

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

Galileo Galilei (1564-1642)

Reference Guide

Step X00 - UNHT³

Ultra Nanoindentation Tester

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

Disclaimer

This document may contain errors and omissions. If you discover any such errors or if you would like to see more information in this document, please contact us at our address below. Anton Paar assumes no liability for any errors or omissions in this document.

Changes, copyright, trademarks, etc.

This document and its contents may be changed or amended by Anton Paar at any time without prior notice.

All rights reserved (including translation). This document, or any part of it, may not be reproduced, changed, copied, or distributed by means of electronic systems in any form (print, photocopy, microfilm or any other process) without prior written permission by Anton Paar GmbH.

Trademarks, registered trademarks, trade names, etc. may be used in this document without being marked as such. They are the property of their respective owner.

Further information

Published and printed by Anton Paar TriTec SA, Switzerland Copyright © 2020 Anton Paar GmbH, Graz, Austria

Address of the instrument producer:

Anton Paar TriTec SA Vernets 6 CH-2035 Corcelles / Switzerland – Europe

Tel: +41 (0)32 552 16 00 Fax: +41 (0)32 552 16 10 E-Mail: info.tritec@anton-paar.com Web: www.anton-paar.com

Date: Jan 2020 Document number: G70IB003EN-A

Reference Guide

Step X00 - UNHT³

Ultra Nanoindentation Tester

From Indentation Software Version 9 for Windows[®] 7 64 bits and Windows[®] 10

Contents

1	ABOUT THIS DOCUMENT	. 6
2	SAFETY INSTRUCTIONS	. 8
3	INSTRUMENT VIEWS	. 9
3.1 3.2 3.3 3.4	GENERAL HEAD DETAILS INDENTER & REFERENCE DETAILS FOR HANDLING INDENTER-REFERENCE SPACING DISTANCE	.9 10 11 14
4	PATENT	15
5	INSTRUMENT & MEASUREMENT PREPARATION	16
5.1 5.2 5.3	WARNING: REMOVE HEAD BOTTOM PROTECTION COVER UNIVERSAL ADAPTER MOUNTING/DISMOUNTING SWITCHING ON THE INSTRUMENT	16 17 18
5.4	POSITION CALIBRATION OF EACH OBJECTIVE	18
5.5	VIDEO SCALE CALIBRATION OF EACH OBJECTIVE	18
5.6		19
5.6.1	DISMOUNTING/MOUNTING	19
5.6.2	MANAGEMENT	33
5.7	SAMPLE HOLDER	36
5.8	POSITION CONTROL	37
5.9	VIDEO	41
5.9.1	VIDEO MICROSCOPE	42
5.9.2	LIGHT ADJUSTMENT	42
5.9.3	FOCUS ADJUSTMENT	43
5.10	INDENTER-MICROSCOPE DISTANCE CALIBRATION	44
5.10.1	SAMPLE HOLDER	44
5.10.2	START CALIBRATION	45
5.11	ADJUST DEPTH OFFSET (ADO)	50
5.11.1	Find ADO LOCATION	50
5 11 2	STARTING	50
5 11 3		51
5.17	HEAD DYNAMIC CALIDRATION (WITH SINUS MODE)	51
5.12	CALIDRATION OF INDENTEDS (QUASISTATIC OF SINUS)	54
5 1 2 1	CALIDRATION OF INDENTERS (QUASISTATIC OK SINUS)	5 5
J.13.1		61 01
J.13.2		67
5.13.3		02
5.13.4		63
5.13.5		68
5.13.6	EDIT FIT METHOD	/5
6	INDENTATION MEASUREMENT PROCESS	78
6.1	SAMPLE HOLDER	78

6.2	OPEN OR CREATE NEW DOCUMENT	78
6.3	PERFORMING AN INDENTATION MEASUREMENT	80
6.3.1	ADO	.80
6.3.2	FIND MEASUREMENT AREA	.80
6.3.3	DISTANCE BETWEEN INDENTS	.81
6.3.4	DEFINE INDENTATION MEASUREMENT PARAMETERS	.81
6.3.5	RUN INDENTATION MEASUREMENT	.86
636	ANALYZE/VISUALIZE FACH SELECTED INDENT	88
6.3.7	SAMPLE DISPLACEMENT AFTER MEASUREMENT(S)	.90
0.017		
7	RESULTS ANALYSIS	91
7.1	VIEWING MODES	92
7.2	CONTACT POINT(S)	93
7.3	REVIEWING IMAGES	93
74	STNUS ANALYSTS	94
75	SAVE DDINT STATISTICS/FYDODT	95
751		05
7.5.1	JAVL	.95
7.5.2		100
1.5.5	STATISTICS/EXPORT	100
8	PERFORMING A NEW MEASUREMENT 1	.01
8.1	WITH THE SAME SAMPLE	.01
8.2	WITH A NEW SAMPLE	.01
•	MOUNTING USAD BOTTOM BROTECTION COVER	00
9	MOUNTING HEAD BOTTOM PROTECTION COVER	.02
10	F.A.Q	.03
10 1		03
10.1	WHY IS THE INDENT NOT UNDER THE VIDEO CROSSHAIR!	03
10.2		.04 0E
10.5		.05
10.4		.00
10.5		.08
10.6	WHY IS THE FORCE DISPLACEMENT CURVE LINEAR?	.09
10.7	WHY DOES THE INSERT AN ADJUST DEPTH OFFSET WINDOW APPEAR?	.11
10.8	WHY DOES "HC OUT OF CALIBRATED RANGE" MESSAGE APPEAR?	.12
11	BLOCKING ERROR MESSAGES 1	.13
11.1	CONNECTION TO CONTROL UNIT FAILED	13
11.2	YOUR INSTRUMENT RANGES HAVE NOT BE VALIDATED!	.14
11.3	MOTORIZED TABLES	.15
11.4	VIDEO SOFTWARE	16
11.5	CAN NOT WRITE ADDRESS	17
11.6	PREVIOUS DAOMX DRIVER VERSION DETECTED	17
11.7	DURING AN ADO, A CALIBRATION OR AN INDENTATION MEASUREMENT	17

1 ABOUT THIS DOCUMENT

This document explains how to carry out an indentation measurement using the Anton Paar Step - UNHT³ instrument.

For further explanations concerning the platform, refer to the **Step X00 Instruction manual and safety information (IMSI)**; this document is common for every Step X00 measuring/imaging head and mentions important information (safety, acquisition system configuration, setup, maintenance/warranty...).

For further information concerning the Anton Paar software, refer to the following documents:

- Common Scratch & Indentation software reference guide
- Indentation software reference guide
- Video software reference guide

For further explanations concerning the available Anton Paar sample holder, refer to the **Sample holders user manual**. The universal sample holder is used as an example in this document.

In this document

- The images may differ from the actual product:
 - Some print screens are coming from a previous software version but they are similar/applicable for V9.0.x version.
 - Some pictures show previous UNHT³ measuring head design but it doesn't change the way to use it.
 - etc.
- Sometimes several ways to access a software window are possible but only the main one(s) is used in this document. However they are all described in the software reference guides (like keyboard shortcuts...).
- The units can be different from a print screen to another (e.g. μN, mN or N and μm or mm), to change the units, refer to *Common Scratch & Indentation* software reference guide in section *Customizing options / Preferences tab (units)*.
- There are some references with a letter and a number '(Z.#)' which are described in section 3.3 (Indenter and reference details for handling).
- If not specified, 'Step X00', 'Step' or 'platform' means Step 700 and 700 Noise-Control if not specified.
- If not specified 'head' means measuring head (UNHT³), as it could be video microscope (VID) imaging head or other measuring/imaging head(s).
- 'key(s)' means keyboard key(s).

Abbreviations & symbols

Abbreviations Symbols	Designations	Units
ADO	adjust depth offset	
AFM	Atomic Force Microscope (imaging head)	
Ap	projected contact area	m ²
E*	plane strain modulus	Pa
EIT	indentation modulus	Pa
F	test force	N
h	indentation depth under applied test force	m
h _c	contact depth of the indenter with the sample at F_{max}	m
h _{max}	maximum indentation depth	m
hp	permanent indentation depth	m
v (Nu)	Poisson's ratio	
sec	seconds	
Step 700	Surface testing platform with up to 3 measuring heads: - with optical video microscope imaging head - with acoustic enclosure	
Step 700	Similar as Step 700 exception: With anti-vibration	
Noise-Control	(electronic) active table	
UNHT ³	Ultra Nanoindentation Tester (measuring head)	
VID	optical video microscope (imaging head)	

Physical quantity symbols are according to the ISO 14577 standard.

2 SAFETY INSTRUCTIONS

Refer to the **Step X00 Instruction manual and safety information (IMSI)** in section **Safety instructions**.

3 INSTRUMENT VIEWS

3.1 GENERAL



1 Refer to the **Step X00 Instruction manual and safety instruction** *in section* **Instrument overview / General views / VID head**.





Observe the UNHT³ head.

Fig.1 UNHT³ head (bottom of the upside down head)

- A.1 Reference bar
- A.2 Reference
- A.3 Reference Dumbbell
- A.4 Reference glass block support
- A.5 Indenter bar
- A.6 Indenter
- A.7 Indenter Dumbbell
- A.8 Indenter glass block support
- A.9 Steel base



When dumbbell (A.3 or A.7) is compressed by the special exchange tool (B.1), this releases the corresponding reference or indenter spring (located inside the corresponding glass block A.4 or A.8) and allows inserting or removing the reference (A.2) or the indenter (A.6).

Carefully observe the tools which should be used.



Fig.2 Special exchange tool V2 orientation relative to head orientation (bottom of the upside down head)

B.1 Special (self-closing) indenter/reference exchange tool V2 (version 2)

has a specific design which needs to be understood before proceeding with an indenter or reference change. This tool is not symmetrical, it has a locating tab on either side:

• 1 large (B.2) which includes 2 legs (B.3)

AND

 1 small (B.4) which contains a pin of half-moon section (B.5)

- **B.2** Larger locating tab with **B.3** Legs x2
- B.4 Smaller locating tab with
- B.5 Pin of half-moon section



C.1 (standard) self-closing tweezers

3.4 INDENTER-REFERENCE SPACING DISTANCE

The spacing distance between the standard indenter and reference is:

2.7 ±0.1 mm (Fig.3).

The spacing distance between long shaft indenter and reference used with optional liquid cell is:

9.3 ±0.1 mm (4).

However, the longest spacing distance: 9.3 ± 0.1 mm (Fig.4) could have been ordered with standard (short shafts) indenter and reference.





Fig.3 Standard indenter/ reference distance

Fig.4 Long shaft indenter/ reference distance

4 PATENT

The Anton Paar Ultra Nanoindenation Tester (UNHT³) design includes active top referencing and 3 sensors and is patented under the following number:

US Patent 7,685,868 B2: Measuring head for nanoindentation instruments and measuring method

5 INSTRUMENT & MEASUREMENT PREPARATION

5.1 WARNING: REMOVE HEAD BOTTOM PROTECTION COVER



Fig.5 Head bottom with protection cover

Before operating the instrument, remove the head bottom protection cover, **Aavoid** any collision with the indenter/ reference:

Holding/retaining the cover, unscrew its 2 hand screws and then \triangle doing **only vertical movement**, pull down and remove the cover.

(to remount the cover, section 9)



The universal adapter 2 lateral holes should be placed in front or at the rear.

<u>Mounting</u>

The universal adapter **should** be mounted on the motorized Z table with a large hole located at the front left side:

> Insert into the large left front and right rear holes and tighten the 2 screws with an Allen key (3 mm) to lock the adapter on Z table.

5.3 SWITCHING ON THE INSTRUMENT

Switch on the instrument; refer to the **Step X00 Instruction manual and safety** *information* in section **Operating the instrument / Switching ON/OFF**. Then start the Anton Paar *Indentation Software* by double clicking with on the acquisition system desktop.

Select instrume	nt]
Use instrumer	nt :				1
Step - UNHT	S/N: 50-00	705		-	(
 Image: A start of the start of	ОК	×	Cancel		

If the platform is equipped with 2 indentation heads, select **"Use instrument : > Step - UNHT S/N: #"** (UNHT³) from drop-down menu.

5.4 POSITION CALIBRATION OF EACH OBJECTIVE

The position of each available objective¹ is calibrated before leaving the factory. However, to perform a new calibration, refer to the **Common Scratch & Indentation software reference guide** in section **Managing the instrument / Hardware configuration / Control unit & modules tab / I/O card**.

5.5 VIDEO SCALE CALIBRATION OF EACH OBJECTIVE

The video scale of each available objective¹ is calibrated before leaving the factory. However to perform a new calibration, refer to the **Video software reference guide** in section **Using the Video software / Toolboxes / Objective / Video scale calibration**.

¹ Each objective is mounted on the video microscope turret.

5.6 **INDENTERS/REFERENCES**

If the indenter/reference is not yet mounted or needs to be changed, read the following sections.



 \bigwedge Each time the indenter is changed on the head:

- The corresponding indenter in use **should** be created and/or selected in the software (section 5.6.2).
- The calibration of the distance between the indenter and the video microscope **should** be performed (section 5.10).
- An ADO **should** be successfully (\Re) performed (section 5.11).

🕂 Be sure that each indenter in use:

- Is **not** too worn out or damaged for the application; periodically verify the indenter wear by performing a calibration (section 5.13).
- Is **not** dirty; periodically or if necessary, clean the indenter extremity (sections 5.6.1.3 and 0).

5.6.1 **DISMOUNTING/MOUNTING**

🕂 It is advised to **read** all following sections in chronological order before manipulating the head.

5.6.1.1 Removing the head

🔨 Before unplugging the head connectors, **close** the *Indentation Software*, **shut down** the acquisition system by software (Windows[®]) and then **switch off** the instrument using the main power switch; refer to the Step X00 Instruction manual and safety information in section Operating the instrument / Switching ON/OFF.







Hold the stand (not shown on images) until it is screwed on the head as follows.

Place the head (red) stand by inserting its 2 captive screws into the head holes with an Allen key long part (3 mm) and tighten these 2 screws, in order to lock the stand to the head.

A Risk of damage

The head is equipped with 2 rear pins of a few millimeter inserted into the measurement assembly frame. Therefore **carefully follow** the operations below to avoid that the head fall down and be damaged during its removal.





Unscrew the 2 head **upper** captive screws with the Allen key long part to release the head the head is still maintained (pushed) with its 2 rear pins inserted into the frame.





From pushing the stand, grab it and carefully pull out the stand, holding it vertically: A end of pins, until the head is removed from the frame.





steps.



Return upside down the head by its stand and with other hand also hold the head by its rear side, near the stand (on head frame cover).

Keeping the hand holding by the rear side, move the other hand over the bottom of the head (on head frame cover) without touching the indenter and reference.



With the hand holding the head bottom (by frame cover), **Carefully place** the head upside down **on its stand** and on the **stable** desk.



The head is ready to mount or change its indenter/reference.

5.6.1.2 Removing the indenter/reference

Carefully read section 3.3 (indenter & reference details for handling) before manipulating the head assembly.



On a stable desk, with the head is placed upside down on its stand; see the previous section 5.6.1.1

Perform all the following manipulations extremely carefully, as there is a risk of damaging the UNHT³. Avoid shaking, vibrating the head assembly and avoid any collision with it.

Avoid any collision with the indenter/reference and avoid touching the indenter/reference extremity during the following operations.



Have special exchange tool V2 (B.1) and (standard) selfclosing tweezers (C.1) at hand.



Place your forearms/elbow on the desk to provide greater stability during delicate manipulations.



Lean your fingers lightly on the head casing for better stability.

Open (press) widely the special exchange tool V2.

Carefully approach with the special exchange tool V2 opened and insert its 2 legs (B.3) around and along the indenter glass block (A.8).

A Be careful **not** to press on the indenter glass block support.

First place the larger locating tab (B.2) on the steel base (A.9) and press lightly on it with the thumb (shown by larger arrow). Keep holding for the following steps.



Fig.6 pin of half-moon section insertion zoom



and place the smaller locating tab (B.4) on the steel base in front of the glass block. Press lightly on the smaller locating tab (shown by arrow) and keep holding for the following steps.



Slowly release the special exchange tool V2.

This releases the spring in the glass block and allows the extraction/insertion of the indenter.

Do **not** move the UNHT³ head with the special exchange tool V2 in place.

End of special exchange tool V2 installation.



Still press lightly on both larger and smaller locating tabs and keep holding.

With the other hand take the (standard) tweezers.



Slowly pull the indenter **vertically** up and release it; it should smoothly slide out from the glass block.

Being careful to **not** touch any part of the indenter and reference axes, **gently** open and remove the special exchange tool V2.

5.6.1.3 Cleaning the indenter with isopropanol

 Λ It is recommended to clean the indenter when it is **not** mounted on the head.

Hold the indenter by its shaft using the self-closing tweezers.



For the cleaning details, refer to the **Step X00 Instruction manual and safety** *information* in section **Upkeep and cleaning / Cleaning the indenters**.

5.6.1.4 Installing the indenter/reference

Choose a suitable and cleaned indenter (section 5.6.1.3) for the indentation measurement.



Fig.7 Berkovich indenter

E.g. a **Berkovich** indenter is chosen; the image shows a serial number **B**-L 89 but in this manual, the **B**-Q 03 will be used.

Go to section 5.6.1.2 to carefully read all warnings and instructions until **End of special exchange tool V2 installation**. Then come back here and continue to read the following steps.



Carefully take the indenter by its head (wider part ending with the indenter diamond extremity) in the (standard) self-closing tweezers.



Still press lightly on both larger and smaller locating tabs and keep holding.

Make sure that the V-shaped notch¹ on indenter shaft faces the head rear and is perpendicular to the glass block length.

With the (standard) selfclosing tweezers, insert the indenter into the dedicated indenter glass block opening; it should smoothly slide into.

Being careful **not** to touch any part of the indenter and reference axes, **gently** open and remove the special exchange tool V2.

The indenter is locked as the released spring pushes the dumbbell into the V-shaped notch (inside the glass block).

¹ A On the other side of the indenter shaft, there is **no** notch - only a flat (dedicated for another measuring head), do **not** orient this flat facing the head rear. Otherwise the indenter will **not** be locked.

5.6.1.5 (Re)installing the head

A Perform the manipulations described in section 5.6.1.1 (removing the head) in the **reverse order** to (re)mount the head (together with the mounted indenter and reference), **being careful** of the following steps:

- Push on the stand while tightening the 2 head locking screws.
- Hold/retain the stand while unscrewing and removing its 2 locking screws to avoid that the stand fall down and damage the head/instrument.
- Only after the reconnection of the connectors, switch on the instrument and then restart the software.



5.6.1.6 Cleaning the indenter by indentation

The indenter cleaning has been previously done before installing the indenter on the head (section 5.6.1.3). However, an indentation at 50 mN in copper can be performed to remove any remaining dirt.

5.6.2 MANAGEMENT

Create and/or select the indenter which is mounted on the head ("**Indenter in <u>use</u>**" drop-down menu) as follows.

Select **"Instrument > Indenters..."** from menu bar to open the following *Edit indenters* window (Fig.8).

Edit indenters		
Configuration		
Indenter in <u>u</u> se		
Berkovich [B-Q 03] (22.05.2014)		
<u>C</u> onfigured indenters		
Berkovich [B-P 05] (11.01.2014) Berkovich [B-Q 03] (22.05.2014)	+	Add
1		Edit
Does the indenter	-	Remove
aiready exist in <u>C</u> onfigured indenters?	⇒	Import
		Export
	→	XPress
✓ <u>о</u> к	×	<u>C</u> ancel

Fig.8 Indenter(s) creation and/or selection

Define a new indenter
Select indenter type
Vickers
Berkovich
Cube Corner 🗟
Spherical
Knoop Flet Durish
Flat Punch
✓ <u>O</u> K <u>Cancel</u>

Fig.9 Berkovich type is selected

If the indenter already exists in <u>Configured indenters</u> and a new calibration is **not** necessary, <u>select the indenter in use</u>, p. 35 and later **skip** section 5.13 (calibration).

OR

If the indenter already exists in <u>Configured indenters</u> and a new calibration is necessary, it is advised to <u>create a new indenter</u> below.

OR

If the indenter does not exist in <u>Configured indenters</u>,

create a new indenter

by clicking <Add> button.

This *Define a new indenter* window appears.

Double click on the new indenter type.

The following *Indenter properties* window appears (Fig.10).

Indenter properties	
General information Type Berkovich Serial number B-Q 03 Material Diamond	Tip Calibration curve Ap=f(hc) This indenter has not yet been calibrated. It is highly recommended that it is calibrated before use.
Beta 1.034 Poisson's ratio 0.07 Young's modulus 1141.00 GPa	
Calibration Date No valid date	Fit Method
Click here to start the indenter calibration procedure	 Edit Fit Method
	OK Cancel

Fig.10 Define at least new indenter serial number

Refer to the new indenter certificate to type its <u>Serial number</u> 1 in the corresponding filed name (e.g. B-Q 03).



- Choose another material than diamond; type/edit or select it from "Material" combo-box.



- Theoretical *Poisson's ratio* and *Young's modulus* field values can be changed.
- Beta field coefficient value can be changed.

Click <OK> button (validation and creation).

¹ See section 0 to localize the serial number directly on the indenter.

Edit indenters						
Configuration						
Indenter in <u>u</u> se	Indenter in <u>u</u> se					
Berkovich [B-Q 03]						
<u>C</u> onfigured indenters						
Berkovich [B-P 05] (11.01.2014) Berkovich [B-Q 03] (22.05.2014)	+	Add				
Berkovich [B-Q 03]		Edit				
	_	Remove				
		Import				
	¢	Export				
	÷.	XPress				
<u>√</u> <u>о</u> к	×	<u>C</u> ancel				

Fig.11 New indenter is selected



If <<u>O</u>K> button is directly clicked, the new indenter creation (in <u>Configured indenters</u>) is validated and saved but **not** selected; therefore,

select the indenter in use

from **"Indenter in <u>u</u>se"** dropdown menu

Indenter in <u>u</u> se	
Berkovich [B-Q 03] (22.05.2014)	
Berkovich [B-P 05] (11.01.2014) Berkovich [B-Q 03] (22.05.2014)	
Berkovich [B-Q 03] Berkovich [B-O 03] W2 05 2014)	

and then click < OK > button (validation and saving).

5.7 SAMPLE HOLDER

If necessary, the height of the beam (where the measuring and imaging heads are mounted on) can be adjusted, depending on the height of the sample/sample holder in use; refer to the *Step X00 Instruction manual and safety information* in section *Operating the instrument / Adjusting beam height*.

Reminders:

- See Universal adapter mounting/dismounting (section 5.2).
- Each time the sample (the same or a different one) is re/installed in the sample holder, an ADO **should** be successfully ([®]/₈) performed (section 5.11).
- **Avoid** any collision with the heads, indenter/reference and objective(s) when a sample/holder is removed or installed/mounted.

The universal sample holder is used as an example in this document.



Fig.12 Sample solidly mounted in universal sample holder

Mount the universal sample holder (2 screws¹ under clamp jaws) on the universal adapter.

Then install the sample as level as possible in the clamp jaws and tighten the 2 hand screws **G** to make sure that the sample remains solidly clamped.

¹ Use Allen key 2.5 mm.
5.8 POSITION CONTROL

Select **"Instrument > Position control..."** from menu bar or click *Position control* icon on toolbar to open the *Position control* window (Fig.13).



After each power on of the Step, the software requires a reinitialization of the motorized tables before moving them.

Verify the sample height to avoid a collision and then click <Yes> button.

The motorized X and Y tables move to their extremities; wait.





The <u>Methods to move</u> the motorized tables/sample either under the indenter, the video microscope or the optional AFM^1 are described on the following page (p. 39).

Read also Motorized video microscope module, p. 40.

¹ Refer to the Common Scratch & Indentation software reference guide in section AFM measurement or refer to the AFM Wide Scan user manual.

Methods to move

Some of the following methods are also applicable to other places in the software, **exceptions:** All '*arrow*' icons and vector move buttons are only available here in the *Position control* window (Fig.13).



Vector move buttons

Click <Vector move> button and set values in the window which appears to move accordingly motorized X and/or Y tables. Clicking <Move full> button is similar but with motorized Z table in addition (the moving speed is automatic) \bigwedge verify the sample height to avoid collision.

Backlash correction

To achieve better final position accuracy, one can click <Backlash> button.

<u>Closing</u>

Click <Close> button to exit *Position control* window. The Z table is retracted (moving down).

¹ Each single hit on one of 'arrow' keys corresponds to a single Minimum displacement: Click 0.250 μm to decrease it (min.) or 50.000 μm to increase it (max.).

Motorized video microscope module

If the video microscope module is motorized, \triangle ensure first that there is no

risk of collision and then click to lower the video microscope.



5.9 VIDEO

To open the following *Video* window (Fig.14) from *Position control* window (Fig.13):

- *Click to move under the microscope* icon. The sample moves under the video microscope (if it is not already there).

OR

- Click <Open video> button.



Fig.14 Video window

Click *Cursors* \square icon on toolbar to open the following *Cursors* toolbox.



Click <*Crosshair*> + button to display the video crosshair on the middle of the screen (Fig.14).

(Click *<Scale>* button to display the scale information on the screen.)

5.9.1 **VIDEO MICROSCOPE**

Click *Objectives* **K** icon on *Video* toolbar (Fig.14) to open the following *Objective* toolbox.



When either the Position control window (Fig.13) or the Analyze indentation window (Fig.53) is opened, the displayed magnification of the objective should match the magnification of the hardware objective into working position; otherwise see section 5.4.



Manipulate only the turret (part with knurling, indicated with grey arrows on image) to rotate objectives into their working (vertical) position.

Front view

5.9.2 LIGHT ADJUSTMENT



There are 2 ways to adjust the light source of the video microscope:

Manual mode, click < Manual light adjustment> button and adjust the intensity in % by 05 dragging the cursor.

OR

Automatic mode, click *<Automatic light* adjustment> PC. button.

5.9.3 FOCUS ADJUSTMENT

When raising the motorized Z table under the video microscope for the focus adjustment, **be constantly vigilant to avoid** a collision between the sample surface and the objective **but also** between the sample holder/sample surface and the indenter/reference.

Use the *Position control* window (section 5.8) to lower or raise the motorized Z table to adjust the focus on the sample surface using the *Video* screen (Fig.14).



5.10 INDENTER-MICROSCOPE DISTANCE CALIBRATION

This section explains how to prepare and perform the calibration procedure of the distance between the indenter and the video microscope. For more details and information about all features or for a microscope-AFM (optional) distance calibration, refer to the *Common Scratch & Indentation software reference guide* in section *Managing the instrument / Calibration (Common tabs) /* there are several sub-sections.

The round copper sample (provided) is usually used for this calibration.

As an example in this section:

- Another type of measuring head with Vickers indenter is used for the print screens, but the following explanations remain the same or are adapted for UNHT³ with Berkovich indenter.
- The video microscope is equipped with the standard 5x objective and optional 20x and 100x objectives. See section 5.9.1 to know how to properly rotate the video microscope turret during the following calibration procedure.

5.10.1 SAMPLE HOLDER



Fig.15 Copper sample/stud solidly mounted in universal sample holder

Properly mount the sample; read section 5.7 for explanations and warnings. Select **"Instrument > Calibration..."** from menu bar to open the following *Step - UNHT - Calibration* window.



Fig.16 Distance tab

Calibrate Indenter Microscope distance		>
1- Define mark settings	3 - Calibrate	
+ Install a sample in the sample holder	+Refine position and focus for each objective, prior	backlash recommended
+ Find a suitable location on the sample,	Objective X5	-
position control can be used	X distance 48732.367 🚔 μm	Open video
•	Y distance 183.336 😭 μm	Speed
Position control	Z distance -1088.417 📮 µm	Very Fast
	Objective X20	Fast
+ The sample will be marked by the indenter	X distance 48508.499 μm	Medium Slow
Mark Load	Y distance 155.333 📄 µm	Very slow
0.50 N Reset to <u>d</u> efault	Z distance -985.017 📄 µm	
Charles and a Carlot	Objective X100	Forward Up
+ Select the number of indents	X distance 48460.435 🚔 μm	
Single indent	Y distance 161.033 🍯 μm	< Lett Right > Op CTRL → Pg
Space between indents	Z distance -974.575 📑 µm	Down
50.00 ਦ µm 🧲 Reset to <u>d</u> efault		Backward Minimum displacement 2.000 µm
2 - Mark the sample		
🐐 Mark sample		Return to origin
X Cancel		Save and Close

Fig.17 Calibrate Indenter Microscope distance window

See the following procedure explanations.

1. Define mark settings

Location

If it has not been done previously, move the sample under the video microscope to find a location¹ free of previous indents and clean, in order to perform the indentation mark(s) for the calibration.

To do this, click <Position control...> button (see explanations from Fig.13) and also use the *Video* window (section 5.9).

Load

In *Mark load* field, set a suitable value to generate a visible indent in the sample: **50 mN** (0.05 N) is advised for copper sample and Berkovich indenter (Fig.17 shows 0.5 N (500 mN) which is suitable for another measuring head/ indenter).

INFORMATION: If the load value is too big, the mark will be too large to fully see it on the Video screen for a high magnification objective (e.g. x100). But if the load is too low, the mark will be too small to see it on the Video screen for the x5 magnification objective.

Number of mark(s)

E.g. <single indent> radio button is selected, only one indentation mark will be performed (we assume a previous calibration has been done, otherwise the 4 indents in triangle shape should be used to better localize them on the sample with the *Video* screen).

2. Mark the sample

Click <Mark sample> button to start the automatic indentation process, in order to mark the sample.

If the sample is located under the video microscope, a *Warning* window asks to move the sample under the indenter. A **Verify** the sample height to avoid a collision and then to continue, click <Yes> button (sample moves under the indenter; wait).







As soon as the sample is under the indenter, the *Microscope distance calibration* window appears for the automatic indentation process. This window is similar to the ADO window (see similar explanations from Fig.25) but here it is used only to mark the sample (automatic parameters).

At the end of the automatic process, another *Warning* window asks to move the sample under the indenter (for

the calibration). \bigwedge **Verify** the sample height to avoid a collision and then to continue, click <Yes> button (sample moves under the microscope; wait).

3. Calibrate

Each available objective of the microscope should be calibrated one at a time by centering the indentation mark (previously done) on the *Video* screen. It is advised to start with the smallest magnification x5.

Calibration procedure



Fig.18 Real pyramid mark (e.g. centered with x100 objective)

Objective X5				
X distance	48734.363	▲ ▼	μm	\checkmark
Y distance	193.335	•	μm	
Z distance	-1033.434	•	μm	

INFORMATION: The sample mark under the Video screen is identical as this image (Fig.18), that is to say a pyramidal mark made with Berkovich indenter in copper sample. The following images show a square mark, made with a different measuring head and indenter (Vickers), but the explanations remain the same.

Correctly rotate the microscope turret to place the objective into working position \rightarrow The corresponding *Objective* X# (e.g X5) *area* fields become active with the current X, Y, Z distance values (coming from the previous calibration).



Fig.19 Mark centered on Video screen with x5 objective



Fig.20 Mark centered with x20 objective



Fig.21 Mark centered with X100 objective

INFORMATION: In case of < Four indents in triangle shape> radio button has been previously selected, center the indentation mark located **at the middle** of the triangle shape (for each objective). E.g pyramidal marks made with Berkovich indenter.



Fig.22 Middle mark centered

Use <u>Methods to move</u>, p. 39 (**exceptions:** All 'arrow' icons and vector move buttons) to precisely center the middle of the indentation mark on the *Video* screen crosshair and also correctly adjust the focus on the sample surface (if necessary, click <Open video> button to reopen the *Video* window). Then click *Redefine calibration distance indenter microscope from this new point* icon in the active *Objective X#* area to valid the current positon/focus \rightarrow a 'tick' icon appears.

One at a time, repeat the previous calibration procedure for the remaining available objective (e.g. x20 and then x100).

 3 - Calibrate + Refine position and focus for each obj 	ective, prior backlash recommended	
Objective X5		
X distance 48734.363 🚔 μm	V Open video	
Y distance 193.335 📄 µm		
Z distance -1033.434 📄 µm	Speed Very Fast	
Objective X20	Fast	
X distance 48508.633	V Medium	
V distance 151 302	Slow	When all available
7 distance 005 542	Very slow	objective(s) are
2 distance -985.342 📄 µm	Keyboard control	centered and validated
Objective X100	Forward Up	
X distance 48459.698 膏 μm	< Left Right > Up	click <save and="" close=""></save>
Y distance 159.301 🚔 µm	CTRL → Pg	button which becomes
Z distance -977.475 📮 µm	Down	active (validation).
	Backward	
	Save and Close	
- MHT - Calibration]
ance Platform Center		
denter-microscope distance	Microscope-AFM distance	
X distance	X distance	
😝 48459.698 🚍 μm	\leftrightarrow 0.000 🚔 μm	Only the new
Y distance	Y distance	calibration values of th
159.301 🚔 µm	0.000 🚔 µm	highest magnification
××		(0.0, 100) are conied
🕴 Calibrate	Calibrate	in X and Y distance
1 Decision de Carden	1000 December of Section	fields
Keset to defaults	Reset to defaults	neids.
Edit		Click < OK > button
		(validation and saving)

Fig.23 Distance tab with new values

5.11 ADJUST DEPTH OFFSET (ADO)

Each time the indenter/reference or the sample is changed, or the topography on the sample area varies, an ADO procedure should be started:

- Because of the vertical variation between indenter and reference.
- To optimize duration of measurement(s).

On toolbar, the icon shows the state of the last ADO:

Successful \rightarrow ADO & icon with a green tick

OR

Not-successful \rightarrow ADO $\stackrel{1}{\downarrow}$ icon with an arrow

See the following sections which explain how to perform a successful ADO. For more information about the ADO and its detailed features, refer to the **Indentation software reference guide** in section **Adjusting depth offset (ADO)**.

5.11.1 FIND ADO LOCATION

Move the sample under the video microscope to find a location¹ free of previous indents and clean, in order to perform the ADO indent; use the following tools:

- Position control (section 5.8)
- Video (section 5.9)

5.11.2 STARTING

Select **"Instrument > Adjust depth offset..."** from menu bar or click ADO $\frac{1}{200}$ / $\frac{2}{200}$ icon on the toolbar to start the ADO procedure.





5.11.3 PROCEDURE



If the sample is located under the video microscope, this *Warning* window appears.

Verify the sample height to avoid a collision.

To continue click <Yes> button, the sample moves under the indenter; wait.

The following Adjust depth offset parameters window (Fig.24) appears.

Adjust depth o	ffset parameters				
	Surface detection parameter	ers			
	Approach <u>s</u> peed		Contact force	2	
	25000.0 📄 nm/min		30.00 💌 µN		
	Characterization force		Contact stiffr	ness threshold	
	500.00 🗭 µN		150.	0 🖶 μN/μm	
	Indenter	Reference			
	Pre-approach	Pre-appro	ach	Contact force	
	10 💭 %		40 🚔 %	500.00	μN
	Presets				
	Default			~	G
			v 0	K X	Cancel

Fig.24 ADO parameters

Set the ADO parameters according to the sample in use/application. Parameters shown on Fig.24 are coming from the "**Presets** > **Default**" drop-down menu and are generally suitable to perform a successful ADO (\Re) with hard samples.

It is advised to never set a value greater than 70 % in *Reference* area *Pre-approach* field and lower than 5 % in *Indenter area Pre-approach* field.

Where 100 % means lower position of actuators displacement, it is the rest position.

Initial positions:

- Motorized Z table is at 0 mm, in lower (retracted) position.
- Indenter and reference are at 100 %, in lower (rest) position.

Click <OK> button. The following *Adjust depth offset in progress* window (Fig.25) appears and an automatic procedure starts.



 This extra UNHT Approach Monitor window¹ is also present during the measurement.

Automatic procedure

Adjust depth offset in progress
+ More Info
Status
Approaching Table Z
Approaching reference
Approaching with the indenter
Contact
Stabilization
Sample characterization
0%
Could not reach the surface
Raising the indenter
Parameters
Fn contact : 30 µN Characterization Force : 500 µN Approach speed : 25000 nm/min FnRef contactload :500.000 µN Stiffness Threshold : 150 µN/µm
Close window automatically
X Cancel

Wait for the end of the automatic procedure sequences (each current status is shown in real time). Otherwise if necessary, click <Cancel> button to stop any movements and to cancel the ADO but then, another ADO should be started again until it is successfully (\$\vec{se}\$) performed.

Fig.25 Adjust depth offset in progress window

¹ Can be deactivated with the .ini file.

At the end of the automatic procedure, the sample position **should** be shifted¹, in order to **avoid** performing the following calibration or measurement indent inside the current ADO indent. The following window (Fig.26) appears.

Sample displacement after ADC)			
It is recommended to move th set a minimum displacement	e sample av	vay from the	a ADO im	pact zone:
X motorized table		Y motor	ized table 0.000 🚔	μm
	~	ОК	×	Cancel

Fig.26 Sample distance displacement after ADO window

Set a **min.** displacement value (depending on current sample material: Approx. 20 x h_m) in X and/or Y motorized table field(s) and click <OK> button to move accordingly; wait.

OR

Click <Cancel> button and **use** the *Position control* (section 5.8) to **move** the sample into another suitable area.

Control that the ADO has been successfully performed (\Re on toolbar). Otherwise see section 10.4 about a non-successful ADO ($\frac{1}{1000}$ on toolbar).

¹ Not too much, otherwise the sample topography may vary. IMPORTANT: Pay attention to the displacement unit (e.g. μm here), which is set in the options; refer to the Common Scratch & Indentation software reference guide in section Customizing options / Preferences tab (units).

5.12 HEAD DYNAMIC CALIBRATION (WITH SINUS MODE)

The head dynamic calibration, only available with the Sinus mode, should have been performed at least once in order to be able to perform any Sinus measurements, including indenter Sinus calibration.

First it is recommended that an ADO have been successfully (\Re) performed (section 5.11).

Select "*Instrument* > *Hardware configuration...*" from menu bar to open the following *Step - UNHT - Hardware configuration* window and select *Dynamic ranges* tab.

	User channels	Control unit & modules	Motors Ir	nstrument adjustment	Indenter ranges	Dynamic ranges
Dynamic						
+ Comman	nd	+ Dynamic coeffici	ents			
Fn coef	(fine)	Mobile M	ass			
2.09	14 🍨 mN/V	A. 2.10	g 🌩 g	Click her	e to start dynamic	calibration
[()	Spring Sti	ffness		procedure	
33.4	(large) 98 🚔 mN/V	B. 2897.6	5 N/m	ų 🖌	Calibrat	e
		C. 0.036	6 Ns/m			
			<u> </u>	-		
		I				
				\checkmark	Validate ra	nge values
				\checkmark	Validate ra	nge values
				\checkmark	Validate ra	nge values
				4	Validate ra	nge values
				~	Validate ra	nge values
					Validate ra	nge values
				~	Validate ra	nge values
				~	Validate ra	nge values

Fig.27 Dynamic ranges tab

Click <Calibrate> button to open the following *UNHT Head Dynamic Calibration* window; if already performed once, this calibration window (Fig.28) contains the last validated values - including the 3 values of Fig.27 - and curves of the previous calibration.



Fig.28 UNHT Head Dynamic Calibration window

Click <Start dynamic calibration> button to start the following semi-automatic calibration procedure.

Then the following additional *Head position* window (Fig.29) appears.



Fig.29 Head position window

Reminder:

The spacing distance between the standard indenter and reference is: $2.7 \pm 0.1 \text{ mm}$

Click <Position control...> button (see from Fig.13) and position the motorized X and Y tables in order the sample extremity is located under the UNHT³ reference only, **not**

under the indenter. A If the motorized Z table is manually raised (to better targeting), **never** touch the sample surface with the reference (/indenter).

Before clicking <Ok> button to continue, be aware that in UNHT Head Dynamic Calibration window (Fig.28), <STOP> button will be active and can be clicked if necessary to interrupt any movement.

Then the motorized Z table automatically approaches (raises) until the sample surface contact the reference.

An *ADO in progress* window appears (it is similar than Fig.25, but here it is used only for the following head dynamic calibration; there is no need to set any ADO parameters).

In UNHT Head Dynamic Calibration window (Fig.28)



- Positioning the indenter... circle status blinks green; wait a few sec, the indenter is positioned at the displayed value.
- 2. Mass Calibration running... circle status blinks green; wait a few sec, the mass calibration runs (the curves in the 2 upper graph areas are displayed/updated).

UNHT Head Dynamic Calibration	
Calibration Start Dynamic Calibration	 Positioning the indenter Mass Calibration running phase calibration running : 11.0 [hz]

3. *Phase calibration running* circle status blinks green; wait some minutes:

The phase calibration runs: the frequency value increases (till 100 Hz) and the measurement curves are displayed on real time in the *Results* bottom graph area.



Av configuration	User channels	Control unit & modules	Motors	Instrument adjustment	Indenter ranges	Dynamic ranges	1 4
Dynamic				,	,		
+ Comman	d	+ Dynamic coefficie	ents				
Fn coef (fine)	Mobile Ma	iss				
2.09	4 🌒 mN/V	A. new 2.190 Spring Stif	fness	Click her	re to start dynamic procedure	calibration	
Fn coef (33.49	large) 98 🚔 mN/V	B. new 3017.94	N/i	m 🐐	Calibrat	e	
		C. new 0.0410	N.s	/m			
				~	Validate ra	nge values 🛛 🗲	
				•			
					ОК	X Car	ncel

Back in *Hardware configuration* window, the 3 new *Dynamic coefficients* field values are automatically updated according to this last performed dynamic calibration.

IMPORTANT:

- Do not forget to click <Validate range values> button (validation of these new range (calibration) values) and then click <OK> button (saving).
- As new range values have been validated, a new and successfully (\Re) ADO should be performed (section 5.11).

5.13 CALIBRATION OF INDENTERS (QUASISTATIC OR SINUS)

The calibration of indenters can be either performed with the standard quasistatic **or** Sinus method; \bigwedge in case the Sinus calibration is selected, the dynamic calibration should have been previously performed (section 5.12). The quasistatic calibration is used as an example in this document, but specific explanations for the sinus calibration are described in addition.

 \bigwedge It is highly recommended to perform a calibration of the indenter in use if:

- It has never been done.
- It has already been done but some time ago, the indenter might have worn out in the meantime.

1 If the indenter is not calibrated, its geometry is approximated to a perfect shape tip. For Berkovich and Vickers indenters, the theoretical function $A_p = 24.5 h_c^2$ is used to approximate the area function.

 Λ The certified reference Fused Silica sample **should** be used for this calibration.

The indenter calibration procedure is performed in order to define the A_p function of the indenter vs. the contact depth; it basically includes in the following order:

- Automatic ADO (with automatic move in X direction straight after the ADO)
- calibration measurements:
 - a matrix of indents for a **quasistatic** indenter calibration
 40 (5x8), 45 (5x9) or use as an example in this manual, 60 indents (10x6):



OR

 a single row of a chosen number of indents for a **Sinus** indenter calibration (advised number is 5 indents)

• contact area determination (verification/redefinition of all contact points of the matrix of indents, removal of bad indents and fit method)



Properly mount the sample; read section 5.7 for explanations and warnings.

Fig.30 Fused Silica sample/stud solidly mounted in universal sample holder

5.13.2 FIND ADO & CALIBRATION AREA

Move the Fused Silica sample under the video microscope to find an area¹ free of previous indents and clean, in order to perform the ADO indent and then the matrix of indents for the calibration; use the following tools:

- Position control (section 5.8)
- Video (section 5.9)

¹ The reference **should** be able to land on the sample as shown here.



Select **"Instrument > Indenters..."** from menu bar to open the following *Edit indenters* window (Fig.31).

Edit indenters		
Configuration		
Indenter in <u>u</u> se		
Berkovich [B-Q 03] 🔹		
<u>C</u> onfigured indenters		
Berkovich [B-P 05] (11.01.2014) Berkovich [B-Q 03] (22.05.2014)	+	Add
Berkovich [B-Q 03]		Edit
	_	Remove
		Import
		Export
	+	XPress
√ <u>о</u> к	X	<u>C</u> ancel

(section 5.6.2), so it is automatically selected (highlighted) in <u>Configured</u> *indenters*. Click <Edit> button.

The new "Indenter in use" has been

previously created and selected

The following *Indenter properties* window appears (Fig.32).

Fig.31 Edit indenter for calibration

In <u>Configured indenters</u>: If a same indenter with an old calibration date is selected and then edited by clicking <Edit> button for a new calibration, the previous A_p curve (Fig.42) will be overwritten. Thus, it is advised to create a new indenter (section 5.6.2).

Indenter properties	
General information	Tip
Туре	Calibration curve
Berkovich	
Serial number	
B-Q 03	
Material	which indicates the section to an indicate of this bight.
Diamond -	This indenter has not yet been calibrated. It is nignly recommended that it is calibrated before use.
Beta	
1.034	
<u>P</u> oisson's ratio	
0.07 👻	
Young's modulus	
1141.00 💭 GPa	
Calibration	
Date	
No valid date	
	Fit Method
Click here to start the indenter	
calibration procedure	
🕴 Calibrate	Edit Fit Method

Fig.32 Perform indenter calibration

5.13.4 PROCEDURE

Click <Calibrate> button in Fig.32 to start the following indenter calibration procedure.

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

E.g. <Measuring a new matrix of indentation> radio button (quasistatic) is selected (by default).

OR

Select <Measuring a new matrix of sinus indentation> radio button (Sinus).

 If necessary, a previous saved calibration file *.mit* can be reloaded by selecting <Loading indentations from file> radio button (quasistatic) OR <Loading sinus indentations from file>

radio button (Sinus)

Then click <<u>N</u>ext> button.

Choose the indenter calibration parameters.

Automatic calibration of the indenter / Step 2			
2. Choose calibration parameters			
	+ Enter sample certificate value Plane strain modulus (E*) © E* Method + Or enter theorical values for your sample		
	Poisson's ratio Young modulus EIT Method		
	+ Choose the calibration mode :		

Oliver and Pharr mode		
🔘 Quick (40 inden	tations at 8 different loads)	
Intermediate (4)	5 indentations at 9 different loads)	
Intensive (60 inc	dentations at 12 different loads)	
O User protocol fi	e	
Sinus Mode		
Measurement ty	pe	
 Std (CSR Sinu 	s 40mN; 0.05 1/s; 1mN @ 5Hz)	
Measurements	5 🕷	
		ı
X <u>C</u> ancel	Previous	Next

With certified value -ADVISED

Select <E* Method> radio button, in its field set the *Plane strain modulus (E*)* value stated on the delivered certificate of the Fused Silica sample and press <Enter> key.

OR

With theoretical values

Select <EIT Method> radio button, in its corresponding fields set the *Poisson's ratio* and *Young modulus* values corresponding to the sample.

Then select the calibration mode:

E.g. the quasistatic mode, <Intensive> radio button (matrix of indents) is selected (by default).

OR

If previously the Sinus procedure has been chosen, the *Sinus Mode* <Std> radio button is selected:

Set the row indents number in *Measurements* field; advised value is 5. Every indent will be performed with Sinus Constant Strain Rate 0.05 1/s, load 40 mN, amplitude 1 mN, frequency 5 Hz.

And then click <<u>N</u>ext> button.

Automatic calibration of the indenter / Step 3			
3. Starting indentation			
+ The indentations are ready to run.			
+ Press "Next" to start.			
X Cancel			

>

6

Hour

Minute

>

30

21

Click <<u>N</u>ext> button.

The following window (Fig.33) appears.

Click <<u>S</u>kip> button to directly start the automatic calibration measurements (matrix of indents).

OR

Delay the measurements, e.g. to have the lowest noise environment (during the night):

Set the time at which the measurements will start and click <<u>W</u>ait> button.

Remaining time before starting the measurements is displayed Waiting .. 4 H 27 min 25 sec (blinks).

5.13.4.1 Measurements

Then *ADO in progress* window (Fig.25) appears. (Here, there is no need to set any ADO parameters; they are automatic and optimized for this indenter calibration).

<u>S</u>kip

And then *Indentation running*... windows (Fig.52) appears; wait for automatic matrix of measurements for the calibration are performed.

Wait Time

<

7

14 15

21 22

28 29 30 31

 \bigcirc

Calibration Time

July

16

23 24 25 26 27

10

17 18 19 20

Fri

11 12 13

Sat Sun

5

Fig.33 Wait time window

Mon Tue Wed Thu

1 2 3 4

8 9

Waiting...

Wait

E.g. quasistatic calibration matrix of indents is performed (totally 60 indents)

- Each row includes 2x [5 indents at the same load].
- The load decreases every 5 indent.
- The weakest load is reached at the 6th row.



Fig.34 Matrix with 5x video microscope objective

Once the last measurement has been completed, the following *Save a calibration file* window (Fig.35) appears.

5.13.4.2 Saving



Fig.35 Save a calibration file window

Calibration date

The date which is included in the file name (in addition to the indenter serial number) corresponds to the date when the calibration measurements started (can be modified).

The date which is included in the *Indenter properties* window (Fig.42) and next to the serial number of **"Indenter in use"** + <u>Configured indenter in Edit</u> *Indenters* window (Fig.44) corresponds to the date of the last matrix indent measurement.

Choose a location to save the file of the indenter calibration measurements on the acquisition system and click <Save> button.

5.13.5 CONTACT AREA DETERMINATION ADJUSTMENT



Fig.36 Contact area determination window

First all load indent automatic contact points should be verified/refined and then all bad indents should be excluded as explained in order in the followings sections.

It starts with the first indents¹ (e.g. 5) of the lowest $#1 \text{ load}^1$



Click <Set contact point> button. The following *Set the contact point* window (Fig.37) appears, see the explanations in the following sections.

¹ Not applicable for Sinus measurements (same load for all indents).

² With Sinus measurements, it could be less or more than 5 indents.

5.13.5.1 Automatic contact points

Each indent contact point (several with matrix) has been automatically defined by the software \rightarrow in Fig.37: Automatic positions of the red cursors on left graph area and *Detect contact point* \swarrow icon is inactive on toolbar. However, it is advised to verify and if necessary to refine (more precisely) each contact point for all current indents¹ (e.g. 5) of all loads² (e.g. 12) as follows.



Fig.37 Set the contact point window

Verify/refine contact point for each current load indent



In the indent selection area, it starts with the first indent #1 (highlighted). One at a time, select (highlight) each indent # to verify and if necessary refine its contact point as instructed in section 5.13.5.2.

Once the 5 current load indents has been verified/refined, click < OK > button (validation and saving of each indent current contact point).

Then, Select the next load, p. 70.

¹ With Sinus measurements, it could be less or more than 5 indents.

² Not applicable for Sinus measurements (same load for all indents).

Select the next load

In Contact area determination window (Fig.36):

Click <'next'> button to skip to the next load Indentations / 12, then click again <Set contact point> button and <u>Verify/refine contact point for each</u> current load indent, as explained in p. 69.

→ Repeat the previous instructions for all available loads.

Click <'previous'> button to skip back previous load (to verify/refine again contact points if necessary).

When all indent contact points of all loads have been verified/refined (e.g. $5 \times 12 = 60$ indents), remove the bad indents of all loads (those which are shifted and/or have a bad shape) as instructed in section 5.13.5.3.

5.13.5.2 Verify/refine contact point procedure

The best contact point is located where the **F** red curve <u>starts</u> to rise¹ and/or **h** green curve <u>starts</u> to peak. If the red cursor are not at this specific location, double click to refine the new and more precise contact point \rightarrow red cursors move accordingly (Fig.39).



End of the verified/refined contact point procedure.

(Clicking *Detect contact point* \checkmark active icon, recover initial automatic contact point \rightarrow icon \checkmark becomes again inactive).

¹ The higher the load the more difficult it is to see the **F** red curve rising, so use the **h** green curve instead to determine the best contact point.

5.13.5.3 Features and removal of bad indents



Fig.40 Bad indent #3 is removed

<u>Display</u>

Initially all *Indent #* boxes are checked in selection area.

By default <Multi> button is selected, otherwise click it to display each 5 indent $curve^2$ on the same time on the graph.

To display only 1 indent curve at a time on the graph, click <Single> button and

place the mouse cursor on a desired Indent # Indent $#_{\mathbb{N}}^2$ in selection area,

Indent #3 With <Multi> button selected, placing the mouse cursor on a *Indent* # Indent #1 Indent #2 highlights the corresponding curve on the graph.
<u>Graph area</u>

Different types of curves can be selected to show the 5 indents¹ of the **current** load:

- By default *Indentation curve i*con is selected to show **F** vs. **h** curves (Fig.40).
- or
- Click *Fn* 🚰 icon to show **F** vs. **time** curves.

or

- Click *Pd* 😭 icon to show **h** vs. **time** curves.

OR

Circles can be selected to show indents (e.g. 5) of **all** loads (e.g. 12):

Click Ap
 icon to show Ap vs. hc circles: The indents of the current load
 are represented by colored circles and the remaining indents of all other
 loads are represented by gray circles.

By default a linear scale is displayed (log lim icon unselected) or

click *log* icon (becomes is selected) to display logarithm scales



Click *Relevant only* \checkmark icon to show the relevant curves or circles \rightarrow the gray curves or circles are hidden from the graph (see above for gray circles, and see removal of bad indent below for gray curves/circles). Click again the selected icon \checkmark to display the 5 indent curves or all indent circles.

Removal of bad indents

E.g. in Fig.40 for the selected load: *Indentations # 3/12*, the *Indent # 3* is totally shifted (even with a correct contact point) and has a bad shape; uncheck each bad *Indent #* Indent #3 box to exclude them \Box Indent #3 \rightarrow On the graph area it becomes \Box Indent #4 gray \rightarrow not relevant (excluded for the A_p determination).

When all bad indents have been removed, click $\langle Next \rangle$ button in Fig.40.

Automatic calibration of the indenter / Step 5				
5. Validation of the results				
+ Check the indenter contact area curve. + Click on "Finish" to validate the results.				
Contact Area = f (Depth) 1.01E-12				
8.12E-13-				
6.11E-13-			The second se	
4.10E-13				
2.08E-13				
6.77E-15 6.82E-09 4.24E-08 x 1	7.80E-08	1.14E-07	1.49E-07	1.85E-07
Scale Mode :				
X <u>C</u> ancel			Previous	<u>F</u> inish

<u>step 5</u>

 A_p curve appears.

The *Scale Mode:* can be changed from <Linear> to <Log> and vice versa (click the desired radio button).

Click <<u>F</u>inish> button.

5.13.6 EDIT FIT METHOD



Fig.42 A_p curve appears in indenter properties window (linear mode)

The scale mode can be changed from linear $Ap \bigwedge$ icon to logarithmic $Log \bigoplus$ icon and vice versa (select desired icon). It is also applicable in the following *Edit fit method* window (Fig.43).

 A_p curve appears in the graph area of the *Indenter properties* window and uses by default the automatic BSplines Interpolation Fit Method. However it is possible to use other methods: Click <Edit fit method> button to open the following *Edit fit method* window.

¹ See <u>Calibration date</u>, p. 67.



Fig.43 Edit Fit Method window (logarithmic scale)

Several edit fit method can be chosen, click the following desired radio button; for further explanations, refer to the **Indentation software reference guide** in section $A_p(h_c)$ **Fit method of the calibration**:

<BSpline interpolation> radio button (standard by default)

OR

<Linear Interpolation> radio button

OR



OR



Once the desired method has been selected (and set for *Oliver & Pharr* and *Polynome*), click <OK> button in Fig.43 (validation).

Then click <OK> in Fig.42 (validation and saving).

Edit indenters		
Configuration		
Indenter in <u>u</u> se		
Berkovich [B-Q 03] (25.07.2014) ¹		An
<u>C</u> onfigured indenters		ind
Berkovich [B-P 05] (11.01.2014) Berkovich [B-Q 03] (22.05.2014)	+ Add	sel
Berkovich [B-Q 03] (25.07.2014) 4	Edit	
	- Remove	Otł <c< td=""></c<>
	Jmport	sav
	Export	G
	XPress	U
<u>о</u> к	X <u>C</u> ancel	

Fig.44 Indenter B-Q 03 25.07.2014 has been calibrated and is (still) selected

And then verify that the indenter in use, which has just been calibrated, is (still) selected.

Otherwise select it and click <<u>O</u>K> button (validation and saving, section 5.6.2).

The calibration date¹ has been automatically added next to the indenter serial number name.

¹ See <u>Calibration date</u>, p. 67.

INDENTATION MEASUREMENT PROCESS 6

This section explains how to prepare and carry out an indentation measurement.

For the following indentation measurement process example, the Fused Silica sample (provided) is in use.

6.1 **SAMPLE HOLDER**

 \bigwedge The sample **should** be solidly mounted to prevent movement when under load.



N Properly mount the sample;

explanations and warnings.

read section 5.7 for

Fig.45 Fused Silica sample/stud solidly mounted in universal sample holder

6.2 **OPEN OR CREATE NEW DOCUMENT**

To start a new indentation measurement, a document should be opened or created as follows.

Select "File > Open..." from menu bar or click Open icon on toolbar to open an existing measurement document:

'File name'.mit

Select "File > New..." from menu bar or click *New document* icon on toolbar to create a new document. The following *Indentation group information* (Fig.46) window appears.

Group <u>N</u> ame		
Groupe name		
Informations Operator Operator name Client Client name	Sample characteristics <u>P</u> oisson's ratio 0.160 <u>S</u> ubstrate Fused Silica]
Reference Reference name	Layers	+ <u>A</u> dd
		<u>E</u> dit
		<u> </u>
	<u>о</u> к	X <u>C</u> ancel

Fig.46 Indentation group information window

<u>C</u> oating	
coating type	~
<u>P</u> rocess	
Process type	~
<u>T</u> hickness	5
0.00	∼ nm

Fill in the fields and especially **set** the correct *Poisson's ratio* value, e.g. 0.160 corresponds to the Fused Silica sample in use (provided) \rightarrow **Adapt** this value for each sample in use as it will affect the EIT main result.

Fused Silica sample has no coating. However if another sample with coating(s) is used, click $<\underline{A}$ dd> button (many times if several layers) to open the following window (Fig.47).

Fill in or select the comboboxes about the details of the layer definition.

Then click <<u>O</u>K> button (validation).

Fig.47 Layer definition window

Then click < OK > button in Fig.46 (validation and creation of the new document).

6.3 **PERFORMING AN INDENTATION MEASUREMENT**

6.3.1 ADO

If an ADO has already been successfully (\Re) performed on the **current** sample, **skip** this section. Otherwise an ADO should be performed (section 5.11).

6.3.2 FIND MEASUREMENT AREA

Move the sample (e.g. Fused Silica) under the video microscope to find an area¹ free of previous indents and clean, in order to perform the indent (or matrix of indents) measurement; use the following tools:

- Position control (section 5.8)
- Video (section 5.9)



- **No**t too far from the current ADO indent (otherwise sample topography may vary).
- The reference **should** be able to land on the sample, as shown here.



6.3.3 DISTANCE BETWEEN INDENTS

Indentation marks should be separated by a minimum distance to avoid influence of previous indent on the following one.



6.3.4 **DEFINE INDENTATION MEASUREMENT PARAMETERS**

The *Standard* measurement type is described in this section. However the *Sinus* measurement type is described in addition.

Select "Instrument > Start a new indentation..." from menu bar or click Indentation $\overline{\mathbf{x}}$ icon on toolbar to open the following *Define a new measurement* window (Fig.48).



Fig.48 Measurement type selection

Double click on a measurement type, e.g. Standard.

The corresponding parameters window appears as follows (e.g. Fig.49).



🔨 For a Sinus

measurement type, the dynamic calibration should have been previously performed (section 5.12). Then double click Sinus in Fig.48.



Click <Change> button to open the following Step - UNHT -Hardware parameters window which includes 2 tabs.

Fig.49 Standard window with measurement parameters

Step - UNHT	- Hardware paramete	ers	
Preferences	Sensor ranges		
Indenter a Approach 20 Pre-appro Contact st	pproach parameters speed 00.0 ♥ nm/min ach 10 ♥ % tiffness threshold 50.0 ♥ µN/µm	Approach distance 3000.0 nm Contact load 0.020 nn	Reference approach parameters Approach speed 60000.0 0.0000 mn/min Contact load 0.500 mN Pre-approach 40 %
Retract sp	eed 00.0 🗭 nm/min	Retract <u>t</u> ime	
Video Video Optica	ıl analysis after meas	urement	Table Z retraction
Default		~ C	
			<u>♦</u> <u>Q</u> K <u>Cancel</u>

Fig.50 Preferences tab parameters

In Preference tab

Settings for the *Indenter approach parameters*, *Reference approach parameters* and *Approach specification* areas shown in Fig.50 are examples (suit for the Fuse Silica provided sample) and should be set according to the sample in use/ application.

It is advised to set the *Indenter* and *Reference Pre-approach* fields with the same values as set for the current ADO (Fig.24).

Optical analysis after measurement box is checked in Video area for section 6.3.6.

For more details and information about the remaining features (*Reference-indenter auto-tuning* and *Presets* areas), refer to the **Indentation software reference** guide in section **Taking a new measurement / Setting the measurement type** parameters / Measurement hardware parameters (head) / UNHT Preference tab and Preset of the preference parameters.

	Step - UNHT	- Hardware paramet	ters	
	Preferences	Sensor ranges		
	Indentatio	n range		
	+ Referen	ce load range	+ Indenter load range	+ Depth range
Fine	• • • • • • • • • • • • • • • • • • •) mN	● 10 mN	● 10 μm
Large	>) 50	0 mN	••••• ○ 50 mN •••••	► O 50 μm
·	·		1	I
				✓ <u>O</u> K X <u>C</u> ancel

Fig.51 Sensor ranges tab parameters

Select the *Reference load range*, *Indenter load range* and *Depth ranges* <radio buttons> according to the sample in use/application (e.g. all fine ranges are selected in Fig.51).

It is recommended to use the fine *Depth range*. However if the depth saturates during the measurement, then select the large *Depth range*. Depending on the instrument version, it is possible that the large *Depth range* value is different than the one shown above.

Click < OK > button (validation and saving for current parameters of the 2 tabs).

SETTING OF THE INDENTATION MEASUREMENT PARAMETERS

in Fig.49

Indentation measurement parameters shown in this section are suitable for the provided Fused Silica sample. For any other applications, the parameters should be set according to the type of the sample in use.

Standard indentation parameters area

<u>Max load field</u>: Set a suitable value, e.g. 10 mN (with Fused Silica sample).

<i>Load Time: 30 s Unload Time: 30 s</i> info	For this kind of measurement, these values are automatic (30 sec). To change them, another type of measurement should be selected (Fig.48).
<u>P</u> ause field:	E.g. set to 10 sec ($0 = no pause$).
<i>Estimated time</i> inactive field	Information for the estimated measurement duration (depend on measurement and hardware parameter settings).
<i>Estimated memory</i> <i>size</i> inactive field	Information for the estimated RAM memory size needed to perform the measurement (depend on the measurement and hardware parameter settings).

For the following *Sinus* measurement type (some parameters are already explained above)

4 parameters are common for the 2 sinus measurements profiles (*Max. Sinus amplitude* and *Sinus frequency* fields and <Constant depth> or <Linear load> radio button).



Then click $< \underline{O}K >$ button in *Standard* window (Fig.49, or other measurement type window: *Sinus*...) to run the indentation measurement (+ validation and saving of current parameters).

For further details about the Indentation measurement types, hardware

preferences and measurement parameters, refer to the **Indentation software** reference guide in section **Taking a new measurement / Selecting the** measurement (type or protocol) and **Setting the measurement type** parameters.

6.3.5 **RUN INDENTATION MEASUREMENT**



If the sample is located under the video microscope, this *Table displacement Warning* appears.

Verify the sample height to avoid collision.

To continue click <Yes> button, the sample moves under the indenter; wait.

The following *Indentation running...* window (Fig.52) appear; wait until the following automatic indentation measurement ends.

1. The motorized head module approaches (moving down)

Status area (following squares blink)

- 2. Approaching Table Z...
 Approaching Table Z...
- 3. Approaching reference % Approaching reference 48.43 %
- 4a. Approaching Indenter Approaching Indenter
 4b. Stabilization [s] Stabilization 15 [s]
- 5. Indentation running... Indentation running... (e.g. Fig.52)
- 6. Removing the indenter... Removing the indenter...
- 7. The motorized Z table retracts (moving down)
- 8. The motorized head module retracts (moving up)

Graph curves are displayed in real time.



Fig.52 Indentation measurement is running

Warning	
A	Do you want to move under the microscope to visualize your indentation ? Check sample height to avoid collision
	Yes No

When the indentation measurement(s) is completed, it is possible¹ to move the sample under the video microscope, in order to analyze/visualize each indent (several if matrix has been performed).

First **verify** the sample height to avoid collision and then click <Yes> button; wait.

Then the following *Analyze indentation* window (Fig.53) appears.

1 If Optical analysis after measurement box has been checked in Fig.50 (Hardware parameters window/Preferences tab).

6.3.6 ANALYZE/VISUALIZE EACH SELECTED INDENT

Analyse indentation			
Indentation position	\sim Λ \sim \sim		
1.0	15.00		
0.5-	Γ.		
		Gallery of Image thumbhalls	
0.0-+	12.00		
	-		
-0.5			
-1.0	9.00-		
Standard [0 ; 0]	5.00		
List	6.00		
	-		
	300-		+
Speed Very Fast			Grab image
O rast O medium			Video image
Very slow	mN 0		MultiFocus image
	0 nm 60.00	120.00 180.00 240.00 300.00	🔲 Include 3D mapping
Keyboard control	Indentation parameters	·	Conscan image
<pre>Forward Up <left right=""> P2</left></pre>	+ Standard		AFM AFM image
← CTRL → Pg	Acquisition Rate : 10.0 [Hz] Linear Loading	E	
Backward Down	Max load : 10.00 mN Loading rate : 20.00 mN/min		井 🎦 📑
Minimum displacement 0.250 µm	Unloading rate : 20.00 mN/min Pause : 10.0 s		Optical hardness
	L		
			Finish analysis

Fig.53 Analyze indentation window

The Video window (Fig.14) is opened, otherwise click Show live image **e** icon.

If there are several indents (matrix), select in the *Indentation definition* list each indent one by one to take <u>Images</u>, p. 89 (optical, optional AFM if available).

If the indent or one of the indents (matrix) is not exactly centered on the video screen crosshair, there is no need to restart a complete Indenter-microscope distance calibration (section 5.10), a recalibration can be performed from this window:

Retarget¹ the indent center on the video crosshair and click *Redefine calibration* distance indenter microscope from this new point $\boxed{\mathbb{K}}$ icon: New calibration values for *X*, *Y* and *Z* distance are automatically saved in the corresponding field of *Calibrate Indenter Microscope distance* window (Fig.17).

¹ On the Video screen adjust the focus and move by using <u>Methods to move</u>, p. 39 (**exceptions:** All 'arrow' icons and vector move buttons). If necessary, click Return to current indentation icon to return to the original video position (as it was before moving).

<u>Images</u>

Images¹ can be taken by clicking <Video image> button (Fig.53), which opens *Edit an image* window (Fig.54).

Multifocus images can be taken by clicking <MultiFocus image> button (Fig.53).

For further details about *MultiFocus image*, refer to the **Indentation software**

reference guide in section **Taking a new measurement** / Analyzing/visualizing the indentation(s) / Optical analysis (includes setting contact point).

Edit an image	and the second s			
	Title 10 mN indent with 100x objective Comments	Relevant	Picture info Color Image Size : 736 x 589 pixels Depth amplitude : 0.00 pixel acquisition type : video	*
		Glow	Convert picture to grayscale before manipulating it.	
÷	 5 μm	Meas	asurement indent	
			OK OK	
	F	ig.54 Edit an im	nage window	
Gallery	of image thumbnails			

If an image thumbnail is clicked, the corresponding image is displayed in large format. Additional options are available (as image title, comments...).

When <OK> button is clicked, the image thumbnail is created inside the gallery of the *Analyze indentation* window (Fig.53).

¹ On the Video screen adjust the focus and move by using <u>Methods to move</u>, p. 39 (*exceptions:* All 'arrow' icons and vector move buttons).

In *Analyze indentation* window (Fig.53), when the optical analysis is completed (for each indent if several and if needed), click <Finish analysis> button.



This *Confirmation* window appears.

If <Yes> button is clicked, it will not be possible to come back to this *Analyze indentation* window \rightarrow The motorized Z table is retracted (moving down); wait.

6.3.7 SAMPLE DISPLACEMENT AFTER MEASUREMENT(S)

After the measurement(s) is performed or after visualizing the indentation(s) (previously described), the sample position should be shifted¹ to avoid performing the next indentation measurement inside the current measurement indent. The following window appears and is comparable to the one after the ADO automatic procedure; refer to the similar explanations from Fig.26.

Sample displacement after Measurement				
It is recommended to move the sample away from the measurement zone: set a minimum displacement				
X motorized table				
0.500 💓 mm			0.000	mm
	~	ОК	X	Cancel

¹ **IMPORTANT:** Pay attention to the displacement unit (e.g. mm here), which is set in the options; refer to the **Common Scratch & Indentation software reference guide** in section **Customizing options / Preferences tab (units)**.

7 RESULTS ANALYSIS

Results and graphic curves of the indentation measurement (sheet 1 bottom tab) are created in the main *Indentation Software* window (Fig.55); *Show the curve* view icon is selected (default) on toolbar.



7.1 VIEWING MODES

The previous graph area (Fig.55) shows the combined curves, force vs. indentation depth as *Indentation curve* \checkmark icon is selected.

Other viewing mode(s) is available (Fig.56):

Click *Fn* icon to show/hide **F** red curve vs. time.

AND/OR

Click *Pd* icon to show/hide **h** green curve vs. time.



7.2 CONTACT POINT(S)

Each indent contact point¹ (several if matrix) has been automatically defined by the software. However, it is advised to verify and if necessary to refine (more precisely) each contact point as follows.

Select "Edit > Indentations > Set contact point..." from menu bar or click Detect contact point \checkmark on additional toolbar (Fig.55) and follow the same explanations from section 5.13.5.1, exceptions: Here¹ there is only one indent (one load).



Here the results are calculated with the contact point automatically defined by the software \rightarrow *Detect contact point* \gtrsim icon is inactive on *Set the contact point* window (Fig.38) toolbar.

Here the results are recalculated with the contact point more precisely refined by the user \rightarrow *Detect contact point* \swarrow icon is active on *Set the contact point* window (Fig.39) toolbar.

7.3 **REVIEWING IMAGES**



Double click one image thumbnail in the gallery of the result analysis main window (Fig.55) to open *Edit an image* window (Fig.54).

¹ For our measurement example (Fig.49), only one indent (#1) is available.

7.4 SINUS ANALYSIS

For Sinus measurement results



Fig.57 Main window with sinus analysis of results

On the left top side of the main window (Fig.57), *Add analysis* of icon to add a new Sinus method analysis.

Create a new analysis	
Analysis method	
Martens hardness Adhesion analysis Sinus Mean Analysis Tangent	
Creep Analysis Hertz Oliver & Pharr	
Sinus Stress ain Analysis	
<u>√</u> <u>O</u> K <u>C</u> ancel	

This window appears.

Double click Sinus.

In the main window (Fig.57) a new tab (e.g. tab #2) appears with:

- Sinus analysis method results, parameters... on the left side
- an additional upper graph area with the sinus curve results

7.5 SAVE, PRINT, STATISTICS/EXPORT

7.5.1 SAVE

Select **"File > Save"** from menu bar or click *Save* \bowtie icon on toolbar to open the following *Save an indentation file* window.

Zave an indentation file		
🕞 🔍 🔻 🕨 🕨 Example	✓ 4y Search Example	1
Organize 🔻 New folder	ee 🗸 📀	Choose a location to
🔶 Favorites	No items match your search.	save the measurement file on
📜 Libraries		the acquisition system.
P Computer		– – – –
🙀 Network		l ype a file name (automatic extension .mit)
File name: Example mit	•	Then click <save></save>
Save as type: Indentation fil	e (*.mit) 🔹	button.
Hide Folders	Save Cancel	J

7.5.2 **PRINT**

For further details, refer to the **Common Scratch & Indentation software reference guide** in sections **Customizing options** and **Printing/PDF document reports**.

7.5.2.1 Report properties

Select "**File > Options...**" from menu bar to open the following *Options* window (Fig.58).

Options					
Preferences	Company header	Document models	Export formats		
Models Default r	model				
Comple	ete document		•		
Comple Synthet Short de	ic document			+	Add
		List			Edit
			[-	Remove
		•			
		\checkmark	<u>о</u> к	×	<u>C</u> ancel

Fig.58 Document models tab

To edit/create document models:

Select *Document models* tab and click <Add> button to create a new model or select an existing document model in the list and click <Edit> button to change it.

Then the *Edit a document model* window appears and includes several tabs in which the report properties can be configured (for printing then).

7.5.2.2 Print preview

Select **"File > Print preview..."** from menu bar or click *Print preview* icon on toolbar to open the following '*Print preview*' window.

Group Informat	nam tions	e								_	
Curves											
15.0	*		*	4		4		4		-	
13.5 -				*	*		*		*	-	Print preview include
120-	+	4	*	4	*	4			*	-	the configuration set
10.5-			*		*		*				section 7.5.2.1 but it
9.0-	,	•	•	*	•		*	.//	1		can also be changed
-0.5	÷	4	-		*			/			CIICKING < Keport
45-										-	see Fig 60
3.0 -						/	<i>.</i>				Sec 119.00.
15-	*					/ .		4		-	Click <i>Print</i> 🖶 icon a
0.0 mN											see Fig.59.
0.0 nm Indentation	n 30.0	60.0	90.0	120.0	150.0	180.0	210.0	240.0	270.0	300.0	
<u>Analysis #</u> Method Main resu	Oliver 8	Pharr 077 MPa	3								Click <i>Save as PDF</i> 🔡 icon and see <i>File info</i> area in Fig.61.
	$E^{+} = 75.2$	281 GPa 106 GPa	l								
Hypothes	is Poisson	s ratio(n	u)= 0.16								

Select **"File > Print..."** from menu bar or click *Print* **=** icon on toolbar to open the following window (Fig.59).

Print	
Printer	
Name	
"Default printer"	Same Print or Preview windows
Select paper size :	Print report properties can also be
🔘 Letter	changed here as follows.
-Document model	Select a <i>Document model</i> from "Name" drop-down menu.
<u>N</u> ame <u>E</u> dit	
Complete document	Name
Print extent	Synthetic document 🗟
Groups	
<all></all>	Then click < <u>E</u> dit> button to open
Indentation tests	the Edit a document model window
<all></all>	which includes several tabs in
	which the report properties can be
✓ <u>O</u> K X Cancel	configured (for printing then).
Fig.59 Print Window	Other printing options are
	available as follows.
Preview	Coloct the desired group(c) of the
-Printer	indeptation massurements that
Name	chould be printed from
"Default printer"	
Select paper size : A4 	<pre></pre>
l etter	
Document model	<pre><all relevant=""> group(s), of one existing " 'Crown name' "</all></pre>
Edit	one existing Group name .
Name	Calast the desired kind of
Complete document 👻	Select the desired kind of
Print extent	should be printed from
Groups	" <indentation tests=""> " drag</indentation>
<ali></ali>	down monu:
Indentation tests	" <all>" measurement(s) or</all>
YOUR Y	
	measurement(s).
<u> </u>	
Fig.60 Preview window	2

Select **"File > Save as PDF..."** from menu bar to open the following window (Fig.61).

Save as PDF	
File info	
Document title	
Example	
Author	
	File info:
Subject	Some fields can be
	modified/filled in.
Keywords	· · · · · · · · · · · · · · · · · · ·
-	
☑ Open in pdf viewer	
- Document model	
Name Edit	Otherwise it is the same explanations that
Complete document	previously described in
	Same Print or Preview
Print extent	windows, p. 98.
<u>G</u> roups <u>I</u> ndentation tests	
<all></all>	Click <ok> button to valid</ok>
✓ <u>Q</u> K X Cancel	

Fig.61 Save as PDF window

A 2nd Save as PDF window appears.

Choose a location to save the PDF file on the acquisition system. Type a file name (automatic extension .pdf) Then click <Save> button.

7.5.3 STATISTICS/EXPORT

Click *Show the statistics* icon on toolbar to show the statistics view; see the following main window.



In the current report (e.g. *New report* in *Manage report* area) or in each new one (clicking *Create a new report* o icon)

In the tree, select by checking each group/measurement# boxes for which data/curves statistics should be performed; e.g. only 1 group and 1 measurement#1 is available here $4 - \frac{36}{2} - \frac{36}{2}$

Click *Select statistics* icon to show/hide the statistics area and/or click *Curves* ~ icon to show/hide the curves area. Select the desired data and options (in statistics area: Results tree check boxes, additional toolbars check boxes and options - in curve area: Drop-down menus, check boxes, additional toolbox icon options...).

Click '*Sheet*' icon to export in different ways the data/curve results in text files.

For further details about statistics (cycles...) and export, refer to the **Common Scratch & Indentation software reference guide** in section **Statistics**.

8 PERFORMING A NEW MEASUREMENT

8.1 WITH THE SAME SAMPLE

To perform new indentation measurement(s) with the **same sample** and the **same indenter**, resume the procedure from section 6.3.

Otherwise if the indenter should be changed, resume the procedure from section 5.6.

8.2 WITH A NEW SAMPLE

To carry out new indentation measurement(s) with a **new sample** but with the **same indenter**, resume the procedure from section 6.

Otherwise if the indenter should be changed, resume the procedure from section 5.6.

G70IB003EN-A

MOUNTING HEAD BOTTOM PROTECTION COVER 9

When the UNHT³ is not in use, it is advised to mount its head bottom cover in order to protect the indenter and reference.

Avoid any collision with the indenter/reference during the following operations.

The cover is equipped with 2 captive hand screws in diagonal and 2 positioning pins in opposite diagonal.

Position the cover in order to see the 'U' shape in front of the head.

- Tilt the protection cover forward to better see and carefully place the 'U' shape over and around the indenter and reference, **A**avoid any collision between the 'U' shape sides and the indenter/reference
- Then lightly press the protection cover against the head bottom.
- Search the 2 pin holes by doing **only** longitudinal movements <u>M</u> no

lateral movements. When cover pins are inserted, keep the cover pressed against the head bottom and (with other hand) tighten the 2 hand screws to lock the cover against the head bottom.





Fig.62 Head bottom





UNH¹

10 F.A.Q.

10.1 WHY IS THE INDENT NOT UNDER THE VIDEO CROSSHAIR?

Why is there an offset between the indent center and the video crosshair center after an indentation measurement?

 \rightarrow The distance between the indenter and the video microscope is wrong:

Perform a new Indenter-microscope distance calibration (section 5.10).

→ The sample moves during the indentation measurement, due to the indentation force:

Tighten stronger the sample in the clamp jaws.

 \rightarrow There was play in motorized X, Y tables:

Select **"Instrument > Hardware configuration..."** from menu bar and in *Hardware configuration* window, click <Edit> button>. In the following *Step - MHT - Hardware configuration* window, select *Motors* tab and verify/set *Backlash Correction* field to 200 µm.

Ste	ep - UNHT - Hard	lware configurati	ion	
М	ly configuration	User channels	Control unit & modules	Motors
	Table model Step		→ Backlash Cor	rection

Then **click** <Backlash> button (backlash correction) in *Position control* window (Fig.13) after the last manual move of the targeting process. This will remove the play of motorized X, Y tables.

10.2 WHY CAN I NOT REACH THE CONTACT LOAD?

→ The 2 pieces of masking tape on each side of the motorized head module are not removed:



Remove the 2 pieces of masking tape.

Masking tape should only be present if the head/instrument is being sent back (for transport).

10.3 Why is the indent not as expected?

Why is the indent non-conform, not repeatable?

 \rightarrow The distance between the indenter and the video microscope is wrong:

Perform a new Indenter-microscope distance calibration (section 5.10).

→ The sample moves during the indentation measurement, due to the indentation force:

Tighten stronger the sample in the clamp jaws.

- \rightarrow The indenter extremity is:
 - ► Dirty,

Clean indenter extremity and/or

 $\ensuremath{\text{Perform}}$ an indentation at 50 mN in copper to remove dirt ; see section 0

▶ Worn out for the application or damaged,

Verify the wear by performing an indenter calibration (section 5.13) and then if it is necessary,

Use a new indenter and review from section 5.6.

10.4 WHY IS THE ADO NOT SUCCESSFUL?

Why has the ADO **not** been successfully performed? The automatic procedure has been interrupted and sometimes the following error status appears.



→ The ADO pre-approach parameters values for the indenter and the reference are not suitable and should be adapted:





Bad pre-approach

While the motorized Z table approaches (moving up), the sample surface touches both reference and indenter or only the indenter.

Depending on *Indenter* and *Reference Pre-approach* fields (in *Adjust depth offset parameters* window), either the indenter value should be decreased by 10 % or more, or the reference value should be increased by 10 % or more.

Adjust depth o	ffset parameters Surface detection paramet	ers			
.	Approach <u>s</u> peed	nin	Contact force	μN	
	Characterization force		Contact stiffn 150.0	ess threshold Ω 💽 μN/μm	
	Indenter Pre-approach %	Reference Pre-appro	ach	Contact force	μN
	Presets Default			~	G
		[V Ok	X	Cancel

An ADO **should** be started again and if it is necessary, readjust those pre-approach values until it is successfully (\Re) performed.

Case b)

 R
Sample
Remaining piezo displacement range

Bad pre-approach

The reference touches the sample surface but the indenter goes (down) to ~ 100 % (max) without having touched the sample surface.

In this case (in *Adjust depth offset parameters* window) the *Reference Pre-approach* field value should be decreased by 10 % or more.

Adjust depth o	ffset parameters Surface detection paramet Approach <u>s</u> peed 25000.0 💭 nm/r	ers nin	Contact forc 30.0	e ₩	
	Characterization force 500.00 ♥ µN		Contact stiff 150	ness threshold .0 💽 µN/µm	
	Indenter Pre-approach	Reference Pre-appro	oach	Contact force	μN
	Presets Default			~	G
			✓ 0	к	Cancel

An ADO **should** be started again and if it is necessary, readjust this pre-approach value until it is successfully (\Re) performed.

If the value is set to the minimum (advised not less than 5 %) and there is still the error, it means the sample is tilted too much and should be leveled.

→ The sample is tilted to much:

Level the sample and then an ADO **should** be started again until it is successfully (\Re) performed.

→ <Cancel> button has been clicked during the automatic ADO procedure (this stops the procedure and cancel the ADO):

An ADO **should** be started again until it is successfully (\Re) performed.

See the ADO (section 5.11).

10.5 Why is the depth signal saturated?

See the following graphs.



→ The sample is very soft which gives a large penetration depth during the measurement (Fig.63)

OR

→ There is a certain relief on the sample (can be a too high roughness). The measurement is performed in a hole: the approach distance is increased which may saturate the depth (Fig.64)



Before starting a new measurement

Select the <u>large</u> *Depth* range radio button, e.g. $<50 \mu m >$ (instead of fine $<10 \mu m >$) in *Sensor* range tab of *Hardware* parameters window (Fig.51).
Even with a Vickers or Berkovich indenter? See the following graphs.



The indenter is already in the sample surface before the approach

→ The ADO is performed in a hole. Then the approach distance is not sufficient and this way the indenter starts the measurement already in the sample



OR

→ During the ADO the indenter penetrates too much in the sample because of sample softness. Then the approach distance is not sufficient and this way the indenter starts the measurement already in the sample



OR

→ There is a certain relief on the sample (can be a too high roughness). The measurement is performed on a bump: the approach distance is not sufficient and this way the indenter starts the measurement already in the sample



Before starting a new measurement, in order to increase the approach distance

Set a greater value in *Approach distance* field, e.g. > 3'000 nm in *Preferences* tab of *Hardware parameter* window (Fig.50).

If the value is too much increased, then it is possible that the depth signal saturates (section 10.5).



10.7 Why does the Insert an adjust depth offset window appear?

Why this window appears before running an indentation measurement?

Edit adjust depth offset parame	eters and position
Delta X -100.000 Delta Y -100.000 Indentation count : 1 dimensions in X 0.000 µm to 0.000 µm	Fn contact : 30 μN Characterization force : 500 μN Approach speed : 25000 nm/min FnRef contact load : 500.000 μN Stiffness threshold : 150 μN/μm
dimensions in Y 0.000 µm to 0.000 µm	Edit <u>a</u> djust depth offset parameters

→ No ADO has been performed or the last ADO was **not** successfully performed (**no** green circle with a tick on ADO ^{*}/_{*} icon on toolbar):

Edit these ADO parameters and position,

- click <Edit adjust depth offset parameters> button, this opens the Adjust depth offset parameters window (Fig.24).
- set suitable values¹ in *Delta <u>X</u>* and <u>Y</u> position fields, this will shift the current position for this ADO (and then when ADO procedure is finished, it will come back where it was before running the indentation measurement) and click <OK> button to start this ADO before running the indentation measurement.

OR

Click <Cancel> button to abort this ADO which aborts also the indentation measurement. Thus an ADO with the standard method, described in section 5.11, should be successfully (\Re) performed before running an indentation measurement.

Not too much, otherwise the sample topography may vary.

IMPORTANT: Pay attention to the displacement unit (e.g. mm here), which is set in the options; refer to the **Common Scratch & Indentation software reference guide** in section **Customizing options / Preferences tab (units)**.

10.8 Why does "hc out of calibrated range" message appear?

Why in the main result analysis window under *Hypothesis* does the message "*hc out of calibrated range* [# nm, # nm]" appears?



→ The h_c of the measurement *Additional results* is out of the known calibration range:

Refer to the *Indentation software reference guide* in section *Analysis of results / Hypothesis / Out of indenter tip calibration*.

11 BLOCKING ERROR MESSAGES

This section shows the main blocking error messages and describes propositions to try removing the cause of each error. However if a problem persists, try switching off the system: First close the software, then shut down the acquisition system by software (Windows[®]) and then switch off the instrument using the main power switch. Wait about 20 sec, then switch on the instrument using the main power switch and then open the software and try again to see if this has solves the problem.

11.1 CONNECTION TO CONTROL UNIT FAILED.

The control unit cannot connect.

Connecting to MHTX S/N: 01-00022 Connexion IOCard Control Unit Motors Present Processing Failure x Information NCO card Success O Voltage on Connexion to Control Unit failed. (Detect error : No Card selected) Stop Stop OK

(Detect error : No Card selected)

Verify that the IO card is selected (**"Instrument > Hardware configuration..."** menu bar / <Edit> button / *Control unit & modules* tab / <<u>C</u>hoose card> button).

(Error detected : Voltage not present)

Connecting to MHTX S/I	N: 01-00022			
Connexion Processing Failure Success	IOCard Present Voltage on	Control Unit Motors	Information Connexion to Control Unit failed. (Error detected : Voltage not present)	×
	Stop			ОК

Verify that the Step is switched on (power cables plugged).

(Error detected : No Communication)

Connecting to MHTX S/	N: 01-00022		
Connexion	IOCard	Control Unit	
Processing	🖨 Present	Motors	
Failure			Information
Success	Voltage on	NCO card	Connexion to Control Unit failed. (Error detected : No Communication)
	Stop		ОК

Verify that the communication Ethernet cable is connected (on acquisition system and on Step rear panels).

(Detected error : missing connection. check cable and...

Connecting to MHTX S/	/N: 01-00022		
Connexion	IOCard Present	Control Unit	
Failure	Voltage on	NCO card	Information
	Stop		check cable and reinit motor control unit.)

Verify that the motor cables are connected to the Step.

11.2 YOUR INSTRUMENT RANGES HAVE NOT BE VALIDATED!

An ADO, calibration or measurement is not carried on.



Refer to the **Common Scratch & Indentation software reference guide** in section **Managing the instrument / Hardware configuration / 'Ranges' tab(s)** before starting a new measurement.

11.3 MOTORIZED TABLES

Motorized tables are no longer controlled: Fatal error.

Cannot get current position on motor 0



Cannot Initialize motor Nb 1.



Error Reading Boolean in API msg 1123 / 1327!



Close the software, then shut down the acquisition system by software (Windows[®]) and then switch off the instrument using the main power switch. Wait at least 20 sec, then switch on the instrument using the main power switch and then open the software.

Verify the voltage network quality (it should be stable).

11.4 VIDEO SOFTWARE

The Video Software does not work.

Cannot run the Video Module. The module is not properly...

Cannot run the Video Software.



This software is too old or not properly installed; try to reinstall a recent Video Software.

The program can't start because uEye_api.dll is missing from...

Cannot properly use the software.

Generi	cVide	eoModule.exe - System Error	X
	8	The program can't start because uEye_api.dll is missing from your computer. Try reinstalling the program to fix this problem.	
		OK	
	Vide A V O in	eo Error In error occurs during camera initialization. Maybe it is used by another program. Itherwise, check its connection and configuration or drivers Isstallation.	
		ок	

The camera driver is not properly installed; try to reinstall the camera driver (in the *setup Video.exe*).

11.5 CAN NOT WRITE ADDRESS

Communication trouble with video microscope light source.

Scratch	
8	Can not write address (Read E2Pot)
	ОК

Verify that the video microscope cables are correctly plugged.

11.6 PREVIOUS DAQMX DRIVER VERSION DETECTED

When starting the software, if the following message appears,

DaqMx Drivers 9.2 (or 8.6) detected! You have to install DaqMX 16.1 or higher



it means that a previous driver version (under DaqMx 16.1) is installed in the acquisition system.

Contact us: www.anton-paar.com to ask for the latest driver version, in order to proceed with an update. Otherwise bad acquisitions can be performed and therefore wrong results can be obtained.

11.7 DURING AN ADO, A CALIBRATION OR AN INDENTATION MEASUREMENT

The ADO, the calibration or the indentation measurement is not carried on.

An error occurred : EIndentationInstrumentError - Could not...

The instrument range cannot be reached.



Start a new ADO until is successfully (\Re) performed before starting a new calibration or a new indentation measurement.

An error occurred: EIndentationInstrumentError - Sample in contact...

The sample surface has touched the indenter before the reference.

Indentation software	Indentation software
Sample in contact with indenter before reference	Stop requested in automaticapproach : Sample in contact with indenter before reference
ОК —	OK
error	
An error occured : EJ	IndentationInstrumentError - Sample in contact with indenter before reference
	ОК

R	
Sample	

Bad pre-approach

While the Z table approaches (moving up), the sample surface touches both the reference and the indenter or only the indenter.

Depending on pre-approach parameters of the indenter and reference (in *Preferences* window and tab), either the indenter value should be decreased by 10 % or more, or the reference value should be increased by 10 % or more.

	Surface detection paramet	ters			
<u>_</u>	Approach <u>speed</u>		Contact force	2	
, v v	25000.0 💭 nm/r	min	30.0	0 🖨 μN	
	Characterization force		Contact stiffr	ness threshold	
	500.00 💌 μN		150.	0 🌩 μN/μm	
	Indenter	Reference			
	Pre-approach	Pre-approa	ch	Contact force	
	%		* %	500.00	μN
	Presets				
	Default			\sim	G
					G

Start again the measurement and if necessary readjust those pre-approach values until the indentation measurement correctly runs until the end.

Otherwise start a new ADO¹ until it is successfully (\Re) performed before starting a new indentation measurement¹

¹ Previously, verify and if necessary, change the ADO parameters and then the hardware parameter preferences for the indentation measurement.

An error occurred : EIndentationInstrumentError - Altitude (z) of...

The altitude (z) of the indenter is undefined.



Start a new ADO¹ until it is successfully (\Re) performed before starting a new calibration or a new indentation measurement¹

An error occurred: EIndentationInstrumentError - Force signal...

The force signal is in saturation, the indenter-reference vertical distance is too large.



Start a new ADO¹ until it is successfully (\Re) performed before starting a new calibration or a new indentation measurement¹

¹ Previously, verify and if necessary, change the ADO parameters and then the preferences parameters of the indentation measurement.

An error occurred: EIndentationInstrumentError - The surface...

The sample surface has touched the indenter during the pre-approach.



Start a new ADO¹ until it is successfully (\Re) performed before starting a new calibration or a new indentation measurement¹

An error occurred: EIndentationInstrumentError - Can not Remove from...

The reference contact cannot be removed with the motorized Z table.

	Indentation software				
	Stop requested in automaticapproach : Can not Remove Contact from Reference with Table Z.(500mu limit)				
	ОК				
error		X			
8	An error occured : EIndentationInstrumentError - Can not Remove Contact from Reference with Table Z.(500mu	limit)			
	ОК				

Start a new ADO¹ until it is successfully (\Re) performed before starting a new calibration or a new indentation measurement¹

¹ Previously, verify and if necessary, change the ADO parameters and then the preferences parameters of the indentation measurement.

If problems should be encountered, refer to this document and to the others mentioned.

Otherwise contact us,



www.anton-paar.com