be useful to define a regular grid for the matrix; the initial position and a same distance increment can be applied for the X and/or Y axis of the selected definitions in the list (to modify their current positions).



Press "*Ctrl*" (or "*Shift*") and select several definitions in the list (highlighted).

#### For the X axis

To Set the initial (absolute) position for the first selected definition (highest position selected in the list)

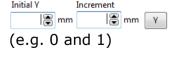
To set the increment (relative) distance between each remaining selected definition (from top to bottom in the list).

To modify the current positions according to the selection and 2 settings described above.



## For the Y axis

Same as described above, **exception:** it is applicable for the Y axis. The selection in the list should be done again.



Indentation definition

Standard [0; 0]

Standard [0; 0]

Advanced [1; 1]

Advanced [2; 2]

Advanced [3; 3]

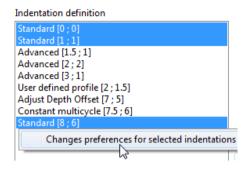
User defined profile [2]

Adjust Depth Offset [3]

E.g. the X and Y positions of the selected definitions have been modified.

## To set same preferences for several measurement definitions

To set in one time the same preference parameters for several measurement definitions, see the descriptions below.

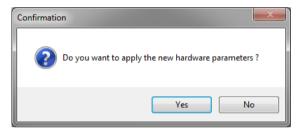


Press "*Ctrl*" or "*Shift*" and select several measurement definitions in the list (highlighted).

Then right click in the definition list area and click on *Changes preferences for selected indentations* context menu.

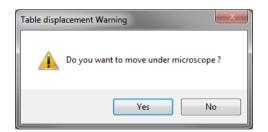
The same *Preferences* window parameters as described for the corresponding head below can be modified for all selected definitions in the list:

```
UNHT chap. 4.6.5.1, p. 112
UNHT Bio chap. 4.6.5.2, p. 114
NHT chap. 4.6.5.5, p. 116
MHT chap. 4.6.5.6, p. 117
```



#### 4.6.4.10 Visual advanced matrix

This measurement is only possible with an **optional** motorized Y table and a video system mounted on the instrument.



Click Yes to open the following Visual advanced matrix and Video windows.

The *Visual advanced matrix* measurement type performs a list of indentation/ADO measurements. For each measurement definition which composes the matrix, a **different** position can be visually<sup>1)</sup> **or** manually<sup>2)</sup> defined and a **different** measurement type (with its specific parameters) can be selected.

<sup>1)</sup> With following *Video* window

<sup>&</sup>lt;sup>2)</sup> With following *Visual advanced matrix* window

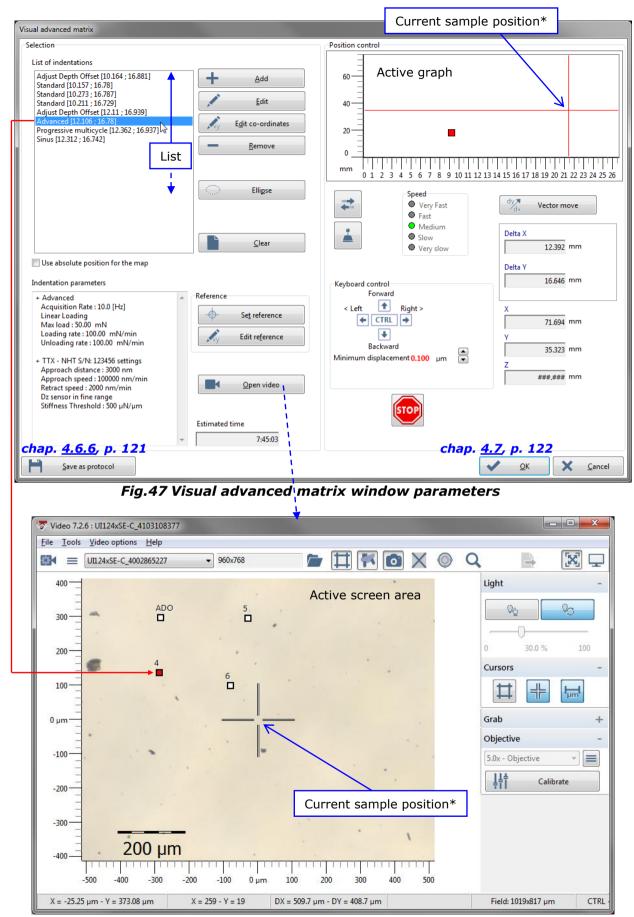


Fig.48 Video window with defined indentations

#### Parameters to create new measurement definitions

To create a new measurement definition at the bottom of the list, **click**:

- A position directly on the Video screen.

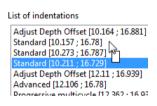
#### OR

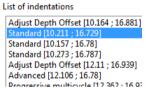


The new measurement definition type (and parameters) are the same as the last selected (highlighted) definition in the list (a copy but with a different position); see how to modify the <u>Measurement type/parameters</u> below and the <u>Position</u>, p. 106.

On the *Video* screen, each defined position is shown with a small square and a "number" or "ADO". Each "number" corresponds to the order of the indentation definitions in the list (from top to bottom) excluding the ADO. When the small square is in red color, it means it is the position of the current selected (highlighted) definition in the list.

#### Parameters for each selected measurement definition



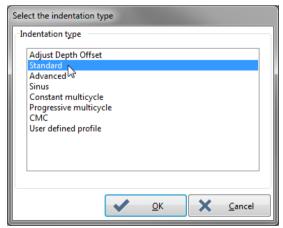


To change the chronologic order of the measurements which will be performed, select any definition (highlighted), drag it and drop it elsewhere in the list.

## **Measurement type/parameters**



To modify the measurement type of the selected (highlighted) definition in the list:



(ADO or indentation measurement)

Select (double click) the desired measurement type. The corresponding parameters should be set as same as described in the following *chapters*:

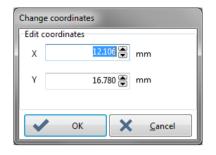
Standard	4.6.4.1, p. 81
Advanced	4.6.4.2, p. 82
Sinus	4.6.4.3, p. 85
Constant multicycle	4.6.4.4, p. 87
Progressive multicycle	4.6.4.5, p. 90
CMC	4.6.4.6, p. 93
User define profile	4.6.4.7, p. 95

Adjust Depth Offset, see chap. 4.6.4.11 Multi-ADO for advanced & visual advanced matrix, p. 110

#### **Position**



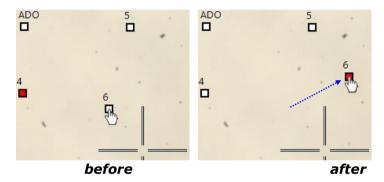
To <u>manually</u> change the position of the selected (highlighted) measurement definition in the list:



Set other coordinate value(s) than the current position.

**OR** 

To <u>visually</u> change a measurement definition position from the *Video* screen, click in the middle of the desired square (the arrow mouse cursor **should** become a hand shape cursor, otherwise a new definition is created), then drag and drop the current red square on the *Video* screen.



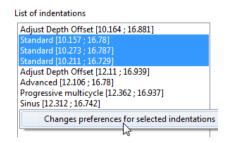


To remove the selected (highlighted) measurement definition(s) from the list - several definitions can be selected using "*Ctrl*" or "*Shift*" before selecting. (The definition square(s) also disappear from the *Video* screen)



To clear all measurement definitions from the list (and from the *Video* screen).

## To set same preferences for several measurement definitions



Same as described in <u>To set same preferences for</u> <u>several measurement definitions</u>, for the *Advanced Matrix* in **p. 103**.

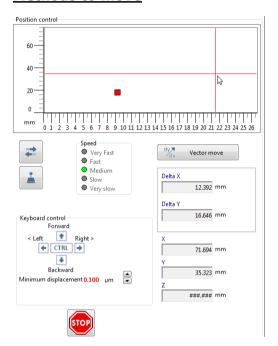
## Absolute reference position for protocol

✓ Use absolute position for the map

Check this box to always keep the same matrix reference if this *Visual advanced matrix* measurement type is saved as a protocol. Each time the saved protocol will be selected, the sample will be positioned at the same reference; see <u>To</u> <u>change the reference position</u>, p. 107.

Otherwise (uncheck) the sample will be positioned at the current position.

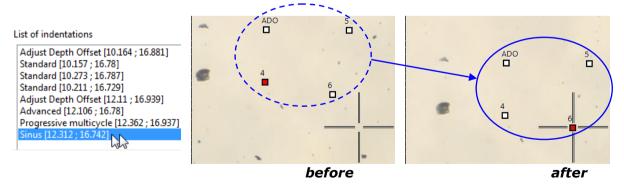
#### **Methods to move**



To move the sample/motorized table(s) use the *Position control* area; for descriptions, refer to the *Common Scratch & Indentation software manual - chap. Managing the instrument - Control of the sample position*, exception: some functions are not applicable here.

OR

To move the sample/motorized tables to an existing measurement definition position, on the *Video* screen center (crosshair), double click on the desired definition in the list.

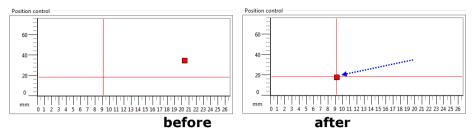


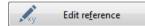
#### To change the reference position

To align the measurement definitions of the matrix with a specific reference position on the sample, see the following descriptions.

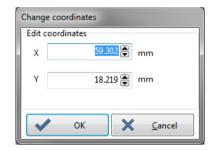


To set a new reference at the current sample/motorized tables position.





To manually set a new reference ■ to another position:

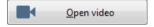


Set different coordinate field value(s) than the current position.

The current position value(s) can slightly differ from the one(s) shows on the *Position control* active graph due to the indentermicroscope distance calibration correction.

When a new reference is set (above), the X-Y deltas of the definitions in the list are not changed, only the origin of map is changed. This allows translation of all measurement definitions by selecting new reference/coordinates (origin).

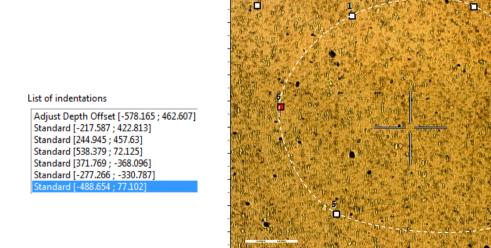
## To reopen the Video



To reopen the *Video* window if it has been closed.

#### **Ellipse**

To fit an ellipse shape for the matrix, first **minimum** 6 indentations should be defined (round shape) in the list (which will be automatically erased later) - ADO(s) are not included.



To define the following ellipse parameters.

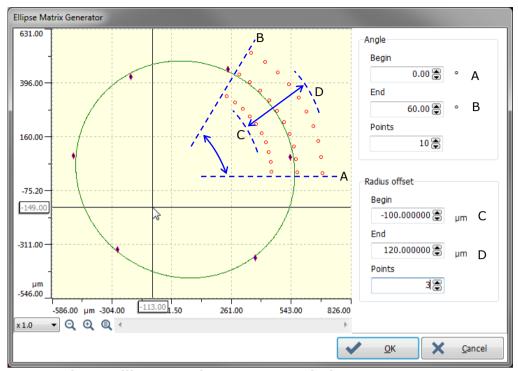
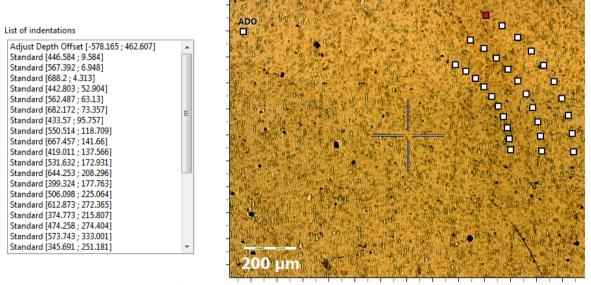


Fig.49 Ellipse Matrix Generator window parameters

Set the field parameters in order the software computes new indentation definitions in the list for the elliptic fit matrix.

E.g. 10 **Points** (Angle) in 3 rows (Radius offset **Points**) = 30 definitions.

When is clicked in <u>Fig.49</u> above, the new indentation definitions replace the previous ones in list. (ADO definition(s) is kept in the list)



New computed indentation definitions (e.g. 30) for the elliptic matrix

#### 4.6.4.11 Multi-ADO for advanced & visual advanced matrix



Reminder of the ADO purpose: read the beginning of the *chap 4.4*, *p. 56*.

One or several automatic ADO(s) can be performed at the beginning and/or between the indentation measurements for the *Advanced matrix* chap. 4.6.4.9, p. 100 or *Visual advanced matrix* chap. 4.6.4.10, p. 103.

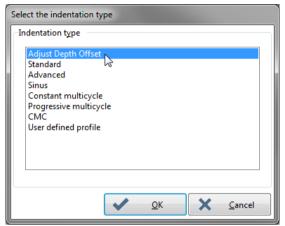


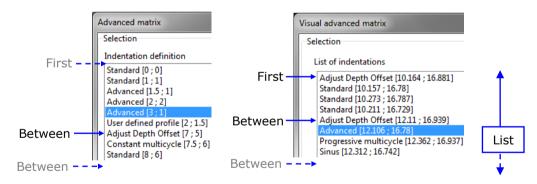
Fig.50 ADO selection

Select (double click) the *Adjust Depth Offset* (ADO) measurement type.

The same ADO window parameters as described for the corresponding head below should be set:

```
UNHT ADO <u>Fig.29</u>, p. 57
UNHT Bio ADO <u>Fig.30</u>, p. 57
NHT ADO <u>Fig.32</u>, p. 66
MHT ADO <u>Fig.33</u>, p. 70
```

- An ADO can be included at the **first** position in the definition list, before starting the indentation measurements of the matrix.
- One or several ADO(s) can be included **between** the indentation measurements of the matrix.



Before starting the matrix of measurements, if there is **no** at least an ADO in the first position of the list, a *Confirmation* window asks to continue:

Click  $\[ \]$  if a successful  $(\[ \]$  ADO has previously been performed and is suitable (location) for the whole matrix area. Otherwise click  $\[ \]$  and add an ADO definition in the first position of the list, and elsewhere in the list where it is necessary.

## 4.6.5 MEASUREMENT PREFERENCE PARAMETERS (HEAD)

Located at the right (or left\*) bottom of the following measurement type window:

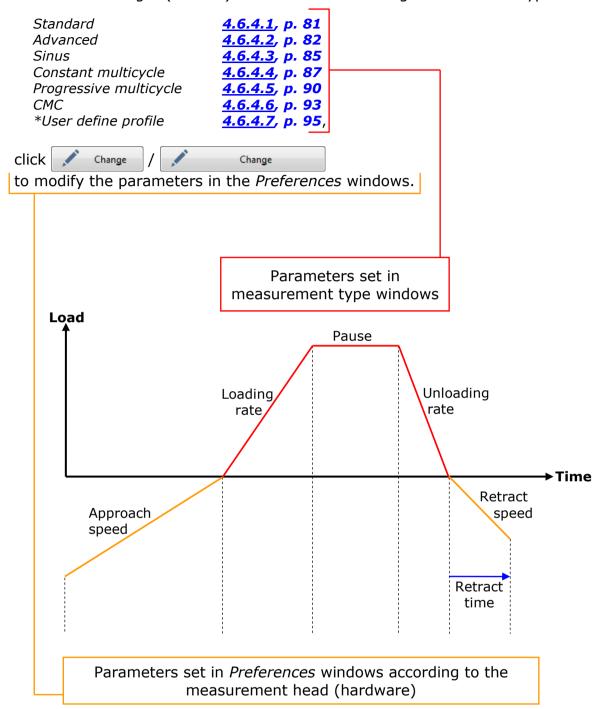


Fig.51 measurement schematic

See the following sections which described the *Preferences* window parameters dedicated for each type of measurement head (UNHT, UNHT Bio, NHT and MHT) and the common parameters for all types of measurement head (approach distance, contact stiffness threshold and adhesion).

## 4.6.5.1 UNHT Preferences tab parameters

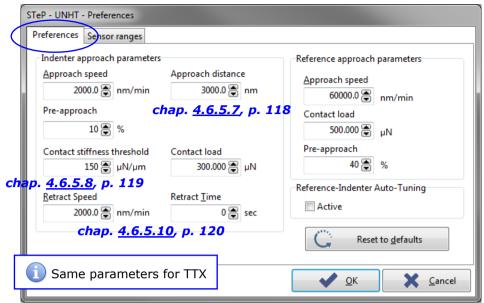
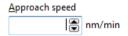


Fig.52 E.g. STeP - UNHT Preferences tab with default parameters

Set all parameters for the indenter & reference approach, contact detection and then for the retraction.

#### **Indenter**



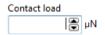
The **Approach speed** determines the velocity at which the indenter approaches the sample surface.

See the typical values in the table below:

1000 1- 2000 (	For max. measurement load	Material
1000 to 2000 nm/min	20 to 1000 μN	fused silica (hard)

#### Tendency:

For maximum loads higher than 1000  $\mu$ N, set a value higher than 2000 nm/min, especially for soft materials.



The indenter **Contact load** is used to detect the sample surface during the approach. The value depends on the desired max load value for the measurement. The following table gives indications on how to realize measurement in the best conditions:

Recommended value	For max measurement load
= <10 µN	= <100 µN

#### Tendency:

The lower the measurement max load value is, the lower the value should be set, in this case reduce the approach speed (set above) to keep enough acquisition data points in the curve.

For max measurement load higher than 100  $\mu$ N, the value can be increased (10 % of max measurement load with max. 50  $\mu$ N); see also *chap.* 4.6.5.9, *p.* 120.

# Reference Approach speed



The <u>Approach speed</u> determines the velocity at which the reference approaches the sample surface; advised (standard) value **60000 nm/min** 

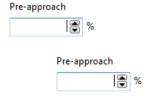


The reference **Contact load** is used to detect the sample surface; UNHT uses a patented active surface referencing system whose principle consists basically of a reference contacting the surface.

See the typical values in the table below:

Max. value	Material	
<b>500</b> or <b>1000</b> μN	for most material	

#### **Indenter & reference**



The indenter and reference **Pre-approach** fields are used to ensure that the indenter position is higher than the reference position before approaching the indenter and reference with the approach speed (set above). Standard values **10** % for the indenter and **40** % for the reference are most of the time suitable. However those values may be different depending on UNHT measurement configuration (type of mounted indenter/reference, mounting and geometry of the sample...).

#### **OR**

Reference-Indenter Auto-Tuning

Active

Check the reference-indenter auto-tuning box to perform an auto adjustment of the pre-approaches between the Pre-approach

reference and the indenter: the indenter



Pre-approach reference 40 🕏

40 🕏 % become inactive (grayed out).

According to the last successful ADO, the software will compute and optimize the best indenter and reference preapproaches.



100 % is the lower/rest position.

## 4.6.5.2 UNHT Bio Preferences tab parameters

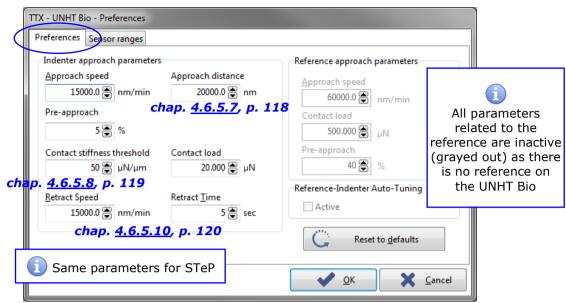
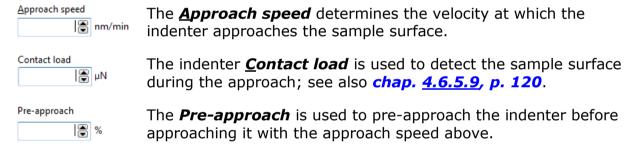


Fig.53 E.g. TTX-UNHT Bio Preferences tab with generic suitable parameters

Set all parameters for the indenter approach, contact detection and then for the retraction.



For the detailed explanations of those parameters, refer to the **Anton Paar Bioindenter UNHT3 Bio User Manual - chap. Indentation measurement process - Performing an indentation measurement - Define indentation measurement parameters**.

## 4.6.5.3 UNHT Sensor ranges tab parameters

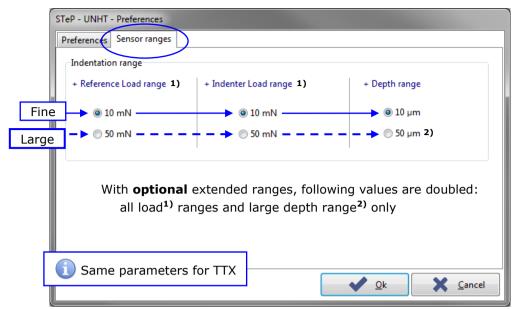


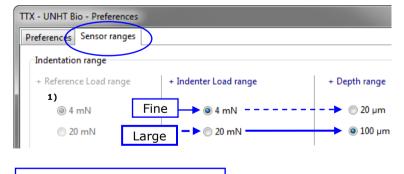
Fig.54 E.g. STeP - UNHT Sensor ranges tab with fine ranges

Select suitable **Reference Load range**, **Indenter Load range** and **Depth range** according to the used sample/application; it is recommended to use fine ranges (e.g. above), to obtain the optimal quality of the signals.

Use the large ranges only if the fine ranges are not sufficient to perform the measurement, or in case of saturation.

This tab is also available in the Calibration window chap. 3.3.1, p. 29.

## 4.6.5.4 UNHT Bio Sensor ranges tab parameters



Similar than UNHT chap. 4.6.5.3 above, exception:

- No reference.
- Range values are different.
- It is recommended to use the large **depth** range.

Same parameters for STeP

# 4.6.5.5 NHT Preferences tab parameters

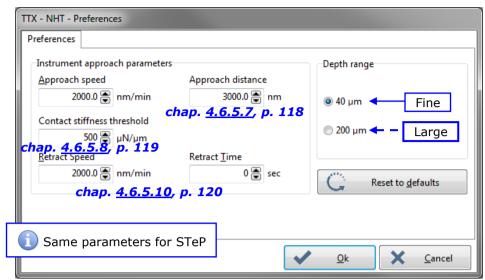
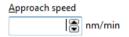


Fig.55 E.g. TTX-NHT Preferences tab with default parameters and fine depth range

Set all parameters for the indenter approach, contact detection and then for the retraction.



The **Approach speed** determines the velocity at which the indenter approaches the surface.

See typical values in the table below:

Hard material		Soft material
< 1 mN	> 1 mN	Soft Illaterial
<b>1000</b> to <b>1500</b> nm/min	2000 nm/min	4000 nm/min

Depth range

40 μm200 μm

Select a suitable **Depth range** according to the used sample/application; it is recommended to use the fine range (e.g. above) but if the depth saturates during the following measurement, then select the large depth range.

## 4.6.5.6 MHT Preference tab parameters

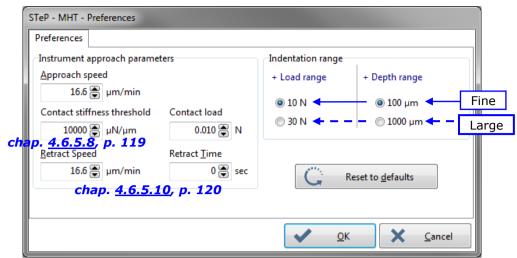


Fig.56 E.g. STeP - MHT Preferences tab with standard parameters and fine ranges

Set all parameters for the indenter approach, contact detection and then for the retraction.



The **Approach speed** determines the velocity at which the indenter approaches the sample surface.

See the typical values in the table below:

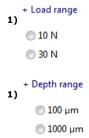
Hard material		
100 μm	- Depth range -	1000 μm
<b>8</b> or <b>16.6 μm/min</b>		16.6 µm/min



The **Contact load** is used to detect the sample surface during the approach; see also **chap.** <u>4.6.5.9</u>, **p. 120**.

See the typical values in the table below:

Hard material		
100 µm	- Depth range -	1000 µm
0.01 N		0.03 N



Select suitable **Load range** and **Depth range** according to the used sample/application; it is recommended to use fine ranges (e.g. above) to obtain the optimal quality of the signals. Use the large ranges only if the fine ranges are not sufficient to perform the measurement, or in case of saturation.

 $oldsymbol{\Lambda}$  The Depth range **should** be the same as the ADO  $oldsymbol{ extstyle Dz\ range}$ ,

p. 70 which has been selected for the current successful ADO. Otherwise an error message will appear; see chap. 6.2,p. 173.

<sup>1)</sup> range values can be different with old MCT version

## 4.6.5.7 Approach distance parameter (indenter)



This parameter is **only** available for the measurement heads equipped with an electronic bridge, such as the UNHT, UNHT Bio and the NHT, and can be changed **for each new measurement** in *Preferences* window/tab; see from *chap.* 4.6.5, *p.* 111.

This parameter allows the increasing or decreasing of the indenter approach distance before starting to record the measurement.



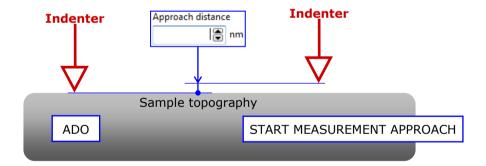
The default value is suitable for most sample materials. However the value may have to be adapted (e.g. for very soft materials).

#### MHT approach distance

Otherwise for MHT <u>without</u> electronic bridge, this parameter can be **only** changed in the current ADO parameters window; see **chap.** <u>4.4.3</u>, **p. 70**. Therefore the approach distance will remain **the same for each following measurement**.

The parameter is applied according to the last successful ADO. When the approach distance value is reached by the indenter, the software starts recording of the measurement acquisition data (points).

The sketch below supposes that the sample surface topography is totally flat (no holes, no bumps, and no roughness) and the sample is not too soft:



The approach distance value which is set can be approximately seen on the following *Set the contact point* window *Fig.57*, *p. 119*, after the measurement has been performed.

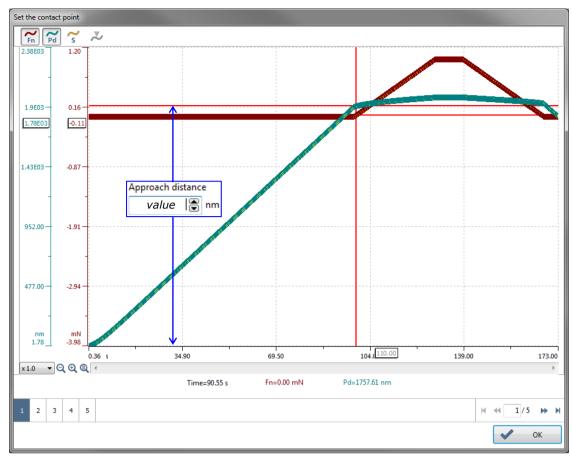


Fig.57 Approach distance on Set the contact point window

The approach distance cannot be exactly as same as the set value because it depends on the sample topography (tilt+ roughness) and the sample material (soft or hard); see *chap. 5.1.1* <u>Setting the contact point</u>, *p. 132*.

## 4.6.5.8 Contact stiffness threshold parameter



The value can be changed for each new measurement in *Preferences* window/ tab; see from *chap.* <u>4.6.5</u>, *p.* 111, but it is recommended to set the default value for each instrument.

All the indentation measurement heads use this parameter for contact detection of the sample surface.

If necessary (for more accuracy or for very soft sample materials),

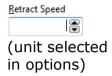
it is possible to slightly decrease the default value; ! however by **decreasing** too much the value, the contact can be detected in the air.

## 4.6.5.9 Contact load detection with UNHT/Bio & MHT



Additionally to the standard contact stiffness method chap. 4.6.5.8, p. 119, the UNHT, UNHT Bio and MHT instruments also can use the contact load parameter (unit selected in options). The sample surface is detected as soon as one of these methods finds it (most of the time the stiffness is detected sooner than the force).

## 4.6.5.10 Retract speed & time parameters for Adhesion

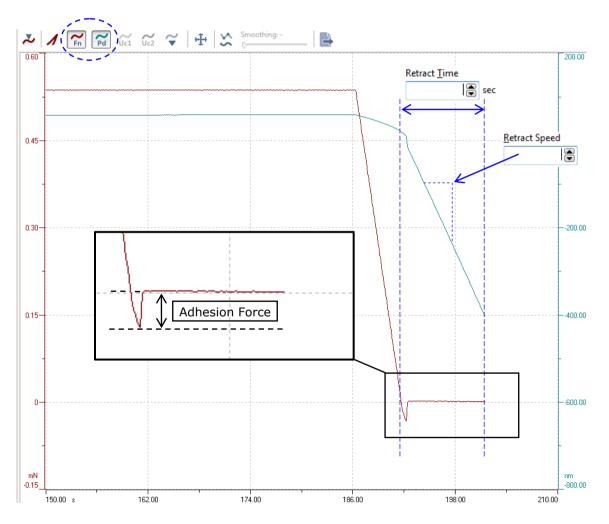


<u>Retract speed</u> and <u>Retract <u>Time</u> values can be changed for each new measurement in <u>Preferences</u> window and tab; see from <u>chap. 4.6.5</u>, <u>p. 111</u>.</u>



The value of these parameters allows studying the adhesion between the indenter and the sample.

Both parameters manage the unloading during the adhesion phenomenon.



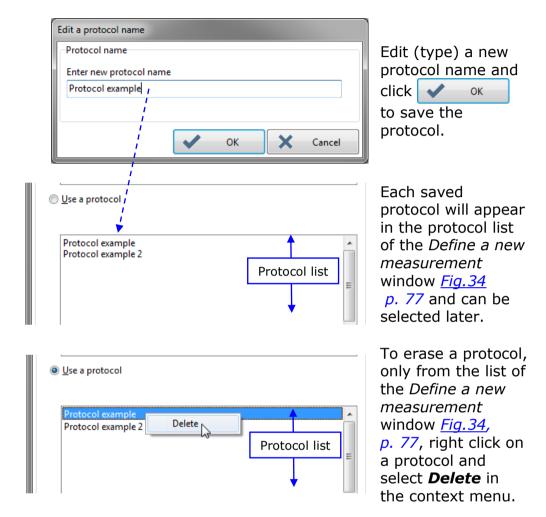
See *chap. 5.5, p. 149* to obtain the Adhesion Force.

# 4.6.6 SAVE AS PROTOCOL (CURRENT MEASUREMENT TYPE SETTING)



Located at the left bottom of each measurement type window (see from *chap.* 4.6.4, p. 81)

To save the current setting of all parameters as a measurement protocol (including preference parameters *chap.* 4.6.5, *p.* 111).



#### 4.7 MEASUREMENT PROCESS (INDENTATION RUNNING)

Once the measurement starts, the window below displays the real-time status, indenter progression bars and the acquisition curves.

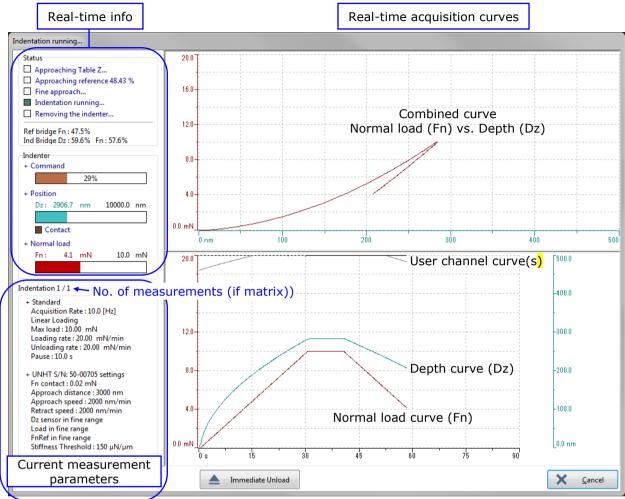


Fig.58 Indentation running... window

igwedge It is recommended not to use any other software whilst acquisition is in progress, as this may interrupt the scheduler and cause discontinuities in the acquisition process. Ensure that no screen saver or other time consuming applications could start during the acquisition process.

There are 5 real-time action blinking squares in the *Status* area:

■ Approaching Table Z... / □ Approaching Table Z... (inactive with TTX-NHT) ■ Approaching reference 48.43 % /□ Approaching reference (active with UNHT) ■ Approaching with the indenter... / ■ Approaching Indenter / ■ Stabilization 30 [s] Indentation running... Removing the indenter...

#### **Stabilization**

Before the indentation starts (■ Indentation running...), ■ Stabilization 30 [s] is blinking and displays a decreasing time info (e.g. starts from 30 [s]); the stabilization time is set in *chap. 3.1.4.2 Stabilization (cancel drift)*, *p. 26*.

As soon as the stabilization time is elapsed 0 [s], the indentation measurement starts.



To unload the current measurement applied force and start the next measurement (if matrix).



To stop all measurement(s) (if matrix), the indenter/motorized Z table are immediately retracted.

i With UNHT and UNHT Bio, the extra *UNHT Approach Monitor* window <u>Fig.28</u>, p. 52 can be displayed or deactivated with the UNHT.INI file; see **chap.** 3.5, p. 51.

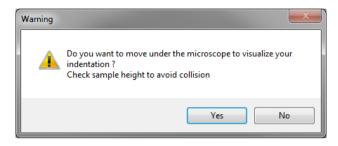
## 4.8 Analyzing/visualizing indentation(s)



This chapter is applicable if the instrument is equipped with the video microscope and if these boxes are checked; refers to the *Common Scratch & Indentation software manual - chap. Managing the instrument - Hardware configuration - My configuration tab*.

Optical Analysis after measurement

With **UNHT Bio**, check this box if an optical analysis is needed (spherical indenter does not let mark on very soft material).



At the end of the indentation measurement process: when the *Indentation running...* window <u>Fig. 58</u>, p. 122 closes, this *Warning* window appears.

Click Yes to open,

the *Video* window <u>Fig. 60</u>, p. 126 and Analyze indentation window <u>Fig. 59</u> below, in order to (if necessary):

- Refine the indenter-microscope distance calibration.
- Analyze/visualize the indentation measurement(s) and capture image(s).

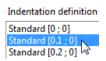
Or click and go to *chap. <u>5</u>, p. 130*. chap. 4.8.2, p. 127 Analyze indentation Indentation position 😭 😭 Curve toolbar 25.00 0.5 0.0 Graph -0.5 0.2 0.2 Indentation definition Standard [0 : 0] **Image** Standard [0.2;0] Indentation list gallery Very Fast Fast Medium Video image Slow 1F03 (1.0 ▼ 🔾 🝳 🔍 🤻 Indentation parameters Keyboard control + Standard
Acquisition Rate: 10.0 [Hz]
Linear Loading
Max load: 20.00 mN
Loading rate: 40.00 mN/min
Unloading rate: 40.00 mN/min
Pause: 5.0 s 1 Parameters of the selected AFM image ← CTRL → indentation in the list 洪 1 Minimum displacement 0.250 μm chap. 4.8.1, p. 125 chap. 4.8.3, p. 129 ✓ Finish analysis

Fig.59 Analyze indentation window



To reopen the *Video* widow.

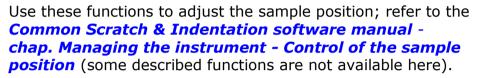
#### Selecting an indentation



E.g. if a matrix of measurements has been performed, select an indentation in the list to automatically move to the corresponding

#### Methods to move for adjustment







The displacement area is limited in the zone of the selected indentation in the list.

#### Original indentation position



To move back to the original position (of the selected indentation in the list).



#### 4.8.1 REFINING INDENTER-MICROSCOPE DISTANCE CALIBRATION

It is possible to refine the calibration of the distance between the indenter and the video microscope from this Analyze indentation window Fig. 59, p. 124, and also using the Video window Fig. 60, p. 126.

In case of a matrix, select any of the indentation in the list (chose only one); see <u>Selecting an indentation</u> above.

## **Centering**

Using the **Methods to move for adjustment above**, precisely center the chosen indentation imprint under the Video screen crosshair Fig. 60 below.



A Be sure the imprint corresponds to the selected indentation in the list.

To have the crosshair in the middle of the *Video* screen, check ☑ Crosshair in the *Video* window; see *Video* software manual for all features description.

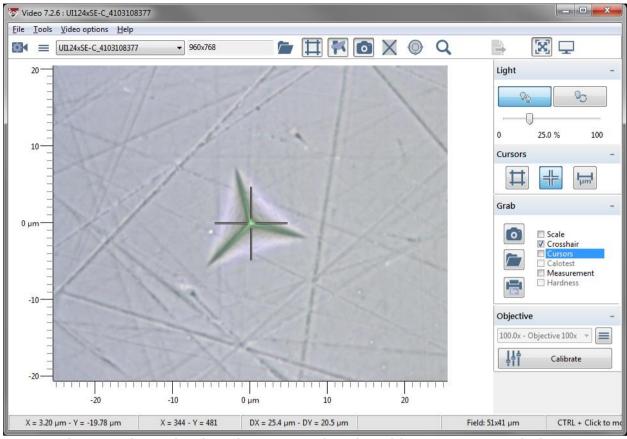


Fig.60 Indentation imprint centered under Video screen crosshair

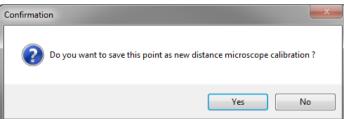


To refine the calibration of the distance between the indenter and the video microscope, only when the indentation imprint is centered *Fig.60* above.



It is recommended to click

Yes in order the software performs a backlash movement before storing the new calibration; wait.



Click Yes to save the new calibration of the X/Y distances between the indenter and microscope in the Calibration window; Fig. 15, p. 30.

Otherwise click and restart from <u>Centering</u>, p. 125 until the imprint is centered.

## 4.8.2 OPTICAL ANALYSIS (INCLUDES SETTING CONTACT POINT)

Standard images and depending on the configuration/settings, Multifocus images can be captured from the *Video* screen. Depending on the available options/settings, AFM or conscan confocal images can also be captured.

One by one, for each selected measurement in the list; see <u>Selecting an</u> <u>indentation</u>, p. 125, the features below can be used:

#### **Contact point**



display, p. 137.

#### Curve toolbar

#### **Grab image(s)**

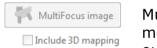
Each of the following active buttons from this *Analyze indentation window* <u>Fig. 59</u>, p. 124 allows image capture(s) from the current *Video* screen <u>Fig. 60</u>, p. 126: Edit an image window appears <u>Fig. 61</u>, p. 128.

Before capturing image(s) below, ensure the focus is adjusted (with Z motorized table, see <u>Methods to move for adjustment</u>, p. 125 with TTX-UNHT use the thumbwheels).



To capture image(s) from the live Video screen,

(standard) images.



Multifocus image only if the instrument is equipped with a motorized Z table. Otherwise the button is inactive (grayed out); see <u>MultiFocus image(s)</u>, p. 128.



To capture images from the conscan and/or AFM, if the corresponding options are available; otherwise the buttons are inactive (grayed out).

This window below allows some settings on the opened image (Title name, comments, image features...); refer to the *Common Scratch & Indentation* software manual - Manipulating document windows - Managing images gallery - Managing picture.

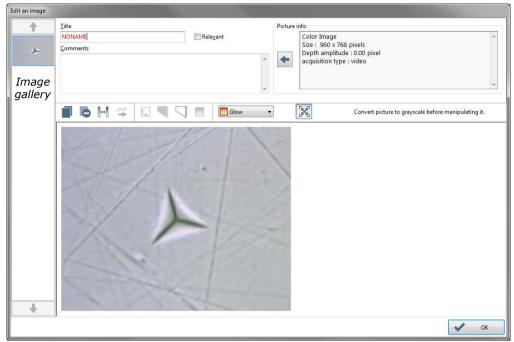
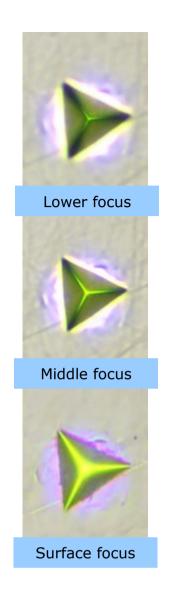


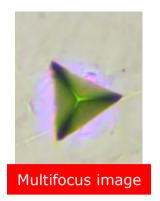
Fig.61 Edit an image

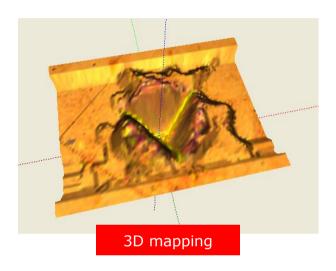
When work is clicked, each image is stored (as thumbnails) in the image gallery of <u>Fig. 61</u> above but also of the <u>Analyze indentation window Fig. 59</u>, p. 124; in each image gallery, double click on a thumbnail to (re)open the corresponding image (if there are more than 3 thumbnails, use to see the previous or following thumbnails in the gallery).

#### MultiFocus image(s)

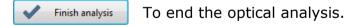
A Multifocus image is an image build with an infinite depth of field. We use the motorized Z table to capture several images of the same indentation at different focus plan (different heights), and after computing all sharpness regions the software rebuilds one single image with an infinite depth of field, called Multifocus image.

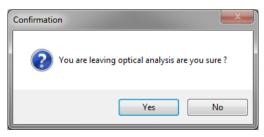






## 4.8.3 ENDING THE OPTICAL ANALYSIS





Click Yes to close the Analyze indentation window Fig. 59, p. 124, it will **not** possible to come back.

## **5 RESULT ANALYSIS**

After the measurement process of the indentation(s) is completed, the main result analysis window appears *Fig.62 below*.

Adjustments (analysis method, curve display, contact point, overlays...) and parameter changes (indenter, Poisson's ration...) can be performed from this window. After most of the parameter changes, an automatic recalculation of the analysis result is performed by the software.

By default, the curve view  $\boxed{\mathbb{Z}}$  is selected on the main toolbar.

For the main toolbar (i) and [iii]; refer to the Common Scratch & Indentation software manual - chap. Understanding documents - Document window - Customizing curve view display areas.

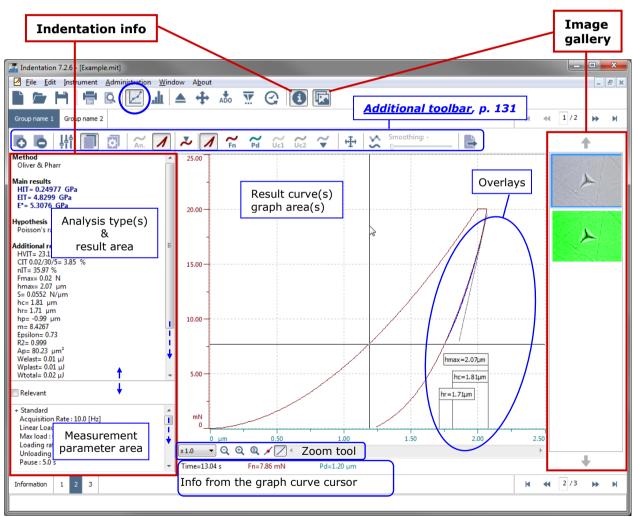
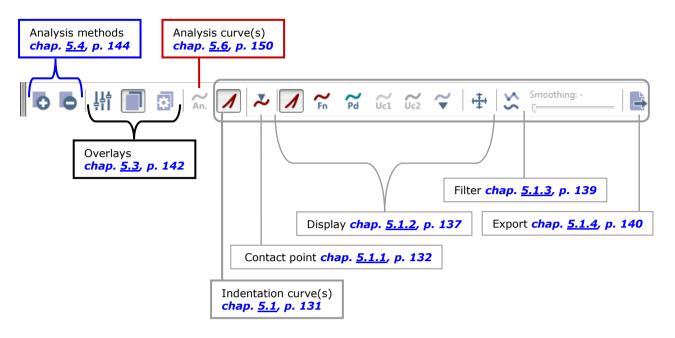


Fig.62 Result analysis window (curve view default setting)

#### **Additional toolbar**



# 5.1 INDENTATION CURVE(S)

For each active measurement



The **indentation curve** icon in the additional toolbar shows (selected):

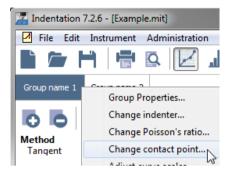
- The displayed indentation curve(s) chap. 5.1.2, p. 137
- The indentation curve features:

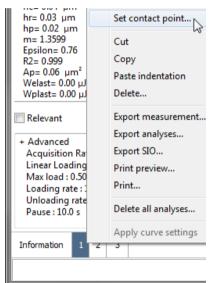
```
Contact point(s) chap. <u>5.1.1</u>, p. 132
Filter chap. <u>5.1.3</u>, p. 139
Export chap. <u>5.1.4</u>, p. 140
```

The indentation curve(s) and features can be hidden (if unselected). Only if the analysis curve(s) is active and selected  $(a_n) / (chap. 5.6, p. 150)$ .

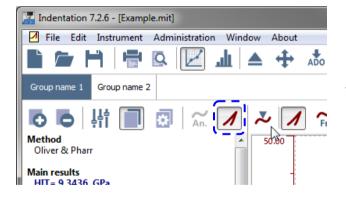
#### 5.1.1 SETTING THE CONTACT POINT

From the result analysis window <u>Fig.62</u>, p. 130, there are several ways to open the Set the contact point window <u>Fig.63</u>, p. 133, which allows to verify, refine (change) the contact point for each available measurement of each desired active group:





- For the active "group name" tab, select Edit/Group ►/Change contact point... from the menu bar.
- Right click on the desired "group name" tab and select Change contact point... from the context menu.
- For the active "measurement #"
  tab Select Edit/Indentation ▶/
  Set contact point... from the
  menu bar (only the contact point
  for this measurement will be
  opened).
- Right click any "measurement #" tab and select Set contact point... from the context menu.



 For the active "group name" tab, click 

on the additional toolbar.

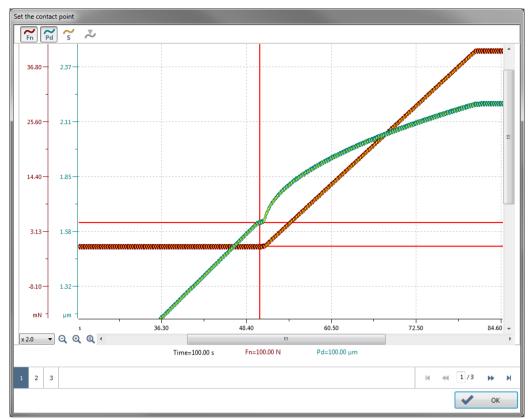


Fig.63 Set the contact point window (default settings)

## **5.1.1.1** Contact point features

#### For the active (selected) measurement



The zoom tools and cursor information are available; refer to the similar explanations as described in the Common Scratch & Indentation software manual - chapters Understanding documents - Features on graph area(s) - Zoom and - Cursor information.

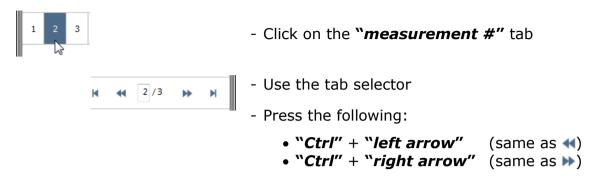
To display/hide the following indentation curve(s) in/from the graph:

- depth **h** vs. time
- and/or
  force F vs. time
  - By default they are both displayed [7], which is advised.
- To use the contact point automatically detected by the software.
  - By default after the measurement process, the contact point for each available measurement is automatically detected ( grayed out).

See chap. 5.1.1.2 Verifying/refining the contact point, p. 135.

## Selecting another measurement

To verify or refine (change) the contact point of the other available indentation measurements (if available), directly from this window:



Refer to the similar explanations described in the **Common Scratch & Indentation software manual - chap. Manipulating document windows - Using tabs - Selecting a measurement**.

## 5.1.1.2 Verifying/refining the contact point

The contact point is automatically detected ( $\nearrow$  as shown in the window below) with a **"slope" rupture** of the **h** (indentation depth Pd) and **F** (indentation force Fn) curves (depends on the material, speed, indenter).

The contact point is defined where the vertical X red graph cursor is located.

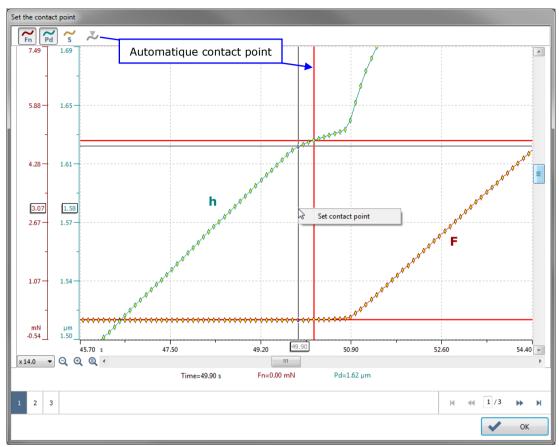
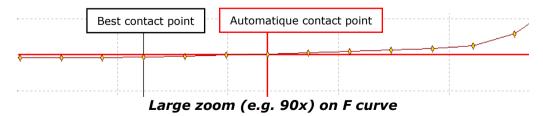


Fig. 64 Automatic contact point will be manually refined

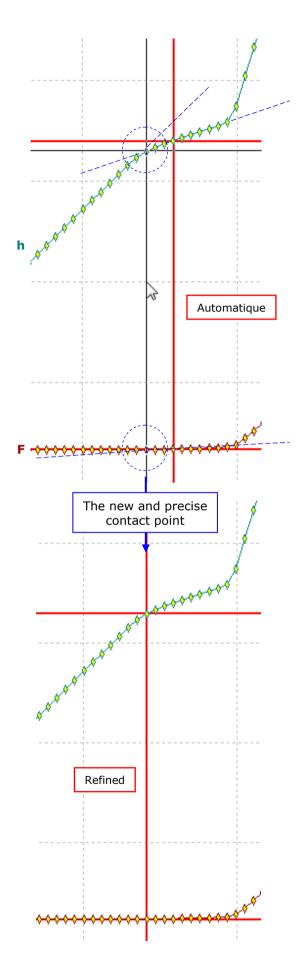
The best contact point is the first break point in the approach slope of the **h** depth curve, and is the first point with a no zero force of the **F** force curve. Therefore it is necessary to zoom to this zone; see the previous **Contact point features**, **p. 133**.

 $\bigcirc$  For high load measurement, it is difficult to see the change in the **F** curve,



therefore it is better using the **h** curve to refine the contact point.

To manually refine (change) the current contact point, see the following descriptions.



## To manually set the contact point

Place the vertical black cursor (X scale) on the graph where:

h curve starts to peakF curve starts to rise,and at this specific location:

- Double click

## OR

 Right click and select Set contact point from the context menu.

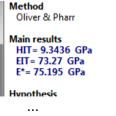
becomes (again) active in the window *Fig.64*, *p. 135*:

If necessary, click  $\stackrel{\checkmark}{\sim}$  to automatically re-define the contact point.

When the contact point changes (different location), the related *Main* and *Additional results* are recalculated in the analysis result area:



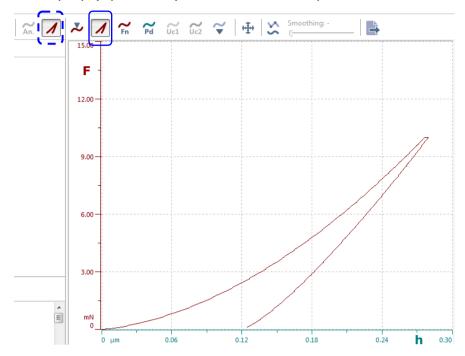
Automatic



Refined

## **5.1.2** INDENTATION CURVE DISPLAY

✓ To display (by default) the force F vs. depth h combined curve.



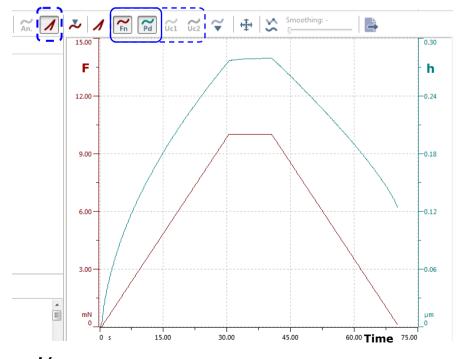
OR

To display/hide the following curve(s):

Force **F** vs. time

and/or

Depth **h** vs. time



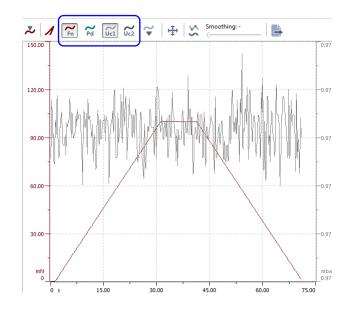
and/or ...

•••

User channel 1 and/or

User channel 2,

if activated and set prior to the measurement(s).

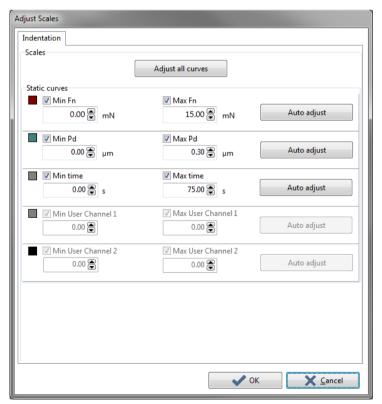


To also display/hide the previous described curves on/from the graph.



The context menu allows a quick selection (overview) of the curves.

To adjust the graph scale(s) for the displayed indentation curve(s).

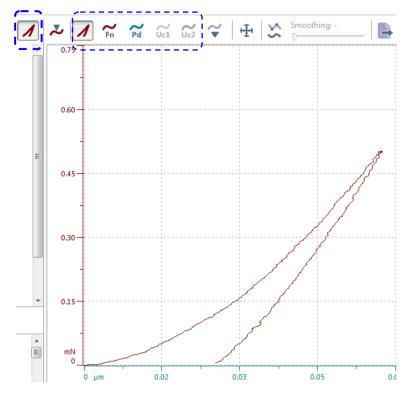


Also select **Edit/Document >/ Adjust curves scales** from the menu bar to open this window.

For the detailed description of this window, refer to the Common Scratch & Indentation software manual - chap. Manipulating document windows - Adapting curve ranges.

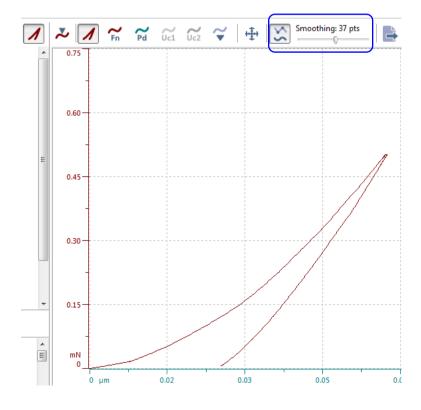
## **5.1.3** FILTERING THE INDENTATION CURVE(S)

This feature is applicable with any indentation displayed curve(s)  $\sqrt{\phantom{a}}$  or  $\sqrt{\phantom{a}}$  or  $\sqrt{\phantom{a}}$  or  $\sqrt{\phantom{a}}$  on the graph; see *chap.* <u>5.1.2</u>, *p.* 137. \*if activated



E.g. there is noise on the displayed combined curve.

To activate the *Smoothing* (filter) slider on the additional toolbar.



With the mouse cursor,



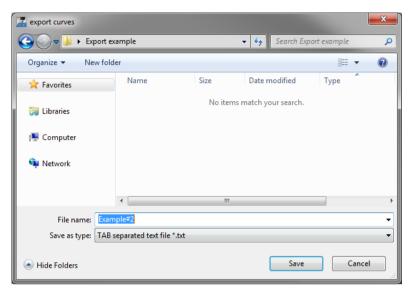
move the slider to select a filter size (sliding average) which is applied on the displayed curve(s).

#### 5.1.4 EXPORTING ALL INDENTATION CURVES

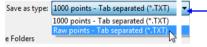


- To export all indentation curves.

#### This window allows:

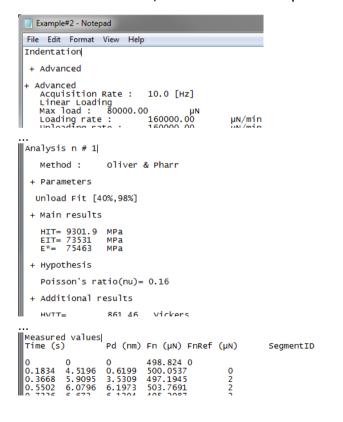


- Choosing a location where to save the export text file.
- Modification of the default *File name*: corresponding to the measurement file name with current measurement #.
- Changing of the default 1000 points<sup>1)</sup> .TXT file format.



Then click Save

In the saved text file, there are from top to bottom:

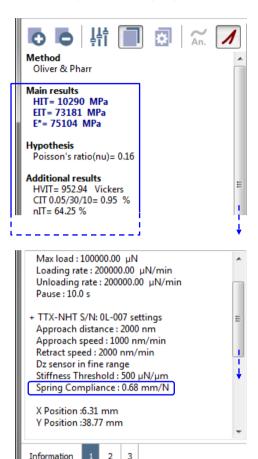


- The indentation measurement type and its parameters.
- All current analysis number(s) and their results.

- 1) The point values for each curve: 1000 points (resampling) or raw points (measured points) selected above when exporting.

#### 5.2 **NHT** SPRING COMPLIANCE

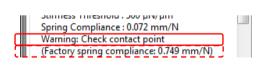
This chapter is only applicable for **NHT**.



The spring compliance value is taken to calculate the *Main* and *Additional* results.

This spring compliance value is computed by the software: it uses the Fn curve zone (approach) just before the contact point.

If the contact point is changed *chap. 5.1.1* **Setting the contact point, p. 132**, the value is recomputed and the results recalculated.



#### Warning

If the difference between the computed value (described above) and the factory<sup>1)</sup> value is larger than 20 %, the message "Warning: Check contact point" appears in the measurement parameter area. Therefore check the contact point chap. 5.1.1 Setting the contact point, p. 132.

The additional message "(Factory spring compliance: value1)" is also displayed in the measurement area.

<sup>1)</sup> factory value was set in the *Factory spring compliance* field of the NHT "*Ranges*" tab Fig.4, p. 17 (hardware configuration); see from chap. 3.1.3, p. 15.

## 5.3 OVERLAY PROPERTIES (INDENTATION COMBINED CURVE)

The surrounded icons on the additional toolbar below are only applicable with the *Oliver & Pharr*, *Tangent* and *Hertz* analysis methods; see *chap.* <u>5.4</u>, *p.* 144, and with the indentation combined curve displayed on the graph; see *chap.* <u>5.1</u>, *p.* 131.

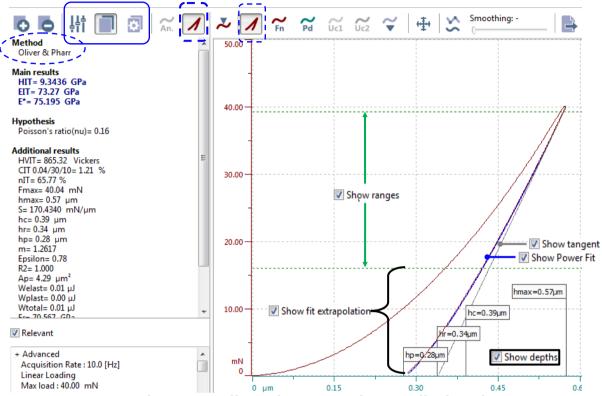
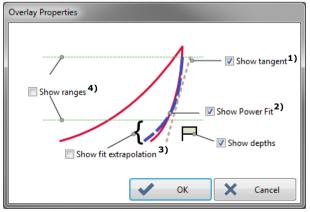


Fig.65 E.g. all overlay properties are displayed

#### **Display**

- To display or hide on/from the graph the overlay properties which are selected below.
- To select the overlay properties which should be shown.



**Default setting**, see Fig. 62, p. 130

Check each overlay property box which should be displayed (uncheck to hide).

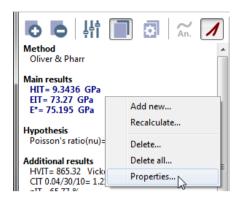
- 1) linear
- 2) power law
- in addition tangent is extended
- upper and lower bounds for the fit and tangent; see <u>Set range</u>, p. 143

#### Set range

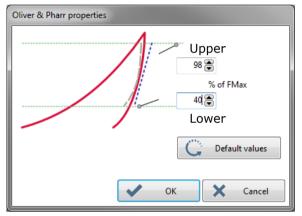
To set the upper/lower bounds for the fit and the tangent (not for *Hertz*) of the current analysis (e.g. *Oliver & Pharr*):



- Click this icon on the additional toolbar.



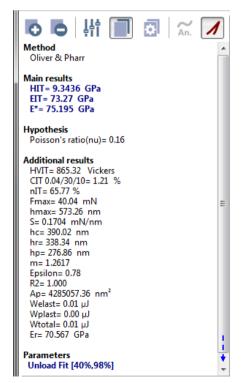
- Right click in the analysis result area and select **Properties...** from the context menu.



Set a percentage of Fmax to define the upper **and/or** lower bounds for the <u>unload</u> fit (*Oliver & Pharr* and *Tangent* analyses) or for the load fit (*Hertz* analysis).

Default analysis values:

Oliver & Pharr 98 % and 40 % 7angent 95 % and 70 % Hertz 98 % and 10 %



With new value(s) set above,

the *Main* and *Additional results* are recalculated in the analysis result area.

If necessary, scroll down in the result analysis area to display the current upper and lower bounds values (Unload fit or Load fit for Hertz analysis) under Parameters section.

## **5.4** Managing the analysis methods

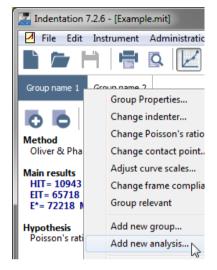
## 5.4.1 ADDING (MANUALLY) ANALYSIS METHOD(S)

From the results, to manually add new analysis method(s) for each:

Active document (all measurements of its groups)

- Select *Edit/Document \rightarrow*/*Add new analysis...* from the menu bar.

Active group (all its measurements)



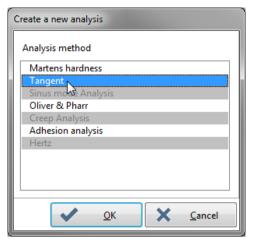
- Select **Edit/Group ►/Add new analysis...** from the menu bar.
- Right click on the "group name" tab and select Add new analysis... from the context menu.

#### Active measurement



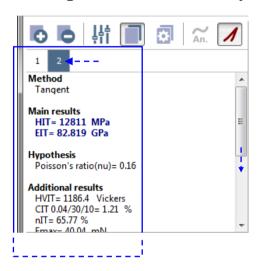
- Select *Edit/Analysis ▶/Add new...* from the menu bar.
- Click 🚺 on the additional toolbar.
- Right click in the analysis result area and select **Add new...** from the context menu.

See the following *Create a new analysis* window *Fig.66*, *p. 145*.



Select (double click) the desired analysis method, e.g. Tangent.

Fig.66 Create a new analysis window



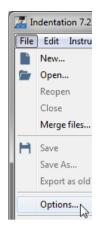
If there is already more than one analysis, a new analysis method "#" tab (e.g. "2") appears and calculates the *Main results* and *Additional results* for this new (current) analysis method;

see each detailed analysis method results from *chap.* <u>5.5</u>, *p.* 149 to *chap.* <u>5.9</u>, *p.* 158.

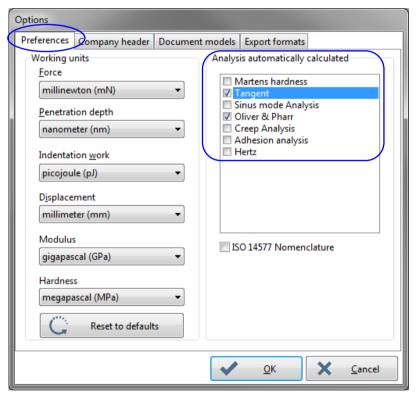
## **5.4.2** AUTOMATIC ANALYSIS METHOD(S)

It is possible to automatically add several analysis methods at the end of the measurement process for each measurement.

The following setting should be done before starting the measurement(s).

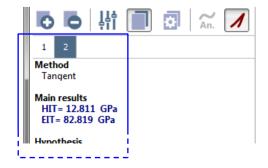


Select File/Options... from the menu bar.



In the Analysis automatically calculated area, check each desired analysis method box which should be automatically calculated after each measurement.

Fig.67 Preferences tab in Options window



E.g. "1" tab Oliver & Pharr and active "2" tab Tangent analysis methods are automatically created in the analysis result area after the measurement(s);

see each detailed analysis method results/curves from *chap.* <u>5.5</u>, *p.* 149 to *chap.* <u>5.9</u>, *p.* 158.

### **5.4.3 DELETING ANALYSIS METHOD(S)**

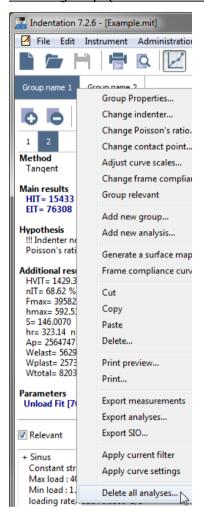
#### **Delete all methods**

To delete **all** the analysis methods for each:

Active document (all measurements of its groups)

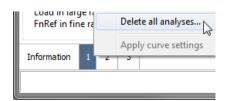
- Select **Edit/Document )**/**Delete all analyses...** from the menu bar.

Active group (all its measurements)



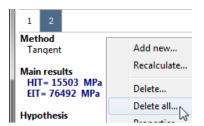
- Select **Edit/Group ▶/Delete all analyses...** from the menu bar.
- Right click on the "group name" tab and select **Delete all analyses...** from the context menu.

#### Active measurement



- Select *Edit/Indentation ▶/Delete all analyses...* from the menu bar.
- Right click on the "group name" tab and select
   Delete all analyses... from the context menu.

#### Active analysis method



 Right click on the "analysis method #" tab or on the analysis result area and select Delete all... from the context menu.

A Warning window appears, Click  $\sim$  to confirm the selected deletions or click  $\sim$  to keep them.

### **Delete the active method**

To delete the active analysis method:



- Select *Edit/Analysis ▶/Delete...* from the menu bar.
- Click **a** on the additional toolbar.
- Right click on the "analysis method #" tab or on the analysis result area and select Delete... from the context menu.

### **5.5** Adhesion analysis method & results

See also *chap.4.6.5.10* <u>Retract speed & time parameters for Adhesion</u>, *p. 120* (measurement preference parameters).

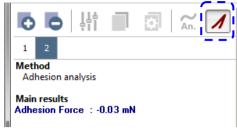
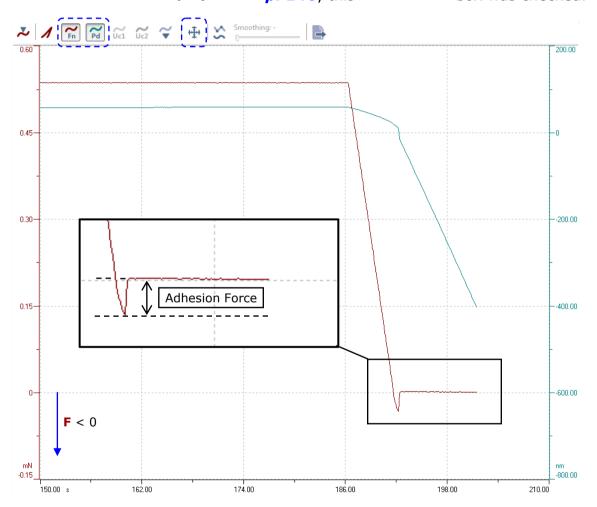


Fig.68 Adhesion Force of current measurement is displayed

After a measurement(s), to manually create an adhesion analysis, see *chap. 5.4.1 <u>Adding</u> (manually) analysis method(s), p. 144* and double click

Otherwise before a measurement(s), see chap. 5.4.2 <u>Automatic analysis method(s)</u>, p. 146, this Adhesion analysis box was checked.



Click in on the additional toolbar and adjust the **Min Fn** scale (Fn<0); refer to the **Common Scratch & Indentation software manual - chap. Manipulating document windows - Adapting curve ranges**.

# 5.6 ANALYSIS CURVE(S)

### For each active measurement



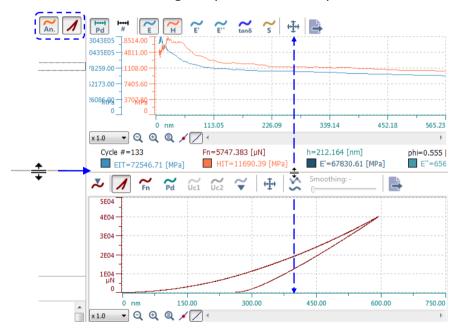
This icon is active on the additional toolbar only with:

- Martens hardness analysis method
- Sinus mode Analysis method (only with a Sinus measurement)
- Oliver & Pharr analysis method only with a Progressive multicycle or a CMC measurement type.

## To show/hide:

- The displayed analysis curve(s).
- The analysis curve features: depth or cycle mode, scale adjustment(s), export.

See the following chapters for descriptions of the related icons.



**Analysis** and indentation curves chap. 5.1, p. 131 can be displayed on the same time. In that case, each analysis curve is displayed on the **upper** graph and each indentation curve is displayed in the **lower** graph. The height size of the graphs can be adjusted by moving up/down the cursor between the 2 graph areas

#### 5.6.1 ANALYSIS CURVE DISPLAY

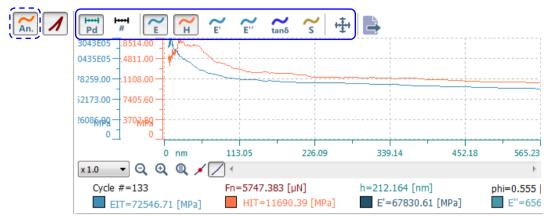


Fig.69 Displayed analysis curves on the graph

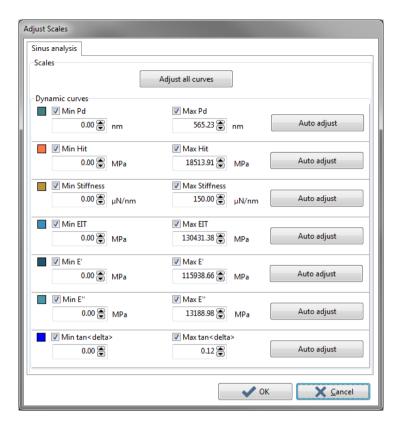
With *Martens* analysis method, only  $\oplus$  is available below.

To switch between the 2 modes below (X axis scale):

Depth mode; curves are displayed as a function of penetration depth (default)

### OR

- Cycle mode; curves are displayed as a function of the cycle numbers.
- To display (by default)/hide the elasticity curve.
- To display (by default)/hide the hardness curve.
- To display/hide the storage modulus curve.
- To display/hide the loss modulus curve.
- To display the tangent curve.
- $ightharpoonup^{<}$  To display the stiffness curve.
- To adjust the graph scale(s) for the displayed analysis curve(s); see the following window.



For the detailed description of this window, refer to the Common Scratch & Indentation software manual - chap. Manipulating document windows - Adapting curve ranges.

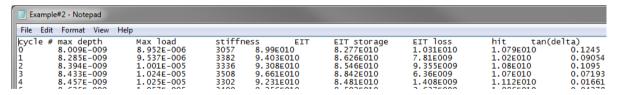
#### 5.6.2 EXPORTING ALL ANALYSIS CURVES





To export **all** analysis curves, even if they are hidden from the graph; refer to the similar explanations as described in **chap. 5.1.4 Exporting all indentation curves**, **p. 140**, **exceptions:** 

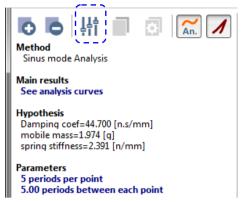
- Type a **File name**:
- There is only one .TXT file format available Save as type: TAB separated text file \*.txt > (raw).
- The saved text file contains only the points for each curve (raw: measured points).



### 5.7 DYNAMIC ANALYSES

See also chap. 5.6 Analysis curve(s), p. 150.

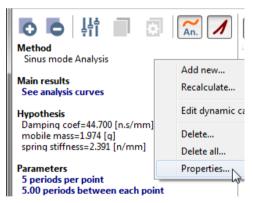
#### 5.7.1 SINUS MODE ANALYSIS METHOD & CURVE RESULTS



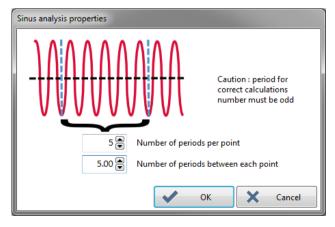
After a sinus measurement(s), to manually create a dynamic sinus analysis, see *chap. 5.4.1 <u>Adding</u>* (manually) analysis method(s), p. 144 and double click Sinus mode Analysis

Otherwise, before a measurement(s), see chap. 5.4.2 <u>Automatic analysis method(s)</u>, p. 146, this box Sinus mode Analysis was checked.





To filter **and/or** to display more points for the displayed sinus analysis curve(s), click on the additional toolbar **or** right click on the analysis result area and select **Properties...** 



To set the following number of periods, with odd numbers:

Number of periods per point

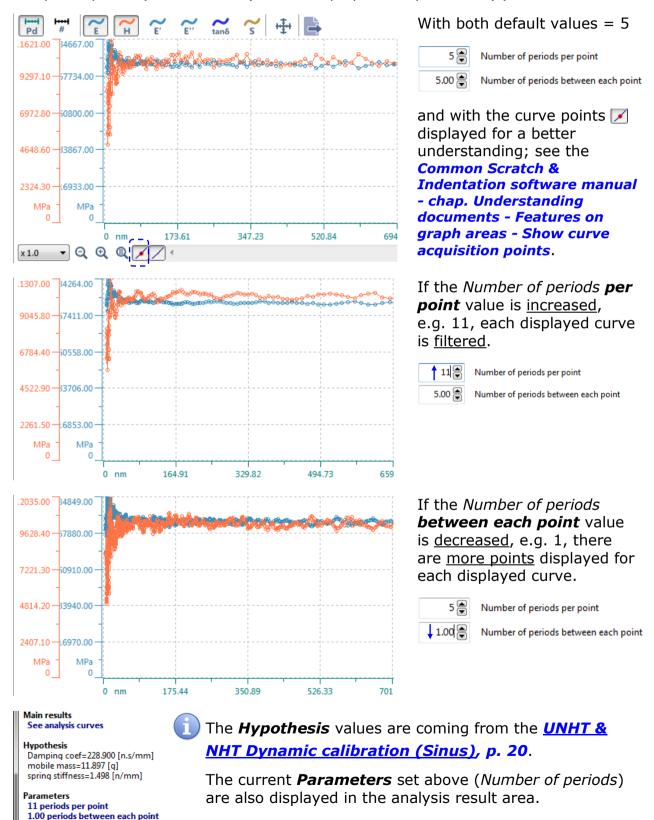
and/or

Number of periods between each point

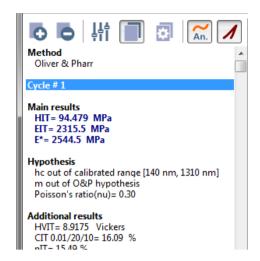
See the following parameter effects on the analysis displayed curves(s) only.

**Number of periods per point**: number of sinus periods used to compute each point of the displayed analysis curve(s).

**Number of periods between each point:** spacing (in period) between the computed points (2 consecutive) of the displayed analysis curve(s).



## 5.7.2 OLIVER & PHARR ANALYSIS CURVE(S) FOR CMC MEASUREMENT TYPE

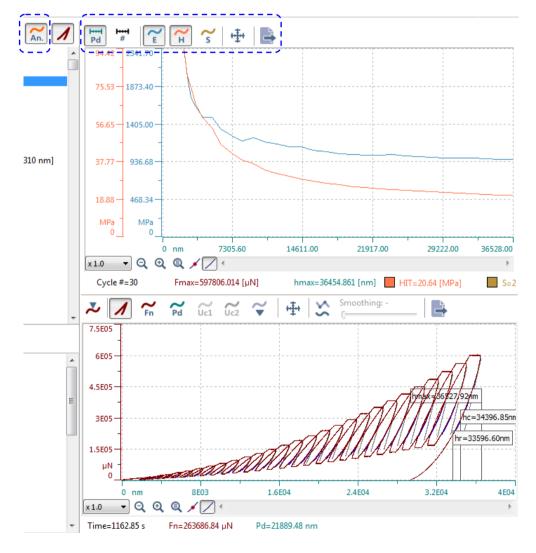


After a CMC measurement(s), to manually create a Oliver & Pharr analysis, see *chap. 5.4.1*Adding (manually) analysis method(s),

p. 144 and double click

Otherwise, before a measurement(s), see chap. 5.4.2 <u>Automatic analysis method(s)</u>, p. 146, this box Oliver & Pharr was checked.

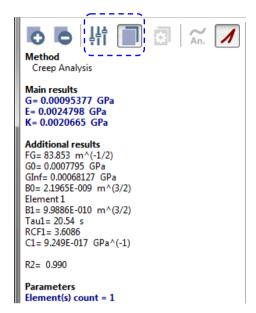
In addition to the indentation curves, the analysis curves are available for such type of measurement (CMC).



### 5.8 CREEP ANALYSIS METHOD & RESULTS



Creep analysis method is only available for measurement(s) with spherical indenter; otherwise it is inactive (grayed out).

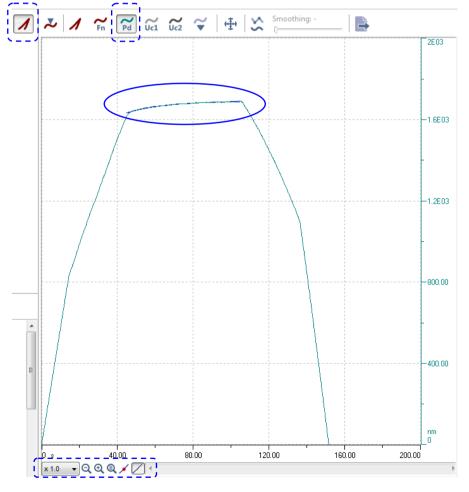


After a measurement(s), preferably with a constant force and a significant pause time, to manually create a creep analysis, see *chap. 5.4.1*Adding (manually) analysis method(s),

p. 144 and double click



Otherwise before a measurement(s), see chap. 5.4.2 <u>Automatic analysis method(s)</u>, p. 146, this box Creep Analysis was checked.



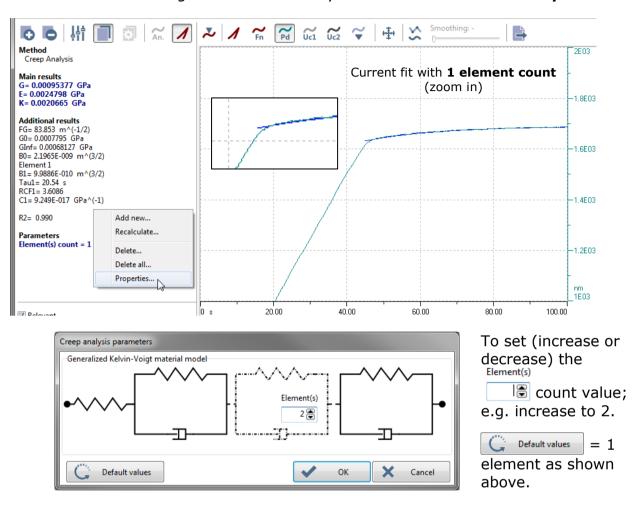
Only display the h depth curve and display the single overlay fir for this analysis (Kelvin-Voigt material model element).

Use the zoom tools

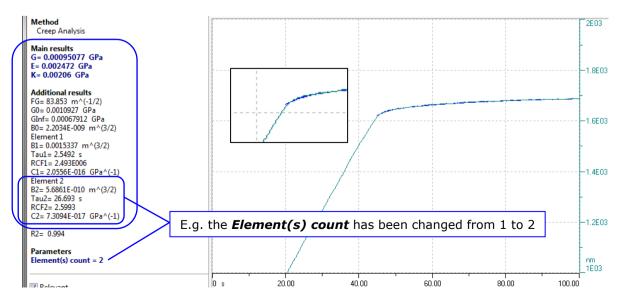
| x4.0 | Q Q Q to
| better see the fit on
| the pause; see the
| Common Scratch & Indentation software
| manual - chap. Understanding | documents - Features
| on graph areas - Show curve | acquisition points.

### Element(s) setting

To increase/decrease the Kelvin-Voigt material model elements, click  $\frac{1}{47}$  on the additional toolbar **or** right click on the analysis result area and select **Properties...** 

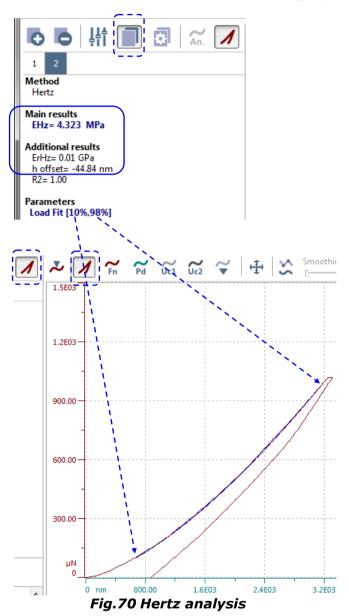


For each change of the element count above: the fit on the curve is adapted and in the analysis result area, the **Main** and **Additional results** are recalculated, and the current **Element(s) count** value is displayed.



### 5.9 HERTZ ANALYSIS METHOD & RESULTS

indenter; otherwise it is inactive (grayed out).



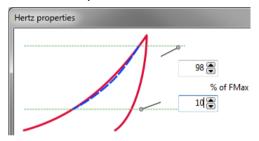
For each change of the upper/lower bound, the load fit on the curve is adapted and in the result analysis area, the *Main* and *Additional results* are recalculated, and the **Load fit** values are displayed.

After a measurement(s), to manually create a *Hertz* analysis, see *chap. 5.4.1 Adding (manually) analysis method(s)*, *p. 144* and double click

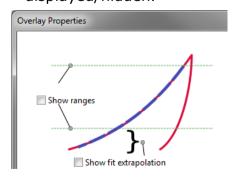
Otherwise before a measurement(s), see *chap.* 5.4.2 <u>Automatic</u> <u>analysis method(s)</u>, *p.* 146, this box was checked.

The upper **and/or** lower bound value(s) for the load fit can be changed; see the similar overlay properties *chap.* <u>5.3</u>, *p.* <u>142</u> descriptions, **exceptions**:

- Here they are for the **load** fit.



 The load fit is always displayed and only the upper/lower bound values (range) and/or extrapolation fit can be displayed/hidden.

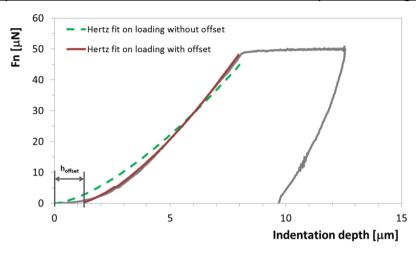


### **Information**

Hertz's model is used for spherical contact with plane for the following reasons:

- Some soft materials do not comply with O&P conditions (no plastic deformation).
- Elastic (poroelastic) deformation is predominant.
- Very often spherical indenter is used.

Depth offset is included to account for soft layers 'floating' above the surface



Hertz's analysis included in the *Indentation Software* is

$$F = \frac{4}{3} \cdot E_r \sqrt{R} \cdot (h - h_{offset})^{3/2}$$

## 5.10 HYPOTHESIS

For each active measurement, in the analysis result area under the *Hypothesis* section, several warning messages can appear and the Poisson's ratio is displayed and can be changed; see the following chapters for the detailed descriptions.

#### **5.10.1** INDENTER NOT CALIBRATED



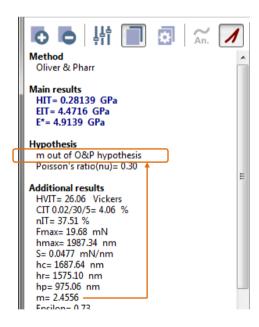
If the used indenter was not calibrated before performing the measurement (*Indenter properties* window <u>Fig. 20</u>, p. 37), the warning message "!!! Indenter not calibrated !!!" appears.



With the UNHT Bio, most of the time spherical indenters are used and do not need to be calibrated.

If necessary, the indenter can be changed with another one which was calibrated; see **chap.** 5.11.2, **p.** 166.

#### 5.10.2 m OUT OF O&P HYPOTHESIS

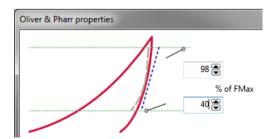


Under **Additional results**, if "m" (exponent in the O&P fit of the indentation curve unloading part) is > 2, the warning message "m out of O&P hypothesis" appears.



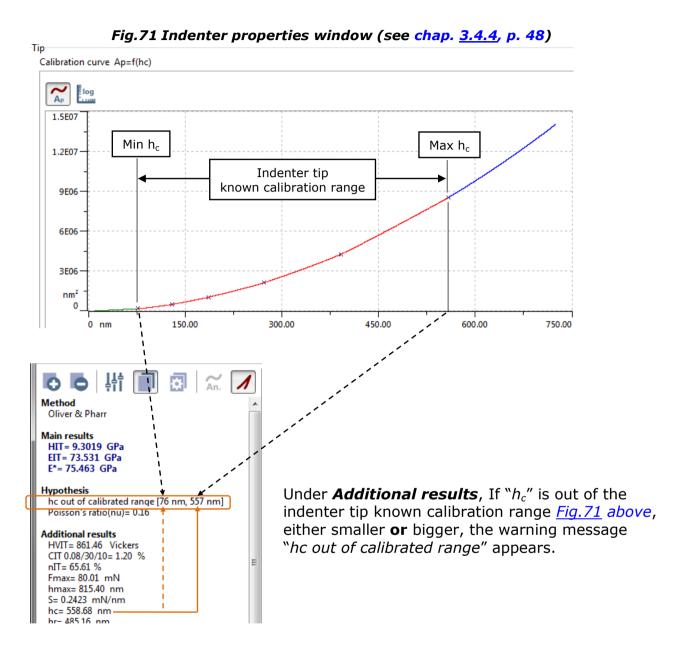
This message appears most of the time with soft material samples as they do not necessarily comply with the Oliver & Pharr (O&P) hypothesis; that is why other types of analysis are more appropriate, e.g. *Hertz* analysis *chap.* <u>5.9</u>, *p.* **158**.

"m" value can be changed (a little) by changing the upper/lower value of the bounds; see the overlay properties **chap. 5.3**, **p. 142**.



### **5.10.3** OUT OF INDENTER TIP CALIBRATION

The indenter tip calibration process is performed between a minimum and a maximum force resulting in a known contact depth range,  $h_c$ 



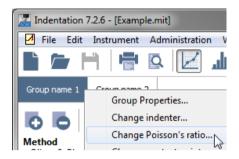
#### 5.10.4 CHANGING THE POISSON'S RATIO

To change the current Poisson's ratio for each:

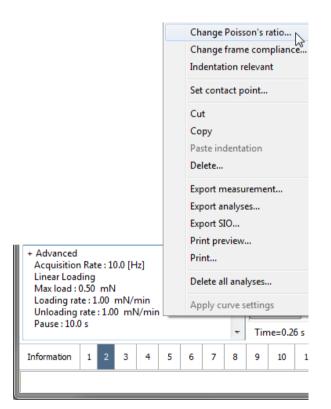
Active document (all measurements of its groups)

- Select *Edit/Document \rightarrow/Change Poisson's ratio...* from the menu bar.

Active group (all its measurements)



## Active measurement



- Select Edit/Group >/Change
   Poisson's ratio... from the menu bar
- Right click on the "group name" tab and select Change Poisson's ratio... from the context menu
- Select *Edit/Indentation* ►/ *Change Poisson's ratio...* from the menu bar

 Right click in the measurement parameter area or on the "measurement #" tab, and select Change Poisson's ratio...from the context menu

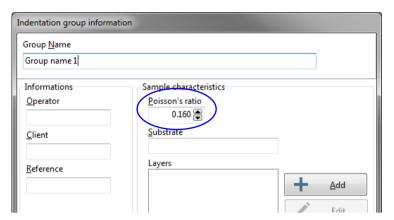


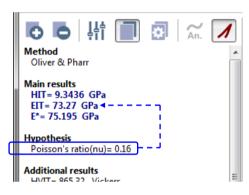
This window allows changing of the current Poisson's ratio value for the measurements(s) previously selected.



To set another Poisson's ratio value.

To reset the Poisson' ratio with the value set during the new document/group creation.

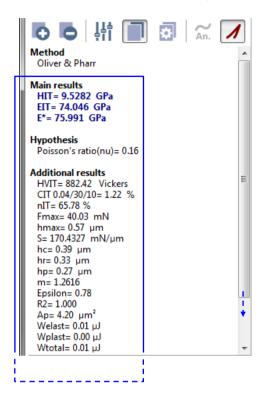




In the analysis result area, for each change of the Poisson's ratio value (set above), a new value for **EIT** under **Main results** is recalculated accordingly.

# **5.11** Managing the indenter in the result analysis

The indenter or some of its parameters can be changed.



#### **Recalculated results**

For changes of the indenter properties (some parameters/fit method), **chap.** 5.11.1, **p.** 165,

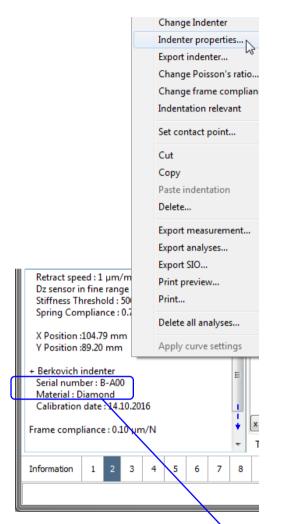
## and/or

for each change of the indenter chap. <u>5.11.2</u>, p. 166,

the *Main* and *Additional* results in the analysis result area are recalculated for each concerned measurement.

#### **5.11.1** INDENTER PROPERTIES

To change some parameters of the current indenter for each <u>active measurement</u> (only):



- Select *Edit/Indentation ▶/Indenter properties...* from the menu bar

- Right click in the measurement parameter area or on the "measurement #" tab, and select Indenter properties... from the context menu
- If necessary, scroll down in the measurement parameter area to display the current indenter information.

The *Indenter properties* window appears and allows changing of:

- the indenter parameters, except the indenter Type; see chap. 3.4.1, p. 37, the indenter information are updated in the measurement parameter area
- the indenter fit method; see
   chap. 3.4.4, p. 48.
- From here it is not possible to calibrate the indenter (the button "Calibrate" is hidden).

For each change of the indenter properties, see **Recalculated results**, p. 164.

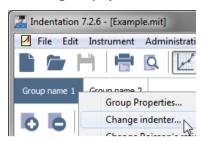
#### **5.11.2** CHANGING THE INDENTER

To change the current indenter for each:

Active document (all measurements of its groups)

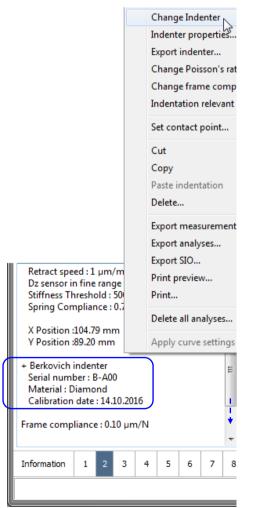
- Select **Edit/Document /Change indenter...** from the menu bar.

### Active group (all its measurements)



- Select *Edit/Group ▶/Change indenter...* from the menu bar
- Right click on the "group name" tab and select Change indenter... from the context menu

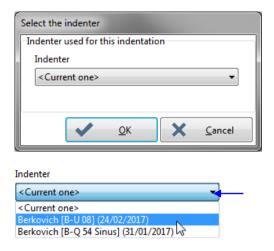
### Active measurement



Select Edit/Indentation >/Change indenter... from the menu bar

- Right click in the measurement parameter area or on the "measurement #" tab, and select Change indenter from the context menu
- i If necessary, scroll down in the measurement parameter area to display the current indenter information.

Fig.72 Measurement parameter area



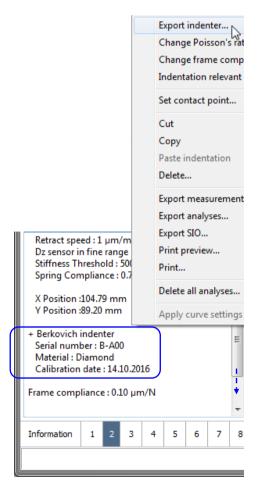
This window allows changing of the current indenter for the measurement(s) previously selected.

To change the current indenter, with another available one.

For each change of the indenter, see **Recalculated results**, p. 164.

The indenter information is updated in the measurement parameter area <u>Fig. 72</u>, p. 166.

#### **5.11.3** EXPORTING THE INDENTER



For each active measurement, right click in the measurement parameter area and select **Export indenter...** from the context menu.

The same *Export a indenter file* window as described in *Import/export*, *p. 35* appears.

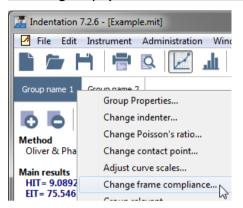
### 5.12 CHANGING THE FRAME COMPLIANCE

To change the current frame compliance value taken to calculate the results for each:

Active document (all measurements of its groups)

- Select **Edit/Document )/Change frame compliance...** from the menu bar.

### Active group (all its measurements)



- Select *Edit/Group ▶/Change frame compliance...* from the menu bar.
- Right click on the "group name" tab and select Change frame compliance... from the context menu.

### Active measurement

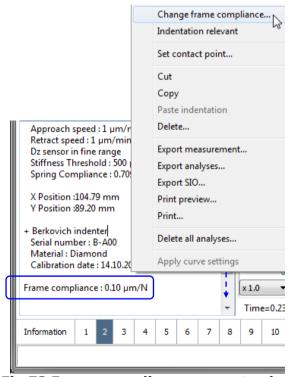
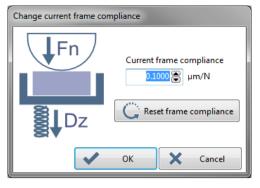


Fig. 73 Frame compliance current value

Select *Edit/Indentation \righterrightarrow/Change frame compliance...* from the menu bar.

 Right click in the measurement parameter area or on the "measurement #" tab and select Change Frame Compliance from the context menu.



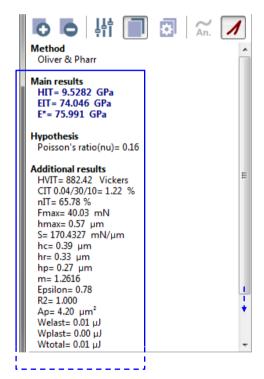
This window allows changing of the current frame compliance value for the measurement(s) previously selected.



To set another frame compliance value.

To reset the frame compliance with the initial<sup>1)</sup> value.

For each change of the frame compliance, the value is updated in the measurement parameter area <u>Fig. 73</u>, p. 168 and the **Main** and **Additional** results in the analysis result area (below) are recalculated for each concerned measurement.



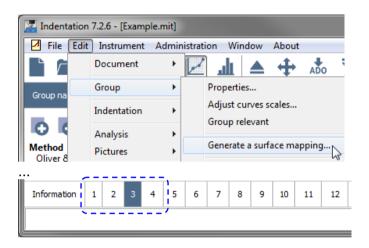
<sup>1)</sup> initial value was set in the *Frame compliance* field of the "Indenter *Ranges*" tab for UNHT <u>Fig.1</u>, p. 15 or "Ranges" tab for NHT <u>Fig.4</u>, p. 17/MHT <u>Fig.9</u>, p. 24 (hardware configuration); see from **chap.** 3.1.3, p. 15

## 5.13 GENERATING SURFACE MAPPING (IMAGE)

The surface mapping feature is used to create 3D image(s) from each (one at a time) selected result parameter of a matrix of measurements, which were performed at different positions on the (same) sample.

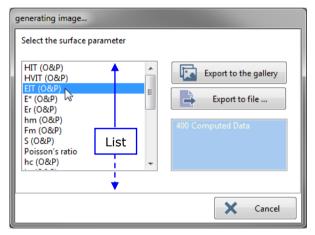
This feature generates image(s) where each pixel location represents a measurement position and each pixel color represents a different measurement value of the selected result parameter.

## For each active group (all its measurements)



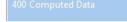
The (matrix) group should contains at least 4 measurements,

select *Edit/Group* // *Document/Generate a surface mapping...* from the menu bar.

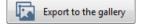


Select one result parameter in the list (standard O&P Oliver & Pharr analysis method, other method(s) in addition if available/added).

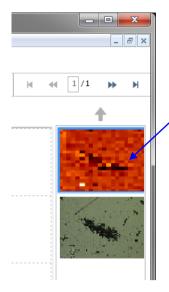
Generally the most interesting parameters are HIT, HVIT and EIT.



Shows the number of the measurements for the selected parameter, in the min. number should be 4 otherwise an *Information* window will appear (If an analysis method is manually added (afterwards), it should be done for the other measurements of the current group, at least 4).



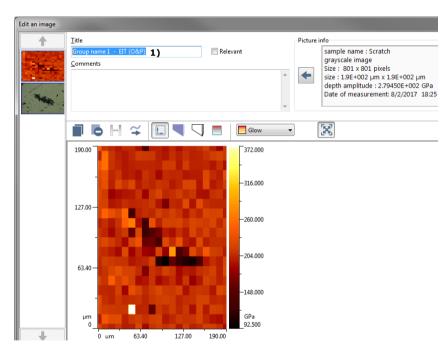
To export the corresponding image (parameter selection above) in the gallery of the result analysis window.



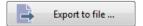
Surface mapping image are added to the gallery with other images (if previously grabbed).

Double click the image to open it in the window below.

The *Edit an image* window below allows some settings on the opened image (Title name, comments, image features...); refer to the *Common Scratch & Indentation software manual - Manipulating document windows - Managing images gallery - Managing picture*.

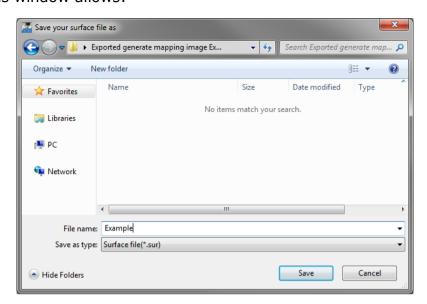


1) to know which measurement group tab and which parameter were previously selected to generate the image, the <u>Title</u> name field is automatically filled in with these information (can be changed if necessary)



To export the corresponding image (previous parameter selection) in .SUR file format, for an external analysis with another software like Image Plus; refer to the *Image Plus software manual*.

## This window allows:



- Choosing a location where to save the surface file.
- Typing a **File name**: The default file format is .SUR

Then click Save .

## **6 TROUBLESHOOTING**

## 6.1 F.A.Q.

Refer to the \*measurement head\* user manual - chap. F.A.Q.

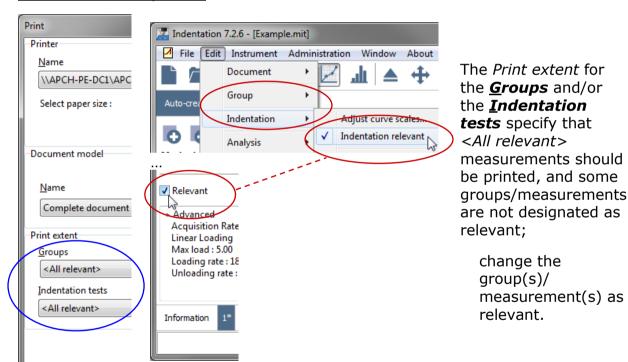
#### 6.2 ERROR MESSAGES WHILE USING THE INSTRUMENT

Refer to the \*measurement head\* user manual - chap. Blocking error messages.

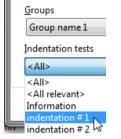
#### **6.3** Unprinted items

Some indentation measurements or images are not printed on the document; this may occur when:

#### **Measurements unprinted**



Otherwise select the Print extent for the desired,



Print extent

#### Groups:

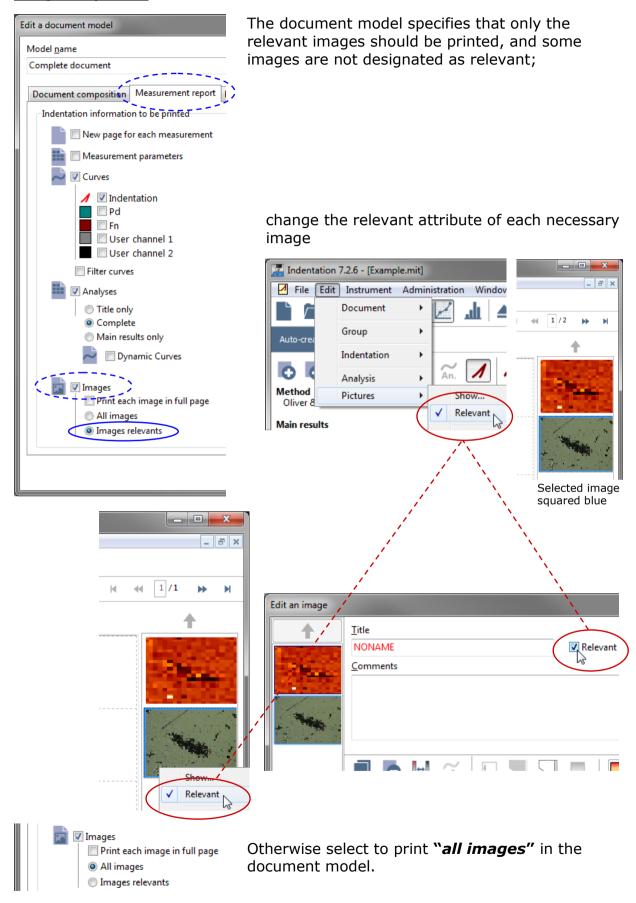
<ALL> or "Group name"

#### AND/OR

#### Indentation test:

<ALL> or "indentation #" measurement(s).

### **Images unprinted**



### **6.4** Instrument seems to behave strangely

Verify that the field values of the 'Ranges' tabs<sup>1)</sup> (in Hardware configuration window) match with the values stated on the calibrations certificates, which are provided with the corresponding instrument. Otherwise change them accordingly. Refer to the Common Scratch & Indentation software manual - chap. 'Ranges' tab(s).

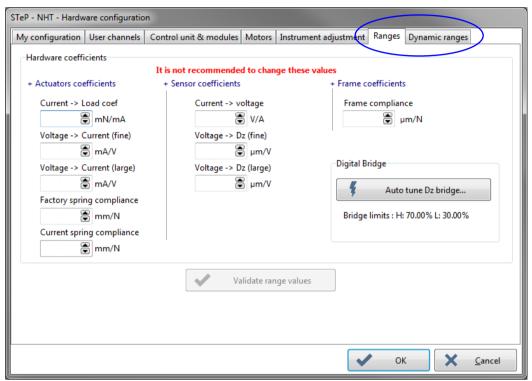


Fig.74 'Ranges' tabs (e.g. NHT)

1) UNHT has 3 'Ranges' tabs

NHT has 2 'Ranges' tabs

MHT has only 1 "Ranges" tab

Ranges

Reference Ranges

Reference Ranges

Ranges

Ranges

# **7 SOFTWARE FORMULAS**

Graphical views of indentation parameters:

## 7.1 Typical indentation curve

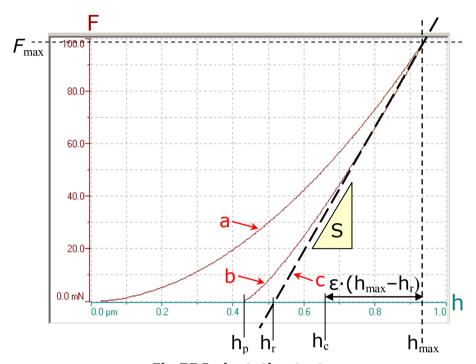


Fig. 75 Indentation test

### Legend

a : Application of Fb : Removal of F

 $\boldsymbol{c}\;$  : Tangent to curve b at  $\boldsymbol{F}_{\text{max}}$ 

F: test force

 $F_{max}$ : maximum test force

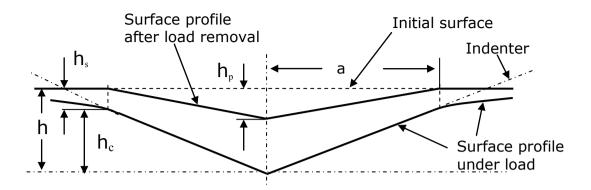
h<sub>p</sub>: permanent indentation depth
 h<sub>r</sub>: tangent indentation depth

 $h_c$ : contact depth of the indenter with the sample at  $F_{max}$ 

h<sub>max</sub>: maximum indentation depth

S : contact stiffness ε : geometric constant

## 7.2 SCHEMATIC REPRESENTATION OF INDENTER - SAMPLE CONTACT



## 7.3 PARAMETERS DETERMINATION

F<sub>max</sub>: Directly on curve

S (Tangent method): Tangent fit: Fit start: 95 % of  $F_{\text{max}}$  Fit end: 70 % of  $F_{\text{max}}$ 

$$S = \left(\frac{dF}{dh}\right)_{m}$$

m,  $h_{max}$ ,  $h_p$  and S (O&P method):

Power law fit of unloading curve:

Fit start: 98 % of  $F_{\text{max}}$ Fit end: 40 % of  $F_{\text{max}}$ 

Equation:

$$F = F_{\text{max}} \cdot \left(\frac{h - h_{p}}{h_{\text{max}} - h_{p}}\right)^{m}$$

Where m,  $h_{\text{max}}$ ,  $h_{\text{p}}$  are fitting parameters.



- In case of creep or viscoelasticity still occurring during unloading, it may happen that the h<sub>max</sub> value in the results cannot be seen as a data point on the curve.
- Due to complex behavior of contact mechanics at the end of the unloading curve (roughness, tip shape, creep, viscoelasticity...), it may happen that the h<sub>p</sub> value in the results cannot be seen as a data point on the curve.

$$S = m \cdot F_{\text{max}} \cdot (h_{\text{max}} - h_{\text{p}})^{-1}$$

Calculation of S:

h<sub>r</sub>: Intercept of the tangent to the load-displacement data at the maximum load on unloading (S) with the depth axis.

$$h_r = h_{max} - F_{max} / S$$

$$h_c = h_{max} - \epsilon \cdot (h_{max} - h_r)$$

ε: Depending on the diamond shape

Indenter Shape	m	ε	
Flat Punch	1.0	1.0000	
Cone	2.0	$2(\pi - 2) / \pi = 0.7268$	
Sphere/Paraboloïd	1.5	0.7500	

In our case,  $\varepsilon$  is estimated using the m value! (Table of 10 values and linear extrapolation between 2 values)

Ref.: J. Woirgard and al./Surface and coatings Technology 100-101 (1998) 103-109

β: Geometric factor (diamond shape dependent)

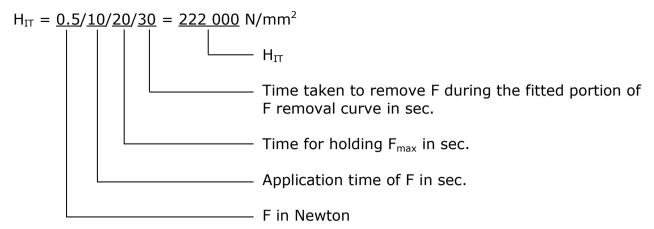
Circular  $\beta = 1.000$ Triangular  $\beta = 1.034$ Square  $\beta = 1.012$ 

A<sub>p</sub>: (theoretical or calibrated)

$$A_n = f(h)$$

# 7.4 Indentation hardness (H<sub>IT</sub>)

## 7.4.1 **DESIGNATION OF HIT**



## 7.4.2 DETERMINATION OF H<sub>IT</sub>

H<sub>IT</sub> is a measure of the resistance to permanent deformation or damage.

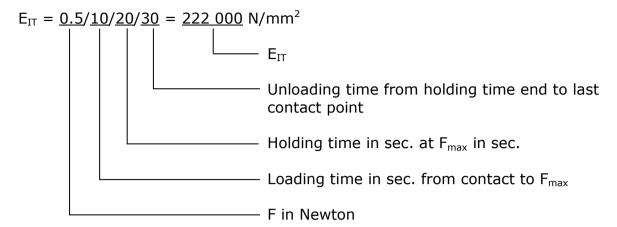
$$H_{\text{IT}} = \frac{F_{\text{max}}}{A_{\text{p}}} \text{ in Pascal}$$

 $A_p$  (theoretical or calibrated)

The determination of the exact area function for the given indenter is required for indentation depth < 0.006mm (tip defect).

## 7.5 INDENTATION MODULUS $(E_{IT})$

### 7.5.1 DESIGNATION OF $E_{TT}$



## 7.5.2 DETERMINATION OF REDUCED MODULUS (ER)

The deduced modulus is calculated from the following equation:

$$E_r = \frac{\sqrt{\pi} \cdot S}{2 \cdot \beta \cdot \sqrt{A_p(h_c)}}$$

# 7.5.3 DETERMINATION OF PLANE STRAIN MODULUS (E\*)

E\* is calculated from the following equation:

$$E^* = \frac{1}{\frac{1}{E_r} - \frac{1 - v_i^2}{E_i}}$$

With:

 $E_i$  = Elastic modulus of the indenter (diamond 1141 GPa)

 $v_i$  = Poisson's ratio of the indenter (diamond 0.07)

## 7.5.4 DETERMINATION OF E<sub>IT</sub>

 $\mathsf{E}_{\scriptscriptstyle\mathsf{IT}}$  calculated from the E\* using a an estimated sample Poisson's ratio ( $\mathsf{V}_{\scriptscriptstyle\mathsf{S}}$ ):

$$E_{IT} = E * \cdot (1 - v_s^2)$$

As a reminder, typical values of  $v_s$  are:

Ceramic: 0.1 to 0.3Metal: 0.2 to 0.4Polymer: 0.3 to 0.4

## 7.6 STANDARD MEASUREMENT OF VICKERS HARDNESS (HV)

#### 7.6.1 DESIGNATION OF HV

$$HV = 0.1$$
 Vickers

Hardness value

#### 7.6.2 DETERMINATION OF HV

$$HV = \frac{F_{\text{max}}}{9.81 \cdot A_c(h_c)} \quad \text{in Vickers (kgf/mm²)}$$

(diagonal measurement)

$$HV = \frac{F[kgf]}{A_d} = \frac{F}{A_p} \cdot \sin 68^\circ = \frac{2 \cdot F}{D^2} \cdot \sin 68^\circ$$

$$HV \approx 1.8544 \cdot \frac{F}{D^2}$$

$$\frac{D}{h} = 2\sqrt{2} \cdot \tan 68^{\circ} \approx 7$$

Where:

h is the indentation depth under applied load (corresponding to diagonal D)

# 7.7 ESTIMATION OF $HV_{IT}$ WITH INDENTATION HARDNESS ( $H_{IT}$ )

$$H_{IT}[MPa] = \frac{F[N]}{A_p} = \frac{9.81 \cdot F[kg]}{A_d \cdot \sin \alpha} = \frac{9.81}{\sin \alpha} \cdot HV_{IT}[Vic \text{ ker s}]$$

With Vickers indenter:

$$HV_{rr} \approx H_{rr}/10.580$$

With Modified Berkovich indenter (M. Berkovich):

$$HV_{rr} \approx H_{rr}/10.800$$

## 7.8 INDENTER DEFINITION & SPECS, WITH AREA FUNCTION RELATIONSHIPS

For indentation depth > 0.006mm, a first approximation to the projected area may be used:

	Vickers	Berkovich	M. Berkovich	Cube Corner
a <sub>t</sub>	136°	141.9°	142.3°	90°
а	68°	65.03°	65.27°	35.264°
$A_d / h^2$	$4 \cdot \frac{\sin \alpha}{\cos^2 \alpha}$	$3\sqrt{3} \cdot \frac{\sin\alpha}{\cos^2\alpha}$	$3\sqrt{3} \cdot \frac{\sin \alpha}{\cos^2 \alpha}$	9/2
	≈26.43	≈26.43	≈26.97	=4.5
$A_p / h^2$	$4 \cdot \tan^2 \alpha$	$3\sqrt{3} \cdot \tan^2 \alpha$	$3\sqrt{3} \cdot \tan^2 \alpha$	$3\sqrt{3}/2$
	≈24.504	≈23.96	≈24.494	≈2.598
A <sub>d</sub> / A <sub>p</sub>	$1/\sin \alpha$	$1/\sin lpha$	$1/\sin \alpha$	$1/\sin\alpha = 3/\sqrt{3}$
	≈1.0785	≈1.1031	≈1.1010	≈1.7320

 $a_t$  = total included angle

a = angle between the axis of the diamond pyramid and the 3 faces

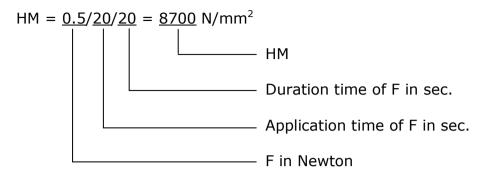
$$A_d = A_c = A_s =$$
 (developed) contact area

#### Reminder:

 $A_p$  = projected contact area

## 7.9 MARTENS HARDNESS (HM) (FORMER DESIGN: UNIVERSAL HARDNESS HU)

#### 7.9.1 DESIGNATION OF HM



### 7.9.2 DETERMINATION OF HM

HM is measured under applied F.

HM is determined from the values given by the F/h curve during load time, preferably after reaching the specified F.

HM includes the plastic and elastic deformation, thus this hardness value can be calculated for all materials. HM value is defined for both pyramidal indenters (Vickers and Berkovich). It is not defined for the Knoop or ball indenters.

HM is defined as F divided by the surface contact area of the indenter penetrating beyond the zero point of the contact:

$$HM = \frac{F}{A_s(h)}$$

Vickers indenter:

$$A_s(h) = \frac{4 \cdot \sin(\alpha)}{\cos^2(\alpha)} \cdot h^2 \qquad \text{and} \quad HM = \frac{F}{A_s(h)} \approx \frac{F}{26.43 \cdot h^2}$$

• Perfect Berkovich indenter:

$$A_s(h) = \frac{3 \cdot \sqrt{3} \cdot \tan(\alpha)}{\cos(\alpha)} \cdot h^2 \qquad \text{and} \quad HM = \frac{F}{A_s(h)} \approx \frac{F}{26.43 \cdot h^2}$$

• Modified Berkovich indenter:

$$A_s(h) = \frac{3 \cdot \sqrt{3} \cdot tan(\alpha)}{cos(\alpha)} \cdot h^2 \qquad \text{and} \qquad HM = \frac{F}{A_s(h)} \approx \frac{F}{26.97 \cdot h^2}$$

HM is usually followed by the test condition in the following order:

- Fin N,
- the time for the application of F in sec.
- the time during F<sub>max</sub> is kept constant in sec.
- after equals sign, HM value.

#### Examples:

• HM  $0.5/20/20 = 8700 \text{ N/mm}^2$ 

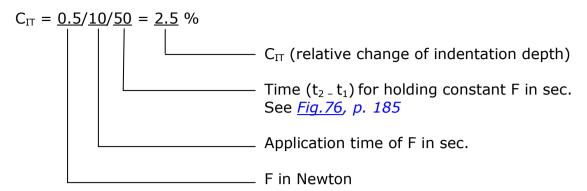
HM is 8700 N/mm<sup>2</sup>, determined with a F of 0.5N, applied in 20s, constant during 20s.

• HM(Berkovich)  $0.5/20/20 = 8700 \text{ N/mm}^2$ 

Same as above but with a Berkovich tip instead of the standard Vickers tip.

# 7.10 INDENTATION CREEP (CIT)

## 7.10.1 DESIGNATION OF $C_{IT}$



## 7.10.2 DETERMINATION OF $C_{IT}$

$$C_{IT} = \frac{h_2 - h_1}{h_1} \cdot 100$$

Where:

 $h_1$  is the indentation depth at time  $t_1$  of reaching F (which is kept constant)

 $h_2$  is the indentation depth\_at time  $t_2$  of holding the constant F

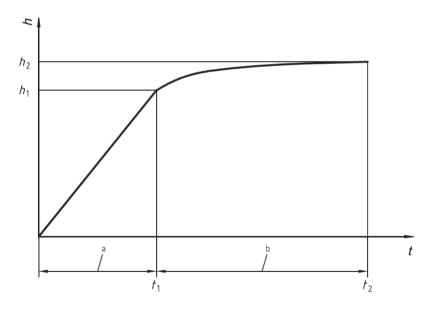


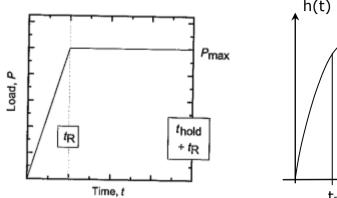
Fig. 76 Expression of CIT

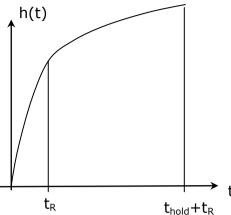
a: Application of F

b: F constant from  $t_1$  to  $t_2$ 

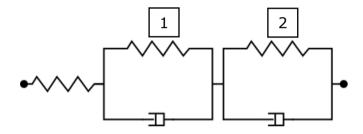
#### 7.10.3 CREEP ANALYSIS

This analysis is used to determine the viscoelastic properties from the creep curve (pause at constant force) with a sphere only.





Example for the case of 2 elements



The material response can be described with the following creep function:

(1) 
$$J(t) = C_0 - C_1 \exp(-t/\tau_1) - C_2 \exp(-t/\tau_2)$$

Then the indentation depth follows the below equation:

(2) 
$$h^{3/2}(t) = B_0 - B_1 \exp(-t/\tau_1) - B_2 \exp(-t/\tau_2)$$

(for 1 element the constant terms are B0 and B1 whereas for 3 elements there are B0, B1, B2 et B3)

The analysis performs a fit of the indentation depth h(t) between  $t_R$  and  $(t_{hold}+t_R)$  using the equation (2) giving the fit parameters of  $B_0$ ,  $B_1$ ,  $B_2$ ,  $\tau_1$  et  $\tau_2$ 

Then the results are calculated using the following equations:

(3) 
$$F_G = 3/(8\sqrt{R})$$
 tip Geometry Factor, where R is sphere radius

(4) 
$$RCF_1 = \frac{\tau_1}{t_R} \left[ exp(t_R / \tau_1) - 1 \right]$$
 Ramp Correction Factor

(5) 
$$RCF_2 = \frac{\tau_2}{t_R} \left[ exp(t_R / \tau_2) - 1 \right]$$

(6) 
$$C_0 = \frac{B_0}{F_m F_G}$$

where  $F_{m}$  is the max. test force

(7) 
$$C_1 = \frac{B_1}{F_m F_G \cdot RCF_1}$$

(8) 
$$C_2 = \frac{B_2}{F_m F_G \cdot RCF_2}$$

(9) 
$$G_0 = \frac{1}{2(C_0 - C_1 - C_2)}$$

$$(10) \qquad G_{\infty} = \frac{1}{2C_0}$$

(11) 
$$G^{v} = 2G_{\infty}(1-v)$$

shear modulus - where  $\nu$  is the tested material Poisson's ratio

(12) 
$$E = 2G^{\nu}(1+\nu)$$

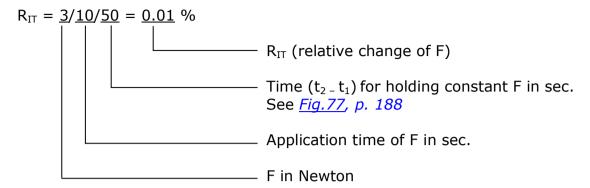
elastic modulus

(13) 
$$K = \frac{E}{3(1-2v)}$$

bulk modulus

## 7.11 INDENTATION RELAXATION $(R_{IT})$

## **7.11.1** DESIGNATION OF $R_{IT}$



## 7.11.2 DETERMINATION OF $R_{IT}$

$$R_{IT} = \frac{F_1 - F_2}{F_1} \cdot 100$$

Where:

 $F_1$  is the force at reaching the indentation depth, which was kept constant, in N

 $\mathsf{F}_2$  is the force after the time during which the indentation depth was kept constant, in  $\mathsf{N}$ 

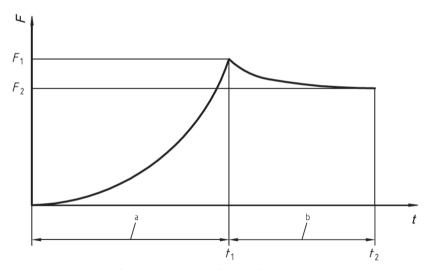


Fig.77 Expression of R<sub>IT</sub>

a: Application of the indentation depth

b: Indentation depth kept constant from  $t_1$  to  $t_2$ 

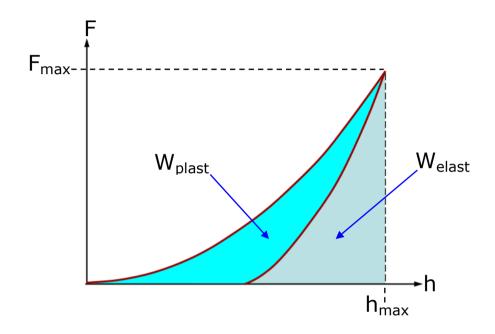
# 7.12 ELASTIC PART OF INDENTATION WORK

$$\eta_{\rm IT} = \frac{W_{elast}}{W_{total}} \cdot 100$$

With:

 $W_{total} = W_{elast} + W_{plast}$ 

Plastic part  $W_{\text{plast}}$  /  $W_{\text{total}}$  follows as 100 % -  $\eta_{\text{IT}}$ 



### 7.13 TANGENT METHOD

The Tangent Method is also called the Linear Extrapolation Method. This assumes that the **first portion of the unloading curve is linear and simply extrapolates that linear portion to intercept the displacement axis**. This method is applicable to materials that show a high degree of stiffness and a large deformation so that the unloading curve is, to a good approximation, linear.

A simple linear fit through the upper part of the unloading data intersects the depth axis at  $h_{\rm r}$ 

S is given by the slope of this line

h<sub>c</sub> is then calculated as:

$$h_c = h_{max} - \varepsilon \cdot (h_{max} - h_r)$$

Where:

 $\epsilon$  depends on the indenter shape.

# 7.14 POWER LAW METHOD (OLIVER & PHARR)

The Power Law Method recognizes the fact that the **first portion of the unloading** curve may not be linear, and can be described by a simple power law relationship:

$$F = k \cdot (h - h_p)^m$$

Where:

k is a constant and m is an exponent which depends on indenter geometry

A power law function is used to describe the upper part of the unloading data.

$$F = F_{\text{max}} \cdot \left(\frac{h - h_{p}}{h_{\text{max}} - h_{p}}\right)^{m}$$

Where:

The constants m and  $h_p$  are determined by a least squares fitting procedure

S (= 1 / C) is given by the derivative at peak load:

$$S = \left(\frac{dF}{dh}\right)_{max} = m \cdot F_{max} \cdot \left[\frac{\left(h_{max} - h_{p}\right)^{m-1}}{\left(h_{max} - h_{p}\right)^{m}}\right] = m \cdot F_{max} \cdot \left(h_{max} - h_{p}\right)^{-1}$$

h<sub>r</sub> is thus given by:

$$h_{r} = h_{max} - \frac{F_{max}}{S}$$

h<sub>c</sub> is then:

$$h_c = h_{max} - \epsilon \cdot (h_{max} - h_r)$$

Where:

 $\epsilon$  now depends on m

The tangent is found by differentiating the unloading curve and evaluating at F<sub>max</sub>

The intercept of this tangent with the displacement axis yields h<sub>r</sub>

### 7.15 REFERENCES

### ISO/DIS Standard 14577-1:2002 DIN Standard 50359-1

\*Meyer, E., Z. Ver. Dtsch. Ing. 52 (1908) 645

\*Doerner, M.F., and Nix, W.D., "A method for interpreting the data from depth sensing indentation instruments", J. Mat. Res., 1, (1986), 601-609

\*Oliver, W.C., Pharr, G.M., "An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments", J. Mat. Res., 7(6), June 1992, 1564-1583

\*ISO/DIS Standard 14577-1, ISO/DIS 14577-2

\*J. Woirgard and al./Surface and coatings Technology 100-101 (1998) 103-109

If problems should be encountered, refer to this manual and to the others mentioned.

Otherwise contact us,



www.anton-paar.com