ALOISA User's Guide

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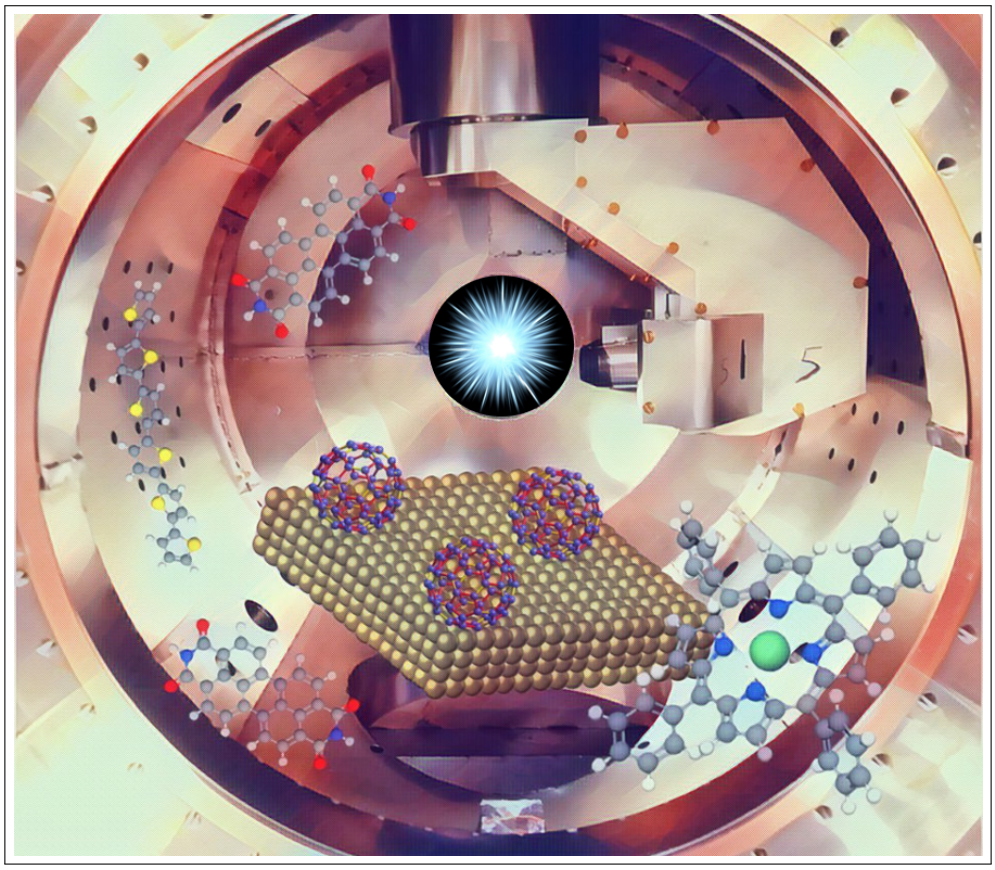
May, 2022



ISTITUTO OFFICINA DEI MATERIALI

Elettra Sincrotrone Trieste

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1 INTRODUCTION

This technical report is intended as a guideline for users of the CNR-IOM beamline ALOISA, located in the Elettra Synchrotron in Trieste. State-of-the-art beamline instrumentation, hardwares and softwares are described in this report. The procedures for running standard experiments on the beamline (surface preparation, XPS and NEXAFS data acquisition, etc...) are reported in detail. The User is always trained by the beamline staff before operating the beamline, this technical manual helps the users to retrieve the operating instruction before and during their beamtimes; always ask to the the beamline staff before performing any procedure, and only follow their instructions.

2 GENERAL DESCRIPTION OF THE ALOISA EXPERIMENTAL CHAMBER

ALOISA is a multipurpose beamline dedicated to surface science studies. The ultra-wide energy range (130-1500 eV) of the beamline and the complete set of detectors in the end-station, allow the users to combine in-situ both structural and chemical investigation techniques, such as: X-ray Photoemission Spectroscopy (XPS), Photoelectron Diffraction (PED), Resonant X-ray Photoemission Spectroscopy (RESPES) and Near Edge X-ray Absorption Fine Spectroscopy (NEXAFS). The experimental chamber hosts an electron energy analysers (66 mm mean radius, with 2 degrees angular resolution and two angular degrees of freedom) and a channeltron detector for electron partial yield measurements. The sample is mounted on a high resolution manipulator with six degrees of freedom. The preparation chamber offers four deposition cells in a liquid Nitrogen cooled flange (Knudsen cells and electron bombardment cells), a RHEED system, a sputter gun, and a fast entry system for rapid change of the sample. The combination of GIXD and photoelectron diffraction allows fast and reliable structural determination of in situ grown films. Moreover the growth can be monitored in real time by RHEED. The very wide range of geometrical configurations of the sample manipulator and the detection system allows Photoelectron Diffraction measurements in near node geometry. A schematic representation of the Aloisa end station is shown in fig.1.

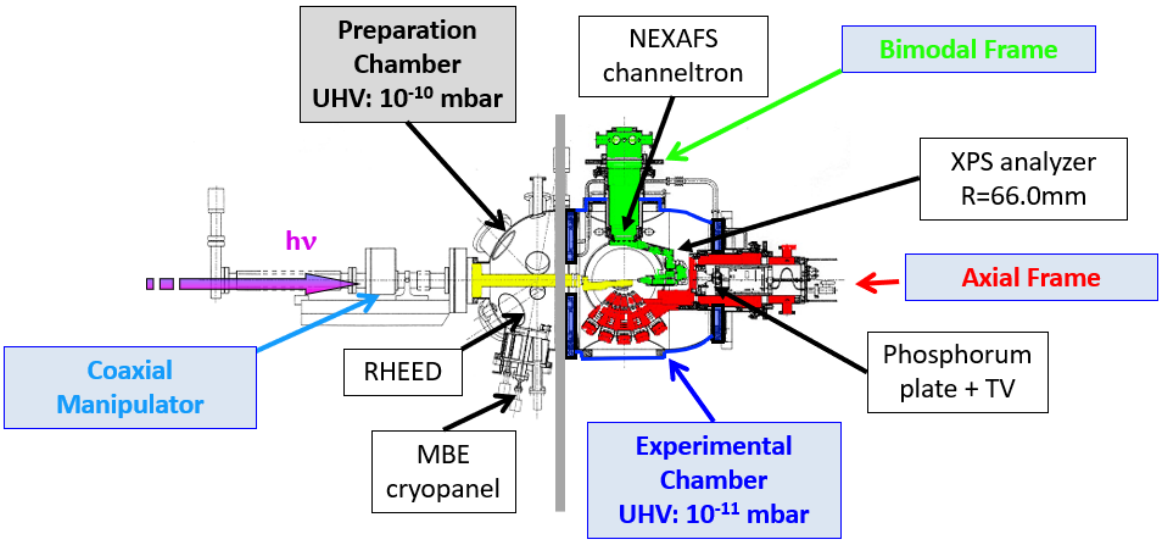

Figure 1: Schematic representation of the ALOISA Experimental Chamber.

The main instrumentation currently available at Aloisa can be summarized as follow:

- **AXIAL FRAME**
  - 1 phosphorum plate with CCD camera for 2D reflectivity measurements and sample alignment.
- **BIMODAL FRAME**
  - 1 electron analyzer (66 mm mean radius) equipped with a 2D-delay line detector for XPS and PED;
  - 1 channeltron multiplier for partial electron yield measurements (XAS).

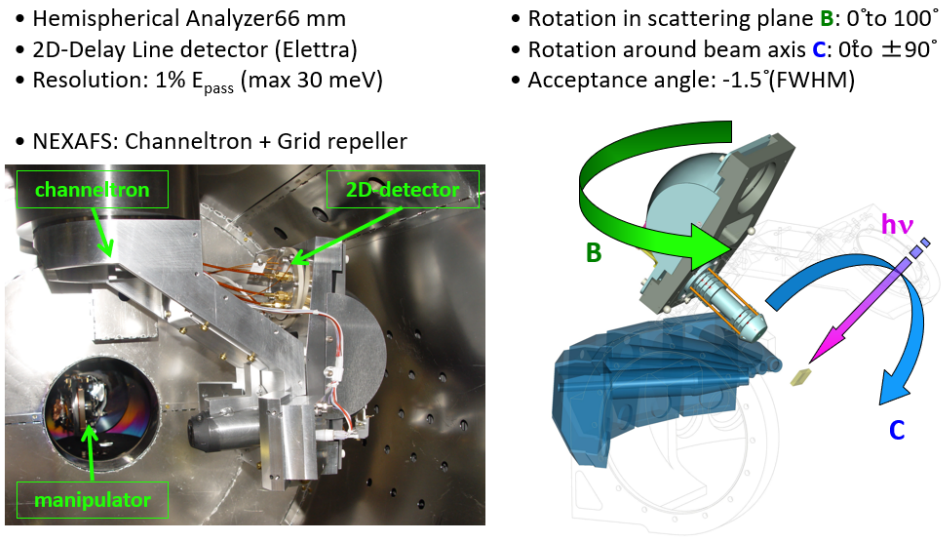

Figure 2: Detectors in the Bimodal Frame.

- **PREPARATION CHAMBER**
  - Sample transfer system with fast entry-lock;
  - RHEED apparatus;
  - MBE cryopanel with 4 slots for Knudsen and electron bombardment evaporators, 2 quartz microbalances, shutters;
  - ion gun for Ar sputtering;
  - gas line.
- **MANIPULATOR**
  - six degrees of freedom (0.004° accuracy for azimut and incidence angles): x, y, z translation, polar, tilt and azimuthal rotation;
  - main rotation arm coaxial to the photon beam for free orientation of the surface with respect to the photon polarization at constant grazing angle;
  - temperature range 150-1100 K (Only flash at max temperature);
  - Liquid N_2 cooling (lower limit $\sim 140K - 150K$).

The experimental chamber, the manipulator, the axial and bimodal frames can rotate with respect to different rotation axes (see fig.3). The rotation extensions are limited in order to avoid mechanical crashes or instrumentation damages, as reported in tab.1.

Element	Movement Axis	Allowed Extension
Experimental Chamber	SR Beam	-90° ÷ 90°
Axial Frame	SR Beam	fixed at -15°
Bimodal Frame	SR Beam	0° ÷ 100°
Sample Holder (manipulator)	SR Beam (polar angle, R1)	-90° ÷ 185°
Sample Holder (manipulator)	surface normal (azimuthal angle, R2)	-90° ÷ 90°
Sample Holder (manipulator)	grazing angle (tilt, R3)	-2° ÷ 15°
Sample Holder (manipulator)	Y translation (Ym)	-5mm ÷ 5mm
Sample Holder (manipulator)	Z translation (Zm)	-5mm ÷ 5mm
Sample Holder (manipulator)	X translation (Xm)	5mm ÷ 379mm

Table 1: ALOISA Chamber/Manipulator positions.

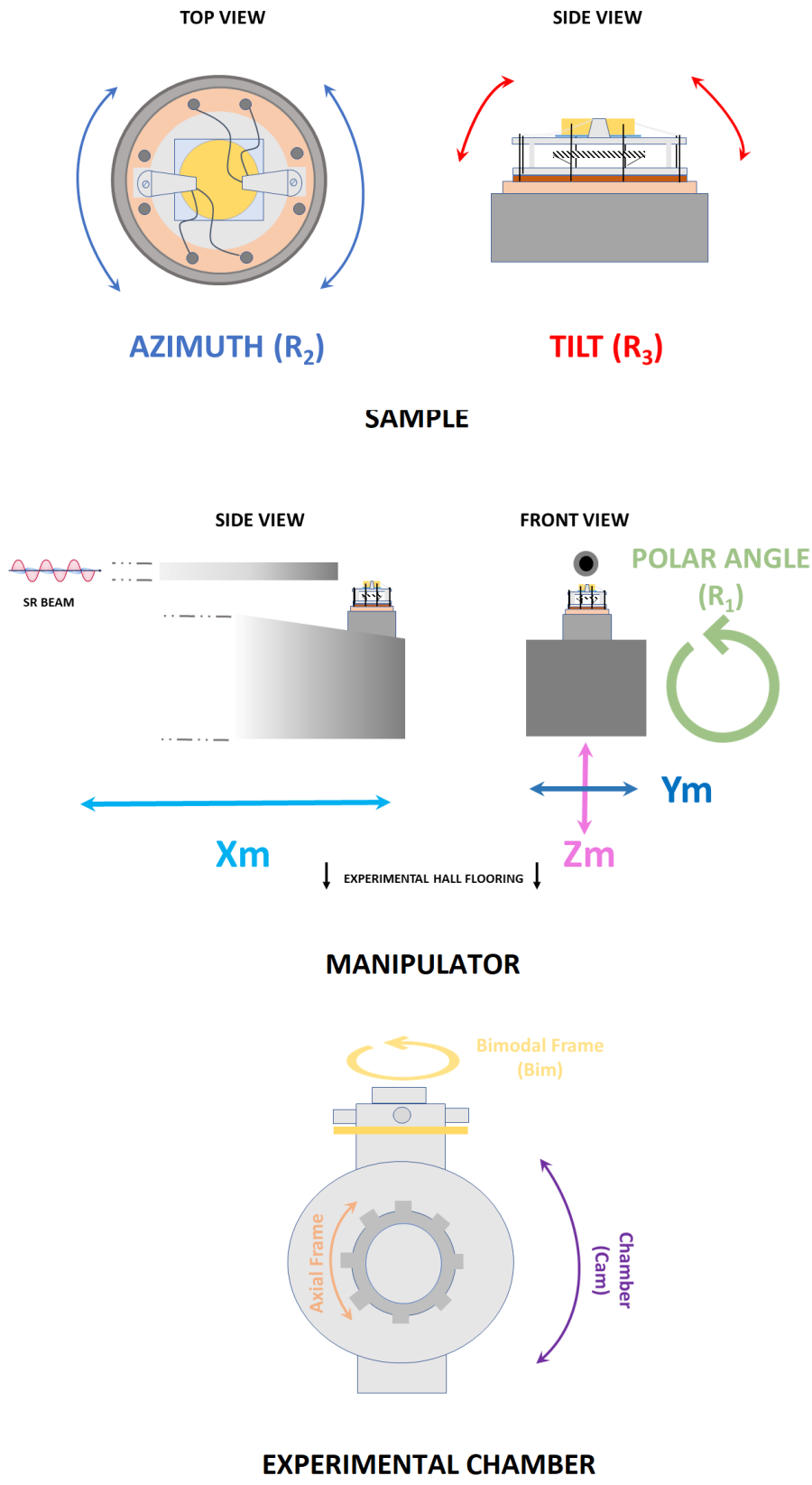

Figure 3: The experimental chamber, the manipulator and the axial and bimodal frames movements.

3 BEAMLINE CONTROL SYSTEM

Next to the acquisition computer (the two monitors one), on the PC available for data analysis, the beamline control system software is installed (Main_BCS.vi, see fig.4). Through the BCS you can open / close the valves of the beamline, in particular the shutter and stopper, that automatically close after a beamdump, i.e. the loss of the synchrotron radiation. A red color means that the valve is open,a green color means that the valve is closed. In order to do this operation, follow this procedure:

1. Click on the green diamond on top of the valve you want to close. The "Current Object" name on the blue window should show the selected valve name.
2. select the desired operation through the drop down menu ((0) Close / (1) Open).
3. Press "Submit"

The Shutter valve is a copper device installed in the beamline frontend utilized to stop the extraction of the synchrotron radiation along the beamline, and is usually cooled to bear the thermal load of the beam.
The Stopper valve is a tungsten device utilized to stop the extraction of the bremsstrahlung radiation.
When closing the beamline, you must close the shutter first, and than the stopper, viceversa when you are opening the beamline.

In order to open the beamline follow this procedure:

1. Click on the green diamond on top of the Stopper valve. The "Current Object" name on the blue window should show the word "stopper" in the command string.
2. Select (1) Open in the drop down menu.
3. Press "Submit"
4. Click on the green diamond on top of the Shutter valve. The "Current Object" name on the blue window should show the word "shutter" in the command string.
5. Select (1) Open in the drop down menu.
6. Press "Submit"

In order to close the beamline follow this procedure:

1. Click on the green diamond on top of the Shutter valve. The "Current Object" name on the blue window should show the word "shutter" in the command string.
2. Select (0) Close in the drop down menu.
3. Press "Submit"
4. Click on the green diamond on top of the Stopper valve. The "Current Object" name on the blue window should show the word "stopper" in the command string.
5. Select (0) Close in the drop down menu.
6. Press "Submit"

Figure 4: Beamline Control System software. In this pictures all the valves are open.

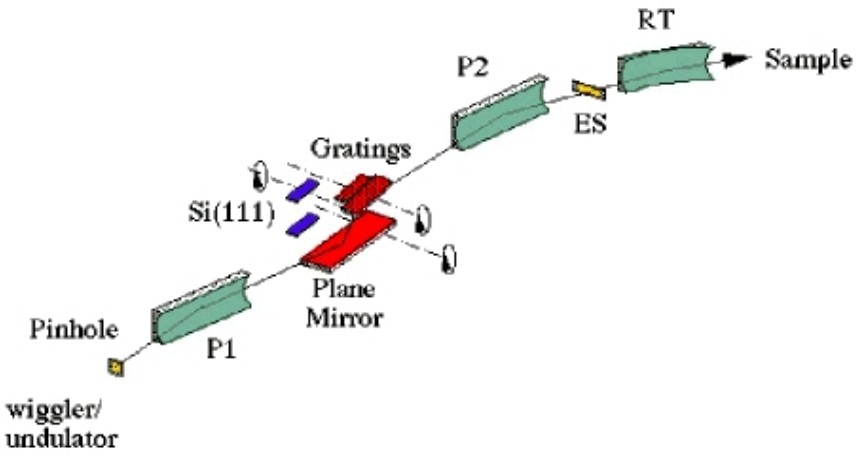

Figure 5: Schematic representation of the ALOISA Beamline.

4 ACQUISITION SOFTWARE

The mechanical movements of both the chamber and the manipulator, as well as all the data acquisition subroutines, are managed through the ALOISA acquisition software, a LabVIEW based user interface designed to control the main aspects of the experiment, such as the geometrical relations between sample and analyzer, the data acquisition procedures and beamline parameters. Next to the Aloisa experimental chamber a work station equipped with two desktop computers is available for the remote control of the experimental station and data analysis. **Always use the "EXIT" buttons in the various panels to close the program windows,**

never use the red "X" close button of Windows or the red "abort" button of LabVIEW to close the software panels.

4.1 Main Panel

In fig.6 is shown the main panel of the ALOISA acquisition software. In the following text an overview is given of the main functions available on the software main panel.

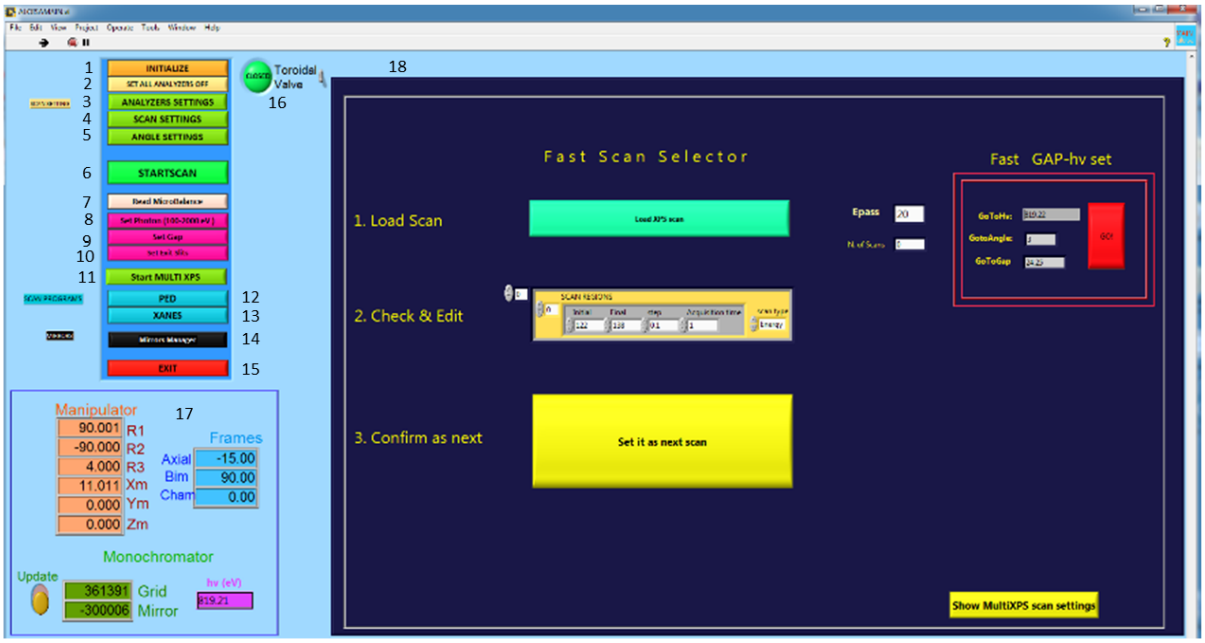

Figure 6: Main Panel of the ALOISA Acquisition Software.

1. **INITIALIZE Button**: click on this button opens the "Initialize" panel, a subroutine that initializes the TCP connections and the manipulator encoders.
2. **SET ALL ANALYZERS OFF Button**: click on this button switches off all the hemispherical analyser high voltage power supplies, including the lens system and the 2D detector.
3. **ANALYZERS SETTINGS Button**: click on this button opens the "Analyzers Settings" panel, a subroutine in which you can choose which instruments (channels) will be activated (recorded) for the acquisitions.
4. **SCAN SETTINGS Button**: click on this button opens the "Scan Settings" panel, in which you can choose the ranges, type and number of scans for the next acquisition.
5. **ANGLE SETTINGS Button**:click on this button opens the "Angle Settings" panel, the subroutine that controls the mechanical movements of the chamber, the axial and bimodal frames, as well as the manipulator degrees of freedom. Trough this panel you can set the x, y, z values of the sample holder, together with the polar (R1), azimuthal (R2) and tilt (R3) angle. You can also move the bimodal and axial frames, and the experimental chamber angles.
6. **START SCAN Button**: Click on this button starts the pre-set scan.
7. **READ MICROBALANCE Button**: click on this button opens the "Read Microbalance" panel, a subroutine used for reading the two microbalances mounted on the MBE cryopanel (Q1 and Q2) during depositions.
8. **SET PHOTON ENERGY Button**: click on this button opens the "Set Photon Energy" panel, a subroutine in which you can set the beam photon energy.
9. **SET GAP Button**: click on this button opens the "Set Gap" panel, a subroutine in which you can set the undulator gap value.

10. **SET EXIT SLITS Button**: click on this button opens the "Set Exit slits" panel, a subroutine in which you can set the beamline exit slits value.
11. **START MULTI XPS Button**: click on this button opens the "Start Multi XPS" panel.
12. **PED Button**: click on this button opens the "PED" panel, a subroutine used for Photoelectron Diffraction measurements.
13. **XANES Button**: click on this button opens the "XANES" panel, a subroutine used for NEXAFS measurements.
14. **MIRROR MANAGER Button**: click on this button opens the "Mirror Manager" panel, a subroutine through which you can control the beamline's mirrors, namely P1, P2 and the Toroidal mirror.
15. **EXIT Button**: click on this button quits the ALOISA Acquisitions software.
16. **PARAMETERS READ AREA**: in this area you can read the current values of different parameters of the manipulator, the chamber frames and the monochromator.
17. **TOROIDAL VALVE Button**: click on this button opens or closes the toroidal valve, the last pneumatic valve between the beamline and the experimental chamber. **GREEN = CLOSED ; RED = OPEN**.
18. **FAST SCAN SELECT AREA**: in this area you can find the fast procedure to load and set a previously recorded scan, as well as a fast photon energy/gap setting button.

4.2 Software remote connections inizialization: Initialize Panel

When you restart the ALOISA Acquisition Software, you should re-initialize the remote connections with the instrumentation. This procedure is not always necessary, but is a good practice to do it every time the software is restarted. The only two important connections to be initialized are the TCP connections and the remote connection with the manipulator (see fig.7. To initialize the Manipulator remote connection select "manipulator" with the scroll bar, press "Initialize". In the new panel (see fig.8) press "Init Manipulator" and than "Read Encoders". A new panel will open (see fig.9), here press "Read Encoders and cal. positions". In the same panel you can also add a relative offset to the R_2 or R_3 values (set the desired offset and press "ADD"). Once you finished the Initialize operation, press "Exit and Go Back" in all the opened panels to go back to the main acquisition software,

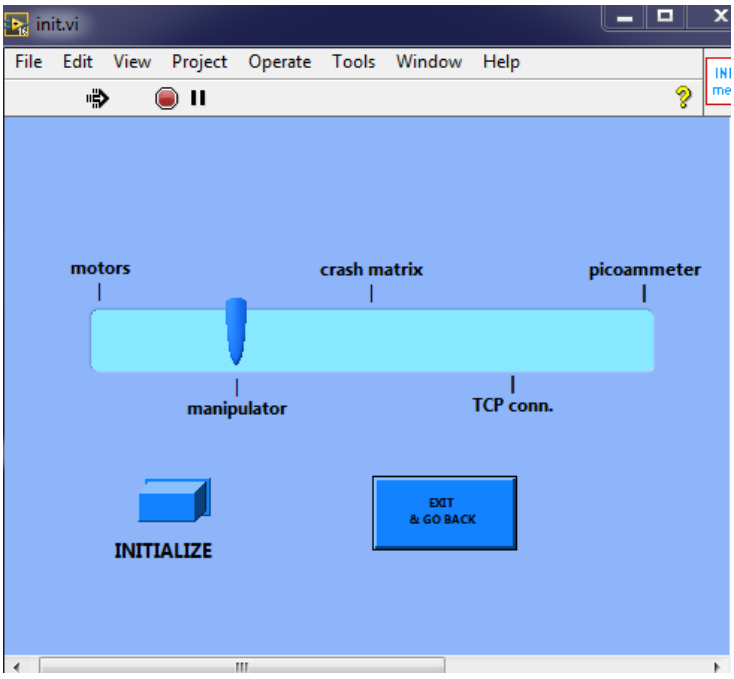

Figure 7: Initialize Panel of the ALOISA Acquisition Software.

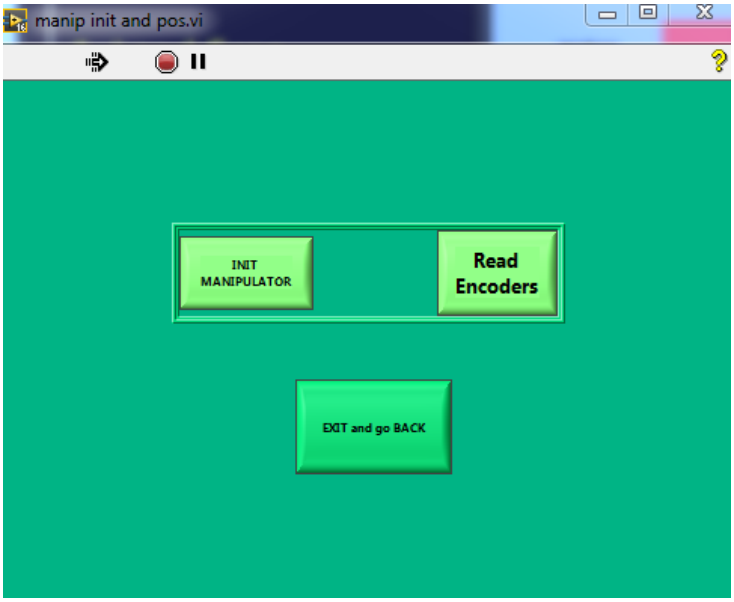

Figure 8: Manipulator Initialize Panel of the ALOISA Acquisition Software.

Figure 9: Manipulator's Encoders Initialize Panel of the ALOISA Acquisition Software.

4.3 Activating/Deactivating the data acquisition instrumentation: Analyzers Setting

The Analyzers Settings Panel allows the user to select which instruments are currently activated, i.e. the channels that are recorded during an acquisition. As shown in fig.10, you can select seven different channels, including the current read on the Toroidal Mirror (channel 3, "I0"), the channeltron signal (channel 4, "NEXAFS"), the 2D detector signal (channel 6, "MCP"), and the temperature of the sample read by the thermocouples through the Oxford instrument (channel 7, "T sample"). Moreover, you can activate three other instruments through a GPIB connection, namely the KEITHLEY 486 (channel K486), the KEITHLEY 196 (channel K196) and the KEITHLEY 6512 (channel K6512) instruments. You can enable a channel simply by clicking on the circular button next to the instrument name. Once enabled, the instrument button turns from green to red. When you finish the selection procedure click on the "EXIT and GO BACK" button. If you select the "MCP" channel, a subwindow is activated, where you must enter a pass energy ("Epass") and a kinetic energy ("KinEne") value. These are the values that are set as soon as you enter in the "Angle Settings" panel, thus you MUST enter physical values, in order to avoid instruments technical problems or possible damages. As an example, you can

use 20eV as Pass Energy, and 110eV as kinetic energy.

Figure 10: Analyzers Settings Panel of the ALOISA Acquisition Software.

4.4 Setting the scan parameters: Scan Settings

Through the Scan Settings Panel the user can sets the scan range, the scan step, the acquisition time of each scan point, the number of scan regions, the scan type and the number of scans. Fig.11 shows the Scan Settings Panel, from left to right the following fields can be filled: Initial value (first point of the scan region), Final Value (last point of the scan region), Step, Acquisition time (acquisition time in seconds for each scan point), Scan Type (Energy, Angle, Manipulator, etc.). In the figure the red square highlights the scroll arrows for the Initial Step value (and in analogy also for the other scan parameters), the blue square highlights the scroll arrows for multiple scan regions, the green square highlights the scroll arrows for nested scan, where you can have different scan types executed simultaneously. For example, you can have an energy scan within an azimuthal angle (R2) scan, in which for each R2 point, an energy scan is performed. The black square highlights the "Number of scans" parameters, in wihich you choose how many times your scan will be repeated. In the panel there are other scan options, including the Constant Ionic State (CIS) Mode button, the "GAP Scan ON" button (for photon energy scan in which also a gap scan is needed), the "chamber precession correction" button, and the backlash correction parameters at the bottom of the panel. Once you finish the scan setting procedure press the "EXIT and GO BACK" button. The scan that you set is stored in memory and it is executed once you press the "START SCAN" button in the software main panel.

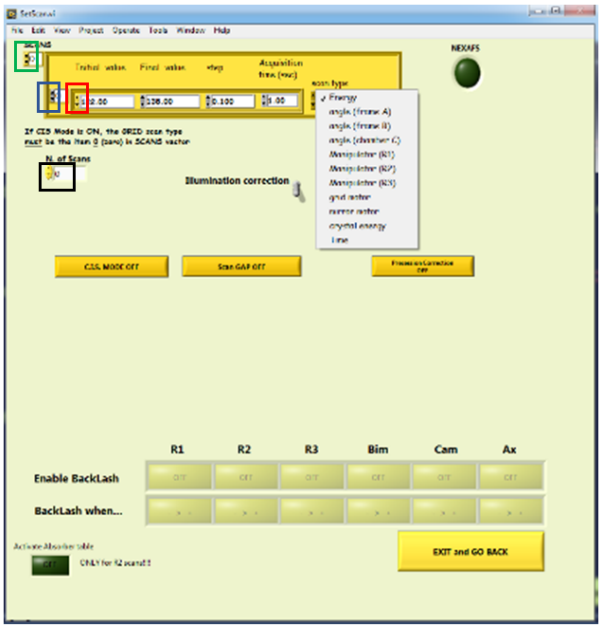

Figure 11: Scan Settings Panel of the ALOISA Acquisition Software.

4.5 Moving the chamber and the manipulator: Angle Settings

The majority of the mechanical movements of the experimental chamber and the manipulator can be managed through the Angle Settings Panel, shown in fig.12. In the upper part of the panel you can find the controls associated with the sample holder/manipulator movements, as well as the ones of the bimodal and axial frames. There are three columns of parameters. In the "Go to..." field you can write the final position value of the element you want to move, and press the arrow button next to it. The software automatically calculates the step that should be added to reach the the desired value, shown in the "Step ((°/mm)" field; in this case, no matter if the step is positive or negative, you have always to press the "+" button (add step) in the "Step (°/mm)" column. A "pop-up" window will open indicating that the motors are moving. DO NOT CLOSE OR CHANGE THIS WINDOW. If you want to stop the motors you can press the appropriate "stop button" in the running pop-up window. This window is automatically closed once the movement is completed. The new current position is visualized in the "Position" field.
You can also directly write the appropriate step in the "Step ((°/mm)" field and add ("+" button) or subtract ("-" button) it, according to the actual position of the element you want to move, and your desired final position of the element.

IMPORTANT: Do not exceed the lower and upper limits reported in tab.1 for the manipulator and the experimental chamber!

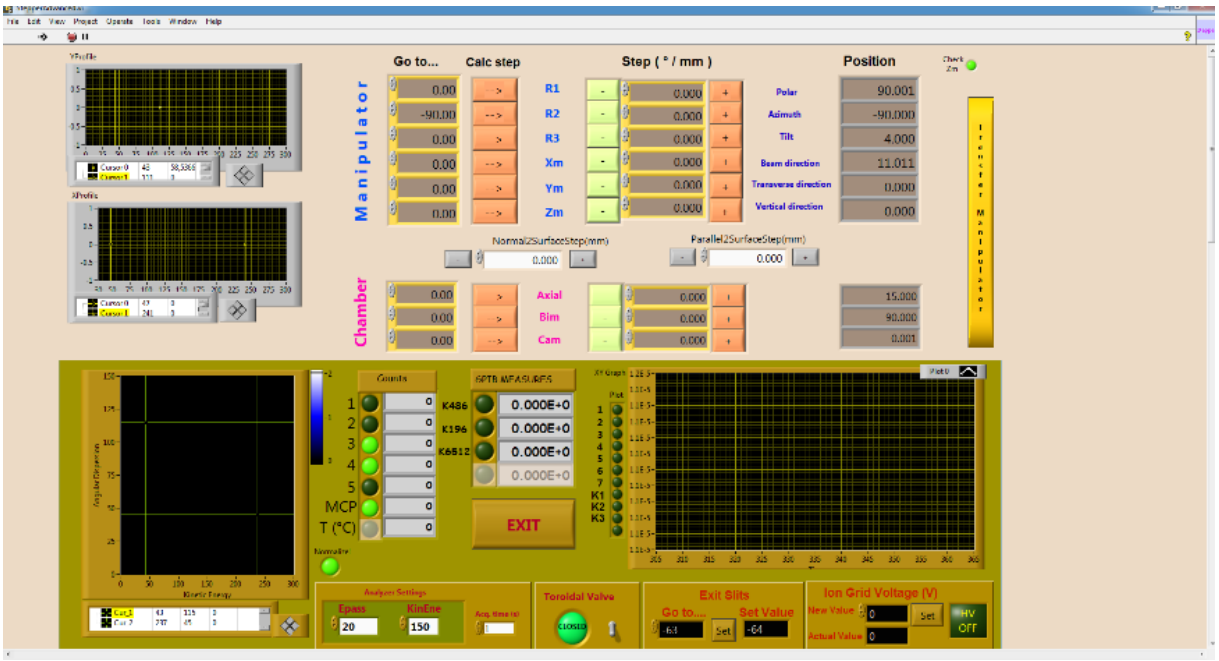

Figure 12: The Angle Settings Panel of the ALOISA Acquisition Software.

There are six degrees of freedom regarding the sample holder/manipulator:

- **R1**: is the polar angle of the sample holder, it rotates the manipulator with respect to the SR beam (-90° ÷ +185°). The polar angle value is very important in different cases: molecules deposition, NEXAFS acquisitions, sample changing or RHEED measurements.
- **R2**: is the azimuthal angle of the sample holder, its rotation axis is defined by the sample surface normal (-95° ÷ +95°). A certain value of the azimuthal angle is frequently associated with a specific crystallographic direction.
- **R3**: is the grazing angle of the sample holder, its rotation axis is defined by the SR beam (-2° ÷ +15°). The tilt of the sample holder defines the grazing angle of the synchrotron beam with respect to the sample surface. Photoemission measurements are performed at a grazing angle (R3) of +4°, while NEXAFS measurements are performed at a grazing angle(R3) of +6°. Due to possible different sample heights with respect to the

photon beam direction of different samples mounted on the sample holder, a nominal value of +4° of R3 could not correspond to a grazing angle of +4° of the incident radiation with respect to the sample surface. In order to evaluate the R3 value corresponding to a grazing angle of +4°, the angle between the direct SR beam and the reflected one by the sample surface can be estimated by the use of the CCD camera mounted on the phosphorous screen at the exit of the chamber. A marker on the CCD monitor permits to evaluate the grazing angle, and set R3 accordingly to it. An offset can be added to the read value of R3 in the "Initialize Manipulator" subroutine (see section 2.1).

- **Xm**: is the translation of the manipulator along the SR beam direction. Only two values are usually allowed: 11mm when the sample holder is in the preparation chamber, and +379mm whan the sample holder is in the measurement chamber. A value of Xm = +8mm is used during the sample replacement operation. You should not use others Xm values, and you should change this parameter only through the "Transfer Manipulator" Procedure (see following text".
- **Ym**: is the translation of the sample holder along the direction perpendicular to the SR beam and parallel to the synchrotron ring, i.e the direction along the ALOISA work station - Synchrotron ring axis.
- **Zm**: is the translation of the sample holder along the direction perpendicular to the SR beam and perpendicular to the synchrotron ring, i.e it is the translational movement perpendicular to Ym.

The measurement chamber has three movable elements:

- **Axial**: this parameter controls the rotation of the axial frame. The rotational axis is defined by the SR beam. At the present day, the experimental apparatus mounted on the axial frame is not in use, **and hence it is kept at a value of -15°.**
- **Bim**: this parameter controls the rotation of the bimodal frame. The rotational axis is defined by the axis perpendicular to the SR beam and passing through the center of the experimental chamber. **The bimodal frame is kept at +90° for photoemission and NEXAFS measurements**.
- **Cam**: this parameter controls the rotation of the chamber. The rotational axis is defined by the SR beam. **The Cam value is 0° for XPS and -45° for NEXAFS**.

The lower part of the Angle Settings Panel controls the visualization of the recorded channels. The graph on the left side shows in real time the 2D detector image of the photoelectron analyzer. Through the "Epass" and "KinEne" fields you can instantly set the Pass Energy and Kinetic Energy of the electron analyzer. The "Acq. Time (s)" control sets the acquisition time of the 2D image. On the right side of the Main Panel a "traffic light" like program is always running. Every time the pass energy or the kinetic energy of the analyzer is changed, this software checks for the analyzer's lens system power supplies stability: only when all the three red lights turn green the set pass energy/set kinetic energy command is sent to the power supplies. By changing the kinetic energy the image shown in the graph, i.e. the projection of the photoelectron band on the 2D detector surface, will move along the kinetic energy dispersion direction. In order to optimize the XPS spectra you should center the 2D image inside the margins of the X-Y green cursors: to center the image move the Ym coordinate towards positive values (the image will move up in the cursor's region) or towards negative values (the image will move down in the cursor's region) with a step of 0.05 mm.

DO NOT USE PASS ENERGY AND/OR KINETIC ENERGY VALUES WITH NO PHYSICAL MEANING IN ORDER TO AVOID INSTRUMENTAL FAILURES ON THE POWER SUPPLIES.

In the same panel region there are the seven possible active channels, and the three GPIB channels. The channels with the green light are the enabled ones. You can plot these channels on the right side graph of the panel by simply clicking on the corresponding green button next to the graph. You can plot only one channel at a time. The "MCP" channel is the integrated signal of the 2D image along the angular dispersion direction; the image is integrated only in the region defined by the X-Y graph cursors.
Through the Angle Settings Panel is also possible to set the beamline exit slits value, to open/close the toroidal valve, and set the channeltron grid voltage (if the corresponding power supply is in remote mode).

4.5.1 Transfering the Manipulator

The "Transfer Manipulator" button in the Angle Settings Panel is used to transfer the sample holder from the preparation chamber to the measurement chamber, and the other way round. The Manipulator is in the preparation chamber at Xm=11, while the manipulator is in the experimental chamber when Xm=379. The software automatically switches between this 2 positions.

EXTREMELY IMPORTANT: BE SURE THAT THE MANUAL VALVE BETWEEN THE TWO CHAMBERS IS COMPLETELY OPENED (END OF STROKE OF THE VALVE) BEFORE YOU TRANSFER THE MANIPULATOR FROM THE PREPARATION CHAMBER TO THE EXPERIMENTAL CHAMBER, OTHERWISE YOU WILL DAMAGE IT!!! NEVER CLOSE THE VALVE WHEN THE MANIPULATOR IS IN THE EXPERIMENTAL CHAMBER!!!

YOU CAN TRANSFER THE MANIPULATOR FROM THE PREPARATION CHAMBER TO THE EXPERIMENTAL CHAMBER ONLY IF THE PRESSURE IS $< 2x10^{-9} mbar$.
In order to move the manipulator from the experimental chamber to the preparation chamber, follow this procedure:

1. Go in the "Angle Settings" panel. Press the "Transfer Manipulator" vertical yellow button. A new panel will appear (see fig.13.
2. Be sure to be in these positions:

   $R_1 = 90°$
   Cam=0°
   Bim=90°
   Axial=$-15°$
   Xm = 379 mm
3. Press "Start Transfer".
4. ONLY WHEN THE TRANSFER IS COMPLETED, close the manual valve between the chambers.

In order to move the manipulator from the preparation chamber to the experimental chamber, follow this procedure:

1. Go in the "Angle Settings" panel. Press the "Transfer Manipulator" vertical yellow button. A new panel will appear (see fig.13.
2. Be sure to be in these positions:

   $R_1 = 90°$
   Cam=0°
   Bim=90°
   Axial=$-15°$
   Xm = 11 mm
3. Press "Start Transfer".
4. **OPEN THE MANUAL VALVE BETWEEN THE EXPERIMENTAL AND PREPARATION CHAMBERS UNTIL YOU REACH THE END OF STROKE OF THE VALVE.**
5. The window shown in fig.14 will appear. ONLY IF YOU ARE SURE THAT THE VALVE IS OPEN, write the word open in the empty field, press the "enter" button, and click on an empty space of the green panel (do not click on the "exit transfer procedure" button, unless you want to stop the transfering).

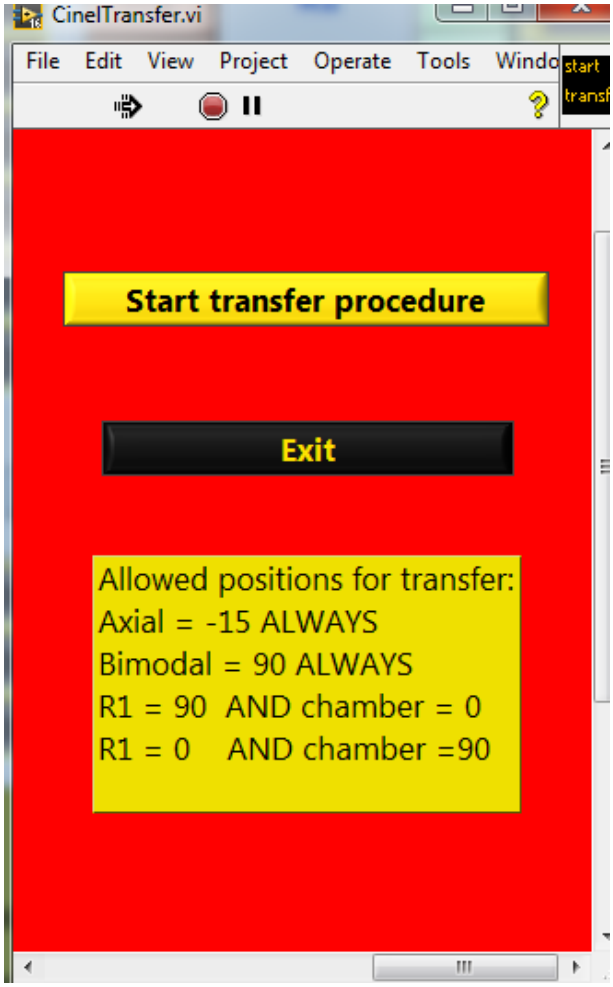

Figure 13: Transfer manipulator Panel of the ALOISA Acquisition Software.

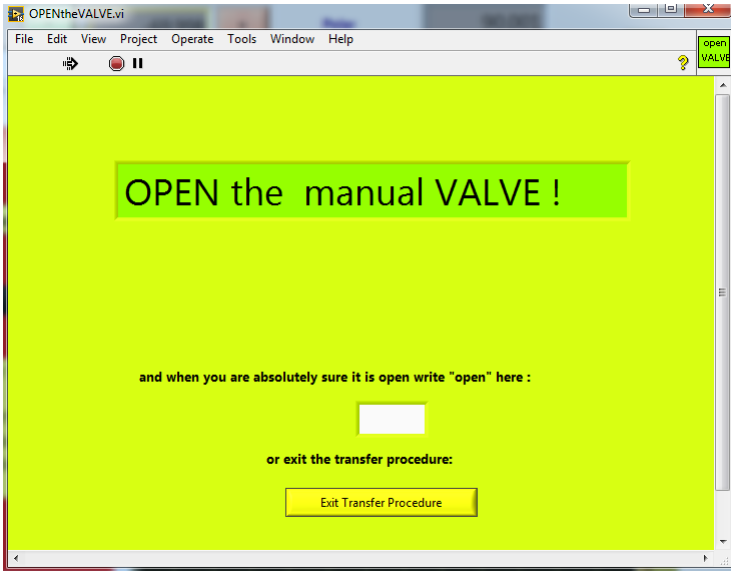

Figure 14: Transfer manipulator Panel of the ALOISA Acquisition Software.

4.6 Setting the Photon Energy

The Set Photon Energy Panel is shown in fig.15; to change the photon energy write the desired photon energy value (in electronvolt) in the "New Energy (eV)" field, select the proper mirror value in the "New Angle (deg)" field and press the "Set Energy" button. A pop-up window will open indicating that the grating and the mirror are moving. DO NOT CLOSE OR CHANGE PARAMETERS IN THIS POP-UP WINDOW. In order to have a more precise value of the set photon energy, a backlash procedure should be followed, especially if change also the used mirror: once you have set the new photon energy, set a photon energy 50-80 eV higher in energy, and set again your desired photon energy. The new values of photon energy and mirror will be displayed in the "STATUS" parameters column, and will be upgraded also in the main software routines.
A photon energy vs mirror graphic located in the bottom part of the panel can help in choosing the proper mirror according to the photon energy range. A more accurate photon energy - mirror - gap table can be found on the desktop of the acquisition computer in a file named " **GAP _vs _hv.ods** ". Once you finish the "set photon energy" procedure, click on the "Exit and GO Back" button.

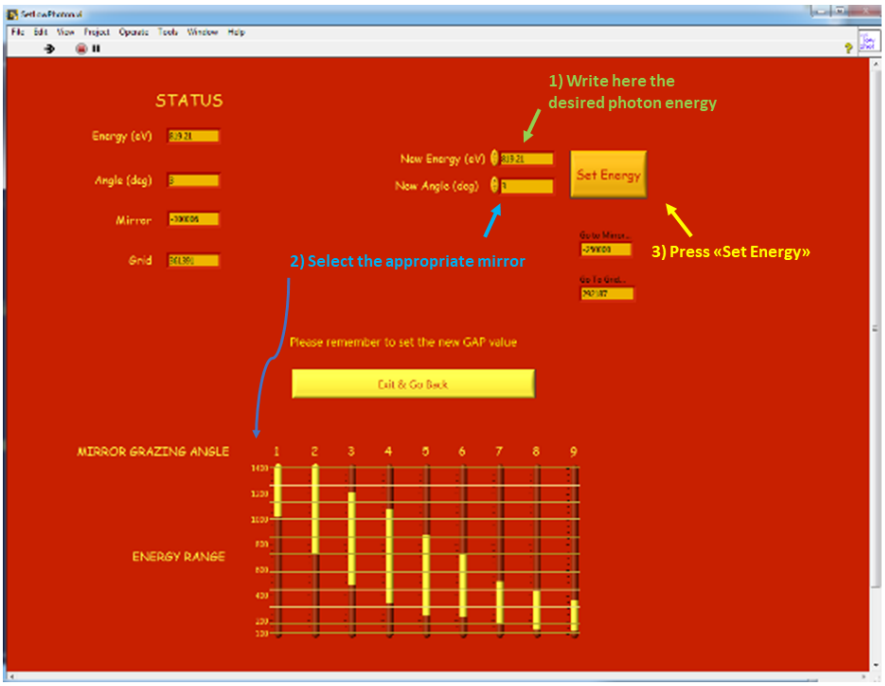

Figure 15: The Set Photon Energy Panel of the ALOISA Acquisition Software.

4.7 Setting the Undulator Gap

The Set GAP Panel is shown in fig.16; to change the gap write the desired gap value in the "Value to Set" field and press the "SET GAP" button. A pop-up window will open indicating that the undulator is moving. DO NOT CLOSE OR CHANGE PARAMETERS IN THIS POP-UP WINDOW. The new gap value will appear in the "Current Value" field. On the left side graph different "GAP vs Photon energy" curves are plotted; select the synchrotron beam energy using the switching button to switch between 2.4GeV and 2.0GeV, and move the cross marker on the graph across the curves to display the gap-energy values in the cursors area of the graph (bottom right). If you want to optimize the gap value for a certain photon energy in order to maximize the photon flux, you can display the flux "I0" on the right side graph by activating the green button of channel 3 next to the plot, and change the gap value iteratively until the "I0" signal is maximized.
The "Set new photon energy" button will open the "Set Photon Energy" Panel (see section 4.6).

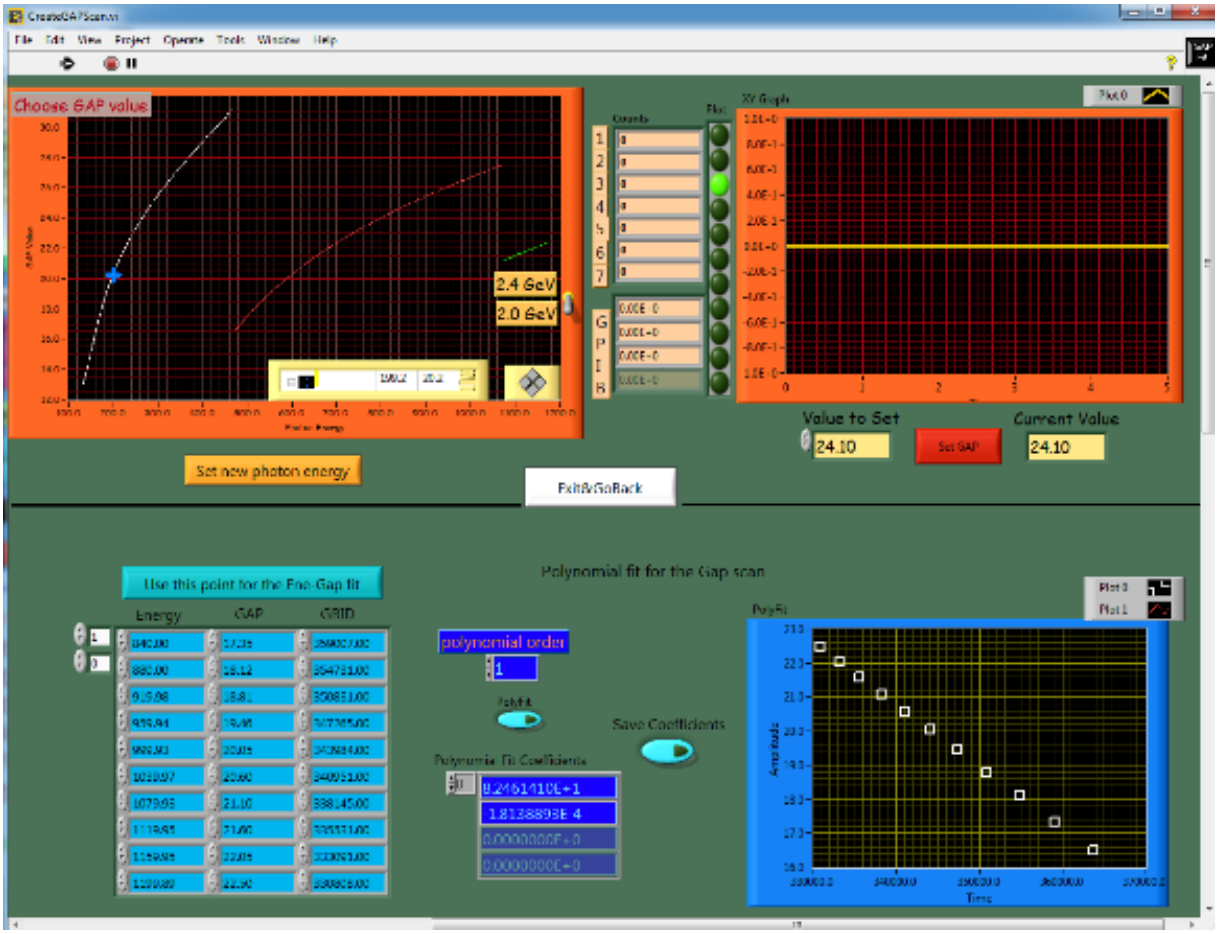

Figure 16: The Set Gap Panel of the ALOISA Acquisition Software.

The "Save Coefficient" button opens a "Core shell - Photon Energy - GAP" coefficients table, shown in fig,17, in which for each common core shell (e.g. C1s, Nis, Co 2p, etc.) a shell label, the reference photon energy, the best gap value at the reference photon energy, the gap-energy fit parameters, and the optimum channeltron grid voltage are reported. This table is used in the NEXAFS Data Acquisition procedure, but can be also used as a mirror-photon energy-gap values reference.

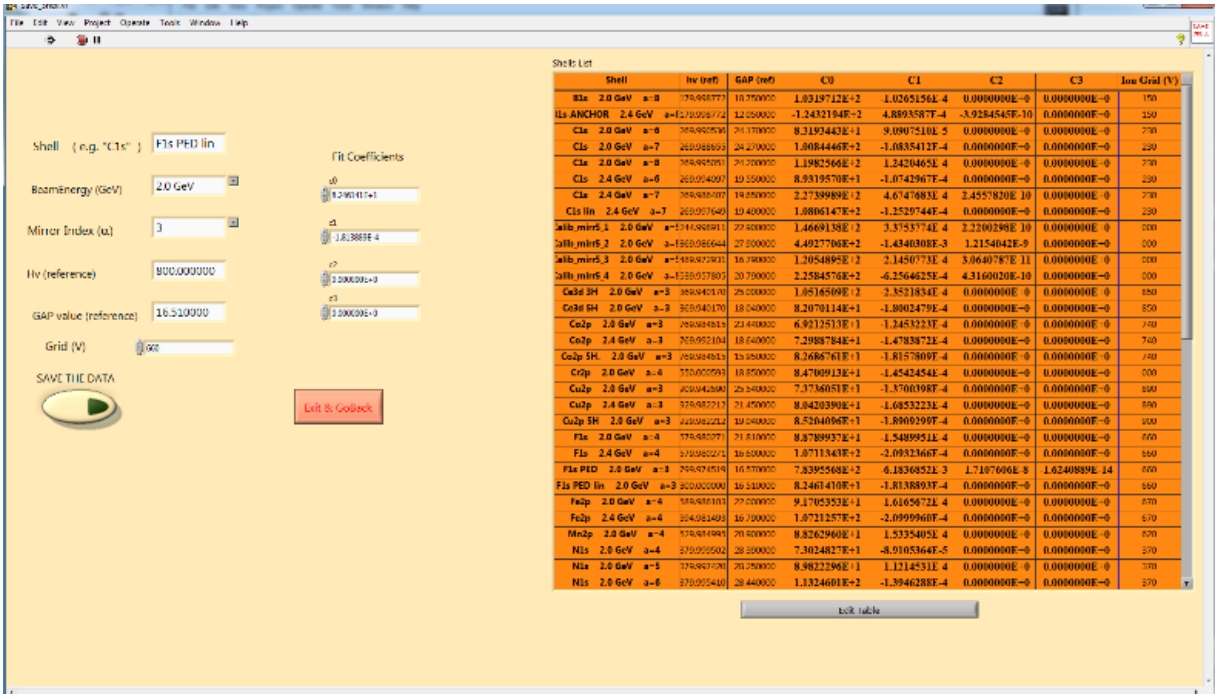

Figure 17: The "Core shell - Photon Energy - GAP" coefficients table Panel of the ALOISA Acquisition Software.

4.7.1 Creating a new Energy-Gap reference curve for NEXAFS measurements

A NEXAFS measurement requires a scan in photon energy. Changing photon energy implies a deviation from the harmonic maximum, defined by the set undulator gap value, with a not negligible decreasing in photon flux. Therefore, the gap value has to be constantly optimized at every new photon energy point of the NEXAFS scan. The software do this automatically during a NEXAFS scan by calculating the best gap value for each energy points. In order to do this, the software uses pre-calibrated reference GAP-Photon Energy polynomial fitting curves.
If you want to add a new core shell point to the "Core shell - Photon Energy - GAP" table (i.e. a new GAP-Photon Energy polynomial fitting curve) , in order to be used in a NEXAFS measurement, use the following procedure:

1. Empty the left-bottom "Energy-GAP-GRID" table (right-click on the scroll arrow field on the top-right of the table -> click "Empty Array").
2. Set the photon energy corresponding to the first point of your desired photon energy range by the use of the "Set New Photon Energy" button (considering also the backlash procedure). YOU HAVE TO START FROM THE LOWEST PHOTON ENERGY VALUE OF YOUR ENERGY RANGE.
3. Find the best gap value that maximize the photon flux "Io".
4. Press the "Use this point for the Ene-gap fit" button. The first raw of the table should be now filled with the first point of the gap-energy table.
5. Repeat points 2,3 and 4 for each point of your photon energy range. The table willl be automatically filled. Every point will be displayed on the "PolyFit" graph (bottom-right of the panel). Depending on the energy range, 2 or more energy points are needed. For instance for a ca. 60eV photon energy range, 3-4 point every 15eV are usually sufficient.
6. Choose the proper polynomial order of the fit function in the "polynomial order" field.
7. Press the "PolyFit" button in order to perform a fitting procedure on the selected energy points. A fit curve will appear on the selected points on the "PolyFit" graph. The fit coefficients are listed in the "Polynomial Fit Coefficients" table.
8. Once you are satisfied with the fitting procedure, press the "Save Coefficient" button.

9. In the "Core shell - Photon Energy - GAP" coefficients panel, fill all the required fields (shell label name, beam energy, mirror, channeltron grid voltage), and press the "Save the data" button. A pop-up message will apear, asking you if you want to replace the elements in the table. If you want to do it, press "yes": the table will be automatically upgraded with the new point. In any case, you can change an element in the table in any time by pressing the "edit table" button.

10. Press the "Exit and GO back" button.

4.8 Switching on the electron detector

The experimental chamber is equipped with a hemispherical photoelectron analyzer, on wich a crossed delay line is mounted as the photoelectron detector, consisting of two Micro CVhannel Plates (MCP), an anode plate and the dleay line. In order to switch on the voltages on the detector open and run the "Set MCP Voltages" software on the acquisition PC's desktop. Set the optimal value for the MCP voltage in the "V_MCP" field and press the "Ramp Voltages" button.

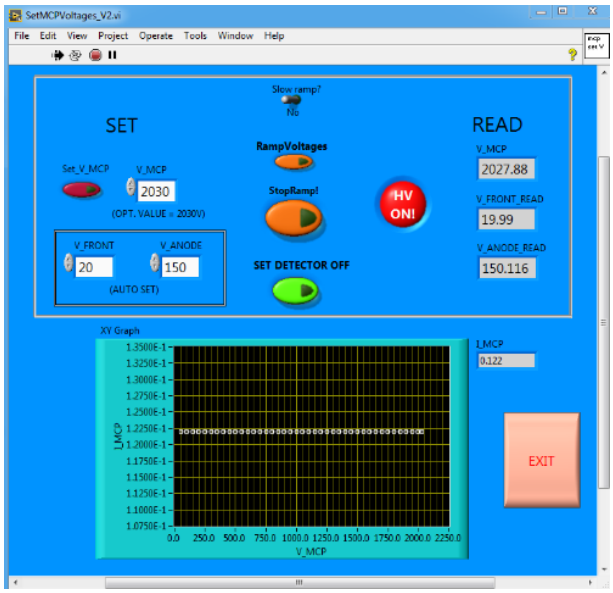

Figure 18: The Photoelectron detector Software.

5 DATA ACQUISITION

5.1 XPS Data Acquisition

A XPS measurement can be launched through two different procedures.
If you want to set a XPS scan from scratch, you have to follow the "Set Photon - Set Gap - Set Scan Procedure" (see paragraph 5.1.1).
If you already have recorded a XPS scan and you just want to launch it again, also with different scan parameters, you can use the "Fast Scan Selector" procedure implemented in the software main panel. This is the standard procedure in XPS experiments.

5.1.1 Set Photon - Set Gap - Set Scan Procedure (new XPS measurement)

NOTE: THE XPS MEASUREMENT ARE ALWAYS PERFORMED AT R1 = 90° AND R3 == +4° OF GRAZING ANGLE (SEE SECTION 4.5). MAXIMUM ACQUISITION TIME IS 2.4s.

In order to launch a completely new XPS scan, follow this procedure:

1. Set the desired photon energy in the "Set Photon Energy" panel (see section 4.6).
2. Set the corresponding undulator gap in the "Set GAP" panel (see section 4.7).
3. Try to find the desired photoemission bands in the 2D image plot through the "Angle Settings" panel (see section 4.5), and adjust the Zm and Ym values if needed.
4. Set the scan parameters in the "Scan Settings" panel (see section 4.4) choosing the appropriate kinetic energy range, energy step and acquisition time for the desired XPS measurement.
5. Open the Toroidal Valve through the Valve button control (point 17 in fig,6).
6. Click the "Start Scan" button.

5.1.2 Fast Scan Selector Procedure (RECOMMENDED)

NOTE: THE XPS MEASUREMENT ARE ALWAYS PERFORMED AT R1 = 90° AND R3 == +4° OF GRAZING ANGLE (SEE SECTION 4.5). MAXIMUM ACQUISITION TIME IS 2.4s.

If you already saved a XPS scan and you want to launch it again, or you want to start from a saved XPS scan and simply add/change/delete one or more regions, you can use the "Fast Scan Selector" procedure in the Main Panel of the Aloisa Software. This procedure is highly recommended during standard photoemission experiments: in few seconds you can start a XPS measurement.
In order to start the XPS scan you have to follow the procedure shown in fig.19.

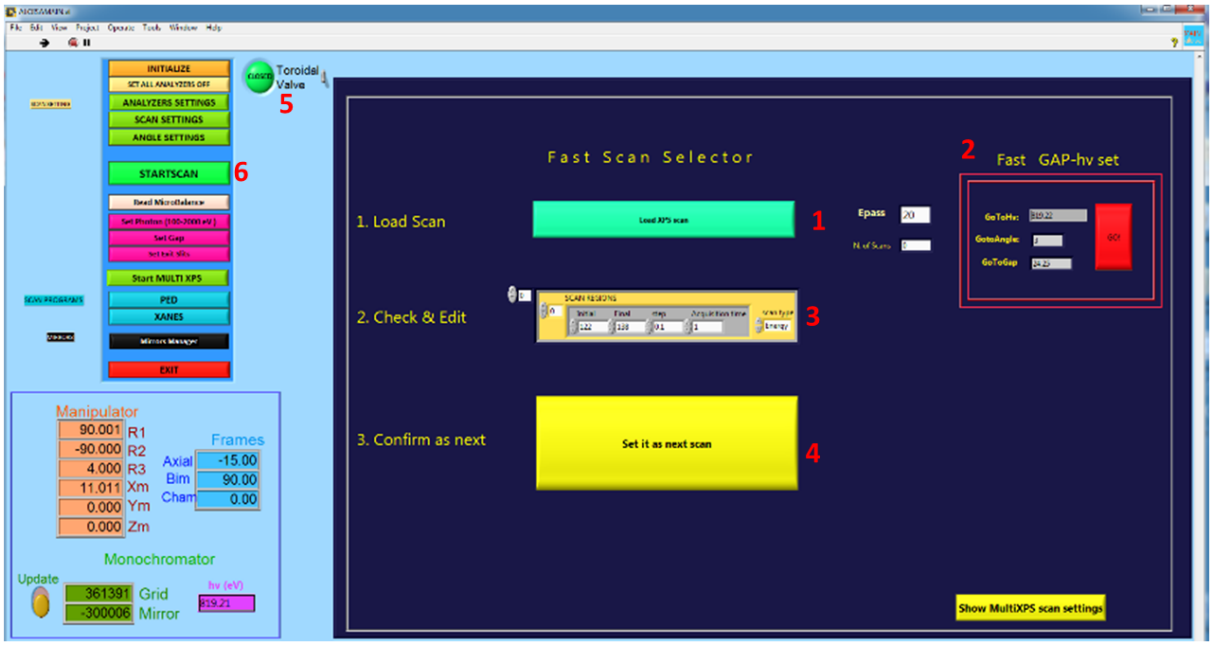

Figure 19: The "Core shell - Photon Energy - GAP" coefficients table Panel of the ALOISA Acquisition Software.

1. Press the green "Load XPS Scan" Button. A pop-up window will open, where you can select the ".prx" file you want to load. Choose the Prx file and press "load". The "Epass", "N. of Scan", "Fast GAP-hv set" and "SCAN REGIONS" fields will be automatically filled with the same parameters as the loaded file.
   IMPORTANT: DO NOT PRESS "CANCEL" IN THE "LOAD POP-UP WINDOW" WHEN LOADING A FILE OR THE SOFTWARE WILL CRASH! PLEASE LOAD A PRX FILE ANY-WAY.
   NOTE: If you enter in the "Angle Settings" after you pressed the "SET IT AS NEXT SCAN" button and change the pass energy, the new Epass value set will be used in the scan, instead of the loaded one.

2. If not already set, set the desired photon energy and the corresponding gap value through the "Fast GAP-hv set" mini-panel (top-right) by pressing the red "GO" button. NOTE: BE SURE THAT THE TOROIDAL VALVE IS CLOSED BEFORE PRESSING THE "GO" BUTTON. It is a good practice to check the "I0" value in the "Angle Settings" Panel: if it is lower than the values, try to optimize again the undulator gap in the "Set GAP" panel (see section 4.7).

3. Check the scan parameters in the "SCAN REGIONS" field, and edit them if necessary. You can check the different scan regions using the scroll arrows.

4. Once you checked all the scan parameters press the yellow "SET IT AS NEXT SCAN" button. It is better to press it a couple of times, in order to be sure that the can parameters are loaded properly.

5. Open the toroidal valve.

6. Press the "START SCAN" button.

5.1.3 MultiXPS Scan

The Multiscan menu (fig 20) can be used to record multiple XPS, also at different photon energies, in sequence. Click on the "Show MultiXPS scan settings" button (on the down left corner of the main panel) to open the MultiXPS menu. You can load a multi-XPS file with the "Load Multi File" button, or create a new one by loading XPS scans with the "–> Add scan to multiXPS" button. Press "Start MultiXPS" to start the XPS scans seiries.

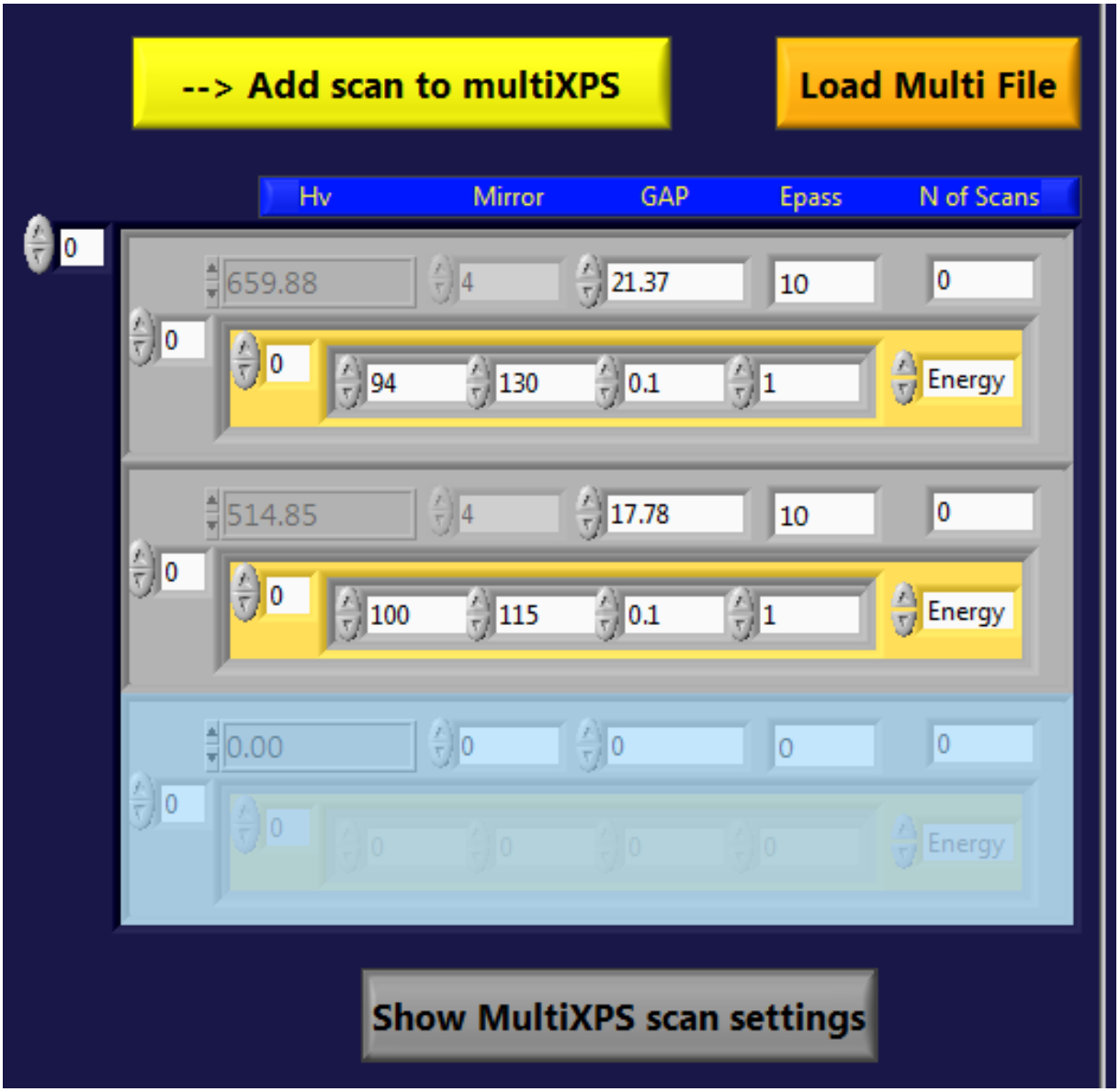

Figure 20: The MultiXPS menu of the ALOISA Acquisition Software.

5.1.4 View and Save a XPS Scan

Once you started a XPS scan, the panel showed in fig.21 will appear. Here are shown the XPS regions scanned in function of the kinetic energy. You can zoom in the regions with the "lens icon" of the top graph, use the "cursors" option (right click on the cursor label and select "bring to center") with the "cross icon" on the top graph, and compare the last measured scan (red trace) with the ongoing one (white trace) by pressing the "Show previous scan" button. Use the x and y locker icons to control the autoscale of the graph axes. Use the "Close valve after scan" button to automatically close the toroidal valve after the scan (to prevent radiation damage on the film).
In order to save the scan, press the "Save" button that appears once the scan is finished. The files are named automatically with the date and a progressive number.
For the first file of the day, after you have pressed the "Save" button, press "Change directory", create a new folder with the format "dd_mm_yyyy" for the current day, enter in the new folder, press "choose this directory" and than "ok".
Press "Exit" to return to the Main Acquisition Panel.

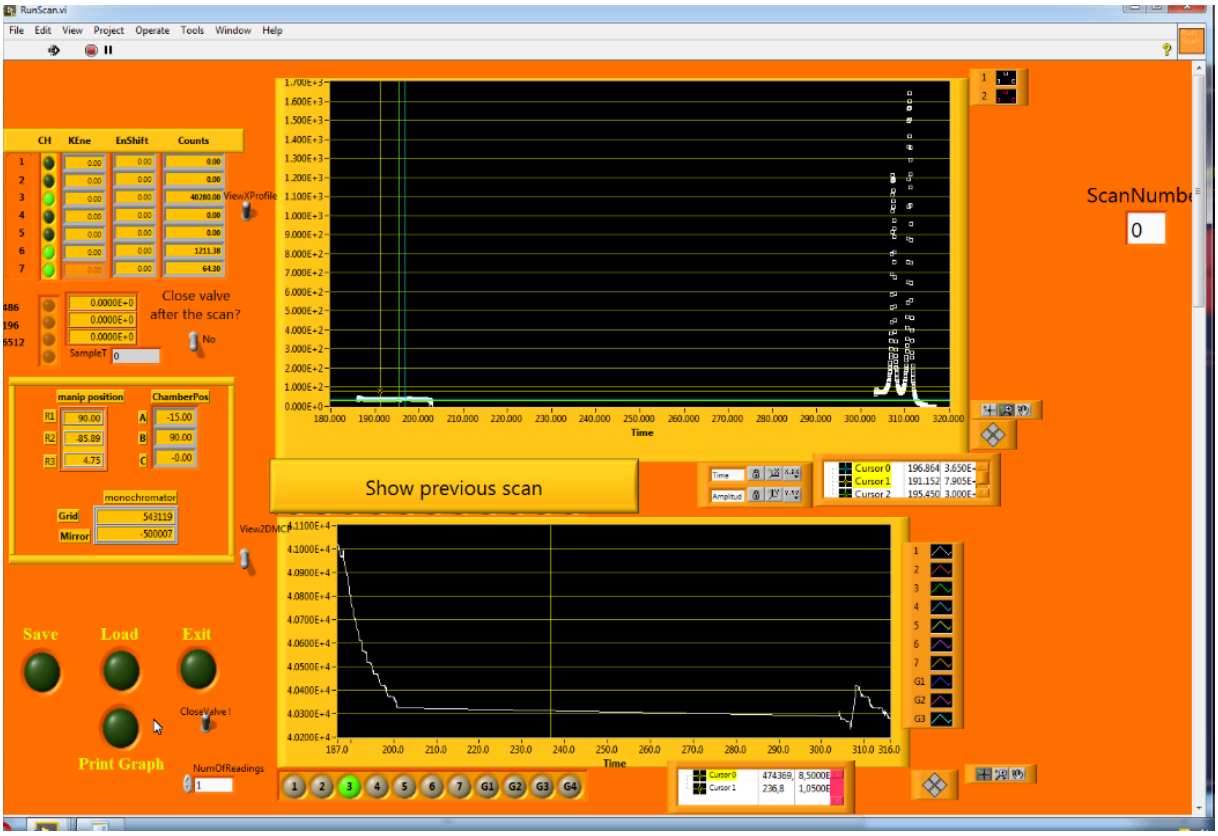

Figure 21: The XPS Scan Panel of the ALOISA Acquisition Software.

5.2 NEXAFS Data Acquisition

NEXAFS Experiments are managed trough the XANES Panel. For the most common absorption edges (C1s, N1s, O1s, Co2p, etc.) pre-set NEXAFS measurement parameters are available in the "Core shell - Photon Energy - GAP" coefficients table (see section 4.7), where for each absorption edge you can find the reference photon energy, the reference gap value, and the reference channeltron grid voltage. The NEXAFS measurements are usually performed at $R_1 = 90°$ and $R_1 = 0°$, corresponding to p-polarization and s-polarization respectively ($R_1 = 35°$ corresponds to the Magic Angle). See fig.22 for the NEXAFS acquisition geometry.

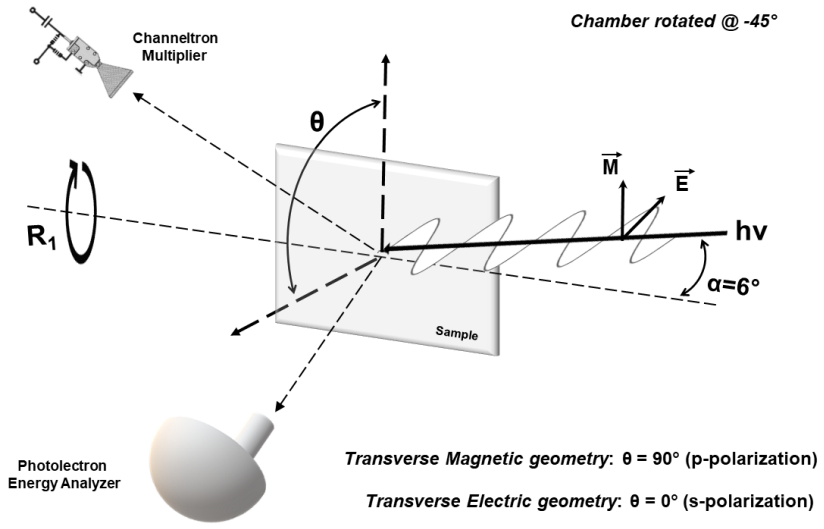

Figure 22: The NEXAFS Acquisition Geometry at the ALOISA beamline. The grazing angle α is kept constant at $6°$ and the chamber is rotated at $-45°$. The angle Θ between the surface and the polarization vector plane of the synchrotron radiation defines the NEXAFS geometry: $\Theta = 90°$ corresponds to the Transverse Magnetic geometry (quasi p-polarization), while $\Theta = 0°$ corresponds to the Transverse electric geometry (s-polarization).

In order to launch a NEXAFS measurement, follow the procedure of the next paragraphs.

5.2.1 STEP 1: set NEXAFS positions

The NEXAFS measurements are recorded at $6°$ of grazing angle, and since the channeltron multiplaier is perpendicular with respect to the manipulator, in order to collect the spectra in both p- and s- polarizations the chamber must be rotated of $-45°$.

1. Increase R_3 of $2°$ with respect to the XPS value (i.e. if $R_3 = 4°$ for XPS, go to $R_3 = 6°$ for NEXAFS).
2. Set the chamber (Cam) to $-45°$.
3. Set y and z to the optimized values for both P- and s-polarizations (i.e. the values at witch the pre-edge signal is equal for both polarizations).
4. Set the kinetic energy of the analyzer far above the ionization threshold of the selected shell (e.g. if you want to measure the N 1s NEXAFS, i.e. in the 400-420 eV range, set the kinetic energy to something like 600eV), to avoid damages to the analyzer's MCP.

5.2.2 STEP 2: switch on the channeltron power supplies

Switch on the power supplies shown in fig.23 in the order from left to the right, and set on the last power supply (far right) the trimmer value for the desired threshold (e.g. for C 1s set the trimmer to 0.42, that corresponds to -0.25kV. Each tick on the trimmer correspond to 0.2). You can find the grid values in the "Set Gap" panel by clicking the "Save Coefficient", opening the "Core shell - Photon Energy - GAP" coefficients table (see section 4.7). Wait until the counts on channel 4 (NEXAFS) stabilize to 0.

Figure 23: The channeltron multiplier power supplies. From left to right: Channeltron electrode, channeltron back, channeltron grid.

5.2.3 STEP 3: set the channeltron yield on the Picolite instrument

The Picolite is an instrument to acquire and amplify very low current signals (1pA-1mA) from HV polarized electrodes. It is used to amplify the signal coming from the channeltron multiplier. The amplification level is set by the value of the instrument yield. The channeltron yield is set taking into account the signal to noise ratio, as well as the channeltron saturation level (around 100000 counts/s). There are two Picolite instruments, mounted on the same rack of the sample power supply: the one on the left is used for the draining current on the toroidal mirror (I0), the one on the right side (the one to be set) is used for the channeltron.
In order to set the Picolite yield follow this procedure and refer to fig.24:

1. Take the metal rods next to the picolite instruments.
2. On the channel 2 panel (bottom one) of the right side Picolite, press and release the "mode" button (central small hole under the "HV2" input) using one of the metal rods, until "Lo" (Local) appears on the bottom display.
3. Press the "mode" button and KEEP IT PRESSED until "C.F." appears on the display and the yield value stars blinking.
4. KEEPING THE "MODE" BUTTON PRESSED, use the other metal rod to press and release the "AO" button (left one), until you select the desired yield value.
5. Release BOTH buttons. The new yield value should appear fixed on the display.

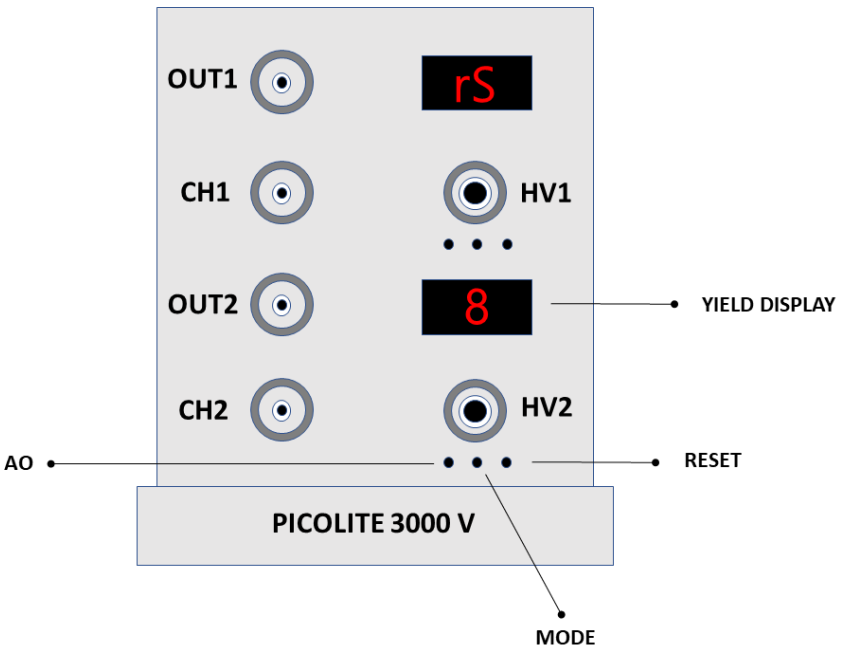

Figure 24: The Picolite Instrument.

5.2.4 STEP 4: set the reference photon energy and optimize the gap

A NEXAFS measurement consists in a scan in photon energy; this implies that the gap value has to be constantly optimized at every new photon energy point, in order to maximize the photon flux. The software does this automatically by using pre-calibrated reference GAP-Photon Energy polynomial fitting curves. For each absorption edge, a reference photon energy and a reference gap value (together with the channeltron grid voltage) can be found in the "Set Gap" panel by clicking the "Save Coefficient", opening the "Core shell - Photon Energy - GAP" coefficients table(see section 4.7). However, changes in the synchrotron ring conditions can lead to differences in the photon energy-gap correspondence with respect to the curves calculated by the program. The calculated curves can be corrected simply by adding an offset gap, i.e the difference between the optimized gap value at the reference photon energy and the reference gap value.
In order to calculate the offset gap value, follow this procedure:

1. Close the Toroidal valve.
2. Enter in the "Set Gap" Panel and in the "Save Coefficient" table and take note of the reference photon energy, the reference gap and reference channeltron grid voltage for the desired threshold (example: C1s - 2.0 GeV - α = 7 –> ref. photon energy = 270eV, ref. gap = 24.27, grid value = -230V (0.42 read on power supply trimmer)).
3. In the "Set Photon energy" panel, set the reference photon energy (New Energy field) and mirror, α (New Angle field).
4. Set the reference photon energy + 30 eV (this operation is important for backlash compensation!).
5. Set again the reference photon energy and exit.
6. In the "Set Gap" panel, set the reference gap, switch on channel 3 by clicking on the green led, and optimize the signal by changing the gap value (steps in the order of ± 0.05). **Take note of the offset with respect to the reference gap.**

5.2.5 STEP 5: set a NEXAFS Scan

The XANES Panel is shown in fig.25. In order to set a NEXAFS scan press the "Set Scan" button in the XANES Panel.

In the "Set Scan" Panel, shown in fig.26 it is possible to set the parameters for the NEXAFS measurements. Follow this procedure:

1. In the "Photon Energy Scan" Field set the scan limits (in eV), the step size (in eV), the mirror angle (same as the mirror value used for the reference photon energy), and the acquisition time (in seconds). By using the red scroll bar, you can set multiple scan regions. **You can use the "Load Scan" button to load an already measured NEXAFS scan: all the scan fields will be automatically filled.**
2. If you want to automatically measure both p- and s-polarization spectra, one after the other, click on the "Main Additional Scan" switch (top left of the panel): a new field will appear. Choose "Manipulator (R_1)" as Main scan type. Fill the starting R_1 position, the final R_1 position, the step and measuring time as follow:

   IF YOU START FROM $R_1 = 0°$
   InitialValue= 0, FinalValue=c 90, Step= 90, Measuring Time= 1

   IF YOU START FROM $R_1 = 90°$
   InitialValue= 90, FinalValue= 0, Step= -90, Measuring Time= 1
3. Select the the desired threshold ("Shell") from the top down menu on the top right of the panel (e.g. C1s - 2.0 GeV - α = 7), and write the ΔGap offset (see STEP 3).

In the case of samples very sensitive to radiation damage, you can move the sample while measuring the NEXAFS Scan bt activating the "ScanZ!" and "ScanY" buttons. You have to set the "Zstart-ZEnd" or "Ystart-YEnd" values, according to the sample usable region and the number of points of the scan: the Z/Y steps will be automatically calculated by the software taking into account the number of points of the NEXAFS scan and the "Zstart-ZEnd" or "Ystart-YEnd" values.

5.2.6 STEP 6: start NEXAFS Scan

Once the scan is set, you can start the scan following this procedure:

1. Press the "SET ANALYZER" button and check that channels 3 (I0) and 4 (NEXAFS) are on (green = OFF; red = ON).
2. Open the toroidal valve using the control in the XANES Panel.
3. Wait few seconds for the channeltron multiplier stabilization.
4. Press the "START" button.

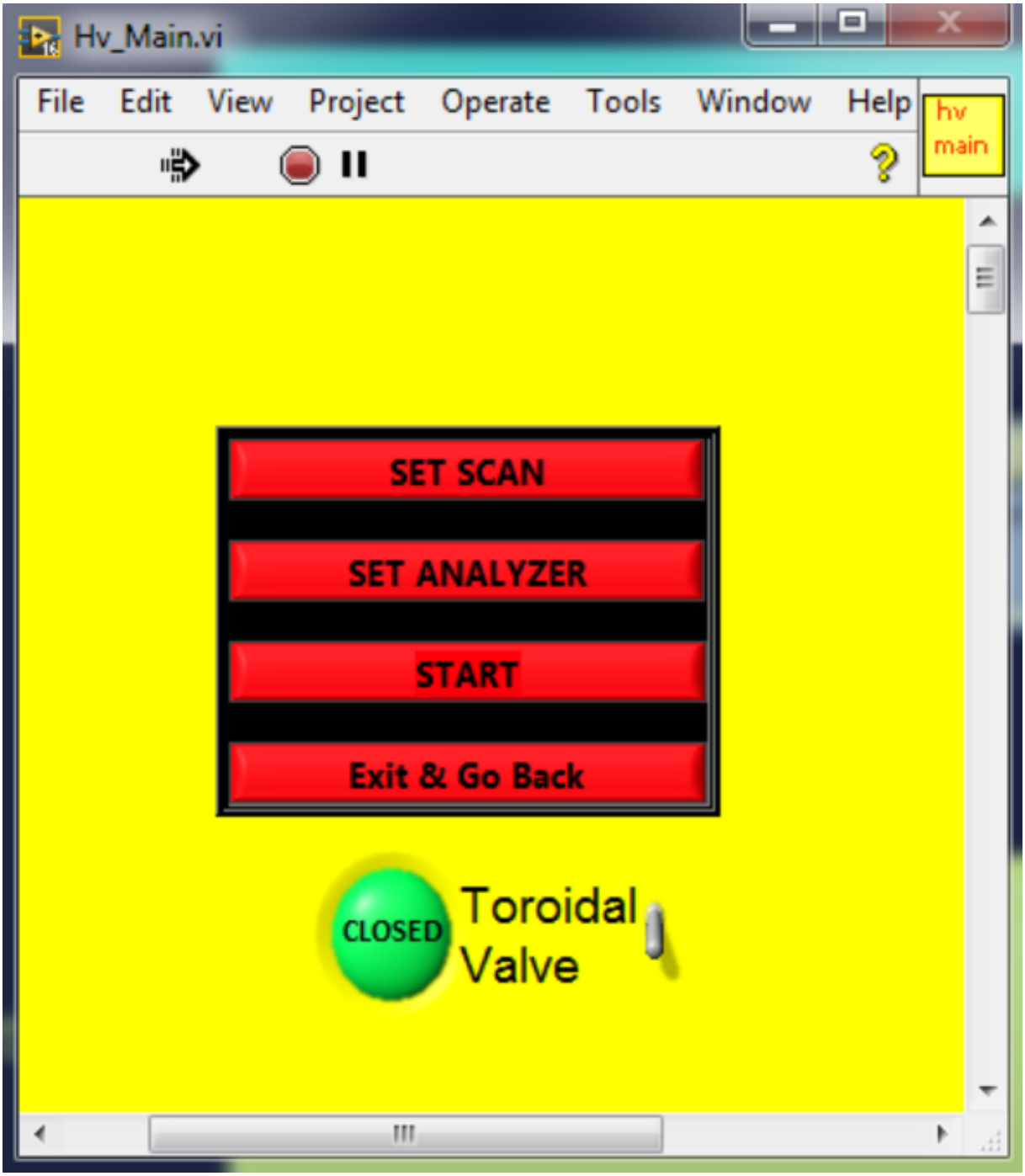

Figure 25: The XANES Panel of the ALOISA Acquisition Software.

5.2.7 STEP 7: save NEXAFS Scan

Once you press the "START" button the panel shown in fig.27 will appear. On the top graph the partial electron yield signal vs photon energy is shown, while on the bottom graph is plotted the Io signal vs the photon energy. The "close valve after scan" option allows to close the toroidal valve once the scan is finished, in order to avoid film radiation damages. On the top graph you can plot two channels at the same time, for instance both p- and s- polarization spectra by choosing channel 4 on the "history plot" field.
Once the scan is finished, press the "Save Sc" button to save the measurement.
The files are named automatically with the date and a progressive number.
For the first file of the day, after you have pressed the "Save" button, press "Change directory", create a new folder with the format "dd_mm_yyyy" for the current day, enter in the new folder, press

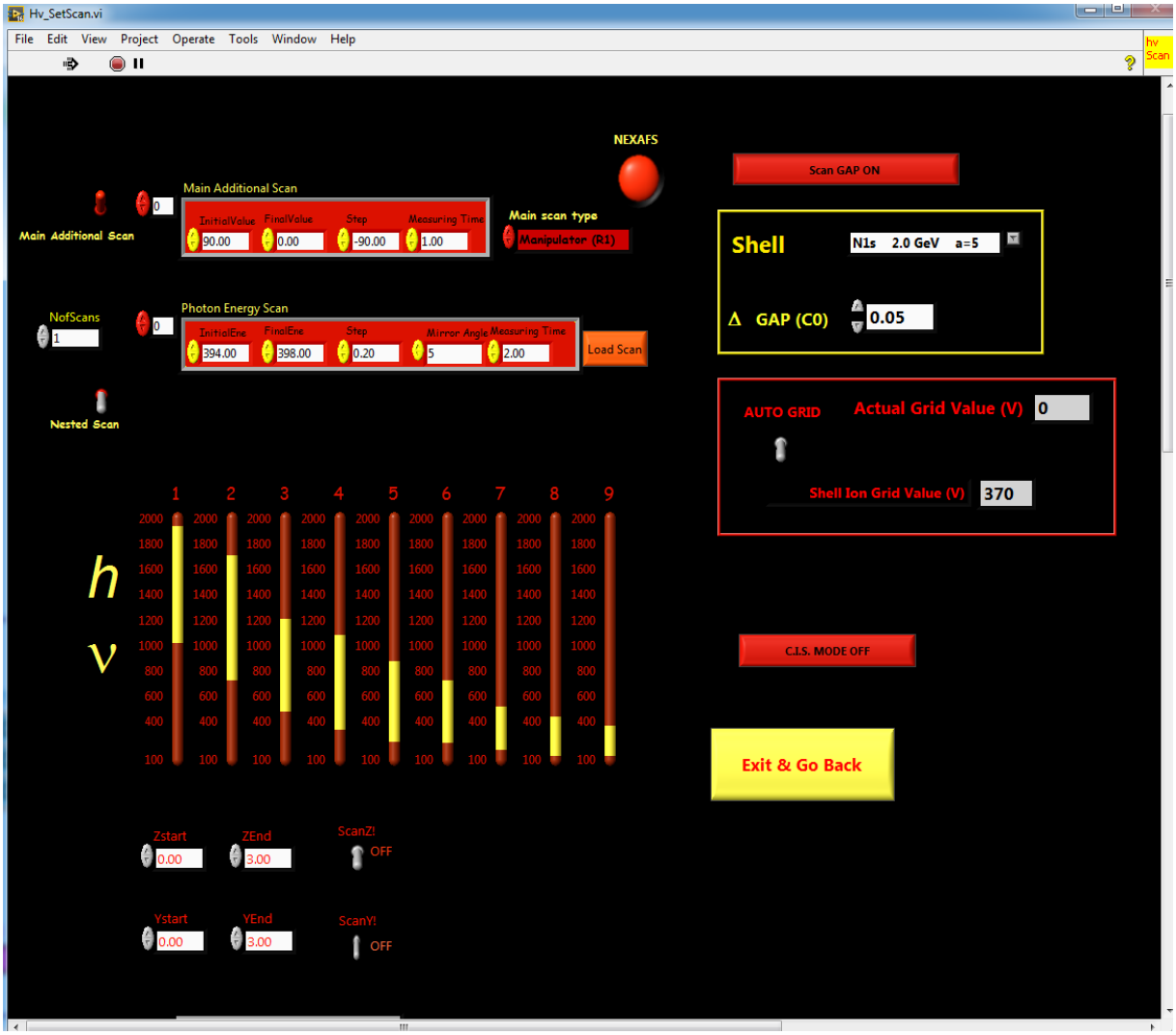

Figure 26: The XANES "Set Scan" Panel of the ALOISA Acquisition Software.

"choose this directory" and than "ok".

Press "Exit Sc" to return to the "XANES" Panel, and again "Exit and Go Back" to return to the main acquisition panel.

5.2.8 STEP 8: After a NEXAFS Scan

Once you finished the NEXAFS measurements, switch off the channeltron power supplies, and set back the chamber and manipulator in the XPS positions.

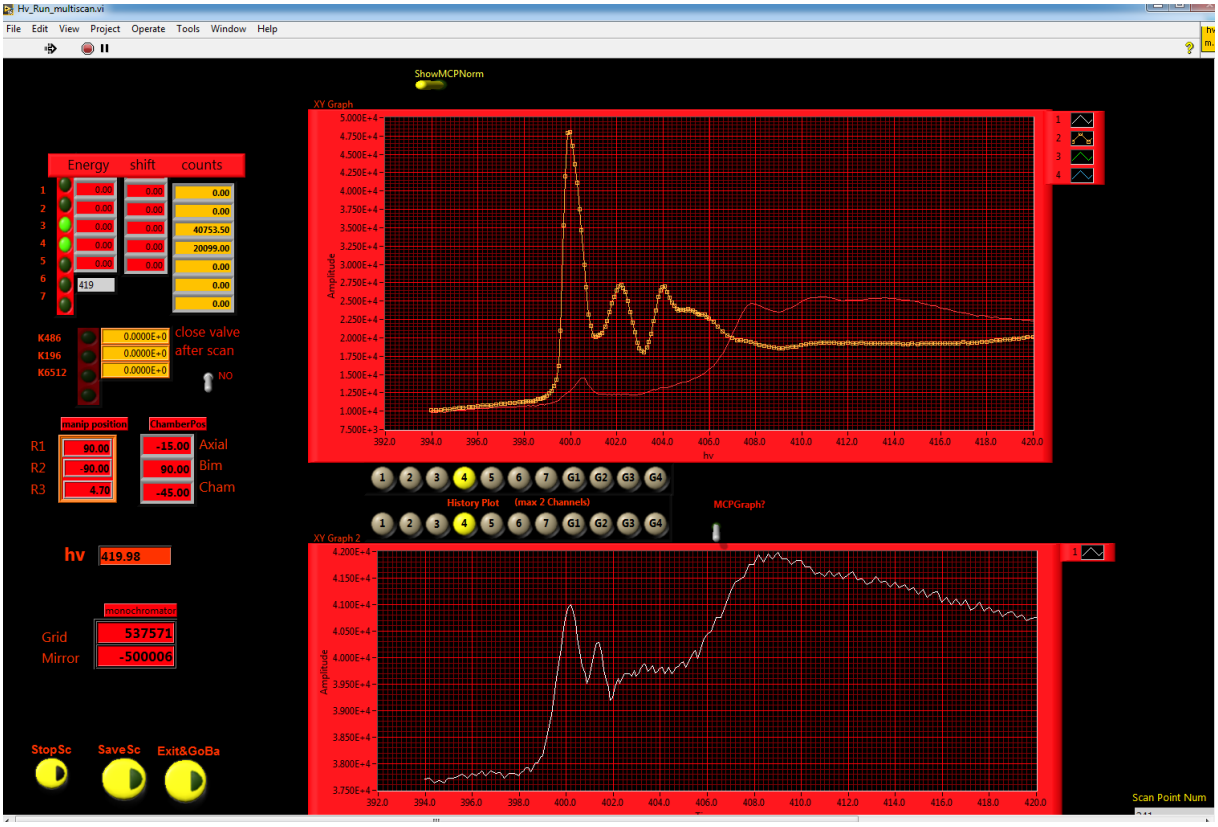

Figure 27: The NEXAFS Scan Panel of the ALOISA Acquisition Software.

5.2.9 START A NEXAFS: RECAP

1. Set NEXAFS positions: R_3 = R_3(XPS) + 2°, Cam= −45°, z and y at the optimized values (check on the experiment logbook). **SEE STEP 1 FOR DETAILS**

2. Switch on the channeltron power supplies and set the grid power supply (far right) trimmer to the refernce value for the desired theshold (e.g. set 0.42 for C1s). **SEE STEP 2 FOR DETAILS**

3. Set the channeltron yield on the Picolite (SEE STEP 3 FOR DETAILS).

4. Set the reference photon energy/mirror and reference gap for the desired absorption threshold (e.g. for C1s at 2.0 GeV set 270eV and α = 7, gap = 24.27). Optimize the gap value to maximize the flux looking at the I0 plot (channel 3). Write down the difference between the reference gap value and the optimized one (Δgap). **SEE STEP 4 FOR DETAILS**

5. Press the XANES button on the software main panel to open the NEXAFS panel, press "Set Scan", load/edit a NEXAFS scan, or set a new scan by entering the photon energy ranges, step, mirror, acquisition time, Shell, gap offset and R1 scan. **SEE STEP 5 FOR DETAILS.**

6. Check in the "SET ANALYZER" panel if the channels 3 and 4 are on, open the toroidal valve and press "START". **SEE STEP 6 FOR DETAILS.**

7. Once the scan is finished, press the "Save" button and go back to the main acquisition program. **SEE STEP 7 FOR DETAILS.**

8. Once you finished the NEXAFS measurements, switch off the channeltron power supplies, and set back the chamber and manipulator in the XPS positions. **SEE STEP 8 FOR DETAILS.**

5.3 RESPES (Resonant Photoemission) Data Acquisition

Resonant Photoemission (RESPES) experiments are performed by recording photoemission spectra at different photon energies, most of the time across a specific absorption resonance. Operatively, it is very similar to a

NEXAFS scan in which for each photon energy point a XPS spectrum is recorded. Hence, you can follow the same procedure for setting a NEXAFS scan and simply add a XPS scan inside the NEXAFS loop.
In order to start a RESPES scan, follow this procedure:

1. FOLLOW THE INSTRUCTIONS FOR A NEXAFS SCAN FROM STEP 1 TO STEP 4, except step 3 (section 5.2.6).
2. Open the "Set Scan" Panel, shown in fig.26.
3. Deactivate the "Main Additional Scan" button.
4. **Activate the C.I.S Mode ON" button**, as shown in fig.28.
5. Activate the "Nested Scan" button and set the photoemission scan you want to record at each photon energy point: set the initial kinetic energy (InitialValue), the final kinetic energy (FinalValue), the kinetic energy step in meV (Step) and the acquisition time in seconds (Measuring Time).You can set also different regions.
6. In the "Photon Energy Scan" Field set the scan limits (in eV), the step size (in eV), the mirror angle (same as the mirror value used for the reference photon energy), and the acquisition time (in seconds). By using the red scroll bar, you can set multiple scan regions. **You can use the "Load Scan" button to load an already measured RESPES scan: all the scan fields will be automatically filled**.
7. Select the the desired threshold (e.g. C1s - 2.0 GeV - α = 7) from the top down menu on the top right of the panel, and write the ΔGap offset (see STEP 4 of the NEXAFS scan, section 5.2.6).
8. Press the "SET ANALYZER" button and check that channels 3 (I0) and 6 (MCP) are on (green led on). On Channel 6, set the initial kinetic energy, AND SET THE DESIRED PASS ENERGY.
9. Open the toroidal valve using the control in the XANES Panel.
10. Press the "START" button.
11. In the scan panel shown in fig.27, activate the channel 6 instead of channel 4.
12. **Once the scan is finished, press the "Save Sc" button to save the measurement.**
    The files are named automatically with the date and a progressive number.
    For the first file of the day, after you have pressed the "Save" button, press "Change directory", create a new folder with the format "dd_mm_yyyy" for the current day, enter in the new folder, press "choose this directory" and than "ok".
13. Press "Exit Sc" to return to the "XANES" Panel, and again "Exit and Go Back" to return to the main acquisition panel.

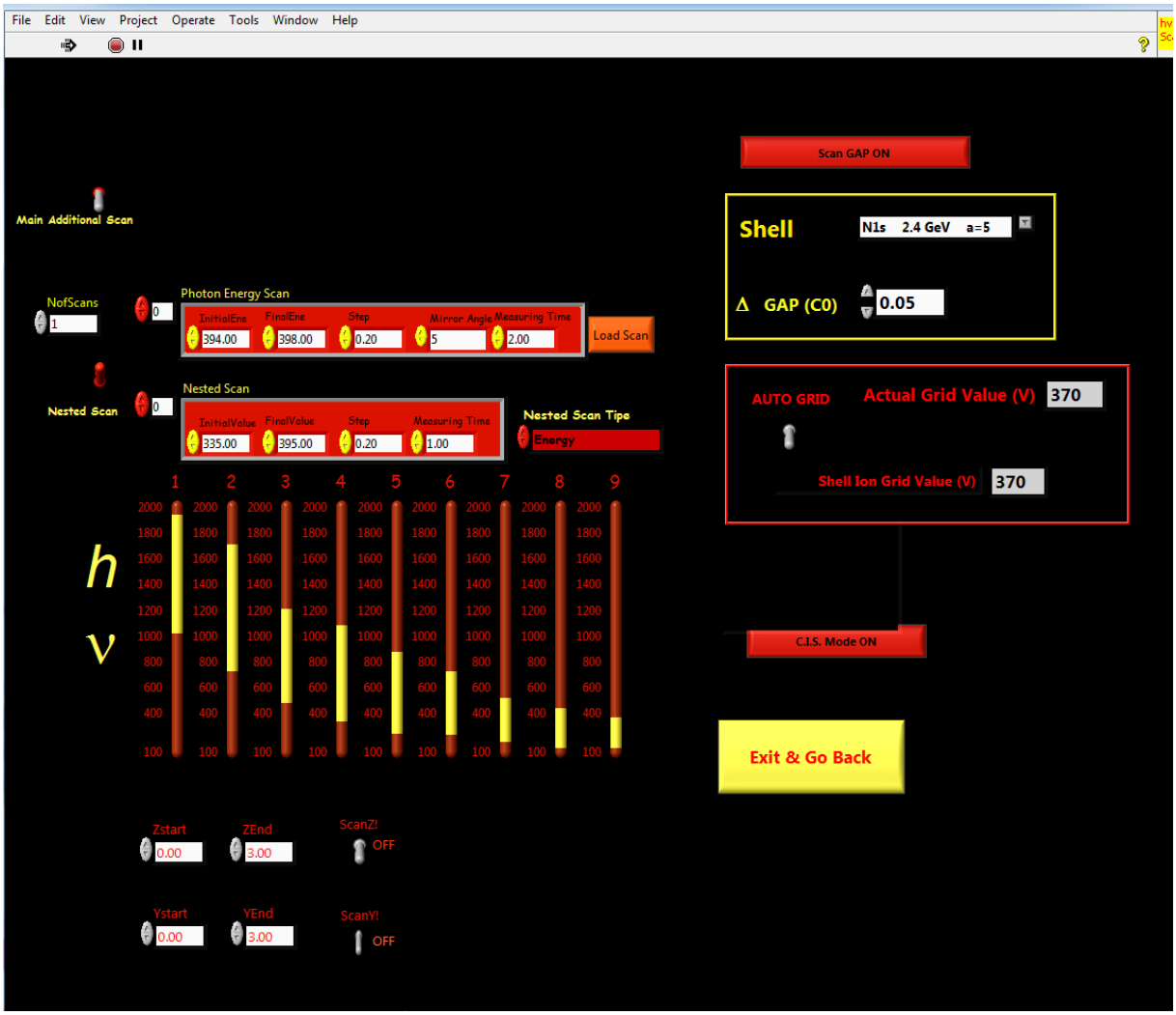

Figure 28: The NEXAFS Scan Panel of the ALOISA Acquisition Software.

5.4 Real Time Temperature-Dependent XPS (Temperature Ramp)

On the ALOISA end-station it is possible to record in real time the evolution of XPS spectra upon thermal treatment of the sample. A proportional–integral–derivative (PID) controller, in the form of a LabVIEW software, is available to perform temperature controlled experiments. With this software you can control in remote the annealing of a sample, by setting the desired temperature or performing a programmed temperature ramp. The program is on the acquisition PC's Desktop, and is named "SetSampleT.vi". The PID software is shown in fig.29.
DO NOT USE THE PID SOFTWARE FOR ANNEALING THE SAMPLE DURING THE SURFACE PREPARATION, USE THE PID ONLY FOR TEMPERATURE RAMPS.

5.4.1 Start Remote Control on sample power supply

In order to remotely control the sample heating, set the sample power supply as shown in fig.30. Run the PID software (if an error appears, click on "continue" until the software is working, otherwise close the program, switch off and then switch on again the Oxford Controller, and try again to run the program).

5.4.2 Remote control of the sample temperature

The software is divided in three sections: a "Manual Control" section (top), the "PID Control" section (middle), and the "RAMP" section (bottom). The temperature is shown on the top of the window, close to the "Exit" button. Two other buttons are available: the "Plot T" button, to plot the temperature on the top right graph, and the "reset graph" button, to clear the graph. Plotting the temperature slows down all the running softwares,

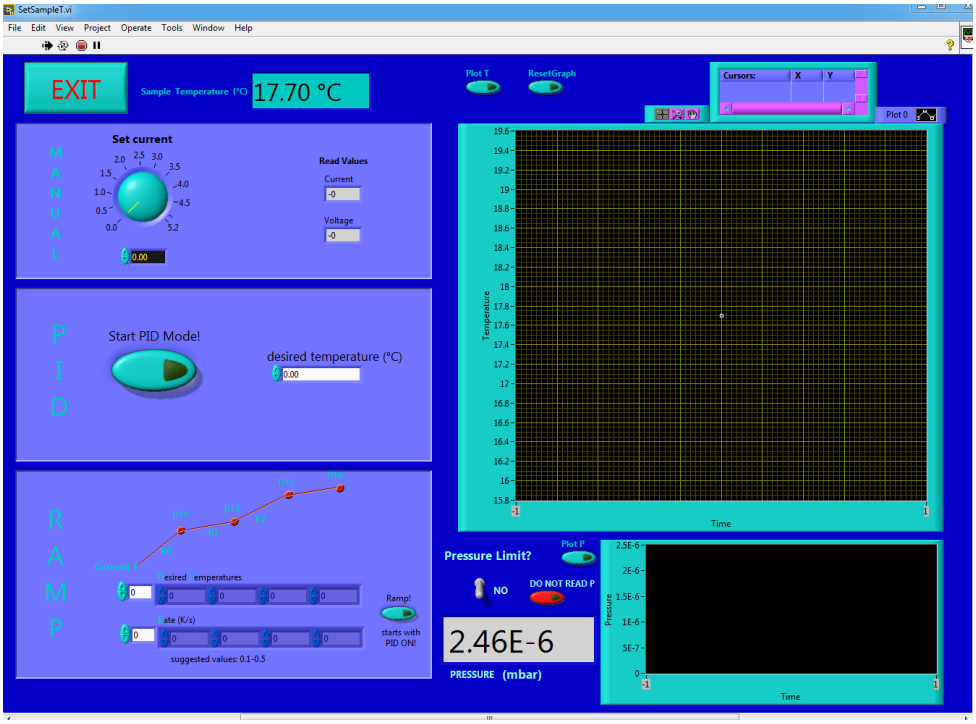

Figure 29: PID Software Panel.

so use this features only if necessary. In the "Manual Control" Section you simply control in remote the sample power supply: you can set a certain current either with the knob or writing the desired current (in Ampere) and pressing "enter" on the keyboard. DO NOT EXCEED 6 AMPERE.
In the "PID Control" section you can directly set the desired temperature (in Celsius degrees); the PID will activate as soon as you press the "Start PID Mode!" button. The software automatically adjust the filament current to stabilize the temperature at the set value. In order to avoid current overshooting, it is better to get close to the desired temperature manually, and than activate the PID. In order to stop the PID control, simply deselect the "Start PID Mode!" button- WARNING: AS SOON AS YOU STOP THE PID, THE SOFTWARE SWITCHES TO THE MANUAL CONTROL, AND SETS THE CURRENT TO THE VALUE WRITTEN IN THE MANUAL CONTROL SECTION. BE SURE THAT THIS VALUE IS SET TO 0 OR TO AN APPROPRIATE CURRENT VALUE.
On the bottom left of the software panel there are some controls related to the monitoring of the pressure. The pressure limit button, if activated, switch off the filament every time that the pressure overcome a certain value, that can be set in a hidden control on the far left of the window (use the scroll bar). The "plot P" button plots the pressure on the bottom graph. The red "Do not read P" button disables the pressure reading; monitoring the pressure is an operation that slows the all the running softwares, and hence should be used only if necessary.

5.4.3 Temperature Ramp

In the "Ramp" section, you can set the parameters of a temperature ramp. In each "box" of the "Desired Temperatures" filed, you can set the final temperature (in Celsius degrees) of your ramp; set different temperatures if you want perform the ramp in different steps. In each box of the "Rate (K/s) field you can set the temperature step in kelvin/second. Set different steps for each one of the final temperatures set in the previous field. Suggested values for the step are 0.1 - 0.5 K/s. It is recommended to set a starting temperature with the PID mode before starting the ramp. In order to start the ramp set the starting temperature in the "PID Control" section, press the "Start PID Mode" button, once you reached the starting temperature press the "Ramp!" button. THE RAMP WILL NOT WORK IF THE "START PID MODEe!" BUTTON IS OFF. In order to stop the ramp press again the "Ramp!" button.
You can measure multiple XPS in sequence while the temperature ramp is running, by setting the appropriate XPS scan parameters and temperature ramp parameters.

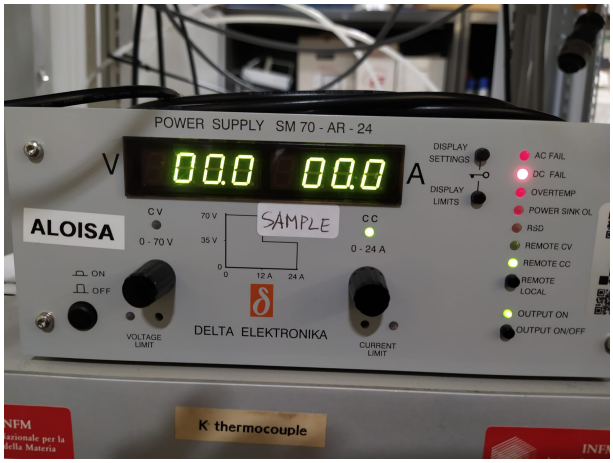

Figure 30: PID Remote Configuration of the sample power supply.

6 SAMPLE PREPARATION

6.1 Sample Holder

The Aloisa sample holder (fig. 31) is specially designed to cool or heat the sample minimizing the heat exchange with the manipulator: a combination of a copper cold finger and a sapphire disk allows to cool down the sample at the liquid nitrogen temperature, and at the same time to reach high temperatures with small heat transfer. The sample holder has 8 electrical contacts: 4 are used to read 2 Alumel-Chromel thermocouples, 2 for a Tungsten (W) filament (pins 3 and 4 on the manipulator electrical feedthrough), 2 for another W filament or to polarize the Molibdenum (Mo) plate of the sample holder (pins 1 and 2 on the manipulator electrical feedthrough).
Specifications:

1. only one sample at a time (due to grazing incidence with manipulator coaxial to the x-ray beam);
2. ideal sample: circular, Ø 10 mm (minimum 5 mm due to grazing geometry);
3. mounting: Mo clips on lateral notches (2 to 3 mm thickness), surface must be fully exposed;
4. heating: either radiative (max 850 K) or electron bombardment (max 1100 K); 2 Cr-Al thermocouples;
5. cooling: LN2 cold finger (minimum 140-150 K).

Figure 31: Sample Holder used at ALOISA.

6.2 Annealing the Sample

The sample can be heated with two different methods, namely radiative or electron bombardment, depending on the used sample holder.
The sample temperature (in Celsius degrees) is shown on the sensor 3 of the Oxford Temperature Controller on the rack next to the experimental chamber (see fig.32).
The manipulator's head is also partially heated during the annealing of the sample, increasing the contamination in the chamber, so be sure that Nitrogen gas is always flowing through the manipulator's head.

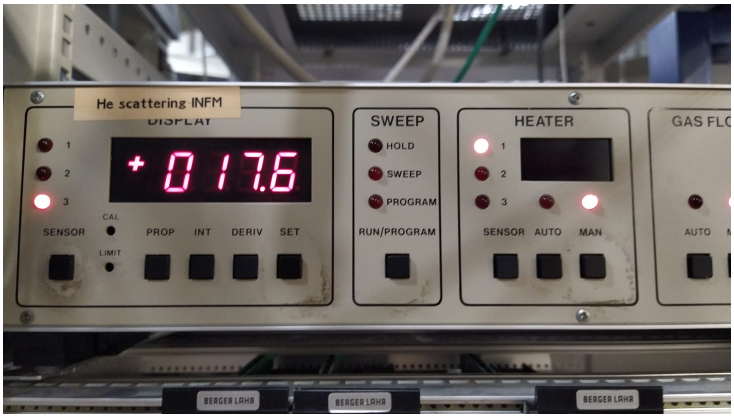

Figure 32: Sample temperature reading.

6.2.1 Radiative Heating

Radiative Sample Holders have two W (Tungsten) filaments connected in series through the 1-4 pins on the manipulator electrical feedthrough. You can use the power supply on the rack next to the chamber, shown in fig.33, to set the current of the filaments. **DO NOT EXCEED 5.5 AMPERES!**. Press the on/off button to activate the current output.

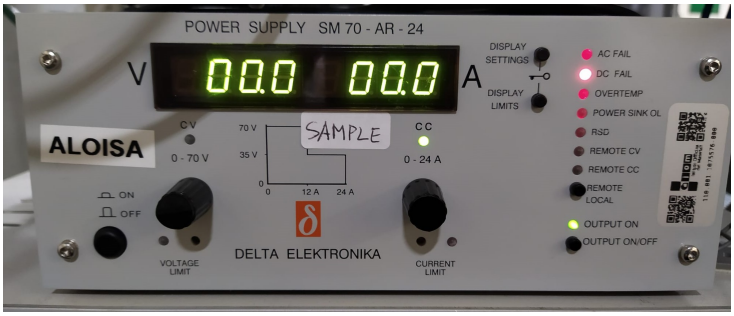

Figure 33: Sample power supply.

6.2.2 Electron Bombardment Heating

WARNING: HIGH VOLTAGE EMPLOYED. ELECTRICAL SHOCK HAZARD.

Electron Bombardment Sample Holders have a W filament connected through the 3-4 pins on the manipulator electrical feedthrough, while the 1-2 pins are used to polarized the sample holder plate. In order to anneal the sample by electron bombardment:

1. Connect the HV cable shield to the ground cable, and the HV output to either the pin 1 or pin 2 cable, as shown in fig.34. **SECURE THE NOT USED ELECTRICAL CONTACT (PIN 1 OR 2) AND THE NOT USED THERMOCOUPLE WITH AN INSULATING SHIELD, AS SHOWN IN THE FIGURE.**

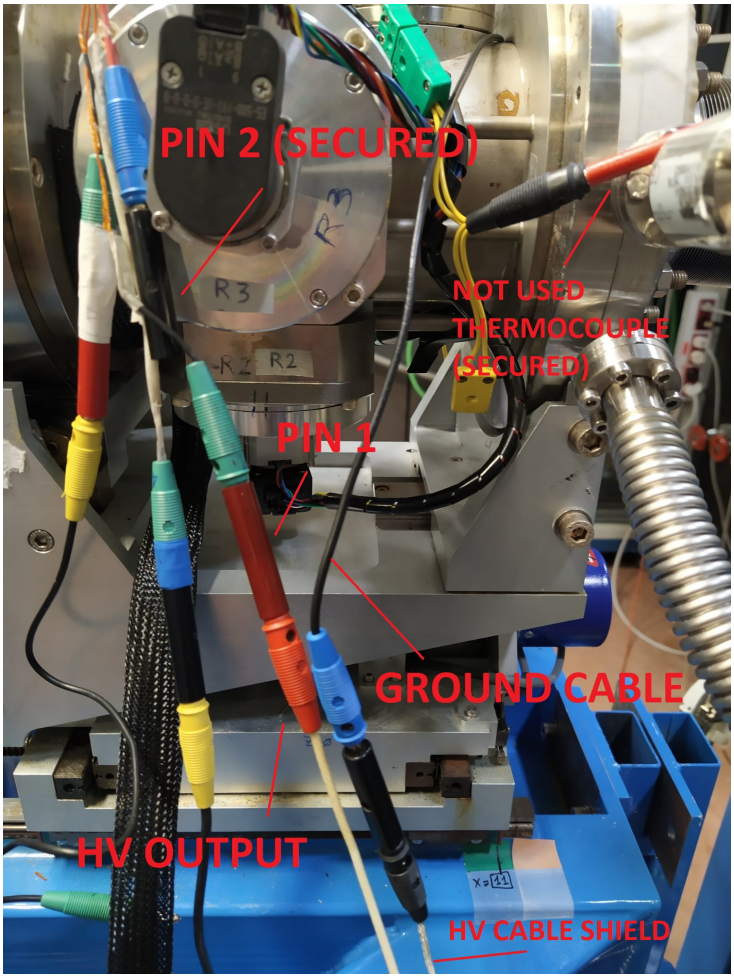

Figure 34: Electron Bombardment setup.

2. Press the on/off button on the Fug DC Power supply (see fig.35); it is pre-set to 800V. **Once the High Voltage is on, do not touch anything close to the manipulator HV cables (including the thermocouples), in order to avoid possible electrical shocks.**
3. Use the filament power supply (fig.33) to regulate the emission current (mA), shown on the right display of the Fug DC Power Supply, to reach the desired sample temperature. The emission current is limited to 60mA. Do not keep the sample at a temperature higher than 600°C for more than few minutes.
4. Once you finished the annealing, switch off the high voltage and set the filament current to zero.
5. **Connect back to ground all the cables before a XPS-NEXAFS measure or Ar sputtering.**

Figure 35: High Voltage power supply.

6.3 Argon Sputtering

In order to sputter the sample, follow this procedure:

1. Move the manipulator to the preparation chamber.
2. Set R1 to 160°.
3. Be sure that the sample is grounded.
4. **Be sure that the manual valve between the preparation and measurement chambers, as well as the toroidal valve, are closed.**
5. Using the Ion Gun leak valve (see fig.36) set a total pressure of argon of $2x10^{-6}mbar$. **Always look at the pressure monitor while opening the leak valve.**
6. Set the emission current to 4-6 mA (see fig.37).
7. Set the Beam energy on the ion Gun Controller (see fig.37): tipically 1-2kV and turn on the beam energy switch.
8. Start a timer.
9. Once the sputtering is finished, set the emission current to zero, turn off the beam energy switch, and close the Argon.

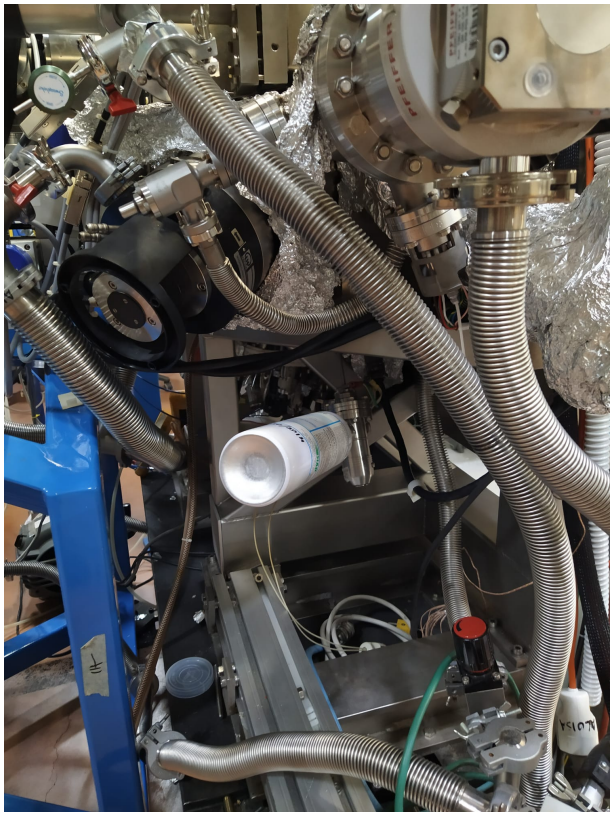

Figure 36: Ar ion Gun.

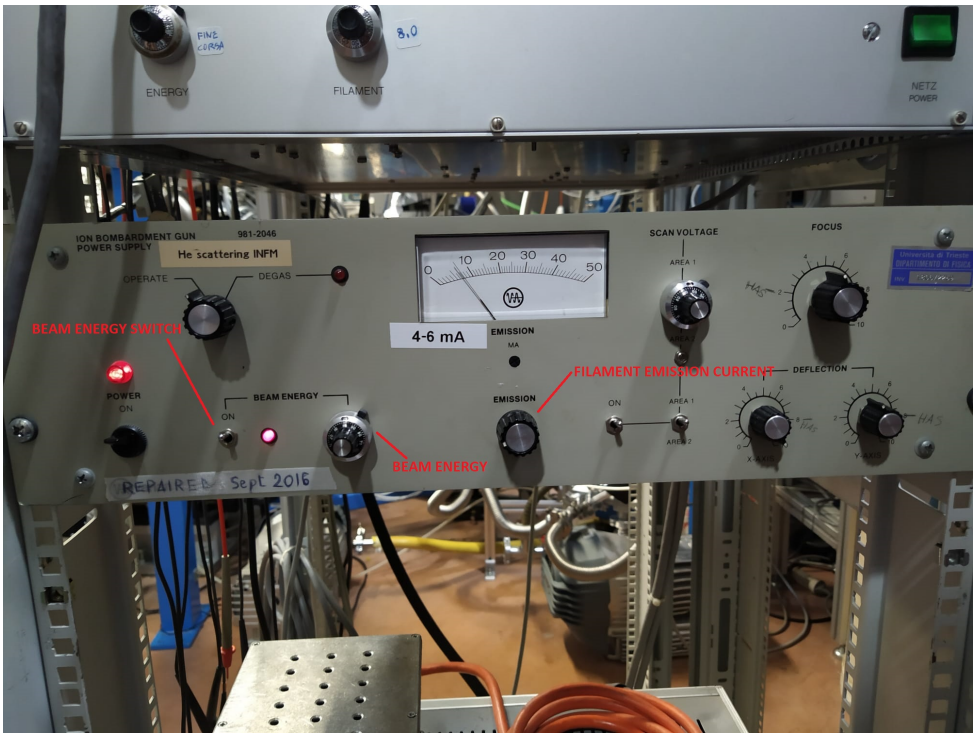

Figure 37: Ar ion Gun Controller.

6.4 Molecular Beam Epitaxy (MBE): molecules evaporation

On the ALOISA Preparation Chamber is equipped with a water cooled cryopanel with 4 slots for custom Knudsen cells, numbered from 1 to 4, each of which can contain up to two crucibles, made of either boron

nitride or quartz. For each double crucible-cell , 8 electrical contacts are used: for one crucible, 2 pins (A-A) are used for heater, 2 pins (B-B) for reading the temperature with a thermocouple, and the same for the other crucible(pins C-C for the power supply, and D-D for the thermocouple).
You can use the power supplies and the temperature reader on the rack next to the chamber (see fig.38).

Figure 38: MBE power supply and temperature reading.

Two shutters in between the cells are used to avoid cross contamination. One shatter is placed between cells 1 and 2, the other one is placed between cells 3 and for. Set the shutter C position (closed) on the opposite cell with respect to the one you are using, namely set the shutter C position on 2 if you are using the cell 1, or on 1 if you are using cell 2; the same for slots 3 and 4.
A copper disk with two holes allows the molecular effusive beam to be blocked or passeed towards the sample. On this copper disk are also mounted two quartz microbalances, namely Q1 and Q2. Depending on evaporator used, you have to select the position of the copper disk in order to read the molecular rate on the microbalance or evaporate the molecular film on the substrate. The possible MBE disk combinations are shown in table 2.

CELL	SLOT 1	SLOT 2	SLOT 3	SLOT 4
OPEN	0	90	0	90
Q1 (red)	270	0	90	180
OPEN	180	270	180	270
Q2 (blue)	90	180	270	0

Table 2: MBE POSITIONS.

6.4.1 Molecular Films Deposition

If you want to prepare a molecular film on your sample by evaporation of a molecule/metal from one of the evaporators in the MBE cryopanel, follow this procedure:

1. Move the manipulator to the preparation chamber.

2. **Be sure that the manual valve between the preparation and measurement chambers, as well as the toroidal valve, are closed.**
3. Set R1 to 160°.
4. be sure that the shutter above the cell is open (the "C" mark should be on the number of the cell next to the one containing your molecule).
5. Be sure that the MBE copper disk is in the microbalance (Q1/Q2) position for the cell containing your molecule (see tab.2).
6. Switch on the power supply controlling the evaporator and set the desired current.
7. Press the "read MicroBalance" button on the main acquisition panel. The Microbalance Panel will appear (fig.39). Here, set the molecular density (g/cm^3) of your molecule in the "rodep" field and press "Start" to monitor the evaporation rate (Angstrom/min). The rate on the sample is the 13% of the rate readed by the microbalance for the standard 21 cm boron nitride evaporator, 30% for the Omicron one.
8. Once all the parameters are stable (rate, temperature, pressure), to start the deposition, move the copper disk to an open position for the cell that you are using (see tab.2) by rotating it of 90° and start a timer [time = 1/(rate x 0.13 (or 0.3) / desired thickness)].
9. Take note of all the parameters (rate, temperature, power, evaporation time, thickness)
10. In order to stop the deposition, set the copper disk on the microbalance position, take note of the rate after the deposition and turn off the cell power supply.
11. Set back R1 to 90° .

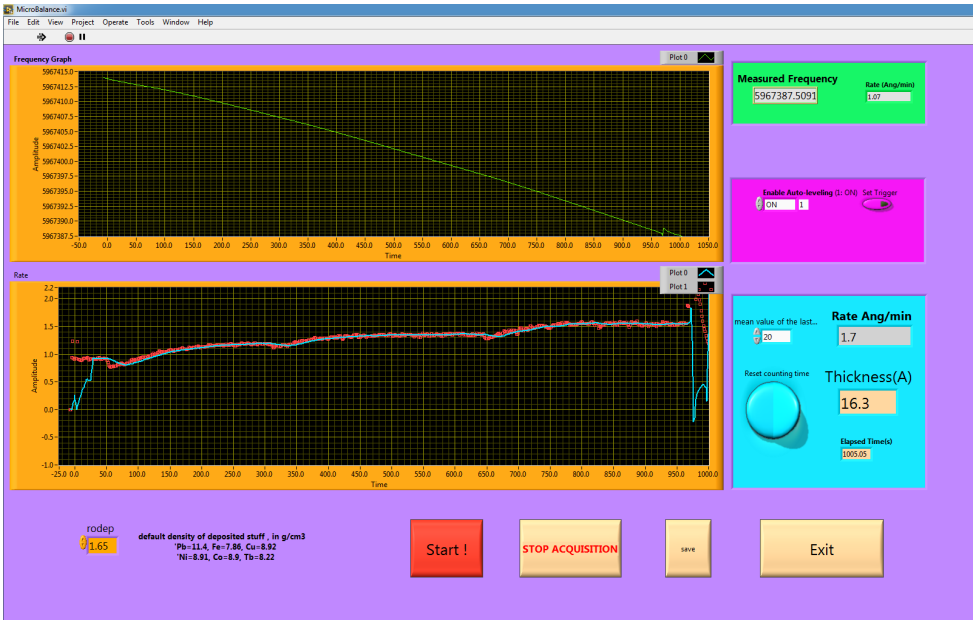

Figure 39: MBE Microbalance panel.

7 ONLINE DATA ANALYSIS

Next to the Acquisition computer, another PC is available for online data analysis. The data taken at Aloisa can be analyzed with a series of macros running on Igor Pro v6.32, that is installed on computer. If you want to analyze your data in real time, open the Igor Pro software on the analysis PC, in the top menu select Analysis → Packages → ALOISA 2.0 (v20180525). All the ALOISA's macro will open automatically, as shown in fig.40. Two windows are visible: the MCP window (left panel in the figure), used to load and plot the XPS files, and the RESPES window (right panel in the figure), to load any kind of MultiScan. You can recall this subpanels in the top menu: ALOISA Utils → Show MCP Window and ALOISA Utils → Show MCP Window or Show RESPES Window.

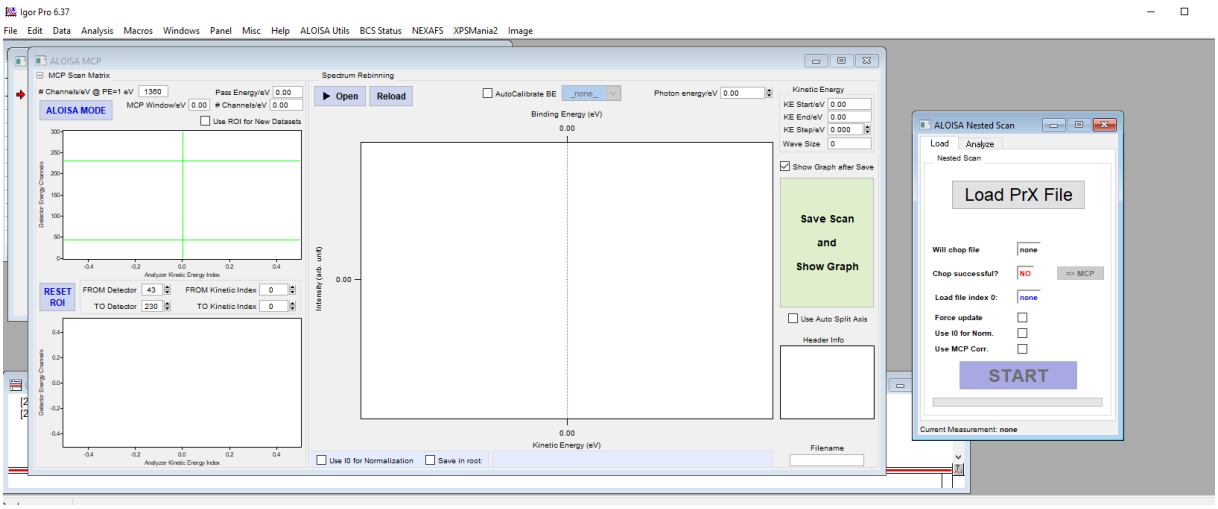

Figure 40: Aloisa's Igor Pro Macros.

7.1 Data Analysis: load a XPS

You can use the MCP subpanel to load a XPS scan, that is saved in a .prx format from the ALOISA acquisition software. To open and plot a XPS follow this procedure:

1. In the MCP Subpanel, select the "Use I0 for Normalization" check box (normilizes the scan for the corresponding photon flux) and the "Show Graph after Save" check box (plots the scan vs the binding energy). You can also select the "Use Auto Split Axis" check box (splits the binding energy axes according to each different energy region) and the "AutoCalibrate" check box (calibrates the binding energy if one of the shells in the drop down menu is recorded in the spectrum).
2. Press the "OPEN" button.
3. Select the re-binning step for your spectrum(KE Step/eV). The software automatically calculates this step according to the Pass Energy: IT IS THE RECOMMENDED ONE.
4. Press "Save and Show Graph". The graph with the loaded scan will open.

In the Igor Pro Data Browser (Data → Data Browser) a new folder called XPS is created. In this folder, a different subfolder is created for each loaded XPS file, with the same name as the loaded file. In each subfolder are stored: the XPS intensity wave (wCnts), the XPS binding energy wave (wEbin), the XPS kinetic energy wave (wEkin) and the photon flux wave (wI0MCP).

7.2 Data Analysis: load a Multi Scan

You can use the RESPES subpanel to load a Multi Scan (e.g. RESPES Scan, Temperature Ramp, etc.), that is saved in a .prx format from the ALOISA acquisition software. To open and plot a Multi Scan follow this procedure:

1. In the "Load" window of the RESPES Subpanel, press the "Load Prx File" button and load the desired .prx file.

2. Press the "Start" button. Before pressing this button you can select one of the check box options. A new folder containing each single scan of the multi scan file is created.

In the Igor Pro Data Browser (Data → Data Browser) a new folder called TXPS is created. In this folder, a different subfolder is created for each loaded multi scan file, with the same name as the loaded file. In each subfolder are stored: the 2D matrix containing all the scans (mRSPESraw), the single-scan binding energy wave (wEBinding), the single-scan kineticg energy wave (wEkinetic)), the photon flux wave (wI0), the photon energy scan wave (wEPhoton) and the single-scan temperature wave (CH7), if recorded.

7.3 Data Analysis: load a NEXAFS Scan

You can use the NEXAFS option in the top menu of Igor Pro to load a NEXAFS Scan. Two oprions are available: "Load One or More NEXAFS Files" and "Display Multiple NEXAFS Files". To open and plot a Multi Scan follow this procedure:

1. In the "NEXAFS" menu select "Load One or More NEXAFS Files" and load the desired NEXAFS file (you can load multiple files at the same time).

2. In the "NEXAFS" menu select "Display Multiple NEXAFS Files". A new window will open.

3. select from the list of NEXAFS files the ones you want to plot on the same graph and press the "GO!" button.

In the Igor Pro Data Browser (Data → Data Browser) a new folder called NEXAFS is created. In this folder, a different subfolder is created for each loaded NEXAFS file, with the same name as the loaded file. In each subfolder are stored: the NEXAFS intensity wave (py), the photon flux wave (I0), the photon energy wave (hn) and the flux-normalized intentity (pyN). The NEXAFS scan are loaded as one single wave with the p and s ploarizations one fater the other.

7.4 Data Analysis: ALOISA Package Utilities

The are some analysis features specific of the ALOISA Package. Here a list of the most commonly used features:

1. Top Igor Pro Menu → ALOISA Utils → Photoionization Cross Sections → Show Panel: A new panel will open, where tou can select ("Element" drop down menu) and plot ("Append" button) the atomic photoionization cross sections of all the most common elements.

2. On a graph, select two or more peaks close in binding energy → with the left-button make a square around the top of the peaks → roght click in the square → ALOISA Utils → Trace Manipulation → Align Traces → enter the binding energy value at wich you want to center the selected peaks: this feature is used to calibrate multiple XPS spectra plotted on the same graph with respect to a reference peak.

3. Pro Top menu → XPSMania2 → Initialize → Put the cursors in the spectral region you want fit → right click → XPS Mania2 Fit → Fit Between Cursors: a new panel (see fig.41) will open. XPSMania is a fitting tool that allows the use of diverse fitting functions and background subtraction methods. The value of the "Mode" field defines the constrictions on the fitting parameters, as shown in the figure.

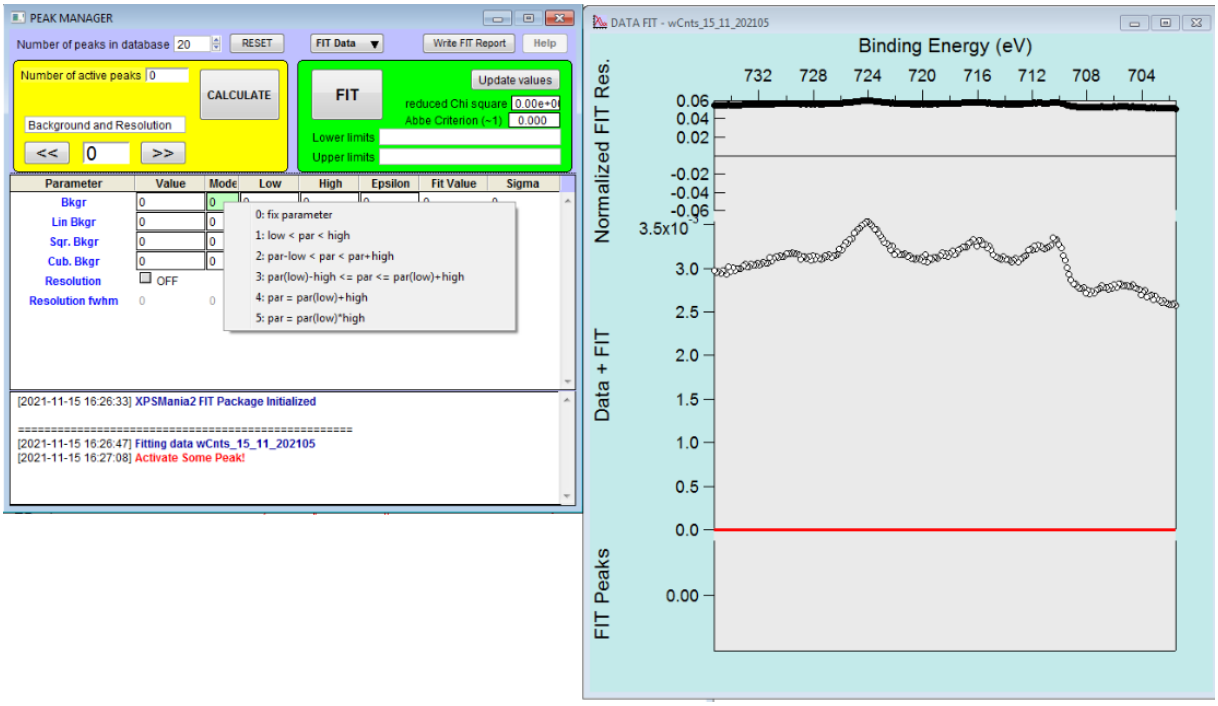

Figure 41: The XPSMania2 Panel.

8 SAFETY

8.1 Personal Safety

Working on the beamline expose you to safety hazards, like electrical shocks, radiation exposures, accidents, etc.. **Please always follow the instructions of the beamline staff, and always ask before performing any procedure.**
You can find a radiation protection and safety training on your Elettra VUO account (www.vuo.elettra.trieste.it) in the "Radiation Protection and Safety training for user" section and the at following web page: www.elettra.eu/activities/spp/safety.
General safety behaviours to prevent workplace accidents are :

- Pay attention to hurdles on the floor (free cables, spare parts, etc.).
- Do not touch energized equipment.
- Do not use water or other liquids on electrical part.
- Do not stay under hanging loads.
- Do not stay around the chamber when there are parts moving.
- Do not eat or drink on the beamline.

EMERGENCY NUMBERS (from the beamline's phones):

- Porter's Lodge (Guardiania): 8333 (to call for any serious accident, like fire, injuries, etc.)
- Fire Brigade: 0-115
- Ambulance: 0-118
- Elettra's Control Room: 8922 - 8923

8.2 Beamline Safety

Some incorrect operations may damage the beamline or the instrumentation. **Please always follow the instructions of the beamline staff, and always ask before performing any procedure.**
In particular:

- Do not transfer the manipulator if the manual valve between the two chambers is closed.
- Do not close the manual valve between the two chambers when the manipulator is inside the measurements chamber.
- Always close the manual valve between the two chambers and the toroidal valve before performing any preparation process in the preparation chamber (sputtering, annealing, depositions, etc.).
- Always watch the pressure monitor when you are introducing the Argon in the preparation chamber.
- Always follow the annealing process of a sample to avoid overheating.
- Do not open the toroidal valve or the manual valve between the two chambers if the pressure in the preparation chamber is $> 2x10^{-9}$mbar.
- Do not change grating and mirror (i.e. photon energy) if the toroidal valve is open, except during a NEXFAS-RESPES scan.
- Do not exceed the limits reported in tab.1.
- Do not exceed a pressure of $1x10^{-5}$mbar in the preparation chamber.

9 FAQ

- **There are no counts in the XPS scan.**
  - Be sure that all the valves are opened.
  - Be sure that the synchrotron beam is on.
  - Be sure that the correct photon energy is set.
  - Be sure that the correct gap value is set.
  - Be sure that the correct pass energy value is set.
  - Be sure that the correct kinetic energy ranges, steps and acquisition times are set.
  - Be sure that the Electron detector is on.
  - Be sure that R1 is set to 90°, the chamber at 0°, the Bimodal at 90°, the grazing angle (R3), the azimuth (R1), Zm, Xm, and Ym are set to the proper values.
  - Be sure that the sample is grounded.
- **I pressed "cancel" while loading an XPS Scan with the Fast Scan Selector, and an error appear.**
  - Press "Continue" until the error window disappear, and repeat the load XPS procedure.
- **There are no counts in the NEXAFS scan.**
  - Be sure that all the valves are opened.
  - Be sure that the synchrotron beam is on.
  - Be sure that you used the correct reference photon energy.
  - Be sure that you used the correct gap.
  - Be sure that you use the correct gap offset value.
  - Be sure that you use the correct yield value.
  - Be sure that the correct photon energy ranges, steps, shell, and acquisition times are set.
  - Be sure that the channeltron detector is on and that the correct grid value is set.
  - Be sure that R1 is set to 90° or 0°, the chamber at −45°, the Bimodal at 90°, the grazing angle (R3), the azimuth (R1), Zm, Xm, and Ym are set to the proper values.

10 FINAL REMARKS

The intent of this guide is to help the users of the ALOISA beamline during their experiments if they are measuring by their own. **The users are trained by the beamline staff, and they have to follow their instructions, this guide is just a reminder on the basic operations that the user should already know.**. Always ask to the beamline staff if you are not sure about the operation you are going to perform.
Good luck for your beamtime!