

UNIVERSITY OF TWENTE.

Faculty of Electrical Engineering,
Mathematics & Computer Science

Investigating local scanning tunneling spectroscopy work function measurements as an alternative to ultraviolet photoelectron spectroscopy work function measurements

J. H. Davenschot
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Supervisors:

prof. dr. ir. M. P. De Jong,
M.Sc. P. Markeev

NanoElectronics Group
Faculty of Electrical Engineering,
Mathematics and Computer Science
University of Twente
P.O. Box 217
7500 AE Enschede
The Netherlands

0.1 Summary

The aim of this assignment was to investigate if a scanning tunneling spectroscopy(STS) can be used as a reliable alternative method of measuring the local work functions of various materials than ultraviolet photoelectron spectroscopy(UPS) and Kelvin Probe force microscopy.

The work function is determined with scanning tunneling spectroscopy (STS) by use of I-Z spectroscopy. In this approach the current through the tip of a scanning tunneling microscope is measured as a function of the distance(z). The work function is extracted from these curves through fitting an exponential model. The results of this method contain a significant amount of spread and the obtained values are not in accordance with theory and the established values in literature.

Another approach used is ultraviolet photoelectron spectroscopy(UPS) in this approach the sample is bombarded with photons which wavelength is in the ultraviolet range. This approach makes use of the photoelectric effect and the work function can be determined by how much energy it takes to free an electron from the lattice. The obtained results of UPS gave values that are in the expected range for the work function of the used materials.

The initial aim of the assignment was to measure the work function of a set of TMDs. Due to obtaining unforeseen results using STS on bulk materials such measurements were not carried out. The results suggest that a different model should be used for doing STS measurements.

Contents

0.1	Summary	i
1	Introduction	1
1.1	Introduction	1
2	Theory	3
2.1	Work Function	3
2.2	Scanning tunneling Microscopy	3
2.2.1	Sample holder	3
2.2.2	Tunneling	4
2.2.3	UPS	7
3	Measurement Setup	11
3.1	Samples	11
3.2	STM	11
3.2.1	Controller	11
3.2.2	Tip	12
3.3	UPS	12
3.4	Measurement results	13
3.4.1	STM and STS	13
3.4.2	STS	15
3.4.3	UPS	19
4	Discussion	23
4.1	Conclusion	25
5	Appendix	29

Introduction

1.1 Introduction

The discovery of Graphene a 2D-material with remarkable properties such as its thickness, extraordinary strength, giant intrinsic carrier mobility and superconductivity has led to an increase in research into other 2D materials [1]. One such group of materials transition metal dichalcogenides (TMDs) possess a band gap making them suitable for applications in the semiconductor industry such as diodes and transistors. A member of the group MoS_2 has been demonstrated to have great potential for use in optoelectronic devices [2]. Not only are 2D-materials interesting for material scientists, they also offer novel possibilities for improving existing products. New improvements could be reduced weight, lower power consumption, improved battery life and flexible electronics.

To be able to utilize 2D materials for such applications characterization of the materials is important. To be able to design multi layered semiconductor devices from 2D-materials the value of a material property known as the work function plays an important role in the band bending in such a device. The work function is the amount of energy needed to remove an electron from the surface of or the energy difference between the Fermi level of a material and vacuum. At the interface of a hetero junction the Fermi levels of the materials align. A difference in work functions then leads to a situation where the Fermi levels of the materials line up but there is an energy difference between the vacuum levels. This results in what is known as band bending resulting in a built in voltage in the junction [3].

Most reliable values of the work function are determined by the measurement method photon electron spectroscopy which uses the photo electric effect to determine the work function. This technique has as disadvantage that the measured value of the work function is the average of the measured surface [4]. This approach

is sufficient for most applications however when one wants to know the local work function of a material surface there is another method using a scanning tunneling microscope to perform scanning tunneling spectroscopy. This assignment aims to investigate if it is possible to obtain reliable local values of the work function using scanning tunneling spectroscopy.

Theory

2.1 Work Function

The work function is defined as the amount of energy needed to remove an electron from a material [5]. As already mentioned in the introduction the work function is an important parameter in hetero junction devices. If the work function of both materials are different there will be band bending possible causing a built-in voltage [3]. The built in voltage in hetero junctions is related to the current that will flow through the junction. In photodiodes the built-in voltage is related to the amount of exciton pairs that are created in the diode. In Transistors the built-in voltage is related to the threshold voltage. Most values of the work function are in the range from 3-5 eV [6].

2.2 Scanning tunneling Microscopy

A scanning tunneling microscope (STM) relies on the phenomenon of quantum tunneling theory. The STM is essentially a sharp metallic tip that is positioned very close to surface of a material that is to be imaged. By measuring a tunneling current between the tip and the surface of the sample the STM can be used to make topographic images of atomic resolution of the sample

2.2.1 Sample holder

The sample holder is depicted in figure 2.1. The tip is hanging from a platform that is standing on 3 legs with each leg having its own piezo actuator. To let the tip approach the sample the piezo acutuator move the legs down the ramp until the current set point of the controller is reached.

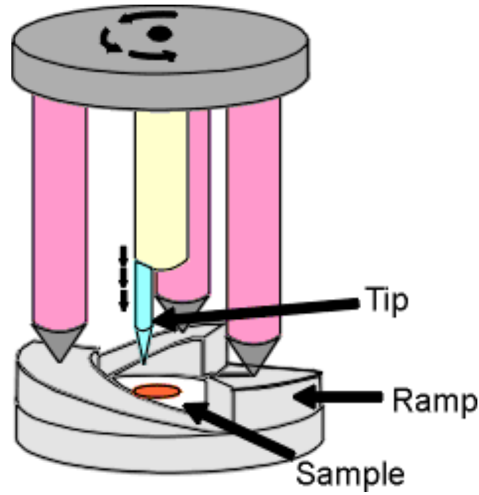


Figure 2.1: A sketch of the STM approach system of the sample holder [7]

2.2.2 Tunneling

In classical mechanics an electron can not penetrate a energy barrier if the energy of the electron is smaller than that of the barrier. However in quantum mechanics the wave function of the electron can penetrate the barrier resulting in the possibility that an electron tunnels trough the barrier. Resulting in a tunneling current that has an exponential dependence on the distance(z) of the tip to the surface of the sample.

Rectangular barrier

To obtain a theoretical understanding for tunneling the situation for a rectangular barrier is solved below. Let us consider the rectangular potential barrier as shown in fig 2.2.

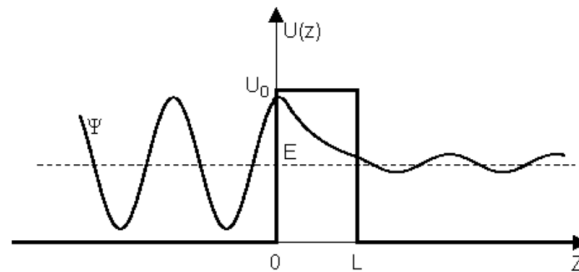


Figure 2.2: Sketch of a rectangular barrier [8]

In the region from 0 to L the Schrödinger equation reads

$$\left[\frac{-\hbar^2}{2m} \nabla^2 + U_0 \right] \Psi(z) = E\Psi(z) \quad (2.1)$$

By assuming that there is only the possibility of tunneling in the right direction and hence taking the trial wave function as:

$$\Psi(z) = c_r e^{\kappa z} \quad (2.2)$$

And substituting in 2.1

$$\left[\frac{-\kappa^2 \hbar^2}{2m} \Psi(z) + U_0 \right] \Psi(z) = E \Psi(z) \quad (2.3)$$

By rearranging equation 2.3 equation 2.4 is obtained

$$\kappa = \pm \frac{\sqrt{2m(U_0 - E)}}{\hbar} \quad (2.4)$$

As the probability of finding an electron is proportional to the squared wave function one obtains for the tunneling probability:

$$|\Psi(z)|^2 = |\Psi(0)|^2 e^{-2\kappa z} \quad (2.5)$$

This result suggest that the tunneling probability grows or decays exponentially with the distance z . In An STM this exponential sensitivity to distance is used to create images of atomic resolution.

Trapezoidal barrier

However the above result assumes a theoretical rectangular barrier in reality the tunneling barrier is different and looks more like a trapezoidal barrier as can be seen in figure 2.3. This is due to a difference in work function between the sample and the tip. As the tip and sample get closer to each other the Fermi levels of the materials have to line up however due to a difference in work functions and the applied bias the energy of the vacuum level has to adapt resulting in a tunnel barrier that looks more like a triangle.

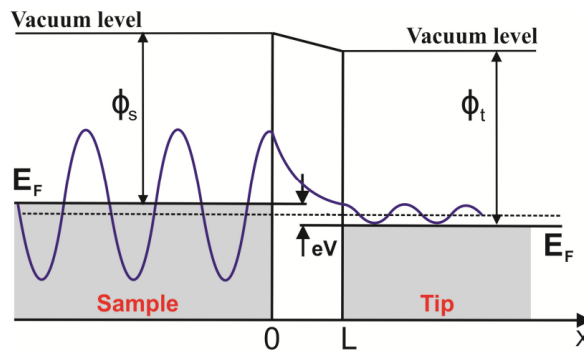


Figure 2.3: Illustration of 1D trapezoidal vacuum tunneling barrier [9]

The resulting barrier is harder to solve than the rectangular barrier. For this case is assumed that the average potential barrier is given by the the average work function of the tip and the sample provided that the applied bias between the sample and the tip is small.

Scanning

To obtain images of a material surface the tip of the STM is brought close to the surface of the material by 3 piezoelectric actuators moving on a ramp until the tunneling current set point J_T is reached. Once the set point has been reached the controller will correct the z-position of the tip such that the measured current is equal to that of the set point current. To create an image the tip is moved over the surface of the material of interest and the height corrections of the tip are recorded. In this way an image of the topography of the surface is built up line by line.

It is possible that the surface of the material is contaminated by another better conducting material shown in figure 2.4 as spot C. Due to the better conductance of the contaminant the measured current will be higher than that of the non contaminated material resulting in the controller correcting the tip position. Which leads to the STM giving a higher value for the topography than is actually the case.

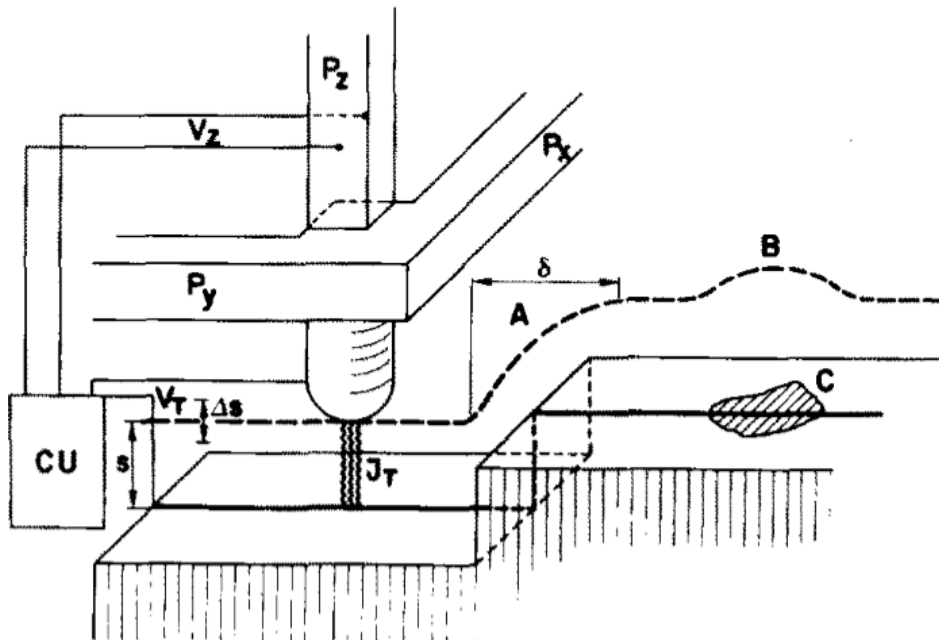


Figure 2.4: Image showing the principle of STM scanning CU is the control unit, s the distance to the sample, P_y, P_x, P_z are the piezo scan directions J_t is the tunneling current. C is a contamination of the surface that is more conductive than the surrounding surface. B is the resulting scan deflection. V_t is the applied bias voltage. [10]

To obtain an image of atomic resolution it is required that the scanning tip is sharp. As is to say that there should at best be a single atom at the the end of the tip.

I(z) spectroscopy

The work function of a material can hence be obtained with an STM by making an I-z plot. This is a plot in which the tunneling current is measured as a function of the tip distance (z) To the material. The current is expected to increase exponentially as the tip approaches the material as indicated by equation 2.5. By curve fitting the obtained I-z plot to an exponential function the value of κ can be determined. By backward substitution one can then obtain an average work function:

$$\Phi = 1/2 (\Phi_{\text{sample}} + \Phi_{\text{tip}}) \quad (2.6)$$

[7] This presents a problem however one can not determine the work function of the material if one does not know the work function of the tip. There are 2 solution to this problem: one must know the work function of the tip in advance or one has to do the measurements on multiple different materials with the same tip and then back substitute to obtain the work function of the material.

2.2.3 UPS

Ultraviolet Photoelectron Spectroscopy(UPS) is a technique which makes use of the photoelectric effect. The photoelectric effect causes electrons to be emitted from a material with less kinetic energy than the photons that irradiate the material. There is a threshold kinetic energy value that the incident photons require before electrons are emitted from the material. This threshold value is the work function of the material. In an UPS measurement the sample is irradiated with ultraviolet light ($E_{kin} = 21.2[eV]$) resulting in a spectrum of the density of states of the sample. By measuring energy range of the obtained spectrum and subtracting it from the kinetic energy of the incident photons one can determine the work function of the material.

Detector

The type of detector used is a hemispherical detection analyzer as shown in figure 2.5.

The electrons that are excited from the material by the UV light first pass through a retardation stage. Reducing their kinetic energy E_{kin} to E_0 before entering the

spherical deflection analyser (SDA)

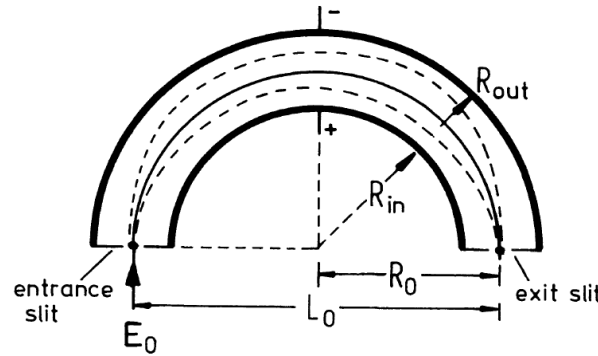


Figure 2.5: Sketch of a hemispherical detection analyzer [11]

detection analyzer. The SDA measures the kinetic energy of the electrons by the amount of deflection that they undergo in the presence of the electric field applied between the two spheres of the SDA. The SDA is designed in such a way that electrons with energy E_0 end up in the middle of the detector. If an electron has more energy than E_0 the amount of the deflection will be less and will end up more on the right hand side of the detector in figure 2.5. If the electron has less energy than E_0 the electron will be deflected more strongly and end up more to the left hand side of figure 2.5.

By varying the amount of retardation it is possible to do a sweep of the density of states of the sample one is looking at. This type of detector has as an added benefit that a smaller detector is required to be able to measure the entire spectrum of the sample. One such example of a spectrum is shown in figure 2.6.

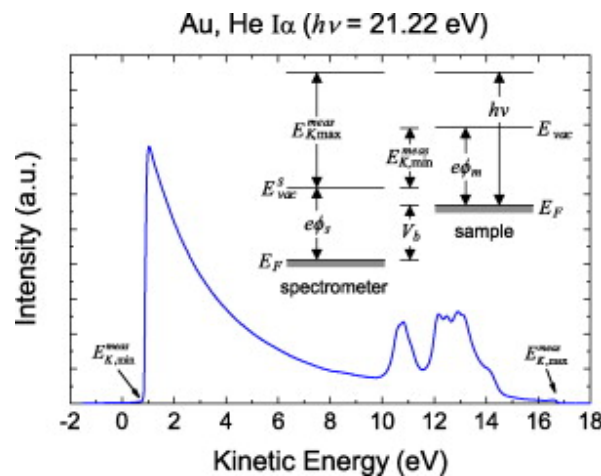


Figure 2.6: Example of a spectra obtained using UPS $E_{kin,min}$ (Left) is the cut off of the spectrum. $E_{kin,max}$ (right) is also known as the Fermi Step [4]

Once the spectrum has been obtained it is possible to determine the work function by measuring the range of the spectrum. The range of the spectrum is determined by finding the cutoff energy and the energy of the Fermi Step. The Fermi Step corresponding to the Fermi energy of the material is the point at which the material starts to release electrons. This can be seen in the obtained spectra as a small step in the intensity. The range of the spectrum is then deducted from the energy of the excitation source yielding the work function. As described by equation 2.7.

$$e\phi = h\nu - (E_{K,\max}^{\text{meas}} - E_{K,\min}^{\text{meas}}) \quad (2.7)$$

Measurement Setup

3.1 Samples

To test the STS theory it was decided to test it on all samples that were already in the STM at the beginning of the assignment. These samples are highly oriented pyrolytic graphite(HOPG), gold(111), MoS₂(2d) and MoS₂(bulk). Later on the samples of aluminum(deposited) and Silicon(p-type) were introduced into the system.

3.2 STM

The measurement setup of the STM consists of a control loop from RHK SPM 100. typical scanning parameters used to inspect the tip quality are timeconstant 3 gain 6 and current setpoint .4 nA. The entire microscope is built upon a vibration correcting table. All the STM/STS measurements shown in this report are done at pressure levels lower than 10^{-8} [mbar]. The systems is brought is kept in ultra high vacuum (UHV) by an ion pump.

3.2.1 Controller

The Scanning tunneling microscope used is an RHK SPM 1000 controlled by an RHK SPM 1000 control loop. There are 4 options which influence the scans of the STM: The current setpoint, the time constant the gain and the bias voltage. Typical scanning parameters used to inspect the tip quality are time constant 3, gain 6, a bias voltage of around 1 volt and current setpoint .5 nA. Depending on the quality of the images that is obtained these parameters can be adjusted to obtain a better image. There is the option to lower the range of the ADC to obtain a smaller quantization interval and hence a higher resolution. This is only done when the output signal of the ADC has a small range typically a voltage swing smaller than -1.25V to

1.25V.

3.2.2 Tip

The tip used for the experiments are Pt-Ir tips cut as obliquely as possible. It was attempted at the beginning the assignment to make a sharper tip by pulling the Pt-Ir wire to make it sharper as can be seen in fig3.1. This method is widely used in various practical manuals of different universities [12]. But was not used in this assignment because this method often resulted in a bend tip also the quality of the images did not seem to improve.

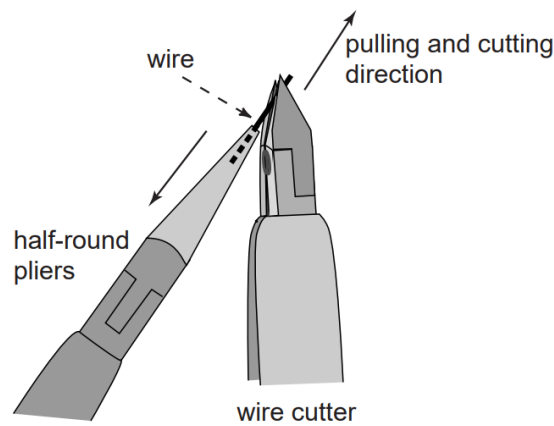


Figure 3.1: Alternative method to cutting as obliquely as possible instead the tip is torn off by pulling the wire [13]

The last method that was available for tip preparation was etching but this was not used as cut tips were already of sufficient quality to obtain atomic resolution on HOPG.

3.3 UPS

The UPS chamber is built on the same vibration damping table as the STM. The UPS chamber is kept in UHV by an ion pump. The system was typically operated at a pressure of $4 * 10^{-7}$ [mbar]. The excitation source is a helium plasma lamp emitting excitation photons with a kinetic energy of 21.2[eV]. The detector used was a SPECS PCU 300 MCD5.

3.4 Measurement results

3.4.1 STM and STS

To test the STS theory and see whether or not it was possible to reliably determine the work function using STS compared to UPS. It was decided to do a small set of measurements on the samples that were in the STM chamber at the moment and then compare the obtained value with the values obtained by UPS. The initial measurements did not show any exponential behaviour but were essentially linear hence it was decided to make a new tip. This resulted in observing the desired exponential behaviour after having made many new tips 3.3.

Topography scans on HOPG material were done until atomic resolution was obtained indicating a good tip quality as can be seen in figures 3.2a and 3.2b. All the measurement in this section have been done with the same tip. Before STS measurements were done on the sample a set of topography scans were done to look for a flat surface as this situation was expected to yield the most consistent results as drift of the piezo motors could not effect the measurement results. If the piezo motors would drift the tip would still be hovering over the same flat surface hence the measurement should be unaffected.

For the STS measurements a set 20 curves would be recorded for each measurement. The tip would reach the current set point and then retract 300pm from the surface of the sample and then approach 600pm. Every point in a measurement represents an ensemble fit of 20 curves. There are 2 figures for each sample. One showing the obtained work function (abbreviated as wf) after fitting and the other figure shows the obtained value of the fit with the respective bias added. The bias was added as it was expected that at higher bias voltages it would lower the effective average barrier height would be lowered by an amount V . So in equation 2.4 the energy E of the electrons would increase. Two figures are hence shown so that the spread in the obtained values can be seen in more detail. So the figures with bias are essentially fitted to:

$$I(z) = Ce^{\kappa z} \quad (3.1)$$

in which κ is equal to

$$\kappa = \frac{\sqrt{2m(\phi_{avg} - V)}}{\hbar} \quad (3.2)$$

And the figures without added bias are fitted to

$$\kappa = \frac{\sqrt{2m(\phi_{avg})}}{\hbar} \quad (3.3)$$

To Process the data use is made of MATLAB as well as Gwyddion and CasaXPS. Gwyddion is used to convert the measurement file format .sm4 to .txt so that the data can be read into MATLAB. The MATLAB program(see Appendix for the code) consists of 2 files one is to load the data from the .txt files into the work space the other is to used to fit the average of each set of measurements to the exponential models.

The results are shown in figures 3.5 to 3.11. The obtained values on HOPG including both bias(figure3.4) and excluding bias(figure 3.5) implied that the work function of HOPG was bias dependent. The obtained values on gold show considerable spread and are lower than what is reported in literature. The measurement results on MoS₂ give a higher result than the other measurements. The average work function on MoS₂ with added bias was found to be 4.26 [eV]. This value was consistently higher than the other obtained values thus far and is in the range that was expected for the work function. However there was one difference in this measurement as compared to the previous measurements. The applied bias voltage was higher than the other measurements for the specific reason that MoS₂ has a band gap. This result prompted the question if such values could be obtained on metals and was tested on aluminum. However the obtained values on aluminum are very low compared to the other samples even with the higher bias.

The obtained average values of the work function were so low that it was not possible to determine the separate work function of the tip and the sample surface as was described in the theory section on $I(z)$ spectroscopy as this would have resulted in a negative value of the work function which does not occur.

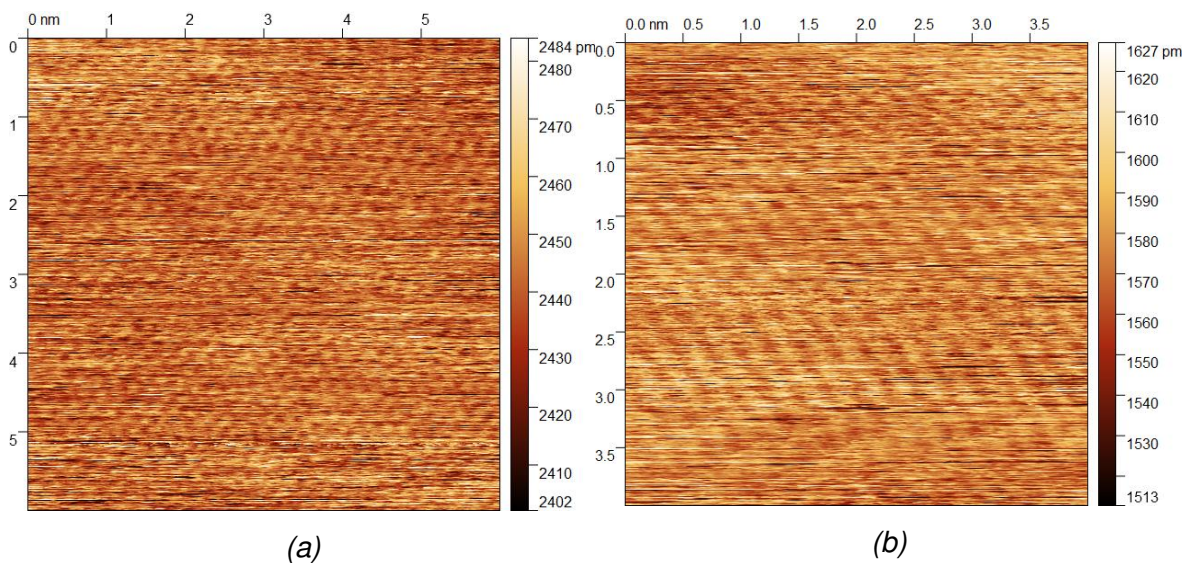


Figure 3.2: Figures (a) and (b) showing obtained atomic resolution on HOPG

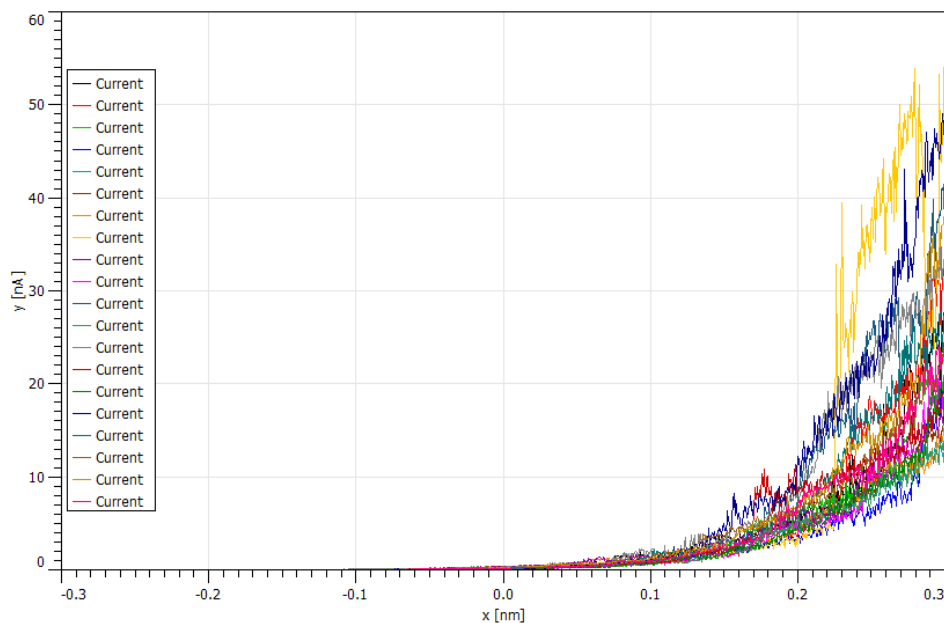


Figure 3.3: The first obtained $I(z)$ measurements showing the desired exponential behaviour of a Platinum-iridium tip on gold

3.4.2 STS

HOPG

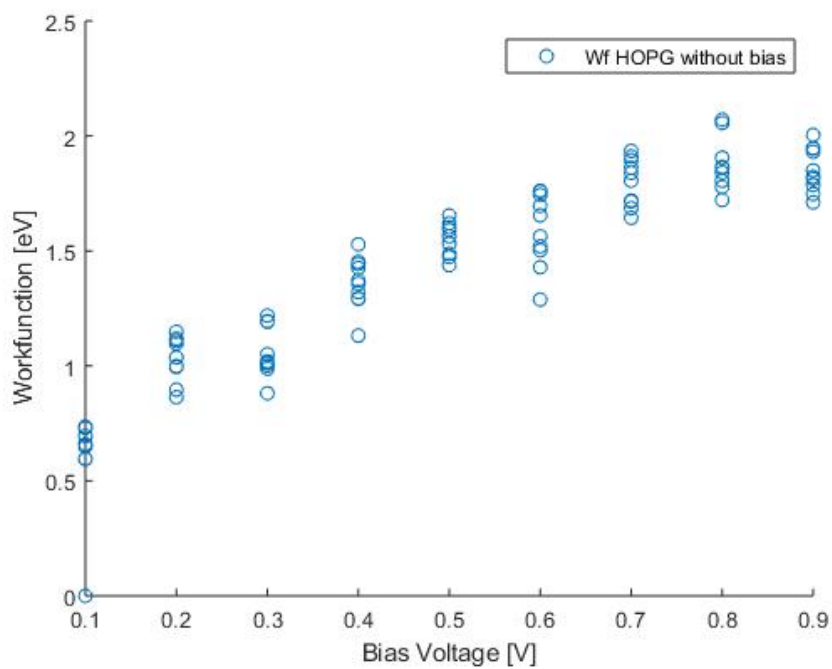


Figure 3.4: work function obtained on HOPG without added bias starting current set point .3nA

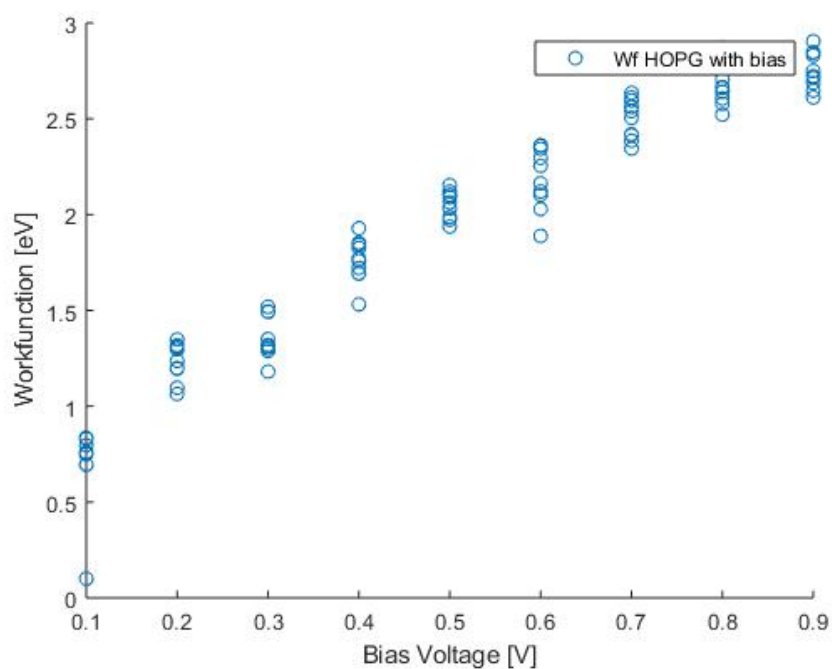


Figure 3.5: work function obtained on HOPG without added bias starting current set point .3nA

MoS₂

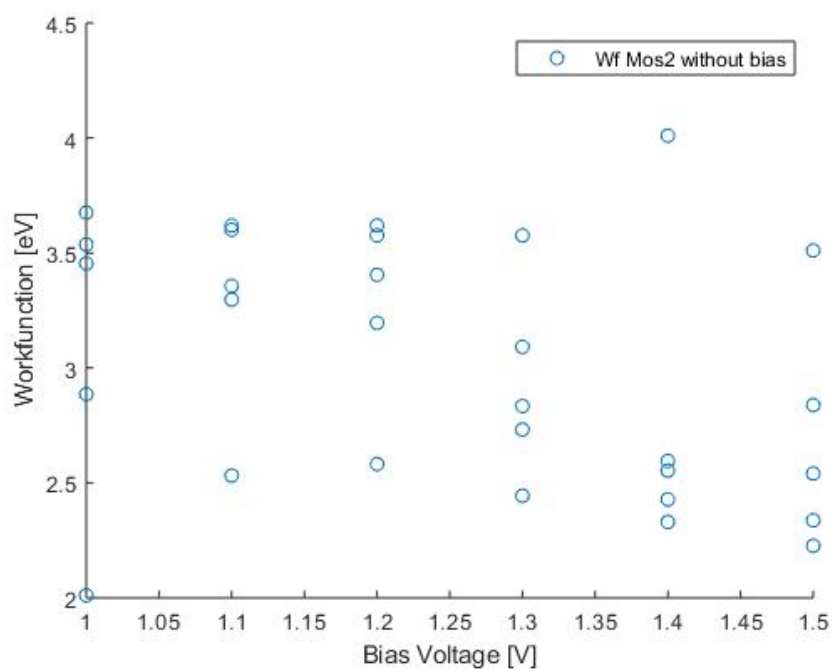


Figure 3.6: work function obtained on MoS₂ without added bias starting current set point .5nA

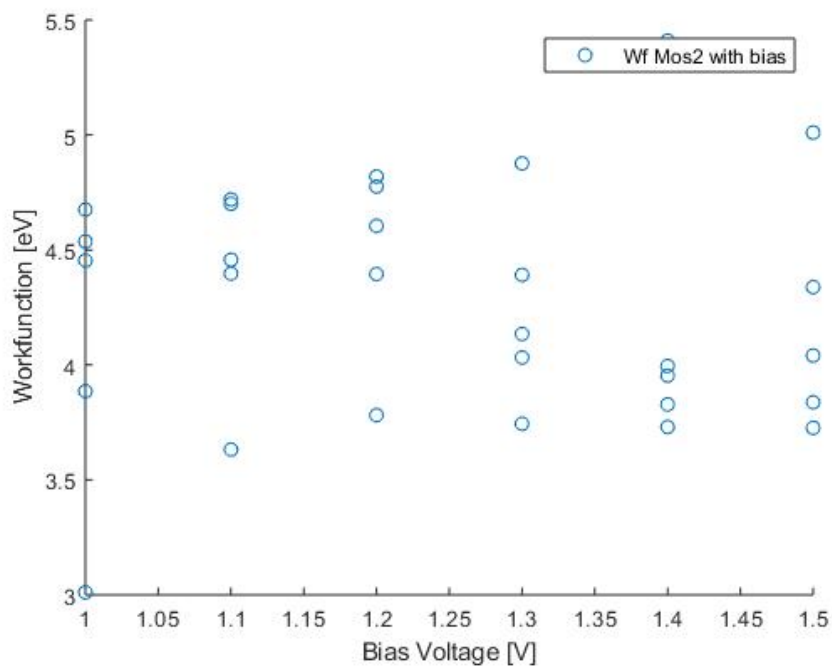


Figure 3.7: work function obtained on MoS₂ with added bias starting current set point.5nA

Au(111)

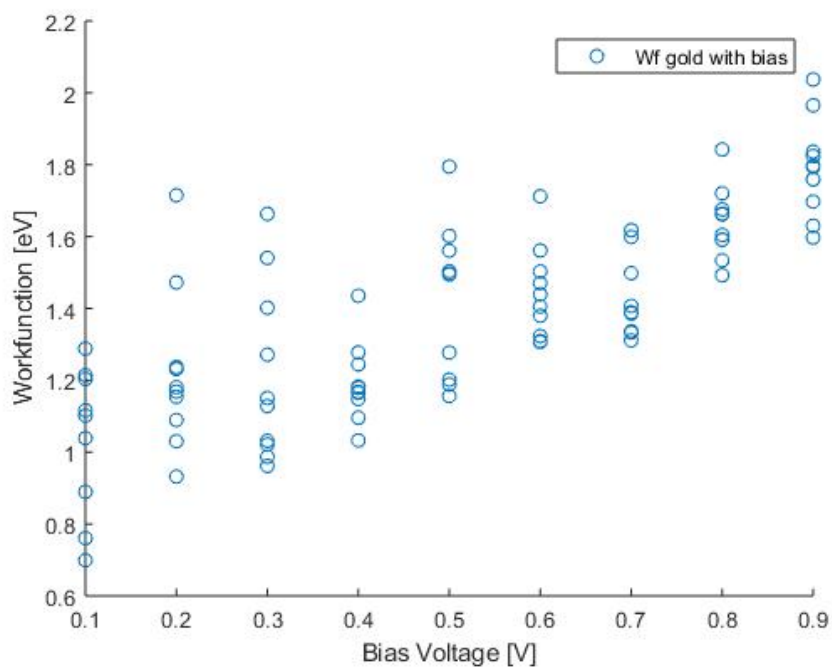


Figure 3.8: work function obtained on gold(111) aluminum without added bias starting current set point.5 nA

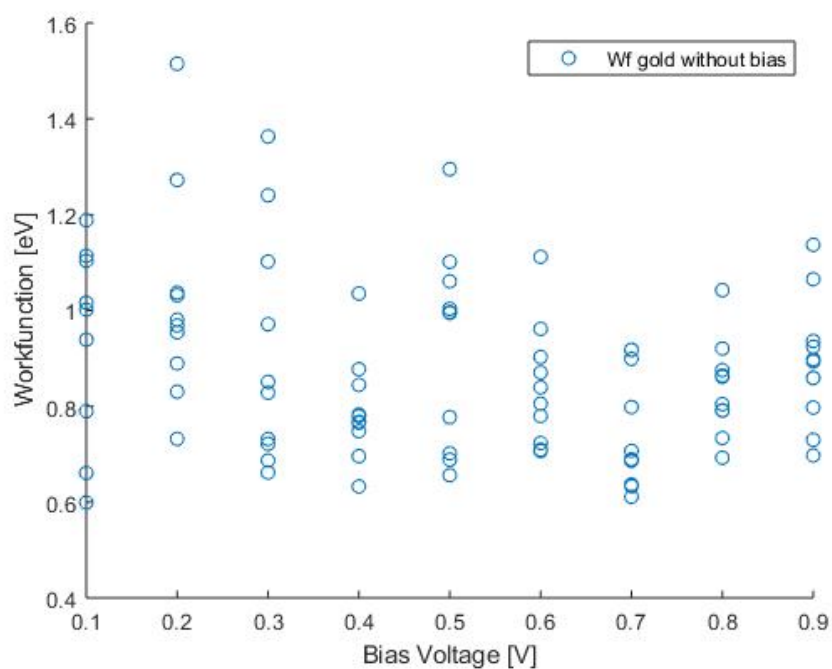


Figure 3.9: work function obtained on gold(111) with added bias starting current set point.5nA

Aluminum

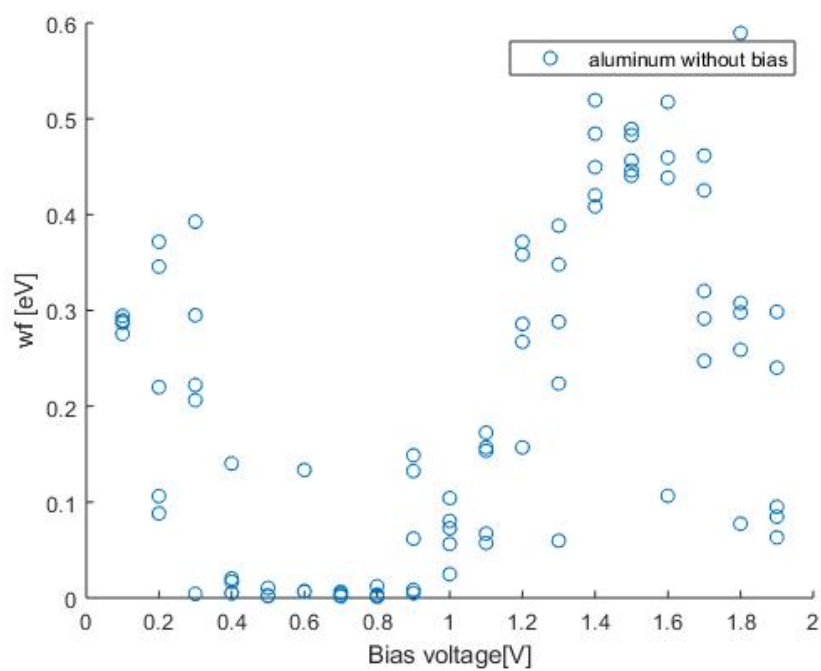


Figure 3.10: work function obtained on sputtered aluminum without added bias starting current set point.5nA

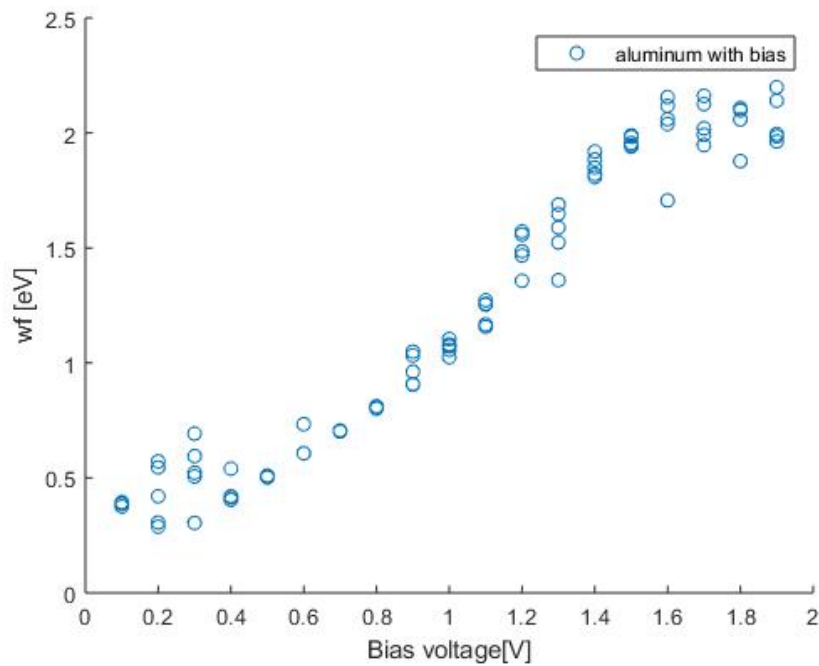


Figure 3.11: Work functions obtained on sputtered aluminum with added bias starting current set point=.5 nA

3.4.3 UPS

The results of the UPS measurements are shown in figures 3.13 3.15 below. On the gold sample 3.12 it was noted that there were multiple spectra present by moving the sample holder it was determined that the black curve occurred due to that the ramp of the sample holder was being illuminated.

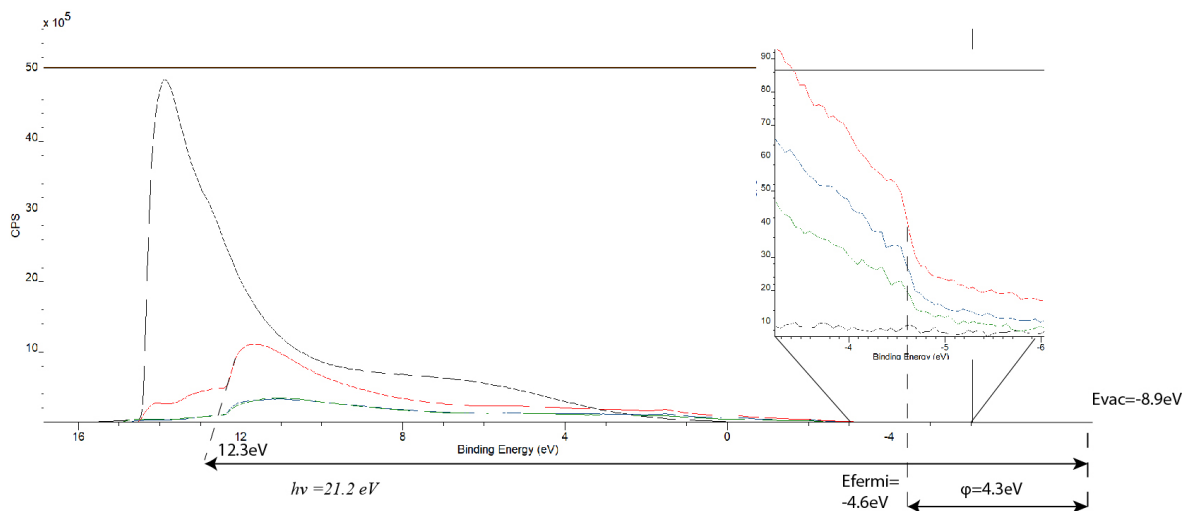
Au(111)

Figure 3.12: UPS spectra on Au(111) the black curve was determined to be the ramp of the sample holder the obtained work function is 4.3 eV

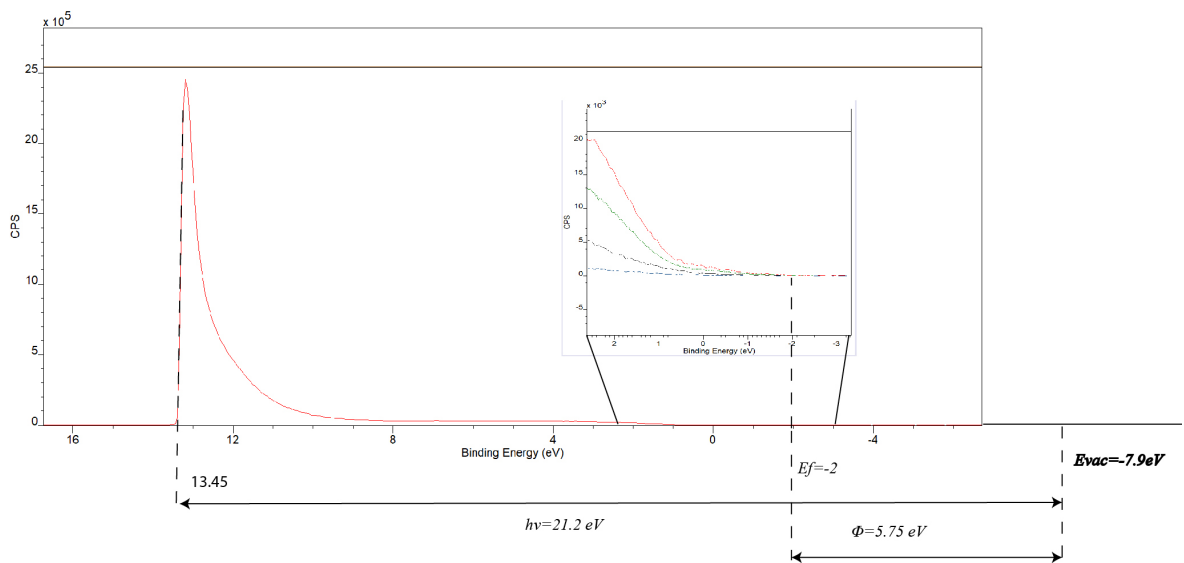
Aluminium

Figure 3.13: UPS spectra on aluminium obtained work function 5.75 eV

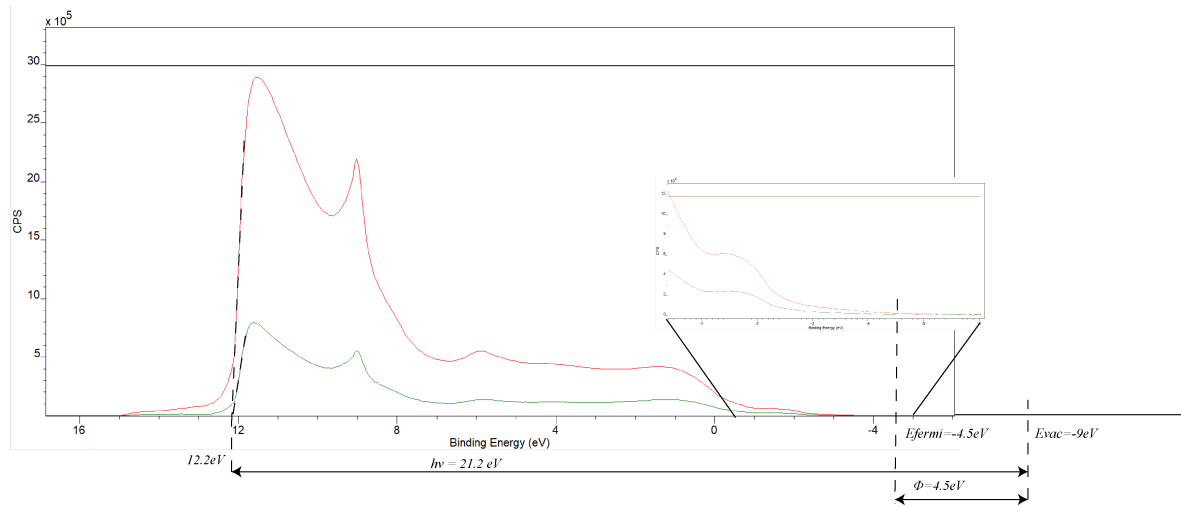
HOPG

Figure 3.14: Measured UPS spectra on HOPG obtained work function value 4.5 eV

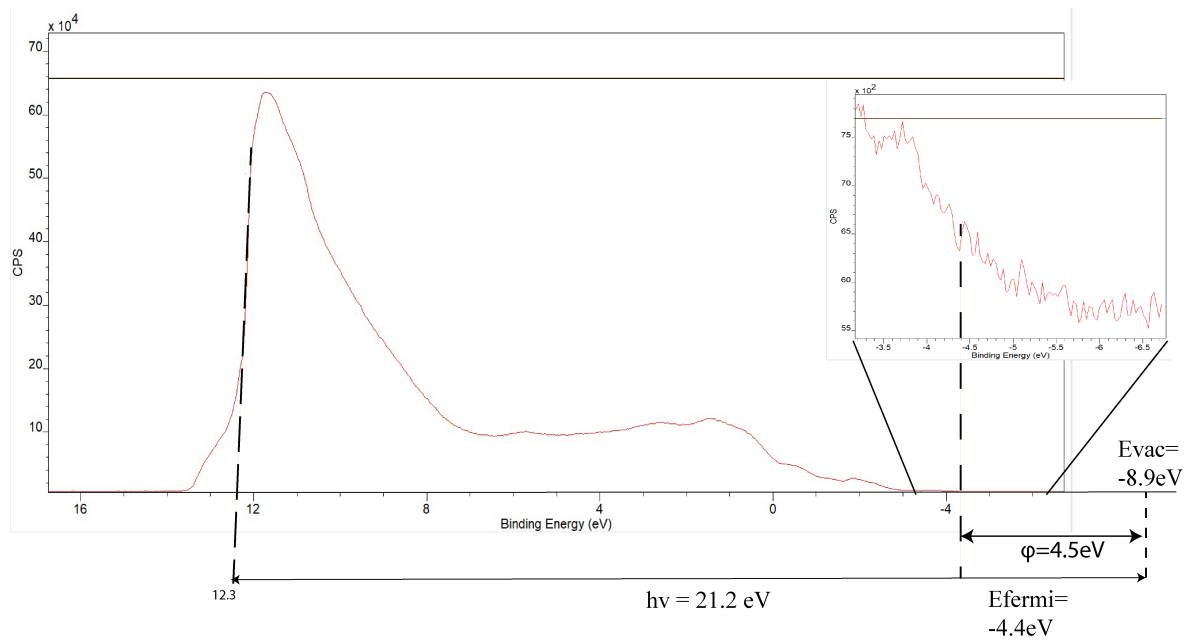
MoS₂

Figure 3.15: Measured UPS spectra of MoS₂ obtained work function 4.4 eV

Discussion

The STS measurement have considerable spread and consistently lower values than have been reported in literature. The values reported in literature for Aluminum, Au(111) and HOPG are 4.2, 5.3 and 5.0 [eV] respectively [6] [14]. This is not in accordance with the obtained results of UPS done in this assignment the maximum deviation between the literature and the measurements done is 1.5 [eV]. Which is closer than the measurements done with STS. A possible explanation for the obtained discrepancy is that the sample preparation was most likely different however the literature sources do not explain the used procedure for sample preparation.

The obtained results with STS were discussed with R. van Bremen a PhD student in the PiN-group. He noted that the same trend low work function and voltage depended work function were also observed on a different STM of the PIN group. A possible explanation given for the discrepancy between the literature values and the STS measurement is that bias of the tip induces a screening charge in the sample itself effectively lowering the vacuum barrier. This explanation is supported by the STS measurements on HOPG however this is contradicted by the measurements of Au(111) fig(3.9) and Mo₂ figures 3.6 and 3.7. It was also suggested that by letting the tip approach the surface of the sample 300 picometers from the current setpoint that there is the risk of the tip hitting the surface and possibly crushing the tip or making a hole in the surface. Which could explain the spread of the STS measurements however the after scans of the surfaces show no visible damage. Lastly the spread could have been caused by the tip picking up flakes from the surface of the samples changing its work function.

Furthermore it was suggested that the tunneling 1D tunneling model used is not adequate as it neglects the curvature of the tip instead a model should be used that takes the tip curvature into account. Also the STS measurements could have been done on different location of the sample surface to check the reproducibility of the

measurements

4.1 Conclusion

From the obtained measurement it is clear that scanning tunneling spectroscopy with the current tunneling model can not be used to reliably obtain local work functions on the used samples. The obtained results have considerable spread are bias dependent on some samples and the values obtained are too low compared to the values given in literature.

The values obtained with UPS are not fully in accordance with the values provided in literature but are closer to the expected value than STS. There are multiple explanations for the lower values obtained with STS but an inadequate model for the tunneling barrier is deemed the most likely cause for the systematically low values.

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Appendix

```
1 clear all
2 path = 'C:\Users\Jan Henk\Documents\Module 12\Aluminium STS\';
3
4 ElectronMass= 9.10938356*10-31;
5 plancksConstant=6.62607004*10-34;
6 ElectronCharge=1.60217662*10-19;
7
8 files = dir([path, '*.txt']);
9 i=0
10 for file = files'
11     i=i+1;
12     data = load([path, file.name]);
13     [fitresult, gof]= fit_Filter(data,0,7)
14     wf(i,1)=fitresult;
15
16 end
17 scatter(bias, (bias+wf))

1
2 function [avphi, gof] = fit_Filter(datamatrix, minwf, maxwf)
3 ElectronMass= 9.10938356*10-31;
4 plancksConstant=6.62607004*10-34;
5 ElectronCharge=1.60217662*10-19;
6 [m,n]=size(datamatrix);
7 l=n/2;
8 x=datamatrix(:, 1:2:(n-1));
9 y=datamatrix(:, 2:2:n);
10 indwf=zeros(l,1);
11 for k=1:l
12     x1=x(:,k);
13     data=y(:,k);
14 [xData, yData] = prepareCurveData( x1, data );
15 % Set up fittype and options.
16 ft = fittype( 'exp1' );
```

```
17 opts = fitoptions( 'Method', 'NonlinearLeastSquares' );
18 opts.Algorithm = 'Levenberg-Marquardt';
19 opts.Display = 'Off';
20 opts.Robust = 'LAR';
21 % Fit model to data.
22 [fitresult, gof] = fit( xData, yData, ft, opts );
23     b=fitresult.b;
24     phi= ((b/2)*plancksConstant/(2*pi))^2 / (2*ElectronMass);
25     phi=phi/ElectronCharge;
26     indwf(k,1)=phi;
27 end
28 [r,c]=find(minwf<indwf & indwf<maxwf);
29 clear xData;
30 clear yData;
31 x2=zeros(length(datamatrix),length(r));
32 y2=zeros(length(datamatrix),length(r));
33 if length(r)>0
34 for i=1:length(r)
35     x2(:,i)=x(:,r(i));
36     y2(:,i)=y(:,r(i));
37 end
38 [xData, yData] = prepareCurveData( x2, y2 );
39 [fitresult, gof]=fit(xData, yData, ft, opts);
40 b=fitresult.b;
41 avphi= ((b/2)*plancksConstant/(2*pi))^2 / (2*ElectronMass);
42 avphi=avphi/ElectronCharge;
43 end
44 if length(r)==0
45     avphi=0;
46 end
47 end
```