THOMPSON RIVERS UNIVERSITY

# Directed Studies in Physics (PHYS 4480)

# Scanning Electron Microscopy of Semiconductors

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# 1 Introduction to SEM

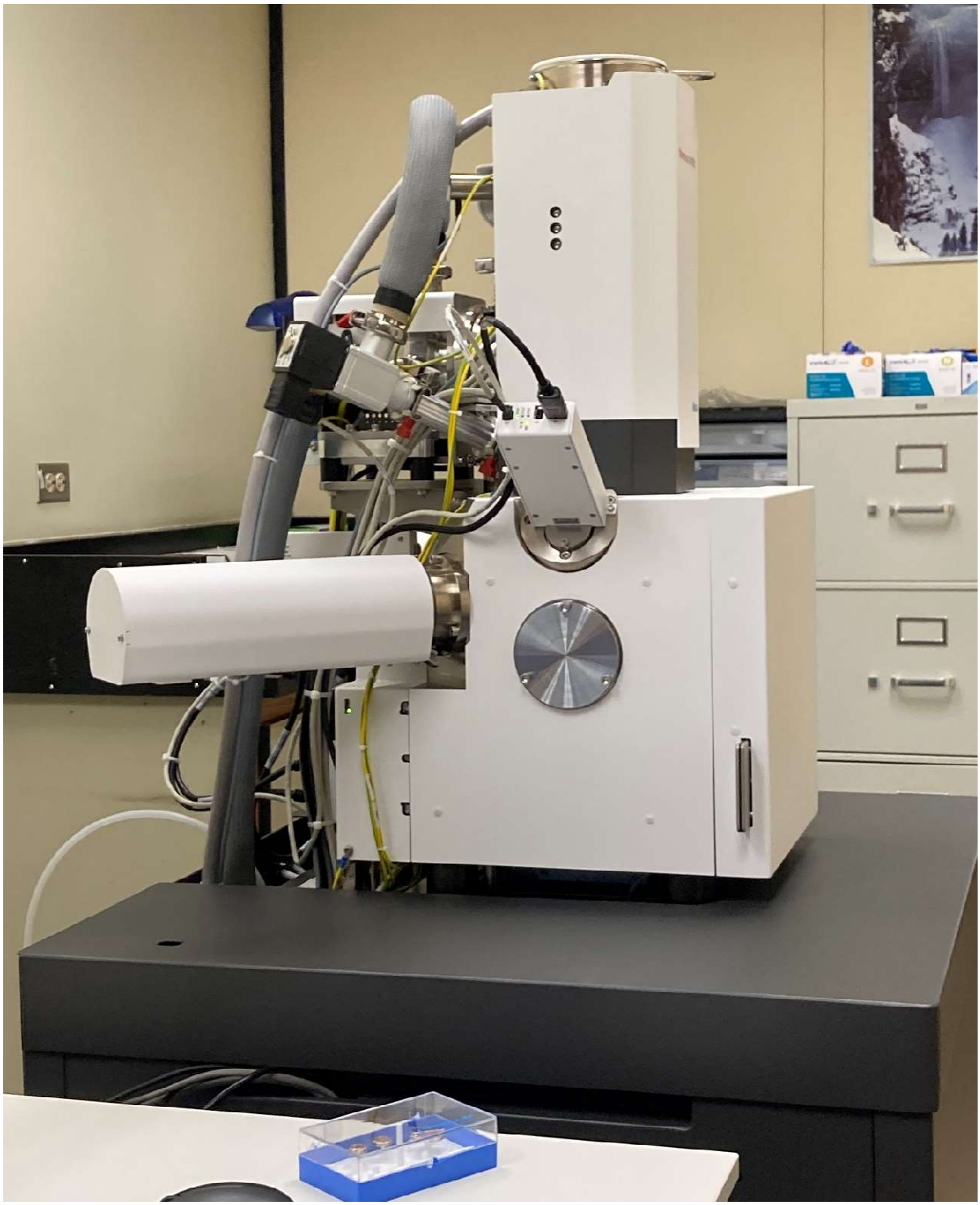

Figure 1-1: The Axia ChemiSEM used for this project.

The scanning electron microscope (SEM) is an important instrument for observing small structures. It can observe the topography of samples and determine structure using an integrated X-ray spectrometer. A beam of electrons is scanned across the sample to produce an image. The electron beam has a much smaller wavelength than visible light used in an optical microscope and can probe much smaller features. The Axia ChemiSEM used in this project can resolve features as small as 3 nm. The X-rays emitted from the bombarding the sample can be measured and used to determine the chemical composition of the sample using Energy Dispersive Spectroscopy (EDS/EDS) [1].

The main advantage of an SEM is the ability to resolve very small features on the sample. An optical microscope is limited by the wavelength of visible light for the smallest features it can observe. The theoretical limit for a visible light microscope is features as small as 200 nm. However scanning electron microscopes can resolve features as small as 1 nm [2],[3]. A high energy electron like what is used with the SEM is more localized than visible light and can be used to examine much smaller features [2].

The scanning electron microscope is an extremely useful tool for analysis of semiconductors; it can be used to examine the topography of semiconductor devices. It is excellent for the analysis of cutting-edge semiconductor devices on the scale of a few nanometers. To analyze the structure of these devices instruments which can reach that scale for analysis are necessary. Small scale structures made of semiconductors have the potential for improved efficiency and performance compared with larger scale counterparts. The SEM is an extremely useful tool which is commonly used to examine the structure of cutting edge nanomaterials in research settings [2],[4],[5]. Figure 1-2 shows an example image of a nanoscale semiconductor device imaged using SEM.

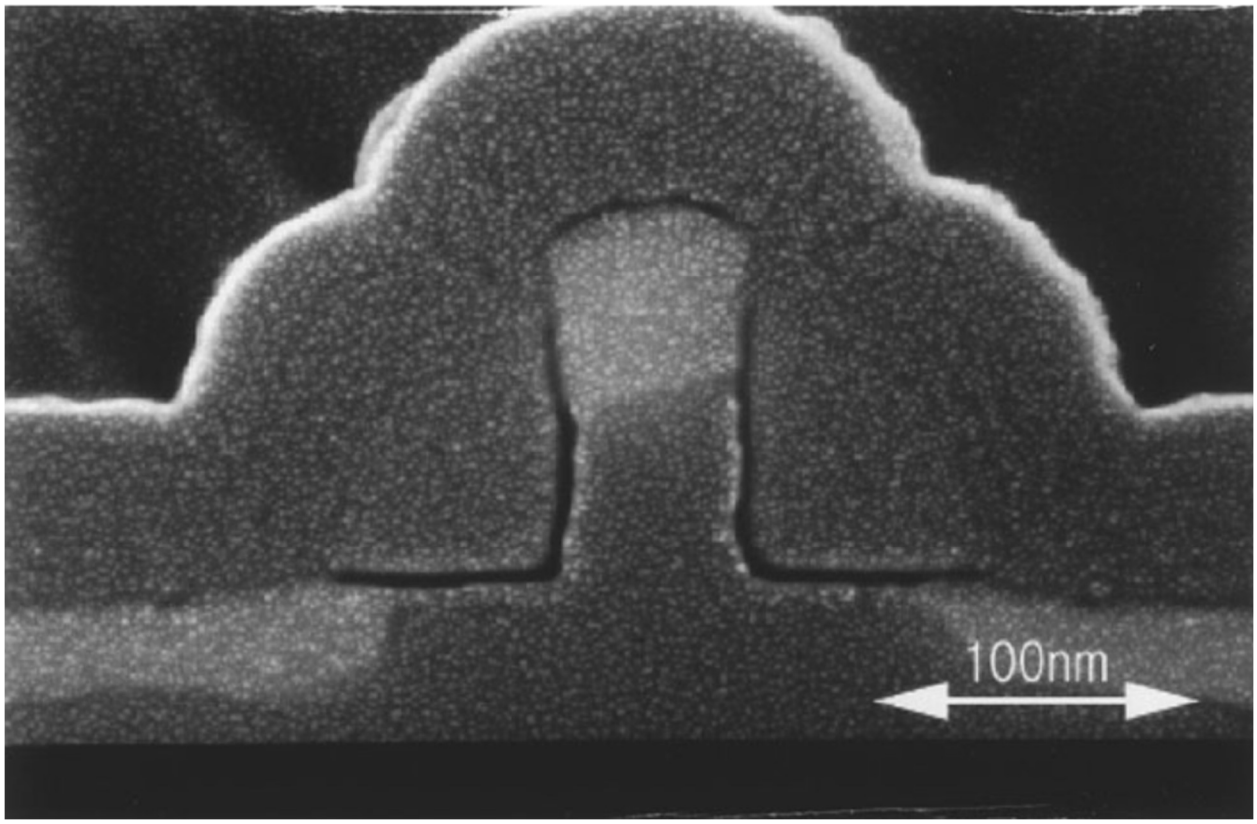


Figure 1-2: Cross section of a semiconductor device coated with 2 nm of platinum taken at 9 keV; nanoscale junctions and gaps in the device are visible with SEM [[6], figure 5.8].

A schematic of an SEM is shown in Figure 1-3, at the top is the electron source, an electron gun. The electron gun is a tungsten filament which is heated to produce electrons and charged plates 200 V to 30 kV which accelerate the electrons to energies in the range of 200 eV to 30 keV. After the electron gun there are typically two condenser lenses to parallelize and narrow the beam. The final objective lens focuses the beam into a small area for imaging. The scan generator is made of magnetic scanning coils that move the beam across the sample. This is either above the objective lens (shown in diagram) or integrated into it. The electrons that scatter off the sample are collected by detectors like the secondary electron detector in the diagram. The signals from the electron detector are measured and combined by a computer to produce a final image of the sample [1],[2].

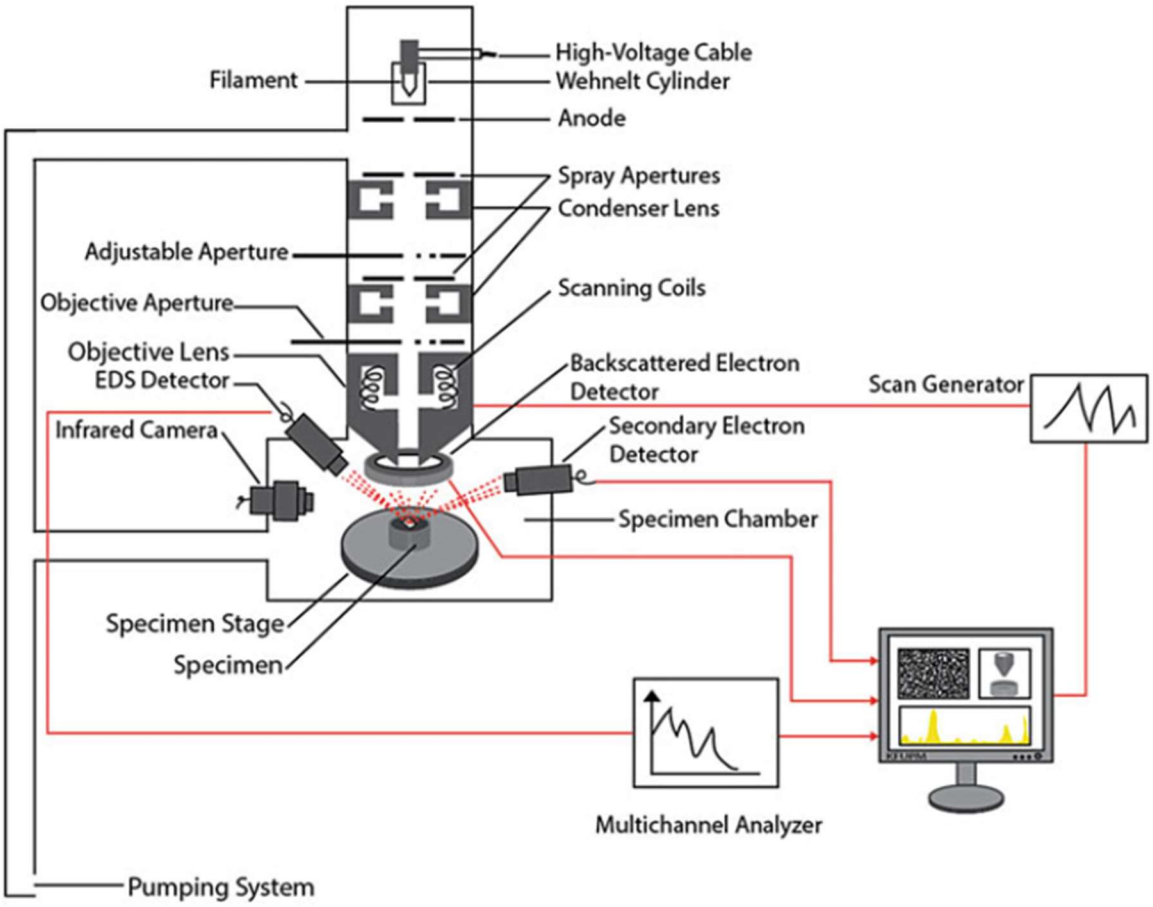


Figure 1-3 schematic drawing of a typical SEM, the electron gun at the top fires a beam of electrons through the optics which parallelize and focus it. The beam impacts the sample and electrons scattered and emitted from the specimen, these are recorded by the detectors to create the image of the sample [[1] figure 2.2].

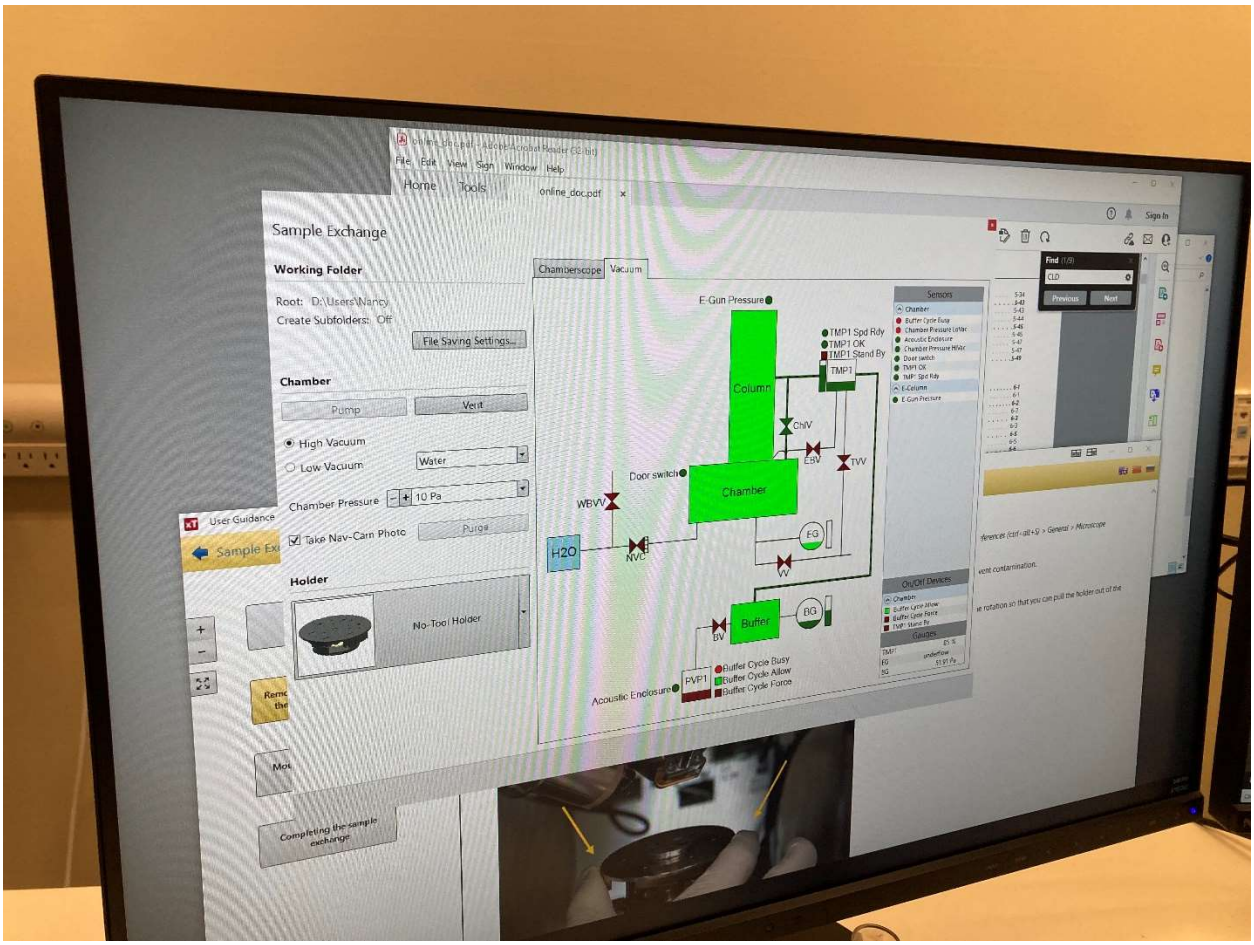


Figure 1-4, the block diagram of the SEM as seen in control software when operating. The gun and optics are in the column, the sample and detectors are in the chamber, the buffer is connected to the vacuum pump which runs periodically, and the water reservoir is used for low vacuum imaging.

In the control software for the Axia ChemiSEM the upside-down funnel button shows block diagram of SEM. The block diagram is similar in structure to the overall diagram, but it focuses on the operating parts of the microscope. This allows for the overall state of the microscope to be monitored while it is operating. It shows values and connections between the different parts of the vacuum and colours them green when the are under vacuum and brown when they are not [7].

## 1.1 Image formation

The beam electrons interact with the sample in a variety of ways which are measured for various types of SEM analysis. Electrons emitted and scattered from the sample are categorized by their energy. Secondary electrons (SE) have less than 50 eV and back scattered electrons (BSE) have greater the 50 eV. Most secondary electrons come from high energy beam electrons exciting electrons from shells of the sample material. The excited electron is emitted from the sample and recorded by the detector. The higher energy back scattered electrons are incident beam electrons which through many scattering processes within the sample 'back-scatter' to the detector. When secondary electrons are excited out of an atomic shell, they leave a gap which a higher energy electron can fill emitting a photon as it does so. For inner shells these are characteristic X-ray photons, their energy can be measured to determine the sample with an energy dispersive X-ray spectrometer (EDS). If the secondary electron is emitted from a less tightly bound shell the photon emitted may be visible or near visible light, such photons can be measured using cathodoluminescence (CL) [1].

The electron beam is scanned across the object to produce an image. The scan is conducted using the scanning coils or scan generator which move the beam across the sample. The scan is conducted in a raster or grid pattern for each pixel in the final image. At each location the beam stays there for a small amount of time called the dwell time, during this time the number of electrons coming from that area is recorded and the count is used to create the image. The higher the count the whiter the final image. The beam can be focussed to smaller areas and scanned in smaller increments to further zoom in and create a finer image. With ideal conductive samples such as metal the ideal resolution of up to 1 nm can be achieved. With non-ideal samples the main limitations are charging, and the noise introduced by focusing the beam on narrow spots. The ChemiSEM which has can resolve features as small as 3 nm [1],[7].

The contrast to topography of the SEM is achieved through the interaction of electrons with the sample. Electrons are more likely to scatter off portions of the sample which are angled compared with the beam. The angled sections have less material for the electrons to scatter though compared perpendicular sections, so electrons are more likely to escape the sample in these directions. This is why BSE images especially are very sensitive to edges in the sample, the high energy electrons are more likely to escape and scatter to the detector (Figure 1-5) [1].

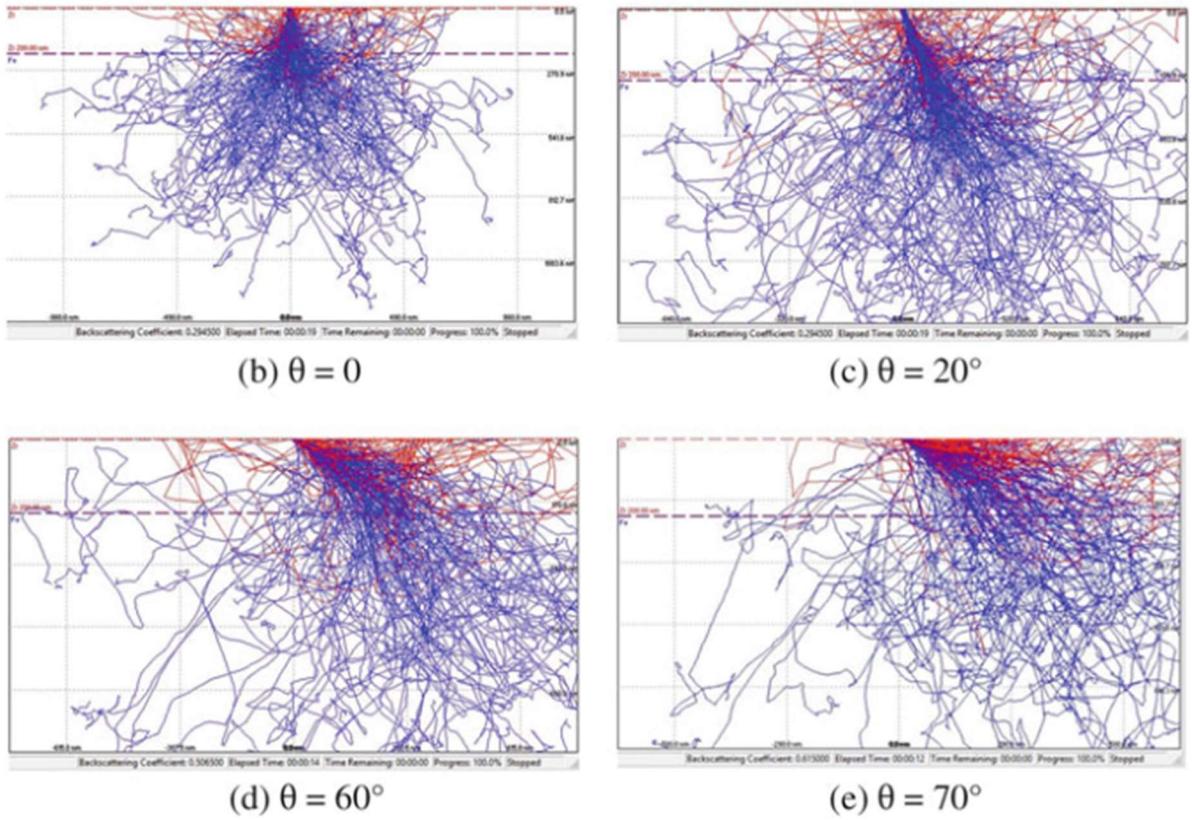


Figure 1-5: Monte Carlo simulations of electron trajectories in an iron sample coated with zircon at various beam incidence angles. As the angle increases a larger proportion of back scattered electrons are able to escape the surface of the sample increasing the count of electrons in those areas [[1] figure 3.16].

For general imaging, there are many parameters which can be changed to change the overall image. The beam current is the current in the electron beam from the gun through the sample to the ground. Increasing the beam current increases the number of electrons which scatter of the sample and can produce better images, it also means the sample must dissipate more charge and can result in heating and charging from the increased number of electrons. The ChemiSEM can be used with beam currents up to 2 $\mu$A but smaller values are generally used. The beam energy is the energy of incoming electrons typically measured in keV, where an electron volt is the energy of one electron accelerated by a voltage of 1 volt. The beam energy is the energy of the accelerating voltage applied in the electron gun. High energy electrons scatter more readily and produce more scattering events, but they may damage the sample. Increasing the beam energy

makes more X-rays available for EDS analysis. The ChemiSEM can operate with beam energy from 0.2 to 30 keV. The spot size is the size of the spot the electron beam is focused on; larger spot sizes are less likely to damage the sample but cannot achieve as high useful magnification. The SEM can create composite images using frame averaging and integration; these allow for an image to be created from multiple passes over the sample, this increases the contrast from low current and energy while avoiding causing damage to the sample [1], [2],[7].

## 1.2 Advantages and disadvantages of SEM

The SEM is a very flexible instrument, which can determine large amounts of information about samples used. This microscope is intended to be primarily used for geology for which the ChemiSEM is well-suited. In comparison to other techniques the SEM can is slightly less flexible in sample type and preparation than a visible light microscope, but it can achieve much greater magnification. The SEM does not have the same sample size restrictions as AFM or TEM for only slightly lower resolution. The strengths and weaknesses are summarized in Table 1-1: [1],[2].

| Advantages | Disadvantages |
|---|---|
| Variety of samples can be imaged | Limited sample size and mass |
| Simple preparation | Can only use solid samples |
| Relatively fast and easy to use | EDS cannot detect elements lighter than Be |
| Large depth of field | EDS is less accurate than solution methods |
| High resolution | Images under vacuum or low vacuum only |
| Mostly non-destructive | Non-conductive samples must be coated |
| Can image sample topography | The beam may penetrate very thin samples |
| High depth of field | |
| EDS can identify composition | |

Table 1-1: Summary table of the advantages and disadvantages of the SEM,[summarized from [1],[2]].

## 1.3 Detectors

The detectors in the microscope collect photons and electrons emitted and scattered by the sample when the electron beam is incident on it. The detectors collect the particles and convert them to electronic signals which are processed by the computer and made into the final images. This section will explain the working principles and use of the detectors in the ChemiSEM, and explain some of their use for semiconductor samples [1],[2].

### 1.3.1 Secondary electron detector

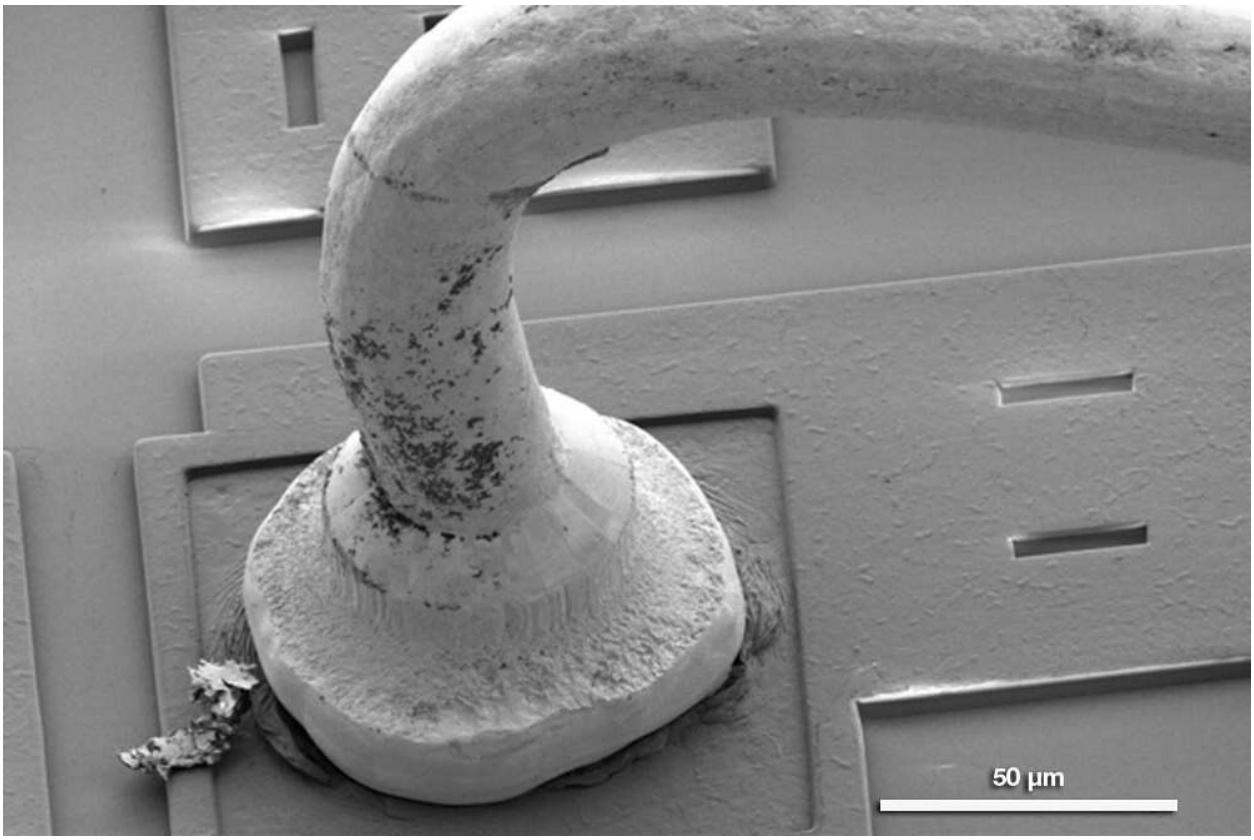


Figure 1-6: 2 kV image of a wire bond connected to s semiconductor circuit, generated using an Apreo SEM with an Everhart-Thornley detector [8].

Secondary electrons are the most common detector for image formation in the microscope. These are electrons which scatter off the sample and have energy less than 50 eV. A secondary electron is created when a primary beam electron 'knocks' an electron out of a shell of an atom which can then scatter to the detector. When a secondary electron is excited out of an inner shell by an

incoming electrons X-rays are released which are then used for EDS. Secondary electrons are the most used for SEM analysis. They are excellent for general imaging and a large number are produced by the beam incidence on the sample [1].

The detector in this microscope is an Everhart–Thornley detector (EDT). The EDT is a scintillator-photomultiplier detector. A scintillator crystal generates photons when an electron enters it, the photons are carried by a light pipe to a photomultiplier which converts the photons to electrons and amplifies the electrical signal. The EDT detector uses a charged faraday cage with a low positive charge around the scintillator crystal to attract low energy secondary electrons, higher energy electrons move too fast for this charge to attract so it mostly detects secondary electrons [1]. In some microscopes this can also be used for BSE, but in the ChemiSEM it is for SE only and a separate detector is used for BSE [7].

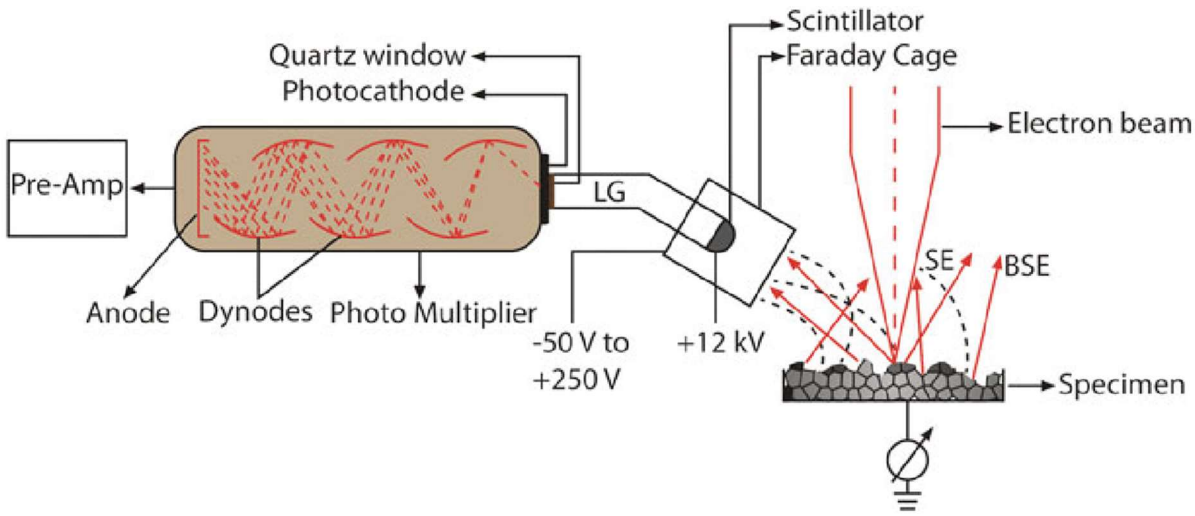


Figure 1-7: Schematic of an ET detector, secondary electrons (black dashed lines) are attracted to the charged faraday cage at +250 V. The scintillator converts these electrons to photons, and they pass through the LG (light guide or light pipe) to the photomultiplier tube which amplifies and records the signal. When the faraday cage is at -50 V the SE are reflected away from it and only high energy BSE pass through it allowing the detector to be used for both types of imaging [[1] figure 2.30].

In the ChemiSEM this is primary detector, the charged faraday cage attracts a large fraction of the secondary electrons allowing for high resolution images to be created. The detector is placed

to the side of the main column angled towards the sample. This is analogous to an optical microscope with the lighting coming from one side giving the topography a shadowed effect that is easy to interpret creating useful images. It has a high signal to noise ratio allowing for very zoomed in high resolution images to be created [1].

Secondary electron images are most useful in examining the topography of semiconductors and semiconductor devices. These images can be used to look examine the overall structure of devices such as solar panels, diodes, and transistors. Secondary electrons are very useful for examining the physical structure of semiconductors and this detector is an important part of general analysis of such samples with the SEM [2].

### 1.3.2 Back-scattered electron detector

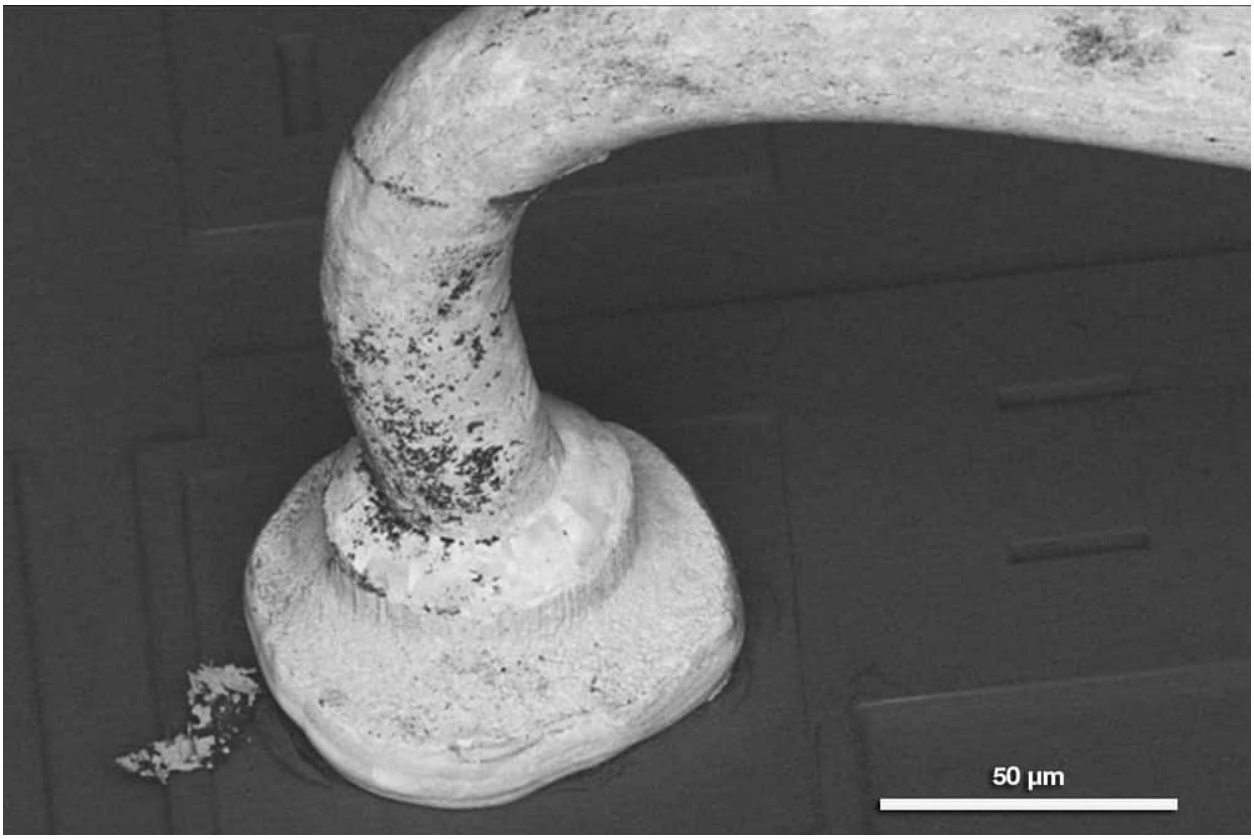


Figure 1-8, BSE image of the same wire bond as in the example picture for SE, generated using an Apreo SEM with a concentric back-scattered (CBS) detector. The contrast on the wire is much greater than the SE image of the same junction, however the back circuit is not shown as clearly [8].

The higher energy back scattered electrons are typically used to reveal the surface topography of a region. They primarily come from incident beam electrons which scatter through the sample in a variety. The less of the sample is blocking the electrons the more of them are detected. Since these electrons are high energy, they are more likely to be seen from areas of the sample which are angled compared to the detector. This makes BSE extremely useful for investigating topography of samples and it can clearly show edges and internal boundaries of the sample [1].

The ChemiSEM has a under the lens back scattered electron detector. This is a retractable detector which can be interested to take images and removed to use other detectors [7]. The detector is either a scintillator photomultiplier detector like the EDT secondary electron detector or a pn semiconductor crystal. If it is a pn junction detector it is made of a junction with a bias voltage applied by thin electrodes on either side. When an electron enters the detector, it excites electron hole pairs proportional to its energy. The pn junction and bias voltage prevent electron hole pairs from recombining, the follow the applied voltage and are amplified and detected to create the overall signal (Figure 1-9). The contrast comes from the sensitivity to the energy of the electrons [1].

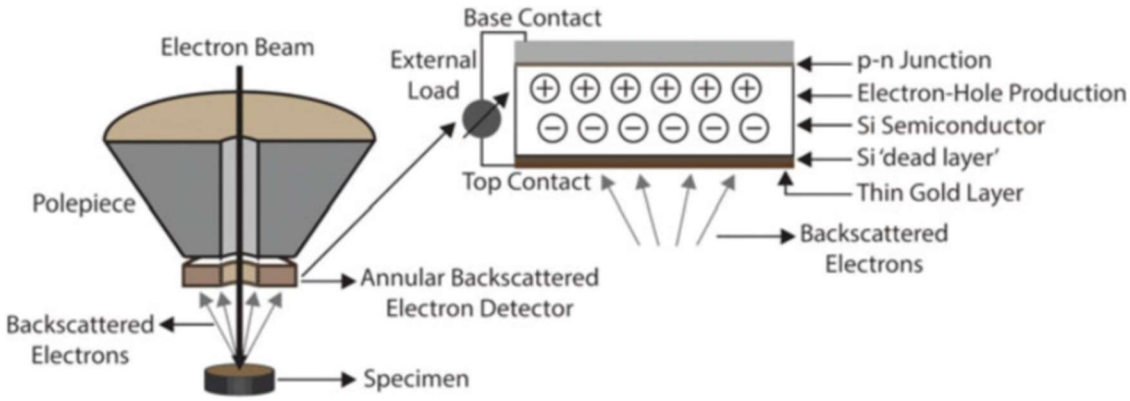


Figure 1-9: Back scattered electron detector, the electron beam passes through the hole in the centre, after scattering off the sample, high energy electrons are scattered up into the detector. These electrons interact with the detector to produce many electron hole pairs proportional to the energy of the incoming electron [[1] figure 2.36].

### 1.3.3 Energy dispersive X-ray spectroscopy

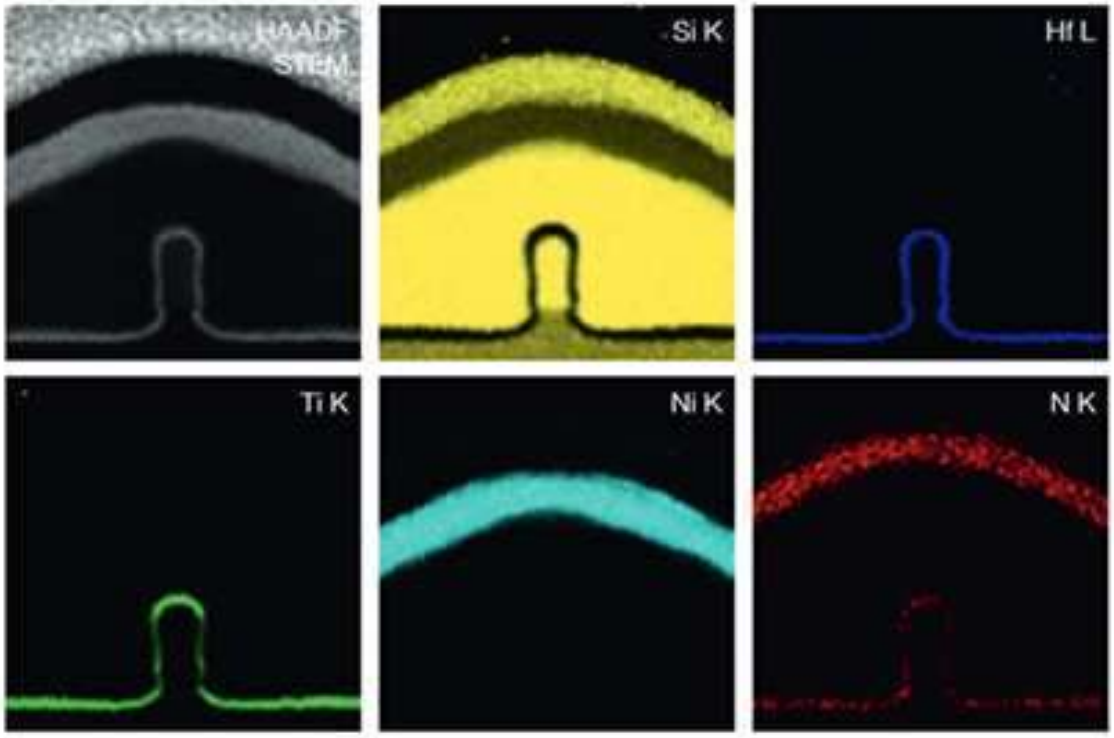


Figure 1-10, EDS map of a transistor, the locations of each elements X-ray emissions are shown. This was taken using a Silicon detector doped with lithium [9].

Energy Dispersive X-ray spectroscopy (EDS/EDS) detects X-rays emitted when a sample is under the electron beam. The beam excites electrons in the sample to higher energy states than the ground state; when these decay, they emit an X-ray photon. The energy of the X-rays emitted are characteristic of the element being examined. These photons are recorded by a spectrometer which measurers their count and energy, these are used to create spectrum of the sample. This spectrometer measures the energy of incoming photons using a silicon crystal. Incoming electrons excite electron hole pairs in the crystal with a number proportional to the energy of the incident X-ray, the amount of these pairs is measured and used to calculate the energy of the incident photon [1].

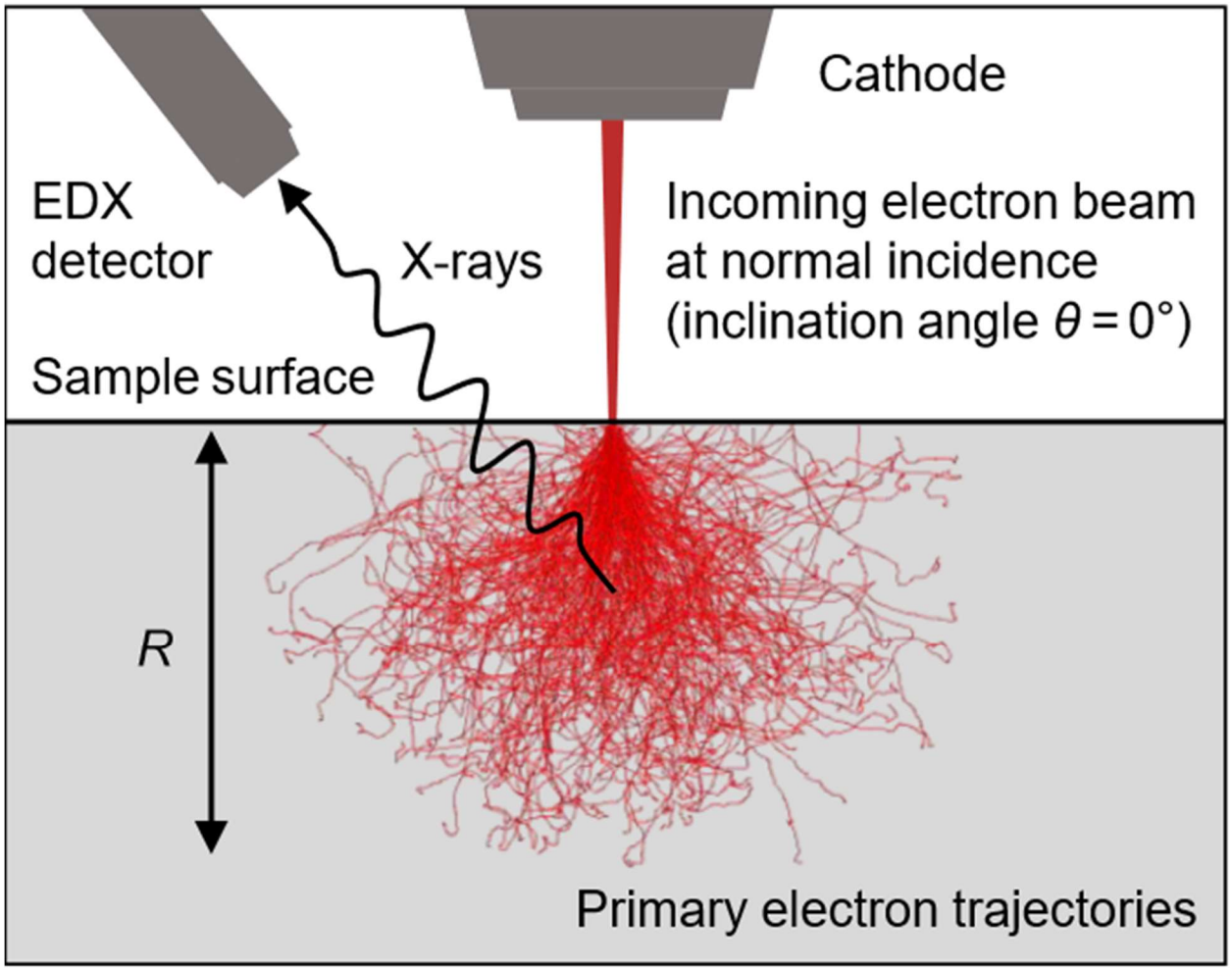


Figure 1-11: Diagram of EDS in an SEM, when elements in the sample are excited by primary beam electrons they emit X-rays, these X-rays are measured using the EDS detector [[10] figure 1].

Using the scanning of the electron beam EDS maps can be created, by measuring the count of X-rays at each point over the scan maps can be created which overall colours for the count of X-rays on an SEM image (Figure 1-10). These maps show where specific elements are in a larger sample. EDS analysis is an important part of SEM analysis especially for the geology applications of this microscope. Different rocks and minerals made of different materials can be identified with EDS. For semiconductor samples EDS can identify impurities in a sample if they are concentrated in a small area. EDS can also identify materials in heterogenous semiconductors made of different materials joined or layered together. Doping may or may not be identifiable depending on the dopant concentration in the semiconductor material. If components of the

semiconductor migrate through the larger crystal EDS can detect concentrations of these materials [1],[9],[10].

### 1.3.4 Cathodoluminescence

Cathodoluminescence (CL) is like EDS, but instead of measuring X-ray emitted by the sample, visible and near visible light is used. When the electron beam interacts with a sample which has a bandgap in or near the visible light range. It excites electrons from the valence shell of electrons in the sample creating an electron hole pair. The electron can drop into a hole re-entering a valence shell and emitting a photon. The pairs which flow away from the beam site and recombine emitting a photon with the energy of the bandgap, these are the photons recorded by the CL detector (Figure 1-12) [1].

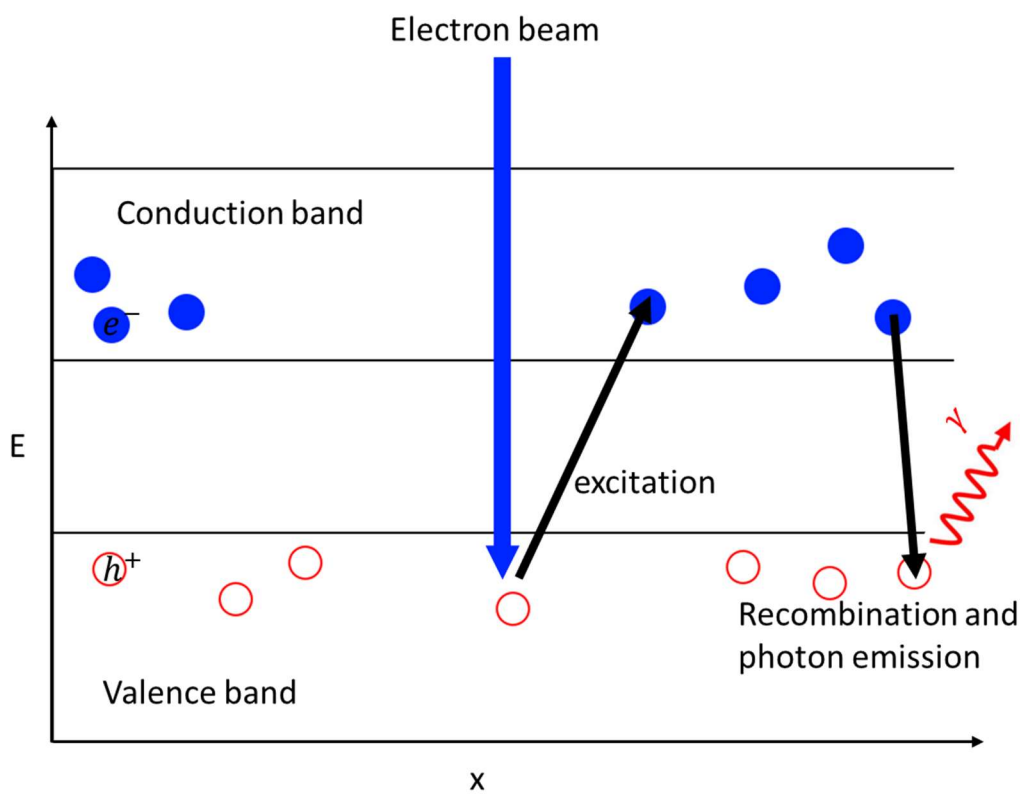


Figure 1-12, band diagram view of cathodoluminescence. Incoming electrons from the beam excite electrons in the sample into the conduction band generating electron hole pairs. The electron hole pair recombine emitting a photon. These photons are measured by the cathodoluminescence detector.

The RGBCL detector in the ChemiSEM can detect cathodoluminescence in the range of 350 - 900 nm (1.38-3.54 eV). Mirrors collect light emitted from the sample when bombarded by the electron beam; this light is fed to a photomultiplier and amplifier which converts the photons to electrons and amplifies the electrical signal to be collected (Figure 1-13). This signal can be used to create cathodoluminescence overlays and spectra for samples [1], [7], [11].

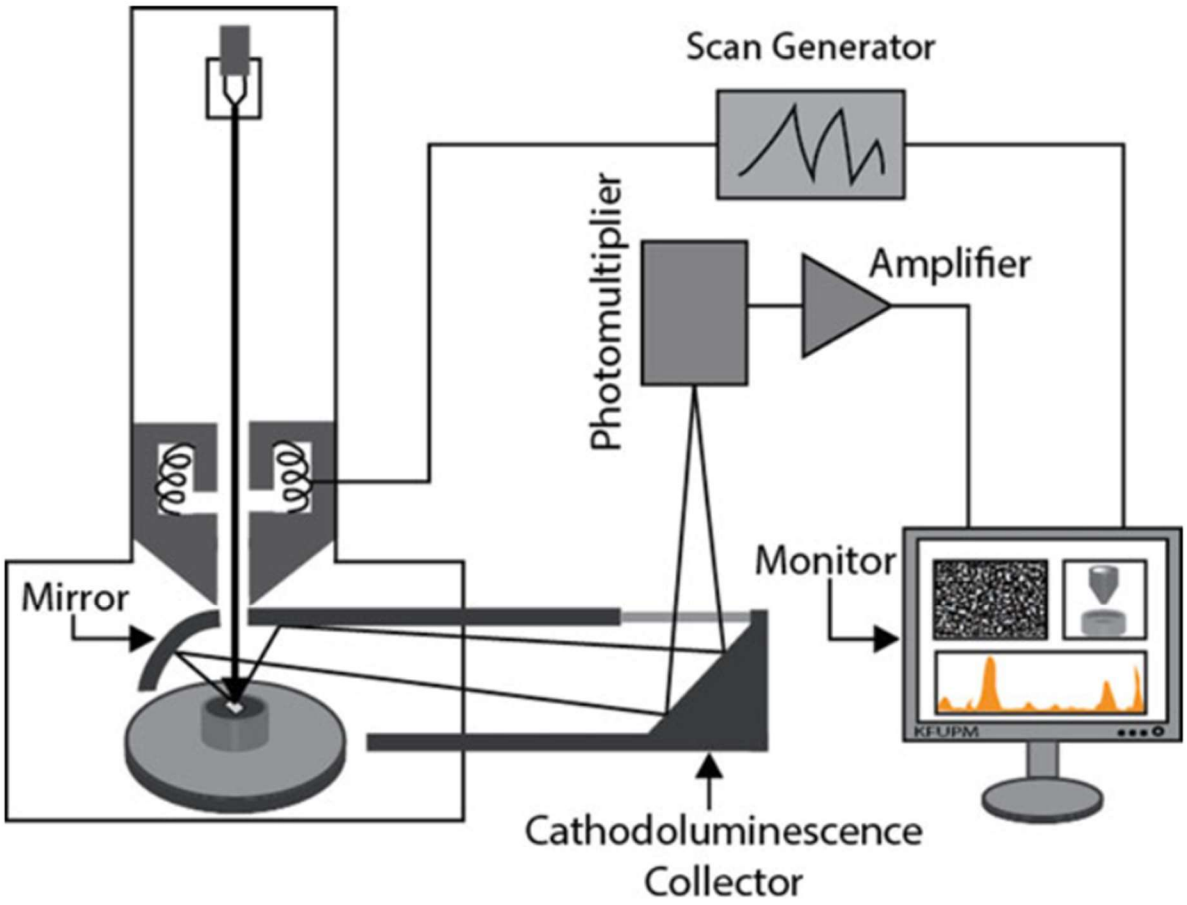


Figure 1-13: Cathodoluminescence detector in an SEM. CL photons emitted from the sample are gathered by mirrors and analyzed to create CL images [[1] figure 5.29].

The CL photons are sensitive to the band structure of the sample and can change based on influences on the band structure across the sample. Since it is sensitive to the band structure of the sample it an extremely useful technique for examining semiconductors. It can detect defects, damage, and features which cause the bandgaps of the sample to change. For geological samples

it can detect differences in crystal structure, and material where other techniques like EDS are not applicable [1][12].

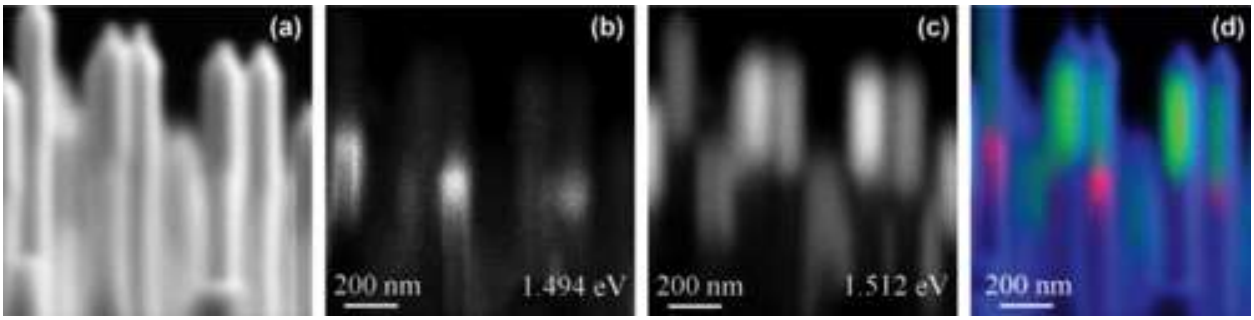


Figure 1-14, CL images of wurtzite and zinc-blende nanowires. The crystal structure changes over the length of the wire, top is zinc-blende and the bottom is wurtzite; crystal the change in crystal structure changes the energy of the emitted CL photons [[5], figure 5].

# 2 Semiconductor samples

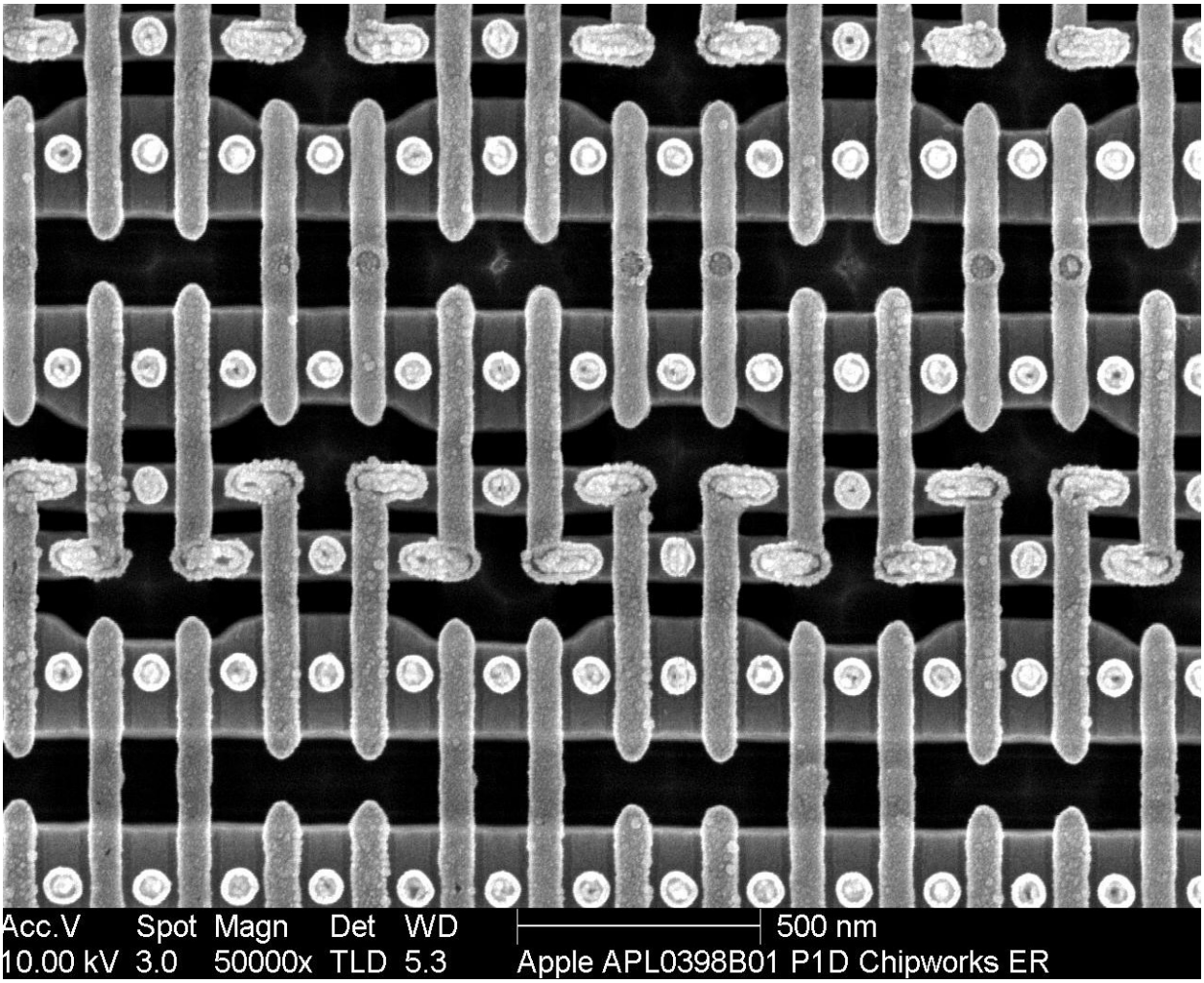


Figure 2-1 A memory cell in the apple A4 microprocessor imaged with 10 kV and a through the lens TLD SE detector. This is a semiconductor sample viewed at high magnification which shows the wiring structure of the memory cell [13].

The scanning electron microscope is an excellent tool for looking at semiconductor samples, however semiconductors require some modifications from standard samples to get good images. The difference in conductivity between semiconductors and conductors makes semiconductors more difficult to examine. This requires specific sample preparation to minimize these effects.

settings will be different than for a fully conductive sample and a non-conductive one, sometimes coating is needed to prevent changing, but it is not always necessary [2],[14].

## 2.1 What is a semiconductor

A semiconductor is a material with a conductivity between that of a metal conductor like copper, and an insulator like glass. Semiconductors are found near the 'metalloid staircase' in the right of the periodic table. A binary semiconductor is one group 14 element like silicon or germanium. A compound semiconductor is made of a combination of elements in groups on either side of 14 such as 13-15 gallium arsenide, or 12-16 zinc oxide [15].

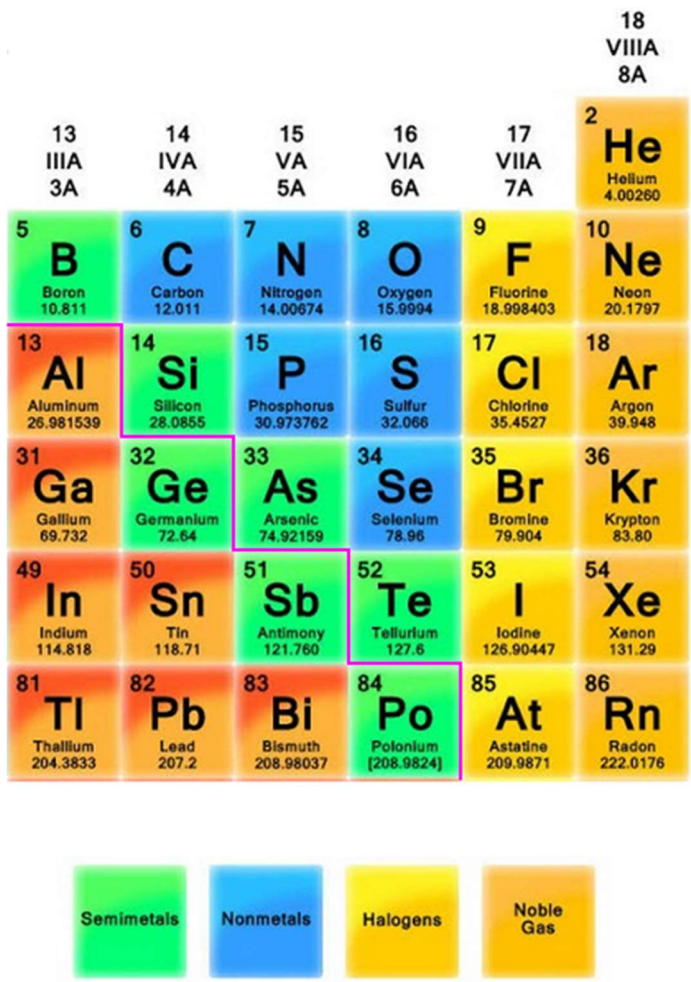


Figure 2-2, The top right of the periodic table, the most common semiconductors are found near the purple metalloid staircase such as pure silicon and germanium. Combinations of other elements such as gallium and arsenic combine to make composite semiconductors [16].

In a crystal, electrons are shared between atoms so that each has a full valence shell, these bind the structure together. Most electrons stay in the valence shell, however when excited to a higher shell by electromagnetic radiation or an applied voltage electrons can move around the crystal more freely. When an electron is excited out of a valence shell the gap it leaves behind can be filled by an electron from a nearby valence shell, the gap can move around the crystal. This creates a quasiparticle called a hole, the hole can move around the crystal like an electron increasing the conductivity [14].

A complete semiconductor crystal is best understood through bands rather than valence and higher shells. When the periodic potential of the repeating regular structure of a crystal with positive point charges from the nuclei for the electrons in a crystal is solved in the Schrodinger equation the band structure emerges. The shells of atoms act like bands across the complete crystal structure. The valence shell becomes the valence band where nonexcited electrons are bound to atoms. The higher shells form the conduction bands, when an electron is excited out of the valence band it goes to a conduction band where it can move around and conduct electricity through the semiconductor. The excited electron leaves a hole behind in the valence band which can also move around the crystal and add to conduction [14].

The overall properties of the crystal can be changed by the inclusion of impurities in the crystal through a process called doping. Replacing silicon atoms in a crystal with a group V element like phosphorous adds extra electrons which move are free to move around the crystal; this is called n-type doping because it adds extra negative electrons. Replacing silicon with a group III element like boron adds positive holes to the crystal which can also move around; since this adds positive charges it is called p-type doping. Different amounts of doping allow for the properties of a semiconductor to be tuned as is necessary for an application. By combining semiconductors with different kinds of doping in them together semiconductor devices can be created such as diodes, transistors, or solar cells [14].

## 2.2 Settings for semiconductor samples

As the conductivity of a semiconductor falls somewhere between that of a full conductor and a metal dealing with charging in semiconductors is important under the SEM. They tend to be somewhat beam sensitive and so require care to be imaged well. The main consideration is

thermal damage, since the samples are less conductive than ideal metal or metal coated samples. To minimize this semiconductor samples should be imaged with low beam current and energy [2],[14].

The main procedure recommended by the manufacturer is as for any potentially beam sensitive samples. The power delivered to the sample by the beam needs to be minimized to the level where it can be dissipated by the sample to the microscope without significant sample heating occurring. Thermal breakdown is a major factor in beam sensitive samples and high energy electron bombardment can cause heating to the point of sample damage. This can be achieved in many ways and for specific samples some experimentation is needed to get good images while maintaining sample integrity. Using low beam energy of less than 5 keV, low beam current < 20 pA are the most basic imaging parameters these lower the overall power the beam delivers. Increasing the spot size and lowering the overall focus of the beam will make it so less power is delivered to the points it is focused on. To make up for this frame averaging and integration can be used to create an overall composite image. This creates a composite image from multiple passes over the sample allowing for charges and energy to dissipate from the sample in between passes. Increasing the amount of frame averaging will increase the contrast of the overall image, this setting is recommended to be used for semiconductor samples, allowing sites to cool between passes and building up an overall image through many passes can create high quality images [1],[8]. In general, for first passes of SEM imaging on semiconductor samples I recommend using 2 keV energy, spot size of 2.0, dwell time of 200 ns and frame averaging of 4. This uses low energy and frame averaging to create a composite image, these settings should minimize sample damage and allow for good images to be created [2], [8].

## 2.3 Limitations

The ideal samples for a scanning electron microscope are metal with some texture to them. Such samples are very conductive and have features to observe. Semiconductors are less conductive than these samples which means they cannot reach the same resolution as is possible with the ideal sample. The same very small resolution cannot be reached on semiconductor samples. High beam energy cannot be used with semiconductor samples it risks damaging them. Dopants cannot generally be detected because the level of a will dopant generally fall below the detection level for EDS. X-rays from these materials may be detected but for low level doping it will most

likely not be a conclusive detection. Some semiconductor devices have built in electrical characteristics which may cause issues with some detectors. Nanostructures may be especially sensitive to the electron beam. Semiconductors which have volatiles in them, or coatings of volatiles can only be imaged in specialized instruments which are equipped to image samples that outgas or with preparation by removing those layers from the sample [2],[8].

# 3 Sample selection and preparation

Selecting and preparing samples appropriately is very important for obtaining good results when imaging. Improperly prepared samples will produce poor images. Samples which are not cleaned properly could release dust and debris on them can damage the microscope. Beam sensitive samples and volatiles my break down under electron bombardment contaminating the chamber. With careful selection and preparation many semiconductor samples can be imaged with SEM [2], [8].

## 3.1 Selection

The samples used were selected to test the microscope on semiconductors. The samples were believed to be beam stable and had properties worth examining with the microscope. To select semiconductor samples for the microscope the main issue is conductivity. The ideal sample for the SEM is metal or metal coated so the electrons can be easily conducted away from the beam and prevent sample damage. Semiconductor samples to be examined need to somewhat conductive, however thin surface oxide layers from exposure to air can be imaged at low beam current without issue. Semiconductors can be imaged to the scale of a few $\mu$m to 100s of nm with such samples. To get very high-resolution images and avoid beam damage over the long term a coating must be used. With low beam current and energy and careful monitoring of the sample inside the chamber for beam damage and breakdown semiconductor samples can be reliably imaged with the microscope [1],[8].

SEM samples need to be sized to fit inside the sample chamber, standard sample stubs come in a range of sizes with 12 mm being the most common. A variety of sizes of stubs can be used with the larger stubs suitable for larger samples. For samples larger than there are stubs other sample holders can be used in the microscope. For the semiconductor samples used the stubs were suitable so no other mounting hardware was investigated. The ChemiSEM sample chamber can fit samples up to 500 g and shorter than 72 mm tall with full 5 axis movement. Larger and heavier samples can be accommodated but this requires the translating stage to be removed limits the flexibility of the microscope [1],[7].

Many semiconductor samples can be used for imaging with the SEM if they do not have nonconductive coatings like glue or plastic over them. Most semiconductors and semiconductor devices can be imaged with a low vacuum SEM where the low vacuum allows for surface charges to be conducted away. Semiconductors can also be imaged with low beam current and energy at high magnification, but they tend to be beam sensitive. Exposed semiconductor samples such as general semiconductor devices with coverings removed and lab created semiconductor samples can be used for SEM imaging, experimenting with settings is important for imaging semiconductor samples [1],[8],[17].

## 3.2 Mounting

The mounting process is a clean process, the mounting of all samples was conducted on new clean sheets of aluminium foil. For PPE a lab coat, gloves, safety glasses, and a face mask were worn throughout the mounting process. All tools were cleaned with isopropyl alcohol before touching samples and were placed on the aluminium foil when not in use. The samples should only be handled with tweezes when being mounted and the final stubs should be only handled with gloves and tweezers. Once the sample is mounted on the stub the final product should be stored in a plastic sample case and only opened wearing the same PPE as for mounting it to load into the microscope. The samples should be inspected for debris before being used in the microscope and if any is present, they should be thoroughly cleaned until none is visible. The geology optical microscopes can be used for this inspection. They can examine a wide variety of samples mounted on SEM stubs and placed in the sample holder.

In the future if they are available hair nets should be worn. During the mounting the sample must be kept away from you face even for examination so it is not contaminated with hair or debris from breathing on it. In the long term as part of a sample preparation setup conducting the process in a fume hood running on low to eliminate dust and using magnifying lenses may allow for better samples to be prepared. Setup of such a permanent sample prep area would be useful.

All samples used for this project were small, so we used SEM stubs to mount them. These stubs are made of aluminum and press fit into the no-tool holder of the SEM. The no-tool holder has holes sized to securely hold stubs in while in the microscope. The standard mounting procedure for a stub is uses conductive circular carbon tape stickers. A sticker is attached to the stub and

then the sample is firmly mounted to the sticker, the remainder of the carbon tape is covered by the sticker to improve conduction away from the sample. Once the sample is attached firmly to the stub hold it in round SEM stub tweezers and tap it on the aluminium foil table cover to remove any loose debris. Using the hand operated air squeeze blower and canned compressed air the final product can be cleaned to remove further contaminants. Compressed air should not be used in the same room as the microscope as it may be flammable and disturb dust in the SEM room [17]. Specific mounting procedures for each sample used in this project are described in chapter 4, for step-by-step instructions on mounting a semiconductor sample see the appendix.

## 3.3 Sputter Coating

A sputter coating is sometimes used for SEM samples, it was not used in these directed studies but is typically used for beam sensitive samples. A thin layer of conductive material is applied to the outside of the sample allowing electrons to better conduct across the surface and preserve the underlying sample. The coating can protect the sample from beam caused damage while preserving the underlying topography so it can be analyzed. The coating can prevent charging of the sample and improve the resolution achievable with the microscope by reducing noise. Ideally a coating allows small structures in non-conducting samples to be imaged like they are in conductive samples. It also can protect beam sensitive samples like organic molecules and allow for them to be analyzed in high vacuum. Where only the topography is important a coating of gold, palladium, or platinum is typically used. This coating will cause large amounts of those materials to be detected under EDS. For EDS and CL analysis of the underlying structure a carbon coating is typically used; the carbon X-ray spectrum does not have significant overlaps with any other elements so the underlying materials can be detected through a thin carbon coating [1],[2].

A sputter coater distributes charged coating ions evenly all over the sample. A sputter coating is applied in an argon chamber with pressure of about 1 Pa. Using free electrons in the chamber the argon is ionized. The sample is placed in a vacuum chamber as the anode of a circuit; it is then connected to a sacrificial cathode made of the coating material. When the circuit is turned on the argon atoms impact the sacrificial cathode breaking off negative ions of the coating material break off of the cathode and are attracted to the positive anode coating the sample [1], [2] (Figure 3-1). The sputter coater in the geology lab can dispense carbon coatings, it was not used for this

project. Sputter coating may be necessary for semiconductor samples especially sensitive to the electron beam or that are planned to be used with high energy for long periods of time [17].

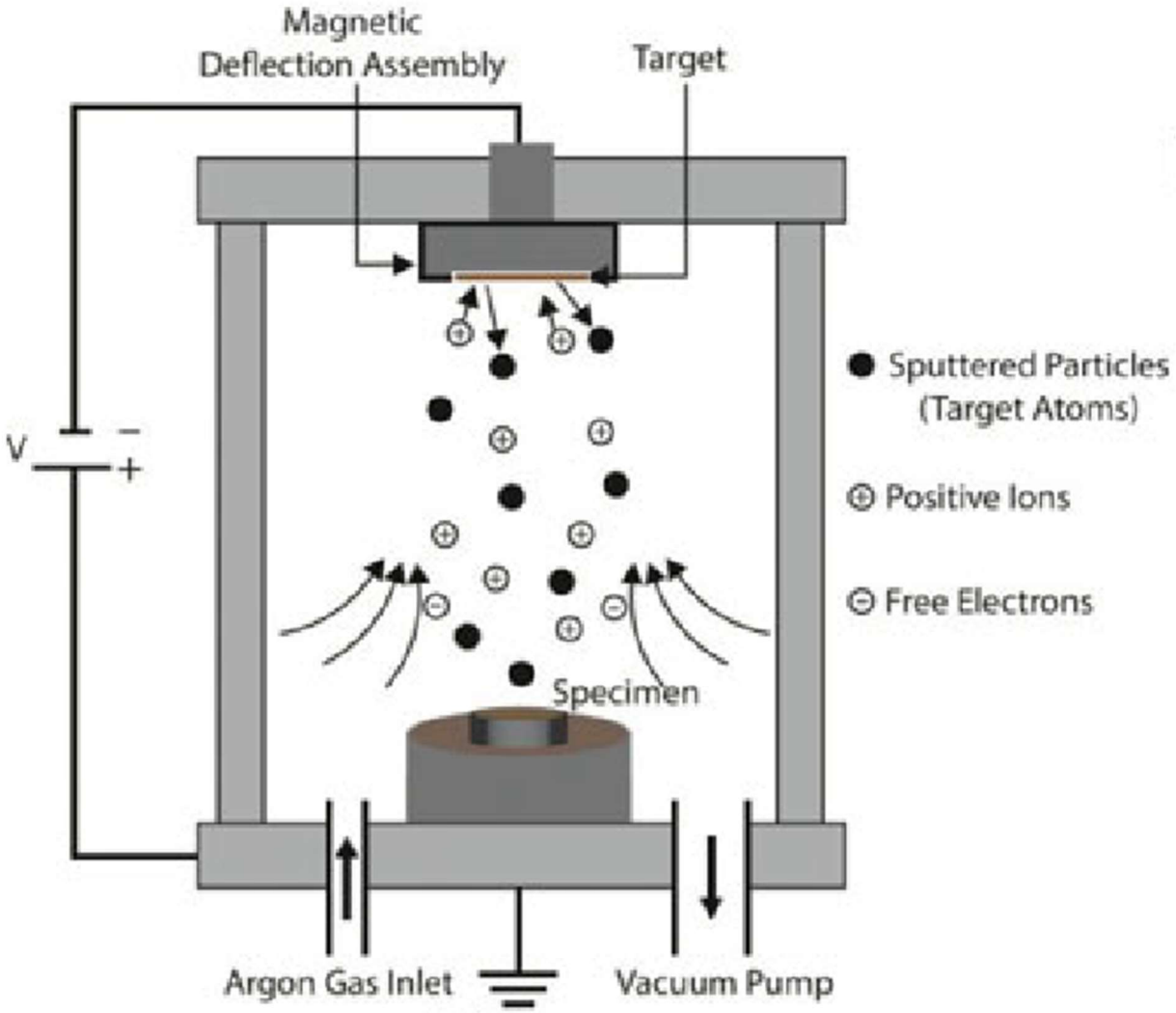


Figure 3-1: Diagram of a sputter coater, the target sputters coating atoms onto the sample producing a thin and uniform coating [[1] figure 8.6a].

# 4 Result analysis

In this project three semiconductor samples were created and tested: the silicon-molybdenum wafer, zinc oxide nanoparticles, and the solar panel (**Error! Reference source not found.**). All samples were examined with the EDT secondary electron detector, and the solar panel and silicon-molybdenum wafer were examined with EDS. The analysis was primarily conducted to identify topography of the samples and test the microscope for semiconductor samples. The final solar panel sample was intended to be the first test of using the SEM to detect damage in semiconductor devices with the eventual goal of relating SEM images to electrical characteristics [12].

## 4.1 Silicon molybdenum wafer

The silicon-molybdenum wafer was from the AFM supplies where they are used to mount samples for AFM examination. We thought this wafer was pure silicon, so we wanted to test the SEM on the simplest semiconductor sample available. The wafer had two features which we wanted to examine, there were two zones which appeared to have differences in reflectivity, and a chip in the edge of the wafer. We were unsure what the significance of the change in reflectivity was and hoped it could be identified with the SEM. The chipped edge was left exposed because it had potentially interesting topography and would serve as a test for SEM examination of damage to semiconductors.

Silicon wafers and devices made from them are routinely examined using SEM. Silicon wafers are the precursor to most modern electronics. Damage in silicon can show on SEM images using many detectors the goal with this sample was to conduct basic testing of the silicon sample and investigate settings and preparation which could be used on more complex samples. The silicon sample is simple and provides useful testing of the microscope.

### 4.1.1 Preparation

The section of wafer we examined was broken off a larger circular wafer using tweezers, it came from near the edge of the overall circular wafer, with the darker region on the outside of the circle. The wafer was cleaned by washing with acetone, then isopropyl alcohol, then and finally

deionized water to remove surface contaminants. The wafer was then mounted on a 12 mm diameter SEM stub using carbon tape. The exposed edges except the one with the chip in it were covered with copper tape to ensure the beam electrons could conduct away from the beam impact site.

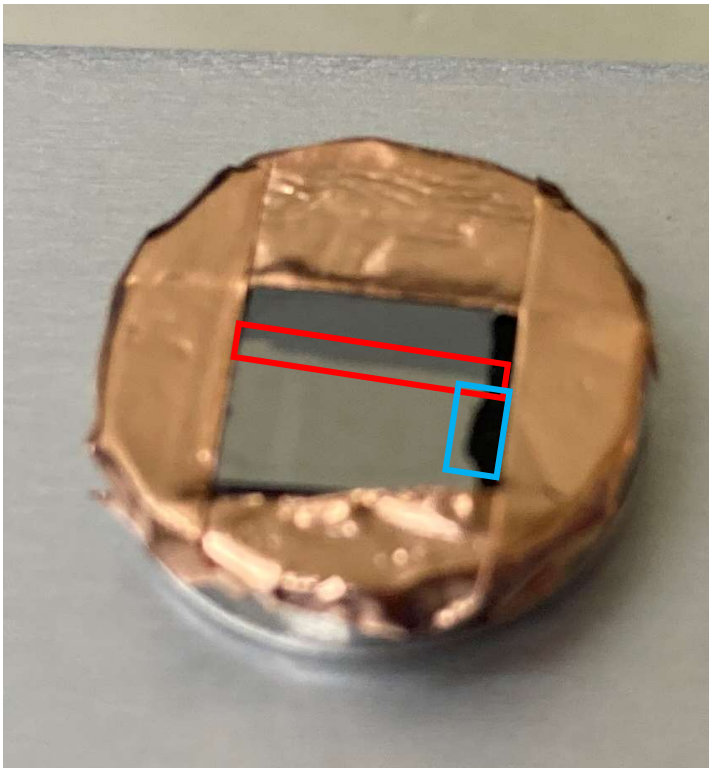

Figure 4-1: The Silicon wafer sample mounted on an SEM stub. The areas of interest are highlighted: red, the junction between the darker and lighter areas and blue, the chip on the right of the wafer. The darker area at the top of the chip is the molebdynium and the lighter is silicon.

### 4.1.2 Imaging

The wafer was examined at high vacuum first with 10 keV beam energy and 15-20 pA beam current. After reassessing the user guidance for semiconductors, it was then re-examined at lower energy of 2 keV. The lower energy was able to reduce charging on the sample and produce very good images. Using the low beam current and energy excellent images of the sample were able to be produced. Two areas were examined on the sample, the junction between the silicon and molybdenum and the chip on the side of the wafer. The change in materials was first identified from the different appearance of the sample when examined with eyes alone, this also showed up

when viewed with the EDT detector. We investigated the change using EDS and identified the molybdenum in the wafer.

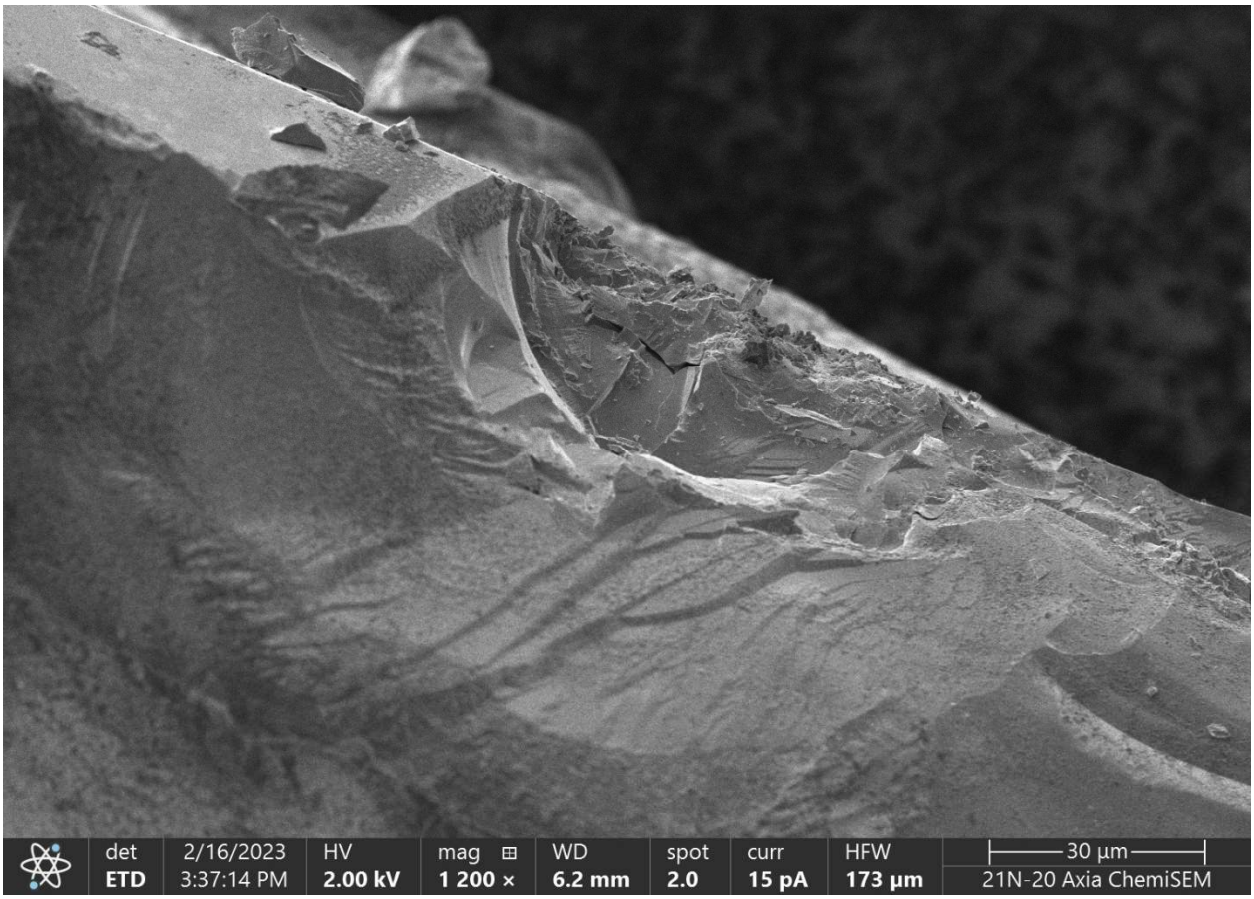


Figure 4-2: The chip in the silicon wafer at 1200x magnification. The overall structure of the chip is shown, with different fractures visible and some debris which has broken off the larger sample (Nancy Van Wagoner).

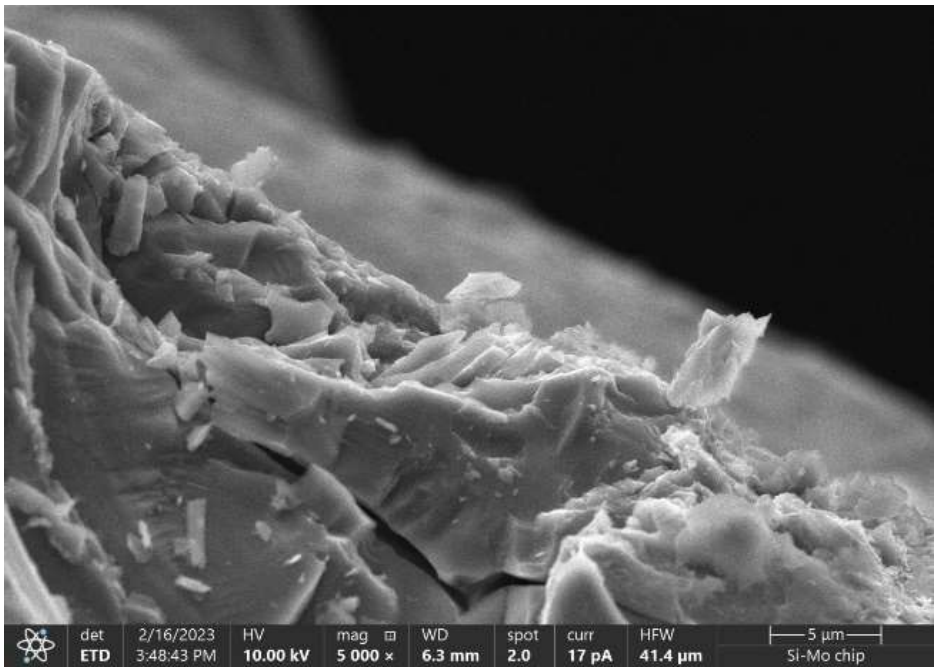


Figure 4-3: The centre of the crack in the chip viewed at 5000x magnification. This is in the centre of Figure 4-2. The conchoidal fracture of the silicon is clearly shown in this image, with the breakage being uneven and jagged across the overall fracture (Nancy Van Wagoner).

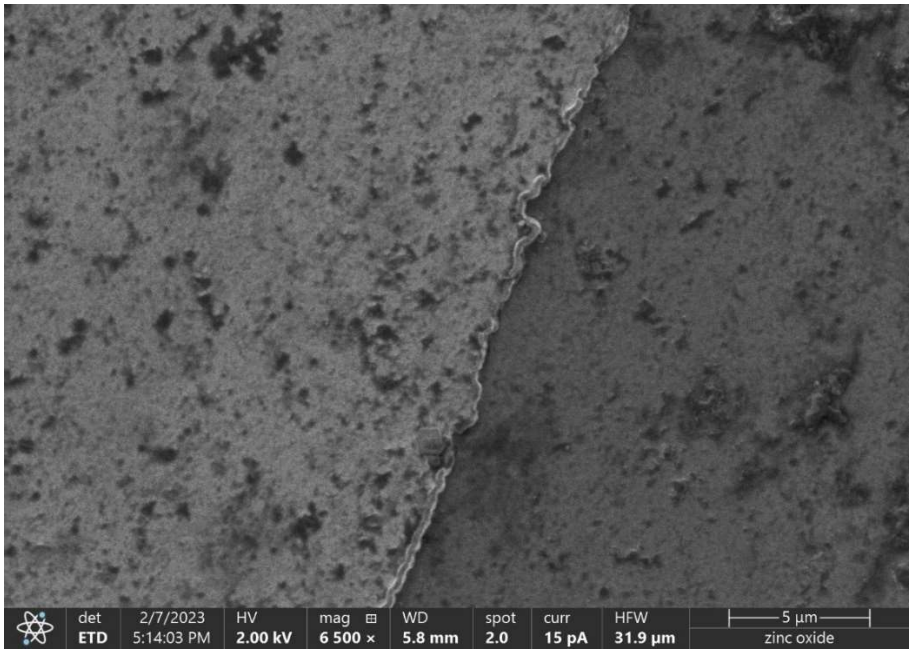


Figure 4-4 Secondary electron image of the material junction in the wafer, this is using low energy and averaging to create the overall image, small gaps and dents in the wafer can be seen even though it was cleaned an polished when created (Nancy Van Wagoner).

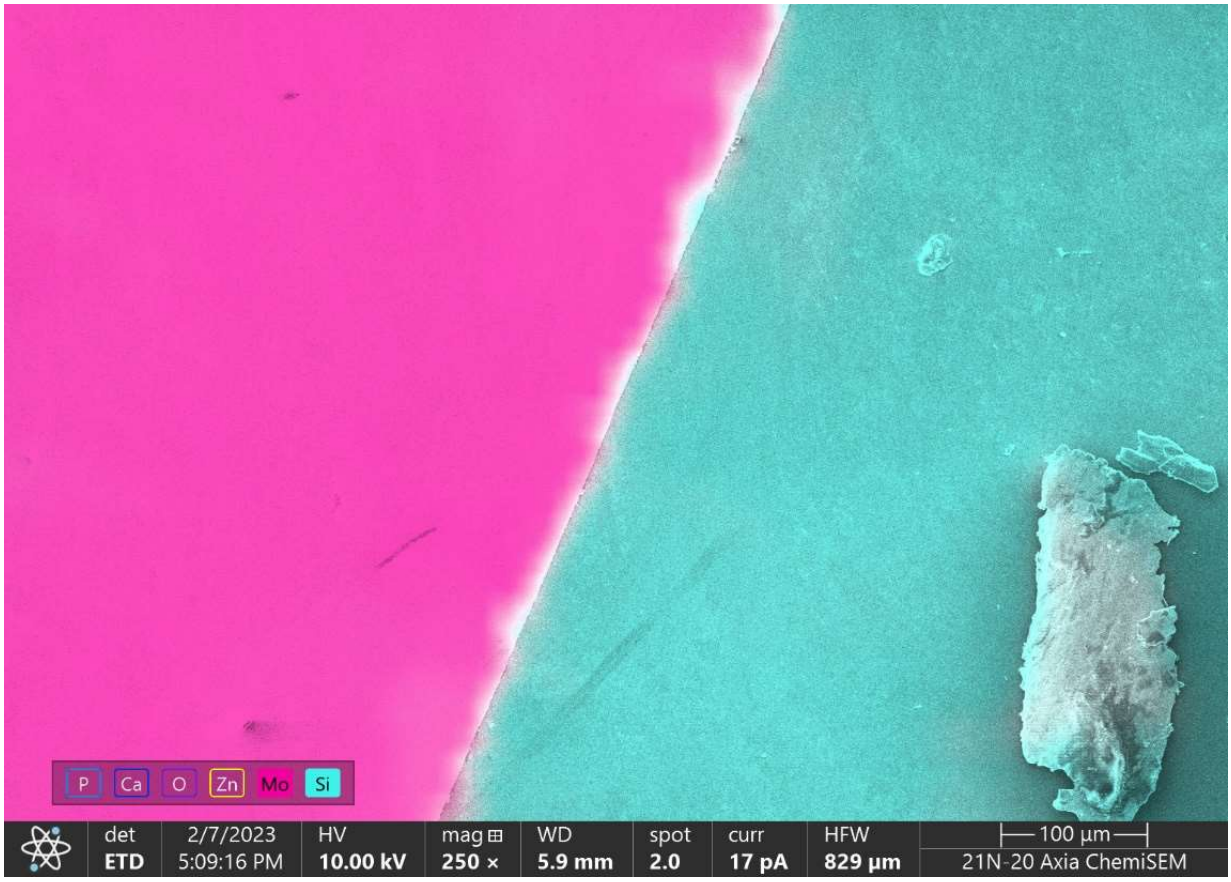


Figure 4-5: EDS map of the junction in the wafer, silicon is blue and molybdenum is pink. The boundary between the materials is shown and aligns with the line seen in SE images. In the bottom right corner, a piece of dust is on the sample (Nancy Van Wagoner).

### 4.1.3 Results

During this testing we learned that the wafer was not pure silicon, it had some molybdenum in it. This means that these wafers cannot be used for AFM imaging where pure silicon is needed. The EDS mapping is uniquely useful for this sample because it is not a pure sample, the molybdenum sections were visible with the unaided eye, but they were small, and we assumed they were also silicon. The edge crack on the wafer was able to be examined in detail, the fracture in the silicon were identified in the highest resolution images which showed small crystals breaking off the larger structure. Examining the sample with low beam energy and current produced excellent images. Some damage was created in the sample using high energy. Using the recommended current and energy for semiconductors there were no issues in examining the sample.

Despite appearing uniform, the sample had some interesting structure on the small scale which could be examined in high resolution to show the abilities of the SEM at high magnification. The silicon was conductive enough for this high magnification to be reached. The low beam energy and current following the microscopes user guidance was able to produce excellent images of the silicon sample. This sample showed the importance of choosing settings to eliminate charging to get good images. Experimentation for a particular sample is required, but choosing the lowest beam current, dwell time, and beam energy possible is important for semiconductor samples. The sample was able to be imaged at higher energies but for general imaging of samples like this one using low energy and current is preferred to eliminate damage. To get good images using frame averaging or integration over many frames with a low dwell time can also minimize damage and allow the sample to dissipate any charges accumulated between scanner passes [1], [2].

## 4.2 Zinc oxide nanoparticles

The zinc oxide nanoparticles were from a sample used by Raju Sapkota, Duan, Kumar, Venkataraman, and Papadopoulos for use in gas sensors. The zinc oxide nanoparticles change conductivity based on the concentration of gasses in the atmosphere. They are commonly investigated for use as an inexpensive gas sensor. The nanoparticles have high surface area and gas diffuse rapidly through them making sensor constructed with nanoparticles fast and accurate. By placing a nanoparticle film between electrodes changes in the conductivity correspond to changes in the ambient gas concentration (Figure 4-6) [4].

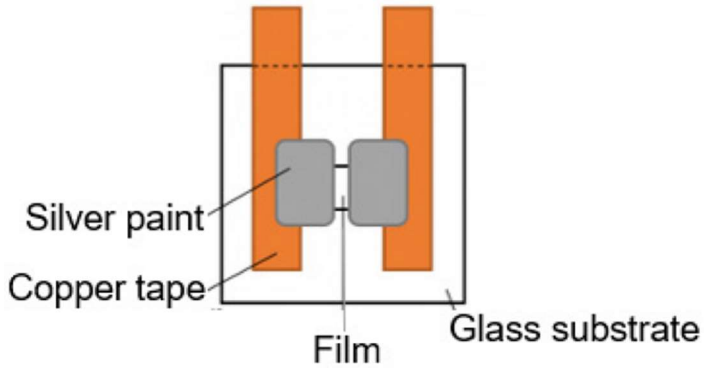


Figure 4-6: Gas sensor with nanoparticle film. The ZnO nanoparticles are places between copper electrodes and connected with silver paint. The resistance changes based on the ambient gas around the sensor [[4] figure 1a].

Zinc oxide nanoparticles were prepared from zinc oxide powder using a planetary ball mill. The particles were in a deionized water or ethylene glycol solvent. The nanoparticles were then doctor bladed onto the glass substrates. The doctor blading process creates a thin film of nanoparticles on a glass substrate. The samples were then baked to remove the solvent and were used for the gas sensors. The sample we used was not a complete gas sensor, it was a film of zinc oxide nanoparticles on a glass substrate, with no contacts to make a complete sensor [4].

The nanoparticles are an excellent sample for analyzing semiconductor nanostructures with the microscope. The particles are on the scale of tens to a few hundreds of nm and this was confirmed with SEM and AFM images showing the individual particles in the original paper (Figure 4-7). The SEM image is using the UBC Hitachi S-2600 SEM, a low vacuum microscope for imaging uncoated samples [4],[18].

Many papers on the production and use of nanoparticles for devices include SEM images to characterize the morphology of particles, few of these include a detailed explanation of the settings used. Nanoparticles are routinely examined with a variety of settings, they are able to be imaged with high voltage coated or uncoated [19][20]. More specific settings and detailed information on sample preparation was difficult to find. Films of nanoparticles like this sample are typically imaged to characterize the particle packing, size and count, these are typically characterized using software built for this purpose [21].

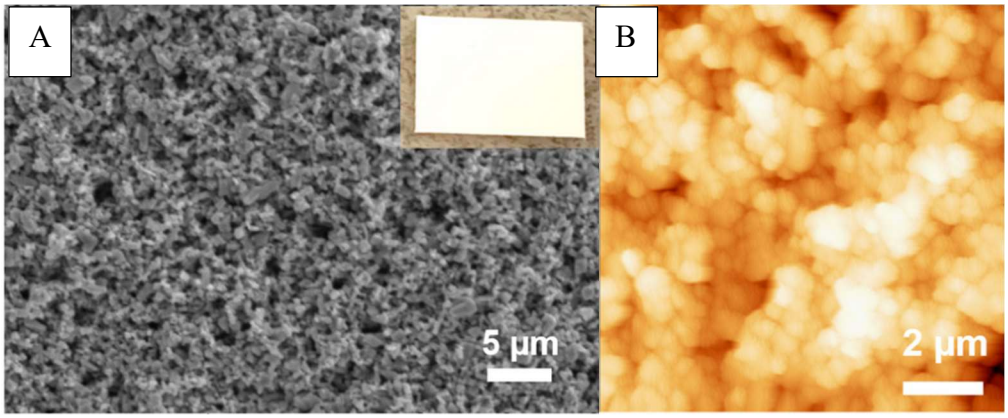


Figure 4-7: A: SEM image of the ZnO nanoparticle film, taken using low vacuum, the inset at the top right is an optical microscope image of a film of ZnO nanoparticles. B: AFM image of a film of ZnO nanoparticles [[4] figure 2 a and b].

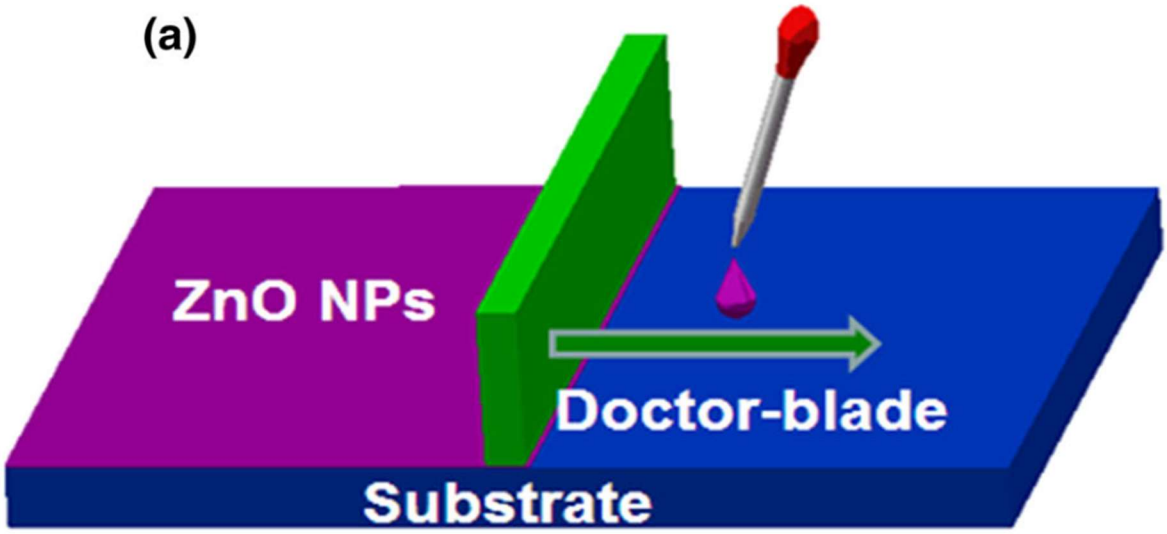


Figure 4-8: Doctor blading of zinc oxide nanoparticles onto a substrate. A solution containing nanoparticles is placed on the substrate, then a sharp blade is dragged a short distance above the top of the substrate to produce a thin film of the nanoparticle solution. Once the sample is dried and the solvent is gone a thin layer of zinc oxide nanoparticles remains [[22] Figure 3].

### 4.2.1 Preparation

The nanoparticle film produced from the doctor blading process was created on a glass substrate. To mount it an SEM stub was covered with a carbon tape sticker, the cover was removed from the sticker to expose the sticky side. The sticky SEM stub was then pressed down onto the ZnO

nanoparticle film on the glass substrate to attach it to the stub. The nanoparticles were then pressed down onto the aluminum foil to ensure they were strongly attached to the stub. This may have contaminated the surface with aluminum. Finally, the exposed parts of the carbon sticker were covered with copper tape and the stub edge was firmly tapped onto the aluminium foil to remove anything which was not firmly attached.

There sample mounting may have caused some issues with the final sample. The film was inverted with respect to how it was created, the side which was attached to the glass substrate is the side exposed for SEM analysis. The pressure from pressing the stub down onto the nanoparticle film and the pressing the final sample onto aluminum foil may have damaged the structure of the complete film. The mounting introduced cracks into the overall film which were visible with the microscope.

Figure 4-9, The ZnO nanoparticle sample on a stub. The image is slightly out of focus but some of the nonuniformity along the edges of the nanoparticles is visible.

### 4.2.2 Imaging

Nanoparticles made by the same process were examined with the UBC Hitachi S-2600 SEM. This is a low vacuum variable pressure SEM. We wanted to further examine them under high vacuum to compare the results and test the zinc oxide for charging. Images were taken starting at 2 keV, 200 ns dwell time, and no averaging as is recommended for semiconductor samples, no image was shown with these settings. To get a better image the dwell time was increased to 500 ns and the beam energy to 10 keV. This produced much better images which showed rectangular structures when zoomed to the scale of $\mu$m, further increasing magnification began to show individual particles more clearly. With further increases magnification the individual particles were clearly shown.

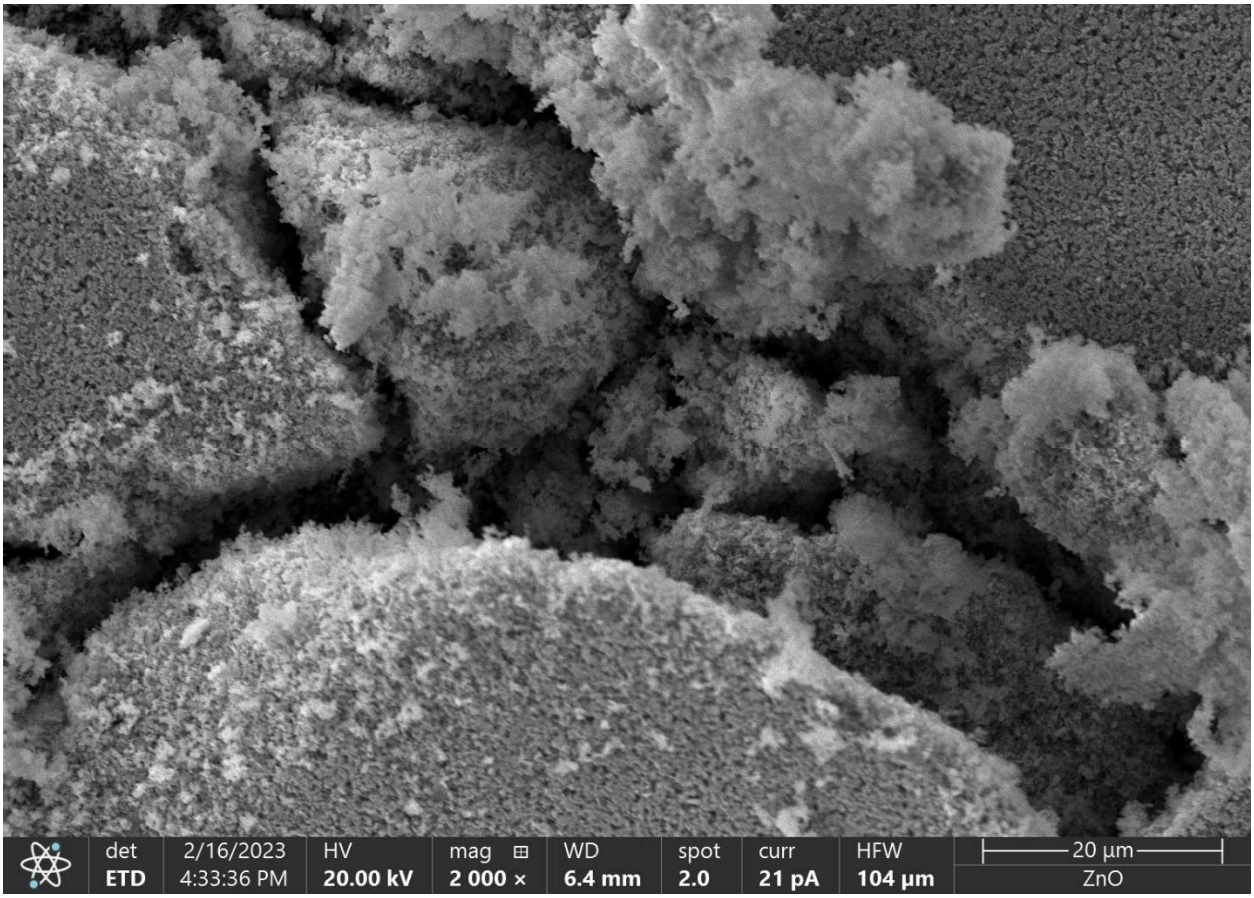


Figure 4-10: Image of the nanoparticles with low magnification showing the cracks created in the wafer when mounting to the SEM stub. The cracks are tens of microns in width and spread over the surface of the entire sample (Nancy Van Wagoner).

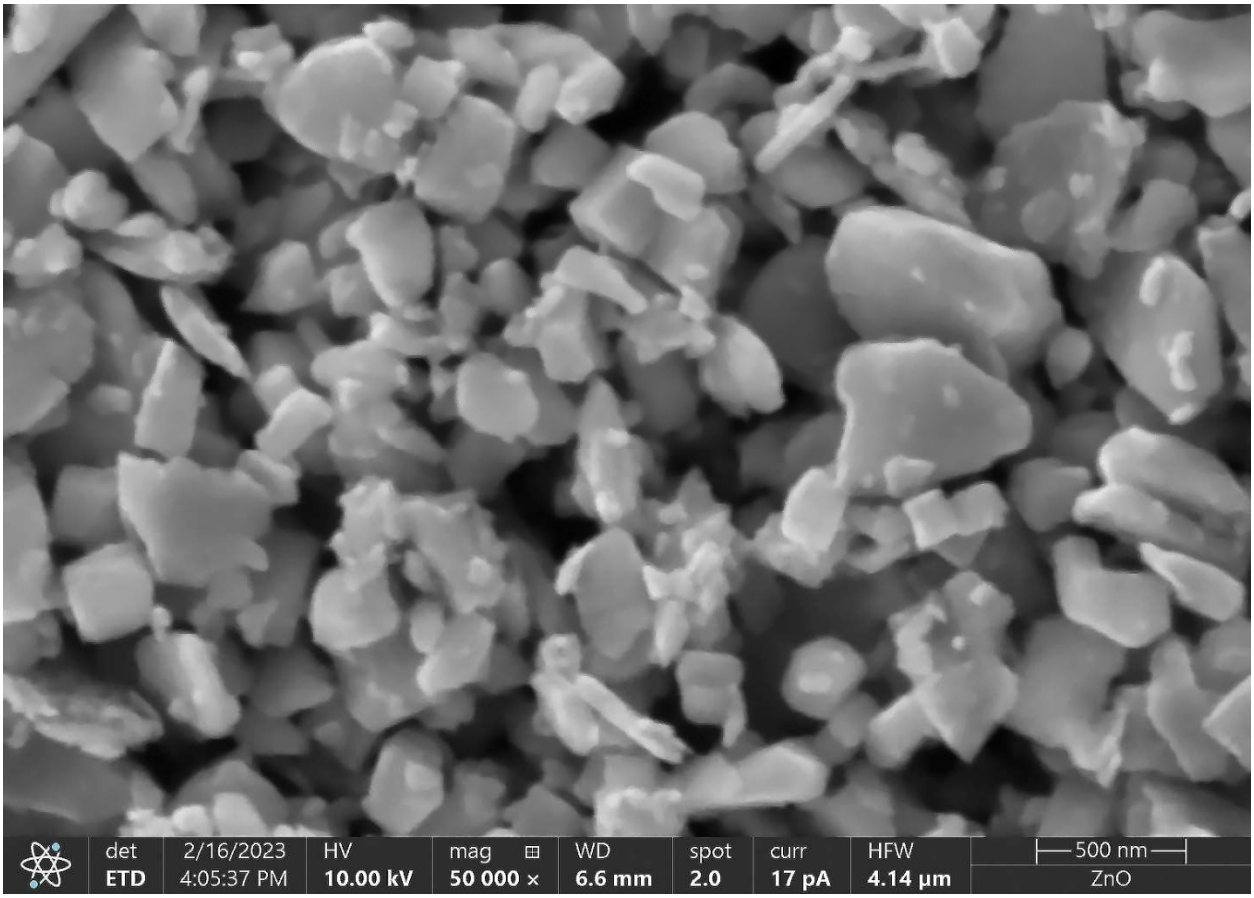


Figure 4-11 zinc oxide nanoparticles, the SEM image at, the image clearly shows the size and shape of the nanoparticles on the scale of tens to hundreds of nm. With this level of magnification, the nanoparticles are not of uniform shape or size, there are a variety of sizes present, and the individual particles have rough, jagged edges. This is at higher magnification than the images included in the paper which may be why this is clearly shown (Nancy Van Wagoner).

### 4.2.3 Results

Due to the close packing of the nanoparticles in the film the size and shape is difficult to obtain from these images. The particles overlap and are grouper very close together but specialized software could potentially extract particle size and count over the area of the scan [21]. I could not identify any freely available software which could be used analyze these images. It could be done by hand but that would be very time consuming. To accurately obtain the size and shape of

many particles a different sample preparation process than doctor blading is necessary, in the film particles are grouped together and overlap making them somewhat difficult to separate. For accurate measurement of the size and shape particles are generally loosely dispersed over a stub to eliminate grouping [20], [21].

The images taken show the agglomeration of the nanoparticles. Agglomeration is the adhesion of nanoparticles to each other. Since the nanoparticles have high surface area compared to volume the adhesive forces between them are relatively strong. In the images small particles stick to the larger particles from this agglomeration. The agglomeration forms an overall aggregate of the particles with particles touching and held together by the intermolecular attractive forces between them. When the solvent is baked out these are the only forces remaining to hold the film together which produces agglomerated clumps of nanoparticles which connect together to produce the overall film [4].

## 4.3 Monocrystalline silicon solar panel

The final SEM sample used in this project was a monocrystalline silicon solar cell. We chose this sample with the eventual goal of relating SEM analysis of damage in the panel to electrical characteristics. Solar panels are fragile, their efficiency can be reduced by improper handling, defects, and damage from use. SEM is used to look in depth on solar cells for specific analysis conducted by manufactures and academic researchers. The SEM can analyze cross sections of the solar panel to identify layers inside the device with a cut solar panel sample. Sem can also examine the surface of the solar cell and identify surface texture and damage [23].

A photovoltaic (PV) generates electric power from light radiation. The most common solar cells are crystalline silicon. In these, a PN junction is constructed across the device so that when incoming radiation is incident on the surface it excites electrons from the top n layer into a circuit generating electricity. There are two types of crystalline solar cell, monocrystalline and polycrystalline. Monocrystalline (MC) cells are made from a single silicon wafer cut from a uniform silicon crystal for electronics. Polycrystalline (PC) are generally made from the waste of other semiconductor manufacturing and include multiple crystals. PC cells have the distinctive appearance of differences in reflections between the different crystals (Figure 4-12). Generally,

MC cells are slightly more efficient than PC cells due to the resistance introduced by the multiple crystals.

Figure 4-12: Left a PC solar cell and right a MC solar cell. The different crystals are visible in the PC cell, whereas the MC cell is darker and more uniform, it also has diagonal corners from cutting from a cylindrical silicon ingot [24].

Crystalline solar cells are produced from p-type silicon wafers, typically with boron. The surface is then textured to reduce reflections of incoming light. These structures are typically acid etched into the surface; different crystal modes of silicon etch differently creating square pyramids [25]. Then the front is heavily $n^+$ doped with phosphorus to form a pn junction from the from to back of the wafer. The PN junction creates a potential difference across the device for excited electrons to follow. The surface is then covered with an antireflection coating. The coating is typically silicon dioxide, titanium dioxide, or silicon nitride. Contacts are then printed on the front and back of the device to conduct the generated current and power connected circuits (Figure 4-13).

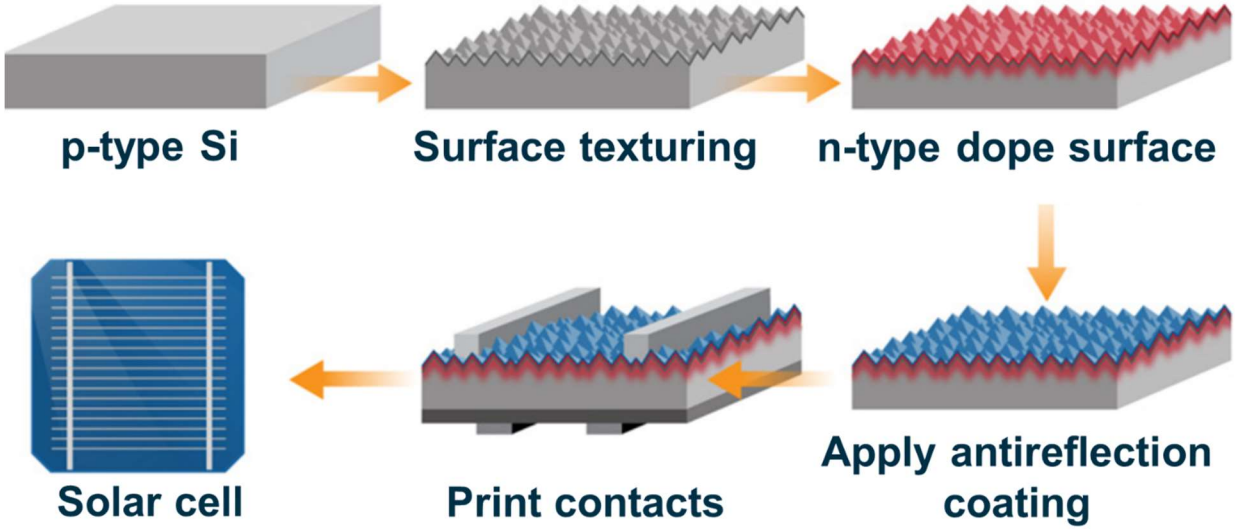


Figure 4-13: The production process for a crystalline silicon solar cell. Beginning from p-type silicon the surface is textured, n-type doped to create a pn junction, an antireflection coating is applied to the surface. Finally the contacts are printed on the front and back [[26] figure 1].

The datasheet identified the sample as a MC silicon solar cell with 25% efficiency and sensitivity to 300-1100 nm light; this is a typical cell used in small electronics. There was no information given on coatings or additional materials given [27]. Solar cells typically have a pyramid structure on the surface on the 1-10 μm scale and an antireflection coating made of SiO2, TiO2, or Si3N4 [28][25] (Figure 4-14). We assumed the surface was exposed and the coating texture and material identifiable, so we hoped to examine topography and composition with the SEM. If the surface was not covered with additional layers the solar cell would be an excellent test sample for the SEM examination of semiconductor samples. We hoped the pyramids could be exposed for physical examination (Figure 4-14) [23].

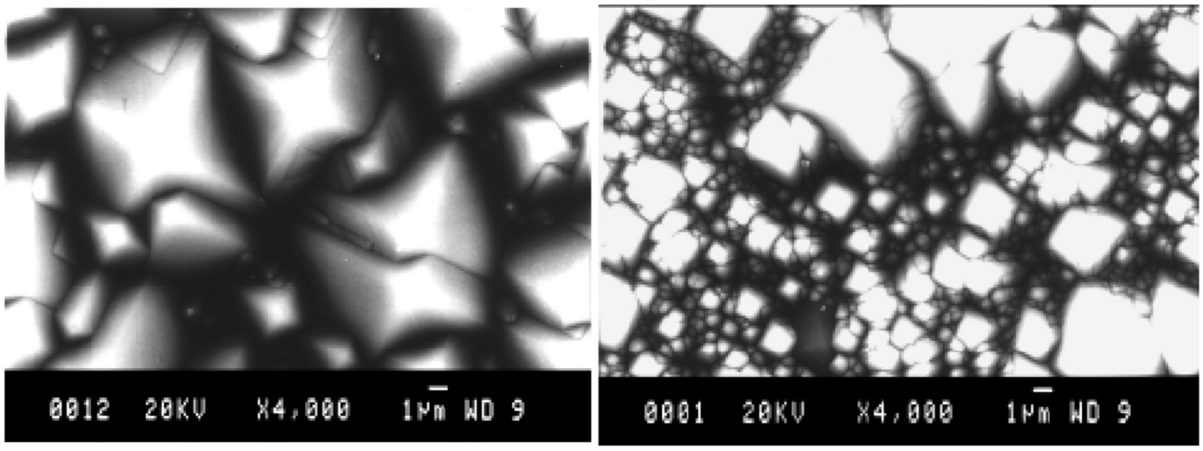

Figure 4-14, MC solar panels under SEM. We expected the final images to look like these which show the regular structure of the solar panel surface. The pyramids are square based and on the scale of a few microns [23].

Based on the datasheet information and background research on antireflection coatings in solar cells we assumed it was a simple monocrystalline solar cell with the surface exposed [27]. Instead, the sample was a complete solar module which contained MC solar cells mounted on a rear printed circuit board (PCB) and covered in organic layers on the front. The contacts of the solar cell are connected to the PCB and then it is attached. The surface is then laminated with ethylene tetrafluoroethylene (ETFE) films encapsulated by ethylene-vinyl acetate (EVA). These bind the complete device together and protects the sensitive solar cells inside from damage. The complete structure was only determined after Anusha contacted the manufacturer following the SEM imaging. This process is usually used for commercial solar panels but not for lab created solar cells [29][30].

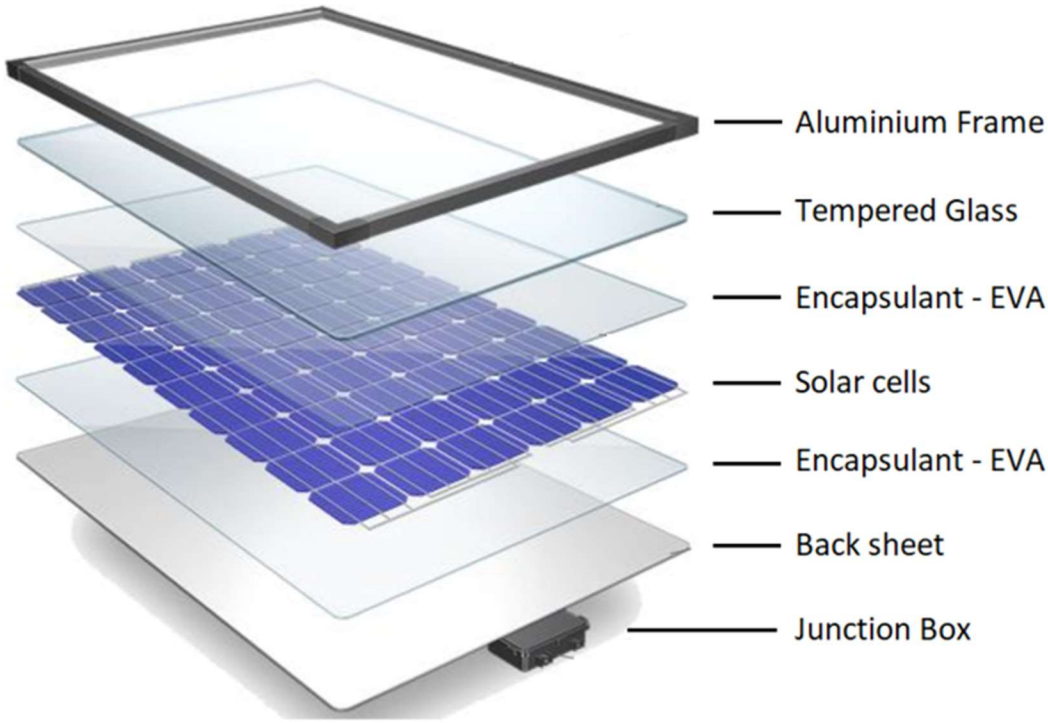


Figure 4-15: The structure of a complete solar panel, the encapsulant covers the interior solar cells and protects them. The back sheet and junction box conduct the electricity generated by the solar panel away from the surfaces. The covering in the case of the solar panel examined in this experiment the tempered glass is replaced by a ETFE film which is exposed to the surface [29].

### 4.3.1 Preparation

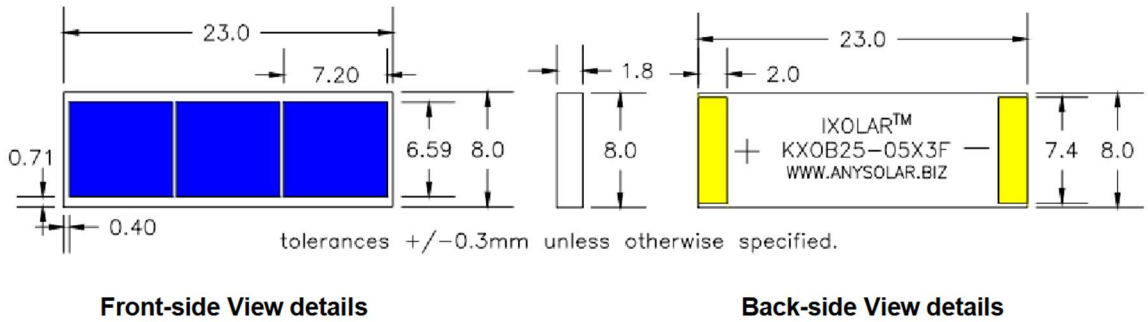


Figure 4-16: package schematic of a KXOB25-05X3F-TB solar cell. The left shows the front, the middle shows the narrow side, and the right shows the rear. It is longer than a standard 12 mm SEM stub so a 24 mm one had to be used. The contacts for connecting the panel to an electronic device are on the rear PCB, the front is divided into thirds by front contacts [27].

The solar panel was newly removed from the package to be used as a sample. It was then cleaned by blowing air on it on the front and back. Solvent was not used to avoid damaging the surface texture. A 24 mm diameter SEM stub was used to support the 23 mm long solar panel. Carbon tape was placed on the stub and the solar panel was mounted on it with tweezers. The contacts on the back were touching the carbon tape to ground the surface through the contacts in the device. The solar panel with the stub on it was then pressed down on the carbon tape backing to ensure it was securely mounted. The solar panel was then cleaned by blowing air on it. The sample was inspected with an optical microscope, there was still dust, so it was further cleaned with compressed air.

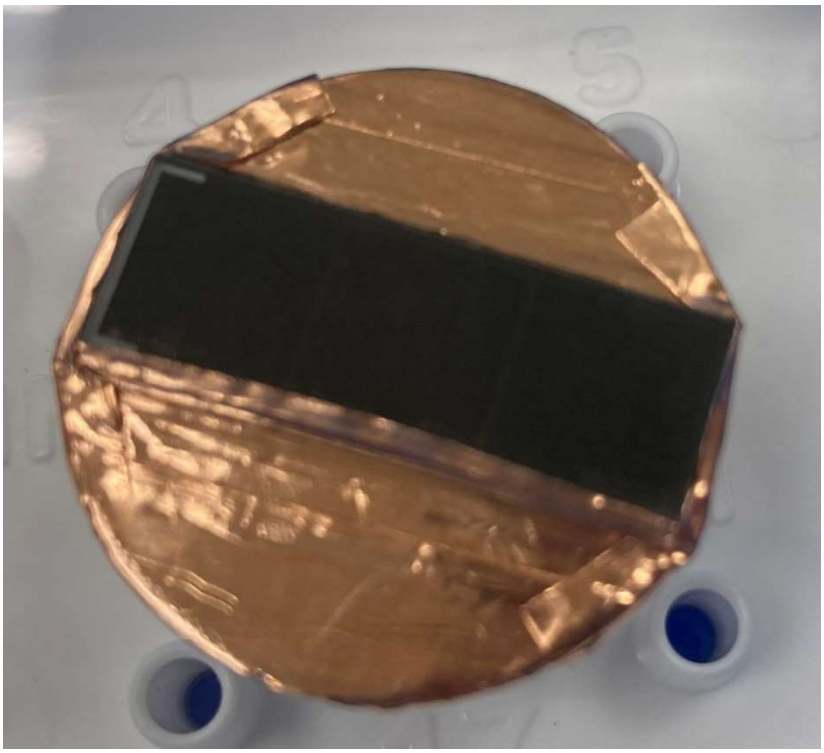

Figure 4-17, solar panel mounted on stub. The grey bar on the left of the panel is a front contact, smaller contacts are barley visible dividing the device in thirds.

### 4.3.2 Imaging

The first analysis was conducted with the optical microscope, once the dust was removed images were taken to examine the structure of the solar cells. The cell was first examined with an optical microscope. The sample was difficult to light for the optical microscope and required several lighting changes to create good images. The optical images showed roughness on the scale of 10s of $\mu$m as was expected from the texturing of the solar panel. The structure showed some diffraction effects around the bumps on the surface in one of the images, which suggests the structure is around the same scale as visible light waves.

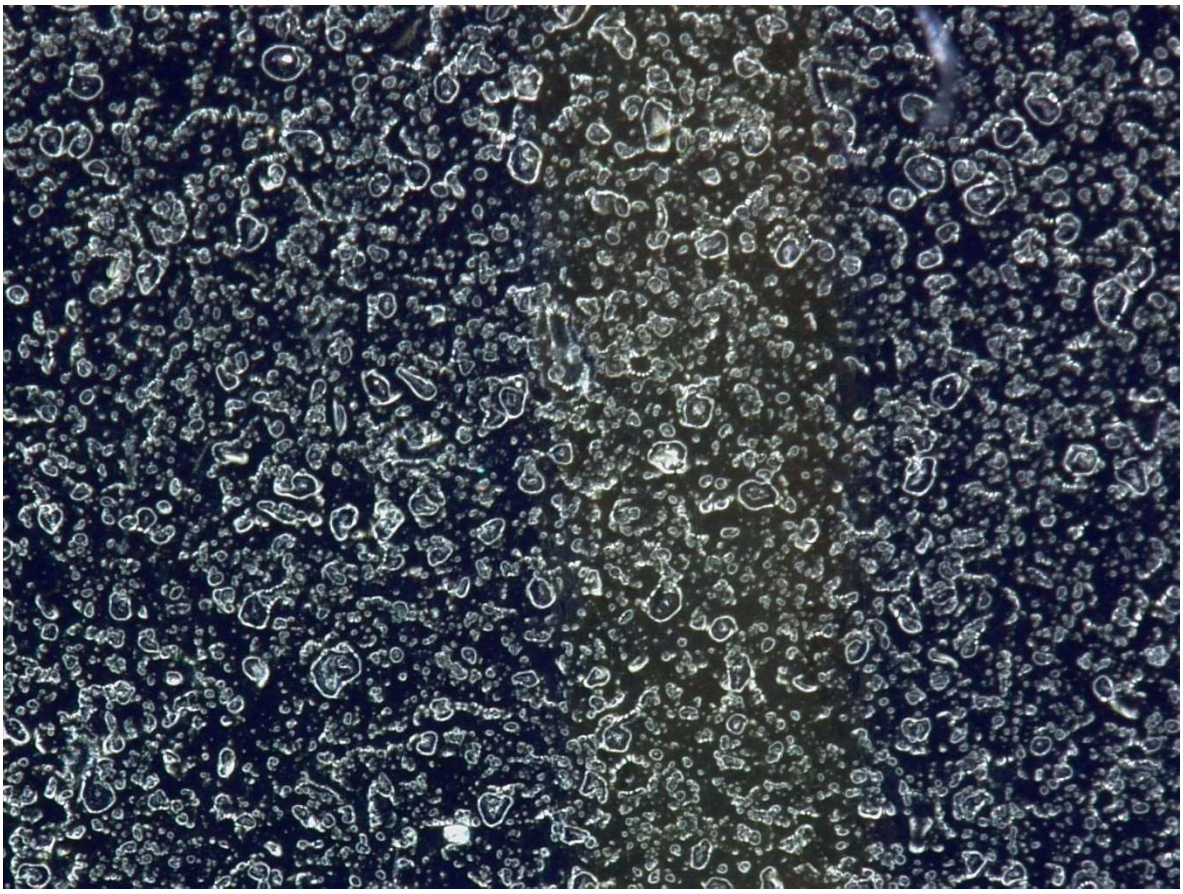

Figure 4-18: Optical microscope image of the solar panel, the field of view is 1.5 mm. The overall structure shows bumps in the ETFE film on the surface, there are some diffraction effects visible around the bumps. The blue on the sides of the image is the silicon of the solar panel visible through the encapsulant and film, the brown in the centre is a front contact (Nancy Van Wagoner).

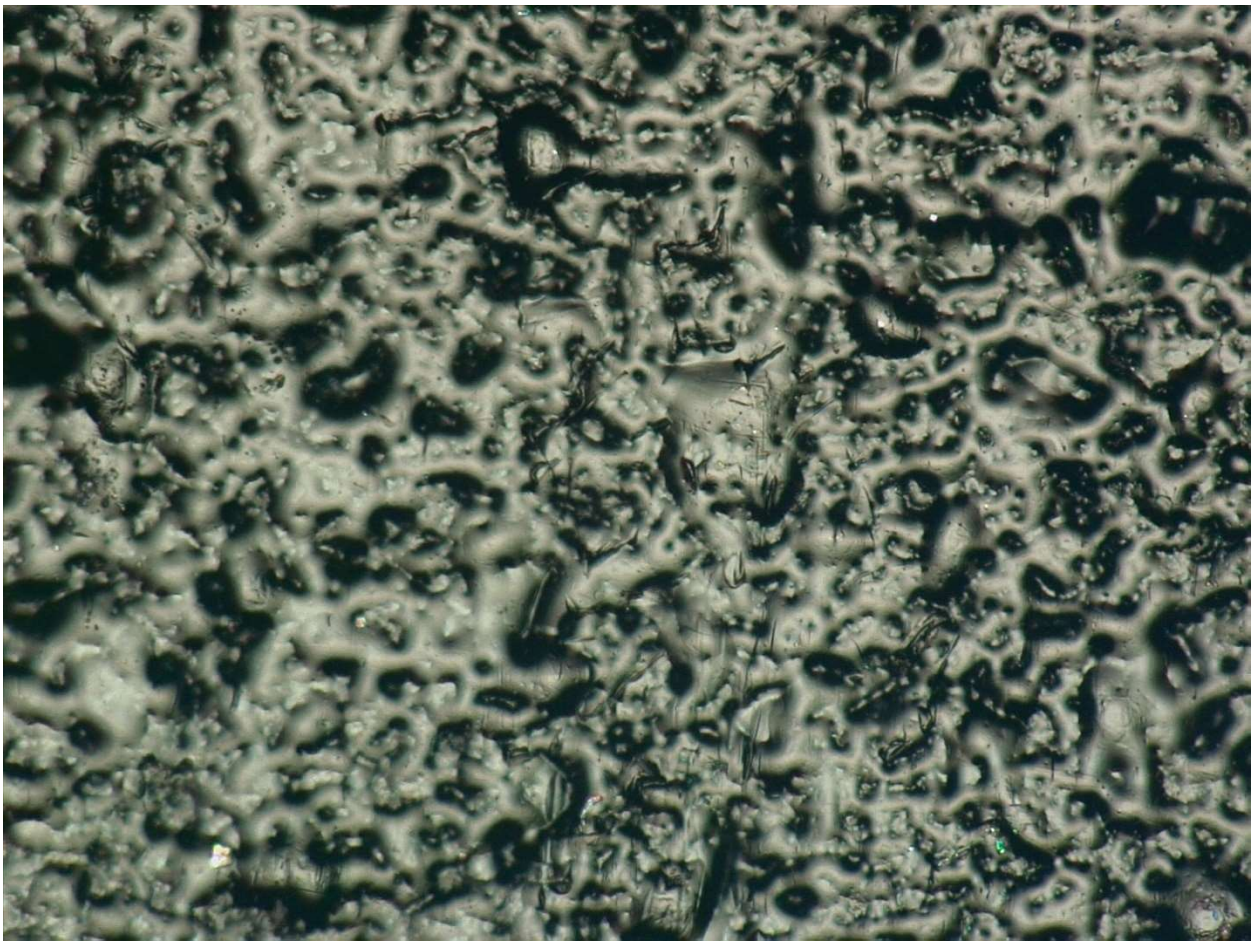

Figure 4-19: Optical image of the solar panel with increased lighting and 0.62 mm field of view. This image better shows the overall surface texture of the panel and has eliminated the small diffraction effects seen in the previous image and shows the structure more clearly (Nancy Van Wagoner).

After the optical imaging we proceeded with SEM imaging of the panel. It was examined first at 2 keV beam energy and 21 pA beam current showing surface texture but it was difficult to identify. There microscope showed some charging, and the overall structure was low contrast and hazy. To improve resolution, beam energy was increased to 20 keV. When the beam energy was increased the view of the sample changed and charging became apparent. The sample was able to be viewed with areas which looked like flow patterns on the surface of the sample.

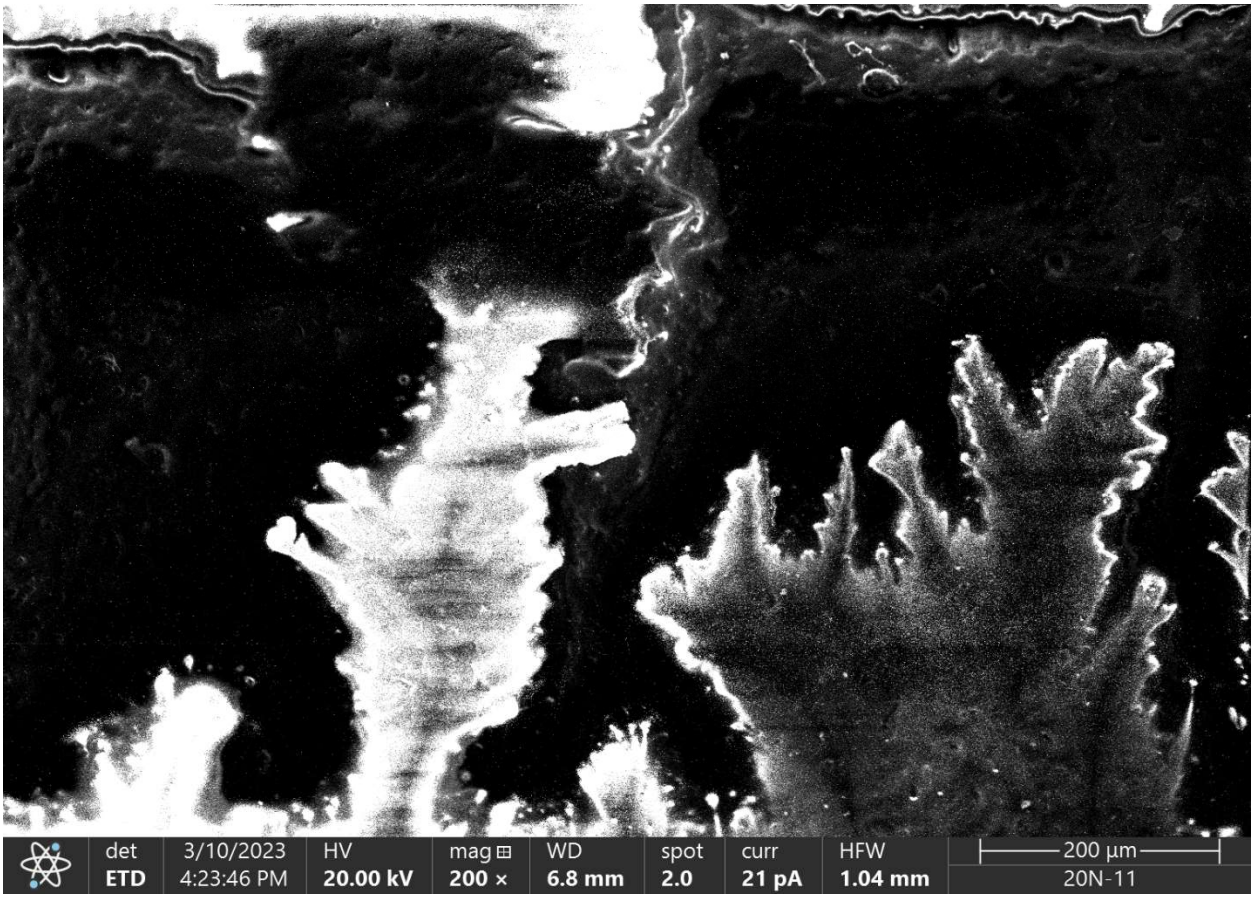


Figure 4-20, SE of solar panel, there are what appears to be flow patterns in the overall image. These flow patters have visible charging especially the large area on the left of the image. In between the charging regions such is the far left middle the surface is slightly rough with some small divots in it which is what EVA typically looks like when imaged with SEM.

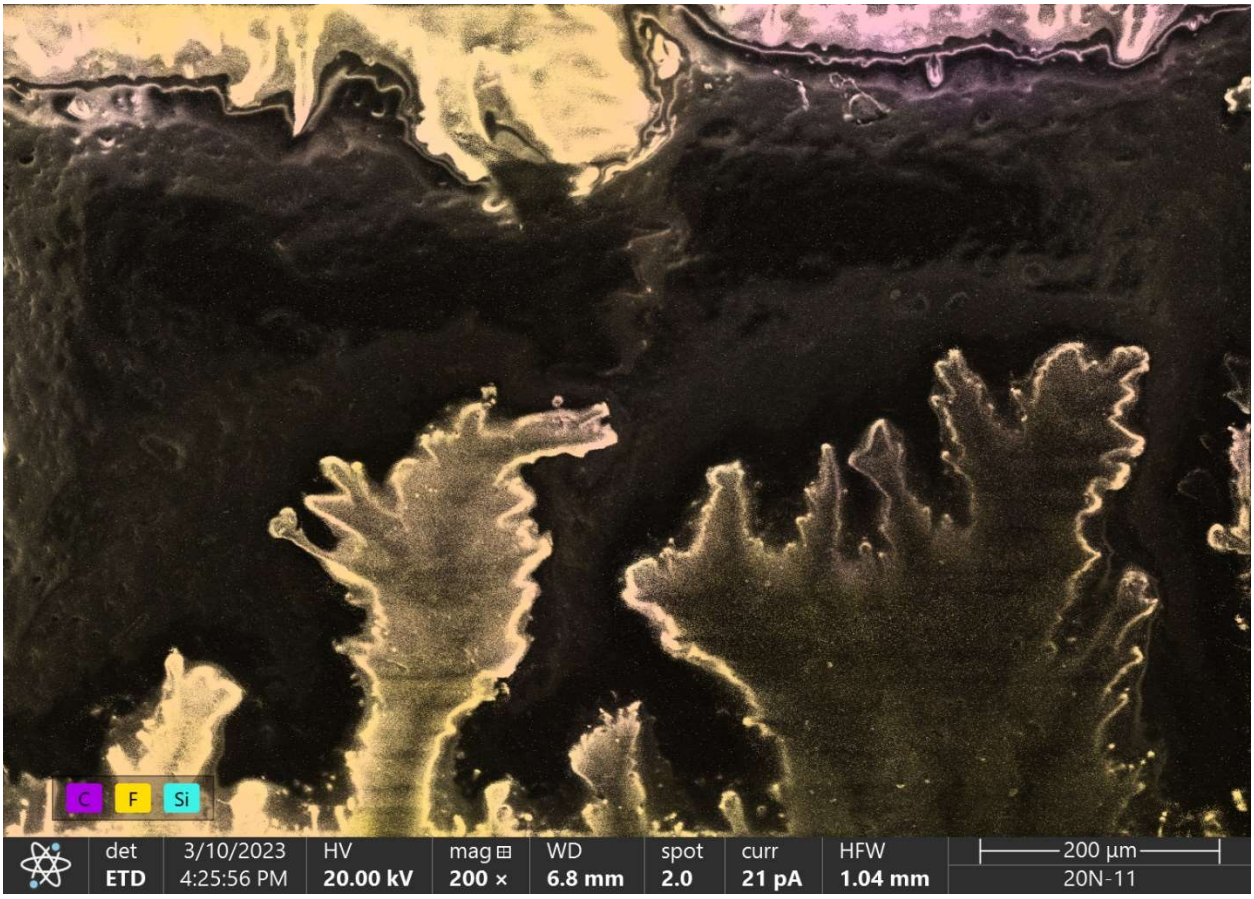


Figure 4-21, EDS of solar panel, the flow patterns seen in the previous image strongly show fluorine, the one in the top right shows carbon as well. These patterns could be produced by the EVA encapsulant melting from the heat of the electron beam and exposing the underlying ETFE film.

### 4.3.3 Results

Based on the datasheet identifying the sample as a solar cell we assumed it was a solar cell with the antireflection coating and contacts exposed. The datasheet identifies it as a MC solar cell so we assumed it would be simply the cell. It was imaged and it turned out not to be the case. The SEM image revealed apparent flow patterns in the top layer and charging. Running the EDS on this image identified fluorine most strongly in the flow patterns. After Anusha reached out to the manufacturer surface of the solar panel is laminated in an ETFE film secured to the cells with an EVA encapsulant. Since fluorine is most strongly identified in the flow patterns these areas are where the ETFE is. This may be from the top ETFE layer liquefying and being blasted into the

flow patterns exposing the underlying EVA which is why the sample areas outside the apparent flow patterns resembled EVA films [31].

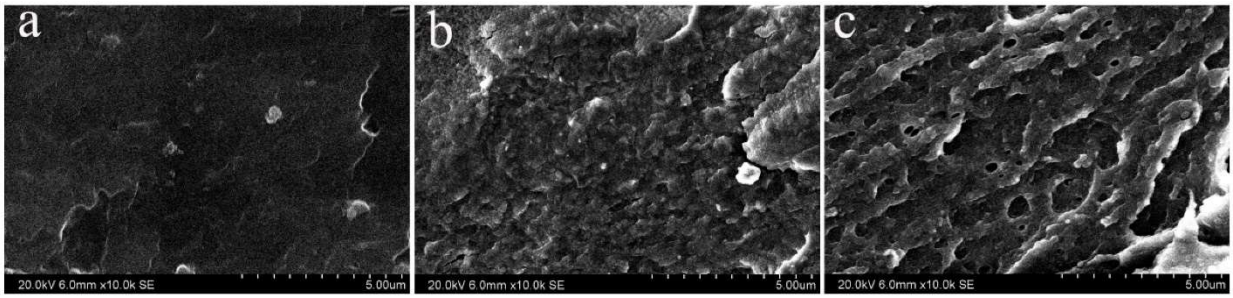


Figure 4-22: SEM images of ETFE which has undergone a 504 h again treatment. A: unirradiated ETFE. B: 60 kGy radiation dose. C: 120 kGy radiation dose. As the radiation dose increases the ETFE degrades and clumps together [[32] figure 8].

The ETFE film covering the complete solar cell. ETFE is a relatively stable polymer. Modern solar panels are laminated with it to protect them from damage [30]. ETFE melts at 265-270 °C, and undergoes thermal breakdown at 350 °C producing mostly hydrogen fluoride [33]. The structure of ETFE changes when it is irradiated. The radiation decreases the crystallization of the polymer and introduces more gel amorphous structure. The more radiation damage to the less uniform the surface becomes with some surface clumping visible at high doses (Figure 4-22) [32]. ETFE can be imaged with the SEM, but it requires specific sample preparation and use of low energy and current as well as potentially low vacuum.

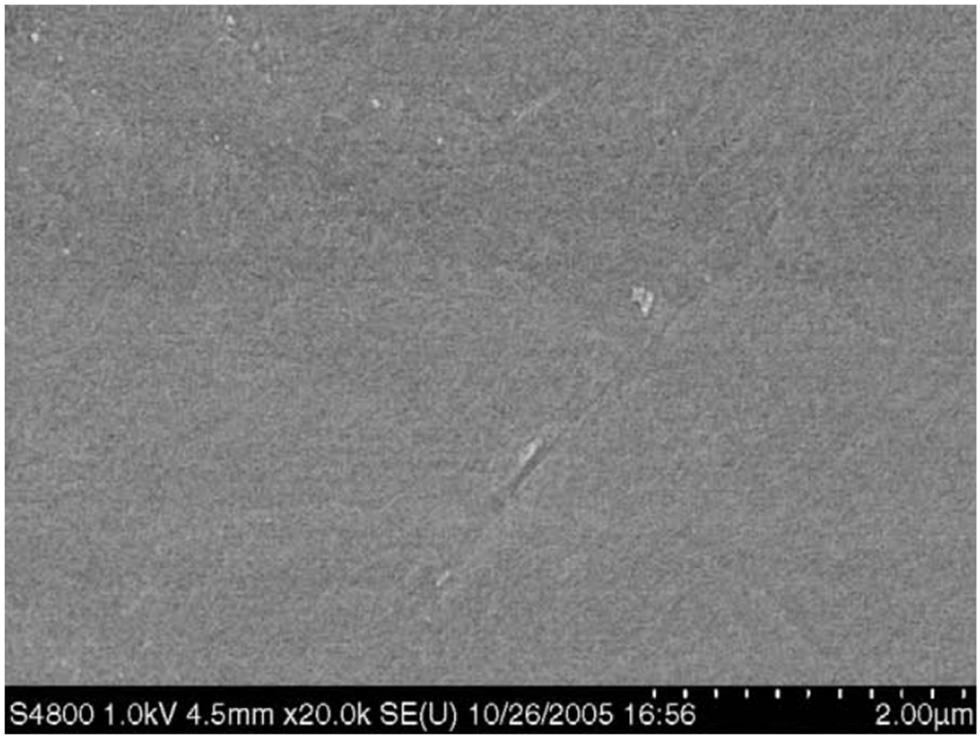


Figure 4-23: SEM image at 1 keV energy of an untreated ETFE film [32].

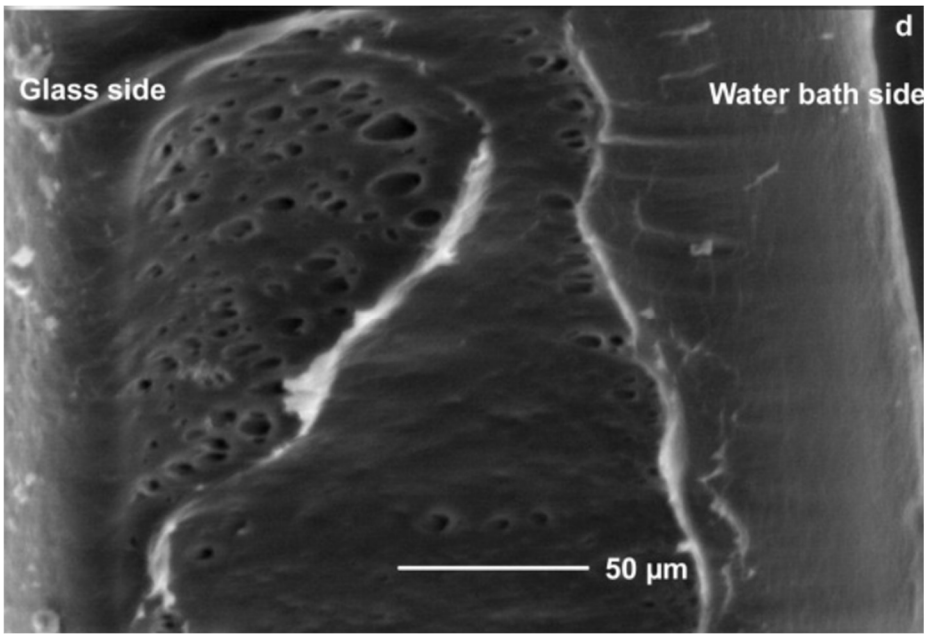


Figure 4-24: SEM image of an EVA film, there is some apparent charging visible in the white areas. The overall surface in the centre of the sample has better contrast than the final image of the solar panel but it shows similar surface texture. The specific sample preparation and settings are not mentioned in the paper [[31] figure 1d].

The EVA encapsulant is made of ethylene and vinyl acetate; it is currently the most common encapsulant for commercial solar panels [29],[30]. EVA can be examined with SEM at low beam current and energy issue free. The areas outside of the flow patterns on the sample surface appear like more general images of EVA under SEM [31]. The main issues with this imaging were the high beam energy. The melting point is between 70-100 °C depending on the amount of vinyl acetate in the polymer, the polymer is stable until thermal breakdown occurs at around 500 °C [34][35]. The electron beam radiation will have disrupted the regular structure of the EVA film and converted some to an amorphous gel structure, as long as it was not heated to the point of thermal breakdown it will have stayed stable [36].

Both the ETFE film and the EVA encapsulant have low thermal and electrical conductivity. In both the beam electrons will stay localized and cause charging and heating with high beam current and energy. Based on my analysis of the images I believe the ETFE surface film either partially melted or clumped from the radiation damage. This caused it to group together on the solar panel surface exposing the underlying EVA. The partial melting produced the flow patterns as it was blasted to the edges of the sample, and it clumped together into the flow pattern on the surface of the EVA encapsulant. The EVA encapsulant layer under the ETFE did not appear damaged so the sample may not have reached a high enough temperature to cause thermal breakdown of either polymer since EVA melts at 70-100 °C [34]. If thermal breakdown of the ETFE occurred hydrogen fluoride may have been released into the chamber if the surface of the sample reached 350 °C [33].

Since the solar panel had a surface made of nonconductive organic polymers rather than semiconductors it was not suitable for the high energy imaging. It could potentially be imaged using low beam energy and current, but it would need to be monitored closely for any damage to the surface caused by the electron beam. The sample could have potentially been imaged using the low vacuum mode on the SEM which would have aided in cooling of the surface and removed breakdown by-products from the surface of the sample. Even if it was imaged in this manner the analysis we wanted to conduct on the solar panel and the coating would not have been possible, the surface texture is under multiple layers of polymers and would not be imaged with electrons without removing it which would be very complex and would also damage the underlying semiconductors. Determination of electrical characteristics using CL is an interesting

area of research in solar panels, but it would only be applicable to lab created uncoated samples without the surface polymers and not complete devices like the sample tested in this research.

### 4.3.4 Pre-screening of samples

The issues with the solar panel highlight the need for proper pre-screening of samples used for SEM. To ensure these mistakes are not replicated I recommend that samples used for SEM be thoroughly pre-screened before they are used with the microscope. The pre-screening process will need to be developed more in detail as the microscope is opened for use with other departments and students based on what samples are being imaged.

For semiconductor samples specifically, datasheets alone should not be relied on for information about the solar panel. Looking for other sources about the complete manufacturing of the solar panel would have allowed for more information to be obtained before the sample was imaged. This is true for semiconductor samples especially; the complete device may include layers of packaging and other components in addition to the semiconductor at the core. These extra components may not be mentioned in the datasheets for the devices and require more in-depth research on the manufacturing process to identify. Glues and polymers used are especially necessary to identify in potential samples as they may be beam sensitive and non-conductive.

For other manufactured samples a component of research proposals for using the SEM should be contacting the manufacturer where relevant. If I had reached out to the manufacturer as part of the research proposal I may have learned about the polymers on the surface of the solar panel before it was examined with the SEM. The manufacturer could identify what materials exactly are used to manufacture the sample and allow for appropriate precautions to be taken for imaging of it.

In general, preliminary examination under an optical microscope should be conducted to look for surface contaminants and debris. Comparing optical microscope images to what we expect to see can show differences in the structure from unexpected materials used in the sample. In the case of the solar panel, if the manufacturer was contacted after imaging with the optical microscope which did not show the pyramids textured into the silicon wafer, this could have been an opportunity to revaluate conducting the final imaging of the panel.

# 5 Conclusion

The scanning electron microscope was tested on three semiconductor samples. These samples are examples of other semiconductor samples which could be examined with this microscope. The SEM is an exceptionally useful tool for the analysis of such samples, it is a very versatile instrument. Over the course of this project, we were able to create exceptional images of semiconductor samples which revealed the deeper structure of them.

The silicon-molybdenum wafer was an excellent example of how the SEM can be used to examine a sample of unknown composition. The wafer was believed to be pure silicon, but the EDS analysis revealed the molybdenum as well. The wafer was able to be examined in detail with the best images revealing very nanoscale features in the uniform seeming wafer. The zinc oxide nanoparticles were an excellent sample for imaging of nanostructures using the SEM. The individual particles were able to be seen in extreme detail including the formation of some agglomerated structures of multiple particles.

The solar panel was not a suitable sample for SEM imaging with the chosen settings, the surface degraded under high energy electron bombardment potentially contaminating the chamber. The high beam current and energy could not be dissipated by the nonconductive organic surface of the panel with the settings used. It may be able to be imaged with lower beam energy and current or low vacuum. Relating the electrical properties of a device like the solar panel is an interesting and promising area of research, however, it could not be applied to the solar panel due to the organic surface layers.

The issues with the solar panel show the need for thorough preliminary analysis of samples. The manufacturer should have been contacted, and more research on complete solar panels should have been conducted to identify the complete composition before testing the sample under the microscope. To examine samples of unknown chemical composition, use low beam energy and current until the sample is well-understood. Where possible other methods should be used to determine the materials of the sample before they are examined with SEM.

The RGBCL detector could be extremely useful for characterization of electrical characteristics in semiconductor samples. Doping below the detection threshold for EDS may be identified from effects on the band structure. Bandgaps could be measured and how they charge from the

electrical characteristics across the complete sample like junctions and material changes. The CL could also identify damage by the modification in the overall crystal structure causing the emitted photons to change [12].

Researching how to examine specific samples under the microscope was challenging. Most scientific papers which feature SEM images only include limited information on how these images were taken, usually only the instrument used. Large amounts of SEM imaging are done by specialized labs. The labs typically do all the sample preparation in house so even the authors may not know how specifically the samples were prepared and the images included were taken. There were very few procedures available to replicate for imaging semiconductors which made creating them difficult. The user guidance for the microscope was very helpful for imaging samples but finding anything more specific than for general semiconductor samples was difficult. The textbooks and the documentation for the microscope encourage experimentation with the settings to get good images but having more information would have made this process much easier.

Further testing could be done of semiconductor samples to conduct more in-depth analysis. The ChemiSEM with Nancy's expertise is an exceptional instrument for nanostructure analysis. The microscope can examine and characterize these structures extremely well. Opening the microscope to more general use would allow for this versatile instrument to contribute to large amounts of research at TRU.

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# 7 Appendix

## 7.1 Sample preparation

Warning: This is a clean process: gloves, a mask, and a lab coat must be worn throughout. Place down a clean new sheet of aluminum foil on the table store all tools and materials on the aluminum foil throughout the mounting procedure.

Tools and materials:

- Double sided carbon sticker (top left drawer under the table behind SEM)
- Copper tape (top left drawer under the table behind SEM)
- Stub tweezers with rounded gripping ends (top left drawer under the table behind SEM)
- Sharp ended tweezers (top left drawer under the table behind SEM)
- Stub holder (top left drawer under the table behind SEM)
- Canned air (table in Nancy's office behind SEM room)
- Isopropyl (table in Nancy's office behind SEM room)
- Kimwipe (table in Nancy's office behind SEM room)

Setup

1. Lay out a clean new sheet of aluminium foil to use as a work surface.
2. Clean all tools with a kimwipe soaked with isopropyl alcohol.
3. Using a clean sample select a stub and place it in the mounting stub holder. The stub should be slightly larger than the sample to be mounted on it.

Mounting

4. Remove a double-sided carbon sticker from the plastic backing with sharp tweezers and stick it on the stub, keeping the paper backing attached so that the surface of the stub is entirely covered.
5. Using a glove and only touching the paper backing to press the sticker firmly onto the stub. Then remover the paper backing keeping it nearby to press down on samples if necessary.
6. Grab the sample using tweezers and press it down onto the sticker until it is firmly attached, for solid samples press down onto the stub using the paper backing to ensure the back is firmly attached to the stub.
7. Golding the stub with the circuit stub tweezers tap in onto the aluminium foil covered table firmly to remove dust and anything which is not well attached.
8. Cover near to the edges of the sample using copper tape wrapping the edges onto the stub removing excess that hangs below the bottom of the stub. Only use gloves and tweezers for this.

Final cleanup

9. Blow dust off sample stub with canned air.
10. Store samples in a clean sample container and assign them numbers to ensure they are not confused with each other.
11. Return tools and materials to the drawers where they originated from.