When Reading please ignore parts in yellow. Sorry if it is confusing but otherwise it would be the biggest pain to edit.

\chapter{Introduction}

The energy outlook of the 21st century is almost certainly going to be renewable. With the World’s largest economies pledging to be carbon neutral within this century, the energy they require needs to be renewable. Solar energy is one of the answers to this problem, the sun is a source of inexhaustible free energy which can be harnessed to address the needs of the world \cite[p.~894]{RN82}

Currently the market leader in solar cells are crystalline silicon photovoltaics which hold a dominant 95\% market share in the solar energy sector \cite{RN54}. Organic solar cells have the opportunity to disrupt this market by providing versatile light-weight devices which have little material consumption and low temperature processing. This, alongside the fact that the materials required for organic photovoltaics are already widely manufactured, means that they have the potential to be significantly lower cost than their silicon counterparts \cite[p.~54]{RN81}. However, the current problems with organic solar cells boil down to long term stability, alongside low power outputs relative to silicon photovoltaics \cite{RN54}. This project looks at creating a method to help solve the issue of long-term stability by developing a low-cost system which can simulate a lifetime (10-20 years) of outdoor degradation in a matter of months.

An important aspect of this project is its ability to contribute to the relevant research worldwide. Therefore, the decision was taken to make everything relating to this project opensource, as well as attempt to use opensource software and open-hardware to create this testing chamber.

Another key goal is to create a testing set up which can easily be \emph{cherry picked} from to suit the needs of those using it. This brings another important tenant to this project of modularity. Ideally, this set up would need one “chamber” which then different substrates and cells could be tested on, with only a small component of the entire set up modified.

\section{Literature Review}

\label{sec:lit}

In order to provide a device to add to the field, a thorough literature review was conducted and modified throughout the project. The main objective of this project was to provide the AFMD (Advanced Functional Materials and Devices) research group, within the University of Oxford Physics Department, with a device that created different conditions to degrade organic photovoltaic cells.

There are multiple degradation mechanisms which cause the short lifetimes of organic solar cells\cite[p.~141]{RN58}. The ones that the system will try and measure the effect of are: diffusion of water vapour into the active layer of the solar cell, diffusion of oxygen into the active layer of the solar cell and operation under high temperatures. High temperature stability is important as solar cells are exposed to direct sunlight, meaning that they will absorb heat radiation alongside light radiation exposing them to high temperatures \cite{RN79}.Other degradation mechanisms will also play a part such as photochemical degradation by UV light and oxygen \cite[p.~106]{RN57}.

An important factor for the entire set up is to ensure that these degradations are measurable, thereby requiring careful control of the ambient conditions. This meant a container that would not let in ambient air (due to the oxygen and water content) as well as have a controllable chemical and physical atmosphere. Degradation experiments are essential in the testing of solar cells; therefore, it made sense to try and find any set ups which had similar goals to the chamber. In 2015 Jaffery et al. \cite{RN59} released a paper outlining a similar setup that was designed for the testing of PVs. This design incorporated temperature control, varying lighting conditions. The set-up is shown in the Figure \ref{fig:set\_upHT9} \cite[p.~3,4]{RN59} below.

\begin{figure}[h]

\begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{HT9\_Fig1}

\caption{The Test chamber and relevant accessories used in the research}

\end{subfigure} \begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{HT9\_Fig2}

\caption{Schematic of the test chamber and relevant accessories used in the research}

\end{subfigure}

\caption{A device developed by Jaffery et al.\cite{RN59} to test organic photovoltaics}

\label{fig:set\_upHT9}

\end{figure}

This set-up is a good start to build on to meet the goals of this project however, it lacks the simplicity of what is required. There are multiple large scale components which mean a complicated building and testing process. For the chamber this project requires, it needs to be easily replicable worldwide, thereby allowing it to contribute to the development of OPV(Organic Photovoltaic) technology.

Another example is by Lai and Potters \cite{RN43} which referenced a large scale industrial chamber. This chamber also provided different conditions for testing a photovoltaic cell. This module was manufactured by Envirotriks (now Weiss Technik) who specialise in the manufacturing of environmental testing equipment. Their technology ranges from aerospace to biological environmental applications. On further investigation, their products were large scale expensive modules which would not be applicable to one of the key goals of this project, being easily replicable worldwide.

Adding to this, a further set-up was used in Pearson et al \cite{RN62} experiments on the degradation of organic solar cells. This set up ensured that the cells were loaded into the container while in a glovebox, then held at an overpressure to ensure atmospheric gases (specifically water vapour and oxygen) did not leak into the container. Furthermore, the atmospheric make-up of the container was monitored by a gas analyser and the cells measured using a Keithley source meter \cite [P.~227]{RN62}. This chamber is the closest to what was specified in my project description; however, the gas make-up monitoring adds further complexity to the chamber which again reduces the replicability worldwide.

These set ups worked well for their goals; however, they do not fully address the needs of this project. Despite this, there were key components in all three set ups which I took inspiration from for my designs. From Figure \ref{fig:set\_upHT9} the small container is something which appealed to me, especially if it were tweaked to ensure ease of set up. Furthermore, from the commercial modules, the holistic nature of all the components was something that was desirable, as this would ensure that the container would be suitable for all manners of testing. From Pearson et al \cite {RN62}, I thought the idea of overpressure to prevent leakage was clever, as well as making the device small enough to load in a glovebox. These were all features which will all be incorporated into the chamber

This all has influenced the functionality that will be designed into the container. However, one of the key features is to align closely with the standard for testing organic solar cells. Different papers reference different standards, a decision was made to see where they overlapped and create a container that was able to meet as many as possible. Zhang et al \cite[p.~1]{RN51} talk about the IEC61646 standard developed by the International Electrochemical Committee. This testing standard includes: “ 1000 h damp heat (DH) test at 85$^‌o$C and 85\% humidity, 200 cycles of thermal cycling (TC) from $−$40 to $+$85$‌^o$C, and a sequence test consisting of UV exposure, 50 cycles of TC, and 10 cycles of humidity freeze (HF) from $-$40 to $+$85 $^o$C at 85\% humidity. After finishing each test, modules are then characterised to determine device efficiency.”

Further to the IEC61646 is the guidelines referenced in Reese et al. \cite[p.~1254]{RN53}[HT 3 P1254]. These were developed at the International Summit on Organic and Hybrid Photovoltaic Stability in the years 2008. 2009 and 2010. These 3 guidelines (ISOS 1,2,3) show different methods for testing solar cell degradation, some of which can be seen in Figure \ref{fig:HT3\_fig2} \cite[p.~1255]{RN53} below.

\begin{figure}[h]

\hfill\includegraphics[width=0.9\linewidth]{HT3\_Fig2}\hspace\*{\fill}

\caption{Showing some of the testing procedures in Reese et al. }

\label{fig:HT3\_fig2}

\end{figure}

Figure \ref{fig:HT3\_fig2} has been taken from Reese et al. \cite{RN53} and outlines some different testing conditions for organic solar cells. As can be seen the guidelines are similar to those referenced in the IEC61646 standard, however, there is more detail on the exact light source, as well as the load the solar cell should be subject to.

From these two sets of guidelines, basic functionality for the container could be drawn up. This includes: temperature variation with a minimum maximum temperature of 85 degrees, ability to vary humidity, regular measurement of Voc (open circuit voltage)and Jsc (short circuit current) and light conditions which would vary from a solar simulator to outdoor sunlight to darkness. Both sets of guidelines reference temperatures below ambient room temperature; however, this would require some sort of refrigerant running through the device, connected to pumping systems and a heat exchanger. This would cause the box to be particularly cumbersome as it would require significantly more components, space and complexity meaning it would struggle to fit the requirement of easily replicable. Thereby, the decision was made to not have testing conditions below ambient room temperature and to have a chamber that could exceed the high temperature testing requirements with a substrate temperature of 120$^o$C.

Another integral consideration was sampling time. For accurate results, it would be desirable to have regular short interval (measurements every minute) measurements of Voc and Jsc. However, due to the differences across different devices and degradation conditions having a singular sampling time would create either too much data, or be too long to create an accurate enough picture of the device. The Figures \ref{fig:O2\_deg} and \ref{fig:N2\_deg} show the differences in degradation for the same solar cells in different conditions.

\begin{figure}[h]

\begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{O2\_deg}

\caption{Solar cell exposed to dry oxygenated atmosphere \footnote{Under continuous white light radiation, with the atmosphere containing 20\% oxygen $<4\%$ relative humidity.},taken from Kawano et al. \cite{RN83}}

\label{fig:O2\_deg}

\end{subfigure} \begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{N2\_deg}

\caption{Solar cell exposed humid nitrogen atmosphere\footnote{Under continuous white light radiation with the atmosphere containing 0/% oxygen and $>40\%$ relative humidity.}, taken from Kawano et al. \cite{RN83}}

\label{fig:N2\_deg}

\end{subfigure}

\caption{Normalised photovoltaic performance characteristics for an unencapsulated solar cells as a function of exposure time. FF is Fill Factor, which represents ratio of maximum power to Jsc$\times$Voc.}

\label{fig:degs}

\end{figure}

From Figures \ref{fig:O2\_deg} and \ref{fig:N2\_deg} it is clear to see that the it would be impossible to create a singular sampling time for different conditions without creating the issues mentioned. This is an important factor that should be considered when designing the measurement system and will be discussed in Section \ref{sec:code\_development}.

\chapter{Chamber Design}

\label{chp:design}

The design of the container and associated electronics was a key process in the development of the chamber. This chapter will go through the entire design process, outlining the key aims, decisions and analysis behind the container design.

During the mechanical design process, there were two different software packages used: OpenSCAD and SOLIDWORKS. OpenSCAD is an open-source software which is compatible with all major computer operating systems. Furthermore, as mentioned in the introduction, using open-source software is a key tenant of this project as it needs to be replicable for teams worldwide. SOLIDWORKS however, is not open-source, but was still used, because there is some key functionality in SOLIDWORKS embedded in SOLIDWORKS (such as rendering photos, heat simulations and producing engineering drawings). However, as I will lay out further in the chapter, the main designs were completed on OpenSCAD, and they do hold enough information for a researchers and engineers elsewhere to replicate without significant difficulty.

A key consideration was what processes would be needed for the manufacturing of the container. The engineering department has different facilities which could be useful including: a workshop with a CNC machine, multiple different 3D printers, a mechanical workshop and an electrical workshop. These all were considered when designing the container and each will be mentioned during this chapter.

During the electrical design process there was more of a conundrum. Due to COVID-19 regulations the design lab was closed (at the time when I needed to conduct electrical designs), meaning that I was unable to access key software (SOLIDWORKS PCB and Electrical) – the favoured software by the electrical workshop. Therefore, an open-source solution needed to be found, the solution was KiCAD. KiCAD was readily accessible, user-friendly and could run on a variety of computers.

\section{Physical Specification}

\label{section:Physical\_spec}

The first stage of the design process was to outline a specification for the testing container. These specifications were drawn from the project brief, the literature review and discussion with my supervisor Professor Moritz Riede. The primary specification of the design is outlined below:

\begin{enumerate}

\item{The container must be able to accommodate a 30 mm x 30 mm substrate provided by AFMD research group.}

\item{The container must be leakproof \footnote{must not allow enough Oxygen and water vapour in from ambient atmosphere to degrade a calcium sample with dimensions 30 mm \*30 mm \*150 nm over a period of 3 days} to outside air.}

\item{The container must allow electrical connections \footnote{The connections must enable the cell to be controlled/measured by a source meter.} from outside to connect to the substrate for measurements.}

\item{The container must enable the substrate to be heated to a given temperature \footnote{Up to 120$^o^$C as mentioned in Section \ref{sec:lit}.}

\item{The container must have a window allowing light\footnote{The AM1.5 light spectrum} to be shone onto the substrate.}

\item{The container must contain a gas inlet.}

\item{The container should fit into the small glovebox load lock with diameter 150 mm.}

\item{It must be possible to assemble the chamber within the glovebox.}

\end{enumerate}

The desirable goals which would supplement the primary specification are as follows:

\begin{itemize}

\item{Build a Python based GUI (Graphical User Interface) to enable programmatic testing of the solar cell.}

\item{Enable a programmable atmosphere for the box which should be embedded into the functionality of the GUI.}

\end{itemize}

This specification is a clear guide to what functionality there needs to be within the container, as well as any size limitations. As mentioned in both the introduction and literature review, it is essential for the container to be leakproof, to ensure that the solar cell does not degrade due to atmospheric O2 and water vapour. This would result in flawed results due to the cell having some unmeasured degradation before the experiments even begin. Another important point in the specification is the ability of the cell to be heated to a given temperature\*. This functionality is important as its role is to attempt to emulate a lifetime (20 years) of temperature degradation in the space of 3 months.

The gas inlet is another feature to enhance the degradation. This will be used to create a ‘cocktail’ of different gases (e.g. oxygen and water vapour) to try and emulate lifetime degradation of the solar cell. The last point on the specification is to ensure the ease of use with the AFMD research group. The gloveboxes they use have a small inlet with a diameter of 150 mm, using this would vastly reduce the time needed to insert the solar cell into the testing container.

These desirable goals are important to ensure the ease of use of the box, as well as to reduce the amount of time needed for setting up and running the container. The programmatic testing of the cell is important as it enables simple running of the chamber, saving time and reducing complexity for those using it.

\section{Initial Mechanical Designs}

This specification provided the structure for the testing container. The literature review showed that there is no similar devices on the market to the chamber, thereby requiring innovative design to satisfy the specification. A first iteration of the design is shown below in Figures \ref{fig:plan\_iteration1} and \ref{fig:iso\_iteration1}.

\begin{figure}[h]

\begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{Design\_1}

\caption{Plan View of Model}

\end{subfigure} \begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{Design\_2}

\caption{Perspective View of Model}

\end{subfigure}

\caption{Showing a Plan and Perspective view of the First Iteration of the Outer Shell}

\label{fig:plan\_iteration1}

\end{figure}

\begin{figure}[h!]

\hfill\includegraphics[width=0.45\linewidth]{Design\_3}\hspace\*{\fill}

\caption{Showing Isometric View of the Outer Shell. Label \textbf{a} represents the ‘electronic windows’ where the wires will go, label \textbf{b} represents the holes for the push-fit valves and label \textbf{c} shows the screw holes.}

\label{fig:iso\_iteration1}

\end{figure}

This model is supposed to coincide with a smaller module named the Substrate Holder, which is designed to sit within the outer shell, holding the substrate, temperature sensor and heater, shown in Figure \ref{fig:substrate\_holder1}. The substrate holder is designed to be a removable component which can be edited to match the substrate provided. During the design process, this module was designed to be 3D printed to ensure low costs and easy modification. In Figure \ref{fig:substrate\_holder1} label \textbf{a} represents the whole which the heater is meant to sit inside, while label \textbf{b} represents the 30 mm x 30 mm slot that the substrate will sit in.

\begin{figure}[h]

\hfill\includegraphics[width=0.45\linewidth]{Design\_4}\hspace\*{\fill}

\caption{Showing the Isometric view of the first iteration of the Substrate Holder}

\label{fig:substrate\_holder1}

\end{figure}

Both the substrate holder and Outer Shell were 3D printed to provide a physical representation of the model (with a photo shown in Figure \ref{fig:3D\_print1}). This was because the process was iterative: design, rapid prototype, design, rapid prototype. The reason behind this was to ensure that the design process found potential flaws within the chamber preventing time being wasted in the future where design flaws could impact the entire timeline. This process paid off as after 3D printing the design, it became clear that this design was very small and fiddly, making it difficult to assemble within the glovebox. Moreover, it is clear that there would not be enough room to wire the components in the container.

\hfill\includegraphics[width=0.45\linewidth]{Design\_5}\hspace\*{\fill}

\caption{Showing the result of the first 3D print of the model}

\label{fig:3D\_print1}

\end{figure}

\section{Final Container Models}

\label{sec:FCM}

To counter the problems listed, the following iteration - shown as a CAD model I Figure \ref{fig:outer\_final} – fixes the listed problems. The Outer Shell is larger in this model with dimensions 100 mm\* 110 mm\* 77 mm, with a wall thickness 16 mm, resulting in more space within the container. Additionally, there was a rethink of the sealing method, to eliminate the need of directly screwing onto glass, risking a break, therefore a lid was constructed for the substrate holder, which would cause the glass window to be sandwiched between 2 O-rings creating an airtight seal. The changes to the outer shell can be seen in Figure \ref{fig:outer\_final}, with the new lid shown in Figue \ref{fig:metal\_lid\_CAD}.

\begin{figure}[h]

\hfill\includegraphics[width=0.45\linewidth]{Design\_6}\hspace\*{\fill}

\caption{The Final Outer Shell model.}

\label{fig:outer\_final}

\end{figure}

\begin{figure}[h]

\hfill\includegraphics[width=0.45\linewidth]{metal\_lid\_CAD}\hspace\*{\fill}

\caption{Showing the Lid which will be used to sandwich the glass plate between 2 o-rings.}

\label{fig:metal\_lid\_CAD}

\end{figure}

The substrate holder was also modified with the intention of being unique to this particular substrate layout resulting in a more detailed design. The pin layout – which includes the light masks - was provided to me by Dr. Grey Christophoro, a researcher working in the Physics department, and can be seen in Figure \ref{fig:sub\_layout}.

\begin{figure}[h]

\hfill\includegraphics[width=0.45\linewidth]{Design\_7}\hspace\*{\fill}

\caption{Showing the pin layout provided by Dr. Christophoro, the light masks show the area which light should be incident on.}

\label{fig:sub\_layout}

\end{figure}

As shown in Figure \ref{fig:sub\_layout} the substrate has 10 contact points, meaning that the substrate holder needs to provide a method to cleanly contact the contact points, without hindering the flow of air around the cell\footnote{To ensure that gases that are needed for degradation are able to react with the active layer or electrodes}, or blocking the light incident on the cell surface. The solution was to develop a small lid which would be screwed into the substrate holder, thereby providing downward pressure onto the substrate to ensure clean contacts with the pins. This lid was designed so that the outline did not block any light being incident on the cells. The lid can be seen on top of the substrate holder in Figure \ref{fig:sub\_lid}.

\begin{figure}[h]

\hfill\includegraphics[width=0.45\linewidth]{Design\_8}\hspace\*{\fill}

\caption{Showing the lid used to exert downward pressure on the substrate.}

\label{fig:sub\_lid}

\end{figure}

Another important consideration was the ability for the substrate holder to be easily attached to the outer shell within the glovebox. Therefore, the substrate holder needs to have electrical wiring within it, to easily connect up the solar cell with the monitoring device. Conversations with the electrical workshop helped me come up with a simple idea of creating a ‘plug’ which would enable the substrate holder to be connected to wiring that in turn would be connected with the monitoring device. This will be discussed about in the further in the testing chapter.

Another small edit is the gap under the where the substrate is supposed to sit, this is to encourage gas flow beneath the substrate. Another issue was where the heater and temperature sensor would sit. This led me to place the two holes running through the substrate holder one on top of the other to place the sensor and the heater. These holes were positioned there tactically so that the sensor temperature could be correlated with the predicted substrate temperature by modelling. These changes can be seen in the blown-up overall model shown in Figure \ref{fig:blown\_up}.

\begin{figure}[h!]

\hfill\includegraphics[width=0.8\linewidth]{Design\_9}\hspace\*{\fill}

\caption{The Blown-Up model of the chamber}

\label{fig:blown\_up}

\end{figure}

\section{Electrical Specification}

\label{section:Electrical\_spec}

This section will take a more in depth look at the electrical requirements of the chamber. From the pin layout, it is clear that there needs to be 20 wires connected to the cell. For this particular substrate layout, there are 6 separate cells which means a PCB needs to be manufactured which is able to choose which of these cells to measure. Each cell needs to be measured to find the value of the Voc and Jsc recorded with the time, temperature and gas composition the substrate is subjected to.

Adding to this, a measurement device needed to be chosen, along with a controller to run and record the data. After discussion with the AFMD group, Dr. Christophoro and my supervisors Professors Riede and Morris the following electrical specification was drawn up:

\begin{itemize}

\item[-] The set-up requires a method of differentiating the cell voltages and currents (ideally through the use of a multiplexer);

\item[-] The set-up requires a method of controlling the heat and gas composition within the chamber as well as the measurement intervals;

\item[-] The set-up requires a method to record the many\* Voc and Jsc measurements;

\item[-] The set-up requires a method of measuring the Voc and Jsc;

\item[-] The set-up requires a method of measuring the temperature conditions within the container.

\end{itemize}

This specification is non-exhaustive but does provide a clear structure to what is needed for the successful manufacturing of the set-up.

\section{Electrical Design}

\label{sec:elec\_design}

For the control systems, there were two options: a Raspberry Pi or an Arduino. Both have extensive documentation and are relatively inexpensive given their power. To decide which would be more suitable a Multi-Criteria analysis was undertaken with the results shown below:

\begin{table}[ht!]

\begin{tabular}{c|c|c}

&Raspberry Pi 4 & Arduino Uno Rev3 \\

\hline

Cost & 4 & 10\\

\hline

Simplicity of use & 10 & 5 \\

\hline

Size & 3 &5 \\

\hline

Adaptability & 20 & 10 \\

\hline

Total & 37 & 30\\

\end{tabular}

\caption{\label{tab:MCA-Pi} The Multicriteria Analysis between the Raspberry Pi and Arduino}.

\end{table}

The cost of a Raspberry Pi 4 from RS- components was \pounds53.22 \cite{RN63}while the cost of the Arduino Uno Rev3 which I planned on using is \pounds20.40 \cite{RN64}. Cost was a factor in my decision, it was not as important as adaptability, yet more important than size, therefore I rated the Arduino with a score of 10, and Raspberry - Pi with a score of 4. The simplicity to use was something that I believe should have the same weighting as cost. From discussions with colleagues, it was clear that running different programs was more difficult on the Arduino due to the fact it stored one program at a time, meaning each time a procedure needed to be changed: the Arduino needed to be plugged in to a computer. The Raspberry-Pi, however, is simpler, it is more like a conventional computer, able to store multiple programs, runs with a modified Linux operating system, thereby creating a simpler system.

The size was a small factor to consider, thereby the reduced emphasis on scoring. Lastly, adaptability of the device was integral. The device needed to be able to store data, as well as run code which was easily modifiable for those using it. This created a clear divide between the two products as the Raspberry-Pi is by far considered a more ‘rounded’ device compared to an Arduino which is usually used for highly specific applications.

From the multi-criteria analysis above it is clear that the Raspberry-Pi is the preferred device and thereby was the chosen product for this application. The Raspberry-Pi required electronics which enables the selection of which solar cell was required. After discussions with Dr. Christophoro, I was pointed to some PCB designs in his GitHub repositories \cite{RN65} \cite{RN66} which could be modified to suit my applications. The two schematics of designs that I was directed to are shown in the appendix as Figure 1 and 3.

These designs needed to be modified for the following reasons. Firstly, the design named Base 8x, was designed to interface with an Arduino, meaning a subsequent header needed to be implemented to ensure that the PCB was able to interface with the Raspberry Pi. Other small modifications include adding resistors and a header for a temperature sensor to be wired in, as well as creating the mounts for a secondary PCB to be mounted (which would hold the Solid-State Relay needed to control the mains powered cartridge heater).

For rapid testing and prototyping, the decision was made to use the departmental PCB printers. This meant some further changes needed to be made to the PCBs: ensuring that the tracks were routed with large enough profiles to be printed on the departmental printers and modifying the PCB so that it only had two working layers \footnote{The departmental printers can only print a PCB with two layers or less}. These modifications in the schematic can be seen in the Appendix \ref{fig:appendix3}.

For the PCB named Solar Mux- schematic shown in Appendix \ref{fig:appendix2} - there needed to be significantly fewer modifications, yet they were still significant. Firstly, the number of relays were reduced from 21 to 17 as only 6 solar cells were being measured rather than 8. Furthermore, there were significant modifications to the routing of the tracks as again this PCB was not designed to be printed in the engineering department thereby meaning the tracks needed to be made larger. These changes to the schematic can be seen in Appendix \ref{fig:appendix4}.

I also designed a third PCB whose schematic can be seen as appendix 5. This is a small PCB designed to isolate mains current from the board named base, thereby enabling me to run tests without the hazards posed by wiring up to the mains.

Once these were all designed, they needed to be created into models that were manufacturable. Each PCB had two working layers made of copper, the components to be soldered on were nearly all through-hole mounted for ease of soldering and the tracks were mainly 1 mm thick to make it easy to solder the components onto. Each PCB can be seen from two views in the Figures \ref{fig:Base\_3D},\ref{fig:MUX\_3D},\ref{fig:SSR\_3D}.

\begin{figure}[h]

\begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{Base\_3D\_1}

\caption{Front View of PCB named Base}

\end{subfigure} \begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{Base\_3D\_2}

\caption{Back View of PCB named Base}

\end{subfigure}

\caption{Showing the model of the PCB named Base }

\label{fig:Base\_3D }

\end{figure}

\begin{figure}[h]

\begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{MUX\_3D\_1}

\caption{Front View of PCB named MUX}

\end{subfigure} \begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{MUX\_3D\_2}

\caption{Back View of PCB named MUX}

\end{subfigure}

\caption{Showing the model of the PCB named MUX }

\label{fig:MUX\_3D }

\end{figure}

\begin{figure}[h]

\begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{SSR\_3D\_1}

\caption{Front View of PCB named SSR}

\end{subfigure} \begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{SSR\_3D\_2}

\caption{Back View of PCB named SSR}

\end{subfigure}

\caption{Showing the model of the PCB named SSR }

\label{fig:SSR\_3D }

\end{figure}

\section {Code Planning}

Chosing the Raspberry Pi enabled the development of the code and planning behind it. There was some general research through the documentation to understand the ways to use the General-Purpose Input Output header to control electronics (GPIO). From reading the documentation for the GPIO \cite{RN67}, it was clear there were 3 real options for programming language, C, C++ or Python. As someone who has experience in Python, alongside the \emph{extensive} documentation for the GPIO pins in Python \cite{RN68} it was a clear choice to use Python as the programming language.

Using the library Gpiozero, tests were carried out to ensure that the Raspberry Pi purchased was working correctly, as well as checking some other features (such as the multiprocessing library) work with Gpiozero. To do multiple LEDs were run in parallel so that they would be blinking with each other, using different time intervals for the blink. This proved that the Gpiozero library worked with the multiprocessing library and enabled the planning of the main script that was going to control the testing container.

The thought process behind the code architecture was to develop a Graphical User Interphase (GUI) to help the user with the control of the testing container. The GUI would then display specific information while the test was running, enabling a convenient ‘check’ for the user to ensure the test is proceeding as planned. The resultant GUI can be seen as a screen-shot in Figure \ref{fig:GUI\_1}.

There were, however, some problems with this set-up. Once the GUI itself was constructed I was unable to integrate it with the Multiprocessing package. A decision needed to be taken: run the risk of chasing a \emph{desriable} but not essential goal, using up valuable time, or create an easily editable python script which incorporated many of the benefits of the GUI while not actually using one.

Due to the iterative nature of this project, the decision was taken to prioritise constructing a working container that may not meet the supplementary desirable goals (but would be operational), rather than create a container that was 80% working yet not ready to test solar cells. For this reason, I decided to put the GUI to one side and develop the code so that it was simple to change the parameters of the test from within. An overall architecture was drawn up and is shown below in Figure \ref{fig:code\_planning}.

\chapter{Manufacturing}.

\label{chp:manufacturing}

In this chapter I will be discussing the different stages of manufacturing of the chamber. The manufacturing process is split into three sections in this chapter which cover the manufacturing of the chamber itself, the manufacturing of the PCB and associated electronics as well as the code development.

From the Gantt chart shown in Appendix \ref{fig:Gantt}, the manufacturing process was estimated to take at least n weeks, this prediction included any time that was needed for iterating as well as physically building the components. Unfortunately, due to the second national lockdown in January 2021, the project was hampered by a series of delays. This can be seen in Appendix \ref{fig:Gantt\_covid} where the delays can be seen clearly.

As mentioned in the Design Chapter, the plan was to use the engineering department workshops for the manufacturing of all the parts of this chamber. There were some limitations, such as the fact the PCB printing machine could only print 2 layers. However, this was dwarfed by the expertise that I was able to utilise while developing these designs, ensuring that the chamber was constructed to the highest standards. This chapter will discuss the decisions made during the manufacturing process, any modifications made and their impact on the final product.

\section{Outer Chamber Manufacturing}

This section will discuss the manufacturing of the container itself, including components which were attached to the chamber From the designs shown in Section \ref{sec:FCM}, engineering drawings were produced and shown to Duncan Constable (the senior workshop technician) who advised me that the inside corners would come out rounded due to the precision of the CNC mill. Furthermore, I was instructed to change the shape of the O-ring ‘gaps’ so that the O-rings would not tear around the sharp corners.

Another important factor was the material selection. As mentioned previously, the material needed to be impermeable, preferably metal with limited corrosion. Speaking to Duncan about the preference of Aluminium, he suggested Duralumin, which the workshop had an abundance of spare stock and it was the most suitable metal for the CNC miller to manufacture. Dural is a 2000 series Aluminium alloy, with the main materials apart from Aluminium being Copper, Manganese and Magnesium \cite{RN73}.

The designs were then submitted to the workshop for manufacturing with the drawings shown in Appendix \ref{fig:drawing\_1}, \ref{fig:drawing\_2}, \ref{fig:drawing\_3}. Alongside the parts shown in the appendix, the electronic windows were manufactured from scrap metal using a lathe, with the drawings shown Figure \ref{fig:metal\_window\_drawing} below. These parts were then modified by hand to create leakproof wire connections between outside the container and within, with the finalised part shown embedded into the container in Figure \ref{fig:metalwindow}.

\begin{figure}[h!]

\hfill\includegraphics[width=0.6\linewidth]{elec\_windows\_drawings}\hspace\*{\fill}

\caption{The Electrical ‘windows’ part drawing.}

\label{fig:metal\_window\_drawing}

\end{figure}

As can be seen, the wires are glued (using a glue gun) into the metal, meaning that they should not allow any leakage of air through, as well as not allowing short circuit due to the metallic casing. Furthermore, there is glue around the outside of the ring component (labelled a), to prevent leakage of air into the container. The advantage of using glue gun glue, is that the material is a thermoplastic, meaning that if there is an error, it can be heated and removed allowing the continuous modification of the chamber.

\begin{figure}[h!]

\begin{subfigure}}{0.5\textwidth}

\includegraphics[width=0.95\linewidth]{elec\_windows\_glued}

\caption{The Electrical windows embedded into the chamber}

\label{fig:metalwindow}

\end{subfigure}\begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.95\linewidth]{safety\_relief}

\caption{The Assembly of the push-fit and safety relief valves}

\label{fig:safety\_relief}

\end{subfigure}

\caption{Figures Showing the assembly of the chamber}

\end{figure}

Following the manufacturing of the outer shell, assembly occurred. Firstly, the O-rings needed to be attached to the chamber. Figure \ref{fig:oring\_assem} shows the O-ring glued into its slot. The O-rings sandwiched an acrylic plate which was used for the testing of the system (before any expensive glassware was bought) shown in Figure \ref{fig:acrylic\_plate}. This assembly concluded with the attachment of the safety relief valves\footnote{The safety relief valve is required in case there is a malfunction with the MFCs causing a large overpressure within the chamber, which could result in an explosive failure causing damage and injury}and push fit valves shown in Figure \ref{fi:safety\_relief} sourced from Tameson \cite{RN36} and RS\cite{RN33} respectively. Lastly, the assembly was completed using small guide pins, labelled in Figure \ref{fig:assembly} alongside the M4 screws used to tighten the lid onto the glass plate thereby creating an airtight seal. The testing of this will be discussed further in the following chapter.

\begin{figure}[h!]

\begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{oring\_assem}

\caption{The O-ring glued into the lid of the container.}

\label{fig:oring\_assem}

\end{subfigure}\begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{acrylic\_plate}

\caption{The acrylic plate sandwiched between 2 O-rings.}

\label{fig:acrylic\_plate}

\end{subfigure}

\caption{Further figures showing the assembly of the chamber}

\end{figure}

\begin{figure}[h!]

\hfill\includegraphics[width=0.7\linewidth]{assembly}\hspace\*{\fill}

\caption{The fully assembled chamber}

\label{fig:assembly}

\end{figure}

\section{Internal Chamber Manufacturing}

\label{sec:ICM}

This section looks at the manufacturing of the substrate holder. Figures \ref{blown\_up} show the designs which were manufactured using the departmental 3D printing machines with ABS (Acrylonitrile Butadiene Styrene) plastic. ABS has a softening point of between 160 – 170$‌^o$C \cite[p.~744]{RN70}, which is lower than desired for the container, however, this version was used as a test to see if it was possible to insert the electrical contacts with this design.

Figure \ref{failure} shows my attempts to insert electrical contact pins \cite{RN71} into the substrate holder. This was unsuccessful due to the fact these pins were extremely fiddly to handle, coupled with the very small amount of space I had to solder wires onto the pin while not melting the plastic. This required a rapid solution otherwise the timeline for the project would be delayed.

\begin{figure}[h!]

\hfill\includegraphics[width=0.5\linewidth]{substrate\_fail}\hspace\*{\fill}

\caption{The substrate holder with 4 electrical contact pins soldered in place. }

\label{fig:failure}

\end{figure}

The solution was creating a new 3D printed part, with more space for the pins to be inserted and soldered correctly. The new substrate holder would also be made from PEEK (Polyether Ether Ketone) rather than ABS due to its higher MP (260$‌^o$C) \cite{RN72} allowing soldering of the pins to wires to take place, as well as no deformation within the container. This part was due to be manufactured in the engineering department, however, due to external factors, the production had to be outsourced to Lazer Lines, with the part made from Formlabs High Temp Resin for High Thermal Stability, ith the datasheet found at \cite{RN80}. The substrate holder and lid can be seen in Figures \ref{fig:substrate\_HT} and \ref{fig:lid\_HT}.

The substrate holder was then modified to incorporate the pins as was attempted in Figure \ref{failure}. This attempt proved successful with the pins clearly showing in Figure \ref{fig:HTpins}.

\section{PCB Manufacturing}

As mentioned in Section \ref{sec:elec\_design}, the PCB was designed to be manufactured in the Engineering Department Electronics Workshop. Using the workshop allowed access to expert help alongside rapid prototyping. Using the engineering workshop meant that there were limitations to the capability of the printer. The holes were not plated through meaning nearly all components could only be connected to the bottom layer. Furthermore, this meant the via’s produced needed to have wires running through them and soldered onto both layers, adding time and complexity to the soldering process.

Using a soldering iron, I was able to complete two of the three PCBs. I was not allowed to solder the PCB named SSR-PCB as it would be connected to mains, therefore, cause a safety risk if someone not qualified completed the wiring\footnote{This needs to be done by qualified technician, however, due to covid this was not completed}. Figures \ref{fig:base} and \ref{fig:mux} show both sides of the completed PCBs.

\begin{figure}[h]

\begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{Base\_front}

\caption{Front View of PCB named Base}

\end{subfigure} \begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{Base\_back}

\caption{Back View of PCB named Base}

\end{subfigure}

\caption{Showing the fully built PCB named Base }

\label{fig:base}

\end{figure}

\begin{figure}[h]

\begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{Mux\_front}

\caption{Front View of PCB named Solar Multiplexer}

\end{subfigure} \begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{Mux\_back}

\caption{Back View of PCB named Solar Multiplexer}

\end{subfigure}

\caption{Showing the fully built PCB named Solar Multiplexer }

\label{fig:mux}

\end{figure}

From here the next steps were to connect the PCBs to the substrate holder via the electronic windows. This required careful soldering of wires to ensure no short circuit occurred. To ensure that this was all correctly working testing was done on all the components with the method and results explained in the following chapter. Figure \ref{fig:final} show the PCBs connected together and wired up to the chamber.

\begin{figure}[h!]

\hfill\includegraphics[width=0.9\linewidth]{final}\hspace\*{\fill}

\caption{The chamber fully assembled to the PCBs and Raspberry Pi }

\label{fig:final}

\end{figure}

\section{Code Development}

\label{sec:code\_development}

The code development for the system was something that spanned the entire project. This is due to the slight changes in scope, mentioned in the \ref{sec:code\_d}. However, the core of the program was unchanging allowing a good base for the measurement system to be built on. The lack of GUI does add slightly more complexity to the user; more importantly however, it does work.

The method used to combat the increase in complexity is a small introduction at the top of the script explaining where to input the testing conditions and exactly what variables this system is able to change, shown in Figure \ref{fig:code\_intro}. A small document was also produced (shown in the Appendix as Figure \ref{fig:code\_doc}) so the user could easily understand the test thereby allowing them to utilise the functionality best of the testing chamber.

The goal of the code was to control the internal environment of the chamber while measuring and storing the current voltage characteristics for analysis. To do this a Keithley 2400 was used to for these measurements. The advantage of using a Keithley is that it is also able to set a voltage bias across the cells and allow degradation to occur near maximum power point. This aspect is beyond the scope of the project, however, keeping this in mind would help those using it create modifications to suit their needs.

Initial communication issues derailed attempts to use the Keithley 2400 with the Raspberry Pi, therefore the measurements for the leak tests were conducted using a simple multi-meter. \footnote{Due to COVID-19, obtaining a Keithley for the leak tests took time, meaning that the testing of the code was unable to take place till late on in the process. With the code not working, a decision was made to prioritise the chamber testing, with the code being an aspect someone could develop further.}

One of the important things was to ensure that the data is saved regularly (outside the script) so that if the test is interrupted for any reason (accidental removal, power cut or error) the data that has been collected will be available for recovery and analysis. To do this, at every measurement point the data is saved to a CSV file, thereby insuring that even if the test is compromised, the data will be saved. Furthermore, to ensure that the information container in the CSV file is readable for those outside the group, formatting is explained at the start of the CSV file with the start date and time also recorded within the file. This can be seen in Figure \ref{fig:csv}.

The development of the code allowed the project to move on to the next steps, including testing

Conclusion of manufacturing chapter

\chapter{Chamber Testing and Simulations}

\label{chp:testing}

The tests conducted range from heat and flow simulations to leak testing. The leak and PCB tests are designed to allow the accurate rebuilding of the chamber for other research groups to use. Therefore, this means that the tests conducted should be done with equipment that would \emph{reasonably} be found in laboratories worldwide (glovebox, source meter, simulation software etc.). This chapter outlines all the simulations and tests as well as their results and analysis.

\section{Heat and Flow Simulations}

The simulations were undertaken in SOLIDWORKS, this is because it was very simple to run the simulations in the same software as some of the designs. Initially heat simulations were undertaken. These were designed to emulate the conditions across the substrate and within the substrate holder. The simulations were conducted using SOLIDWORKS heat simulations, where many different conditions could be placed upon the chamber.

The parameters for the initial simulation were as follows: ambient temperature of 25$‌^o$C C, heater temperature 200$‌^o$C and a convective heat transfer coefficient of 2.5 Wm$^{-2}$L$^{-1}$. The value of 2.5 Wm$^{-2}$L$^{-1}$ was chosen as a lower bound of natural convection from Kosky et al.’s book \cite[p.~264]{RN76}. Using these values, the test was run with a cylindrical cartridge heater. This simulation was only simulating the heat transfer across the substrate holder with the results shown in Figure \ref{fig:heat\_sim\_1}.

\begin{figure}[h!]

\hfill\includegraphics[width=0.7\linewidth]{heat\_sim\_1}\hspace\*{\fill}

\caption{The first heat simulation showing the temperature variation across the substrate. holder}

\label{fig:heat\_sim\_1}

\end{figure}

These simulations were conducted with the substrate holder being made out of PEEK. This is because the safe working temperature range for ABS plastic (which the prototype was made of) is between -20$^o‌$C and 80$‌^o$C \cite{RN77}. Furthermore, this thermal simulation has an aluminium plate at point \textbf{a} to ensure that the temperature distribution was uniform below the area the substrate holder would sit. As can be seen the temperature varies across the substrate holder from a max of 200$^o‌$C to 80$‌^o$C, with the aluminium plate having a uniform temperature of 180 $‌^o$C.

A further simulation was conducted, this time setting the Aluminium plate to 180 $‌^o$C and modelling the temperature distribution across the substrate. This resulted in the temperature distribution shown in Figure \ref{fig:heat\_sim\_2}.

\begin{figure}[h!]

\hfill\includegraphics[width=0.7\linewidth]{heat\_sim\_2}\hspace\*{\fill}

\caption{The second heat simulation showing the temperature distribution across the substrate, the substrate labelled as point \textbf{s}.}

\label{fig:heat\_sim\_2}

\end{figure}

It can be clearly seen that the substrate (marked at point \textbf{s}) has non uniform temperature distribution. However, the temperature distribution across where the cells would be found (shown in Figure \ref{fig:sub\_layout}) illustrates that there is a uniform temperature distribution across the solar cells themselves with a temperature of 120$‌^o$C. This is the desired temperature for the cells and indicates that a cartridge heater, heated to 200 $‌^o$C should cause the substrate to be heated up to the temperature set out in the specification.

As well as heat simulations, flow simulations were also conducted. This was to understand the mechanism in which input gases would flow inside the chamber and around the substrate. SOLIDWORKS flow simulation was used for these simulations. The gas was modelled as an ideal gas, allowing the assumptions of the gas particles having no intermolecular forces and random movement. To understand the movement of the gas particles, the simulation was set up to show the trace of multiple singular particles over a time period of minutes.

\begin{figure}[h!]

\hfill\includegraphics[width=0.7\linewidth]{flow\_sim\_1}\hspace\*{\fill}

\caption{View 1 of the flow simulations}

\label{fig:flow\_sim\_1}

\end{figure}

This view shows that there is good mixing around the chamber, however, it is hard to tell the distribution of the input gases around the substrate. Figure \ref{fig:flow\_sim\_2} shows the view from underneath the substrate with the substrate holder hidden from view. In this view, it is clear that there is minimal direct flow over the cells (which are located on the underside of the substrate). However, there is good mixing away from cell and due to the tests being 1000s of hours, it is assumed that the mixing under the cells will be driven by diffusion.

\begin{figure}[h!]

\hfill\includegraphics[width=0.5\linewidth]{flow\_sim\_2}\hspace\*{\fill}

\caption{Underneath view of the flow simulations}

\label{fig:flow\_sim\_2}

\end{figure}

These simulations allow the user to understand the mechanisms by which the chamber reacts to heat and gas flow thereby enabling them to

These simulations were conducted to allow the user to understand the flow and heat variation across the chamber.

\section{Electrical Testing}

This section discusses the PCB testing, specifically the testing of the code with the PCBs and checking the soldering for any short circuits.

The first step of the tests was to conduct visual checks on the PCBs to ensure that there are no visible short circuits within the design. This involved using a magnifying glass to look or visible short circuits. Once this was complete, a digital multimeter was utilised to ensure that the resistance between unconnected tracks was unmeasurable as well as checking that the resistance between tracks which are supposed to be connected was minimal (as close to zero as possible). This test discovered 2 small errors where the via’s were not soldered fully, requiring further work to ensure good connections.

Once this was rectified, tests involving the Raspberry Pi were undertaken. Firstly simple connection testing using a multimeter to ensure that the Pi was outputting high or low voltages and the connections between the Pi and the PCBs were at an acceptable standard. Using jumper leads as shown in Figure \ref{fig:jumpers}, the Pi was connected to the PCB named Base (with the schematic shown in the Appendix as Figure \ref{fig:Appendix3}) and a multimeter used to check voltages were 5V or 3.3V - depending on which pin the PCB was connected to.

\begin{figure}[h!]

\hfill\includegraphics[width=0.7\linewidth]{jumpers}\hspace\*{\fill}

\caption{Showing how the Pi and PCBs were connected.}

\label{fig:jumpers}

\end{figure}

Once finished, the following step was to attempt to run some of the code I had written on the PCB. The purpose of the original code is to select a cell for the Keithley 2400 to measure, however, this modified slightly to select a relay to allow current through to light up an LED. Initially, the plan was to use the MCP23S17 (shown in the multiplexer schematic in the Appendix as Figure \ref{fig:Appendix4}), however, after some testing this was not working as planned. However, I noticed that the Pi had significantly more Input/Output (I/O) pins than the Arduino that this PCB was originally designed to work with, enabling me to use the ‘extra’ I/O pins to bypass the serial port expander (MCP23S17) creating a simpler code and PCB.

\begin{figure}[h!]

\begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{sc\_1}

\caption{Before image of the Solar Multiplexer}

\end{subfigure} \begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{sc\_2}

\caption{The Solar Multiplexer after the edits.}

\end{subfigure}

\caption{Showing the edited Solar Multiplexer PCB}

\label{fig:sc}

\end{figure}

This step was only done after PCB manufacturing; therefore, I took the decision to solder wires to \emph{short-circuit} the MCP23S17. The before and after of this can be seen in Figures \ref{fig:sc\_1} and \ref{fig:sc\_2}. Once this was complete, the test code was run again, this time using the GPIO zero package to switch the SSRs on and off, thereby allowing the LEDs to light up. This can be seen in Figure \ref{fig:LEDs} as well as the video linked \href{http://www.latex-tutorial.com}{here}.

\begin{figure}[h!]

\hfill\includegraphics[width=0.7\linewidth]{LEDs}\hspace\*{\fill}

\caption{Showing the LEDs lighting up}

\label{fig:LEDs}

\end{figure}

This concluded the testing for the PCBs as did it not only show the Relays and raspberry Pi working well together, but it also illustrated that it would be possible to set all the cells to specific voltage bias for enhanced degradation, followed by the ability to cycle through each one to conduct measurements.

\section{Leak Testing}

This section will discuss the leak testing of the chamber. There were two leak tests which were planned: a simple test of placing the chamber in water – to find any large leaks, and a further test which measured the resistance of calcium inside the chamber.

The water-based leak test was conducted by filling a sink full of water and placing the chamber inside. The first test revealed a hole which I didn’t notice around the electrical windows. A glue gun was used to seal the hole and a hairdryer used to fully dry out the inside.

Once this was completed the chamber was ready for the calcium test. This test is similar to the one mentioned in Klumbies paper on Encapsulations for Organic Devices \cite{RN78}. The testing procedure was measuring the resistance of calcium deposited on a glass substrate. If the box was leakproof, the calcium wouldn’t degrade as there would be no oxygen and water within the chamber, thereby keeping the resistance low (and the calcium plate shiny). However, if there was a leak, the resistance would increase until the point where the calcium would have oxidised to either CaO or Ca(OH)$\_2$, both of which are insulators.

The plan initially was to use a keithley 2400 to run the tests, this would be done by setting the current to a specific value, then continually measuring and recording the voltage. The setup can be seen below in Figure \ref{fig:calc\_test1}.

\begin{figure}[h!]

\hfill\includegraphics[width=0.7\linewidth]{calc\_test1}\hspace\*{\fill}

\caption{Showing the desired set up for the calcium plate test.}

\label{fig:calc\_test1}

\end{figure}

The equations \ref{eq:resistivity} and \ref{eq:voltage} were used to calculate the source current given the minimum voltage which I wanted the keithley 2400 to measure was 0.5 V.

\begin{equation}\label{eq:resistivity}

R = \frac{\rho L}{A}

\end{equation}

\\

\begin{equation}\label{eq:voltage}

V\_m = I\_s~R\

\end{equation}

Where:

\begin{tabbing}

\phantom{$ P\_{diff}\ $}\= \kill

$R$ \>= Resistance of the sample\\

$\rho$ \>= Resistivity of Calcium\\

$A$ \>= Cross sectional area of the sample ($w~t$)\\

$L$ \>= Length of the sample\\

$V\_m$ \>= Voltage measured\\

$I\_s$ \>= Source current\\

\end{tabbing}

The value of the resistivity of Calcium ranged from 4 – 9 $\*10^{-8}$ $\Omega$ m \cite[p.~42]{RN78} (with the lower bound chosen for these calculations), the thickness of the calcium sample was 150 nm, the length and width of the calcium sample was 30 mm. This resulted in a I$\_s$ of 1.875 Amps and a minimum power dissipated of 0.93 W.

Once the measurement procedure was decided assembly of the chamber within the glove box occurred. During my first attempt, I realised that the space between the inside edge of the chamber and the substrate holder was very small, thereby preventing me from holding the substrate holder still while attempting to place the calcium plate upon it. This resulted in 2 calcium plate breakages and a redesign of some of the internal components within the chamber.

To enable easy attachment of the substrate to the substate holder, the decision was taken to use plugs, in this case 3 pairs 9 pin D-sub plugs and sockets. This allowed the substrate holder to be completely removed from the chamber during assembly which helped me hold the substrate holder still while I placed the substrate and lid onto it. The result of which can be seen in Figure \ref{fig:D\_sub} below.

\begin{figure}[h!]

\hfill\includegraphics[width=0.7\linewidth]{D\_sub}\hspace\*{\fill}

\caption{Showing the D-sub plugs connected to the wires exiting the chamber}

\label{fig:D\_sub}

\end{figure}

Once this was completed, I attempted to assemble the chamber again. However, there was a problem which prevented the assembly from working as planned. The substrate holder was deformed slightly from the hair dryer and heating required to dry out the porous ABS material. This meant that the substrate no longer sat well inside the substrate holder creating poor electrical connections that would work for a short period of time, but with a tiny bit of disturbance would sever and require a reassembly of the chamber.

To mitigate this, I attempted to order a part made out of PEEK from the engineering department, however, the part never arrived. Therefore, I decided to use crocodile clips clipped onto the calcium substrate, to connect it to the wires within the chamber. This worked well with the assembly shown clearly in Figure \ref{fig:calc\_test\_assembly}.

\begin{figure}[h!]

\hfill\includegraphics[width=0.7\linewidth]{ calc\_test\_assembly}\hspace\*{\fill}

\caption{Showing the calcium test set up using crocodile clips.}

\label{fig:calc\_test\_assembly}

\end{figure}

From here I measured the resistance of the calcium plate using a multimeter within the chamber. I measured the resistance for 15 minutes and there was no change in that time, with the calcium plate having a resistance of 13 $\Omega$. The Figure \ref{fig:glovebox\_c\_test} shows the set up with the resistance showing on the multimeter.

\begin{figure}[h!]

\hfill\includegraphics[width=0.7\linewidth]{glovebox\_c\_test}\hspace\*{\fill}

\caption{Showing the cacium test running, with the first measurement within the chamber.}

\label{fig:glovebox\_c\_test}

\end{figure}

After removing the chamber from the glovebox, the resistance started increasing. This was seen on the multimeter, and after 3 hours of running the test, the resistance reached a immeasurable value. This indicated there was a leak in this initial test. To try and find the source of the leak I watched the chamber while it was submerged in water with dissolved washing up liquid\footnote{The washing up liquid reduces the surface tension of the water making bubbles form more easily}. This showed a clear but small leak which were creating bubbles which can be seen rising in Figure \ref{fig:bubbles}.

\begin{figure}[h!]

\begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{bubbles1}

\end{subfigure} \begin{subfigure}{0.5\textwidth}

\includegraphics[width=0.9\linewidth]{bubbles2}

\end{subfigure}

\label{fig:bubbles}

\end{figure}

Once these leaks were filled, a further water test\footnote{Again with washing up liquid} was conducted with no leaks found. Therefore, I conducted another calcium plate test, with the same setup shown previously. However, when the plate was placed within the chamber, while it was in the glovebox, it started to degrade (the measured resistance increasing) to the point where it fully degraded while within the glovebox in the space of 3 hours. Clearly, this is not because of outside air leaking into the chamber, but the most likely reason is that the chamber itself was not fully dried out after using the hairdryer. This means that even with the chamber kept in a vacuum overnight, the water within the chamber did not evaporate and leave, meaning that it was able to degrade the calcium resulting in a failed test before it even started.

There were no more tests conducted due to a lack of lab time available, meaning that the only leak tests conducted that were passed were water-based tests. This, however, is not a failure; the lack of bubbles indicate that if there is a leak, it is very small, meaning that if the user holds the chamber at a slight overpressure (using MFCs), outside air will not be able to enter the chamber allowing the degradation tests to go ahead as planned.\footnote{The reason behind only two calcium leak tests is due to lab regulation surrounding COVID-19. A user could only access the AFMD physics labs for half of a week, meaning that if an experiment takes 2 days to set up (such as leaving the chamber unsealed, in a vacuum overnight, one must wait another week to conduct another one.}

The leak tests were conducted late on in the year, meaning that it was not possible to conduct an overall test of the chamber using a working solar cell. This is something that will be spoken about further in the conclusion.

\chapter{Conclusion}

In conclusion, it is clear that the specification of the set-up has not been fully achieved. This chapter will lay out some of the reasons why this happened, along with recommendations for those who need a set-up like this.

The points on the specification that were not achieved were: