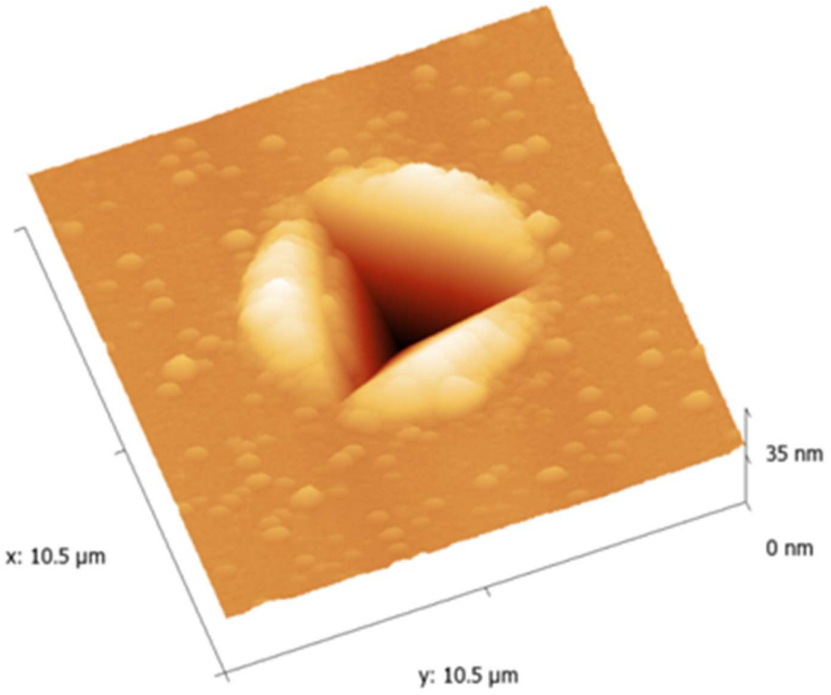
Queen Mary University of London

alemnis
Mechanical testing at the micro- and nano-scale



Instrumented Indentation Short Course

Guide to Practical Sessions

INSTRUMENTED INDENTATION COURSE SCHEDULE

	WEDNESDAY (12 Feb)	THURSDAY (13 Feb)	FRIDAY (14 Feb)
08.30-09.00		Data Interpretation 2 (NR)	Data Interpretation 4 (NR)
09.00-09.30		5. Unusual Behaviours (Cracking, Phase Transformations, etc.) (AB)	Examination Debrief (AB&NR)
09.30-10.00		*Coffee Break*	*Coffee Break*
10.00-10.30		6. Potential Pitfalls (AB)	10. Testing of coatings (AB)
10.30-11.00	Registration in DLL (MED2 2423)		
11.00-11.30	1. Introduction to Indentation (AB)	7. Strategies for different materials (AB)	11. Overview of Industrial Standards (NR)
11.30-12.00		Practical 3: Metal Experiments (NR)	Practical 5: Coating Experiments (NR)
		Group 1: Copper / Group 2: Copper	Group 1: DLC-on-steel / Group 2: Al-on-Si
12.00-13.30	*Lunch*	*Lunch*	*Lunch*
13.30-14.00	2. Basic Indentation Theory (AB)	Data Interpretation 3 (NR)	Data Interpretation 5 (NR)
14.00-14.30		8. Measurement of Soft Materials (AB)	Round table Discussion on predefined Attendee Topics
14.30-15.00	3. Instrumentation: how does it work? (NR)		
15.00-15.30	4. Selection of Test Parameters (AB)	9. Research Methods & Applications (NR)	Wrap-up, End of Course
15.30-16.00	Practical 1: Basic Experiments Fused Si (NR)		
16.00-16.30	*Tea Break*	*Tea Break*	
16.30-17.00	Data Interpretation 1 (NR)	Examination (multiple choice)	
17.00-17.30	Practical 2: Creep & Strain Rate Sensitivity (NR)	Practical 4: Advanced Mapping (NR)	
	Group 1: PMMA / Group 2: PMMA	Group 1: Co-W Alloy / Group 2: Diffusion bond	
17.30-18.00		19.00 Dinner TBC (self-pay)	

Location: Discovery Learning Labs (DLL) in MED Building, École Polytechnique Fédérale de Lausanne, CH-1015 Lausanne, Switzerland

Lectures are held in MED 2 2423 and the practical sessions in MED3 1424 (map available here **http://plan.epfl.ch/?room=MED%203%201424**)

INTRODUCTION

The experimental work is conducted on two nanoindentation instruments equipped with Berkovich diamond indenter:

1) Anton Paar Nanoindentation Tester (TTX-NHT2)
2) Alemnis Standard Assembly (ASA)

There are 2 groups with 3-6 students in each group.
The samples to be tested are shown in Fig. 1 and 2:

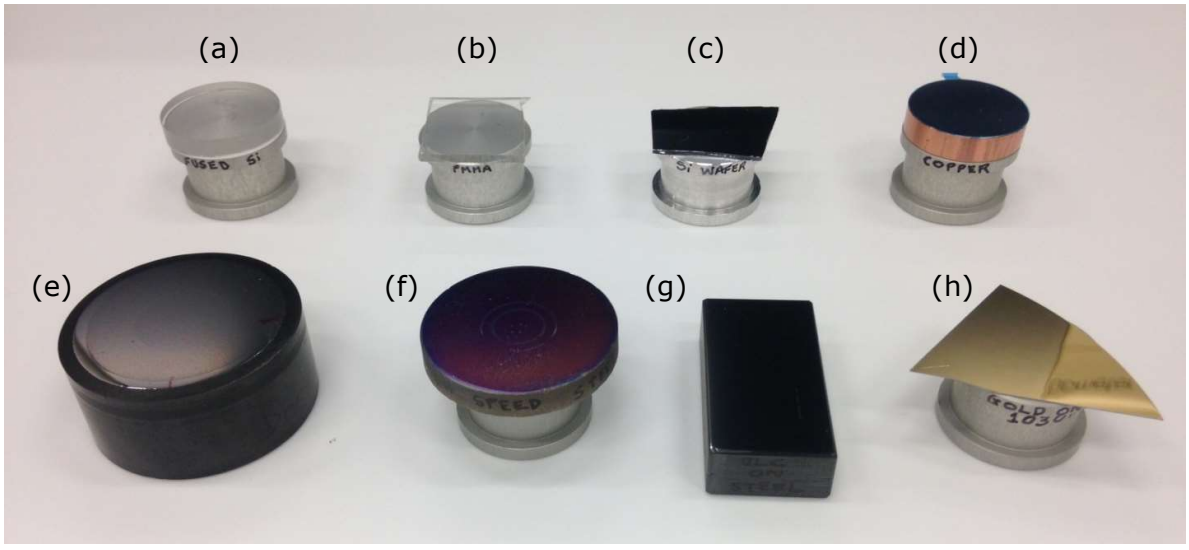


Fig. 1: Samples to be tested on the Anton Paar TTX-NHT; (a) Fused Si, (b) PMMA, (c) Silicon Wafer, (d) Copper, (e) Co-W alloy, (f) High Speed Steel, (g) DLC-on-Steel and (h) Gold-on-Si.

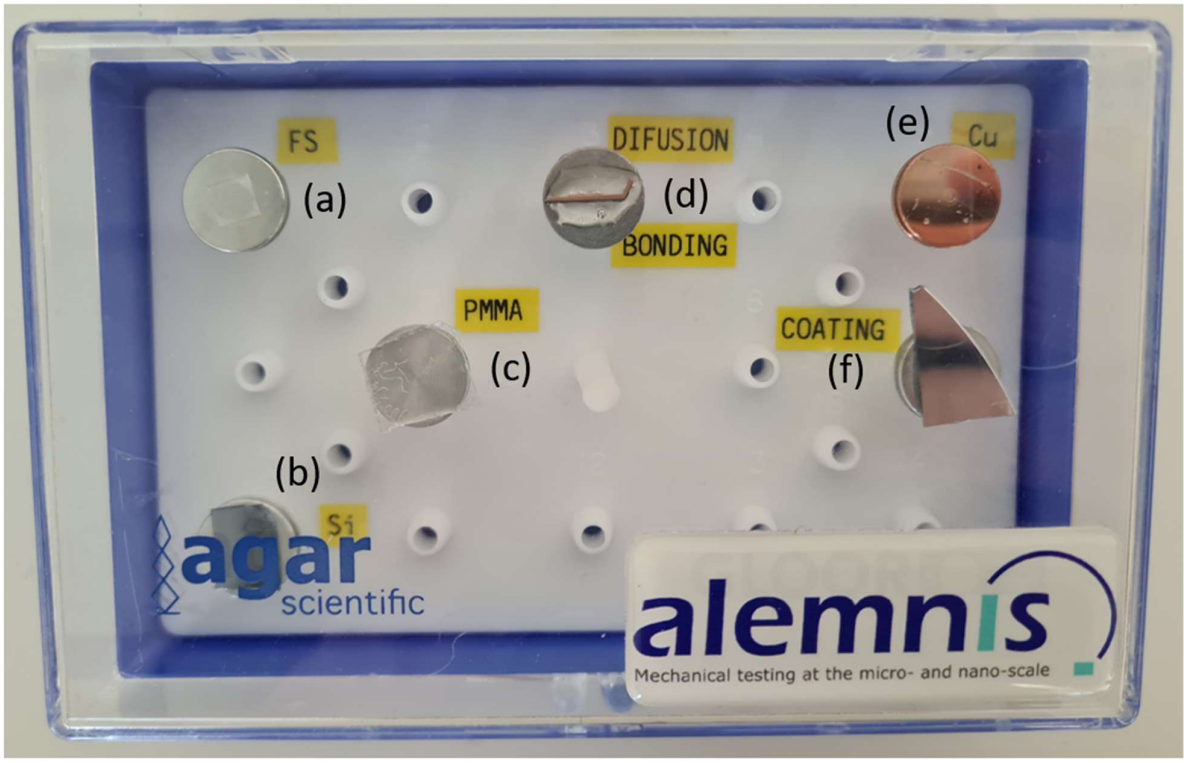


Fig. 2: Samples to be tested on the Alemnis ASA; (a) Fused Si, (b) Silicon Wafer, (c) PMMA, (d) Diffusion Bonded Joint, (e) Copper, (f) Al-on-Si coating.

PRACTICAL 1 : Fused Si (Quartz)

Fused silica is a non-crystalline glass form of Silicon Dioxide (quartz, sand) and its highly crosslinked 3D structure gives it a low thermal expansion coefficient. It is a pure material which exhibits homogeneous mechanical properties and is therefore commonly used as a calibration material for assessing elastic modulus, E. It exhibits a relatively elastic response.

GROUP 1: Experimental Procedure:

1) Clean the "Fused Si" sample by blowing off with compressed air. Additional cleaning can be done with isopropanol if necessary.
2) Place "Fused Si" sample under the NHT measuring head as shown in Fig. 3

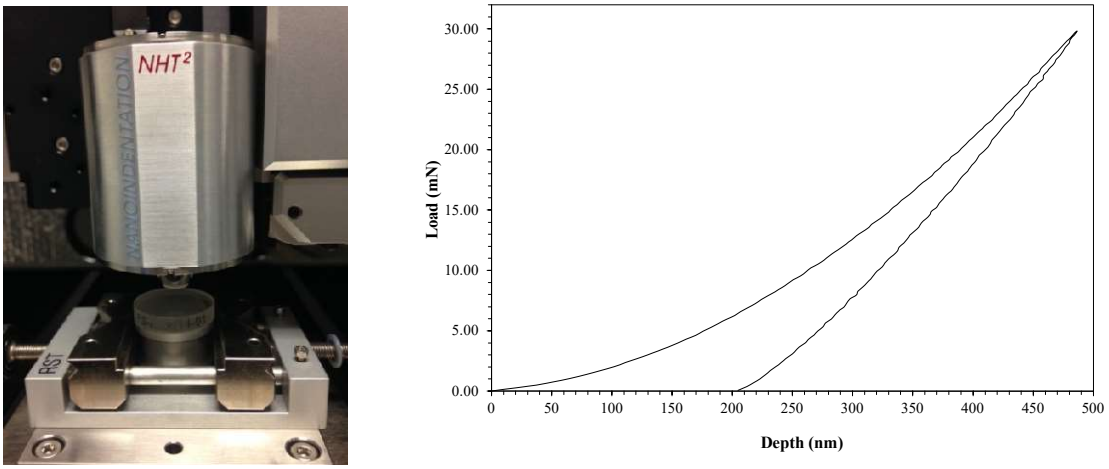
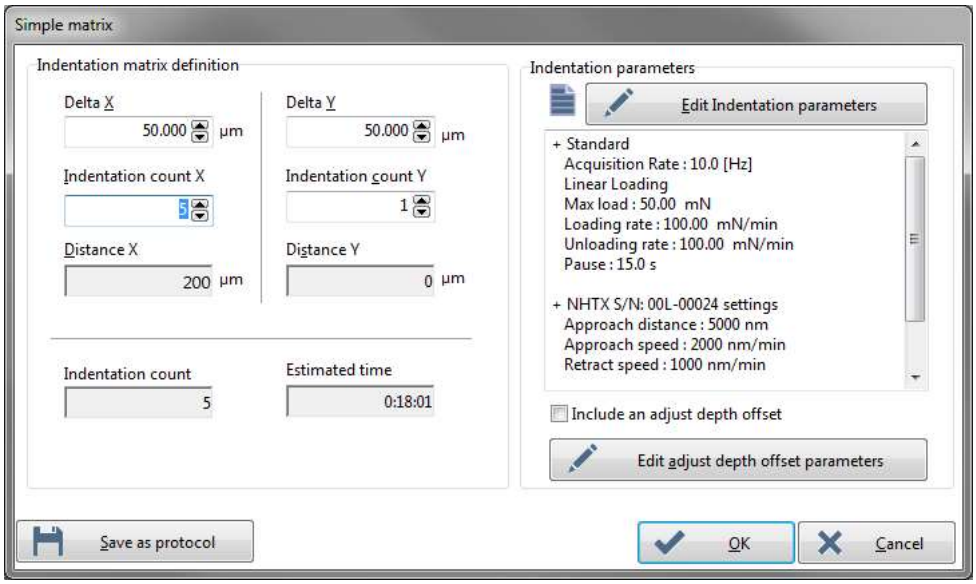


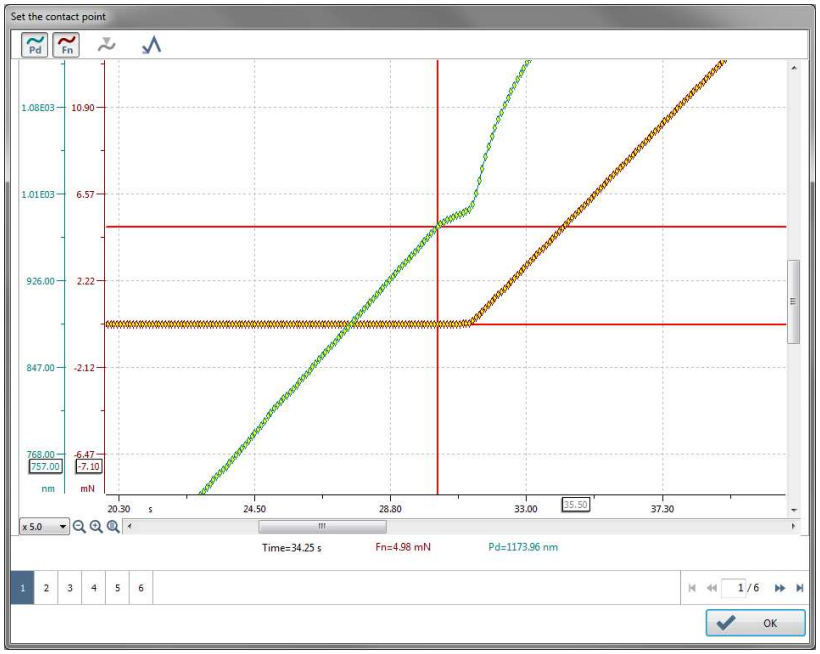
Fig. 3: Fused Si sample mounted under the NHT head and typical load-depth curve at load of 30 mN

3) Create new file group, enter group name and enter Poisson's Ratio value of 0.16
4) Perform an Adjust Depth Offset (ADO). Click OK to move to a fresh area.
5) Program the software to make a "Simple Matrix" with 5 "Standard" nanoindentations with an applied load of 50 mN, loading rate of 100 mN/min. and a pause at maximum load of 15 seconds. Set the Approach and Retract speeds to 3000 nm/min. Separate the indentations by at least 50 µm (use Delta X and Y value of 50 µm):

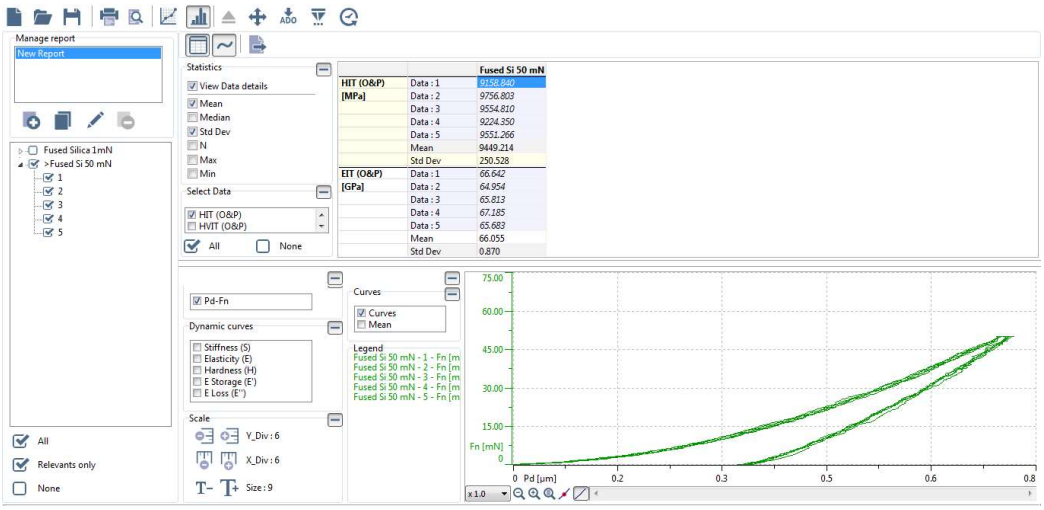


GROUP 1: DATA INTERPRETATION

1) After completion of tests, do not move under the microscope. Check that the contact point of each indentation has been correctly chosen (right click on each indentation tab, select "Set contact point" and double-click on new position if necessary):



2) Right-click on the Group Tab, select "Change Poisson's ratio" and enter a value of 0.16 (literature value for Fused Si)
3) Click on the "Statistics" page and select the 5 measurements (Tick "All" in bottom left of screen)
4) Choose "Combined Curves" in the curve view in order to superimpose the 5 measurements on the same load-depth axes.
5) Create a results table by checking the "View data details", "Mean" and "Std Dev" checkboxes. Select HIT (hardness) and EIT (elastic modulus) for the data outputs:



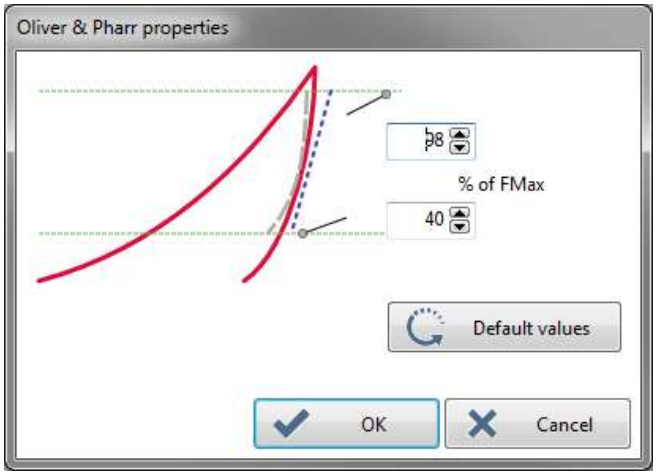
6) Note the HIT and EIT mean values and their standard deviation. If the standard deviation exceeds 5%, inspect the superimposed curves and look for any measurements which seem outside of the group. Remove such measurements from the group calculation by unchecking on the left of the screen. How does this affect the final value..?

QUESTIONS:

(i) How does changing the contact point of the measurement affect the H and E values? Why?

(ii) Do you see any creep during the 15s hold at maximum load? If so, why?

(iii) Click on the "Analysis Properties" icon in the curves view of the software. This allows the user to change the portion of the unloading curve used in the calculation of H and E:



Change these parameters and comment on their impact on the H and E values

(iv) What would be the minimum number of indentations required to make these results "meaningful"…?

GROUP 2: Experimental Procedure:

1) Place “Fused Si” sample under the ASA measuring head as shown in Fig. 4

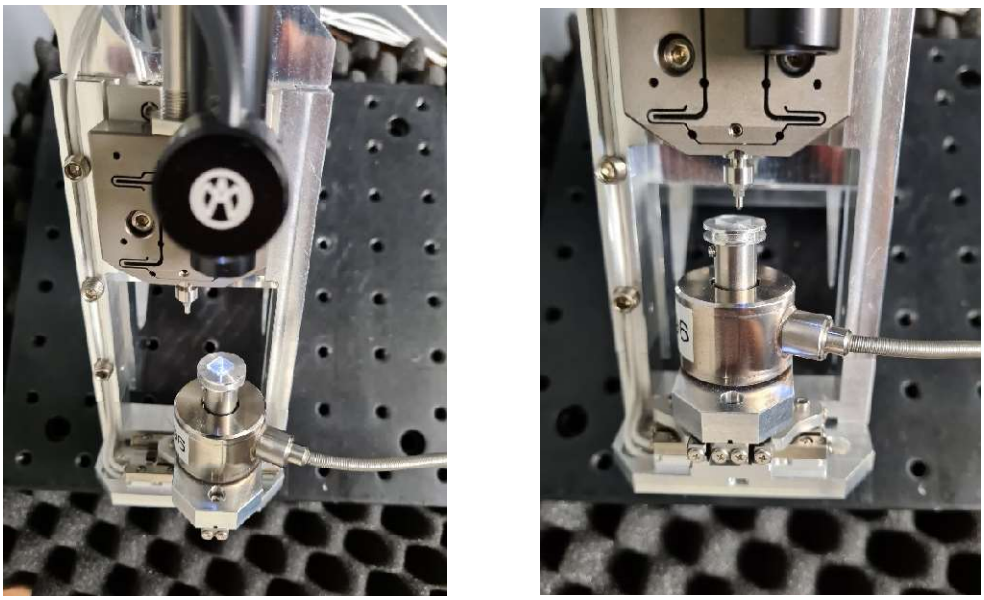
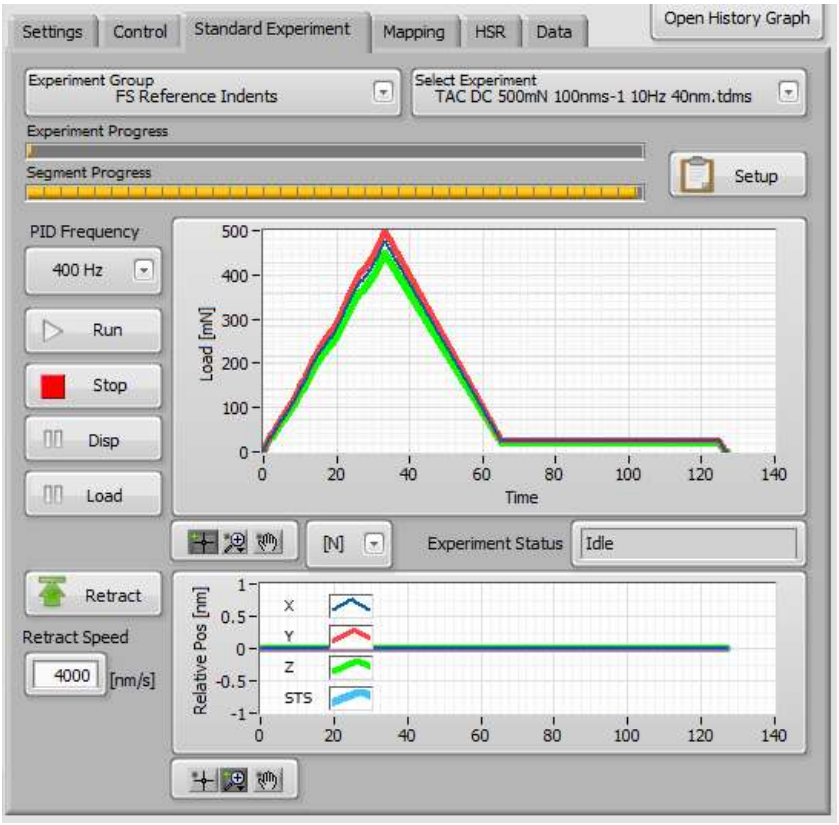


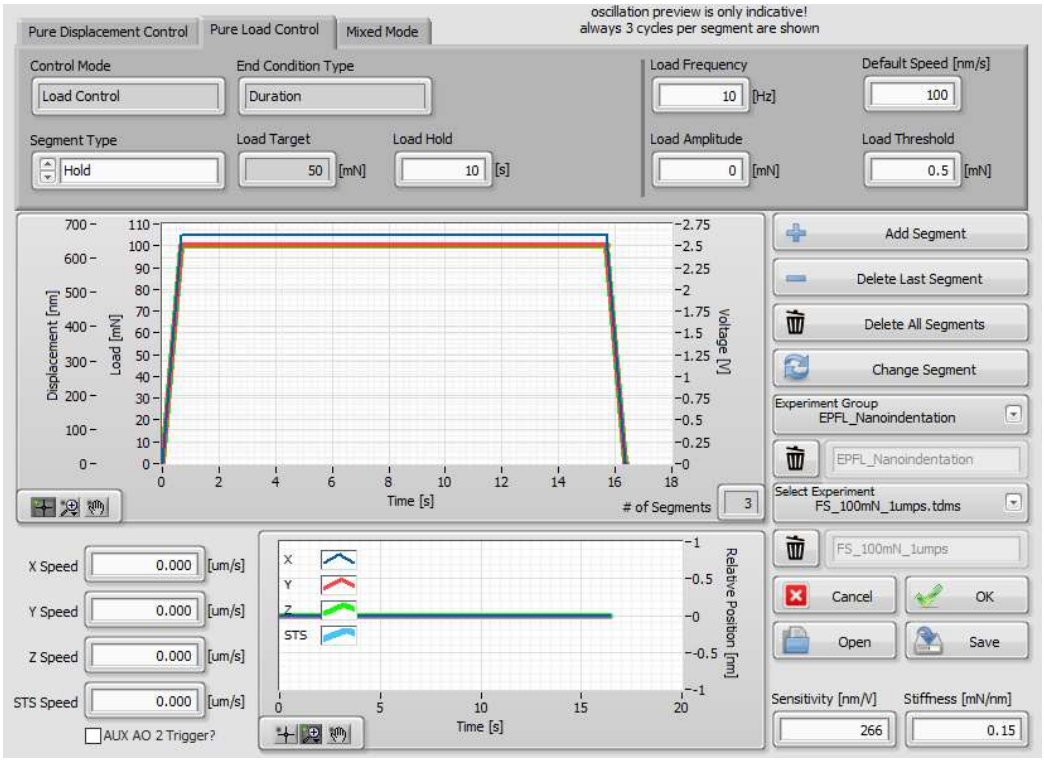
Fig. 4: Fused Silica (FS) sample mounted under the ASA in microscope position and indentation position

2) After coarse approach using MCS2 controller, perform an auto-approach (load target 1 mN) to detect the sample surface. Use a target distance to surface of 1000 nm.
3) Click on Standard Experiment → Setup

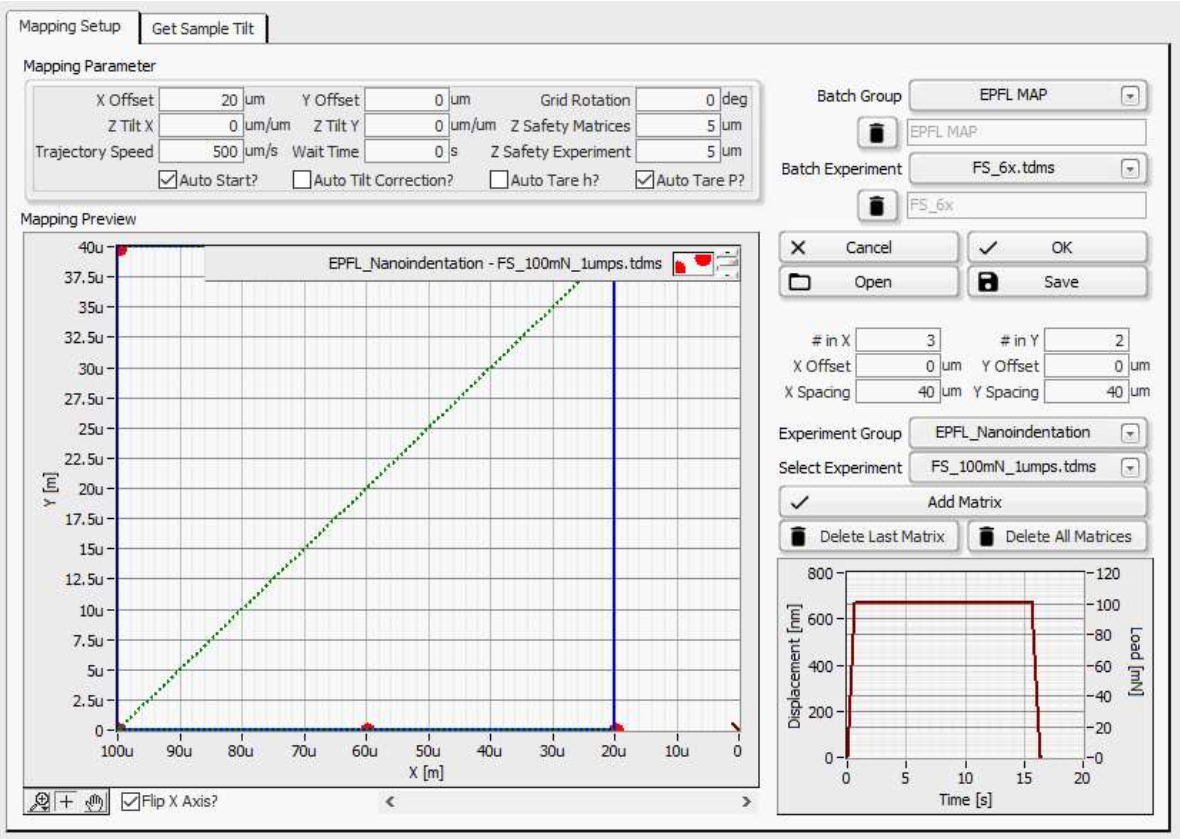


4) Create new experiment group and select a new experiment

5) In “mixed mode” create a new experimental profile, with 3 segments both in displacement control with a speed of 1000 nm/s:
    a. displacement control with a speed of 1000 nm/s, end condition type: “load >=” and mixed target: “100mN”, segment type: “linear”
    b. load control, hold segment, load hold: 15 s, end condition type: “duration”
    c. displacement control with a speed of 1000 nm/s, end condition type: “displacement <=” and mixed target 0 nm, segment type: “linear”



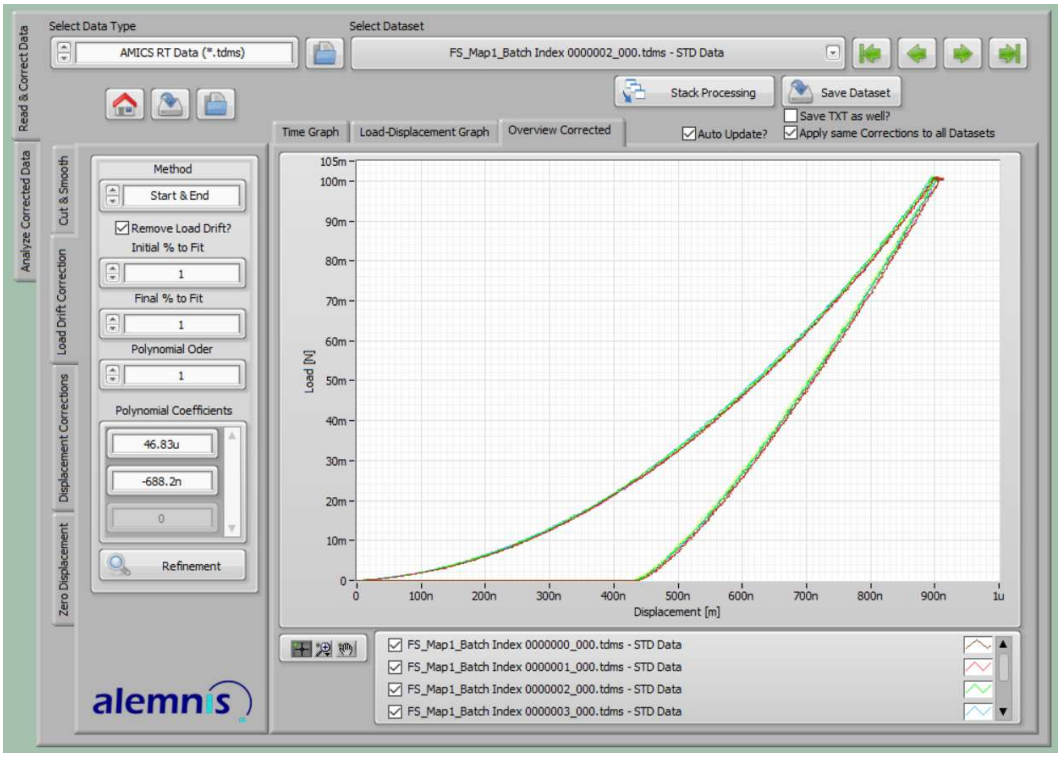
6) Click on Mapping → Setup
7) Create new batch group and select a new batch experiment
8) Create a 3x2 map with 40 um spacing, 5 um “Z safety experiment” and 20 um offset.



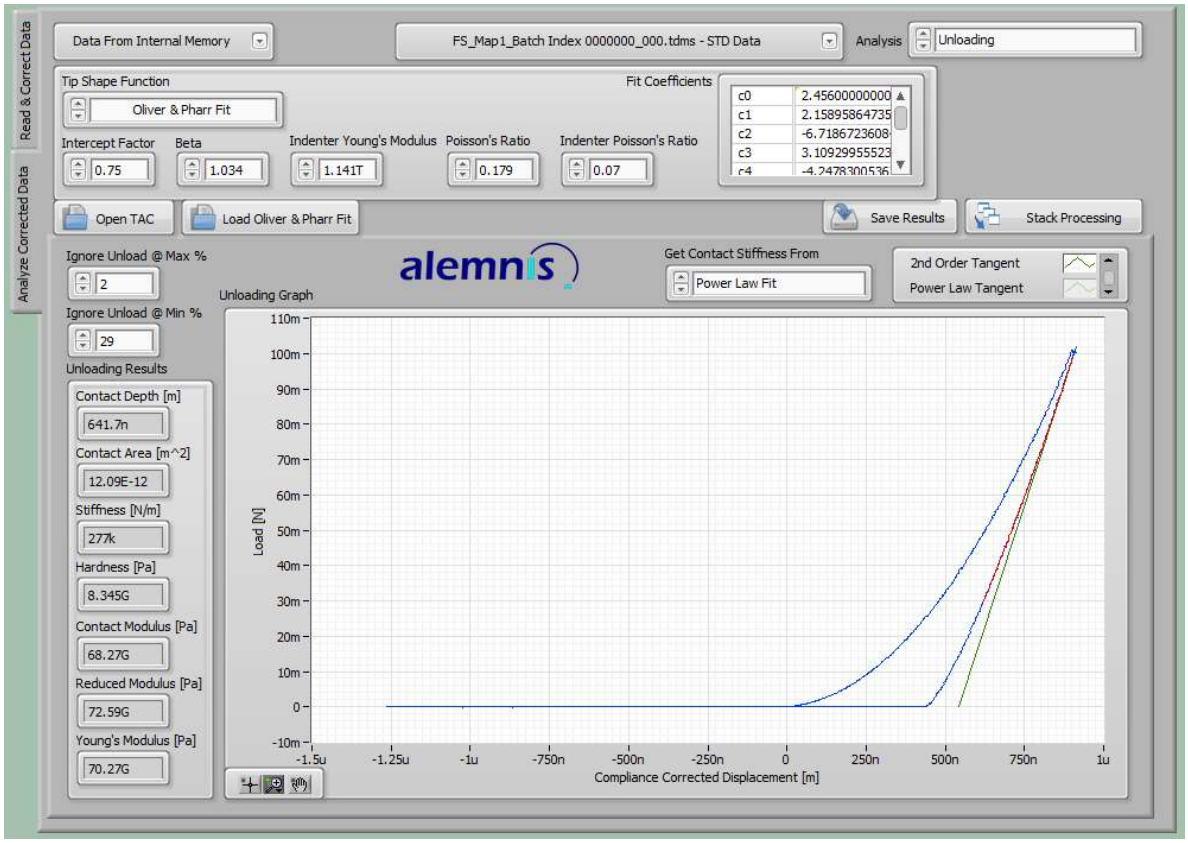
9) Select a new file name and select both sampling rate and PID frequency: 400 Hz
10) Run batch

GROUP 2: DATA INTERPRETATION

1) After completion of tests, open the DATA in AMMDA, the Alemnis software for post-processing. Check that the contact point of each indentation has been correctly chosen, and apply the required corrections (if necessary) such as load drift and compliance:



2) Open “analyze corrected data” tab and insert a Poisson’s ratio of 0.179 (nominal value for Fused Si reference sample)



3) Click on stack processing → memory and save the results
4) Open the file in excel and calculate the average Elastic Modulus, Hardness with inherent standard deviation.

	H	E
Mean GPa	8.40	70.9
SD GPa	0.07	0.4
RSD	0.8%	0.6%

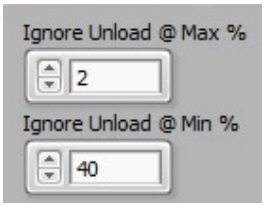
5) If the standard deviation exceeds 5%, inspect the superimposed curves and look for any measurements which seem outside of the group. Remove such measurements from the group calculation by unchecking on the left of the screen. How does this affect the final value ?

QUESTIONS:

(i) How does changing the contact point of the measurement affect the H and E values? Why?

(ii) Do you see any creep during the 15s hold at maximum load? If so, why?

(iii) In "analyze corrected data" tab, try to modify the values in "ignore Unload at min/max %" in AMMDA software. This allows the user to change the portion of the unloading curve used in the calculation of H and E:



Change these parameters and comment on their impact on the H and E values

(iv) What would be the minimum number of indentations required to make these results "meaningful"?

PRACTICAL 2 : Creep & Strain Rate Sensitivity

GROUP 1: EXPERIMENTAL PROCEDURE (CREEP OF PMMA)

Poly Methyl Methacrylate (PMMA) is a transparent thermoplastic with a glass transition temperature (Tg) ranging from 85 to 165°C, depending on composition. It usually shows linear viscoelastic behavior at low stress levels and nonlinear viscoelastic behavior at higher stress levels.

1) Clean the "PMMA" sample by blowing off with compressed air. Do not use any solvents to clean a polymer surface as this can modify the surface properties.
2) Place "PMMA" sample under the NHT measuring head as shown in Fig. 5

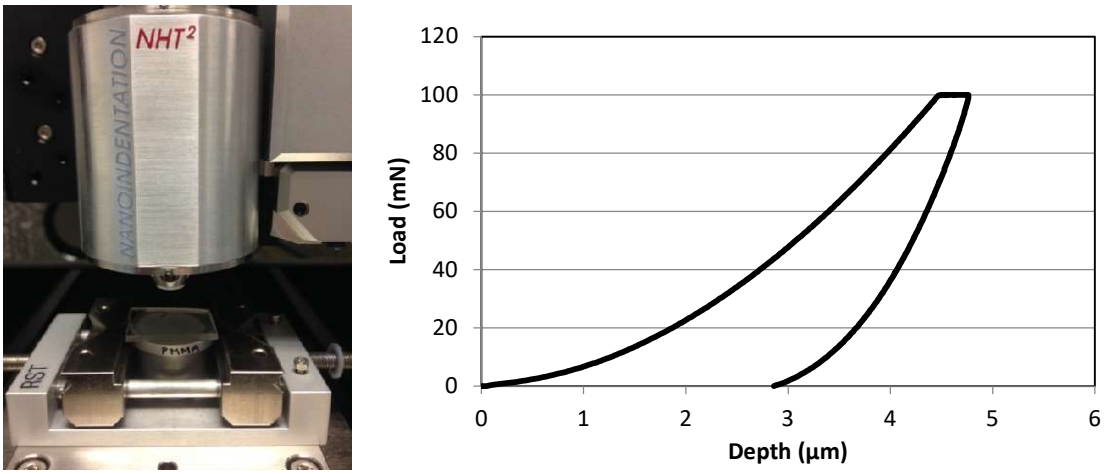
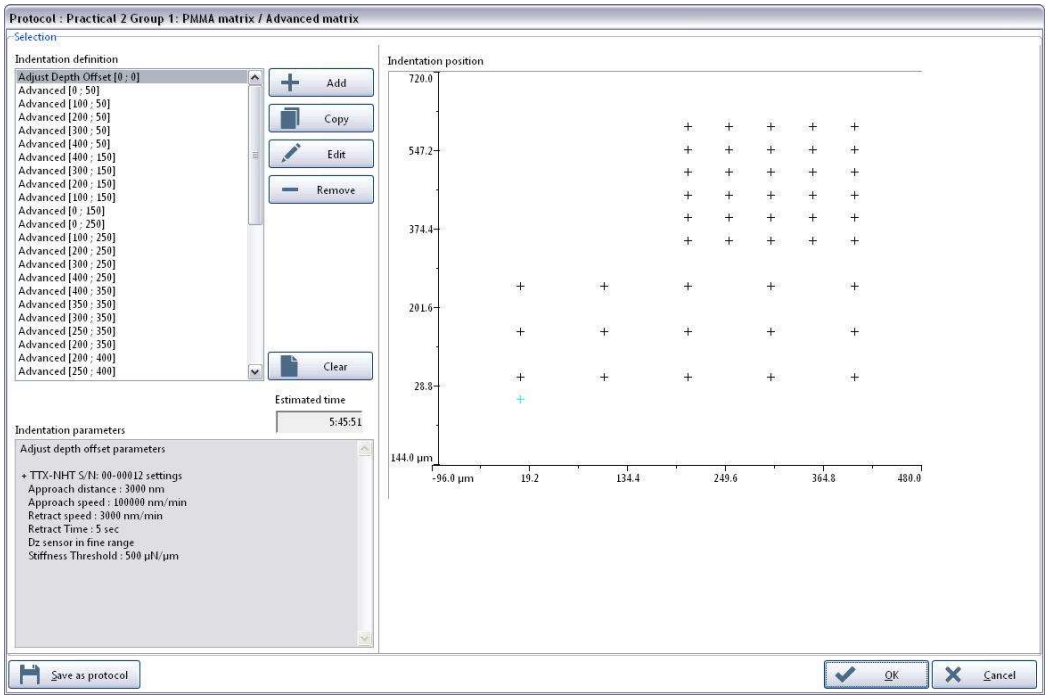
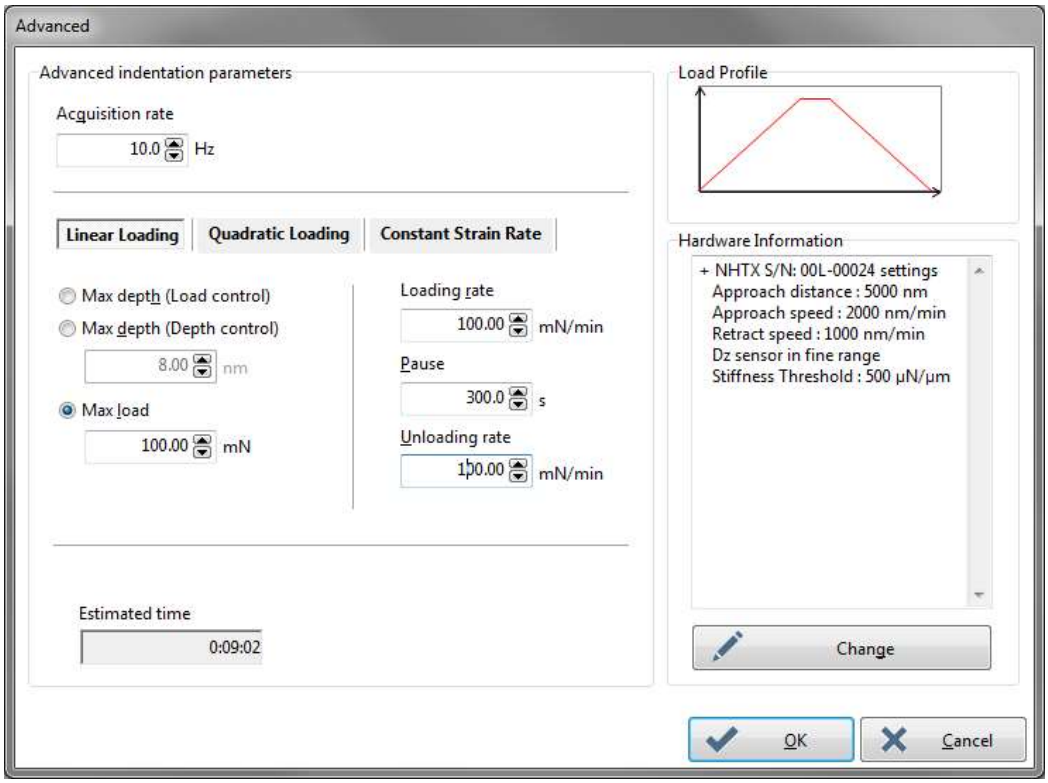


Fig. 5: PMMA sample mounted under the NHT head and load-depth curve at load of 100 mN

3) Create new file group, enter group name and enter Poisson's Ratio value of 0.42
4) Using Position Control, displace the sample under the optical video microscope, focus on the surface using the 5x objective followed by the 100x objective. Choose an area for measurement which looks clean. (displace in X and Y directions by using keyboard arrows).
5) In order to investigate the rate dependence of PMMA, program an Advanced Matrix with the following nanoindentations and include an Adjust Depth Offset (ADO):
   (i) Adjust Depth Offset (ADO)
   (ii) 5 advanced nanoindentations at 100 mN with loading rate of 500 mN/min. and 300s pause at maximum load, separation 100 µm
   (iii) 5 advanced nanoindentations at 100 mN with loading rate of 100 mN/min. and 300s pause at maximum load, separation 100 µm
   (iv) 5 advanced nanoindentations at 100 mN with loading rate of 10 mN/min. and 300s pause at maximum load, separation 100 µm
   (v) 5 advanced nanoindentations at 5 mN with loading rate of 3 mN/min. and no pause at maximum load, separation 50 µm
   (vi) 5 advanced nanoindentations at 5 mN with loading rate of 3 mN/min. and 10s pause at maximum load, separation 50 µm
   (vii) 5 advanced nanoindentations at 5 mN with loading rate of 3 mN/min. and 100s pause at maximum load, separation 50 µm
   (viii) 5 advanced nanoindentations at 5 mN with loading rate of 30 mN/min. and no pause at maximum load, separation 50 µm
   (ix) 5 advanced nanoindentations at 5 mN with loading rate of 30 mN/min. and 10s pause at maximum load, separation 50 µm
   (x) 5 advanced nanoindentations at 5 mN with loading rate of 30 mN/min. and 100s pause at maximum load, separation 50 µm

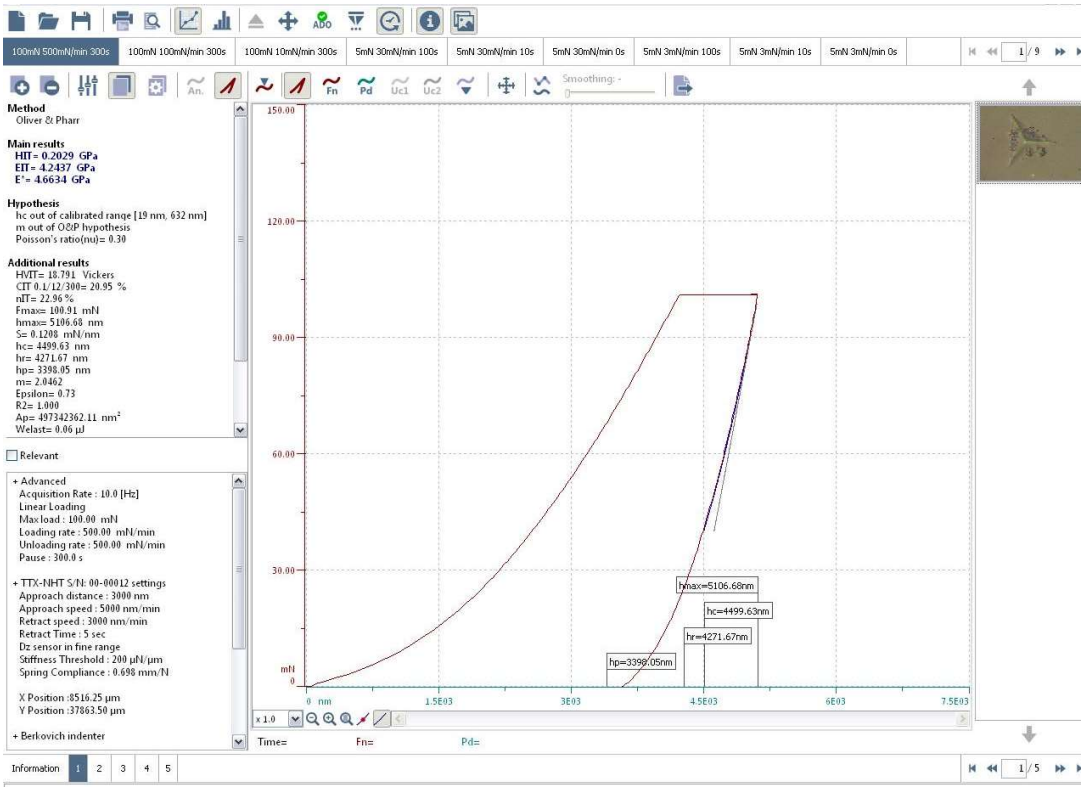




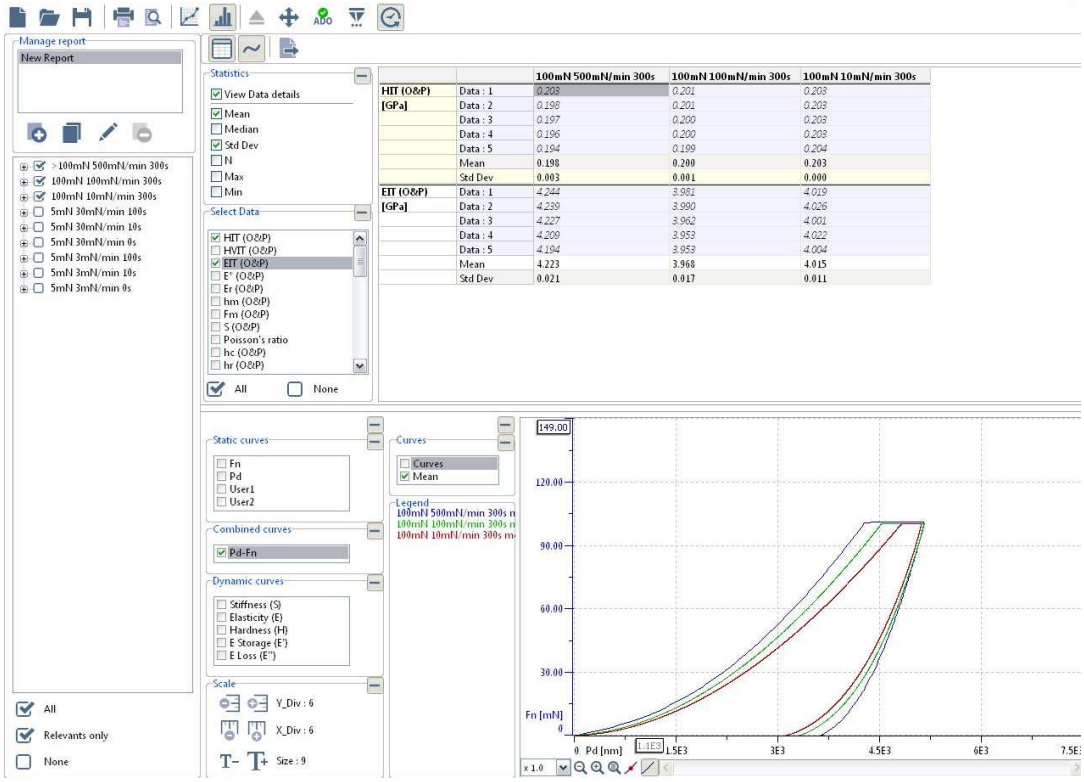
GROUP 1: DATA INTERPRETATION

1) Click “Yes” to move the sample under the optical microscope and focus with the 5x objective. Grab at least one image from each line of 5 indents using an appropriate objective (100x).

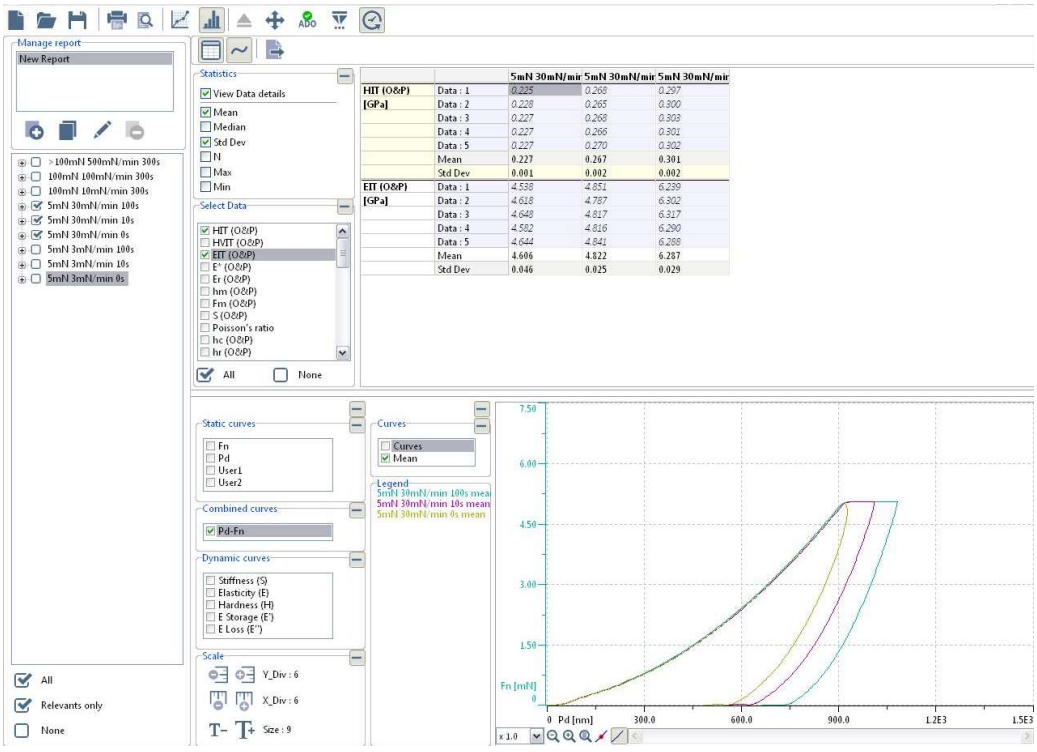
2) Check that the contact point of each indentation has been correctly chosen (right click on each indentation tab, select “Set contact point” and double-click on new position if necessary).
3) All the indents will have been saved in one single group making statistical analysis more difficult. Therefore, create 9 new data groups, each containing 5 indents, as shown:



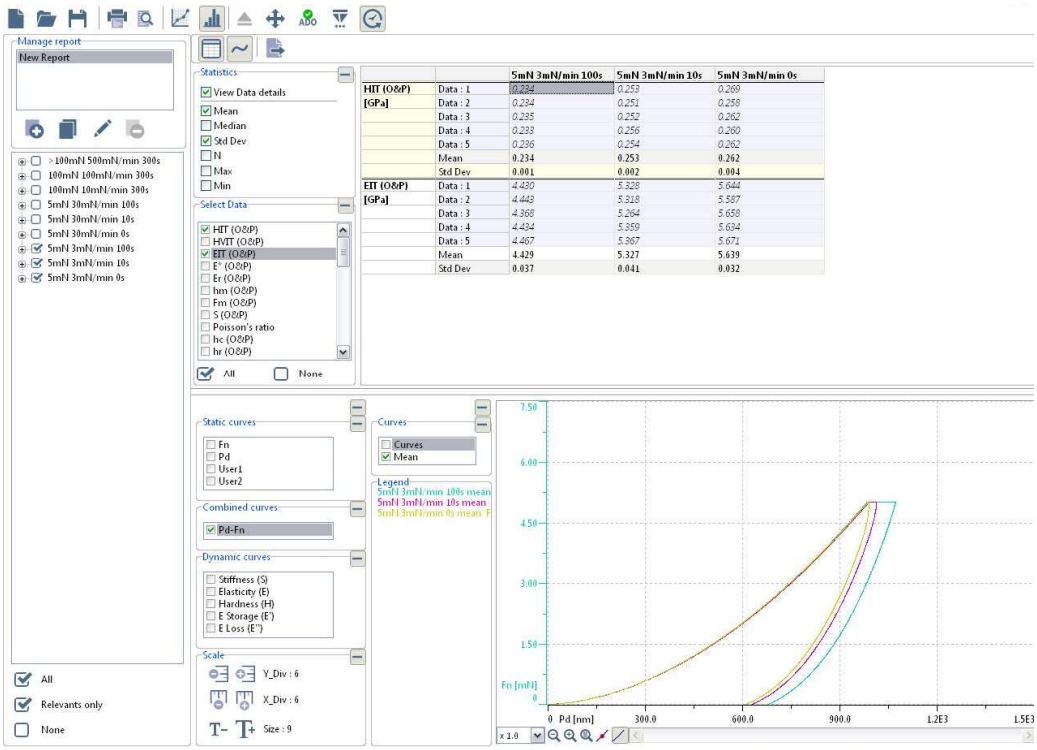
4) Select the 3 groups containing the 100 mN indents and choose “Combined Curves Pd-Fn” in the curve view in order to superimpose the mean of each measurement group on the same load-depth axes, provided that there are no outliers. Create a results table by checking the “View data details”, “Mean” and “Std Dev” checkboxes. Select HIT (hardness) and EIT (elastic modulus) for the data outputs:



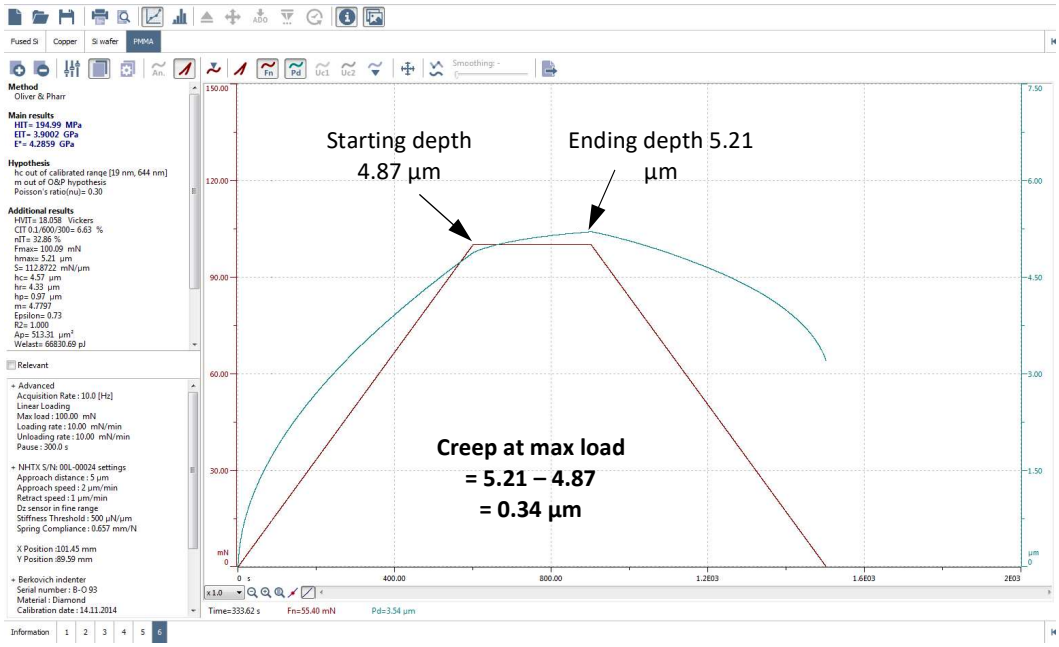
5) Now select the 3 groups containing the 5 mN, 30 mN/min indents and choose “Combined Curves Pd-Fn” in the curve view in order to superimpose the mean of each measurement group on the same load-depth axes, provided that there are no outliers. Create a results table by checking the “View data details”, “Mean” and “Std Dev” checkboxes. Select HIT (hardness) and EIT (elastic modulus) for the data outputs:



6) Now select the 3 groups containing the 5 mN, 3 mN/min indents and choose “Combined Curves Pd-Fn” in the curve view in order to superimpose the mean of each measurement group on the same load-depth axes, provided that there are no outliers. Create a results table by checking the “View data details”, “Mean” and “Std Dev” checkboxes. Select HIT and EIT for the data outputs:



7) Return to curves view and select Fn and Pd icons in order to display the 100 mN load and depth datasets as a function of time:



8) The creep in the depth signal can clearly be seen over the 300s hold period. By passing the cursor over the beginning and end point of this creep period, note down the starting and ending depth for each of the 3 measurements and calculate the "creep at max load" value.
9) Plot and complete a results table (in Excel or other) which includes the following:

Indent Number	Loading Rate (mN/min.)	Measured Creep at max. load (nm)	Hardness, HIT (MPa)	Elastic Modulus, EIT (GPa)	Creep, CIT (%)
1	500				
2	100				
3	10				

The Creep, CIT, value is calculated automatically by the software. It is defined as follows:

$$CIT = \frac{h_2 - h_1}{h_1}.100$$

where:

h_1 is the indentation depth at time t_1 of reaching load F (which is kept constant)
h_2 is the indentation depth at time t_2 of holding the constant load, F.

QUESTIONS:

(i) Comment on the differences in curve shape between the three 100 mN loading rates. This is clear on the superimposed curves. Refer specifically to the loading portion, the hold portion and the unloading portion.
(ii) Why is the creep at maximum load not linear…?
(iii) How does the loading rate influence the calculated values of H and E…?
(iv) Are there any differences observed between the manually measured creep at maximum load and the calculated CIT value? If so, why…?
(v) How does the loading rate influence the manual and calculated values of creep..? Comment on this in relation to the known time-dependent properties of this material.

GROUP 2: EXPERIMENTAL PROCEDURE (STRAIN RATE SENSITIVITY OF PMMA)

Poly Methyl Methacrylate (PMMA) is a transparent thermoplastic with a glass transition temperature (Tg) ranging from 85 to 165°C, depending on composition. It usually shows linear viscoelastic behavior at low stress levels and nonlinear viscoelastic behavior at higher stress levels.

1) Clean the "PMMA" sample by blowing off with compressed air. Do not use any solvents to clean a polymer surface as this can modify the surface properties.
2) Place "PMMA" sample under the ASA indentation head as shown in Fig. 6

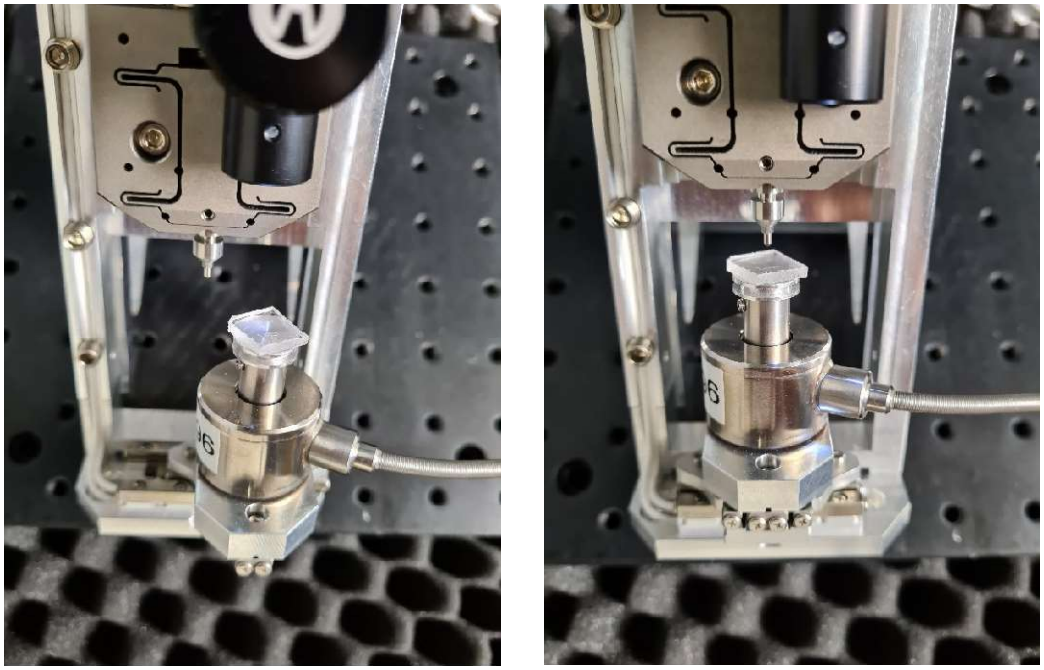
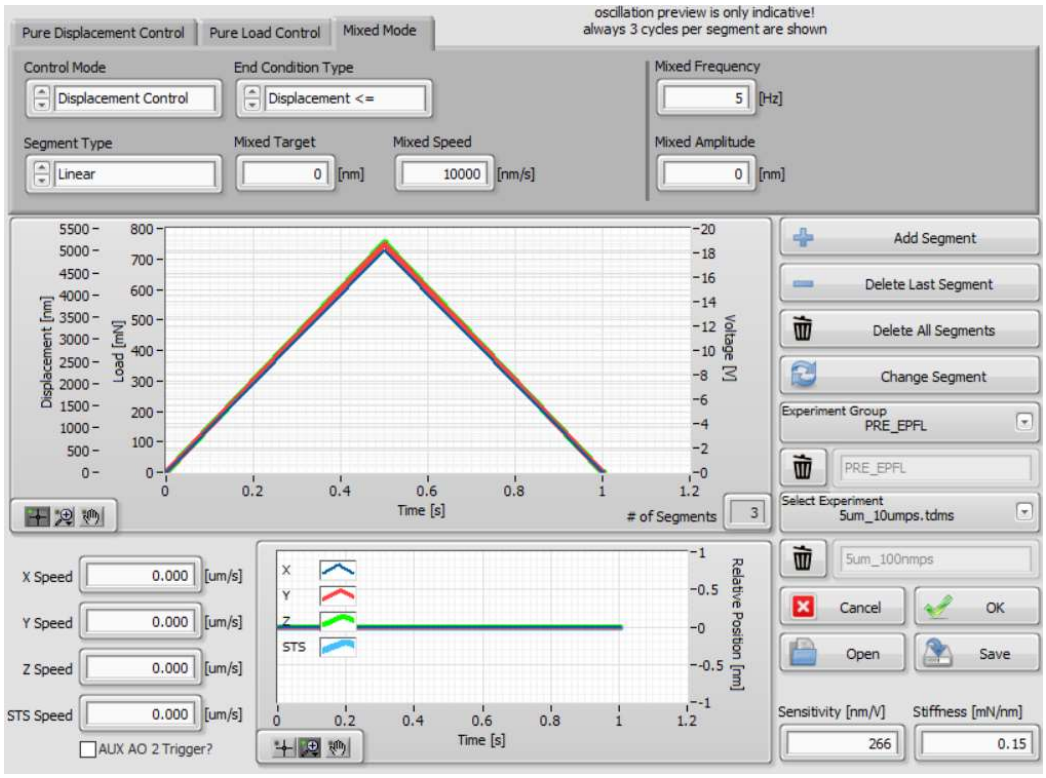
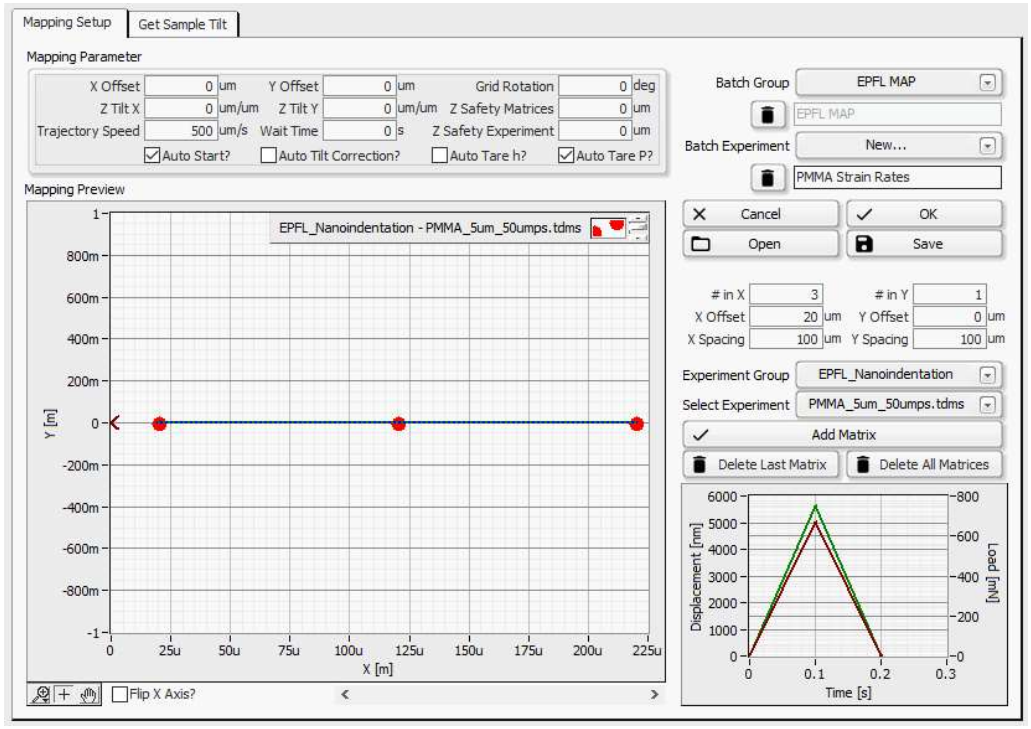


Fig. 6: PMMA sample mounted under the ASA in microscope position and indentation position

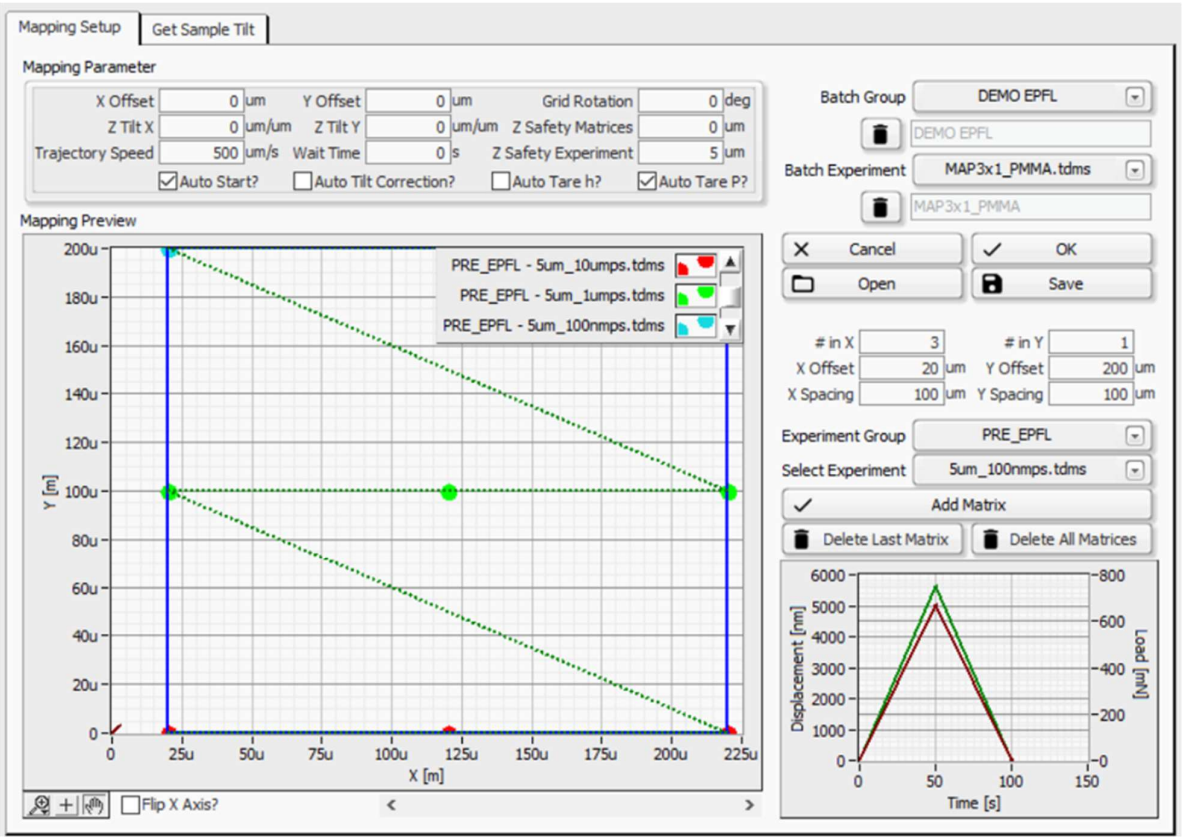
3) After coarse approach using MCS2 controller, perform an auto-approach (load target 1 mN) to detect the sample surface. Use a target distance to surface of 2000 nm.
4) Click on Standard Experiment → Setup
5) Create new experiment group and select a new experiment
6) In "mixed mode" create a new experimental profile, with 3 segments in displacement control with a speed of 10000 nm/s:
    a. end condition type: "load >=" and mixed target: "1 mN"
    b. end condition type: "displacement+" and mixed target: "5000 nm"
    c. end condition type: "displacement <=" and mixed target: "0"



7) Create 2 other protocols with 1000 nm/s and 100 nm/s displacement rates, respectively.
8) Click on Mapping → Setup
9) Create new batch group and select a new batch experiment
10) Create a 3x1 matrix of the 10000 nm/s protocol with 100 um spacing and 20 um X offset.



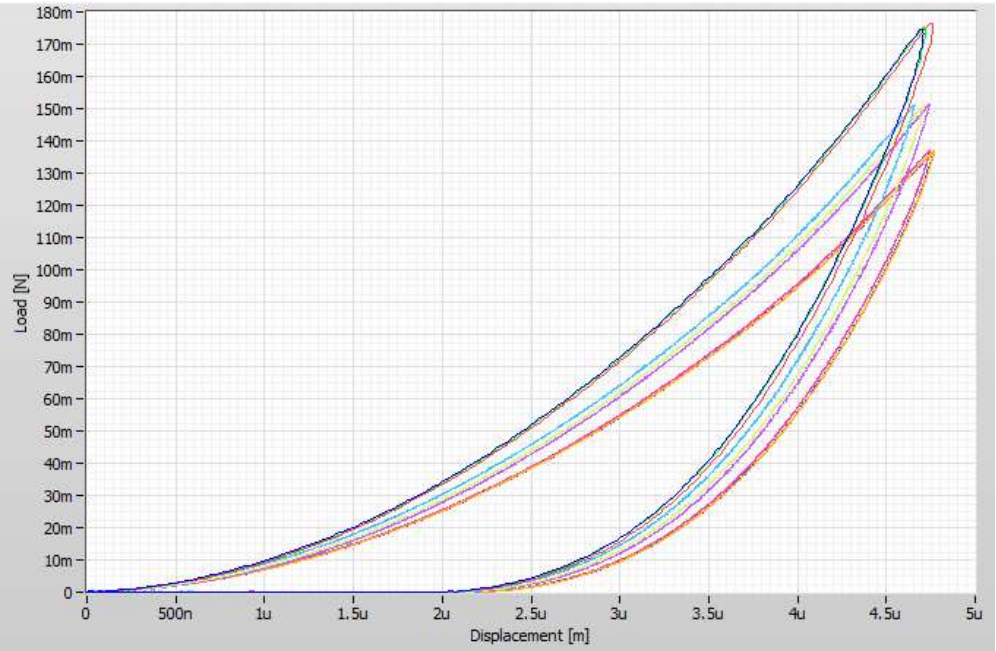
11) Add 2 other 3x1 matrices for the 1000 and the 100 nm/s protocols. Add a y offset of 100 um each time a matrix is added and keep the x offset 20 um.



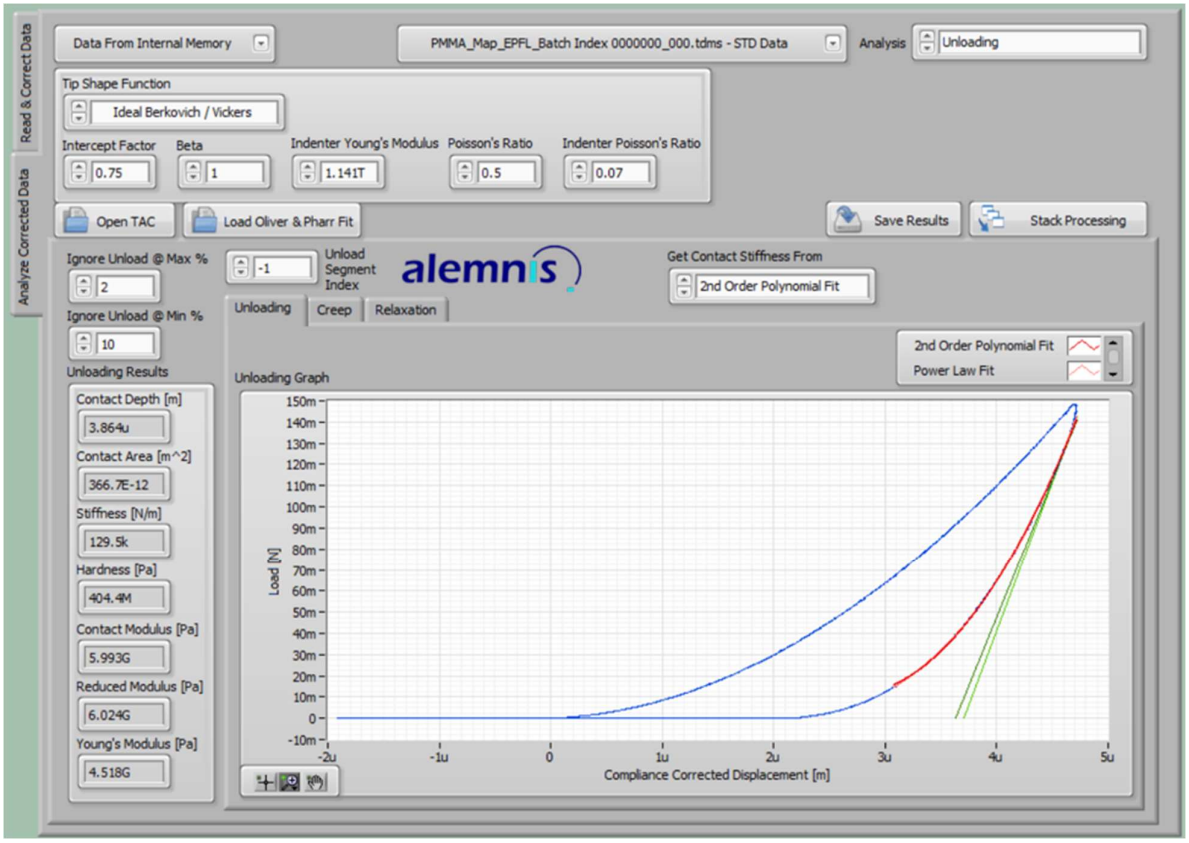
12) Select a new file name and select both sampling rate and PID frequency: 1 kHz.
13) Run batch

GROUP 2: DATA INTERPRETATION

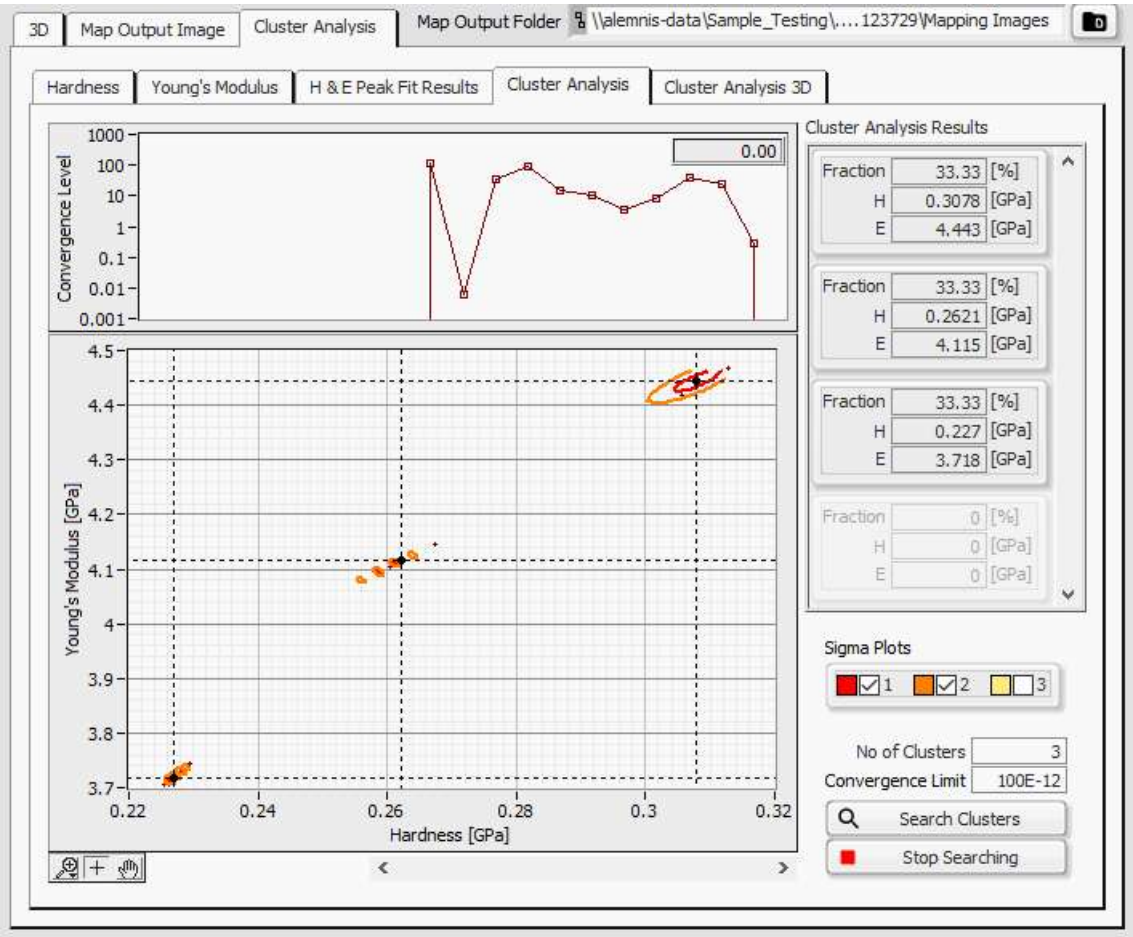
1) After completion of tests, open the DATA in AMMDA, the Alemnis software for post-processing. Check that the contact point of each indentation has been correctly chosen, and apply the required corrections (if necessary) such as load drift and compliance:



2) Move to analyze corrected data and insert a Poisson's ratio of 0.5 (nominal value for Fused PMMA)



3) Click on stack processing → memory and save the results
4) Open the file in AMMDA 3D and apply some statistics in the cluster analysis tab, like average Elastic Modulus, Hardness with inherent standard deviation.



QUESTIONS:

(i) Comment on the differences in curve shape between the three displacement rates. This is clear on the superimposed curves.

(ii) Why is the elastic modulus changing as a function of displacement rate? Does the polymer chain have an influence ?

(iii) How does displacement vs. load control influence the “nose” in the unloading curve ?

PRACTICAL 3 : Metals

Copper is a soft and ductile metal which exhibits a relatively plastic response and therefore work hardens as a function of deformation. Exposure of its surface to ambient air will produce a native cuprous oxide layer (Cu_2O) which may have a thickness of 100 nm or more.

GROUP 1: EXPERIMENTAL PROCEDURE (COPPER)

1) Clean the "Copper" sample by blowing off with compressed air. Additional cleaning can be done with isopropanol if necessary.
2) Place "Copper" sample under the NHT measuring head as shown in Fig. 7

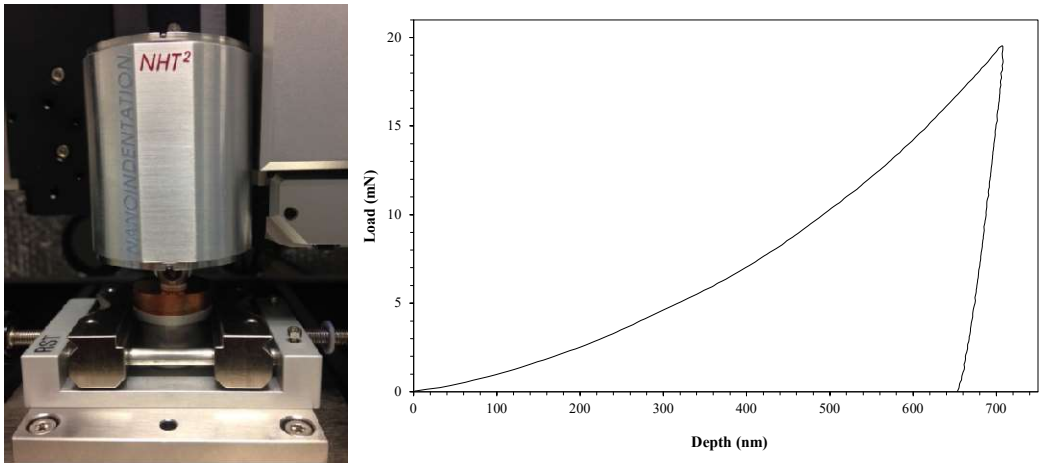
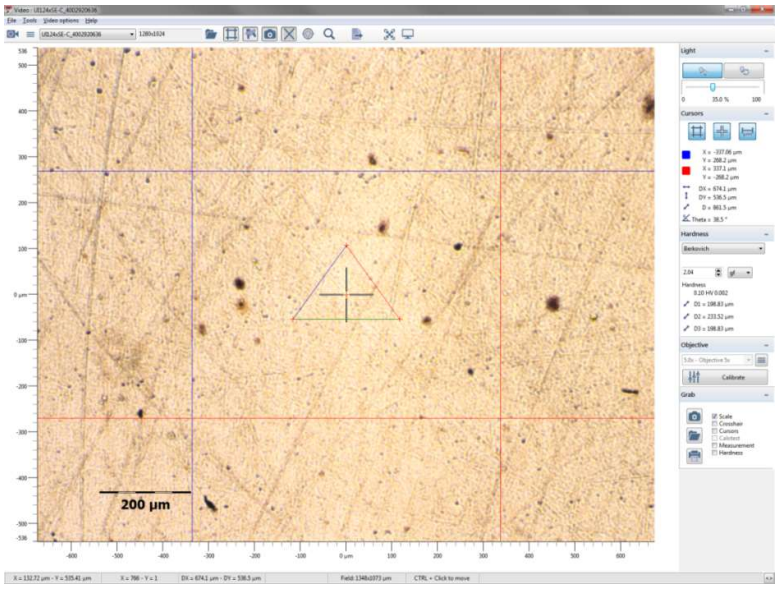


Fig. 7: Copper sample mounted under the NHT head and load-depth curve at load of 20 mN

3) Create new file group, enter group name and enter Poisson's Ratio value of 0.35
4) Using Position Control, displace the sample under the optical video microscope, focus on the surface using the 5x objective followed by the 100x objective. Choose an area for measurement which looks clean and undamaged by polishing scratches (displace in X and Y directions by using keyboard arrows):



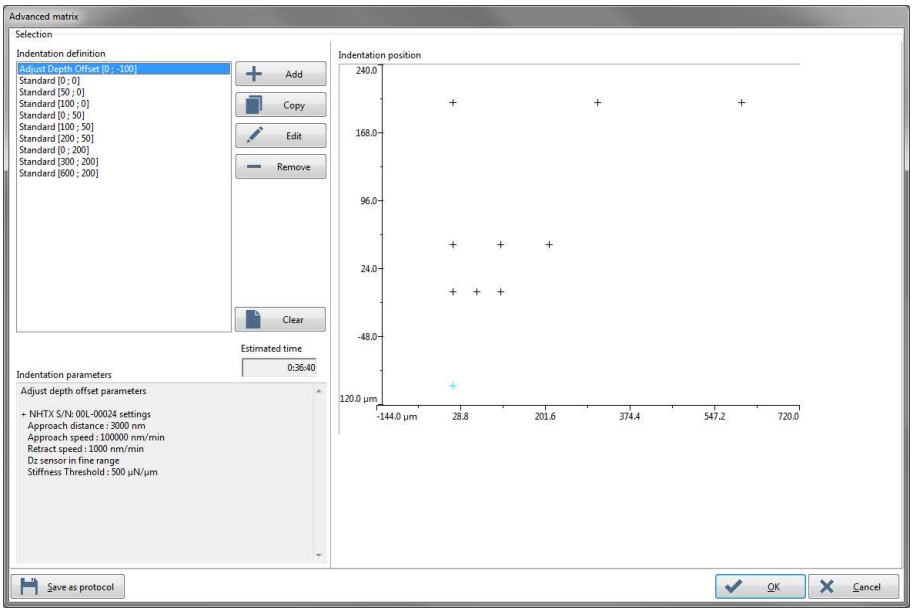
5) Program the software to make an Advanced Matrix with the following parameters:

(i) Add an "Adjust Depth Offset" at the beginning of the matrix.

(ii) 3 “Standard” nanoindentations with an applied load of 10 mN, pause at maximum load of 15 seconds and separation 50 µm

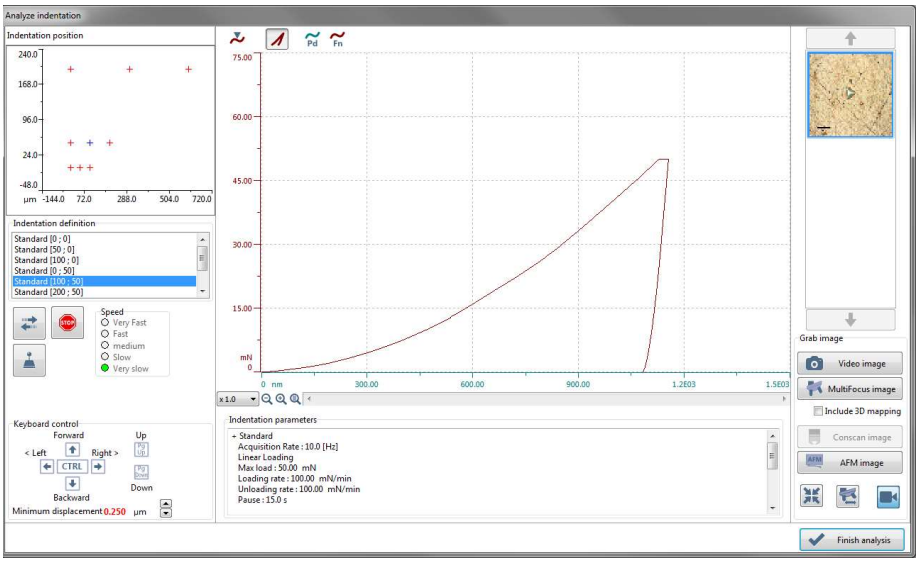
(iii) 3 “Standard” nanoindentations at 50 mN (loading rate 100 mN/min., pause 15s, separation 100 µm)

(iv) 3 “Standard” nanoindentations at 300 mN (loading rate 600 mN/min., pause 15s, separation 300 µm).

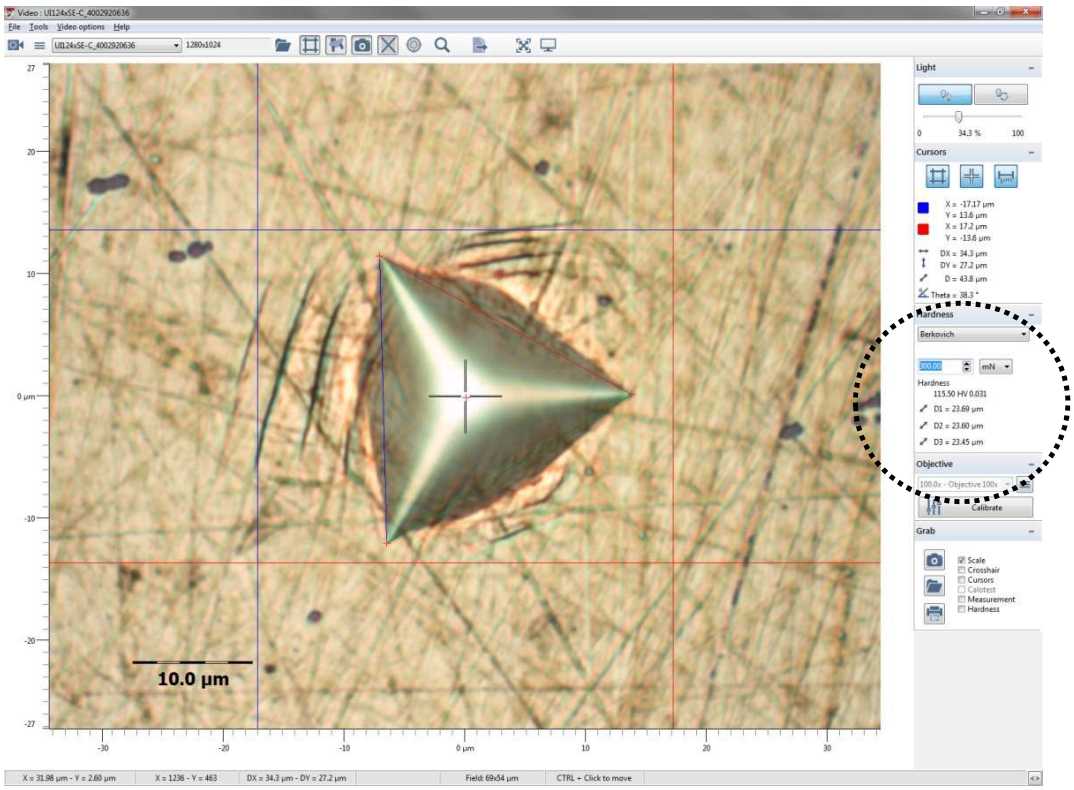


GROUP 1: DATA INTERPRETATION

1) After testing is completed, click “Yes” to move the sample under the optical microscope and focus with the 5x objective. Grab at least one image from each group of 3 indents using an appropriate objective (100x).



2) Whilst focused on a 300 mN imprint, in the Video window, select “Berkovich” in the Hardness box (right side) and enter 300 mN as the applied load used. Then place the 3 corners of the triangle onto the Berkovich indent in the image. The calculated conventional hardness value (H_V) will be displayed in the Hardness box.



3) Repeat the calculation of conventional hardness on one of the 50 mN and one of the 10 mN indents.
4) After completion of tests, check that the contact point of each indentation has been correctly chosen (right click on each indentation tab, select “Set contact point” and double-click on new position if necessary)
5) Plot and complete the HV column of the following results table:

Max indentation load (mN)	Hardness, HIT (MPa)	Elastic Modulus, EIT (GPa)	Recalculated Vickers Hardness, HVIT (Vickers)	Calculated Conventional Hardness, HV (Vickers)	%age difference between HV and HVIT
10	1693	156	157		
50	1657	151	154		
300	1313	179	122		

GROUP 2: EXPERIMENTAL PROCEDURE (COPPER)

Copper is a soft and ductile metal which exhibits a relatively plastic response and therefore work hardens as a function of deformation. Exposure of its surface to ambient air will produce a native cuprous oxide layer (Cu_2O) which may have a thickness of 100 nm or more.

1) Clean the "Copper" sample by blowing off with compressed air. Do not use any solvents to clean a polymer surface as this can modify the surface properties.
2) Place "Copper" sample under the ASA measuring head as shown in Fig. 8

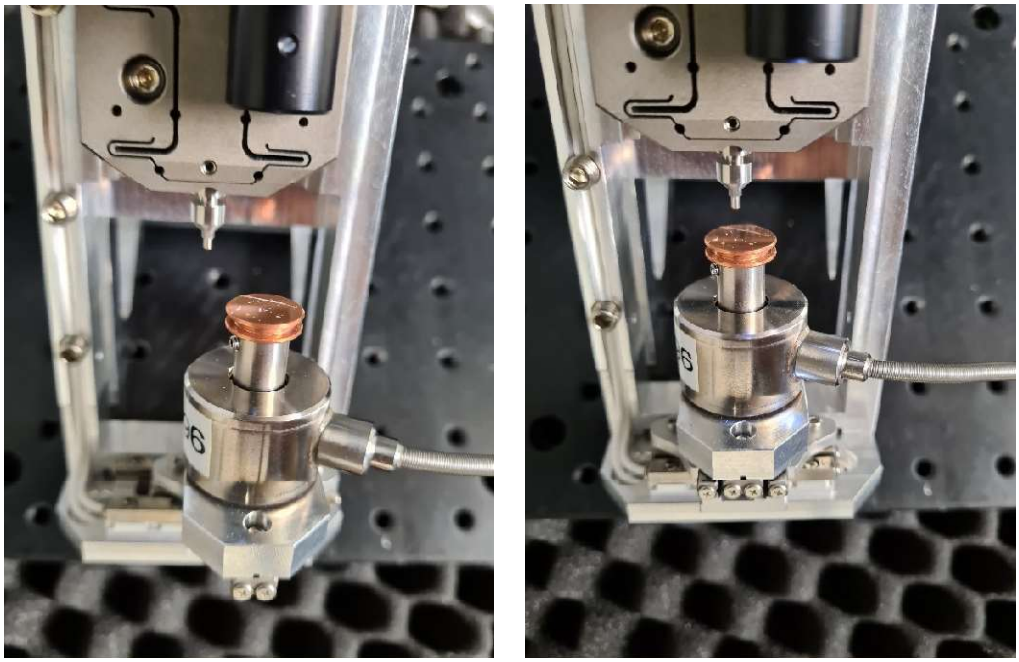
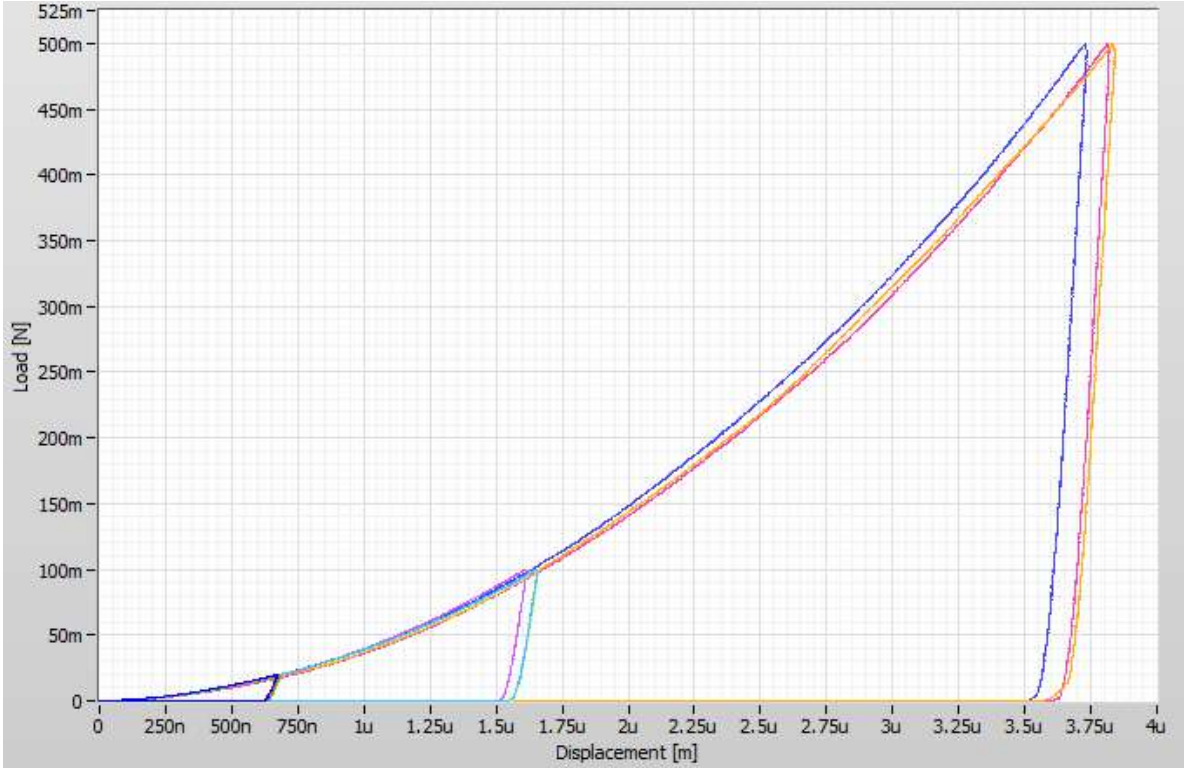


Fig. 8: Copper sample mounted under the ASA in microscope position and indentation position

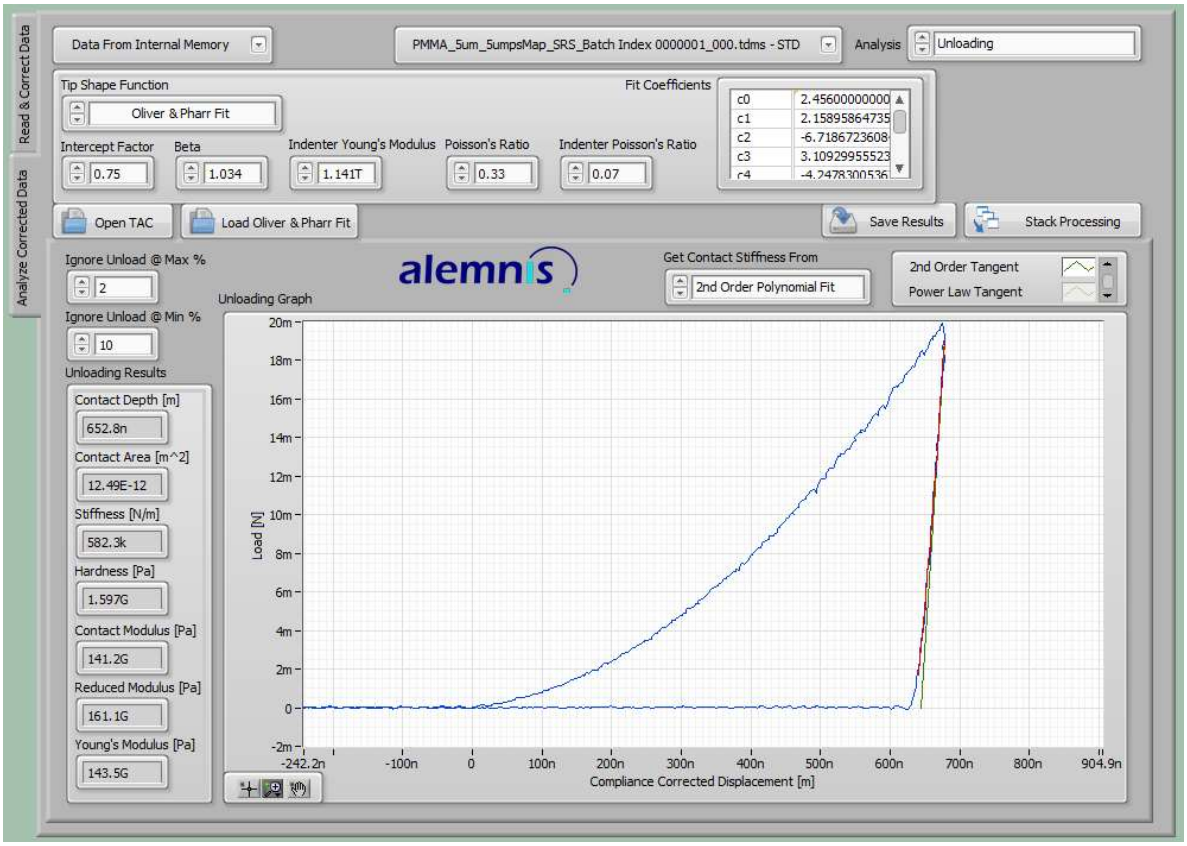
3) After coarse approach using MCS2 controller, perform an auto-approach (load target 1 mN) to detect the sample surface. Use a target distance to surface of 1000 nm.
4) Click on Standard Experiment → Setup
5) Create new experiment group and select a new experiment
6) In "mixed mode" create a new experimental profile, with 2 segments in displacement control with a speed of 500 nm/s:
    a. end condition type: "load >=" and mixed target: "20 mN"
    b. end condition type: "displacement<=" and mixed target: "0 nm"
7) Create 2 other protocols with 100 and 500 mN maximum load, respectively.
8) Click on Mapping → Setup
9) Create new batch group and select a new batch experiment
10) Create a 3x1 matrix of the 20 mN protocol with 100 um spacing and 20 um X offset.
11) Add 2 other 3x1 matrices for the 100 mN and the 500 mN protocols. Add a y offset of 100 um each time a matrix is added and keep the x offset 20 um.
12) Select a new file name and select both sampling rate and PID frequency: 200 Hz
13) Run batch

GROUP 2: DATA INTERPRETATION

1) After completion of tests, open the DATA in AMMDA, the Alemnis software for post-processing. Check that the contact point of each indentation has been correctly chosen, and apply the required corrections (if necessary) such as load drift and compliance:



2) Move to analyze corrected data and insert a Poisson's ratio of 0.33 (nominal value for Copper)



3) Click on stack processing → memory and save the results
4) Plot and complete the HIT and EIT columns of the following results table:

Max indentation load (mN)	Hardness, HIT (MPa)	Elastic Modulus, EIT (GPa)
20		
100		
500		

QUESTIONS:

<u>On the Anton Paar TTX-NHT:</u>

(i) How does the focus of the microscope change the positioning of the triangle corners when measuring the conventional hardness?
(ii) What phenomenon do you notice around the indentation, from the microscope image?
(iii) Do you see any creep during the 15s hold at maximum load? If so, why?

<u>On the Alemnis ASA:</u>

(i) From the results table, what conclusions can you make about the HIT and EIT values as a function of the maximum indentation load? Do you think that the applied load influences these values? If so, why?
(ii) How does changing the percentage of the unloading curve fit affect the measured values?
(iii) How does changing the fit model affect the measured values?

PRACTICAL 4 : Advanced mapping of multiphase materials

Although the positional accuracy of a nanoindenter can be used to hit actual grains within a microstructure, the results obtained may not be representative of the grain properties owing to errors such as grain boundary constraints, residual stresses, preferential polishing, etc. It is therefore preferable to "map" the surface with a significantly large matrix of indentations and use statistical analysis to calculate average property values for each phase present.

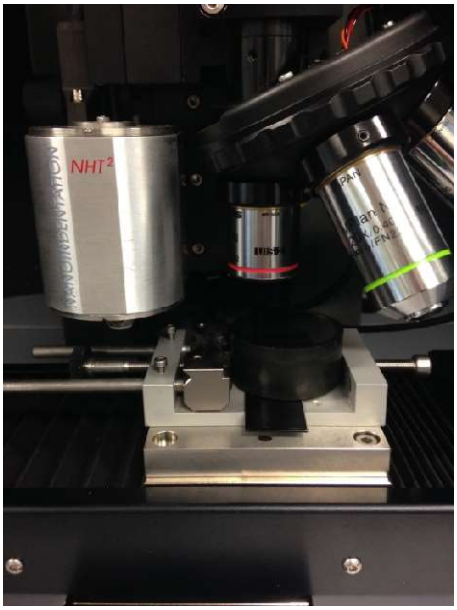
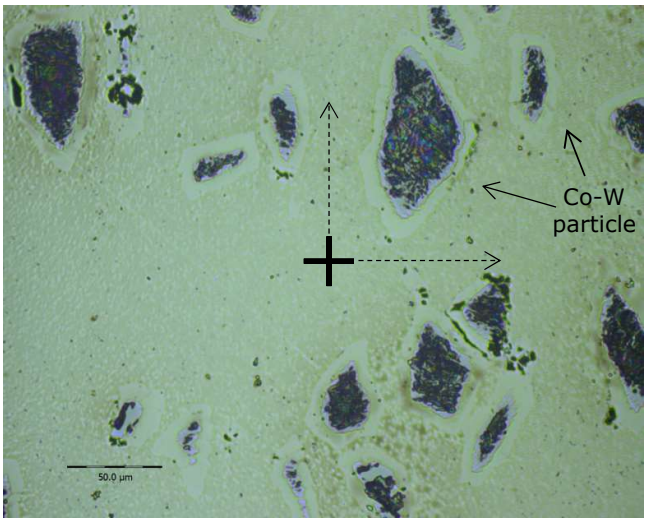


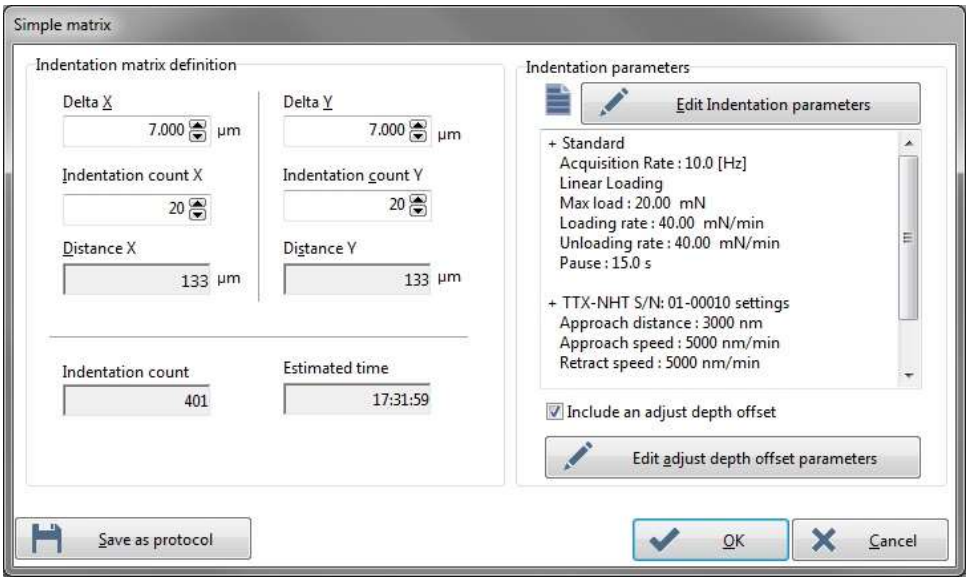
Fig. 9: Multiphase Co-W mounted under the NHT head

GROUP 1: EXPERIMENTAL PROCEDURE (Co-W ALLOY)

1) Clean the "Co-W Alloy" sample by blowing off with compressed air.
2) Place "Co-W Alloy" sample under the NHT measuring head as shown in Fig. 9. A rigid spacer may be required under the sample in order to have a sample height which exceeds the vice.
3) Using Position Control, displace the sample under the optical video microscope, focus on the surface using the 5x objective followed by the 100x objective. Choose an area for measurement which looks clean and where there are significant amounts of Co-W particles visible (displace in X and Y directions by using keyboard arrows). The crosshair needs to be set in the precise position where the matrix will commence. The matrix size then needs to be calculated so that the particles of interest will be included in an appropriate manner. Note that a positive X nd Y matrix will be in the direction of dotted arrows.

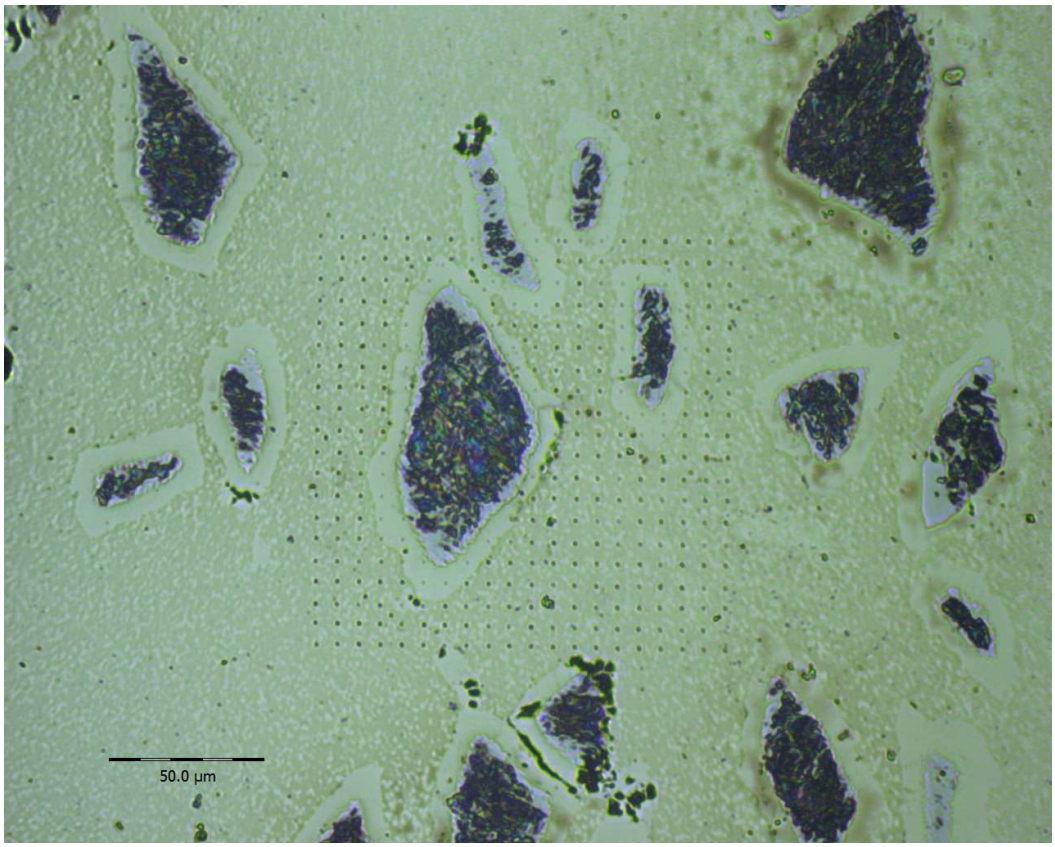


4) Program the software to make a “Simple Matrix” with a grid of 20 x 20 “Standard” nanoindentations with an applied load of 20 mN, loading rate of 40 mN/min. and a pause at maximum load of 15 seconds. Set the Approach and Retract speeds to 5000 nm/min. Separate the indentations by an appropriate distance; in this case 7 µm (use Delta X and Y value of 7 µm). Remember to add an Adjust Depth Offset:

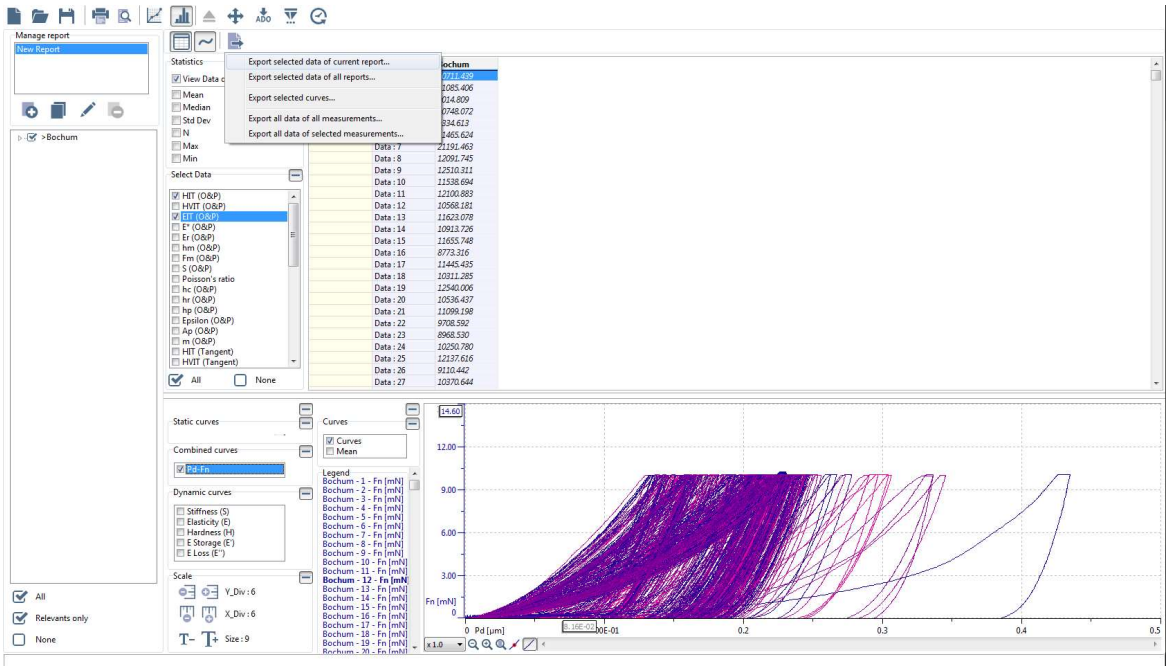


GROUP 1: DATA INTERPRETATION

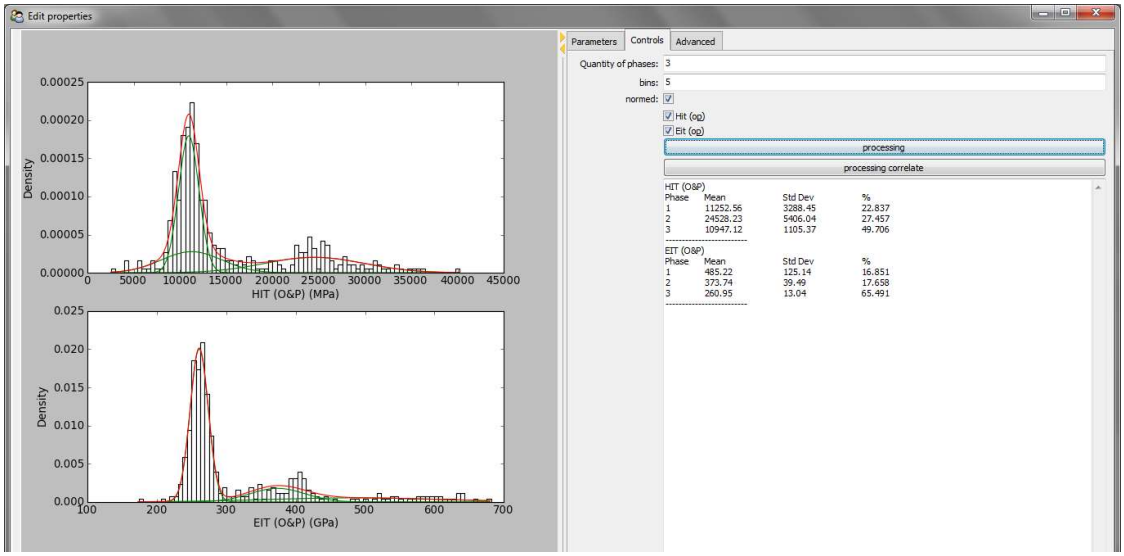
1) After completion of tests, click “Yes” to go under the microscope and grab some images of the resultant matrix. It should look something like this:



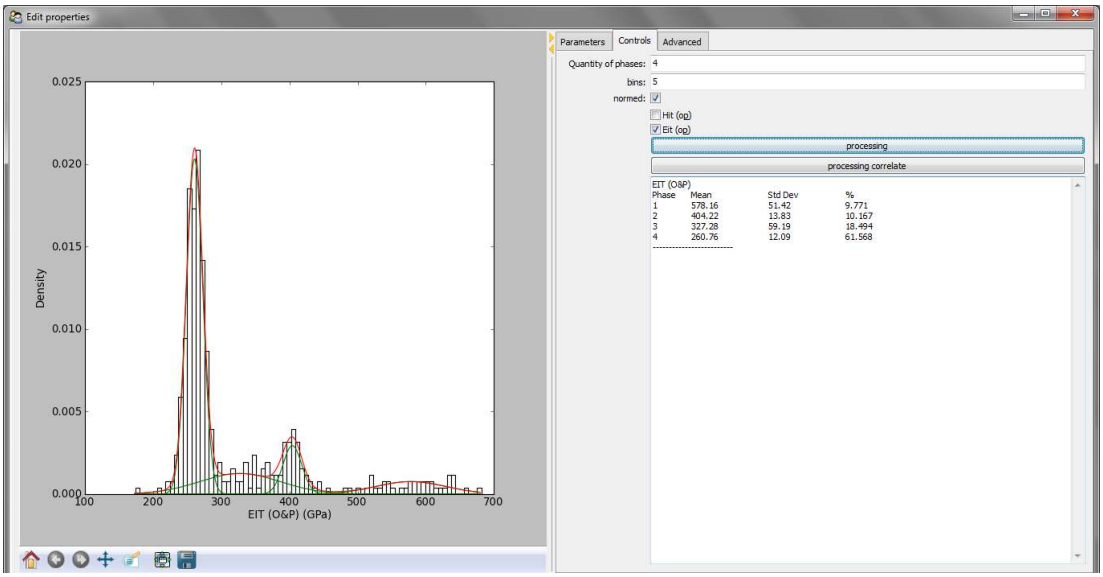
2) Check that the contact point of each indentation has been correctly chosen (right click on each indentation tab, select “Set contact point” and double-click on new position if necessary)
3) Click on the “Statistics” page and select “All” measurements (Tick “All” in bottom left of screen)
4) Choose “Combined Curves” in the curve view in order to superimpose the 400 measurements on the same load-depth axes:



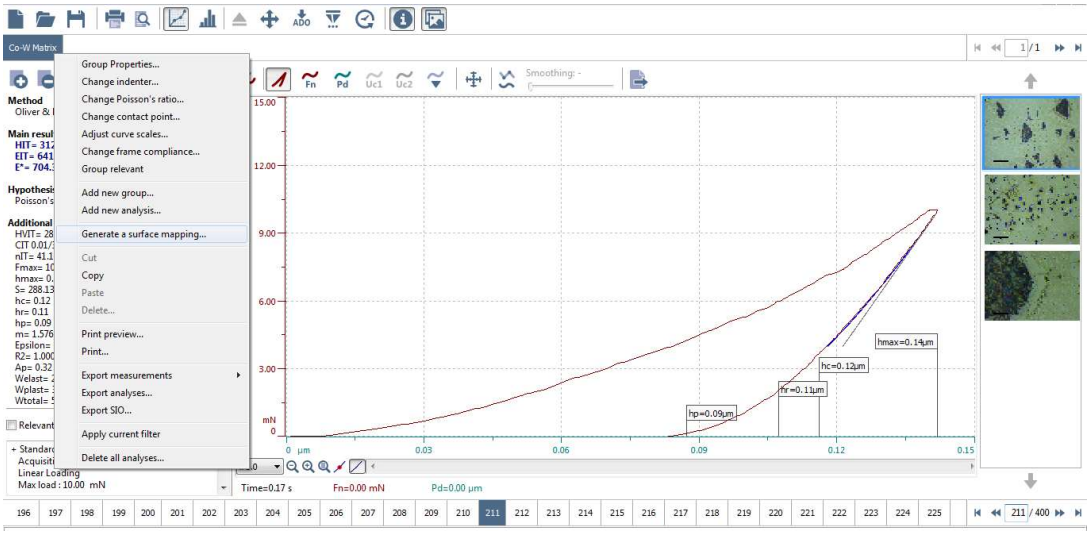
5) Create a results table by checking the “View data details” and select HIT (hardness) and EIT (elastic modulus) for the data outputs.
6) Export the HIT and EIT values by clicking on the Export icon and selecting “Export selected data of current report”. Save the data as a TXT file.
7) Open Gaussian Fit software and select the TXT file from the same location. Click on the “Controls” tab, select 3 phases, 5 bins, HIT, EIT and click on “Processing”. The Gaussian fits for HIT and EIT will display on the left screen as shown below:



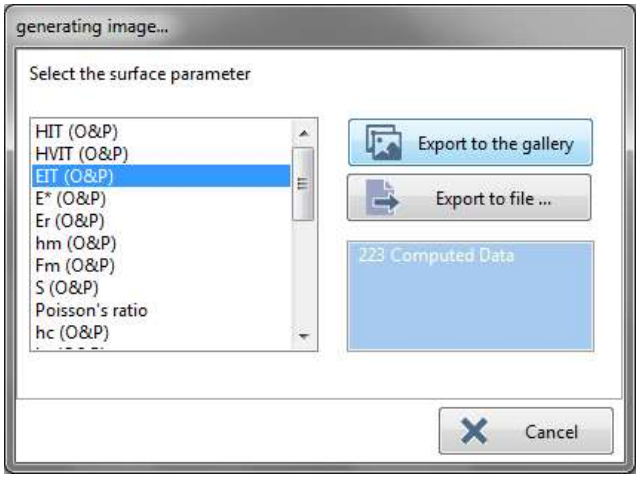
8) Note that in this case, although we know that 3 phases are present, accuracy may be improved by selecting 4 phases so that any noise is taken out as a separate phase. Here is an example for the EIT data where this works well:



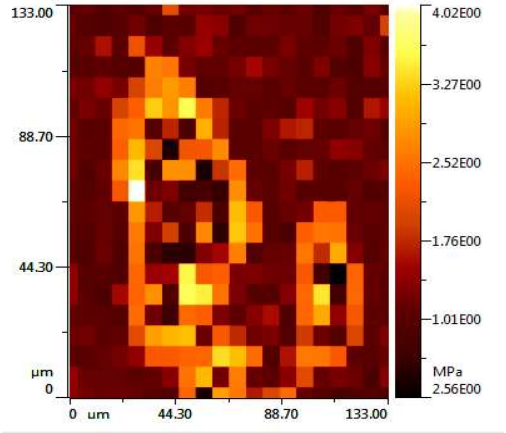
9) Go back to the Indentation software and select “Curves” view. Right-click on the Group Tab and select “Generate a Surface Mapping”:



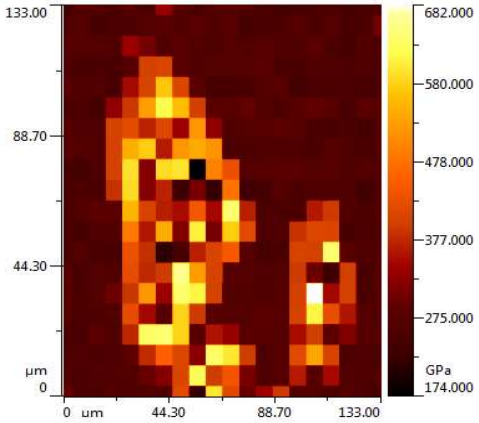
10) Choose HIT and “Export to the Gallery”. This will generate a surface map of the HIT values and pin it to the image gallery on the right of the screen. Repeat this step to generate the surface map with the EIT values.



11) Typical surface maps will look something like this:

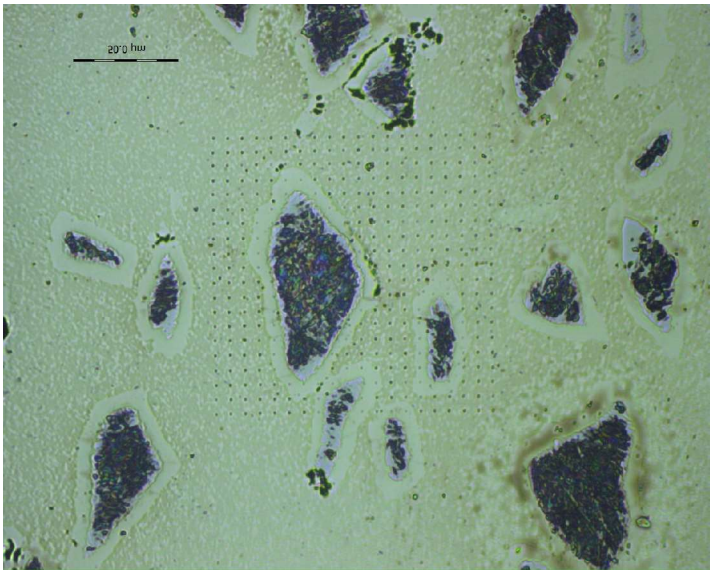


HIT



EIT

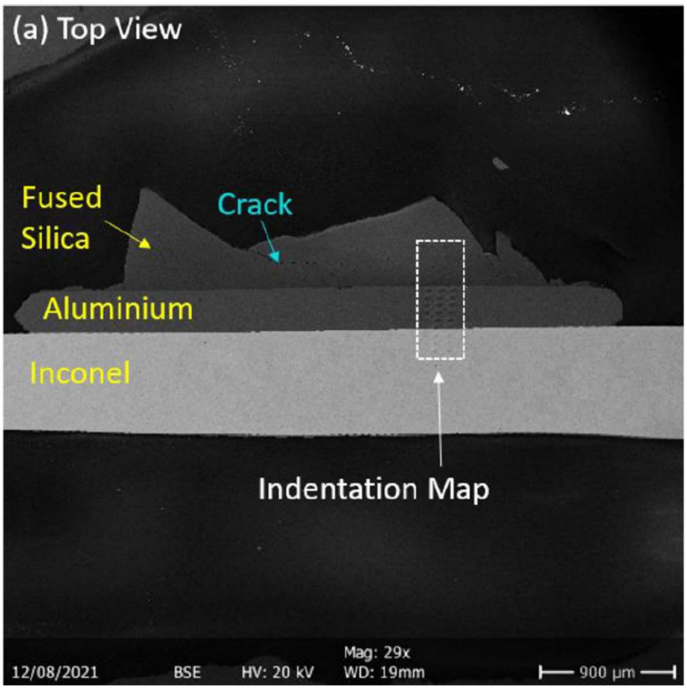
As compared to the optical micrograph which looks like this:



12) Note that the generated maps may not be oriented in the same direction as the optical video microscope images, because the camera optics will invert the image relative to the actual direction. This is solved by selecting the image in Word and clicking Format/Rotate/Flip Vertical

GROUP 2: EXPERIMENTAL PROCEDURE (Diffusion Bonded Joint)

Sample is a diffusion bonded joint between Inconel, aluminium and fused silica, see below a SEM image of the sample cross section.



1) Clean the "Diffusion Bonded Joint" sample by blowing off with compressed air.
2) Place Diffusion Bonded Joint sample under the ASA indentation head as shown in Fig. 10. Align the sample as shown in picture below, taking care to align the interfaces along the X axis.

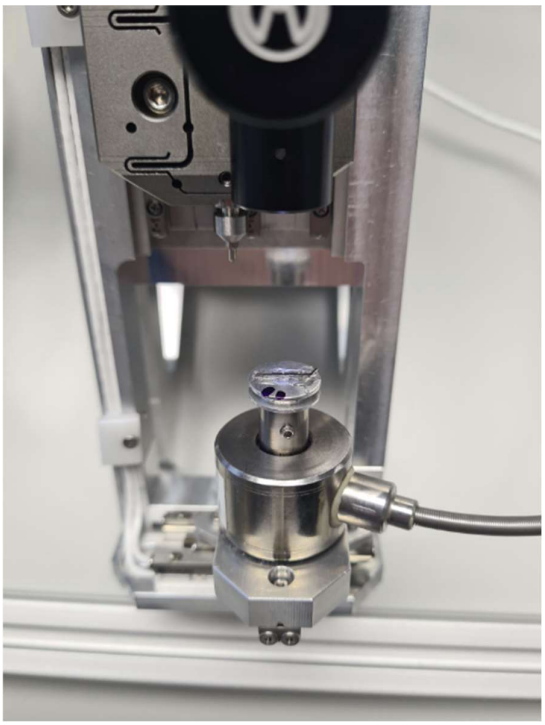
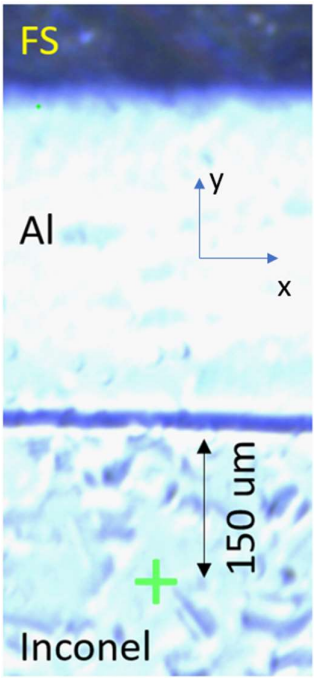
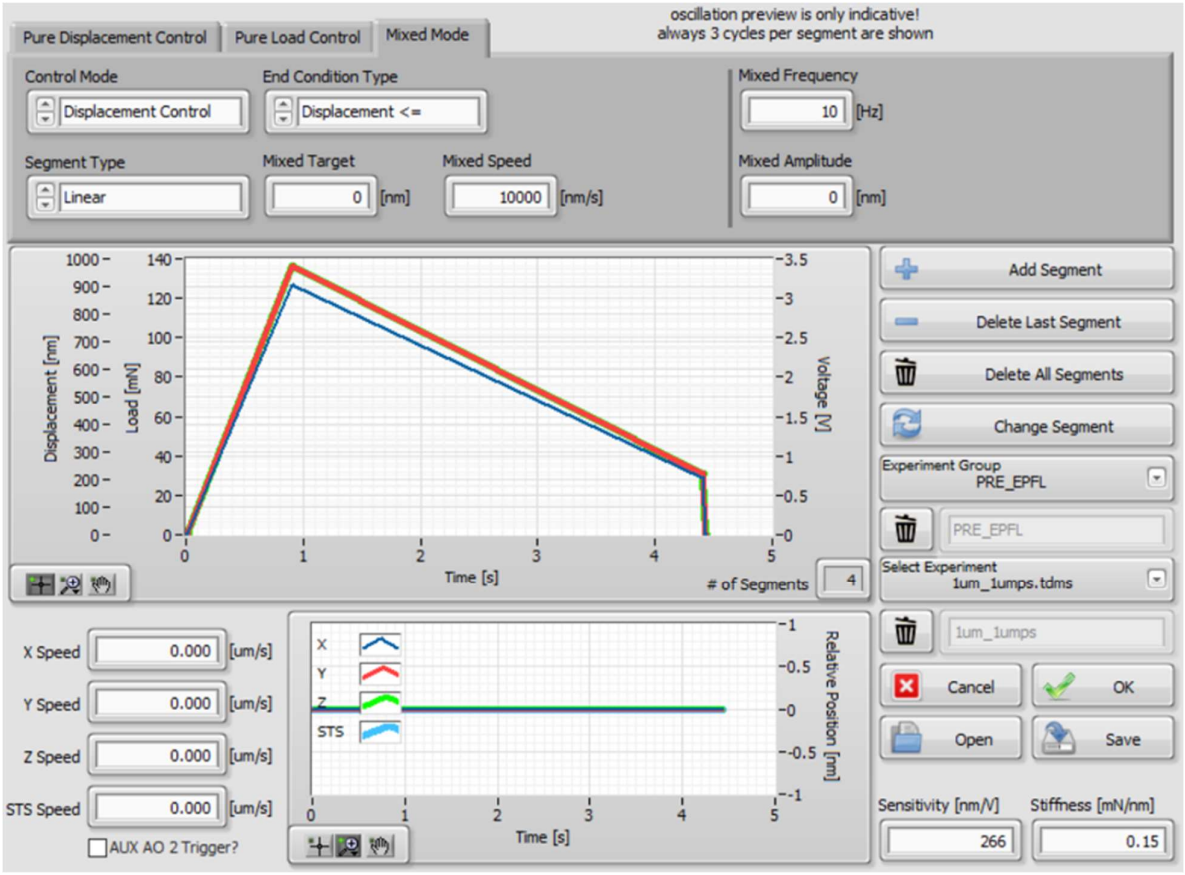


Fig. 10: Diffusion Bonded Joint sample mounted under the ASA in the microscope position

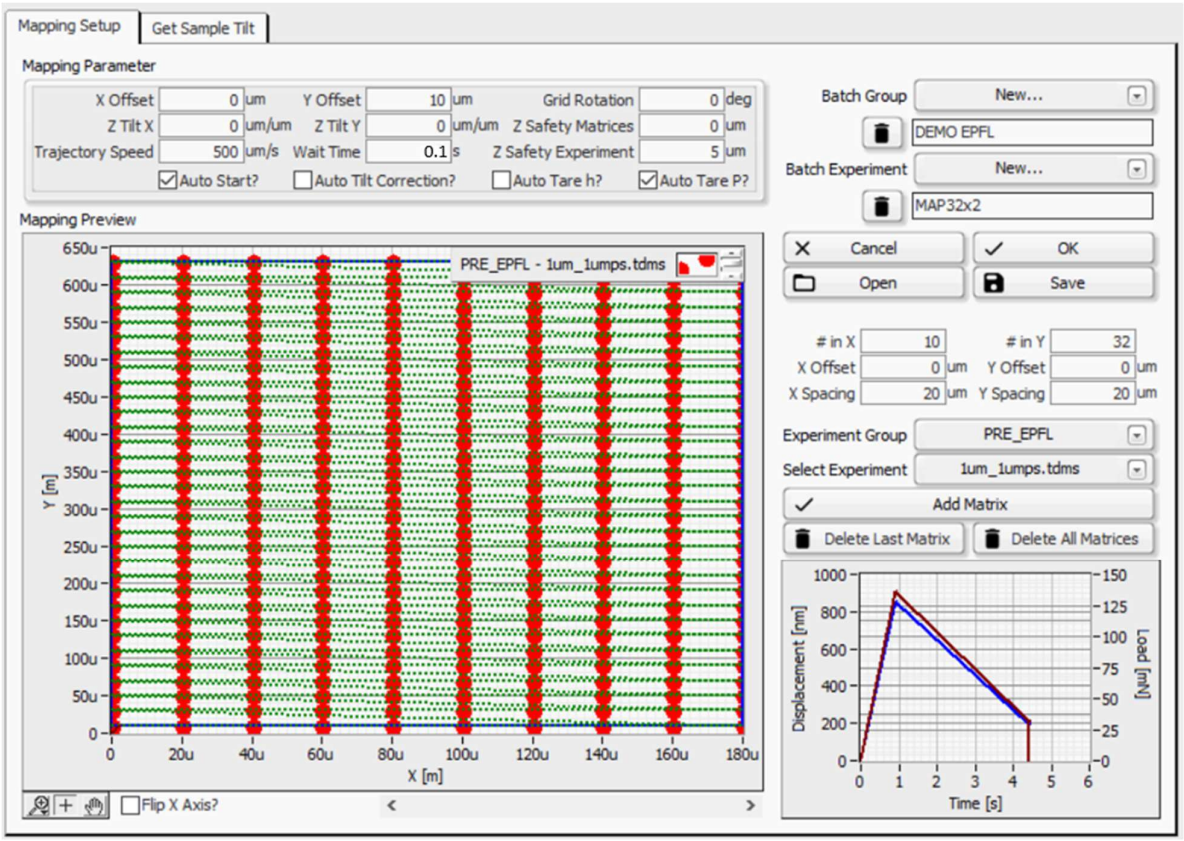
Choose an area for measurement which looks clean and where there are no cracks (displace in X and Y directions). Select a starting point in the Inconel approximately 150 µm away from the Inconel/Aluminium interface and save coordinates. The crosshair needs to be set in the precise position where the matrix will commence. The matrix size then needs to be calculated so that the materials of interest will be included in an appropriate manner.



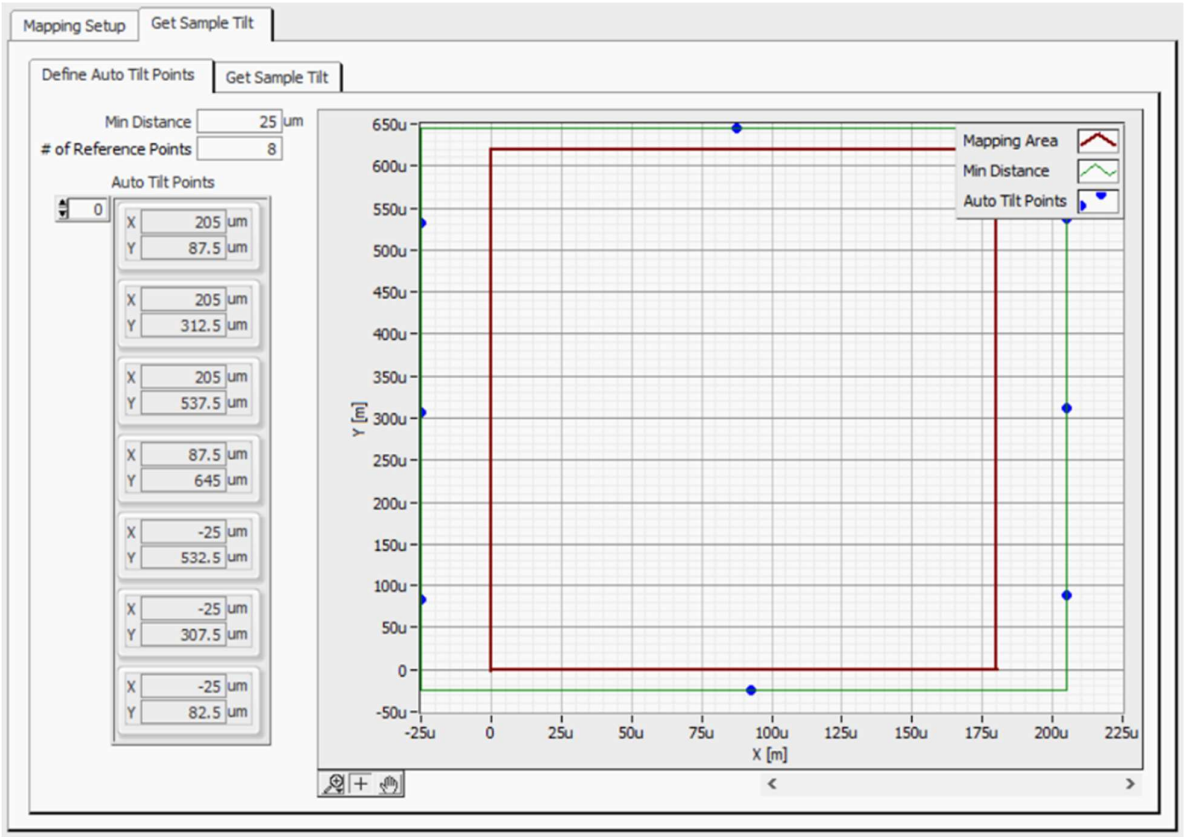
3) Move to these coordinates in tip position. After coarse approach using MCS2 controller, perform an auto-approach (load target 2 mN) to detect the sample surface. Select a target distance to surface of 10'000 nm.
4) Click on Standard Experiment → Setup
5) Create new experiment group and select a new experiment
6) In "mixed mode" create a new experimental profile, with 3 segments both in displacement control:
    a. speed of 1'000 nm/s, end condition type: "load >=" and mixed target: "1 mN"
    b. speed of 1'000 nm/s, end condition type: "displacement+" and mixed target: "1000 nm"
    c. speed of 200 nm/s, end condition type: "displacement-" and mixed target: "700 nm"
    d. speed of 10'000 nm/s, end condition type: "displacement <=" and mixed target 0 nm
7) Notice that the preview is imprecise, as the software cannot know in advance which depth will be reached at 3 mN, as this not only depends on the materials, but also on the initial distance between sample and tip.



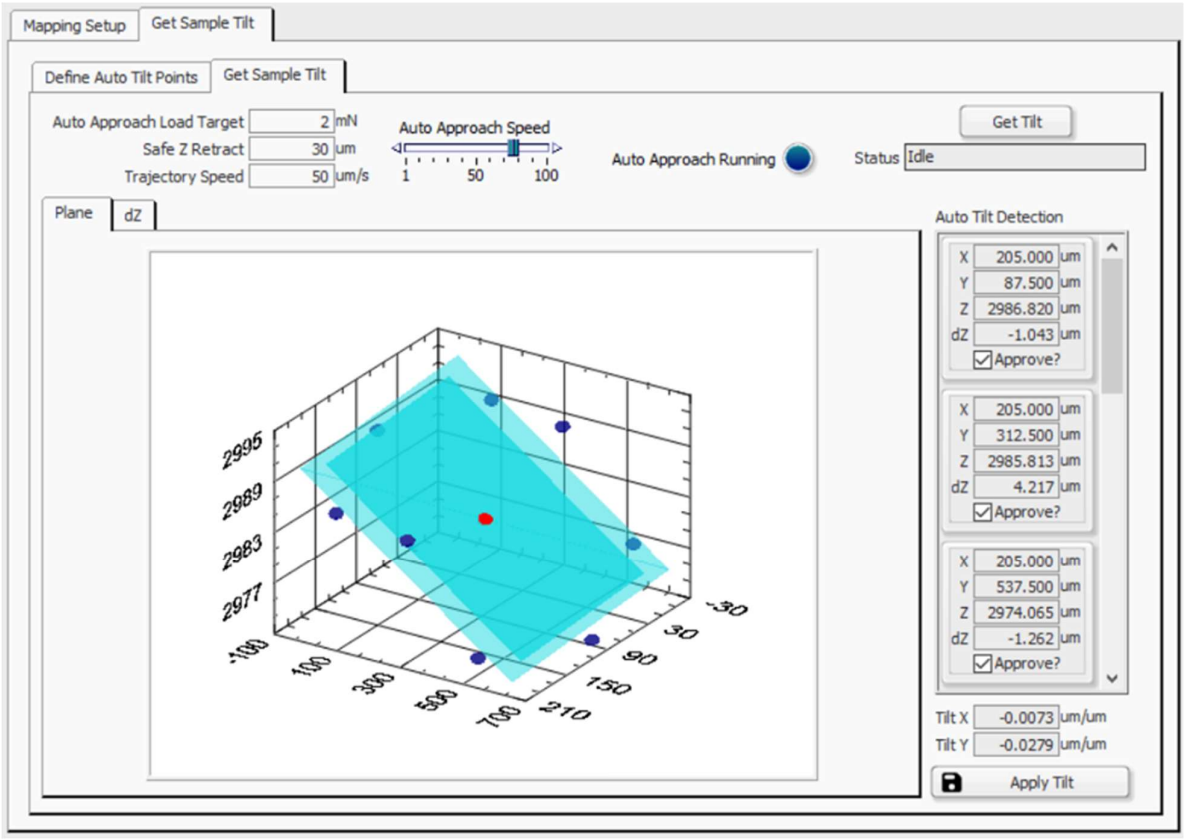
8) Click on Mapping → Setup
9) Create new batch group and select a new batch experiment
10) Create a 10 (in x) x 32 (in y) map with 20 µm spacing, 20 µm X offset, trajectory speed 500 µm/s, waiting time 0.1 s.



11) Click on get sample tilt tab.
12) Enter min distance 25 um and number of reference points 8.



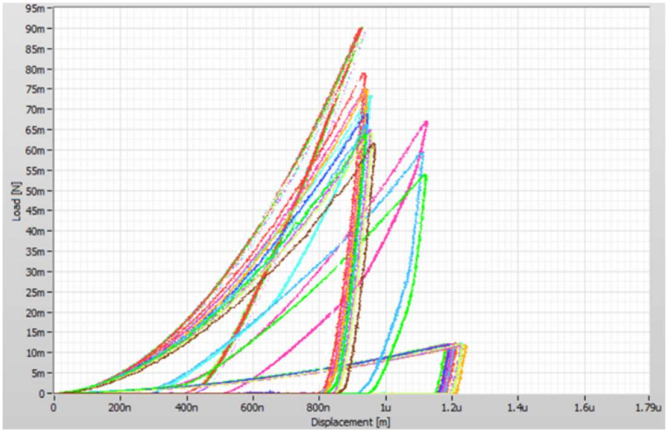
13) Click on Get Sample Tilt



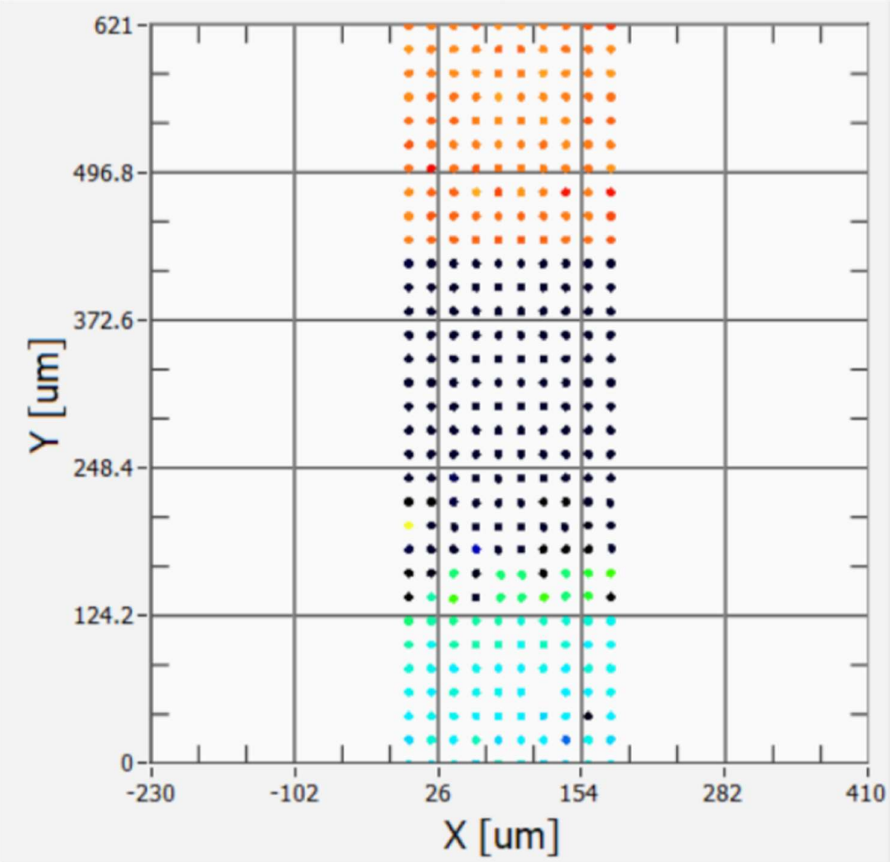
14) Enter load target of 1mN, safe Z retract 20 um, trajectory speed 50 um/s and auto approach speed approximately 80. Then click on Get Tilt, observe load/displacement live window and microscope image.
15) Click on “Apply Tilt”.
16) Select a new file name and select both sampling rate 1 kHz and PID frequency 2 kHz
17) Run batch

GROUP 2: DATA INTERPRETATION

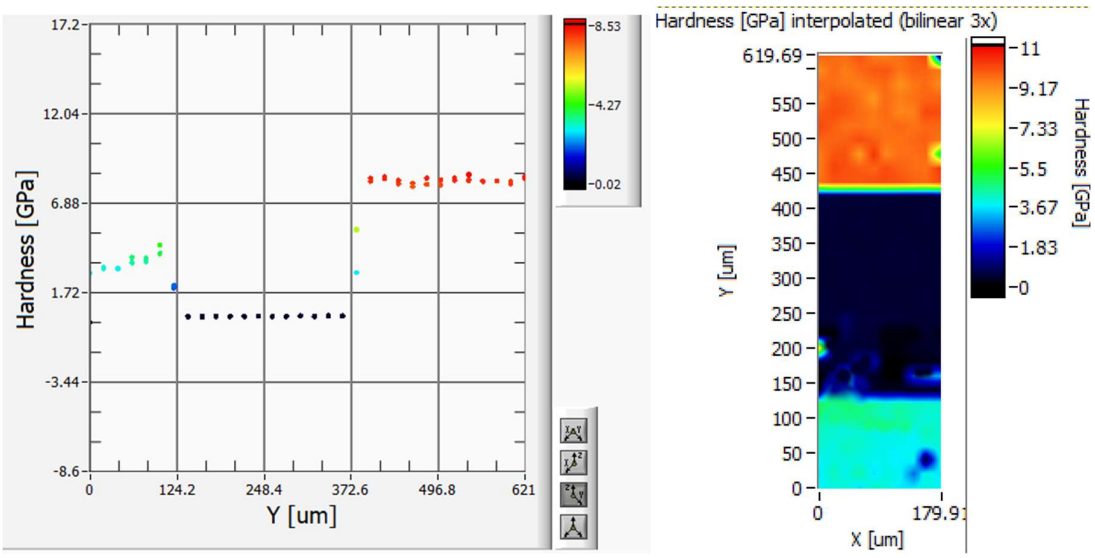
1) After completion of tests, download the map on the computer.
2) Check the contact points of some of the indentations (do not open all the indentation curves in AMMDA simultaneously) and apply desired corrections.



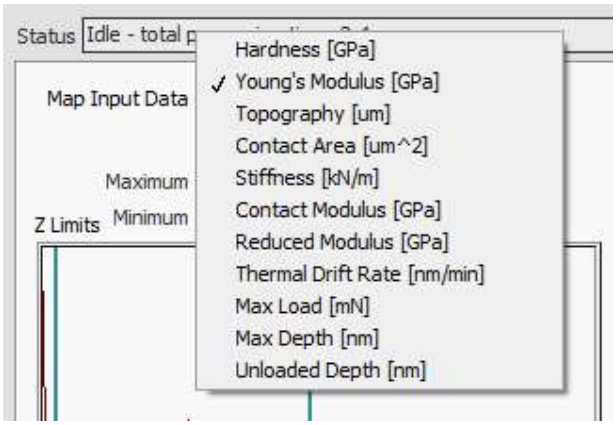
3) Once the optimal correction parameters are found, apply the same correction to all the curves: Click on stack processing → closed file, then select all the raw data files in the folder
4) Move to analyze corrected data tab and identify the unloading analysis parameters.
5) Click on stack processing → closed file, then selects all the corrected files in the subfolder named "corrected"
6) After that post processing has been completed, save the results.
7) Open the saved results in AMMDA 3D



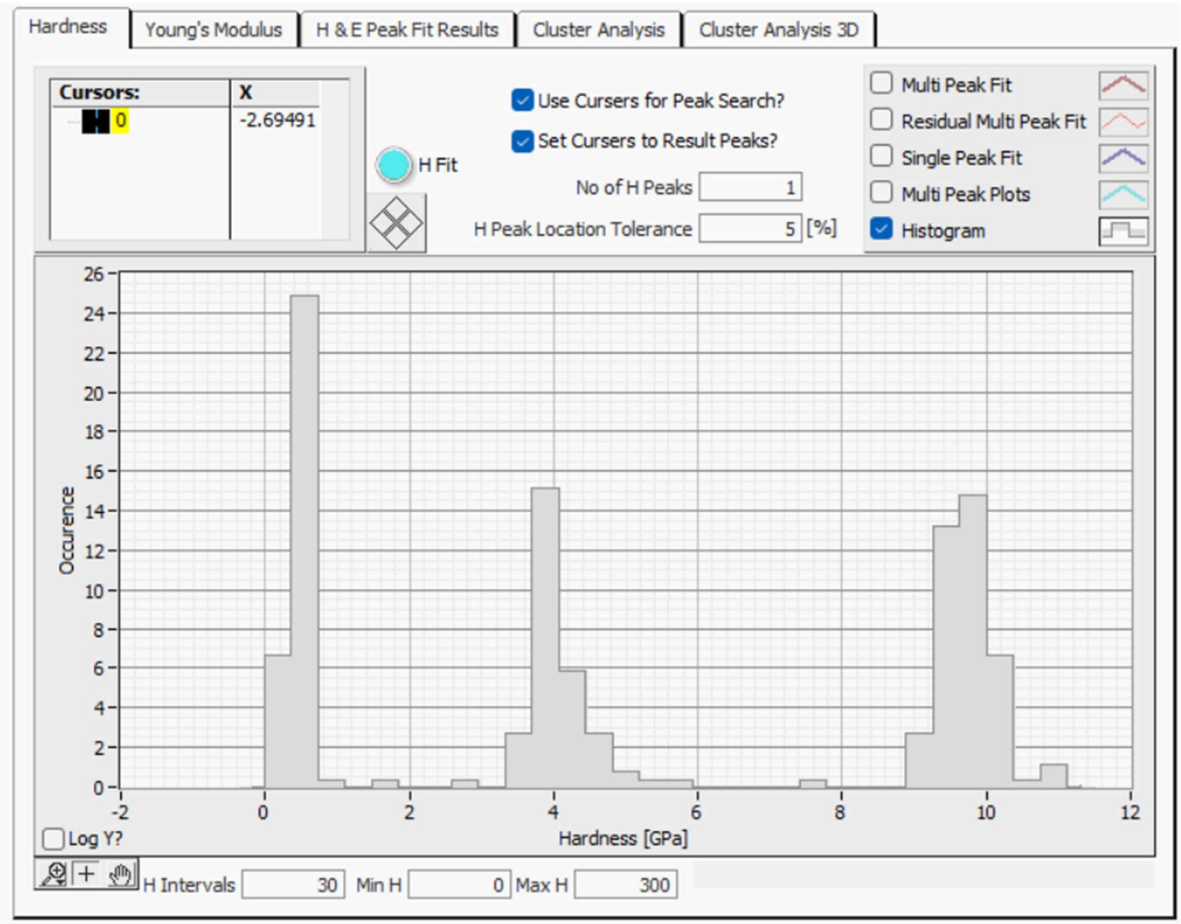
8) Display hardness map (Map Input Data)
9) Select the number of indentations in x and y and the spacing. Then apply grid.
10) In 3D →Scatter tab observe the scatter plot, each point accounts for one single indentation result.
11) Notice that it might be necessary to remove some outliers, so impose a lower/upper limit (then click on Apply Grid again)
12) In Map output image tab the individual points are interpolated. Try to optimize the display with interpolation factor, method, colors and change the view.



13) Try to display different properties, such as Young's modulus:



14) In cluster analysis tab → Hardness, plot the histogram and perform a multi peak analysis.



15) Try to modify the number of “H intervals”, and select appropriate min H and max H to filter out outliers.
16) Select 3 “No of H Peaks”. (Notice that depending on the dispersion of the data it might be required to enter 4 peaks, to exclude the “noise”).
17) Repeat for Young’s Modulus.
18) Then check results in H&E Peak Fit Results Tab

PRACTICAL 5 : Coating Experiments

Mechanical properties of coatings need to be assessed with the minimum of influence from the substrate. The depth of indentation must therefore be tailored to the coating thickness in an appropriate manner, depending whether the coating-substrate system is a hard-on-hard, hard-on-soft or soft-on-hard case. These experiments investigate two different coatings: (i) a hard DLC coating of thickness 2 µm on steel which is a typical industrial coating for cutting tools and falls into the hard-on-soft case; (ii) a polymer coating of thickness < 300 nm on a Si wafer substrate which falls into the soft-on-hard category.

GROUP 1: EXPERIMENTAL PROCEDURE (DLC-on-steel)

1) Clean the "DLC-on-steel" sample by blowing off with compressed air.
2) Place "DLC-on-steel" sample under the NHT measuring head as shown in Fig. 11

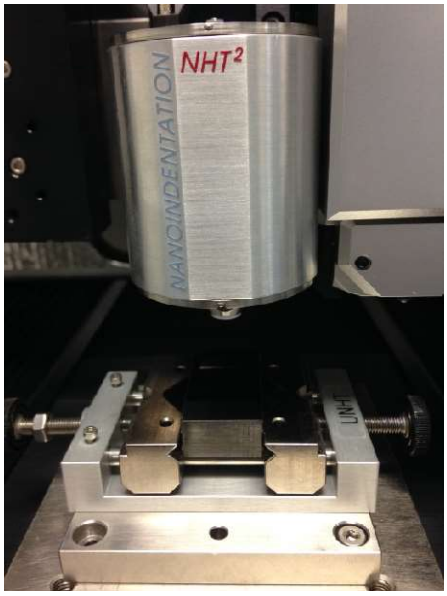
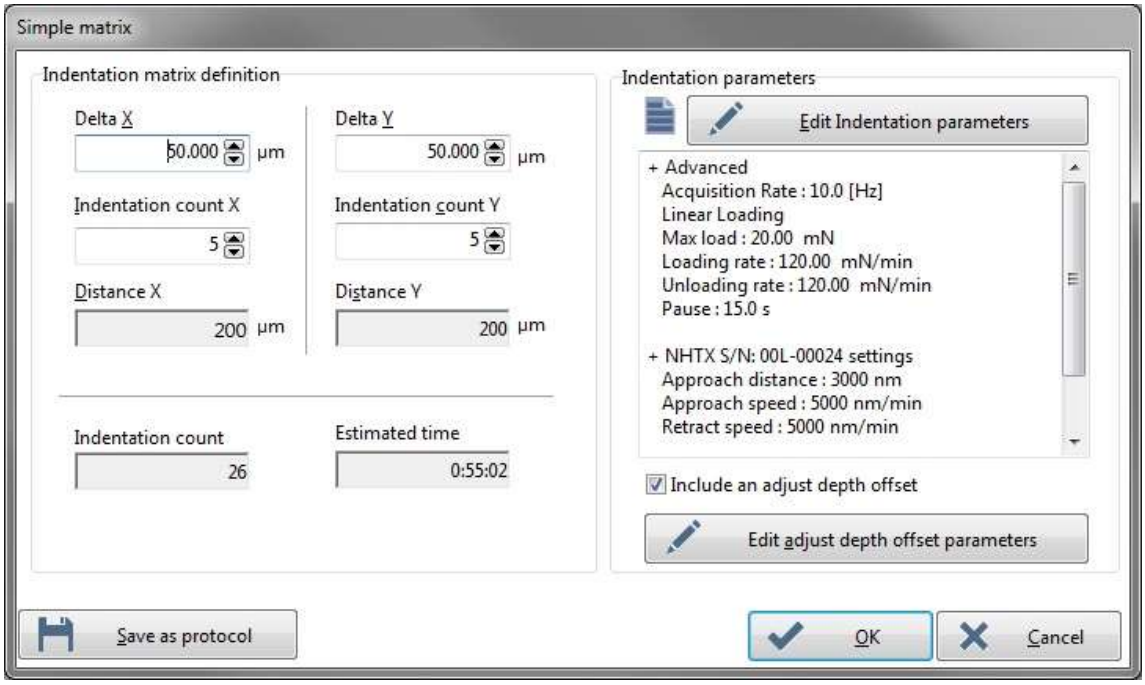
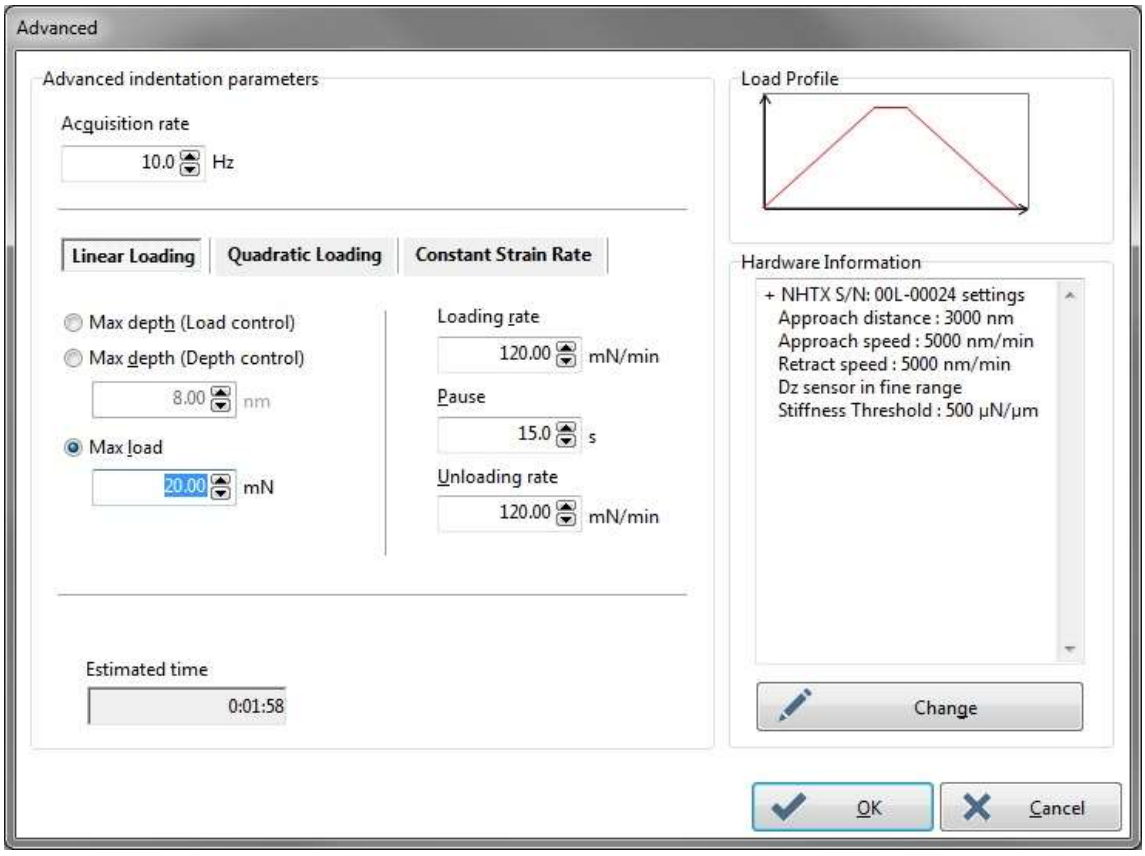


Fig. 11: Diamond-like Carbon (DLC) coating of thickness 2 µm on steel mounted under the NHT head

3) Create new file group, enter group name and enter Poisson's Ratio value of 0.2
4) Using Position Control, displace the sample under the optical video microscope, focus on the surface using the 5x objective followed by the 100x objective. Choose an area for measurement which looks clean and undamaged by polishing scratches (displace in X and Y directions by using keyboard arrows):
3) Program the software to make a "Simple Matrix" with a grid of 5 x 5 "Advanced" nanoindentations with an applied load of 20 mN, loading rate of 120 mN/min. and a pause at maximum load of 15 seconds. Set the Approach and Retract speeds to 5000 nm/min. Separate the indentations by 50 µm (use Delta X and Y value of 50 µm). Remember to add an Adjust Depth Offset:

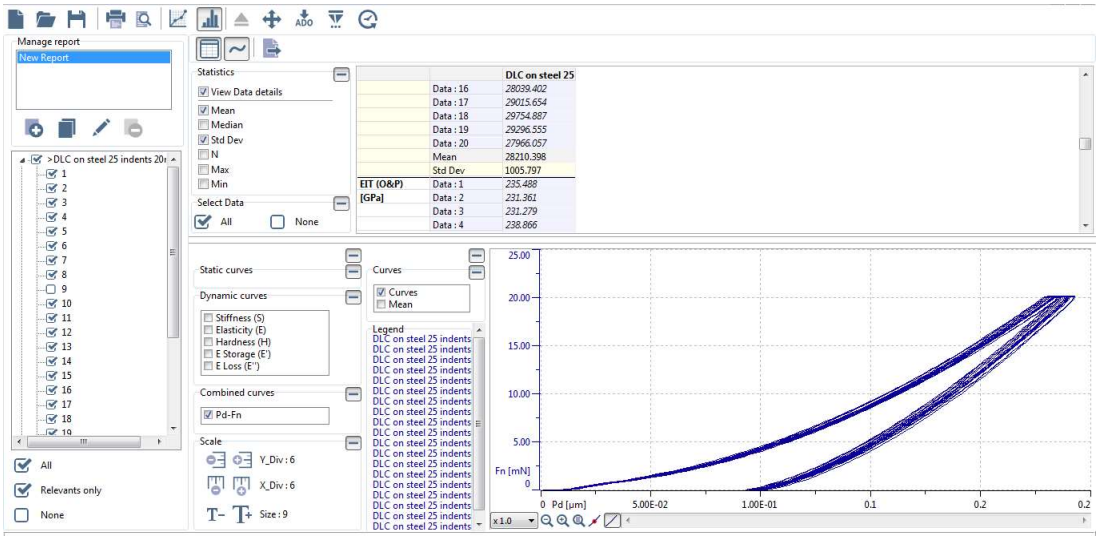




4) Note that these test parameters are based on industrial Quality Control (QC) techniques where faster loading/unloading rates are used to speed up the measurements, resulting in 10s for loading and 10s for unloading.

GROUP 1: DATA INTERPRETATION

1) After completion of tests, click “Yes” to go under the microscope and see if any of the residual indents are visible at highest magnification. If they are not, why is this…?
2) Check that the contact point of each indentation has been correctly chosen (right click on each indentation tab, select “Set contact point” and double-click on new position if necessary).
3) Click on the “Statistics” page and select the 25 measurements (Tick “All” in bottom left of screen)
4) Choose “Combined Curves” in the curve view in order to superimpose the 25 measurements on the same load-depth axes.
5) Create a results table by checking the “View data details”, “Mean” and “Std Dev” checkboxes. Select HIT (hardness) and EIT (elastic modulus) for the data outputs:



6) Note the HIT and EIT mean values and their standard deviation. If the standard deviation exceeds 5%, inspect the superimposed curves and look for any measurements which seem outside of the group. Remove such measurements from the group calculation by unchecking on the left of the screen. How does this affect the final value..?

QUESTIONS:

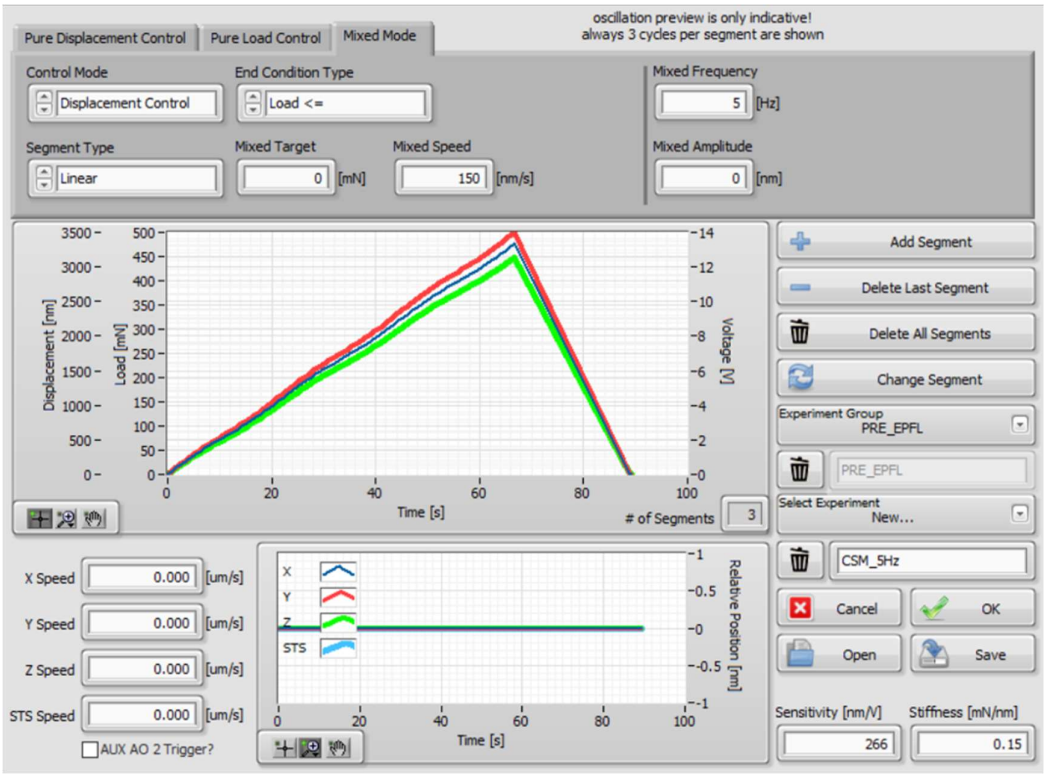
(i) Do you think that there are sufficient indents to make the measurement of coating properties meaningful..?
(ii) What recommendations might you make in an industrial environment to optimize this test?
(iii) What other characterization methods could help to understand this sample better?

GROUP 2: EXPERIMENTAL PROCEDURE (Al-on-Si)

1) Clean the “Al-on-Si” sample by blowing off with compressed air.
2) Place “Al-on-Si” sample under the ASA indentation head as shown in Fig. 12

Fig. 12: Al-on-Si sample mounted under the ASA in microscope position and indentation position

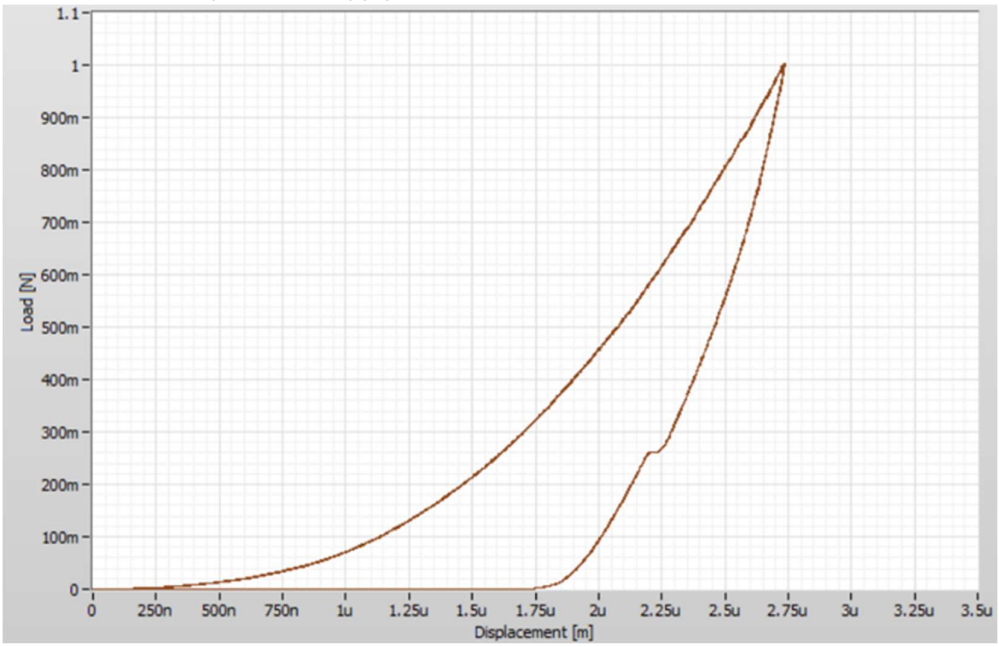
3) After coarse approach using MCS2 controller, perform an auto-approach (load target 1 mN) to detect the sample surface. Select a target distance to surface of 500 nm.
4) Click on Standard Experiment → Setup
5) Create new experiment group and select a new experiment
6) In “mixed mode” create a new experimental profile, with 3 segments both in displacement control:
    a. Speed 50 nm/s, end condition type: “load >=” and mixed target: “1 mN”
    b. Speed 50 nm/s, end condition type: “load >=” and mixed target: “1000 mN”. Mixed Frequency 5 Hz and amplitude 15 nm.
    c. speed 150 nm/s, end condition type: “displacement <=” and mixed target 0 nm.



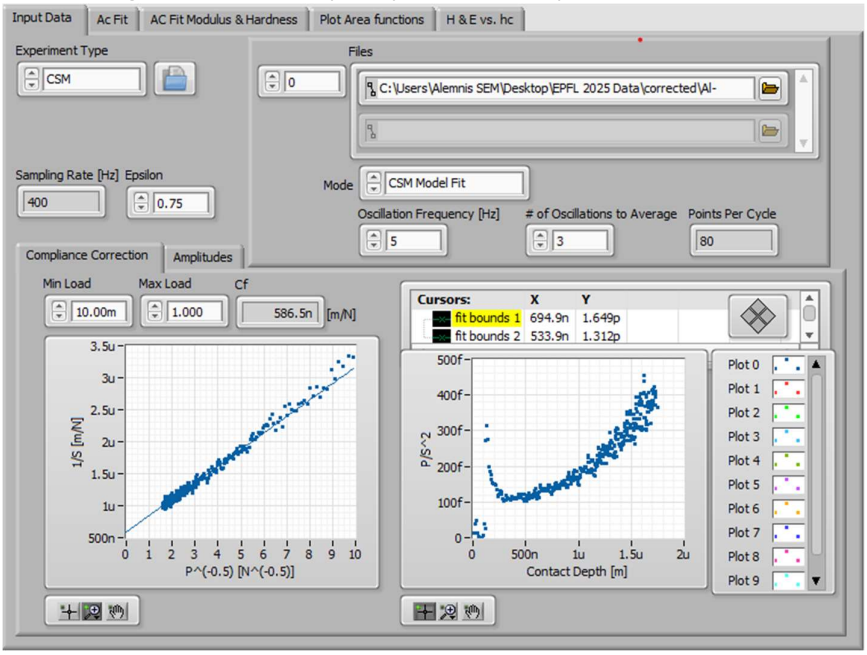
7) Select a new file name and select both sampling rate 400 Hz and PID frequency 400 Hz
8) Run

GROUP 2: DATA INTERPRETATION

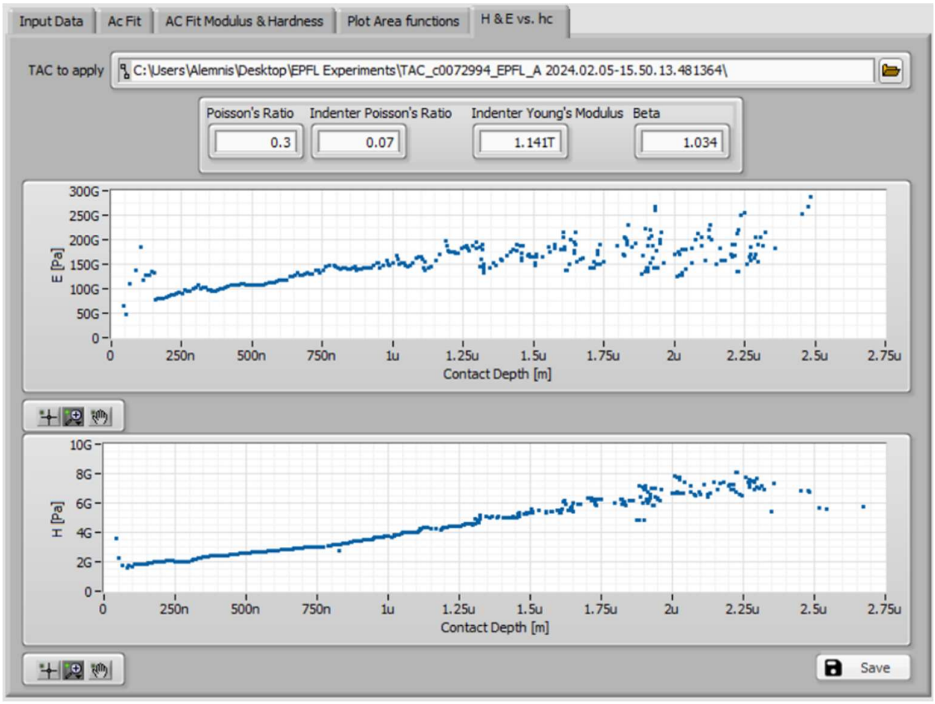
1) After completion of test, download the data on the computer.
2) Check the contact points and apply desired corrections.



3) Once the optimal correction parameters have been found, save the corrected data.
4) Click on analyze corrected data tab → open tac and identify the unloading analysis parameters.
5) Select the right oscillation frequency (5 Hz) and import the corrected data file.



6) Move to "H & E vs hc" Tab
7) Select the tip area calibration (TAC to apply)



QUESTIONS:

(i) How do you explain the evolution of H and E data with depth?
(ii) Where is the transition from coating to coating/substrate properties?
(iii) Why do you think there is more scatter at high depths?