Chapter 3

DESIGN AND CONTRUCTION OF A MIT CRUD LOOP

3.1 Experimental Overview

~~There are three main experimental objectives. The first objective is to characterize CRUD through using different image analysis including fractal box counting. The second objective is to evaluate the performance of the various types of surface chemistry modification coatings. The third objective is to assess the performance of hydrophobic and biphilic surfaces in mitigating or preventing CRUD.~~

~~Experimental data are required to complete these experimental~~. The most accurate way to obtain data is to acquire actual CRUD samples from a reactor. Obtaining the samples directly from a reactor, however, is impractical for many reasons. CRUD from a reactor is extremely hard to extract given its hazardous environment. Not only that, but it will also be very hard to handle as CRUD will accumulate a dangerous amount of radioactive materials. ~~For the second and third experimental objectives~~, which involve cladding coating tests, it is even more impractical since a reactor remains operation for around 18 months, which means it will take 18 months to obtain a sample set. Testing cladding coating in the reactor without proper experimental evaluation of what the coatings will do in the reactor also raises concerns regarding safety. Other than that, CRUD growth in a reactor is unpredictable making it even more impractical to attempt any CRUD experiment with a reactor.

It is unlikely that radioactivity has significant effects on CRUD growth and characteristic. Therefore, if an experimental loop can be built to simulate most of the conditions within the reactor except its radiation condition, it should still be able to produce CRUD formations with similar characteristics in the reactor. Omitting the radiation conditions allow us to ~~handle~~ the loop more easily. The MIT CRUD loop will allow CRUD growth experiment to be carried out at experimental thermal hydraulic and chemical conditions similar to that of pressurized water reactor (PWR). Samples obtained will be easy to handle and should imitate reactor~~'s~~ CRUD quite well.

Additionally, the construction of CRUD loop will open up for our group, as well as others involved, many new experimental capabilities/possibilities, since it will be the first loop at Nuclear Science and Engineering (NSE) Department at MIT that is built to simulate PWR condition. Professor Buongiorno planned an experiment that will study contact angle of surfaces at the high-pressure and high-temperature condition, something which was never done before experimentally since the hurdle of building a PWR condition loop is quite high. There are many experimental loops in NSE department. However, none of these loops would work for us for many reasons such as having too low temperature and pressure or low flow rate.

3.2 Introduction

The MIT CRUD loop is built to simulate the conditions inside a PWR including temperature, pressure, flow, and coolant chemistry. It works by driving flow through the autoclave, where PWR conditions that induce CRUD formation will be simulated. Autoclave contains heating rod which contains electric-resistance heater inside to simulate the actual PWR's fuel rod. This introduction to design and construction of the CRUD loop has a goal of giving the reader a good idea of how the system works and why without delving into any exhaustive technical details or actual steps needed to operate the loop.

3.2.1 Overview of the CRUD loop

CRUD loop is made up of two main systems, the circulation loop (or the main loop) and the auxiliary loop 3-1. The circulation loop is where the autoclave is situated and where the experiment is carried out. ~~The circulation loop is~~ the part that will be pressurized and heated to the PWR condition. Water will also circulate the loop to imitate the hydraulic condition of PWR in the autoclave. The auxiliary loop is where the water storage tank is situated. It is used mainly for chemistry control. The auxiliary loop is where the circulation loop obtains its water from and where it retrieves its water input supply. The loop will be at room temperature slightly pressurized with argon.

3.2.2 Main Loop or Circulation Loop

The circulation loop or the main loop's function is to simulate the thermal hydraulic condition of the PWR. To simulate thermal hydraulic conditions, pressure, temperature, and flow will be carefully controlled. By regulating these three factors, the PWR condition within the autoclave flow channel can be achieved, and the CRUD can be grown on the heating rod in the autoclave. The high-pressure condition in the main loop is maintained using the pressurizing pump. The high

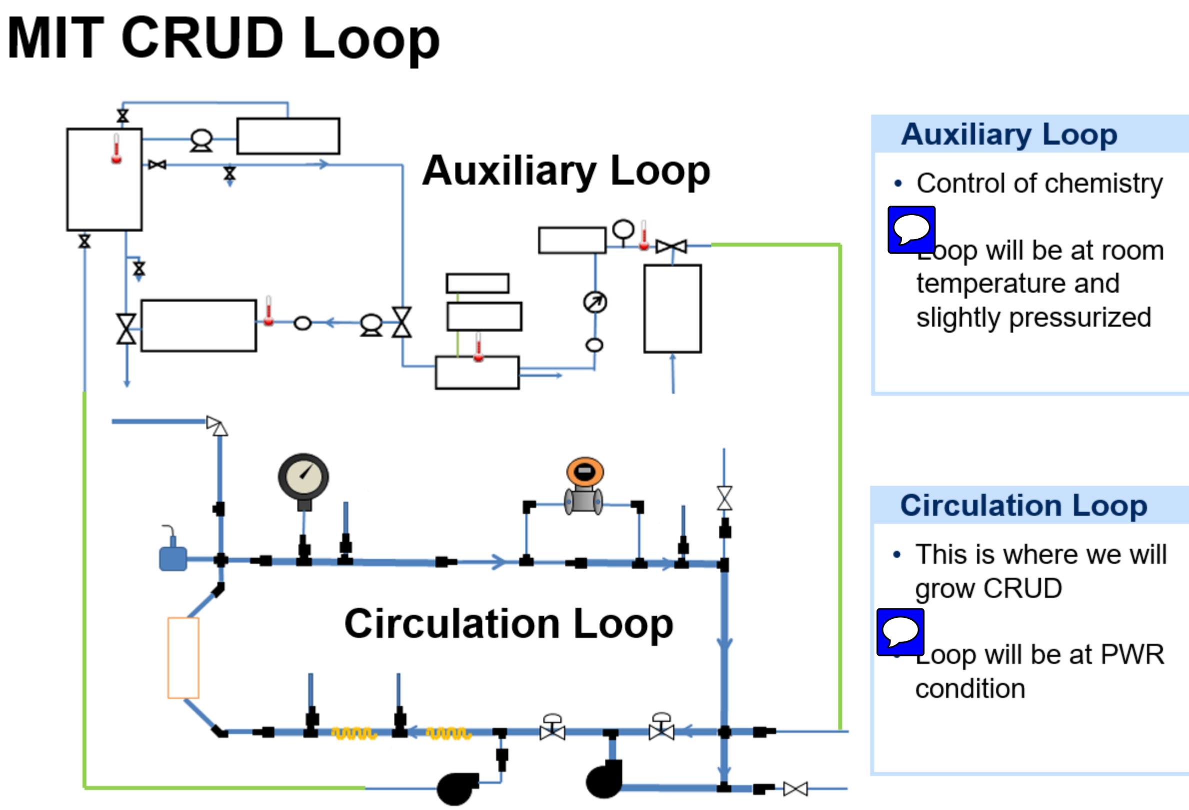

Figure 3-1: Diagram demonstrating the two most important parts of CRUD loop, the auxiliary loop and circulation loop. The diagram shows how the loops how both loops are connected. The auxiliary loop provides water to the circulation loop at the input of the pressurizing pump. The circulation loop outputs the water to be cooled down at sample cooler. After that, the pressure is regulated at back-pressure regulator in the auxiliary loop.

temperature is maintained using the autoclave heating rod as well as the heating tapes around the loop. The flow is maintained with the circulation pump.

The water in the circulation loop will be kept at a high temperature at all time. The heat loss ~~comes from the heat loss~~ through insulation and the pressurizing pump cold water input. Having ~~a loop similar to ours where~~ water circulate~~s in a circle~~ without lowering the water flow temperature has an advantage in having less heat loss. The power usage to get the temperature to 320 °C is quite ~~huge~~, making it one of our limiting factors in building the loop. The downside of this design is that the corrosion products coming from loop's tubing will not get removed easily, and can, therefore, affect the experimental results. However, since our goals, including the testing of coatings and the fractal analysis of CRUD using scanning electron microscope images, do not require a very precise chemistry, it was an attractive design choice. The decision to go with this design made it possible to build the CRUD loop within our budget constraints.

3.2.2.1 Pressure Control

The pressure control is accomplished by the pressurizing pump and the back pressure regulator. The pressurizing pump is a positive-displacement pump that will keep pumping in water at a defined flow rate. The pressure is kept under control by the back-pressure regulator. The back-pressure regulator will release the pressure pumped up by the pressurizing pump just enough to maintain the pressure. The pressure is monitored by the pressure transmitter connected to the autoclave, and additionally from the pressure gauge in the main loop and the pressure gauge on auxiliary loop. The pressure switch helps shut down the electrical system in the case of emergency pressure hike. The pressure relief valves will release the pressure, pushing the steam to the top of the building when there is an emergency pressure increase. Additionally, the pressurizing pump also has its safety relief valve for the pressure emergency handling in the case that all other prevention mechanisms failed.

3.2.2.2 Temperature control

For the temperature control, two separate systems are being used to maintain autoclave temperature. First, the autoclave heating system is the system attached to the autoclave itself. Second, the loop heating system operates the heaters that are placed throughout the loop. The autoclave heating system includes the heating rod and its internal thermocouples and the controller box. Heating rod is used to heat up water flowing in the main loop system, as well as provide heat flux for sub-cooled boiling. The element temperature or the centerline temperature of the heating rod is measured by the thermocouple embedded within the heating rod. This element temperature is displayed on the controller box's left most panel. The element temperature is the temperature that is used to regulate heat supplied to the heating rod. As the heat density coming from the autoclave heating rod is a lot higher than the usual heating rod, it takes a little time before the heating rod becomes too hot and gets destroyed. Therefore, trying to achieve the control using the liquid temperature will give the temperature feedback that is too slow to react to rapid temperature changes. Because the temperature control in the heating rod is very delicate, on-off control of heater will also not work as 100% power in many cases can quickly burn out the heater. The control box help achieves this fine temperature control using the transformer to control the input voltage. Control box's element temperature panel (left most panel) can be utilized to adjust the temperature setup. The proportional-integral-derivative setup allows control box to stabilize the temperature at any given point. In the autoclave heating system, the liquid temperature is measured with the thermocouple situated on the top of the autoclave, right where liquid leaves the autoclave. The liquid temperature is displayed on the middle panel of the autoclave's control box. This liquid temperature panel is used for monitoring only and does not have anything to do with the control of temperature ~~anywhere~~. The last panel of the autoclave control box is used to monitor the pressure within the autoclave, in bars. Again, this panel does not directly control pressure within the system.

The loop heating system controls the heating tape throughout the loop's tubing. Because the autoclave heater cannot provide enough power to be able to heat the whole loop beyond around 230 °C, additional heating is needed. This extra heating is where the loop heating system comes in. Although ~~the picture~~ 3-2 below shows the pre-heaters or the heating tapes only at a few spot, in the real CRUD loop, the heating tape was installed at almost every possible spot to maximize the heat input. As the heating tapes have low energy density, it will not fail even if we ~~put it to~~ full power without any significant heat sink. Therefore, temperature control of these heating tapes does not have to be as delicate. As a result, the liquid temperature is chosen to control these heating tapes. Additionally, the liquid temperature is ultimately the temperature that we want to control in the loop system. Unlike the autoclave where heating rod also plays a major role in defining heat flux and sub-cooled boiling, the temperatures of heating tapes are not essential for the experiment. Controlling the heaters with the liquid temperature also allows us to control all the loop heaters through just one point without installing the thermocouples and the controller everywhere in the loop. This simplification adds huge benefits regarding debugging the loop. Although there are four thermocouples throughout the loop used to monitor temperature, only the exterior thermocouple situated at the autoclave output is relevant to loop heater control. The autoclave output exterior thermocouple is a dual junction thermocouple which means that it can output temperature to two thermocouple lines. The first thermocouple wire goes to the computer for displaying on the computer screen. The second thermocouple wire goes to the heating tape proportional-integral-derivative (PID) controller. This single controller controls the heating tape throughout the loop. Unlike autoclave heating rod which has the luxury of a large transformer to control power input, this single controller only turns heating tape power on/off. When this controller clicks on, the all the loop's heating tape will heat up. This control method is very crude, but works well, and has huge benefits when it comes to simplifying the loop. PID control allows this controller to stabilize the temperature in an acceptable way even with no variable heating. The autoclave heating system and the loop heating system combine to make the CRUD loop capable of heating itself to 320 Celsius or more and providing enough heat flux for growing CRUD on the testing samples on the heating rod in the autoclave.

3.2.2.3 Flow control

The flow control is achieved by using the combination of the circulation pump, the differential pressure transmitter, and the control valve. The circulation pump is a centrifugal pump that drives flow through the loop. Since circulation pump with the variable speed function add-on is very expensive, we use the control valve to regulate the flow instead. Control valves regulate the flow by constricting the flow path. There are two control valves to prevent damage to the circulation pump in the case of a control valve failure, or an accidental control valve shut-off. The main control valve in series with circulation pump is the one regulating the flow. The control valve parallel to the circulation pump is left slightly open to let the water through in case the main control valve shut-off. The differential pressure transmitter is the chosen method for measuring the flow through the loop. At this temperature, pressure and flow rate, there is no flow meter on the market except those that are custom made. Those custom made flow meters are extremely expensive and do not make much sense for this project. Therefore, a differential pressure transmitter is used as a way to circumvent this problem. By knowing the difference in pressure between the flow area of different sizes, Bernoulli's equation can be applied to calculate the flow rate from a given differential pressure.

3.2.3 Auxiliary loop

The main loop needs a support of auxiliary loop 3-3 to store its input/output water and control the chemistry. The water tank in the auxiliary loop is where water that will be injected into the main loop is stored. On startup, the water tank is used to mix the boric acid, the lithium hydroxide, and the nickel oxide CRUD particles together. The process starts by first cleaning up the water to make sure that the chemicals added~~,~~ are the only chemicals that exist in the water. Cleaning up the water first also prevent non-CRUD fouling of the CRUD loop. The important part for cleaning is the ion exchange resin. During cleaning, input tap water is circulated using a small pump, through the ion-exchange resin container, to exchange out any ions that exist within the water. As ions in water get filtered out, the water's conductivity as measured by the conductivity sensor will go down. During cleaning run, valves can be configured so that the water is drawn from the tank through measurement equipment to monitor the conductivity and dissolved oxygen in the water. This ion cleaning process is slow and can take as long as two days to complete depending on the content of tap water and how new the ion-exchange column is. After the water in the tank has been rinsed of

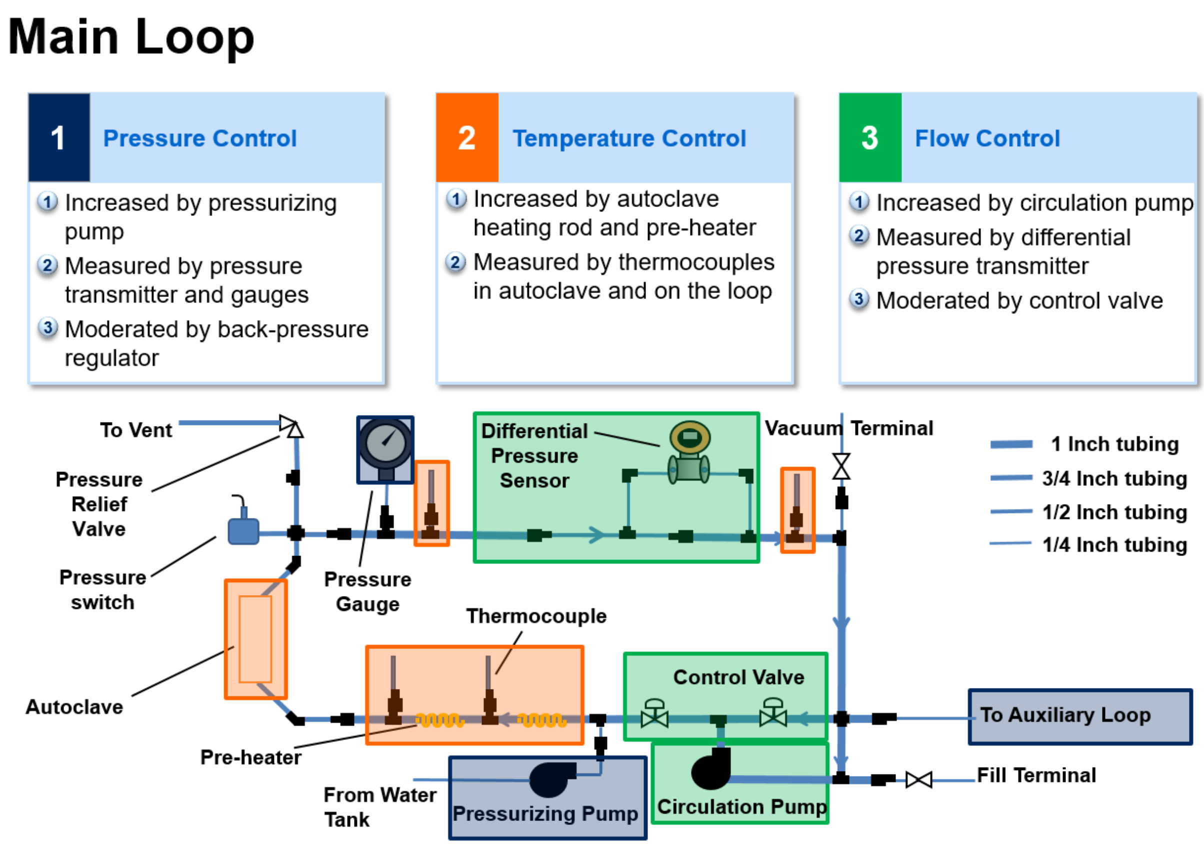

Figure 3-2: This diagram shows different parts of main loop or circulation loop and their functionalities.

ions, the next process is to get rid of the dissolved oxygen in the water. This process is done through bubbling argon through the water. After both dissolved oxygen and conductivity is low enough, the chemical can be added to water tank via injection port on the top of the water tank. After the chemical is added, the valves can switch flow to bypass the ion-exchange resin container, turning that loop into a mixing loop instead. The mixing will involve taking the water from the bottom of the tank and releasing it at the top. When the chemical has been well-mixed as determined by the conductivity stabilizing, the water tank will be ready for input into the loop. The main loop is filled through its fill port which connects to the water tank.

When the loop is filled, and the fill port is closed, the pressurization process can begin. The part that drives this process is the pressurizing pump which is the only water input during operation. It draws water directly from the water tank to pressurize the loop. The output for the main loop is connected to auxiliary loop via sample cooler which cools down the water to near room temperature. Cooled down water pass through the pressure gauge and pressure transmitter to measure its pressure. The back-pressure regulator acts as a pressure boundary between the high-pressure system and the low-pressure system of the CRUD loop. It releases flow just enough to maintain the pressure it was set at. The water then gets directed through measurement equipment to ensure that water has desired chemical properties. After that, the water is released into the water tank completing the water cycle during operation.

3.3 CRUD Loop Preliminary Design

3.3.1 Design Considerations

The ~~designing~~ the MIT CRUD loop involves finding a balancing point ~~in~~ between many trade-offs. Many design considerations go into designing the extreme temperature, pressure and flow loop with limited resources. These design considerations play major roles in choosing CRUD Loop design parameters and in the decision of which equipment to purchase for the loop. Considerations that play~~s~~ a major role in our design and purchase decision include~~s~~ the following:

Data Accuracy: The data accuracy is the most important aspect of CRUD loop's design consideration. Ideally, the loop should be able to produce good quality useful data in a reasonable amount of time. One good way to get the CRUD data representative of PWR CRUD is to imitate the condition within the PWR in the experimental setup. The MIT CRUD loop is designed in such way that makes it capable of imitating the PWR thermal hydraulic and chemical conditions as closely as possible. The heating rod within the autoclave where samples are placed imitates the fuel rod of the reactor. The autoclave test section is designed in such way that Reynold's number and the mass flux imitate the condition within the PWR. All the supporting mechanism in both main loop

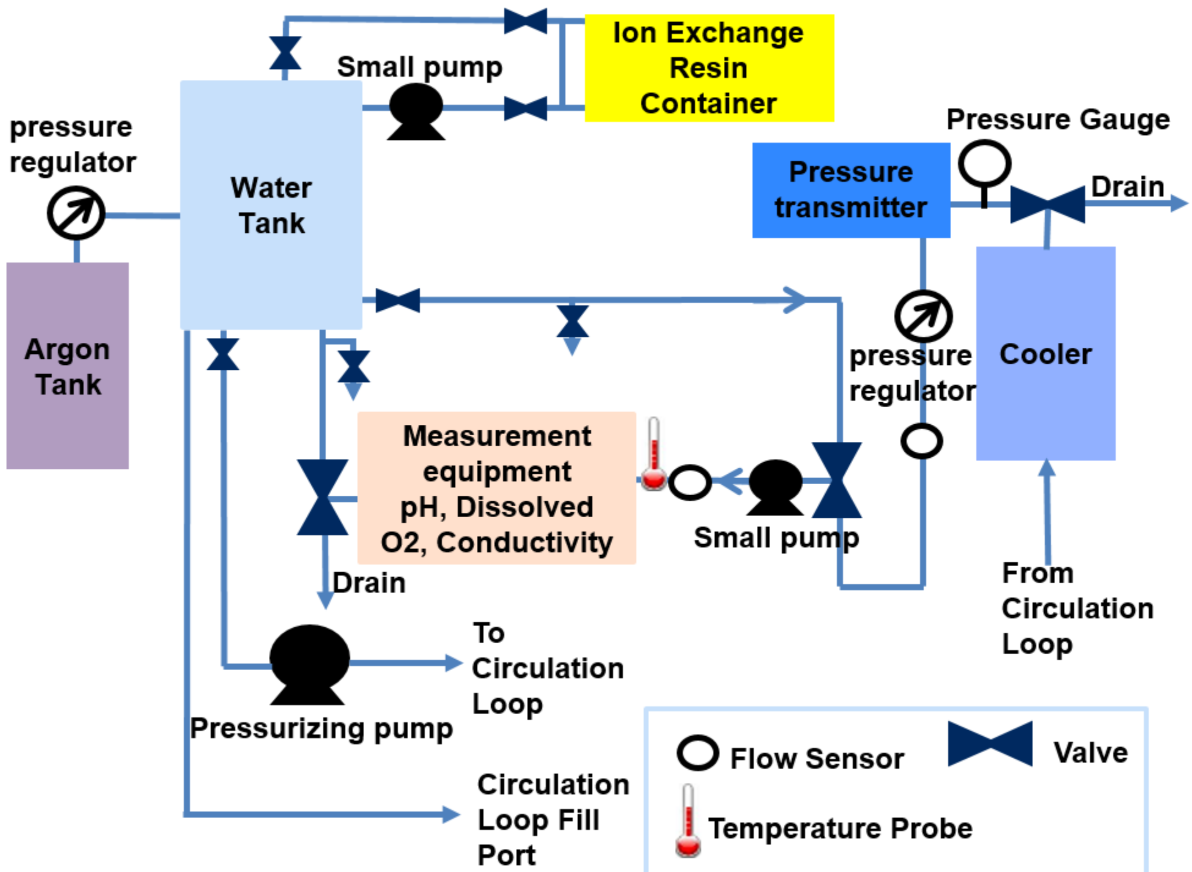

Figure 3-3: This diagram shows different parts of the auxiliary loop and their functionalities.

and auxiliary loop work together to help CRUD grown in the loop exhibit characteristics that is as close to CRUD grown in actual PWR as possible.

One might ask if going to the PWR condition is necessary, since getting to PWR condition is extremely hard to achieve compare to the normal atmospheric pressure pool boiling facility and accurate imitation of PWR condition might not be worth the hassle. However, both the low-pressure aspect and the no flow aspect of the simpler facility has many disadvantages when it comes to growing CRUD. A pool boiling facility several critical disadvantages. Firstly, the real world CRUD formation is a balance between the CRUD layer forming and the flow washing off the CRUD formation. With a pool boiling facility, CRUD formation experiment lacks the flow washing off CRUD component. The lack of flow will lead to more CRUD growth. In our experiment specifically, the materials that would otherwise be CRUD resistant might not look that way in pool boiling experiment. Things are worse if the pool boiling facility has its heating plate on the bottom of the autoclave. In this case, CRUD particles may settle just like any other sediment system without any flow.

Having less pressure and temperature lead to results that will likely not represent the real reactor. Inaccurate pressure changes properties such as the contact angle shown later in chapter 4. Most of the relevant properties that could affect the CRUD growth changes with temperature, including properties such as the boiling bubble sizes, the surface tension of water, and the water viscosity, etc. Having both the high pressure and high temperature together was difficult to achieve. However, for the CRUD growth experiment, increasing one cannot be done without increasing another. The CRUD growth experiments need sub-cooled boiling to work. When the temperature is high, the pressure must also increase to prevent liquid from turning into vapor. When the pressure is high, the temperature will also need to increase, or there will be no boiling and no CRUD.

Cost: High temperature and pressure equipment are extremely expensive. To be able to build the whole loop with limited funding, many equipment must be recycled from another experiment when possible. Not only that, but some very expensive equipment are also replaced with another set of equipment that can function the same way, instead. For example, a flow meter that can properly measure flow rate at this extreme condition is only available as a custom made, and extremely expensive. It was replaced with a differential pressure transmitter and an orifice, which can also be used to obtain flow rate with some additional calculations using Bernoulli's equation. This saved cost by a factor of more than ten times compares to a custom piece. Another example of a cost saving initiative is the use control valves instead of a circulation pump's variable speed add-ons. A circulation pump with variable speed setup is extremely expensive, double the cost of a normal circulation pump. Since control valves cost a lot less than a circulation pump, using control valves, in this case, saved an enormous amount of costs. Reuse of equipment available is also a big part of cost-saving. It is fortunate the MIT has many used equipment that was discarded, not because it is broken, but because it is no longer needed for the experiment. Reusing equipment such as the dissolved oxygen sensor, the conductivity sensor, the pH sensor, the sample cooler, relays, and the temperature controller, etc., make the cost of acquiring that equipment virtually free.

Safety: The MIT CRUD loop will be running at PWR's conditions which are very dangerous. Therefore safety is of utmost importance. Multiple redundant safety features are present throughout the loop. For pressure safety, there are three pressure relief valves attached to tubings in the main loop, the autoclave itself and the pressurizing pump. Having three redundant pressure relief valves is one way to make sure that even if one of the relief valves failed to open, there would be others to compensate. Other than this, the pressure pump will shut off on its own without human intervention, once the pressure becomes too high. The pressure switch also shuts down the whole electrical system in the case of pressure emergency.

The polycarbonate sheet is used protect the people within the CRUD loop room in the case that high pressure sends some equipment flying off. Polycarbonate is the same material that is used for making bulletproof equipment in the security industry, so the person behind the glass can feel assured nothing will easily penetrate the polycarbonate sheet. Nevertheless, even with many redundant safety systems, it is still a dangerous experimental setup, and extreme cautions must be taken, when operating it.

Minimize downtime: During its lifetime, the loop had already been moved two times. During the initial construction phase of the loop, the final room that the loop was supposed to be placed was not ready yet. Therefore, CRUD loop had to be built with mobility in mind. For this purpose, the loop can separate itself into three major parts that can be moved separately. All three major parts are designed to fit through common door size of 6.5 feet by 3 feet. Two parts that made up the main loop are intended so that it can be easily lifted with a palette jack. Another reason why the main loop is designed to have three-dimensional structure is that it is a lot easier to lift a three-dimensional structure than a two-dimensional structure. With the three-dimensional structure, a ~~palette~~ jack can easily lift the loop without any tilting.

Mobility: During its life-time, the loop had already been moved two times. During the initial construction phase of the loop, the final room that the loop was suppose to be placed was not ready yet. To avoid having to completely reassemble the loop many times, CRUD loop had to be built with mobility in mind. For this purpose, the loop can separate itself into three major parts that can be move separately. All three major parts are designed to fit through common door size of 6.5 feet by 3 feet. Two parts made up the main loop are designed so that it can be easily lifted with pallette jack. This is another reason why the main loop was designed to have three dimensional structure. With three-dimensional structure, pallete jack can easily lift the loop with good balance.

Space: The whole loop, including the control computer desk, must fit in its designated room which has usable space of only around 17 feet by 12 feet. The loop must also be able to fit through the normal door which is around 6.5 feet by 3 feet. This is necessary for moving the loop. Disassembling the whole loop for moving is impractical since it entails a high risk of damaging equipment during disassembling and reassembling of the equipment. Disassembling and reassembling also take many man-hours. Heated part of the loop usually becomes so sealed with heat that it becomes extremely hard to disassemble. In multiple cases, parts were thrown away because tube itself was bent from too much force that goes into disassembling.

Simplicity: The first few iterations of CRUD loop before it was built had too much emphasis on cost and too little emphasis on simplicity which led to more probability of failure and more arduous task when debugging the loop. One example of this mistake is the usage of the accumulator in an attempt to match the flow rate of pH, dissolved oxygen, and conductivity measurement equipment. The old pressurizing pump has a flow rate that is too low, so the loop output flow rate is too low for measurement equipment to measure properly. The previous solution to this was to use an accumulator that accumulates output water, then from time to time, the computer will let some water from the accumulator through the measuring equipment to take the measurement. Although this system can save some cost of buying a new pressurizing pump, it has myriads of problems that delay experiments. In the end, the old pressurizing pump was replaced with a new one with higher flow rate, which functions very well, making the experiment a lot smoother. Learning from mistakes such as this, the design add-ons that was later added are designed to be as simple as possible.

Another decision worth noting regarding simplification of the loop is the decision to use only one liquid temperature for central control of all heaters in the loop. Before the decision, the initial plan was to have a separate temperature controller for each heating element. Each temperature controller will take input from the thermocouple embedded between the heating tape and the stainless steel tubing. The motivation back then was to make sure that the heater does not burn out. Fortunately, it was later found that because heating tapes have very low heat density, there is no need to worry about it burning out even if it was put to 100% power for a long time. Therefore the loop was simplified to having just one controller that takes the liquid temperature at the autoclave output as an input. This was a great decision as lots of time were spent maintaining the complex electrical system for loop heating. If there were one controller for each heater in the loop, the maintenance work for the loop heating system would have taken at least quadruple the time. Moreover, the loop's electrical circuit will become an even more complex and less understandable to outsiders.

Maintenance: Easy maintenance and debug of the loop is another vital design consideration. A significant amount of time was spent doing maintenance and figuring out what is wrong with the loop. Therefore designs must also make the loop maintenance easy. There are two main categories of ways that make the loop easy to maintain. First, the loop should be designed so that it is easy to access every equipment for maintenance. This accessibility is one of the reasons why the loop structure is built as a sturdy three-dimensional rigid structure as oppose to having just struts holding up the tubings. The three-dimensional structure allows operators to climb up the loop to get to any necessary parts without using a ladder, which is more dangerous as it can slip. Every maintenance procedure for CRUD loop does not require ladder except refilling the loop which is done only around once in two months.

3.3.2 CRUD Loop Design Parameters

CRUD Loop design parameters were chosen to have a condition that is as close to the reactor as possible. The table 3.1 summarizes the conditions in the MIT CRUD loop compared to those in a PWR.

	PWR	MIT CRUD Loop
Pressure	15.5 MPa [7]	15.5 MPa
Temperature	287-324°C [7]	320-335°C
Reynolds Number	5·10^5 [7]	1.62·10^5
Mass flux	3500kg/m2s [7]	1920kg/m2s
Fe3O4, NiO	13-116ppb [8]	0-500ppb
Fuel rod diameter	9.5mm [7]	13.5mm

Table 3.1: This table shows the MIT CRUD loop operating conditions, compared to a prototypical PWR.

As seen in the table above, the MIT CRUD Loop can easily reach and operate at the PWR pressure. The pressure has profound effects on boiling and how bubbles form. Therefore, the loop was designed to get to 15.5 MPa matching the PWR condition. For temperature, the MIT CRUD loop can run at a temperature higher than the hot section of PWR. The high temperature was chosen to speed up CRUD growth. Since CRUD is formed from boiling, more temperature and more boiling are expected to produce more CRUD growth. CRUD grows in PWR over a year and a half length. However, we do not have the time luxury of growing CRUD for a year. This significant time limitation drove the CRUD loop design parameters to support a higher CRUD growth rate. Reaching these high temperatures was extremely hard and introduced many unexpected problems, but nevertheless necessary to achieve the higher growth rate.

Reynold's number and mass flux are intertwined with each other. Since the experimental loop's liquid is fixed as water at the PWR condition. The only two parts left to adjust, that will affect Reynold's number and mass flux, are the flow velocity, as well as the flow area size and shape. The flow area shape and size is limited severely by the equipment prices. The more flow area leads to larger, thicker and more expensive autoclave. CRUD loop autoclave's window hole also act as an excellent stress concentrator. The finite element stress analysis shows that there is no way around it other than reinforcing the hole with thicker steel. Therefore, limiting the flow area to avoid the autoclave that is too thick is even more crucial for the CRUD loop with window.

3.3.3 Pressure Drop Calculations

The pressure drop calculations were done on excel spreadsheet using the Darcy-Weisbach frictional pressure drop equations. The following table 3.2 summarizes the result obtained from excel pressure drop calculation. The pressure drop of the loop as a whole is still lower than the pump specification with a significant margin. The remaining pressure drop will be incurred across the control valves, used to control the flow rate. This margin will allow for future upgrades without worrying about the pressure drop constraint.

Loop Section	Pressure Drop (kPa)
Autoclave	2.09
Inlet/Outlet Constriction	1.95
Straight Tube	3.94
Tube Elbows	3.99
Differential Pressure Sensor Orifice	3.20
Total Pressure Drop without Pump and Valve	15.2
Pump	67.6
Control Valve	52.4

Table 3.2: This table shows the pressure drop across each section of the MIT CRUD loop.

3.3.4 Heater Calculations

3.3.4.1 Heat transfer calculation

In the CRUD loop, it would be very hard to be able to measure the temperature exactly at the surface of the cladding. Having a thermocouple on the surface of cladding can obstruct the flow leading to changes in the result. Therefore it is best to obtain the temperature of the surface of cladding through calculations, given that we know the heating rod centerline temperature and the liquid temperature. The heat transfer calculations [61] are used to determine the difference between the temperature of the heating rod centerline temperature (T_{max}) to the temperature of the outer surface of the heating rod cladding (T_{co}). To accomplish this, we start with the heat conduction equation in a cylindrical geometry.

$$\frac{1}{r}\frac{d}{dr}(kr\frac{dT}{dr}) + q''' = 0 \tag{3.1}$$

Integrating this equation then dividing it by r we obtain.

$$k\frac{dT}{dr} + q'''\frac{r}{2} + \frac{C_1}{r} = 0 \tag{3.2}$$

By taking this equation, moving all terms with r in it to the other side, then integrating both sides, we get:

$$-\int_{T_{max}}^{T} k\,dT = \frac{q'''}{4}(r^2 - R_v^2) + C_1 ln(\frac{r}{R_v}) \tag{3.3}$$

This equation can then be used to derive the temperature difference between the heating rod centerline (T_{max}) and the inner surface of cladding (T_{ci}) , as well as the temperature difference through the cladding ($T_{ci} - T_{co}$). When combined, we can get the temperature difference between the heating rod centerline temperature and the temperature at the outer surface of the cladding.

$$T_{max} - T_{co} = (T_{max} - T_{ci}) + (T_{ci} - T_{co}) \tag{3.4}$$

To obtain the temperature difference from T_{max} to T_{ci}, we assume that the heat flux at the center of the heating rod is zero. This assumption can be converted to the following boundary condition:

$$q''|_{r=0} = -k\frac{dT}{dr}|_{r=0} = 0 \tag{3.5}$$

Applying this boundary condition to the heat conduction equation with $r = 0$, we get the following:

$$C_1 = 0 \tag{3.6}$$

Now plugging $C_1 = 0$ and $R_v = 0$ back into the equation (27) we get:

$$-\int_{T_{max}}^{T} k\,dT = \frac{q'''}{4}r^2 \tag{3.7}$$

At the inner surface of cladding, $r = R_{ci}$, the temperature is represented by the valueT_{ci}. Plugging these values into the equation (31), the following equation is obtained.

$$-\int_{T_{max}}^{T_{ci}} k\,dT = \frac{q'''}{4}R_{ci}^2 \tag{3.8}$$

Converting this equation to linear heat rate using energy balance $q' = \pi R_{ci}^2 q'''$, we obtain:

$$-\int_{T_{max}}^{T_{ci}} k\,dT = \frac{q'}{4\pi} \tag{3.9}$$

With the assumption that k is constant at k_{heater} , we can derive the following equation:

$$T_{max} - T_{ci} = \frac{q'}{4\pi k_f} \quad (3.10)$$

In the similar way we can derive $(T_{ci} - T_{co})$. Starting with knowing that volumetric heat generation q''' should be 0, we simplify the equation (27) to the following:

$$-\int_{T_{max}}^{T} k\, dT = C_1 ln(\frac{r}{R_v}) \quad (3.11)$$

We also know that the following the heat flux equation must apply:

$$q''|_{r=R_{ci}} = -k\frac{dT}{dr}|_{r=R_{ci}} \quad (3.12)$$

And that the heat flux q'' is equal to $q'' = \frac{q'}{2\pi R_{ci}}$. By plugging this information into the equation (26), we yield:

$$-\frac{q'}{2\pi R_{ci}} + \frac{C_1}{R_{ci}} = 0 \quad (3.13)$$

$$C_1 = -\frac{q'}{2\pi} \quad (3.14)$$

With the assumption that k is a constant at k_{heater}, we can derive the following equation from equation (35):

$$T_{ci} - T_{co} = \frac{q'}{2\pi k_c} ln(\frac{R_{co}}{R_{ci}}) \quad (3.15)$$

Combining these derivation using equation (4) we get the following equation :

$T_{max} - T_{co} = \frac{q'}{2\pi}(\frac{1}{2k_{heater}} + \frac{1}{k_c} ln(\frac{R_{co}}{R_{ci}}))$

We use this equation to calculate the temperature of cladding outer surface from knowing the heating rod centerline temperature (element temperature). The input values to the equation is shown in the table 3.3. From using these values and the equation (39), we found the $T_{max} - T_{co}$ to be 95.1 ºC. Since the element temperature measurement which measures the centerline temperature outputs around 420 ºC depending on the heater rod used, the outer cladding temperature where CRUD grows should be around 325 ºC.

3.3.5 Autoclave Geometry Considerations

The design of the autoclave must allow experiment carried out in its test section to imitate the condition in PWR as closely as possible. At the same time, it must be within a reasonable cost range. The following section explains the considerations that went into designing different parts of

Parameter	Value
k_{heater}	15 W/m·K
$k_{cladding300C}$	18.8 W/m·K
q'	1700 W
R_{ci}	12.5 mm
R_{co}	18.0 mm
$T_{max} - T_{co}$	95.1 ºC

Table 3.3: This table summarizes different parameters that was used to calculate the temperature difference between the centerline temperature and the cladding outter temperature $T_{max} - T_{co}$. Thek_{heater} is the thermal conductivity of the nichrome heater. The $k_{cladding300C}$ is the thermal conductivity of stainless steel cladding and ring at 300 ºC. The q' is the linear heat rate of the autoclave heater rod. The R_{ci} and R_{co} are the cladding inner diameter and the cladding outer diameter that includes the sample ring. From these information, we found the $T_{max} - T_{co}$ valu of 95.1 ºC.

the autoclave.

Autoclave Windows One of the aspects that make this CRUD loop the first of its kind is that its autoclave has three two-centimeters diameter size window that can be used to observe CRUD growth in situ. These windows will open up a wide range of possibilities for using this autoclave, including using laser triangulation to observe CRUD thickness in situ, using Raman spectroscopy in situ to observe CRUD formation, and using laser techniques for high-pressure contact angle experiment planned by Professor Buongiorno's group.

Autoclave Inner Shape The autoclave inner shape is designed to be as thin as possible while maintaining a reasonable mass flux and Reynold's number. The heating rod in the middle of the annulus has 17.5 mm diameter while the real reactor's fuel rod have only 9.5 mm diameter. The reason that MIT CRUD loop's heating rod must be thicker is that electric resistant heating that it uses have lower heat density than the uranium pellet inside the commercial reactors. Therefore, to obtain a reasonable heat flux for CRUD generation, we ended up having to use a heater rod with larger volume and diameter. Nevertheless, the mass flux and the Reynold's number is comparable to that of the real PWR as shown in the CRUD loop's design parameter's section.

The inner test section is designed to be very small because it saves the amount of metal that must be used to contain the pressure. The finite element stress analyses determined that stress concentration are very high at the window holes. This issue is made worse by having a larger autoclave. Therefore, the autoclave inner shape is designed to be just large enough for proper flow conditions while small enough that the autoclave will no need too much reinforcement to combat the stress concentrations. The computational fluid dynamics models showed that the inner section of 20 mm is optimal for this case before the addition of sample rings. Therefore, this size was chosen as the autoclave inner diameter (test section outer diameter). Since the sample ring addition idea came later after the autoclave was already built, it was too late to change the design. Nevertheless, the actual experiments showed that CRUD growth was not significantly disturbed by the change in flow at the window.

The slanted inlet and outlet allow the flow to stabilize quicker in the autoclave, as there will be no sharp perpendicular turn in and out of the autoclave. Having the slanted inlet and outlet also lead to less flow friction. The slanted inlet and outlet also avoid the equipment and flanges on the top and bottom of the autoclave, making the autoclave easier to manufacture and maintain.

Autoclave Outer Shape The autoclave outer shape design was a direct result of stress analysis. The autoclave manufacturer decided the final autoclave outer shape, as shown in the figure 3-4, with the result of their final stress analysis investigations. The design has its window holes on a large cylindrical section because that is where the stress is most concentrated. This design also allows for easier manufacturing.

Adding Rings There are several reasons we want to add a ring structure on to fuel rod. First of all, the main reason that we added a ring structure is because of the limitation in the size of the e-beam or the sputtering chamber. To coat the CRUD-resistant materials on the whole heating rod would require an enormous sputtering chamber which is not readily available to us. Second, it allows us to replace only the ring part for each trial of the experiment. Replacing only small a sample ring as oppose to the full heating rod will also save significant consumable costs. The only downside to using the ring is that it might disturb the flow profile within in the autoclave. The computational fluid dynamics models were used to verify that this flow disturbance effect is acceptable for CRUD growth experiment. After that, observations from the real test shown that the window section's CRUD samples do not show any significant deviation from other CRUD samples.

3.3.6 Computational Fluid Dynamics (CFD) Model

Computational fluid dynamics (CFD) analysis was performed for the autoclave, specifically on the region with the sapphire viewing windows. The CFD was done to ensure that the flow conditions in the autoclave will represent the flow conditions in real reactors and to ensure that the windows will not disturb the flow patterns. The CFD of the system without sample ring shows little disturbance of flow on the surface of the heating rod by window section.

The difficulty came when a decision was made to use sample rings for various reasons. In the system with sample rings, the CFD results showed that the flow stabilizes very quickly after getting disturbed by the beginning section of the sample ring. The windowed flow section of the autoclave was shown to slightly change the flow profile in the system with sample rings. This flow profile disturbance led to the decisions to place four dispensible stainless steel rings without a surface modification coating where the window section is, on the September 22nd, 2015 trial. Fortunately,

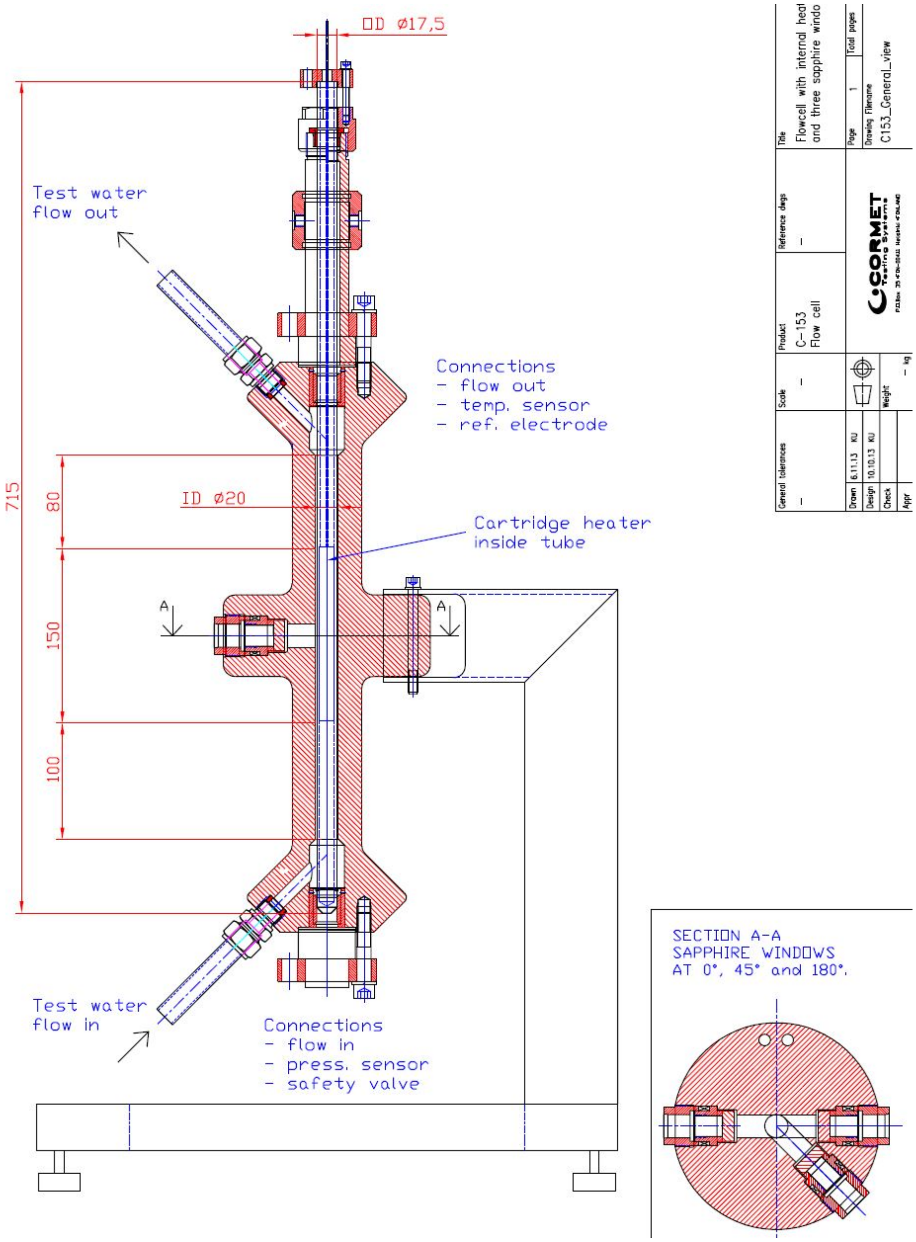

Figure 3-4: Final autoclave design as rendered by the autoclave manufacturer Cormet Oy.

the trial showed that there was no significant difference between CRUD grown near the window and those far away from the window. This fact confirms that window section is not an issue that will affect data accuracy.

3.3.7 Finite Element Stress Analysis

The goal of using an in-house finite-element stress analysis is not to build an exact autoclave design, but to gain more understanding of the type of equipment that is needed to carry out the task. Before working with a manufacturer, we must ensure that the kind of autoclave with sapphire windows, at PWR pressures and temperatures, is a possible task. The finite element stress analysis verified that the autoclave with the parameters we are looking for could be built within our lab constraints. It also narrows down the optimal design that fits the need of the study, before the detailed manufacturing tasks are relegated to Cormet Oy, the company that manufactured the CRUD loop's autoclave. The more detailed modeling and testing of stress for licensing were left to the autoclave manufacturer.

In a more specific detail, the finite-element stress analysis makes sure that the stress concentration caused by drilling the autoclave for windows is not too high. Other than this, the diamond-sapphire window was examined regarding how well it holds against the high PWR pressure. One important piece of information that was obtained from finite-element stress analysis was that stress concentration is very significant at the location of the window holes. A larger diameter autoclave previously designed with a window would need the autoclave that is extremely thick. The preliminary simulation showed that the stress concentration at the window hole is barely reduced when adding more thickness to the autoclave. Instead, the preliminary simulation shows that the stress concentration is reduced significantly faster when reducing the size of autoclave instead. Because of this, we arrived at a much thinner autoclave design with a flow section of only 2 cm in diameter.

3.4 CRUD Loop Parameter Controls

3.4.1 Pressure Control

The pressure control of the system is done using two main equipment, the pressurizing pump, and the back-pressure regulator. The pressurizing pump supplies the system with a small flow rate, but with very high force. The back-pressure regulator will let the water out of the system just enough to maintain a steady pressure in the system. In case the back-pressure regulator fails to release the pressure, multiple safety systems are put in place to avoid any danger. These safety systems include the main loop safety valve, the autoclave safety valve, the pressure switch, and the pressurizing pump's safety system.

The pressurizing pump can be turned on and off via the computer which controls the relay in the CRUD loop relay box. The back-pressure regulator can be adjusted manually. Cautions must be taken when adjusting the back-pressure regulator. Proper protective equipment is required if the adjustment is made during high pressure. The pressurizing pump can be adjusted for a different flow rate. Increasing the pressurizing pump flow rate can help the loop pressurize quicker and get the necessary flow rate for measurement equipment. The adjustment can be made by turning the big knob on the pressurizing pump that will increase or decrease the stroke length. Increasing the stroke length will increase the flow rate as the stroke rate will remain steady. Note that during the heating of the loop, it is advisable to decrease the flow rate to lessen the heat loss.

3.4.2 Temperature Control

As mentioned in the introduction, the CRUD loop uses two heating systems to control its temperature, the loop heating system and the autoclave heating system. Both systems are completely separate with its heaters and control boxes. The autoclave heating system provides the necessary heat flux to carry out the experiments while at the same time providing the loop with the significant heat source. The loop heaters' sole purpose is to heat up the coolant water enough to get to the PWR temperature conditions. These sections below will describe the specifics of each heating systems.

Loop Heating System The loop Heating System is the heating system that spans over the whole loop. This heating system consists of many heating tapes placed all around the loop. The main controller for this system is the Omega CN9000 temperature controller, which controls all of these heating tapes. The Omega CN9000 temperature controller takes only one input from the dual junction thermocouple situated on the top of the loop near the autoclave. This dual junction thermocouple measures the liquid temperature. The slower temperature feedback caused by using liquid temperature is acceptable for two reasons. First, even if the loop heaters remains fully on without any water flow, the heaters will still not burn out. Second, the temperature changes when the loop is at high temperature is very slow, and therefore a slow temperature feedback will suffice. See the section on how to heat the system for more information on how to operate the autoclave temperature controller.

Autoclave Heating System The autoclave heating system is the heating rod inside the autoclave where we grow CRUD. Autoclave's heating rod is controlled using West 6100 temperature controller on the autoclave controller box provided by Cormet Oy, the company that manufactured the autoclave. The temperature control uses the heating element temperature as feedback as oppose to the liquid temperature. See the section on how to heat the system for more information on how to operate the autoclave temperature controller.

3.4.3 Flow Control

In CRUD loop, liquid flow through the main loop is driven by Centrifugal pump. The centrifugal pump can be turned on/off using the control program on the computer. The flow rate is mitigated to the desired point by using the control valves. Control valves' stems can be adjusted using the CM10 control panel. Since the flow driven by the centrifugal pump is usually very steady, in normal experimental conditions, adjusting the control valves' opening manually will suffice.

3.5 CRUD Loop Measurement and Instrumentation

3.5.1 Pressure Measurement

The pressure measurement can be obtained from multiple types of equipment on the experimental setup. Having multiple places to get pressure readings is beneficial because we will be able to retrieve pressure even if some of the pressure reading equipment break down. Other than that, in case there is some malfunction or clog in some part of the loop, the problem can be recognized earlier because we know the pressure readings everywhere in the loop. There are two pressure transmitters on the loop. One is on the autoclave. It reads the pressure inside the autoclave and displays the number on the West6100 panel on the autoclave controller. Another pressure transmitter is connected before the back-pressure regulator. This one will read to the computer. Other than the pressure transmitters, there are also three pressure gauges at the pressurizing pump, at the top of the main loop and above the sample cooler. These pressure gauges should be more reliable than the other forms of measurement with electronics that can give a reading error.

3.5.2 Temperature Measurement

Temperature measurement in the loop is done in six spots, two spots in the autoclave and four spots on the tubings of the loop. The two temperatures are measured in the autoclave, the liquid temperature and the element temperature. Both readings are shown and labeled on the autoclave controller box. The liquid temperature is the temperature measured at the liquid on the top section of the autoclave. The element temperature is the temperature measured at the inside of the autoclave heater rod. The element temperature thermocouple is embedded inside the heating element press-fitted into the heating rod. The temperatures are measured outside the autoclave in four spots. All of these spots measures liquid temperature within the CRUD loop's tubing. This is clearly shown in the diagram 3-2.

3.5.3 Flow Measurement

The flow must be maintained so that the mass flux and the Reynolds number imitate the PWR as close as reasonable. The velocity of 3 m/s was chosen for most of our experiments because that will bring the Reynolds number and the mass flux condition close to that of the PWR's while not being too fast that it presents a significant vibration that can make CRUD loop deteriorate quickly.

In the MIT CRUD loop, the flow measurement is done using the differential pressure transmitters. The reason for using the differential pressure transmitters is that at PWR temperature and pressure conditions, other types of flow meter will not be able to withstand the harsh pressure temperature and flow rate. In this flow measurement system, the water flows through a small section made of $\frac{1}{2}$-inch tubing as oppose to the surrounding 1-inch tubing. Each end of the differential pressure transmitter is connected to the $\frac{1}{2}$-inch tubing and the 1-inch tubing. When the fluid flows through tubings with different flow area, it will have different pressure according to the Bernoulli's equation. The differential pressure measured from differential pressure transmitter can be used to calculate the actual fluid velocity using the Bernoulli's equation.

Calculation for differential pressure drop: The differential pressure drop in unit inch-H2O $\Delta P_{inch-H20}$ displays on the meter. This can be converted to the Pascal unit using the following conversion equation:

$$\Delta P_{Pa} = 249.09 * \Delta P_{inch-H20} \tag{3.16}$$

To convert the differential pressure drop to the fluid velocity, we start from Bernoulli's equation:

$$\frac{p_1}{\rho} + V_1^2 + gz_1 = \frac{p_2}{\rho} + V_2^2 + gz_2 \tag{3.17}$$

Since the two tee fittings where the differential pressure transmitter's nodes are connected at the same height level, the gravity term can be ignored leaving the following equation:

$$\frac{p_1}{\rho} + V_1^2 = \frac{p_2}{\rho} + V_2^2 \tag{3.18}$$

By rearranging the equation above, the following equation can be obtained:

$$\frac{2(p_1 - p_2)}{\rho} = V_2^2 - V_1^2 \tag{3.19}$$

Since the pressure at two locations are measured in the same tube, the mass flow rate at both locations must be the same, leading to the following relationship:

$$\dot{m}_1 = \dot{m}_2 \tag{3.20}$$

$$\rho_1 A_1 V_1 = \rho_2 A_2 V_2 \tag{3.21}$$

$$A_1 V_1 = A_2 V_2 \tag{3.22}$$

$$V_2 = \frac{A_1}{A_2} V_1 \tag{3.23}$$

Plugging this equation back into the rearranged Bernoulli's equation, the following equation is obtained:

$$\frac{2(p_1 - p_2)}{\rho} = (\frac{A_1}{A_2})^2 V_1^2 - V_1^2 \tag{3.24}$$

Velocity V_1can then be obtained from the above equation:

$$V_1 = \sqrt{\frac{\frac{2(p_1 - p_2)}{\rho}}{((\frac{A_1}{A_2})^2 - 1)}} \tag{3.25}$$

This equation is used to obtain the flow velocity in the autoclave from the differential pressure value measured.

Parameter	Value
Differential Pressure ($inches - H_2O$)	6.0
Density ($\frac{kg}{m^3}$)	667.4
1-inch Tubing Inner diameter (inches)	0.834
1/2-inch Tubing Inner diameter (inches)	0.402
Autoclave Inner Diameter (mm)	18.0
Autoclave Outer Diameter (mm)	20.0

Table 3.4: This table summarizes different parameters affecting mass flow rate. The values shown are values that are usually present in the loop.

3.5.4 pH Measurement

According to EPRI PWR Primary Water Chemistry Guidelines Volume-1 (1999) [41], the pH should be between 6.9 to 7.4. By itself, the pH has no effect on corrosion. The pH lower than 6.9 is expected to generate heavier core CRUD deposits. In the CRUD loop's case, the goal is the grow CRUD regardless, so there is no need to follow the 6.9 pH guideline directly. The amount of boron and lithium hydroxide is used to control the amount of pH in the reactor. EPRI's guideline allows boron to be added according to the situation to optimize the reactor. Lithium hydroxide is then added just enough to maintain the pH of above 6.9.

In CRUD loop's case, the interest lies in simulating the PWR conditions as closely as possible.

Therefore, the typical boron concentration of 1400ppm and lithium hydroxide of 3ppm is used. The lithium hydroxide amount is taken from the graph 3-5 by EPRI. If 1400 ppm of boron is traced up to 3 ppm of lithium, we can see that the point lies within the band for pH of 6.9-7.0.

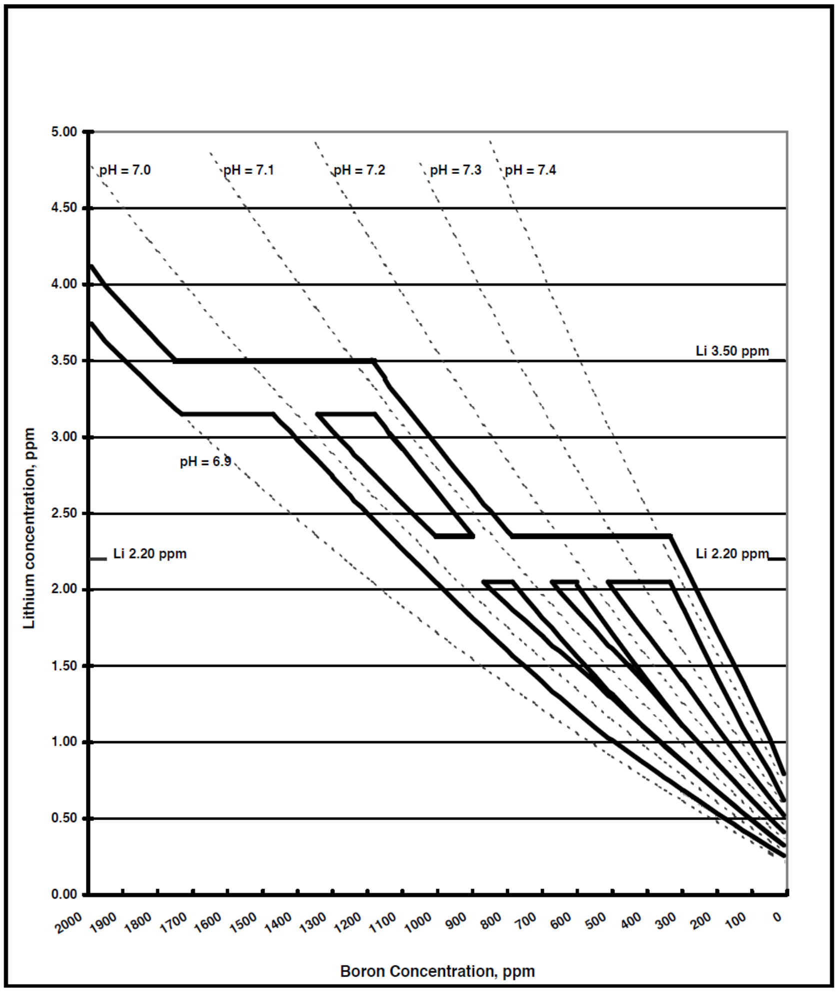

Figure 3-2d
Coordinated Chemistry at Elevated pH_T (>6.9)
Example Li/B Programs (plotted at T_{ave}=300°C)

Figure 3-5: Figure from the EPRI PWR Primary Water Chemistry Guidelines Volume 1 (1999) [41], that shows how the boron concentration related to the lithium concentration at 300-degree Celsius. This table was used to make the decision regarding the lithium concentration for the CRUD loop.

3.5.5 Dissolved Oxygen Measurement

The dissolved oxygen level is an important measurement to keep track of because it can increase or decrease both the general corrosion and the possibility of stress corrosion cracking. In the real reactor, dissolved oxygen concentrations can be controlled with plant heat-up by venting and vacuum filling, followed by the used of hydrazine or hydrogen for residual oxygen scavenging [41]. In CRUD loop's case, an easier method is employed where dissolved oxygen is bubbled out with argon from argon tank.

As seen in the figure below, the EPRI Primary Water Chemistry Guidelines suggests that the oxygen concentration for the PWRs should be measured every three weeks and should be less than 5 ppb, or else action level one is required. Action level one means that actions should be made for the dissolved oxygen level to go down. If that does not happen within a week, the shutdown is recommended. In the case that the measured dissolved oxygen is between 5ppb and 100ppb, the reactor can continue to operate but needs further investigation into the cause of the higher dissolved oxygen. At 100ppb of measured dissolved oxygen, the EPRI suggestion is to shut down the plant. Therefore, our aim for the loop is to get 5 ppb or less during an experimental run to imitate the normal PWR condition, as well as to keep the loop from corroding.

Table 3-3
Reactor Coolant System Power Operation Control Parameters[9] (Reactor Critical)

Control Parameter	Sample Frequency	Action Level 1	2	3
Chloride, ppb	3/wk[1]	[2]	>150	>1,500
Fluoride, ppb	3/wk[1]	[2]	>150	>1,500
Sulfate, ppb	1/wk[3]	[2]	>150	>1,500
Lithium, ppm	3/wk[4]	[5]	---	---
Hydrogen, cc (STP)/kg H_2O	3/wk[6]	<25[7] >50	<15[8]	<5
Dissolved Oxygen, ppb	3/wk[1]	>5	---	>100

1. These frequencies are a minimum based on typical plant Technical Specifications. Plant-specific frequencies may vary.

Figure 3-6: This figure shows the table from EPRI PWR Primary Water Chemistry Guidelines Volume 1, 1999 [41]. The important point from this table is the recommended dissolved oxygen level in ppb. If the dissolved oxygen level is beyond 5 ppb, it is recommended by action level one that the level should come down within a week or else shutdown is recommended. If dissolved oxygen is beyond 100 ppb, it is recommended by action level three to shut down the reactor right away.

3.5.6 Conductivity Measurement

In the EPRI PWR Primary Water Chemistry Guidelines Volume 1, the conductivity of water should be in accordance with the chemical additions. Therefore, there is no direct commandment on what the conductivity should be. Nevertheless, in the case of CRUD loop, the conductivity measurements are very useful for monitoring what is going on in the loop during the ion-exchanger cleaning.

For the CRUD Loop, the initial conductivity before adding any substance can be cleaned with the ion-exchanger down to around $0.09\mu S/m$. The conductivity should be a good representation of the amount of ions floating in the system. If there are too many unexpected ions inside the water prior to any addition of our chemical, these ions might cause an unexpected build up of harmful deposits. For example, calcium deposits can build up and ruin the tubing system. Other than that, without proper cleaning of ions, we cannot predict how these ions will interact with other things in the loop and affect the results of the CRUD buildup experiment. Once the conductivity reached that point or lower, chemical additions including the boric acid, the lithium hydroxide, and the nickel oxide CRUD particles can be added.

3.6 CRUD Loop Components (and Equipment Selections)

First is the main loop where the experiment will take place. It is where the water is heated to $320°C$ and pressurized to 155 bars to simulate the condition of PWR. The figure 3-7 below, revisited from the introduction, shows how the two loops are connected. The main loop's (circulation loop) input is connected to the output of the auxiliary loop. The main loop also dumps the output water into the auxiliary loop.

3.6.1 Main Loop

The main loop 3-9 is where the CRUD growth experiment will take place. The most important part of this loop is the autoclave. Within the autoclave is a test section with a heating rod in the center and the water flowing in an annulus around the heating rod. This test section is where we make the condition match that of the PWR to simulate the CRUD growth at PWR condition. The sample rings are placed on the heating rod inside the test section. The rest of the loop exists to get the conditions within the test section to the PWR conditions. This loop's water is heated to 320 ºC, pressurized to 155 bars, and given flow rate of roughly 2.5-3 m/s in the autoclave, to simulate the condition within the PWR.

Design Considerations Many design considerations go into building the main loop the way it is currently. First off, the loop supporting structure was constructed in the rectangular box shape because this structure is very sturdy. It can withstand heavy weight of the equipment such as the autoclave and the control valves without any problem. Other than that, the sturdy structure allows easier maintenance since the structure can be climbed to reach any point in the loop without the hassle of using a ladder. The loop can also be moved relatively easy. The middle section can be detached separating the main loop into two large sections. Each of these can then be transported as a whole through the use of a forklift or a palette jack. Unistrut beams were chosen as the structural building blocks because they are modular, allowing easier loop modification. Unistrut beams also have a very competitive price. Stainless steel was chosen as the structural material because of its

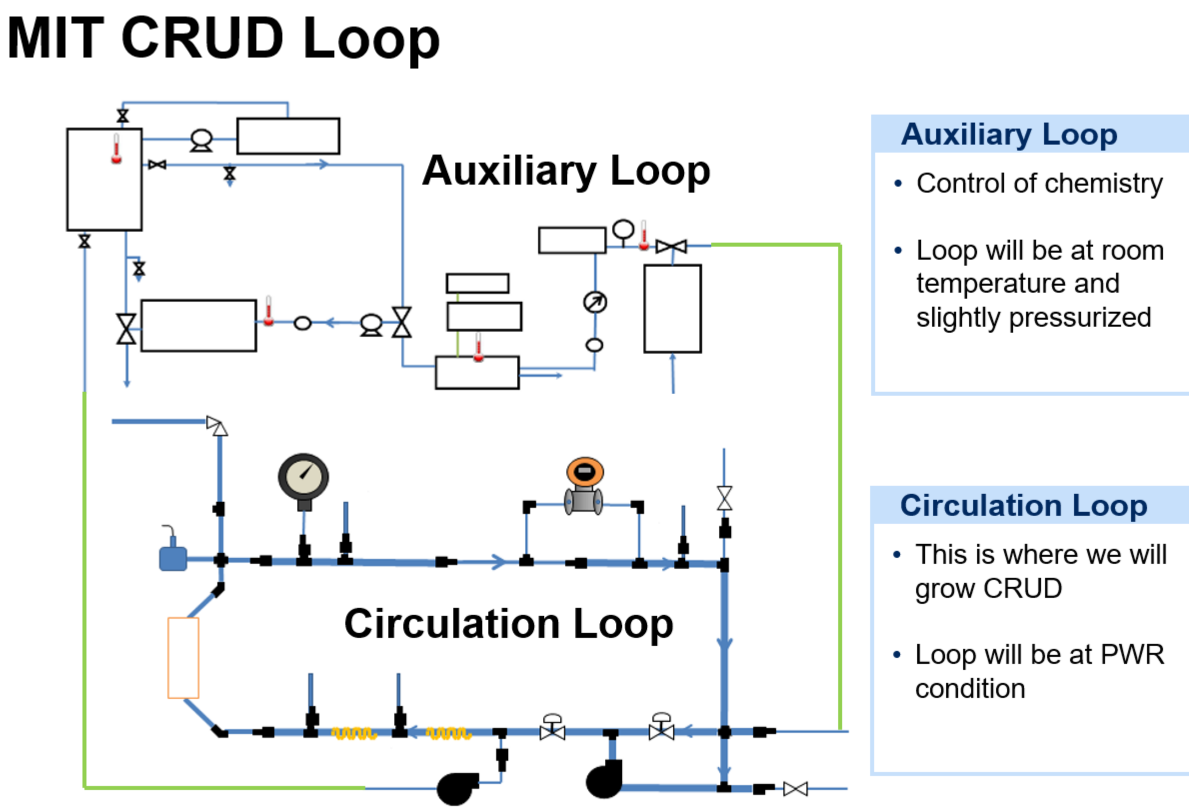

Figure 3-7: This is a diagram demonstrating the two most important parts of CRUD loop, the auxiliary loop, and the circulation loop. The diagram shows how both loops are connected. The auxiliary loop provides water to the circulation loop at the input of the pressurizing pump. The circulation loop outputs the water to be cooled down at the sample cooler. The pressure boundary is located at the back-pressure regulator in the auxiliary loop.

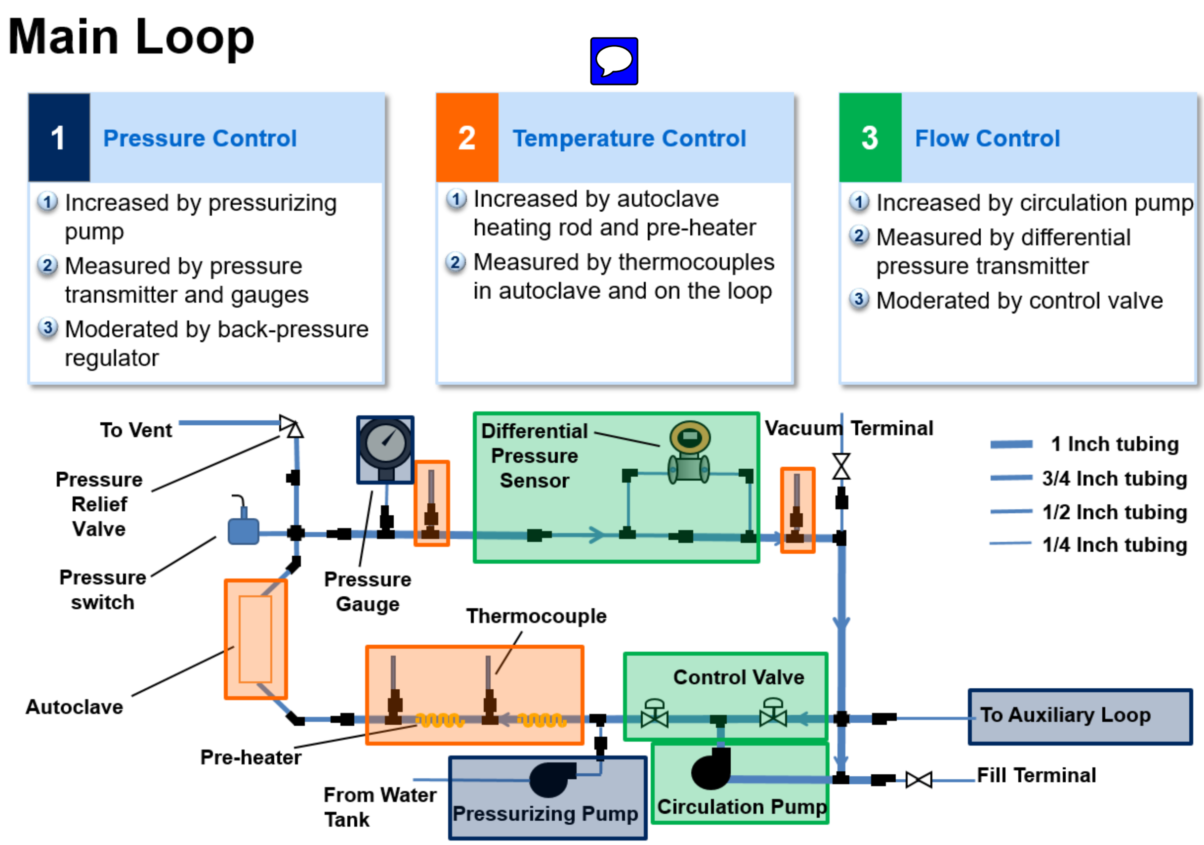

Figure 3-8: This diagram shows different parts of the main loop or circulation loop and their functionalities.

strength and low price.

3.6.1.1 Main Loop Pictures

Figure 3-9: This figure shows the photos of the main loop from different angles. Notice the autoclave on the left of both pictures wrapped in the heat insulators. This autoclave is where the experiment is carried out.

3.6.1.2 Autoclave Components

The autoclave is the main part of the loop. It is the place where the CRUD growth will take place. The rest of the system is built to help this part simulate the condition within the PWR reactor vessel. The autoclave is shown in the figure 3-9 on the left of both photos wrapped in the beige heat insulation blankets. The autoclave has many components attached to it to control and regulate the conditions inside the autoclave vessel which will be explained below.

Autoclave Vessel The autoclave vessel is the actual vessel where simulated PWR fuel rod will be placed. Water will flow in from the bottom of the vessel at 45-degree angle, then travel up the annular flow section with the inside of annular being the electric resistant heating rod which simulates PWR fuel rod. The autoclave test section diameter is 20 mm. This autoclave is also special because it has three sapphire windows attached which open up a wide range of opportunities including the use of laser triangulation to measure the CRUD thickness in situ, the use of Raman spectroscopy to measure the CRUD composition in situ, and the measure of the contact angle with a laser.

It has two flanges on the top and bottom. The bottom flange will remain sealed most of the time. The top flange can be taken off along with the sample fuel rod. The top flange contains a graphite gasket at its larger opening to seal the autoclave. On the top section of the top flange, a rubber o-ring acts as a seal. The top most part of the autoclave top flange is a polyacetal plastic cap that pushes down on the fuel rod to prevent it from getting pushed up by the pressure inside the autoclave. The reason that this part is made of polyacetal and not metal is to keep the heating rod from being grounded. Grounding the heating rod could affect electrochemistry on the surface of the heating rod which is not desirable in the CRUD growth experiments. Since this rubber O-ring, as well as the polyacetal cap, cannot withstand the high temperature, the top part must be cooled with the cooling system from the auxiliary loop. This autoclave has been custom manufactured by Cormet Oy, a company from Finland.

Autoclave Heater Autoclave heater lies within the test section of the autoclave vessel. It is composed of the internal cartridge heater, press-fitted into the heating rod cladding. The internal cartridge heater spans a section 150 mm and has a diameter of 12.5 mm. The heating rod cladding also has 12.5mm in inner diameter and 17.5mm in outer diameter. The heating rod cladding is 715 mm in length. The maximum power of the heater is 1750 Watts.

The heating rod is designed so that the electricity runs through only the middle heating cartridge part, as oppose to the whole rod. This design is done to preserve the electrochemistry of the heating rod surface, so it is not interfered with by electricity flow. This preservation of the electrochemistry is important to the CRUD-resistant coating experiments because changing the electrochemistry at the surface can potentially change the adsorption of CRUD particles at the sample surface, and therefore give inaccurate data.

Autoclave Control Box The autoclave itself has its controlling system that can be used to monitor the pressure as well as the temperature of the heating element and the liquid inside the autoclave vessel. This information is also passed on to the computer so it can be displayed on the autoclave control program and recorded into data files.

The heater element temperature controller can be used to ramp up the heater element temperature in a controlled fashion. This variable control of temperature is made possible with the large transformer inside the heater box that can be used to adjust the voltage and the power supplied to the heater. The autoclave control box has a breaker and two fuses as a protection against a sudden power surge.

Autoclave Flow Meter The flow meter attached to the autoclave stand is used to monitor the flow rate of the cooling water. This flow meter's reading indicates the flow output throughout the cooling water system. To adjust the cooling water flow rate, turn the yellow valve at the sample cooler's cooling line outlet. Adjusting the yellow valve at the sample cooler's cooling line will redirect to either the autoclave cap or the circulation pump.

Autoclave Pressure Transmitter This pressure transmitter is used to measure the pressure within the autoclave. It is attached to the tubing that connects directly to the autoclave. It transmits the data to the autoclave control box to display on the control box panel. Additionally, the signal is also relayed from the autoclave controller to the computer for display purpose.

Autoclave Safety Valve The autoclave safety valve is a redundant safety valve which will release steam into the room once the pressure goes to around 3500 psi. It is the last resort protection in the case that no other systems are operational including the main loop safety valve, the pressure switch, etc. The release pressure can be set to anywhere between 3000-4000 psi by turning the knob on top of the valve with a wrench.

3.6.1.3 Heating Tape Control Components

As mentioned earlier, there are two main heating systems in the main loop. One is in the autoclave heating rod which is situated inside the autoclave vessel. Another is the heating tape system. This heating tape system comprises of all the heating tapes installed on tubings in the loop. The following section explains what each component in this heating tape system is and their importance in the heating tape control system.

Main Loop Temperature Probes The main loop temperature probes are the temperature probes outside the autoclave. They are used to monitor the temperature at different spots throughout the loop. There are four main loop temperature probes. They are inserted into their casing which can withstand the loop's high pressure. The tip of the casing is submerged into the area with flowing water along the tubing system.

Main Loop Heating tape Heating tapes are wrapped around most of the main loop tubings. These heaters are used to give more heat to the system so that the temperature close to that of the PWR can be achieved. The heating tapes used is an extreme temperature heating tape wrapped in fiberglass which can withstand temperature up to 760 ^{o}C.

There are two main types of heaters we used. Both of which have the length of 8 feets and the power of 624 Watts. The 120V type heater has higher current of 5.2A, while the 240V type has the current of 2.6A. The reason that the heating tape is separated into two types is that the 240V plugs which were meant for heaters do not give enough Amps to be able to get the loop to the PWR temperature on its own. Therefore a 120V plug must also be used to draw more current to heat up the water.

When running the loop near PWR temperature, the heating tape will be heated to a very high temperature. As a result, it may turn white and brittle. It is recommended not to move them in any way, because the outer fiberglass will flake off, rendering the heating tape unusable. Since the flow rate that is used in the loop to match the PWR condition is quite high, the vibration is quite significant. This vibration can over the time cut the fiberglass insulation out of the heating tape wire if they are not kept clear of aluminum insulation cover.

Latching Relay System The latching relay system is used to make sure that once there is an emergency shutdown for some reason, the heating system will stay shut. For instance, if there was an overpressure and the pressure switch turned the heating system off, we do not want the heating system to be on again as soon as the pressure is lowered. If there is something wrong with the loop that causes it to shutdown, we need to inspect it before continuing the run to make sure that the loop will be safe.

Temperature Controller The temperature controller is of type CN9000A by Omega Engineering. Only one temperature controller is used to control all heating tapes wrapped around the main loop tubings. This temperature controller takes the input as the liquid temperature from the top-left dual junction thermocouple near the outlet of the autoclave. It is a proportional-integral-derivative controller which means it has better ability to stabilize temperature than the normal on/off controller.

Solid State Relays Since the voltage and current we are controlling is very high, we cannot run the current through the temperature controller without having it burn out. Therefore, an intermediate relay is needed to connect the heaters with the rest of the temperature control system. The intermediate relays in the CRUD loop heating system are the solid state relays in the temperature controller box. Two solid state relays are used in the system, one for the 240V heaters and another for the 120V heaters.

Heater Duplex Receptacles These duplex receptacles act as an intermediate connection between individual heaters and the heating control system. It is included to facilitate the task of connecting and disconnecting individual heater, making it a matter of just unplugging and plugging the individual heater plug. There are two duplex receptacles for 240V heaters which are connected to the thick white power line. Another duplex receptacle is for the 120V heaters which are connected to the gray power line.

Heater Emergency Switch The emergency switch is the smaller red manual switch which is not attached to the wall. It is used to shut down only the heaters in the system. To shut down the whole system (not recommended), the emergency power-off switch attached to the wall both inside and outside the CRUD loop room can be used. The reason that this system is not connected to the pressure or flow control system is that we still want the water to be pressurized and running for a while after shutting down heaters. If pressurizing pump is shut down as well during an emergency, the quick decrease in pressure will cause the water inside the loop to flash to steam which could damage the system. Leaving circulation pump on will help with cooling the whole loop evenly.

3.6.1.4 Pressure Control Components

Pressurizing Pump The pressurizing pump is used to pressurize the system to 15.5 MPa. It is a positive-displacement pump (diaphragm pump) which works by using a piston. The diagram of pressurizing pump is shown in the following figure 3-10. When piston gets pulled back, the diaphragm also gets pulled back. As a result, the water is drawn in, to fill the chamber. The discharge ball of this pump chamber is pushed on its seat while the suction ball is opening the suction side. Because of this, water will not be drawn from the outlet, because the discharge ball, on its seat, will block the flow. Water comes in through the inlet passing through the suction ball, filling up the chamber. When the piston gets pushed forward, the reverse happens. Water is pushed away from the pump chamber out through the discharge balls, while the inlet will be blocked with suction balls on its seat. This stroking repeats to pump the water slowly through the diaphragm pump.

The current pressurizing pump is of Ecoflow model LDB1V-M910 manufactured by LEWA. The pump can achieve the highest flow rate of 82 ml/minute (1.3 gallons/hour) at 2250 psi (roughly 15.5 MPa). The pump's driving motor is a 1730 rpm AC motor with 12.5 gear reduction. The pressurizing pump stroking speed is 138 stroke/minute. The internal pressure relief valve of the pressurizing pump is set to 170 bars. The maximum allowable working pressure for the pressurizing pump is 391 bar.

When using the pressure pump, the valve leading up to the pressure pump inlet must be checked so that it will not obstruct flow. Failure to do this may result in the diaphragm damage to the pressurizing pump. If the diaphragm of the pump is damaged, pump oil will mix with water making it very hard to clean and potentially very costly. When repairing the pressure pump or emptying this loop, this same valve must be closed. The pump flow rate can be adjusted via the adjusting knob on its side. The flow rate can be adjusted from 82 - 8.2 ml/minute, all while maintaining the stroke rate at 138 strokes/minute.

Pressure Gauge Extra pressure gauges on the main loop serve two purposes. First, it is used to supplement the electronic measurement and to make sure that there is the electronic is not malfunctioning. Second, pressure gauges also give a measurement of local pressure. They can be used to determine if there is any extreme pressure difference in the loop caused by some problem such as clogging of tubes.

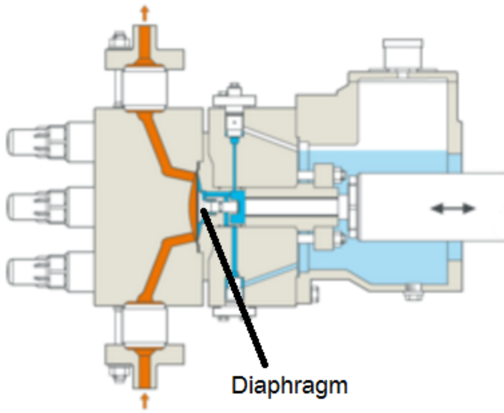

Figure 3-10: This figure shows the cross-section diagram of a generic LEWA diaphragm pump [101]. The orange shaded parts are the water. The white part at the inlet suction and outlet discharge holds the suction balls and the discharge balls.

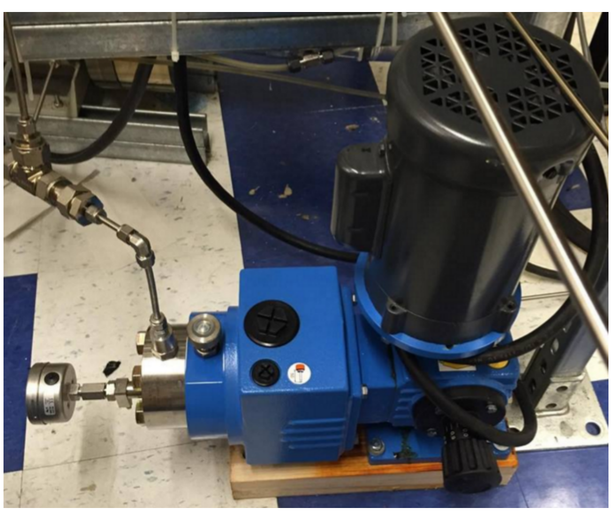

Figure 3-11: This photo shows the pressurizing pump installed on CRUD loop.

3.6.1.5 Flow Control Components

Differential Pressure Transmitter Differential pressure transmitter can be used to obtain the difference in pressure in two spots. Together with the orifice and some additional Bernoulli equation's calculation, the loop flow speed can be obtained. This differential pressure transmitter can measure a pressure difference from 0 to 150 inch-H2O.

Control Valves The control valves are used to control the flow speed of the water around the loop. The control valve in series with the circulation pump is the main control valve that will be used to adjust the speed of the flow. This control valve is connected to the PID controller that will adjust the valve's opening depending on the input that it gets from the computer which represents the velocity of the system. However, in reality, since the flow rate driven by the circulation pump is very steady, a manual adjustment of the control valves to constant opening amount will suffice for the flow control without any PID functions.

When using the control valves, caution should be taken to make sure that valve is not pushed too far. Pushing the valve too far will cause damage in the packing and therefore a leak during pressurization. The reason for using any control valve, when variable speed pumps are available, is that the variable speed pumps cost significantly higher than a pump with a control valve.

Compressed Air Pump The compressed air pump is used to supply compressed air to the control valves. The control valves need the pressure of at least 30 psi to operate. It uses the gas pressure to close and open the valve. The reason we did not use a gas tank for supplying the pressure to the control valves was that the gas tank would exhaust very quickly because of the gas leak from the control valves. The compressed air pump is situated in a storage room near the CRUD loop room to save room space. The compressed air runs from its room through the copper pipe into the CRUD loop's room where it is attached to the control valves. The compressed air pump will trigger, each time its pressure falls below a set pressure.

Circulation Pump The circulation pump is a centrifugal pump that is used to drive the flow around the loop. The pump is a magnet drive pump which means it uses a magnet to couple the pump turbine with its motor. Using the magnetic coupling as oppose to the physical coupling is done to prevent any leaking. A small leak at a pump's direct coupling could lead to water exiting the loop, and other undesirable things such as oxygen going in. If the pump's turbine is directly coupled to the pump motor, there must be a connecting hole in the pressurized part of the pump to make this direct coupling happens. That connection, even if the leak is tiny, will have an effect on the water chemistry of the loop over time. For our use, we cannot tolerate much chemistry change since the system will be at high pressure and temperature where corrosion is a huge issue. The loop will run for week-long experiments which give the leak plenty of time to degrade the system's water chemistry. The pump's ability was chosen, based on the flow rate versus the pressure head graph as shown in the figure 3-12, with the criteria of getting enough flow rate, while having redundant power. As seen on the flow rate versus pressure head graph, the flow rate can be adjusted by adjusting the pressure head. In CRUD loop, the pressure head can be adjusted via the control valve.

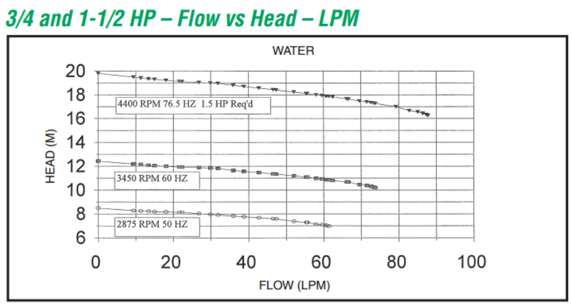

Figure 3-12: This graph shows the magnet pump's flow vs. head graph as seen in its documentation. The pump is used for CRUD loop is represented by the middle line.

3.6.1.6 Safety Components

Main Loop Pressure Relief Valve The pressure relief valve will release steam into the tube directed to the ceiling vent when the pressure in the loop is higher than 3000 psi. The ceiling vent tube is connected all the way up to the roof of the building where the steam will be released from a chimney. This pressure relief valve is used as a last resort to prevent the loop pressure from rising dangerously high. This main loop pressure relief valve is manufactured by Swagelok, and is part of their RVP series, with part number 5RVP9072.

Pressure Switch The pressure switch is one of the many safety features we have on the CRUD loop. It is a switch that will shut down the system once the pressure becomes too high. This pressure switch is connected to the heating system and will trip the heating system if the pressure goes above around 2500 psi. The latching relay is used along with this switch to make sure that the heating system will stay off until someone comes in to check up on the CRUD loop and make sure that there is nothing seriously wrong with the loop. The reason that the pressure switch is only connected to the heating system is that the system should still be pressurized and the water in the system should still flow while the loop cools itself down. If the pressurizing pump and the circulation pump is tripped as well, the decrease in pressure and flow rate while the temperature remains high will cause the water inside the system to flash which is very undesirable because of the decreased heat removal and the increased corrosion. After the heating system has been shut down for a while, the computer will automatically shut down the pressurizing pump and the circulation pump. Since the computer is sometimes unreliable, in the case the computer fails to send a signal to stop the pump resulting in further overpressure, this should not be as dangerous if the system is no longer heated. In the case the where water is heated, and pressure is high, any leak or part detached will be very dangerous as the steam quickly expands. The system will take a while to come under a normal pressure as water flashes to steam. In the case where water is not heated, water will only expand by a tiny amount and the system will come down to normal pressure very quickly, and therefore is not a dangerous circumstance.

3.6.1.7 Other Components

Piping & tubing All tubings in the loop are made of 316 stainless steel with a working pressure rating of at least 2800 psi and over 10,000 psi burst pressure. It is highly advised that the tube should not be used above 2800 psi for an extended period, but if pressure runaway ever happens, the tube will be able to withstand the pressure as high as 10,000 psi for a short time.

In the high-temperature and high-pressure condition, corrosion poses a huge problem to the integrity of the loop. Therefore, the 316 stainless steel is chosen instead of the widespread 304 stainless steel because of its superior corrosion resistance that it gets from having 2-3 percent mix of molybdenum. Other than that, it is also easy to clean and fabricate. Tubings in the cold parts of the CRUD loop are also 316 stainless steel. Malleable nylon and vinyl tubing had been tried in the cold regions of the system but resulted in an oxygen leak into the system. Therefore, it was replaced with steel. Most tubings on the main loop are larger than the rest of the loop at one inch in diameter because the pressure drop must be maintained at a low level to get the high-speed flow of the PWR condition. The larger the diameter of the tubings, the less pressure drop it creates.

Main Loop Tubing Insulation The ceramic wool insulation wrapped with aluminum sheet jacket is used to insulate most sections of pipe. The ceramic wool is chosen as the insulator for its ability to withstand a very high temperature we operate at as well as for its ability to insulate heat very well. The aluminum jacket is used to contain the ceramic wool around the tubings. Since inhalation of the ceramic wool or any other long biopersistent dust particles over an extended amount of time has a potential to cause tumors, it is strongly advised to put on masks covering nose when removing or assembling ceramic wool insulations.

3.6.2 Auxiliary Loop

As introduced in the introduction, the auxiliary loop's main function is to control the chemistry, store the water, and act as input/output for the main loop. The auxiliary loop consists of three major loop systems. First, the main part of the auxiliary loop is the loop that help controls the chemical parameters and provides the water input into the main loop. This loop will be at the room temperature and almost at the atmospheric pressure. The second loop is the cooling water loop that helps cool the water coming out of the main loop into the auxiliary loop, as well as other equipment including the autoclave and the circulation pump. The third loop is the deionization system which is used to clean (deionize) and mix the water in the water tank. For simplicity, we have grouped the three loops together in the auxiliary loop system as they all operate at the room temperature and close to the atmospheric pressure. This section will delve deeper into details of what each piece of equipment on the auxiliary loop does, and how to use them. The figure 3-13 shows what make up the auxiliary loop.

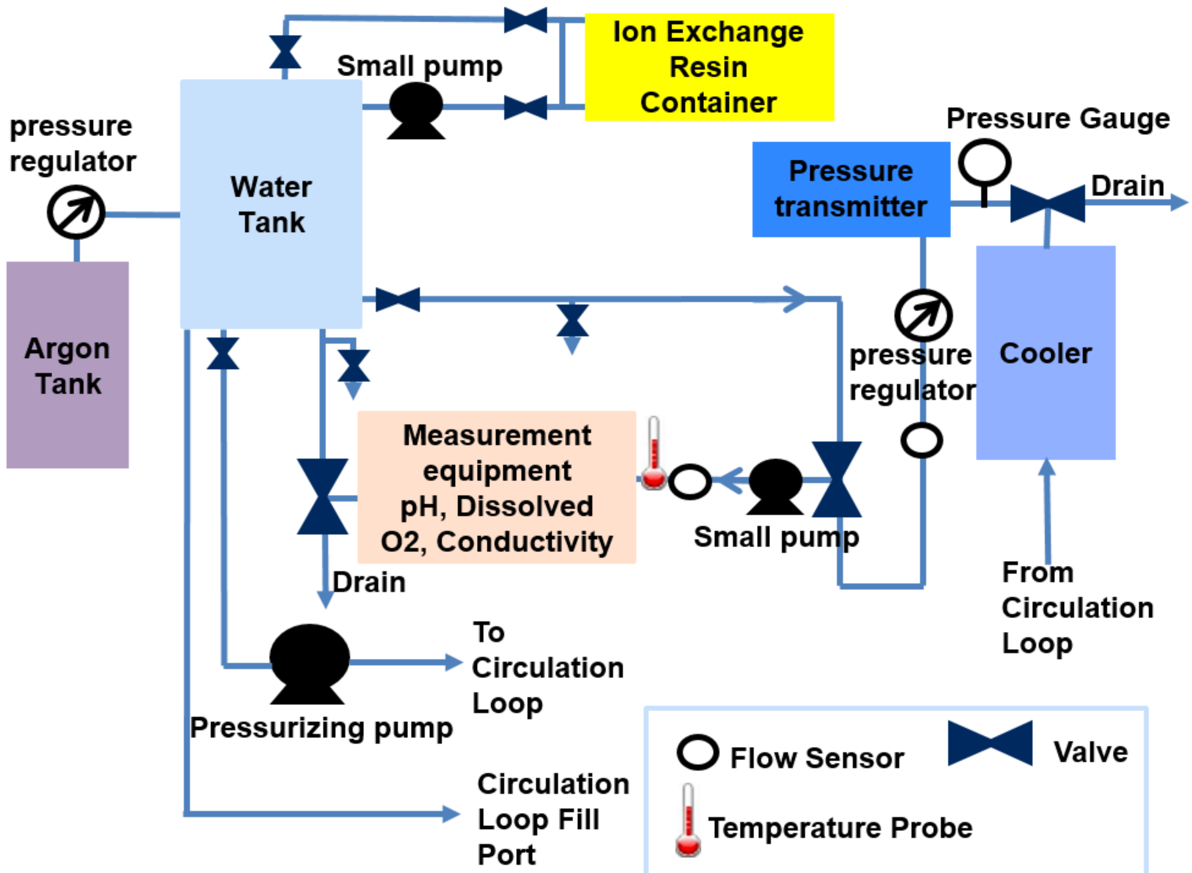

Figure 3-13: This diagram shows different parts of the auxiliary loop and their functionalities. It should give a better overall picture of what is going on in the loop before delving deeper into what each instrument does.

3.6.2.1 Water Chemistry Measurement Equipment

The chemistry measurement equipment on the CRUD loop come in a set manufactured by Mettler Toledo. This set can be used to measure the oxygen, the conductivity, and the pH of water. The measurement of water chemistry is important because an uncontrolled loop chemistry may affect the data, as well as damages the loop. For example, a high amount of oxygen can quickly corrode the loop, while a high amount dissolved calcium can lead to the scaling and clogging of the loop.

Figure 3-14: This is the photo of the auxiliary loop. The auxiliary loop is used to measure and regulate the loop chemistry.

Transmitter monitor This blue transmitter with a green screen is used to read the data signals from the dissolved oxygen, the pH, and the conductivity sensors. Through its connections on the back side of the board, this transmitter can also relay the data analog signal to the computer's Agilent control unit to be displayed on the computer. Data output from the transmitter monitor is in the form of the current with the ampere unit. Therefore, the data line must be connected to the analog reader with ampere input. It is also possible to connect the data line to the reader with voltage input and use resistor to convert the ampere to the voltage. The transmitter monitor can also be used to set the settings of each of the three equipment.

O2 Sensor The oxygen sensor is used to measure dissolved oxygen within the water in the unit of ppm. The settings for the unit can be controlled through the Mettler Toledo transmitter monitor. It is recommended that the dissolved oxygen level should be below 5 ppb during the experimental runs to prevent corrosion. The oxygen can be lowered by bubbling the argon through the water tank for several hours to a day depending on the oxygen level. The high oxygen level may also be an indicator of a leak in the system.

Conductivity Sensor The conductivity sensor can determine the amount of ions in the loop. The conductivity measurement can be used to make sure that the water is completely cleaned of ions before we add in any other chemicals. Before adding any chemicals to the loop, it is recommended that the loop should be deionized down to around 0.09 μS/m as recommended by the autoclave manufacturer. This conductivity sensor has a range of 0.05 μS/m to 10,000 μS/m and will output a 4-20 mA signal.

pH Sensor The pH sensor is used to monitor the pH to make sure that it is similar to that of the reactor which is around 6.9-7.4 [41]. Note that this sensor needs at least 50 ml/minute flow rate to give proper readings. The EPRI's lower bound pH recommendation is there to prevent CRUD buildup. Therefore, it is recommended that this loop should operate at EPRI's lower bound since our goal is to grow CRUD.

3.6.2.2 Flow Sensor

The flow sensor measures flow output in ml/minute and output the readings in analog form to the computer. The flow sensor attached in series with the measurement equipment is useful in determining if the measurement equipment has enough flow rate to work properly. Having a flow rate through the measurement equipment 50 ml/minute or more is recommended for accurate measurement readings. The flow sensor's input and output lines are only 1/8-inch in diameter. Because of its tiny size, it is the equipment that is most likely to clog in the measurement system.

3.6.2.3 Valves and Actuators

The valves and actuators in the auxiliary loop are used to control the direction of flow within the loop. The three electric valves attached to the actuators are all three-way valves. These three-way valves can let water flow from center inlet to either the right or the left end outlet. The arrows marked on the valve shows which end outlet is open for flow.

The reason electric actuator is needed is that the manipulation of valves is required when taking the measurement of loop's chemistry. While the loop is running no one should be near it, and therefore, there will be no one to adjust these valves for taking the measurements. The valve system, therefore, must be controllable from the computer. This remote control of the valves is where the electric actuator comes in.

3.6.2.4 Back Pressure Regulator

The back pressure regulator can be used to set the pressure in the loop. As shown in the figure 3-15, the inlet of the back pressure regulator has high pressure, while the outlet will have almost the atmospheric pressure. When the pressure is not as high as the adjusted pressure, no water will leave the regulator. When the pressure on the inlet side increases to become high enough to push the diaphragm, some water is let through the regulator until the pressure drops down again. When the adjusting knob is turned, the spring becomes tightened pushing down the diaphragm with greater force. As a result, more pressure is needed from the inlet side to push up the diaphragm and let water flow out of the outlet. The back pressure regulator is placed after the sample cooler as it cannot tolerate high-temperature environment.

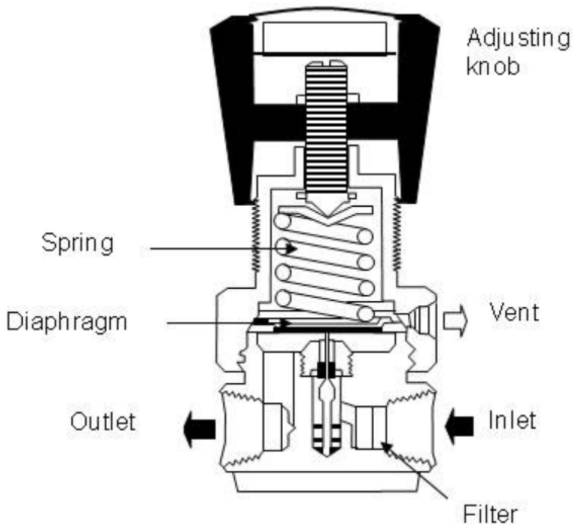

Figure 3-15: A labeled diagram depicting an overview of back-pressure regulator [102].

3.6.2.5 Sample Cooler

The sample cooler is used to cool the water as it flows into the auxiliary loop. By cooling the water down, we make sure that the equipment in the auxiliary loop will not be damaged from the extreme temperature. Equipment such as the back-pressure regulator, the pressure transmitter, and the measurement equipment cannot withstand the extreme temperature within the main loop. The sample cooler is a heat exchanger that dump heat from the high-temperature line from the main loop onto the cooling loop.

3.6.2.6 Water Tank

The water tank in the auxiliary loop is used to store water that will be used to circulate in the main loop. It is where water is added to the loop. The top lid can be unscrewed open to pour in tap water. The raw tap water can be deionized by the deionization system attached to the water tank. The mixture of chemicals such as boron and lithium hydroxide can be added from the top of the lid after the water is cleaned through an injection port. The water tank can hold approximately 120 liters of liquid when full to the top. Its height is approximately 90 cm, and its diameter is approximately 41 cm. The glass column on the side of the tank is used to see the water level in the tank.

3.6.2.7 Argon Tank

The argon tank is used for bubbling argon into the water tank to remove oxygen. When argon is bubbled inside the tank, the argon bubbles remove dissolved oxygen from water releasing it on top of the water tank where the bubbles pop. The gas exhaust then removes that additional argon along with oxygen from the top of the tank. Argon is used instead of nitrogen because it was found that bubbling nitrogen causes pH to rise slightly.

3.6.2.8 Deionization System

The deionization system is used to lower the ion concentration of water in the water tank to an acceptable amount for the experiment, which is around 0.1 µ S/m. Additionally, the valves can also be setup to bypass ion-exchanger columns. In that case, the deionization system will act as a water tank mixer instead. This functionality is useful when mixing the chemicals to make sure they are distributed evenly throughout the water tank. The deionization system consists of the two mixed-bed ion exchanger columns and a pump.

The mixed-bed ion exchanger columns are cylindrical containers of ion-exchange resin beads. When water flows through the beads, ions in the water will be absorbed by the beads. Ion-exchange resin beads work by trapping unwanted ions and releasing the ions that combine with water. Ion-exchange resin beads are fabricated from organic polymer substrate and are usually porous providing high surface area to trap and release ions. The small pump in the deionization system is used to draw the water from the bottom of the water tank into the deionization columns. The output water from the deionization columns is ejected into the top of the water tank.

Small pump in the deionization system is used to draw the water from the bottom of the water tank, push the water through the deionization columns, and out into the top of the water tank.

Figure 3-16: This is a photo of the two ion-exchanger columns connected to the deionization system. The lower left green pump is the small pump used to drive the deionization system.

3.6.2.9 Gas Exhaust Flasks

When bubbling argon through the loop, there must be an exhaust where the argon gas can leave the tank. A bare exhaust that just let argon gas out of a port will interfere with the dissolved oxygen level. The interference happens because the exhaust will also let the oxygen from the air into the tank. Although this is counter-intuitive that the oxygen from the air can seep in while the argon gas is rushing out, it is an observable effect. Without the gas exhaust flasks, we never got the dissolved oxygen level below 200 ppm. After the gas exhaust flasks were installed, the oxygen can go down to almost zero ppm. To prevent the oxygen from outside from seeping in through the exhaust, we use a system where the exhaust gas would bubble through two flasks of water. The exhaust gas can leave the system in the form of bubbles, but the air outside cannot go back through the exhaust because it cannot penetrate the water to get to the bubbling tube. Two flasks function in the same way to make sure that no oxygen will get into the loop through the exhaust.

3.6.2.10 Neslab Cooling Water Heat Exchanger System

This heat exchanger system cools down and drives the cooling water around the loop to cool the sample cooler, the circulation pump, and the autoclave. This cooling water heat exchanger input and output water from the building's cooling water. The water from the building is used to heat-exchange cool down the cooling water that goes through the whole CRUD loop. Note that the building water and the CRUD loop's cooling water runs in a separate loop and do not mix. CRUD loop's cooling water can be filled in from the hole on top of heat exchanger system designated for monitoring and filling its cooling water.

3.7 Data Acquisition and Control System

3.7.1 Control Program

The control program is used to monitor parameters and control equipment in the CRUD loop from the computer. The control program usage includes:

- Turning on/off valves 1, 2, 3 in the auxiliary loop
- Turning on/off mini-pump for the auxiliary loop
- Turning on/off mini-pump for the deionization loop
- Turning on/off circulation pump for the main loop
- Saving the data

The control program is not used for safety at all since it can be unreliable. Once the control program starts, flick the scan control switch on to initialize its operation. The program will then starts reading from the circuit board, and clicking sounds will be heard. After a set scan interval, this scanning as indicated by clicking will repeat.

The control program panel 3-17 shows a simplified layout of the CRUD loop with important equipment in the white boxes. Beside the boxes are the data readings associated with each equipment along with its unit. Additionally, for the equipment that can be controlled through the program, the on/off button is provide to turn that equipment on and off. To save the data, specify the save interval in minutes, then click on “start saving data” button. After this is done, the save minutes elapsed will start counting the minutes since the save start. Once the experiment is done, click finish saving data to stop the count and save data.

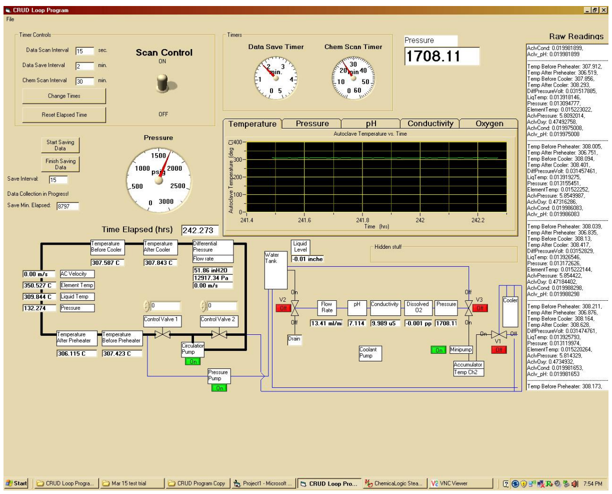

Figure 3-17: This is the illustration of how the CRUD loop control program looks. It is laid out in a simplified diagram of the loop, where data readings will be shown at the related equipment locations. This control program can be used to control valves and pumps as seen by their on/off function buttons.

3.7.2 Electronics

Other than the controller box provided by Cormet Oy for the autoclave, the two primary electrical systems in this loop consist of the system controlled by CRUD loop relay box, and the system controlled by the heating tape controller box. The CRUD loop relay box controls most functionalities in the loop except the heating tapes. The heating tape controller box has a single job of controlling all the heating tapes on the loop.

3.7.2.1 CRUD Loop Relay Box Wiring Diagram

The CRUD Loop relay box 3-18 acts as an intermediary between the CRUD loop control program and the equipment. The computer’s CRUD loop program can read data and send on/off command from the Agilent control unit attached to the computer. All the data inputs used in this loop are analog inputs. All the outputs are on/off command signal. The main reason to have the CRUD loop relay box is that there will be a high voltage overload if equipment high voltage wires are wired directly to the relay box. Therefore intermediate relays are needed to lower the voltage first. The CRUD loop relay box is a collection of relays used to control the equipment in the loop other than the heating tapes. It also provides duplex boxes, so it is easy to plug the equipment into the system. The emergency switch and the pressure switch are also wired through here to shut down everything in the case of an emergency.

For most reading purposes, the data output line from the equipment is connected directly to the reader board on the Agilent control unit. For some other equipment, such as the three electric actuator valves, the connection first goes to relay box which does nothing to it, then to the Agilent control unit. The reason for putting those connections through CRUD loop relay box is to make it easier to disconnect and organize wires when moving the loop to other rooms.

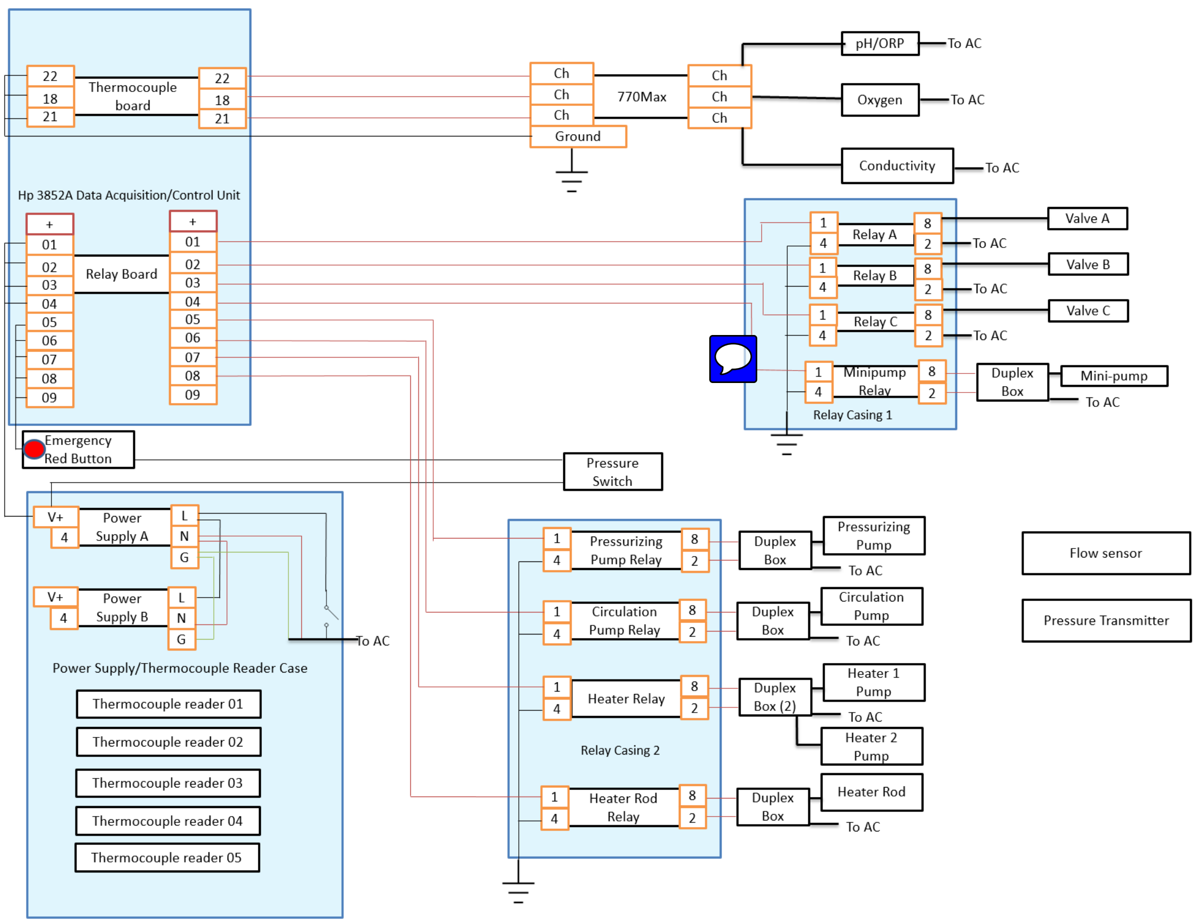

Figure 3-18: This diagram illustrates the simplified electric wiring diagram of CRUD loop relay box system.

All connecting plugs on the CRUD loop are labeled with the socket that it connects. In the case that the labels are damaged or lost, the following list will give guidance on where to connect different equipment to. Alternatively, this list can be obtained by looking into the visual basic code that made up the program. The CRUD-loop relay box part shows that the connections that go directly to the CRUD loop relay box. The CRUD-loop computer inputs list shows the connections that go onto the reader channels on the board attached to the Agilent control unit.

CRUD loop relay box

1. Valve 1: Ch1
2. Valve 2: Ch2
3. Valve 3: Ch3
4. Minipump: Ch4
5. Pressurizing pump: Ch5
6. Coolant pump: Ch10

CRUD loop computer reader

1. Temperature before heater: Ch1, Thermocouple reader
2. Temperature after heater: Ch2, Thermocouple reader
3. Temperature before cooler: Ch3, Thermocouple reader
4. Temperature after cooler:Ch4, Thermocouple reader
5. Differential Pressure: Ch9, Voltage reader
6. Liquid Temperature: Ch12, Voltage reader
7. Pressure: Ch13, Voltage reader
8. Element Temperature: Ch 14, Voltage reader
9. Autoclave Pressure: Ch 15, Voltage reader
10. Auxiliary Loop Flow: Ch16, Voltage reader
11. Liquid Level: Ch 17, Voltage reader
12. Oxygen: Ch 18, Voltage reader
13. Conductivity: Ch 21, DC current reader
14. pH: Ch 22, DC current reader

3.7.2.2 Heating Tape Controller System Wiring Diagram

The heating tape system wiring is setup to control the heating tapes outside the autoclave as well as to shut down in the case of the emergency switch activation or the pressure switch activation. The following figure represents the wiring within the control box. The gray box represents the wire coming in from different places. The power is drawn from the 120V AC and the 240V AC lines. The solid state relays (SSR) are intermediate relays that will allow the high voltage and current to go through it to the heaters. Without the intermediate relays, the wires in the normal relays will burn out from the high current. Each solid state relay operates with either the 120V AC line or the 240V AC line. The DC power supply supplies the power to turn on and off the solid state relays. The PID controller gives on/off signal to the solid state relays by cutting or bridging the DC power supply to the solid state relays. The rest of the system are parts of the latching relay circuit which is also connected to the emergency button and the pressure switch as seen in the diagram 3-19.

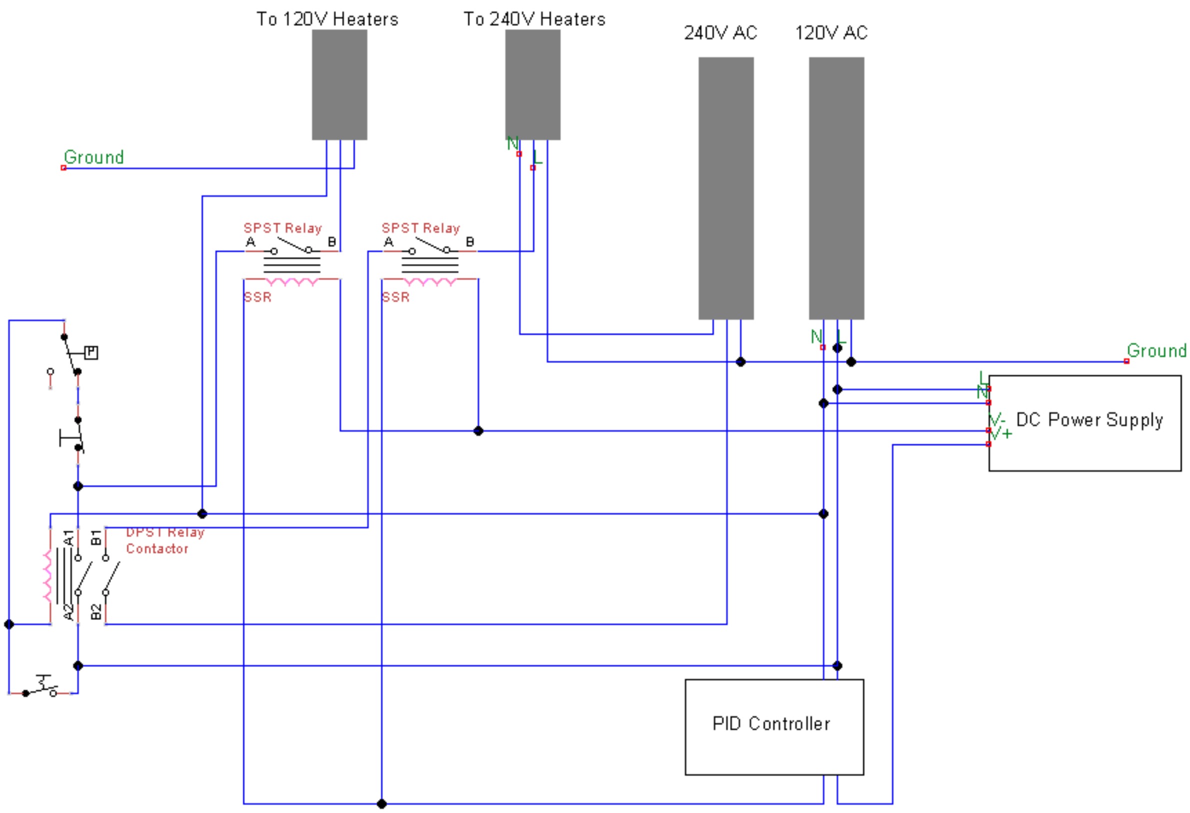

Figure 3-19: This figure illustrates the electric wiring diagram of heating tape controller system box.

3.7.2.3 Heating Tape Controller System Wiring Diagram

For the autoclave heater controller wiring diagram, see Cormet Oy's autoclave documentation.

3.8 Safety Systems

Running any system with the capability to get to the conditions similar to that of PWR is dangerous. In the CRUD loop, multiple redundant safety systems exist, with the primary task to ensure that no one operating the system will get hurt. Additionally, some safety systems are there to ensure that there is no equipment damage in an unexpected circumstances. The figure 3-20 shows the diagram of the safety systems within the CRUD loop. The subsections below will delve into the details of the safety systems in the CRUD loop.

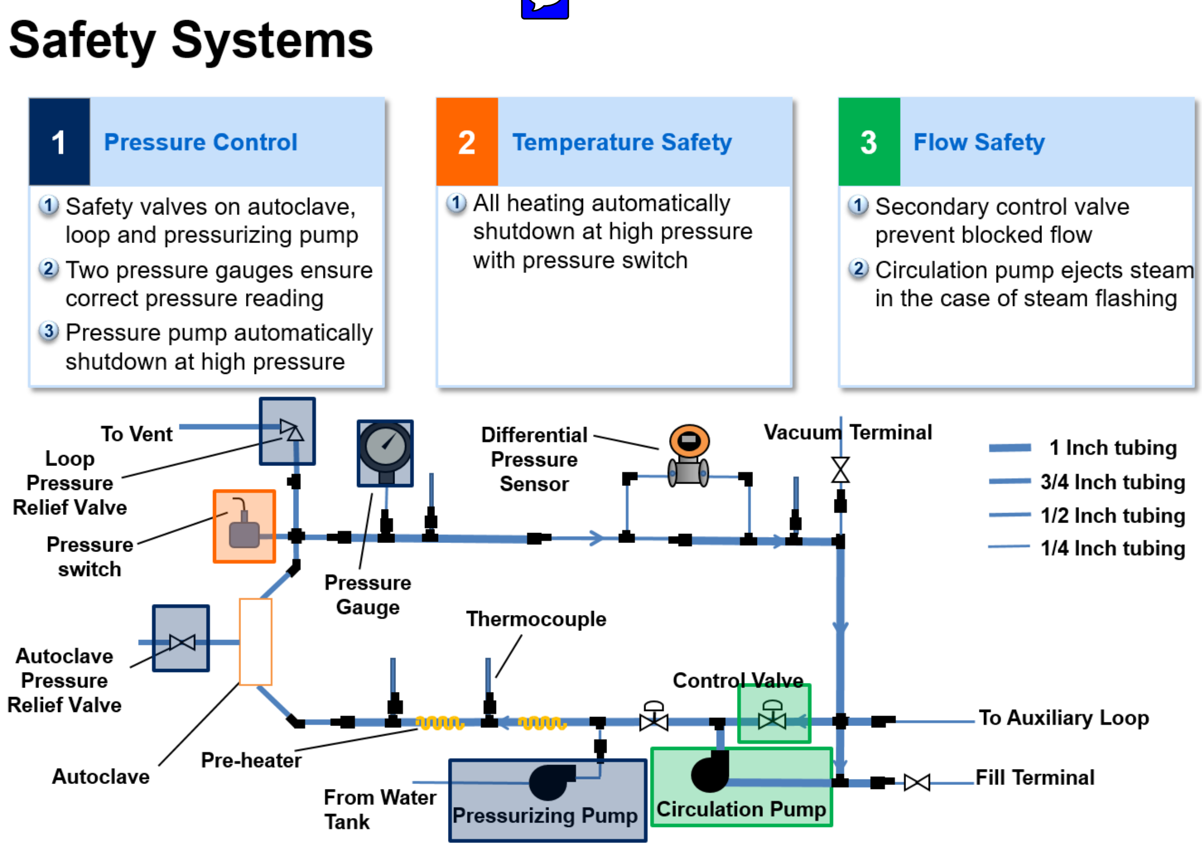

Figure 3-20: This is the diagram of the safety system elements within the loop.

3.8.1 Safety Relief Valves

There are multiple redundant safety valves within the system so that at least one should be functional in case other valves failed. There are three safety valves. The first one is attached directly to the autoclave. The second one is attached to the top part of the loop. The third valve is internal to the pressurizing pump. The safety relief valve on the loop will be release once pressure reach 3,000 psi. The pressure relief valve attached to the autoclave will also release at 3,000 psi. The internal pressure relief valve in the pressurizing pump will release at 170 bar.

3.8.2 Pressure switch

The pressure switch will shut down the electrical system once its set point is reached. The pressure switch's set point can be adjusted by opening up its covering and adjusting the bolt within. The latest adjustment of pressure switch will triggers around 2,500 psi. At that point it will cut the power to the pressurizing pump and the pressure should not increase further to trigger the safety valves.

3.8.3 Closed Room

The system is placed in its own specialized room to prevent any danger it might pose to other lab members. The room is equipped with a specialized tubings connected to safety valve that will shoot steam straight to a vent on building's ceiling in the case emergency safety valves opens. When the loop is running, this room will be locked with a danger sign posted on the door.

3.8.4 Polycarbonate shield

The loop also has a polycarbonate shield placed around the control computer to prevent any danger from the loop in case of steam leak or flying object caused by a pressure release. Polycarbonate is the same material that is used in security industry for bulletproof purposes, so it is highly unlikely for any flying object to penetrate it. Even with the polycarbonate shields around the workstation, it is not recommended to stay in the room while the loop is running.

3.8.5 Electrical System

All the electrical plugs in the room are connected to the breaker. Therefore, in the case of short circuit, the breaker will automatically shut down the whole system preventing the damage to the circuit or a nearby human. The autoclave control box also comes with its own internal breaker and two fuses for this purpose. Nevertheless, the loop operator must be sure to unplug all equipment when working with any of the control boxes' internals.

3.9 Operation of the Loop

Now that most information regarding how the loop works, and what each pieces of equipment does exactly, was presented, this section will pull everything together and set out a procedure on how to operate the loop. Through our experiences operating the loop, we have found many nuances that contributes toward operating the loop smoothly. Most of which are present in the following sections.

3.9.1 Precautions before operating the loop

Before diving in, to manipulate anything in the loop, always check that all the heating systems and the pressure systems are properly closed. This includes not just checking the computer screen, but also all the gauges available as the computer may have error. All plugs to the wall except that of the computer should be unplugged. Other than this, also be sure to wear proper protections when working with chemicals related to the loop. Nickel oxide particles used as the CRUD precursor are carcinogens and exposure to them should be very limited. The ceramic wool insulation dust can cause lung complications.

3.9.2 Preparing the Water

To imitate the water chemistry within nuclear reactors, there are several steps that has to be taken in preparing the water. It starts with filling up the water tank and deionizing it, then removing its dissolved oxygen. This is done because, before adding any chemical into the system, we need to have very pure water that must be properly deionized with dissolved oxygen removed. After water has been cleaned, boric acid, lithium hydroxide and nickel oxide CRUD particles can be added.

3.9.2.1 Filling Up the Water Tank

Before filling up the water tank, make sure that all the leftover water is drained. The tap water can be used to fill the tank since we have the mixed-bed ion exchanger attached to the water tank. To fill the tank, remove the lid of the water tank. And fill the water tank using any type of water container available. The usual way the loop is filled is by using a plastic tank to transfer water from the main ~~Professor Short's blue~~ lab.

3.9.2.2 Deionizing the water

The deionizing loop in the back of the auxiliary loop board is used to deionize the water in the loop and bring the conductivity down before any additive chemical are added. To deionize the water, turn the valves on the deionization loop so that water is redirected into the deionization columns. Then turn on the mini pumps on the control program. The deionization loop pump will drive the water through the deionization column where the ion-exchange beads will remove ions from the water. The ideal amount for conductivity before adding other chemicals should be around 0.09 μS/cm . This may take up to two days to complete depending on the conditions of the ion-exchanger columns.

3.9.2.3 Removing Oxygen

In the CRUD loop, the target dissolved oxygen is less than 5 ppb similar to the reactor. The oxygen can be removed by bubbling argon into the tank. The connections on top of water tank provides a way to connect to argon tank for this purpose. To bubble argon, simply connect the tube fitting from the argon tank into middle connection on top of the water tank. Make sure that another valve on top of the water tank is open to allow for gas outlet.

To remove the dissolved Oxygen, first, make sure that the argon tank is hooked up to the water tank's bubbler-port which is at the center of the water tank lid. Then check if the exhaust gas port, which is the port on the side of the water tank lid with valve attached, is properly connected to the gas exhaust system. To let gas bubbles into the water tank, slowly turn on the gas valve, first by turning the knob on the gas regulator, so the knob moves outward. When the knob moved out to the point where it almost gets detached, we can start to open the gas tank knob very slowly. After the pressure on the high-pressure reader jumped up, stop turning the gas tank knob, and start to turn the gas regulator knob slowly, so it moves inward toward the gas regulator body. At some point, the gas exhaust flasks will start bubbling. Usually, there is a delay between when the knob is turned and when the flask bubbles so turn a little at a time and wait for 30 seconds or so each turn. When the flask bubbles very slowly, turn the knob back out a bit. The best point the regulator knob should be at to save the most gas is where gas bubbles once in a long time, and even without the bubbles, the dissolved oxygen level does come down. One trick to adjust the flow rate of the argon gas is by using the valve at the water tank lid's center. This valve will give a much better precision than using the gas regulator valve. The markings on the valve can also be used as guidance to get the right flow amount.

Next, we want to redirect the water from the water tank into measurement system so we can get the dissolved oxygen measurement and see it decrease to the point we want. Make sure all the manual valves that allow the water from water tank through the measurement equipment are open. Then redirect the valve just before the measurement equipment to let the water flow from the water tank to the measurement equipment. When done, open the measurement pump from the computer user interface to pump the water from the water tank. Now the measurement of water properties in the water tank can be read from the computer user interface. Wait as the dissolved oxygen concentration lowers to the proper amount. When the dissolved oxygen is the proper amount, the gas tank valve and the gas tank regulator valve should be closed. Then disconnect the tube fittings from the top of the water tank and reconnect the fitting to the connection it was at before it was used for oxygen removal.

3.9.2.4 Adding CRUD Particles, Boric Acid and Lithium Hydroxide

Once the oxygen and ions in the water have been removed, the CRUD particles, the boric acid, and the lithium hydroxide can be added. Usually, 20 µg/l CRUD particles are added. The calculation of how many grams of CRUD particles that should be added, is done by using basic unit conversions combined with the fact that the water at the full tank is around 120 liters. Note that the calculation should also be adjusted for the density of water which will decrease a good amount going from 25 ºC to 310-320 ºC.

The boric acid is added to the system to simulate the reactor condition. In the commercial PWR, boron is added to control the number of neutrons. For our experiments we use 1400 ppm of boron for the reasons explained in "pH Measurement" section 3.5.4. The lithium hydroxide is added to partially neutralize the boric acid both in the reactor and in the CRUD loop. For the CRUD loop, 3 ppm of lithium hydroxide is usually added. The chemical injection port on the top is the port sealed with rubber. Injecting the solutions through the rubber with a syringe is the easiest way to add the chemicals. Another alternative approach is to remove the small rubber seal as well, for an easier injection. A slower alternative approach is to remove the top of the tank and pour in the solution of nickel oxides CRUD particles, boric acid, and lithium hydroxide.

3.9.3 Top flange and heating rod assembly Insertion

To insert the top flange and the heating rod assembly, slowly lowers the heating rod down the autoclave test section without letting the heating rod touch the autoclave or at least touch as little as possible. Then, place the cylinder that is covering the heating rod into its hole, holding the heating rod in place. Adjust the graphite gasket on to its spot, then lower the whole top flange assembly down to make it sits on the graphite gasket. After that make sure all of the following procedures are carried out to make the autoclave ready for operation:

- All six large bolts must be tightened. When tightening the large bolts, tighten them a bit a time and go around in a circle. If one bolt is tightened very far in while other bolts are still quite loose, there will be an imbalance in load on the soft graphite gasket which can damage the gasket.
- The top plastic cap's bolt must be tightened.
- The top cap holding the heating rod in should be screwed into place and tightened.
- The cooling line to the top flange must be connected.
- Make sure that the thermocouple line and the heating rod power line is plugged

3.9.4 Filling Up the Loop

It is crucial that after the loop is filled, there should be little to no air inside the loop. Having even a little amount of air inside the loop cause the loop to pressurize very slowly. Two ways that were used to achieve this includes using the vacuum pump to get rid of the air and using the gas to push the water from the tank out through the loop to replace air in the loop with water. The vacuum pump method is preferred over the gas method since it requires less work and is usually better at making sure that there is no air pocket in the loop.

Filling the loop with the vacuum pump method For the vacuum pump method, starts with making sure that there is no water left in the loop that will damage the vacuum pump. Then, close all the loop drain valves as well as the valve connecting the loop to the water tank. After that the connect tubings from vacuum pump to the valve on top of the loop near the water tank. Open that valve, and vacuum the loop for at least 20 minutes to clear up any air inside. After that close the valve to the vacuum pump and open the valve leading to the water tank to let the water fill the loop for around 5 minutes. If there is no leak, the loop should be filled with water without any air gap. The valve connecting the water tank directly to the loop can then be closed.

When first starting up the vacuum pump by plugging it into the wall, it is recommended to stand back in the case that pump has accidently been filled with some water. In that case, the pump may splash out a huge amount of oil. If the pump does not turn on when plugged into the wall, try resetting it with the red button on the back of the pump. One trick that works quite well with when using the vacuum pump method is to turn the black valve near the sample cooler, so the flow toward the back-pressure regulator is blocked. Doing this will prevent the water from rushing in through the back-pressure regulator when doing the vacuuming. If this black valve leading to sample cooler was closed for vacuuming, make sure that it becomes open when pressurization starts, or it will cause a pressure overrun.

Filling the loop with the argon gas method For the gas method, starts by connecting the tube from argon tank to the inlet near the side of the water tank. Then on the CRUD loop, open the top valve near to the water tank as well as the valve connecting to the water tank to let the water fill up the loop. Adjust the gas tank valve to pressurize the water tank, so the water is pushed out to replace air and fill the whole loop. When the loop is filled, and water came out of the top valve, close the top valve. Then go through each connection to the equipment that may have an air gap in them and release its nut to let water push~~es~~ all the air out.

3.9.5 Pressurizing

3.9.5.1 Conditions required

Before pressurizing, make sure that all connections are tight and all the equipment are operational. Other than that, check to make sure that all the valve positions lie in the proper spots. The following figure 3-21 show which valves should be checked before commencing pressurization procedures. Other than the valves from the figure 3-21, if the black three-way valve 3-22 between the sample cooler and back-pressure regulator was closed for vacuuming, make sure that it is open. Otherwise, there will be pressure overrun.

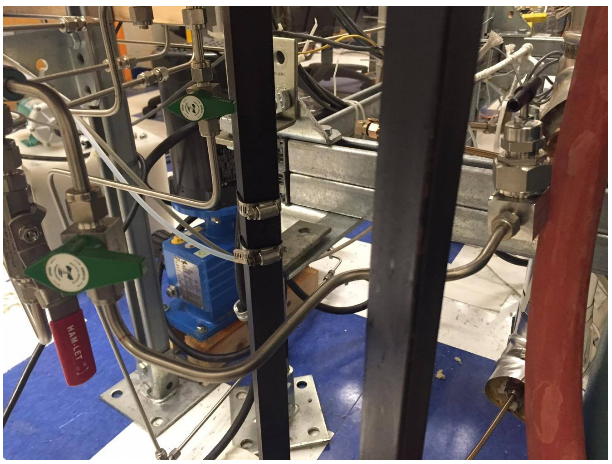

Figure 3-21: This photo shows the two green valves and the fill valve that should be checked before pressurizing. The nearer green valve on the left and the black needle valve on the right should be properly closed when pressurizing. Moreover, the farther green valve in the middle going to the pressurizing pump must be open for flow. Otherwise, the pressurizing pump may be starved of input water leading to damage.

Next, make sure all the pressure readings are working correctly. There are four places on the CRUD loop where the pressure can be monitored, which includes the two pressure gauges and the two pressure transmitters. The two pressure gauges include one on the top mid section of the main loop and another on the auxiliary loop. The pressure transmitter data can be read from both the autoclave controller for the autoclave pressure transmitter or the computer. The redundant measurements are done to make sure that the whole loop is getting pressurized, as well as for the pressure reading comparison.

3.9.5.2 Back-pressure regulator valve position

In the case that the back-pressure regulator's position is unknown. Turn the back-pressure regulator out all the way down to a low pressure for safety. In the case that the back-pressure regulator valve remains unchanged from the last experiment and the current experiment requires the same condition, there is no need to adjust the back-pressure regulator during start-ups.

Figure 3-22: This photo shows the black valve connected to the sample cooler. The valve can be closed in the position shown in this picture.

3.9.5.3 How to begin pressurizing

After all of the necessary conditions are met, the pressurization can commence. Simply go to the control program and click the pressure pump button to turn it on. If everything goes well, the pressure displayed on the autoclave heater controller box, the computer screen, and the gauge will begin rising. It is recommended to look at the autoclave's heater controller box's panel as that usually gives the quickest and the most accurate reading of pressure.

If the back-pressure regulator was set to the lowest pressure, slowly turn up the pressure, then wait for the actual pressure to catch up. Repeat this process until the pressure reached the desired point. Otherwise, if the back-pressure regulator was left at the position from the last experiment, watch closely as the pressure rise with the mouse on the pressure pump button on the control program ready to turn the pump off if there is pressure overrun.

3.9.5.4 Start data acquisition

Data acquisition can commence once the loop has been pressurized. To start on data acquisition, put in 15 minutes or any other save interval preferred in save interval box, then press the "Start Saving Data" button. At the end of the experimental run, click on "Finish Saving Data" to stop the data reading interval and record the data to the file.

3.9.6 Flow control

After the loop is pressurized, the flow should be turned on before the loop heating starts. Turning on the flow will distribute the heat more evenly across the loop. Then, adjust the control valve to around 85% close and turn on the circulation pump. Adjust the control valve opening until the differential pressure regulator reads the desired value. The control valves' openings can be adjusted via its PID controllers on the auxiliary loop. In a manual control mode, the higher percent on the control screen corresponds to a smaller control valve opening.

3.9.7 Heating the system

Once the water inside CRUD loop has been pressurized to the required point, and the flow in the loop is stable, the loop will be ready for the heating. Heating the loop to the PWR conditions requires both the heating from the autoclave and the heating from the heater tapes. This section will give guidance on how to heat up the system. It will provide information on how to operate both the autoclave heater rod controller, as well as the heater tapes controller.

3.9.7.1 How to operate autoclave heating controller

The autoclave heating system that is used to control the autoclave has three mounted controllers, one for the heating element temperature, one for the liquid temperature and one for the pressure. The heating element temperature is the value that will be used as the main control parameter. The others can only be used to set a limit to the pressure and the temperature. The following paragraphs describe the basic functions needed to control the West 6100+. It provides just enough information to get started on using the West 6100+ control unit. For detailed information, the West 6100+ documentation, available online, has an extensive list of what could be done with it.

There are four buttons from the left to the right of the controller panels including the auto/manual, the down, the up and the return button. These buttons can be used in a combination to achieve more functionalities. The default screen will display the current temperature on top and the set point temperature below. This default screen is known as the operation mode. The first button auto/manual allows the user to switch between the manual and the automatic control. In the manual control mode, the power is set to an exact percentage. In the automatic control mode, the controller will automatically adjust the power output percentage so that the temperature will reach the set point established by the human operator. In the CRUD loop's case, the automatic control is always used. Always be very cautious about switching to manual control. When switching to manual mode, the controller will maintain its power output. Therefore, when the power is 100% during the automatic mode, the 100% power trend will continue, which could quickly lead to film boiling and heater burn out in a low-pressure setting. The up and down buttons are for the adjustment of the current adjustable number displayed on the screen.

In any mode, pushing the return button will allow the user to navigate the functionalities within the particular mode. In the operation mode, the return button will allow the user to investigate and set the target temperature set point (SP), the current temperature set point (SPrP), and the temperature ramp point (rP). A human operator can adjust these set points. In automatic mode, the controller will adjust the power so that the temperature will reach the set point. Once the target set point is changed, the current set point that the controller is trying to match will not become the new target set point right away. It will slowly ramp up to target set point according to the speed set by ramp point (rP). Speed set by ramp point is in the unit of degree Celsius per hour.

The mode menu can be accessed by pushing the up button and the return button at the same time, then using the up/down button to navigate the menu to see other modes. Pushing the return button on any mode will swap the controller into that mode. Moreover, from within any mode, pushing the up button and the return button at the same time will take the operator to the mode menu. The menu will start with OPtr which means operation mode, the home mode of the controller. Other modes include the setup mode (SEtP), the configuration mode (ConF), the information mode (inFo) and the auto-tune mode (Atun). For most usages, the operation mode and the setup mode are the most useful. The operation mode is the default display mode, while the setup mode can be used to adjust the PID parameters. To find out more about adjusting the PID controller for proper parameters in the case of manual adjustment, consult the documentation of West6100+ control unit.

3.9.7.2 How to operate loop heaters

The Omega CN9000 temperature controller controls all of the heating tapes. Since the temperature difference across the loop is not very high, and we are operating the heaters at far below its maximum temperature, we use only one temperature controller to control all the heaters in the loop for simplicity. The first heating system is connected to a latching relay. Connecting the system to the latching relay means that if the system was previously shut off, the latching relay button, on the same control box as the Omega CN9000 temperature controller, must be manually switched on by a human operator for the heating system to work again. Before trying to turn on the latching relay, also make sure that the red emergency button is not in off mode. If the latching relay system successfully turned on, a clicking sound will be heard once when the latching relay button is pressed down. If there were two clicking sounds when the latching relay button was pressed, and when the latching relay button was released, it is likely that the emergency button is still in an off mode or the circuit had been damaged. Once the latching relay is properly turned on, the Omega CN9000 temperature controller can be used to control the output of all the heating tapes on the loop.

The controller is also attached to the dual junction thermocouple on top of the CRUD loop near the autoclave. Knowing the temperature of only one part of the loop is enough for temperature control in this case because the temperature difference across the whole CRUD loop with water flowing in normal circumstances, do not exceed 2-3 degree Celsius. The Omega CN9000 temperature controller does not do variable control heating. It will only turn the heater on/off according to the PID functions programmed into it. As a result, the heating tape temperature will fluctuate significantly. The temperature of the loop heating tapes can be adjusted manually on the CN9000 temperature controller. By pressing the star key on the Omega CN9000, the current temperature set point will show up. By pressing both the star key and up/down key on the Omega CN9000, the temperature set point can be adjusted.

3.9.7.3 Heating up the system

When heating up the system, always make sure that the temperature does not rise too fast. When the temperature rises too quickly, the thermal expansion of connections anywhere in the loop, especially in the autoclave, could expand at a different rate causing a leakage. To achieve the slow temperature rise, the operator can use the autoclave heating rod with a variable heat adjustment to heat up the system in the beginning until the temperature starts to plateau. When that happens, turn on the heating tapes, and set a new target temperature for the autoclave heating rod. Let the system continue~~s~~ heating until the required point.

3.9.8 Cooling the system

When cooling down the system, make sure that system is not cooled down too quickly. Powering down the temperature ramp is limited by the possibility of the water liquid flashing and leaking from an unequal thermal contraction in the equipment. When the water is cooled down too rapidly, and the pressurizing pump cannot keep up, the water in the system can flash to steam which can potentially damage to the circulation pump. The circulation pump has its steam vent to protect itself. This steam vent will shoot out steam in the case of flashing. Cooling down too rapidly will also cause some equipment to contract faster than others. This different amount of contraction can lead to a leak, or even worse, a broken Xpando seal which is tough to fix. To cool the system down slowly, start with decreasing the temperature on the Autoclave control box since the autoclave has a variable heat resistor. The usual speed the loop is powered down is at 200 ºC per hour.

3.9.9 Depressurizing the system

Once the system has been cooled below 100 ºC, it can be depressurized. Depressurizing the system involves just shutting down the pressurizing pump from the control program screen and waiting for the pressure to come down. Even without adjusting the back-pressure regulator, the pressure will usually come down on its own. After the loop has been depressurized, do not drain the loop until the water hits around 40 ºC to avoid any hot water splash burn.

3.9.10 Draining the loop

The loop contains two valves on the bottom of the loop for draining water, as well as a Swagelok cap that can be taken off to let the water out. These two drain valves and the cap are shown in the figure 3-23 below.

When draining the loop, be sure to close all the valves leading to the loop to prevent the water from rushing in from the water tank. These valves to the water tank include the valve leading to pressurizing pump and the valve between the sample cooler and the back-pressure regulator. Those valves are shown in the following figure 3-24. After all the valves are in the right position, some parts of the loop must also be disconnected to let the air inside the loop. The input air will replace water allowing more water to be drained. This way the more water will easily drain out. The parts to disconnect to let air into the system includes the quarter-inch tubing line connecting to the pressure switch, the loop safety relief valve, and the differential pressure transmitter.

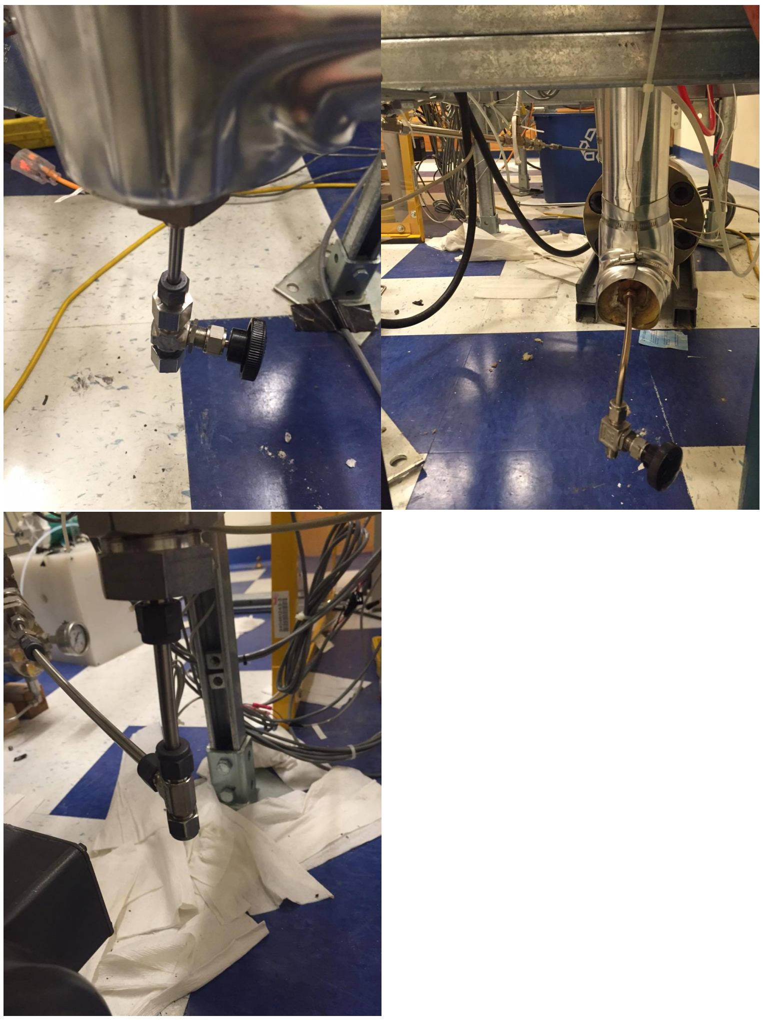

Figure 3-23: These three photos show the ports that must be opened for all the water in the loop to be drained. The first valve is the valve on the bottom of the loop near the autoclave which should drain the water from the autoclave and the tubings near it. The second valve is used to drain the circulation pump and the tubings near it. The cap is used to drain the water near the pressurizing pump.

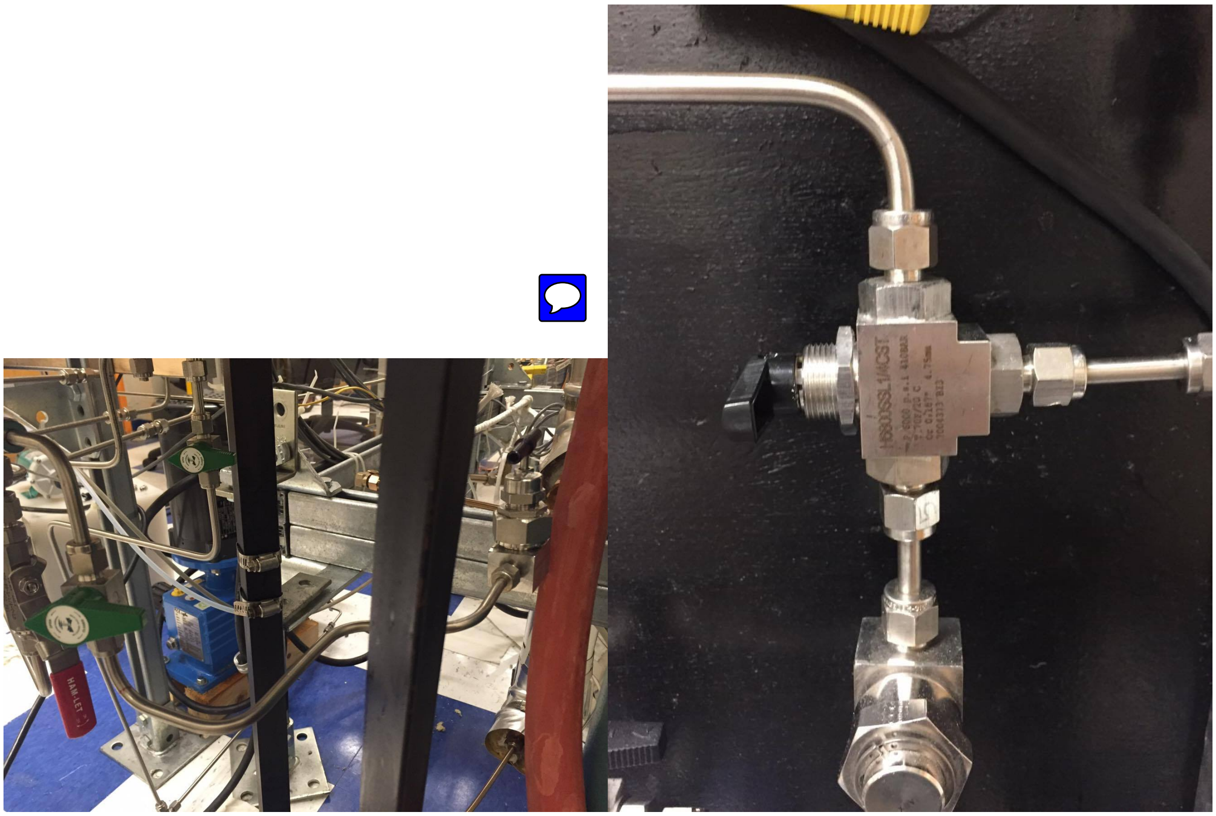

Figure 3-24: These two photos show the valves that should be close during the draining to avoid water rushing in from the water tank. The first picture shows the three valves which should be closed. The closer green valve and the black needle valve are on the fill line and lead directly to the water tank. Therefore it should be closed. The farther green valve connects the water tank to the pressurizing pump. This valve should be closed as well since the water can seep through the pressurizing pump into the loop.

3.9.11 Top flange and heating rod assembly removal

Before pulling off and separating the top flange assembly from the rest of the autoclave, make sure to do the following:

- All of the six large bolts on the autoclave must be loosened. When loosening the large bolts, loosen them a bit a time and go around in a circle. If one bolt is loosened too far out while other bolts are still quite loose, there will be an imbalance in load on soft graphite gasket which can damage the gasket.
- The cooling line to the top flange must be disconnected.
- The thermocouple line and the heating rod power line should be disconnected

After these steps are done, top flange assembly will be ready to be separated. To separate the top flange assembly, slowly pull it vertically without having the heating rod touching the autoclave wall. Do this as slowly and carefully as possible to prevent damage to samples.

3.9.12 Safety Precautions in During Loop Operation

Always stay behind the polycarbonate shield when operating to loop to avoid any danger in the case of any accident. Always keep the emergency stop button on the table near you in the case the loop needs to be stopped right away. Avoid being in the CRUD loop room during loop operation. Always monitor the coolant flow rate using the flow meter attached to the autoclave. Shut down the loop if there is not enough coolant flow.

3.10 Maintenance of the Loop

There is equipment in the loop that will need maintenance replacement from time to time. For most equipment, a maintenance procedure is well written within its documentation. However, there are a few maintenance procedures that are not in the documentation. The following section explains those maintenance procedures for the loop.

3.10.1 Replacing autoclave windows

Window replacement can be done by first removing the outer window casing. Then, using the puller tool provided to screw into the window assembly. Once the puller is in place, apply just enough force to pull out the inner window casing. To reinsert the window casing simply push the window assembly into its spot. After that, the outer casing can be tightened into place. When tightening the window casing, first do it by hand. This is to prevent cross threading. Then tighten properly by applying the torque of 100 Nm with a torque wrench.

Note that great care must be taken when removing and inserting the outer casing of the window. Always check to make sure the thread is in the right spot before starting to apply the torque on the wrench. If the torque is applied inappropriately and the cross threading happens, it will take a lot of work to fix, especially if the damaged thread is on the autoclave itself. The recommended way of fixing cross threading is by grinding the crossed part down so the thread can slide in again. The sapphire window assembly may break during removal so the windows should be removed only when necessary.

3.10.2 Replacing argon tank

When argon tank runs out, it can be detached by removing the gas regulator and its holder. After removing the connections, roll the gas tank out towards a gas cart. Used argon tanks must be transferred to the building NW13's docking area for disposal. To replace argon tank, simple latch the holder on it and thread in the gas regulator.

3.10.3 Replacing vacuum pump oil

The vacuum pump oil can be replaced, simply by draining the oil from the bottom of the pump into a disposal container. Then pour the new oil in from the top input opening. Fill the oil to the marking on the side of the vacuum pump.

3.10.4 Replacing ion-exchange column

After a while of use, the ion-exchange column will start to become less effective in filtering ions from water. The symptom of this happening is that it may take as long as two days to clean up ions within the water. To replace the ion-exchange column, first, close down the two valves leading to the ion-exchange columns. Doing so prevents the water from gushing out during replacement. Then, use the white plastic ring given with the ion-exchange column to screw out the ion-exchange column holder. Then replace the ion-exchange resin column inside with a new column. After that, proceed to put the whole ion exchange column and its holder back in the same place with the help of white plastic ring.

3.11 Troubleshooting

There are many cases where there will be issues that prevent the operation of the loop. This section here present common problems, how to check for them and suggest ways to fix them. Consult documentations for the specific piece of equipment if the common fixes did not work.

3.11.1 Troubleshooting in the case pressurization is not achieved

There are many cases where there will be issues that prevent the operation of the loop. Most of the time, similar problems occur. This section presents these common problems, how to check for them and suggest ways of fixing them. Consult the documentations for a specific piece of equipment if these common fixes do not work.

Wrong valve position In the case that some of the valves are not properly turned, the loop will not pressurize. The valves to check includes the valve letting the water from the water tank into the pressurizing pump, and the needle valves on the fill line.

Needle valve leakage The needle valve leakage is another common problem. The sign of this is water seeping out of the needle valve stem. In this case, the problem usually goes away after the valve stem is tightened. If the problem persists, new valve replacement is typically the best choice as they are not too expensive compared to manhour of fixing the valve.

Control valve stem leakage Control valve stem leakage could also happen. The sign of this is the water seeping out of the control valve stem. Usually, this problem can be addressed by tightening the valve stem. In a rare case, tightening the valve stem will not work, and the packing could cause the issue. Other than that, the O-ring might be broken. There are two options to solving these problems. One of the ways is to take apart the control valve according to the manual to check for damage in the packing or the o-ring. If either the packing or the O-ring is broken, replace the broken packing or O-ring with a new spare part. The second way is to ship it to the manufacturer to have it fixed.

Tube or pipe connection leakage There could be a leak in some tube or pipe connections. Running the pressurizing pump at a high flow rate for a while might reveal where the leak is occurring.

- If the leak comes from the tube connections, this issue can usually be fixed by tightening the nuts. Make sure that the nuts are not overtightened, or there is a risk of damaging the tubing seal which leads to a leak as well. In the case that a tubing connection is damaged, the tube section including nuts, and ferrules must be replaced.

- For a pipe connection, replacing the seal is the usual way to approach the problem. There are two types of seals that are applied on the pipes, the Teflon tape seal for low-temperature connections, and the Xpando seals for high-temperature connections. For the Teflon tape seal, the pipe connection can be undone and the Teflon tape will usually easily unwound. Replace the Teflon tape and put the pipe connection back again and the connection should work. For the Xpando seals, releasing the pipe connection must be done very carefully to avoid having broken Xpando debris fall into the tube. The connections can then be cleaned of the Xpando material using any available equipment that can scrape off the debris. In the case the Xpando debris falls into the tube, it can be removed either by vacuuming, removing with Kimwipe, or removing the whole three-way section to take the debris out. Once the connections are clean, mix the Xpando with water to get a thickness of a thin paste and apply them to the pipe male connection. After putting the pipe connection back together, Xpando will need 24 hours to dry before it can be used without leaking.

Autoclave window leakage A leakage could also occur at the autoclave windows. One possible test for this is first to close down the system and drain the whole loop. Then vacuum out the loop. If there is a leak at the window, you will be able to hear the wheezing sound from the window. Otherwise, if no sound is heard, it is also useful to plug the window with your palm to see if there is a leak which will suck your palm. If another person is present, you can also have another person look at the autoclave controller for you to see if the pressure changes when you plug the window with your palm. Usually, a leak in the autoclave window means that either a part of the window glass corroded away, or that the window is not properly tightened. In the case that tightening the window does not work, see the section "Replacing autoclave window" 3.10.1, for how to change the autoclave window.

Pressurizing pump problem The pressurizing pump could be a problem as well. One possible test for this issue is to replace the tubing going to the pressurizing pump with a short tubing leading to a pressure gauge. Once the replacement is done, try pressurizing a little to see if the pressure will go up with that small system. Be ready to click the off button for the pressurizing pump all the time. An extreme caution must be used when doing this test, as there is nothing to decrease the pressure in the small system, so it will be easy to get a pressure overrun without great care. If the pressurizing pump does not pressurize, it is recommended to send it manufacturer, unless the pump is already old at the time of reading.

3.11.2 The autoclave controller box remains shutdown

If the heater is not turned on at all, first check if the heater is properly supplied with power by examining different wires with a voltmeter. If the heater is properly supplied with power, the next most common cause is that there was previously a power surge. The following fixes work most of the time to combat this issue. If none of the following fixes work, it is likely that the heater burned out and must be replaced.

Turn on the internal breaker The autoclave controller comes with an internal breaker built-in that will turn off in the case of power surge. To activate the internal breaker again, first, check to make sure that power is not supplied to the autoclave heater controller. Then open the top cover by loosening the screws on the back of the heater controller. Once the heater controller's top is opened, the internal breaker switch is located near the heater controller front panel towards the right-hand side, when looking in from the front panel. Flick the switch and close the heater controller box properly before testing if the fix worked.

Replace the fuse If turning on the breaker switch does not work, another issue could be that the fuse was blown. On the back of the autoclave heater controller towards the right-hand side, there are two black rubber circles with a slotted screw head. Those parts can be unscrewed out to reach the fuses. Once the fuse is found, test its integrity by using a voltmeter to check the resistance across the fuse. If one fuse was found to be blown with infinite resistance, the operator should replace that fuse with a new one. The fuse should be the 500 Voltage and 16 Amps type. After the fuse is replaced, close up the heater controller box. Then, test if the fix worked.

3.11.3 Neslab heat exchanger stopped working

The two most common problems which made the Neslab heat exchanger stops working is that either the temperature of the coolant became too high or that its coolant level is too low.

Coolant level too low To determine if the coolant level is the issue, simply check the coolant level by opening to the top hatch leading to the coolant. If it is low, fill it up to almost full. Usually, if the coolant level became low not too long after the last change, it indicates that the loop might have a leak. In that case, it is recommended to go through all the connections to test for a leak as well.

Temperature level is too high The temperature of the coolant can be checked from the front of the Neslab heat exchanger. The most common reason for the rise in coolant line temperature is that the building's cooling line was shut down, and therefore there isn't a place to dump heat. To determine if the building's cooling line is the problem, check the incoming cooling line to see if it is cold. If the building's cooling line is hot, then you will have to wait for building's cooling line to be fixed.

3.11.4 Failure to get to desired temperature

If the temperature does not increase to the desired PWR temperature, even when all the heaters are running at full power, several issues might be the cause. The following three issues are the most common ones.

Leak in the system A leak in the loop will drain away its energy as heated water exits the loop. A symptom for a leak is that it is harder than usual to pressurize the system. Moreover, when the pressurizing pump is closed, the pressure falls quicker than usual. In the case that there is an unknown leak, you may have to go through all the connections in the loop to see which one is causing the problem. One way to find out where a leak that cannot be detected at low pressure is to pump the loop with very high flow rate from pressurizing pump. After a while, close the pressurizing pump and inspect the loop for any dripping.

Heating tape failure It could also be that some of the heating tapes failed. To check this, test the resistance of all heaters using their plugs after the loop had been shut down. If there is indeed a failed heating tape, replace it accordingly. When replacing a heating tape, wear a gas mask when removing the insulation covers. The ceramic wool heat insulator's dust can be damaging to the lungs.

Too much pressurizing pump flow rate If the pressurizing pump's stroke length is too high, the pressurizing pump may dump too much cold water into the loop. This influx of cold water will cool down the loop making it harder to increase the temperature. The easy fix for this is to turn down the pressurizing pump's stroke length. An ideal pressurizing pump stroke length is where the measurement loop measures around 60 ml/minute.

3.12 Example Raw Data Outputs from the Loop

If the data file is saved for the experiment run, there should be several text files that contain the data for the loop. Each of them is a dataset for a specific type of loop data output. The following figure is the excel file made from compiling the several text files together in different columns.

Pressure	Liquid Temperature	Element Temperature	Differential Pressure	Dissolved O2	pH	Conductivity	Temperature before cooler	Temperature after cooler	Temperature before preheater	Temperature after preheater
154.535	33.604	33.757	12.6	0.007	6.859	8.724	34.163	34.471	33.857	34.133
155.185	34.27	33.863	12.58	0.006	6.859	8.722	34.16	34.443	33.836	34.121
154.002	69.469	69.606	16.35	0.006	6.898	8.619	67.865	69.082	67.988	69.011
155.221	92.786	92.963	16.39	0.007	6.898	8.293	91.894	92.827	91.575	92.627
152.59	122.779	183.191	16.42	0.005	6.895	8.019	121.423	122.282	120.273	121.373
155.513	156.356	246.529	16.19	0.006	6.883	8.255	155.273	155.952	153.611	154.629
155.413	180.825	280.228	15.87	0.005	6.885	8.235	179.564	180.039	177.469	178.386
153.196	192.871	250.31	15.67	0.005	6.894	8.178	192.752	193.229	191.286	192.23
152.639	202.837	264.765	15.59	0.005	6.897	8.106	202.276	202.605	200.352	201.403
154.36	214.229	314.067	15.4	0.005	6.911	7.97	213.901	214.381	211.578	212.478
155.439	227.721	327.105	15.09	0.005	6.915	7.96	227.371	228.046	225.213	226.381
155.587	239.587	339.754	11.42	0.004	6.914	7.799	239.032	239.505	237.622	237.601
156.042	249.655	349.787	11.16	0.005	6.938	7.738	248.938	249.581	247.595	247.626
155.543	259.089	360.251	11.06	0.005	6.95	7.71	257.86	258.588	256.614	256.522
155.767	266.121	367.479	10.92	0.005	6.958	7.773	264.764	265.592	263.719	263.697
156.446	272.009	372.995	10.74	0.005	6.974	7.948	270.511	271.194	269.276	269.367

Figure 3-25: This table shows an example of raw data output, with data gathered every 15 minutes interval.

The following list gives a brief explanation of what each of these output columns represent:

- Pressure: This value represents the pressure of the whole system. It reads from the pressure transmitter near the back-pressure regulator.
- Liquid temperature: This value represents the liquid temperature in the autoclave as measured from the top of the test section within the autoclave.
- Element temperature: This value represents the temperature within the heating element measured by the thermocouple embedded in the center of the heating element.
- Differential pressure: This value represents the differential pressure from the differential pressure transmitter on top of the loop. This use useful for figuring out the flow rate.
- Dissolved Oxygen: This is the dissolved oxygen value measured by the measurement equipment cluster. It will measure either the dissolved oxygen content of the main loop or the water tank depending on the valve direction.
- pH: The pH measurement also measures from the measurement equipment cluster.
- Conductivity: The conductivity is another value from the measurement equipment cluster. It is useful for determining how much ions are flowing within the loop or the water tank.
- Temperature before cooler: Temperature from thermocouple on the top left side of the main loop
- Temperature after cooler: Temperature from thermocouple on the top right side of the main loop
- Temperature before preheater: Temperature from thermocouple on the lower right side of the main loop
- Temperature after preheater: Temperature from thermocouple on the lower left side of the main loop

Chapter 4

EXPERIMENTAL AND ANALYTICAL METHODOLOGY

4.1 Introduction

This chapter will present the information regarding the experiments that we ran on the MIT CRUD Loop. The experiment done is divided into two major sections, image analysis of CRUD, surface-chemistry modification for CRUD resistance. Although, in reality, each sections share growth trial, the experiment should be easier to understand when separated into actual goals. At the end of this chapter, summary section will explain the actual experimental matrix with each trials explained more clearly with all the goals in mind.

First part, the analysis methods, explains each types of methods that will be applied repeatedly when going through the whole experimental matrix. Most of the analysis will be obtained using scaning electron microscope (SEM) image and focused ion beam (FIB) processing.

Image analysis deals with characterizing CRUD using scanning electron microscope's image and different image processing methods. The most important parameters that will be obtained includes fractal dimension of CRUD, porosity of CRUD, CRUD particle size etc. Ultimately, there are two major goal of these analyses. First goal is to verify and provide more experimental information for MAMBA CRUD model developed by our group to simulate CRUD. Second goal is to find patterns or characteristics of CRUD that has never been looked at before, and use them to gain interesting insights into CRUD formation and fouling deposit formation as a whole.

The surface-chemistry modification of fuel cladding for CRUD resistance includes experiments conducted to verify if any surface-chemistry modification can lead to mitigation or prevention of CRUD. This part will explains the idea behind the surface-chemistry modification we are making and the method that will be used to coat and test them. Other than this, second part will also go