

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

Galileo Galilei (1564-1642)

## Software Reference Guide

## Indentation

From Indentation Software Version 9 for Windows<sup>®</sup> 7 64 bits and Windows<sup>®</sup> 10

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Software Reference Guide

# Indentation

From Indentation Software Version 9 for Windows  $^{\mbox{\tiny B}}$  7 64 bits and Windows  $^{\mbox{\tiny B}}$  10

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## **1** ABOUT THIS REFERENCE GUIDE

## **1.1 IMPORTANT: ACQUISITION SYSTEM CONFIGURATION**

**REMINDER:** *Refer to the same section in the* **Common Scratch & Indentation** *reference guide.* 

## **1.2** INTRODUCTION

## **1.2.1** ABOUT THE INDENTATION SOFTWARE

By choosing an Indentation Tester from the complete range proposed 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, statistics and advanced reporting functions.

The *Indentation Software* (for Windows<sup>®</sup> 7 64 bits and Windows<sup>®</sup> 10) provided by Anton Paar enables a complete integration of various data in a single document.

## **1.2.2** ENCLOSED CONTENT

This document describes the *Indentation Software* features.

Some *Indentation Software* features are common with *Scratch Software*. If the information is not described in this document, refer to the *Common Scratch & Indentation software reference guide*.

For explanations (set up and use) of each Indentation head, refer to its corresponding **user manual** or **reference guide**.

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

#### **1.2.3** How to use this document?

If the components and software (*Indentation*) are not yet installed on the acquisition system, refer to the **Common Scratch & Indentation software reference guide** in section **Installation**. Otherwise if they are installed, first refer to the **Common Scratch & Indentation software reference guide** in section **Starting** which describes how to start the software.

## **1.2.4 GENERAL NOTE**

In this document the images may differ from the actual product.

The software print screen images that show V7.0.x or V8.0.x version are also applicable for V9.0.x version.

'Indentation head' means Indentation measuring head.

If not specified, the descriptions in this document concerns all following Indentation instruments of Anton Paar:

- Step MHT/MCT/NHT/UNHT/UNHT Bio, or UNHT HTV if <High temperature> radio button is selected in *My configuration* tab (section 2.1.1.2)
- TTX-NHT/UNHT/UNHT Bio

Otherwise each concerned platform/Table Top and/or Indentation head is mentioned.

'Step' means it is applicable for all platform generations/types. Otherwise the concerned platform(s) is specified (e.g. Step 700, STeP 4...)

If not specified, 'measurement(s)' means indentation measurement(s):

• for single measurement

OR

• for matrix of measurements.

'toolbar' means software main toolbar.

'key(s)' means keyboard key(s).

For troubleshooting see section 5.

#### **1.2.5** Typographical conventions

Refer to the same section in the *Common Scratch & Indentation software reference guide*.

#### **1.2.6** ABBREVIATIONS & SYMBOLS

Physical quantity symbols are according to the ISO 14577 standard.

| Abbreviations<br>Symbols | Designations                           | Units |
|--------------------------|--|-------|
| ADO                      | adjust depth offset                    |       |
| AFM                      | Atomic Force Microscope (imaging head) |       |
| Ap                       | projected contact area                 | m²    |
| Снм                      | creep Martens hardness                 | %     |
| CIT                      | indentation creep                      | %     |

| COS                  | Conscan confocal (imaging head)                            |          |
|----------------------|--|----------|
| .DCF / .dcf          | dynamic (sinus) calibration file                           |          |
| E*                   | plane strain modulus                                       | Pa       |
| <u> </u>             | indentation modulus  | Pa       |
| ε (Epsilon)          | geometric constant   |          |
| F <sub>r</sub>       | reduced modulus  | Pa       |
| <u> </u>             | Saxonian (SIO) Institute of Surface Mechanics              |          |
| .FDOP / .fdop        | Software file extension                                    |          |
| F                    | test force   | N        |
| <br>F <sub>max</sub> | maximum test force   | N        |
| h                    | indentation depth under applied test force                 | m        |
| hc                   | contact depth of the indenter with the sample at $F_{max}$ | m        |
| Ηπ                   | indentation hardness                                       | Pa       |
| HM                   | Martens hardness   | Pa       |
| h <sub>max</sub>     | maximum indentation depth                                  | m        |
| h                    | permanent indentation depth                                | m        |
| h <sub>r</sub>       | tangent indentation depth                                  | m        |
| HV                   | Vickers hardness   | Vickers  |
| HVIT                 | Vickers hardness calculated from H <sub>TT</sub>           | Vickers  |
|                      | Indentation Software configuration backup file             | Viciters |
| .INDBCK / .indbck    | extension  |          |
| .INI/.ini file       | initialization file  |          |
| m                    | power law exponent   |          |
|                      | Micro Combi Tester (measuring head)                        |          |
|                      | applicable for:  |          |
| MCT                  | MCT : first generation                                     |          |
|                      | MCT <sup>3</sup> : new generation                          |          |
|                      | MCT <sup>3</sup> V : Vacuum prepared new generation        |          |
|                      | Microindentation Tester (measuring head)                   |          |
| мцт                  | applicable for,  |          |
| וחוייו               | • MHI : first generation                                   |          |
|                      | • MHT <sup>3</sup> V: Vacuum propared new concration       |          |
| MIT / mit            | Indentation Software document file extension               |          |
|                      | Nanoindentation Tester (measuring head)                    |          |
|                      | applicable for:  |          |
| NHT                  | NHT <sup>2</sup> : second generation                       |          |
|                      | NHT <sup>3</sup> : new generation                          |          |
|                      | NHT <sup>3</sup> V : Vacuum prepared new generation        |          |
| v (Nu)               | Poisson's ratio  |          |
|                      | Coefficient of determination for the linear fit            |          |
| κΖ (Κ΄ Ζ)            | regression   |          |
| RIT                  | indentation relaxation                                     | %        |
| O&P                  | Oliver & Pharr   |          |
| S                    | contact stiffness  | N/m      |

|                    | Surface Testing Platform  |     |
|--------------------|---|-----|
|                    | applicable for new generation Step X00:   |     |
|                    | <pre>Step 100 : for 1 head + optional VID Step 300 : for 1 head + VID with air system Step 500 : up to 3 heads + VID with air system Step 700 : up to 3 heads + VID with air system and enclosure Step 700 Noise-Control : up to 3 heads + VID with active</pre>    |     |
|                    | electronic system and enclosure   |     |
| Sten               | also applicable for 1 <sup>st</sup> generation STeP:  |     |
| Step               | STeP 4: dedicated for 4 unitsSTeP 6: dedicated for 6 unitsSTeP 5 V: Vacuum Surface Testing Platform<br>dedicated for 5 units<br>(primary vacuum pump)STeP 5 HV: High Vacuum Surface Testing Platform<br>dedicated for 5 units<br>(primary & secondary vacuum pumps) |     |
|                    | it is also applicable for previous CPX/OPX Platform:  |     |
|                    | CPX Compact Platform (similar to STeP 4)<br>OPX Open Platform (similar to STeP 6)   |     |
| ттх                | Table Top<br>also applicable for Table Top Platform first generation  |     |
| UNHT               | Ultra Nanoindentation Tester (measuring head)applicable for:UNHT: 1st generationUNHT <sup>3</sup> : new generationUNHT <sup>3</sup> HTV: High Temperature new generationUNHT <sup>3</sup> V: Vacuum prepared new generation   |     |
| UNHT Bio           | Anton Paar Bioindenter (measuring head)   |     |
| VID                | optical video microscope (imaging head)   |     |
| $W_{elast}$        | elastic reverse deformation work of indentation   | N.m |
| $W_{plast}$        | plastic deformation work of indentation   | N.m |
| W <sub>total</sub> | total mechanical work of indentation  | N.m |
| $\eta_{IT}$        | elastic part of indentation work  | %   |

## **INFORMATION:**

1 N/mm<sup>2</sup> = 1 MPa 1 J = 1 N.m 1 Vickers = 1 kgf/mm<sup>2</sup>

## **2 MANAGING THE INSTRUMENT**

### **2.1 HARDWARE CONFIGURATION**

### **INFORMATION:** The tabs/features which are common for the Scratch and Indentation instruments are described in the **Common Scratch & Indentation reference guide** in section **Managing the instrument / Hardware configuration / Instrument configuration (common tabs)**.

To reach the following window (Fig.1) and to validate/save the current settings of its tabs, refer to the same document and section previously mentioned.

## 2.1.1 MY CONFIGURATION TAB

 STeP - NHT - Hardware configuration

 My configuration
 User channels
 Control unit & modules
 Motors
 Instrument adjustment
 Verification
 Ranges
 Dynamic r

Fig.1 Instrument - Hardware configuration window with tabs

## 2.1.1.1 Common (UNHT/NHT/MHT)

| Tosca | samp | le int | terfa | ce- |
|-------|------|--------|-------|-----|

Tosca sample interface

This feature is inactive; it is reserved for future use.

## 2.1.1.2 UNHT

| This inactive box is automatically checked to activate the $($ standard $)$ Sinus hardware present on the UNHT. |                              |   |  |  |  |  |
|---|------------------------------|---|--|--|--|--|
| Heating   | Select a following radio     | button:   |  |  |  |  |
| <ul> <li>No heating</li> <li>Heating stage</li> </ul>   | <no heating=""><br/>Or</no>  | To perform ambient measurements.  |  |  |  |  |
| ⊖ High temperature  | <heating stage=""></heating> | To perform measurements under elevated temperature with optional Peltier (100 °C) or 200 °C Heating Module; see section 3.11. |  |  |  |  |
|   | Or                           |   |  |  |  |  |
|   | <high temperature=""></high> | To perform measurements under high temperature with STeP 5 HV - UNHT HTV.   |  |  |  |  |

## 2.1.1.3 NHT

Heating

Although it is possible to perform the measurements on NHT under Heating stage Although it is possible to perform the measurements on NHT under elevated temperature, it is not recommended, because the thermal drift of the instrument can be too high. Therefore, it is advised to keep this box unchecked to perform measurements at ambient temperature. Module Sinus W Hardware generator
This box should be checked to activate the optional Sinus hardware present on the NHT; the corresponding software features become active. If this option is not present, this box should be unchecked.

## 2.1.1.4 MHT

| Head        | In almost all cases this box should be unchecked.<br>(It is only checked if the head is equipped with an <i>Electronic bridge</i> , which concerns only a few previous special MHT versions.) |
|-------------|---|
| Heating     | To perform measurements under elevated temperature with optional Peltier (100 °C) or 200 °C or 450 °C Heating Module, check this box and see section 3.11.                                    |
| Temperature | Uncheck (default) to perform measurements at ambient temperature.   |

## 2.1.2 VERIFICATION TAB

This tab allows setting parameters used for verifying the instrument and also defining a reminder when verifying the instrument.

**INFORMATION:** The verification only works with instrument equipped with Sinus mode option.

See section 2.2 for the instrument verification procedure and results.

| Step - NHT - Hardware configuration                                  |                        |                              |   |        |         |              |                 |        |           |     |
|--|------------------------|------------------------------|---|--------|---------|--------------|-----------------|--------|-----------|-----|
| My configuration   | User channels          | Control un                   | it & modules  | Motors | Instrum | ent adjustme | nt Verification | Ranges | Dynamic r | 4 F |
| h low limit 14<br>F/S <sup>2</sup> min 2(<br>F/S <sup>2</sup> max 1( | 48 🗭<br>00 💽           | ] nm<br>] nm²/mN<br>] nm²/mN | 148+3 -<br>168+3 -<br>148+3 | ē ē    | ē ē     | 2 2 Q        | ō ō             | ° o    | ē ē o     | Ā   |
| Reminder 90  | 0                      | Day                          | nm²/mN  |        | ,       |              |                 |        |           |     |
|  |                        |                              | nm  | 200    |         | 400          | 600             |        | 100       |     |
| C Rese   | et to <u>d</u> efaults |                              |   |        |         |              |                 |        |           |     |

Fig.2 Verification tab with (advised) default parameter settings for NHT

Passive graph (on the right side):

It shows the  $F/S^2$  curve which is constant with fused silica (SiO<sup>2</sup>) sample. The green frame zone of the graph is the tolerance of this constant.

*h low limit*,  $F/S^2$  *min* and  $F/S^2$  *max* fields:

To change the 3 default values corresponding to the instrument measuring head (see the following  $<\underline{R}$ eset to defaults> button), that are used to compute the verification results (section 2.2).

**IMPORTANT:** Only an expert user may change these 3 default values.

#### Reminder box:

To remind the user to perform a new verification procedure (section 2.2) on the instrument measuring head, check this box (default) and set its following field. Uncheck to not remind.

#### Reminder field:

To set the number of days (default 90) after which the user should be reminded (if the previous box is checked)  $\rightarrow$  After the defined number of days has elapsed, before starting a new measurement (*Indentation*  $\overline{\mathbf{x}}$  icon on toolbar, section 3.6), a *Confirmation* window recommends the user to perform again the verification procedure (section 2.2).

| Confirma | tion   | $\times$ |
|----------|--|----------|
| ?        | The instrument reached the verification date, an instrument verification is highly recommended. Do you want to continue measurement anyway ? |          |
|          | Yes No   |          |

#### <Yes> button:

To continue the current measurement(s).

#### <No> button:

To cancel the current measurement(s).

#### <Reset to <u>d</u>efault> button:

To set automatic default (advised) values for each parameter of the instrument current measuring head.

## 2.1.3 'RANGES' TAB(S)

**IMPORTANT:** Refer to the **Common Scratch & Indentation software reference guide** and carefully read section **Managing the instrument / Hardware configuration / 'Ranges' tab(s)**.

## 2.1.3.1 UNHT 'Ranges' tabs

UNHT has the 3 following '*Ranges*' tabs.

| er channels  | Control unit & modu | les Motors     | Instrument adjustment | Indenter ranges | Dynamic ranges | Reference ranges | 1 |
|--------------|---------------------|----------------|-----------------------|-----------------|----------------|------------------|---|
| identer Hard | ware coefficients   |                |                       |                 |                |                  |   |
| F- 6         |                     | It is not reco | ommended to change tr | lese values     |                |                  |   |
| FII SENSO    |                     |                | D2 Set                | 1501            |                |                  |   |
| ⊖ Calib      | ration v1           |                | 00                    | alibration v1   |                |                  |   |
| Fn           | coef (fine)         | Fn coef (lar   | ge)                   | DZ coef (fine)  | DZ co          | ef (large)       |   |
|              | mN/V                |                | mN/V                  | μη              | n/V            | 🗭 μm/V           |   |
| Calib        | ration v2           |                | () C                  | alibration v2   |                |                  |   |
| A.\          | /0 (fine)           | A.V0 (large    | )                     | A.V0 (fine)     | A.V0 (         | large)           |   |
|              | V 🗣                 |                | V                     | V               |                | V                |   |
| e0.          | S (fine)            | e0.S (large)   |                       | e0 (fine)       | e0 (lar        | ge)              |   |
|              | 🖨 mN                |                | 🖨 mN                  | 🖨 μn            | n              | 🗣 μm             |   |
| е0.          | S Slope (fine)      | e0.S Slope     | (large)               | e0 Slope (fine) | e0 Slo         | pe (large)       |   |
|              | 🖨 mN                |                | 🖨 mN                  | 🖨 μn            | n              | 🚔 μm             |   |
| С (          | (fine)              | C (large)      |                       | C (fine)        | C (larg        | je)              |   |
|              |                     |                | ▲<br>▼                |                 |                |                  |   |
| C            | Slope (fine)        | C Slope (la    | rge)                  | C slope (fine)  | C slop         | e (large)        |   |
|              |                     |                |                       | ▲<br>▼          |                |                  |   |
|              |                     |                |                       |                 |                |                  |   |
| + Spring     |                     | 6t             | + Frame coeffic       | cients          | lmport ca      | alibration       |   |
| Complia      | nce Head            | Tactor         | Frame comp            | ance            |                |                  |   |

Fig.3 UNHT <u>Indenter ranges</u> tab

| My configuration       User channels       Control unit & modules       Motors       Instrument adjustment       Indenter ranges       Dynamic ranges         Dynamic       + Command       + Dynamic coefficients  | onfiguration       User channels       Control unit & modules       Motors       Instrument adjustment       Indenter ranges       Dynamic ranges         bynamic         + Command         Fn coef (fine)         Image: mN/V         Fn coef (large)         Image: mN/V         Image: mN/V         Fn coef (large)         Image: mN/V   |
|---|--|
| Dynamic         + Command         Fn coef (fine)         Mobile Mass         mN/V         g         Click here to start dynamic calibration procedure         Spring Stiffness         Fn coef (large)         mN/V         mn/V | Aynamic         + Command         Fn coef (fine)         Image: mN/V   |
| + Command<br>Fn coef (fine)<br>Mobile Mass<br>Mobile Mass<br>g Click here to start dynamic calibration<br>procedure<br>Fn coef (large)<br>M/W<br>Damping Coef<br>N/m<br>Calibrate<br>N.s/m<br>See UNHT & NHT Dynamic calibration (Sinus),   | + Command<br>Fn coef (fine)<br>Mobile Mass<br>g Click here to start dynamic calibration<br>procedure<br>Fn coef (large)<br>mN/V<br>Damping Coef<br>N/m<br>Calibrate<br>N.s/m<br>See UNHT & NHT Dynamic calibration (Sinus),<br>p. 19   |
| Fn coef (fine)       Mobile Mass         Image: Mobile Mass       g       Click here to start dynamic calibration procedure         Fn coef (large)       N/m       Calibrate         Image: Mobile Mass       N.s/m       See UNHT & NHT Dynamic calibration (Sinus),  | Fn coef (fine)       Mobile Mass         mN/V       g       Click here to start dynamic calibration procedure         Fn coef (large)       N/m       Calibrate         mN/V       N/m       Calibrate         Damping Coef       N.s/m       See UNHT & NHT Dynamic calibration (Sinus), p. 19  |
| Implicit minipage       minipage       Click here to start dynamic calibration procedure         Fn coef (large)       Implicit minipage       N/m       Calibrate         Implicit minipage       N/m       Calibrate         Implicit minipage       N/m       Calibrate         Implicit minipage       N.s/m         See       UNHT & NHT Dynamic calibration (Sinus),  | Image: mn/V       Image: g       Click here to start dynamic calibration procedure         Fn coef (large)       Image: mn/V       Image: mn/V         Image: mn/V       Image: mn/V       Image: mn/V     < |
| Fn coef (large)<br>mN/V<br>Damping Coef<br>N/m<br>Calibrate<br>Calibrate<br>N.s/m<br>See <u>UNHT &amp; NHT Dynamic calibration (Sinus)</u> ,  | Fn coef (large)       N/m       Calibrate         mN/V       Damping Coef       Image: Calibration (Sinus), p. 19  |
| See <u>UNHT &amp; NHT Dynamic calibration (Sinus)</u> ,   | See <u>UNHT &amp; NHT Dynamic calibration (Sinus)</u> ,<br>p. 19   |
| p. 19   |  |

Fig.4 UNHT <u>Dynamic ranges</u> tab

| Jser channels | Control unit & modules                       | Motors                               | Instrument adjustment | Indenter ranges | Dynamic ranges | Reference ranges |  |
|---------------|--|--------------------------------------|-----------------------|-----------------|----------------|------------------|--|
| Reference     | Hardware coefficients                        | lt is no                             | t recommended to chan | ge these values |                |                  |  |
|               | + Measuremen<br>Fn coef (fin<br>Fn coef (lar | its<br>e)<br>♥ mN/\<br>ge)<br>♥ mN/' | /<br>/                |                 |                |                  |  |
|               |  |                                      |                       |                 | A              |                  |  |

Fig.5 UNHT <u>Reference Ranges</u> tab

<sup>&</sup>lt;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.

## 2.1.3.2 NHT 'Ranges' tabs

NHT has 2 'Ranges' tabs.



Fig.7 NHT Dynamic ranges tab

<sup>1)</sup> Select (1 time max.) the right cursor to display (fully) the last tab or select the left to display (again) the first 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).

|                                     | Digital Bridge                      |
|-------------------------------------|-------------------------------------|
| In <i>Ranges</i> tab (Fig.6), click | 🐐 🕴 Auto tune Dz bridge             |
|                                     | Bridge limits : H: 70.00% L: 30.00% |

The *Dz Bridge tuning* window appears (Fig.8).

| INFORMATION:               |          | Factory service | I          | a special area use  | ed for factory  |
|----------------------------|----------|-----------------|------------|---------------------|-----------------|
| setting (not dedica<br>it. | ated for | the user) is av | ailable bu | ut an access code i | s needed to use |

```
Click
```

Auto tune Dz bridge

to start the automatic tuning process.

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.8 Scanning completed

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

Click **v** ok to validate the *New limits* (overwrite the previous ones) in the software.

H70IB103EN-C

## In the Ranges tab (Fig.6),

Digital Bridge
Auto tune Dz bridge...
Bridge limits : H: 51.00% L: 31.00%

the new Dz bridge limits appear.

**INFORMATION:** *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.8

## UNHT & NHT Dynamic calibration (Sinus)

The *Dynamic coefficients* fields and <Calibrate> button are located in the *Dynamic ranges* tab of the UNHT (Fig.4) and NHT (Fig.7).

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

## **INFORMATION:**

## <u>With NHT</u>

If **Calibrate** is inactive, it means the optional Sinus hardware is not present (section 2.1.1.3) and this calibration cannot be performed.

| Motors  | Instrument adj   | ustment | Indenter ra | inges    | Dynamic ra             | anges          | Reference Rang |
|---------|------------------|---------|-------------|----------|------------------------|----------------|----------------|
| + Dynan | nic coefficients |         |             |          |                        |                |                |
|         | Mobile Mass      | g       | (           | Click he | ere to start o<br>proc | dynam<br>edure | ic calibration |
|         | Damping Coef     | N/m     |             | ļ        |                        | Calibra        | ite            |
|         | ۲                | N.s/m   |             |          |                        |                |                |
|         |                  |         |             |          |                        |                |                |
|         |                  |         |             |          | Val                    | idate r        | ange values    |

Fig.9 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 (section 3.4.2).

Then click **Calibrate** to open the following *UNHT* or *NHT Head Dynamic Calibration* window (Fig.10).

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



Fig.10 Head Dynamic Calibration window (e.g. NHT)



To load a dynamic calibration from a .DCF file format.

To save the current dynamic calibration into a .DCF file format.

To export the current dynamic calibration into a .TXT file format.

To stop the dynamic calibration when it is proceeding.

## **Only for NHT**

To set another indenter position for the calibration; for advanced user only.

To start the semi-automatic dynamic calibration procedure, which is described below.



If no ADO or non-successful ADO has been performed (section 3.4.2), it is recommended to perform one by clicking <Yes> button (section 3.4.4).

**INFORMATION:** Having a successful ADO  $\Re$  prior starting the dynamic calibration allows defining the indenter position to perform this calibration in the same condition as a measurement (that is to say the most correct one possible).

| Head position |  |
|---------------|--|
|               | This step requires the presence of a sample.<br>Positon the sample so the reference but not the<br>indenter is in contact with it.<br>Position control |
|               | Ok Cancel  |
| Head position |  |
|               | This step requires the presence of a sample.<br>Positon the sample so the reference but not the  |
| *             | indenter is in contact with it.  |
|               | Indenter is in contact with it.  |

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.



| <u>For UNHT</u> |
|-----------------|
| This addition   |

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.



 Indenter
 Reference

 Pre-approach
 Contact force

 24 ● %
 40 ● %
 500.00 ● μN

 C
 Reset to defaults
 ✓
 OK
 X Cancel

Contact force

50.00 😤 µN

150 🚔 µN/µm

Contact stiffness threshold

Then in the Head Dynamic Calibration window (Fig.10):

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

Adjust depth offset parameters

Surface detection parameters

Characterization force

500.00 厌 µN

25000.0 🖀 nm/min

Approach speed

| Positioning the indenter  | (The reference approaches for UNHT and) the indenter pre-approaches.   |
|---|--|
| Mass Calibration running  | Free oscillation analysis to determine the mobile mass and the damping coefficient: the curves in the 2 upper graph areas are displayed/updated.   |
| phase calibration running : 11.0 [hz  | Forced oscillation analysis on a broad frequency<br>range. It will compute the dynamic head response<br>(phase and gain): the frequency value increases<br>and the measurement curves are displayed in real<br>time in the <i>Results</i> bottom graph area. |
| Removing the indenter   | When the phase calibration measurement is completed, the indenter (and reference for UNHT) are retracted.  |
| Start Dynamic Calibration   | When this button becomes again active , the calibration is completed as follows:   |
| new<br>Freq = 57.990 [Hz]<br>K = 1557.572<br>Log = 9.761<br>Mass= 11.732 [g]<br>Damp= 0.228 [N.s/m]<br>Software version: 7.1.13<br>Calibration Date: 30.08.2016 | The new calibration values are updated on the top right corner of the window and all new curves are displayed accordingly in all graph areas.<br>Click • • • • • (approve).  |
| In the Dynamic range  | es tab (Fig.9):  |
| + Dynamic coefficients<br>Mobile Mass   | All <i>Dynamic coefficients</i> field values are automatically updated with the 3 new values (approved) of the previous dynamic calibration.   |
| Damping Coef  | Click Validate range values (validation of these new values).  |

## 2.1.3.3 MHT Ranges tab

MHT has only one Ranges tab.



Fig.11 MHT <u>Ranges</u> tab

## 2.1.4 INSTRUMENT ADJUSTMENT TAB

| My configuration User channels                                 | Control unit & modules   | Motors Instrument adjustm                    | ent Verification              | Ranges Dynamic r 🔸 🕨       |
|--|--|--|-------------------------------|----------------------------|
| PID control Approach Kp Approach Ki Depth Ki Depth Ki          | p Load Kp<br>i Load Ki   | Sinus Kp Sinus Fn H                          | Kp Sinus Dep                  | pth Kp<br>pth Ki<br>2.1.4. |
| PID debug mode (see docu                                       | imentation)  | Restore factore                              | ory settings                  |                            |
| Threshold  |  |  |                               |                            |
| [nm/s]   | TimeOut<br>[s]<br>settings   | ection<br>.1.4.2                             |                               |                            |
| ip Sinus Fn Kp Sinus<br>i Sinus Fn Kp Sinus                    | settings [s] S<br>settings INFO<br>contro  | RMATION: All S                               | inus <i>parar</i><br>/e with: | meters in PID              |
| (p<br>Sinus Fn Kp<br>Sinus Fn Kp<br>Sinus Fn Ki<br>Sinus Fn Ki | TimeOut  Settings  Is Depth Kp  Source Sourc | RMATION: All S<br>ol area are inactiv<br>MHT | inus <i>parar</i><br>/e with: | meters in PID              |

## 2.1.4.1 PID control area

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> button to restore the values.

|             | Depth    | Load    | Sinus<br>amplitude | Sinus<br>force     | Sinus<br>depth |
|-------------|----------|---------|--------------------|--------------------|----------------|
| Approach Kp | Depth Kp | Load Kp | Sinus Kp           | Sinus Fn Kp        | Sinus Depth Kp |
|             |          |         |                    |                    |                |
| Approach Ki | Depth Ki | Load Ki | Sinus Ki           | Sinus Fn Ki        | Sinus Depth Ki |
|             |          |         |                    |                    |                |
|             |          |         |                    | Restore factory se | ttings         |



Fig.12 Control loop schematic description

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

## 2.1.4.2 Cancel drift area (stabilization)

During the automatic ADO process (section 3.4.4.2) and during the indentation measurement process (section 3.8), a *Stabilization* step  $\blacksquare$  *Stabilization* 30 [s] (in *Status* area) is performed to wait for an acceptable thermal drift before performing an indentation.

| Cancel Drift |               |
|--------------|---------------|
| Threshold    | TimeOut       |
| [nm/s]       | ▲ [s]         |
| Restore fac  | tory settings |

The stabilization ends:

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

OR

after the elapsed set *TimeOut* field value.

Click <Restore factory settings> button to restore the factory values.

## 2.2 INSTRUMENT VERIFICATION

**INFORMATION:** The instrument verification is only possible with the Sinus mode, available with all UNHT and optional with NHT (the Hardware generator box should be check in section 2.1.1.3).



Select "Instrument > Instrument verification..." from menu bar.

Fig.13 Verification window - E.g. good NHT verification results

## 2.2.1 RESULTS

The parameters used to compute each verification result are defined in section 2.1.2

## Verification history list

To select (highlight) one of the verification measurement (files are created after each automatic measurement  $\rightarrow$  see following <Proceed to verification> button), in order to see its results in the following  $F/S^2$  graph and in *Results* area. The current/last verification is the one at the bottom of the list.

## *F/S*<sup>2</sup> graph

There are 3 ways to display the verification results in the graph,



## <u>Results</u> area

| Verification History<br>2019-02-28-17-09.mit<br>2019-03-01-13-24.mit<br>2019-05-28-07-06.mit | F/S2<br>~ 文 互<br>1.8E+3       |   |      |                  |                   |                   |
|--|-------------------------------|---|------|------------------|-------------------|-------------------|
|  | 1.6E+3                        | -<br>-<br>-<br>-<br>-<br>-<br>-<br>-<br>-<br>-<br>-<br>-<br>-<br>-<br>-<br>-<br>-<br>-<br>- | õ    | o ∳ o            | ō o o             | ° ° °             |
|  | nm²/mN                        |   | 400  |                  | 600               | <br>800           |
|  | x 1.0 ∨ Q Q                   | ≠ 🖊 <   |      |                  |                   | >                 |
|  | Result<br>Verification Date : | 28.02.2019 17:09:15   |      | hmin > 1.50E-007 | f/s2 min          | f/s2 max          |
|  | Status :                      | X   |      | Target           | 1350.00000 nm²/mN | 1649.99997 nm²/mN |
|  |                               | • •   |      | Verified         | 1453.18756 nm²/mN | 1673.75867 nm²/mN |
|  | Indenter :                    | Berkovich [B-V 74] (12.02.2   | 019) |                  |                   |                   |

Fig.14 E.g. Bad NHT verification results

*Verification date*: The date and time when the verification measurement has been performed are displayed and correspond to the selected measurement in the *Verification history* list, **except** if the file is renamed (it is possible but not advised; see following <Proceed to verification> button to access to the files).

*Status*: If the verification results are good (Fig.13), a green tick appears. If the verification results are bad, a red cross appears (Fig.14).

*Indenter*: The defined details of the indenter which has been used for the verification measurement are displayed.

*Results* table: The detailed values are summarized in this table. The bad *Verified* value(s) is highlighted in red and the good one(s) in green. As soon as one value is highlighted in red, the previous *Status* displays a red cross (bad result).

**IMPORTANT:** In case of a **current** bad verification results (the last measurement selected at the bottom of the list), it means there is a problem on the instrument. The problem should be solved in order to perform a new verification procedure until it is good. The problem(s) could be: The Indenter is not properly mounted on the measuring head (indenter holder), the indenter is dirty, worn out, the sample is not properly mounted (sample holder), the measuring head has a trouble or is bad calibrated (Ranges hardware coefficients are wrong)...



Fig.15 E.g Current (last) verification is good

#### File area

<Open files in explorer> button:

To open the software dedicated folder location where each verification measurement file is stored.

| _   🔄 🔄 ╤   NH | IT-Demo SN 01-01010         |                   |  |
|----------------|-----------------------------|-------------------|--|
| File Home      | Share View                  |                   |  |
| ← → • ↑ 📘      | ≪ Local Disk (C:) → Program | nData → Anton Paa | r > Indentation > instrument verification > NHT-Demo SN 01-01010 |
| 🛃 Quick access | Name                        | Date modified     | Туре   |
| A Quick access | 🔁 2019-02-28-17-09.mit      | 06.03.2019 17:02  | Indentation Document   |
| 💻 This PC      | 🔁 2019-02-29-13-24.mit      | 09.07.2019 15:41  | Indentation Document   |
| 💣 Network      | 🔁 2019-05-28-07-06.mit      | 28.05.2019 08:05  | Indentation Document   |

## 2.2.2 PROCEDURE

It is advised to verify the measuring head at least every 90 days (section 2.1.2 under *Reminder* field).

| 1- Define verification location  |
|--|
| + Install a SiO2 sample in the sample holder   |
| <ul> <li>Target a suitable location on the sample,<br/>using position control</li> </ul> |
| Position control   |
| 2 - Proceed Verification   |
| + The sample will be marked by the indenter  |
| Proceed to verification  |

Prior to using the following buttons, the provided fused silica (SiO<sup>2</sup>) sample should be properly mounted on the instrument sample holder; refer the corresponding Indentation head **user manual** or **reference guide**.

### 1- Define verification location area

<Position control...> button:

To open the *Position control* window<sup>1</sup> in order to target a suitable location on the fused silica ( $SiO^2$ ) sample, where to start the verification measurement (see following button).

#### 2- Proceed verification area

<Proceed to verification > button:

To start the automatic measurement for the verification. The measurement file is created in the software and appears in the *Verification history* list.

<sup>1</sup> Refer to the Common Scratch & Indentation software reference guide in section Managing the instrument / Position control.

### To display ISO 14577 nomenclature

Select "File > Options..." from menu bar and then Preferences tab.



## **INFORMATION:**

The feature below is applicable with Marten hardness, Tangent and e.g. Oliver & Pharr analysis methods (section

Check ISO 14577 Nomenclature to display the analysis results in ISO 14577 format (Fig.17).

Fig.16 Preferences tab in Options window



In the analysis result area (Fig.84) the Main results and Additional results values 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.17 ISO 14577 nomenclature

See also:

- section 1.2.6 (Abbreviations & symbols)
- section 6 (Software formulas)

**INFORMATION:** ISO 14577 Nomenclature unchecked in Fig. 16 is the default format.

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

## 2.4 CALIBRATION

**INFORMATION:** The Distance and Center tabs which are common for the Scratch and Indentation instruments are described in the **Common Scratch & Indentation software reference guide** in section **Managing the instrument / Calibration** (common tabs).

To reach the following window (Fig.18) and to validate/save the current settings of its tabs, refer to the same document and section previously mentioned.

STeP - UNHT - Calibration Sensor ranges Distance Platform Center Default Values

#### Fig.18 Instrument (e.g. Step - UNHT) - Calibration window with tabs

See the following sections which described the detailed parameters of each tab concerning only the Indentation heads.

**INFORMATION:** Sensor ranges *and* Default values *tabs are available only with UNHT/Bio.* Platform *tab is not available for TTX-NHT.* 

## 2.4.1 SENSOR RANGES TAB (UNHT/BIO)

| STeP - UNHT - ( | Calibration |          |           |                |               |
|-----------------|-------------|----------|-----------|----------------|---------------|
| Sensor ranges   | Distance    | Platform | Center    | Default Values |               |
| Indentation     | range       |          |           |                |               |
| + Reference     | load range  |          | + Indente | r load range   | + Depth range |
| ) 10 n          | nN          |          | (         | 0 10 mN        |               |
| ◯ 50 r          | nN          |          | (         | ) 50 mN        | 🔿 50 μm       |
|                 |             |          |           |                | 1             |

Fig.19 E.g. Step - UNHT with fine ranges

**INFORMATION:** For UNHT Bio there is no reference. Therefore the + Reference load range radio buttons are inactive.

*The UNHT Bio* + Indenter load range *and* + Depth range *values are different than the UNHT.* 



This tab is also available in the UNHT/Bio *Hardware parameters* window; for the detailed descriptions, see respectively sections 3.7.6.3 and 3.7.6.4.

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

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

| STeP - MHT - Calibration                                   |  |  |
|--|--|--|
| Distance Platform Center                                   |  |  |
| Approach specification<br>Table Z retraction<br>1.500 💭 mm |  |  |
| Auto return to safe position                               |  |  |

Fig.20 E.g. Step - MHT

Table Z retraction field:

## NOTICE

## Risk of collision and damage

To set a value at which the motorized Z table automatically returns (lowered) to its rest position after it has been raised (e.g. when the position control is closed after a focus adjustment, or after a measurement...)

To save time, the field value 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).

**INFORMATION:** 1 mm is the initial value and 0.5 mm is the min. value. The unit of the field is defined in the options (e.g. mm).

#### Auto return to safe position box:

To automatically return to the safe position at the end of each measurement, check this box  $\rightarrow$  The motorized tables/sample move to the extreme right front position. This is the same position as clicking  $\triangleq$  on the main toolbar. Uncheck to stay where the sample was before running the measurement(s).

## 2.4.3 DEFAULT VALUES TAB (UNHT/BIO)

| STeP - UNHT - Calibration (Same param  | eters for TTX)              |
|--|-----------------------------|
| Sensor ranges Distance Platform Center | Default values              |
| Indenter parameters                    | Reference parameters        |
| Default pre-approach                   | Default pre-approach        |
| 10 🗬 %                                 | 40 💓 %                      |
|  |                             |
|  |                             |
|  | C Reset to <u>d</u> efaults |

Fig.21 E.g. Step - UNHT with both default values

| STeP - UNHT Bio - Calibra | tion (Same par  | ameters for TTX)            |
|---------------------------|-----------------|-----------------------------|
| Sensor ranges Distance    | Platform Center | Default values              |
| Indenter parameters       |                 | Reference parameters        |
| Default pre-approach      |                 | Default pre-approach        |
| 5                         | %               | 100 🖉 %                     |
|                           |                 |                             |
|                           |                 |                             |
|                           |                 | C Reset to <u>d</u> efaults |

Fig.22 Step - UNHT Bio with indenter default value

## Indenter parameter area

#### Default pre-approach field:

To define a value for the **indenter** pre-approach used in the 3 presets of the hardware parameters (section 3.7.6.11).

#### Reference parameter area

*Default pre-approach* field:

To define a value for the **reference** pre-approach used in the 3 presets of the hardware parameters (section 3.7.6.11).

**INFORMATION:** For UNHT Bio there is no reference. Therefore the related Default pre-approach field is inactive.

<Reset to <u>d</u>efault> button:

To set the default values in the fields  $\rightarrow$ 

UNHT: Indenter 10 % and reference 40 % (Fig.21)

UNHT Bio: Indenter 5 % (Fig.22)

## **2.5 MANAGING THE INDENTERS**

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



Select **"Instrument > Indenters... "** from menu bar.

#### **Edit indenters**



Fig.23 Edit indenter windows



To create a new indenter in the <u>Configured indenters</u> list.

| Vickers                    |         |  |  |
|----------------------------|---------|--|--|
| Berkovich                  | <u></u> |  |  |
| Cube Corner 4<br>Spherical | 5       |  |  |
| Knoop                      |         |  |  |
| Flat Punch                 |         |  |  |

Select (double click) the type of the indenter which is used (section 2.5.1).

Fig.24 Define a new indenter window



To modify the selected (highlighted) indenter in the <u>*Configured indenters*</u> list (section 2.5.1).

#### Before running a measurement



**IMPORTANT:** If there are several <u>C</u>onfigured indenters in the list, do not forget to select the Indenter in <u>use with the last calibration date if</u> available (the calibration date in section 2.5.3, 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 (section 2.5.2).

|   | Remove |
|---|--------|
|   |        |
| 1 | XPress |

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

Not applicable.

## Import/export



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

This window allows:

| xport a indenter file 🛛 🕹 🕹 🕹 🕹 🕹 🕹 🕹 🕹 🕹 🕹 🕹 🕹 🕹                      | <ul> <li>Choosing a location<br/>where to save the<br/>indenter file.</li> </ul>                    |
|--|---|
| Arganize ▼ New folder  | <ul> <li>Modifying the default File<br/>name corresponding to<br/>the selected indenter.</li> </ul> |
| E PC   | - Changing of the default<br>V6 .IND file format.   |
| File name: Berkovich B-Q 03/ind  | e Folders Indenter file V5, V4, V3 (Vand)   |
| Save as type:     Indenter file Vo (*.ind)       Hide Folders     Save |   |

> Import

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 \_\_\_\_\_\_ 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 section 4.12.



- in the printed/PDF report (from page 2 and depending on the report option configuration); refer to the *Common Scratch & Indentation software reference guide* in sections:
  - Customizing options / Document models tab
     Printing/PDF Document Reports

| nt model Groups S<br>te document <a>All&gt;</a>   | cratch<br>All>   |  | • 74  |
|---|--|--|---|
| Group name  |  | Group name   |   |
| Indentation # 1   |  | Indentation # 1  |   |
| Indentation parameters  |  | Indentation parameters   |   |
| + Standard<br>Acquisition Rate : 10.0 [Hz]<br>Linear Loading<br>Max load : 10.00 mN<br>Loading rate : 20.00 mN/min<br>Unloading rate : 20.00 mN/min<br>Pause : 10.0 s<br>+ UNHT S/N: 50-00705 settings<br>En contact - 602 mN | App<br>App<br>Retr<br>Dz s<br>Loai<br>FnR<br>Stiff<br>Date :<br>Hour | + Standard<br>Acquisition Rate : 10.0 [Hz]<br>Linear Loading<br>Max load : 10.00 mN<br>Loading rate : 20.00 mN/min<br>Unloading rate : 20.00 mN/min<br>Pause : 10.0 s<br>+ UNHT S/N: 50-00705 settings | Ar<br>Ar<br>Re<br>Dz<br>Lo<br>Fn<br>Sti<br>Date |
| ndenters  |  | Indenters  |   |
| Type : Berkovich<br>Serial number : B-Q 03<br>Material : Diamond  |  | Type : Berkovich<br>Serial number : B-Q 03<br>Material : Diamond   | >   |
#### 2.5.1 INDENTER PROPERTIES WINDOW

| + Add | ] or 📝 | Edit | is clicked in the <i>Edit indenter</i> windows (Fig.23). |
|-------|--------|------|--|
|-------|--------|------|--|



Fig.25 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.

**INFORMATION:** *The initial* Beta *value corresponds to the indenter type selected in Define a new indenter window (Fig.24).* 

**INFORMATION:** *The initial* <u>P</u>oisson's ratio + <u>Y</u>oung's modulus *values correspond to the diamond material.* 

If Spherical or Flat Punch indenter type is selected in *Define a new indenter* window (Fig.24) these 2 additional fields appear.

\* indicated on the indenter provided certificate



To (re)calibrate the indenter (section 2.5.2).

#### 2.5.2 INDENTER CALIBRATION (QUASISTATIC/SINUS)

**Calibrate** is clicked in the *Indenter properties* window (Fig.25). The calibration procedure starts.

#### 2.5.2.1 Measuring a matrix of indentations

**INFORMATION:** Use a certified Fused silica sample with UNHT and NHT, or a certified BK7 sample with MHT.

**INFORMATION:** Sinus calibration is not available ( Measuring a new matrix of sinus indentation inactive) for MHT and UNHT Bio, and NHT without the optional sinus module/generator (section 2.1.1.3).

| Automatic calibration of the indenter tip / Step 1   |   |  |  |  |  |
|--|---|--|--|--|--|
| 1. Prepare the automatic indenter tip calibration  |   |  |  |  |  |
| + Choose your procedure  |   |  |  |  |  |
| <ul> <li>Loading indentations from file</li> <li>Loading sinus indentations from file</li> <li>Measuring a new matrix of indentation</li> <li>Measuring a new matrix of sinus indentation</li> </ul>   |   |  |  |  |  |
| <ul> <li>+ This procedure allows you to automatically calibrate an indenter. It is recommended that it is done everytime you change the indenter.</li> <li>+ The calibration is used to take in account the shape of the indenter tip.</li> <li>+ Ensure that the indenter is correctly installed in the head.</li> <li>+ Put the test sample under the instrument head.</li> <li>+ Click "Next" to follow the automatic calibration procedure.</li> </ul> |   |  |  |  |  |
| X Cancel   | ĺ |  |  |  |  |

Fig.26 Choose a calibration procedure

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

#### For MHT only

If the ADO is not yet successful  $\frac{1}{100}$  (section 3.4.2):

| Confirmat | ion   | Х |
|-----------|---|---|
| ?         | A new depth offset in FINE Dz range is needed, to adjust it and continue<br>calibration press "OK"<br>To abort press "CANCEL" |   |
|           | OK Cancel   |   |

Click <OK> button to start a new ADO.

**IMPORTANT**: The **fine** Dz range <100 μm> radio button should be selected in ADO parameters window (section 3.4.7.1). Otherwise if the current ADO is successful  $\Re$  (section 3.4.2):



If the sample has already been moved with a min. displacement from the current ADO indent (similar sample topography), click <No> button to keep this current ADO.

OR

If the sample has not been moved or has been moved too much from the current ADO indent (sample topography may vary), click <Yes> button to start a new ADO **IMPORTANT**: The **fine** Dz range <100  $\mu$ m> radio button should be selected in ADO parameters window (section 3.4.7.1).

| Automatic calibration of the indenter / Step 2                            |  |  |  |  |  |  |
|---|--|--|--|--|--|--|
| 2. Choose calibration parameters  |  |  |  |  |  |  |
| + Enter sample certificate value  |  |  |  |  |  |  |
| Plane strain modulus (E*)   |  |  |  |  |  |  |
| + Or enter theorical values for your sample                               |  |  |  |  |  |  |
| Poisson's ratio Young modulus <ul> <li>EIT Method</li> <li>GPa</li> </ul> |  |  |  |  |  |  |
| + Choose the calibration mode :   |  |  |  |  |  |  |
| Oliver and Pharr mode   |  |  |  |  |  |  |
| Quick (40 indentations at 8 different loads)                              |  |  |  |  |  |  |
| Intermediate (45 indentations at 9 different loads)                       |  |  |  |  |  |  |
| Intensive (60 indentations at 12 different loads)                         |  |  |  |  |  |  |
| O User protocol file  |  |  |  |  |  |  |
|   |  |  |  |  |  |  |
| X Cancel  |  |  |  |  |  |  |

Fig.27 Calibration parameters (e.g. quasistatic UNHT)

 Plane strain modulus (E\*)

 Image: Base of the strain modulus (E\*)

 Image: Base of the strain modulus (E\*)

 Image: Base of the strain modulus (E\*)

Select O E\*Method and set the *Plane strain* modulus (E\*) field value noted on the certificate provided with the certified used sample. Then press <Enter> key.



|              | 3. Starting indentation  |   |
|--------------|--|---|
|              | + The indentations are ready to run.<br>+ Press "Next" to start. |   |
|              | Cancel   | ► |
| Click Next M |  |   |

Select a quasistatic calibration mode.

**INFORMATION:** The number of the indentations and loads for the matrix of measurements are different for each measuring head.

#### <u>Wait time</u>

| it Time          |     |      |     |     |     |     |        |
|------------------|-----|------|-----|-----|-----|-----|--------|
| Calibration Time |     |      |     |     |     |     |        |
| <                |     | July | /   |     |     | >   |        |
| Mon              | Tue | Wed  | Thu | Fri | Sat | Sun | Hour   |
|                  | 1   | 2    | 3   | 4   | 5   | 6   | 21     |
| 7                | 8   | 9    | 10  | 11  | 12  | 13  |        |
| 14               | 15  | 16   | 17  | 18  | 19  | 20  |        |
| 21               | 22  | 23   | 24  | 25  | 26  | 27  | Minute |
| 28               | 29  | 30   | 31  |     |     |     | 30     |
|                  |     |      |     |     |     |     |        |
| Waiting          |     |      |     |     |     |     |        |
| 🔆 Wait           |     |      |     |     |     |     |        |

#### Set a delayed starting

| 24 | 25 |
|----|----|
| 31 | 3  |

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



Wait

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 measurements start is displayed Waiting ... 4 H 27 min 25 sec

OR

#### Direct starting



To start the automatic matrix of measurements (even if  $\bigcirc$  wait has been clicked).

#### Then:

- Only for UNHT/Bio or NHT, first the *ADO in progress* window appears (see descriptions from Fig.38). For these instruments, anyway an ADO is automatically performed before running the calibration matrix of measurements. There is no need to set any ADO parameters, they are automatic (transparent) and optimized for this calibration.
- Just after, *Indentation running...* window (same as Fig.76) appears; wait for all automatic matrix of measurements are performed. Once the last measurement is completed, the following window appears and allows:



Then, the following window appears.

- Choosing a location where to save the calibration measurement 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

#### **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.28 E.g. Indent #3/12 is excluded (non-relevant)



To select next or previous load batch.

For Sinus, no load to select.





To display only the relevant indents of the **current** load; see <u>Keep only the relevant indents</u>, p. 44:

The non-relevant ( $\Box$  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 reference guide* in section *Measurement Documents in Curve view / Document window interface / Graph area tools / Zoom*.

When all contact points and curves are verified, click  $\bowtie$  :

**IMPORTANT:** 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).

| s | aving file in progress |
|---|------------------------|
|   | 100%                   |
|   |                        |

The following window appears.

log

 $\boldsymbol{\sim}$ 

#### Validation of the results

According to the previous <u>Contact area determination</u>, p. 43, the red curve fits on the crosses displayed on the graph of the window Fig.29:

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

Select <sup>(e)</sup> Log to display graph logarithm scales, OR select (initial) <sup>(e)</sup> Linear to display graph linear scales.

Verify the curve and if necessary click Merevious and modify the <u>Contact area</u> <u>determination</u>, p. 43.

When the curve is suitable, click Einish and see sections:

- 2.5.3 (Calibration date)
- 2.5.4 (Fit methods  $A_P(h_c)$  of the calibration)

## 2.5.2.2 Loading indentations from file

This procedure is similar than for Measuring a matrix of indentations described in section 2.5.2.1, **exceptions**:

- There are no parts related to perform measurements.
- Select 
  Loading indentations from file **OR** 
  Loading sinus indentations from file in Fig.26 (chose a calibration procedure in the Step 1 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.

### 2.5.3 CALIBRATION DATE

**Einish** is clicked after a calibration (Fig.29) or **Edit** is clicked in the *Edit indenter* windows (Fig.23) for an indenter already calibrated.

The *Curve results of indenter contact area (Ap) vs. contact depth (hc)* from Fig.29 is reported on the graph of the window below.



Fig.30 Indenter properties window (with calibration/date)

Calibration

Date 25.07.2014

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.23);

## e.g. **Berkovich [B-Q 03] (25.07.2014)**

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

## 2.5.4 FIT METHODS A<sub>P</sub>(h<sub>c</sub>) OF THE CALIBRATION

**Einish** is clicked after a calibration (Fig.29) or **Edit** is clicked in the *Edit indenter* windows (Fig.23) for an indenter already calibrated.

The *Curve results of indenter contact area (Ap) vs.* contact depth from Fig.29 is reported on the graph of the window below.



Fig.31 Indenter properties window Ap(hc) curve

#### Graph scale display modes

Also applicable in the following Edit Fit Method window (Fig.32).

A<sub>P</sub>

To display graph linear scales, in order to focus on the highest indentation depths: **red** and **blue** part of the curve (e.g. in Fig.31).

OR

log

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.32.)

## Part of the curve in blue (Fig.31)

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.32)

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<sup>st</sup> cross (lowest depth) of the calibration.

## Part of the curve in red (Fig.31 and Fig.32)

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

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: 
Coefficients



Fig.32 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.  $hc^{1/8}$   $\blacksquare$   $\blacksquare$ 

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

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

**IMPORTANT:** 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).

**INFORMATION:** ';' 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  | o open the <i>UNHT.INI</i>    |
|-----|-------------------------------|
| fil | e in the Windows <sup>®</sup> |
| N   | otepad:                       |
| Ν   | otepad:                       |

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

 $\rightarrow$ 



- 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.

|                           | ► OS (C:) ► ProgramData ► A | nton Paar 🕨 In | dentation 🕨 🕓          |  |  |  |  |
|---------------------------|-----------------------------|----------------|------------------------|--|--|--|--|
| File Edit View Tools Help |                             |                |                        |  |  |  |  |
| Organize 🔻 🤎              | Open 🔻 Print Burn           | New folder     |                        |  |  |  |  |
| 🔶 Favorites               | Name                        | Size           | Туре                   |  |  |  |  |
|                           | 퉬 Sample                    |                | File folder            |  |  |  |  |
| 📄 Libraries               | IndentationInstrument.cfg   | 8 KB           | CFG File               |  |  |  |  |
|                           | IndentationSoft.cfg         | 82 KB          | CFG File               |  |  |  |  |
| pe PC                     | Indentors.cfg               | 1 KB           | CFG File               |  |  |  |  |
|                           |                             | 1 KB           | Configuration settings |  |  |  |  |
| 📬 Network                 | 45                          |                |                        |  |  |  |  |
|                           |                             |                |                        |  |  |  |  |

| 🔲 UI | NHT.IN | II - Notep |      | x    |   |
|------|--------|------------|------|------|---|
| File | Edit   | Format     | View | Help |   |
| 1    |        |            |      |      | ~ |

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

Select "File > Exit" from menu bar or click and on the right top of the main window and start again the software by double clicking and 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.

| UNHT Approach Monitor   |   |  |  |  |  |  |
|---|---|--|--|--|--|--|
| Indenter Com<br>37.09 %<br>FnCommand: 12424<br>○ Servo On<br>○ Large Range<br>Fin<br>Bridge Adjust<br>50%<br>● Fine Range<br>20008.588 µN | nmand<br>IS 860 µN<br>Dz<br>Bridge Adjust<br>61%<br>● Fine Range<br>3352.467 nm | nce Command<br>67.00 %<br>and: 500.000 μN<br>REF<br>Bridge Adjust<br>49%<br>• Fine Range<br>497.368 μN |  |  |  |  |
| Status  | Quick Z Approach  | Reference Approach   |  |  |  |  |
| HW Sinus Active     Sinus Frequency : 0.000 Hz     Sinus Amplitude : 0.000 µN   | 🗌 Slow Ζ Approach<br>Ζ Table: 11426.776 μm                                      |  |  |  |  |  |
|   | AdjustFnRefToLoad Done  |  |  |  |  |  |

Fig.33 UNHT Approach Monitor window





Thanks to the <u>INI file activation</u>, p. 52:

Select "Instrument > .INI file > Edit.INI file" from menu bar to open the Notepad .INI file.

The UNHT Approach Monitor window (Fig.33) 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 (Fig.33) 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.





## **3 TAKING A NEW MEASUREMENT**

## **3.1** INTRODUCTION

For detailed manipulations and instructions, refer to each corresponding Indentation head **user manual** or **reference guide**.

#### 3.1.1 WARNING

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

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

#### 3.1.2 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.

#### **3.1.3 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 sections.

#### **3.2 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.

#### **3.3** 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 reference guide* in section *Managing the instrument / Position control.* 

**IMPORTANT:** Do not change the focus once it has been adjusted. To avoid any collision, always verify the sample height before moving.

- 1. To choose the sample measurement area, click  $\bigoplus$  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)**, otherwise click **(under microscope icon)**. The *Video* Software window is open, otherwise click **(Open video**)

For the video microscope module with Step (not with STeP 5): To lower the module (if not already at the lowest position) click  $\blacksquare$ .

3. To adjust the focus of the sample surface on the Video screen, move the

motorized Z table: click  $\uparrow$  to raise /  $\oint_{Down}$  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  $\rightarrow$  / / /



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 ( $\frac{1}{1000}$ ).

## **3.4** Adjusting depth offset (ADO)

## 3.4.1 Аім

The ADO is a procedure carried out to correctly setup the depth sensor measurement range (offset of the contact point inside the measurement range) for the next indentation measurement(s) (from an indenter calibration or a measurement type):

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

Reasons for which the ADO should be performed:

- After restarting the software.
- If the sample surface topography may vary between the measurements.
- If the 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.

## 3.4.2 ADO ICON STATUS



An arrow over the *ADO* icon displayed on toolbar means no or not successful ADO has been performed. Therefore a (new) procedure should be started (section 3.4.3).

## Fig.34 No/Not successful ADO icon

When the last (current) ADO has been successfully performed, a green disk with a tick appears over the *ADO* icon on toolbar. Anyway, for any reasons described in the previous section 3.4.1, a new procedure should be started (section 3.4.3).

## ADO

## Fig.35 Successful ADO icon

## **3.4.3 ADO** STARTING

## 3.4.3.1 Manual

Before starting the ADO, use the *Position control* to ensure that the current location on the sample surface is free of previous indents and clean; refer to the *Common Scratch & Indentation software reference guide* in section *Managing the instrument / Position control*.

To start the ADO (section 3.4.4), select **"Instrument > Adjust depth offset..."** from menu bar or click ADO  $\frac{1}{100}$  /  $\frac{2}{100}$  icon on toolbar.

## 3.4.3.2 Automatic

If the current ADO is not successful  $\overset{*}{\underset{\text{dot}}}$  (section 3.4.2), it can be started from a measurement type. The following window appears to set an offset positon (from current position) and the ADO parameters before performing the automatic process.

| Insert an adjust depth offset  |   |  |  |
|--|---|--|--|
| Insert an adjust depth offset with the following position and parameters   |   |  |  |
| Edit adjust depth offset paramete  | ers and position  |  |  |
| Delta X<br>-100.000 €<br>Delta Y<br>-100.000 €<br>Indentation count : 1<br>dimensions in X<br>0.000 µm to 0.000 µm | Characterization Force : 25 mN<br>Approach distance : 1500 nm<br>Approach speed : 100000 nm/min<br>Dz sensor in fine range<br>Stiffness Threshold : 500 µN/µm |  |  |
| dimensions in Y<br>0.000 µm to 0.000 µm  | Edit <u>a</u> djust depth offset parameters   |  |  |
|  | OK X Cancel   |  |  |

Fig.36 Insert an ADO offset window

Delta X and Delta Y fields:

To set the ADO relative position values from the 1<sup>st</sup> point of the matrix. **IMPORTANT:** *This location on the sample surface should be free of previous indents and clean.* 

<Edit <u>a</u>djust depth offset parameters> button:

To set the ADO parameters (section 3.4.4.1).

<OK> button:

To start the ADO automatic process (section 3.4.4.2).

## **3.4.3.3** From indenter calibration or matrix measurement types

The ADO can also be started from:

- An indenter calibration; for details see section 2.5.2.

or

From a matrix measurement type; for details see sections 3.7.5.8 (*Simple matrix*), 3.7.5.10 (*Quick matrix*), or 3.7.5.12 (Multi-ADO for Advanced matrix 3.7.5.9 or Visual advanced matrix 3.7.5.11)

## **3.4.4 ADO** GENERIC PROCEDURE (UP TO 3 WINDOWS)

The ADO is composed by 2 or 3 windows (depends from where the ADO is started) described as follows.

## 3.4.4.1 ADO parameters 1<sup>st</sup> window

The *Adjust depth offset parameters* window allows setting the parameters which are used to perform the ADO automatic process.

| Adjust depth | offset parameters  |                             |        |
|--------------|--|-----------------------------|--------|
| <b>•</b>     | Surface detection parameters<br>Approach <u>speed</u><br>100000.d 💭 nm/min |                             |        |
|              | Characterization force   | Contact stiffness threshold |        |
|              | Presets<br>Default   | ~                           | G      |
|              |  | ✓ ОК 🗙                      | Cancel |

Fig.37 ADO window (e.g. NHT with default presets)

Surface detection parameters area:

The fields are different depending on the current type of the measuring head; they are described in details in sections 3.4.5.1 (UNHT/Bio), 3.4.6.1(NHT) and 3.4.7.1 (MHT).

#### "Presets" drop-down menu:

To select one of the following presets according to the material of the current sample.

| Presets              |   |   |
|----------------------|---|---|
| Default              | ~ |   |
| Default              |   | _ |
| Soft materials       | N |   |
| Ultra soft materials | 6 |   |

#### "Default"

For sample with hard material coating.

#### "Soft materials"

For sample with soft material coating.

#### "Ultra soft materials"

For sample with ultra-soft material coating.

## <'Apply'> 🧲 button:

To apply the current **"Presets"** drop-down menu selection  $\rightarrow$  The field values previously described are automatically set (updated) accordingly. If the material matches with the selection, the parameters are suitable to perform a successful ADO.

#### <OK> button:

To valid the current settings. And also to run the following automatic process (section 3.4.4.2), only if the ADO has been started:

- Manually (section 3.4.3.1)

OR

- From the indenter calibration, only for MHT (section 2.5.2).

## 3.4.4.2 ADO in progress 2<sup>nd</sup> window

The *Adjust depth offset in progress* window is performing the automatic (semiautomatic for MHT without electronic bridge) process sequences using the parameters previously described (section 3.4.4.1) - An indentation is performed.



Fig.38 Minimized or developed Adjust depth offset in progress window

This window features are described as follows. The real-time sequences are different depending on the current type of the measuring head; they are detailed in sections 3.4.5.2 (UNHT/Bio), 3.4.6.2 (NHT) and 3.4.7.2(MHT).

#### Parameters area

The parameters which have been set in *Adjust depth offset parameters* window (Fig.37) are summarized.

#### <Cancel> button:

If necessary, to stop the process and cancel the ADO  $\rightarrow$  not successful ADO  $\stackrel{*}{\downarrow}$  icon is displayed (section 3.4.2). Otherwise wait until the end of the process.

+ in *More Info* area (top of window):

To develop the window for more information, such as *Command*, *Bridges*, *Additional information* and *graph* areas.

- in *Less Info* area to minimize the window (default).

Close window automatically box:

In order to automatically close the window after the process is completed, check this box (default).

Uncheck this box so that the window is not closed automatically  $\rightarrow$  OK button appears to manually close the window.

|                            | ~        |  |  |
|----------------------------|----------|--|--|
| Close window automatically |          |  |  |
| 🗸 ОК                       | X Cancel |  |  |

When the window is closed (Fig.38), the user can verify that the current ADO is successful  $\Re$  (section 3.4.2). If not  $\frac{1}{80}$  the procedure should be performed again until it is successful.

## 3.4.4.3 Sample displacement after ADO 3<sup>rd</sup> window

**INFORMATION:** This section is not applicable if the ADO has been started from a matrix measurement type or from the indenter calibration (also a matrix), because the ADO indent is then anyway shifted (part of the matrix - Delta X/Y).

When the *ADO in progress* window (Fig.38) is closed, the following *Sample displacement after ADO* window appears.

**IMPORTANT:** To avoid performing the next 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).



Fig.39 Sample displacement after ADO window

#### X motorized table field:

To set another min. displacement value<sup>1</sup> for the motorized X table.

#### Y motorized table field:

If necessary, to set a min. displacement value<sup>1</sup> for the motorized Y table.

<OK> button:

To move the motorized table(s) to the position values set in the fields described above; wait.



<Cancel> button:

To cancel the displacement (no move). However it is recommended to then use the *Position control* window to move the sample into another suitable location; refer to the *Common Scratch & Indentation software reference guide* in section *Managing the instrument / Position control*.

<sup>1</sup> It depends on the current sample material: Approx. 20 x h<sub>m</sub>

Pay attention to the unit (e.g µm in Fig.39), which is defined in the options; refer to the **Common Scratch & Indentation software reference guide** in section **Customizing options / Preferences tab (units)**.

#### 3.4.5 UNHT/UNHT BIO PARAMETERS AND SEQUENCES

| Adjust depth | offset parameters   |                           |                            |                    |                |
|--------------|---|---------------------------|----------------------------|--------------------|----------------|
| <b>•</b>     | Surface detection paramete<br>Approach <u>s</u> peed<br>25000.0 | rs<br>Co<br>in            | ontact force<br>30.00 룾    | μN                 | Quick Mode     |
|              | Characterization force<br>500.00 € µN                           | Co                        | ntact stiffness<br>150.0 🗲 | threshold          | 1              |
|              | Indenter<br>Pre-approach  | Reference<br>Pre-approach | C                          | ontact for<br>500  | ce<br>.00 💽 μΝ |
|              | Presets<br>Default  | Sec                       | Sect                       | ion 3.<br>]<br>4.1 | 4.4.1<br>G     |
|              |   | ~                         | ОК                         | >                  | Cancel         |

#### 3.4.5.1 Detailed parameters

Fig.40 UNHT ADO window (e.g. default preset for hard sample)

| Adjust depth o | offset parameters                 | ers  |
|----------------|-----------------------------------|--|
| <b>.</b>       | Approach <u>speed</u><br>25000.0  | Contact force  |
|                | Characterization force            | Contact stiffness threshold  |
|                | Indenter<br>Pre-approach<br>5 💽 % | Reference       Pre-approach     Contact force       99 ♥     %     500.00 ♥ |
|                | Presets<br>Default                | Section 3.4.4.1  |

Fig.41 UNHT Bio ADO window (e.g. default preset for hard sample)

**INFORMATION:** There is no reference with UNHT Bio (Fig.41). Thus the corresponding Reference area fields are inactive.

The field values of the applied preset (section 3.4.4.1), if the preset corresponds to the type of the sample which is used, are suitable to perform a successful ADO. However each following value can be separately adjusted if necessary.

#### Surface detection parameters area

#### Approach speed field:

To set the speed at which the indenter/(UNHT) reference approach the sample surface.

#### Contact force field:

It is the same parameter as the one described in the hardware parameter preferences (section 3.7.6.9), but here it is used during the ADO automatic process.

#### Characterization force field:

To set the indenter force to be applied for indenting the sample.

#### Contact stiffness threshold field:

It is the same parameter as the one described in the hardware parameter preferences (section 3.7.6.8), but here it is used during the ADO automatic process.

#### Quick mode box:

Only available with matrix measurement types (section 3.6).

To speed-up the ADO automatic process (section 3.4.4.2), check this box; the reference stays in contact with the sample and the indenter is not fully retracted (moved up) till the end of the matrix.

Uncheck for standard ADO process.

#### Indenter area

#### Pre-approach field:

To set the indenter pre-approach before the first approach starts (the indenter moves up out of its rest position: 100 % which corresponds to the fully extended (lowest) position of the actuator displacement).

#### Reference area (for UNHT)

#### *Pre-approach* field:

Similar description as for the indenter field (described above), **exception:** It is applicable for the reference.

#### Contact force field:

Similar description as for the indenter field (described above), **exception:** It is applicable for the reference.

## 3.4.5.2 Detailed sequences

**Reminder:** *Extra* UNHT Approach Monitor *window (Fig.42) can be hidden or displayed with the UNHT.INI file (section 2.6).* 



Fig.42 Extra UNHT Approach Monitor window

For the description of the window common features, see section 3.4.4.2. Otherwise wait for the end of the automatic process (described as follows).



Fig.43 UNHT/Bio ADO in progress (extended) window

**INFORMATION:** The following sequences concerning the reference are not applicable with UNHT Bio (to be ignored).

#### <u>The window (Fig.43) real-time</u> <u>sequences are described on the left</u>

<u>Command area</u> (extended window) <u>Indenter Command</u> (beige) and <u>Reference command</u> (blue) progression bars has decreased (from 100 %); this can be observed in <u>UNHT Approach</u> <u>Monitor</u> window (Fig.42) under the same (vertical) progression bars.

<u>Bridges area</u> (extended window) Fn Bridge (red), Dz Bridge (green) and FnRef Bridge (blue) progression bar has fluctuated; this can be observed in UNHT Approach Monitor window (Fig.42) under the same progression bars.

#### <u>Status area</u>

Approaching Table Z... (green) square blinks.

<u>Command area</u> (extended window) Reference command (blue) progression bar decreases.

#### Status area

Approaching reference... (green) square blinks.

<u>Command area</u> (extended window) Reference command (blue) progression bar increases.

#### The instrument actions are described on the right

With TTX/Step (not with STeP 5), the motorized head/module approaches (moves down).

The *Indenter* and *Reference* retract (move up) to the *pre-approach* values (section 3.4.5.1), **except +15 %** is added for the reference.

The Fn, Dz and FnRef sensor bridges are automatically adjusted (could be quick).

The motorized Z table slowly approaches (moves up).

When the sample surface contacts the reference, the motorized Z table stops and the reference actuator is retracted (moved up) by **15 %**  $\rightarrow$  The *Reference* returns to its initial *pre-approach* value (section 3.4.5.1).

The reference approaches (moves down) with the *Approach speed* (section 3.4.5.1) until it reaches the *Reference Contact force* (section 3.4.5.1). **INFORMATION:** *The reference touches* ~10 % above the Reference preapproach value (section 3.4.5.1).

#### <u>Status area</u>

Approaching with the indenter... (green) square blinks.

(extended window) <u>Indenter area</u> Indenter command (beige) progression bar increases. <u>Additional information area</u> Fn (red) and Dz (green) values are displayed. <u>Graph area</u> Fn (red) and Dz (green) curves are displayed.

<u>Status area</u> Contact (brown) square lights.

#### <u>Bridges</u> area

*Dz Bridge* (green) progression bar has fluctuated; this can be observed in *UNHT Approach Monitor* window (Fig.42) under the same progression bar.

#### <u>Status area</u>

*Stabilization* (brown) square lights and the decreasing stabilization time is displayed (in sec.).

#### <u>Status area</u>

Sample characterization (brown) square lights and its progression bar increases.

<u>Additional information area</u> (extended window) Sample characterization coefficients are displayed. The indenter approaches (moves down) with the *Approach speed* (section 3.4.5.1) until ...

... it contacts the sample.

 $\rightarrow$  The indenter has either reached the Contact stiffness threshold or the Contact force (section 3.4.5.1).

The Dz sensor bridge is adjusted.

Wait for the drift stabilization (cannot be set by the user, automatic algorithm).

The indenter position slightly varies (indenter is still in contact); this can be observed in UNHT Approach Monitor window (Fig.42) under Indenter command (beige) progression bar.

An indentation with the *Characterization force* (section 3.4.5.1) is performed to evaluate the sample mechanical properties. As previously described, the indenter position slightly varies.

The ADO is defined.

<u>Status area</u> *Rising the indenter* (green) square blinks.

<u>Command area</u>(extended window) Indenter Command (beige) and Reference Command (blue) progression bars decrease. The *Indenter* and *Reference* retract (moving up) to their *Pre-approach* values (section 3.4.5.1).

In case of *Quick mode* box has been checked, the indenter is retracted only a little bit and the reference stays in position (contact with sample) till the end of the matrix of measurements.

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

In case of *Quick mode* box has been checked, the motorized Z table stays in position (sample against reference) till the end of the matrix of measurements.

And then with Step (not with STeP 5), the motorized head module retracts (moves up).

In case of *Quick mode* box has been checked, the motorized head module stays in lower position (down) till the end of the matrix of measurements.

At the end of the process, both indenter and reference return (move down) to their extended rest positions; this can be observed only in UNHT Approach Monitor window (Fig.42) under Indenter Command (beige) and Reference command (blue) vertical progression bars (increase to 100 %).

## 3.4.5.3 Summary schematics (with Step)



#### **3.4.6 NHT ADO PARAMETERS AND SEQUENCES**

#### 3.4.6.1 Detailed parameters

| Adjust depth | offset parameters  |  |
|--------------|--|--|
| <b>T</b>     | Surface detection parameters<br>Approach <u>speed</u><br>100000.d 💽 nm/min |  |
|              | Characterization force   | Contact stiffness threshold                        |
|              | Presets<br>Default   | Section 3.4.4.1<br>Section 3.4.4.1<br>VOK X Cancel |

Fig.44 NHT ADO window (e.g. default preset for hard sample)

The field values of the applied preset (section 3.4.4.1), if the preset corresponds to the type of the sample which is used, are suitable to perform a successful ADO. However each following value can be separately adjusted if necessary.

#### Surface detection parameters area

#### Approach <u>speed</u> field:

To set the speed at which the indenter approaches the sample surface.

#### Characterization force field:

To set the indenter force to be applied to indent the sample.

#### Contact stiffness threshold field:

It is the same parameter as the one described in the hardware parameter preferences (section 3.7.6.8), but here it is used during the ADO automatic process.

#### *Quick mode* box:

Only available with matrix measurement types (section 3.6). To speed-up the ADO automatic process (section 3.4.4.2), check this box; the reference ring stays in contact with the sample and the indenter is not fully retracted (moved up) till the end of the matrix. Uncheck for standard ADO process.

## 3.4.6.2 Detailed sequences

For the description of the window common features, see section 3.4.4.2. Otherwise wait for the end of the automatic process (described as follows).



Fig.45 NHT ADO in progress (extended) window

# The window (Fig.45) real-time sequences are described on the left

#### <u>Status area</u>

<u>Stat</u>us area

blinks.

Approaching Table Z... (green) square blinks.

## The instrument actions are described on the right

With TTX/Step (not with STeP 5), the motorized head/module approaches (moves down).

Then with Step (not with STeP 5), the Z motorized table approaches (moves up) until the sample contacts the reference ring.

The indenter pre-approaches (moves down) and ...

An *adjusting bridge* pop up appears and its progression bar increases.

Approaching with the indenter... (green) square

... the Dz sensor bridge is automatically adjusted a  $1^{st}$  time (could be quick).

| <u>Status area</u><br>Approaching with the indenter (green) square<br>blinks.   | The indenter approaches (moves down) with the <i>Approach <u>s</u>peed</i> (section 3.4.6.1) until   |
|---|--|
| (extended window)<br><u>Command area</u><br>Indenter Command (beige) progression bar<br>increases.<br><u>Additional information area</u><br>Fn (red) and Dz (green) values are displayed.<br><u>Graph area</u> Fn (red) and Dz (green) curves<br>are displayed. |  |
| <u>Status area</u><br>Contact (brown) square lights.  | it contacts the sample.<br>$\rightarrow$ The indenter has reached the<br><i>Contact stiffness threshold</i><br>(section 3.4.6.1).                      |
| <u>Status area</u><br>Stabilization (brown) square lights and the<br>decreasing stabilization time are displayed (in<br>sec.).  | Wait for the drift stabilization<br>(cannot be set by the user,<br>automatic algorithm).<br>(This sequence could be quick.)                            |
| An <i>adjusting bridge</i> pop up appears and its progression bar is increasing.  | The Dz sensor bridge is automatically adjusted a 2 <sup>nd</sup> time (could be quick).  |
| <u>Status area</u><br>Sample characterization (brown) square lights<br>and its progression bar increases.   | An indentation with the <i>Characterization force</i> (section 3.4.6.1) is performed to evaluate the sample mechanical properties.                     |
| <u>Additional information area</u> (extended window)<br>Sample characterization coefficients are<br>displayed.  | The ADO is defined.  |
| <u>Status area</u><br>Rising the indenter (green) square blinks.  | The indenter retracts (rises) until its top (rest) position (0 %).   |
| <u>Command area</u> (extended window)<br>Indenter Command (beige) progression bar<br>decreases.   | In case of <i>Quick mode</i> box has<br>been checked, the indenter is<br>retracted only a little bit till the<br>end of the matrix of<br>measurements. |

With TTX/Step (not with STeP 5), the motorized head/module retracts (moves up).

In case of *Quick mode* box has been checked, the motorized head/module stays in lower position (down) till the end of the matrix of measurements

Then the Step (not with STeP 5) Z motorized table retracts (moves down).

In case of *Quick mode* box has been checked, the motorized Z table stays in position (sample against reference ring) till the end of the matrix of measurements.
## 3.4.6.3 Summary schematics (with Step)



## 3.4.7 MHT ADO PARAMETERS AND SEQUENCES

| Adjust depth | offset parameters  | Contact force   |
|--------------|--|---|
| <b>•</b>     | Surface detection parameters<br>Approach <u>s</u> peed<br>200.d 💭 µm/min | 25.00 mN<br>Contact force<br>0.03 N                               |
|              | Characterization force   | Contact stiffness threshold                                       |
|              | Approach distance<br>5.0 💓 μm  | Dz range<br>● 100 µm ← Fine <sup>1</sup> )<br>○ 1000 µm ← Largol) |
|              | Indenter<br>Pre-approach<br>10 💽 %                                       |   |
|              | Presets<br>Default   | Section 3.4.4.1<br>Section 3.4.4.1<br>V OK X Cancel               |

## 3.4.7.1 Detailed parameters

Fig.46 MHT ADO window (e.g. default preset for hard sample)

The field values of the applied preset (section 3.4.4.1), if the preset corresponds to the type of the sample which is used, are suitable to perform a successful ADO. However each following value and Dz range can be separately adjusted if necessary.

#### Surface detection parameters area

#### Approach speed field:

To set the speed at which the indenter approaches the sample surface.

#### Contact force field:

It is the same parameter as the one described in the hardware parameter preferences (section 3.7.6.9), but here it is used during the ADO automatic process.

#### Characterization force field:

To set the indenter force to be applied to indent the sample.

#### Contact stiffness threshold field:

It is the same parameter as the one described in the hardware parameter preferences (section 3.7.6.8), but here it is used during the ADO automatic process.

<sup>1)</sup> Dz range values can be different for old MCT versions.

## Approach distance area field:

For this parameter, see description in section 3.7.6.7 (hardware parameter preferences) under <u>MHT approach distance</u>, p. 126.

#### <u>Dz range</u>

Dz range area radio buttons:

## **IMPORTANT:**

- If the ADO procedure is started from the indenter calibration (<Calibrate> button is clicked section 2.5.2), the fine Dz range must be selected here (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 (e.g. <100 µm>) but if the depth saturates, then select the large Dz range.
- Later in the hardware parameter preferences (section 3.7.6.6), the Depth range radio button which is selected should be the same as the ADO Dz range radio button selected here (fine: e.g. <100 μm>) for the current successful ADO. Otherwise an error message appears; see section 5.2.

### Indenter area

#### Pre-approach field:

To set the indenter pre-approach before the first approach starts (the indenter moves down out of its rest position: 0 % which corresponds to the fully retracted (highest) position of the actuator.

The min. and recommended value is 10 %.

## 3.4.7.2 Detailed sequences

For the description of the window common features, see section 3.4.4.2. Otherwise wait for the end of the automatic process (described as follows).



Fig.47 MHT ADO in progress (extended) window

### The window (Fig.47) real-time sequences are described on the left

<u>Command area</u> (extended window) Indenter Command (beige) progression bar increases.

# The instrument actions are described on the right

The *Indenter* pre-approaches (moves down) to the value set in *Pre-approach* (section 3.4.7.1).

This extra *Adjusting Dz range* window (Fig.48) appears over window Fig.47.



Fig.48 Adjusting Dz range window

## <u>Status area</u>

Approaching Table Z... (green) square blinks.

## <u>Status area</u>

Approaching with the indenter... (green) square blinks.

<u>Status area</u> Contact (brown) square lights.

<u>Status area</u> Stabilization (brown) square lights and the decreasing stabilization time are displayed (in sec.).

## Dz range adjustment 1

Turn<sup>1</sup> the wheel to adjust the first DZ range targeted *Approach distance* (section 3.4.7.1) used for the next measurement(s) performed later:

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 click <OK> button to continue (this window Fig.48 is closed).

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

The indenter approaches (moves down) with the *Approach speed* (section 3.4.7.1) until ...

... it contacts the sample.  $\rightarrow$  The indenter has either reached the *Contact stiffness threshold* or the *Contact force* (section 3.4.7.1).

Wait for the drift stabilization (cannot be set by the user, automatic algorithm).

<sup>1</sup> The rotation direction (an arrow) is displayed on the extra window (Fig.48).



#### <u>Status area</u>

Sample characterization (brown) square lights and its progression bar increases.

<u>Additional information area</u> (extended window) Fn (red) and Dz (green) values are displayed.

<u>Additional information area</u> (extended window) Sample characterization coefficients are displayed.

#### Dz range adjustment 2

Adjust the other Dz range; see the same instructions as previously described in <u>Dz range</u> <u>adjustment 1</u>, p. 77.

An indentation with the *Characterization force* (section 3.4.7.1) is performed to evaluate the sample mechanical properties.

The ADO is defined.

The Z motorized table retracts (moves down).

The indenter retracts (rises) until its top (rest) position (0 %).

<u>Status area</u>

Rising the indenter (green) square blinks.

<u>Command area</u> (extended window) Indenter Command (beige) progression bar decreases.

## 3.4.7.3 Summary schematics (with Step)



## **3.5 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  $\bigcirc$  is enabled: Before any measurement is started (section 3.7.5), the *Wait* 



See the same information than described in <u>Wait time</u>, p. 41, for the indenter calibration matrix of measurements), **exception:** It is applicable here for a single measurement or for a matrix of measurements.

**INFORMATION:** A delay of max. 2 months can be set.

## **3.6 SELECTING THE MEASUREMENT (TYPE OR PROTOCOL)**

Select "**Instrument > Start a new indentation...**" from menu bar or click  $\overline{\mathbf{x}}$  on toolbar. The window allows selecting a new measurement (single indentation measurement or **matrix** of several indentation measurements). The measurement type which is selected in the top list should be set. Each (available) measurement protocol at the bottom of the list is a measurement type (top list) which has been previously selected, set and saved (its parameters could be modified is necessary).



Fig.49 Define a new measurement window

<Define a <u>new measurement> radio button:</u>

To select (highlight) one active  $^{1)}$  measurement type in the (top) list.  $\ensuremath{\mathsf{OR}}$ 

<<u>U</u>se a protocol> radio button:

To select (highlight) one available measurement protocol in the (bottom) list:

- To save (or delete) a measurement type as a protocol, see section 3.7.7 and to manage the protocols, refer to the *Common Scratch & Indentation software reference guide* in section *Managing the measurement protocols*.
- <sup>1)</sup> Each inactive measurement type cannot be selected as it is not applicable for the current measuring head.

- Depending on the current measuring head, **default** protocol(s) in bold and at the end of the (bottom) list are present (e.g in Fig.49), with pre-defined parameters for certain dedicated sample type.

**IMPORTANT:** The sample in use should correspond to the selected default protocol.

**INFORMATION:** These default protocol(s) cannot be deleted or managed, only their corresponding measurement type parameters can be modified if necessary.

## <<u>O</u>K> button:

To open the window of the measurement type or protocol selected in a list. **INFORMATION:** Double clicking a measurement type or a protocol in a list does the same effect.

The required parameters of the corresponding measurement type window should be set (or modified for a protocol if necessary) before running the measurement(s); see the following sections for:

- the main **description** of a measurement type window section 3.7.1
- the common **loading rate** parameter description section 3.7.2
- the common **acquisition rate** parameter description section 3.7.4
- all required **parameters** for each measurement type window; see from section 3.7.5

•

• the **preference parameters** dedicated for each measuring head; see from section 3.7.6

## **3.7 SETTING THE MEASUREMENT TYPE PARAMETERS**

#### **3.7.1** MAIN DESCRIPTION OF A MEASUREMENT TYPE WINDOW

A measurement type is selected in *Define a new measurement* window (Fig.49).



From section 3.7.5, see the specific required parameters for each measurement type window.

<sup>&</sup>lt;sup>1</sup> With Load or Depth control

## **3.7.2 ESTIMATED INFO FIELDS**

Estimated time

Estimated memory size 459.6 KB

Estimated duration field (in Hour:Minute:Second):

It provides an estimation time that is necessary to perform the measurement(s) defined in the current measurement type window (time is refreshed with different parameters).

**INFORMATION:** In addition, number of day(s) may appear.

Estimated memory size field (in KB):

It provides an estimation of the software RAM memory size that is necessary to perform the measurement(s) defined in the current measurement type window (time is refreshed with different parameters).

**IMPORTANT:** In case of a red warning message appears at the left bottom of the window, decrease parameter(s) until the message disappears. Otherwise the software may crash during the measurement(s).

## **3.7.3** LOADING PROFILES

The measurement(s) is performed with one of the loading profiles described below. Depending on the selected measurement (Fig.49), several loading profiles are proposed (one can be selected).

## 3.7.3.1 Linear loading/Constant time loading

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

 $F = k \cdot t$ 



# 3.7.3.2 Quadratic loading

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



## 3.7.3.3 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:

t

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

## **3.7.4 COMMON ACQUISITION RATE PARAMETER**

Common parameter to all measurement types; see from section 3.7.5.

Acguisition rate

Acquisition rate field: To set the acquisition rate. The value corresponds to the number of data point recorded per second during the measurement(s). Initial value is 10 Hz Min. value is 1 Hz and max. 400 Hz

However, there is no field to set for the following measurements types:

- Standard measurement type section 3.7.5.1, value is fixed to 10 Hz
- *Sinus* measurement type section 3.7.5.3, value is automatically calculated (20x the Sinus frequency value)
- User defined profile measurement type section 3.7.5.7, value depends on the load profile segments which are created
- Quick matrix measurement type section 3.7.5.10, value is fixed to 400 Hz

## **3.7.5 PARAMETERS OF EACH MEASUREMENT TYPE**

A measurement type is selected in the *Define a new measurement* window (Fig.49).

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).

## 3.7.5.1 Standard

The *Standard* measurement type is the simplest **single** indentation measurement, performed in **1 cycle** of automatic linear loading/unloading; section 3.7.3.1.

|                                 | · · · · · · · · · · · · · · · · · · · |
|---------------------------------|---------------------------------------|
| Standard                        |                                       |
| Standard indentation parameters | Profile description                   |
| Max load                        |                                       |
|                                 |                                       |
| Load time : 30 s                | -                                     |
| Unload time : 30 s              | Hardware parameters                   |
|                                 | + Step - NHT S/N: 123456 settings     |
| Pause                           | Dz sensor in fine range               |
| 5.4 🗨 s                         | Stiffness threshold : 500 μN/μm       |
| Section 3.7.2<br>Estimated time |                                       |
|                                 | Section 3.7.6                         |
| 78.2 KB                         | Change                                |
| Section 3.7.7                   | Section 3.8                           |
| Save as protocol                | ✓ <u>O</u> K X <u>C</u> ancel         |

Fig.51 Standard window parameters

**INFORMATION:** About the acquisition rate, see section 3.7.4.

| <u>M</u> ax load field:        | To set the max. load to be applied.   |
|--------------------------------|---|
| Load Time fixed parameter:     | A linear loading rate is computed to reach the max. load (above) within 30 sec.             |
| Unload Time fixed parameter:   | After the pause (below), a linear unloading rate is computed to fully unload within 30 sec. |
| <u>P</u> ause field (in sec.): | To set a pause at the max. load before unloading. Value sets to $0 = no$ pause.             |

## 3.7.5.2 Advanced

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



Fig.52 Advanced window parameters

| <i>Linear loading</i> tab      | below: |
|--------------------------------|--------|
| <i>Quadratic loading</i> tab   | p. 89: |
| Constant strain rate (CSR) tab | p. 90: |

To select the corresponding loading profile. The related set of parameters is described as follows.

#### Linear loading tab

|  | To select a <b>depth</b> mode:   |
|--|--|
| <max (load="" control)="" depth=""> radio button/field:</max>  | Load controlled measurement. The load increases linearly at the defined load rate until the max. depth defined is reached.             |
| or   | or   |
| <max (depth="" control)="" depth=""> radio button/field:</max> | Depth controlled measurement. The depth increases linearly at the defined loading rate ( $\mu$ m/min) until the max. depth is reached. |
| OR   | OR   |
| Max load radio button/field:                                   | To select the <b>load</b> mode and to set the max. load to be applied.   |
| Loading <u>r</u> ate field:                                    | To set the loading rate to reach the max. depth OR max. load (above).  |
|  |  |

#### Hazardous result

**INFORMATION:** *With* Max depth (Depth control) *radio button selected* (*previously described*), *if* Approach speed *field value in* Hardware parameters window / Preferences *tab* (*section 3.7.6*) *is at least 4 times faster than* Loading rate *field value* (*previously described*), *the measurement may have hazardous results.* 

| The loading rate and the approach s   | peed are too different. Your measuremen | t may have hazardous result !   |
|---|---|---|
| <ul> <li>○ Max depth (Load control)</li> <li>● Max depth (Depth control)</li> </ul> | Loading rate                            | + STeP - MHT S/N: 123456 settings<br>Fn contact : 0.01 N<br>→ Approach speed : 16.6 µm/min<br>Dz sensor in fine range |

<u>P</u>ause field (in sec.): To set a pause at the max. depth OR max. load before unloading. Value sets to 0 = no pause.

<u>Unloading rate field</u>: To set the unloading rate to fully unload after the pause.

Land see Cha

### <u>Quadratic loading tab</u>

|                                  |   | Load prome |
|----------------------------------|---|------------|
| Linear loading Quadratic loading | Constant strain rate  |            |
| Max <u>l</u> oad                 | Time to max load<br>30.00 s<br>Pause<br>15.0 s<br>Time to unload<br>30.00 s |            |

*Max load* field: To set the max. load to be applied.

*Time to max load* field: To set the time to reach the max. load (above).

Pause field:To set a pause at the max. load (above) before unloading.<br/>Value sets to 0 = no pause.

*Time to unload* field: To set the time to fully unload after the pause (above).

## Constant strain rate (CSR) tab

| <u>constant s</u>   |   | <u>ony tab</u>            | Load profile  |
|---|---|---------------------------|---|
| Linear loading  | Quadratic loading                       | Constant strain rate      |   |
| ⊖ Depth control   |   | Loading <u>r</u> ate/load |   |
| Min depth   | <b>0.00</b> μm                          | 0.0500 🖨                  | 1/s   |
| Max depth   | <b>0.00</b> μm                          | <u>P</u> ause             |   |
| Load control  |   | 10.d 🚍                    | s   |
| Min load  | 0.100 🚔 N                               |                           |   |
| Max load  | 1.00 🗭 N                                |                           |   |
| <depth<br><i>Min dep</i><br/><i>Max dep</i><br/>OR</depth<br> | control> rad<br>th field:<br>oth field: | lio button:               | To select the <b>depth</b> mode and<br>To set the min. depth at which the constant<br>strain rate starts.<br>To set the max. depth to be reached.<br>OR |
| <load c<br="">Min load</load>                                 | field :                                 | Dutton:                   | To set the min. load at which the constant  |
| Max loa   | d field:                                |                           | To set the max. load to be applied.   |
| Loading <u>r</u> a  | <i>te/load</i> field:                   | -<br>-                    | To set the loading rate to reach the max. depth OR max. load (above).<br>Typical range values = from 0.01 to $0.1[s^{-1}]$                              |
| <u>P</u> ause field   | l:                                      | -                         | To set a pause at the max. depth OR max. load<br>(above) before unloading.<br>Value sets to 0 = no pause.   |

An automatic linear unloading rate is performed to fully unload after the pause (above).

## 3.7.5.3 Sinus

**INFORMATION:** Sinus measurement type is not available (Sinus inactive) in the top list of Define a new measurement window (Fig.49) for MHT, UNHT Bio, and NHT without the optional Sinus mode/generator (see section 2.1.1.3).

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. This measurement type allows obtaining a depth related analysis; see section 4.8.1.



Fig.53 Sinus window parameters

*Linear loading* tab below:

*Constant strain rate (CSR)* tab p. 93:

To select the corresponding loading profile. The related set of parameters is described as follows.

#### Linear loading tab

| <i>Max <u>l</u>oad</i> field:      | To set the max. load to be applied.                                     |
|------------------------------------|---|
| Loading <u>r</u> ate field:        | To set the loading rate to reach the max. load (above).                 |
| Un <u>l</u> oading rate field:     | To set a linear unloading rate to fully unload after the pause (below). |
| <u>P</u> ause field:               | To set a linear pause at the max. load (above) before unloading.        |
| <i>Max. sinus amplitude</i> field: | To set the sine wave max. amplitude for the loading (above).            |

## Sinus driving mode area

<Constant depth> radio button: OR

<Linear load> radio button:

To set the sine wave frequency for the loading (previously described).

To select the sinus amplitude driven with constant depth (Fig.54). Square root evolution of sine amplitude.

#### OR

To select the sinus amplitude driven with linear load (Fig.55). Linear evolution of sine amplitude



Fig.54 Constant depth driving (Acquisition debug window<sup>1</sup> purple area)



Fig.55 Linear load driving (Acquisition debug window<sup>1</sup> purple area)

<sup>1</sup> To display the acquisition debug window (after measurement is performed), see section 4.2.

#### Constant strain rate (CSR) tab



Min load field:

To set the min. load at which the Sinus constant strain rate starts.

Max load field:

Loading <u>rate/load</u> field:

To set the max. load to be applied.

To set the loading rate to reach the max. load (above).

Typical range values = from 0.01 to 0.1 [ $s^{-1}$ ]

An automatic linear unloading rate is performed to fully unload after the pause (below).

Pause field:To set a pause at the max. load (above)<br/>before unloading.Max. sinus amplitude field:3 same parameters than described for the<br/>previous Sinus Linear loading tab, p. 91.Sinus frequence field:91.

Sinus driving mode area

<Constant depth> radio button:

OR

<Linear load> radio button:

## 3.7.5.4 Constant multicycle

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

| Constant multicycle  |   |   |
|--|---|---|
| Multi-cycle indentation parameters   | Load profile  |   |
| Acquisition rate   |   |   |
| Section 3.7.3►   |   | \\ <sub>&gt;</sub>  |
| Linear loading Quadratic loading   |   |   |
| <ul> <li>In depth (Load control)</li> <li>In depth (Depth control)</li> <li>Max depth</li> <li>4.00 → nm</li> <li>Unload down to depth</li> <li>4.00 → nm</li> <li>Loading rate</li> <li>40.00 → mN/min</li> <li>Pause</li> <li>2.0 → s</li> </ul> | In load          Max load         10.00 mN         Unload down to         2.00 mN         Unloading rate         40.00 mN/min | Hardware parameters<br>+ Step - NHT S/N: 1234567890 settings<br>Approach distance : 1500 nm<br>Approach speed : 2000 nm/min<br>Dz sensor in fine range<br>Stiffness threshold : 500 µN/µm |
| Section<br>Estimated time<br>0:11:47<br>Cycles<br>Number of cycles<br>20<br>Section 3.7.7  | 3.7.2<br>Estimated memory size<br>586.5 KB<br>Pause between cycles<br>0.0 s   | Section 3.7.6<br>Change<br>Section 3.8  |

Fig.56 Constant multicycle window parameters

## <u>Cycles area</u>

| <u>N</u> umber of cycles | To set the number of cycles to be |
|--------------------------|-----------------------------------|
| field:                   | performed.                        |
|                          | Min. value = 2                    |



The following parameters are **the same** for all cycle repetitions which are performed (above).

| <u>P</u> ause between cycles | To set a pause to be done between |
|------------------------------|-----------------------------------|
| field:                       | each cycle (above).               |
|                              | Value sets to $0 = no pause$ .    |



*Linear loading* tab p. 95: *Quadratic loading* tab p. 95:

To select the corresponding loading profile. The related set of parameters, is described as follows.

## <u>Linear loading tab</u>

|            | <in (load="" control)="" depth=""> or<br/><in (depth="" control)="" depth=""><br/>radio button:</in></in> | -<br>I                 | To select the <b>depth</b> mode, either controlled by oad or by depth and  |
|------------|---|------------------------|--|
|            | Max depth field:  | -                      | To set the max. depth to be reached for each cycle.  |
|            | Unload down to depth field:   | -                      | To set the min. depth to be reached for each cycle <sup>1</sup>  |
| OI         | २   | OR                     |  |
|            | <in load="" radio=""> button:</in>  | -                      | To select the <b>load</b> mode and   |
|            | Max load field:   | -                      | To set the max. load to be applied for each cycle.   |
|            | Unload down to field:   | -                      | To set the min. load to be applied for each cycle <sup>1</sup>   |
| Lc         | <i>ading <u>r</u>ate</i> field:   | To<br>OR               | set the loading rate to reach the max. depth max. load of each cycle (above).  |
| <u>P</u> a | use field (in sec):   | To<br>eac<br>Val       | set a pause at the max. depth OR max. load of ch cycle (above) before unloading.<br>ue sets to $0 = no$ pause.   |
| <u>U</u> ı | nloading rate field:  | To<br>OR<br>pau<br>Aft | set the unloading rate to reach the min. depth<br>min. load of each cycle (above) after each<br>use (above).<br>er the last cycle, it is fully unloaded. |

# <u>Quadratic loading tab</u>

| Linear loading Quadratic loading | Load profile     |
|----------------------------------|------------------|
| Max load                         | Time to max load |
| Unload down to                   | Pause            |
| 2.00 🖢 mN                        | Time to unload   |

| <i>Max <u>l</u>oad</i> field:               | To set the max. load to be applied for each cycle.   |
|---|--|
| <u>U</u> nload down to field:               | To set the min. load to be applied for each cycle $^1$   |
| <i>Time to max load</i> field:              | To set the time to reach the max. load for each cycle (above).   |
| <i>Pau<u>s</u>e</i> field (in <i>sec</i> ): | To set a pause at the max. load for each cycle (above) before unloading. Value sets to $0 = no$ pause.                                 |
| <i>Time to unload</i> field:                | To set the time to reach the min. load for each cycle (above) after each pause (above).<br>After the last cycle, it is fully unloaded. |

<sup>1</sup> **Exception:** The last cycle is fully unloaded.

## 3.7.5.5 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 **max.** depth or **max.** load increases for each measurement cycle (the min. depth or load remains the same for all measurement cycles).

| Progressive multicycle  |   |  |
|---|---|--|
| Multi-cycle indentation parameters  |   | Load profile   |
| Acquisition rate  | 7.4   | Auto   |
| ◄ Section 3.7.3► Linear loading Quadratic loading                                 |   |  |
| In depth (Load control)   | ● In load   | Hardware parameters<br>+ Step - UNHT S/N: 123456 setting   |
| First depth<br>4.00 nm<br>Unload down to depth<br>4.00 nm<br>Max depth<br>4.00 nm | First load<br>100.00 ♥ µN<br><u>U</u> nload down to<br>50.00 ♥ µN<br>Max <u>l</u> oad<br>1000.00 ♥ µN | Fn contact : 300 µN<br>Approach distance : 1500 nm<br>Approach speed : 2000 nm/min<br>Dz sensor in fine range<br>Load in fine range<br>FnRef in fine range<br>FnRef contact load : 500.000 µN<br>Stiffness threshold : 150 µN/µm |
| Loading <u>r</u> ate<br>400.00 ♥ µN/min<br>Pause<br>2.0 ♥ s                       | <u>U</u> nloading rate<br>400.00 ♥ µN/min   |  |
| Section 3<br>Estimated time   | .7.2<br>Estimated memory size<br>383.8 KB   |  |
| Cycles<br>Number of cycles  | Pause between cycles<br>0.d ♥ s   | Section 3.7.6<br>Change  |
| Section 3.7.7   |   | Section 3.8  |

Fig.57 Progressive multicycle window parameters

*Linear loading* tab p. 97: *Quadratic loading* tab p. 97:

To select the corresponding loading profile. The related set of parameters, is described as follows.

## Cycles area

<u>N</u>umber of cycles field: <u>P</u>ause between cycles field (in sec):

Same parameters described in <u>Cycles area</u> (p. 94) of the <u>Constant multicycle</u> measurement type.

## Linear loading tab

| <in (load="" control)="" depth=""> or<br/><in (depth="" control)="" depth=""><br/>radio button:</in></in>  | To select the <b>depth</b> mode, either controlled by load or by depth and  |
|--|---|
| <i>First depth</i> field:<br><u>Unload down to depth</u> field:<br><u>Max depth</u> field:<br>OR<br><in load=""> radio button:<br/><i>First load</i> field:<br/><u>Unload down to</u> field:<br/><u>Max load</u> field:</in> | To set the max. depth <sup>1</sup> for the first cycle.<br>To set a min. depth for each cycle <sup>2</sup><br>To set the max. depth <sup>1</sup> for the last cycle.<br>OR<br>To select the <b>load</b> mode and<br>To set the max. load for the first cycle <sup>1</sup><br>To set a min. load for each cycle <sup>2</sup><br>To set the max. load for the last cycle <sup>1</sup> |
| Loading <u>r</u> ate field:  | To set the loading rate to reach each cycle max.<br>depth OR max. load (above).<br><b>INFORMATION:</b> See the same information<br>described in <u>Hazardous result</u> , p. 89 of Advanced<br>measurement type.  |
| <u>P</u> ause field (in sec):  | To set a pause at each cycle max. depth OR max.<br>load (above) before unloading.<br>Value sets to 0 = no pause.  |
| <u>U</u> nloading rate field:  | To set the unloading rate to reach the min. depth OR min. load for each cycle <sup>2</sup> (above) after each pause (above).  |

## Quadratic loading tab

| Linear Loading Quadratic Loading |                  |
|----------------------------------|------------------|
| First load                       | Time to max load |
| Unload down to                   | Pau <u>s</u> e   |
| Max <u>l</u> oad<br>1000.00 🗬 μΝ | Time to unload   |



*First load* field: To set the max. load<sup>1</sup> to be applied for the first cycle.

<u>Unload down to field</u>: To set a min. load to be applied for each cycle<sup>2</sup>

*Max load* filed: To set the max. load<sup>1</sup> to be applied for the last cycle.

*Time to max load* field: To set the time to reach each cycle max. load (above).

<sup>1</sup> Between the first and the last max. depth/load and depending on the number of cycles, the software computes a linear increment for each cycle intermediate max. depth/load.

<sup>2</sup> **Exception:** The last cycle is fully unloaded.

| <i>Pau<u>s</u>e</i> field:   | To set a pause at each cycle max. load (previously described) before unloading. Value sets to $0 = no$ pause. |  |
|------------------------------|---|--|
| <i>Time to unload</i> field: | To set the time to reach the min. load for each cycle <sup>1</sup> after each pause (previously described).   |  |

## 3.7.5.6 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 min. load increases for each measurement cycle, as well as the max. load.

| Acguisition rate   | ation Parameters<br>Ann 3.7.4   | Load profile   |
|--|---|--|
| Section 3.7.3.1  |   |  |
| Constant time loading  |   | - Auto   |
| First load<br>0.10 mN<br>Unload down to<br>10.00 %<br>Max load<br>100.00 mN<br>Linear max load incre<br>Quadratic max load incre | Time to max load<br>10.00 s<br>Pause<br>1.0 s<br>Time to unload<br>10.00 s<br>s<br>ement<br>increment | + Step - NHT S/N: 123456 settings<br>Approach distance : 1500 nm<br>Approach speed : 2000 nm/min<br>Dz sensor in fine range<br>Stiffness threshold : 500 µN/µm |
| Estimated time 0:09:10   | ion 3.7.2<br>Estimated memory size<br>505.3 KB  | -  |

Fig.58 CMC window parameters

The related set of parameters of the unique *Constant time loading* tab is described as follow.

## <u>Cycles area</u>

<u>N</u>umber of cycles field: <u>P</u>ause between cycles field (in sec):

Same parameters described in <u>Cycles area</u>, p. 94 of the *Constant multicycle* measurement type.

## Constant time loading tab

| <quadratic increment="" load="" max=""> radio button:</quadratic> | To select an automatic quadratic increment for each cycle max. load until the last cycle.   |
|---|---|
| OR  | OR  |
| <linear increment="" load="" max=""><br/>radio button:</linear>   | To select an automatic linear increment for each cycle max. load until the last cycle.  |
| First load field:   | To set the max. load. to be applied for the first cycle.  |
| <u>U</u> nload down to field (in %):                              | To set each cycle min. load. The value is a<br>percentage of each current cycle max. load.<br>The recommended range value for Oliver &<br>Pharr analysis is from 5 to 40 %<br>The last cycle is fully unloaded. |
| <i>Max <u>l</u>oad</i> field:                                     | To set the max. load to be applied for the last cycle.  |
| <i>Time to max load</i> field:                                    | To set the time to reach each cycle max. load (above).  |
| Pau <u>s</u> e field:   | To set a pause at each cycle max. load<br>(above) before unloading.<br>Value sets to 0 = no pause.  |
| <i>Time to unload</i> field:                                      | To set the time to reach each cycle min. load after the pause (above).  |

## 3.7.5.7 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.59 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.60).

<Add> button: To create a new segment (type/parameters) at the bottom of the list. <Copy> button: To copy the selected (highlighted) segment at the bottom of the list. If necessary, the segment can be modified. <Edit> button: To modify the selected (highlighted) segment in the list. Segment list

Linear Load 10.00 mN ; 20.00 mN/min Pause in Load 20 s

Quadratic Load 20.00 mN in 20 s Linear Sinus 15.00 mN ; 45.00 mN/min ; 2.00 @ 5 Pause Sinus 5 s ; 2.00 @ 5.0 Hz Linear Depth 150.00 nm ; 500.0 mm/min CSR Load 12.00 mN ; 0.10 1/s Linear Depth 0.00 nm ; 200.00 nm/min

Segment list Linear Load 10.00 mN ; 20.00 mN/min Pause in Load 20 s Linear Sinus 15.00 mN ; 45.00 mN/min ; 2.00 @ 5 Pause Sinus 5 s ; 2.00 @ 5.0 Hz Quadratic Load 20.00 mN in 20 s Linear Depth 150.00 nm ; 500.00 nm/min CSR Load 12.00 mN ; 0.10 1/s Linear Depth 0.00 nm ; 200.00 nm/min 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.

- <Remove> button: To remove the selected (highlighted) segment from the list.
- <Clear> button:

To clear all segments from the list.

A *Warning* window asks to confirm one of the previous deletions (<Yes> button) or not (<No> button).

| dit segment<br>Profile description  |  | ×  |
|---|--|--|
| Section 3.7.2   | User segment parameters<br>Final depth<br>0.00 nm<br>Final load<br>0.00 mN | Pause<br>0.0 s<br>Time to final load<br>0.00 s     |
| Linear Load End Depth<br>Linear Depth<br>Linear Sinus<br>Pause in Load<br>Pause in Depth<br>Pause Sinus<br>Quadratic Load | Loading rate<br>0.00 mN/min<br>Loading rate<br>0.00 nm/min                 | Loading <u>r</u> ate/Load<br>0.0010 💽 1/s          |
| CSR Load<br>CSR Depth<br>CSR Sinus  | Sinus amplitude<br>0.00 mN<br>Sinus constant depth                         | Sinus frequency<br>0.0 🗭 Hz<br>O Sinus linear load |
|   | CSR Load 0.00 mN ; 0.00 1/s  |  |
|   | ~  | <u>O</u> K X <u>C</u> ancel                        |

Fig.60 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.

The active set of parameters for each concerned segment type is described as follows.

## Loading profile parameters

| Final depth            | or | Final load field:  |
|------------------------|----|--------------------|
| For:                   |    | For:               |
| Linear Load End Depth/ |    | Linear Load/       |
| Linear Depth/          |    | Linear Sinus/      |
| CSR Depth              |    | Quadratic Load/    |
|                        |    | CSR Load/CSR Sinus |

To set the final depth or final load to be reached.

<Sinus constant depth> or <sinus linear load> radio button (inactive for Pause sinus):

To set the sinus wave amplitude and sinus frequency to add for the loading.

To select one of the sinus driving modes described in section 3.7.5.3 under *Sinus driving mode* area.

**INFORMATION:** A message in red: 'Warning: Outside operational frequencies 1-40 Hz' could appear. To obtain a correct Sinus analysis, it is recommended to set the Sinus frequency value from 1 to 40 Hz (other values do not block the measurement).

Loading <u>rate</u> (unit selected in options) field: For: Linear Load/Linear Load End Depth/Linear Sinus Or Loading <u>rate</u> (always in nm/min) field: For: Linear Depth Or Loading <u>rate/Load</u> (always in 1/s) field: For: CSR Load/CSR Depth/ CSR Sinus OR

Time to final load (always in s) field:

To set one of the 3 active loading rates in order to reach the final depth or final load (previously described).

OR

To set the time to reach the final load (previously described).

#### Pause parameter(s)

For: Quadratic Load

<u>Pause field:</u> For: Pause in Load/Pause in Depth/Pause Sinus To set a pause after the segment. With *Pause Sinus* segment type, set also the *Sinus amplitude* and *Sinus frequency* (above).

**INFORMATION:** The last segment should be unloaded to "0".

<<u>O</u>K> button: To valid the current segment settings in the list (Fig.59). **INFORMATION:** An Information window message appears in case of a parameter in not properly set.

## 3.7.5.8 Simple matrix

The *Simple matrix* measurement type performs a **matrix** of **identical** indentation measurements with a (same) **defined** X & Y spacing. The matrix of measurements starts from the current position of the tables.

| Simple matrix                 |                             |   |       |
|-------------------------------|-----------------------------|---|-------|
| Indentation matrix definition |                             | Indentation parameters  |       |
| Delta X                       | Delta <u>Y</u>              | <u>Edit Indentation parameters</u>                                  |       |
| 0.500 🗑 mm                    | 0.700 🗑 mm                  | + Standard  | ^     |
| Indentation count X           | Indentation <u>c</u> ount Y | Acquisition rate : 10.0 [Hz]<br>Linear loading<br>Max load : 1.00 N |       |
| Distance X                    | Di <u>s</u> tance Y         | Loading rate : 2.00 N/min<br>Unloading rate : 2.00 N/min            |       |
| 0 mm                          | 0 mm                        | + Step - MHT S/N: 123/567800 settings                               |       |
|                               | Estimated time              | Fn contact : 0.01 N<br>Approach speed : 16.6 µm/min                 |       |
| Indentation count             | Estimated memory size       | Load in fine range  | *     |
|                               | Section 3.7.2               | Edit <u>a</u> djust depth offset parameters                         |       |
| Section 3.7.7                 |                             | Section 3.8   |       |
| Save as protocol              |                             | <u>о</u> к <u>х</u> с   | ancel |

Fig.61 Simple matrix window parameters

#### Indentation matrix definition area

#### *Delta* <u>X</u> and *Delta* <u>Y</u> fields:

To set the (relative) distance between each measurement (<u>Indentation</u> parameters area, p. 103) for the X and Y axis.

<u>Indentation count X and Indentation count Y fields:</u>

To set the number of measurements for the X and Y axis.

#### Distance X and Distance Y info fields:

According to the setting of the parameters above, the distance between the first and the last measurement for the X and Y axis are displayed as information.

#### Indentation count info field:

The total number of the measurements which are performed (+ 1 if *Include an adjust depth offset* box is checked, under <u>Parameters for ADO</u>, p. 104) is displayed as information.

#### Indentation parameters area

<Edit Indentation parameters> button:

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

| andard<br>Ivanced    |          |       |   |
|----------------------|----------|-------|---|
| Wanceu VV            |          |       |   |
| onstant multicycle   |          |       |   |
| ogressive multicycle |          |       |   |
| мČ                   |          |       |   |
| er defined profile   |          |       |   |
| nple matrix          | atrix of | matri | v |
| JICK matrix          |          | matri | × |
| wanceu matrix        |          |       |   |

Measurement type:

To select (highlight) a measurement type.

<<u>O</u>K> button:

To open the corresponding window of the selected measurement type.

Double clicking a measurement type does the same effects.

The corresponding parameters should be set as same as described in the following sections.

**INFORMATION:** Adjust Depth Offset *is not useful for a* Simple matrix *of measurements (see Parameters for ADO below).* 

| Standard               | 3.7.5.1 | Advanced            | 3.7.5.2 |
|------------------------|---------|---------------------|---------|
| Sinus                  | 3.7.5.3 | Constant multicycle | 3.7.5.4 |
| Progressive multicycle | 3.7.5.5 | СМС                 | 3.7.5.6 |
| User define profile    | 3.7.5.7 |                     |         |

Matrix of matrix is possible,

| Simple matrix          | a $2^{nd}$ window as same as Fig.61 is superimposed |
|------------------------|---|
| Quick matrix           | 3.7.5.10  |
| Advanced matrix        | 3.7.5.9   |
| Visual advanced matrix | 3.7.5.11  |

## Parameters for ADO

#### Include an adjust depth offset box:

Only if necessary, check this box to include an ADO before performing the matrix of measurements.

<Edit adjust depth offset parameters> button (useful if box above is checked): To set the parameters for the included ADO (section 3.4.4.1).

The ADO automatic process (3.4.4.2) begins once the matrix of measurements is started ( $< \underline{O}K >$  button Fig.61).

## 3.7.5.9 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.

| Advanced matrix   |  |             |   |
|---|--|-------------|---|
| Selection   |  |             |   |
| Indentation definition  |  | Indentation | ion position  |
| Standard [0 ; 0]<br>Standard [1 ; 1]<br>Advanced [1.5 : 1]  | Add  | 7.2         | 2   |
| Advanced [2; 2]<br>Advanced [3; 1]<br>User defined profile [2; 1.5]   | Сору   |             | + +   |
| Adjust Depth Offset [7; 5]<br>Constant multicycle [7.5; 6]<br>Standard [8: 6]   | Edit   | 5.5-        | +   |
|   | Select & modify position                               |             | A cross can be selected directly in this graph,   |
| +   | Initial X Increment                                    | 3./-        | the related definition become selected (highlighted) in the list.                                       |
|   | Initial Y Increment                                    | 2.0-        | 0- +<br>+   |
|   | Clear  | 03-         | + + + + + + + + + + + + + + + + + + +   |
| Indentation parameters  | Estimated time Estimated memory size 0:26:05 1286.0 KB |             | +   |
| + Advanced<br>Acquisition Rate : 10.0 [Hz]<br>Linear loading  | *  | -1.4 mm     | n   |
| Max load : 50.00 mN<br>Loading rate: 100.00 mN/min<br>Unloading rate : 100.00 mN/min<br>Pause : 5.0 s   |  | F           | Red cross:     selected definition in the list       Blue cross(es):     ADO definition(s) of the list  |
| + Step - NHT S/N: 1234567890 settings<br>Approach distance: 1500 nm<br>Approach speed: 2000 nm/min<br>Dz sensor in fine range<br>Stiffness threshold: 500 μN/μm |  | E           | Light blue cross:selected ADO definition in the listBlack cross(es):remaining definition(s) of the list |
| Section 3.7.7   | <b>v</b>   |             | Section 3.8   |

Fig.62 Advanced matrix window parameters

The corresponding position of each measurement definition from the list is represented with a cross in the *Indentation position* graph. Each measurement definition is 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.63).

<Add> button:

To create a new definition (type/parameters/position) at the bottom of the list.

<Copy> button:

To copy the selected (highlighted) definition at the bottom of the list. If necessary, the definition can be modified (see <Edit> button below).

<Edit> button:

To modify the selected (highlighted) definition in the list.



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

## <Remove> button:

To remove the selected (highlighted) definition(s) from the list - Several definitions can be selected by pressing <Ctrl> (or <Shift>) key prior to clicking.

### <Clear> button:

To clear all definitions from the list.



A *Warning* window asks to confirm one of the previous deletions (<Yes> button) or not (<No> button).

When one of the <Add>, <Copy> or <Edit> buttons previously described is clicked.



Fig.63 Indentation position window parameters

For each current (selected) definition:

```
Delta X field:
```

To set a relative position for the X axis.

*Delta Y* field:

To set a relative position for the Y axis.

<<u>E</u>dit> button:

To select one of the following measurement types.



 Adjust Depth Offset
 3.7.5.12

 Standard
 3.7.5.1

 Sinus
 3.7.5.3

 Progressive multicycle
 3.7.5.5

 User define profile
 3.7.5.7

Measurement type:

To select (highlight) a measurement type.

<<u>O</u>K> button:

To open the corresponding window of the selected measurement type.

Double clicking a measurement type does the same effects.

The corresponding parameters should be set as same as described in the following sections.

| Advanced            | 3.7.5.2 |
|---------------------|---------|
| Constant multicycle | 3.7.5.4 |
| СМС                 | 3.7.5.6 |

Matrix of matrix is possible,

Simple matrix3.7.5.8Quick matrix3.7.5.10Advanced matrixa 2<sup>nd</sup> window as same as Fig.62 is superimposedVisual advanced matrix3.7.5.11

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

**INFORMATION:** 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>) key and select (highlight) several definitions in the list.

## 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 the 2 settings (above).

Х

For the Y axis





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

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

# To set same preferences (hardware parameters) for several measurement definitions

To set in one time the same hardware parameter preferences for several measurement definitions, see the following descriptions.

| Indentation definition                        |
|---|
| Standard [0 ; 0]                              |
| Standard [1 ; 1]                              |
| Advanced [1.5;1]                              |
| Advanced [2; 2]                               |
| Advanced [3;1]                                |
| User defined profile [2; 1.5]                 |
| Adjust Depth Offset [7 ; 5]                   |
| Constant multicycle [7.5 ; 6]                 |
| Standard [8 ; 6]                              |
| Changes preferences for selected indentations |

Press <Ctrl> or <Shift> key and select (highlight) several measurement definitions in the list.

Then right click in the definition list area and select "Changes preferences for selected indentations" from context menu.

The same parameters (in *Hardware parameters* window / *Preferences* tab) described for the corresponding head below can be modified for all selected definitions in the list:

| UNHT     | section 3.7.6.1 |
|----------|-----------------|
| UNHT Bio | section 3.7.6.2 |
| NHT      | section 3.7.6.5 |
| MHT      | section 3.7.6.6 |

Once the parameters are validate (<<u>O</u>K> button is clicked in the *Preferences* tab),



A *Confirmation* window asks if the new parameters should be applied. Click <Yes> button to apply the modifications. Otherwise click <No> button to not apply the modifications.
# 3.7.5.10 Quick matrix

The *Quick matrix* measurement type is similar to *Simple matrix* section 3.7.5.8, it performs a matrix of identical indentation measurements. However between each measurement of the matrix, the reference stays in contact with the sample to speed up the approaches (and therefore the measurements).

Set the proposed parameters for **1 cycle** of linear loading/unloading (section 3.7.3.1).

**INFORMATION:** It is not possible to use the quick matrix with the MHT.

| Quick matrix  | · · · · · ·  |
|---|--|
| Quick matrix  |  |
| Indentation parameters  | Profile description  |
| Max load<br>300.00 ♥ µN Sinus   |  |
| Time to load / unload     Sinus frequency       1.0     s       20.0     Hz | ,  |
| Pause   | Hardware parameters  |
| 0.0 💽 s   | + Step - UNHT S/N: 123456 setting<br>Fn contact : 300 µN             |
| Indentation matrix definition   | Approach distance : 1500 nm<br>Approach speed : 2000 pm/min          |
| Delta X         Delta Y           100.000 ♀         μm                      | Dz sensor in fine range<br>Load in fine range<br>FnRef in fine range |
| Indentation count X Indentation <u>c</u> ount Y                             | FnRef contact load : 500.000 μN<br>Stiffness threshold : 150 μN/μm   |
| Distance X Distance Y 400 μm 300 μm   |  |
| Section 3.7.2   | Section 3.7.6  |
| 16 0:07:32 3981.3 KB  | Change   |
|   | Edit <u>a</u> djust depth offset parameters                          |
| Section 3.7.7   | Section 3.8  |
| Ave as protocol   | V <u>O</u> K X <u>C</u> ancel  |

Fig.64 Quick matrix window parameters

#### **Indentation parameters area**

Max load field:

To set the max. load to be applied.

#### Time to load/unload field:

To set the time to reach the max. load (above) and also to fully unload after the pause (below).

Pause field:

To set a (linear) pause at the max. load (above) before unloading. Value sets to 0 = no pause.

Sinus box:

To load with a sinus wave, check this box



Sinus frequency field (active if Sinus box above is checked): To set the sinus wave frequency for the loading.

# Indentation matrix definition area

Refer to the same <u>Indentation matrix definition area</u> described in the *Simple matrix* measurement type, p. 103.

# <Edit adjust depth offset parameters> button

To set the parameters for the mandatory ADO (section 3.4.4.1). The ADO automatic process (section 3.4.4.2) begins once the matrix of measurements is started (< OK > button Fig.64).

# 3.7.5.11 Visual advanced matrix

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



The *Table displacement Warning* window asks to move the sample under the microscope. Before clicking <Yes> button, verify the sample height to avoid any collision. Clicking <Yes> button opens the following *Visual advanced matrix* (Fig.65) and *Video* windows (Fig.66).

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>2</sup> With the following Visual advanced matrix window (Fig.65)

<sup>&</sup>lt;sup>1</sup> With the following Video window (Fig.66)



Fig.66 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

- <<u>A</u>dd> button.

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. 113.

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. The selected (highlighted) definition in the list appears with a red square.

#### Parameters for each selected measurement definition

| List of indentations                   | List of indentations                  |
|--|---------------------------------------|
| Adjust Depth Offset [10.164 ; 16.881]  | Adjust Depth Offset [10.164 ; 16.881] |
| Standard [10.157 ; 16.78]              | Standard [10.211 ; 16.729]            |
| Standard [10.273 ; 16.787]             | Standard [10.157 ; 16.78]             |
| Standard [10.211 ; 16.729]             | Standard [10.273 ; 16.787]            |
| Adjust Depth Offset [12.11 ; 16.939]   | Adjust Depth Offset [12.11 ; 16.939]  |
| Advanced [12.106 ; 16.78]              | Advanced [12.106 ; 16.78]             |
| Drogressive multicycle [12:362 + 16:07 | Drogressive multicycle [12:362 + 16:0 |

To change the chronologic order of the measurements, select (highlight) any definition, drag it and drop it elsewhere in the list.

#### Measurement type/parameters

<<u>E</u>dit> button: To modify the measurement type of the selected (highlighted) definition in the list (Fig.65).

| Select the indentation type       |
|-----------------------------------|
| Indentation type                  |
| Adjust Depth Offset               |
| Standard                          |
| Advanced                          |
| Constant multicycle               |
| Progressive multicycle            |
| CMC                               |
| User defined profile              |
| Simple matrix<br>Matrix of matrix |
|                                   |
| Advanced matrix                   |
| Visual advanced matrix            |
|                                   |
|                                   |
|                                   |
| V <u>O</u> K <u>C</u> ancel       |

Measurement type:

To select (highlight) a measurement type.

<<u>O</u>K> button:

To open the corresponding window of the selected measurement type.

Double clicking a measurement type does the same effects.

The corresponding parameters should be set as same as described in the following sections.

| Adjust Depth Offset    | 3.7.5.12 |                     |         |
|------------------------|----------|---------------------|---------|
| Standard               | 3.7.5.1  | Advanced            | 3.7.5.2 |
| Sinus                  | 3.7.5.3  | Constant multicycle | 3.7.5.4 |
| Progressive multicycle | 3.7.5.5  | СМС                 | 3.7.5.6 |
| User define profile    | 3.7.5.7  |                     |         |

Matrix of matrix is possible,

| Simple matrix          | 3.7.5.8  |
|------------------------|--|
| Quick matrix           | 3.7.5.10   |
| Advanced matrix        | 3.7.5.9  |
| Visual advanced matrix | a 2 <sup>nd</sup> window as same as Fig.65 is superimposed |

#### <u>Position</u>

<Edit co-ordinates> button:

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.



- <<u>R</u>emove> button: To remove the selected (highlighted) measurement definition(s) from the list - Several definitions can be selected using <Ctrl> or <Shift> key before selecting (the definition square(s) also disappear from the *Video* screen).
- <<u>C</u>lear> button: To clear all measurement definitions from the list (and from the *Video* screen).

# To set same preferences (hardware parameters) for several measurement definitions

List of indentations

| Adjust Depth Offset [10.164 ; 16.881]        |
|--|
| Standard [10.157 ; 16.78]                    |
| Standard [10.273 ; 16.787]                   |
| Standard [10.211 ; 16.729]                   |
| Adjust Depth Offset [12.11 ; 16.939]         |
| Advanced [12.106 ; 16.78]                    |
| Progressive multicycle [12.362 ; 16.937]     |
| Sinus [12.312 ; 16.742]                      |
| Changes preferences for selected indentation |
|  |

Same as described in <u>To set same preferences</u> (hardware parameters) for several measurement definitions for the *Advanced Matrix*, p. 108.

#### Absolute reference position for protocol

*Use absolute position for the map* box.

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 change the reference</u> position, p. 115.

Uncheck to position the sample at the current position.



To move the sample/motorized table(s) use the *Position control* area; for descriptions, refer to the *Common Scratch & Indentation software reference guide* in section *Managing the instrument / Position control*, exception: Some functions are not applicable here.

OR

#### Methods to move

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.

<Set reference> Button: To set a new reference at the current sample/motorized tables position.



<Edit reference> To manually set a new reference to another position:



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

**INFORMATION:** 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

<<u>O</u>pen video> To reopen the *Video* window if it has been closed. button

#### <u>Ellipse</u>

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



List of indentations Adjust Depth Offset [-578.165 ; 462.607] Standard [-217.587 ; 422.813] Standard [244.945 ; 457.63] Standard [538.379 ; 72.125] Standard [371.769 ; -368.096] Standard [-277.266 ; -330.787] Standard [-488.654 · 77.102]

<Ellipse> button: To define the following ellipse parameters.



Fig.67 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 (*Points* field in *Angle* area) in 3 rows (*Points* field in *Radius* offset area) = 30 definitions.

When < OK > button is clicked in Fig.67, the new indentation definitions replace the previous ones in list (ADO definition(s) is kept in the list).

List of indentations



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

# 3.7.5.12 Multi-ADO for Advanced matrix & Visual advanced matrix

**REMINDER:** For the ADO purpose, read the beginning of the section 3.4.

One or several automatic ADO(s) can be performed at the beginning and/or between the indentation measurements for the *Advanced matrix* (section 3.7.5.9) or *Visual advanced matrix* (section 3.7.5.11).

| Advanced<br>Constant multicycle<br>Progressive multicycle<br>CMC<br>User defined profile<br>Simple matrix<br>Advanced matrix<br>Quick matrix<br>Visual advanced matrix |
|--|
| Constant multicycle<br>Progressive multicycle<br>CMC<br>User defined profile<br>Simple matrix<br>Advanced matrix<br>Quick matrix<br>Visual advanced matrix             |
| Progressive multicycle<br>CMC<br>User defined profile<br>Simple matrix<br>Advanced matrix<br>Quick matrix<br>Visual advanced matrix                                    |
| CMC<br>User defined profile<br>Simple matrix<br>Advanced matrix<br>Quick matrix<br>Visual advanced matrix  |
| User defined profile<br>Simple matrix<br>Advanced matrix<br>Quick matrix<br>Visual advanced matrix   |
| Simple matrix<br>Advanced matrix<br>Quick matrix<br>Visual advanced matrix   |
| Advanced matrix<br>Quick matrix<br>Visual advanced matrix  |
| Quick matrix<br>Visual advanced matrix   |
| Visual advanced matrix   |
|  |
|  |
|  |
|  |
| 🗸 <u>O</u> K 🗙 <u>C</u> ancel  |

Select (double click) the *Adjust Depth Offset* (ADO) measurement type and set its parameters (section 3.4.4.1).

- Fig.68 ADO selection
- 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.



**INFORMATION:** Before starting the matrix of measurements, if there is not at least an ADO in the first position of the list, a Confirmation window asks to continue:

Click <Yes> button if a successful ADO & has previously been performed (section 3.4.2) and is suitable (location) for the whole matrix area. Otherwise click <No> button and add an ADO definition in the first position of the list, and elsewhere in the list where it is necessary.

# **3.7.6 MEASUREMENT HARDWARE PARAMETERS (HEAD)**



Sensor ranges tab(s) according to the measuring head

Fig.69 Measurement schematic

See the following sections which described the hardware parameters used during the measurement(s).

Some preference parameters are dedicated<sup>1</sup> for each type of measuring head and some are common<sup>2</sup> for all types of measuring heads.

The preference preset feature<sup>3</sup> allows setting automatic suitable values according to the kind of sample material in use.

- <sup>1</sup> UNHT (sections 3.7.6.1, 3.7.6.3 and 3.7.6.9), UNHT Bio (sections 3.7.6.2, 3.7.6.4 and 3.7.6.9), NHT (section 3.7.6.5), MHT (section 3.7.6.6 and 3.7.6.9)
- <sup>2</sup> Approach distance (section 3.7.6.7), Contact stiffness threshold (section 3.7.6.8), Adhesion (section 3.7.6.10)
- <sup>3</sup> Preset (section 3.7.6.11)

# 3.7.6.1 UNHT Preferences tab



Fig.70 E.g. Step - UNHT with default (presets) parameters

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

#### **Indenter**

Approach speed

The <u>Approach speed</u> field determines the velocity at which the indenter approaches the sample surface.

See the typical values in the table below:

| 1000 to 2000 mm (min | For max. measurement load | Material            |
|----------------------|---------------------------|---------------------|
|                      | 20 to 1000 µN             | fused silica (hard) |

Tendency:

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

```
Contact load
```

The indenter *Contact load* field 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 section 3.7.6.9.

#### **Reference**

Approach speed

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

Contact load

The reference *Contact load* field 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 μN</b> | 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 | Check the reference-indenter auto-tuning box to perform an auto adjustment of the pre-approaches between the<br>Pre-approach |
|--------------------------------|--|
|                                | reference and the indenter: the indenter 50 % and<br>Pre-approach  |
|                                | reference 40 8 % become inactive. According to the last  |
|                                | successful ADO, the software will compute and optimize the best indenter and reference pre-approaches.                       |

**INFORMATION:** 100 % is the lower/rest position.

# 3.7.6.2 UNHT Bio Preferences tab



Fig.71 E.g. Step - UNHT Bio with default (presets) 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 UNHT<sup>3</sup> Bio User Manual** in section **Indentation measurement process / Performing an indentation measurement / Define indentation measurement parameters**.

# 3.7.6.3 UNHT Sensor ranges tab



Fig.72 E.g. Step - UNHT 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.

**INFORMATION:** This tab is also available in the Calibration window (section 2.4.1).

#### 3.7.6.4 UNHT Bio Sensor ranges tab



Radio buttons related to the reference are inactive as there is no reference on the UNHT Bio Similar than UNHT section 3.7.6.3, **exceptions:** 

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

# 3.7.6.5 NHT Preferences tab

| Step -          | NHT - Hardware parameters (S   | ame parameters for                                | TTX)  |
|-----------------|--|---|---|
| Prefe           | rences   |   |   |
| Soction 3.7.6.8 | trument approach parameters<br>pproach speed<br>2000.0 nm/min<br>ontact stiffness threshold<br>500.0 uN/um | Approach distance<br>1500.0 nm<br>Section 3.7.6.7 | Depth range<br>● 40 µm ← Fine<br>○ 200 µm ← Large |
|                 |  |   |   |
|                 | nm/min<br>● ● nm/min<br>Section 3.7.6  | Retract time 0 sec                                |   |
| Pre<br>De<br>Se | esets<br>efault<br>action 3.7.6.11   | ~ C   |   |
|                 |  | ×   | <u>O</u> K X <u>C</u> ancel                       |

Fig.73 E.g. Step - NHT with default (presets) parameters

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

Approach speed

The <u>Approach speed</u> field determines the velocity at which the indenter approaches the surface.

See typical values in the table below:

| Hard mater                        | Soft material |               |
|-----------------------------------|---------------|---------------|
| < 1 mN                            | > 1 mN        | Solt material |
| <b>1000</b> to <b>1500 nm/min</b> | 2000 nm/min   | 4000 nm/min   |

Depth range

© 40 μm

🔘 200 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.

# 3.7.6.6 MHT Preference tab

|               | Step - MHT - Hardware parameters   |   | 7     |
|---------------|--|---|-------|
|               | Preferences  |   |       |
|               | Instrument approach parameters<br>Approach speed   | Indentation range<br>+ Load range + Depth range |       |
|               | Contract d'fferent brack ald   |   | ⁻ine  |
| Section 3.7.6 | .8 10000.0 ♥ μN/μm 0.010 ♥ N   | ○ 30 N ◀····· ○ 1000 µm ◀·····                  | Large |
|               | Retract speed Retract time<br>0.0 	µm/min 0 	set<br>Presets<br>Default ✓<br>Section 3.7.6.11 | 20  |       |
|               |  | ✓ <u>O</u> K X <u>C</u> ancel                   |       |

Fig.74 E.g. Step - MHT with default (presets) parameters, except fine ranges

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

Approach speed

The <u>Approach speed</u> field 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     |
| 8 or 16.6 µm/min |                 | 16.6 µm/min |

Contact load

The **Contact load** is used to detect the sample surface during the approach; see also section 3.7.6.9.

See the typical values in the table below:

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

Load range Select suitable Load range and Depth range according to the 1) used sample/application; it is recommended to use fine ranges (e.g. 10 N above) to obtain the optimal quality of the signals. Use the large 30 N ranges only if the fine ranges are not sufficient to perform the measurement, or in case of saturation. + Depth range 1) **IMPORTANT:** The Depth range should be the same as the ADO Dz 100 μm
 10 range, p. 75 which has been selected for the current successful 1000 μm
 ADO. Otherwise an error message will appear; see section 5.2.

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

# 3.7.6.7

Approach distance

The *Approach distance* field is only available for the measuring 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* tab; see from section 3.7.6.

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

**INFORMATION:** *The value of* the presets corresponding to the type of the used sample (section 3.7.6.11) is suitable.

#### MHT approach distance

Otherwise for MHT <u>without</u> electronic bridge, this parameter can be only changed in the current ADO parameters window; see section 3.4.7. Therefore the approach distance will remain the same for each following measurement.

Approach distance parameter (UNHT/Bio/NHT)

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.75), after the measurement has been performed.



Fig.75 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 section 4.1.1.

# 3.7.6.8 Contact stiffness threshold parameter (All heads)

All heads use this parameter to detect the sample surface with the indenter.

Contact stiffness threshold

#### Contact stiffness threshold field:

To set the stiffness threshold at which the contact between the indenter and sample surface should be detected (detection sensitivity with a stiffness algorithm).

It is recommended to set the value of the preset corresponding to the type of the used sample (section 3.7.6.11). If necessary (for more accuracy), it is possible to slightly decrease the preset value; **IMPORTANT:** *However by decreasing too much the value, the contact can be detected in the air.* 

**Except** NHT, other heads also use the following *Contact load* parameter (section 3.7.6.9) to detect the sample surface. 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).

# 3.7.6.9 Contact force parameter (not with NHT)

Additionally to the *Contact stiffness threshold* parameter (section 3.7.6.8), the UNHT, UNHT Bio and MHT instruments also use the *Contact load* field (unit selected in options) to detect the sample surface with the indenter/reference (UNHT). 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).



Contact load field:

To set the force at which the contact between the indenter/reference(UNHT) and sample surface should be detected.

# 3.7.6.10 Retract speed & time parameters for adhesion



🜲 sec

Retract time

<u>Retract speed</u> and <u>Retract time</u> field values can be changed for each new measurement in <u>Preferences</u> tab; see from section 3.7.6.

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 section 4.6 to obtain the adhesion force.

# 3.7.6.11 Preset of the preference parameters

In each *Preferences* tab of the *Hardware parameters* window (see from section 3.7.6) there is the **"Presets"** drop-down menu located at the left bottom of the window. Suitable automatic values according to the kind of sample material in use can be set with this feature.

| Preferences Sensor ranges  |  |  |
|--|--|--|
| Indenter approach parameters<br><u>Approach speed</u><br>2000.0 	mildown nm/min<br>Pre-approach<br>10 	mildown %<br>Contact stiffness threshold<br>150.0 	mildown µN/µm<br><u>R</u> etract speed | Approach distance<br>1500.0 ♥ nm<br>Contact load<br>300.000 ♥ µN<br>Retract time | Reference approach parameters<br><u>Approach speed</u><br>60000.0 	mtextbf{nm/min}<br>Contact load<br>500.000 	mtextbf{pm} µN<br>Pre-approach<br>40 	mtextbf{mm} %<br>Reference-indenter auto-tuning |
| 2.0 € nm/min<br>Video<br>☑ Optical analysis after meas<br>Presets<br>Default   | urement  | Active   |

| Presets              |   |   |   |
|----------------------|---|---|---|
| Default              |   | ~ | G |
| Default              |   |   |   |
| Soft materials       | N |   |   |
| Ultra soft materials | 3 |   |   |

"Presets" drop down menu:

To select one of the 3 following presets according to the material of the used sample,

```
"Default"
For hard material.
```

OR

"Soft materials"

OR

"Ultra soft material"

<'Apply'> 🧲 button:

To apply the current **"Presets"** drop-down menu selection  $\rightarrow$  The parameters are automatically set accordingly.

**INFORMATION:** For the UNHT/bio, the indenter and reference Pre-approach fields automatic values are defined in the Default values tab of the Calibration window (section 2.4.3).

# **3.7.7** SAVE AS PROTOCOL (CURRENT MEASUREMENT TYPE SETTING)



Located at the left bottom of each measurement type window (see from section 3.7.5) To save the current setting of all parameters as a measurement protocol (including hardware preference parameters section 3.7.6).

| Edit a protocol name Protocol name Protocol example                               | Edit (type) a new protocol name and click ok ok to save the protocol.  |
|---|--|
| Use a protocol Protocol example Protocol example 2 Protocol list                  | Each saved<br>protocol will appear<br>in the protocol list<br>of the <i>Define a new</i><br><i>measurement</i><br>window (Fig.49)<br>and can be selected<br>later.                                       |
| Use a protocol      Protocol example Protocol example 2      Protocol list      E | To erase a protocol,<br>only from the list of<br>the <i>Define a new</i><br><i>measurement</i><br>window (Fig.49),<br>right click on a<br>protocol and select<br><b>"Delete"</b> in the<br>context menu. |

# **3.8** MEASUREMENT PROCESS (INDENTATION RUNNING)

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



Fig.76 Indentation running... window

**IMPORTANT:** 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 ( $\blacksquare$  Indentation running...),  $\blacksquare$  Stabilization 30 [s] is blinking and displays a decreasing time info (e.g. starts from 30 [s]); the stabilization time is set in section 2.1.4.2.

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.

**INFORMATION:** With UNHT and UNHT Bio, the extra UNHT Approach Monitor window (Fig.33) can be displayed or deactivated with the UNHT.INI file; see section 2.6.

#### 3.9 ANALYZING/VISUALIZING THE INDENTATION(S)

#### This section is applicable if:



Optical analysis after measurement

- *Microscope installed* box has been checked and <Video Microscope> radio button has been selected (providing the video microscope is present on the instrument); refer to the Common Scratch & Indentation software reference quide in section Managing the instrument / Hardware configuration / My configuration tab.

Optical analysis after measurement box has been checked in the UNHT or UNHT Bio - Hardware parameters window / Preferences tab (respectively sections 3.7.6.1 or 3.7.6.2).

| Wa | arning | ×   |
|----|--------|---|
|    |        | Do you want to move under the microscope to visualize your<br>indentation ?<br>Check sample height to avoid collision |
|    |        | Yes No  |

At the end of the indentation measurement process: when the Indentation running ... window (Fig.76) closes, this Warning window appears.

Click <Yes> button to open,

the Video window (Fig.78) and Analyze indentation window (Fig.77), in order to (if necessary):

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

Or click <No> button and go to section 4.



Fig.77 Analyze indentation window



To reopen the Video window.

#### Selecting an indentation

Indentation definition Standard [0 ; 0] Standard [0.1 ; 0] Standard [0.2 ; 0] E.g. if a matrix of measurements has been performed, select an indentation in the list to automatically move to the corresponding area.

# Methods to move for adjustment

Speed Very Fast Fast Medium Slow Very slow Use these functions to adjust the sample position; refer to the **Common Scratch & Indentation software reference guide** in section **Managing the instrument / Position control** (some described functions are not available here).

# **INFORMATION:**

 <PgUp> and <PgDn> GUI buttons appear in *keyboard control* area when a motorized Z table is present on the instrument (e.g. not the case here with a TTX)

- 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).



Click <Yes> button to confirm.

#### **3.9.1 REFINING INDENTER-MICROSCOPE (OBJECTIVE IN USE) DISTANCE** CALIBRATION

It is possible to refine the calibration of the distance between the indenter and the video microscope (only for the objective into working positon) from this *Analyze indentation* window (Fig.77), and also using the *Video* window (Fig.78).

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 <u>Methods to move for adjustment</u> above, precisely center the chosen indentation imprint under the *Video* screen crosshair and properly adjust the focus using <PgUp> / <PgDn> keys in case of motorized Z table (Fig.78). **IMPORTANT:** *Be sure the imprint corresponds to the selected indentation in the list.* 

| Keyboard control           |    |   |
|----------------------------|----|---|
| Forward                    |    |   |
| < Left Aight >             |    |   |
| •                          |    |   |
| Backward                   |    | _ |
| Minimum displacement 0.500 | μm | ◄ |

洸

**INFORMATION:** To have the crosshair in the middle of the Video screen, check Crosshair box in the Video window; see **Video software reference guide** for description of all features.



Fig. 78 Indentation imprint centered under Video screen crosshair

To refine the calibration of the distance between the indenter and the objective into working position, only when the indentation imprint is centered (*Fig.78*). The calibration is not applied to the other objective(s).

| Table displ | acement Warning   |
|-------------|---|
| <b></b>     | A backlash movement is recommended before storing the new<br>calibration ?<br>Proceed now ? |
|             | Yes No  |
| Confirmatio |   |
| Comman      |   |
| 2           | Do you want to save this point as new distance microscope calibration ?                     |
|             |   |

It is recommended to click <Yes> button in order the software performs a backlash movement before storing the new calibration; wait.

Click <Yes> to save the new calibration **X**, **Y** and **Z distance** values<sup>1)</sup> in the corresponding fields of the Calibrate Indenter Microscope distance window<sup>2)</sup>

Otherwise click <No> button and restart from <u>Centering</u> , p. 135 until the imprint is centered.

- <sup>1</sup> X, Y distances between the indenter and objective into working position and Z distance, in case of motorized Z table, between the sample surface and objective into working position (focus).
- <sup>2</sup> Refer to the Common Scratch & Indentation software reference guide in section Managing the instrument / Calibration (Common tabs) / Indenter-microscope distance calibration procedure.

# **3.9.2 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. 135, the features below can be used:

#### **Contact point**



Curve toolbar

If necessary, the contact point can be refined from here  $\checkmark$ . Otherwise it can be refined later during the result analysis; see the similar explanations as described from section 4.1.1.1.

#### Grab image(s)

Each of the following active buttons from this *Analyze indentation* window (Fig.77) allows image capture(s) from the current *Video* screen (Fig.78): *Edit an image* window appears (Fig.79).

Before capturing image(s) below, ensure the focus is adjusted (with Z motorized table, see <u>Methods to move for adjustment</u>, p. 135 or 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; see <u>MultiFocus image(s)</u>, p. 138.

|     | Conscan image |
|-----|---------------|
| AFM | AFM image     |

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

This window below allows some settings on the opened image (Title name, comments, image features...); refer to the *Common Scratch & Indentation software reference guide* in section *Measurement Documents in Curve View / Manipulating Document windows / Image Gallery*.



Fig.79 Edit an image

When v is clicked, each image is stored (as thumbnails) in the image gallery of Fig.79 but also of the *Analyze indentation* window (Fig.77); 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.







# **3.9.3 OPTICAL VICKERS HARDNESS ANALYSIS**

First adjust the optical hardness Vickers measurement: Place the cursors (center and 4 edges) over the Vickers measurement imprint on *Video* screen (Fig.80); refer to the *Video software reference guide* in section *Using the Video Software / Hardness (Indentation)* (selecting Vickers measurement).



Fig.80 Vickers optical measurement adjusted on Video screen = Vickers analysis

(Do not close the Video window)



<Hardness analysis> button (Fig.77):

To save the current optical Vickers analysis (Fig.80) in the 2 following places.

- The optical Vickers analysis is saved in the image gallery (Fig.77). When the image is opened, *Edit an image* window (Fig.79) shows the analysis details.



- The optical Vickers analysis is also saved later in the analysis of results (section 4.5). **INFORMATION:** *This analysis cannot be defined as automatic or added manually afterwards, it is coming only from Analyze indentation window (Fig.77)* 



#### **3.9.4** ENDING THE OPTICAL ANALYSIS





# **3.10** SAMPLE DISPLACEMENT AFTER THE MEASUREMENT(S)

After the measurement(s) have been performed (section 3.8) or after visualizing the indentation(s) (section 3.9), the following window appears.

**IMPORTANT:** To avoid performing the next calibration/measurement indent inside the current indent (current position), the sample/motorized table(s) position should be shifted of a minimum displacement<sup>1</sup>

| Sample displacement after Measu                                | irement           |            |           |        |
|--|-------------------|------------|-----------|--------|
| It is recommended to move the zone: set a minimum displacement | sample awa<br>ent | y from the | measure   | ment   |
| X motorized table  |                   | -Y motori  | zed table | 1      |
| 0.500 🖝 mm   |                   |            | 0.000     | mm     |
|  |                   |            |           |        |
|  | $\checkmark$      | ОК         | X         | Cancel |

Fig.81 Sample displacement after measurement

#### X motorized table field:

To set a min. displacement value<sup>1</sup> for the motorized X table.

#### Y motorized table field:

If necessary, to set a min. displacement value<sup>1</sup> for the motorized Y table.

<OK> button:

To move the motorized table(s) to the position values set in the previous fields; wait.



<Cancel> button:

To cancel the displacement (no move). However it is recommended to then use the *Position control* window to move the sample into another suitable position; refer to the *Common Scratch & Indentation software reference guide* in section *Managing the instrument / Position control*.

<sup>1</sup> It depends on the current sample material: Approx. 20 x h<sub>m</sub> Pay attention to the displacement unit (e.g. mm in Fig.81), which is set in the options; refer to the Common Scratch & Indentation software reference guide in section Customizing options / Preferences tab (units).

# **3.11 HEATING MODULE MODE**

# **3.11.1 ACTIVATION**

This mode and following descriptions are applicable only if:

- <Heating stage> radio button is selected for UNHT (section 2.1.1.2)
- *Heating stage* box is checked for MHT (section 2.1.1.4)

# 3.11.2 ADO

The *Temperature area* appears in the *ADO* window (Fig.82). For the standard ADO descriptions and parameters, see section 3.4

| Adjust depth of | offset parameters  |            |
|-----------------|--|------------|
|                 | Surface detection parameters                             |            |
| <b>1</b>        | Approach speed Contact force                             |            |
|                 | 200.0 📦 µm/min 0.03 📦 N                                  |            |
|                 | Characterization force Contact stiffness threshold       |            |
|                 | 1.000 ♥ N 10000.0 ♥ μN/μm                                |            |
|                 | Approach distance Dz range                               |            |
|                 | 5.0 μm Ο 100 μm  |            |
|                 | Indenter   |            |
|                 | Pre-approach   |            |
|                 | 10 💽 %   |            |
|                 | Temperature  |            |
|                 | heating mode enabled                                     |            |
|                 | Sample target temperature Heating slope Temperature stat | pilization |
|                 | 25.0 € °C 20.0 € °C/min 0.10 € °                         | C/min      |
|                 | ☐ Turn off heaters after measurement                     |            |
|                 |  |            |
|                 | Presets  |            |
|                 |  | Apply      |
|                 | 🗸 ок 🗙   | Cancel     |

Fig.82 E.g. MHT ADO window with heating module mode

Heating mode enabled box

To enable the heating of the module before performing the ADO process, check this box (communication with *Heater Software*). Otherwise uncheck (default) to perform a (standard) ADO at ambient temperature.

*Sample target temperature* field

To set the target temperature value that will be automatically set and applied in *Target temperature* field of the *Heater Software*.

| <i>Heating slope</i><br>field                 | To set the heating slope value that will be<br>automatically set and applied in <i>Slope</i> field of<br>the <i>Heater Software</i> .   |
|---|---|
| <i>Temperature stabilization</i> field        | To set the stabilization temperature to wait before the ADO process starts.   |
| <i>Turn off heaters after measurement</i> box | To turn off the heater of the module after the<br>last measurement of any started matrix<br>measurement types (section 3.6), check this<br>box (default).<br>Otherwise uncheck to keep the heating<br>module at the target temperature until<br>manually turned off by the user through the<br><i>Heater Software</i> .                 |
| <ok><br/>button</ok>                          | If the previously described <i>Heating mode</i><br><i>enable</i> box is checked, the <i>Heater Software</i><br>should be opened by the user before clicking<br>this button (otherwise, a <i>Confirmation</i><br>window appears).<br>Then, to heat the module according to the<br>previously described parameters, click this<br>button. |

#### **3.11.3 TEMPERATURE SCAN MEASUREMENT TYPE**

The *Temperature scan* is active in *Define a new measurement* window (section 3.6) at the very bottom.

| Define a new measurement<br>Standard<br>Advanced<br>Sinus<br>Impact Mode | ^        |  |
|--|----------|--|
| Standard<br>Advanced<br>Sinus<br>Impact Mode                             | ^        |  |
| Advanced<br>Sinus<br>Impact Mode   |          |  |
| Sinus<br>Impact Mode   |          |  |
| Impact Mode  |          |  |
|  |          |  |
| Constant multicycle  |          |  |
| Progressive multicycle   |          |  |
| CMC  |          |  |
| User defined profile   |          |  |
| Simple matrix  | 1.1      |  |
| Advanced matrix  | 1.1      |  |
| Quick matrix   | <b>+</b> |  |
| Visual advanced matrix   |          |  |
| Temperature Scan 🔊   |          |  |

Select this measurement type.
| Temperature Scan                               |                              | - Indentation parameters  |
|--|------------------------------|---|
| Temperature min                                | Temperature max              | Edit Indentation parameters   |
| 25 ▼     Temperature steps     25.0 ▼ °        | Indentation repetition       | Temperature Scan<br>2 X 4 indentations<br>Delta X : 1.000 mm, Delta Y 0.000 mm<br>Indentation parameters<br>+ Standard                            |
| Indentation matrix definition Delta X 1.000 mm | Delta <u>Y</u><br>1.000 🗭 mm | Acquisition rate : 10.0 [Hz]<br>Linear loading<br>Max load : 1.00 N<br>Loading rate : 2.00 N/min<br>Unloading rate : 2.00 N/min<br>Pause : 10.0 s |
| Indentation count X                            | Indentation <u>c</u> ount Y  | Fn contact : 0.01N<br>Approach speed : 16.6 µm/min<br>Z table retract : 1.5 mm<br>Dz sensor in large range<br>Load in large range                 |
| 2 mm   | Ji <u>stance r</u><br>3 mm   | Edit <u>a</u> djust depth offset parameters   |
| Bave as protocol                               |                              |   |

Fig.83 Temperature scan parameter window

The standard parameters are similar to the ones described for the *Simple matrix* measurement type (section 3.7.5.8), except for the following ones.

| <i>Temperature min</i><br>field     | To set the minimum temperature for each measurement.   |
|-------------------------------------|--|
| <i>Temperature max</i> field        | To set the maximum temperature for each measurement.   |
| <i>Temperature steps</i> filed      | To set the desired delta temperatures between the min<br>and max (previously described); the software adjusts<br>automatically the different temperatures (based on the<br>steps value) and performs the measurement at each<br>temperature.   |
| <i>Indentation repetition</i> field | To set the number of measurements (defined through $<\underline{E}$ dit Indentation parameters> button). The distance between each measurement performed at the same temperature is defined in <i>Delta X</i> field and the distance between measurements performed at different temperature steps is defined in <i>Delta Y</i> field. |
| <ok> button</ok>                    | Before clicking this button, ensure that the <i>Heater</i><br><i>Software</i> is opened.<br>Then, to start the defined temperature scan matrix<br>measurements, click this button.   |

Later, the statistics view allows to display the results versus temperatures (Refer to the *Common Scratch and Indentation software reference guide* in section *Measurement documents in statistics view*).

# **4 ANALYSIS OF RESULTS**

After the measurement process of the indentation(s) is completed, the main result analysis window appears (Fig.84).

Adjustments (analysis method, curve display, contact point, overlays...) and parameter changes (indenter, Poisson's ration...) can be performed in 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  $\boxed{\mathbb{M}}$  is selected on the main toolbar.

For the main toolbar (1) and [1]; refer to the **Common Scratch & Indentation software reference guide** in section **Measurement documents in curve view** / **Document window interface / Information/measurement tabs / Measurement tab**.



Fig.84 Result analysis window (curve view default setting)

#### Additional toolbar



# 4.1 INDENTATION CURVE(S)

For each active measurement

Λ

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

- The displayed indentation curve(s) (section 4.1.2)
- The indentation curve features:

Contact point(s) (section 4.1.1) Smoothing (section 4.1.3) Export (section 4.1.4)

The indentation curve(s) and features can be hidden (if unselected). Only if the analysis curve(s) is active and selected  $\boxed{\swarrow} /$  (section 4.7).

# 4.1.1 SETTING THE CONTACT POINT

From the result analysis window (Fig.84), there are several ways to open the *Set the contact point* window (Fig.85), which allows to verify, refine (change) the contact point for each available measurement of each desired active group:



| Indentation 7.2.6 - [Example.mit]                  |            |
|--|------------|
| 🗹 File Edit Instrument Administration Window About |            |
| È 🖕 H 🖶 🛛 🖉 🖬 ▲ 🕂                                  | ADO        |
| Group name 1 Group name 2                          |            |
| 🖸 🗗 👫 🔳 👩 🖾 🖊 🕹 🖊                                  | ) <b>^</b> |
| Method<br>Oliver & Pharr                           |            |
| Main results<br>HIT= 9 3436 GPa                    |            |

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

 For the active group tab, click on additional toolbar.



Fig.85 Set the contact point window (default settings)

# 4.1.1.1 Contact point features

# Left graph



If there are **several measurements**, the measurement numbers are displayed on the left top of the window. The active measurement is highlighted and its corresponding curves are displayed on the left graph.

To verify or refine (change) the contact point of each indentation measurement, select (one at a time) each active measurement by:

- Clicking the measurement number #
- Pressing the <'Up arrow'> or <'down arrow'> key

In case of a **single measurement**, there is no measurement # number displayed: it is the active measurement.

To center the left graph displayed curves on contact point. If the zoom tools are used, click this icon to center the left graph, where the important zone to verify/refine the contact point is displayed.



The zoom tools and cursor information are available on both left and right graphs; refer to the similar explanations as described in the **Common Scratch & Indentation software Reference guide** in section **Measurement documents in curve view / Document window interface / Graph area tools / Zoom**. To display/hide the indentation curve(s) in/from the left graph:



To restore the contact point automatically detected by the software.

86.00

Fn=-3.80 µN

87.30

89.30

Pd=2820.56 nm

Fig.86 Stiffness curve is displayed in addition

91.40

S=1.00 mN

....

95.40

93.40

2.81F03

**INFORMATION:** By default after the measurement process, the contact point for each available measurement is automatically detected  $\gtrsim$  (inactive).

mN/mm µN -2.24E04 -4.55E03

x 16.0 🔻 🔍 🔍 🔍 🗸

Time=85.35 s

See section 4.1.1.2 (verify/refine contact point).

## Right graph area

The right graph always displays the available measurement combined curve(s), force  ${\bf F}$  vs. depth  ${\bf h}$ 



The selected active measurement # (highlighted in the left top of the window) curve is displayed in dark red color.

If the mouse cursor is placed on any measurement #, the corresponding curve becomes highlighted.



# 4.1.1.2 Verifying/refining the contact point

The contact point is automatically detected ( $\gtrsim$  inactive in Fig.87) 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.87 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 section 4.1.1.1.

**INFORANTION:** For high load measurement, it is difficult to see the change in the *F* curve,



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

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



To manually set (refine) the contact point

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

**h** curve starts to peak

**F** curve starts to rise,

and at this specific location:

- Double click

OR

- Right click and select **Set contact point** from context menu.

If necessary, click  $\nearrow$  (it became active in Fig.87) to automatically redefine the contact point.

When the contact point changes (different location), the curves and statistics are updated and the analyses are recalculated without leaving the *Set the contact point* window (Fig.87). This allows seeing in real time the impact of the contact point.

E.g. Oliver & Pharr in the analysis result area:



Hypothesis Poisson's ratio(nu)= 0.16 Additional results Main results values

*Main results* values with **refined** (manual) contact point.

Some *Additional results* values also change accordingly.

### 4.1.2 INDENTATION CURVE DISPLAY



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



 $\forall$  To also display/hide the previously described curves on/from the graph.



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

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

|                    | Adjust all curves  |             |
|--------------------|--------------------|-------------|
| itatic curves      |                    |             |
| 0.00 mN            | 15.00 mN           | Auto adjust |
| 📝 Min Pd           | Max Pd             |             |
| 0.00 🗭 µm          | 0.30 💭 µm          | Auto adjust |
| 🔽 Min time         | 📝 Max time         |             |
| o.00 💌 s           | 75.00 💌 s          | Auto adjust |
| Min User Channel 1 | Max User Channel 1 |             |
| 0.00               | 0.00               | Auto adjust |
| Min User Channel 2 | Max User Channel 2 |             |
| 0.00               | 0.00               | Auto adjust |
|                    |                    |             |
|                    |                    |             |
|                    |                    |             |
|                    |                    |             |

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

For the detailed description of this window, refer to the **Common Scratch & Indentation software Reference guide** in section **Measurement documents in curve view / Manipulating document windows / Adjusting curve scale**.

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

This feature is applicable with any indentation displayed curve(s)  $\mathcal{N}$ , or  $\mathcal{N}/\mathcal{M}$  (if activated)/ $\mathcal{M}$  (if activated) on the graph; see section <u>4.1.2</u>.



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



#### 4.1.4 **EXPORTING ALL INDENTATION CURVES**



v ∂

No items match your search.

Size

Search Example

Date modified

Save

To export **all** indentation curves.

→ ✓ ↑ → This PC → Desktop → Example

<

Save as type: 1000 points - Tab separated (\*.TXT)

Name

#### This window allows:

New folder

File name: Example#2.TXT

🔏 Export

Organize 🔻

🖈 Quick access

This PC

i Network

Hide Folders

 Choosing a location where to save the export text file.

×

Q

?

8== 🗸

Туре

Cancel

- Modification of the default *File name*: corresponding to the measurement file name with current measurement #.
- Changing of the default 1000 points .TXT file format.

Save as type: 1000 points - Tab separated (\*.TXT) 1000 points - Tab separated (\*.TXT)

Then click <<u>S</u>ave> button.

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

| ĺ  | Example                               | e#2 - Notep                                      | ad                              |   |             |           |
|----|---------------------------------------|--|---------------------------------|---|-------------|-----------|
| ſ  | File Edit                             | Format   | View Help                       | )   |             |           |
| 1  | Indentat                              | tion   |                                 |   |             |           |
|    | + Adva                                | nced   |                                 |   |             |           |
|    | Advano<br>Acqu<br>Line<br>Max<br>Load | ced<br>isition<br>ar Loadi<br>load :<br>ing rate | Rate :<br>ng<br>80000.0<br>:    | 10.0 [Hz]<br>0 μN<br>160000.00                | μN/min      |           |
| į, | Analysi:                              | sn#1   |                                 |   |             |           |
|    | Meth                                  | od :   | oliver                          | & Pharr                                       |             |           |
|    | + Para                                | meters   |                                 |   |             |           |
|    | Unloa                                 | d Fit [4   | 0%,98%]                         |   |             |           |
|    | + Main                                | results  |                                 |   |             |           |
|    | HIT=<br>EIT=<br>E*=                   | 9301.9<br>73531<br>75463                         | MPa<br>MPa<br>MPa               |   |             |           |
|    | + нуро                                | thesis   |                                 |   |             |           |
|    | Pois                                  | son's ra   | tio(nu)=                        | 0.16  |             |           |
|    | + Addi                                | tional r   | esults                          |   |             |           |
|    | HVTT                                  | -  | 861 46                          | Vickers                                       |             |           |
| !  | Measure<br>Fime (s)                   | d values <br>)                                   | Pd (nm)                         | Fn (µN) FnR                                   | ef (μN)     | SegmentID |
|    | )<br>).1834<br>).3668<br>).5502       | 0<br>4.5196<br>5.9095<br>6.0796                  | 0<br>0.6199<br>3.5309<br>6.1973 | 498.824 0<br>500.0537<br>497.1945<br>503.7691 | 0<br>2<br>2 |           |

- The indentation measurement type and its parameters.

e Folders

- All current analysis number(s) and their results.

 The point values for each curve: 1000 points (resampling) or raw points (measured points) previously selected when exporting. To display the instrument driving curves and all signals, select **"Edit > Indentation > Show signals..."**.



On the left of the window, the Y axes for all curves are always displayed and the related curve color square legends with names are displayed at the bottom of the window.

'*X axis time*' 🔻 icon:

To select the total time of the graph X axis. A context menu allows selecting one of the several proposed times.

#### *Show/hide curves* $\widetilde{\phantom{a}}$ icon:

To show/hide curves on/from the graph. A context menu allows a quick selection of the curves; each curve name with its color square highlighted is shown on the graph.



Clear graph 🕺 icon:

To clear all curves from the graph.

# Export 📑 icon:

To export all curves from the graph (even if some are not shown). A *Save as* window appears: Browse where to save the export curve file on the acquisition system, type a file name with the extension .TXT (not automatic) and click <Save> button.

#### 'Zoom' tools:

Same as described in the *Common Scratch & Indentation software reference guide* in section *Measurement documents in curve view / Document window interface / Graph area tool.* 

## 4.3 NHT SPRING COMPLIANCE

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





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

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

If the contact point is changed (section 4.1.1), the value is recomputed and the results recalculated.

**IMPORTANT:** If the difference between the <u>computed</u> 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 (section 4.1.1.)

The additional message "(Factory spring compliance: value<sup>1</sup>)" is also displayed in the measurement area.

<sup>&</sup>lt;sup>1</sup> Factory value was set in the Factory spring compliance field of the NHT Ranges tab (Fig.6); see from section 2.1.3.

# 4.4 **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 (section 4.5), and with the indentation combined curve  $\checkmark$  displayed on the graph (section 4.1).



#### **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.84)

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

1) Linear

- <sup>2)</sup> Power law
- <sup>3)</sup> In addition tangent is extended
- <sup>4)</sup> Upper and lower bounds for the fit and tangent; see <u>Set</u> <u>range</u>, p. 160

#### 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*):



## 4.5 MANAGING THE ANALYSIS METHODS

# 4.5.1 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 > Add new analysis..." from menu bar.

Active group (all its measurements)



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

#### Active measurement



See the following Create a new analysis window (Fig.89).

| Create a new analysis     |
|---------------------------|
| Analysis method           |
| Martens hardness          |
| Tangent                   |
| Sinus mode Analysis       |
| Oliver & Pharr            |
| Creep Analysis            |
| Adhesion analysis         |
| Hertz                     |
|                           |
|                           |
|                           |
|                           |
| <u>O</u> K <u>C</u> ancel |

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

Fig.89 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* values for this new (current) analysis method; see each detailed analysis method results from section 4.6 to 4.10.

# 4.5.2 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).



Fig.90 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 section 4.6 to 4.10.

# 4.5.3 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 menu bar.

Active group (all its measurements)

| 🔀 Indentation 7.2.6 - [Example.mit] |                          |  |  |  |
|-------------------------------------|--------------------------|--|--|--|
| 🗹 File Edit Ir                      | nstrument Administration |  |  |  |
|                                     | 🖶 🖪 🔀                    |  |  |  |
| Group name 1                        | Group Properties         |  |  |  |
| 00                                  | Change indenter          |  |  |  |
| 1 2                                 | Change contact point     |  |  |  |
| Method<br>Tangent                   | Adjust curve scales      |  |  |  |
| Main results                        | Change frame complia     |  |  |  |
| HIT= 15433<br>FIT= 76308            | Group relevant           |  |  |  |
| Hunothosis                          | Add new group            |  |  |  |
| III Indenter no                     | Add new analysis         |  |  |  |
| Poisson's rati                      | Generate a surface map   |  |  |  |
| Additional res<br>HVIT= 1429.3      | Frame compliance curv    |  |  |  |
| nIT= 68.62 %<br>Emax= 39582         | Cut                      |  |  |  |
| hmax= 592.5                         | Сору                     |  |  |  |
| 5= 146.0070<br>hr= 323.14 n         | Paste                    |  |  |  |
| Ap= 2564747<br>Welast= 5629         | Delete                   |  |  |  |
| Wplast= 2573<br>Wtotal= 8203        | Print preview            |  |  |  |
| Parameters                          | Print                    |  |  |  |
| Unload Fit [7                       | Export measurements      |  |  |  |
|                                     | Export analyses          |  |  |  |
| Relevant                            | Export SIO               |  |  |  |
| + Sinus                             | Apply current filter     |  |  |  |
| Max load : 40                       | Apply curve settings     |  |  |  |
| Min load : 1.                       | Delete all analyses      |  |  |  |

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

Active measurement



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

# Active analysis method



#### **Delete the active method**

To delete the active analysis method:



A *Warning* window appears. Click  $\gamma_{es}$  to confirm the active method deletion or click  $N_0$  to keep them.

# 4.6 ADHESION ANALYSIS METHOD & RESULTS

See also section 3.7.6.10 (measurement preference parameters).



After a measurement(s), to manually create an adhesion analysis, see section 4.5.1 and double click

Otherwise before a measurement(s), see section 4.5.2, this Adhesion analysis box was checked.



Click  $\oplus$  on the additional toolbar and adjust the *Min Fn* scale (Fn<0); refer to the **Common Scratch & Indentation software Reference guide** in section **Measurement documents in curve view / Manipulating document windows / Adjusting curve scale**.

Fig.91 Adhesion Force of current measurement is displayed

# 4.7 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.
- 荒 To show/hide:
  - The displayed **analysis curve(s)**.
  - The analysis curve features: depth or cycle mode, scale adjustment(s), export.

See the following sections for descriptions of the related icons.



Analysis and indentation curves (section 4.1) 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

# 4.7.1 ANALYSIS CURVE DISPLAY



With *Martens* analysis method, only + is available below.

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

- Pd
- 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 show (by default)/hide the elasticity curve.
- To show (by default)/hide the hardness curve.
- $\checkmark$  To show/hide the storage modulus curve.
- To show/hide the loss modulus curve.
- $\underset{\text{and}}{\sim}$  To show the tangent curve.
- $\checkmark$  To show the stiffness curve.
- To adjust the graph scale(s) for the displayed analysis curve(s); see the following window.

| Scales                  |       |   | _     |             |
|-------------------------|-------|---|-------|-------------|
|                         | Adj   | ust all curves  |       |             |
| Dynamic curves          |       |   |       |             |
| Min Pd                  |       | 📝 Max Pd  |       |             |
| 0.00 🗭 r                | im    | 565.23 💌  | nm    | Auto adjust |
| Min Hit                 |       | 🗸 Max Hit   |       |             |
| 0.00                    | ИРа   | 18513.91 💌  | MPa   | Auto adjust |
| Min Stiffness           |       | Max Stiffness   |       |             |
| 0.00 🗭 µ                | ıN/nm | 150.00 🐑  | µN/nm | Auto adjust |
| Min EIT                 |       | Max EIT   |       |             |
| 0.00                    | ИРа   | 130431.38 🐑   | MPa   | Auto adjust |
| Min E'                  |       | Max E'  |       |             |
| 0.00                    | ИРа   | 115938.66 💌   | MPa   | Auto adjust |
| Min E"                  |       | Max E''   |       |             |
| 0.00                    | ИРа   | 13188.98 💌  | MPa   | Auto adjust |
| Min tan <delta></delta> |       | 📝 Max tan <delta< td=""><td>&gt;</td><td></td></delta<> | >     |             |
| 0.00                    |       | 0.12 💭  |       | Auto adjust |

For the detailed description of this window, refer to the *Common Scratch & Indentation software Reference guide* in section *Measurement documents in curve view / Manipulating document windows / Adjusting curve scale*.

### 4.7.2 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 section 4.1.4, **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).

| 📃 Exam  | ple#2 - Notepad |            |        |           |             |           |           |         |
|---------|-----------------|------------|--------|-----------|-------------|-----------|-----------|---------|
| File Ed | lit Format View | Help       |        |           |             |           |           |         |
| kyc1e   | # max depth     | Max load   | stiffn | ess EIT   | EIT storage | EIT loss  | hit tan(  | delta)  |
| 0       | 8.009E-009      | 8.952E-006 | 3057   | 8.99E010  | 8.277E010   | 1.031E010 | 1.079E010 | 0.1245  |
| 1       | 8.285E-009      | 9.537E-006 | 3382   | 9.403E010 | 8.626E010   | 7.81E009  | 1.02E010  | 0.09054 |
| 2       | 8.394E-009      | 1.001E-005 | 3336   | 9.308E010 | 8.546E010   | 9.355E009 | 1.08E010  | 0.1095  |
| 3       | 8.433E-009      | 1.024E-005 | 3508   | 9.661E010 | 8.842E010   | 6.36E009  | 1.07E010  | 0.07193 |
| 4       | 8.457E-009      | 1.025E-005 | 3302   | 9.231E010 | 8.481E010   | 1.408E009 | 1.112E010 | 0.01661 |
|         | 0 0000          | 4 0575 005 | 2400   | 0.0565040 | 0.500-010   | 2 6275000 | 4 0000040 | 0.01070 |

#### 4.8 **DYNAMIC ANALYSES**

See also section 4.7.

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



**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).



The current *Parameters* set above (*Number of periods* fields) are also displayed in the analysis result area.

H70IB103EN-C

Parameters 11 periods per point 1.00 periods between each point

Damping coef=228.900 [n.s/mm] mobile mass=11.897 [g]

spring stiffness=1.498 [n/mm]

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

| Image: Constraint of the second secon |
|--|
| Cycle # 1  |
| Main results<br>HIT = 94.479 MPa<br>EIT = 2315.5 MPa<br>E* = 2544.5 MPa  |
| Hypothesis<br>hc out of calibrated range [140 nm, 1310 nm]<br>m out of O&P hypothesis<br>Poisson's ratio(nu)= 0.30   |
| Additional results<br>HVIT= 8.9175 Vickers<br>CIT 0.01/20/10= 16.09 %  |

After a CMC measurement(s), to manually create a Oliver & Pharr analysis, see section 4.5.1 and double click

Otherwise, before a measurement(s), see section 4.5.2, this box  $\bigcirc$  Oliver & Phare was checked.

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



#### 4.9 CREEP ANALYSIS METHOD & RESULTS

**INFORMATION :** Creep analysis method is only available for measurement(s) with spherical indenter; otherwise it is inactive.



### Element(s) setting

To increase/decrease the Kelvin-Voigt material model elements, click  $\frac{1}{10}$  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 results* and *Additional results* values are recalculated, and the current *Element(s) count* value is displayed.



H70IB103EN-C

# 4.10 HERTZ ANALYSIS METHOD & RESULTS

**INFORMATION:** Hertz analysis method is only available for measurement(s) with spherical indenter; otherwise it is inactive.



For each change of the upper/lower bound, the load fit on the curve is adapted and in the result analysis area, the *Main results* and *Additional results* values are recalculated, and the *Load fit* values are displayed. After a measurement(s), to manually create a *Hertz* analysis, see section 4.5.1 and double click

Otherwise before a measurement(s), see section 4.5.2, this box  $\boxed{\ensuremath{\mathbb{Z}}\xspace{-1mu}}$  was checked.

The upper and/or lower bound value(s) for the load fit can be changed; see the similar overlay properties described in section <u>4.4</u>, **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



# 4.11 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 sections for the detailed descriptions.

#### 4.11.1 INDENTER NOT CALIBRATED



If the used indenter was not calibrated before performing the measurement (*Indenter properties* window Fig.25), the warning message "*!!! Indenter not calibrated !!!*" appears.

**INFORMATION:** 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 section 4.12.2.

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



Under Additional results area, 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.

**INFORMATION:** 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 (section 4.10).

*m* value can be changed (a little) by changing the upper/lower value of the bounds; see the overlay properties section 4.4.



## 4.11.3 OUT OF INDENTER TIP CALIBRATION

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



Fig.94 Indenter properties window (section 2.5.4)

## 4.11.4 CHANGING THE POISSON'S RATIO

To change the current Poisson's ratio for each:

Active document (all measurements of its groups)

- Select "Edit > Document > Change Poisson's ratio..." from menu bar.

# Active group (all its measurements)



### Active measurement

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

|  |   | Ch<br>Ch                    | ange<br>ange | Poiss<br>fram | on's i<br>e con | ratio<br>nplianc |     |
|--|---|-----------------------------|--------------|---------------|-----------------|------------------|-----|
|  |   | Ind                         | lentat       | ion r         | elevar          | nt               |     |
|  |   | Set                         | cont         | act p         | oint            |                  |     |
|  |   | Cu                          | t            |               |                 |                  |     |
|  |   | Co                          | ру           |               |                 |                  |     |
|  |   | Pas                         | ste ind      | lenta         | tion            |                  |     |
|  |   | De                          | Delete       |               |                 |                  |     |
|  |   | Export measurement          |              |               |                 |                  |     |
|  |   | Export analyses             |              |               |                 |                  |     |
|  | - | Export SIO<br>Print preview |              |               |                 |                  |     |
|  |   |                             |              |               |                 |                  |     |
| + Advanced<br>Acquisition Rate : 10.0 [Hz]                 |   | Print                       |              |               |                 |                  |     |
| Linear Loading<br>Max load : 0.50 mN                       |   | Delete all analyses         |              |               |                 |                  |     |
| Loading rate : 1.00 mN/min<br>Unloading rate : 1.00 mN/min |   | Ap                          | ply cu       | irve s        | etting          | js               |     |
| Pause : 10.0 s   |   |                             |              | Ŧ             | Tin             | ne=0.2           | 6 s |
| Information 1 2 3 4  | 5 | 6                           | 7            | 8             | 9               | 10               | 1   |
|  |   |                             |              |               | 1               | 1                | 1   |



 Select "Edit > Indentation > Change Poisson's ratio..." from menu bar

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

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

| Poisson's ratio | n's ratio 🛛 – |
|-----------------|---------------|
|-----------------|---------------|

| ( | Reset to sample value |
|---|-----------------------|

100

To set another Poisson's ratio value.

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

| ndentation group information       |  |      |
|------------------------------------|--|------|
| Group <u>N</u> ame<br>Group name 1 |  |      |
| Informations<br>Operator<br>Client | Sample characteristics<br><u>P</u> oisson's ratio<br>0.160<br><u>Substrate</u> |      |
| <u>R</u> eference                  | Layers   | Fdit |



In the analysis result area, for each change of the Poisson's ratio value (set above), a new value for *EIT* in *Main results* section is recalculated accordingly.
### **4.12 MANAGING THE INDENTER IN THE RESULT ANALYSIS**

The indenter or some of its parameters can be changed.

| Main results   |   | <b>Recalculated</b>                                  |
|--|---|--|
| EIT= 74.046 GPa<br>E*= 75.991 GPa  |   | For chang  |
| Hypothesis<br>Poisson's ratio(nu)= 0.16  |   | (some par<br>4.12.1,                                 |
| Additional results   | E | AND/OR   |
| CIT 0.04/30/10=1.22 %<br>nIT= 65.78 %<br>Fmax= 40.03 mN<br>hmax= 0.57 µm<br>S= 170.4237 mN(vm  |   | for each c<br>4.12.2,                                |
| hc= 0.39 μm<br>hc= 0.33 μm<br>hp= 0.27 μm<br>m= 1.2616<br>Epsilon= 0.78                        | L | the <i>Main res</i><br>in the analys<br>for each con |
| R2= 1.000<br>Ар= 4.20 µm <sup>2</sup><br>Welast= 0.01 µJ<br>Wplast= 0.00 µJ<br>Wtotal= 0.01 µJ | + |  |

#### d results

jes of the indenter properties rameters/fit method); section

hange of the indenter; section

ults and Additional results values is result area are recalculated cerned measurement.

### 4.12.1 INDENTER PROPERTIES

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

|                               |                          | <ul> <li>Select "Edit &gt; Indentation &gt; Indenter<br/>properties" from menu bar</li> </ul> |
|-------------------------------|--------------------------|---|
|                               | Change Indenter          |   |
|                               | Indenter properties      |   |
|                               | Export indenter          |   |
|                               | Change Poisson's ratio   |   |
|                               | Change frame complian    |   |
|                               | Indentation relevant     |   |
|                               | Set contact point        |   |
|                               | Cut                      |   |
|                               | Сору                     |   |
|                               | Paste indentation Delete |   |
|                               | Export measurement       |   |
|                               | Export analyses          |   |
|                               | Export SIO               |   |
| Retract speed : 1 µm/m        | Print preview            |   |
| Stiffness Threshold : 50      | Print                    | - Right click in the measurement parameter  |
| spring compliance. o.         | Delete all analyses      | alea, of on the measurement (a), and  |
| X Position :104.79 mm         | Apply cupie settings     | select Indenter properties Iron   |
| Y Position :89.20 mm          | Apply curve settings     | context menu  |
| + Berkovich indenter          | =                        |   |
| Material : Diamond            |                          | INFORMATION: If necessary, scroll down in   |
| Calibration date : 14.10.2016 |                          | the measurement parameter area to display   |
| Frame compliance : 0.10 µm/   | N 🗼 🗵                    | the current indenter information.   |
|                               | - T                      |   |
| Information 1 2 3             | 4 5 6 7 8                |   |
|                               |                          |   |
| -                             |                          |   |
|                               |                          | The <i>Indenter properties</i> window appears and allows changing of:                         |
|                               | $\sim$                   | - The indenter parameters <b>evcent</b> the   |
|                               |                          | indenter Type (section 2.5.1) The   |
|                               |                          | indenter information is undated in the  |
|                               |                          | macurement parameter area   |
|                               |                          | measurement parameter area.   |
|                               |                          | - The indenter fit method (section 2.5.4).  |
|                               |                          | <b>INFORMATION:</b> From here, it is not  |
|                               |                          | possible to calibrate the indenter (the   |
|                               |                          | <i>button</i> <calibrate> <i>is hidden</i>).</calibrate>                                      |
|                               |                          | ,   |

For each change of the indenter properties, see <u>Recalculated results</u>, p. 181.

### 4.12.2 CHANGING THE INDENTER

To change the current indenter for each:

### Active document (all measurements of its groups)

- Select "Edit > Document > Change indenter..." from menu bar.

### Active group (all its measurements)



#### Active measurement

- Select "Edit > Group > Change indenter..." from menu bar
- Right click on the group tab and select "Change indenter..." from context menu
- Select "Edit > Indentation > Change indenter..." from menu bar

| Retract speed : 1 µm/m<br>Dz sensor in fine range<br>Stiffness Threshold : 50<br>Spring Compliance : 0.7<br>X Position :104.79 mm<br>Y Position :89.20 mm<br>+ Berkovich indenter<br>Serial number : B-A00<br>Material : Diamond |               | C<br>Ir<br>S<br>C<br>C<br>P<br>D<br>E<br>E<br>E<br>P<br>P<br>D<br>A | hang<br>ndent<br>xport<br>hang<br>hang<br>ndent<br>et cor<br>ut<br>opy<br>aste i<br>velete.<br>xport<br>xport<br>rint p<br>rint<br>velete<br>pply | e Ind<br>er pro<br>inder<br>e Poi<br>e frar<br>ation<br>ntact<br>meas<br>analy<br>SIO<br>review<br>all ar | enter<br>operti<br>nter<br>ssson'<br>relev<br>point<br>tatior<br>surem<br>yses<br>w<br>aalyse | s rat<br>omp<br>ant<br>:<br>nent |
|--|---------------|---|---|---|---|----------------------------------|
| Calibration date : 14.10.<br>Frame compliance : 0.10   | .2016<br>µm/N | ן<br>ז  |   | I   | I   | ↓<br>▼                           |
| Information 1 2  | 3 4           | 1   | 5   | 6   | 7   | 8                                |

Fig.95 Measurement parameter area

 Right click in the measurement parameter area, or on the measurement tab, and select "Change indenter" from context menu

**INFORMATION:** *If necessary, scroll down in the measurement parameter area to display the current indenter information.* 

| Select the indenter                |    |   |        |  |  |  |  |
|------------------------------------|----|---|--------|--|--|--|--|
| Indenter used for this indentation |    |   |        |  |  |  |  |
| Indenter                           |    |   |        |  |  |  |  |
| <current one=""></current>         |    |   |        |  |  |  |  |
|                                    |    |   |        |  |  |  |  |
|                                    |    |   |        |  |  |  |  |
|                                    | ок | X | Cancel |  |  |  |  |
|                                    |    |   |        |  |  |  |  |
| *                                  |    |   |        |  |  |  |  |
| Indenter                           |    |   |        |  |  |  |  |
| <current one=""></current>         |    |   |        |  |  |  |  |

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 <u>Recalculated results</u>, p. 181.

The indenter information is updated in the measurement parameter area (Fig.95).

### 4.12.3 EXPORTING THE INDENTER

Berkovich [B-Q 54 Sinus] (31/01/2017)

<Current one>

Berkovich [B-U 08]

|   |   |      | E               | xport  | inde   | nter    |       |
|---|---|------|-----------------|--------|--------|---------|-------|
|   |   |      | C               | hang   | e Poi  | sson'   | s rat |
|   |   |      | C               | hang   | e frar | ne co   | mp    |
|   |   |      | I               | ndent  | ation  | relev   | ant   |
|   |   |      | s               | et co  | ntact  | point   |       |
|   |   |      | C               | Cut    |        |         |       |
|   |   |      | C               | ору    |        |         |       |
|   |   |      | P               | aste i | nden   | tatior  | 1     |
|   |   |      | C               | elete) |        |         |       |
|   |   |      | E               | xport  | meas   | surem   | nent  |
|   |   |      | E               | xport  | analy  | /ses    |       |
|   |   |      | E               | xport  | SIO    |         |       |
|   | Retract speed : 1 µm/m<br>Dz sensor in fine range |      | Print preview   |        |        |         |       |
|   | Stiffness Threshold : 50                          |      | P               | rint   |        |         |       |
|   | Spring Compliance : 0.7                           |      | Philum          |        |        |         |       |
|   | X Position :104.79 mm                             | mm   | Delete all anal |        |        | nalyse  | s     |
| I | Y Position :89.20 mm                              |      | A               | pply   | curve  | e setti | ngs   |
| 1 | + Berkovich indenter                              |      | ٦               |        |        |         | =     |
|   | Serial number : B-A00                             |      |                 |        |        |         | _     |
|   | Calibration date : 14.10.                         | 2016 |                 |        |        |         |       |
|   |   |      | _               |        |        |         | Ţ     |
|   | Frame compliance : 0.10                           | µm/l | N               |        |        |         |       |
|   |   |      |                 |        |        |         | Ψ.    |
|   | Information 1 2 3                                 | 3    | 4               | 5      | 6      | 7       | 8     |
|   |   |      |                 |        |        |         |       |
| U | <u>[</u>  |      |                 |        |        |         |       |

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

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

### 4.13 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 menu bar.

Active group (all its measurements)



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

#### Active measurement



Fig.96 Frame compliance current value

Select "Edit > Indentation > Change frame compliance..." from menu bar.

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

| Change current frame compliance |   |  |  |  |  |  |  |
|---------------------------------|---|--|--|--|--|--|--|
| Current frame compliance        | This window allows changir<br>current frame compliance<br>measurement(s) previously |  |  |  |  |  |  |
| Current frame compliance        |   |  |  |  |  |  |  |
| I∰ µm/N To set and              | other frame compliance value.   |  |  |  |  |  |  |

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

Reset frame compliance

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 (Fig.96) and the Main results and Additional results values in the analysis result area (below) are recalculated for each concerned measurement.

|   | DD 🗗 👫 🔳                        | \$<br>An. | 1        |
|---|---------------------------------|-----------|----------|
|   | Method<br>Oliver & Pharr        |           | <b>^</b> |
|   | Main results<br>HIT= 9 5282 GPa |           |          |
| I | FIT = 74.046 GPa                |           |          |
|   | E*= 75.991 GPa                  |           |          |
|   | Hypothesis                      |           |          |
|   | Poisson's ratio(nu)= 0.16       |           |          |
|   | Additional results              |           | =        |
|   | HVIT= 882.42 Vickers            |           | _        |
|   | CIT 0.04/30/10= 1.22 %          |           |          |
| l | Emax= 40.03 mN                  |           |          |
|   | $hmax = 0.57 \ \mu m$           |           |          |
|   | S= 170.4327 mN/µm               |           |          |
|   | hc= 0.39 μm                     |           |          |
|   | hr= 0.33 µm                     |           |          |
|   | hp= 0.27 μm                     |           |          |
| l | m= 1.2616                       |           |          |
| l | Epsilon= 0.78                   |           |          |
|   | $\Delta n = 4.20 \ \mu m^2$     |           | •        |
| l | Welast= 0.01 µJ                 |           |          |
|   | Wplast= 0.00 µJ                 |           |          |
|   | Wtotal= 0.01 µJ                 |           | -        |

<sup>1</sup> Initial value was set in the Frame compliance field of the Indenter Ranges tab for UNHT (Fig.3) or Ranges tab for NHT (Fig.6) / MHT (Fig.11); see from section 2.1.3.

### 4.14 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)

| 🔀 Indent   | ation | 7.2.6 - [Examp | ole.mit] |         |  |         |       |          |        |    |
|------------|-------|----------------|----------|---------|--|---------|-------|----------|--------|----|
| 🛛 File     | Edit  | Instrument     | Admir    | nistrat | ion                                    | Wind    | ow    | Abou     | ıt     |    |
| È ĉ        |       | Document       | ×        | 2       | L (                                    |         |       | <b>+</b> | AD     | 0  |
| Group pa   |       | Group          | ×        |         | Prop                                   | erties  |       |          |        |    |
|            |       | Indentation    | ×        |         | Adjust curves scales<br>Group relevant |         |       |          |        |    |
|            |       | Analysis       | ×        |         |  |         |       |          |        |    |
| Method     |       | Pictures       | •        |         | Gene                                   | erate a | surfa | ace m    | apping | 3  |
| Informatio | on    | 1 2 3          | 4 5      | 6       | 7                                      | 8       | 9     | 10       | 11     | 12 |
|            | i     |                | /        |         |  |         |       |          |        |    |

#### generating image ... Select the surface parameter HIT (O&P) Export to the gallery HVIT (O&P) E\* (O&P) Export to file .. Er (0&P) hm (0&P) Fm (0&P) S (O&P) List Poisson's ratio hc (0&P) х Cancel

**INFORMATION:** The (matrix) group should contain at least 4 measurements.

Select "Edit > Group > Document > Generate a surface mapping..." from menu bar.

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

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

400 Computed Data

Shows the number of the measurements for the selected parameter. **INFORMATION:** *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.

|   |   |     |   | ×<br>F× |
|---|---|-----|---|---------|
| M | - | 1/1 | ₩ | м       |
|   |   |     | • |         |
|   |   | 1   | 4 |         |
|   |   |     | - |         |

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 reference guide* in section *Measurement Documents in Curve View / Manipulating Document windows / Image Gallery*.



Export to file ...

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*.

<sup>&</sup>lt;sup>1)</sup> To know which measurement group tab and which parameter were previously selected to generate the image, the <u>T</u>itle name field is automatically filled in with this information (can be changed if necessary)

This window allows:

| Save your surface f | īle as                           |                 |                   |            | ×    |
|---------------------|----------------------------------|-----------------|-------------------|------------|------|
| 😋 💬 🗢 📙 🕨 Ex        | ported generate mapping image Ex | <b>▼</b> 49     | Search Exported g | enerate ma | ip 🔎 |
| Organize 🔻 Ne       | ew folder                        |                 |                   |            | ?    |
| ☆ Favorites         | Name                             | Size            | Date modified     | Туре       | ^    |
| 🥽 Libraries         | No ite                           | ms match your s | earch.            |            |      |
| PC                  |                                  |                 |                   |            |      |
| 👽 Network           |                                  |                 |                   |            |      |
|                     | ٠                                | 11              |                   |            | Þ    |
| File name:          | Example                          |                 |                   |            | -    |
| Save as type:       | Surface file(*.sur)              |                 |                   |            | •    |
| ) Hide Folders      |                                  | (               | Save              | Cancel     |      |
| Then click          | Save                             |                 |                   |            |      |

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

H70IB103EN-C

## **5 TROUBLESHOOTING**

## 5.1 F.A.Q.

Refer to each corresponding Indentation head **user manual** or **reference guide** in section *F.A.Q.* 

#### 5.2 ERROR MESSAGES WHILE USING THE INSTRUMENT

Refer to each corresponding Indentation head **user manual** or **reference guide** in section **Blocking error messages**.

#### **5.3** UNPRINTED ITEMS

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

#### **Measurements unprinted**



#### **Images unprinted**



The document model specifies that only the relevant images should be printed, and some images are not designated as relevant;

change the relevant attribute of each necessary



### 5.4 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 calibration certificates, which are provided with the corresponding instrument. Otherwise change them accordingly.

Refer to the **Common Scratch & Indentation software reference guide** in section **Managing the instrument / Hardware configuration / 'Ranges'** tab(s).

| STEP - NHT - Hardware configuration |  |   |                     |                              |          |   |  |
|-------------------------------------|--|---|---------------------|------------------------------|----------|---|--|
| My configuration<br>Hardware coeffi | User channels<br>icients   | Control unit & modules                        | Motors<br>d to char | Instrument adjustment        | Ranges   | Dynamic ranges                                      |  |
| Current -> L                        | .oad coef<br>mN/mA<br>Current (fine)<br>mA/V<br>Current (large)<br>mA/V<br>ng compliance<br>mm/N<br>ing compliance<br>mm/N | Current -> vi<br>Voltage -> D<br>Voltage -> D | oltage              | Frame<br>Digital B<br>Bridge | complian | .ce<br>.m/N<br>tune Dz bridge<br>: 70.00% L: 30.00% |  |
|                                     |  | Va  | ilidate ran         | ige values                   | ● OF     | K <u>C</u> ancel                                    |  |

Fig.97 'Ranges' tabs (e.g. NHT)

<sup>1</sup> UNHT has 3 'Ranges' tabs NHT has 2 'Ranges' tabs MHT has only 1 Ranges tab

| Indenter ranges | Dynamic ranges | Reference Ranges |
|-----------------|----------------|------------------|
| Ranges Dynam    | ic ranges      |                  |
| Ranges          |                |                  |

# **6 SOFTWARE FORMULAS**

Graphical views of indentation parameters:



## 6.1 **TYPICAL INDENTATION CURVE**

Fig.98 Indentation test

### <u>Legend</u>

- a : Application of F
- b : Removal of F
- c~ : Tangent to curve b at  $F_{\text{max}}$ 
  - F : test force

 $F_{\text{max}}\,$  : maximum test force

- h<sub>p</sub> : permanent indentation depth
- h<sub>r</sub> : tangent indentation depth
- $h_c \quad : \mbox{ contact depth of the indenter with the sample at <math display="inline">F_{max}$
- $h_{max}$  : maximum indentation depth
- S : contact stiffness
- ε : geometric constant

### 6.2 SCHEMATIC REPRESENTATION OF INDENTER-SAMPLE CONTACT



#### 6.3 **PARAMETERS DETERMINATION**

F<sub>max</sub>:

Directly on curve

S (Tangent method):

Tangent fit:Fit start: 95 % of  $F_{max}$ Fit end: 70 % of  $F_{max}$ 

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

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

Power law fit of unloading curve:

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

Equation:

$$\mathbf{F} = \mathbf{F}_{\max} \cdot \left(\frac{\mathbf{h} - \mathbf{h}_{p}}{\mathbf{h}_{\max} - \mathbf{h}_{p}}\right)^{m}$$

Where m,  $h_{max}$ ,  $h_p$  are fitting parameters.

#### **INFORMATION:**

- In case of creep or viscoelasticity still occurring during unloading, it may happen that the  $h_{max}$  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.

Calculation of S:

$$\mathbf{S} = \mathbf{m} \cdot \mathbf{F}_{\max} \cdot (\mathbf{h}_{\max} - \mathbf{h}_{p})^{-1}$$

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<sub>c</sub>:

$$\mathbf{h}_{c} = \mathbf{h}_{max} - \varepsilon \cdot (\mathbf{h}_{max} - \mathbf{h}_{r})$$

:3

Depending on the diamond shape

| Indenter Shape    | m   | 3                           |
|-------------------|-----|-----------------------------|
| Flat Punch        | 1.0 | 1.0000                      |
| Cone              | 2.0 | $2(\pi - 2) / \pi = 0.7268$ |
| Sphere/Paraboloïd | 1.5 | 0.7500                      |

In our case,  $\epsilon$  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

| A <sub>p</sub> : | (theoretical or calibrated)               |  |  |
|------------------|---|--|--|
|                  | $\mathbf{A}_{\mathbf{p}} = f(\mathbf{h})$ |  |  |

### 6.4 INDENTATION HARDNESS (HIT)

### 6.4.1 DESIGNATION OF H<sub>IT</sub>



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

 $H_{\mbox{\scriptsize IT}}$  is a measure of the resistance to permanent deformation or damage.

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

 $A_p$  (theoretical or calibrated)

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

### 6.5 INDENTATION MODULUS (EIT)

#### 6.5.1 DESIGNATION OF EIT



#### 6.5.2 DETERMINATION OF REDUCED MODULUS (ER)

The reduced modulus is calculated from the following equation:

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

### 6.5.3 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)  $E_{IT}$  calculated from the E\* using a an estimated sample Poisson's ratio (Vs):

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

As a reminder, typical values of  $\nu_{\text{s}}$  are:

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

### 6.6 STANDARD MEASUREMENT OF VICKERS HARDNESS (HV)

### 6.6.1 DESIGNATION OF HV

### 6.6.2 DETERMINATION OF HV

$$HV = \frac{F_{\text{max}}}{9.81 \cdot A_{\text{c}}(h_{\text{c}})} \quad \text{in Vickers (kgf/mm^2)}$$

(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)

6.7 ESTIMATION OF HVIT WITH INDENTATION HARDNESS (HIT)

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

With Vickers indenter:

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

With Modified Berkovich indenter (M. Berkovich):

 $HV_{IT} \approx H_{IT}/10.800$ 

#### 6.8 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°                         |
| a                               | 68°   | 65.03°  | 65.27°  | 35.264°                     |
| A <sub>d</sub> / h <sup>2</sup> | $4 \cdot \frac{\sin \alpha}{\cos^2 \alpha}$ | $3\sqrt{3} \cdot \frac{\sin \alpha}{\cos^2 \alpha}$ | $3\sqrt{3} \cdot \frac{\sin \alpha}{\cos^2 \alpha}$ | 9/2                         |
|                                 | ≈26.43                                      | ≈26.43  | ≈26.97  | =4.5                        |
| A <sub>p</sub> / h <sup>2</sup> | $4 \cdot \tan^2 \alpha$                     | $3\sqrt{3} \cdot \tan^2 \alpha$                     | $3\sqrt{3} \cdot \tan^2 \alpha$                     | $3\sqrt{3}/2$               |
|                                 | ≈24.504                                     | ≈23.96  | ≈24.494   | ≈2.598                      |
| A <sub>d</sub> / A <sub>p</sub> | $1/\sin \alpha$                             | $1/\sin \alpha$                                     | $1/\sin \alpha$                                     | $1/\sin\alpha = 3/\sqrt{3}$ |
|                                 | ≈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

### 6.9 MARTENS HARDNESS (HM) (FORMER DESIGN: UNIVERSAL HARDNESS HU)

### 6.9.1 **DESIGNATION OF HM**



#### 6.9.2 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:

$$A_{s}(h) = \frac{4 \cdot \sin(\alpha)}{\cos^{2}(\alpha)} \cdot h^{2} \qquad \text{and} \qquad HM = \frac{F}{A_{s}(h)} \approx \frac{F}{26.43 \cdot h^{2}}$$

• Perfect Berkovich indenter:

$$A_{s}(h) = \frac{3 \cdot \sqrt{3} \cdot \tan(\alpha)}{\cos(\alpha)} \cdot h^{2} \qquad \text{and} \qquad HM = \frac{F}{A_{s}(h)} \approx \frac{F}{26.43 \cdot h^{2}}$$

• 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  $F_{max}$  is kept constant in sec.
- after equals sign, HM value.

### Examples:

• HM 0.5/20/20 = 8700 N/mm<sup>2</sup>

HM is 8700  $\text{N/mm}^2,$  determined with a F of 0.5N, applied in 20s, constant during 20s.

• HM(Berkovich) 0.5/20/20 = 8700 N/mm<sup>2</sup>

Same as above but with a Berkovich tip instead of the standard Vickers tip.

### 6.10 INDENTATION CREEP (CIT)

### 6.10.1 DESIGNATION OF CIT



#### 6.10.2 DETERMINATION OF CIT

$$C_{IT} = \frac{h_2 - h_1}{h_1} \cdot 100$$

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.99 Expression of CIT

a: Application of F

b: F constant from  $t_1$  to  $t_2$ 

### 6.10.3 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)  $\operatorname{RCF}_{1} = \frac{\tau_{1}}{t_{R}} \left[ \exp(t_{R} / \tau_{1}) 1 \right]$  Ramp Correction Factor

(5) 
$$\operatorname{RCF}_{2} = \frac{\tau_{2}}{t_{R}} \left[ \exp(t_{R} / \tau_{2}) - 1 \right]$$

| (6)  | $\mathbf{C}_{0} = \frac{\mathbf{B}_{0}}{\mathbf{F}_{m}\mathbf{F}_{G}}$           | 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)  | $\mathbf{G}_{0} = \frac{1}{2(\mathbf{C}_{0} - \mathbf{C}_{1} - \mathbf{C}_{2})}$ |  |
| (10) | $G_{\infty} = \frac{1}{2C_0}$  |  |
| (11) | $G^{\nu} = 2G_{\infty}(1-\nu)$   | shear modulus - where ${\bf v}$ is the tested material Poisson's ratio |
| (12) | $E=2G^{\nu}(1\!+\!\nu)$  | elastic modulus  |
| (13) | $K = \frac{E}{3(1-2\nu)}$  | bulk modulus   |

### 6.11 INDENTATION RELAXATION (RIT)

#### 6.11.1 DESIGNATION OF R<sub>IT</sub>



#### 6.11.2 DETERMINATION OF R<sub>IT</sub>

$$R_{IT} = \frac{F_1 - F_2}{F_1} \cdot 100$$

Where:

 $F_1$  is the force at reaching the indentation depth, <u>which</u> was kept constant, in N

 $\mathsf{F}_2$  is the force after the time during which the indentation depth was kept constant, in N



Fig.100 Expression of RIT

- a : Application of the indentation depth
- b : Indentation depth kept constant from  $t_1$  to  $t_2$

### 6.12 ELASTIC PART OF INDENTATION WORK

$$\eta_{IT} = \frac{W_{elast}}{W_{total}} \cdot 100$$

With:

 $W_{total} = W_{elast} + W_{plast}$ 

Plastic part W\_{plast} / W\_{total} follows as 100 % -  $\eta_{\text{IT}}$ 



### 6.13 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:

$$\mathbf{h}_{\rm c} = h_{\rm max} - \varepsilon \cdot \left( h_{\rm max} - h_r \right)$$

Where:

 $\boldsymbol{\epsilon}$  depends on the indenter shape.

#### 6.14 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**:

$$\mathbf{F} = \mathbf{k} \cdot \left(\mathbf{h} - \mathbf{h}_{\mathbf{p}}\right)^{\mathbf{m}}$$

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.

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

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:

$$\mathbf{S} = \left(\frac{d\mathbf{F}}{d\mathbf{h}}\right)_{\max} = \mathbf{m} \cdot \mathbf{F}_{\max} \cdot \left[\frac{\left(\mathbf{h}_{\max} - \mathbf{h}_{p}\right)^{m-1}}{\left(\mathbf{h}_{\max} - \mathbf{h}_{p}\right)^{m}}\right] = \mathbf{m} \cdot \mathbf{F}_{\max} \cdot \left(\mathbf{h}_{\max} - \mathbf{h}_{p}\right)^{-1}$$

h<sub>r</sub> is thus given by:

$$h_r = h_{max} - \frac{F_{max}}{S}$$

 $h_c$  is then:

$$\mathbf{h}_{\mathrm{c}} = \mathbf{h}_{\mathrm{max}} - \varepsilon \cdot \left(\mathbf{h}_{\mathrm{max}} - \mathbf{h}_{\mathrm{r}}\right)$$

Where:

 $\boldsymbol{\epsilon}$  now depends on m

The tangent is found by differentiating the unloading curve and evaluating at  $F_{max}$ 

The intercept of this tangent with the displacement axis yields h<sub>r</sub>

### 6.15 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