

Characterization of Detector and Electron Source for Inverse Photoemission Spectrometer

by

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A thesis
presented to the University of Waterloo
in fulfillment of the
thesis requirement for the degree of
Master of Science
in
Physics

Waterloo, Ontario, Canada, 2023

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Author's Declaration

I hereby declare that I am the sole author of this thesis. This is a true copy of the thesis, including any required final revisions, as accepted by my examiners.

I understand that my thesis may be made electronically available to the public.

Abstract

The inverse photo-emission spectroscopy (IPES) is a powerful tool to probe the unoccupied energy states of materials, such the surface states of metals or the unoccupied states of high temperature superconductors. IPES instruments are not commercially available, requiring the custom design and characterization of instrumentation in order to perform measurements. An IPES instrument was previously developed by our group. However, the initial design of the photon detectors was unsuccessful. In this thesis I present testing and characterization of a new generation of Geiger photon detectors for IPES measurements that we show to reliably measure photons resulting from inverse photoemission. Characterization of the photon detectors and electron gun, required for performing successful IPES measurements are presented. Finally, initial angle resolved IPES measurements on Cu (100) surfaces are presented, show good agreement with previous work.

Acknowledgements

First of all, I would like to thank my supervisor Dr David Hawthorn for his support and guidance throughout my two years Master program. I am grateful for his support not only on the project, but also for the general guidance in academia.

I would also like to thank my group members, especially Michael Bouliane and Naman Gupta, for assisting lab activities and giving advises to my results.

Last but not the least, I would like to thank all my friends in Waterloo, who have been super supportive, physically and mentally.

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List of Abbreviations

PES	Photoemission Spectroscopy
IPES	Inverse Photoemission Spectroscopy
ARIPES	Angle Resolved Inverse Photoemission Spectroscopy
FWHM	Full Width at Half Maxima

Chapter 1

Introduction

In the field of condensed matter physics, the energy, momentum and occupation of electronic states is central to key physical properties of materials, governing for instance if a material is an insulator, semiconductor or metal. Important for characterizing the electronics structure is the development of experimental tools to accurately measure the energy and momentum of electronic states for both those states that are occupied by electrons and the states that are unoccupied.

Photoemission Spectroscopy (PES) is a common method that is used to probe into the occupied energy state of materials. When the photons reach the sample at given energy, the electrons in the occupied states of the material will be excited, and if the photons have enough energy for electrons to escape from the material, the material will emit electrons.¹ By detecting the energy and momentum of the ejected electrons, the occupied energy states can be studied [6]. However, we are missing the other part – unoccupied energy states of the material.

Inverse Photoemission Spectroscopy (IPES) acts as a inverse process of photoelectric effect. Electrons with given energy will be incident on the sample. When the electrons reach the surface of the sample with kinetic energy and momentum that match one of the unoccupied energy states, then electrons will decay into the lower unoccupied energy state and emitting photons during the process[1,10]. By detecting the energy of the emitted photons, the unoccupied energy state of the material can be studied. Once we can probe into both occupied and unoccupied energy states, we can then find the band dispersion in momentum space. Then we could use this technique to study the unoccupied states

¹The photons with less energy for electrons to escape could also be absorbed if there are empty sates available, and the photon energy is at right value, but the electrons will not be detected in this case.

of materials we are interested in, such as high temperature superconductors. The band dispersion for such materials do not necessarily have electron-hole symmetry. In such case, we will need the information from the unoccupied states.

In order to perform IPES experiments, a custom instrument for this technique must be developed. An IPES spectrometer was designed and built by David Hawthorn and Christopher McMahon.[1] However, neither the detectors nor the electron gun operation parameters have been fully characterized, and no IPES experiments have been performed prior to the start of this project. In order to obtain the energy – momentum dispersion plot, we will need both detectors, as well as the electron gun to work properly. There are variety of parameters we could manipulate for both photon detectors and electron gun. In this thesis we present characterization of both the detectors and the electron gun, the results of which are shown in chapter 4.

To detect the emitted photons, we will be using Geiger-Muller gas filled detectors [1]. The detector is a cylindrical shaped tube, with a thin, long wire in the middle acting as the anode. The wall acts as cathode, and a high voltage can be applied between the wire and the wall. When operating, the tube will be filled with detection gas to a desired pressure. Once the photons get into the GM tube, they will collide and ionize the gas particle, producing ion pairs. Then the charge particle will be drifting towards the anode wire by the high voltage, then the electric signal can be picked up by electronics and could be measured. One of the objective for this experiment is to find a combination of anode voltage and gas pressure that maximize the signal counting rate, while keep the background signals at low level. Details are provided in chapter 4. We found a stable range of pressures and anode voltages providing acceptable count rate. Moreover, unlike ref[4] we did not find an optimal pressure and voltage setting where counts are maximized below the breakdown voltage of the detectors.

After characterizing detectors, we also characterized the electron gun. The electron gun has multiple parameters that can be varied (Energy, filament, extraction, grid, focus 1 and 2). The objective of characterization are to keep the size of the electron beam to be as small as possible. This is because the size of cuprate superconductor samples are generally small of order 2mm x 2mm in size. If the beam size is too large then when the beam hits the sample holder, it could induce background counts. While keeping the beam size small, we also need a large sample current, so that the photon signals are distinguishable from the background counts. We also want a high energy resolution with ΔE_{gun} to be within 200 meV, and a low beam divergence in order to have high momentum resolution. The characterization of electron gun is described in details in chapter 4.

We did a characterization for $E_{gun}=10$ and 15V, and have obtained beam spot sizes of

2.3mm and 2.5mm, while keeping few μA of sample current. Since we will use Cu sample, which has a larger size than beam size, we will be using the parameters we found to do the IPES scan. The energy resolution is found to be around 120 meV, and the momentum resolution is found to be around $10^{-2} \text{\AA}^{-1}$, two orders of magnitude smaller than FBZ of Cu.

We used the optimized setting for $E_{gun}=10V$ to perform ?? on Cu(100) sample. By rotating the sample, we can change the angle between the sample plane and the direction of the electron beam, thus changing the horizontal component of the momentum. We have successfully obtained an energy-momentum plot. The result is shown in chapter 6. A visible band dispersion for B_1 bulk state is present, and the result agrees with A. Mark [3] and L.Duffin[2]. This result demonstrates successful operation of the IPES instrument. We could apply this technique to materials that are not being well studied in the future experiments.

Chapter 2

Background

2.1 IPES

Inverse Photoemission Spectroscopy(IPES), in some sense, can be understood as a reverse process of Photoemission Spectroscopy(PES). In PES, photons are used to hit the surface of the sample, and electrons at the occupied states of the sample are likely to absorb those photons, getting ejected from the sample and can be detected. By detecting the energy of emitted electrons, the energy level of occupied states can be studied. In IPES, electrons are emitted from the source. Once the electrons reach the surface of the sample, their energies are conserved as they couple with a quantum state above vacuum state. Since the electrons are at a high energy state they are energetically unstable, which means they will likely to decay into the lowest unoccupied energy state and release photons.[10] The process can be expressed using following equation:

$$E_s = E_k - \hbar\omega_0 \quad (2.1)$$

Where E_s and E_k correspond to the energy state of the material, and the kinetic energy of electrons, ω_0 corresponds to the frequency of emitted photons. The process is also shown in fig.(2.1). Those photons can then be detected using Geiger-Muller detecting tubes (Described in Chapter 3). Once the energy of the photons are determined, and with known energy of incoming electron beam, the information for the unoccupied energy state can be studied.

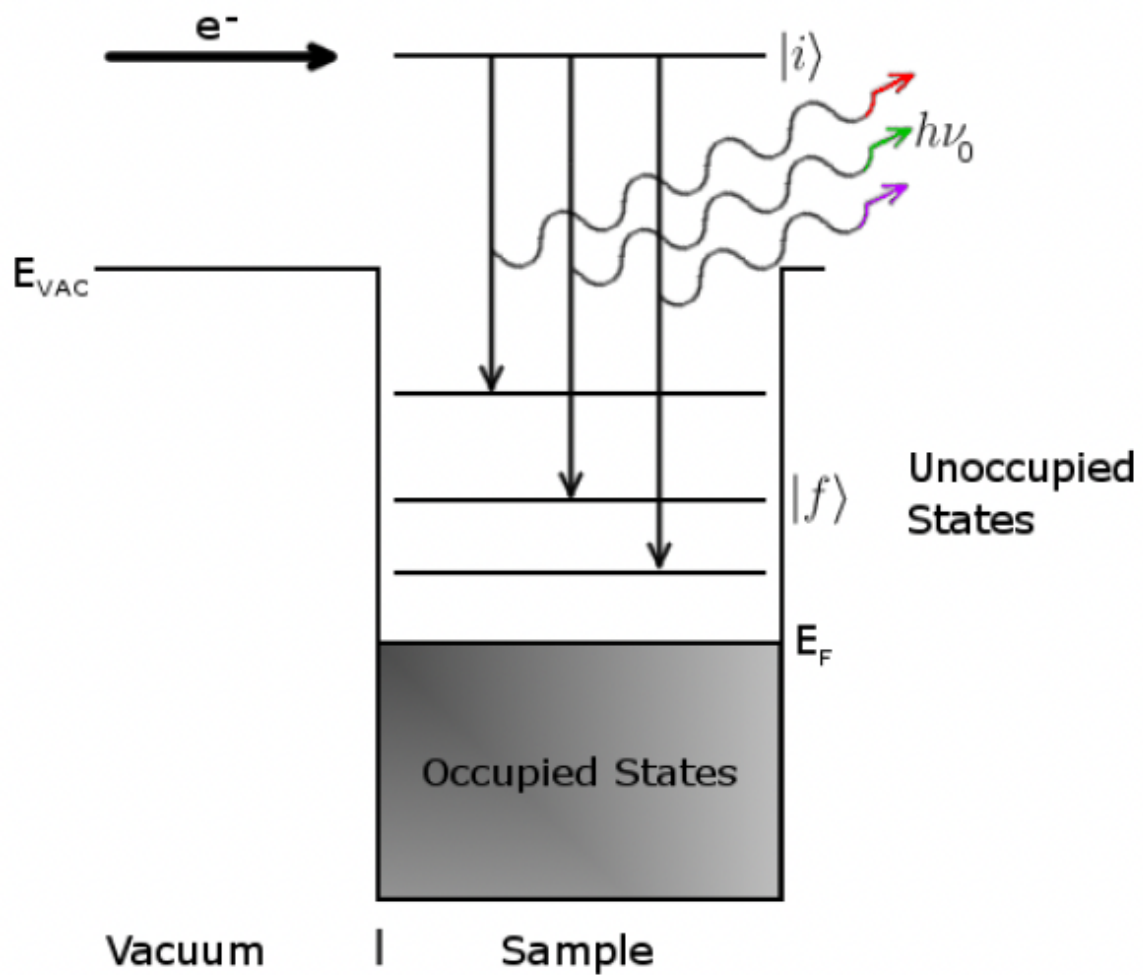


Figure 2.1: IPES process. Electrons couple with the initial state on sample surface, decaying into some lower energy states above Fermi energy and release photons. Figure retrieved from [1] with permission.

2.2 Detecting Mode

There are two ways where the energy of emitted photons can be detected. The first way is to keep the incidental beam energy unchanged, and measure all the emitted photon energy. This method is called spectrograph mode. The alternative way will be keep the detecting photon energy fixed and vary the incidental electron beam energy. In this way of detecting photons, only one photon energy is detected, and whenever the detected photons carries energy that satisfies equation (1), a signal pulse can be detected. The latter method is refereed as isochromat mode. A more detailed discussion about spectrograph mode can be found elsewhere[10].

In our lab, we will be using isochromat mode to conduct the entire experiment. The main reason is that for spectrograph mode, multiple detectors are required to be spread along the plane of diffraction. Since the entire experiment is conducted under high vacuum environment, it is technically challenging and also costly to use spectrograph mode in vacuum chamber. By comparison, isochromat mode only require one detectors for specific photon energy, which is simpler and cheaper to implement in our vacuum chamber. Each detector consists an entrance window and detection gas. Fig.(2.2) shows the basic structure of this detector. For left tube the material for the entrance window is CaF_2 , while for right tube is MgF_2 . The entrance window acts like a low-pass filter, which means photons with energy higher than a specific value cannot penetrate through. The difference in material for entrance window causes the energy cut-off for the entering photon to be different, hence make a difference in energy resolution. In our experiment, two entrance windows made of different materials are used, CaF_2 and MgF_2 . For CaF_2 , the cut-off energy is around 10.1eV, which means any photon with energy higher than this value will be absorbed by the window[2]; for MgF_2 , the cut-off energy is around 10.8 eV [19]. The detection gas is acetone, which has an ionization energy of 9.7eV. The detection gas acts as a high-pass filter, which means only photons with energy higher than the ionization energy of acetone can ionize the gas, hence produce a pair of charged particles, which can be detected by detector. The entrance window and detection gas function together as a band-filter, which only allow photon with narrow range of energy to pass through. ¹ The expected energy resolution for acetone and CaF_2 window band-pass filter is 300–400 meV, for MgF_2 window is 800 meV to 1.1 eV.

¹The cut off energy for both windows are not constant. There are slightly depended on temperature, thickness and purity. Ref[2] describes more detailed information.

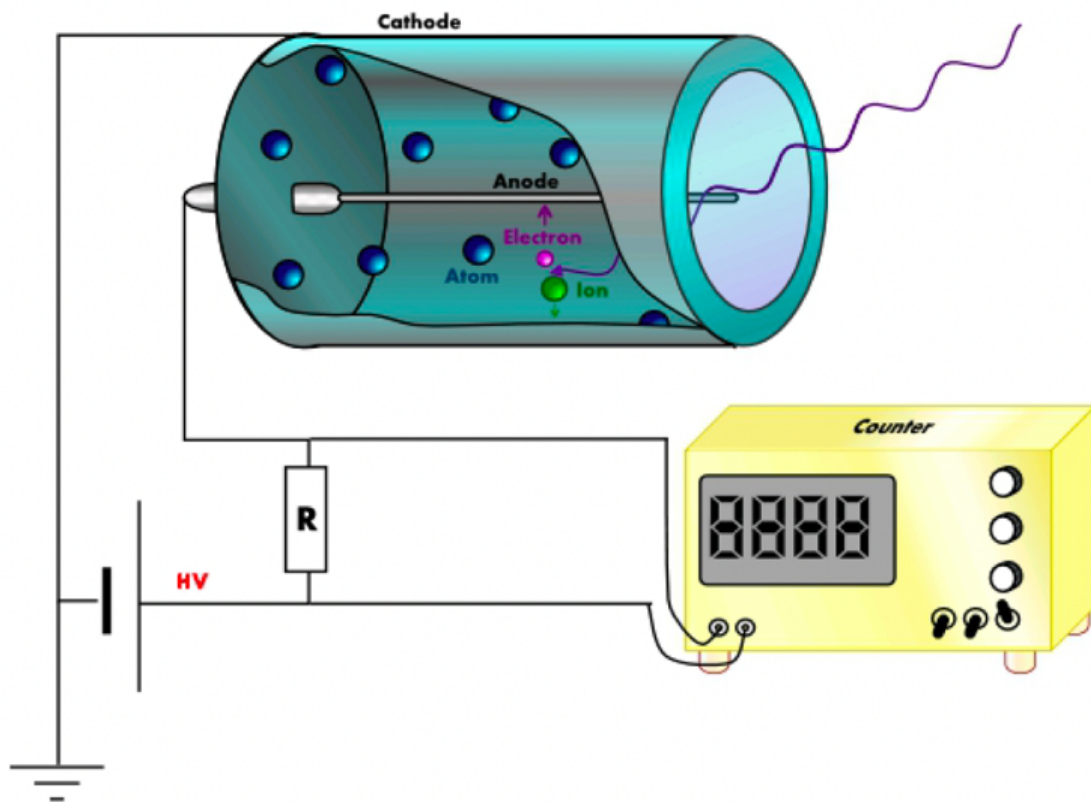


Figure 2.2: GM Tube image. Graphics adapted from [20].

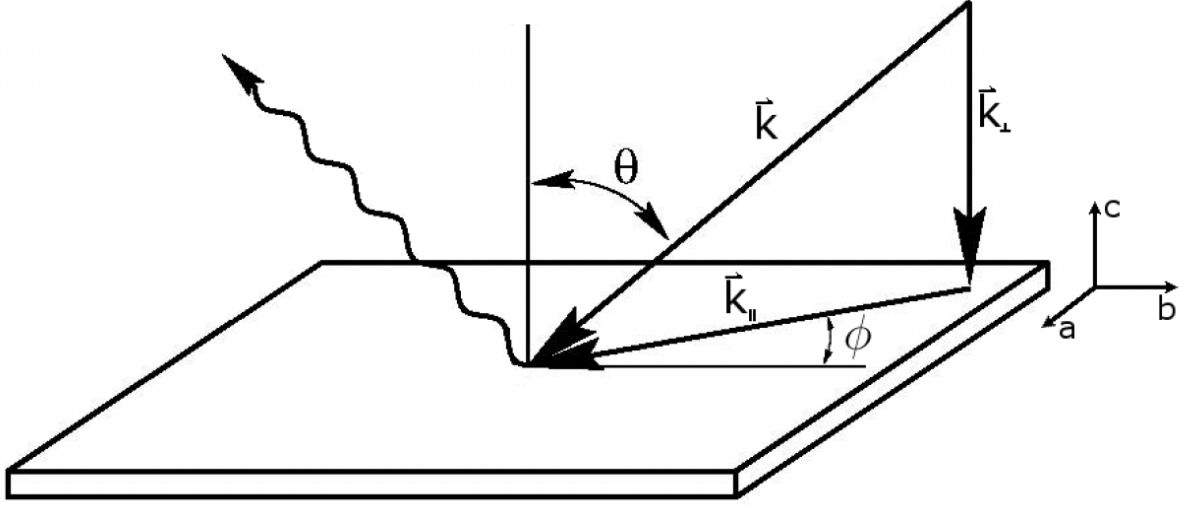


Figure 2.3: Schematic of an ARPES experimental setup. The momentum of incident electrons can be separated into parallel and perpendicular direction. Drawing adapted from ref [12].

2.3 Angle Resolved IPES

For the 2-D material, the electronic band structure does not only depend on energy, but also on momentum. Therefore it is necessary for us to map out the momentum of incoming beam as well. Due to the translation symmetry along the parallel direction of the sample, the horizontal component, $k_{\parallel}$, serves as a good quantum number. For electrons, during the transition from initial state to final state, the horizontal component of momentum is conserved², so that electrons at the final state must have the same horizontal component for momentum. Fig.(2.3) shows how the horizontal momentum is being controlled. [1] The momentum of incident electron beam is given by

$$k = \frac{\sqrt{2mE_k}}{\hbar} \quad (2.2)$$

²The momentum is conserved for electrons and photons actually, but for electrons with energy around 10 eV, the photon momentum can be negligible, so the initial momentum of electrons are set to be the same as final momentum of electrons.

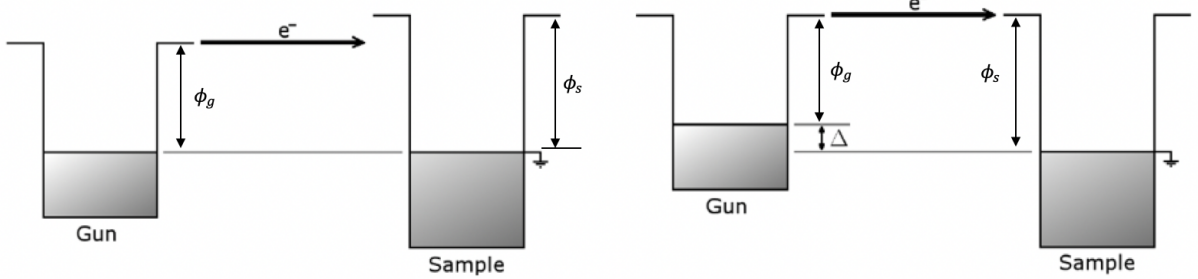


Figure 2.4: Δ is the contact potential, the minimum energy required for electrons to overcome the energy barrier. Figure adapted from [1] with permission. Left: Electrons cannot travel from cathode to the sample because they do not have sufficient energy to overcome the barrier. Right: Electrons could reach the sample from electron gun cathode.

where E_k is the kinetic energy of electrons. And the horizontal component of momentum is given by

$$k_{\parallel} = \frac{\sqrt{2mE_k}}{\hbar} \sin\theta \quad (2.3)$$

Thus by rotating the sample, we could adjust theta, hence control the horizontal component of electron beam. In our lab it is possible to accomplish that by using the motor to rotate the sample, hence change the angle between incoming electron beam and the sample plane. This way of mapping out the momentum space is referred as Angle-Resolved Inverse Photoemission Spectroscopy, or ARIPES.[1] The perpendicular component of momentum, $k_{\perp}$, is not necessarily conserved. This is due to the limitation on electron penetration depth (on order of 10 Å. [17]), thus leaves a limited translation symmetry.

2.4 Contact Potential

The electron gun is capable to give initial kinetic energy to the electrons, and the value is equal to the voltage supply multiplies by the charge of electrons. While the energy given by electron gun can be controlled, the initial kinetic energy of electrons is not exactly the same as the energy given by electron gun, but with the value less by contact potential of the sample-cathode system.

Once the sample and electron gun cathode are grounded to the same reference, they will have the same Fermi level once thermodynamic equilibrium is reached[1]. When cathode

is heated up, it will thermionically emit electrons with energy equal to its work function ϕ_g . If the sample has a higher work function, ϕ_s , than the cathode, then in order for the electrons to overcome the energy barrier, a minimum energy is required, and the value is equal to Δ .

$$\Delta = (\phi_s - \phi_g)e \quad (2.4)$$

It is necessary to determine E_k just before electrons hit the sample

$$E_k = E_e - \Delta \quad (2.5)$$

Where E_e is the initial energy of the electron given by electron source. Since the exact values of the work function of sample and cathode are unknown, we will need to experimentally determine Δ . This is accomplished by vary the bias voltage between the sample and electron cathode.

Chapter 3

Instrumentation

In this section we will introduce the set up for the experiment. The experiment contains multiple components, which includes sample and sample holder, vacuum chambers, electron gun and detectors. In this section, we will describe the apparatus being used in this experiment.

3.1 Vacuum Chamber

The entire experiment is conducted in a high vacuum environment. There are three vacuum chambers being used in this experiment: load lock, preparation chamber and main chamber. A 3D model is shown in fig.(3.1).

3.1.1 Load lock

Load lock is a small part of vacuum chamber where samples can enter the vacuum chamber, and a place to temporarily store the samples, as shown in fig.(3.2 a) and (3.2 b) . The holder can move along the arm, towards the preparation chamber, hence transfer the sample to preparation chamber for next step. There is a valve between load lock and preparation chamber, so whenever we need to vent down the load lock to take sample out or insert sample, the vacuum condition for preparation chamber will not be affected.

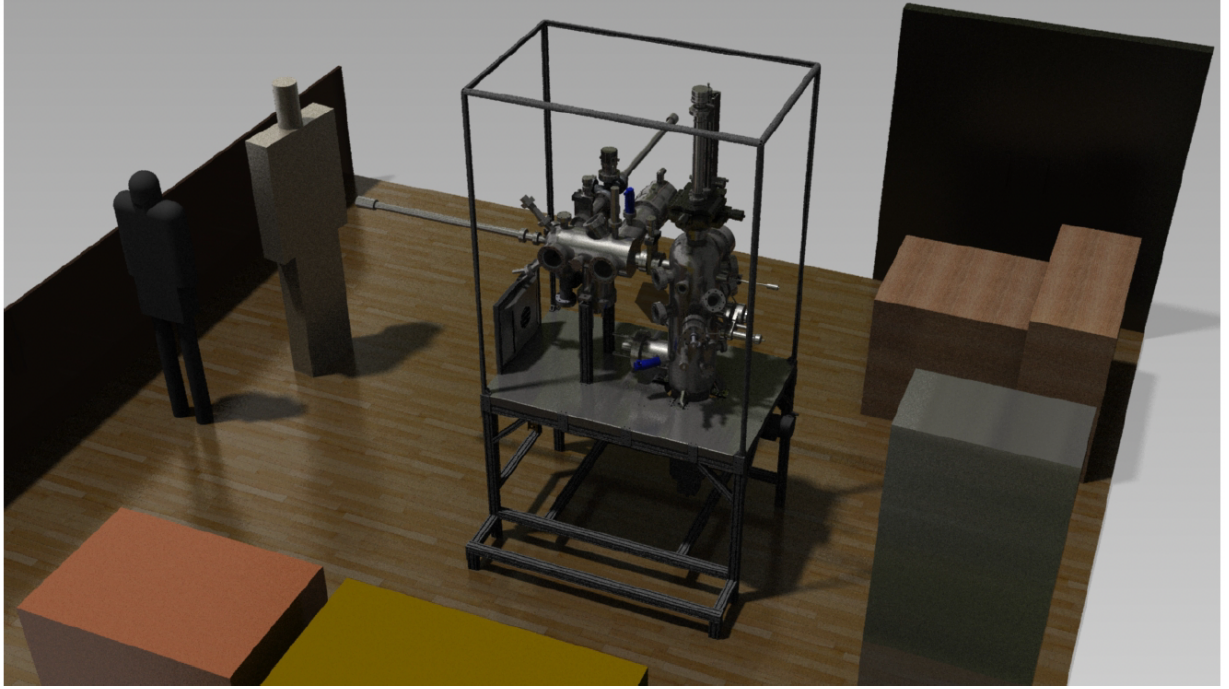
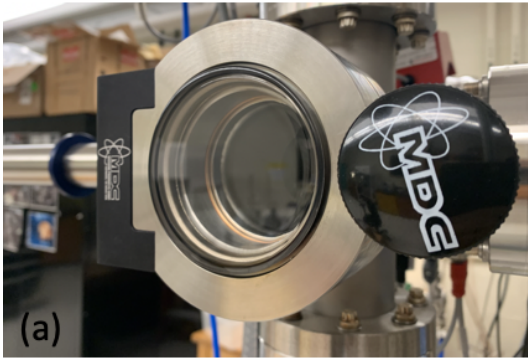
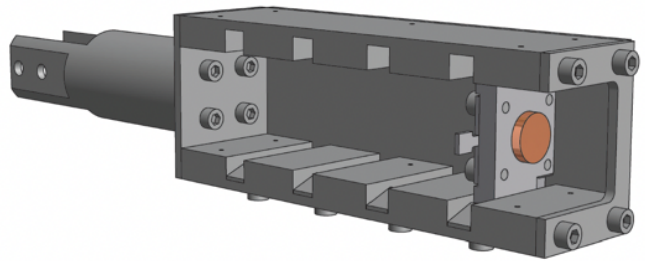


Figure 3.1: A three-dimensional scale model of the IPES spectrometer in our laboratory. Note the size relative to the experimenters on the left. Figure adapted from [1] with permission.



(a)



(b)

Figure 3.2: (a) Sample entrance window. This is where we insert the sample and sample holder into the vacuum chamber. (b) This is how sample garage stores samples. The garage could store up to 4 samples at the same time. Figure adapted from ref[1] with permission.

3.1.2 Preparation Chamber

Fig(3.3 c) shows the basic structure of preparation chamber. Before the sample can be transferred to the main chamber for IPES experiment, it requires proper cleaning on the surface, and the cleaning process is completed in the preparation chamber. Inside the preparation chamber there is a heater stage for holding the sample. In order to be able to transfer the sample from load-lock garage to the heater stage, a sample transfer arm is used. The pliers can grab the sample holder tab, pulling it out of the garage and place it onto the heater stage. The process of cleaning the sample surface will be described in chapter 5. Once the cleaning process is complete, the sample is ready to be transferred to main chamber for experiment. To transfer the sample to the main chamber, using the transfer arm pliers to grab the sample holder tab, open the valve between preparation chamber and main chamber, then pushing the arm towards the main chamber.

3.1.3 Main Chamber

Inside the main chamber, it is the place where all the measurements are performed. After sample being transferred from preparation chamber, the sample will be stored into the garage, which is connected to a motor system. The motor could adjust the position of the garage, as well as the rotational angle. Two detecting tubes are located on each side of main chamber, and the electron gun is pointing towards the sample.

3.2 Detector

3.2.1 GM Tubes

The signal analyzing process is illustrated in fig.(3.4). In this lab two gas-filled Geiger Muller Tube with different entrance window materials are used as detectors. Inside the detector, there is a long, straight wire acting as the anode, while the metallic outer tube acting as the cathode. High voltage can be applied between the wire and the outside cylindrical tube. The ionized electrons will then be drifted towards the cathode wire, hence the detector can pick up those electron signals.

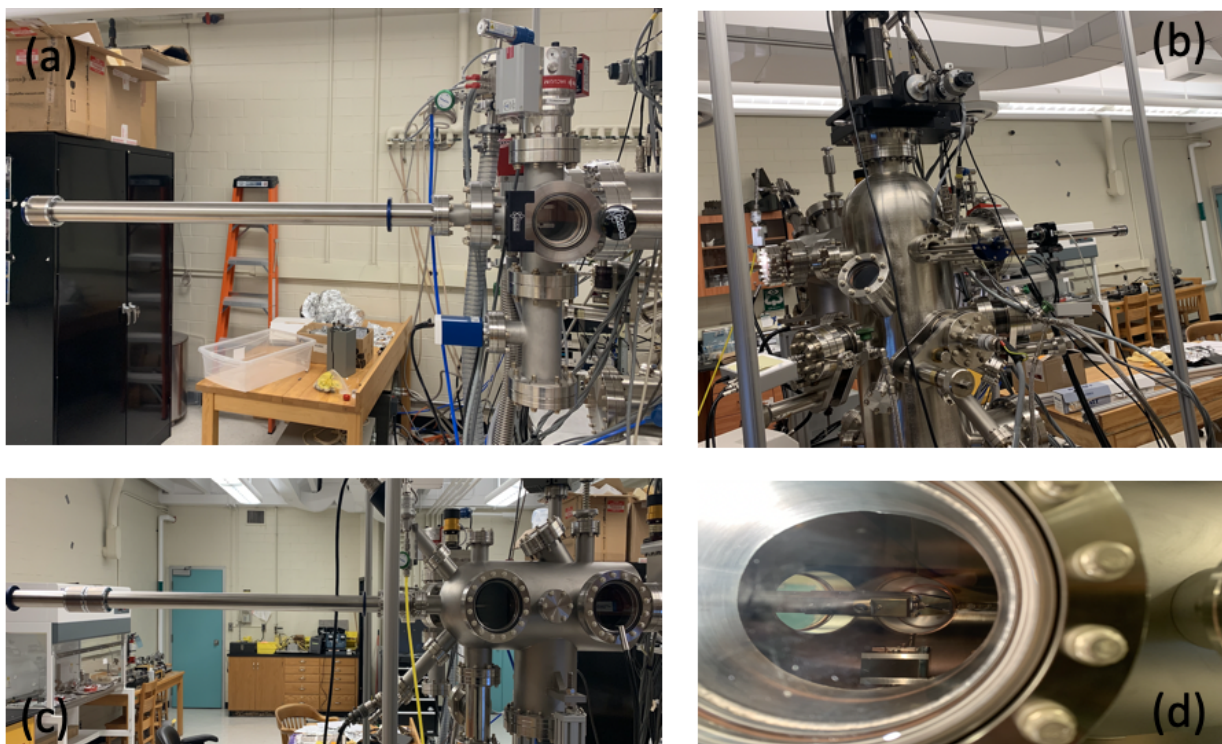


Figure 3.3: IPES instruments: (a) Load lock. (b) Main chamber. (c) Preparation chamber. (d) Sample transfer arm grabber.

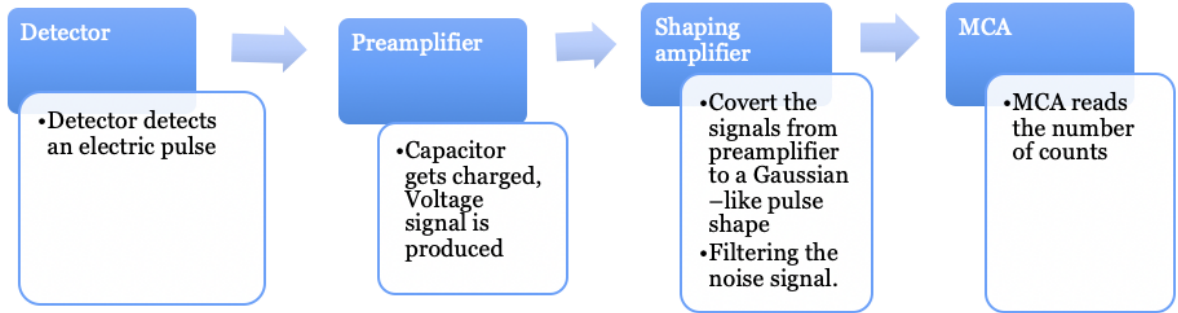


Figure 3.4: The process of converting the photon signal to counts. Detector sends the current signal to pre-amplifier, then the current signal is converted into voltage signal, which goes into shaping amplifier, then the MCA program picks up the signal coming from shaping amplifier.

3.2.2 Electronics

Once the positive ions reaches the anode wire, it will charge up the capacitor installed in pre-amplifier board. Then the voltage signals of the capacitor are measured, produces a voltage signal step function, and then be displayed on the oscilloscope. During this experiment we will be using Cremat CR-111 board for pre-amplifier.[11]

Those voltage signals, however, are not ideal to be detected as pulse signals. This is because it takes significant amount of time for capacitor to discharge comparing to the time interval between two pulses. If the electric pulses are being produced at a high frequency, the capacitor could be charged up again before being discharged from previous pulse, hence making it difficult to count the number of pulses. In order to solve this issue, a shaping amplifier is used to convert the voltage pulse signal to a Gaussian like pulse, and filtering out noise signals at the same time. In this experiment we used Cremat CR-200-500 shaping amplifier module. Electronic details could be found on their website.[12] Both output from pre-amplifier and shaping amplifier are shown in fig.(3.5).

The signals coming out of the shaping amplifier will then be measured by a multi channel analyzer (MCA). This software for the MCA has many functions, while the key functions that we used in the experiments are counting the number of pulses and displaying the pulse height distributions. We could set a threshold value for each channel, so that only voltage signals higher than this threshold value will be counted towards the signal pulses, hence filtering out the background signals.

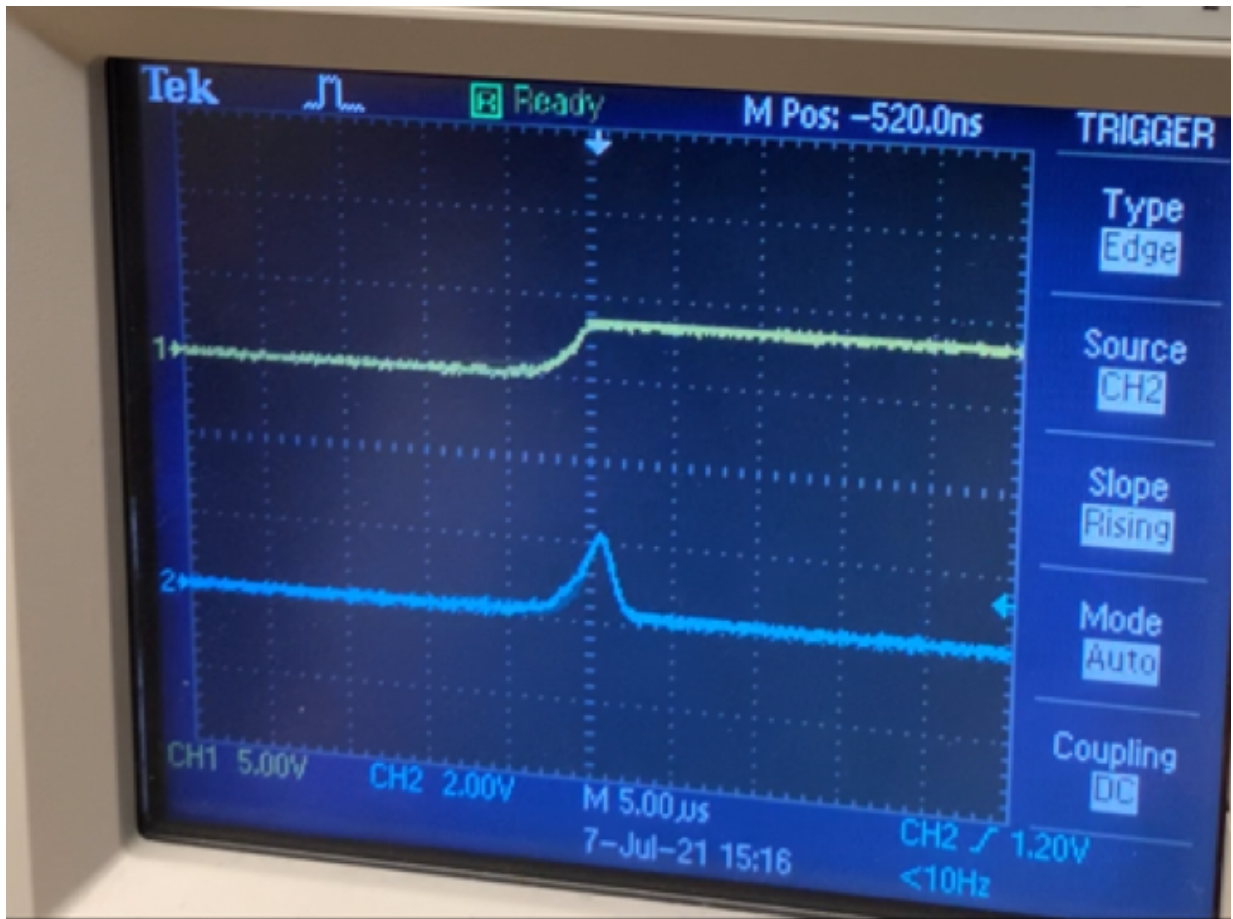


Figure 3.5: Oscilloscope screen showing the detection of charge pulses from the detectors. The yellow signal is the output from pre-amplifier, showing a step like increase in voltage with a long tail decay; the blue signal is the output from shaping amplifier, showing a Gaussian like peak.

3.3 Electron Gun

The electron gun is the source of electrons, and it is capable to control the energy of released electron. There are several parameters that can be adjusted(filament, extraction, grid, focus 1 and 2) in order to improve the quality of scans. Details will be provided in chapter 4.

Chapter 4

Characterization

As mentioned in introduction section, we have not found a working conditions for detectors and electron gun. In this section the process of characterizing GM tube detectors and the electron gun is presented, as well as the optimizing operation conditions for both detectors and electron gun. After transferring the sample to the main chamber, we could proceed to the characterize the detectors.

4.1 Detector Characterization

A basic description for GM tube has been discussed in previous section. During the experiment, we require the detector to be operating at voltages below the breakdown voltage of the detector, but also at a voltage high enough that pulse heights are larger than the background noise. The breakdown voltage corresponds to the voltage where the detection gas will start to be ionized by the high voltage even in the absence of incident photons. The electrons from the ionized gas will be accelerated towards the anode, setting off an avalanche and resulting in charge pulses that are unrelated to the IPES process. The value of breakdown voltage can depend on the gas pressure, the tube design, and the configuration of the electronics. In order to perform IPES measurements, we need to characterize the breakdown voltage and operate the detectors below this voltage.

Besides breakdown, there are other sources of background noise signals. For example, vibration of the anode wire can induce changes in the capacitance of the detector, leading to oscillations or pulses in the charge between the anode and the cathode. The amplitude of these pulses noise is dependent on the anode voltages. It is important that the pulses

originating from photon signals be distinguishable from background signals. [4] describes details of GM tube characterization.

In addition to characterizing the noise and breakdown of the detectors, we also need to characterize the mode of operation of the detector, specifically in the Geiger mode or proportional counting mode (See figure 4.1). In Geiger mode, the detection of a pulse fully ionizes the gas in the detector, leading to pulse heights that depend on gas pressure, but are otherwise constant and independent of anode voltage. However, this mode has a large dead time in order for the gas to charge neutralize before it can detect another pulse. In contrast, the proportional mode, only partially ionizes the gas, with the average charge per detection dependent linearly proportional to anode voltage. Moreover, there is a distribution of pulse heights. Proportional counting mode has smaller dead-time than Geiger mode.

For this project, acetone GM detectors without a quench gas are expected to operate in proportional counting mode. [8] Here we characterize if this is indeed the case. We vary the anode voltage and measure the rate of signal counts that have a voltage exceeding a threshold voltage. If the distribution follows an exponential decay distribution, then we can conclude that the detector is operating within proportional counting region rather than the Geiger mode. [8]

Before we perform the detector characterization, the following electron gun parameters are used: Filament = 5V, extraction = 30V, $E_n = 15V$ (and $E=0$ for background). For each scanning cycle, we first filled the GM tubes to a desired pressure, closed the acetone gas valve, then turned on the high voltage power supply, and set the voltage to 300V. For each value of the anode voltage and acetone pressure, we count for 30s the number of pulses that have a voltage exceeding a fixed threshold voltage (50 mV). By doing this we could filter out some of the noise with low amplitude that could trigger false counts. For each pressure and voltage, we count the pulses with and without the electron gun on. In this way we differentiate pulses from noise and breakdown from pulses that originate from photons. The counts vs pressure and anode voltage are shown in fig.(4.2), with background counts subtracted. The white regions in fig.(4.2) indicate the onset of breakdown of the detectors. In fig. (4.3) we plot the breakdown voltage vs acetone pressure for the MgF_2 (GM1) and CaF_2 (GM2) detectors. We could conclude from fig.(4.3) that after 3 torr, the breakdown voltage does not change significantly with pressure, indicating that the performance of the detectors are not highly sensitive to the precise value of acetone pressure in this pressure range and stable operation of the detectors is achievable. From the fig. (4.3) we note that the number of count rate from GM 2(CaF_2 window) is higher than GM 1 (MgF_2 window) and that the detectors have different breakdown voltages. This difference is attributed to differences in the electrical feedthrough and anode assemblies of the two

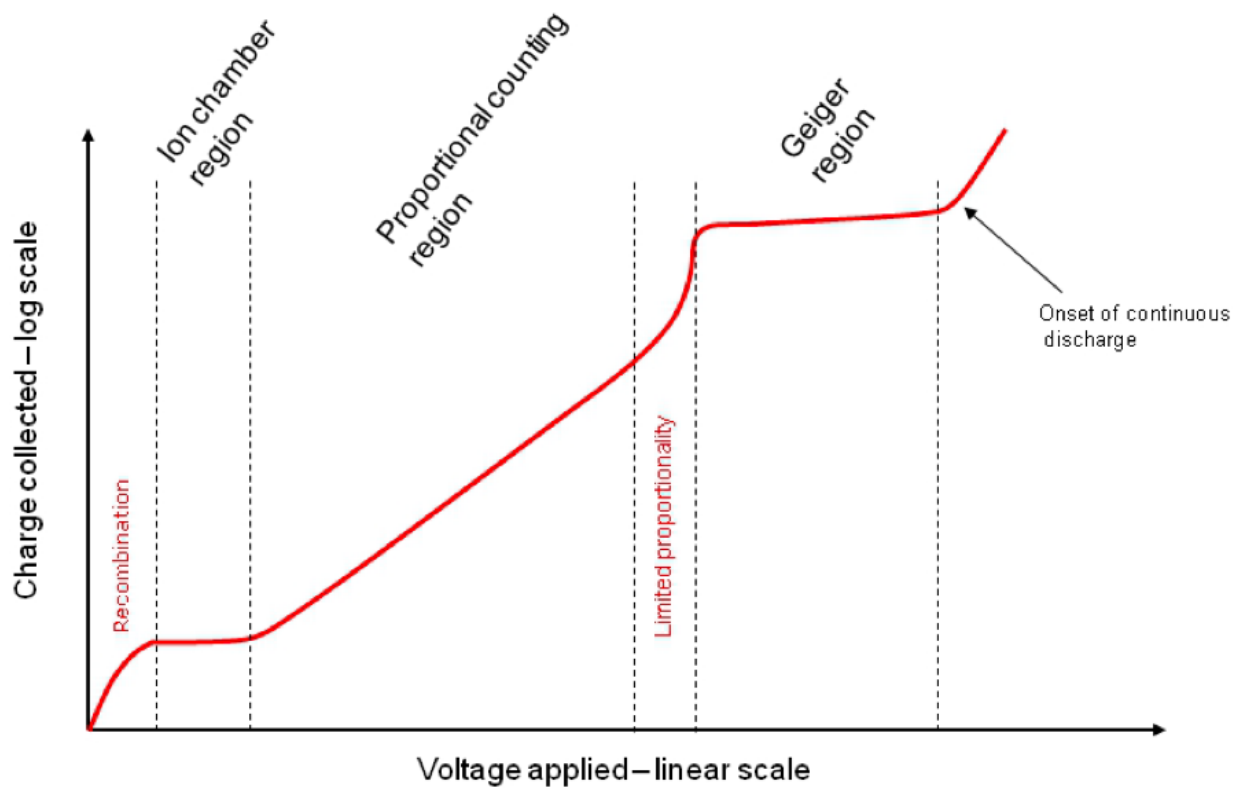


Figure 4.1: Different modes of operation for GM tube under different anode voltage. Graphic adapted from [21].

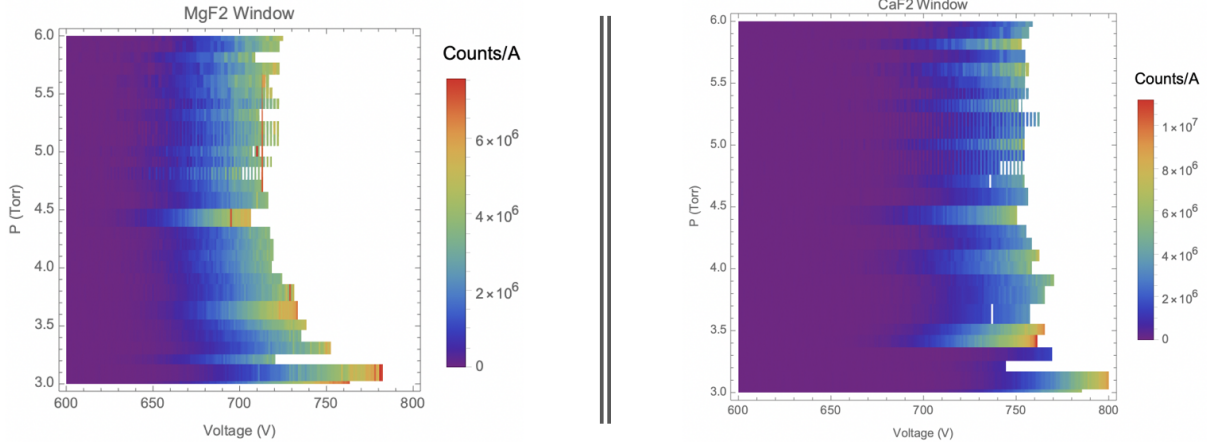


Figure 4.2: Signal count rate for both detectors. We will be using $P=3.7$ torr with anode voltage of 800V.

detectors.

In fig.(4.4), we show the counts as a function of anode voltage on a log-linear scale for acetone pressure of 3.7 Torr. The results showed that both detectors are operating in linear proportional mode and the counts are increasing with anode voltage (see fig. (4.1)) as opposed to the Gieger mode. This conclusion is reinforced by examining the pulse-height distribution of the detectors, (see fig. (4.5)) using the MCA (for this we have used a threshold voltage of 300 mV and gas pressure of 3.7 torr). A pulse-height distribution plot for each detector at gas pressure of 3.7 torr and 800V anode voltage is shown in fig.(14). We found that the number of pulses decreases exponentially with voltage signal height, as expected for proportional counting region.[8]. In contrast, if the tube were operating in Geiger mode, we should expect a peak counts appearing at high voltage values.

Note, unlike in ref. [4], we did not observe a maximum in the count rate at a pressure below the breakdown voltage, nor a clear optimal pressure or voltage to operate the detectors. Rather, there is a range of pressure and voltage that provide similar detector performance. Consequently, we have chosen a gas pressure of 3.7 Torr to perform the following parts of the experiment.

The operating voltage and pressure is rechecked before performing an IPES measurement to account for any changes in the detector electronics. For instance, since performing the detector characterization above, we installed a resistor to preamplifier board to prevent it getting damaged by high current pulses. This resistor also induced extra noise, requiring higher threshold value settings after inserting the extra resistor. A new threshold value

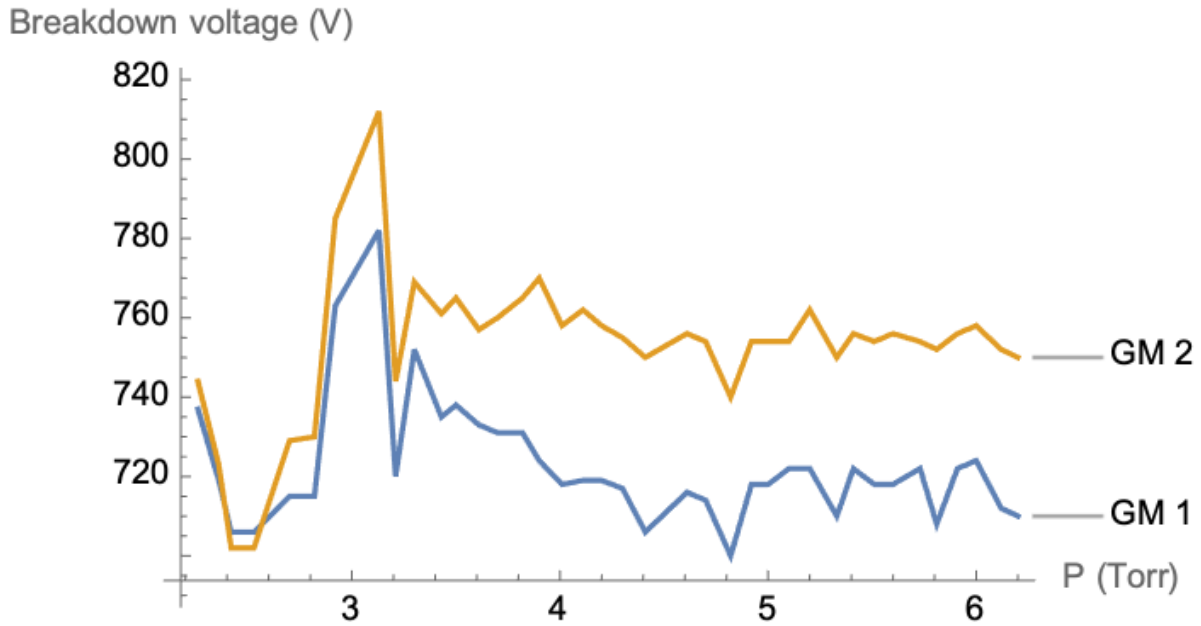


Figure 4.3: Tube breakdown voltage for different gas pressure. Note after 3 Torr an external resistor is added to pre-amplifier board for both detectors to prevent the board getting fried by large signal pulses.

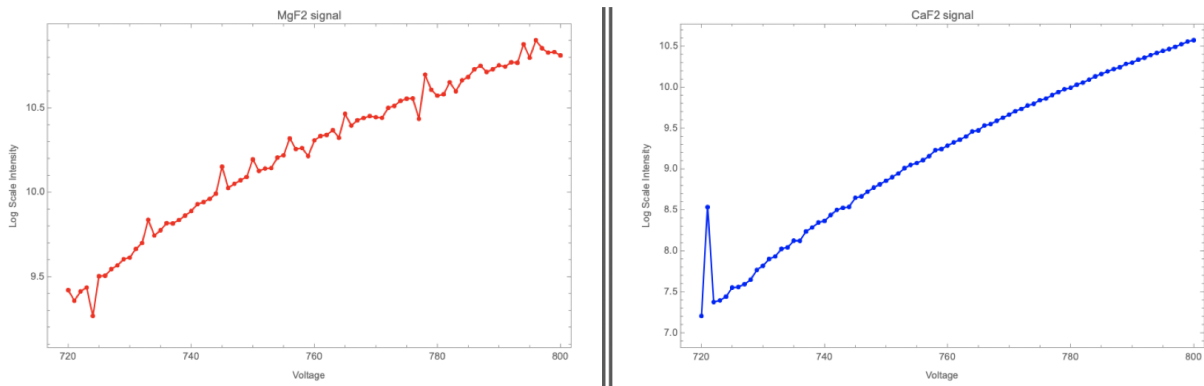


Figure 4.4: Counting rate vs anode voltage from 720V to 800V for both detectors on a log-linear scale. The approximate linear dependence indicates the detectors are operating in proportional mode.

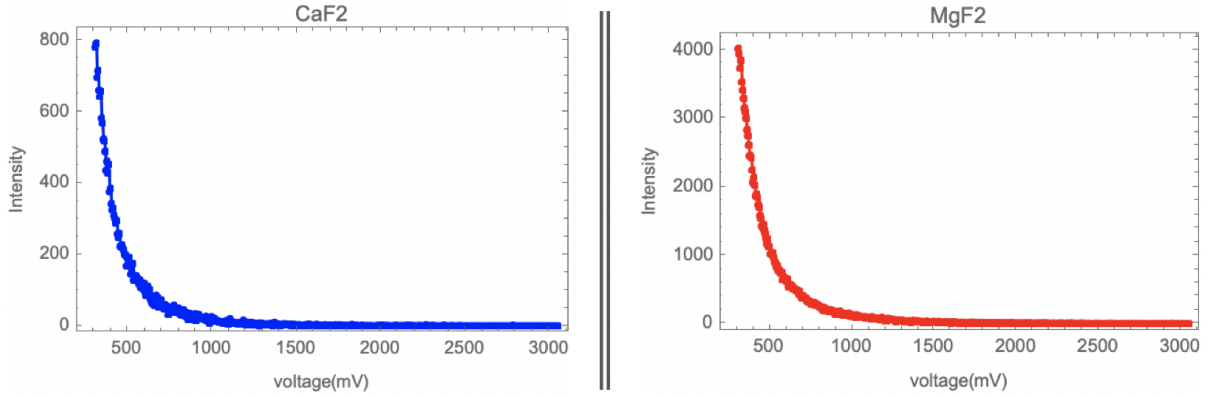


Figure 4.5: Pulse-height distribution for both detectors. The threshold value is 300 meV.

of 300 mV is used to test the tube. Moreover, changes to the preamplifier modules have resulted in slightly higher values of breakdown voltages than the initial detector characterization measurements presented in this section. Future work will recharacterized the detectors with the modified electronics, although we do not expect qualitative changes to the results presented here.

4.2 Electron Gun Characterization

After detector characterization, the next step is to characterize the electron gun. The electron gun has multiple parameters that can be varied. Each parameter contributes to the electron beam in their own ways. Filament Voltage will have current flowing through the filament, heating up the filament so that electrons get excited and hence produce electrons at the surface of filament. Extraction Voltage is in charge for dragging those excited electrons out of the filament surface. Within some limits, increasing extraction means the ability to extract electrons from filament is increased, hence will produce a larger emission current. However, if the filament voltage is too low, and all the available electrons are ejected already, then further increasing of the extraction will not increase the emission current. Grid voltage is set to knock off electrons that are drifting away from center, and hence reduces the beam size and produces a straight forward beam. A negative grid voltage is needed to achieve this goal. Focus voltages will push electrons towards each other, hence decrease the size of electron beam. The function of focus voltage is similar to the function of convex lens to light. Deflection voltages (both directions) will deflect the electron beam. However due to the non-uniform electric field at the edge of the deflection

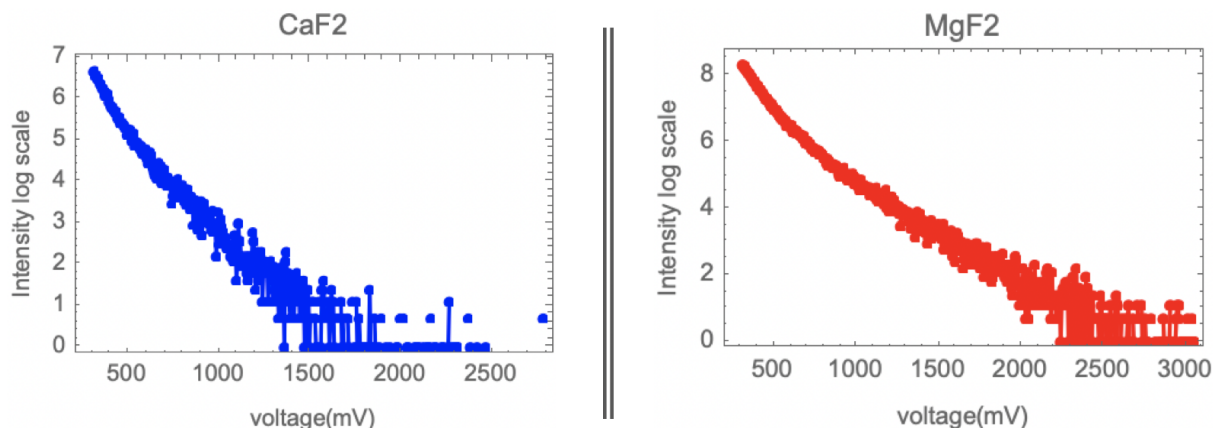


Figure 4.6: Pulse-height distribution on semi-log plot for both detectors. The threshold value is 300 meV.

plat, it will cause distortion to the beam. During this project, deflections will not be used.

The ultimate goal of electron gun characterization is to find a set of parameter values, so that a beam with small beam size (less than 2.5mm in diameter) can be produced, while have a sample current of $10\mu A$, with a low beam divergence and high energy resolution (ΔE to be within 200 meV).

Faraday Cup

Faraday cup is used to measure the spot size of the beam.[1] The size of the hole on Faraday cup is much smaller than beam size. Once the Faraday cup is driven by the motor to scan the plane perpendicular to the beam, only electrons that pass through the hole can be detected. A Gaussian shaped electric signal is expected, and the FWHM of the signal is considered as the size of the beam.

4.2.1 Electrodes Characterization

We will be using $E=15V$ for characterization, since we do not expect to perform IPES scan at large electron energy values. By experience, a higher filament voltage means hotter filament, which give more electrons to be ejected. However, if the filament voltage is too high, it is possible for the gun to generate a weird, non uniform distribution of electron beam, which is harmful for the experiment. One possible explanation is the space-charge

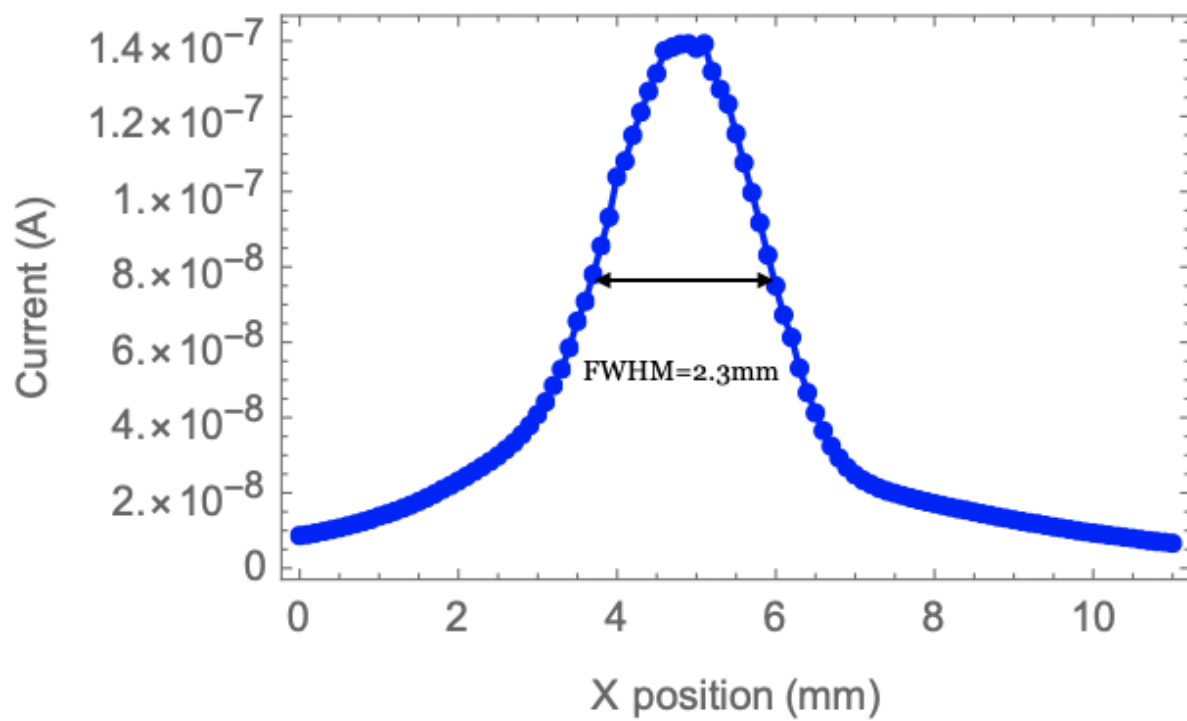


Figure 4.7: A sample scan using Faraday cup, where the electron gun settings are: $E=10\text{V}$, Filament= 4.4V , Extraction= 8V , Grid= -3V , $F1=4\text{V}$, $F2=0\text{V}$.

limit at high temperature. We used a filament voltage of 4.2V, and the beam shape is uniform. The sample current is in an order of a few μA .

Applying the extraction voltage will extract the electrons from filament, thus a higher extraction voltage means more electrons can be extracted from the source. If the extraction voltage is too high and all the available electrons from filament are already extracted, then a further increment on extraction voltage will not increase emission current. In this experiment, it is noticed that for filament voltage of 4.2V, after extraction voltage reaches 5V further increasing the extraction voltage does not increase the sample current. We will be using 5V for extraction.

The function of grid voltage is to repel electrons that are drifting away from the center of the beam. Thus the grid voltage should be negative. In our experiment, the constrain for grid voltage is set to be 15V to -15V. Fig.(4.8) shows the scanning result of grid voltage versus x position of the Faraday cup, which is driven by the x-direction motor. Fig.(4.9) shows the plot of x position versus z position of the Faraday cup. We could treat the beam to be a circular shape, so we only need to scan along one direction. To get a small beam size within 2.5mm, while have sample current of several μA I chose to use grid voltage to be -3V.

Applying focus voltages will focus the electron beam. There are two focus voltage sources, focus 1 and focus 2. The scan results are shown in Fig(4.10). For the purpose of smaller beam size, values for focus 1 and 2 are chosen to be 2V and 4V.

4.2.2 Momentum Divergence

Due to the space-charge effect, electrons that are close to each other will tend to repeal, hence it is not very likely that the beam is a concentrated beam like laser. Motor controlling the y axis of the Faraday cup could change the distance between sample and electron gun. Fig.(4.11) shows the scanning plot of y position vs x position. We will be using y=0mm, since the beam size does not vary significantly from fig.(4.11).

From fig.(4.11) it is seen that the electron gun is not pointing right towards the sample, but has a small deflection angle. The deflection angle could be found by:

$$\theta_d = \frac{d_f - d_i}{\Delta Y} \quad (4.1)$$

Where θ_d is the deflection angle, d_f and d_i are locations of two peaks of the electron beam. We found that the deflection angle is around 0.4 degrees¹.

¹This is achieved by plotting sample current vs x position for each y position of the Faraday cup, then

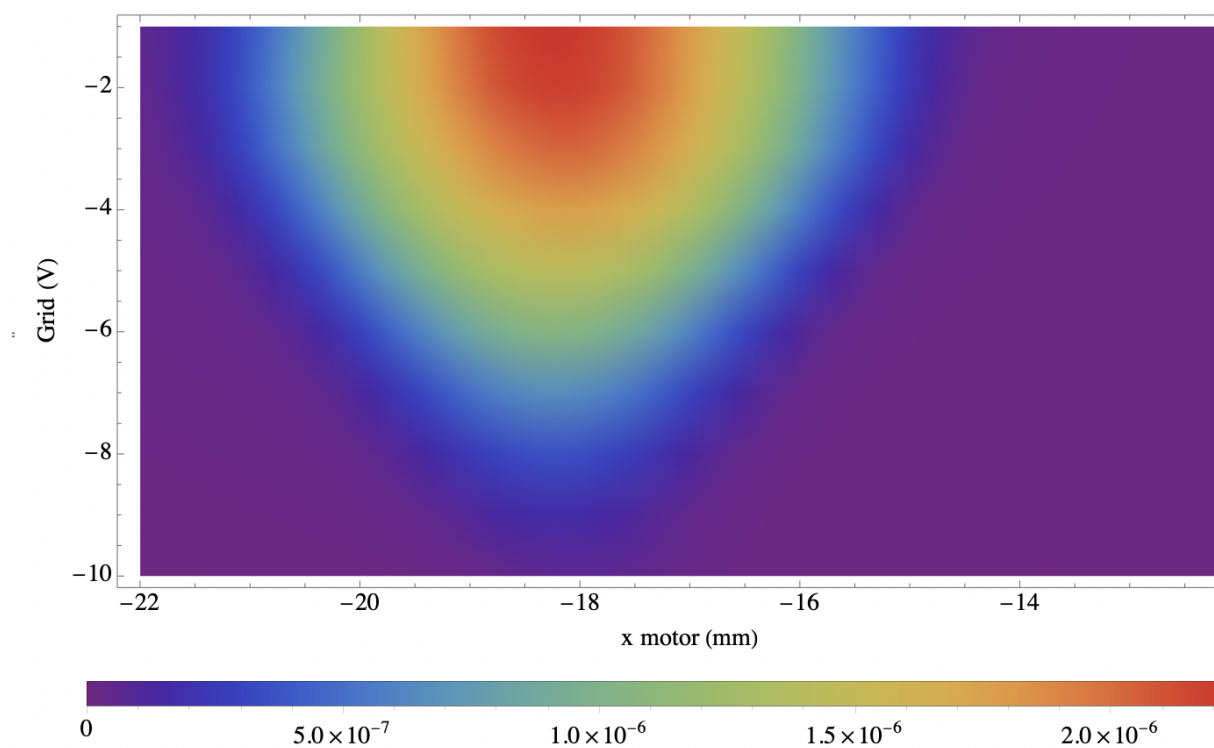


Figure 4.8: Grid Voltage scan. We will be using grid=-3V since the beam size is around 2 mm, while the sample current is above $1 \mu A$.

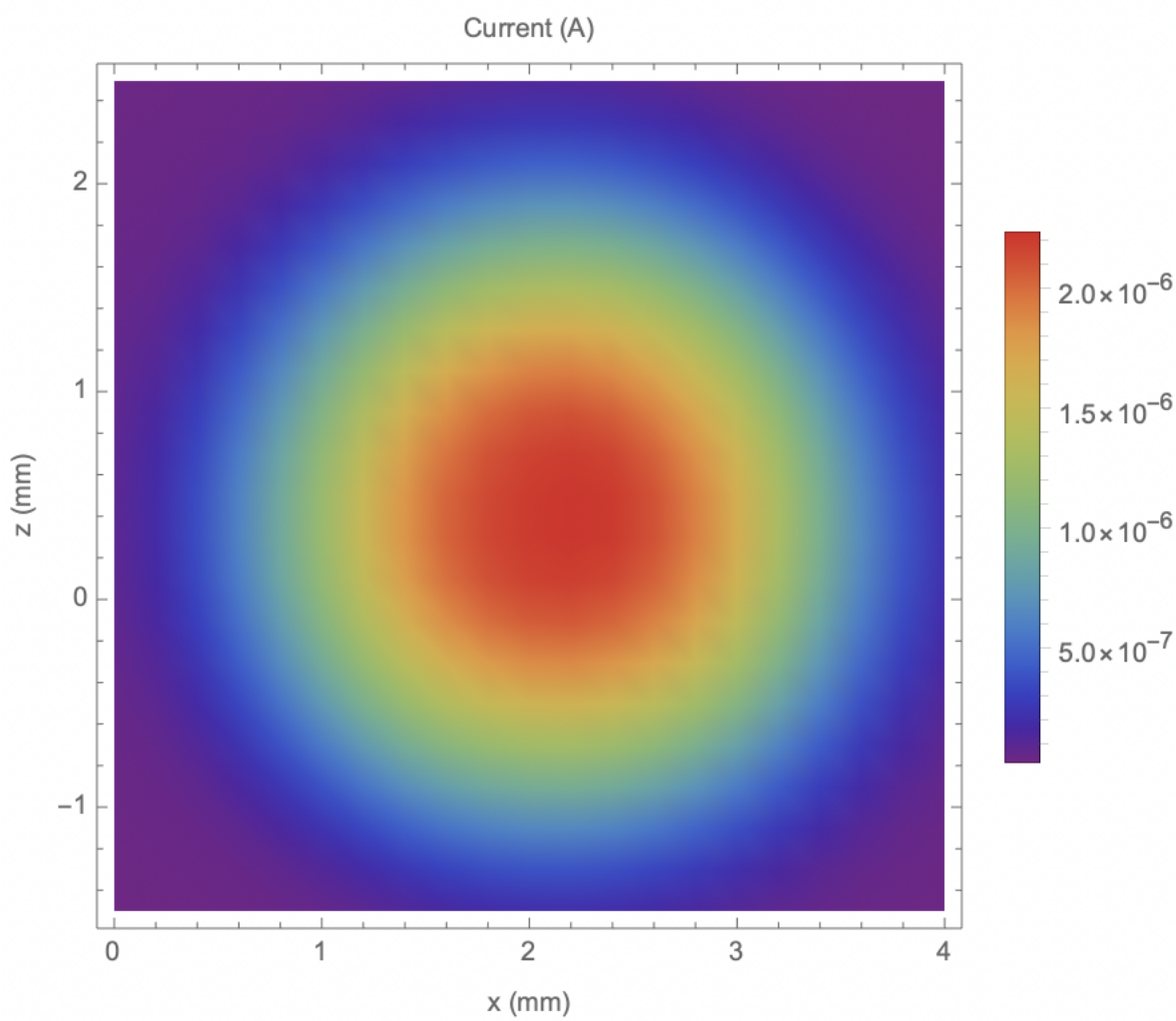


Figure 4.9: Scan of x versus z. The beam is in a circular shape.

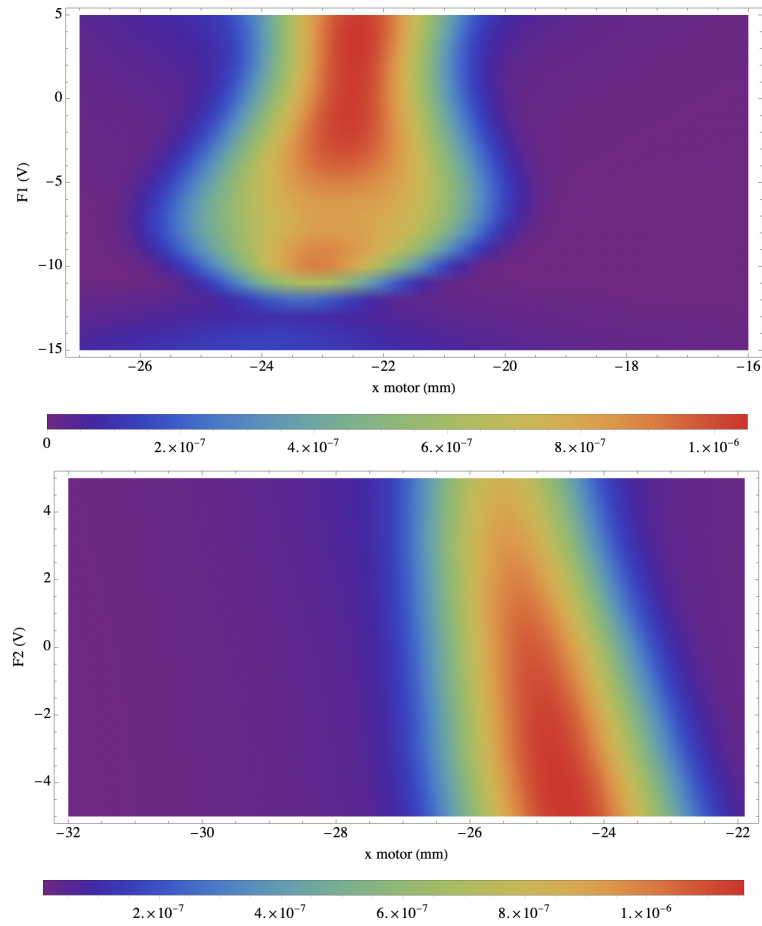


Figure 4.10: F1 and F2 scan. We will be using $F1=2V$ and $F2=4V$.

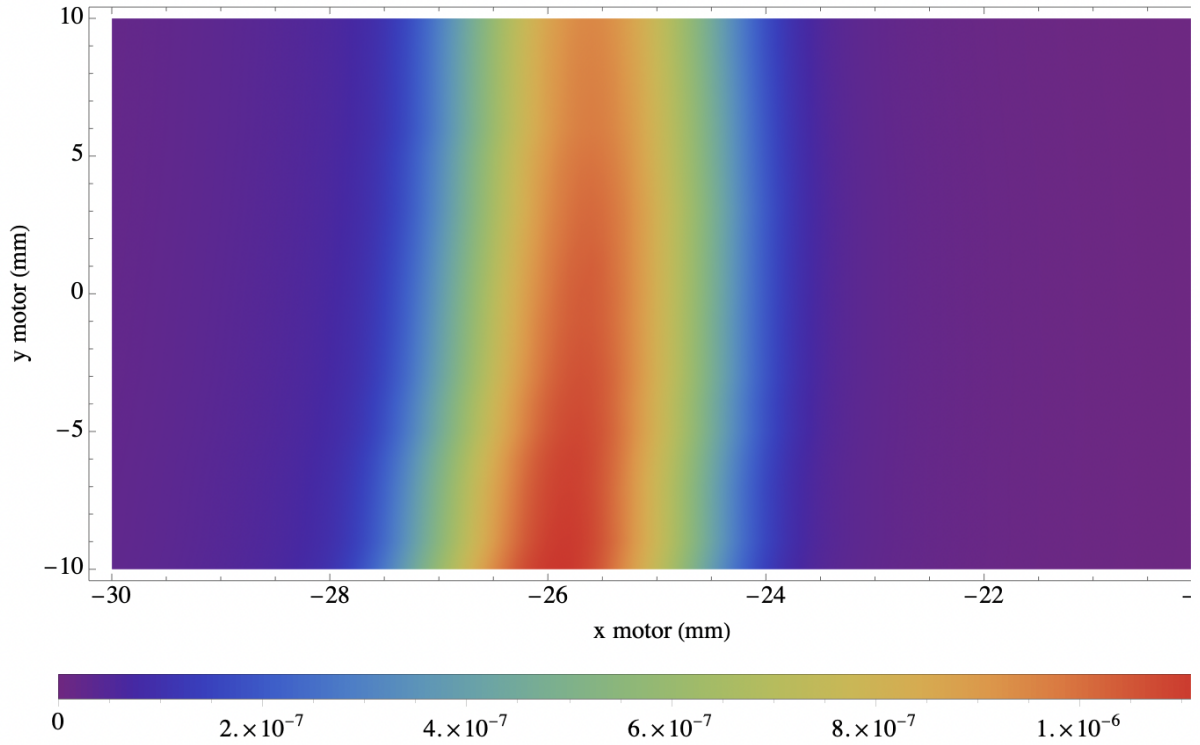


Figure 4.11: Y vs X. The beam size does not have significant changes when moving away or towards the electron gun, we will be staying at $y=0$ mm.

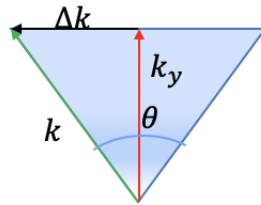


Figure 4.12: The electron beam is spreading out. k is the beam momentum, k_y is the vertical component of the momentum (towards the sample), and Δk is the momentum divergence.

We also want to know the momentum resolution of the beam, so that we could compare it with the size of the first Brillouin zone of Cu and see if the uncertainty in momentum is small enough. Because the electron beam is not a straight beam, some portion of the beam is spreading out from straight line direction. The momentum divergence is shown in fig.(4.12). In order to know how much the momentum is spreading out, we will need to find angular divergence of the beam first. The angular divergence can be calculated as following:

$$\theta = \arctan\left(\frac{D_f - D_i}{2L}\right) \quad (4.2)$$

Where D_f and D_i are the diameter of the beam spot at two different distance away from electron gun, and L is the distance between these two points. We could then use the angular divergence to find the momentum dispersion. The expression for the beam momentum is given by

$$k = \frac{\sqrt{2m_e E_k}}{\hbar} \quad (4.3)$$

And the horizontal component of momentum is given by

$$k_{\parallel} = k \sin \theta_k \quad (4.4)$$

Where θ_k is the angle between beam and the axis perpendicular to the sample surface plane (as shown in fig.(3)). For electrons with $E_k = 15eV$, using equation (8) and (9), the momentum dispersion is calculated to be $0.018\text{\AA}$, which is two order of magnitude smaller than the size of first Brillouin zone of Cu. Since the momentum dispersion is proportional to square root of the kinetic energy of electrons, the dispersion could be treated small for all electron energy range during this experiment.

4.2.3 Contact Potential

As described in Chapter 2, it is necessary to determine the kinetic energy of electrons just before they hit the sample. This is accomplished by adding some bias voltage between the sample and electron gun. If the applied bias voltage is smaller than electrons kinetic energy divided by e , the electrons will have enough energy to reach the sample, hence the sample current is detected. Once the bias voltage reaches a value that is comparable to electron kinetic energy over e , the electrons will not be able to reach the sample, hence there will be a sudden lost of sample current at this bias voltage. The difference between

locate the x position where sample current reaches the peak value. Then choose two points that are apart from each other.

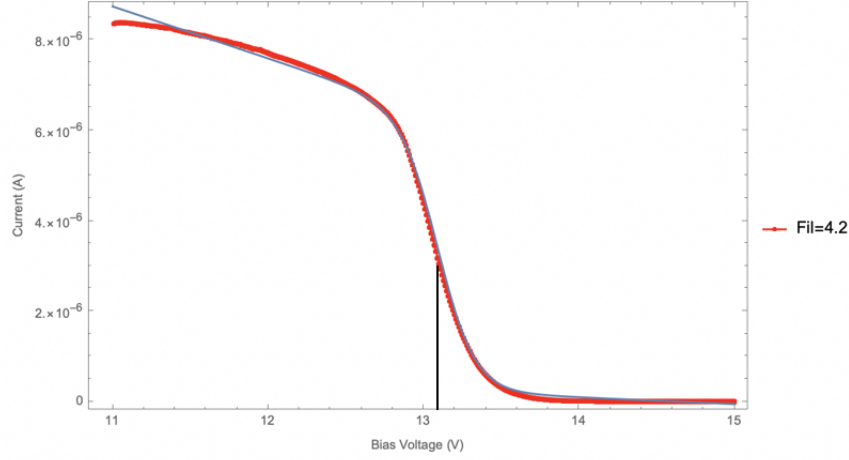


Figure 4.13: Blue line plot with data point is the contact potential scan; the blue line plot is a step function fit. The step width of the fitting function, ΔE , is 120 meV, and E_{stop} is found to be 13.1V.

the energy given by electron gun and the value of bias voltage at where the sudden lost of sample is the contact potential between the sample and electron gun cathode, as shown in fig.(4.13). The experimental data is fit to:

$$I = \frac{B(C - E)}{e^{\left(\frac{E - E_{stop}}{\Delta E}\right)} + 1} + AE + D \quad (4.5)$$

This function captures the Fermi function as well as the approximately linear dependence of the current at low bias voltage.

The contact potential is found to be 1.9V for the gun cathode and Cu(100) sample, with energy resolution of 120 meV for the electron gun. The work function for Cu(100) is about 4.5 eV [16]. Using equation (4), it leaves a value around 2.6 eV for cathode work function.

By choosing this set of parameters for $E=15V$, a small beam size within 2.5 mm is obtained, while keeping the sample current at several μAs . Also the momentum resolution and energy resolution for the electron gun is high, which is what we expected to achieve.

We then using the same method to find a set of parameters for lower electron energy ($E=10V$), which we found a set of parameters(filament=4.4V, extraction = 8V, grid = -3V, focus 1=4V and focus 2=0V) that produces a beam size 2.3 mm, while the sample current is a few μA for $E=10V$. Since we are interested in energy states near Fermi energy,

for Cu sample we are interested in electron gun energy lower than 15 eV, thus we will be using the settings for $E=10\text{V}$.

Chapter 5

Sample Preparation and IPES scan

Before we put sample into the vacuum chamber, we need to wet clean the sample surface to get rid of the contamination that is hard to be pumped out, such as oil and dust particles. In the wet cleaning step, we use methanol, ethanol and acetone, in that order, to wipe sample holder, sample plate, screws and sample, to remove the contamination. While performing the cleaning procedure, gloves needs to be worn to prevent the sample from being contaminated by hand oil. If there is any contamination that is hard to wipe, we put the parts in ultrasonic machine and leave it for about 15 minutes for a deeper clean.

After everything is cleaned, we assemble the plates and sample holder together, tight the screws, then put “sliver paint” on it to keep the sample stacked onto the plates. The sliver paint is electrical conducting material, so that when electrons hit the sample the current signal can be passed through the paint. Then we bake the entire assemble for about 15 minutes to let the paint dry, so that the sample is tightly stack on the sample holder and will not fall off .

5.1 Sputtering and Annealing the Sample

The IPES measurements on a 2D material sample is surface sensitive, since the conservation of momentum along the horizontal direction is relied on translation symmetry. Due to this reason, we want to prepare the sample with a smooth, clean surface. After we clean the sample using solvent, the next step will be transferring the sample into the vacuum chamber. In order to do that, we will need to turn off the turbo and screw pump for the load lock, turn on the nitrogen gas supply and let it flow through the turbo pump into

the load lock. By doing this, the load lock will be filled with nitrogen gas rather than air, which is harmful for a clean sample surface. Then we wait for the pressure to come up to around 700 torr, close to atmosphere pressure. Then we open up the entrance window on the load lock, installing the sample holder into one of the sample storage holder and close the entrance. Then we turn the pumps back on and waiting for the pressure to come back down to vacuum. After the pressure comes down to the order of 10^{-8} torr, we open the valve between load lock and preparation chamber, sliding in the storage and use sample transfer arm to grab the sample tab and place it on the heater stage with caution. Then we close the valve. After the sample holder is place well on the heater stage, we can start the dry clean process. Wet cleaning the sample gets rid of oil and dust, but not the oxidized product on the sample surface. Our lab uses Argon gas and ion gun combination to kick those oxidized particles off the sample surface. The operation principle is straight forward. We first point the ion gun towards the sample surface, turn on the ion gun, as well as the Argon gas supply. Once the Ar particles go through the ion gun, it will get ionized and be positively charged. Then those Argon ions will be shoot right at the sample surface, kicking the oxidized contamination particles off the sample surface and then be pumped out by vacuum pump. After sputtering the sample, there will be holes left on the sample surface due to the oxidized particles being kicked off. The next step is to annealing the sample [15], where the sample will get heated up, so that the energetic atoms on the surface could temporarily break the lattice bonds and diffuse along the sample surface. With sufficient amount of annealing time, it is likely that those holes on the surface could be filled, hence creating a smooth surface that can be used in IPES experiment. More details can be found in [1].

For sputtering the sample, we first turn on the ion gun, set emission to 20 mA. Then we gradually open the argon valve and letting argon gas flow in the preparation chamber. Note that we open the valve slowly enough so that the pressure in preparation chamber will not go up too fast. Once the pressure in preparation chamber has reached to low 10^{-6} torr we leave the valve at that position, and let the sputtering process to go over night.

The second step is annealing the sample. Once the sputtering process is finished, we shut off the ion gun and argon supply, then letting the pressure returns to baseline level. Then we connect the heater supply to the heater stage, adjust the supply voltage to about 4A. This will keep the temperature at about 350 degrees. The temperature in the preparation chamber can be measured using thermo couples . We could also use multimeter to measure the more accurate temperature when there is too much noise signal in thermo couples. We left the annealing process going over the night as well.

After the cleaning process is complete, we wait for the pressure coming down to baseline, and then the sample is ready to be transferred to the main chamber.

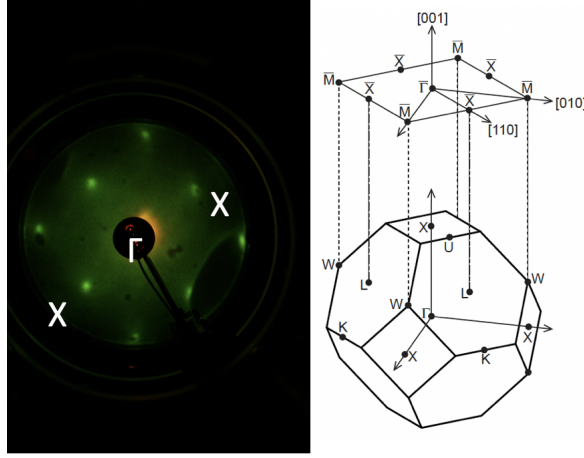


Figure 5.1: Left: LEED image of Cu(100) sample. Note when we rotate the sample along horizontal direction of the page it is the ΓX direction. Right: Drawing adapted from [18]. The bulk and surface Brillouin zones of the fcc(100) surface.

5.2 LEED

Before we perform IPES measurements, we need to check if the sample surface is smooth enough for IPES measurement. We also want to rotate the sample along a symmetric direction so we could compare our result with what other groups have obtained. In order to accomplish the goals, we use Low energy electron diffraction (LEED). The operation principle has been discussed elsewhere [14]. A LEED image is shown in fig.(5.1). We can see there are visible and clear reciprocal lattice points. In this experiment we will rotate the sample along ΓX direction.

5.3 ARIPES Scans for Cu(100)

The ARIPES scan of Cu(100) is shown in fig.(5.2). The normal incident spectra shows an intensive peak near the Fermi energy, which is due to a transition from B_1 into bulk band, X'_4 . [3] The sample is rotated along ΓX direction. During the experiment sample is rotated towards MgF_2 detector. The spectra were compiled as E-k map, using equation (8), shown in fig.(5.3). It shows a strong B_1 transition until around $0.8 \text{ \AA}^{-1}$. Due to the detecting resolution, only one peak with highest intensity is visible for each angle of scan. The image state peak, S_1 , is less intensive and was not detected during the experiment. The result

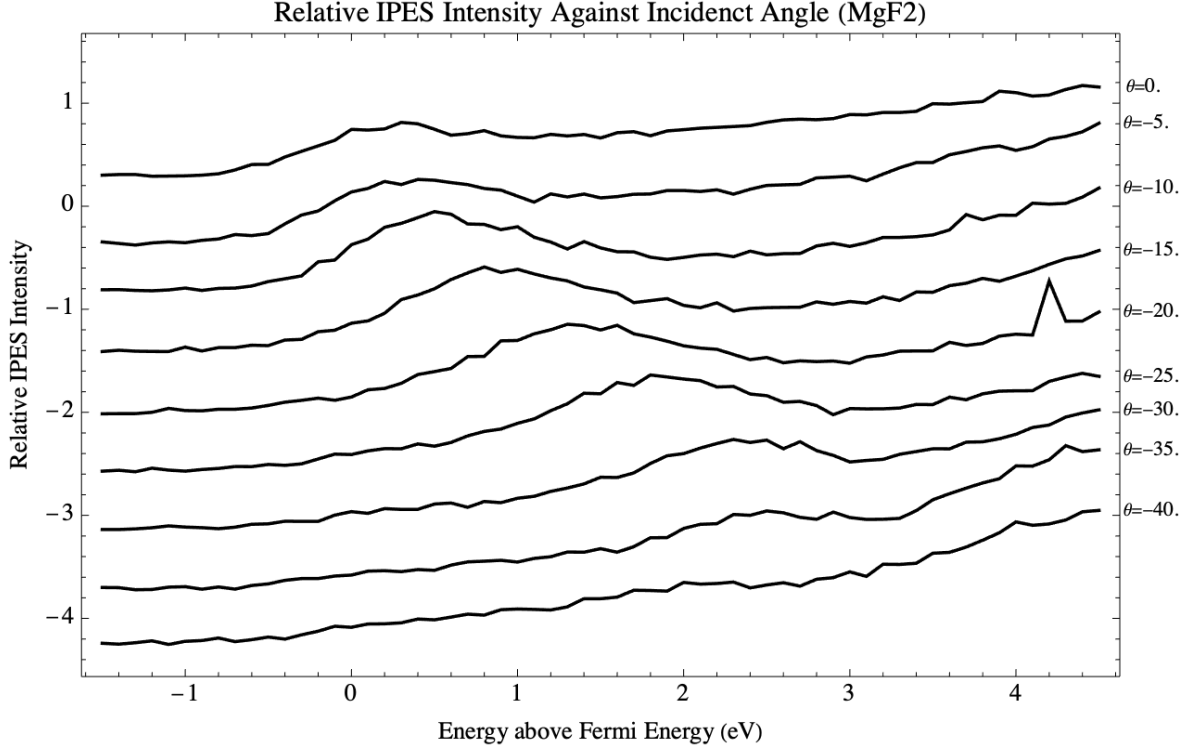


Figure 5.2: Angle-resolved IPES spectra with varies angles along ΓX direction.

for B_1 transition agrees with Cu(100) ARIPEs from other group [2], as shown in fig.(5.4). The result also agrees with theoretical calculated band dispersion, within the resolution of around 900 meV.[20]

5.3.1 Energy Resolution

After we obtain the ARIPEs scan results, we want to know how well our data are comparing to other groups work or theoretical values. This is achieved by looking at the energy resolution of the spectrometer. Both photon detectors and electron gun contribute to the energy resolution, and the combined uncertainty in energy will determine the overall energy resolution of the system.

The fitting plot for the IPES scan at 10 degrees is shown in fig.(5.5). Since we expect to obtain spectrum with a Gaussian like peak centred at 8V, the Gaussian fit equation is chosen to have a Gaussian function part, with background estimated by other terms

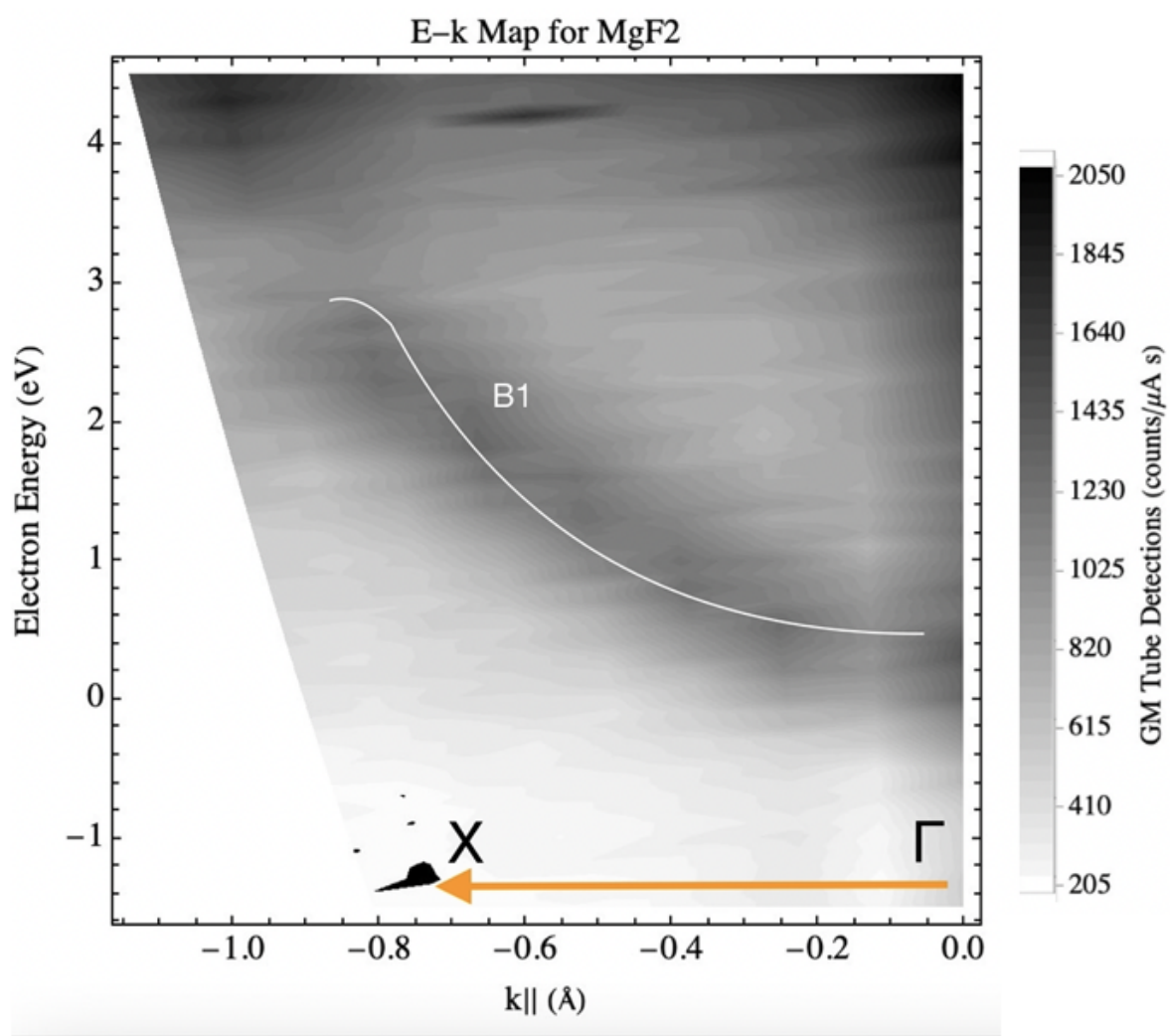


Figure 5.3: E-k map of Cu(100) spectra along ΓX

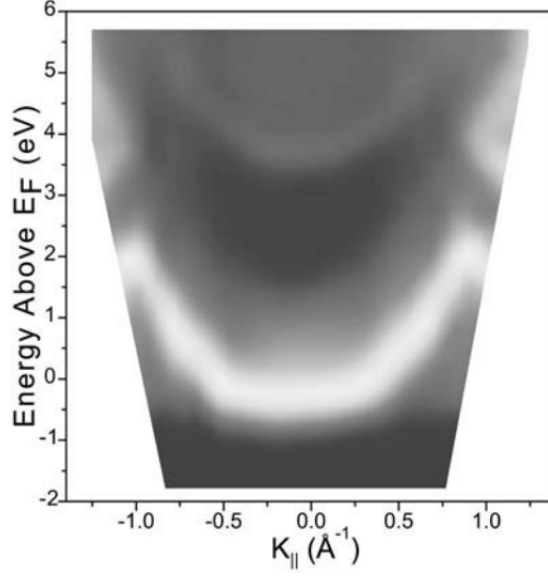


Figure 5.4: E-k map (along ΓX direction) of Cu(100) spectra from ref[2] with permission.

in equation(11). The parameters are then obtained by running program for least-square fitting.

$$Intensity = (Ae^{-\frac{1}{2}(\frac{E-E_{Fermi}}{\sigma})^2} + C(x - F)^2 + D) * (\frac{1}{e^{(\frac{7-E}{0.1})} + 1}) + G \quad (5.1)$$

With the best-fit value found to be $\sigma=460$ meV, the Gaussian peak fits the data plot. Therefore the FWHM is 920 meV. This is the energy resolution for both electron gun and the MgF_2 detector.

By using equation (12), and $\Delta E= 120$ meV for the electron gun from previous chapter, it is determined that the acetone/ MgF_2 bandpass has an energy resolution of 912 meV. The photon absorption cut-off energy for MgF_2 is about 10.8 eV [19], and the ionization energy for acetone gas is near 9.7 eV. The energy resolution of acetone- MgF_2 band pass filter used in this experiment falls in this range, as expected.

$$\Delta E = \sqrt{(\Delta E_{window})^2 + (\Delta E_{gun})^2} \quad (5.2)$$

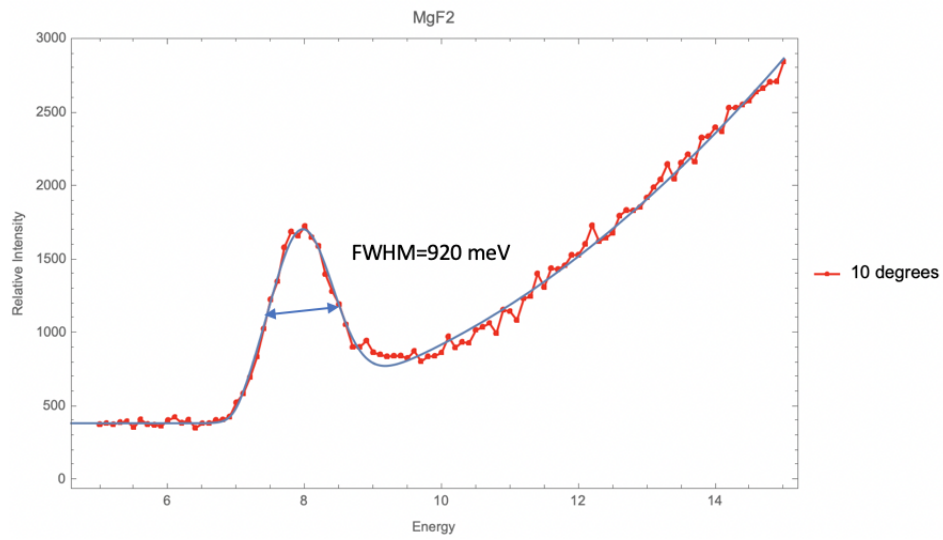


Figure 5.5: Red line plot with data point is IPES scan at an angle of 10 degrees; the blue line plot is a Gaussian fit. The FWHM of the Gaussian fit is 920 meV.

Chapter 6

Conclusions

For the GM tube detector, it has been shown that at acetone pressure of 3.7 torr with anode voltage of 800V, both detectors are operated in linear proportional region. Under this voltage and pressure there is a significant large signal to noise ratio, hence we could be certain that it is a good set of parameters we would like to operate the detector.

For the electron gun we have characterized the parameters, and found a set of values (filament=4.2V, extraction= 5V, grid= -3V, focus 1=2V and focus 2=4V) that produces a electron beam with size 2.5 mm, while keeping the sample current in an order of few μA for $E = 15\text{eV}$. We have also found a set of parameters for lower energy electrons (filament=4.4V, extraction = 8V, grid = -3V, focus 1=4V and focus 2=0V), with beam size 2.3 mm and also few μA of sample current. We have used the settings for $E=10\text{V}$ since our ARIPEs scans are focused on electron energies within 5 eV above Fermi energy, which is within 12 eV of electron energy and is close to $E=10\text{V}$.

Using GM detectors and electron gun, we have successfully re-produced ARIPEs scan for Cu(100) along ΓX direction, and a clear band dispersion of B_1 is present. Unfortunately no dispersion of other energy states are clearly visible due to surface quality and energy resolution.

Chapter 7

Future Work

Since the ARIPES scan only gave us a clear dispersion for energy states with highest signal intensity, we could try to improve our system for better resolution. The next step for our system could be characterizing the electron gun further more, following the same trail, for each electron energy. There are also other chip models for pre-amplifier and shaping amplifier, which may change the resolution, as well as the ideal operational voltage and pressure. All of them could potentially increase the signal resolution, hence we have a higher chance to detect the band dispersion for those low intensity peak signals.

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