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

\chapter{Introduction}

Introductory section describing the project.

\section{Literature Review}

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 with a device that created different conditions to degrade organic photovoltaic cells.

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. However, the current problems with organic solar cells boil down to long term stability, alongside low power outupts \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 is able to simulate a lifetime (10-20 years) of outdoor degradation in a matter of months.

To ensure that the conditions were analogous to a lifetime of degradation, research was conducted looking at failure mechanisms of organic photovoltaics (OPVs) as well as industry standards for testing OPVs. 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 system are: diffusion of water into the cell, diffusion of Oxygen into the cell operation under high temperatures. Other degradation mechanisms will also play a part such as photochemical degradation by UV light and oxygen \cite[p.~106]{RN57}. These degradation mechanisms and more are illustrated below in Figure \ref{fig: }

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 was expected that something would have been manufactured for the testing of any solar cell. 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{Showing the Test chamber used by Jaffery et al.\cite{RN59}}

\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 specifies, 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 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.

Despite these flaws, 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 I desired to emulate, 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 I incorporate into my device.

I also noted some of the drawbacks which I determined will not plague my design. ….

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, therefore I decided 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 °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 Reece 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 and Jsc 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.

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, this is not possible due to the observed characteristics of OPVs. Degradation can be broken down into three distinct phases, “an initial period of steep degradation that slows down with time, a period of relatively constant degradation that lasts for most of the solar cell’s usable lifetime, and rapid and complete degradation that results in device failure” \cite[P.~1]{RN60}. This non-linear nature of degradation can clearly be seen in Figure \ref{fig:HT10\_fig1} \cite[P.~3]{RN60}.

\begin{figure}[h]

\hfill\includegraphics[width=0.85\linewidth]{HT10\_Fig1}\hspace\*{\fill}

\caption{Illustrating the non linear nature of OPV degradation \cite{RN60}}

\label{fig:HT10\_fig1}

\end{figure}

From Figure \ref{fig:HT10\_fig1} it is clear to see that the 2 regions that need short interval measurement times are Burn-in and Failure. These can be categorised as the time to reach 80 \% and 50\% of initial performance \cite[P.~4]{RN60}. Thereby the most accommodating testing regime, that won't create unnecessary excess data-points would be a collecting data at small intervals during Burn-in and Failure, while having longer interval times during the long-term testing regime. This will be discussed further during the x chapter.

Write closing remars for the intro…

\chapter{Chamber Design}

The design of the container and associated electronics was a process that encompassed several months. 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 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 to outside air.}

\item{The container must allow electrical connections from outside to connect to the substrate for measurements.}

\item{The container must enable the substrate to be heated to a given temperature.}

\item{The container must have a window allowing light to be shone into the box.}

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

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

\end{enumerate}

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.

Along with the specification, some further goals were drawn up to provide aims that would provide important functionality but were not essential for the solar cell. These are shown below:

\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}

These 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}), where it became possible to see some of the flaws that were hidden by the virtual design. The first thing was that this design was very small, making it difficult for use within the glovebox. This was a problem as all assembly needed to occur within a glovebox to ensure there would be no unwanted oxygen or water residue able to degrade the cell. Adding to this, it seemed that there wouldn’t be enough room to wire the components in the container, this would cause significant problems as the modularity of the design would be compromised. This would go against one of the tenants of the project (modularity) which would be a problem when thinking of using this set up on different substrate layouts.

\begin{figure}[h]

\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}

These problems caused a redesign of the container, resulting in the model shown in figure (n). 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}.

\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}

The substrate holder was also modified with the intention of being unique to this particular substrate layout resulting in a more detailed design. The substrate layout 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 substrate layout provided by Dr. Christophoro.}

\label{fig:sub\_layout}

\end{figure}

As shown in Figure \ref{fig:sub\_layout} the substrate has x contact points and y cells, 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, 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=0.3\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 substrate layout it is clear that there are 6 solar cells to contact, each with 2 pins, meaning a total of 12 wires will be connected to the cell itself. This means a PCB needs to be manufactured which is able to connect 12 wires to the cells. From there each cell needs to be measured and 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}

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}[h!]

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

&Raspberry Pi & Arduino \\

\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).

Other changes included ensuring that the tracks were routed with large enough profiles to be printed on the departmental printers, as well as modifying the PCB so that it only had two working layers. These modifications in the schematic can be seen in appendix 2.

For the PCB named Solar Mux- appendix 2, 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 4.

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}

\section {Code Planning}

Another integral component to the container is the control electronics which are created to run it. After the Raspberry Pi was chosen 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 this I ran multiple LEDs blinking in parallel 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 enable easy 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 trying to solve this problem, 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 entire specification, 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}.

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 the Appendix, Figure \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, the work in this chapter was hampered by the impact of the second national lockdown in January 2021, with the details of the mitigations strategies I took outlined in this chapter as well as any forced changes I had to make.

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{Chamber Manufacturing}

This section will discuss the manufacturing of the container itself, including any components that are mechanically attached to it. From the designs shown in the previous chapter, 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 the appendix as Figure \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}

Once the outer shell had been manufactured, parts needed to be fitted to it. 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 and push fit valves shown in Figure \ref{fig: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}

The other component which needed manufacturing was 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. Unfortunately, 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. Unfortunately, this part was never manufactured as the departmental 3D printing lead Peter Walters informed me that the person in charge of printing in PEEK was not responding, costing over 4 weeks in development time. This meant that this part could no longer be manufactured and caused significant impact on the outcome of the project, which will be discussed further in the conclusion.

\section{PCB Manufacturing}

This section will talk about the manufacturing of the PCB and associated electronics of the chamber. As mentioned in the previous chapter, the PCB was designed to be manufactured in the Engineering Department Electronics Workshop. This meant that there were some limitations in manufacturing such as: the fact that the printer was unable to create plated through holes, thereby 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. 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}

The PCB in Figure\ref{fig:base} does have significant tidiness issues. These can be explained by the soldering iron I was using during the first part of the soldering process. Unfortunately, my first soldering iron did not reach high enough temperatures for easy, smooth soldering. However, once I changed soldering iron (for which the PCB named Solar Multiplexer was created with) the quality and ease significantly increased.

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}

The code development for the system was something that spanned the entire project. This is due to the slight changes in scope, some of which mentioned in the previous chapter. However, the core of the program was unchanging allowing a good base for the measurement system to be built on.

As mentioned in the previous chapter, the code was to be designed using Object Oriented Programming with the absence of a GUI to control the system. The lack of GUI does add slightly more complexity to the user, however, does not create a \emph{unusable} system. The main issue with not having a GUI is the fact that the user cannot easily input the testing conditions into the program. Instead, they have to delve into the code to \emph{find} the exact point to input it.

The method used to combat this 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 key aspect of the code is to control the environment inside the temperature while recording the Voc and Jsc values of the cell. For accurate measurements, the AFMD group requested a Keithley 2400 was used to measure the solar cells. 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.

Unfortunately, this aspect of the code created some unforeseen issues which were cantered on the Raspberry Pi communicating with the Keithley 2400. Due to the lack of access to Keithley’s until late into the Easter Vacation (due to COVID rules), this problem was not noticed untill first week of Trinity term. The short term solution was to use a multi-meter for the leak tests, following that

From this point on, the objective was making the code as simple to use and modify as possible. This is because one of the objectives was to ensure simple modification of the set-up. A user should easily be able to understand the code which was created thereby allowing them to create modifications to the script to suit their needs.

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.

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

Conclusion of manufacturing chapter

\chapter{Testing}

This chapter will be discussing and explaining the different testing procedures that the chamber, electronics and code were subjected to. The testing of the container is integral to the project as it provides a mechanism to demonstrate the working capabilities as well as determine any failure modes.

The tests conducted range from heat and flow simulations to leak testing. The tests conducted were designed to be replicable worldwide, 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{Simulations}

In this section, I will discuss the different simulations conducted on the chamber. 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=0.7\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. This showed a clear but small leak which were creating bubbles which can be seen rising in Figures \ref{fig:bubbles1} and \ref{fig:bubbles2}.

\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}

\section{Overall test}

Talk about short time test with a solar cell

\chapter{Conclusion}