HØGSKOLEN I BUSKERUD OG VESTFOLD

SENSETION

EN-2015-01

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HØGSKOLEN I BUSKERUD OG VESTFOLD

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Sensor Interface
Microfluidic Flow Cell
Electrochemical Sensors

SAMMENDRAG

Zimmer & Peacock is currently developing an amperometric glucose sensor that can act as a reference instrument in a laboratory setting. They also have an electronic prototype system called eZ Sense, for reading the signal output of the sensors. The eZ Sense system consists of the MAS_V2_R2 board, used for interfacing with the sensor, and ZT GUI software to monitor sensor measurements.

SAMMENDRAG (forts.)

The current prototype can read only a single sensor, which limits the precision of the measurements due to individual sensor variations. Another challenge is noise introduced into the system which is suspected to come from the turbulent flow of fluid at the sensor interface as well as potential interfering electromagnetic fields coupled to the sensor electrodes.

The purpose of the Sensetion project is to design and build a multichannel laboratory instrument for sensor characterization, based on the eZ Sense technology, and to implement a microfluidic flow cell to minimize the noise characteristics of the system. The fluid flow in a microfluidic flow cell is laminar, which may reduce any noise caused by the turbulent flow of liquid at the sensor interface.

Through prototyping on breadboard, and PCB design and fabrication, the group was able to create an eight-channel instrument for sensor interfacing, accompanied by the necessary firmware and software to control the system, process sensor signals and present them on a computer in real-time.

The microfluidic flow cell was created through a series of laboratory exercises. Several designs were made before obtaining a suitable design for the system.

The laboratory instrument proved to perform as desired, with the ability to give a very visual presentation of sensor response through real-time graphing of sensor signals.

The microfluidic flow cell did not seem to have the expected effect of reducing the noise architecture. Further testing is necessary before making any definite conclusion on this subject.

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Preface

This thesis is written for Zimmer & Peacock.

The authors would first and foremost like to thank project supervisor Dr. Erik Johannesen for his guidance and invaluable support throughout the project. Furthermore they would like to thank Richard Krogstad and Maria K. Askim for their aid on logo design.

Mette Varegg wants to thank Svein Mindrebø for his help on the workshop and Zeikia for guidance and help in the cleanroom.

Torgrim and Sindre would like to thank M.Sc. Rune Langøy and Masters student Sindre Søpstad for their support on the Computer Code, and Tor Olaf Berger for allowing us to use the circuit board production equipment and for supplying the group with electronic components and equipment necessary for both the prototype and the final PCB.

HØGSKOLEN I BUSKERUD OG VESTFOLD

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Terms and Acronyms

CAD: Computer Aided Design
CE: Counter Electrode
GOX: Glucose Oxidase
GUI: Graphical User Interface
HBV: Buskerud and Vestfold University College
Hard Bake: Process for strengthening resist at temperatures < 200°C
I^2C: Inter-Integrated Circuit
LCD: Liquid Crystal Display
MFC: Microfluidic Flow Cell
Microns: Micrometre
PCB: Printed Circuit Board
PGA: Programmable gain Amplifier
RE: Reference Electrode
SCL: Serial Clock Line
SDA: Serial Data Line
Soft Bake: Process for strengthening resist at temperatures < 100°C
SPE: Solid Polymer Electrolyte
SU-8: Epoxy Based on Resin for Creating Microstructures
TIA: Transimpedance Amplifier
Vref: Voltage Reference
Wafer: Semiconductormaterial, often referred to as substrate
WE: Working Electrode
USCI: Universal Serial Communication Interface

1 Introduction

1.1 Background

The origin of the amperometric glucose enzyme electrode dates back to 1962. It was developed by Clarc and Lyons of the Cincinnati Children's Hospital [1]. The glucose enzyme electrode was then a device that relied on a layer of glucose oxidase (GOX) entrapped over an oxygen electrode via a semipermeable dialysis membrane (a membrane that will allow for certain molecules or ions to pass through it by diffusion). Then the Oxygen consumed by the enzyme-catalyzed reaction formed the basis of the measurements, i.e. the oxygen consumed in reaction (1) was measured.

$$glucose + oxygen \xrightarrow{glucose\ oxidase\ (GOX)} gluconic\ acid + H_2O_2 \qquad (1)$$

Amperometric enzyme electrodes based on glucose oxidase, have since that played a leading role towards simple and easy to use blood sugar testing, and represents the current state of the art [2].

Modern electrochemical sensors work by the three-electrode principle. The first electrode, the working electode (WE), represents the surface where the electrochemical process takes place. The second electrode, the reference electrode (RE) is made up of a material that does not polarize in an aqueous solution and therefore has a controlled defined reference potential to which the WE can be referred to. The third electrode, the auxiliar electrode (also referred to as the counter electrode (CE)) completes the circuit and channels the current needed to continue the reaction at the working electrode. By applying a fixed potential of the WE with respect to the RE, one can trigger an oxidation or reduction reaction from electroactive molecules and components that are present in the solution [3].

The amperometric glucose sensor is a three electrode electrochemical cell that oxidizes glucose into gluconolactone by the enzyme glucose oxidase (GO_X). This process leads to the consumption of O_2, and the production of H_2O_2 as a bi-product [4]. The current running through the auxiliary electrode is decided by the amount of glucose being oxidized and therefore the amount of H_2O_2 being produced. Depending on the applied electric potential at the WE, one can either oxidise the H_2O_2, or reduce the available O_2 to water. This will, in both cases, result in a current indirectly proportional to the glucose concentration [5].

Based on this detection principle, Zimmer & Peacock is now developing an amperometric glucose sensor that can act as a reference instrument in a laboratory setting. They also have an electronic prototype system called eZ Sense, for reading the signal output of the sensors. The eZ Sense system consists of the MAS_V2_R2 board, used for interfacing with the sensor, and ZT GUI software to monitor sensor measurements.

A challenge with the current prototype is that only a single sensor can be used at a time, which limits the precision of the measurements due to individual sensor variations. This is mainly due to differences in the solid polymer electrolyte (SPE) that is deposited on the sensors surface and which immobilizes the GO_X enzyme used for glucose detection. A second challenge is noise introduced into the system and which is suspected to come from the turbulent flow of fluid at the sensor interface as well as potential interfering electromagnetic fields coupled to the sensor electrodes. Finally, the goal for the future is to miniaturize the sensor in order to create an implantable system for continuous glucose monitoring in vivo. This last challenge is currently being considered by Mr. Sindre Søpstad, a master student at HBV, who is working on the miniaturization process of the SPE sensors in his master thesis.

Consequently it is desirable to build a reliable and efficient system for sensor testing, which introduces the idea of a laboratory instrument able to read multiple sensors in parallel

and to find a way to avoid the noise generated by the fluid flow or magnetic mixers used in the current setup. This challenge formed the basis of the work presented in this bachelor project thesis.

The current form of sensor measurements uses a stir bar in a glass of test solution. A magnetic mixer generates a rotating magnetic field, forcing the stir bar to spin. If the sensor in the solution picks up the electromagnetic field it is likely to generate some interference in the sensor measurements. The mixer does also produce turbulence in the solution that might be measured by the sensor as noise. A way of obtaining this uniform supply of glucose/O_2 is necessary for the accuracy of the measurements.

The fluid flow in a MFC is laminar, which may reduce any noise caused by the turbulent flow of liquid at the sensor interface in the current step. This is due to a more even supply of the glucose/O_2 to the sensor interface.

The MFC, along with a control system for a multi-channel sensor input would then make a good laboratory instrument for sensor characterization.
These ideas were the fundamental thoughts behind the research question for this project:

How can we develop a novel laboratory instrument based on the eZ Sense system, with multiple sensor channel support and minimal sensor noise architecture for the investigation of electrochemically active components?

This has been somewhat modified from the pre-project report, which can be found on the CD attached to the printed version of this thesis. The research question will be answered by designing the laboratory instrument with the necessary hardware and software, and by creating a MFC to minimalize the noise surrounding the electrochemical sensors.

1.2 Project Stages

The project were, as a process in the pre-project, divided into 5 stages:

- ∇ Pre-Project
- ∇ Design and assembly
- ∇ Testing
- ∇ Thesis
- ∇ Presentation

Each of these steps served as guidelines, where each stage were to succeed the previous stage, except in those stages where overlapping seemed more practical. This gave the group a complete overview of the project, and the different parts of the project.

1.2.1 Pre-Project

The pre-project was the initiating of the project. There was a thorough planning process where a research question was raised, and several project goals as well as intermediate goals were set. The pre-project was also used to create an intermediate plan, and several ideas for approaching an answer to the research question. The fragmentation of the project into the already mentioned stages also served as headlines in the Gantt diagram, found on the project CD.

Before the group could begin the tedious work of answering the research question, there were some fundamental rules that had to be set. The project group drafted a group contract,

which is also found on the project CD, and defined areas of responsibility. These areas were never meant as fixed work area, but more as an area over which each group member had the possibility to take charge, and somewhat control the workflow. However, in the microfluidic flow cell area, the work was somewhat more tied to the one person studying that topic; Mette Varegg.

1.2.2 Design and Assembly

The majority of resources was intended used on this stage of the project. The group used the preset split of tasks, and embarked on the problems. All work was done in parallel, and the group members, though separated by their main tasks, worked together as a team to solve the problems at hand. The group held weekly meetings, working as status reports, keeping all members up to date on the project as a whole.

1.2.3 Testing

Testing of the system is mainly described in the test and the result section. This process worked as a closure of the project, and was performed in parallel with the thesis.

1.2.4 Thesis

One stage of the project was the thesis itself. The time set of for this was the last two months of the project. However, the process of documenting the work done and writing reports along the project was a continuous work from the initializing of the project, until the very end.

1.2.5 Presentation

The project presentation is set to be in the form of a public presentation at the annual HBVExpo at HBV, followed by an oral examination.

1.3 About the Thesis

The Thesis is written as a scientific report, with introduction, followed by theory of the sensor principle and the microfluidic flow cell. Subsequent to the theory, the process of designing the microfluidic flow cell, prototype on breadboard, Printed Circuit Board (PCB) and hardware/software code is described in details. This is followed by section 2.5, which gives an overview of the tests performed. Section 3 covers the results, and section 4, the conclusion of the project

The project thesis is accompanied by a CD and a booklet, in order to relieve the main document and to give a more well-presented thesis.

1.4 About the CD

The project CD contains the following:

- ∇ Project description
- ∇ Pre-project report
- ∇ Project report
- ∇ Datasheets
- ∇ Circuit diagrams
- ∇ Design files

- ∇ Ez Sense code
- ∇ Sensetion code
- ∇ VersaSTAT program
- ∇ Solutions calculator
- ∇ Test results
- ∇ Raw data

1.5 About the Booklet

The Sensetion Project, Source Code is a booklet accompanying the project thesis. It contains all of the project's code for both microcontroller and computer software. For practical reasons, *The Sensetion Project, Source Code* will henceforth be referred to as *Booklet*.

2 Methodology

Due to the extent of the task, the methodology is divided into two main areas of focus; The MFC, and the Front-End Interface and control system. These areas, though they are somewhat dependent of each other, are separated in the process of designing the parts.

2.1 Microfluidic Flow Cell

Initial studies focused on how the design of the microfluidic system affects the fluid flow and how this could be optimized to facilitate the exchange of fluid in the measurement chamber where the sensor is located.

The flow rate is an important factor due to its effect on the sensor response, meaning the time it takes to exchange all the fluid in the measurement chamber. The relation between flow rate and sensor response is explained in 2.5.1.3. The flow rate depends on the following factors:

- ∇ Pressure given by the pump
- ∇ Properties of the fluid, like viscosity and density
- ∇ Properties of the surface in contact with the fluid, like hydrophilic/hydrophobic and roughness
- ∇ The cross section of the channels or tubes, where larger cross sections permits higher flow rate
- ∇ Length of the channels or tubes, the longer the more hydrodynamic resistance

The properties of the fluid is decided by the composition of the test solution used to characterize the sensors.

The PDMS gives the properties of the surface. PDMS is a material commonly used in MFC due to properties like low surface tension, making it highly hydrophobic. It is resistant to many factors like water, oxidation, aging, UV radiation, temperature extremes and many chemicals [6].

Another factor that influences the sensor response is the size and design of the measurement chamber. It will take more time to exchange all the liquid in a larger chamber, and the design may affect the fluid flow by creating stagnated zones where the liquid lies motionless or zones where the flow moves in the wrong direction.

The MFC consists of a system with inlets, outlets and a measurement chamber for the sensor to be tested. To manufacture the MFC, knowledge of the following fields is required:

- ∇ Microfluidics, fluid mechanics
- ∇ Computer aided design (CAD)
- ∇ Microfabrication

From the pre-project, these were the intermediate goals for the MFC:

- ∇ Complete calculations for channel shape and size using fluid mechanics
- ∇ Complete mask design in L-Edit
- ∇ Complete the creation of the MFC using a photolithography process
- ∇ Complete the design of a frame to attach the sensor to the MFC, sealing the measurement chamber
- ∇ Successful testing of the MFC in conjugation with the prototype laboratory instrument

The process of creating a MFC is illustrated in Figure 1, and starts with planning and sketching the designs for the MFCs, explained in 2.1.1. The designs are drawn in L-edit, a computer-aided design (CAD) program, described in 2.1.2 and sent to Infinite graphics Inc. (USA), a manufacturer of photomasks, to create a glass mask. The glass mask is used in the photolithography process to create the master; a structure of negative photoresist on a silicon wafer. This process is explained in 2.1.4.1. The PDMS casting is done by pouring the PDMS over the master, and in that way creating the micro system, covered in 2.1.4.2. Some MFC designs require plasma bonding to attach the PDMS to a glass slide, as described in 2.1.4.3. The MFC is placed in a holder created to assure an even pressure on the system, to avoid leakage, explained in 2.1.3.2.

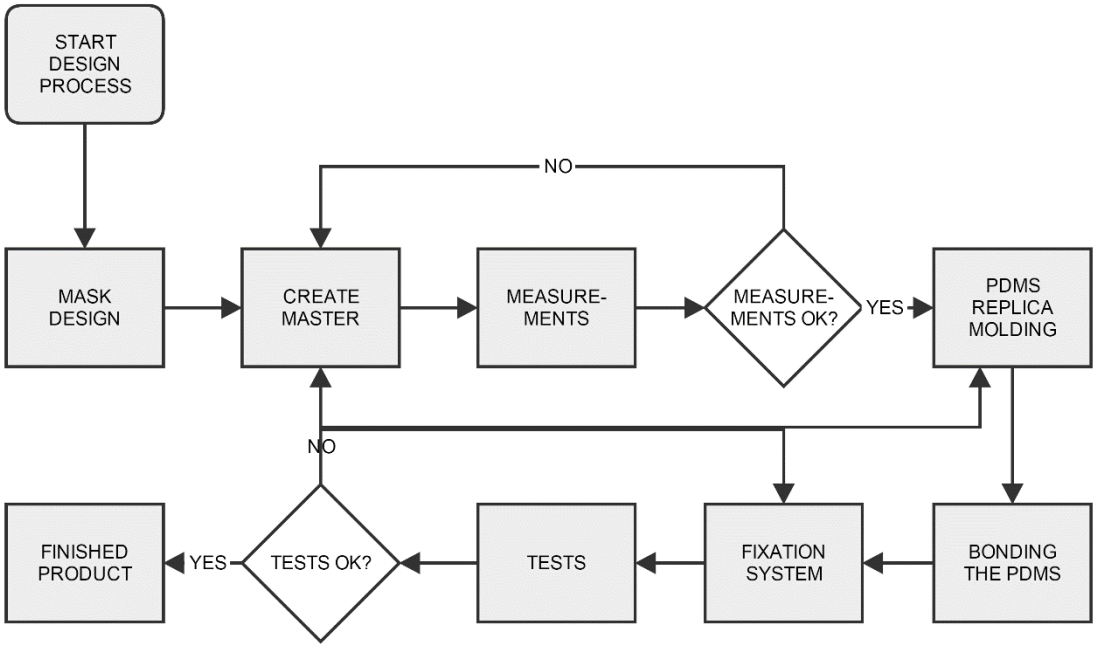

Figure 1. Flow diagram of the process of creating a microfluidic flow cell.

2.1.1 Design of the Microfluidic Flow Cell

The working principle of the MFC was discussed in the early stage of the pre project. At this moment the idea was to create a classic MFC using PDMS mounted on glass, with one inlet, one outlet and a measurement chamber in the center. How the pressure was to be applied was not yet decided at this point, but two of the options were using a siphon tube, or a peristaltic pump described in 2.5.1.

The glass slides were given the measure of a typical microscope slide used on HBV, 75mm x 25 mm x 12mm. The in-/outlets are made of silicone tubing with an inner diameter (ID) of 1 mm and an outer diameter (OD) of 3 mm. this was chosen due to being commonly used in fabrication of MFC at HBV. The measurement chamber needed to be larger than the sensing area of the sensor chip (8mm) but smaller than the chip's width (10mm), therefor 9mm was opted for.

The length and width will be decided by the size features of the photomask used to define the channel pattern.

The height will be decided by the manufacturing process of the master. The manufacturer of the negative photoresist provides a standard to create masters with 100µm thick structures. It should be possible to create thicker structures, by changing the parameters.

The size of the measurement chamber depends on the design of the photomask, the thickness of the photoresist structure, the glass slide and the gasket.

Once the MFC is fabricated, the sensor will be placed under the measurement chamber, sealed with a gasket and held in place by a holder to avoid leaks. The principle of the MFC is sketched in Figure 2 and Figure 3, design on the left.

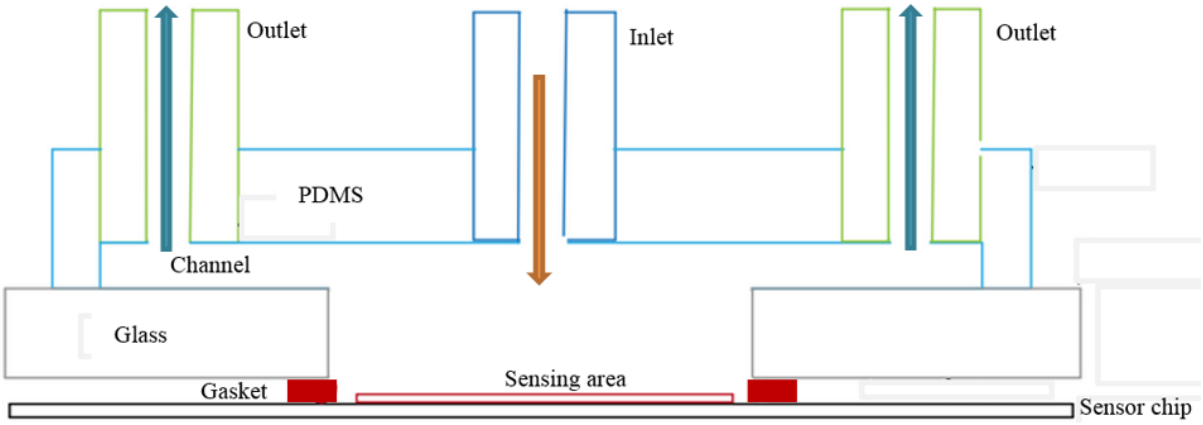

Figure 2. Sketch of the principle of MFC with the inlet in the center as described below.

It was decided to make one basic design as described above, and two versions based on the basic design but with some modifications as shown in Figure 3. The change done to the basic design was placing the inlet in the center of the measuring chamber to extend the fluid over the sensing area. By moving the inlet to the center, the system now has two outlets, increasing the flow rate through the MFC.

The second design had the inlet in the center and four outlets distributed evenly at 3 mm distance from the chamber. This was an attempt to create a good flow rate, and have the possibilities to change the direction of the current by blocking some of the outlets. That might be helpful to avoid bubbles getting stuck in the system. The measurements and shapes of the designs are illustrated in Figure 3.

The third design was the same principle as the first, only that there were no channels. The outlets were placed on each side of the inlet inside the measuring chamber. This should make the best possible flow rate due to not having channels limiting the fluid flow.

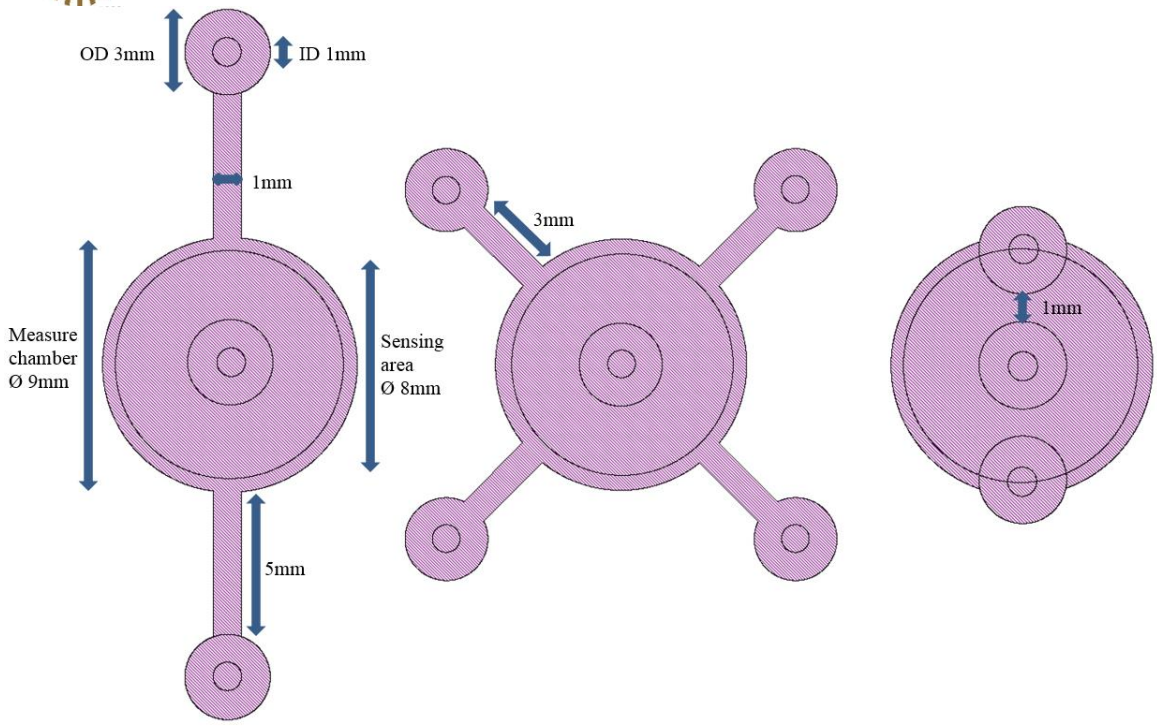

Figure 3. From left to right, design 1 to 3.

The classic designs are all based on the same principle. The fluid enters the measurement chamber in the center and has to flow up to exit through the outlets.

The sensor response is highly depending on the flowrate and the measurement chamber. This is covered in 2.5.1.3. This means that the greater the volume of the chamber, the higher flowrate needed to exchange all the fluid. Therefore, it was decided to make the fourth MFC based on a different principle. The working principles of the fourth design is that the fluid enters in the center of the sensing area, flows over the chip and out through the outlets on each side of the chip as shown in Figure 4.

The design consists of one PDMS castings creating the top layer, and one creating the bottom layer. The top layer will have the inlet and a chamber wider than the chip, so that the fluid can flow off the chip on both sides. The bottom layer needs some kind of slot of comparable thickness of the chip to eliminate part of the stress on the sensor chip, as well as the outlets. This way, the PDMS will work as a gasket. The advantages of this design is the lower volume due to no need for gasket or glass. No channels are limiting the flow.

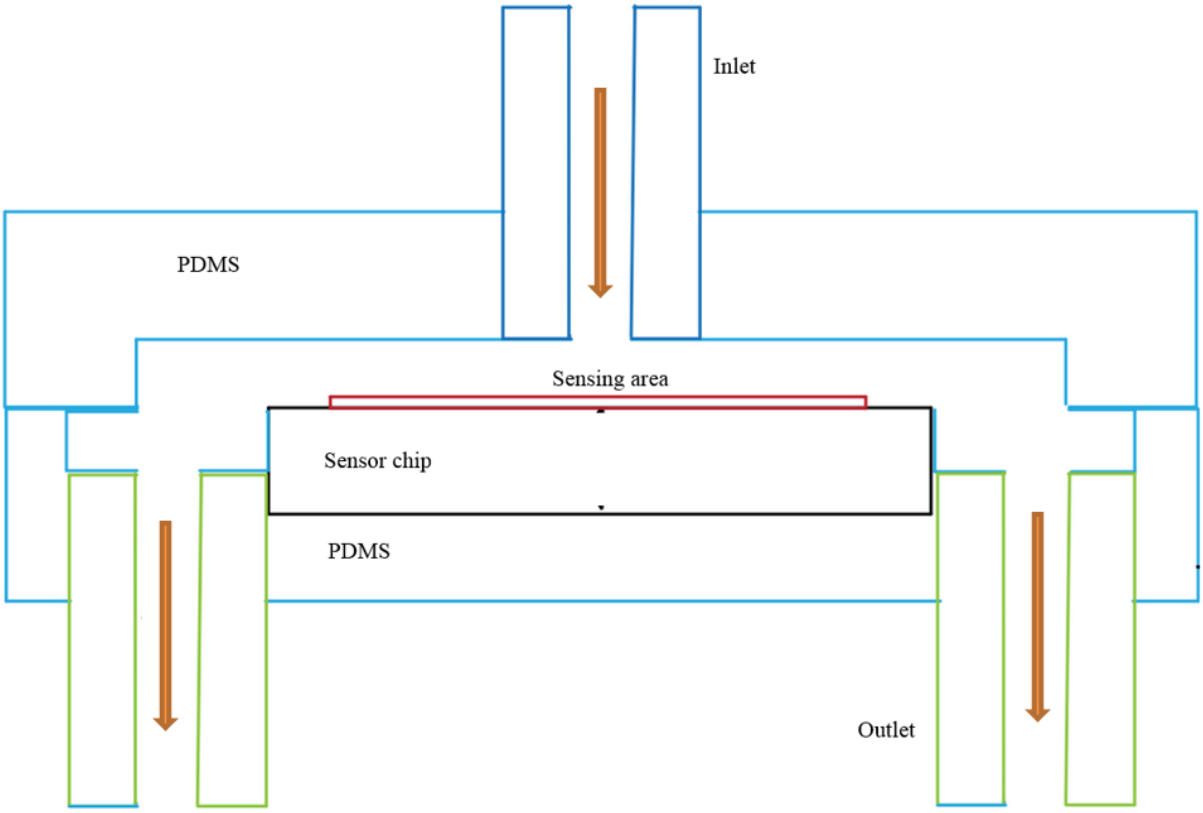

Figure 4. Sketch of the principle of the design four.

There are two bottom designs. In one of the designs the slot for the sensor chip is made with SU-8; an epoxy based resin that is used to create microstructures. The problem with this design is that the photoresist is not going to be thick enough to avoid stress on the chip, that might causes it to break. The other design only has the pads for the outlets. Before pouring the PDMS on the master, a dummy chip is fastened with glue between the pads. The dummy chip is thinner than the sensor chip, 500 µm instead of 650 µm thick, but otherwise shaped as the sensor chip. The idea is to create the necessary pressure to seal the chip, but not too much pressure, so that it will break. The fourth design consisting of three drawings is illustrated in Figure 5.

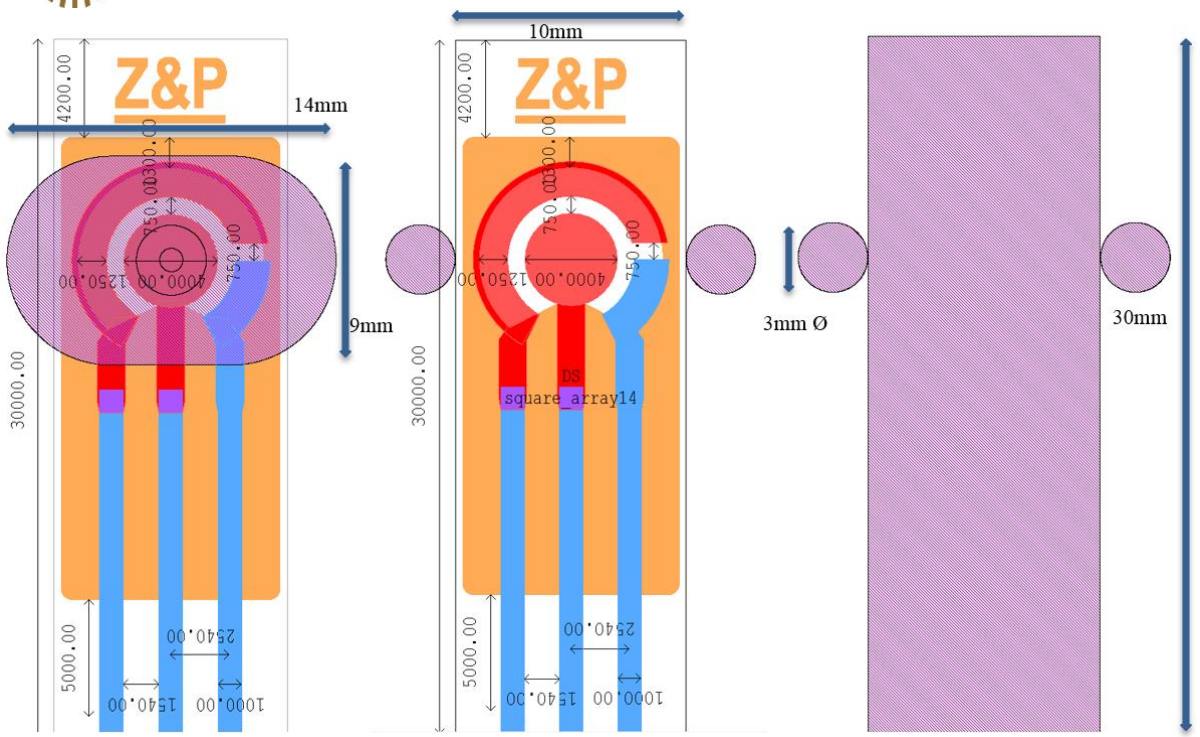

Figure 5. The fourth design. The purple shaded area shows the design and the chip is used to show the idea. From left to right: Top, bottom with space between the pads and bottom with socket made with SU-8

2.1.2 CAD Design

L-edit is very suitable for creating masks for photolithography, due to the ability to draw details down to 0.5 µm. This program has many options and possibilities, but lacks an intuitive user interface. L-edit has a multilayer function that enhances the possibility to make very accurate multilevel designs. In this project, the designs are all on one layer, but it possible to use the multiple layers to create patterns and guidelines. The design of the sensor chip was previously drawn in this program, and therefore it was easy to use it as a pattern for the designs. It was imported together with a sketch of the perimeter of a 4" wafer. This made it a lot easier to create the drawings of the designs.

The glass slides had a width of 25 mm and the designs had to be slimmer to fit. It was decided to be 22 mm. The length of the sensor chip is 30mm so the length of the PDMS design was set to 33 mm. A square was drawn inside the perimeter of the wafer of 66x66 mm. The 6 drawings fitted perfectly inside as shown in Figure 6.

Guidelines were made using the drawing of the sensor chip as pattern. Temporary guidelines were also used to find the center of a design or as a help to achieve the same height for different parts of a design.

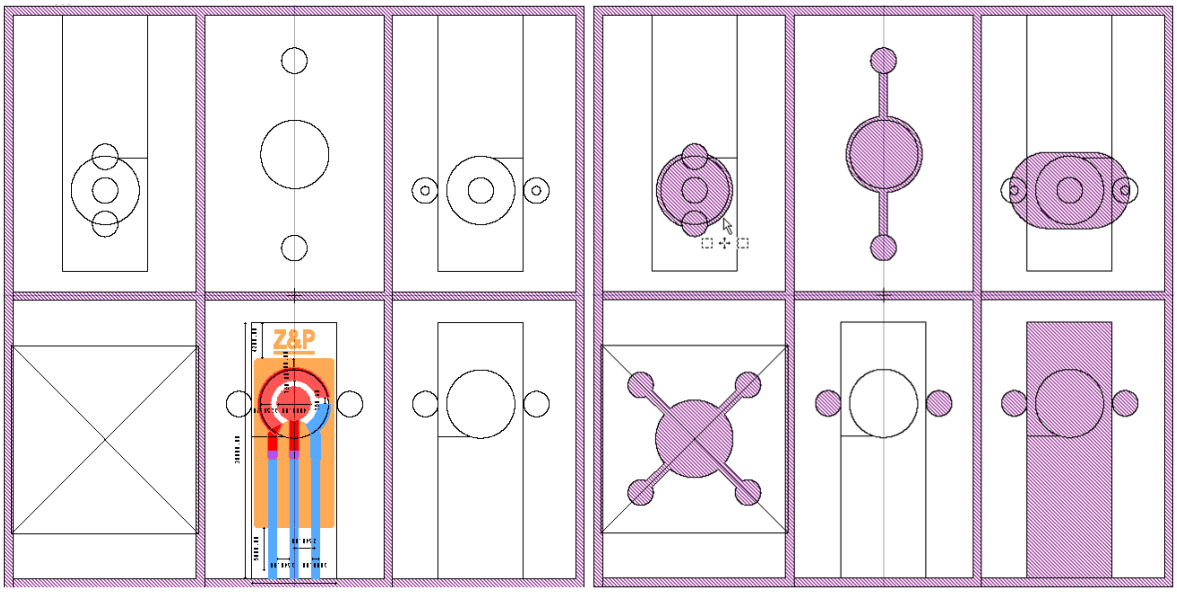

Figure 6. The figure at the left shows the guidelines and the figure at the right shows the patterns drawn in the mask layer.

With the guidelines on another level, it was easier to draw the designs on the mask layer, as shown in Figure 6. It was also added some alignment windows as shown in, to easier align the mask with the wafer.

Once all the designs were drawn, the guidelines were removed, and the designs were merged. That means that if one design consists of various drawings, like rectangles or circles, these will be merged into one single drawing.

The designs were drawn to show the structure of the master. When exposed to UV light, the photoresist becomes hardened as explained in 2.1.4.1. The shaded parts will be the covered part of the mask and the blank part will be the transparent, which is why the drawings had to be inverted before they are sent to Infinite graphics Inc. This is easily done with the Boolean operation “NOT”. This is illustrated in Figure 7. The file had to be converted to the GDSII file format.

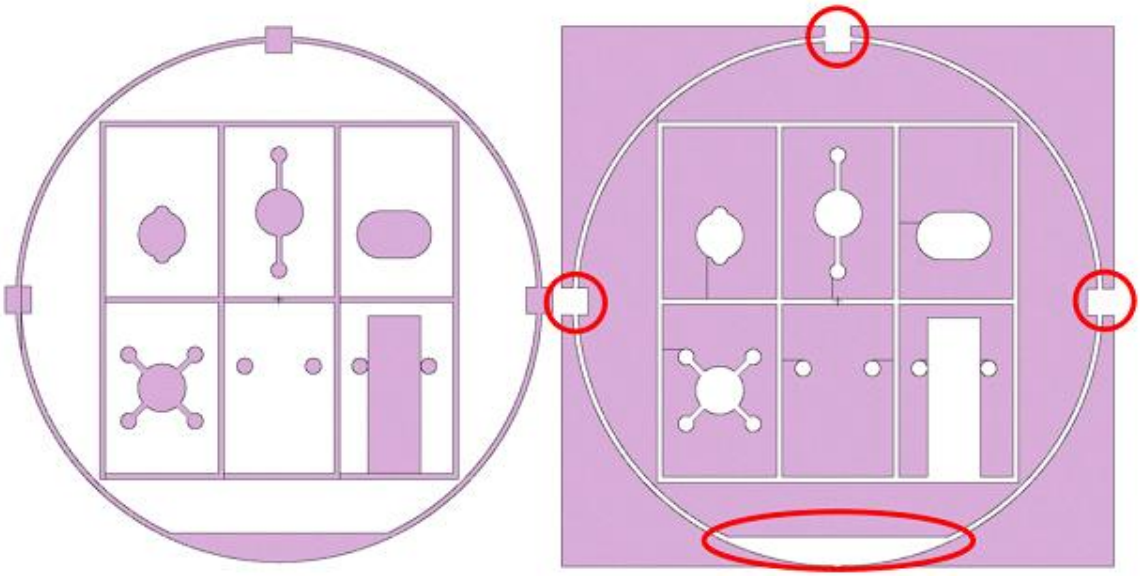

Figure 7. Drawing before inversion to the left, and inverted to the right. The designs from the left top to right bottom are the second, the first, the fourth (top), the second, fourth (bottom two) and fourth (bottom one). The alignment windows are marked with red

2.1.3 Holder Designs for the MFC

The designs 1, 2 and 3 are based on the same system: a PDMS design mounted on a glass slide with a hole where the sensor is to be fixated. The idea is to create a holder that presses the sensor chip against the glass, with a gasket in between. For design 4, the holder uses the same concept with two plates, but in this case, the PDMS acts as gasket. The holders are designed using a top and bottom plate held together with one bolt in each corner. The bolts have to be long enough as to act as legs and the bottom plate can be fixated at a desired altitude. Plexiglas is used as plates due to its transparency and firmness. It is also easier to drill holes in Plexiglas than in harder materials like glass.

2.1.3.1 Holder for Design 1, 2 and 3

The holders are quite expensive to make, and there seemed to be a possibility to create the top plate in a way that all the designs could be used. Making holes for the outlets of the different designs, gives the opportunity to change between them. A bigger hole is made in the center, over the sensing area, to make it easy to visually follow the movement of the liquid. A gasket must be cut out of a rubber sheet to fit around the measurement hole in the glass slide.

The gasket needs an inner diameter of 8mm to adapt to the sensing area of the chip. To give space to the connector, a socket must be cut out in the bottom plate. This is illustrated in Figure 8.

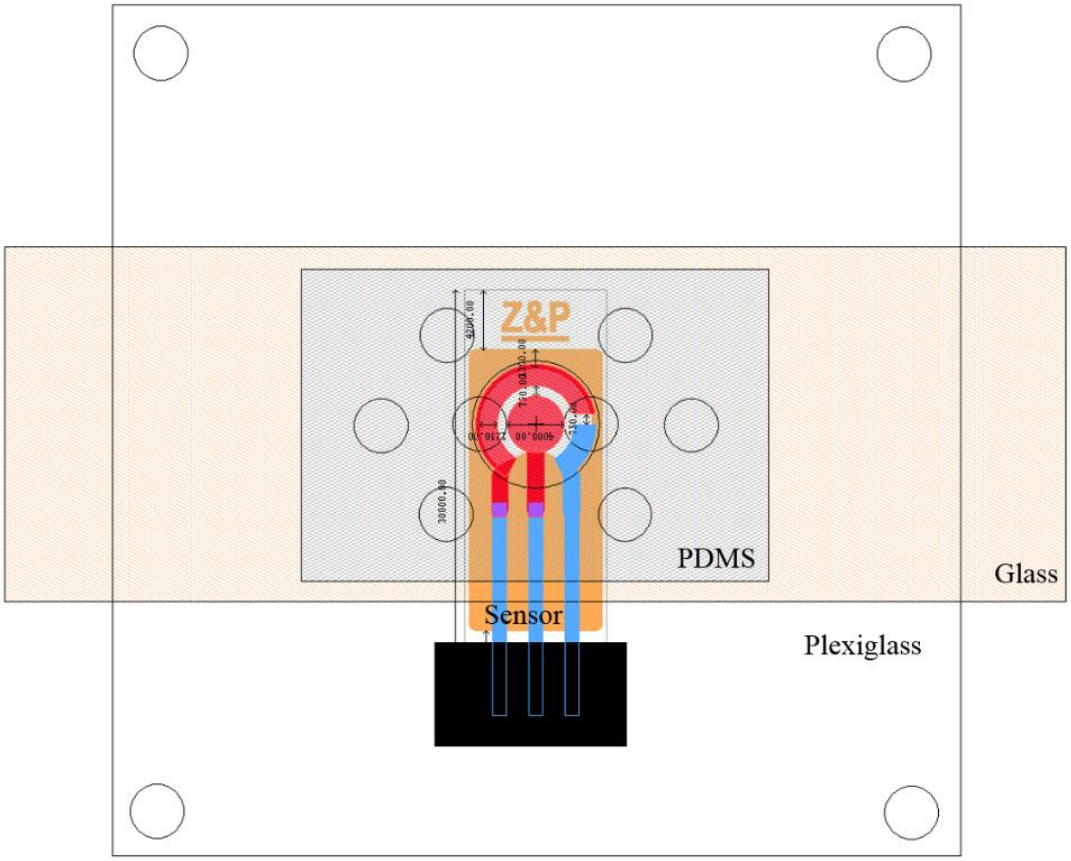

Figure 8. Top plate of the holder with holes suited for all three designs.

2.1.3.2 Holder for Design 4

The holder for design 4 was designed with one oval hole in the top plate to view the chamber, and 2 holes in the bottom plate for the outlets of the bottom layer. This works as a sandwich with the bottom plate under, then the bottom layer, the sensor chip in the socket of the bottom layer, the top layer covering the chip, and the top plate holding it together. Figure 9 illustrates the working principle of the holder. A track needs to be carved into the bottom layer of PDMS to make a socket for the connector. On the top layer, the design is cut smaller, to not obstruct the space for the connector.

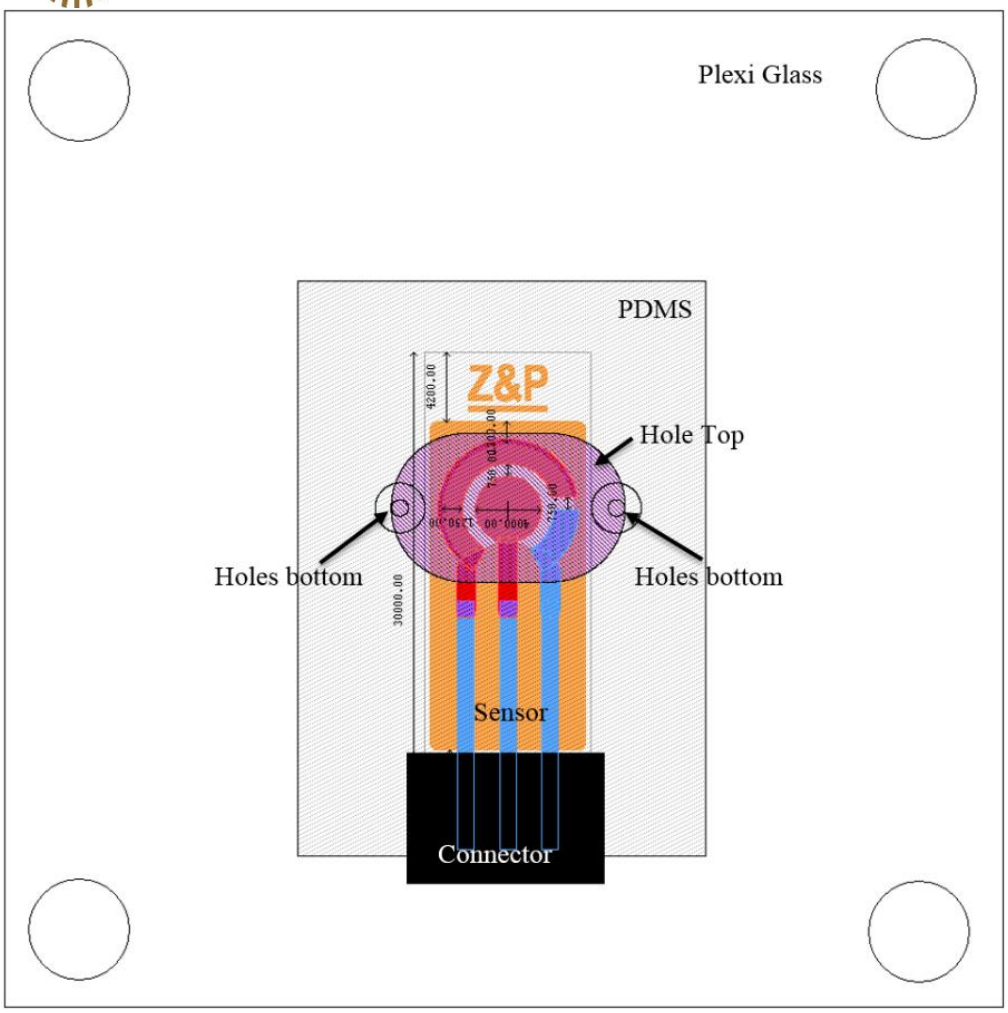

Figure 9. Holder for design 4.

2.1.4 Fabrication Protocol.

The fabrication of the MFC consists of four steps:

- ∇ Polymeric master
- ∇ PMS casting
- ∇ Oxygen plasma bonding
- ∇ Manufacturing of the holder

The materials for all the processes and the standard procedures for the master fabrication are placed in Appendix 7.4 to 7.6

2.1.4.1 Polymeric Master

The master consists of a silicon wafer with a microstructure of negative photoresist, SU-8, created through pattern transfer. For wave length larger than 360 nm, the SU-8 has very good translucent properties, making it very suitable for photolithography of thick resist requiring high precision. The structures are chemically and thermally stable. The thickness depends on the spin parameters (speed/time/acceleration) and of the viscosity of the resin. In the project, it was decided to use SU-8 100, giving the possibility to choose between 100 µm, 150 µm and

250 µm thicknesses through the recommended fabrication protocol provided by the manufacturer MicroChem Corp., USA [8]. A simple illustration of the protocol is described in Figure 10.

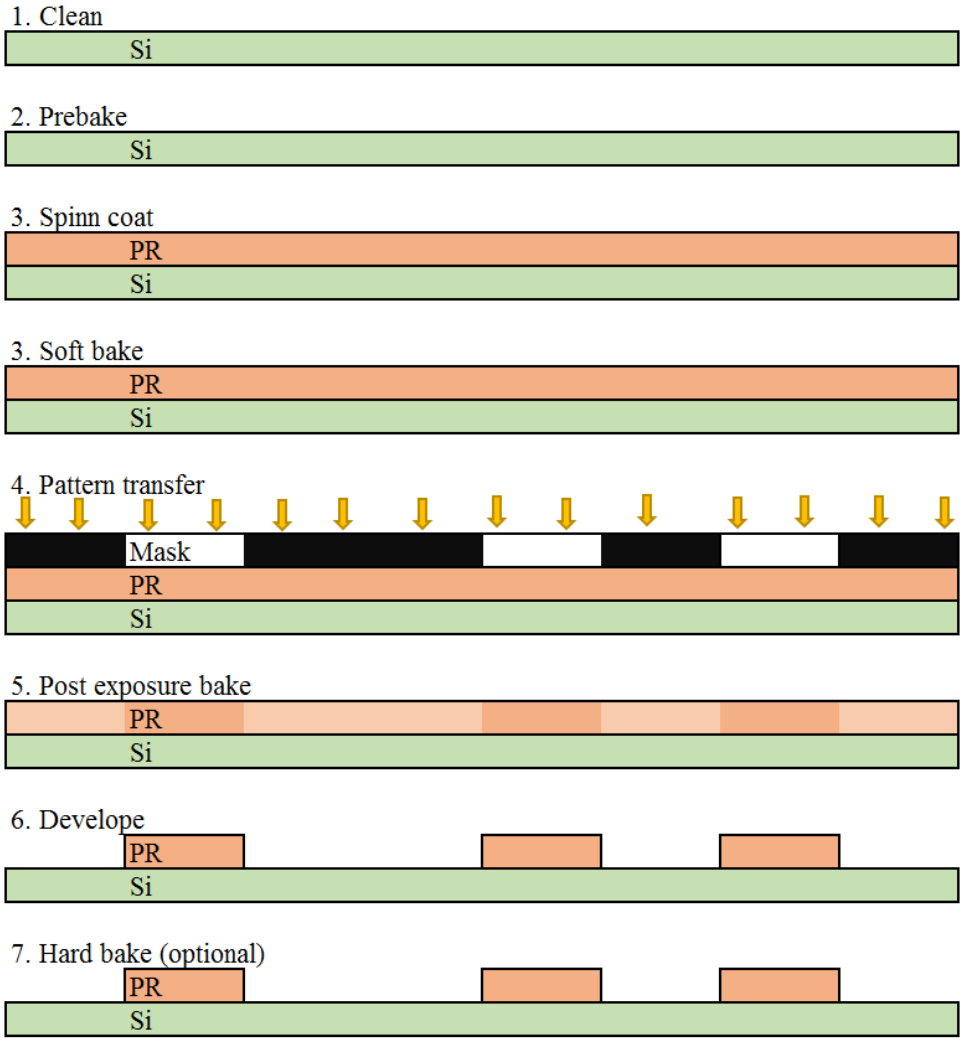

Figure 10. Simple illustration of the photolithography process.

Pattern transfer occurs by illuminating the wafer covered with SU-8 through a photomask. By being exposed to the UV-light, acid is created. The acid starts a crosslinking reaction powered by heat of the post exposure bake on a hot plate [8]. Crosslinking happens when neighboring linear chains of epoxies are joined in a grid-like structure by covalent bonds as illustrated in Figure **11** [9]. This makes the structure more rigid and resistant to the developer. The unexposed SU-8 remains soluble to chemicals in the developer.

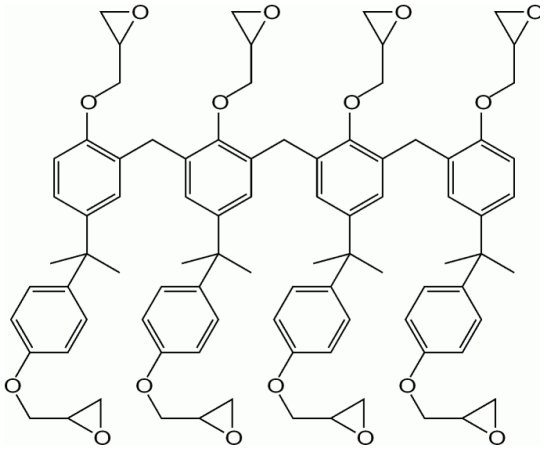

Figure 11. SU-8 molecules [10].

The mask aligner, Karl Suss, is fitted for 4" wafers and masks up to 5". It has a 350W mercury arc lamp that gives 365 nm wavelength.

The light intensity was measured before starting the fabrication process, and the result was used to find the exposure time in the table in the procedure provided by MicroChem. The procedure is found in Appendix 7.6.

- Center: 8.5mW/cm^2
- Top: 6mW/cm^2
- Bottom: 12mW/cm^2
- Right: 8.5mW/cm^2
- Left: 9mW/cm^2

The middle value was 8.8mW/cm^2 rounding it to 8.5mW/cm^2. To calculate the time needed for the exposure, the energy is divided by the light intensity.

$$\frac{Energy}{Light\ intensity} = \frac{J/cm^2}{W/cm^2} = seconds \quad (2)$$

The polymeric masters were fabricated through a process of laboratory exercises attached in Appendix 7.9, following the procedure explained in the standard fabrication procedure. Only the deviations from the standard procedure are commented below.

Two different hot plates were used. Due to the stickiness of the SU-8, the hot plates were covered with aluminum foil. The temperatures were increased by 10 ^oC to compensate for the loss of heat transmission provoked by the aluminum foil.

The first two laboratory exercises, Test 1 and Test 2, were tests to learn the procedure of the fabrication and how to operate the equipment. It was experimented with the amount of SU-8 applied and possible application methods.

The four consecutive Laboratory Exercises, Exercise 1 to 4, had as objective to manufacture a set of masters usable for the fabrication of the MFC designs.

The objective of Test 1 was to create a 100 µm thick layer of SU-8. This was seen as a test, and not expected to give a usable master.

- The cooling time after the post exposure bake was 5 minutes.
- The developer was used.

The objective of Test 2 was to create a master with 250 µm thick layer of SU-8. In this process it was decided to experiment with the application method to try to obtain a repeatable amount of resist.

- ∇ Disposable syringes were used to apply the SU-8 on the wafer.
- ∇ The procedure could not be finished due to bubble formation.

The objective of Exercise 1 was to create two masters with a 250 µm thick SU-8layer.

- ∇ The acceleration from 500 to 1000 rpm was set to 10 seconds, giving a lower acceleration.
- ∇ For the baking processes, two different hot plates were used.
- ∇ The soft bake time for the second wafer was increased with 10 minutes.
- ∇ Exposure time was set to 71 seconds.
- ∇ The development time was increased by 7 minutes on both wafers.

The objective of Exercise 2 was to create master with a 250 µm thick layer of SU-8 (wafer 1), and another master with 100 µm thick layer of SU-8 (wafer 2). It was decided to hard bake the masters.

- ∇ The acceleration time on both wafers were 10 seconds on step 3.
- ∇ The spin time of step 3 was increased from 30 to 60seconds on wafer 1.
- ∇ The time of the soft bake step 2 was increased to 150 minutes for wafer 1 and 55minutes wafer 2.
- ∇ The exposure time of wafer 1 was increased to 150 seconds.
- ∇ The development time for wafer 1 was 22 minutes.
- ∇ The wafers were cooled down only 5 minutes from the hard bake

The objective of Exercise 3 was to create a master with a 250 µm thick layer of SU-8. It was decided to hard bake the master.

- ∇ The acceleration time was set to 10 seconds
- ∇ The spin time of step 3 was increased from 30 to 60 seconds.
- ∇ The time for the soft bake step 2, was increased to 150 minutes
- ∇ The exposure time was set to 150 seconds

The objective of Exercise 4 was to create a master with a 250 µm thick layer of SU-8. The master was not hard baked. The hot plate was changed to test if the results change.

- ∇ The acceleration time was set to 10 seconds
- ∇ The spin time of step 3 was increased from 30 to 60 seconds
- ∇ The time for the soft bake step 2, was increased to 150 minutes
- ∇ The exposure time was set to 150 seconds

2.1.4.2 PDMS Replica Molding

Polydimethylsiloxane (PDMS) is a transparent silicone elastomer and comes in two components, the base and the curing agent. Figure **12** shows how the PDMS consists of long chains of monomers. It is applied in many different fields as in LED Lightning encapsulation, power supplies, connectors, sensors, transformers, relays etc. [11].

The PDMS has the ability to fill micro spaces, and therefor used for replica molding. It is non-fluorescent, gas permeable, biocompatible and chemically and thermally stable [12].

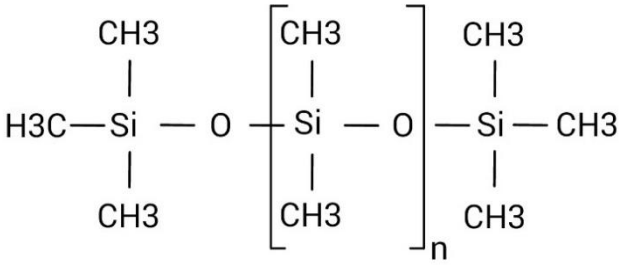

Figure 12. The PDMS polymer consisting of n monomers.

The PDMS is cured through a reaction between the vinyl groups (-CH=CH_2) in the base and the Silicon hydride groups (Si-H) in the curing agent, creating crosslinking. The rate of crosslinking is temperature dependent. Because of low surface energy, the PDMS replica is easily removed from the master without damage, and the master can be used again.

The PDMS replica molding consists of casting PDMS on a polymeric master. The procedure is illustrated with the flow chart shown in Figure 13.

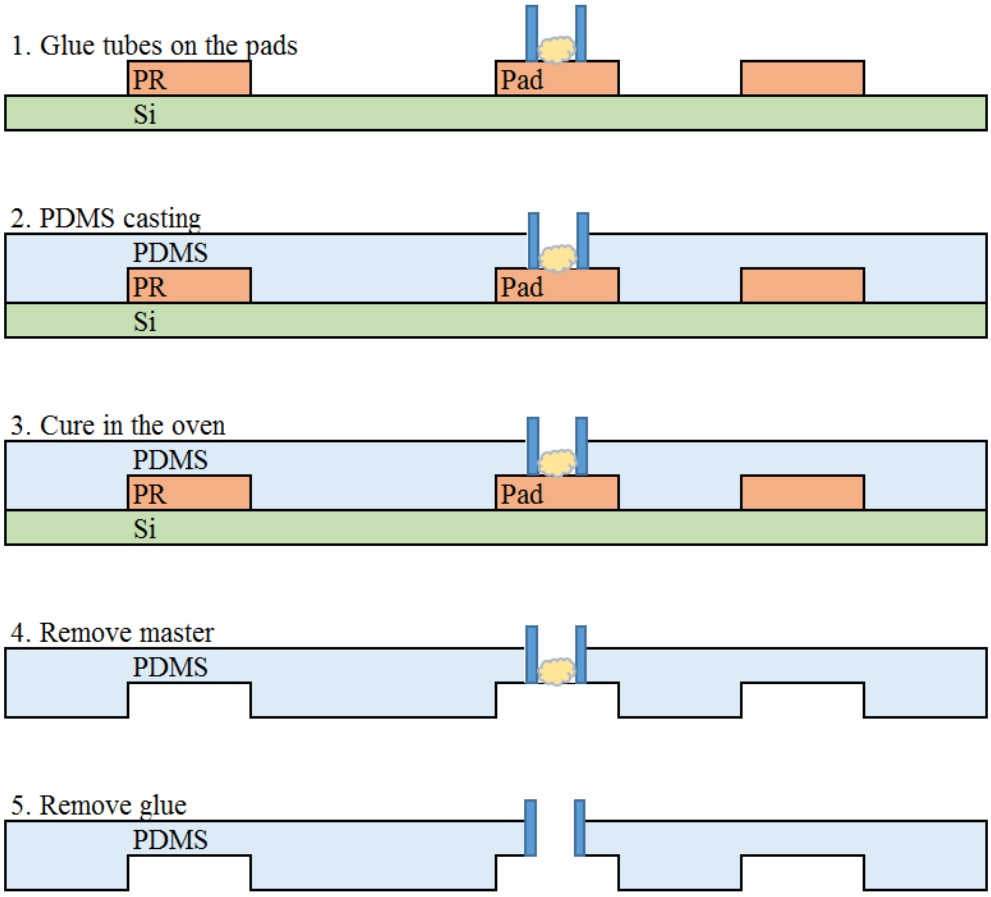

Figure 13. Simple illustration of the PDMS replica molding.

Two PDMS replicas were created using the same procedure. Both laboratory exercises, Replica molding 1 and Replica molding 2, are attached in the Appendix 7.9.5 and 7.9.6.

The first replica used wafer 1 from Exercise 1, and the second replica used wafer 2 from Exercise 1.

The master was cleaned. The silicone tube was cut into 1 cm long pieces that were glued to the in-/outlet pads on the master with Duco cement. Some of the glue filled the entrance of the tube, blocking it so that the PDMS could not enter. The dummy chip was glued to the master 2 from Exercise 1. It dried for 5-20 minutes while preparing the PDMS.

Approximately 40g of PDMS was used. It was mixed with the curing agent 10 to 1 in a beaker, and stirred until it is white with bubbles. The beaker was placed in the vacuum chamber for 20-30 minutes, until the bubbles were eliminated. Due to the formation of foam from the bubbles, the vacuum was set to ¾ of maximum power.

When the bubbles were removed, the PDMS was poured into a mold with the master, carefully avoiding the tubes. The mold was placed in the vacuum chamber again to degas for another 15-20minutes.

To cure the PDMS, it was placed in the oven at 65^{o}C over-night. The PDMS will also cure at room temperature, but it will take very long, approximately 24 hours.

The PDMS was released by cutting out the designs first, to avoid ruining the PDMS replica. It comes off easier in smaller parts than in a complete piece. To cut the designs, a normal paper knife was used. The master has separation lines between the designs that are easy to follow with a knife. The master was removed from the mold and cleaned.

When the designs were lifted off, the glue was removed by pushing a thin metal wire through the tube.

The designs were guarded in a dust free plastic box.

2.1.4.3 The Oxygen Plasma Bonding

PDMS does not bond easy with other substances due to low energy and non-reactive surface, and it is highly hydrophobic. When the surface is activated by oxygen plasma, it gets more reactive and hydrophilic, and it can bond to glass, silicon and itself. Once it is activated, it needs to be bonded quickly, if not it goes back to its normal and hydrophobic state within few hours. The bonding process can be accelerated with a post exposure bake [13].

PDMS consists of units of -O-Si(CH_3)- units in long chains. When the surface is activated with oxygen plasma, silanol groups (Si-OH) are created. When PDMS and glass bond, these groups condense with compatible groups like OH, COOH and ketone. It is joined with covalent bonds with the reaction of Si-O-Si, and is irreversible [14].

Three of the four designs were mounted on a glass slide with a hole as a measurement chamber for the sensor chip. The advantage of PDMS is that it bonds easily with glass through an oxygen plasma treatment. The process flow is illustrated in Figure 14.

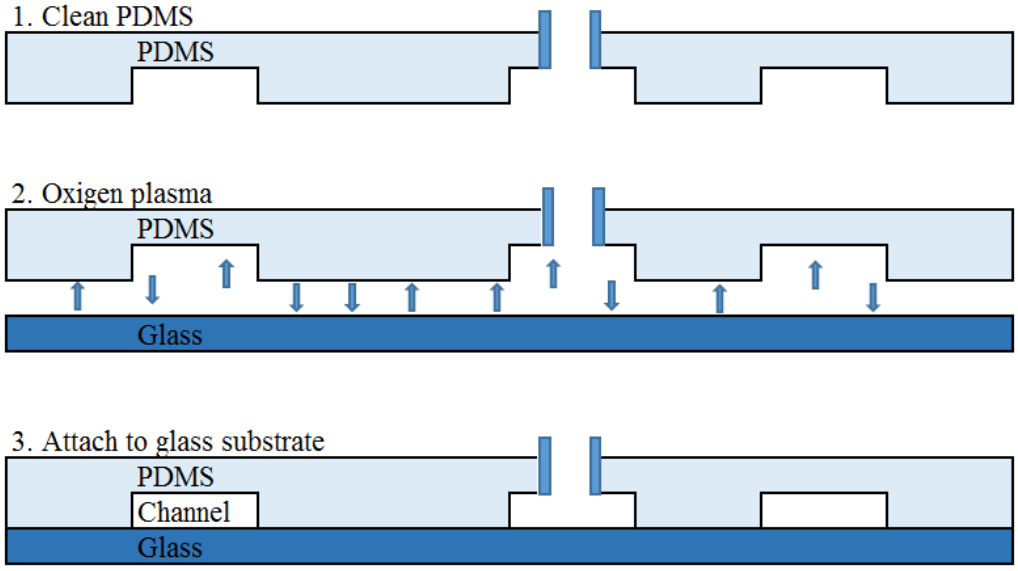

Figure 14. Simple illustration of the bonding process.

Before the oxygen plasma treatment, the glass and PDMS designs were thoroughly cleaned and dried. At first, the designs were only cleaned with DI-water, and the bonding did not work well, possibly because of impurities. They were cleaned again with IPA.

The parameters for the Reactive Ion Etcher (RIE) were as shown in Table 1.

Table 1. Parameters for the oxygen plasma treatment

Power (W)	Pressure (mT)	Time (s)	Oxygen (sccm)
90	100	30	20

All the designs and glasses were placed bonding side up as horizontally as possible inside the vacuum chamber of the RIE, shown in Figure 15. Once the oxygen plasma process was completed, the PDMS designs were placed on the glass slides, making the hole coincide with the measurement chamber of the designs. It was squeezed carefully to help the bonding. After a few minutes, the bonding was examined by feeling the resistance at the edges. The designs were left to finalize the formation of covalent bonds. It is also possible to use a hot plate at 65^{o}C to accelerate the bonding process, but that was not necessary.

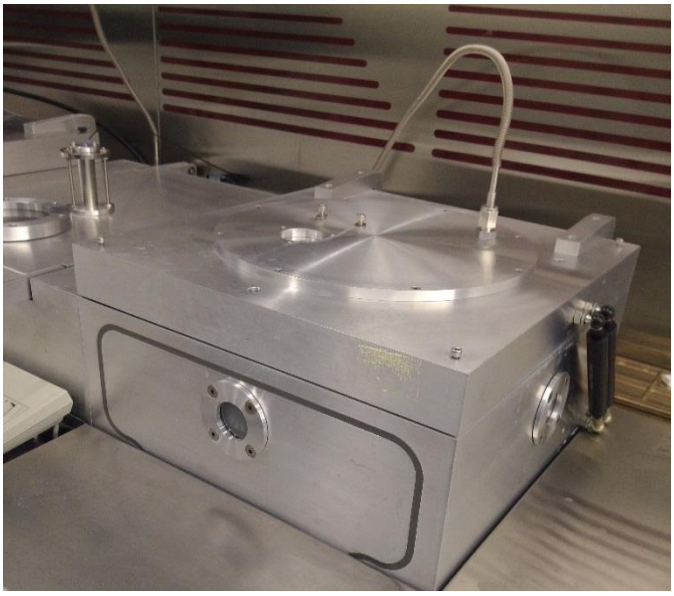

Figure 15. The vacuum chamber of the RIE

2.1.4.4 Manufacturing of the Holder

The holders consists of four Plexiglas plates with different designs, 8 bolts with a thickness decided to be 6 mm for esthetic reasons, 16 nuts, 24 washers and 8 wing nuts.

The Plexiglas drawings of the designs, described in section 2.1.3, were sent to Tønberg Glassliperi (glazier in Tønsberg, Norway), and it was decided to use a thickness of 5 mm to prevent the plates from bending under pressure.

The rest of the materials were bought on Tønsberg Maskinforetning, Tønsberg, Norway.

When the Plexiglas designs were finished, the holder could be assembled. The bottom plate was fixated between nuts and their corresponding washers at the wanted height and the wing nuts were used to fixate the top plate giving the desired pressure. A picture of the holder is shown in section 3.1.2.

2.2 Front-end Interface and Control System

The front-end interface and control system is the hardware and software required to drive the sensor, and to read the sensor current and present measurement data on a computer. It is based on the eZ Sense; a prototype system for reading sensor signals, developed by Zimmer & Peacock. The eZ Sense system, shown in Figure 16, consists of the MAS_V2_R2 board and ZT GUI software.

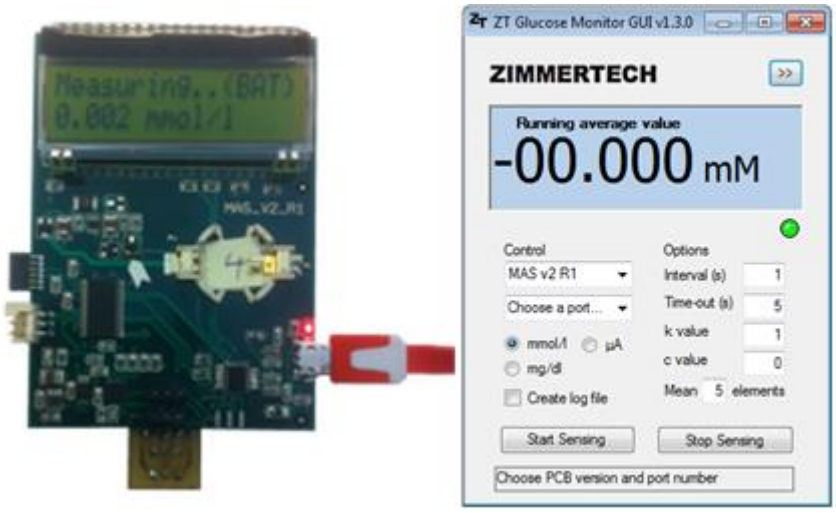

Figure 16. The eZ Sense system: MAS_V2_R1 board and ZT GUI. The MAS_V2_R2 board used in the project did not have a LCD display connected. Therefore a picture of the similar MAS_V2_R1 board was used for illustrating purposes.

The intermediate goals for the front-end interface and control system were defined in the pre-project:

- ∇ Choose methods for improving and expanding the eZ Sense system
- ∇ Complete circuit layout
- ∇ Complete setup system on breadboard
- ∇ Complete PCB design, fabrication and assembly
- ∇ Perform successful system tests

The design improvements of the eZ Sense were divided into four main objectives and one secondary objective:

Main objectives

- ∇ Expand to multiple channels
- ∇ Upgrade firmware and software
- ∇ Find solution to a problem with the power switching logic
- ∇ Reduce 50 Hz noise from USB connection

Secondary objectives*

- ∇ Create a graphical representation of sensor signals in the Graphical user Interface (GUI)

*if time permits

The objectives were somewhat modified during the first couple of weeks. The main focus became to create a more versatile instrument, which meant implementing some additional functionality upon request from Zimmer & Peacock. Among these were temperature measurements using the embedded temperature sensor on the LMP91000, and controlling the LMP91000's transimpedance gain. The LMP91000 will be introduced in section 2.2.1.1.

Expanding to multiple channels and upgrading the firmware and software became the main objectives during the project, along with the temperature measurement, gain control and graphic data presentation in the GUI. Reduction of the 50 Hz noise as a main objective was effectively replaced by the extra functionality to make the instrument more versatile.

The number of channels was decided partially after the first project meeting with Z & P, where the number eight was suggested. Eight channels was also practical seen from a programming perspective; one byte could be used to address the channels with one bit each.

The thought of having a battery powered system was discarded early in the planning phase; effectively solving the power switching logic problem, which did not automatically change between battery and USB power. The main reason for this choice was that a battery would only be needed if the system were to operate without a computer, something that would require displays to present measurement data, such as with the LCD display on the MAS_V2_R2. As the new system is intended to be a more stationary laboratory instrument, it seemed more logical to have it communicate with a computer for easier data access, both in real-time and from log files. Because of this, neither battery nor LCD displays are implemented in the new design.

2.2.1 System Blocks

The block diagram in Figure 17 shows how the different key components of the multi-channel system are connected. The choice of components and their role in the system is described in more detail in the sections below.

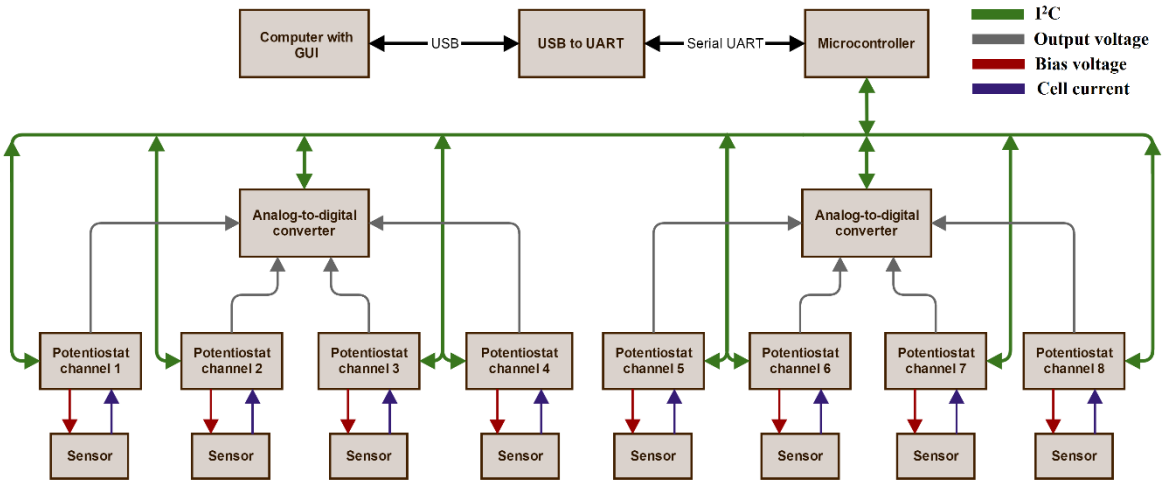

Figure 17. System Block Diagram.

2.2.1.1 Potentiostat

The eZ Sense use an LMP91000 potentiostat, manufactured by Texas Instruments (USA). It was not considered to look for possible replacements, as it has proven to function very well in the system. Figure 18 shows a block diagram of its inner circuitry.

The purpose of the potentiostat is to control the voltage difference between working and reference electrode, and to supply the electrochemical cell with the required current through the counter electrode [15]. A transimpedance amplifier (TIA) provides a voltage output proportional to the current flowing through the working electrode, on the V_{OUT} pin. The transimpedance gain is equal to the magnitude of R_{TIA} [16].

The LMP91000's internal zero is the voltage at the non-inverting TIA input. It can be programmed to a certain percentage of the supply or external reference voltage. An LT6656 voltage reference manufactured by the American Linear Technology, is used in the eZ Sense system. There were no compelling arguments to change this, so it was chosen as the reference for the ZP2015 as well.

The LMP91000 have several operation modes, of which three are used in the Sensetion system; 3-lead amperometric cell mode, Temperature Measurement mode (with TIA turned on) and Deep Sleep mode.

An embedded temperature sensor can be read on the V_{OUT} pin when in Temperature Measurement mode. There are two different temperature modes; one with TIA on, and one with TIA off. ZP2015 use TIA on for reasons that will be described in section 2.4.2.3.1.

The Deep Sleep mode turns off the control amplifier, TIA and temperature sensor, leaving only the I^2C interface operational. This mode is intended to reduce power consumption.

All communication with the LMP91000 is done through an I^2C interface. The LMP91000 comes with a pin to enable and disable I^2C for the device. In order to communicate with the LMP91000, the MENB pin must be held low during the whole operation. This makes it possible to have multiple LMP91000 devices on the same I^2C bus, even though they all have a fixed I^2C address of 1101 000.

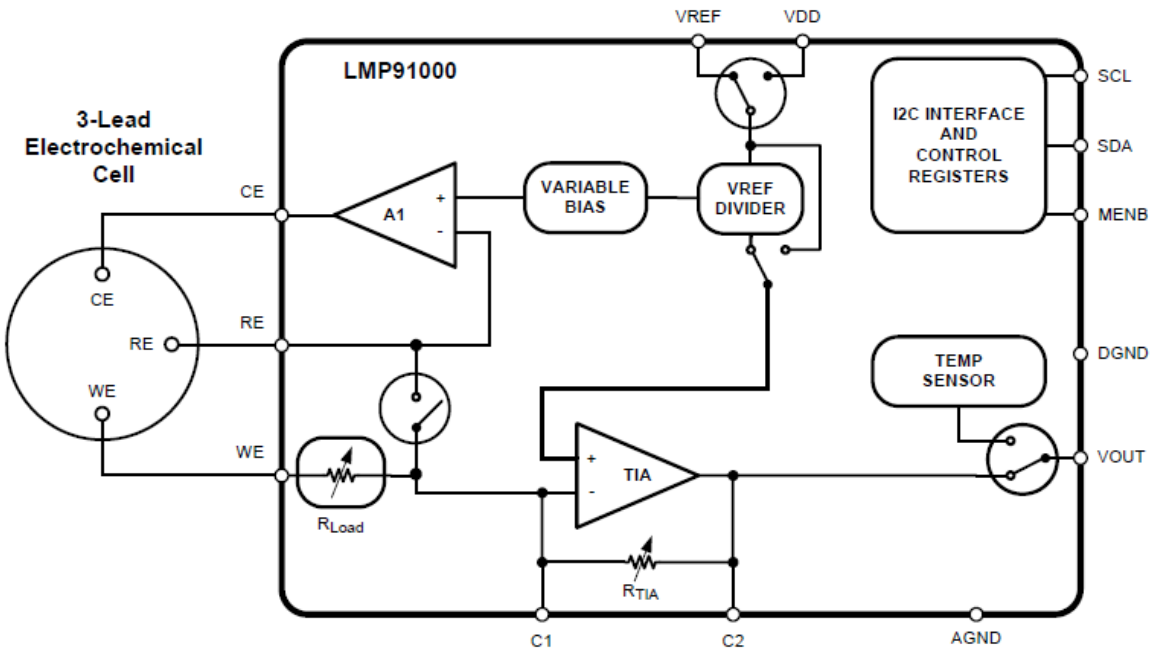

Figure 18. LMP91000 System Block Diagram [16]. With permission from Texas Instruments.

2.2.1.2 Analog-to-Digital Converter

An analog-to-digital converter is used to digitize the analog output voltage from the potentiostat so that it can be read by a microcontroller.

The eZ Sense system use the ADS1113 analog-to-digital converter from Texas Instruments. After reading the data sheet for this particular device, it became clear that it would not be suitable for the new application as it can only measure one differential or single-ended input, so one ADS1113 per potentiostat would be required. In addition to the large amount of traces needed on the PCB to support eight devices, there are only four different I^2C addresses available on the device, and one is already occupied by the LMP91000. The idea of doing this was quickly discarded. However, the input multiplexer featured in the ADS1115 makes it possible to have two differential or four single-ended input signals, allowing the use of only two devices in an eight-channel system. Both devices use the same registers, so the ADS1113 code from the eZ Sense system would also work on the ADS1115. Therefore, it was decided to use the ADS1115 in this project.

The ADS1115 shown in Figure 19 is a 16-bit precision delta-sigma analog-to-digital converter. It consists of a delta-sigma analog-to-digital core with adjustable gain, internal voltage reference, clock oscillator and I^2C interface. There are four I^2C addresses available for the ADS1115, chosen by connecting the ADDR pin to either V_{CC}, ground, SCL (I^2C serial clock line) or SDA (I^2C serial data line) [17].

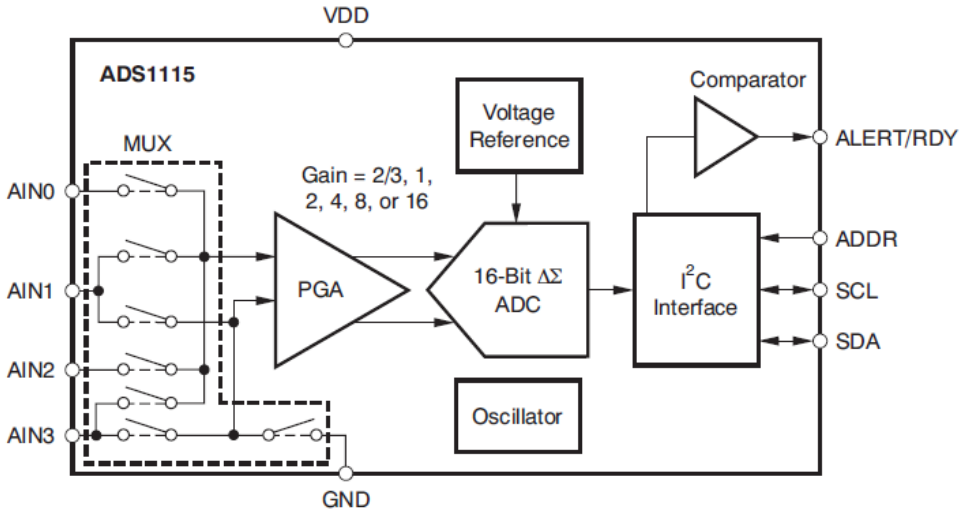

Figure 19. ADS1115 Functional Block Diagram [17]. With Permission from Texas Instruments.

The A/D core measures the difference of AIN_P and AIN_N. Depending on the input multiplexer configuration in Figure **20**, either four single-ended or two differential signals can be measured. The negative input is internally connected to ground when using single-ended signals. It was considered to use four ADS1115 devices with two differential inputs each for better noise reduction, but only three I^2C addresses were available because one of the addresses are the same as for the LMP91000.

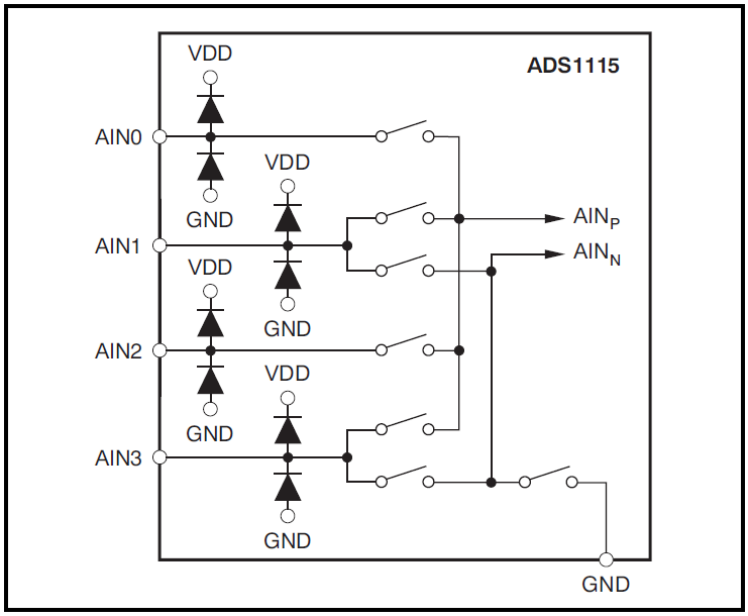

Figure 20. ADS1115 MUX [17]. With Permission from Texas Instruments.

Conversion of a single-ended input voltage is performed according to (3).

$$V_{in} = FS \cdot \frac{(ADC\ value)}{2^{15}-1} \qquad (3)$$

The programmable gain amplifier (PGA) can be set to different gains with corresponding full-scale (FS) ranges. It was chosen to keep the eZ Sense's PGA value of 2, giving a full-scale input range of 2.048 V. This means that analog input voltages up to 2.048 V are represented by digital values between 0 and 32767, given by the 16-bit precision of the ADS1115.

$$V_{in} = 2.048\,V \cdot \frac{(ADC\ value)}{2^{15}-1} \qquad (4)$$

The ADS1115 has two operation modes; continuous conversion mode and single-shot mode. In continuous conversion mode, a new conversion of the input signal initiates as soon as the previous conversion is completed, at a rate equal to the programmed data rate. The most recent completed conversion is always read when accessing the result register. In single-shot mode, only one conversion is performed upon request and the value is stored in the result register before the device enters a low-power shutdown. This is intended to save power in systems that have long idle periods between conversions [17]. The eZ Sense system use continuous mode, but single-shot mode was chosen for the ZP2015. The choice was made for two reasons; one was to save power and the other was to make sure that the values that are fetched from the result register belong to the desired channel.

2.2.1.3 Microcontroller

A microcontroller acts as the I^2C master and controls the ADS1115 and LMP91000. It also communicates with the computer software application. MAS_V2_R2 featured an MSP430F2274 microcontroller from Texas Instruments. It is a low-power microcontroller with a 16-bit RISC architecture CPU [18]. As the previous circuit design and code are designed for this particular device, there seemed to be no reason to change it.

The MSP430F2274 uses a universal serial communication interface (USCI) that supports multiple serial communication modes [19]. The two modes used in this project are I^2C for on-board communication with the LMP91000 and ADS1115, and UART for communication with the computer.

2.2.1.4 USB to UART Interface

In order for the microcontroller to communicate with the computer via USB, an USB to UART converter is needed. eZ Sense uses the FT230X from Future Technology Devices International Ltd. for this task. There were no apparent reasons to choose a different device, so it remains the same in ZP2015.

2.2.1.5 Grahical User Interface

The GUI acts as the link between PCB and user. It allows for system configurations and processes the measured signals before presenting them on screen.

This part of the system is described in section 2.4.3.

2.3 Design and Production of Front-end Interface

2.3.1 Prototype on Breadboard

Before considering a PCB layout, the group set goals for a fully functional prototype setup on a breadboard. The setup was initialized early in the project, and went forward in parallel with the work on the microcontroller code.

A list of materials and equipment for this part of the project can be found in Appendix 7.3. Most of the components except for the resistors, capacitors and connector for the MSP-EZ430U debugging interface, which is introduced in section 2.4.1, were surface mounted and could not be applied directly onto the breadboard. Z & P provided the group with breakout boards for the microcontroller and two potentiostats, but the rest had to be produced because they were not – with the exception of the ADS1115 – obtainable through any of the standard component supplier at HBV. This caused some delay in the prototyping stage. The breakout board production is covered in section 2.3.2.

It was necessary to learn how the eZ Sense system worked before a prototype could be set up on a breadboard. This process was a combination of studying the existing code, circuit designs and datasheets for the various components, and experimenting with a MAS_V2_R2 board and ZT GUI. It was not necessary to learn everything before starting the prototype setup, but the basics; such as communication between the digital components and how to upload code with the MSP-EZ430U, had to be covered. The rest came through trial and error as the project progressed.

The work with the prototype setup was done systematically by starting with the most essential, and adding more components when the previous setup responded as desired. The initial setup was done according to the original MAS_V2_R2 circuit designs, found on the CD accompanying the project thesis. It was thought to create new designs for the expanded system before assembling the components on the breadboard. However, because the breadboard prototyping had a large role in the process of learning the eZ Sense system, new circuit designs were made and adjusted as the breadboard setup evolved. The circuit designs can be found in Appendix 7.8.

The first and most essential piece of the task was to establish contact with the microcontroller and being able to upload code to it. Apart from the various resistors and capacitors, this part required four key components to work:

- Microcontroller on breakout board (Z & P provided this)
- FT230X Serial to UART IC
- Micro USB connector
- Connector for the debugger

The four key components were connected on the breadboard. Wires were soldered onto a debugger connector that had been cut off a broken MAS_V2_R2 board. The initial plan was to reuse some components from old MAS_V2_R2 boards to avoid having to create breakout boards for all of the surface mounted components. The first setup therefore used an FT230X from a broken MAS_V2_R2 board, with wires soldered onto the solder pads under the IC pins. The wires broke off easily, and bad connection between one of the IC pins and its solder pad made the setup very unstable. The wire solution was attempted for the USB connector as well, but it was soon decided to discard the idea of recycling surface mounted components; with the exception of one LT6656 voltage reference.

After successfully connecting the four key components, in addition to a LED for status tests, on the breadboard, it was possible to establish contact with and upload code to the

microcontroller. The first code uploaded simply turned the status LED on and off to show that the microcontroller did indeed respond as desired.

Next, it was decided to upload the eZ Sense code, modified as explained in the first part of section 2.4.1. All code related to the ADS1113 and LMP91000 removed, and the microcontroller was instead programmed to return only static values when addressed by the ZT GUI. This set the base for further expansions of the prototype setup, and the learning process that came along with it.

The next step was to add an ADS1115. The ADS1113 code was reinstated and modified to fit the ADS1115, which at the time meant to update the part of the ADS1115's Config register that controls the input multiplexer. Tests were performed by applying a potential at each of the four analog input pins to confirm that the voltages were converted as expected for single-ended channels.

The first LMP91000 was added to the system after confirming that the ADS1115 functioned properly. At this point, the original LMP91000 code was still used, with an external TIA feedback resistor set to 82kΩ.

The 2.5 V external reference was initially produced by a DC power supply, which proved to be too unstable. The supply was later replaced with an LT6656 voltage reference from a MAS_V2_R2 board, connected by soldering wires onto the IC pins.

A test similar to the constant current test in section 7.7.2 was conducted by connecting a AAA battery, a 1 MΩ resistor and an ammeter. The test showed that the current presented in ZT GUI was indeed the same as the one measured with the ammeter, confirming that the setup was correct.

The request for temperature measurements came around this time. Modifications were made to the code, allowing for a change of LMP91000 operation modes. Examination of the voltage on the V_{OUT} pin while in temperature mode seemed reasonable when compared to the temperature-voltage relationship table in the LMP91000 datasheet, explained in section 2.4.3.7.

Experimentation with measurements on multiple channels on the ADS1115 began after testing the temperature mode. At first, modifications were made to the ZT GUI to support the extra data, but the development of the final GUI, explained in section 2.4.3 began shortly after.

The initial plan was to add a second LMP91000 immediately after getting the first operational, but the second one provided by Z & P did not work. It was therefore decided to first add the second ADS1115 instead, parallel to breakout board production. The second ADS1115 presented some issues that are explained in section 3.2.1.

Having two operational analog-to-digital converters made it possible to measure eight channels. A DC supply and a voltage divider applied different voltages at each ADS1115 analog input so it was easy to see that the measured values came from correct channels.

When the first successful attempt to solder an LMP91000 to a breakout board was completed, it was added to the prototype. The first LMP had its MENB pin connected to ground because there were was no need to disable I^2C communication. This was no longer an option after adding the second device because they both use the same I^2C address. They were connected to their own I/O pin on the microcontroller, and the code was modified to set the pin low when initiating I^2C communication.

The remaining LMP91000's were added one at a time, each time followed by testing to make sure they functioned as normal. It was decided to add a 3:8 demultiplexer to control the MENB pins. Each LMP91000 MENB pin was connected to one of the eight outputs on the demux. These outputs are held high until the microcontroller initiates I^2C communication with one of the devices, pulling the assigned output low. This reduced the amount of MENB

connections to the microcontroller from eight to four (three address inputs and one enable signal to the demux), and allowed for a little less complex PCB design. The demux used on the breadboard was a through-hole mounted IC manufactured by Motorola (USA).

Adding the rest of the potentiostats marked the end of the breadboard prototyping. The PCB design was initiated, but the code development continued to use the breadboard setup.

2.3.2 Printed Circuit Board

2.3.2.1 Design

The software used for designing the breakout boards and final system PCB was ARES PCB Design, which is a part of the PROTEUS Design Suit 8.1 software. PROTEUS contains both the ISIS Schematic Capture and the ARES PCB Layout. The design of a circuit is dependent on both.

ARES PCB Design comes with a variety of pre-designed components, where each component consists of a physical part, the component itself, and a footprint; which is the shape, pitch and size of the solder pads. All the ICs in need of breakout boards were missing from the component library, meaning that the group needed to create the necessary footprints.

The breakout boards and PCB followed the same procedure for design and fabrication. Only the process for the PCB design is explained in this section.

The circuit was first designed in ISIS schematic Capture, following the circuit layouts developed during the breadboard prototype stage. Next, the ISIS Schematic Capture design was transferred into ARES PCB Layout, where the actual PCB design took place.

During the design process of the PCB, several factors had to be considered:

- ∇ Noise suppression
- ∇ Ground plane/V_{CC} plane
- ∇ Number of layers
- ∇ Spacing between the components
- ∇ Traces
- ∇ Through-hole positions
- ∇ Analog and digital ground

Even though suppression of the 50 Hz noise from the USB was not set as a priority for the ZP2015, general noise reduction was still considered an important factor of the PCB design. To obtain a system as low-noised as possible, the placement of each component was carefully planned.

A general rule followed was to keep traces as short as possible. Long traces can have the effects of an antenna and pick up unwanted RF signals [23].

Also, because I/O pins are connected to internal circuitry in the IC, such as internal clock switching e.g. this internal circuitry can also generate noise in the system. To minimize the IC inlet noise, the use of decoupling capacitors was continued from the MAS_V2_R2 design decoupling capacitors on the IC I/O pins and VCC inlet.

Traces to ground were made as short as possible. This was obtained by creating a ground plane. The bottom of the PCB was dedicated to ground.

The decision of producing the PCB at HBV eliminated the possibility of creating a design with more than two layers. Some of the advantages with a multi-layer board as four layers e.g. are the possibility to have whole layers dedicated to ground and V_{CC}. This allows for very short traces through holes directly to the desired layer. At the same time it leaves more space

for other traces on the component side of the PCB, and the fourth layer works as a traces-only layer, where there is room for high frequency signals to roam freely without disturbing other traces or components [20].

A final advantage of multilayer board, is the ability to separate digital ground from analog ground, as Hank Zumbahlen describes it in "Staying well grounded": "It is a fact of life that digital circuitry is noisy". Meaning that digital circuitry and its components draw large and fast current spikes from its supply. To prevent this type of noise to corrupt the rather fragile analog circuitry, keeping digital and analog ground separated is of great usefulness [21]. This was attempted in the PCB design. However, to manage a controlled separation was close to impossible on a two-layer design.

The size of the LMP91000 breakout boards had a significant impact on the PCB size and component placing. For practical reasons, all sensor connectors were to be placed on one side of the board. It was desirable to have the same conditions for all channels; meaning that the traces from sensor connector to potentiostat should be of equal length. Due to high sensor current amplification in the transimpedance amplifier of the potentiostat, it was also considered important to have as short traces as possible between sensor connectors and potentiostats to minimize any external interference on the signal before the amplification stage. The same was true for the traces between potentiostat and analog-to-digital converters. This required the LMP91000 breakout boards to be placed side by side, effectively deciding the board size. Figure 21 shows the final PCB design in ARES.

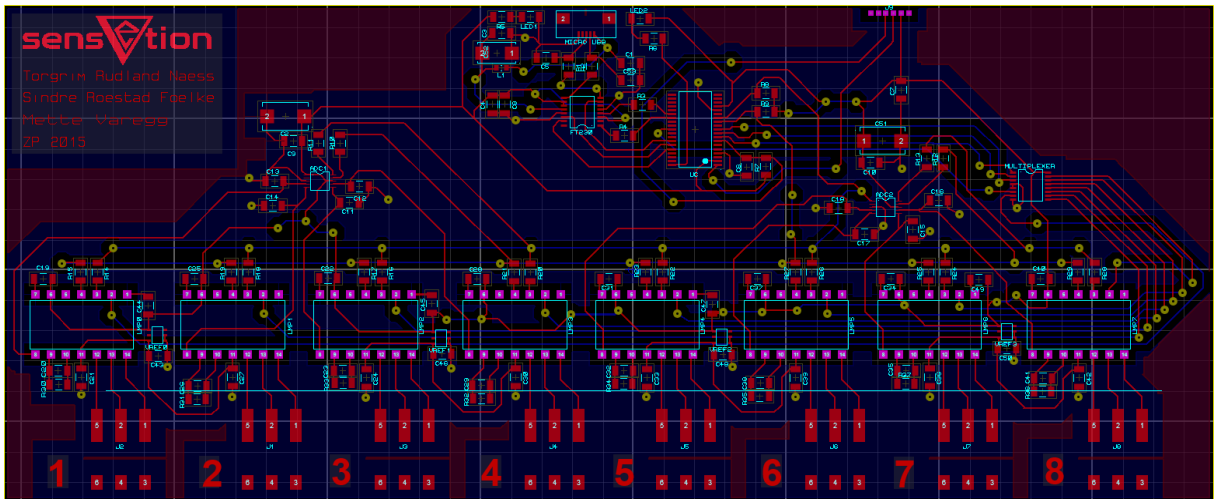

Figure 21. PCB design layout in ARES.

ARES PCB layot provides the following color coding:

- Top copper: Red
- Bottom Copper Blue
- Board edge: Yellow
- Via holes: Green circles with black center

Here one can see all the traces on the component side as red traces, and on the ground layer as blue lines. The I^2C lines (The two blue lines that runs from both sides on the middle of the PCB) were the first lines to divide the ground layer. Ideally, these lines would have been a bit closer to the ADC components, and in that way providing a split of the analog and the digital ground. At the same time, this might have had an effect on the high frequency I^2C data, and

the surrounding components. It was concluded that the importance of the I^2C data trumps the importance of a clearly separated analog and digital ground.

2.3.2.2 Photolithography and Etching

All work with the PCB was performed at the electronics laboratory at HBV. When a finished PCB layout was ready in Proteus, the pattern was printed on a translucent paper, creating a mask sheet.

The substrate used was delivered by Bungard electronik (Germany), and was a presensitized (pre-covered with positive photoresist). The positive photoresist is resistant to the etching agents, and is therefore used to protect the circuits of the PCB. The substrate is exposed to UV light through a printed mask. The translucent parts of the mask allows the UV light pass through on all areas where the copper is to be removed. The UV light alters the chemical structure of the photoresist so it becomes soluble in a developer [22]. The developer used was a mixture of 32 ml Sodium Hydroxid (NaOH) per 2.5 l of H_2O.

After approximately 60 seconds in the developer, the substrate will have all exposed photoresist removed. The substrate is then, to avoid contamination of the etchant, rinsed in tap water before placed in the etching machine. The etching process removes the exposed copper on the substrate, leaving a near finished PCB. The etchant was made of 6 kg Sodium persulfate ($Na_2S_2O_2$) per 24 l of H_2O. To finish the process, the PCB was again illuminated with UV light, and submerged in the developer to remove the final residues of photoresist. The process is illustrated in Figure 22.

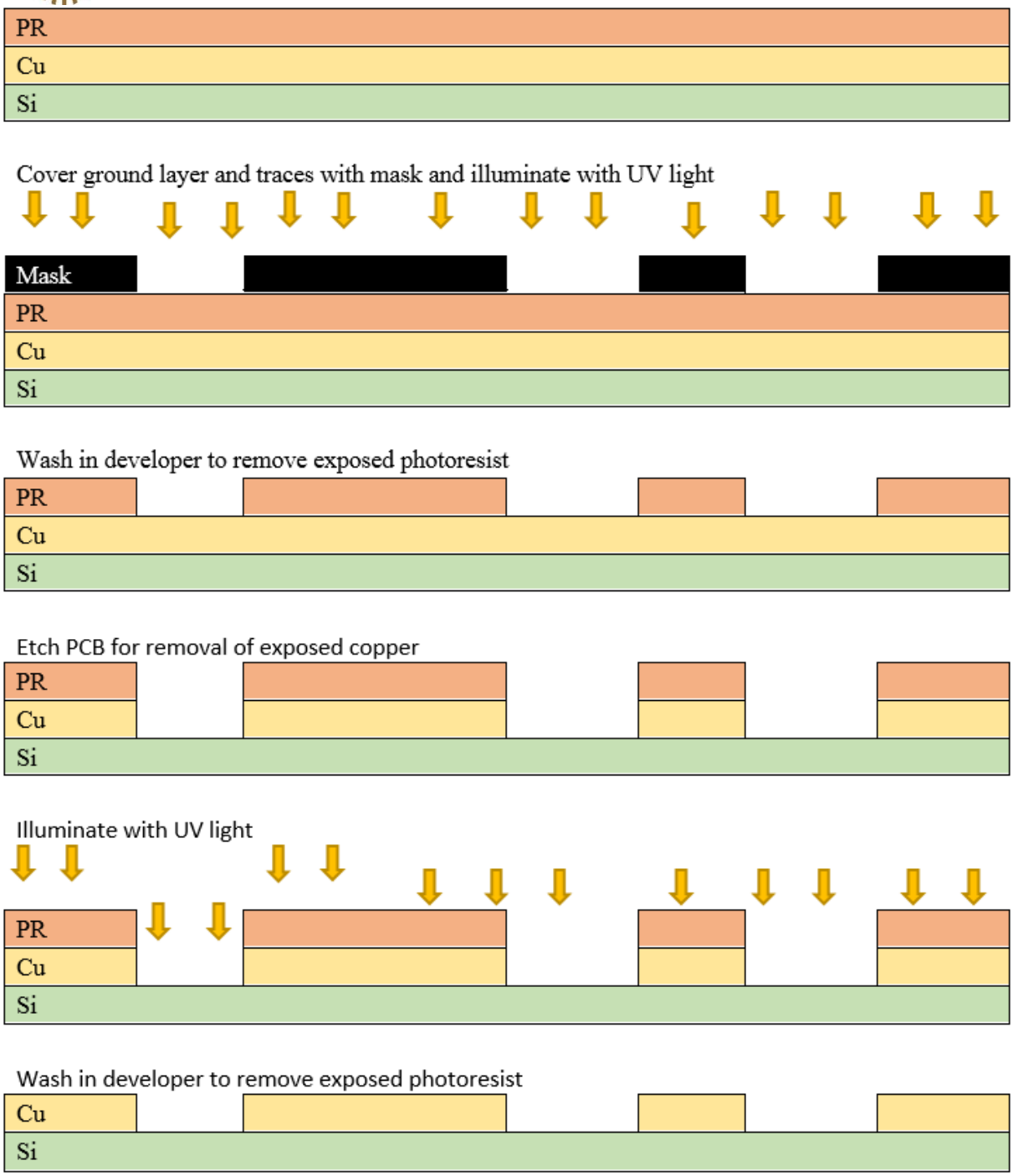

Figure 22. PCB fabrication: Photolithography and etching.

To avoid the etchant removing more than intended of the remaining photoresist and copper, it was necessary to end the process at the right time. The correct etching time is variable due to several factors: Quality of the etchant; the etchant might be old, or well used, making the etchant less efficient. Size of PCB; a larger PCB requires more etching time. Quality and complexity of layout: A well-made layout will have few to none 90 degree angles on the traces; enough space between traces, components, ground and board edge, so that the etcher can easily reach the space in between such places. Therefore, it is of importance to

observe the process, and inspect the PCB with regular intervals in order to stop the etching process at the right moment.

2.3.2.3 Assembly

The mounting was performed by using two different techniques; reflow and manual soldering. Reflow soldering is a process that starts with applying solder paste to the solder pads on the PCB with a dispenser. Some trial and error was required in order to find a suitable pressure and dispensing time; this to make sure the amount of solder paste was correct. Too much can result in short circuiting of IC pins, and the opposite can result in a bad connection, or no connection at all. Surface mounted components are then placed on the PCB, before the board goes through the reflow soldering process in a reflow oven.

For the breakout boards, all ICs except the LMP91000 were soldered by the use of the reflow oven. The LMP91000 was attempted soldered onto breakout boards by the use of reflow oven at first. However, it was nearly impossible to obtain sufficient contact between IC pins and solder pads. Too much solder paste would easily short several pins because of the pin pitch, as illustrated in Figure 23, and too little would not allow for a good connection. The pins on the LMP91000 are mounted underneath the IC, and are just barely reaching around the body on the side, which made it very difficult to perform resistance tests to determine whether a connection was established between IC pin and solder pad.

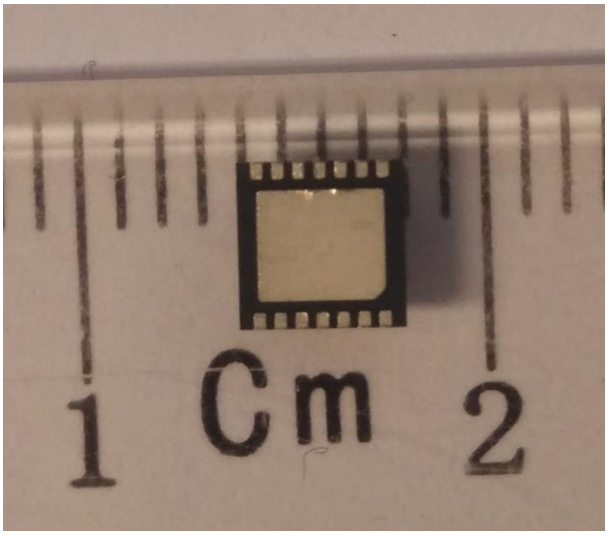

Figure 23. Bottom view of the LMP91000. It is clear that there is narrow margins to solder on, and only a small part of the pad available for soldering on the side.

There was a significant amount of insecurity of how to solve this issue. One idea was to perform a wire bond in the cleanroom at Vestfold Innovation Park. However, the wire bonding would be impractical and time consuming, which lead to the idea being discarded.

However, a temporary solution was needed in order to continue expand the breadboard to two channels. One of the group members, who had a lot of soldering experience, performed a manual wire bond with a soldering iron and a piece of old speaker cable that was found in the electronics lab. This was done to in order to make a quick solution without having to depend on help from the cleanroom laboratory engineer. The process was to glue the IC upside down on the breakout board, then twirl three or four threads of copper wire from the cable and solder these from the IC pins down to the solder pads on the breakout board. The design was

extremely fragile, but gave the group the possibility to expand the breadboard prototype to contain two potentiostats.

In order to solder the LMP91000 components manually, a better solution than the wire bond attempt was needed. The chosen method became to solder a small portion of tin onto the solder pads, then press the IC onto the pads with a set of tweezers. By then heating one of the tin soldered pads, the solder would make contact with the IC pad, and heat it up enough for the solder process to take place. This was repeated on all connections of the IC. By using a microscope, the connections were controlled, and all solder points that did not pass the visual test was then soldered again.

Because of the high number of pins on the ICs to be mounted on the PCB, soldering by the method with reflow oven was preferred on these. The risk of accidentally pushing one or more components off their solder pads while applying solder paste or mounting other components, lead to the choice of using reflow only on the larger ICs. The microcontroller, FT230X, analog-to-digital converters and demultiplexer were soldered with the reflow oven, and the remaining components manually with a soldering iron.

In order to check whether connection with the microcontroller was obtainable, the via holes and the necessary passive components were soldered first, and code was uploaded to see if the microcontroller responded. When contact was successfully obtained, the analog-to-digital converters and its necessary via holes and passive components were soldered. Finally, when both analog-to-digital converters were operational, the LMP91000 potentiostats were soldered one by one, all the while checking the potentiostats functionality. In this way, all faults were detected consecutive, and the origin of fault could easily be detected.

Continuity tests with multimeter were used to control connection to all ICs, for those pins with a bad connection, or no connection to the traces, a manual solder was performed to make sure a proper connection was in place. When all connections was confirmed, a visual inspection was performed in addition to the measurement of resistance over the solder points, which are not to be higher than one ohm as to insure for a proper solder point [23].

2.4 Control System

2.4.1 Microcontroller Programming

The microcontroller was programmed in C, using Texas Instruments' eZ430-F2013 Development Tool, which consists of the Code Composer Studio Integrated Development Environment (IDE) and the MSP-EZ430U debugging interface (Figure 24).

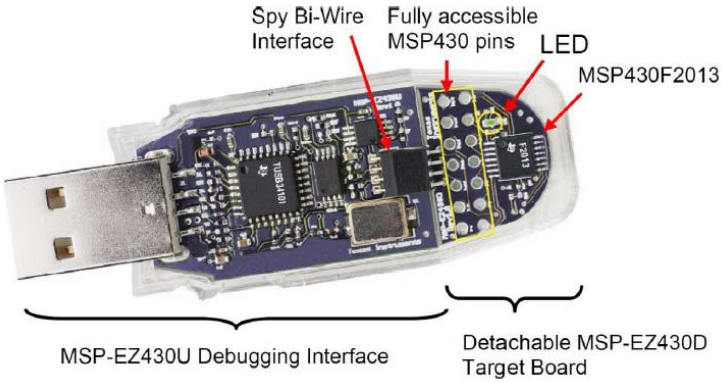

Figure 24. MSP-EZ430U Debugging Interface [24]. With permission from Texas Instruments.

The microcontroller code written for this project is based on the the code from the eZ Sense system. Before new code for the microcontroller could be created, it was necessary to understand the old one. The group was informed that the eZ Sense code is quite extensive and has a lot of excess functionality. In order to simplify the learning process, it was chosen to filter out as much of the unnecessary code as possible before attempting to upload it to the breadboard setup and modifying it to fit the new system.

The first action was to remove all code related to the LCD display, as it had already been decided not to implement one on the new prototype. Next, the entire code was examined systematically and every function that seemed to not be used anywhere was removed, each time compiling and uploading the code to the MAS_V2_R2 to make sure it still functioned as normal. The remaining code was spread out over a multitude of source- and header files. The original idea was to modify the eZ Sense code directly in these files, but as many of them had very little code left, a new set of files were created.

This was done for two reasons; one was to ensure a logical file structure with code for the different parts of the system bundled together in their own file sets, and the other was to avoid any remnants of unused code that might cause confusion in future development.

Although many new code requirements revealed themselves as the project evolved, these were the most obvious changes that would have to be implemented:

- ∇ Configure microcontroller pins for the extra components
- ∇ Change the ADC register setup to fit ADS1115 with four single-ended input signals instead of the ADS1113 with one single-ended input.
- ∇ Change communication to handle more data
- ∇ Add functions to control the increased number of channels

The majority of the work was related to the ADS1115 and LMP91000. The source files for these components contains mostly new code with some elements loosely based on the ez Sense code. The remaining files were cleared of unused code and modified as needed.

2.4.2 Microcontroller Program Structure

The microcontroller's program structure is illustrated in the flow chart of Figure 25. The following sections covers only the most important parts, but more details can be found in the source code comments in *Booklet*.

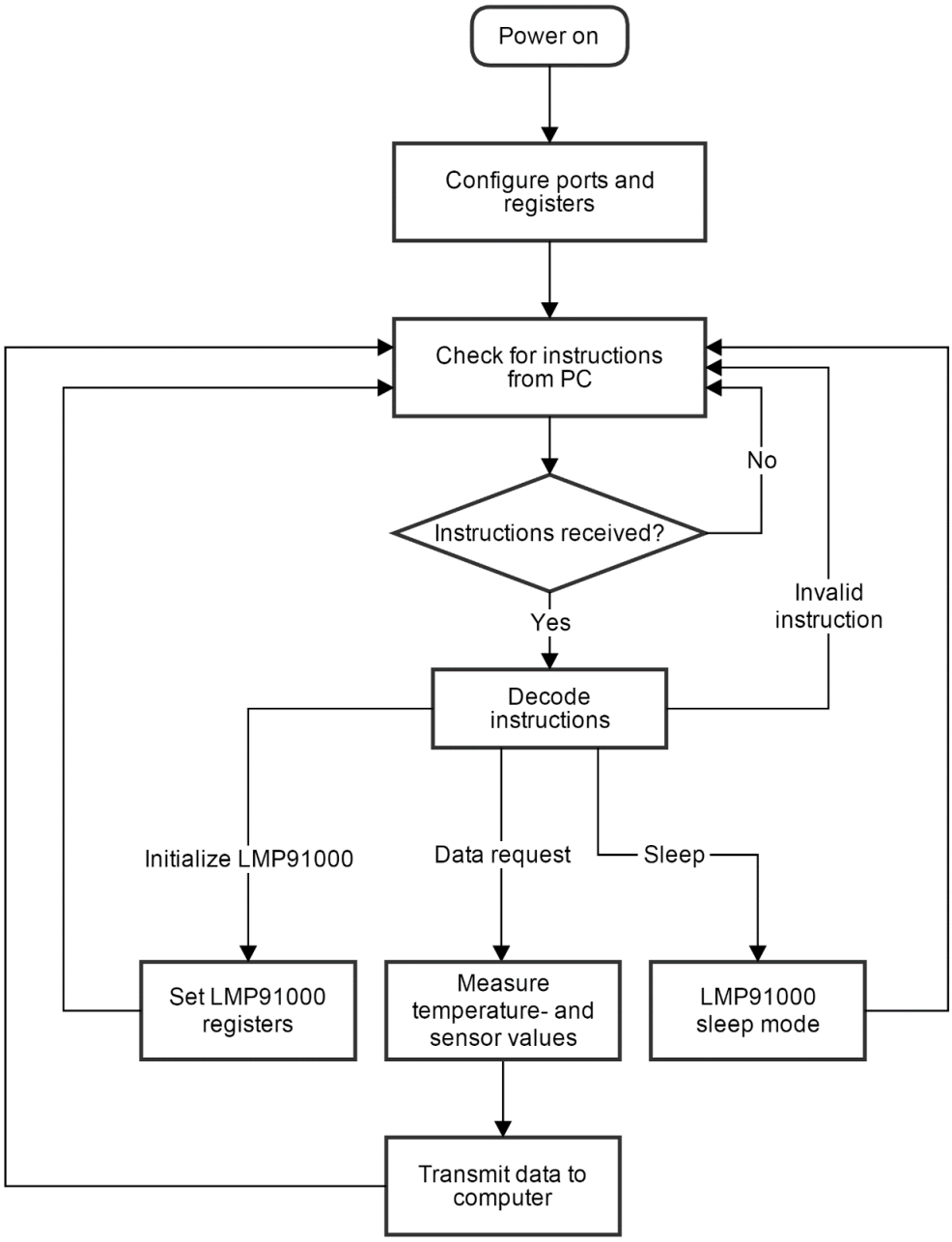

Figure 25. Microcontroller program flow.

2.4.2.1 Configure Ports and Registers

When the system powers on, it starts by configuring ports and registers of the microcontroller in `main` (*Booklet*, section 1.3). This specifies communication methods, clock frequencies, inputs and outputs (I/O) pins and their functionality in the microcontroller.

I/O pins 1, 2, 4 and 5 in port 3 are set to their primary peripheral module functions (I^2C and UART), and pin 7 of port 1 is set as output for the status LED. All other unused I/O pins have been left unconnected to the PCB and are configured as I/O function with output direction, as recommended in the MSP430x2xx Family User's Guide [20]. This is to prevent floating inputs and reduce power consumption.

2.4.2.2 Check for instructions from GUI

The microcontroller periodically checks if it has received instructions from the GUI. This is done by using an infinite loop (*Booklet*, section 1.3, line 72–108) in `main`. On each run-through, `uart_Enable_Receiver` (*Booklet*, section 1.5, line 68–84) returns the address to the character pointer *pNoChar*, which holds the number of characters received by UART. An if statement (*Booklet*, section 1.3, line 82) uses *pNoChar* to check if the number of received characters is greater than zero. If it is, the received instructions are decoded. If not, the loop starts over.

The eZ Sense performs measurements and displays them on the LCD display if no instructions have been received after a certain period of time. This part of the code was removed in from the ZP2015, as it was decided not to use LCD displays. Otherwise, this section of the code remains unchanged.

2.4.2.3 Decode Instructions

Received instruction data are placed in an array named *UART_buffer*. *UART_buffer* has nine elements as shown below. A switch statement (*Booklet*, section 1.3, line 87–106) checks the value of *UART_buffer* element 0, which holds the value representing the type of instruction to be executed. The switch statement has one case for each instruction; each one described in the next three sections.

Element [0] **Main instruction**
Determines the type of instruction to be executed.
'1': Initialize LMP91000
'2': Perform measurements
'3': LMP91000 sleep mode

Element [1] **TIA gain (when element [0] = '1')**
Contains a number representing TIA feedback resistor value.
'0': 2.75 kΩ
'1': 3.5 kΩ
'2': 7 kΩ
'3': 14 kΩ
'4': 35 kΩ
'5': 120 kΩ
'6': 350 kΩ
'7': External resistor

Element [1-8] **Channel selection (when element [0] = '2')**
Determines which channels to measure.
'0': Do not measure channel
'1': Measure channel

The eZ Sense code had separate cases for starting a conversion, and for reading and transmitting conversion data to the computer. It was chosen to combine the two by having a single case to perform all operations and automatically transmit data when conversions are finished. This was done both to ensure that a read instructions would not be sent prematurely and to have a cleaner `main` function.

A few other cases that were originally used to send calibration constants for calculating glucose values on the microcontroller and choosing unit values for the data shown on the LCD, were removed.

2.4.2.3.1 Case 1: Set LMP91000 Registers

Case 1 initializes the eight LMP91000 devices. The microcontroller addresses them one at a time via the I^2C interface and writes to the registers specifying operation modes, TIA gain, voltage source references and bias settings.

The case was added for the possibility to change the LMP91000s TIA gain settings from the GUI. MAS_V2_R2 use an external 82 kΩ resistor, but using the integrated variable feedback resistor instead makes the system much more flexible.

Initially, case 1 only changed the TIA gain and the LMP91000 initialization was done on system startup as in the eZ Sense, but this was changed because the initialization function sets the gain anyway, and it seemed unnecessary to perform the task twice. In addition, it saves power because the LMP91000 enters the Deep Sleep mode by default when powering on.

The eZ Sense needed only to address a single LMP91000. When more channels were added to the system, a way of communicating with all the devices independently was needed, in addition to a way of choosing the value of R_{TIA}.

The function `initialize_LMP91000` (*Booklet*, section 1.1, line 170–237) was created for this task. It runs through a loop and sends the values to be written into the control registers of each LMP91000 through the I^2C interface. Communication is enabled for each device with `enable_LMP91000_i2c` (*Booklet*, section 1.1, line 13–80) and disabled with the *DISABLE_LMP91000_I2C* macro (*Booklet*, section 2.1, line 22).

A switch statement reads the value of element 1 in *UART_buffer*, which is taken in as a function parameter. Element 1 specifies the TIA gain. There are eight different gain settings, including external resistor. Solder pads for an external feedback resistor were added to the PCB to allow the use of external resistors.

The register setup after initialization is listed in Table 2. It is the same as in the eZ Sense, except for the feedback resistance. It was considered to let the LMP's remain in Deep Sleep mode even after initialization because they are set to 3-lead amperometric mode before each measurement, as explained in section 2.4.2.3.2. The only problem was that it takes some time after the transimpedance amplifier is turned on before accurate voltage levels appear on the LMP91000s V_{OUT} pin; a quick test indicated as much as 100 milliseconds with the highest feedback resistor values. It was decided to let that operation be performed one additional time rather than add unnecessary delay to the code.

Table 2. LMP91000 register setup after initialization.

TIACN - TIA Control Register	
TIA feedback resistance	As chosen in the GUI (or 120 kΩ default)
R_{LOAD}	100 Ω
REFCN - Reference Control Register	
Reference voltage source	External
Internal zero selection (percentage of source reference)	20 %
Bias polarity	Positive
Bias selection (percentage of source reference)	24 %
MODECN – Mode Control Register	
Mode of Operation	3-lead amperometric cell

2.4.2.3.2 Case 2: Measure Sensor- and Temperature Values

Case 2 means that the GUI has sent a request for sensor- and temperature data. Elements 1 through 8 of *UART_buffer* are checked to determine whether a measurement is to be performed on the respective channels; each element corresponds to a channel number. This is done in the `take_measurements` (*Booklet*, section 1.2, line 147–260).

The operation modes of all LMP91000's are first set to 3-Lead Amperometric before analog-to-digital conversions are initiated on the chosen channels. The digitized voltages in the ADS1115s conversion registers are stored in an integer array named *conversion_data*, visualized in Table 3. If a channel is not to be measured, a 0 is stored instead. It was thought to have an array of variable size and store only measurement values, but this would make it more difficult for the GUI to know what data belongs to which channel. Because of this, the easy method of each channel having a designated location in a fixed-size array was chosen.

After completing conversions of sensor data, the LMPs are set to Temperature Measurement mode before the conversion process is repeated for temperatures.

Table 3. The *conversion_data* array.

Index 0	**Index 1**	**Index 2**	**Index 3**	**Index 4**	**Index 5**	**Index 6**	**Index 7**
Channel 1 sensor data	Channel 2 sensor data	Channel 3 sensor data	Channel 4 sensor data	Channel 5 sensor data	Channel 6 sensor data	Channel 7 sensor data	Channel 8 sensor data
Index 8	**Index 9**	**Index 10**	**Index 11**	**Index 12**	**Index 13**	**Index 14**	**Index 15**
Channel 1 temperature data	Channel 2 temperature data	Channel 3 temperature data	Channel 4 temperature data	Channel 5 temperature data	Channel 6 temperature data	Channel 7 temperature data	Channel 8 temperature data

Analog-to-digital conversions are not done from channel 1 through 8 in ascending order. To be more efficient, one conversion is initiated on each ADS1115, followed by a small delay before the conversion register values are read. The conversion order of operation is illustrated in Table 4. It can likely be done even more efficiently by reading one conversion result while the conversion is performed on the other device. This was deemed unnecessary for our current system version as the smallest measurement interval is one second.

Table 4. Order of operations.

Channel	Operation
1	begin conversion
5	begin conversion
1	read conversion data
5	read conversion data
2	begin conversion
6	begin conversion
2	read conversion data
6	read conversion data
3	begin conversion
7	begin conversion
3	read conversion data
7	read conversion data
4	begin conversion
8	begin conversion
4	read conversion data
8	read conversion data

2.4.2.3.3 Case 3: LMP91000 Deep Sleep Mode

Case 3 happens when a session is terminated by the *Stop Sensing* button in the GUI. Each LMP91000 mode of operation is set to Deep Sleep in `LMP_all_sleep` (*Booklet*, section 1.1, line 152–160). This case is intended to reduce power consumption when the system is idle.

2.4.2.4 Transmit Data to Computer

Two ways of transmitting data to the computer were considered; one was to send data values separately and the other was to send them all at once. The first option would have to involve adding an index bit or byte the GUI could use to know which channel number the data belonged to. For simplicity, the second option was chosen.

After finishing conversions on all channels, the entire *conversion_data* array is transmitted to the computer. This happens in almost the same way as in the eZ Sense system; by a function called `uart_transmit` (*Booklet*, section 1.5, line 50–66). Although the eZ Sense transmits only a single value, `uart_transmit` could still be used to transmit an array of values without modifiactions. The only difference is that instead of providing the function with an address pointer to a single integer, it gets a pointer to an array of integers instead.

Each 16-bit value in *conversion_data* is divided into two bytes, sent one at a time. The two bytes are converted back into a 16-bit integer on the receiving side. There are some issues with the transmission, as will be discussed in section 3.2.3.1.

2.4.2.5 Troubleshooting

During the breadboard prototyping, it was sometimes needed to determine whether contact could be established with an LMP91000 on the I^2C bus. If a single LMP was offline or otherwise non-functional, the entire system would time out. A function named `check_LMP_status` (*Booklet*, section 1.1, line 246–270) was created for this task. The

function returns a 1 if the device is reachable on the I^2C bus and a 0 if otherwise. This, combined with the status LED, allowed a simple way to troubleshoot the system.

The function was also used to fill an array with the status of each LMP which was sent as a parameter to the early version of `initialize_LMP91000` (*Booklet*, section 1.1, line 301–374). When initiating the devices, each array element corresponding to one device, would be checked. If 1, normal initiation occurred, and if 0, it would be skipped. Although the user would not be alerted if one or more devices were offline, it provided a method to avoid timeouts so the system could run as normal.

The final code version does not implement this, but the functions are still kept in case of future development and troubleshooting processes. There is also a similar function to check the status of ADS1115 devices, named `check_ADS_status` (*Booklet*, section 1.2, line 264–284).

2.4.3 Computer Software Programming

The second part of the control system is the computer software, hence known as ZP GUI. It processes data received from the microcontroller and presents it to the user in real time, both numerical and graphical. All data is also stored in log file that can be opened in programs such as Excel and Matlab.

The application is based on ZT GUI v.1.3.0, and was programmed in Microsoft Visual C# using Microsoft's IDE, Visual Studio 2013. The whole application had to be completely revamped in order to support multiple channels. Not much of the original code remains, but the general structure and program flow are more or less the same. The following sections will describe the different parts of the program and how they work.

As previously mentioned, early testing with the MAS_V2_R2 and breadboard setup was performed with a modified version of ZT GUI. There were many temporary solutions from trial and error during this phase. When the time came to create the final GUI, a new code project was created and only the necessary blocks of code were copied, in the same way as for the microcontroller code.

2.4.3.1 ZP GUI Program Structure

The program structure is very similar to the ZT GUI, with some exceptions that will be pointed out later. Figure 26 shows the program flow while measuring.

After the measuring process has been started, the program repeatedly sends commands to the microcontroller. The first is always to initialize the LMP91000 devices and contains the TIA gain chosen by the user. Subsequent commands are data requests, which are sent at timed intervals, explained in section 2.4.3.4.

If the microcontroller responds by returning conversion data, the program will read and process the data before presenting them on screen, otherwise the program will time out.

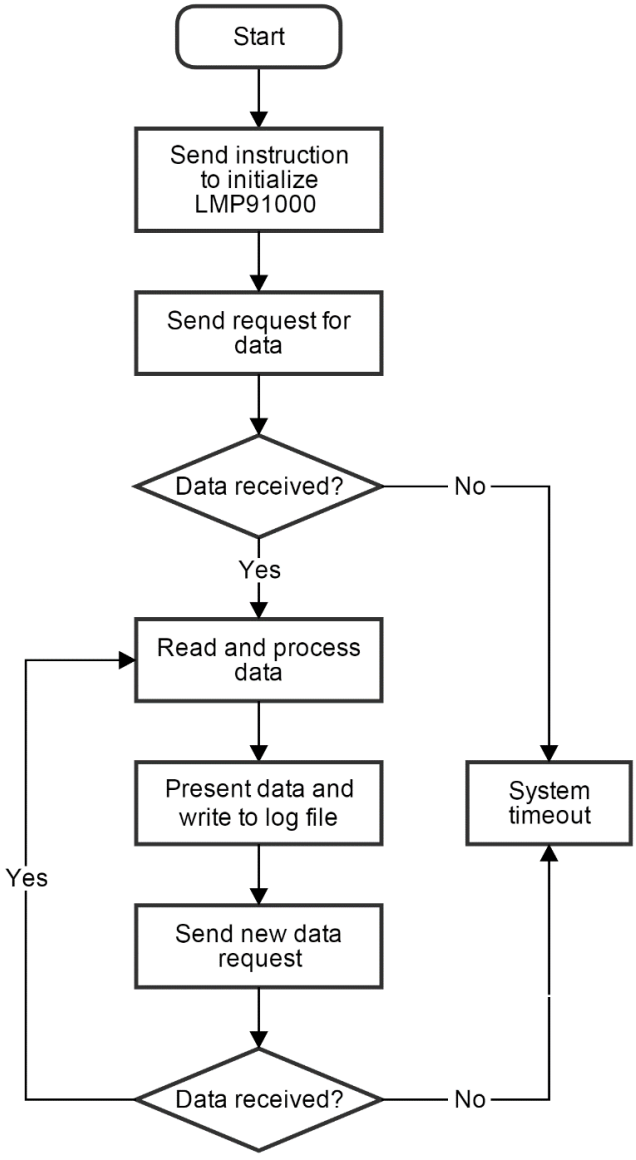

Figure 26. ZP GUI program flow.

2.4.3.2 Main Window

Figure 27 shows the ZP GUI design. When compared to the ZT GUI in Figure 16, one can see that the basic design has been kept, although modified to fit eight channels.

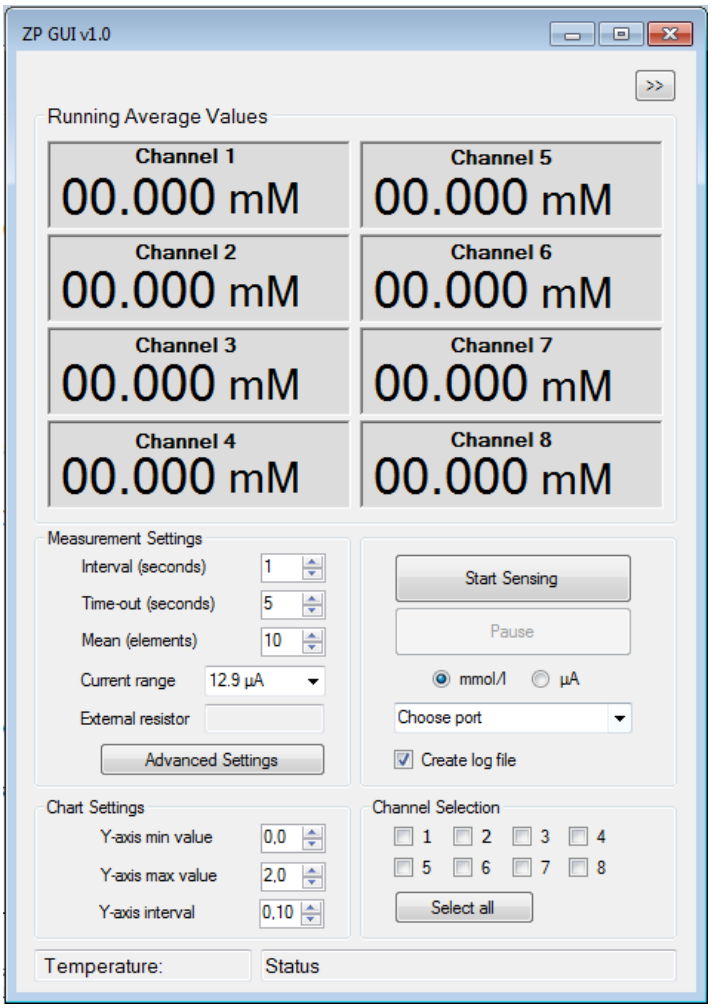

Figure 27. ZP GUI.

2.4.3.3 Options and Settings

There are a variety of options and settings that can be chosen for the system. Most settings, except for choosing a serial port, are optional because they have been given default values. Starting the program will lock all controls except for the pause- and stop button, so the user must finish setting modifications before initiating the sensing process.

2.4.3.3.1 Measurement Settings

The group box labeled *Measurement Settings* contains options for timing, averaging and current ranges. Interval and Time-out sets the interval properties of *timerData* and *timerTimeout*, explained in section 2.4.3.4.

Mean elements specifies the length of the arrays stored in *voltsForAvg*, which holds previous voltages from every channel, used for calculating the running average. This is explained in further detail in section 2.4.3.8.1.

The TIA gain is selected in the drop-down list under Measurement Settings. The gains are presented as current ranges so it will be easy for the user to choose correct values. The different TIA gains are listed in section 2.4.2.3. There is also an option to choose external resistors. If this is selected, the resistor value must be entered in the text box below. The program will not start the sensing process before a valid resistor value has been entered.

Current ranges are calculated using (5), where *FS* is the maximum voltage the ADS1115 can read with the chosen PGA settings, as explained in section 2.2.1.2, and V_{zero} is the LMP91000 internal zero. The factor 10^6 is there to give a result in terms of microamperes. Selecting a current range automatically updates the *R_TIA* variable with the TIA feedback resistor value used for converting ADC values to voltages. The current ranges for this system are set using an FS of 2.048 V and V_{zero} of 0.5 V.

$$Max\ current\ (\mu A) = \frac{FS - V_{zero}}{R_{TIA}\ (\Omega)} \cdot 10^6 \qquad (5)$$

The advanced Settings button opens the form window in Figure 28, with options to change k- and c-values for every channel, in addition to the temperature range. The arrays *k_val* and *c_val* and variables *T1* and *T2* from the main form are updated with values from the numeric boxes when *Save and close* is pressed (*Booklet*, section 3.2, line 39–62). The *k_val* and *c_val* arrays contains the k- and c values used for converting currents to glucose values *T1* and *T2* represents the lowest and highest temperatures used in the linear approximation of the LMP91000 temperature sensor values. This is covered in section 2.4.3.7.

Closing the *Advanced Settings* window with the x in the upper right corner will generate a dialog box, warning the user that any changes made will be discarded. Clicking *No* will leave the window open and *Yes* will close it and discard any changes.

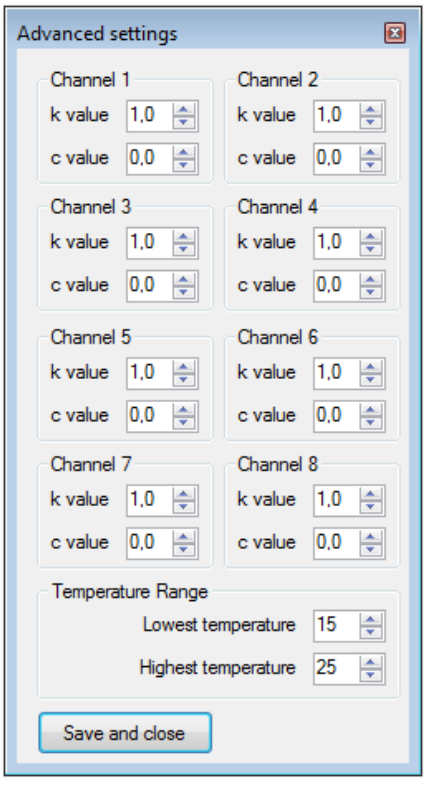

Figure 28. Advanced settings form window.

2.4.3.3.2 Chart Settings

The group box labeled *Chart Settings* allows the user to change minimum- and maximum values of the Y-axis, as well as axis intervals of the chart described in section 2.4.3.8.5. It was thought to let this be done automatically or allow changes during measurements, but this resulted in somewhat erratic behavior from the chart. It was not considered to be particularly important, so no further time was spent on trying to find better solutions.

2.4.3.3.3 Channel Selection

The check boxes in the Channel Selection group box makes an array of check boxes named *channelCheckBoxes*. The array is used in all parts of the program that needs to know which channels are selected, such as the creation of command strings and data presentation. Clicking the *Select all* button selects all channels and changes the button text to "*Remove all*". Clicking it again will deselect the channels (*Booklet*, section 3.1, line 179–206).

2.4.3.4 Initialize the Sensing Process

After the start button is pressed (*Booklet*, section 3.1, line 384–496), the program is controlled by two timers; *timerData* and *timerTimeout*. *timerData* is used to periodically send commands to the microcontroller and *timerTimeout* stops the program if the microcontroller is nonresponsive. The method of using timers is adopted from ZT GUI. The start button text will also change to "*Stop Sensing*". Pressing it again will close the serial port, stop both timers and unlock all controls. *Pause* (*Booklet*, section 3.1, line 220–237) will do the same, but allows the process to be started again without clearing all previous data.

Upon start, the program first checks if the grid view tabs contain any data from previous measurement sessions. If they do, the dialog box in Figure 29 appears. The user must confirm that a new session is to be started to avoid the risk clearing grids and charts by accident. A similar dialog box appears if the user attempts to close the program while a measurement session is in progress.

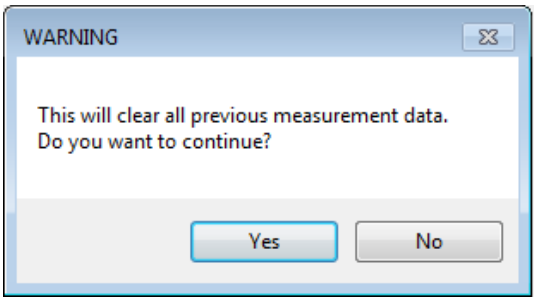

Figure 29. Dialog box to confirm the start of a new session.

2.4.3.5 Send Instructions to the Microcontroller

When sending commands to the microcontroller, the GUI writes string data to the serial port using the `SerialPort.Write` method. The command to initialize LMP's is sent before the timers are started in `start_sense` (*Booklet*, section 3.1, line 505–551). It is a string of two numbers, the first is "1", which tells the microcontroller what type of command it is, as explained in section 2.4.2.3, and the other is a number from 0 to 7 that represent different TIA gains.

Every time the interval time of *timerData* elapses, `ReadADC` (*Booklet*, section 3.1, line 607–636) will send data requests to the microcontroller. In ZT GUI, there were two commands; one to begin a conversion and one to read conversion data. ZP GUI sends a string named *serialCommand* instead. This string is composed of nine characters. The first is the number "2", which tells the microcontroller that the command is a data request. The remaining eight are either "1" or "0" decided by the *channelCheckBoxes* array. For example, *211000011* means that measurements should be made on channel 1, 2, 7 and 8.

The last command is the number "3", which tells the microcontroller to set the LMP's in Deep Sleep mode (*Booklet*, section 3.1, line 573–592). It is sent when the clicking the *Stop* button.

2.4.3.6 Receive Data from the Microcontroller

When 32 bytes are available on the serial port, a DataReceived event is fired (*Booklet*, section 3.1, line 641–647). The number of bytes are specified by the port's *ReceivedBytesThreshold* property. The event invokes `RS232DataReceived` (*Booklet*, section 3.1, line 696–885), which is the method used to read and process data from the serial port. This procedure is identical in both ZT GUI and ZP GUI, apart from the number of bytes required to fire the event, and the extensive modifications of `RS232DataReceived`.

Each data value is represented by two bytes in the input buffer. These bytes are converted into 16-bit integers and written to *AD_values*; an array similar to *conversion_data* on the microcontroller. There have been some problems with the number of bytes read, which lead to the creation of a separate method to read data from the input buffer and inform if something goes wrong (*Booklet*, section 3.1, line 652–686). This is more thoroughly described in section 3.2.3.1.

If no data is received during the Time-out interval, *timerTimeout* will fire an event which terminates the measurement process and shows the message "*Timed out…"* in the status text box (*Booklet*, section 3.1, line 560–566). `RS232DataReceived` restarts the timer before data is read from the input buffer to prevent this from happening.

2.4.3.7 Signal Processing

Voltages, currents, and glucose values are calculated from *AD_values* and stored in their own arrays. The equations listed below are the same as in ZT GUI, but they are performed on arrays instead of single values (*Booklet*, section 3.1, line 752–773).

$$volts = 2.048\,V \cdot \frac{(ADC\ value)}{2^{15} - 1} - V_{zero} \qquad (6)$$

$$microcurrent = \frac{volts}{R_{TIA}} \cdot 10^6 \qquad (7)$$

$$glucose = k \cdot microcurrent + c \qquad (8)$$

Equation (6) is used to convert the digital value from the ADS1115 to a voltage representing the output from the LMP91000. It is the same as (4) from section 2.2.1.2, except that the LMP91000 internal zero voltage, $\mathbf{V_{zero}}$, set to 20 percent of the external voltage 2.5 V reference, is subtracted.

Sensor currents are found using with Ohm's law and multiplied by a factor 10^6 to get a value in microamperes in (7). $\mathbf{R_{TIA}}$ is the transimpedance feedback resistor.

The microcurrents are converted to a glucose concentration in mmol/L in (8). The constant **k** makes the glucose value proportional to the sensor current. **c** is a calibration constant for adjusting the glucose concentration offset.

The temperature sensor response has a slight downward parabolic shape [16]. To compensate for this, a linear approximation can be performed over a range of temperatures from a table of temperature-voltage relations in the LMP91000 datasheet. Equation (9) is a general expression for a temperature T, in the range from T_1 to T_2. V_1 and V_2 are the corresponding output voltages in millivolts. In the code, V_1 and V_2 are given their values from `CelsiusToMilliVolts` (*Booklet*, section 3.1, line 914–971) which converts the temperature range values according to the table in the LMP91000 datasheet.

$$T = (V - V_1) \cdot \frac{T_2 - T_1}{V_2 - V_1} + T_1 \qquad (9)$$

2.4.3.8 Data Presentation

2.4.3.8.1 Running Average

ZT GUI has a display that shows running average values with three one of three unit values; mmol/L, µA or mg/dl. The mg/dl is not used in ZP GUI because it had been a request from a specific customer, and not particularly relevant for a laboratory instrument. ZP GUI use eight displays; one for each channel.

Originally, the mmol/L and µA was calculated from the average of the last *nAvg* ADC values, where *nAvg* is the mean elements value chosen from the Measurement Settings. This was changed because integer division discards any decimals and thus produces a small error in succeeding calculations. Now, the *voltsForAvg* array is used instead. voltsForAvg holds one array for each channel. `FindAverage` (*Booklet*, section 3.1, line 890–909) takes two parameters; the array of previous voltages and along with the latest voltage value and returns the average. The currents and glucose values to be displayed are calculated from this.

2.4.3.8.2 Grid View

ZT GUI lists all data values in textboxes in the two tabs that appear on the right side when the arrow button in the upper right corner of figure something is clicked; one tab for mmol/L or µA and one for ADC. ZP GUI use grid view with nine columns for this task. The left-most column shows the time, and the remaining eight holds data values for their respective channels. There are six tabs; one for the chart, explained in section 2.4.3.8.5, and grid views for sensor ADC, mmol/L, microamperes, temperature and temperature ADC. During the development process, the temperature ADC tab displayed voltages so it was easy to confirm that the ADS1115 readouts were correct by measuring the LMP91000 outputs with a voltmeter.

All values from selected channels, except for the average temperatures, are converted and stored in string arrays. If a channel is not selected, "*N/A*" is used instead of measurement values.

`AutoscrollGrids` (*Booklet*, section 3.1, line 1153–1169) makes sure that the grid scrolls down to the latest values when all visible rows are filled.

2.4.3.8.3 Temperature

The temperature in the bottom-left corner of the GUI is the average of temperatures measured on the LMP91000 devices on all active channels. It is calculated by dividing the sum of all temperatures by the number of active channels (*Booklet*, section 3.1, line 762–779). The Channel Selection check boxes are used to determine whether a temperature is to be a part of the sum.

2.4.3.8.4 Log file

All data from the selected channels is stored in a log file. If the Create log file check box is checked while pressing Start, the `LogInit` (*Booklet*, section 3.1, line 1081–1129) is called. `LogInit` creates a log file directory if it does not already exist, and creates a .csv file in this location. The csv format was chosen because it opens easily in Excel. The log file name consists of the date and time it was created. ZT GUI logs data in a temporary file and creates the log file when stopping the program. It was chosen to create the log file on start and write

directly to this file while running instead in case something goes wrong, such as a program crash or the computer losing power.

Data is written to the file entire rows at a time in the form of strings where each value is separated by a semicolon. Excel will automatically place every value in their own columns when the file is opened. The strings are created by adding together rows from the grid views.

2.4.3.8.5 Line Chart

A real-time graphic presentation of data was set as a secondary objective if given enough time, but it was moved up on the priority list because it makes it much easier to monitor multiple channels when testing the response of multiple sensors at the same time. In addition, two of the group members had already implemented a similar feature in a project the previous semester, so much of the code could be reused with only small modifications.

The line chart has glucose concentrations on the Y-axis time on the X-axis. Each channel is represented with a unique color. The chart is initialized in `InitChart` (*Booklet*, section 3.1, line 1044–1077) when a measurement session begins.

2.5 Testing

According to the pre-project report, testing and measurements of the complete system followed as the final product was completed. In order to manage a successful test of the entire system, the group had to make sure both the electronics part; PCB, and the mechanical part; Microfluidic flow cell, worked as the theory suggested.

This part of the project was considered to be of great importance, and was documented as detailed as possible in order to be able to repeat the tests under as equal circumstances as possible, and to be able to reconstruct the tests later.

The PCB self-noise test and the PCB test with constant current on sensor input would, under ideal circumstances, be performed with the PCB in a faraday cage. This was however, not an option at the time due to the only available faraday cage being occupied. Therefore, all tests described here, except from the temperature tests, which were conducted in a temperature cabinet, were conducted at one location, which leaves less room for variations in external noise sources.

The hydrogen peroxide test in section 2.5.4 was merely a response test for all channels. For this test, a random selection of sensors were chosen. Therefore, in order to obtain data comparable between the usage of a magnetic mixer and a MFC, and to answer the research question raised in the introduction, a set of tests performed with two randomly selected sensors, hereby referred to as sensor 1 and sensor 2. Originally these tests were thought to be performed with the electrochemical glucose sensors. However, the enzyme layer used to oxidize glucose is not reliable, and suffers from a relatively large decay rate. This means that the repetition of tests, and use of the same sensors will not be adequate for tests that are to characterize the system. All measurements were therefore performed with the use of Hydrogen Peroxide as a model compound since this is the by-product from the glucose conversion process that is oxidized by the sensor as a function of glucose concentration.

2.5.1 Test of the Microfluidic Flow Cell

The main objective of the tests of the MFC were to find out the flow rate, flow speed and sensor response of the system, the functionality of the different designs and if there were some other difficulties. The MFCs used for the test are design 2, 3 and 4 from wafer 2 from Exercise 1. The design 1 was used from the wafer 1, Exercise1. To drive the liquid through the system, a peristaltic pump were used. From the technical information sheet [25], the maximum speed at 10 rpm with the original tubing (bore mm 1.02) gave a flow rate of 0.7ml/minute ≈ 0.012 cm^3/sec. To increase the flow rate, the tubings of 1 mm diameter were changed with a tubing that measured 3 mm.

2.5.1.1 Finding the Flow Rate

The flow rate was found by pumping DI water mixed with red food coloring through a tube marked every 5 cm over 20 cm while filming with a video camera. The pump was used at 10rpm and 5rpm to see how it affected the flow rate of the different designs. When the experiment was finished, the film was played in Windows Movie Maker, which has the possible to stop the film to find the exact time it passes a point. The flow rate is decided by the speed of the flow multiplied by the cross section. All data and calculations are done in Excel.

$$Flow\ rate, Q = \frac{s}{t} \times A, where\ \frac{s}{t} = v \qquad (10)$$

2.5.1.2 Finding the Flow Speed

Once the flow rate is defined, the speed of the flow will depend on the cross section of each part. The flow rate was calculated where the tubes had a diameter of 1 mm, which gives a cross section of 0,007854cm^2. The measurements in Figure **32** and Figure 33 were used to calculate the cross sections of the designs with channels, e.g. design 2 and 4.

$$Flow\ speed, v = \frac{Q}{A} \qquad (11)$$

2.5.1.3 Finding the Sensor Response

The sensor response depends on the size of the measurement chamber and the flow rate. The response is proportional to the volume of the measurement chamber, V_c. This is plotted in Excel.

$$Sensor\ response, S_R = \frac{V_C}{Q} \qquad (12)$$

The design of the MFC might have an important influence on the sensor response.

2.5.1.4 Testing for Functionality and Difficulties

This is just a visual test, describing the behavior of the MFCs. It is important to look for leakage, bubble formations, flow difficulties, constipations and general performance of the different designs.

The tests will be done with the pump working at 5rpm and 10rpm, and all the different designs will be tested.

2.5.2 PCB Self-Noise

In order to check the PCB's self noise a test was performed without any connections on the PCB inputs. The test are run for one hour. This test was performed in order to see whether the PCB generated any self noise when running, and whether this noise was in the same area as the MAS_V2_R2. The procedure is explained in Appendix 7.7.1.

2.5.3 PCB with Constant Current on Sensor Input

As a measure of the PCB's response to an actual sensor input, there were conducted several tests. The IM6 Impedance measurement unit from Zahner elektronik was considered the best option as a constant current source because of ability to operate as a galvanostat, which is an instrument that can hold a very accurate, constant current. The test was run for one hour. The procedure is explained in Appendix 7.7.2.

2.5.4 Sensor in Hydrogen Peroxide

In order to control the accuracy of the system, a series of tests was conducted with hydrogen peroxide (H_2O_2). The procedure is explained in appendix 0. The experimental protocol is summarized in Table 5, and the test setup is shown in Figure 30. The sensors are

submerged into 25mL PBS, this quantity was chosen due to the size of the measuring glass available, and to prevent unnecessary use of analyte. The mixer is turned on, and the GUI is set to start. For two minutes, the sensors are held in the PBS without any H2O2 added, this is to make sure the sensors are stabilized. Then each second minute, 120th second, 5µL are added to the solution. The GUI tracks the response of the sensor in both number values, and graphical. At the fifth injection, after 10 minutes, a higher volume, 20µL is added to the solution, This also runs for two minutes, before the test is brought to an end, and repeated if necessary/wanted. For this project the test was repeated three times.
In a test solution of quantity 25mL, and a concentration of 0.065mmol/L H2O2, which is the desired quantities for this experiment, the volumes of PBS and undiluted H_2O_2 was calculated as follows:

$$Undiluted\ H_2O_2(\mu L) = Desired\ Concentration\left(^{mmol}/_{L}\right) \cdot \frac{Total\ wanted\ Amount\ (mL)}{Molarity\ (^{mol}/_{L})} \quad (13)$$

$$PBS\ (mL) = Total\ wanted\ Amount - \frac{Undiluted\ H_2O_2(\mu L)}{1000} \quad (14)$$

Table 5. Test protocol 1.

Time (s)	0	120	240	360	480	600
Volume added (µL)	0	5	5	5	5	20
Concentration (mM)	0	65	130	195	260	320

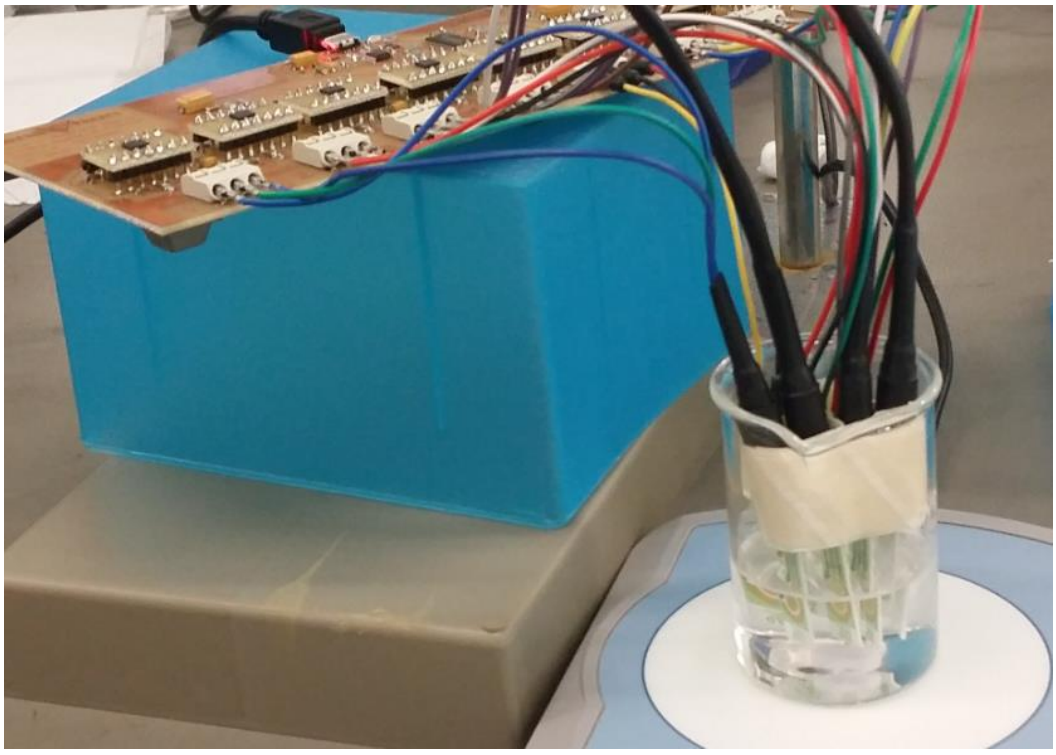

Figure 30. Eight sensors in response test.

2.5.5 Temperature

The temperature sensor within the LMP91000 Potentiostat have an accuracy of +-3 degrees Celsius [16]. Though this is a rather poor accuracy for a temperature sensor, its purpose in the ZP2015, is to show the stability of the temperature, rather than the exact temperature, and also of interest, the relationship between the measurements and the temperature alternation. For this purposes there were conducted a series of tests in a controlled environment, namely a temperature cabinet. For the test, the following program was created:

Program 11 – ZP Test

- ∇ Step 1. Set temperature to 15 degrees
- ∇ Step 2. Hold 15 degrees for 30 min
- ∇ Step 3. Set temperature to 20 degrees, hold for 10 minutes
- ∇ Step 4. Set temperature to 25 degrees, hold for 10 minutes
- ∇ Step 5. Set temperature to 20 degrees, hold for 10 minutes
- ∇ Step 6. Set temperature to 15 degrees, hold for 10 minutes

In order to check the rate of the thermal equilibrium and the stabilization, each temperature was held for 10 minutes. Though the sensors were thought to have an almost instant response, the delay before the next step would allow for a slower response. Also the delay would allow for a check of the stability of the temperarture sensors.

The procedure is explained in Appendix 7.7.4.

3 Results and Discussion

3.1 Microfluidic Flow Cell

The results consists of both the results obtained from the fabrication process as well as from the test procedure.

3.1.1 Cad Design

The photomask from the CAD design resulted just as expected. The mask shown in Figure **31**, displays the designs mirrored compared to the drawing. The reason is that when doing the exposure, the side of the glass covered with metal will be facing the wafer with SU-8.

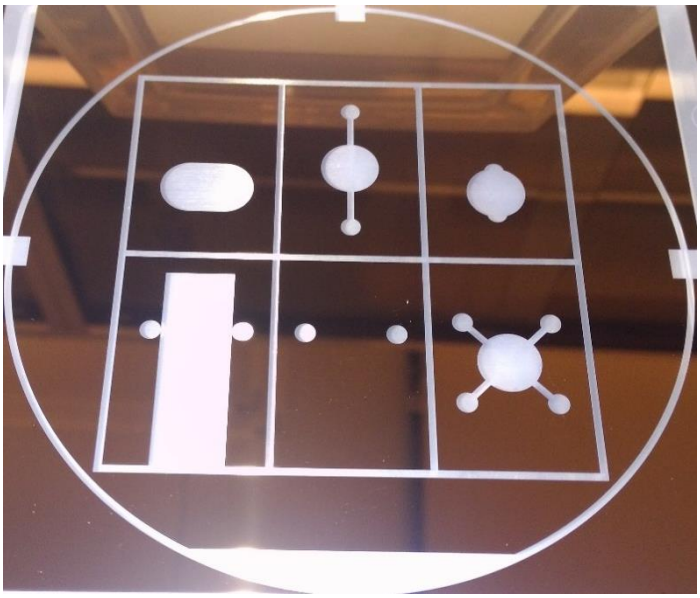

Figure 31. Glass mask with the designs

3.1.2 Fabrication results

3.1.2.1 Polymeric master

The laboratory reports from the fabrication process can be found in Appendix. All measurements done are documented in the reports.

In the process, six masters were fabricated with different results. The intention was to create a master with a SU-8 structure approximately around 250µm. The SU-8 100 is primarily adapted to give a 100µm structure, but with options to make thicker layer.

The first part of fabrication that could affect the results are the application of the resist. On Test 2, a syringe was tested to find a method for always applying the same amount of SU-8. This did not work at all, due to a large amount of bubbles trapped in the resist. It was tried two times with the same result. It was considered to apply the resist while the wafer was on a scale, but due to the fixation system for the wafer in the spin coater, it was not possible to do this, see Test 2. Because of this, an approximate amount of resist has to be applied, covering the center of the wafer with 4-5cm in diameter. Due to this, the thickness may vary depending on how much resist is applied.

The measurements from all the masters are visually illustrated by drawing the thicknesses on an illustration of the designs. The measurements are also presented in tables showing the differences between the designs. These are shown from Figure **32** to Figure **37**, and Table 6 to Table 11. The thickness was measured with the profilometer.

The wafer 1 from Exercise 2, and the master from Exercise 3 were cracked due to the hard bake process. It seems that the SU-8 on both wafers broke where the structure was thicker. Wafer 2, Exercise 2 had a structure thinner than 100µm, and did not crack. Because the PDMS does not require hard bake, it was decided to ignore this part of the process.

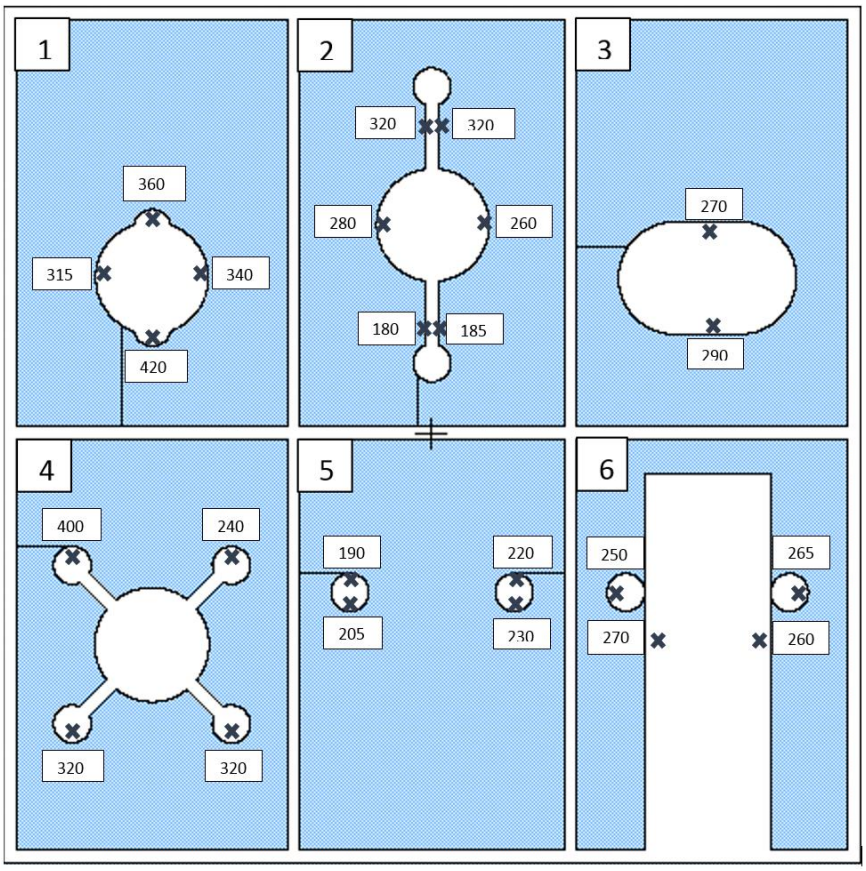

Figure 32. Thickness Wafer 1, Exercise 1

Looking at the measures of Figure **32**, it can clearly be seen that the SU-8 layer on the master is uneven. In the center, the layer is thinner than closer to the borders. The layer is thickest on the left side of the master. The maximum thickness is 420µm and the minimum is 180 that is approximately 133% thicker on the left side than closer to the center. The Table 6 shows the details of the each design.

Table 6. Differences wafer1, Exercise 1

Design	Thinnest (µm)	Thickest (µm)	Percent thicker (%)
1	315	420	33
2	180	320	78
3	270	290	7
4	240	400	67
5	190	230	21
6	250	270	7

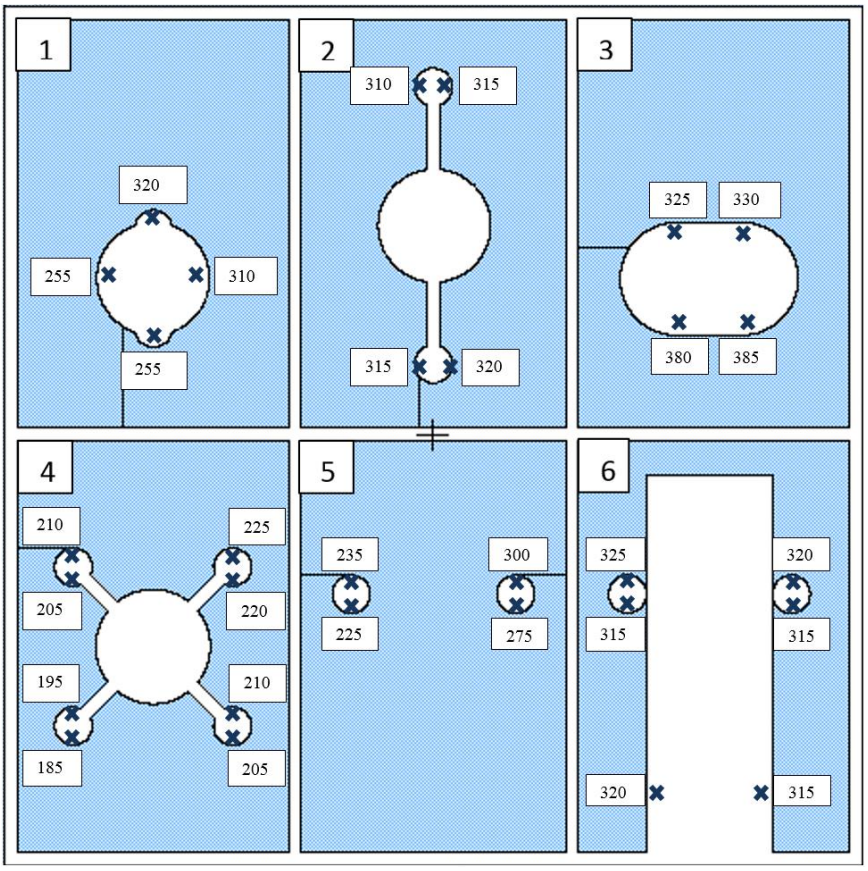

Figure 33. Thickness wafer 2, Exercise 1

As the Figure 33 shows, the wafer 2 is much more even than wafer 1, but there still are some quite large differences. The thinnest part is down on the left corner, and the thickest part is up on the right corner/side. The minimum is 185µm and the maximum is 385µm, which makes the thickest part 108% thicker than the thinnest part. Table 7 gives more information about the errors within the designs.

Table 7. Differences wafer 2, Exercise 1

Design	Thinnest (µm)	Thickest (µm)	Percent thicker (%)
1	255	320	25
2	310	320	3
3	325	385	18
4	185	225	22
5	225	300	33
6	315	320	2

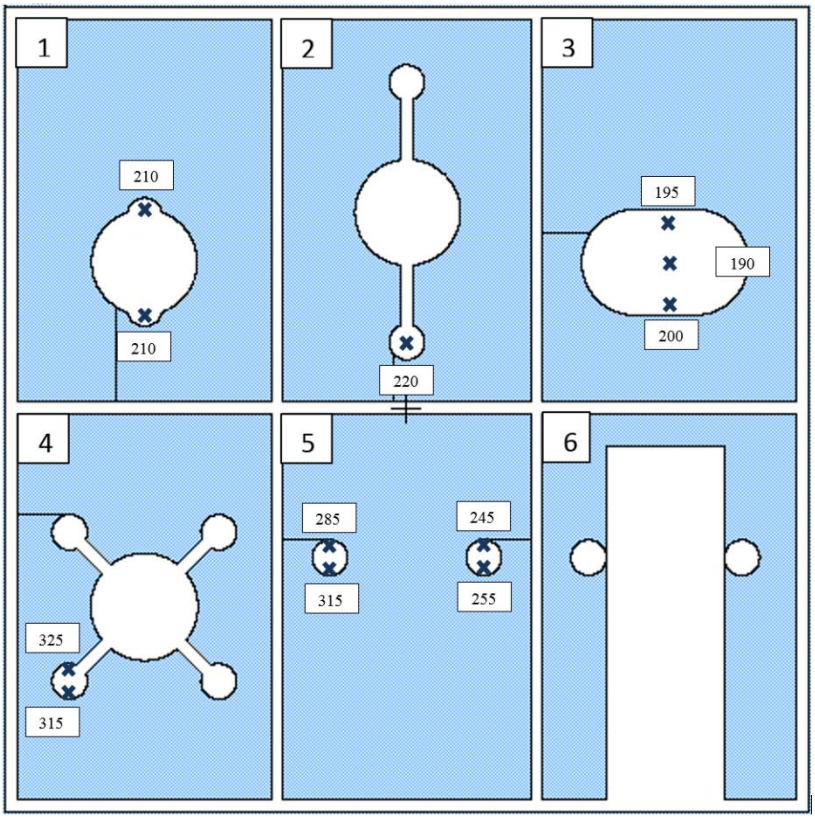

Figure 34. Thicknesses wafer 1, Exercise 2.

It was not possible to measure the designs that were broken in the hard bake process, hence it would have damaged the stylus of the profilometer. From Figure **34**, the few measurements made seemed to indicate that the higher right corner had the thinnest layer of resist, around 190µm, and that the lower left corner had the thickest, around 325µm. The difference is approximately 71% more than the thinnest. Table 8 shows more details of the differences between the designs.

Table 8. Differences wafer one, Exercise 2

Design	Thinnest (µm)	Thickest (µm)	Percent thicker (%)
1	210	210	0
2	220	-	-
3	190	200	5
4	315	325	3
5	245	315	29
6	-	-	-

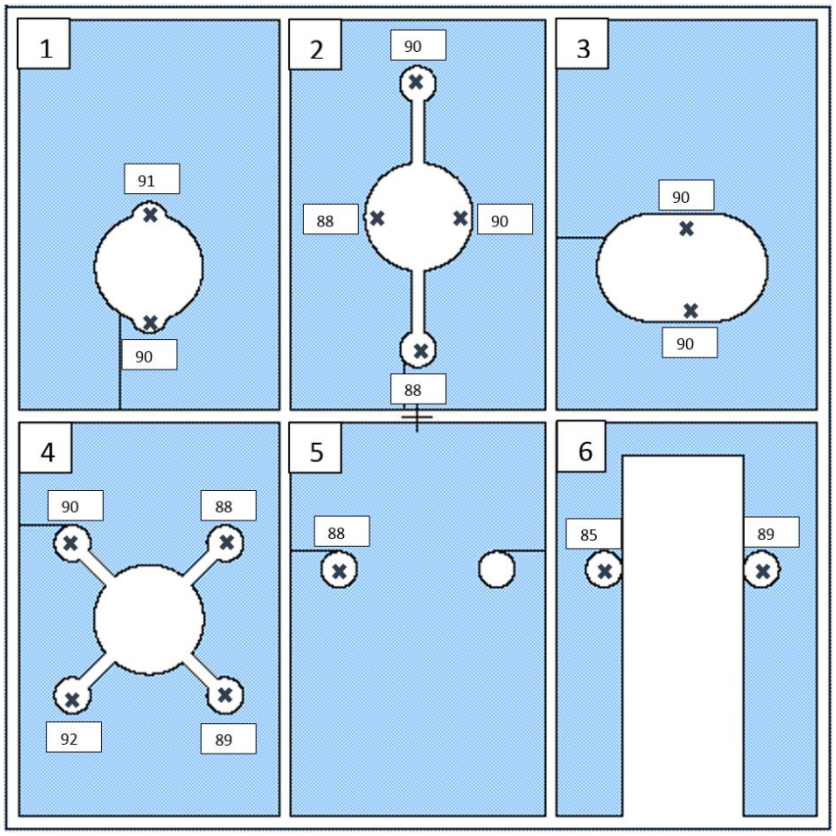

Figure 35. Thicknesses wafer 2, Exercise 2

Looking at Figure **35**, it seems clear that the second wafer of Exercise 2, was much more even, and it seems obvious that it is easier to obtain a master with little difference of thickness, when aiming for SU-8 with a thickness of 100µm. The thickest point were only 8% thicker than the thinnest point. Table 9 shows a more detailed information on each design.

Table 9. Differences, wafer 2, Exercise 2

Design	Thinnest (µm)	Thickest (µm)	Percent thicker (%)
1	90	91	1
2	88	90	2
3	90	90	0
4	88	92	5
5	88	-	-

6	85	89	5

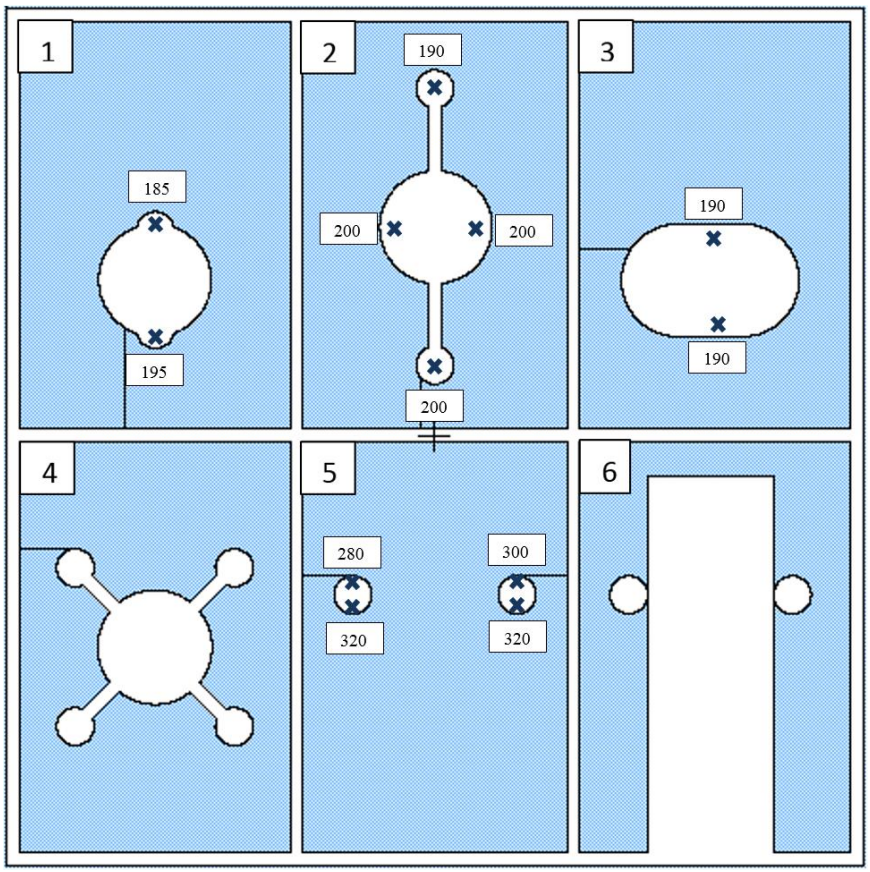

Figure 36. Thicknesses Exercise 3

Due to the same problem as wafer 1 of Exercise 2, it was not possible to measure the broken designs, see Figure **36**. From the measurements made, the thickest resist was 73% thicker than the thinnest, as shown in Table 10. Just by the looks of the master, it seems to be a bigger difference.

Table 10. Differences Exercise 3.

Design	Thinnest (µm)	Thickest (µm)	Percent thicker (%)
1	185	195	5
2	190	200	5
3	190	190	0
4	-	-	-
5	280	320	14
6	-	-	-

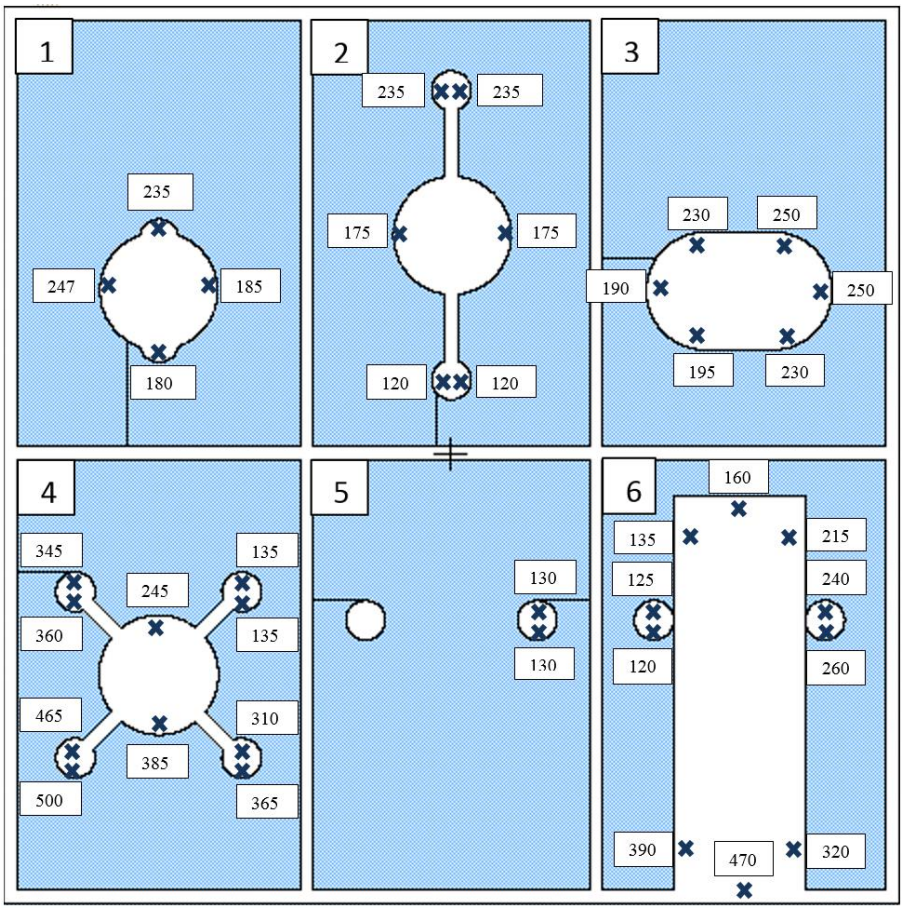

Figure 37. Thicknesses Exercise 4

The thickest part of the wafer was approximately 316% thicker than the thinnest as shown in Figure 37. None of the other masters created with the procedure to get 250µm thick SU-8 layer had as thick or as thin spots as this wafer. Maximum was 500µm and minimum was 120µm. There were large differences in the designs as well, as shown in Table 11. This might be caused by the hot plate, because on this wafer the same as wafer 1, Exercise 1 was used. This seems to indicate that the hot plate has to be leveled before use.

Table 11. Differences Exercise 4

Design	Thinnest (µm)	Thickest (µm)	Percent thicker (%)
1	180	245	36
2	120	235	96
3	190	250	32
4	135	500	270
5	130	-	-
6	120	470	292

To view the shapes and measuring the width, the caption feature of the optical microscope were used. Figure **38** and Figure **39** show the captions. The measurements showed that the SU-8 structure was very accurate comparing to the CAD designs. It was not possible to measure the bigger parts of the designs due to the amplification.

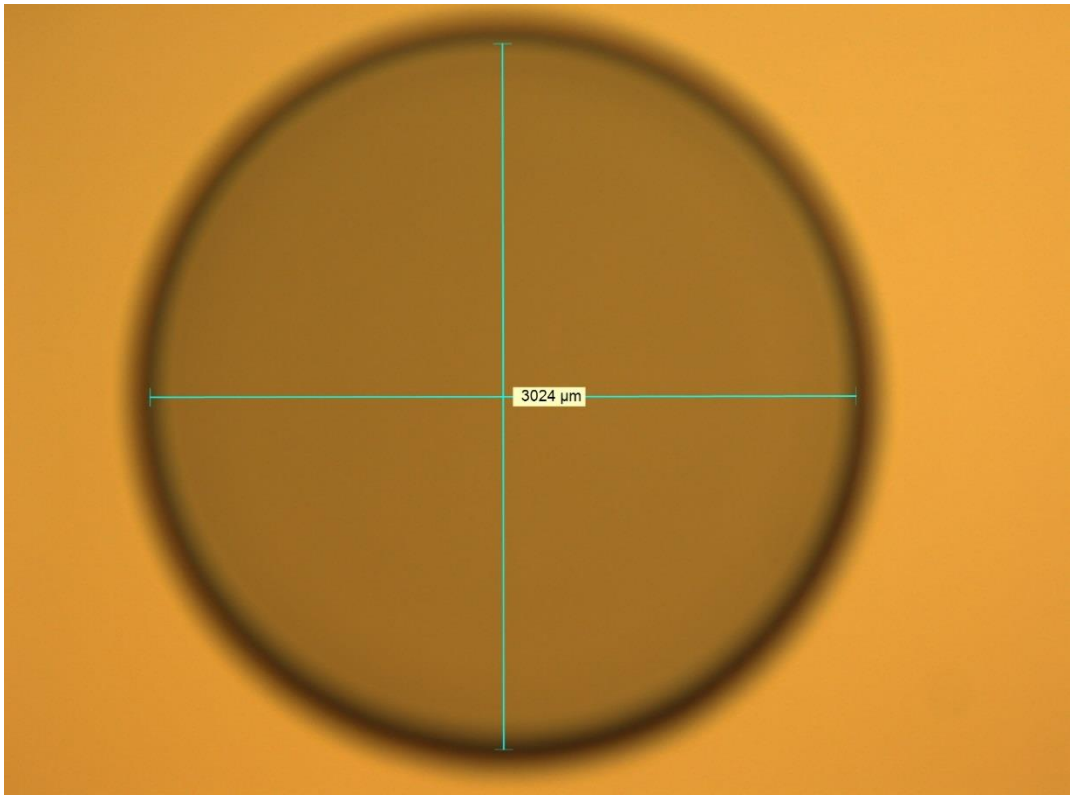

Figure 38. Design 5, right outlet, Exercise 4

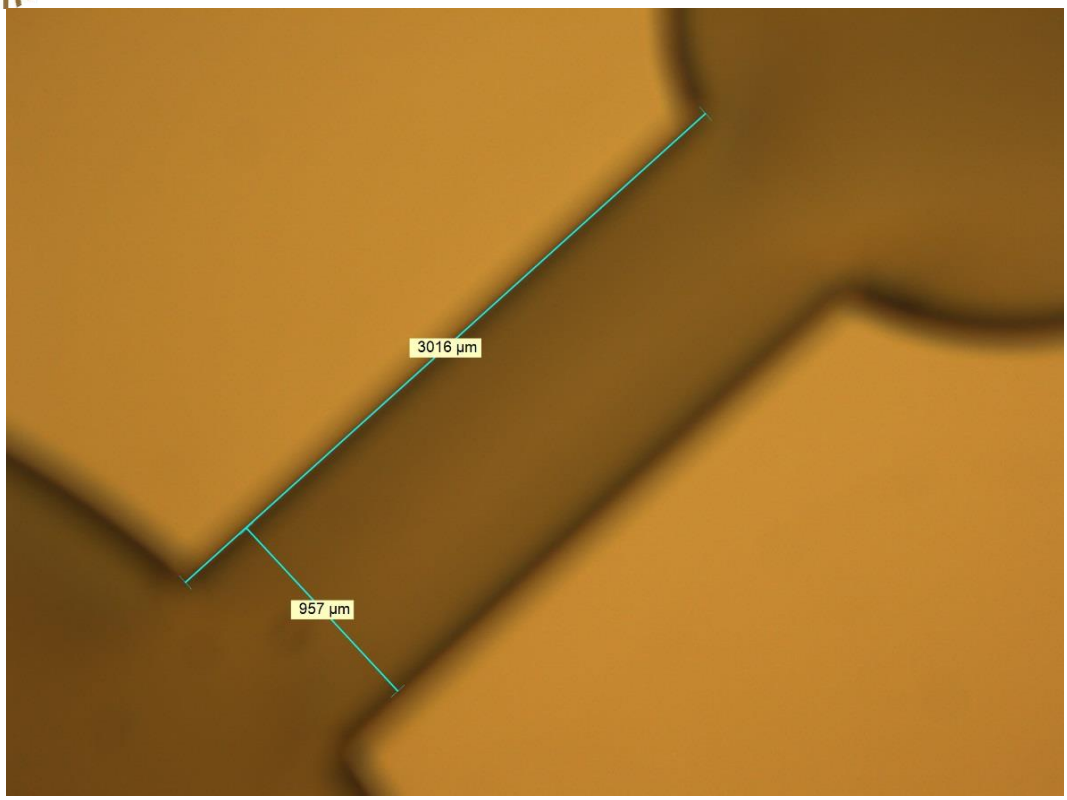

Figure 39. Design 4, left lower channel, Exercise 4

Pictures were taken with the optical microscope showing the cracks from the hard bake. These pictures are shown in Figure **40**. A picture taken with camera is shown in Figure **41**.

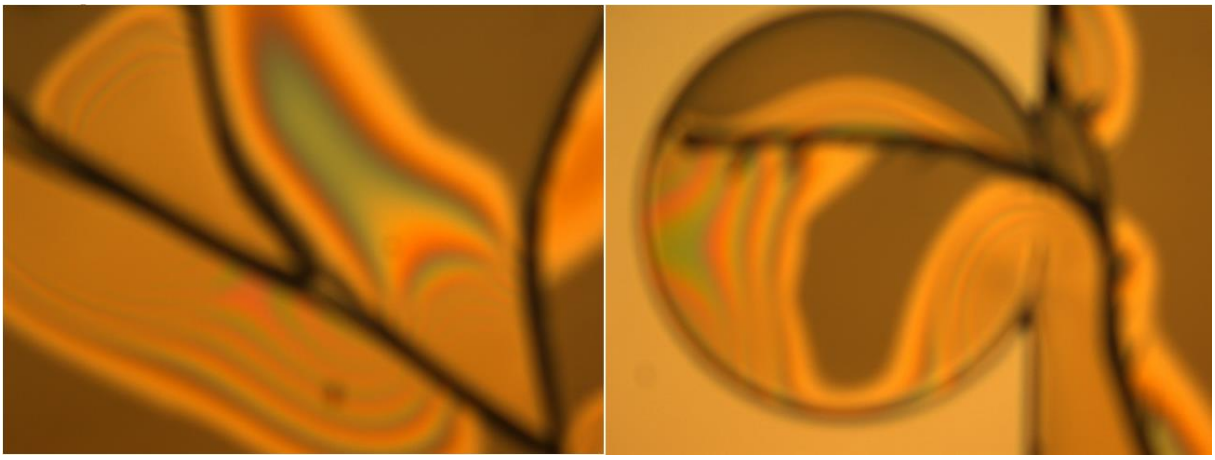

Figure 40. The cracks in the SU-8 after the hard bake of design 6, Exercise 3.

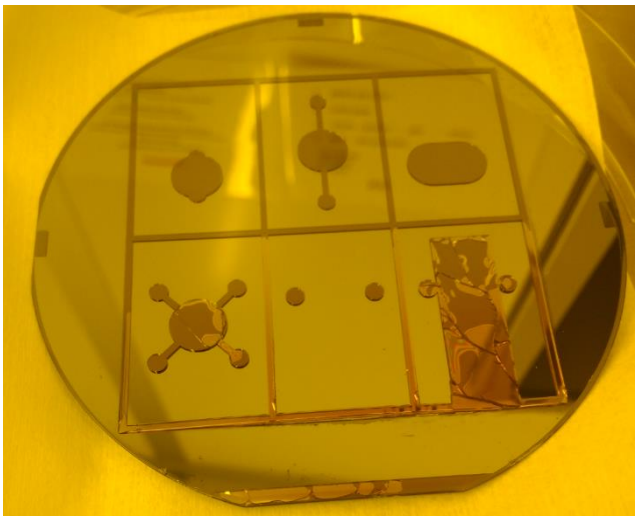

Figure 41. Showing the cracks in design 4 and 6, Exercise 3

The most important thing about the master is that it has to be as even as possible, and that the thickness does not differ too much on one design. Especially design 2 and 4 are more sensitive due to the channels. Looking at the result of the wafer1, Exercise 1, there are large differences in thickness. The upper channel is approximately 78% thicker than the lower channel on design 2, and on design 4 the upper left outlet is approximately 68% thicker than the upper right outlet, and approximately 25% thicker than the two lower outlets. Even worse results are encountered in Exercise 4, where design 2 have one channel that is 96% thicker than the other and design 4 has a difference of 270%. This can be seen clearly in Figure 42.

The difference will probably make the fluid choose the channel with the biggest cross section, where it finds less resistance.

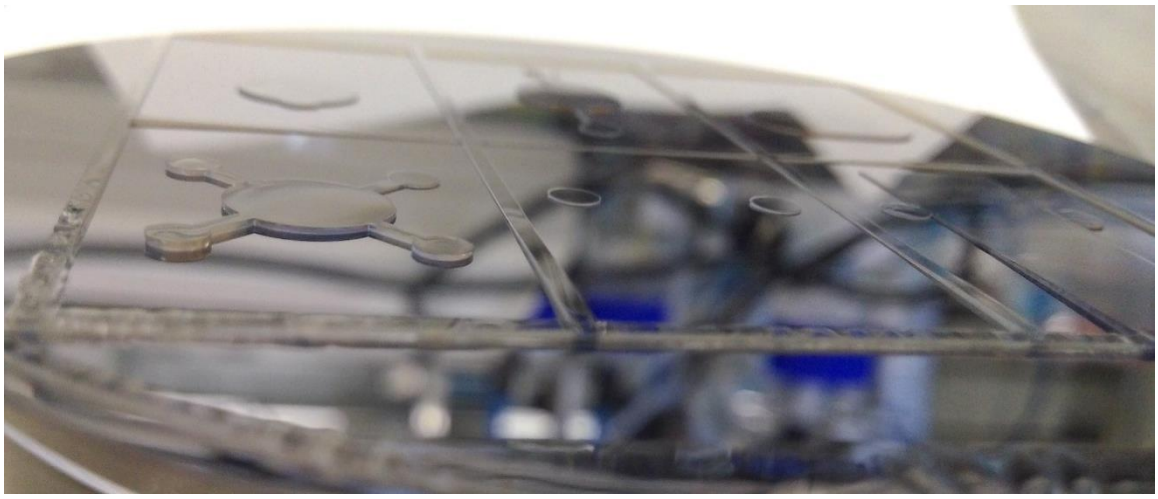

Figure 42. Close up design 4, Exercise 4.

The best master seems to be wafer 2 from Exercise2, measuring around 90µm. It has almost no differences in thickness on each design, but the photoresist is very thin. For the designs with channels, that may create a problem with the flow rate. Picture is show in Figure **43**.

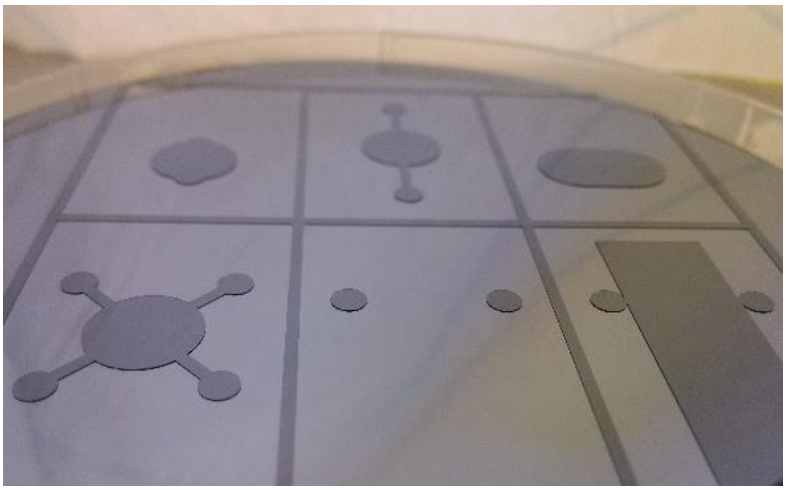

Figure 43. Wafer 2, Exercise 2.

Wafer 2 of the first laboratory exercise seems to be the best of the remaining masters, having resist with thickness around 200µm. It is not ideal, but it can be used. The design 2 seems to be very even, and the lower outlet is only 3% thicker than the higher.

Through the laboratory exercises two different hot plates has been used. It seems that the masters that have been the baked on the largest hot plate, in general have gotten better results. The Figure **44** shows the larger hot plate to the right. The masters created on the hot plate to the left are wafer 1 from Exercise 1 and the master from Exercise 4. Those are the two masters with the greatest thickness differences. This may indicate that there are some differences between the two hot plates. It seems likely to think that the level of the hot plates may cause the thickness difference. For future fabrication, it will be a good idea to level the hot plate before use.

Figure 44. Wafer 1 to the left and wafer 2 on the right, Exercise 1

3.1.2.2 PDMS Replica Molding

It was made two sets of PDMS designs, one from wafer 1, Exercise 1, and the other from wafer 2, Exercise 1. Both laboratory exercises can be found in The Appendix under Replica molding 1 and Replica molding 2. Everything occurred according to the procedure. Wafer 2 from Exercise 1 is the master for both castings in Figure **45** and Figure **46**.

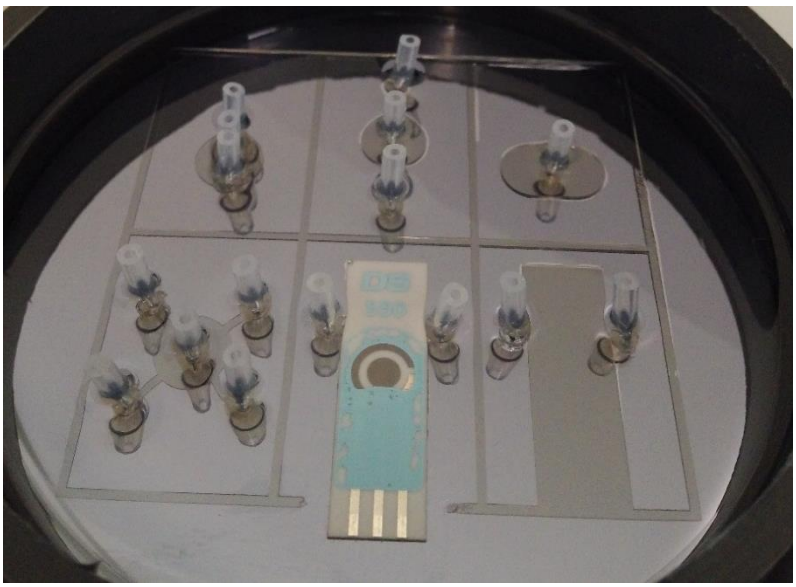

Figure 45. Wafer 2, Replica molding 2. The designs showing through the cured PDMS

The transparency of the PDMS makes it easy to see the designs on the master as shown in Figure **45**. After lifting off the designs, the master was attempted removed, but it was badly stuck and it cracked. This is shown in Figure **46**. If there is no need to take the master out of the mold, it should be left to avoid breakage.

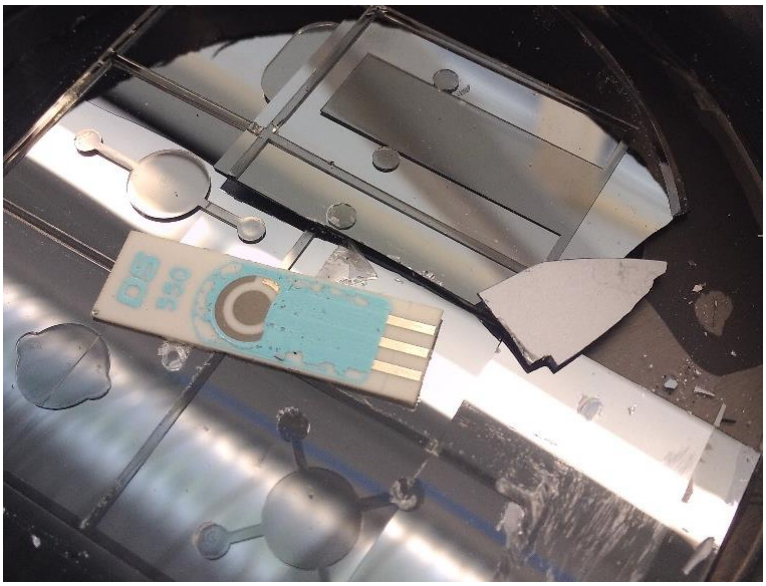

Figure 46. Wafer 2, Replica molding 2. The master broke when trying to take it out of the mold

The finished designs from wafer 1, Exercise 1 are shown in Figure **47**.

Figure 47. The designs created from wafer 1, laboratory exercise 1.

3.1.2.3 Oxygen Plasma Bonding

The design 1, 2 and 4 were bonded to glass slides. The designs were washed with DI water, and that did not seem to leave the designs clean enough, because two of the designs did not bond. The designs were cleaned with IPA, and that worked fine. The best would be to do keep the replica in the mold, until the bonding is to be done, then cut the designs out while in the clean room. Figure **48** shows the results.

Figure 48. Designs 1, 2 and 4 bonded on glass slides

3.1.2.4 Holder for MFC

The holder for design 1, 2 and 4 was assembled with the sensor chip, gasket and the MFC from design 1. The moment the holder pressed on the system, the glass slide broke as shown in Figure 49. This was not enough pressure to seal the system.

Figure 49. The broken glass slide with design 1.

That means that the holder designed for these designs did not work as expected. The glass slides were to fragile due to the hole. It was necessary to find another way to test the designs.

The holder for design 3 was assembled, and it seemed to work fine. There was a problem with leakage due to the capillary force. The liquid escaped between the sides of the chip and the PDMS. This was fixed using silicone grease.

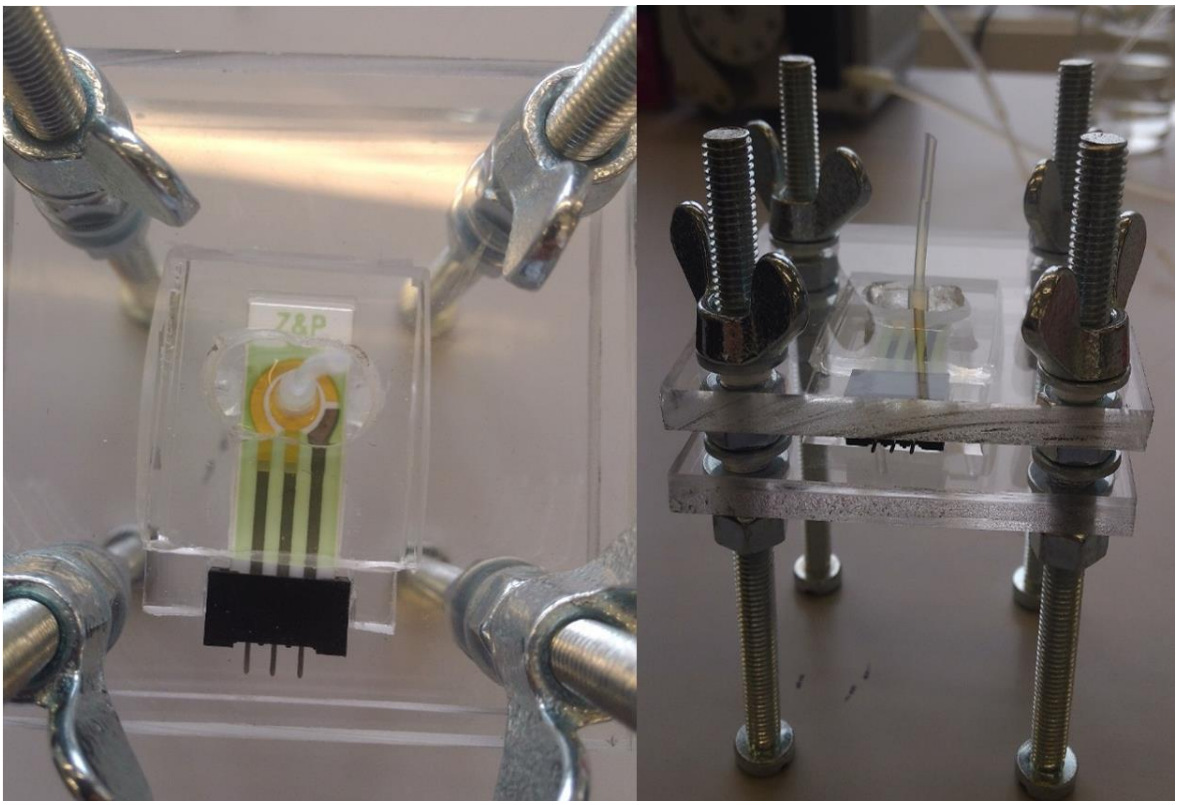

Figure 50. Holder for design 4, showing how the connector is adapted.

3.1.3 Test Results

To calculate the flow rate, flow speed and sensor response, the measured values and equations from 2.5.1 were used.

3.1.3.1 Flow Rate

The first time the designs were tested, all the designs with glass slides had leakage because there were not used enough silicone glue to seal the chambers. The second time, it was only design 2 that had a leakage. Design 2 was tested later. All the designs gave approximately the same the same flow rate. At 10rpm, the flow rate was calculated to be between 0,049 and 0.051 cm^3/s and at 5rpm it was calculated to be between 0,024 and 0,026 cm^3/s. At 5rpm it seems to give a flow rate half the one given by 10rpm. The average of the flow rate at 5rpm is 0,025 cm^3/s and at 10rpm it is 0,050 cm^3/s. The detailed calculations are found in the table in Appendix 1.

3.1.3.2 Flow Speed

Design 1 and 3 have no limitation to the flow speed, but design 2 and 4 have channels with cross sections narrower than the silicon tubing, and therefore the flow speed will change inversely proportional to the cross section. There were not enough points of measurement as to get an exact flow speed, but it can give an idea.

The flow speed for design 2 was approximately 27.8cm/s at the upper channel and 15.6cm/s at the lower channel. The design 4 had 12.5cm/s at the upper left channel, 20.8 at the upper right channel and 15.6 at both the lower channels. The flow speed a 5rpm is half of the speed at 10rpm. The data is found in the table in Appendix 2.

The liquid will always choose the path with less hydraulic resistance, and if the flow speed is low enough, there will be no need to use more than one outlet, as seen in the tests in 3.1.3.4. It is logical to think that the channel with the largest cross section will be the one chosen, due to having the lowest flow resistance.

3.1.3.3 Sensor Response

The different designs have different sizes of the measurement chambers, partly due to the design itself and partly due to the fabrication process. All the volumes are inserted into the tables in Appendix 1, and the sensor responses are calculated. The volumes of the measurement chambers of designs 1, 2 and 4 lays between 0.14 and 0.17 cm^3, and the volume of design 3 is approximately 0.02. At 5rpm, the designs 1, 2 and 4 had a response between 5.7 and 6.6 seconds while design 3 had a response of approximately 0.9 seconds. At 10rpm, design 1, 2 and 4 had a response between 2.8 and 3.4 seconds and design 3 had a response between 0.4 and 0.5 seconds. It is clear that the calculated sensor response for design 3 is far better than for the other designs, and that is due to the size of the measurement chamber.

The real response of the sensor will be found when testing the sensor with the MFC. If the design is not made correctly, the fluid flow through the chamber may move in an unsuitable way, like not covering the sensing area properly, or not exchanging the fluid quickly. This is due to the laminar flow expected in micro systems [26].

3.1.3.4 Functionality and Difficulties

All the designs have one inlet in the center, and two or more outlets. When pumping the fluid through the system, it tends to come out of one outlet only. It was possible to change the outlet by obstructing the one draining. That way the MFC starts to evacuate through both outlets. That even works with design 4, having 4 outlets. The idea of having more outlets was to create a better flow rate, but it does not seem to be necessary, considering that the flow rate is the same on all the designs, even though the liquid is drained through one outlet only.

Because of this design, the liquid is not always exchanged properly, and it takes longer to replace it completely. This is especially a problem with the designs with the bigger measurement chambers. A picture sequence of design 2 is presented in Figure 51, showing the evacuation of the measurement chamber.

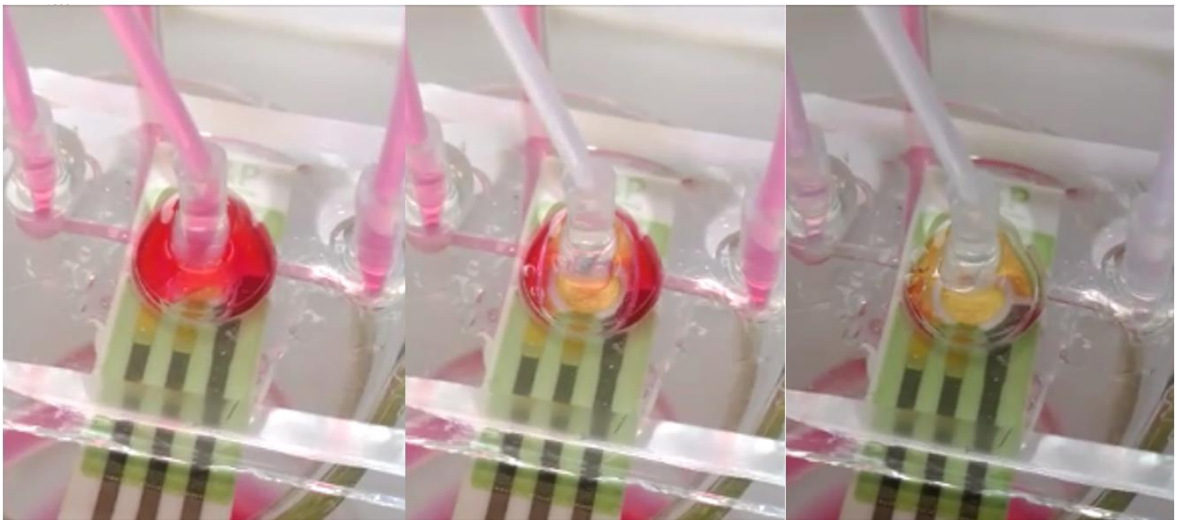

Figure 51. Exchanging the fluid in the measurement chamber of design 2.

Another challenge was the formation of bubbles on the sensing area. From the video material, it seems that the bubbles are stuck easier at 5 rpm than at 10 rpm. It was tried to block the draining outlet, to make the liquid current change direction, but the bubbles only disappeared when the chamber were completely evacuated and then filled again. On Figure 52, one can clearly see a bubble covering the sensing area of design 4, and only one outlet is permitting flow of fluid. The liquid has risen approximately a centimeter up one of the other outlets.

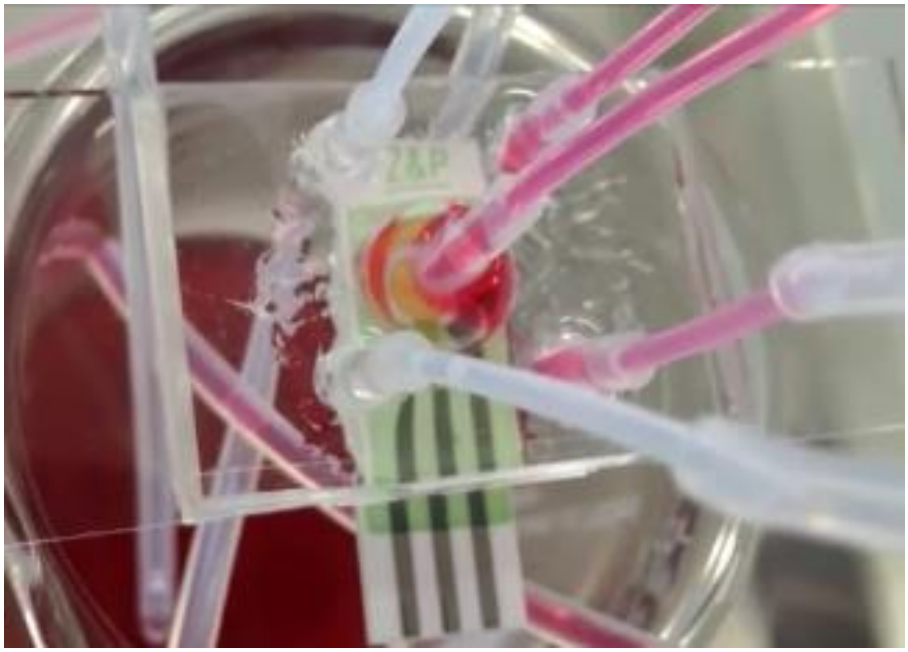

Figure 52. Bubble covering a big part of the sensing area.

When the MFC were tested with the PCB and versaSTAT, it was discovered that it was necessary to create breaks of several seconds with air between one concentration and the next. This was done by taking the tube out of one concentration while the pump was still working, wait three or more seconds before introducing the tube into the next concentration. Doing that the problem with the bubbles almost disappeared.

The designs bonded with glass slides were not very suited for the comparison tests with the magnetic mixer due to the provisional system of fixating the sensors with silicone glue. It was decided to test the system with one sensor only, because of the leakage possibility and the difficulty and time required to cut off one sensor and glue on a new.

Looking at the flow rate and the thickness of the channels, it seems that there should be enough with one outlet, but the designs will have to be reconsidered, because the flow pattern inside the measurement chamber will change compared to the original design.

3.2 Front-end Interface and Control System

3.2.1 Breadboard Prototype

Figure 53 shows the LMP91000 on breakout boards. The one on the left side is the temporary solution made by gluing the IC upside down and perform a manual wire bond with a soldering iron and threads of speaker cable. The one to the right is the permanent solution. Figure 54 shows the USB connector and FT230X on their respective breakout boards.

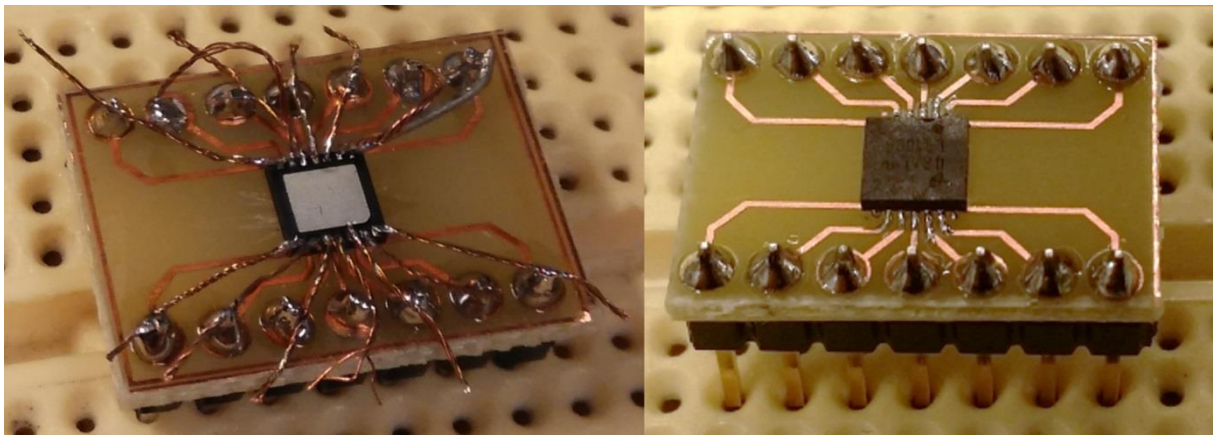

Figure 53. LMP91000 on breakout boards. Left side: manual wire bond. Right side: final solution.

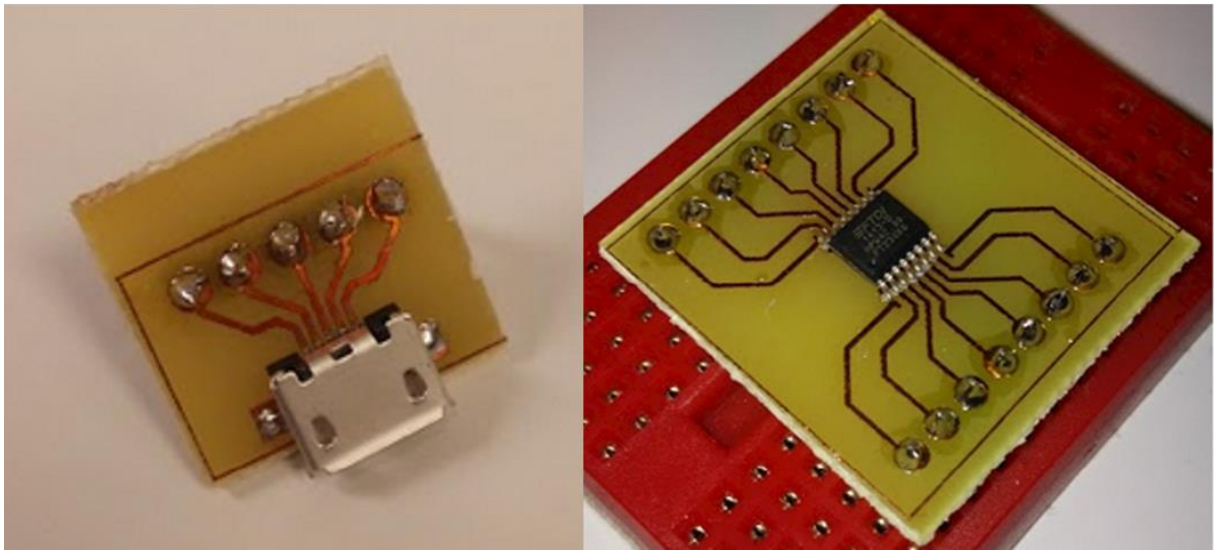

Figure 54. From the left: USB connector and FT230X on breakout boards.

Adding the second ADS1115 to the breadboard presented some problems that took some time to figure out. When reading the conversion register of the second ADS1115, very strange numbers appeared; not consistent at all with the voltages applied to the analog input pins.

Connections were checked and re-checked, and components were moved to different locations on the breadboard in case of broken traces. It was also attempted to switch places of the two ADS1115 devices to see if the results were the same. The source of the problem turned out to be the I^2C. The ADDR pin of the second ADS1115 had been connected to

ground, thereby selecting the I^2C slave address 1001 000, which is the same as that of the LMP91000. Connecting the ADDR pin to SCL solved the issue. It became apparent that it would be convenient to have a way of testing whether a slave device responds to I^2C commands. This is explained in section 2.4.2.5.

3.2.2 PCB

The first attempt on etching ended in failure because of too long exposure time in the etching machine. Fear of repeating the failure led to the etching being ended a bit too early on the second attempt, leading to some leftover copper on the upside of the PCB. However, the amount of excess copper left on the PCB was insignificant, and easily detectable through a continuity test with a multimeter. When detected the short circuits was repaired with a scalpel. On the bottom side however, there was an issue with dirt, possibly residues from the cutting process of the substrate that was performed prior to the etching process. These residues were spread over the ground layer under the mask during the photolithography process, which left several openings in the multiplexer traces that runs through the ground layer, as seen in Figure 55. These openings were soldered manually, and a continuity test was performed to check for contact. Also, the resistance over the solder point were checked to see whether the solder was of good quality or not. A good solder point should have less than one ohms resistance [23].

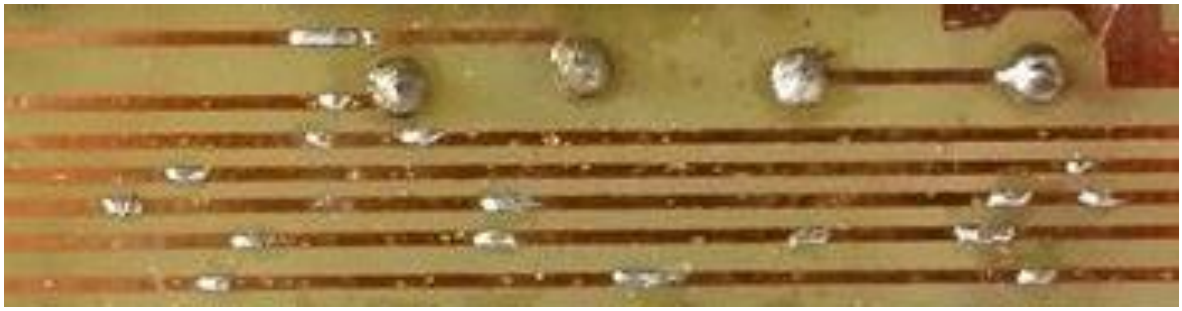

Figure 55. Ground layer, one can clearly see the solder points on the traces.

Figure 56 shows a partially assembled PCB. The connector needed for the microcontroller programmer tool was not available through the standard sources used by HBV. To avoid an expensive shipment for one single piece, the group decided to make an effort to reuse one from an old MAS_V2_R2 board. A connector was removed from the board by simply cutting it, and red wires was soldered from the PCB to the connector, as seen in Figure 58, this provided the group with the possibility to upload new code to the microcontroller. As a final solution there were drilled holes where short wires were soldered through.

The connector were soldered on top of these wires. This can be seen on the final PCB is depicted in Figure 57.

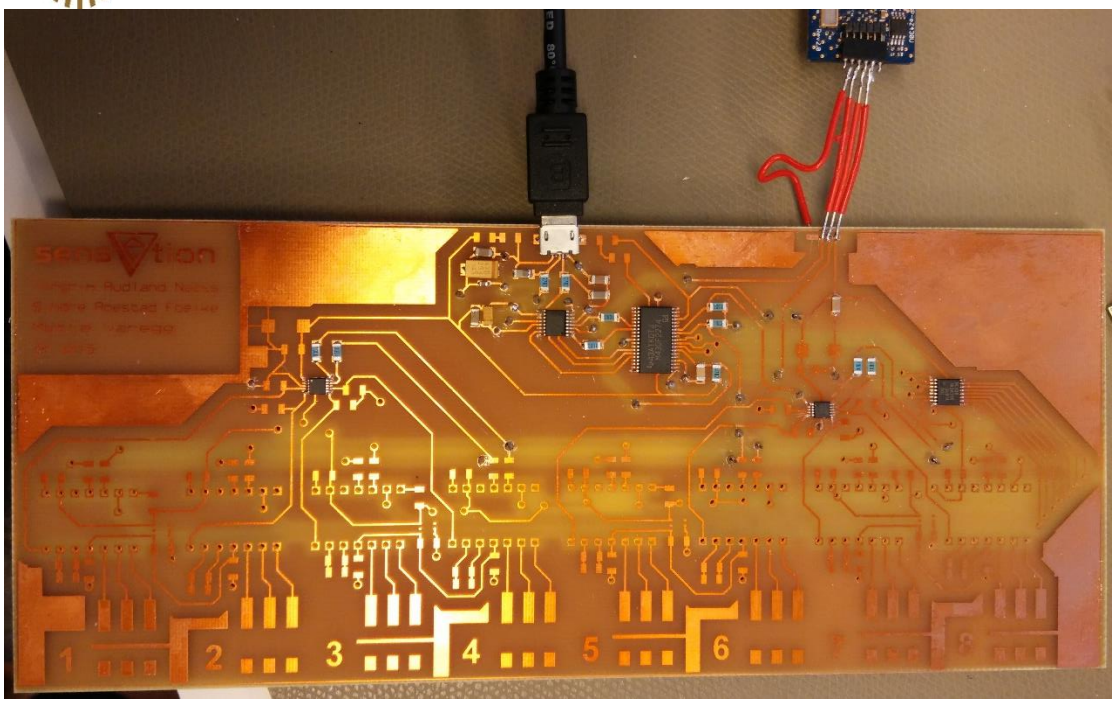

Figure 56. Partially assembled PCB.

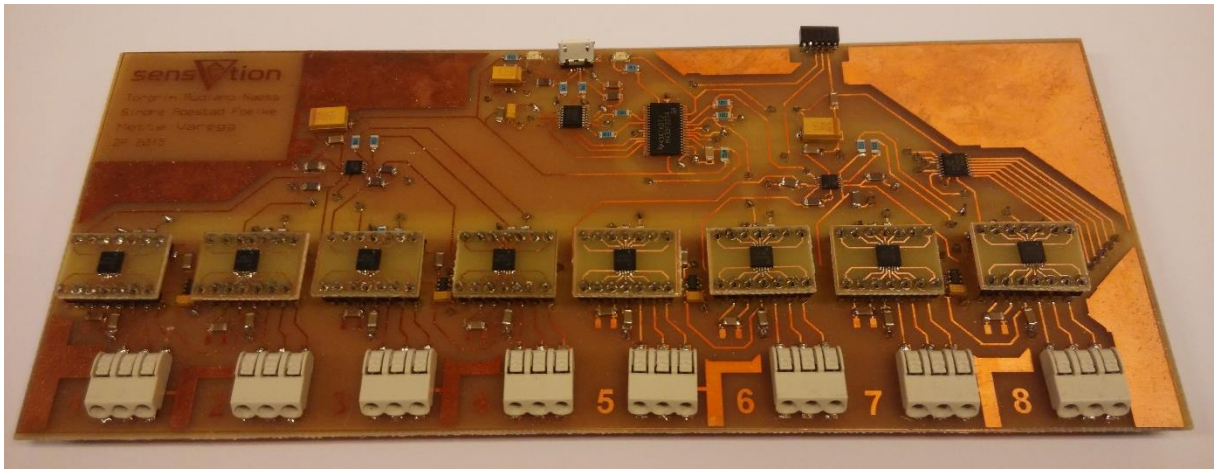

Figure 57. Final PCB.

3.2.3 Computer Software

The computer software required more work than expected, as the whole application had to be completely revamped in order to support multiple channels. The graphical representation of sensor signals is shown in Figure 58. Separating the channels by color makes it easy to monitor multiple sensor signals simultaneously. The grid view for ADC values in Figure 59 also displays data in an organized way, and provides the possibility to sort through data because each column can be sorted in ascending or descending order.

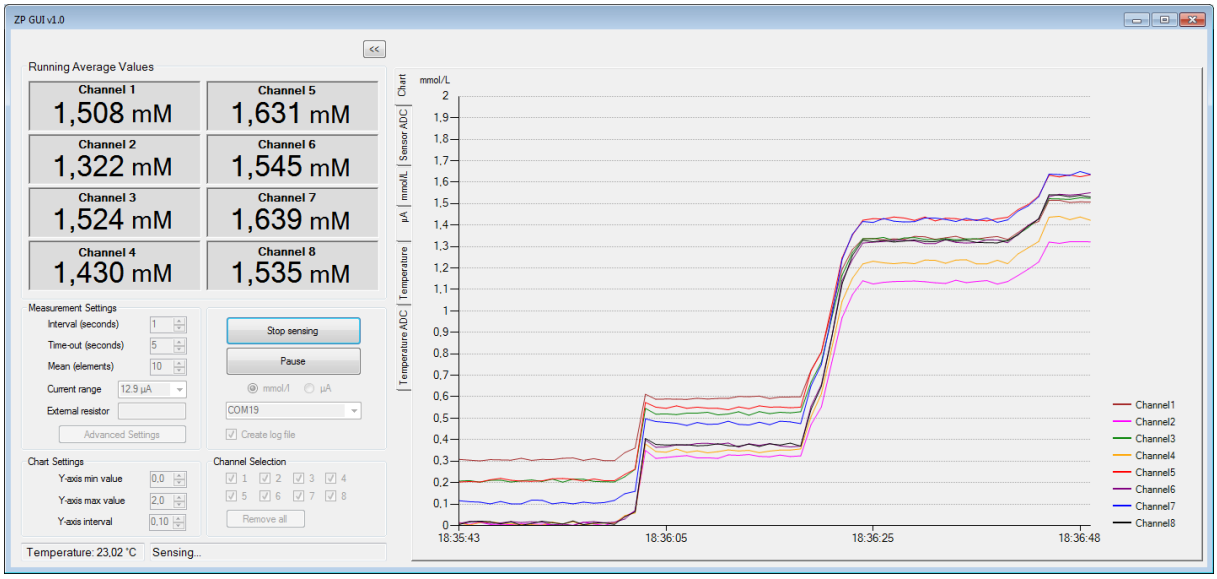

Figure 58. Real-time graphic presentation of data. It was forgotten to take a screenshot during the testing, so the values presented here are generated by the microcontroller.

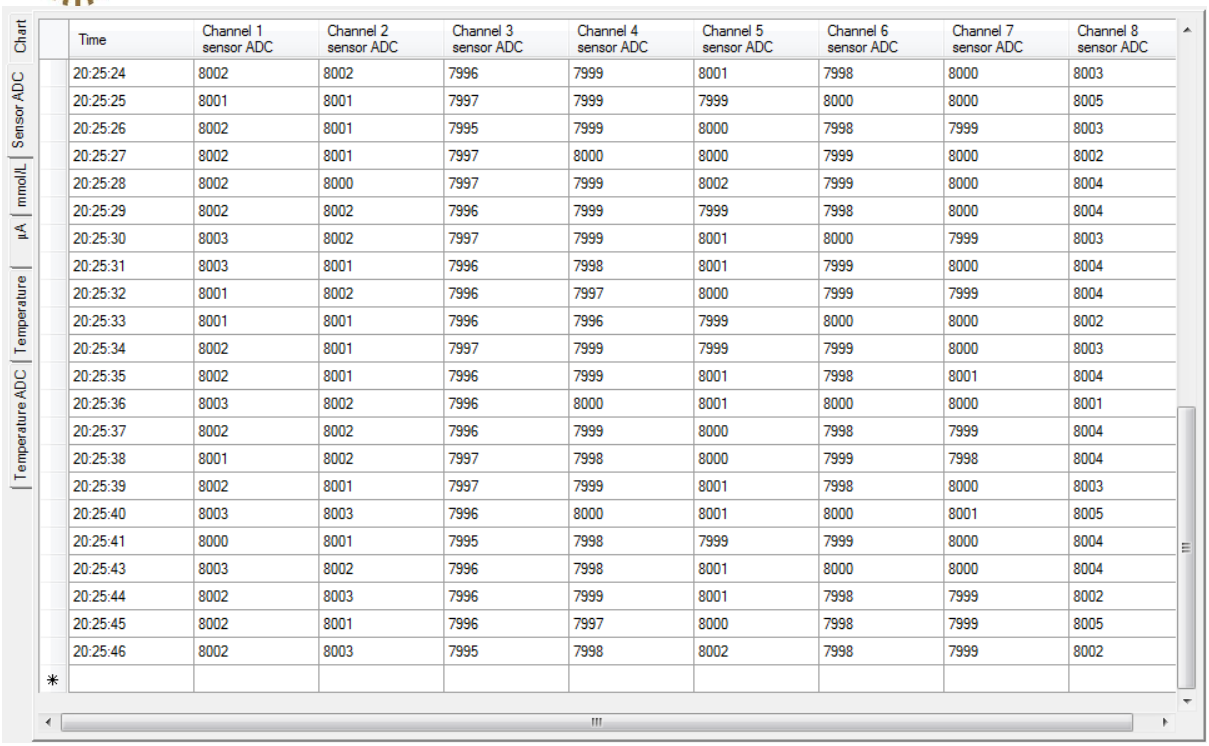

Chart | Sensor ADC | mmol/L | µA | Temperature | Temperature ADC

Time	Channel 1 sensor ADC	Channel 2 sensor ADC	Channel 3 sensor ADC	Channel 4 sensor ADC	Channel 5 sensor ADC	Channel 6 sensor ADC	Channel 7 sensor ADC	Channel 8 sensor ADC
20:25:24	8002	8002	7996	7999	8001	7998	8000	8003
20:25:25	8001	8001	7997	7999	7999	8000	8000	8005
20:25:26	8002	8001	7995	7999	8000	7998	7999	8003
20:25:27	8002	8001	7997	8000	8000	7999	8000	8002
20:25:28	8002	8000	7997	7999	8002	7999	8000	8004
20:25:29	8002	8002	7996	7999	7999	7998	8000	8004
20:25:30	8003	8002	7997	7999	8001	8000	7999	8003
20:25:31	8003	8001	7996	7998	8001	7999	8000	8004
20:25:32	8001	8002	7996	7997	8000	7999	7999	8004
20:25:33	8001	8001	7996	7996	7999	8000	8000	8002
20:25:34	8002	8001	7997	7999	7999	7999	8000	8003
20:25:35	8002	8001	7996	7999	8001	7998	8001	8004
20:25:36	8003	8002	7996	8000	8001	8000	8000	8001
20:25:37	8002	8002	7996	7999	8000	7998	7999	8004
20:25:38	8001	8002	7997	7998	8000	7999	7998	8004
20:25:39	8002	8001	7997	7999	8001	7998	8000	8003
20:25:40	8003	8003	7996	8000	8001	8000	8001	8005
20:25:41	8000	8001	7995	7998	7999	7999	8000	8004
20:25:43	8003	8002	7996	7998	8001	8000	8000	8004
20:25:44	8002	8003	7996	7999	8001	7998	7999	8002
20:25:45	8002	8001	7996	7997	8000	7998	7999	8005
20:25:46	8002	8003	7995	7998	8002	7998	7999	8002

Figure 59. Close-up of the ZP GUI grid view showing sensor ADC values with no sensors connected.

3.2.3.1 Readout Errors

Sometimes, strange readouts from the ZP2015 occur, either in the form of very low numbers, sometimes negative and sometimes zero, very high numbers or temperature values appearing in the place of a sensor value and vice versa. The errors appear at seemingly random times, and often not at all, which made the debugging process quite time consuming because we were unable to recreate the errors.

Because we could see no apparent pattern as to when the errors would appear, we could only make educated guesses as to why. We came up with a few possible explanations and decided to try various tests to eliminate them systematically:

- ADS1115 conversion register is read before conversion has finished
- Temperature data is read instead of sensor data
- Errors in the I^2C communication between ADS1115 and microcontroller
- Errors in the data transmission between microcontroller and computer

A break point and a few extra lines of code, shown in Figure 60, was added to `take_measurements` so that after each data transmission to the computer, a loop cycled through the first eight elements of *conversion_data* and compared them with a fixed value of 13000. If this value was exceeded, the status LED would turn on and the program halt, making it possible to check the contents of *conversion_data* using the debugging functionality in Code Composer Studio. This way we would be able to determine if the data read from the ADS1115 was wrong by reading register values. The number 13000 was more or less randomly selected. A normal error value is around 14000-17000, so 13000 would trigger the check for all of them. The reason that only the first eight elements are checked is because temperature values are usually above 21700. A check for negative values could trigger for

more error events, but the simple test was still sufficient to confirm that wrong values in *conversion_data* was not the source of the problem, as the program did not halt, nor did the LED turn on, even though errors above 13000 appeared in the GUI. The results from the first test also gave strong indications that the problem is not because of the I^2C communication because that would mean incorrect values in *conversion_data*. The same goes for the theory of temperatures being read instead of sensor signals. Further tests therefore focused on the serial communication between microcontroller and computer.

```
231     // Transmit data
232     uart_transmit((char*) conversion_data, 32);
233
234     int i;
235     for(i = 0; i < 8; i++)
236     {
237         if (conversion_data[i] >= 13000)
238         {
239             LED_ON;
240         }
241     }
```

Figure 60. Code used for debugging.

Sometimes, several zero values are read from the input buffer into the *ADCbuffer* array, indicating that bytes might be missing. By adding code to compare the serial port's `Read()` method, which returns the number of bytes read, with the expected amount of bytes, it became clear that bytes are indeed missing at times. A way of indicating when this happens was implemented in the form of a Boolean variable called *readOK* that is set true or false, depending on whether 32 bytes are read. Whenever *readOK* is false, an X is added behind the timestamp. This happens on most rows with errors, but not when entire rows of zeros appear.

A free serial monitor software called *SerialMon* was installed to monitor all data traffic on the serial port, in order to find out if entire transmission could be lost. A snippet from the serial monitor in figure something shows a situation where commands are sent from the computer to the microcontroller, but nothing is returned. A comparison with the log file showed that whenever this happens, an entire row of zeros is logged. It was suspected that in these situations, no data is read from the input buffer and the zeros are instead initial values from *ADCbuffer*. Another test with *ADCbuffer* having initial values different from zero proved this to be the case.

Further tests with the above implementations confirmed that marks were added to the timestamps on almost all readout errors, with a few exceptions. By examining the log files, it became clear that these non-marked errors always appeared between two readouts with missing bytes. This made it seem probable that bytes are sometimes not sent all at once, but end up divided over several transmissions, either because the microcontroller fails at sending them, or because a DataReceived event is fired in the GUI before all 32 bytes are received.

After examining the non-marked errors in both log files and serial monitor results, a clear pattern emerged, strengthening the suspicions that data are divided over more than one transmission. As can be seen in the snippet from the serial monitor in Figure 61, bytes are clearly missing in the first and third transmission, both marked in the log file, but not in the middle one, which is an unmarked error readout.

19:25:40.568	PC → A	32 31 31 31 31 31 31 31 31
19:25:41.128	A → PC	40 1F 41 1F 3B 1F 3D 1F 41 1F 40 1F 40 1F 40 1F 13 55 1A
19:25:41.582	PC → A	32 31 31 31 31 31 31 31 31
19:25:42.143	A → PC	55 05 55 FF 54 F2 54 EB 54 07 55 06 55 40 1F 40 1F 3A 1F 3D 1F 40 1F 3D 1F 40 1F 41 1F 14 55 1A
19:25:42.596	PC → A	32 31 31 31 31 31 31 31 31
19:25:42.942	A → PC	55 05 55 00 55 F2 54 EB 54 08 55 04 55

Figure 61. Snippet from the serial monitor, showing that several bytes are missing.

Table 12 shows the hexadecimal and decimal values from each transmission with added colors to better illustrate the previously mentioned pattern. Hexadecimal values are arranged with the most significant byte to the right. If the green byte sequence is placed behind the orange one, and the blue sequence is shifted to the left and merged with the yellow, two data sequences with perfectly valid results are formed, as shown in Table 13. The test was performed with open channels in a room of approximately 24-25 ºC and the ADC values for both sensor and temperature in the merged data sequences are exactly as expected during such conditions. Figure 62, Table 14 and Table 15 contains equal results.

If one compares the two cases, it is also possible to see that the small variations of ADC values of the different indexes are the same in all combined sequences.

Table 12. Three consecutive byte sequences, highlighted with colors for reference.

Time: 19:25:41:128															
Index	**0**		**1**		**2**		**3**		**4**		**5**		**6**		**7**	
Hex	40	1F	41	1F	3B	1F	1D	3F	41	1F	40	1F	40	1F	40	1F
Dec.	8000		8001		7995		7997		8001		8000		8000		8000	
Index	**8**		**9**		**10**		**11**		**12**		**13**		**14**		**15**	
Hex	13	55	1A													
Dec.	21779		26													
Time: 19:25:42:143																
Index	**0**		**1**		**2**		**3**		**4**		**5**		**6**		**7**	
Hex	55	05	55	FF	54	F2	54	EB	54	07	55	06	55	40	1F	40
Dec.	1365		-171		-3500		-5292		1876		1621		16469		16415	
Index	**8**		**9**		**10**		**11**		**12**		**13**		**14**		**15**	
Hex	1F	3A	1F	3D	1F	40	1F	3D	1F	40	1F	41	1F	14	55	1A
Dec.	14879		15647		16415		15647		16415		16671		5151		6741	
Time: 19:25:42:942																
Index	**0**		**1**		**2**		**3**		**4**		**5**		**6**		**7**	
Hex	55	05	55	00	55	F2	54	EB	54	08	55	04	55			
Dec.	1365		85		-3499		-5292		2132		1109		85			
Index	**8**		**9**		**10**		**11**		**12**		**13**		**14**		**15**	
Hex																
Dec.																

Table 13. The bytes in Table 12 combined into two valid sequences.

Index	**0**		**1**		**2**		**3**		**4**		**5**		**6**		**7**	
Hex	40	1F	41	1F	3B	1F	1D	3F	41	1F	40	1F	40	1F	40	1F
Dec.	8000		8001		7995		7997		8001		8000		8000		8000	
Index	**8**		**9**		**10**		**11**		**12**		**13**		**14**		**15**	
Hex	13	55	1A	55	05	55	FF	54	F2	54	EB	54	07	55	06	55
Dec.	21779		21786		21765		21759		21746		21739		21767		21766	
Index	**0**		**1**		**2**		**3**		**4**		**5**		**6**		**7**	
Hex	40	1F	40	1F	3A	1F	3D	1F	40	1F	3D	1F	40	1F	41	1F
Dec.	8000		8000		7994		7997		8000		7997		8000		8001	
Index	**8**		**9**		**10**		**11**		**12**		**13**		**14**		**15**	
Hex	14	55	1A	55	05	55	00	55	F2	54	EB	54	08	55	04	55
Dec.	21780		21786		21765		21760		21746		21739		21768		21764	

```
19:27:37.185  PC → A  32 31 31 31 31 31 31 31 31
19:27:37.745  A → PC  40 1F 3F 1F 3A 1F 3D 1F 40 1F 3F 1F 40 1F 41 1F 14 55 1A
19:27:38.199  PC → A  32 31 31 31 31 31 31 31 31
19:27:38.760  A → PC  55 05 55 FF 54 F2 54 EC 54 08 55 05 55 40 1F 40 1F 3B 1F 3C 1F 41 1F 3F 1F 3F 1F 41 1F 13 55 1A
19:27:39.212  PC → A  32 31 31 31 31 31 31 31 31
19:27:39.559  A → PC  55 04 55 00 55 F3 54 EB 54 08 55 05 55
```

Figure 62. Another snippet from the serial monitor, showing that several bytes are missing.

Table 14. Three consecutive byte sequences, highlighted with colors for reference.

Time: 19:27:37:745																
Index	**0**		**1**		**2**		**3**		**4**		**5**		**6**		**7**	
Hex	40	1F	3F	1F	3A	1F	3D	1F	40	1F	3F	1F	40	1F	41	1F
Dec.	8000		7999		7994		7997		8000		7999		8000		8001	
Index	**8**		**9**		**10**		**11**		**12**		**13**		**14**		**15**	
Hex	14	55	1A													
Dec.	21780		26													
Time: 19:27:38:760																
Index	**0**		**1**		**2**		**3**		**4**		**5**		**6**		**7**	
Hex	55	05	55	FF	54	F2	54	EC	54	08	55	05	55	40	1F	40
Dec.	1365		-171		-3500		-5036		2132		1365		16469		16415	
Index	**8**		**9**		**10**		**11**		**12**		**13**		**14**		**15**	
Hex	1F	3B	1F	3C	1F	41	1F	3F	1F	3F	1F	41	1F	13	55	1A
Dec.	15135		15391		16671		16159		16159		16671		4895		6741	
Time: 19:29:39:559																
Index	**0**		**1**		**2**		**3**		**4**		**5**		**6**		**7**	
Hex	55	04	55	00	55	F3	54	EB	54	08	55	05	55			
Dec.	1109		85		-3243		-5292		2132		1365		85			
Index	**8**		**9**		**10**		**11**		**12**		**13**		**14**		**15**	
Hex																
Dec.																

Table 15. The bytes in Table 14 combined into two valid sequences.

Index	**0**		**1**		**2**		**3**		**4**		**5**		**6**		**7**	
Hex	40	1F	3F	1F	3A	1F	3D	1F	40	1F	3F	1F	40	1F	41	1F
Dec.	8000		7999		7994		7997		8000		7999		8000		8001	
Index	**8**		**9**		**10**		**11**		**12**		**13**		**14**		**15**	
Hex	14	55	1A	55	05	55	FF	54	F2	54	EC	54	08	55	05	55
Dec.	21780		21786		21765		21759		21746		21740		21768		21765	
Index	**0**		**1**		**2**		**3**		**4**		**5**		**6**		**7**	
Hex	40	1F	40	1F	3B	1F	3C	1F	41	1F	3F	1F	3F	1F	41	1F
Dec.	8000		8000		7995		7996		8001		7999		7999		8001	
Index	**8**		**9**		**10**		**11**		**12**		**13**		**14**		**15**	
Hex	13	55	1A	55	04	55	00	55	F3	54	EB	54	08	55	05	55
Dec.	21779		21786		21764		21760		21747		21739		21768		21765	

The same problems occur also when using only a single channel, which is not unexpected since the number of bytes sent is always the same; only zeros are used on channels that are not selected. It does, however, seem to work fine when transmitting only the 16 bytes containing sensor data. Several tests over a total of 36 hours have not produced a single error.

Because it was fairly certain that the problem is lies in the transmission and not the actual data from the ADS1115, it was decided to exclude all readouts with missing bytes from the GUI's running average, temperature and chart. They are still appended to the log file as normal, but with an X mark on the timestamp.

3.3 Test Results

Due to the findings in 3.2.3.1, it was decided to filter out error measurements from the analysis of test results.

The self-noise, and forced current tests performed on the ZP2015 were both compared with the tests performed on the eZ sense system. Table **16** Shows the self-noise ADC min, max, mean and std. deviation values measured with the eZ sense system. If compared with the ZP2015 self-noise data measured, seen in Table 17, there is very little difference in the values from all channels of the ZP2015. If one were to point at something, the Max values for channel 7 and 8 seems a bit high compared to the other channels, though this does not affect the mean values, it does seem to have an influence on the STDEV, raising it 0.02 ADC values.

Table 16. eZ Sense self noise test.

	ADC
Min	7999
Max	8005
MEAN	8002,119
STDEV	0,809

Table 17. ZP2015 self noise test.

Channel	1 (ADC)	2 (ADC)	3 (ADC)	4 (ADC)	5 (ADC)	6 (ADC)	7 (ADC)	8 (ADC)
Min	7998	7997	7993	7995	7994	7996	7997	7999
Max	8003	8004	8000	8003	8003	8001	8021	8021
MEAN	8000,425	8000,131	7995,616	7997,987	8000,582	7998,857	7999,863	8003,698
STDEV	0,791	0,782	0,806	0,800	0,779	0,801	0,819	0,819

Figure 63 illustrates the eZ sense ADC values from the self noise test. This graph describes a steady signal, oscillating around 8002 ADC values, with a peak-to-peak at 3 ADC values, wich is also implied by the standard deviation in Table **16**. Comparing Figure 63 with Figure 64, the ZP2015 Ch.1 ADC values, adds up to the reasoning that the two systems seems to have a similar self-noise value. Because of the magnitude of measurements acquired, it was decided to only display the illustrations of channel 1.

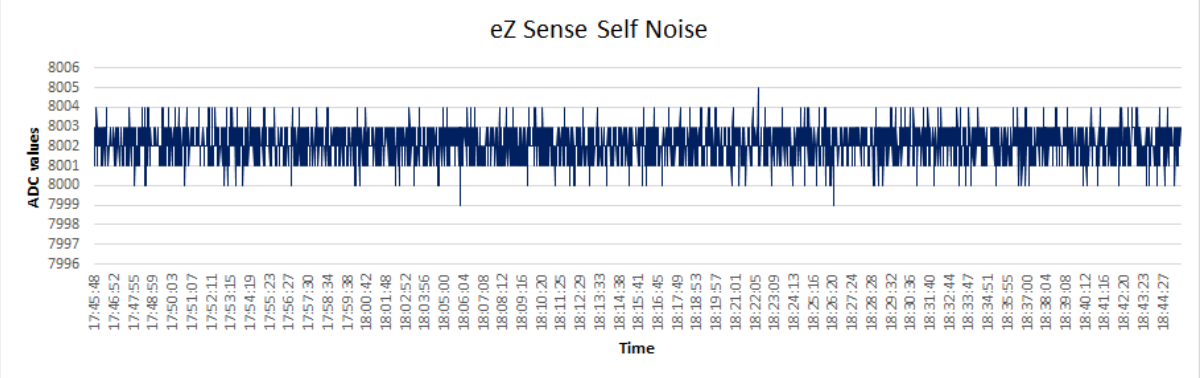

Figure 63. eZ Sense ADC values in self noise test.

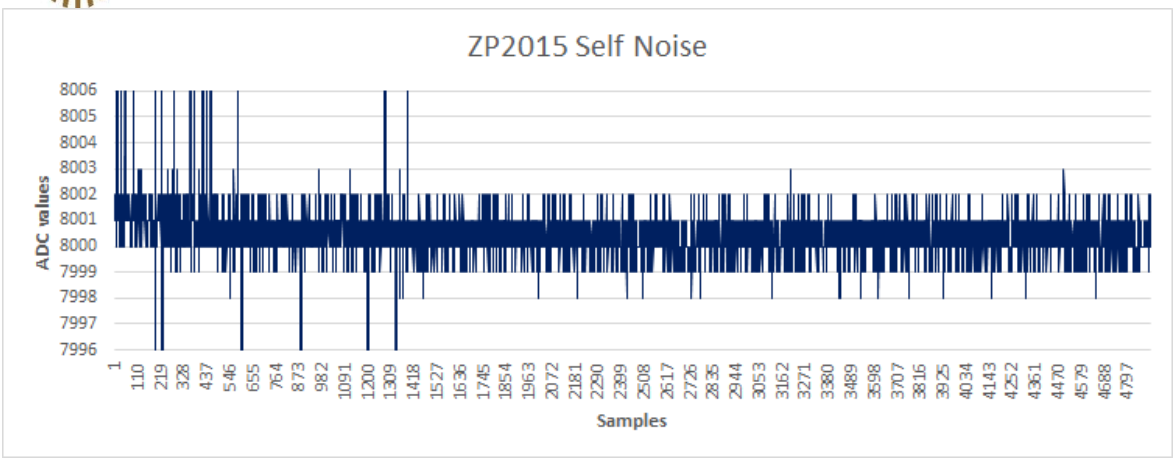

Figure 64. ADC values of channel 1 in self noise test.

The forced current tests was implemented in the test procedure as to obtain comparable data with the eZ Sense system. A constant current is set on the sensor input to resemble a sensor, and in that way one can see how the system reacts to, and process a signal input.

Table **18** shows the uA values for the eZ Sense, and channel 1 on the ZP2015. Both systems have a MEAN value of 1.005uA, the STDEV however, seems to be more steady for the ZP2015 than with the eZ Sense system. This is also verified by studying Figure 65 and Figure 66, where the current is visualized with the same range in both figures. The eZ Sense seems to oscillate with a peak-to-peak at about 0,25uA, while the ZP2015 oscillates with PP at about 0.013uA.

Table 18. Forced current test.

	eZ Sense (µA)	ZP2015, Ch. 1 (µA)
Min	0,861	0,997
Max	1,149	1,010
MEAN	1,005	1,005
STDEV	0,094	0,001

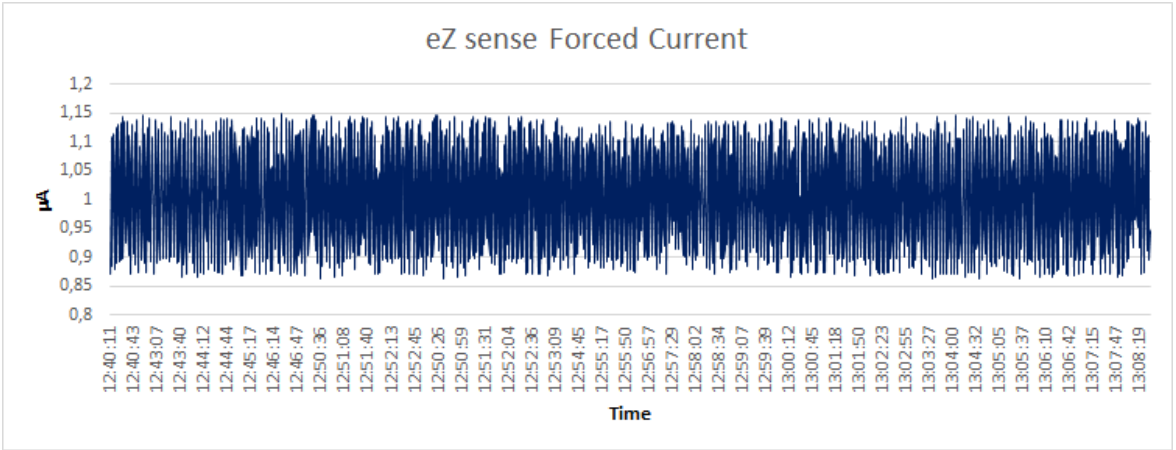

Figure 65. eZ Sense uA values for the forced current test.

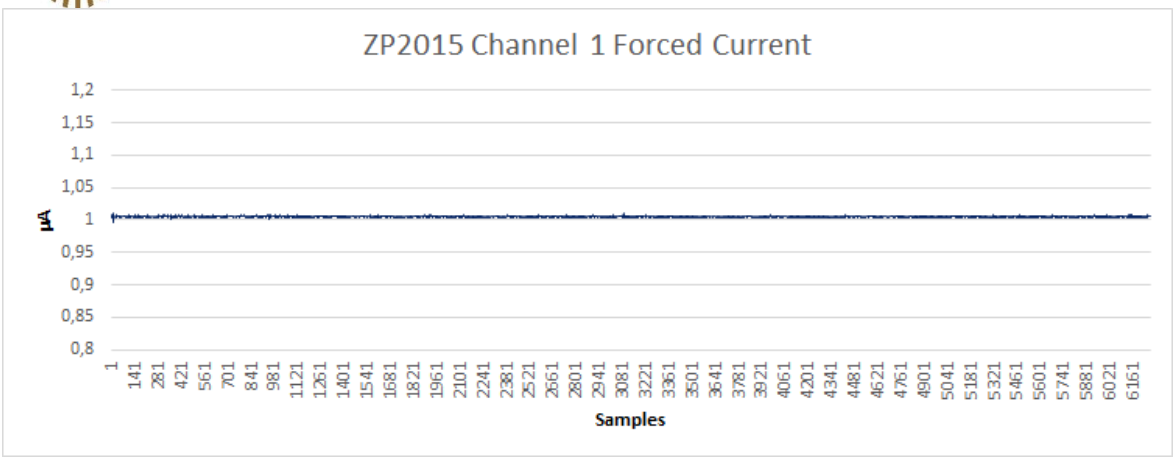

Figure 66. ZP2015 uA values for the forced current test.

The system interface is designed to handle eight sensors simultaneously, as to obtain confirmation of all inlets ability to process sensor input, the response of all sensor inlets was tested by the use of hydrogen peroxide.

The response of all sensor inlets proved to be quite steady, this is shown in Figure 67. As one can see, the sensors are stabilized after about 2 minutes, then the first response is emitted. The response is clear on all sensors. In all measurements, and in all three tests, channel 7 (dark blue), have an equal response of about 0.2 µA. The stability of the measurements is indeed a signal of the stability of the PCB.

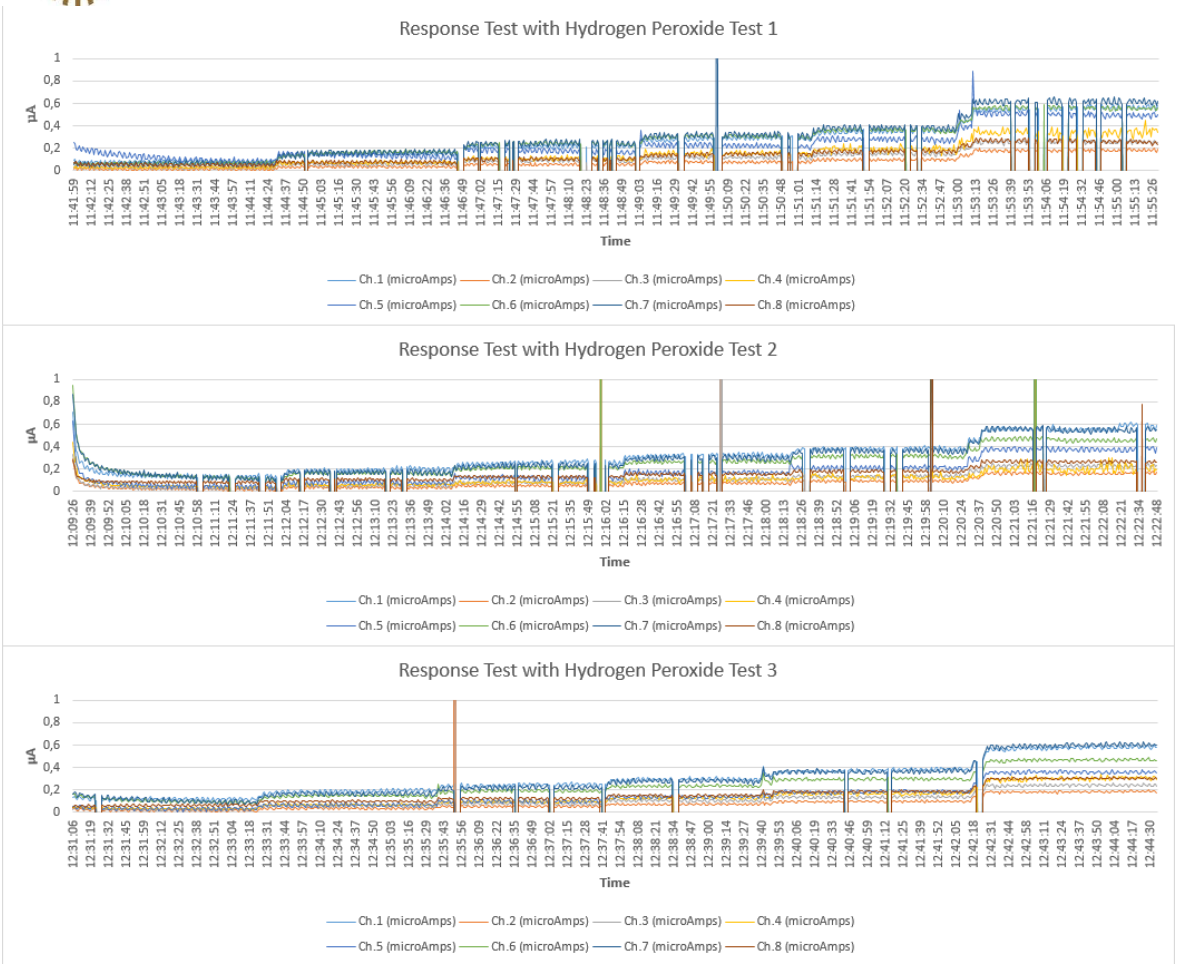

Figure 67. ZP2015 Raw data response test with Hydroogen Peroxide.

The Temperature test results is illustrated in Figure 68. Due to the programming of the temperature cabinet, the first response is somewhat distorted. The cabinet was set to run step one until a temperature of 15 degrees were reached. Due to the heating process being slow compared to the cooling process, the temperature overshot the temperature, and reached a minimum temperature at 12 degrees before climbing up to 15 degrees. Because the program were set to run step 2, hold temperature for 5 minutes, as soon as the temperature reached 15 degrees, the overshooting time is calculated in this time. Because all three tests shows a very steady temperature graph, where all channels seems to have the same response in each of the measurements, also the initial response, there were found no reasons for repeating the tests with a new program. In fact, this gives an indication on the steady response of the temperature sensors in the potentiostats.

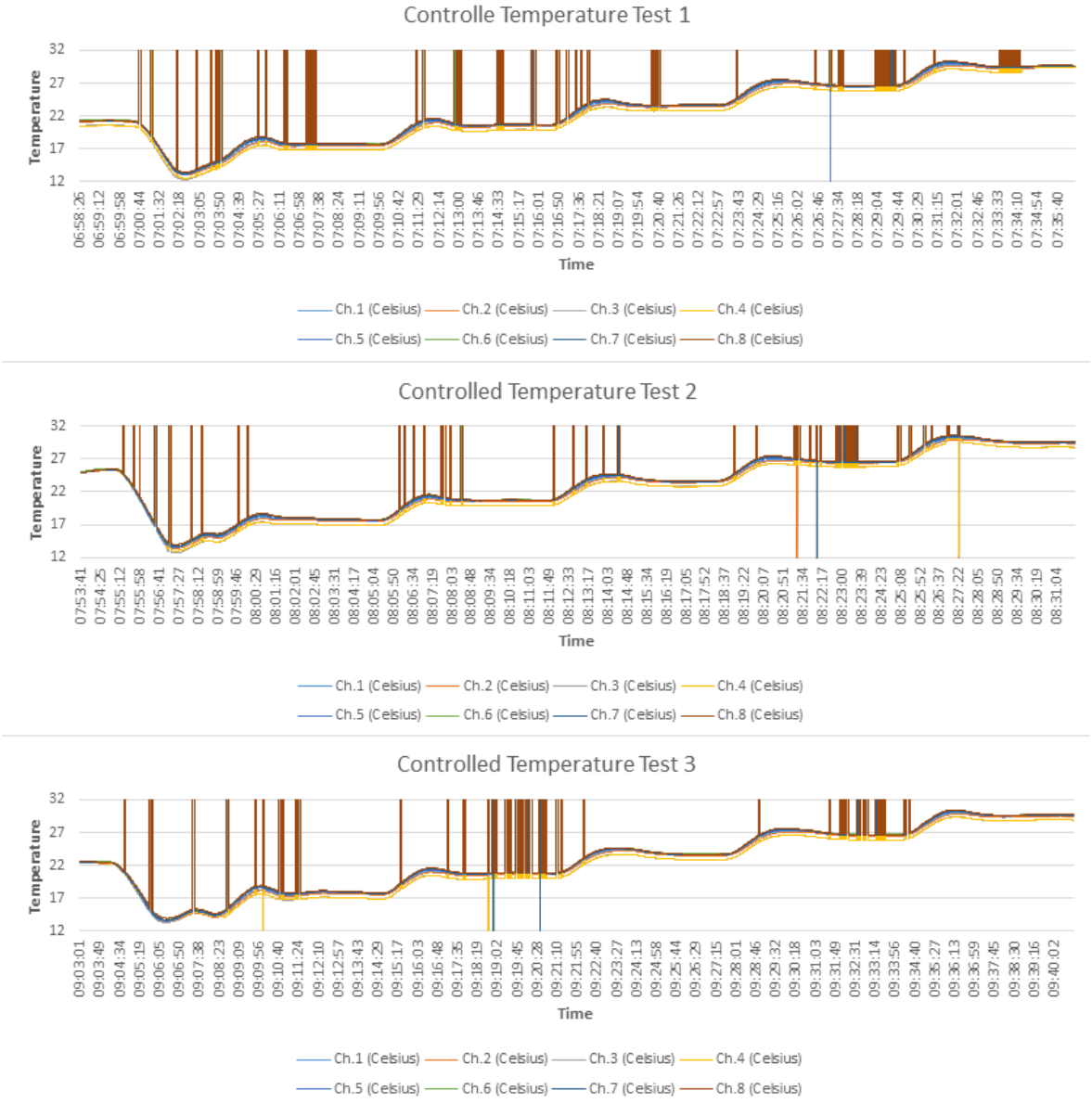

Figure 68. ZP2015 Raw data obtained from temperature test.

As to obtain comparable results between the MFC and the magnetic mixer, two sensors were chosen as testing sensors, these sensors are hereby referred to as sensor 1 and sensor 2. For all these tests, sensor 1 is connected to channel 7, and sensor 2 is connected to channel 2.

Figure 69 illustrates the response of sensor 1 and 2 with Hydrogen Peroxide, with the sensors in the glass with the magnetic mixer. Test 2 and 3 shows similar results, with a maximum peak of about 0.8uA on channel 7, and 0.6 on channel 2. Test 1, however, seems to give a response that is, at maximum, over twice the magnitude in test 2 and 3. There might be several reasons for such variations, but most likely, the 1% Hydrogen Peroxide mixed, have a slightly different relation of PBS and H2O2. As mentioned in section 2.5.4, the difference between each time the 1% H2O2 is mixed is always a factor in the measurements.

The large signal in test 1 is of special interest in this measurement because of the deviation from test 2 and 3 in all responses. Especially because the same sensors are used for all three measurements. One vital fact to consider is that each sensor was tested separately.

Test 1, 2 and 3 were performed on separate days, each time with a new solution of H_2O_2, but the same solution was used for both sensors within each test, implying that the deviation between test 1 and the two others might be a result of the H_2O_2 used for the test.

This assumption is further enhanced by the fact that test 2 and 3 are proving to show steady values for the responses with deviations of less than 0.2uA between the two measurements.

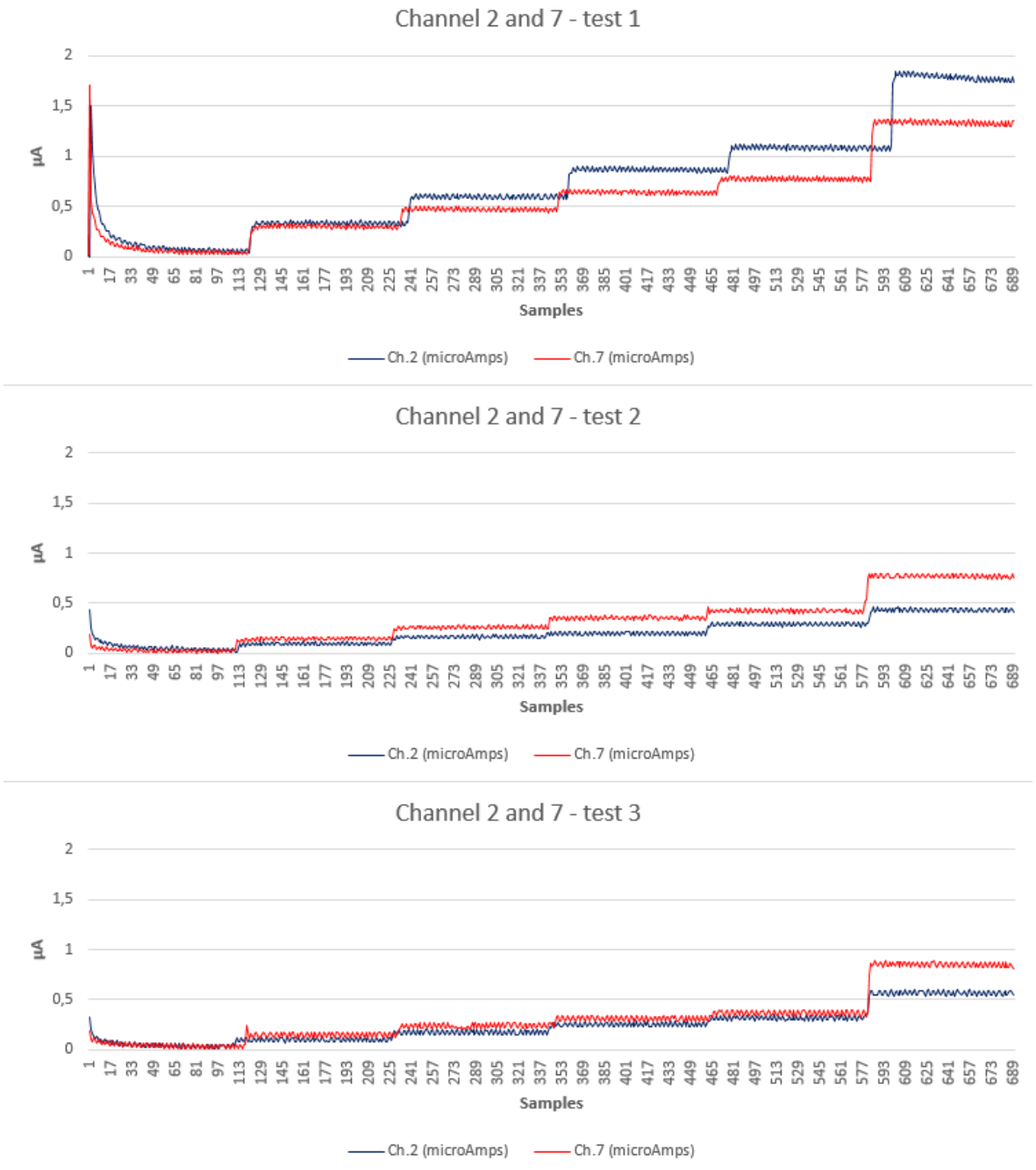

Figure 69. PCB response test with sensor 1 and 2 performed with a magnetic mixer.

Figure 70 and Figure 71 shows a zoom view of test 2 and 3 after the first response, i.e. between 120s and 240s. These graphs proves a rather steady noise interference, at about 0.04uA on both sensors, in both tests.

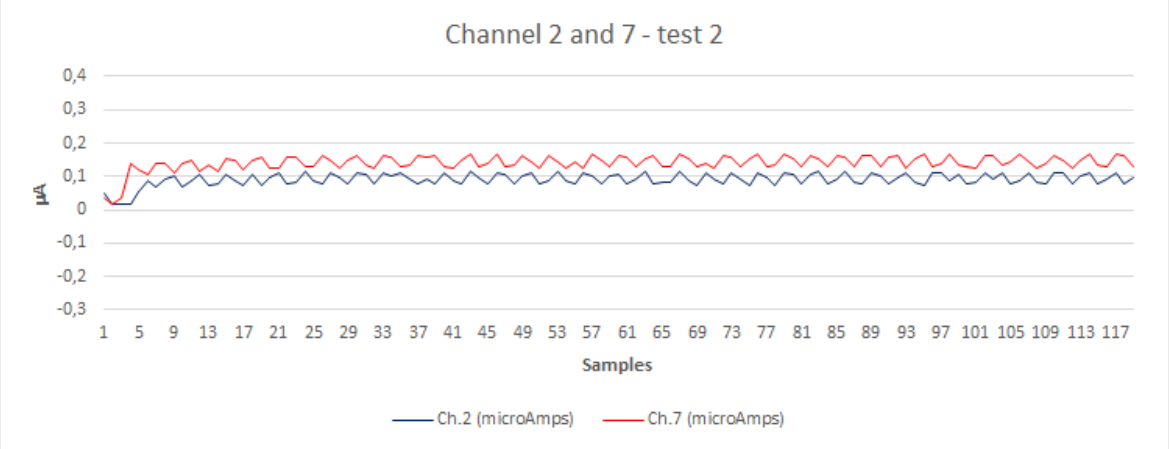

Figure 70. Test 2 after first response.

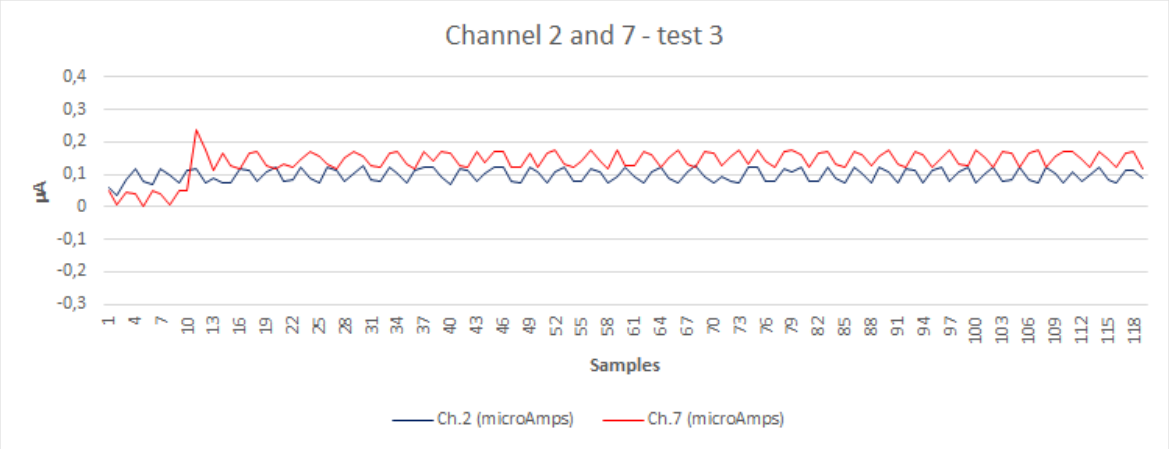

Figure 71. Test 3 after the first response.

This response test was also performed on the MAS_V2_R2 prototype. In this test, only sensor 2 was used. The purpose of this experiment was to work as a control test, as to see whether the measurements of the ZP2015 were within the expected ranges. Figure 72 shows the MAS_V2_R2 response during the response performed by the project group.

As this illustration shows, there are a considerable amount of noise in the system, and it is rather difficult to observe the actual response occurring. The responses should be at 120, 240, 360, 470 and 600[s], this is, however, not clear without actually knowing these response times at forehand. Comparing the illustrations in Figure 69 and Figure 72, the difference in the noise level is clearly shown. The ZP2015 shows a very steady signal, where the variation of the signal in between the step responses are very small compared to the ones generated by the eZ Sense.

By a visual inspection of the zoom view in Figure 73, compared to Figure 70 and Figure 71, the difference in the noise level between the two instruments become clear.

The MAS_V2_R2 is, however, an older PCB than the ZP2015, and the connector for the sensor chip is a little loose. If there is a bad connection, this may result in distortion of the signal.

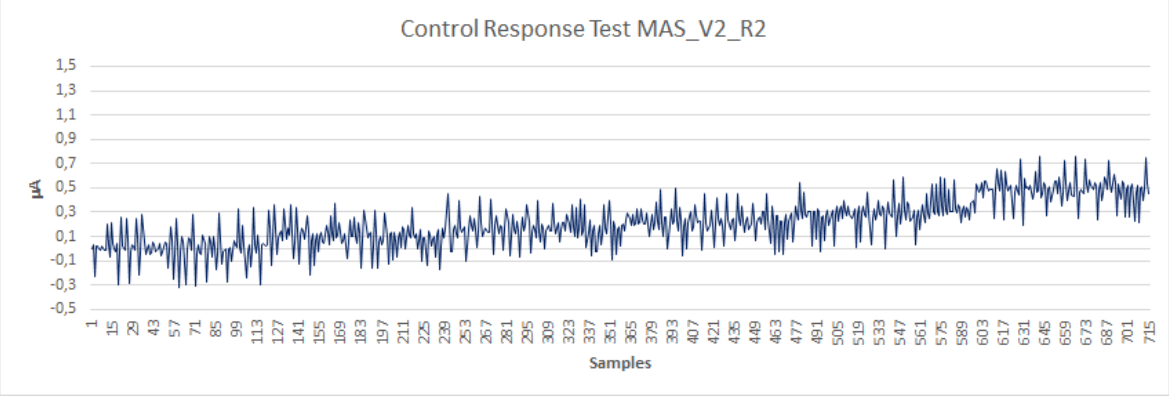

Figure 72. MAS_V2_R2 Control response test

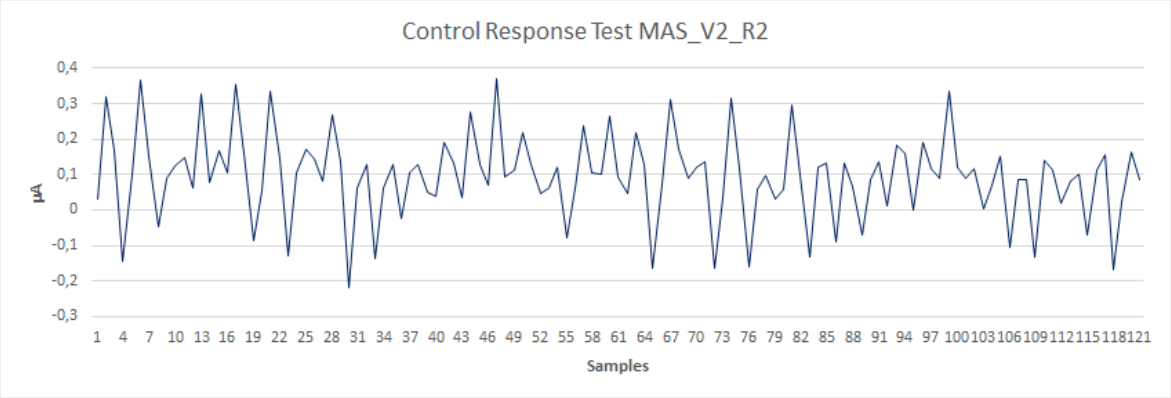

Figure 73. MAS_V2_R2 Control response test after first response.

The MFC was considered a noise friendly system because of the removal of the magnetic mixer from the sensor. The first experiments with the MFC were inconclusive due to air bubbles forming in the MFC, on top of the sensor. See Figure 52. After some experimenting, the group figured out that in order to guarantee that no air pockets were entrapped over the sensor, the measuring chamber had to be completely drained between the concentrations. The particular system used in these experiments, demands about one second to empty, therefore the group let the microfluidic pump take in air for about two seconds between each change of concentrations. An alternative to draining the measuring chamber would be to turn off the pump when changing concentration, however, due to the test setup created, which required one person holding the tube in the concentration, while another person mixing the concentrations, it was not enough hands available to perform the tests in this way. This is, however, something to take into consideration for further tests performed with the MFC.

A typical measurement with too short timespan between two concentrations is illustrated in Figure 74. The three first responses; after 120, 240 and 360 seconds shows typical response values. After about 450 seconds, the response seems to drop, implying an air pocket entrapped over the sensor. At about 480 seconds, the response is partial back again, giving an indication that the air pocket have moved, or a new pocket has been entrapped. After 650

seconds, the signal is once again back to normal, indicating that the air pocket has passed the system.

By emptying the measuring chamber completely between the concentrations, the problem with the air pockets seems to disappear completely.

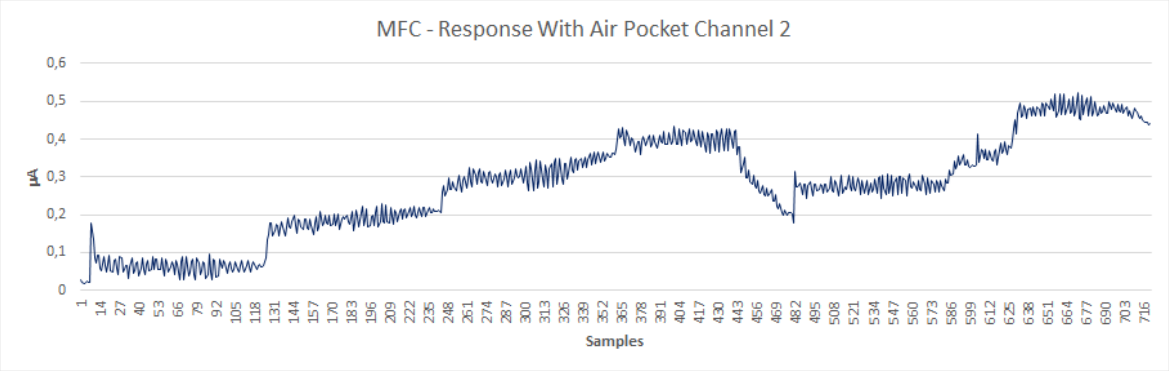

Figure 74. MFC response test with sensor 2 with air pocket entrapped in the measuring chamber.

The MFC response tests with appropriate time interval between the concentrations, shown in Figure 75 and Figure 76. Shows a rather steady oscillation, which is shown in detail in Figure 77 and Figure 78, where both sensors are shown in between the first and the second response. i.e between point 120 and 240[s]. However, by studying Figure 77 and Figure 78, there is a remarkable difference in the noise level between the two measurements. While sensor 2 has a peak to peak at about 0.02uA, while sensor 1 has a peak to peak at about 0.14uA.

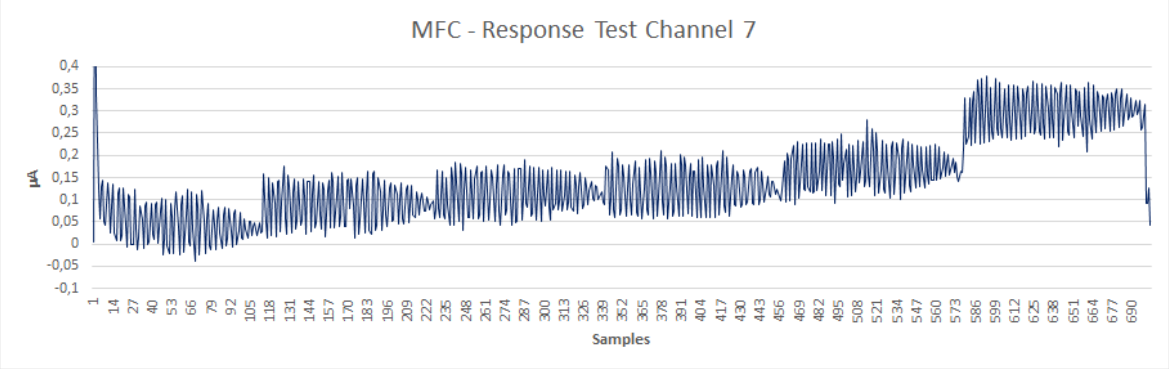

Figure 75. MFC response test sensor 1.

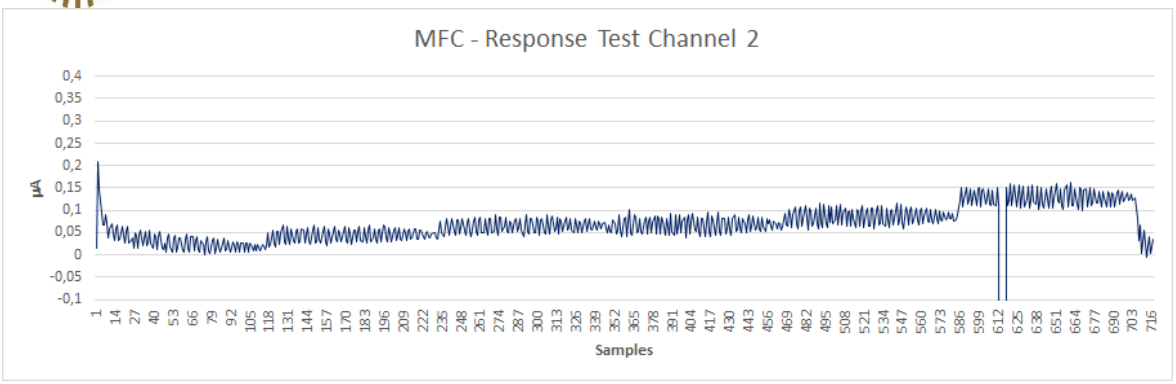

Figure 76. MFC response test sensor 2.

If compared to the results from Figure 70 and Figure 71, Figure 77 and Figure 78 seems to imply that the MFC generates some additional noise. However, due to the fact that the high amplitude noise only appears in one of the measurements, there is a possibility of variable noise affecting the system. One consideration in that matter would be the filling and emptying of the measuring chamber; when fluids fill the chamber, the waves of liquid might generate some noise and instability in the initial phase of a response.

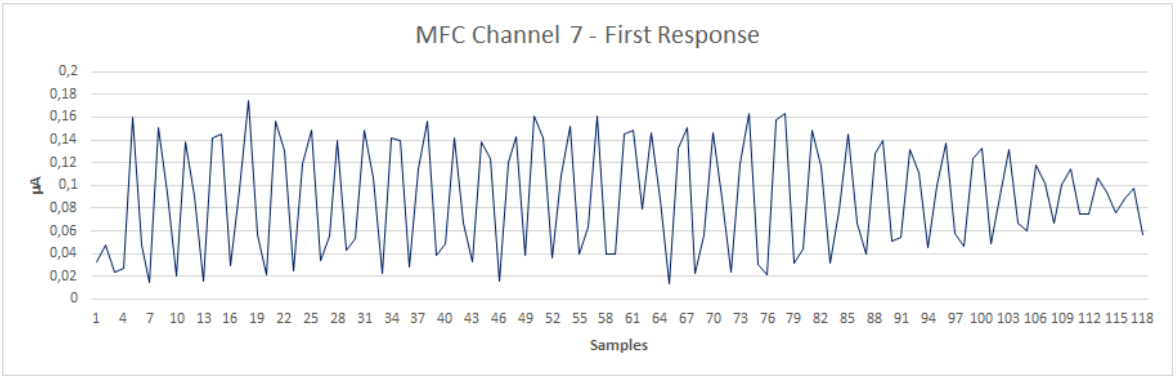

Figure 77. MFC response test sensor 1 after first response.

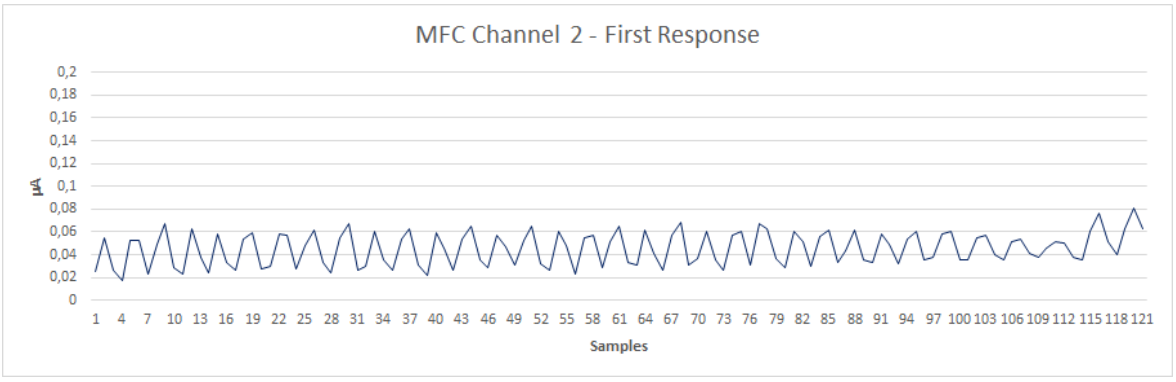

Figure 78. MFC response test sensor 2 after first response.

Figure 79 and Figure 80 shows the response of sensor 2 by the use of versaSTAT and MFC. Though the magnitudes of the responses are much higher than with the PCB, the signal is very steady, and shows very little noise when considering the illustration of Figure 79. When considering Figure 80, however, one can observe that the signal is rising for a while

before stabilizing at a level. During this rising period, one can clearly see a more chaotic signal than when the system is stabilized. This might be an indication on that the noise generated in the MFC comes from the filling of the chamber.

When compared to Figure 83 and Figure 84**,** which shows the versaSTAT test with the magnetic mixer, one see that the MFC measurements shows a considerable more noise initializing of a new concentration than the magnetic mixer tests. This also adds to the point of having a better test setup, so that the pump could be turned off when changing the concentration. This could have removed the noise occurring when altering concentrations.

The reason for the large differences in currents measured with the versaSTAT and ZP2015 have not yet been investigated, but the link between sensor and potentiostat might be a good place to begin. It is unlikely that the problem lies in the analog-to-digital conversion or signal processing in the computer software because of the results from the forced current test in Table **18**, which shows the expected results from a test where a constant current of 1 µA is applied. A comparison with results from the MAS_V2_R2 board shows that the ZP2015 has the expected response of a system based on the technology provided by Zimmer & Peacock. It is, however, something that any future developer may want to take a look at.

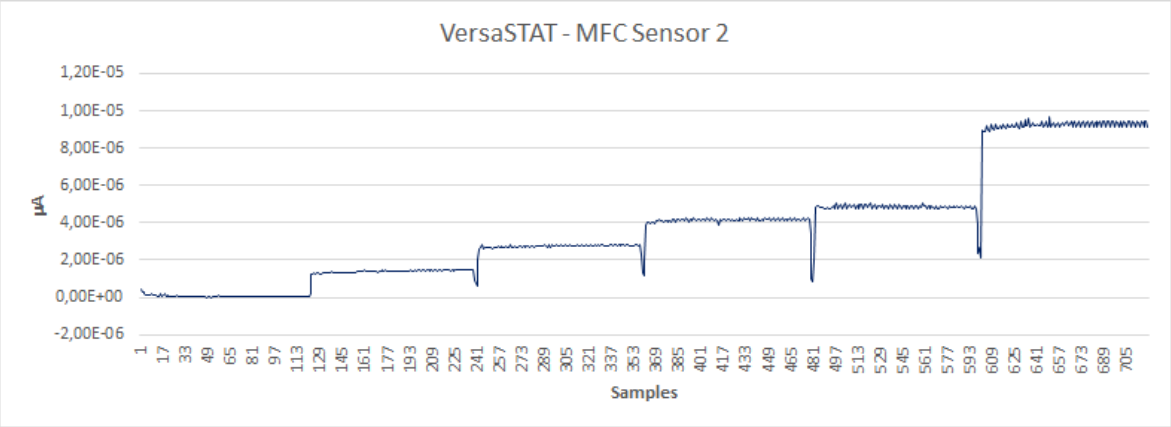

Figure 79. VersaSTAT test - MFC Sensor 2.

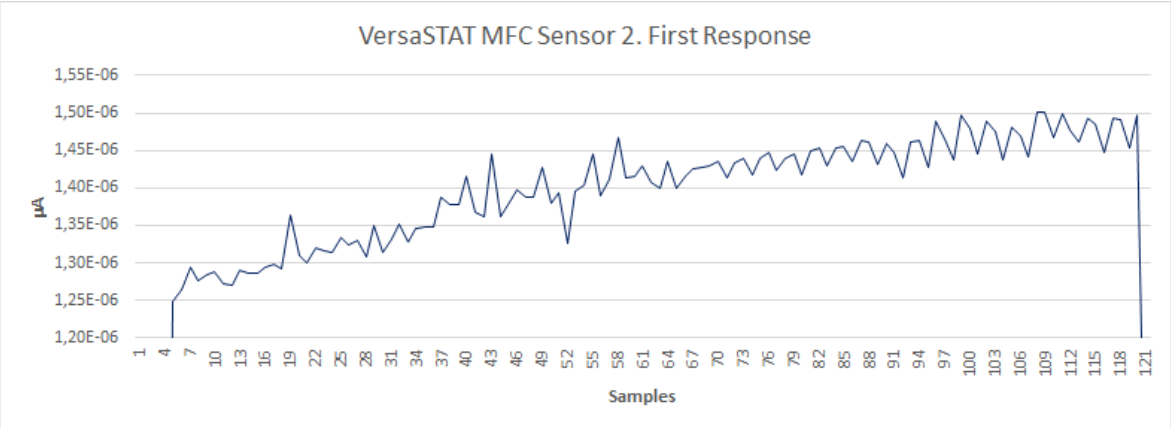

Figure 80. VersaSTAT test - MFC sensor 2 after first response.

In order to check if filling the chamber is an actual source of noise, an extra test was performed, where the flow speed of the pump were reduced with 10 percent. These results are illustrated in Figure 81 and Figure 82, and does indeed show more chaotic tendencies at the initializion of a new concentration than with 100 % speed.

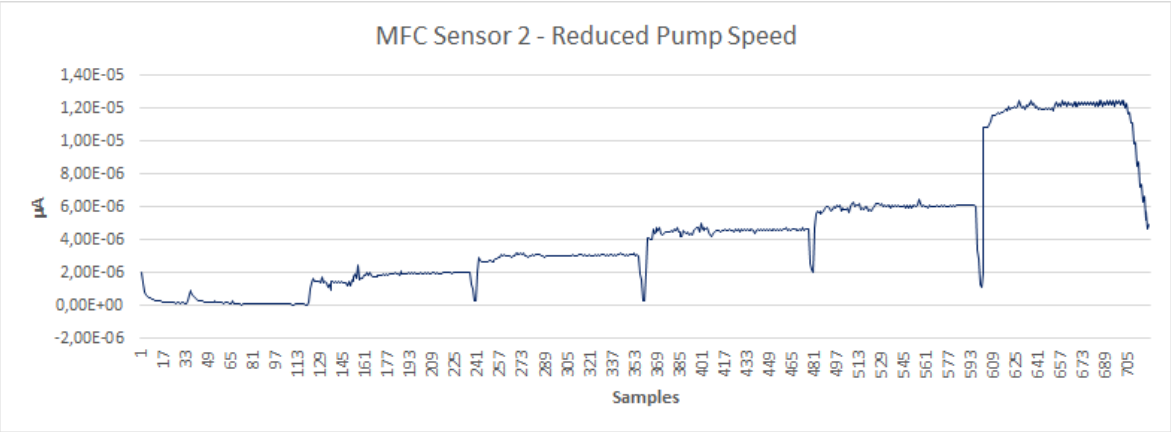

Figure 81. VersaSTAT test - reduced speed with sensor 2.

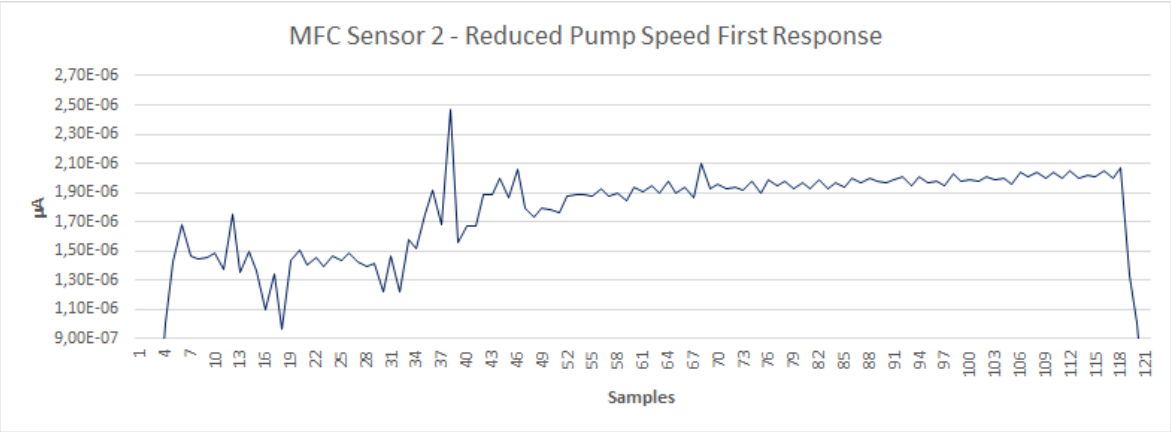

Figure 82. versaSTAT test - reduced speed after first response.

The versaSTAT tests were also performed with the magnetic mixer. The step response in is shown in Figure 83 and Figure 84, and the noise level in Figure 85 and Figure 86**.** These illustrations, though more chaotic than the second part of Figure 80, shows a steady amplitude, adding to the idea of the MFC needing time to stabilize.

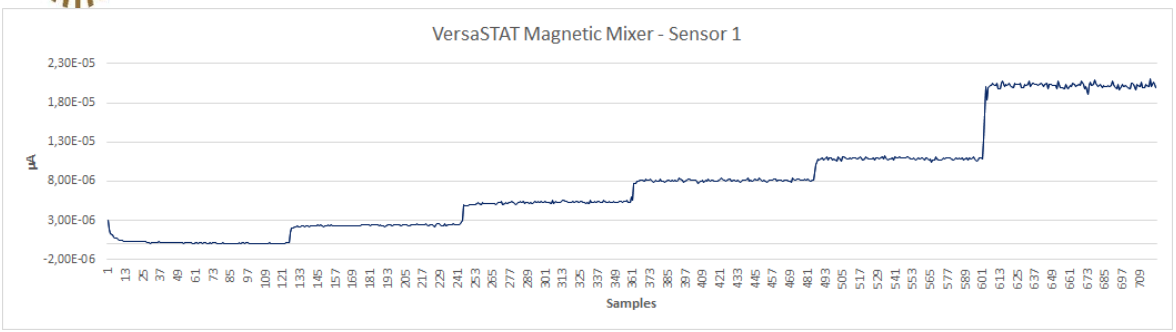

Figure 83. versaSTAT test - sensor 1 in magnetic mixer.

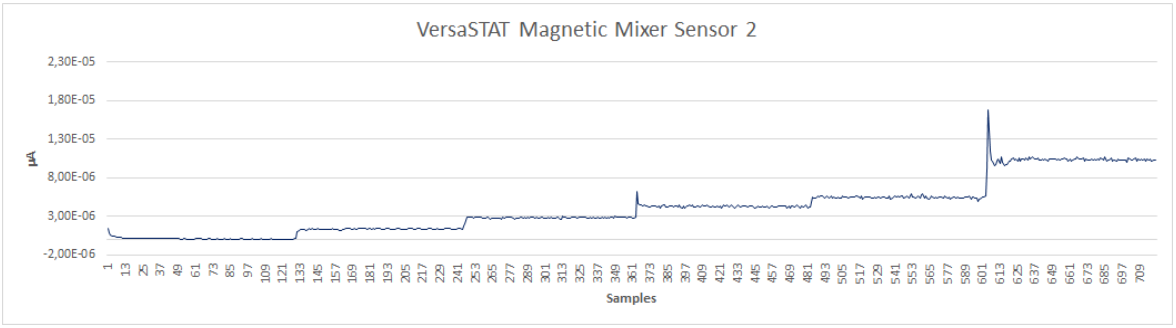

Figure 84. versaSTAT test - sensor 2 in magnetic mixer.

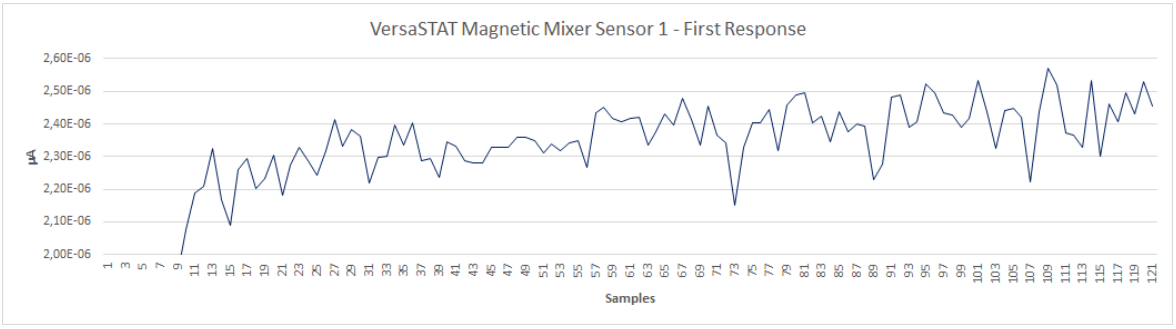

Figure 85. VersaSTAT test - sensor 1 after first response.

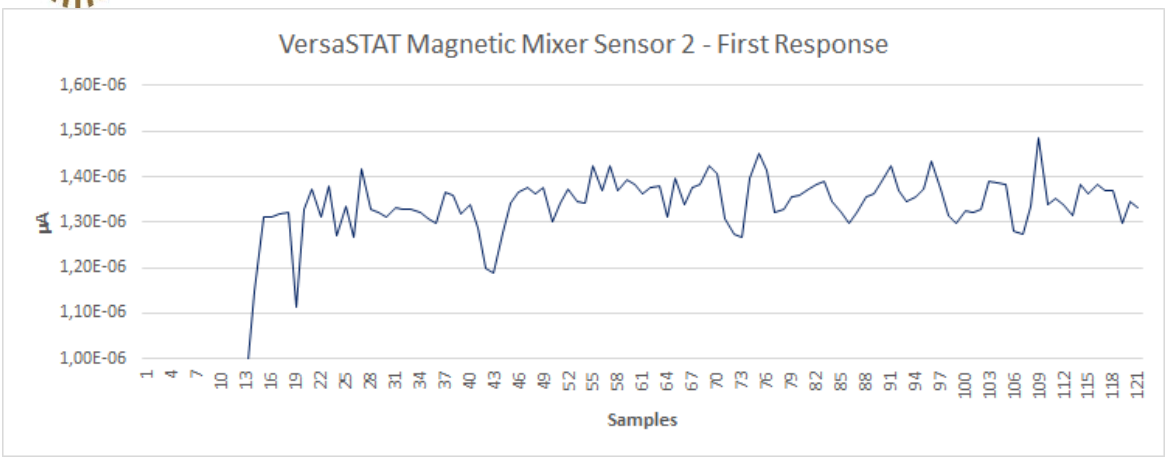

Figure 86. VersaSTAT test - sensor 2 after first response.

4 Conclusion

The MFC designs were made by casting PDMS, a silicone elastomer, on a master. The master is previous made by a lithography process with pattern transfer, using a negative photoresist to create the structure. Depending of the design of the MFC, the PDMS is bonded on a glass slide through oxygen plasma bonding, creating covalent bonds between the glass and the PDMS. Holders were designed to seal the MFCs to prevent leakage.

The MFCs seemed to work, but with irregular results. There were some problems with air pockets blocking the sensing area of the sensor chip, and the liquid seemed to be distributed unevenly. The liquid always seemed to choose one channel/outlet, and it might be an idea to redesign the MFCs, making them suited to only have one outlet. This is due to air blocking the outlet. If a higher pressure is required, it might be a good idea to avoid the designs with channels. This was not tested hence the pump had a limit of flowrate being approximately $0.5cm^3/s$.

Fabricating the master, the photoresist always seemed to be thicker on one side than the other. The thickness clearly changed when one hot plate were replaced with another. That seems to indicate that the hot plate has to be leveled before the soft bake. It might be necessary to look into the spin parameters as well.

The PDMS casting and Oxygen plasma bonding process had no problems. It was necessary to clean the PDMS with isopropanol before bonding, or better, wait to cut out the designs until it were to be bonded in the clean room to avoid contamination.

The holder for the three designs mounted on glass did not work, and the alternative, using silicone glue to seal the chamber was too time consuming to use for testing of sensors. If one of these designs are to be used, a new design must be created for the holder.

The design used for testing the sensor worked quite well, but even if the sensor response were supposed to be under one second, it took a much longer time to stabilize the readings. This might work better with higher pressure, but when the speed of the pump were used on maximum (50rpm), the liquid managed to pass the silicone grease and enter the connector.

The use of an MFC for noise reduction was not a complete success; the noise levels were even higher than while using a regular magnetic mixer. There were problems with the measuring chamber not being filled as fast as needed, due to the low pressure generated from the microfluidic pump, which lead to erratic sensor behavior when changing the fluid. A solution was not found for this problem, mainly because the pump itself was close to maximum pressure, but also because higher pressures increased the possibility of unwanted leakage from the MFC. One solution to this problem might have been to acquire a new pump system.

The goals set for the front-end interface and control system in the pre-project report were accomplished, even though the focus was adjusted in the starting phase. The new system is more versatile in the sense that it can handle multiple channels efficiently, as well as being able to adjust the transimpedance gain of its potentiostats; allowing for a wider range of sensor currents. Real-time graphic presentation of signals makes it easier to monitor the responses of multiple sensors simultaneously, and measuring temperatures provides a way of comparing sensor responses under different temperature conditions. The system's noise characteristics have also been improved compared to the eZ Sense, even though the task of noise reduction ended up as a secondary objective.

Due to the fact that the fluid flow in a microfluidic flow cell is to be laminar, the measurements of Figure 80 and Figure 82, though they show the same tendency, does not make. A subject for further testing would be to examine whether the flow is actually laminar, or if there could be any noise in the fluid flow. In that interest to matter it would also be interesting to calculate Reynolds number for the system - which takes into consideration the

fluid density, kinematic velocity and the hydraulic diameter, i.e. the characteristic travelled length, along with the dynamic viscosity of the fluid- in order to observe whether the theory and the measurements adds up. Reynolds formula is stated in equation (15) [27]. This is, however, somewhat difficult on the MFC used, because it does not have any internal channels. This does however, imply that there should indeed be a laminar flow. One last question to consider is then; can the outlet size be too big, so that the fluid is drained before the chamber reaches a steady level, and therefore result in a period of time where the sensor is partial exposed to the fluid, resulting in an erratic sensor measurement in the beginning of a new concentration? This would makes sense when considering Figure 80 and Figure 82, where the reduced pump speed confirmed a longer, more erratic period before obtaining a steady signal.

$$Re = \frac{\rho V D_H}{\mu} \qquad (15)$$

$$\text{Where } D_H = \frac{4A}{P} \; ; \; P = Circumference \; and \; A = Area$$

There are still some flaws in the new system; the biggest one being the issues with data transmission between microcontroller and computer, as discussed in section 3.2.3.1. If the problem can indeed be fixed by reducing the number of bytes sent, one must consider what is most important in the current system version; being able to log temperatures on all channels or have error-free transmissions. There is, however, no reason to believe that it is impossible to find a permanent solution. This might be a job for future developers.

Another issue revealed itself during the final tests when comparing sensor readouts with those of the versaSTAT; sensor currents read by the versaSTAT were much higher. Because the new system yields results consistent with those of the eZ Sense, the task has been solved when considering the original goals; to develop an instrument based on eZ Sense technology.

5 Index

6 References

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7 Appendices

7.1 Appendix 1 Table flow rate and sensor response

28.04.2015	Design	5 cm between each step					Flow speed	Cross section	Flow rate	Volume meas.	Sensor
		1	2	3	4	5	[cm/sec]	tube [cm^2]	[cm^3/s]	chamber [cm^3]	respons
5	1		1,33	2,9	4,43	5,87	3,30396476	0,007854	0,0259493	0,16738	6,45026
	2	6,4	8,23	9,67	11,5	13	3,01659125	0,007854	0,0236923	0,14727	6,21594
	3	2,3	3,8	5,37	7,03		3,17124736	0,007854	0,0249069	0,022585	0,90677
	4	6,3	7,8	9,5	11,1	12,7	3,13971743	0,007854	0,0246593	0,14034	5,69115
	1	3,03	3,8	4,63	5,4		6,32911392	0,007854	0,0497088	0,16738	3,36721
10	2							0,007854	0	0,14727	
	3	6,33	7,07	7,83	8,57	9,33	6,66666667	0,007854	0,05236	0,022585	0,43134
	4							0,007854	0	0,14034	

29.04.2015	Design	5 cm between each step					Flow speed	Cross section	Flow rate	Volume meas.	Sensor
		1	2	3	4	5	[cm/sec]	tube [cm^2]	[cm^3/s]	chamber [cm^3]	respons
5	1	2,85	4,3	5,97	7,59	9,08	3,21027287	0,007854	0,0252135	0,16738	6,63851
	2							0,007854	0	0,14727	
	3	1,73	3,32	5,02	6,58	8,27	3,05810398	0,007854	0,0240183	0,022585	0,94032
	4	0,78	2,54	4,03	5,56		3,13807531	0,007854	0,0246464	0,14034	5,69413
10	1	5,69	6,41	7,19	8,03	8,75	6,53594771	0,007854	0,0513333	0,16738	3,26065
	2							0,007854	0	0,14727	
	3	3,56	4,41	5,12	5,97	6,75	6,26959248	0,007854	0,0492413	0,022585	0,45866
	4	3,02	3,8	4,54	5,36	6,17	6,34920635	0,007854	0,0498667	0,14034	2,8143

07.05.2015	Design	5 cm between each step					Flow speed	Cross section	Flow rate	Volume meas.	Sensor
		1	2	3	4	5	[cm/sec]	tube [cm^2]	[cm^3/s]	chamber [cm^3]	respons
5	2	0,78	2,54	4,07	5,56		3,13807531	0,007854	0,0246464	0,14727	5,9753
10	2	0,53	1,32	2,13	2,92	3,71	6,28930818	0,007854	0,0493962	0,14727	2,9814

7.2 Appendix 2 Table flow speed

Speed 10	Channels, cross section cm^2				Flow rate cm^3/s	Flow speed cm/s			
Design	Upper	Lower				Upper	Lower		
2	0,0018	0,0032			0,05	27,777778	15,625		
Design	Upper L	Upper R	Lower L	Lower R		Upper L	Upper R	Lower L	Lower R
4	0,004	0,0024	0,0032	0,0032	0,05	12,5	20,833333	15,625	15,625

Speed 5	Channels, cross section cm^2				flow rate	Flow speed cm/s			
Design	Upper	Lower				Upper	Lower		
2	0,0018	0,0032			0,025	13,888889	7,8125		
Design	Upper L	Upper R	Lower L	Lower R		Upper L	Upper R	Lower L	Lower R
4	0,004	0,0024	0,0032	0,0032	0,025	6,25	10,416667	7,8125	7,8125

7.3 Materials for Front-end Interface

7.3.1 Prototype on Breadboard

The following materials were used to setup the breadboard prototype:

- Breadboards
- Jump wires
- USB Connector on breakout board
- SMD ICs
  - One MSP430F22x4 microcontroller on breakout board
  - Two ADS1115 Analog-to-Digital Converters on breakout boards
  - Eight LMP91000 Configurable AFE Potentiostats for Low-Power Chemical-Sensing Applications on breakout boards
  - One LT6656-2.5 Voltage Reference on a discarded MAS_V2_R2 board
- Through hole ICs
  - One 74HC138PW 3-to-8 line decoder/demultiplexer
- Through hole Resistors
  - Two 27ohms
  - Twenty-four 100ohms
  - One 47Kohms
  - Two 10Kohms
- Through hole Capacitors
  - One 0.01uF
  - Two 10uF
  - Nine 0.1uF
  - Two 47pF
  - One 0.0022uF
  - Five 1uF
  - Thirty-Two 100nF NB!
  - One 4.7uF

7.3.2 PCB

The following materials, equipment, software and chemicals were used for the PCB production:

- Bungard electronics Presensitized board
- Bungard Bell Splash Etcher
- Vacumat3 UV-light/Vacuum machine
- Sodium hydroxide(NaOH) developer (32ml NaOH to 2.5L tap water)
- Sodium thiosulfate($Na_2S_2O_2$) Etchant (6Kg Sodium Persulfate to 24L water(40degrees)
- EFD Dispenser Ultra 1400 Series
- Techno HA-02 Reflow oven
- Microscope
- Proteus PCB Design Package
- USB connector
- Breakout boards
- SMD ICs
  - One MSP430F22x4 microcontroller
  - Two ADS1115 Analog-to-Digital Converters
  - Eight LMP91000 Configurable AFE Potentiostats for Low-Power Chemical-Sensing Applicatioins
  - Four LT6656-2.5 Voltage references
- SMD resistors
  - Two 27ohms
  - Twenty-four 100ohms
  - One 47Kohms
  - Two 10Kohms
  - Eight Free for tilbakekobling potentiostater
- SMD Capacitors
  - One0.01uF
  - Two 10uF
  - Nine 0.1uF
  - Two 47pF
  - One 0.0022uF
  - Five 1uF
  - Twenty-four 100nF
  - Eight 58pF
  - One 4.7uF

7.4 Materials for MFC fabrication

7.4.1 Master

Materials and chemicals:

- The glass mask designed with L-edit
- Isopropanol (IPA) (2-Propanol Emplura®, Merck KGaA, Germany)
- Acetone (Acetone for analysis EMURE® ACS Merck KGaA, Germany)
- Silicon wafer, 4”
- SU-8 100 negative photoresist (Organic Resin Solution, SU-8 100 Y131273, MicroChem Corp., USA)
- Developer for SU-8 100 (Developer mr-Dev 600, micro resist technology GmnH, Germany)

Equipment:

- Fume hood 4 for general solvents
- Fume hood 5 for corrosive solvents
- Spinner 1 Semitool 1
- Mask Aligner (Karl Suss MA56, SUSS MicroTec, Germany)
- Profilometer (DEKTAK 150 Stylus ProfilerScan, Veeco Instruments Inc., USA)
- Optical microscope (DM 4000M, Leica microsystem, Germany)

7.4.2 PDMS casting

Materials and chemicals:

- PDMS resin - Sylgard® 184 Silicone Elastomer , kit whith PDMS base and curing agent [11].
- Silicone tubes, ID 1mm and OD 3mm (VWR, Norway)
- Plastic holder for PDMS
- Disposable syringe
- SU-8 master
- Duco cement

Equipment:

- Fume hood 2-Bio
- Oven
- Vacuum jar with vacuum pump (Epovac, Struers, Denmark)
- Scale (SI-234, Denver Instrument, USA)

7.4.3 Oxygen plasma bonding

Materials and chemicals:

- IPA and acetone for cleaning
- PDMS casting
- Glass slides (Tønsberg Glassliperi, Norway)

Equipment:

- Reactive ion etcher (RIE) (PlasmaTherm SLR series (RIE), PlasmaTherm, USA)

7.4.4 Holder

- ∇ Plexiglas, 5mm thick (Tønsberg Glassliperi, Norway)
- ∇ Bolts and nuts, Ø 6mm (Tønsberg Maskinforretning, Norway)
- ∇ Silicone glue (Dow Corning® 3140 RTV Coating , Dow Corning Corporation, USA)
- ∇ Silicone grease
- ∇ Plastic tubes, ID 1mm and OD 1/16” ≈ 1,6mm
- ∇ Silicone tubes, ID 1mm and OD 3mm

7.5 Materials for MFC testing

- ∇ Holder and designs
- ∇ Peristaltic pump (403U/VM2, Watson-Marlow, UK)
- ∇ Food coloring
- ∇ DI water
- ∇ Silicone grease, PTFE fett, Biltema, Norway
- ∇ Extra tubing,
- ∇ Video camera (cell phone)

7.6 Standard fabrication procedure for master

Cleaning wafer:

Even new wafers have to be cleaned to avoid impurities that can spoil the results. Normally it is enough to clean thoroughly with acetone and rinse off with IPA and finally DI-water (distilled water). Dry the wafer using Ni (Nitrogen).

Prebaking:

To avoid any kind of humidity in the wafer, it is necessary to prebake it. This is done on a hot plate at 200ºC for 10 minutes. It is important to spin coat quick after the wafer is finished prebaking, because it will absorb humidity when removed from the hot plate.

Spin coating:

Cover the bowl and removable shield with aluminum foil before spin coating, to protect it from the SU-8. Set the parameters for the spin coater according to the desired thickness.

Table 19. Spin coating parameters

Step	Thickness (µm)	Speed (rpm)	Accelerate (sec)	Time (sec)	Decelerate (sec)
1	100	0	0	0	-
	150	0	0	0	-
	250	0	0	0	-
2	100	500	5 (500/5=100)	10	-
	150	500	5 (500/5=100)	10	-
	250	500	5 (500/5=100)	10	-
3	100	3000	8 (2500/8=313)	30	30
	150	2000	5 (1500/5=300)	30	30
	250	1000	2 (500/2=250)	30	30

The recommended amount of resist is 1 ml per inch of the diameter of the wafer
There is no marking or socket on the spin coater. This implies that the wafer has to be placed on the spinner, then the program has to be started just to see how the wafer moves. If it wiggles it has to be moved and the same procedure has to be done again until it seems centered.
The SU-8 is applied in the center of the wafer directly from the bottle. Let it cover approximately 4cm in diameter.

Soft baking:

To evaporate the solvent in the SU-8, the wafer has to be soft baked on a hot plate.
It is recommended to ramp or to step up the temperatures.

Table 20. Soft bake parameters

Thickness (µm)	Temperature (ºC)	Time (min)
100	65	10
150	65	20

250	65	30
100	95	30
150	95	50
250	95	90

Pattern transfer:

Pattern transfer occurs by illuminating the wafer with the SU-8 through a mask.
Place the wafer with SU-8 and the mask in the mask aligner and select the time from the Table 21 according to the desired thickness. Start the process.

Table 21. Exposure parameters

Thickness (µm)	Dose (mJ/cm^2)	Light intensity (mW/ cm^2)	Time (s)
100	≈ 550	8.5	≈ 65
150	≈ 650	8.5	≈ 76
250	≈ 700	8.5	≈ 82

Post exposure baking:

After the exposure, place the wafer on a hotplate and follow the Table 22according to the desired thickness. Let the wafer cool on the plate until it reaches approximately 75^{o}C

Table 22. Post exposure bake parameters

Step	Thickness (µm)	Temperature (^{o}C)	Time (min)
1	100	65	1
	150	65	1
	250	65	1
2	100	95	10
	150	95	12
	250	95	20

Developing:

Submerge the wafer in the developer until the unexposed SU-8 is removed. The expected times are given in Table 23. Clean the wafer with IPA and blow dry with Ni.

Table 23. Development time

Thickness (µm)	Development (min)
100	10
150	15
250	20

Hard baking:

This is not necessary.
Place the master on the hotplate at 150^{o}C and ramp it up to 200 ^{o}C. Leave it on the plate to cool slowly.

7.7 Test Procedures

7.7.1 PCB Self Noise

Equipment:

- ∇ Sensetion PCB ZT2015 or newer verison
- ∇ USB
- ∇ Laptop
- ∇ ZP GUI

Procedure:

- ∇ Connect the PCB to the laptob through a USB
- ∇ Start the GUI

7.7.2 PCB with constant Current on Sensor Input

Equipment:

- ∇ Sensetion PCB ZT2015 or newer versions
- ∇ USB
- ∇ Laptop
- ∇ ZP GUI
- ∇ IM6 Impedance measurement unit from Zahner elektronik, or similar equipment which delivers a small constant current
- ∇ Four probes for connection between the IM6 outputs and the sensor input
- ∇ 3 wires about 5cm long.

Procedure:

This procedure is meant for the first version ZT2015 with the zahner as current source. For other types of current source, or newer versions than the ZT2015, there may be different ways of connecting the PCB. Please check your equipment thoroughly before attempting tests.

IM6 comes with six outputs, Sense, test, CE, RE, probe1 and probe E. For this test, only the CE, RE, test and sense will be used. Following is the setup used for the self noise tests performed in this project:

- ∇ Connect probes on the four outputs to be used, These are connected together as follows; Counter electrode and Reference electrode, Test electrode and Sense.
- ∇ Short RE and CE (pin 13 and 14) on the PCB sensor input with wires
- ∇ Connect RE and CE from IM6 to The shorted RE and CE on PCB
- ∇ Connect the test electrode and Sense to WE (pin 12) on the PCB sensor input.
- ∇ Start the IM6 by powering on the three separated parts, then power up the computer for controlling the IM6
- ∇ Open the software, set the galvanostat to 1uA
- ∇ Connect PCB to PC through USB
- ∇ Start the GUI

7.7.3 Sensor in Hydrogen Peroxide

Equipment:

- ∇ Sensetion PCB ZT2015 or newer versions
- ∇ USB
- ∇ Laptop
- ∇ ZP GUI
- ∇ 1% Hydrogen Peroxide (H2O2) made up from
  - ∂ 9.7mL PBS
  - ∂ 330µL 30% H_2O_2
- ∇ PBS, made up from:
  - ∂ $7.49g\ Di-Sodium\ Hydrogen\ Phosphate\ anhydrous\ (Na_2HPO_4)$
  - ∂ $1.79g\ Sodium\ dihydrogen\ phosphate\ (NaH_2PO_4)$
  - ∂ $0.29g\ Sodium\ Chloride\ (NaCl)$
- ∇ Eight E&Z electrochemical glucose sensor
- ∇ Eight sensor connectors
- ∇ Measuring glass for H_2O_2
- ∇ Measuring Glass for PBS and H_2O_2 mix
- ∇ Mixer

Procedure:

This procedure is meant for the specific test of eight sensors simultaneously, in one measurement glass. The volumes used must be altered, and adjusted to the proportion of the test. In order to calculate the H_2O_2 test solution, a solution calculator, which is attached on the accompanying CD, were used.

- ∇ All eight sensors are taped together in a bundle, so that each of the sensors can be held at the same distance from the magnetic mixer
- ∇ Fill a measure glass with 25mL PBS, or the quantity necessary to submerge the sensors properly, calculated by equation II
- ∇ Insert the sensors, be aware, if the connectors are soaked, the measurements will not be accurate
- ∇ Turn the magnetic mixer on
- ∇ Start the software
- ∇ After two minutes, add $5\mu L\ H_2O_2$
- ∇ After four minutes, add $5\mu L\ H_2O_2$
- ∇ After six minutes, add $5\mu L\ H_2O_2$
- ∇ After eight minutes, add $5\mu L\ H_2O_2$
- ∇ After ten minutes, add $20\mu L\ H_2O_2$
- ∇ Wait for two minutes before terminating the test

Test protocol 1.

Time (s)	0	120	240	360	480	600
Volume added (µL)	0	5	5	5	5	20
Concentration (mM)	0	65	130	195	260	320

7.7.4 Temperature

Equipment:

- ∇ Sensetion PCB ZT2015 or newer verisons
- ∇ USB
- ∇ Laptop
- ∇ ZP GUI
- ∇ Temperature Cabinet (Weiss Umwelttechnic GmbH from Weiss Technik)

Procedure:

This procedure is meant for the first version ZT2015 with the given heat cabinet. For other types of heat cabinets, or newer versions than the ZT2015, there may be different configurations needed. Please check your equipment thoroughly before performing tests.

For this particular test, the temperature cabinet was programmed to run the ZP test (Program 11).

- ∇ Place the PCB in the temperature cabinet, wired to the outside through the cable gates, and connect to the laptop
- ∇ Start the GUI
- ∇ Set the closet to run program 11 – ZP test

7.7.5 PCB with Mixer

Equipment:

- 1% Hydrogen Peroxide (H_2O_2) made up from
  - 9.7mL PBS
  - 330µL H_2O_2
- PBS, made up from:
  - $7.49g\ Di-Sodium\ Hydrogen\ Phosphate\ anhydrous\ (Na_2HPO_4)$
  - $1.79g\ Sodium\ dihydrogen\ phosphate\ (NaH_2PO_4)$
  - $0.29g\ Sodium\ Chloride\ (NaCl)$
- Sensetion PCB ZT2015 or newer versions
- USB
- Laptop
- ZP GUI

Procedure:

In order to get as accurate measurements, and little interference as possible, the two sensors were tested separate, i.e. first sensor one was tested on channel 7, then sensor two were tested on channel 2.

- Connect the sensor connector to the sensor input. There are three input holes, WE, RE and CE, make sure to connect in correct order
- Insert sensor in connector Make sure to insert correct.
- Connect PCB to Laptop via USB
- Fill a small measure glass with 10mL PBS, and place it on top of the mixer, it is essential that sensor measure area is properly covered when inserted.
- Insert sensor, be aware, if the connector is soaked, the measurements will not be accurate
- Turn on the magnetic mixer
- Start the software
- After two minutes, add $2\mu L\ H_2O_2$
- After four minutes, add $2\mu L\ H_2O_2$
- After six minutes, add $2\mu L\ H_2O_2$
- After eight minutes, add $2\mu L\ H_2O_2$
- After ten minutes, add $8\mu L\ H_2O_2$
- Wait for two minutes before terminating the test

Test protocol 2.

Time (s)	0	120	240	360	480	600
Volume added (µL)	0	2	2	2	2	8
Concentration (mM)	0	65	130	195	260	320

7.7.6 PCB with MFC

Equipment:

- 1% Hydrogen Peroxide (H2O2) made up from
  - 9.7mL PBS
  - 330µL H_2O_2
- PBS, made up from:
  - $7.49g\ Di-Sodium\ Hydrogen\ Phosphate\ anhydrous\ (Na_2HPO_4)$
  - $1.79g\ Sodium\ dihydrogen\ phosphate\ (NaH_2PO_4)$
  - $0.29g\ Sodium\ Chloride\ (NaCl)$
- Sensetion PCB ZT2015 or newer versions
- MFC
- Six measuring glasses
- Laptop
- ZP GUI
- USB
- Sensor one and sensor two
- connector

Procedure:

To obtain results that were comparable wit the results from PCB with mixer tests, the procedure was kept as similar as possible, i.e. sensor one was tested on channel 7, then sensor two were tested on channel 2. In order to avoid any cases where air pockets get stuck in the measuring chamber of the MFC, one has to make sure that the chamber is completely emptied before new concentration enters. This is done by either allowing the pump to draw air for approximately two seconds, or turning off the pump while changing the concentration.

- Connect the sensor connector to the sensor input. There are three inlets, WE, RE, and CE, make sure to connect the wires to their respective inlets
- Insert sensor in connector. Make sure to insert correct
- Mount the sensor and the connector to the MFC
- Connect PCB to Laptop via USB
- Fill 6 small measure glasses with PBS, it is essential that the sensors measure area is properly covered when inserted into the fluid.
- The same principle as test protocol 2 applies, however, because of the working principle of the MFC, one cannot use one glass for all concentrations. Therefore the following mixtures should be used in the measuring glasses:
  - Glass one: 10mL PBS
  - Glass two: 10mL PBS + 2µL 1% H_2O_2
  - Glass three: 10mL PBS + 4µL 1% H_2O_2
  - Glass four: 10mL PBS + 6 µL 1% H_2O_2
  - Glass five: 10mL PBS + 8 µL 1% H_2O_2
  - Glass six: 10mL PBS + 16 µL 1% H_2O_2
- Set glass one on the magnetic mixer
- Start the microfluidic pump
- Insert the hose that runs from the MFC via the pump, into the glass
- After 55 seconds, start the software. NB! This applies for this specific test, there might be some variations in time. One minute is the time it takes from one insert the hose in the glass, til it reaches the senor.

- ∇ After two minutes, take out the hose, wait for two seconds, then insert the hose into glass two, also put glass two on top of the magnetic mixer. Be aware: It is of most importance that the sensor chamber have time to remove all fluids before adding new one, for this specific setup, this takes about one second
- ∇ After four minutes, repeat the changing process, but with glass three
- ∇ After six minutes, repeat the changing process, but with glass four
- ∇ After eight minutes, repeat the changing process, but with glass five
- ∇ After four ten, repeat the changing process, but with glass six
- ∇ Wait three minutes before terminating the test

7.7.7 versaSTAT with magnetic mixer

Equipment:

- 1% Hydrogen Peroxide (H2O2) made up from
  - 9.7mL PBS
  - 330µL H_2O_2
- PBS, made up from:
  - $7.49g\ Di-Sodium\ Hydrogen\ Phosphate\ anhydrous\ (Na_2HPO_4)$
  - $1.79g\ Sodium\ dihydrogen\ phosphate\ (NaH_2PO_4)$
  - $0.29g\ Sodium\ Chloride\ (NaCl)$
- VersaSTAT
- Versa studio
- Sensor one and two
- connector
- One measuring glass
- Magnetic mixer

Procedure;

- Connect the sensor connector to the versaSTAT input. There are three connections, WE, RE and CE, make sure to connect in correct order
- Insert sensor in connector Make sure to insert correct.
- Fill a small measure glass with 10mL PBS, and place it on top of the mixer, it is essential that sensor measure area is properly covered when inserted.
- Insert sensor, be aware, if the connector is soaked, the measurements will not be accurate
- Turn on the magnetic mixer
- Start the versa studio software
- Run the program amperometri, or a program with offset at 0.6V, and runs for 720seconds
- After two minutes, add $2\mu L\ H_2O_2$
- After four minutes, add $2\mu L\ H_2O_2$
- After six minutes, add $2\mu L\ H_2O_2$
- After eight minutes, add $2\mu L\ H_2O_2$
- After ten minutes, add $8\mu L\ H_2O_2$
- Wait until program terminates, or wait for two minutes before terminating the test

7.7.8 versaSTAT with MFC

Equipment:

- 1% Hydrogen Peroxide (H2O2) made up from
  - 9.7mL PBS
  - 330µL H_2O_2
- PBS, made up from:
  - $7.49g\ Di-Sodium\ Hydrogen\ Phosphate\ anhydrous\ (Na_2HPO_4)$
  - $1.79g\ Sodium\ dihydrogen\ phosphate\ (NaH_2PO_4)$
  - $0.29g\ Sodium\ Chloride\ (NaCl)$
- VersaSTAT
- Versa studio
- Sensor one and two
- Six measuring glasses
- connector
- Six measuring glass
- Magnetic mixer
- MFC

Procedure:

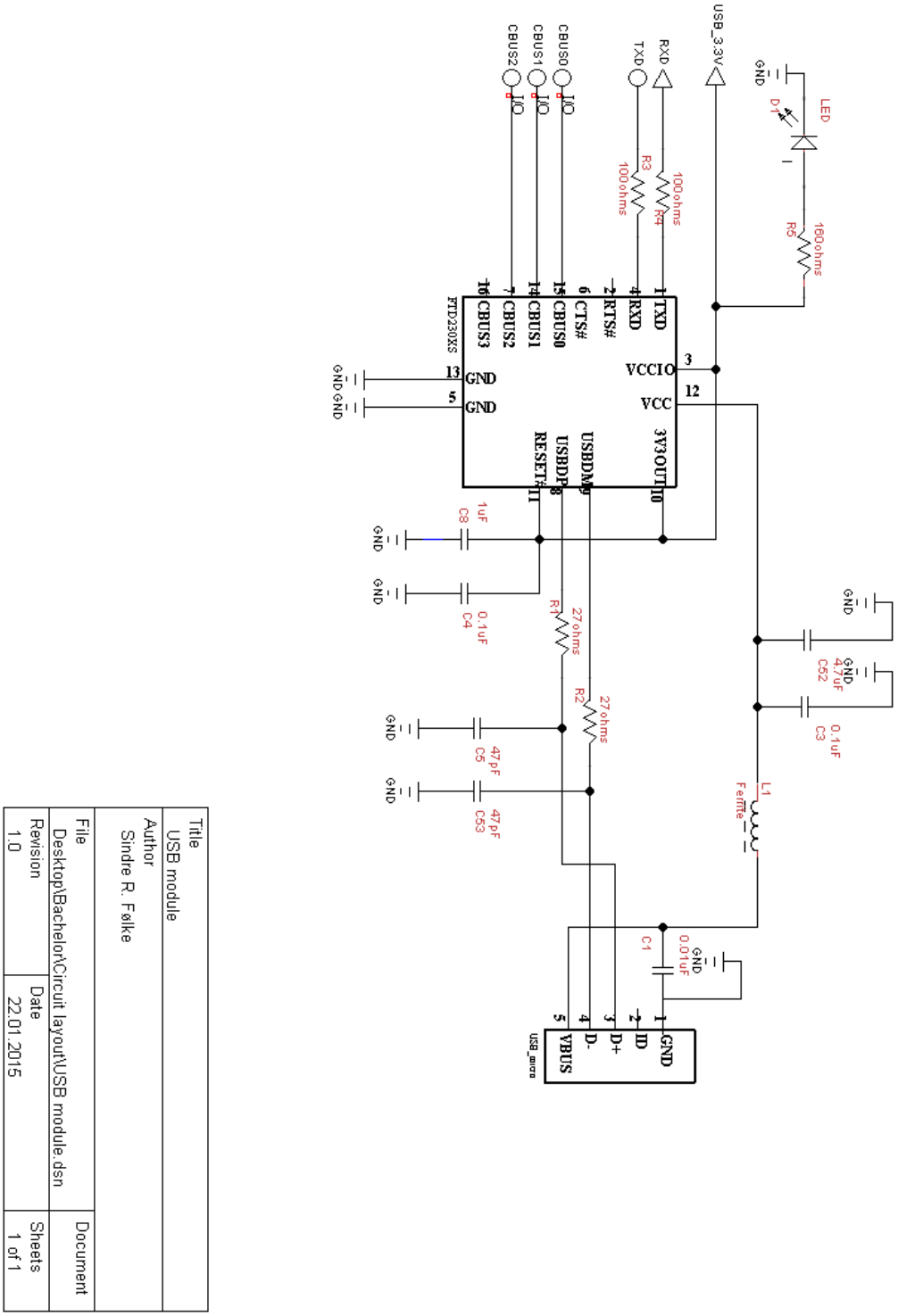
- Connect the sensor connector to the versaSTAT input. There are three inlets, WE, RE, and CE, make sure to connect the wires to their respective inlets
- Insert sensor in connector. Make sure to insert correct
- Mount the sensor and the connector to the MFC
- Fill 6 small measure glasses with PBS, it is essential that the sensors measure area is properly covered when inserted into the fluid.
- The same principle as test protocol 2 applies, however, because of the working principle of the MFC, one cannot use one glass for all concentrations. Therefore the following mixtures should be used in the measuring glasses:
  - Glass one: 10mL PBS
  - Glass two: 10mL PBS + 2µL 1% H_2O_2
  - Glass three: 10mL PBS + 4µL 1% H_2O_2
  - Glass four: 10mL PBS + 6 µL 1% H_2O_2
  - Glass five: 10mL PBS + 8 µL 1% H_2O_2
  - Glass six: 10mL PBS + 16 µL 1% H_2O_2
- Set glass one on the magnetic mixer
- Start the microfluidic pump
- Insert the hose that runs from the MFC via the pump, into the glass
- After 55 seconds, start the software. NB! This applies for this specific test, there might be some variations in time. One minute is the time it takes from one insert the hose in the glass until it reaches the senor.
- After two minutes, take out the hose, wait for two seconds, then insert the hose into glass two, also put glass two on top of the magnetic mixer. Be aware: It is of most importance that the sensor chamber have time to remove all fluids before adding new one, for this specific setup, this takes about one second
- After four minutes, repeat the changing process, but with glass three
- After six minutes, repeat the changing process, but with glass four
- After eight minutes, repeat the changing process, but with glass five

∇ After four ten, repeat the changing process, but with glass six
∇ Wait three minutes before terminating the test

7.8 Appendix Circuit Layout

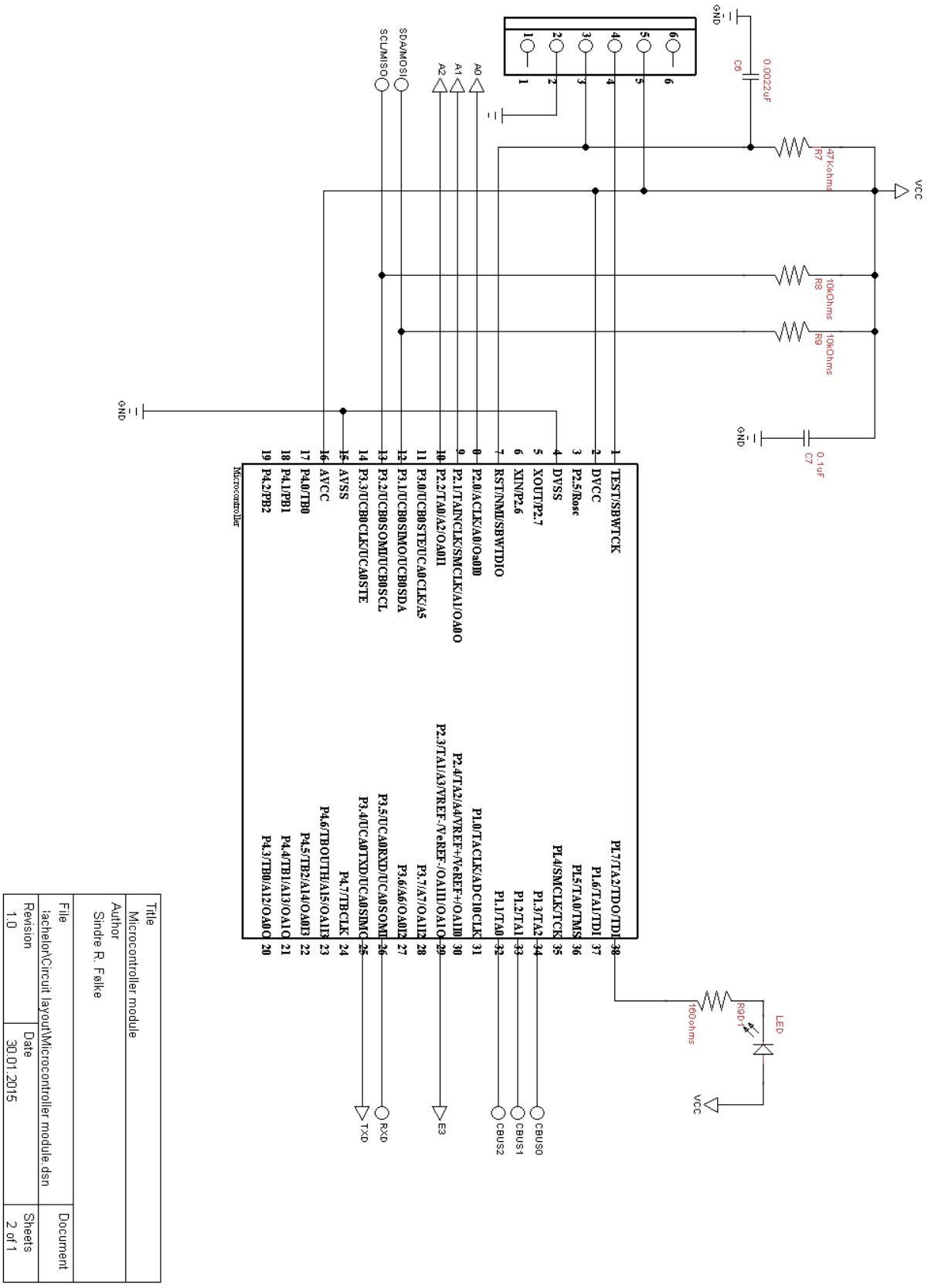
All circuit diagram layouts, except for the demultiplexer in 7.8.3 are based on the MAS_V2_R2 circuit layout designs, which can be found on the project CD

7.8.1 USB Module



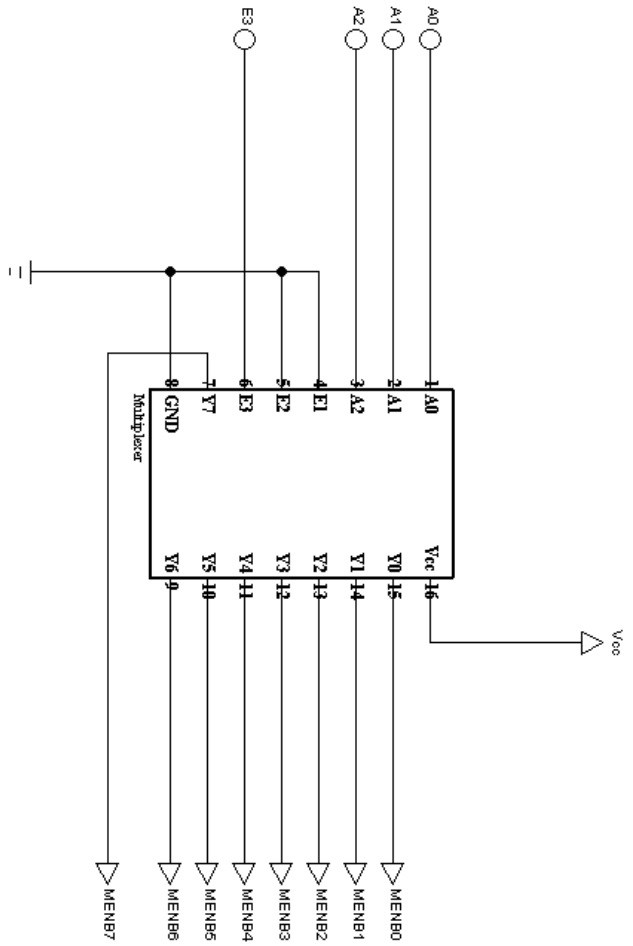
Title	USB module
Author	Sindre R. Følke
File	Desktop\Bachelor\Circuit layout\USB module.dsn
Revision	1.0
Date	22.01.2015
Document	
Sheets	1 of 1

7.8.2 Microcontroller Module



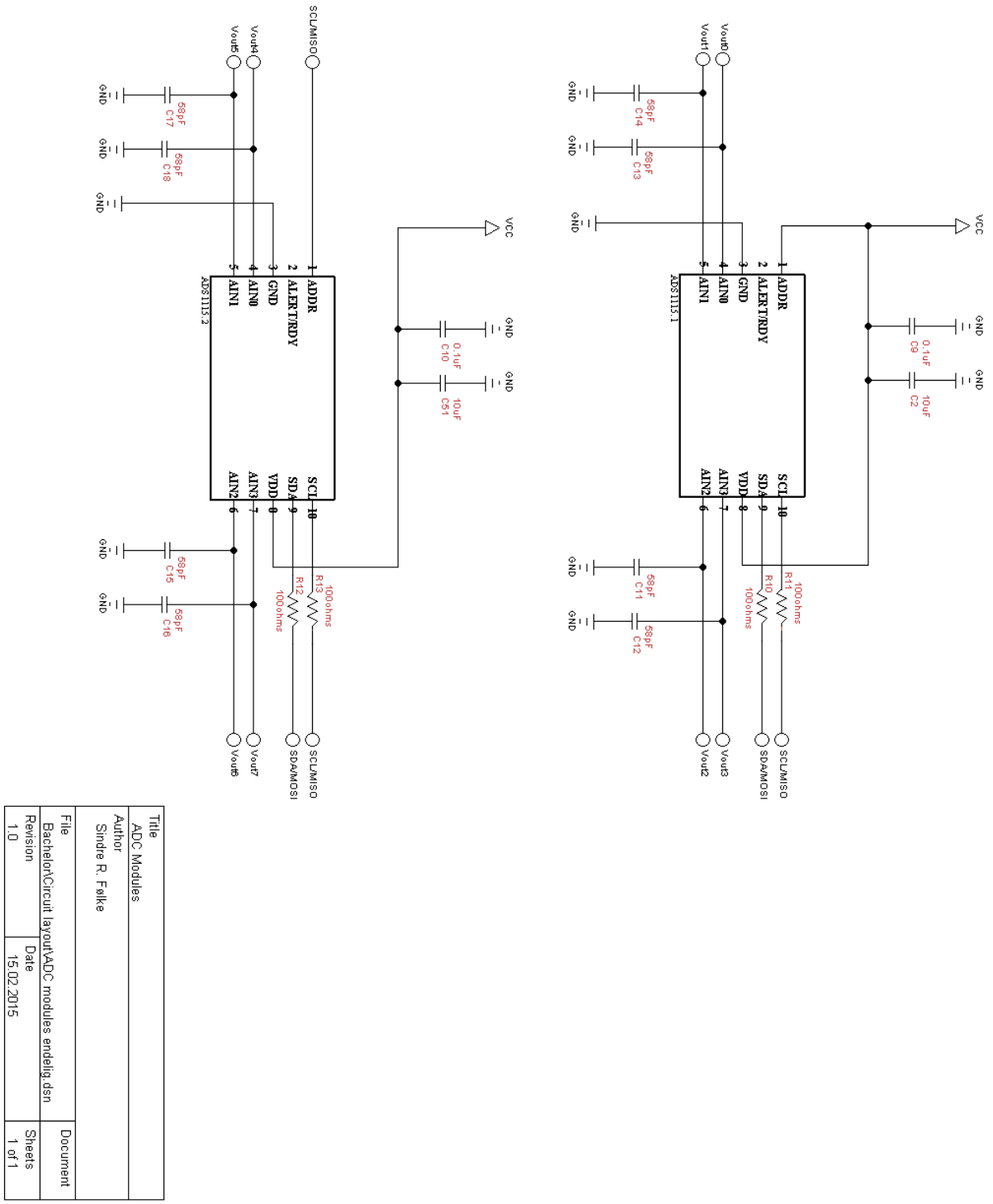
Title	Microcontroller module
Author	Sindre R. Følke
File	Iachelor\Circuit layout\Microcontroller module.dsn
Revision	1.0
Date	30.01.2015
Document	
Sheets	2 of 1

7.8.3 Demultiplexer Module

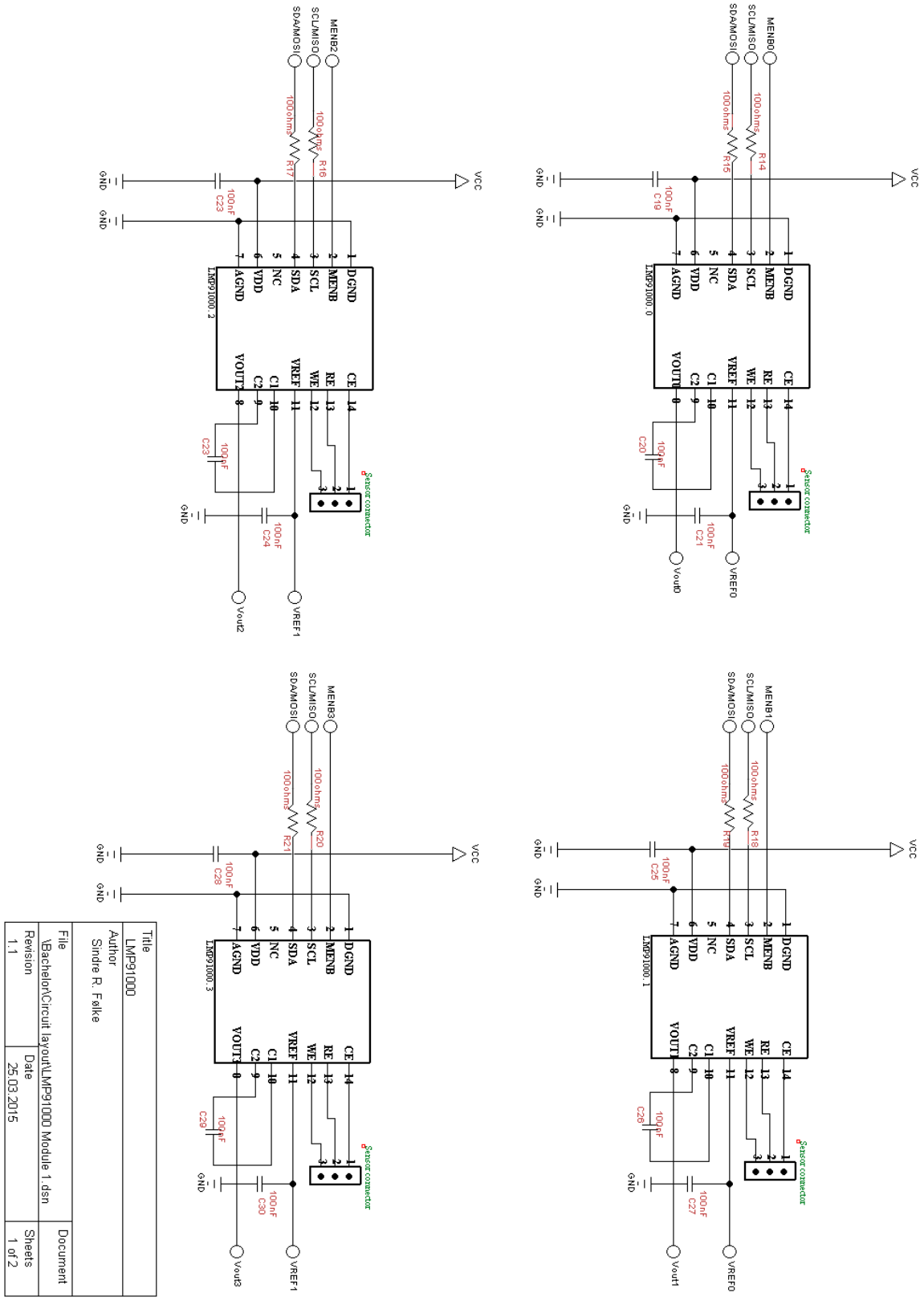


Title	Multiplekser ting
Author	Sindre R. Følke
File	sktop\Bachelor\Circuit layout\multiplekserting.dsn
Revision	1.0
Date	15.03.2015
Document	
Sheets	1 of 1

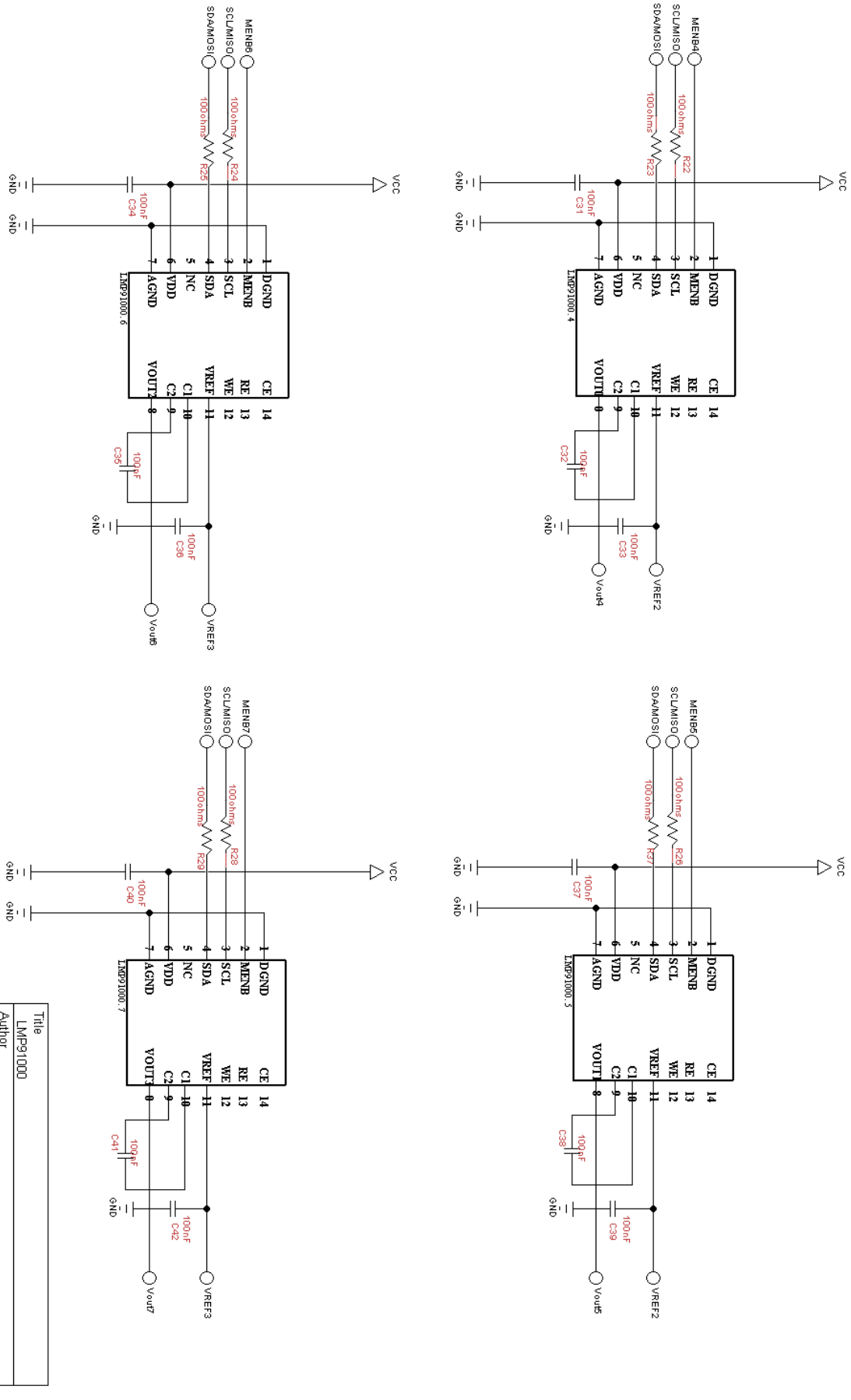
7.8.4 ADC Modules



7.8.5 Potentiostat Modules



Title	LMP91000
Author	Sindre R. Følke
File	\Bachelor\Circuit layout\LMP91000 Module 1.dsn
Revision	1.1
Date	25.03.2015
Document	
Sheets	1 of 2



Title	LMP91000
Author	Sindre R. Følke
File	\Bachelort\Circuit layout\LMP91000 Module 2.dsn
Revision	1.1
Date	25.03.2015
Document	
Sheets	2 of 2

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7.9 Laboratory Reports

Test 1

Test laboratory exercise, February 26, 2015

Negative photoresist SU-8 100

Procedure. We followed this procedure: http://www.microchem.com/pdf/SU8_50-100.pdf

First, all the machines have to be turned on in time. VERY IMPORTANT! REMEMBER THE PRESSURE VALVE IN THE CORRIDORE if you are going to use the new mask aligner! Always clean all the surroundings. USE PROTECTION GLASES!

Materials, chemicals and equipment:

- The glass mask designed with L-edit
- Isopropanol (IPA)
- Acetone
- Silicon wafer, 4”
- SU-8 100 negative photoresist
- Developer for SU-8 100
- Fume hood 4 for general solvents
- Fume hood 5 for corrosive solvents
- Spinner 1 Semitool 1
- Mask Aligner Karl Suss MA56
- DEKTAK profilometer
- Optical microscope I Leika DM4000M
- Hot plate

Procedure:

1. Cleaning wafer.
   1.1. First, try to clean with Ni
   1.2. Clean with acetone and rinse with isopropyl (IPA)
   1.3. After drying with Ni, bake on 200ºC during 10 minutes. Cool down.
2. Coating the photoresist. We aimed for a layer of 100µm.
   2.1. Coat bowl and shield with aluminum foil to protect them from the very sticky SU-8.
   2.2. Set up the timer the following way: This can be adjusted somehow. The instructions are on the side of the spin coater.

TABLE XXIV Spin coating parameters

Step	Speed (rpm)	Accelerate (sec)	Time (sec)	Decelerate (sec)
1	0	0	0	-
2	500	5 (500/5=100)	10	-

3	3000	8 (2500/8=312.5)	30	30

2.3. Try to place the wafer as centered as possible.
2.4. Pour the SU-8 carefully in the center. It is very viscous, so when it gets to the entrance of the bottle, slow the fluid by inclining the bottle. Try to control the flow by turning the bottle. Stop when you have a diameter of approximately 4cm.
2.5. Clean the bottleneck with IPA and place in the box.
2.6. Cover with the lid and start the program.
2.7. When the spinning cycle is finished, use a needle to gently remove the bubbles.

3. Soft bake. To eliminate residual solvent. Use alumina foil on the hot plate to avoid the sticky SU-8 and add 10°C to compensate.

TABLE XXV Soft bake parameters

Temperature (°C)	Time (min)
75	10
105	30

4. For the pattern transfer, use the designed glass mask. For the SU-8 you can use the mask aligner, Karl Suss. We measured the light intensity to be approximately 8.5mW/cm^2. Center = 8.5, Top = 6, Bottom = 12, Right = 8.5 and Left = 9. The table in the instruction manual showed that the exposure energy should be approximately 600 mJ/cm^2, if we aimed at the highest energy recommended. 600 : 8.5 =70.5 sec. (J = kgm^2/s^2 and W = kgm^2/s^3)

TABLE XXVI Exposure parameters

Step	Dose (mJ/cm^2)	Light intensity (mW/ cm^2)	Time (s)
Exposure	600	8.5	70

5. Post exposure bake. Still use the alumina foil and augment with 10 °C

TABLE XXVII Post exposure bake parameters

Temperature (°C)	Time (min)
75	1
105	10

6. Turn off the hot plate and leave the wafer until it reach approx. 75 °C
7. Immerse the wafer in the developer. This took a long time, more than 15 minutes, and we still did not get a clean wafer. In parts of the wafer, the exposed resist fell off or was almost totally erased. Developing is finished when no whitish residue is observed when rinsing with IPA
8. Rinse the wafer with IPA one last time and blow dry with Ni.
9. We did not examine under microscope or measure anything, since there really was nothing to measure.
10. Clean everything and put the chemicals away. Throw the used chemicals in waste bottle.

Results and discussion:

2. The SU-8 did not cover the entire wafer. That might have been caused by too little SU8 and/or not managing to place the SU8 in the center of the wafer. The removal of the air bubbles worked well. A possible way to apply the SU-8 with more control is using a syringe. That way it is easier to use exactly the same amount each time, and with better control. To use the exact same amount every time, it might be a possibility to weigh the wafer and apply the SU-8 while on the scale.

3. Since we only had one hot plate, we could not follow the procedures exactly as described. After 10 minute on 75 °C it went gradually up to 105. We also had to wait for quite a time for the plate to cool after prebaking. It might be good to use at least two hot plates next time.

4. We took the highest value on the plot for the exposure, maybe we should choose one more in the center that can also be used for thicker resists. May be around 500-550?

5. The temperature was taken gradually from 75 to 105 °C. The instructions indicates ramping for the best results.

6. We did not leave it at the plate long enough for the temperature to cool down to 75 °C, but took it off almost instantly, letting it cool on the metal plate for transporting the wafer. This was due to the time schedule. There seemed to be some cracks in the exposed structure after a while. The quick cooling might have caused this.

7. The developer was used/old and might have lost some of its properties. There still was residues of the unexposed resist after more than 15 minutes, and almost all the exposed resist had either fallen off (loosened from the wafer) or been erased. In the instruction guide, the wafer is supposed to be ready in 10 minutes for 100µm thickness. There seemed to be some cracks in the exposed structure

8. There really was nothing to measure left on the wafer, and we did not have too much time

Finally a comment: You can hard bake (cure) the SU-8, but it has good mechanical properties and normally it is not required.

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Test 2

Test laboratory exercise, Mars 10, 2015

Negative photoresist SU-8 100

Procedure. We followed this: http://www.microchem.com/pdf/SU-8_50-100.pdf

First, all the machines have to be turned on. VERY IMPORTANT! REMEMBER THE PRESSURE VALVE IN THE CORRIDORE! Always clean all the surroundings. USE PROTECTION GLASES!

Introduction:

The intention with this laboratory exercise is to learn the process of creating a master using SU-8 100 structure on a wafer. The idea is to follow the procedure recommended by MicroChem to create a 100µm thick structure. Can the syringe be used to apply the SU-8 on the wafer for the spin coating? That would give a better control on where to place it and how much.

Materials, chemicals and equipment:

- The glass mask designed with L-edit
- Isopropanol (IPA)
- Acetone
- Silicon wafer, 4”
- SU-8 100 negative photoresist
- Remover for SU-8 100
- Fume hood 4 for general solvents
- Fume hood 5 for corrosive solvents
- Spinner 1 Semitool 1
- Mask Aligner Karl Suss MA56
- DEKTAK profilometer
- Optical microscope I Leika DM4000M
- Hot plate

Procedure:

1. Preparing the wafer
   1.1. Cleaning the wafer with acetone, IPA, SI-water and Ni
   1.2. Bake on 200^{o}C during 10 minutes. Cool down.
2. Spin coating
   2.1. Filled a small beaker with approx. 10 ml of SU-8. small bubbles were created.
   2.2. Filled the syringe with SU-8 from the beaker. Managed to suck up close to 4 ml. Some bubbles on the inside.
   2.3. Used the syringe to apply the SU-8 on the wafer. Many more bubbles was created.

2.4. Set up the timer the following way: This can be adjusted somehow.

TABLE XXVIII Spin coating parameters

Step	Speed (rpm)	Accelerate (sec)	Time (s)	Decelerate (s)
1	0	0	0	-
2	500	5s (500/5=100)	10	-
3	1000	8s	30	30

2.5. After the spinning, I tried to remove all the bubbles. No use! It almost covered the wafer. The SU-8 did not cover completely, something that may indicate that 4 ml is not enough. I think it should be something like 5ml. It is supposed to be one ml per inch of diameter (4" wafer).

Results and discussion:

It is impossible to use a syringe. The creation of bubbles is very difficult to avoid.

It should be more than 4 ml. More like 5ml.

There is no need for a specific thickness for my structure. It is better to pour the SU-8 directly from the bottle to avoid bubbles.

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7.9.1 Exercise 1

Test laboratory exercise, Mars 16, 2015

Negative photoresist SU-8 100

Procedure. We followed this: http://www.microchem.com/pdf/SU-8_50-100.pdf

First, all the machines have to be turned on. VERY IMPORTANT! REMEMBER THE PRESSURE VALVE IN THE CORRIDORE! Always clean all the surroundings. USE PROTECTION GLASES!

Introduction:

The intention with this laboratory exercise is to learn the process of creating a master using SU-8 100 structure on a wafer. The idea is to follow the procedure recommended by MicroChem to create a 250µm thick structure.

Materials, chemicals and equipment:

- The glass mask designed with L-edit
- Isopropanol (IPA)
- Acetone)
- Silicon wafer, 4”
- SU-8 100 negative photoresist
- Remover for SU-8 100
- Fume hood 4 for general solvents
- Fume hood 5 for corrosive solvents
- Spinner 1 Semitool 1
- Mask Aligner Karl Suss MA56
- DEKTAK profilometer Optical microscope I Leika DM4000M
- Hot plate

Procedure:

11. Cleaning wafer. Always finish with Ni.
    11.1. First, try to clean with Ni
    11.2. Clean with acetone and rinse with isopropyl (IPA)
    11.3. After drying with Ni, bake on 200°C during 10 minutes. Cool down.
12. Coating the photoresist. We aimed for a layer of 250µm.
    12.1. Coat bowl and shield with aluminum foil to protect
    12.2. Set up the timer the following way: This can be adjusted somehow. The instructions are on the side of the spin coater.

TABLE XXIX Spin coating parameters

Step	Speed (rpm)	Accelerate (sec)	Time (s)	Decelerate (s)
1	0	0	0	-

2	500	5s (500/5=100)	10	-
3	1000	10s(500/10) = 50	30	30

12.3. Try to place the wafer as centered as possible.

12.4. Pour the SU-8 carefully in the center. It is very viscous, so when it gets to the entrance of the bottle, slow the fluid by inclining the bottle. Try to control the flow by turning the bottle. Try to stop when you have a diameter =4cm approx.

12.5. Clean the bottleneck with IPA and place in the box.

12.6. Cover with the lid and start the program.

12.7. Use needle to gently remove the bubbles.

13. Soft bake. To eliminate residual solvent. Use alumina foil on the hot plate. Add 10ºC to compensate

TABLE XXX Soft bake parameters

Temperature (ºC)	Time (min) Wafer1	Time (min) Wafer2
75	30	30
105	90	90+10

14. Pattern transfer using designed mask for the mask aligner. We measured the light intensity, ≈9mW/cm^2. C=8.5, T=6, B=12, R=8.5 and L=9. The table in the instruction manual showed that the exposure energy should be approx. 600 mJ/cm^2. 600:9=67 sec. (J=kgm^2/s^2 and W=kgm^2/s^3)

TABLE XXXI Exposure parameters

Step	Dose (mJ/cm^2)	Light intensity (mW/ cm^2)	Time (s)
Exposure 250µm	600	8,5	71

15. Post exposure bake. Still use the alumina foil and augment with 10 ºC . Slowly from one temperature to the other, and let cool slowly on the hot plate to avoid cracks in the SU-8.

TABLE XXXII Post exposure bake parameters

Temperature (ºC)	Time (min)
75	1
105	20

16. Turn off the hot plate and leave the wafer until it reach approx. 75 ºC
17. Immerse the wafer in the developer and move it slowly to help remove the SU-8

TABLE XXXIII Development time

Thickness	Developement
250µm	20 min +7 on both

18. Rinse the wafer with IPA one last time, rinse with water and blow dry with Ni.
19. Clean everything and put the chemicals away. Throw used chemicals in waste bottle.

Results and discussion:

2. The application of the resist was done by hand and by eye, trying to cover about 4-5 cmØ in the center of the wafer. Zekija wanted to put the acceleration from 500 to 1000 in 10sec. This gives 50rpm/sec and not 300rpm that was in the paper. At least there was almost no bubbles on either wafer.

3. After I soft baked for 30 minutes on 75 and 90 minutes on 105, the wafer got stuck on the mask. I added 10 more minutes on the second wafer, and it still got stuck, but less. I discussed with Erik and he said that it could bake for a really long time without any problems. He said that he used 50 minutes on the 100µm layer instead of 30. That adds 67%. Next time I will try 90+67%=150 minutes. I used two different hot plates. The one for wafer one was a small plate that might have bad contact with the wafer. Wafer number two used the big plate and had very good contact. When baking, number one was glossy like a mirror all the time. Number two turned like "ice on a car a freezing morning" but evened out at the end. I think may be the plate was a bit too hot in the beginning.

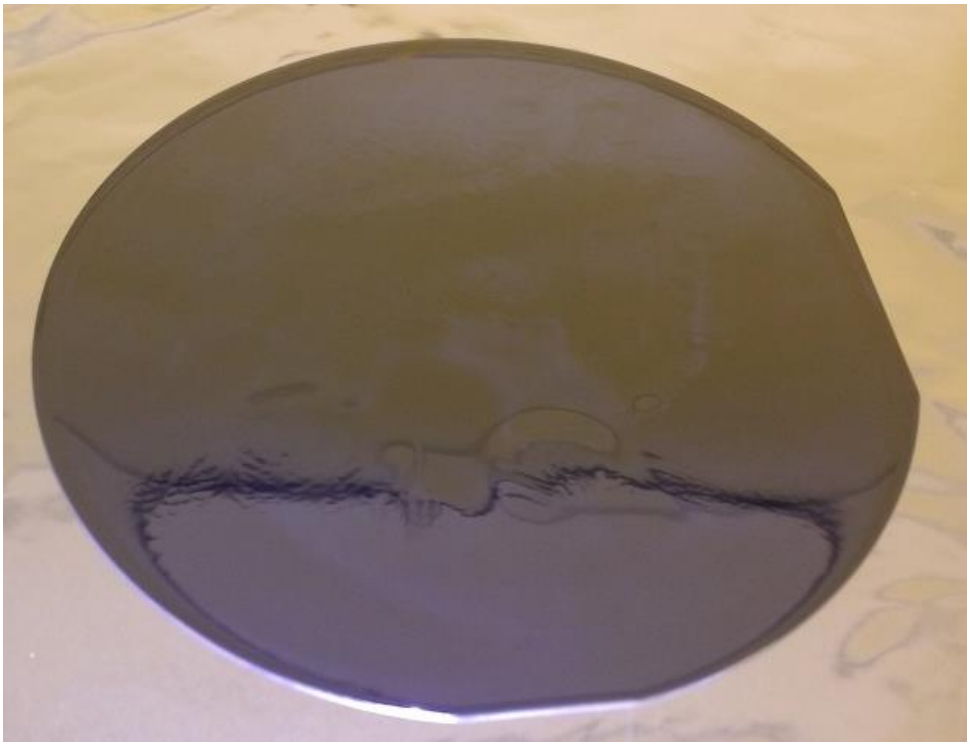


Figure 87 Wafer number two

4. Zekija thought we should take 600 mJ/cm^2 that ended up with 71 seconds. Both wafers got stuck on the mask, but that might have been because of the lack of baking.

6. When the last 20 minutes of the post bake was over I just turned off the plate and waited until it seemed fairly cooled (less than 75 degrees).

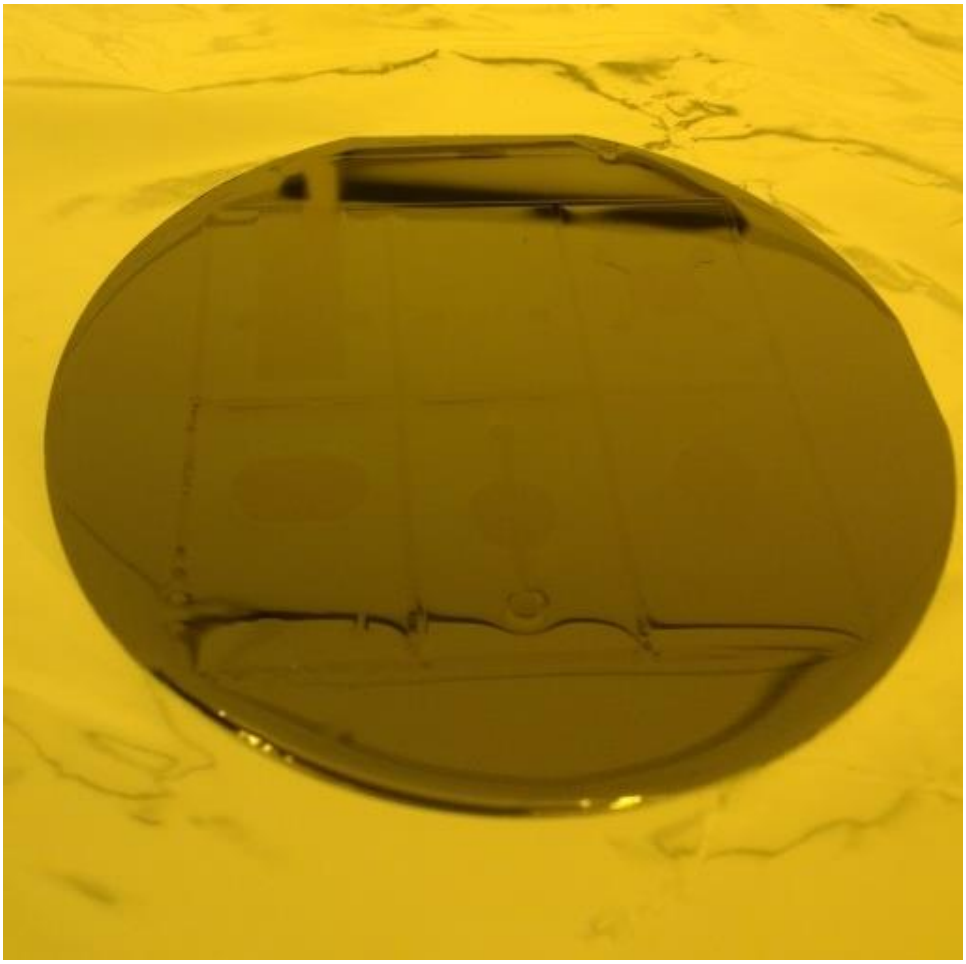


Figure 88 Post exposure baking. The left low corner have the marks where it got stuck on the mask

7. For the developing process, both wafers needed exactly 7 minutes more than the stipulated 20 minutes. After measurements, it was clear that it needed more time to develop, having over 0,4mm thick SU-8 at some places. If the resist is spread properly, it should not be necessary to develop for more than the stipulated time of 20 minutes.

Both the wafers seemed to have useable results, but even with the bare eye it was clear that the SU-8 was thicker on one side of the wafer than the other side. This might be caused by several reasons. The spin coater might not have finished throwing of the excess of the resist, and therefore there might be too much on the border, the SU-8 might not have been centered or the hot plates might not have been leveled.

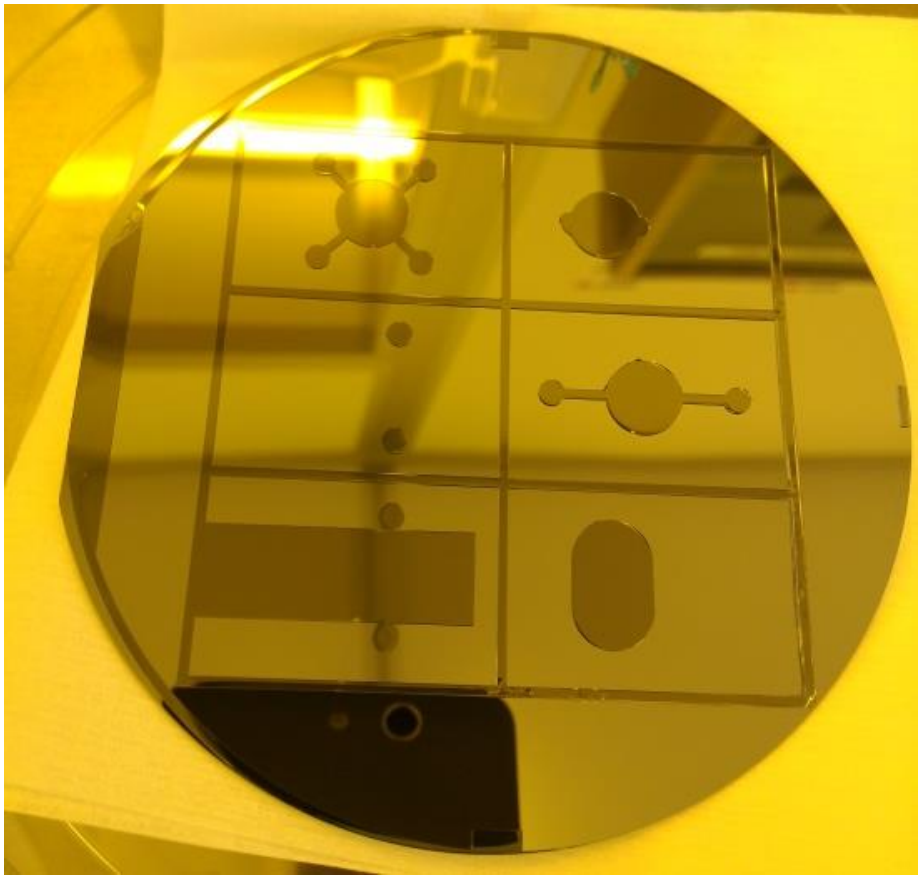


Figure 89 The master

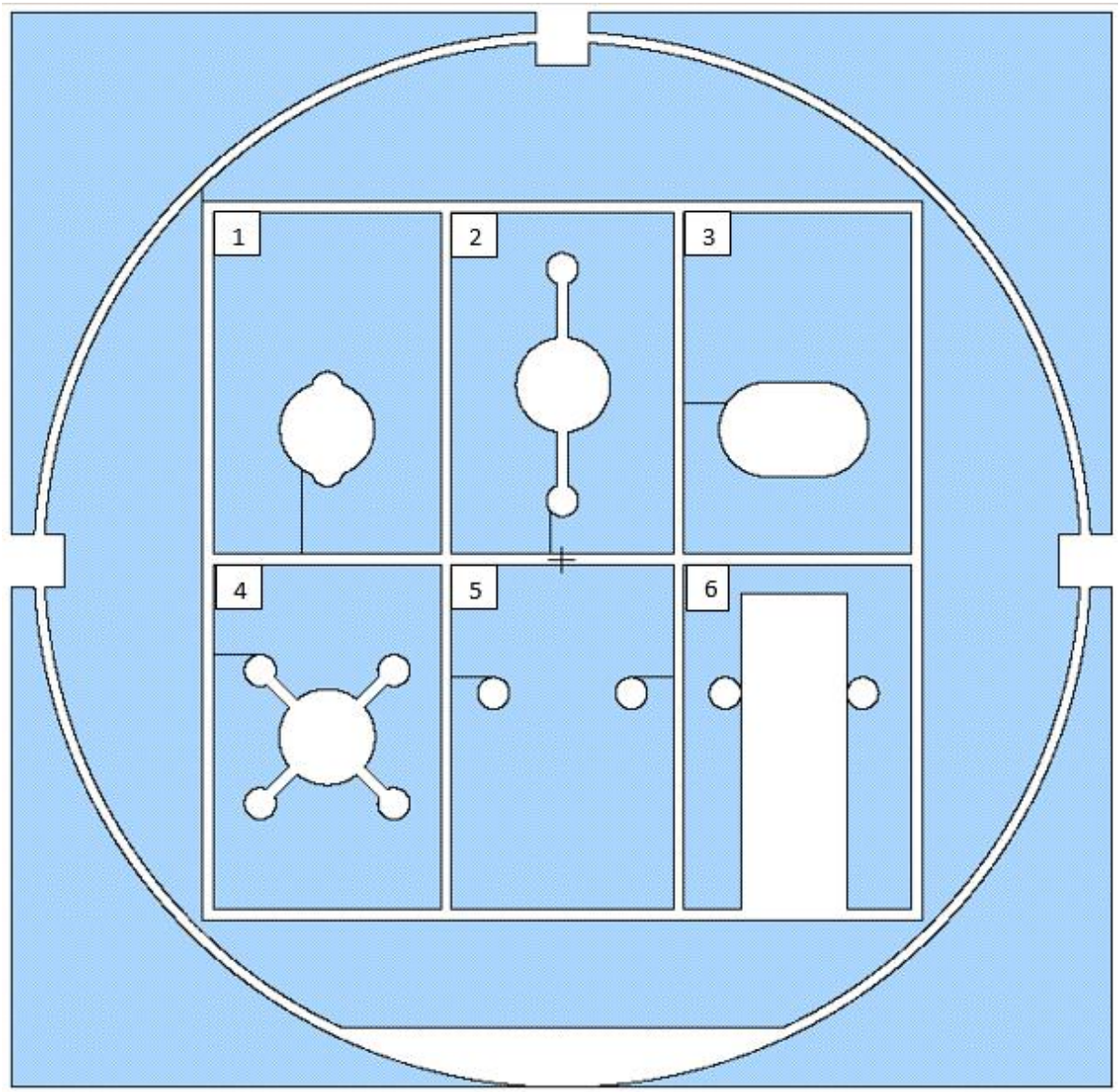


Figure 90 The master with the numbers of the designs.

Wafer one:

- 1 through 5 looked good except signs of stress in some corners.
- Some damage on the sensor support on number 6 under the outlet pad on the right side. This was the damage from getting stuck on the mask.
- Can see that there are thickness differences over the wafer, even with the bare eye

Wafer two:

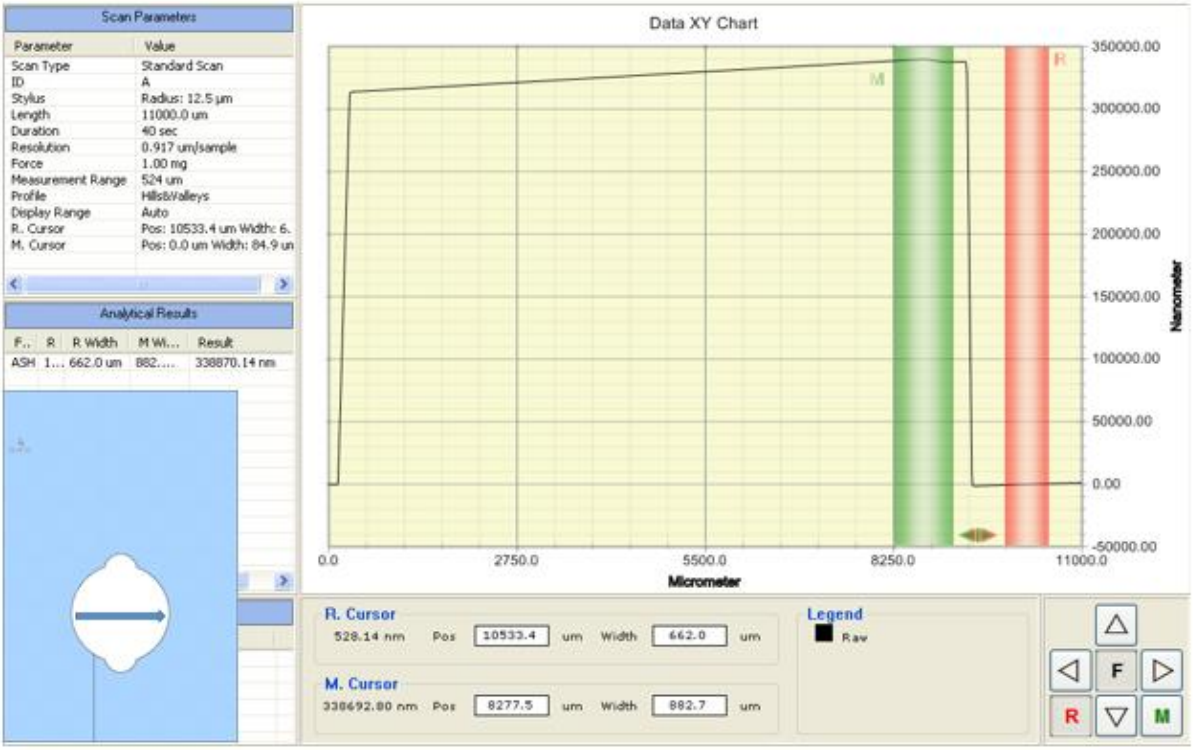
- 1: Small error/spot on the right side. Very small and I suppose it is not important
- 2 and 5 looks good
- 3: Some spots on the right side

- 4: Small spot high on the upper channel on the left and a small spot low on the upper channel at the right
- 6: One spot just under the outlet on the right side on the sensor support. Clearly some cracks between the sensor support and the outlets on both sides.

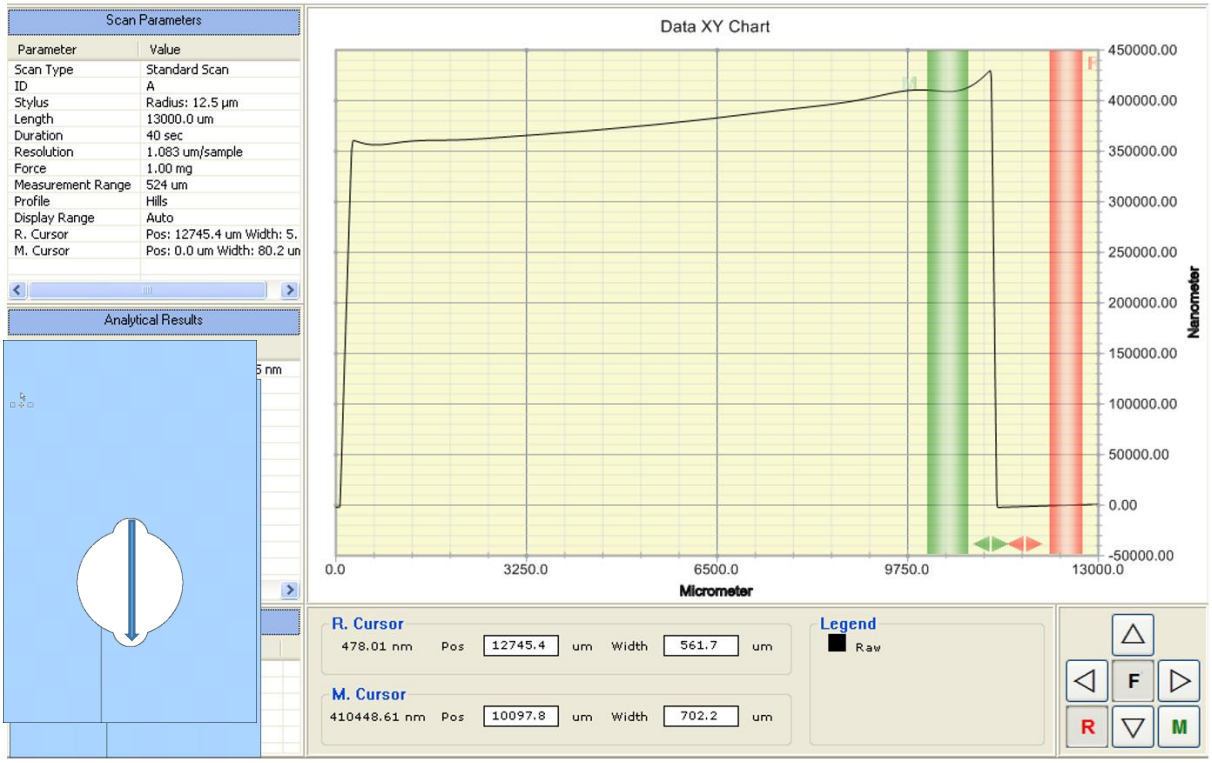
Measurements:

Wafer one:

- Design one:

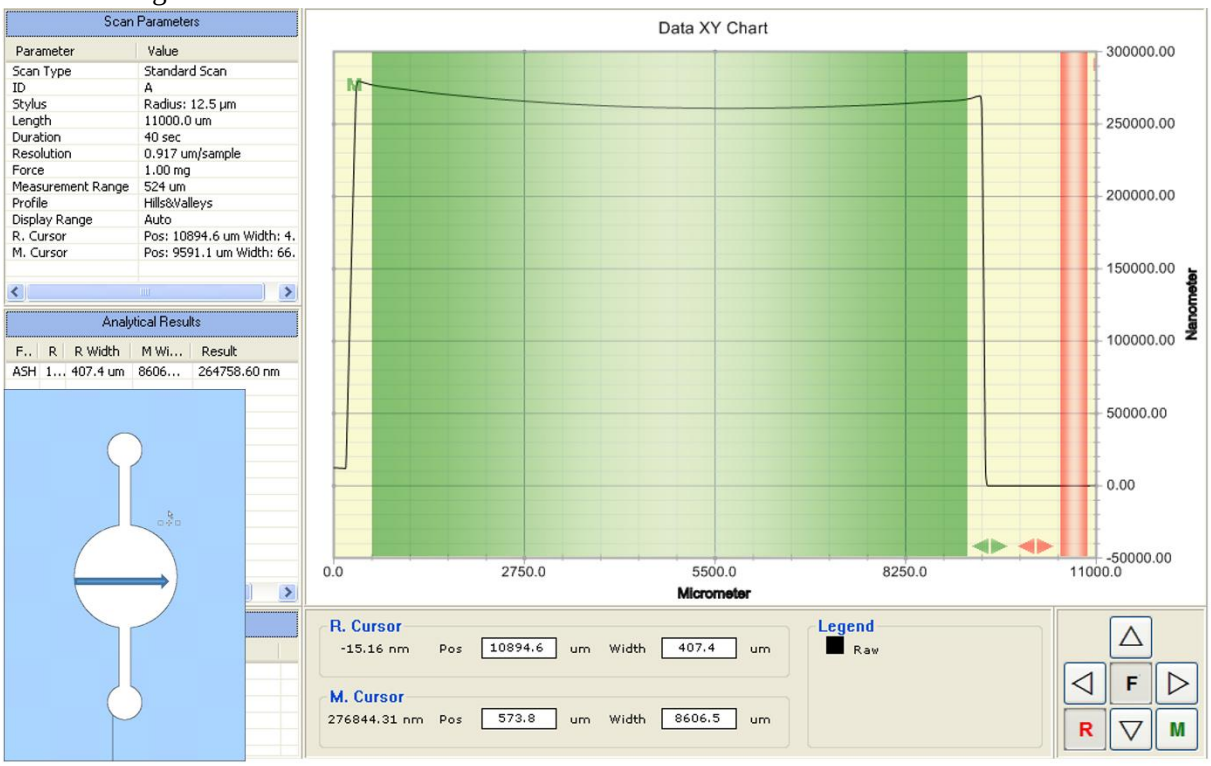


Minimum approximately 315µm and maximum 340µm.

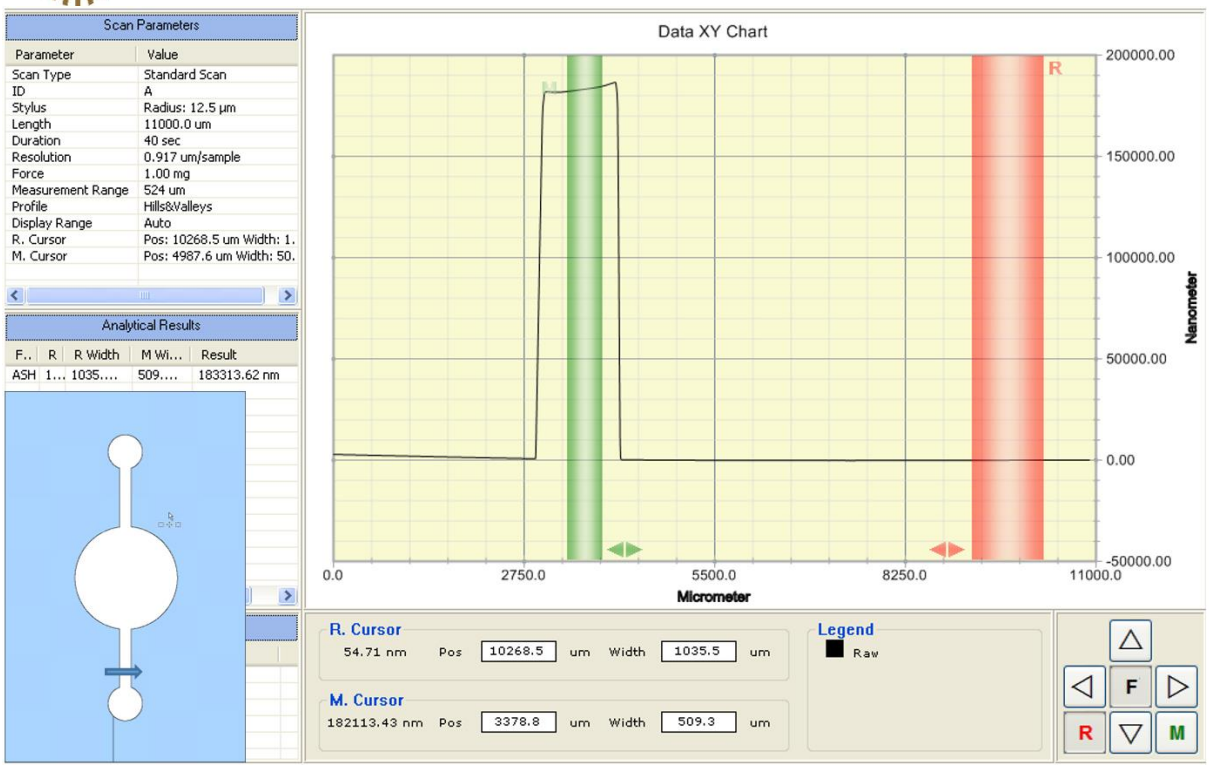


Minimum approximately 360µm and maximum 420µm.

- Design two:

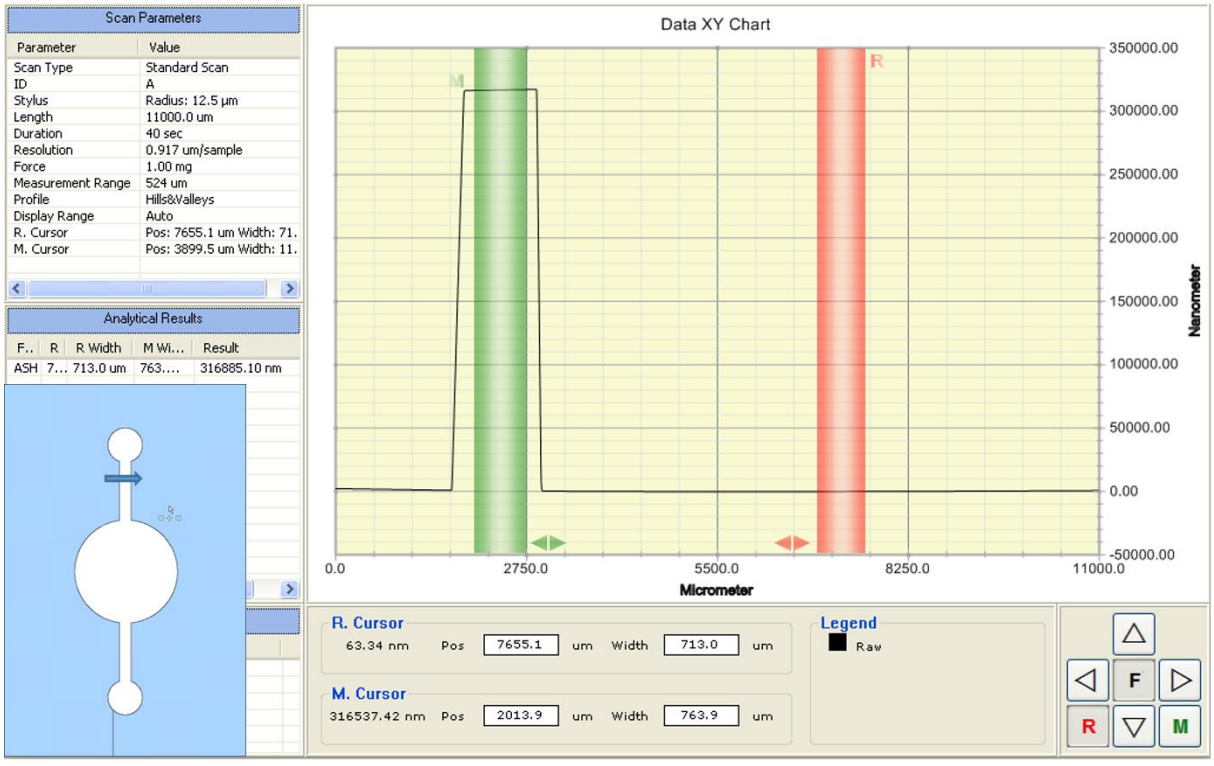


Minimum approximately in the center 260µm and maximum at the left side 280µm



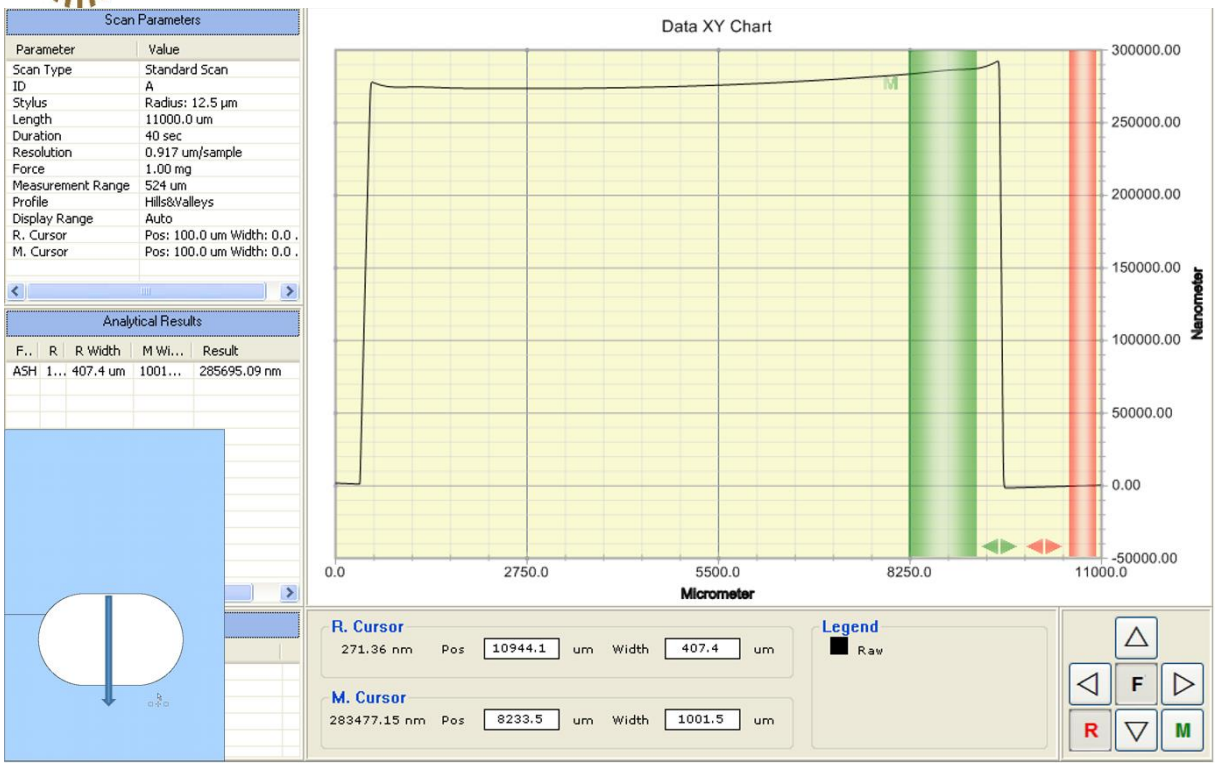
Minimum 180µm and maximum 185µm

Minimum 180µm and maximum 185µm



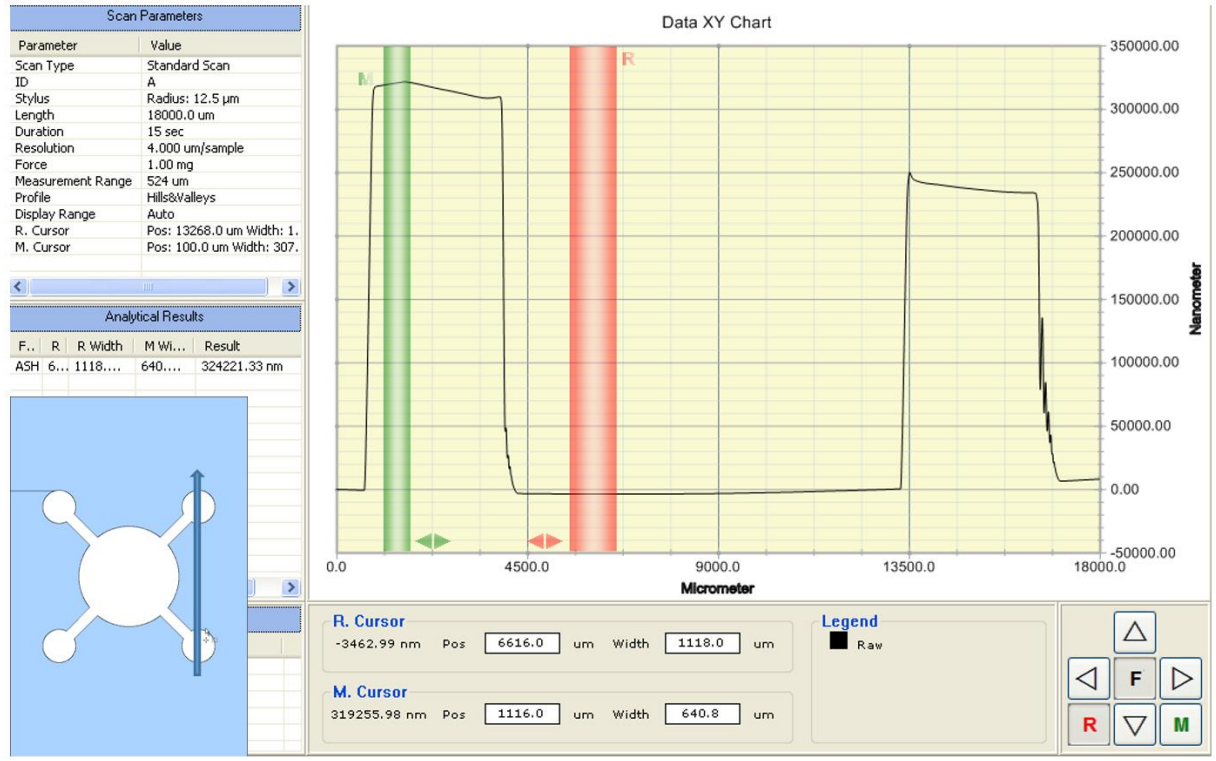
Minimum = maximum = 320µm

- Design three

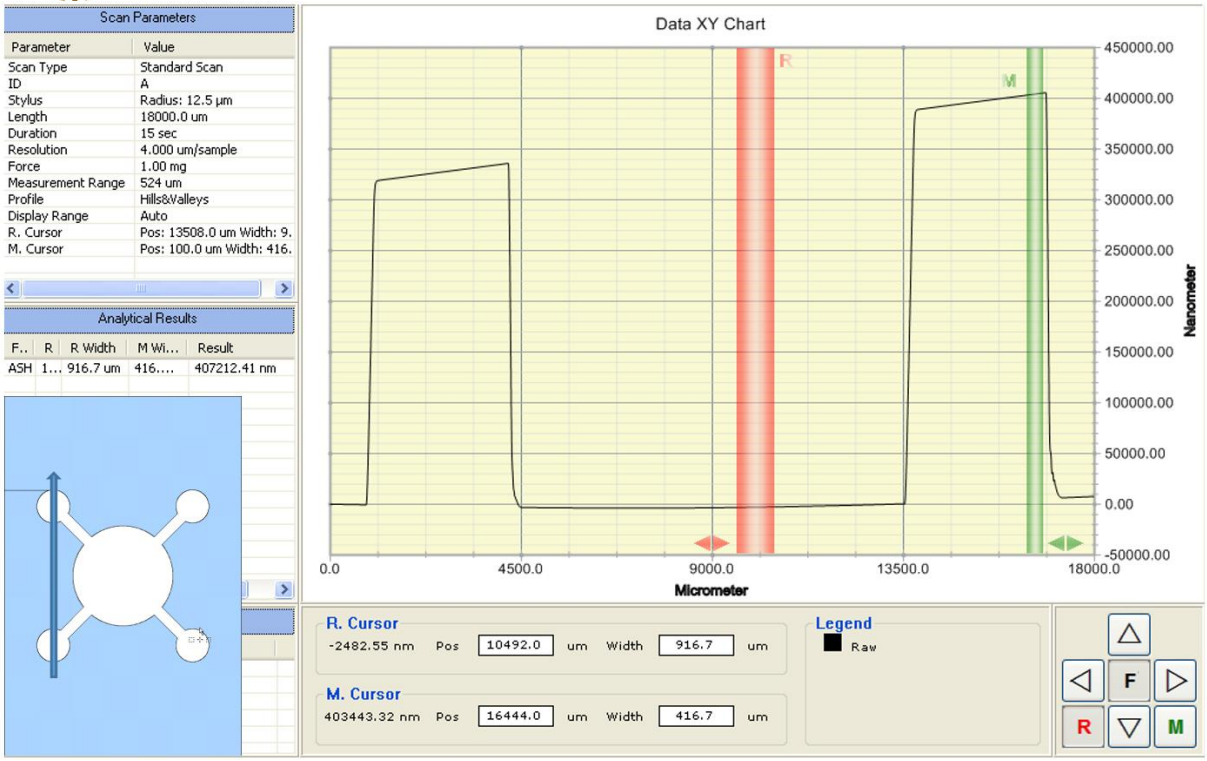


Minimum 270µm and maximum 290µm

- Design four

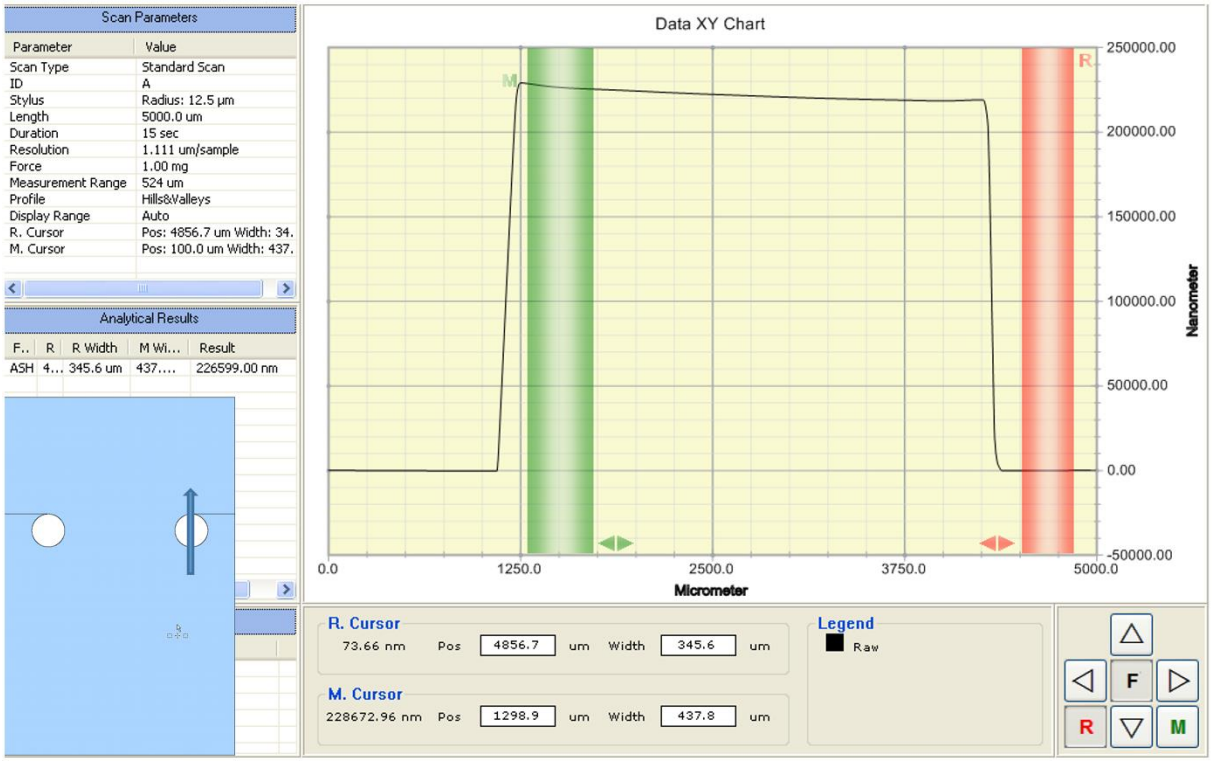


Minimum 240µm and maximum 320µm

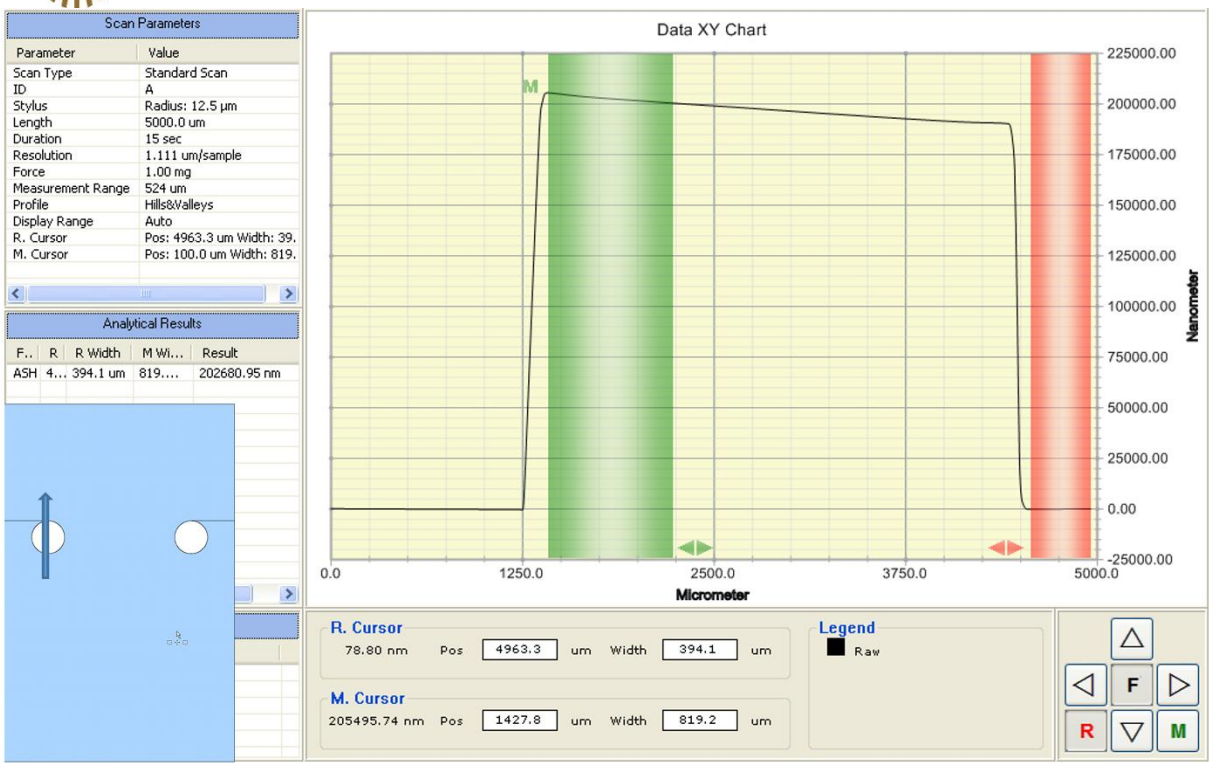


Minimum 320µm and maximum 400µm

- Design five

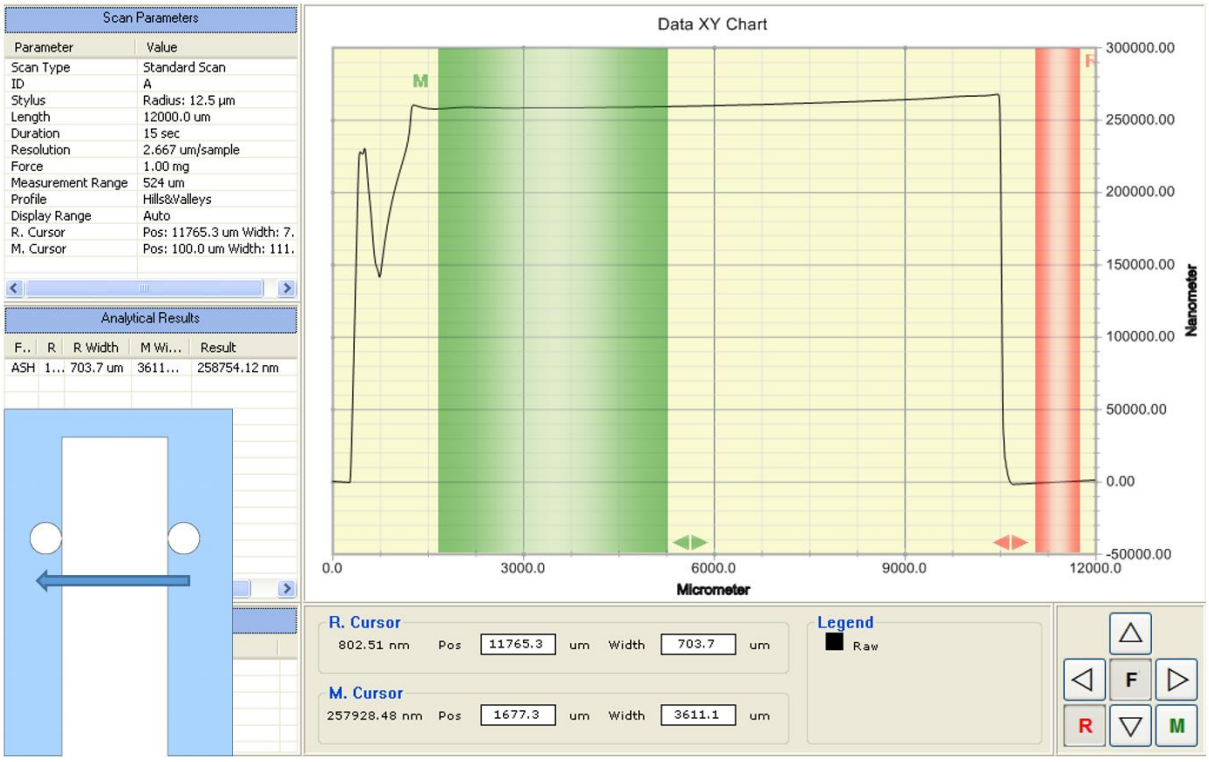


Minimum 220µm and maximum 230µm

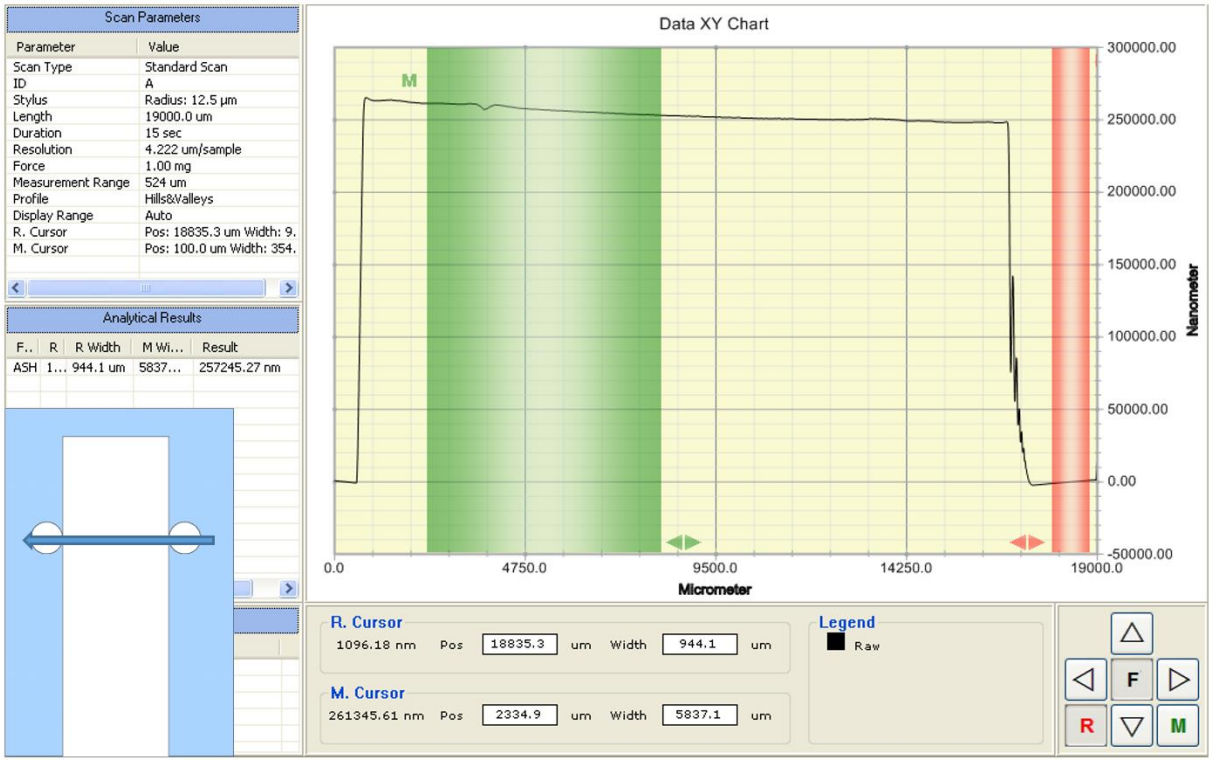


Minimum 190µm and maximum 205µm

- Design six



Clearly see the damage where the resist got stuck to the mask. Minimum 260µm and maximum 270µm.



Minimum 250µm and maximum 265µm

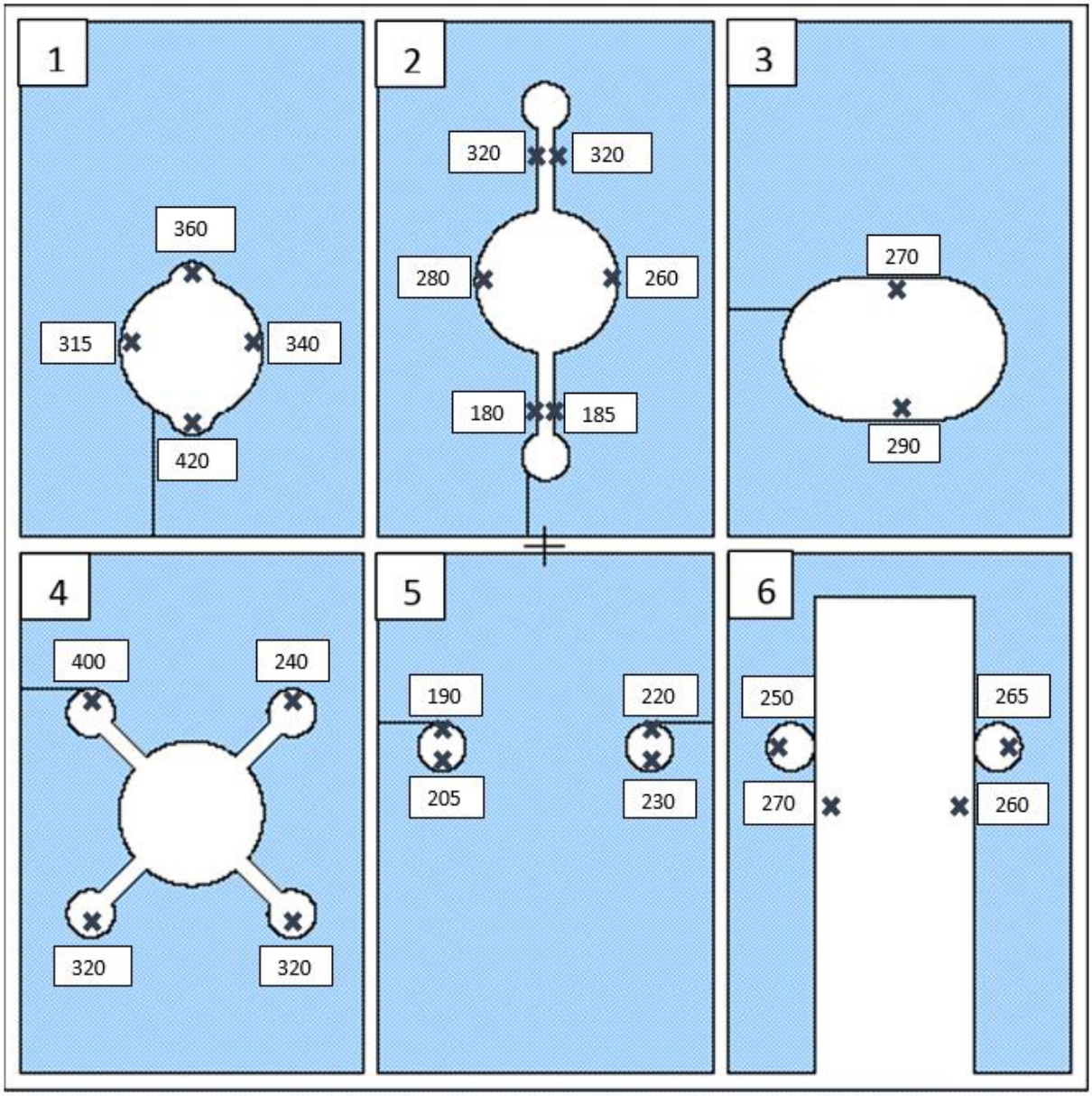
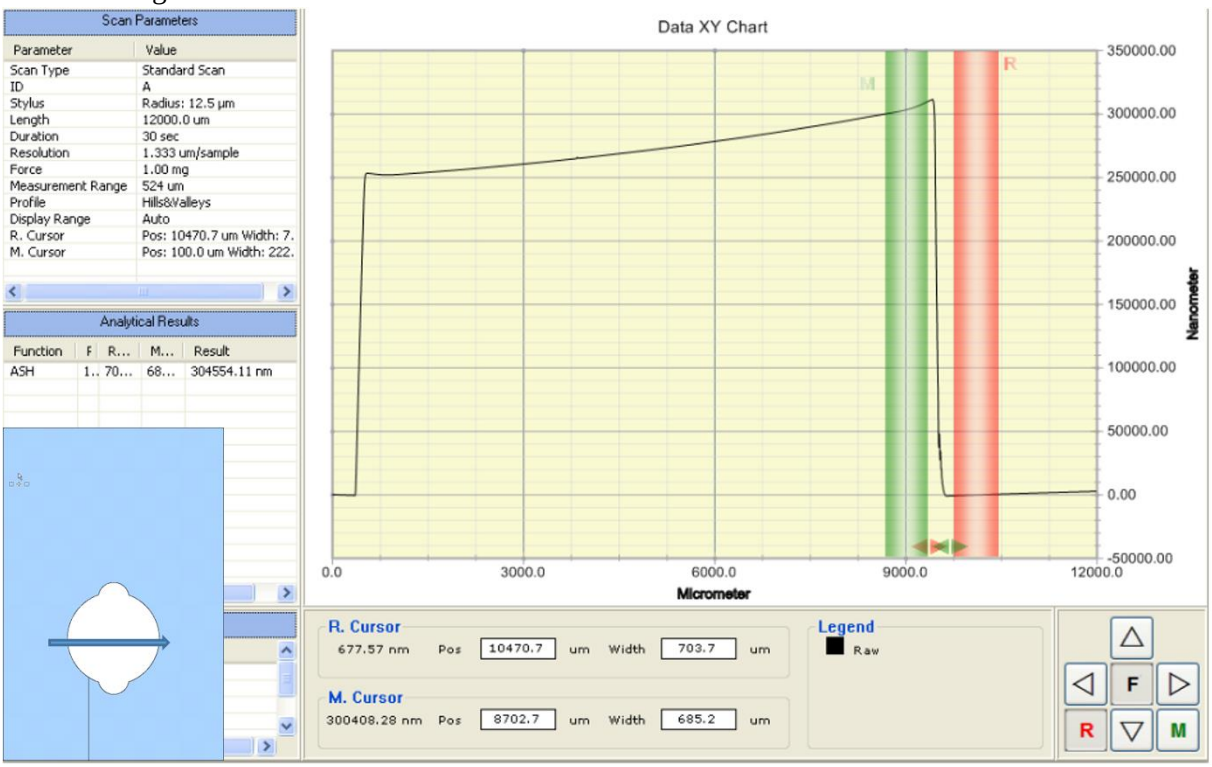


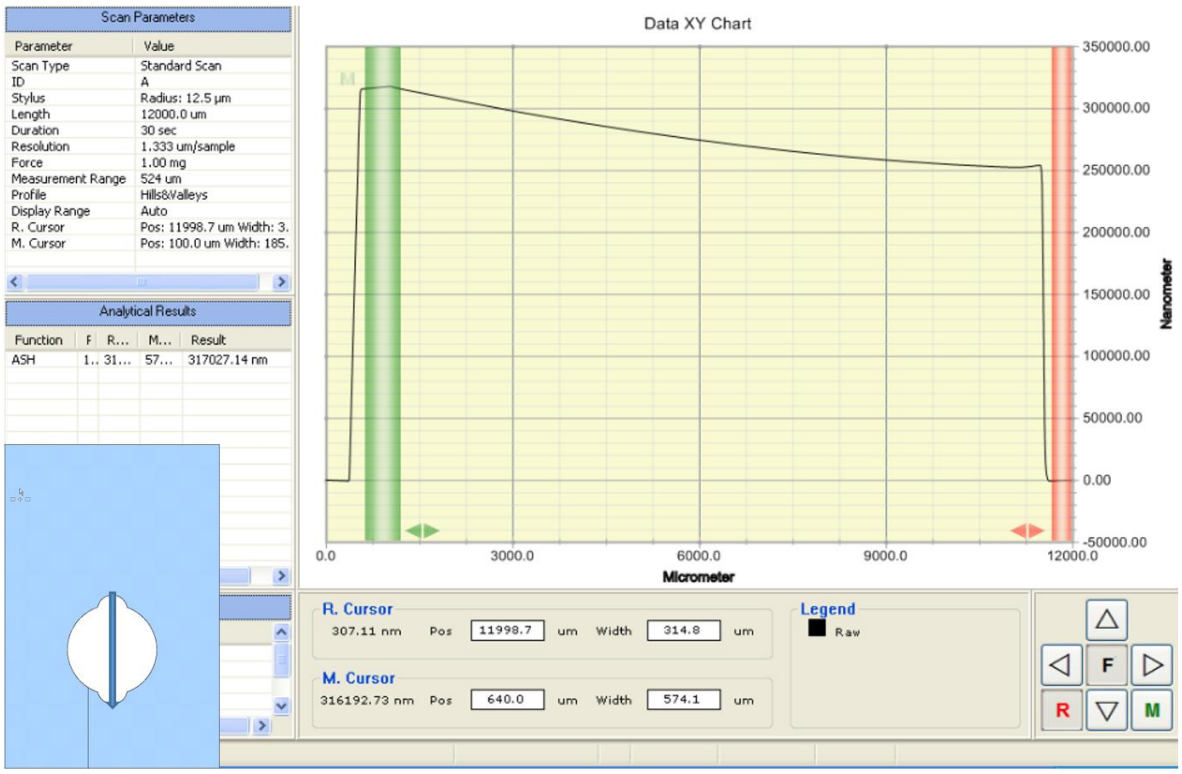
Figure 91 Wafer 1, measurements

Wafer two:

- Design 1

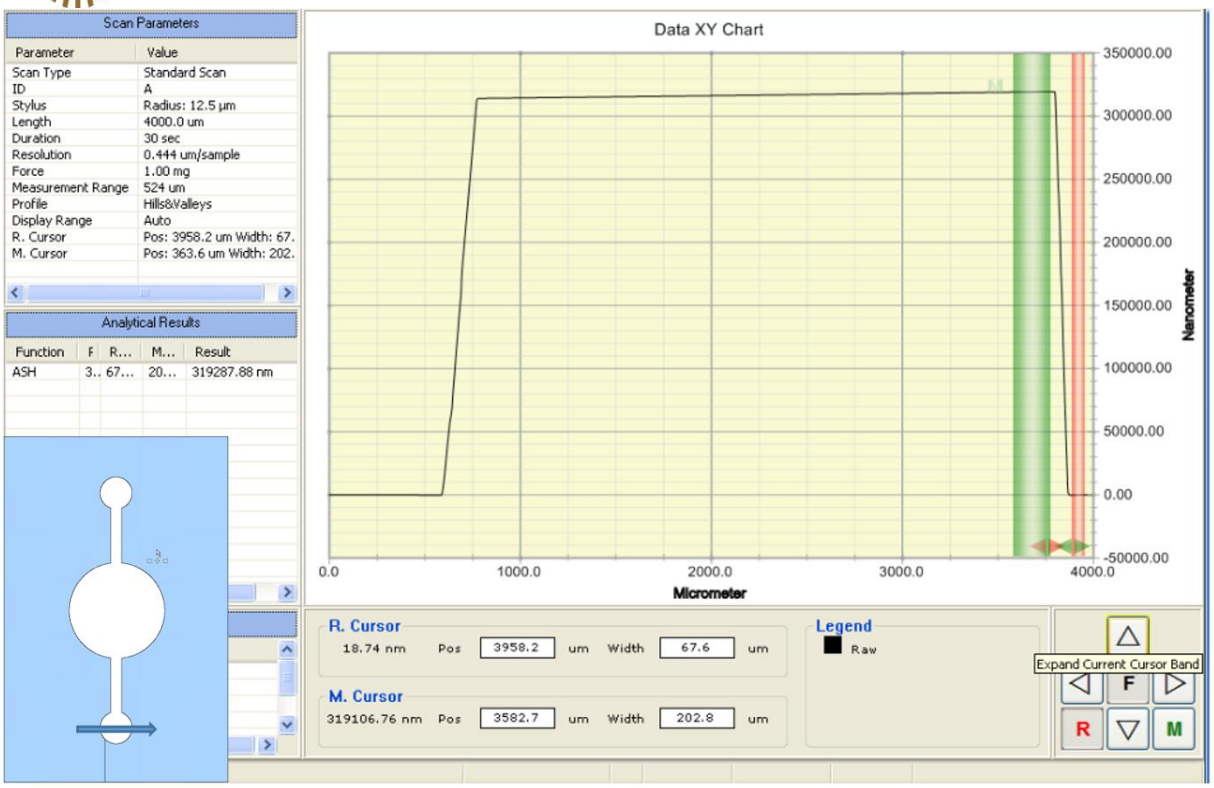


Minimum 255µm and maximum 310µm

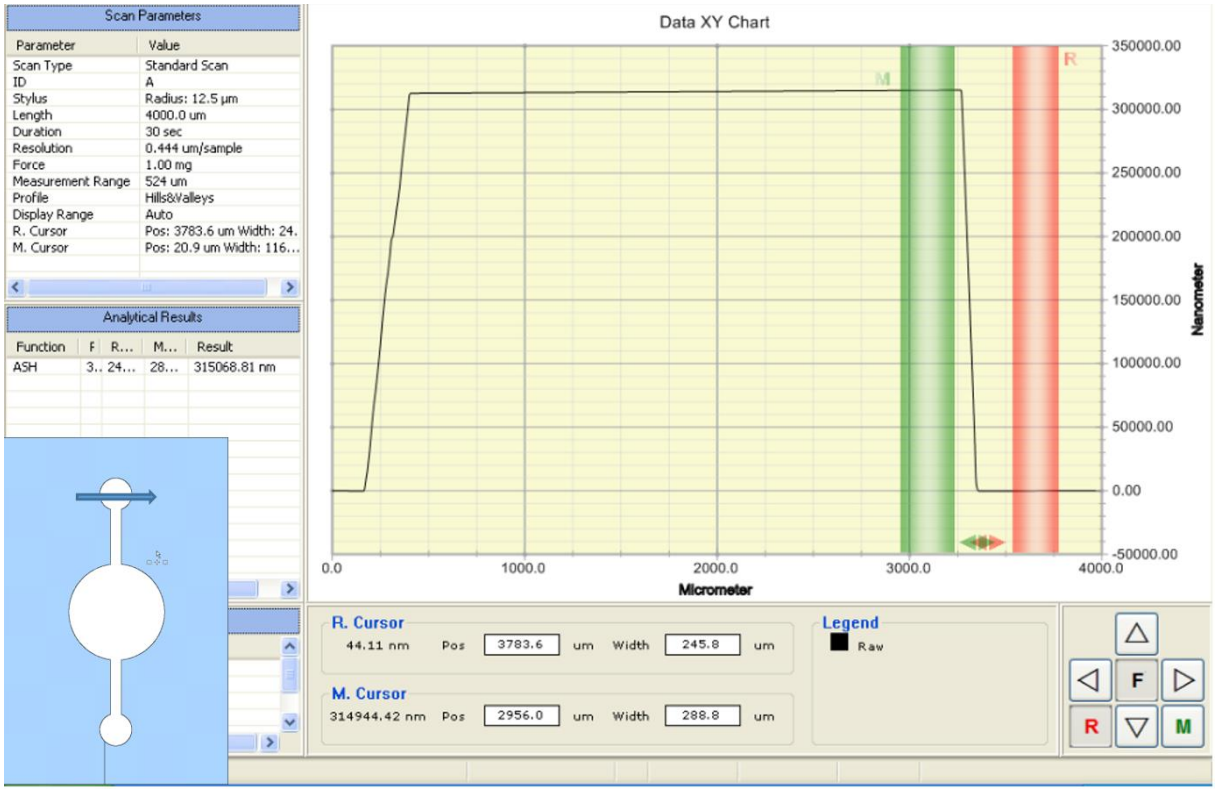


Minimum 255µm and maximum 320µm

- Design 2

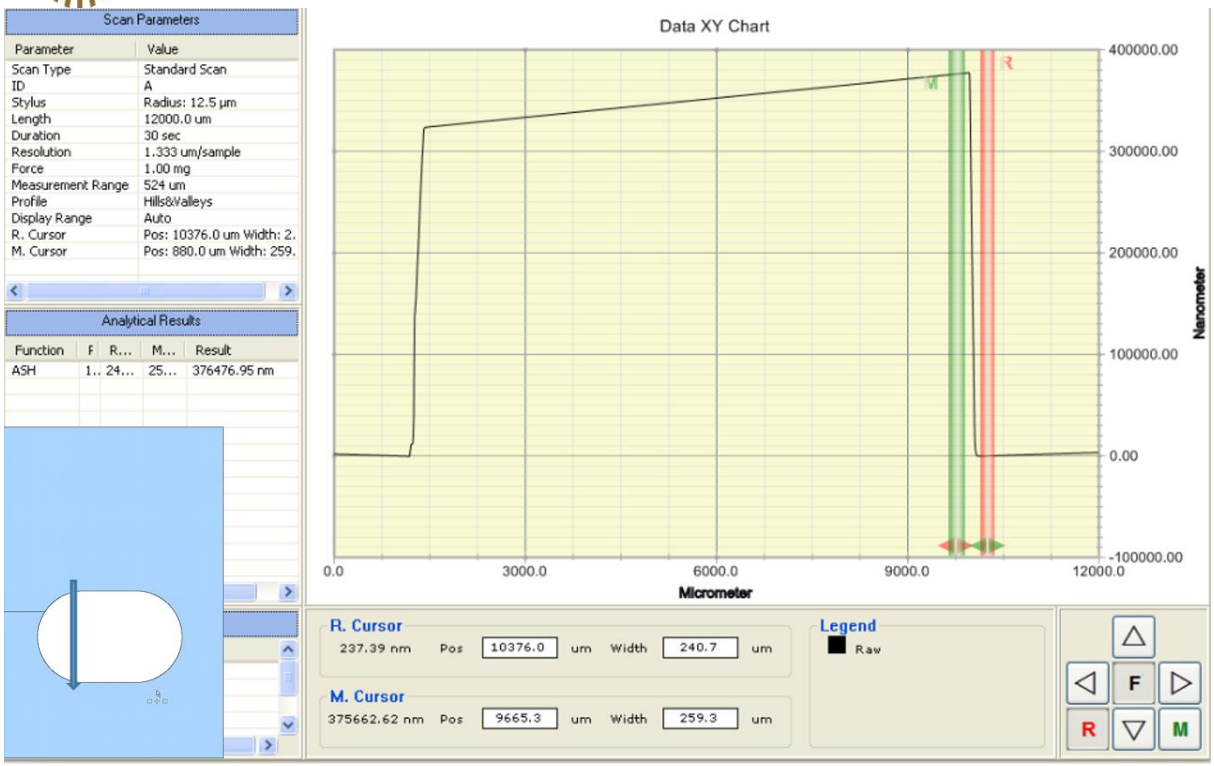


Minimum 315µm and maximum 320µm

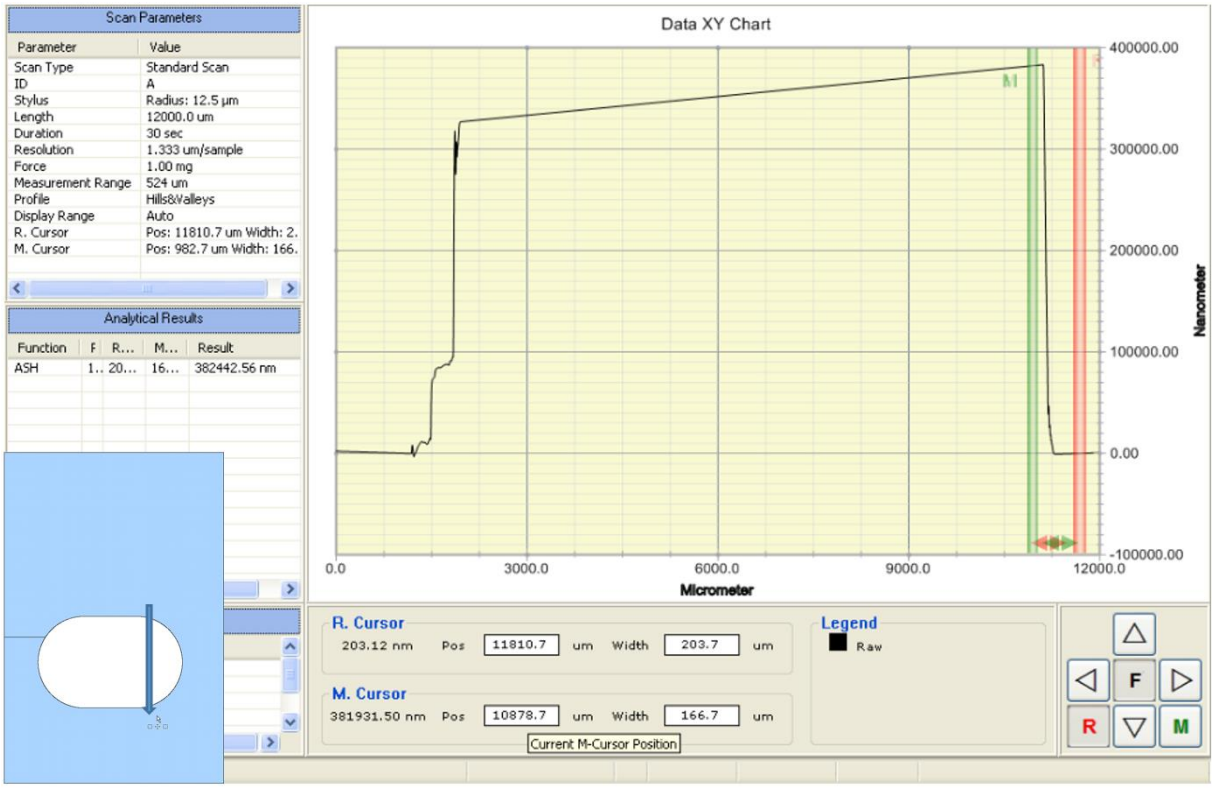


Minimum 310µm and maximum 315µm

- Design 3

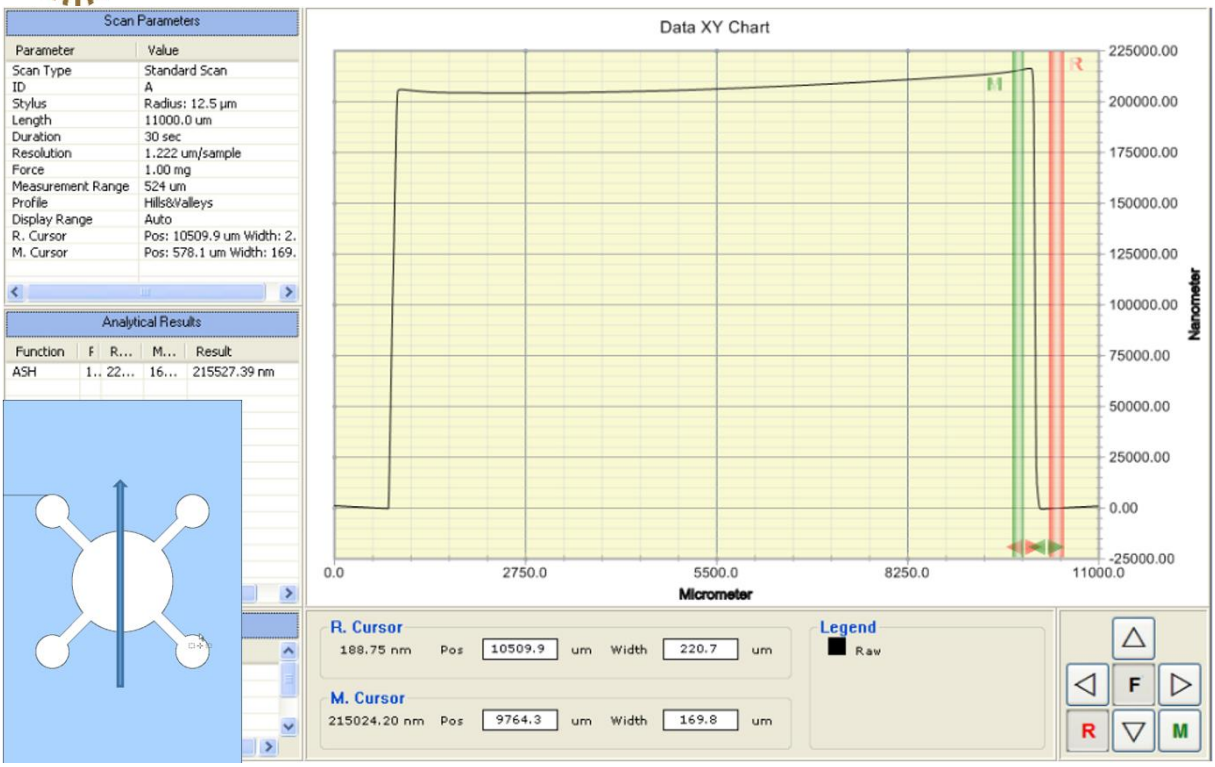


Minimum 325µm and maximum 380µm

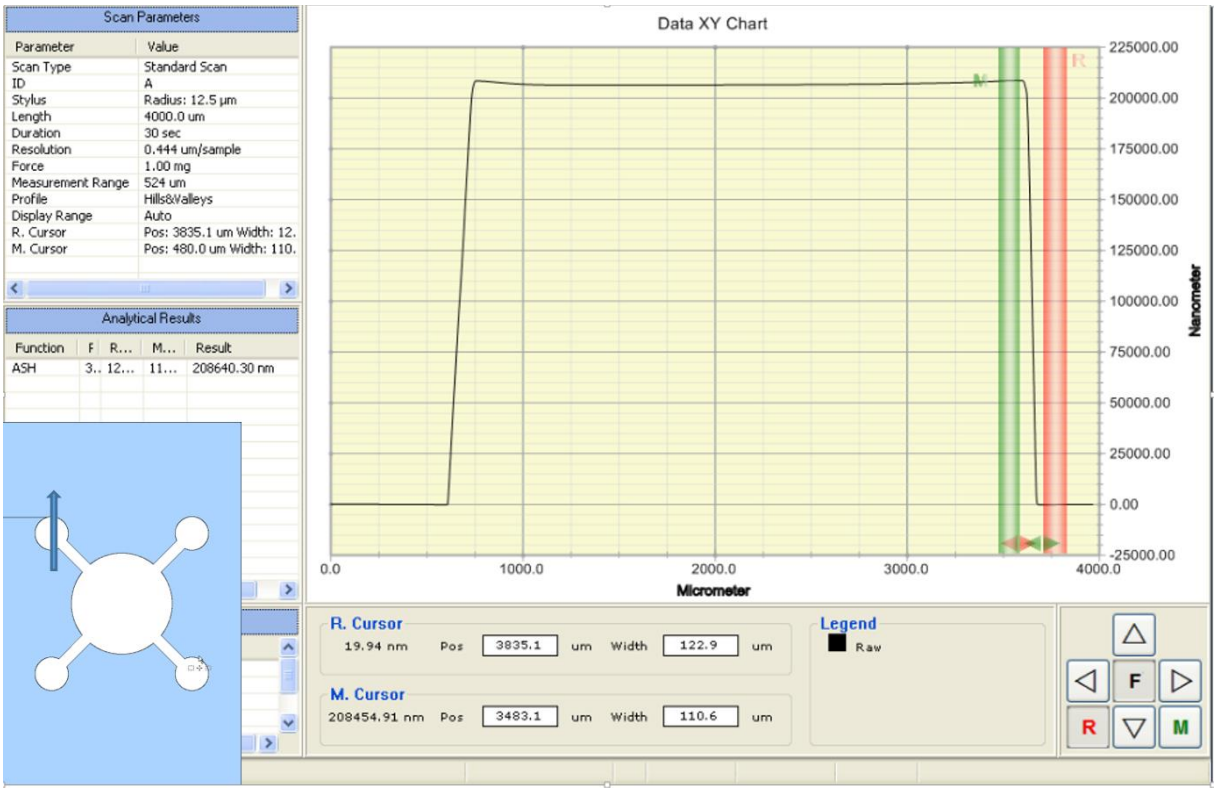


Minimum 330µm and maximum 385µm

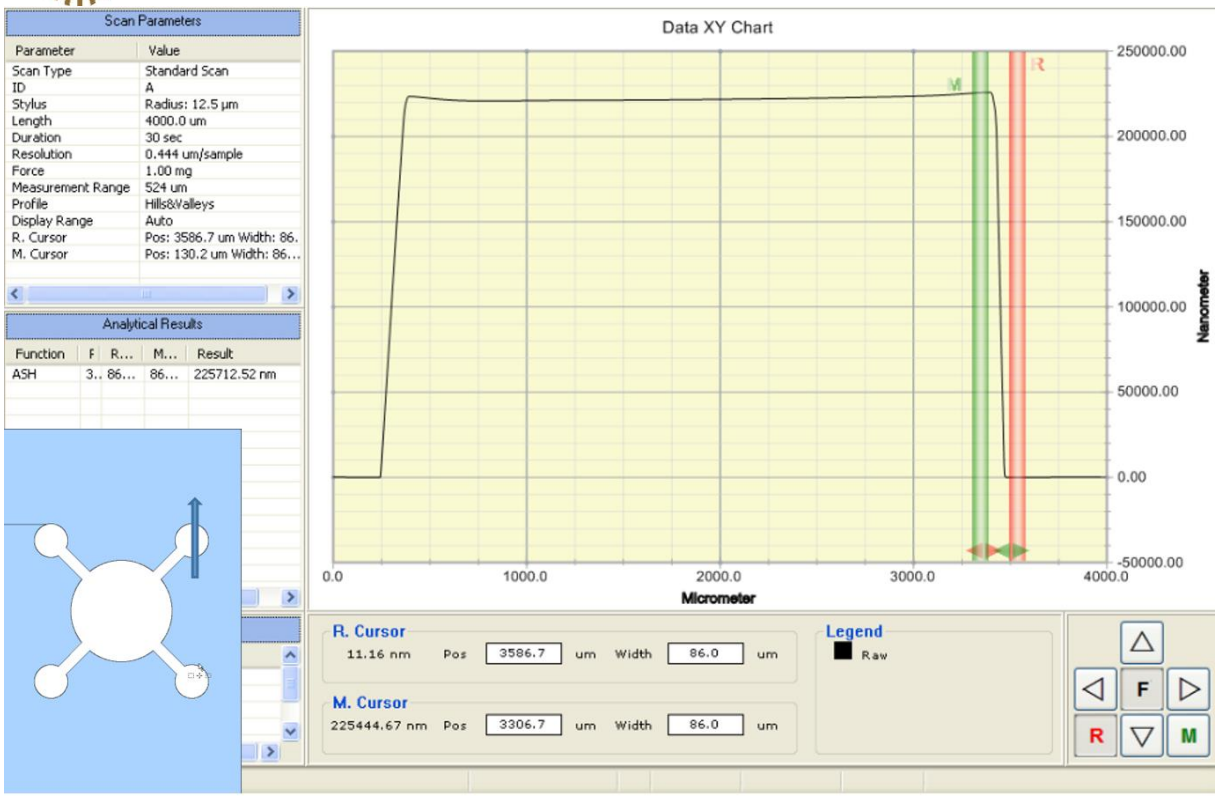
- Design 4



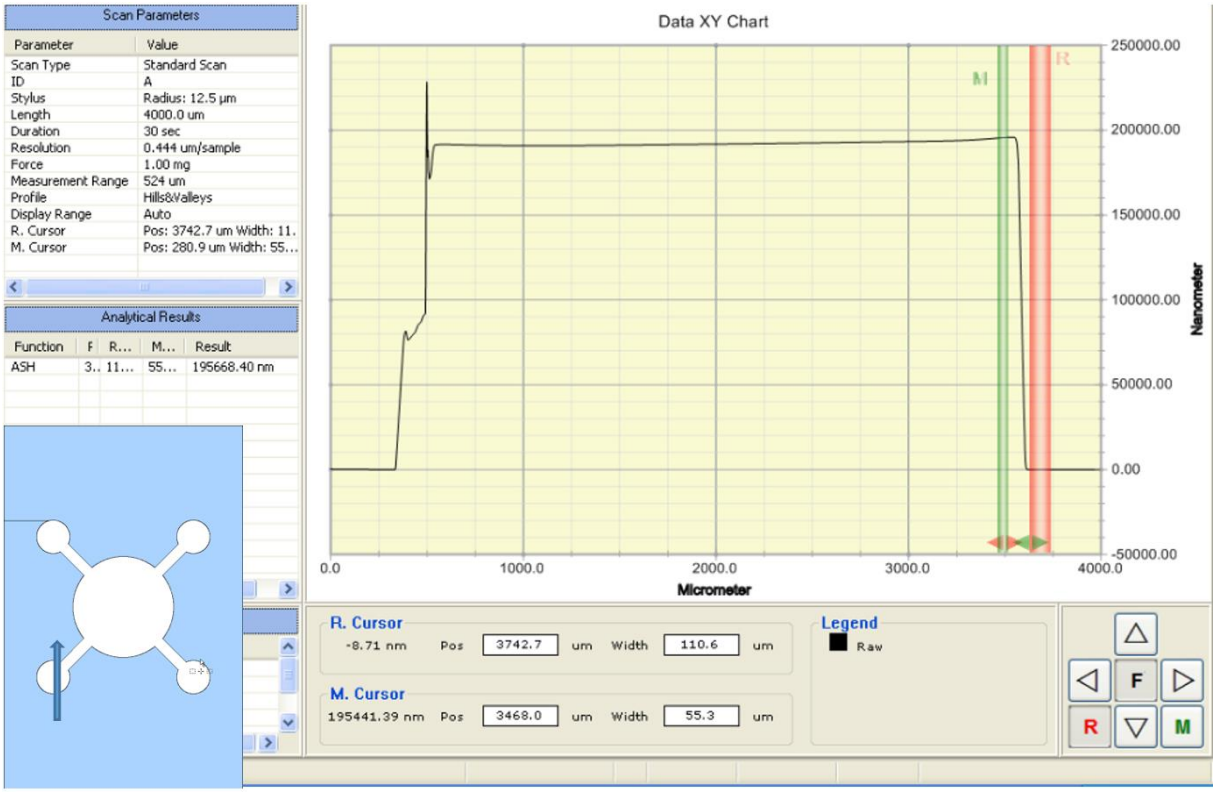
Minimum 205µm and maximum 215µm



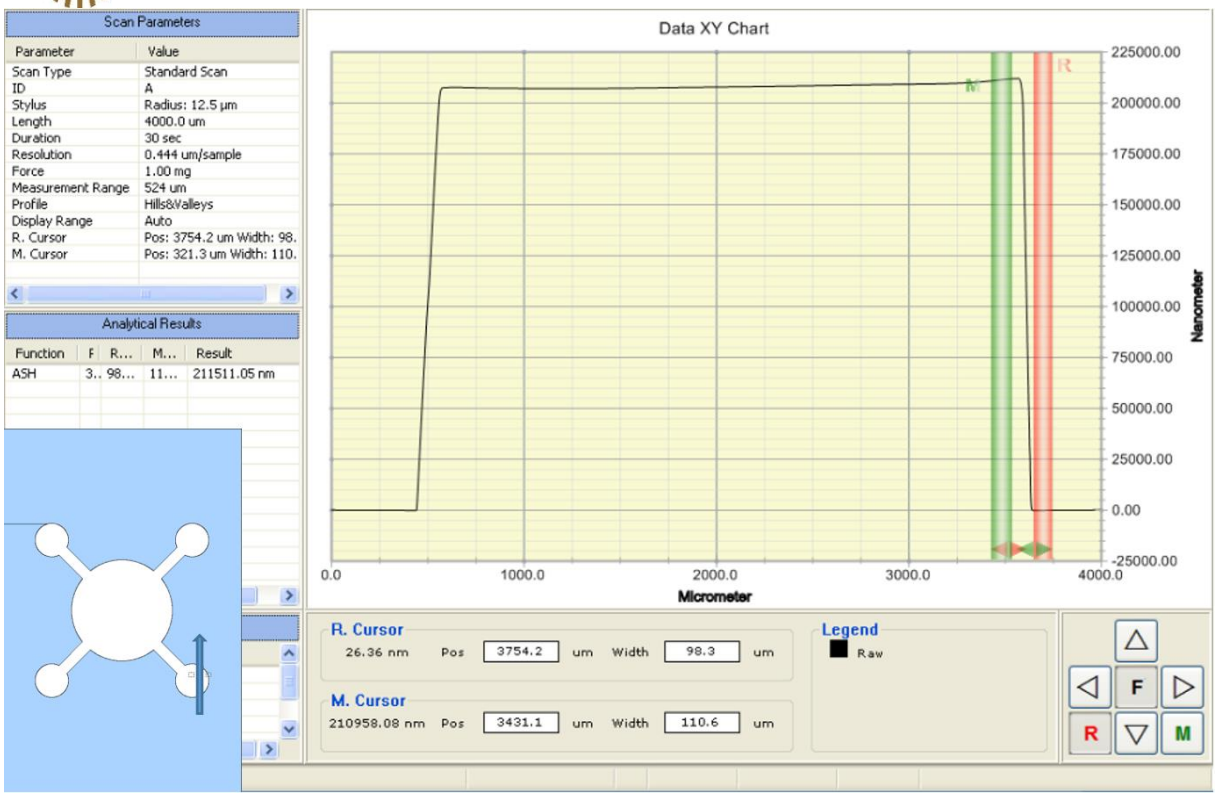
Minimum 205µm and maximum 210µm



Minimum 220µm and maximum 225µm

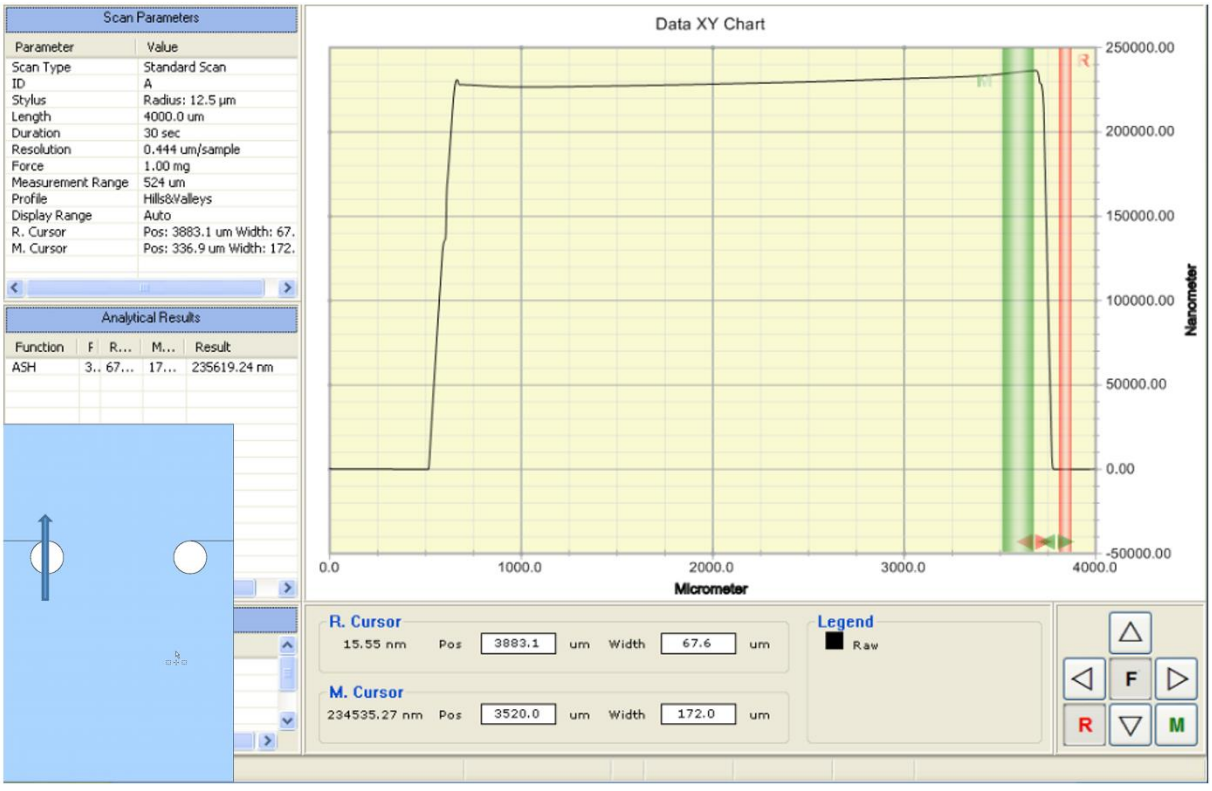


Minimum 185µm and maximum 195µm

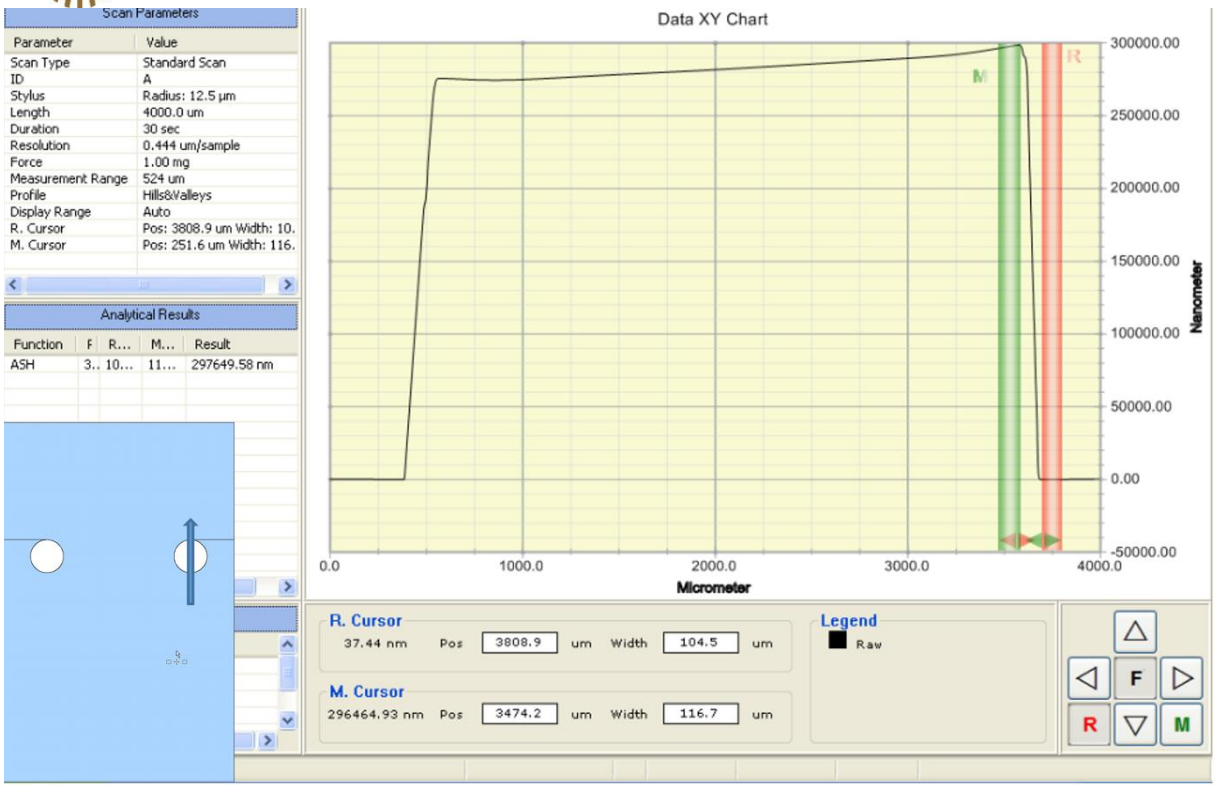


Minimum 205µm and maximum 210µm

- Design 5

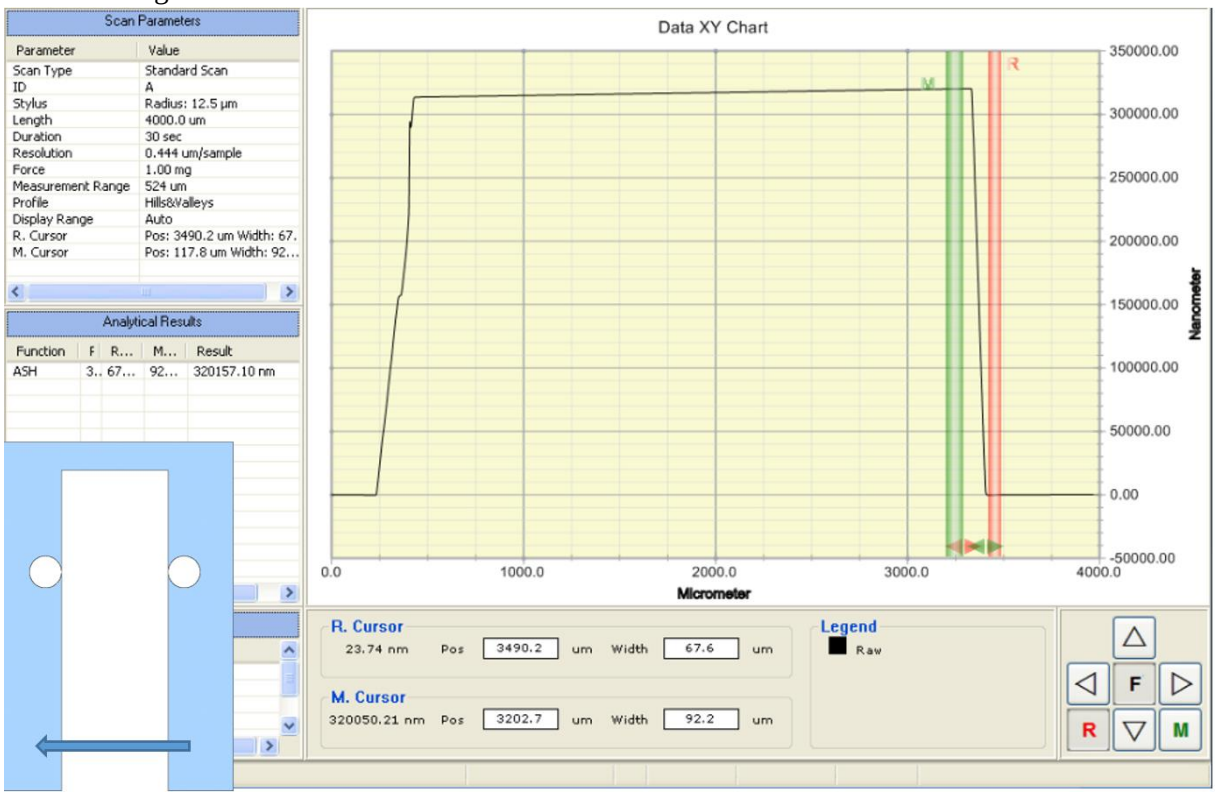


Minimum 225µm and maximum 235µm

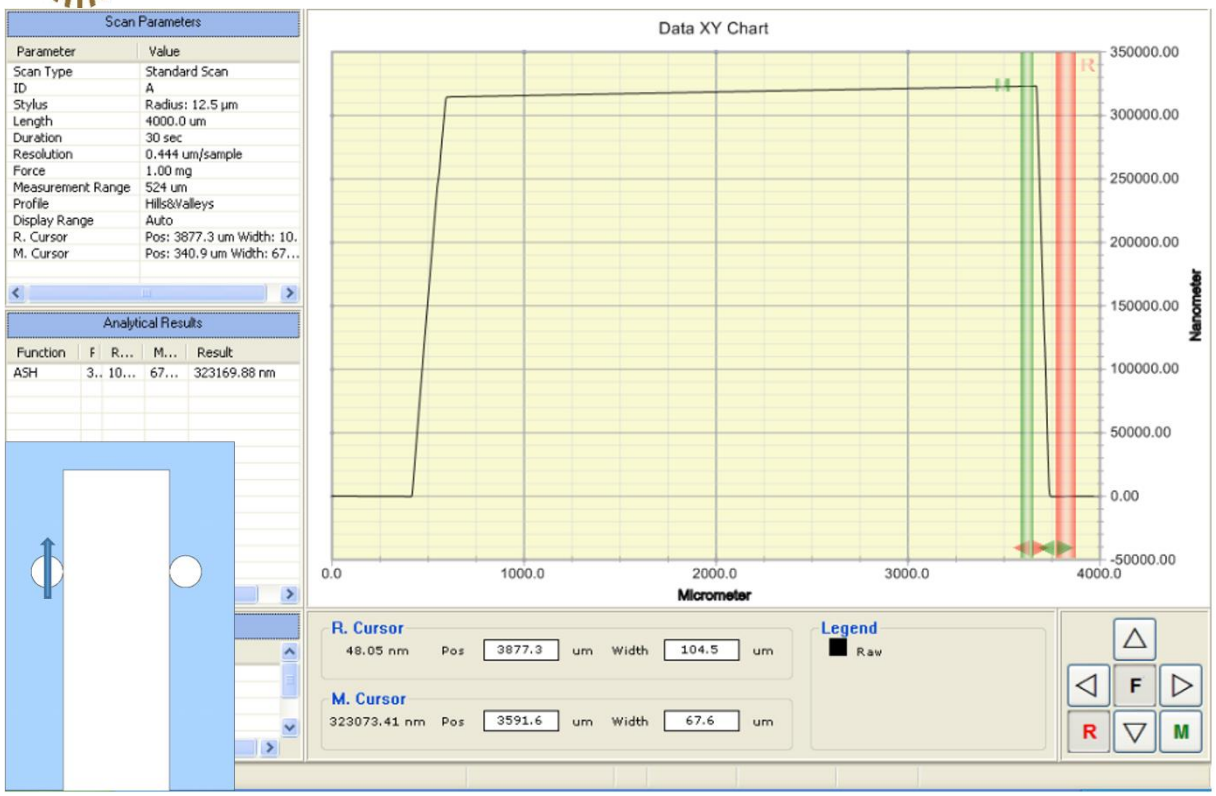


Minimum 275µm and maximum 300µm

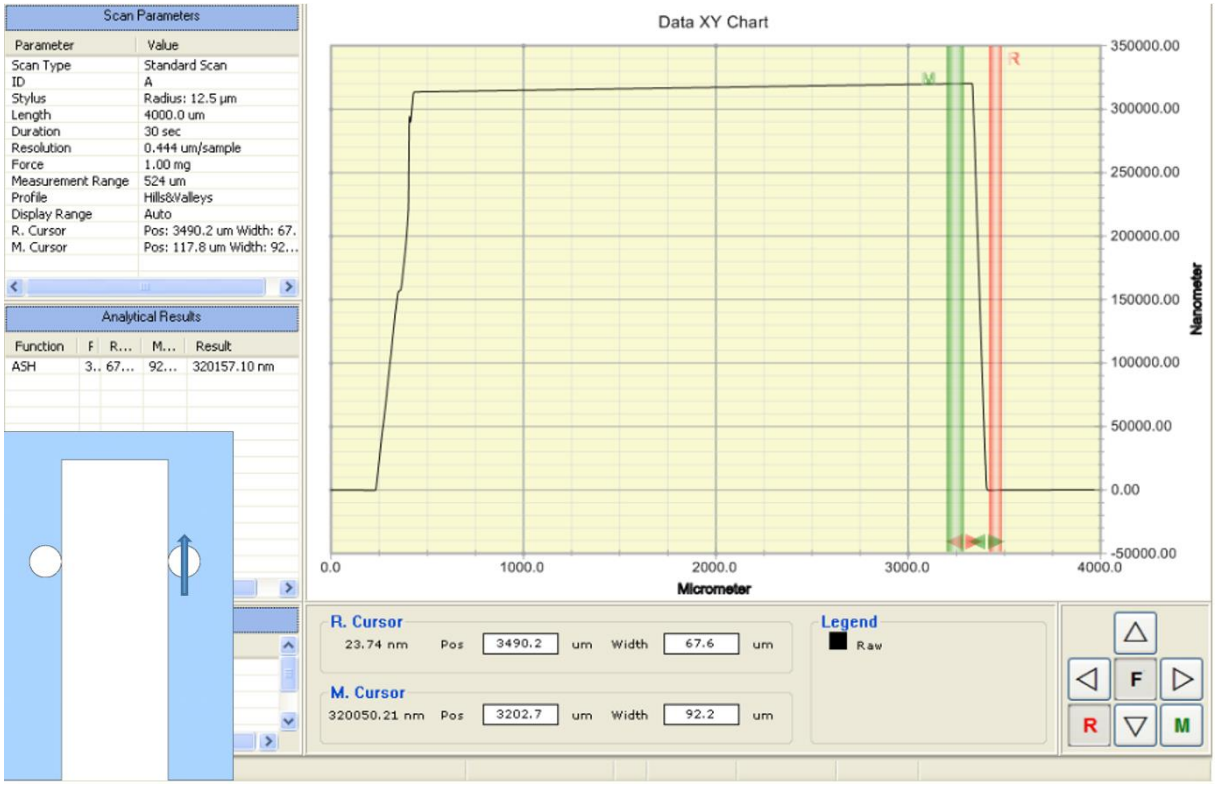
- Design 6



Minimum 315µm and maximum 320µm



Minimum 315µm and maximum 325µm



Minimum 315µm and maximum 320µm

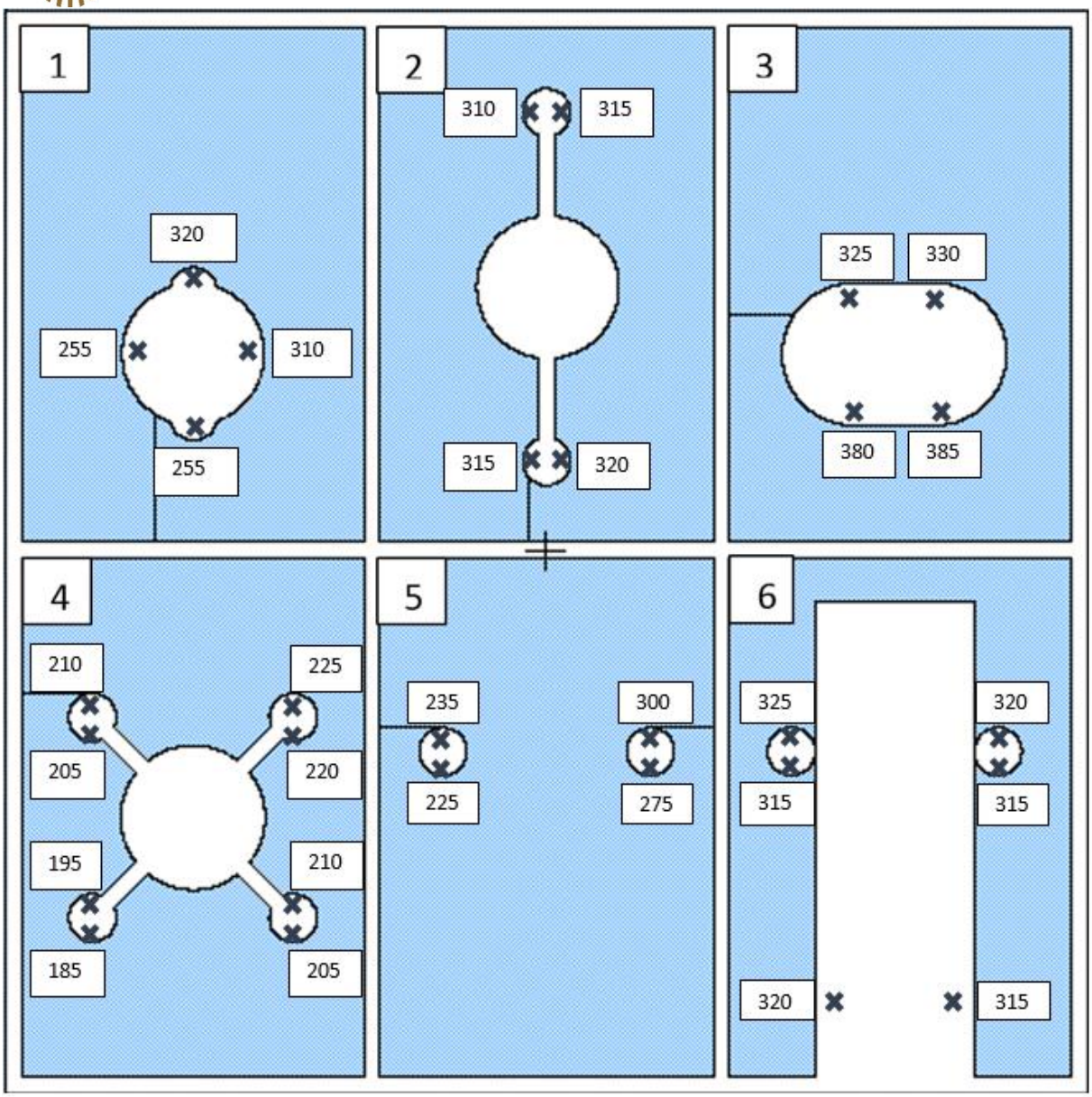


Figure 92 Wafer 2, measurements

HØGSKOLEN I BUSKERUD OG VESTFOLD

7.9.2 Exercise 2

Test laboratory exercise, Mars 20, 2015

Negative photoresist SU-8 100

Procedure. We followed this: http://www.microchem.com/pdf/SU8_50-100.pdf

First, all the machines have to be turned on. VERY IMPORTANT! REMEMBER THE PRESSURE VALVE IN THE CORRIDORE if I am using the new mask aligner! Always clean all the surroundings. USE PROTECTION GLASES!

Introduction:

The intention with this laboratory exercise is to learn the process of creating a master using SU-8 100 structure on a wafer. The idea is to follow the procedure recommended by MicroChem to create one wafer with 250µm thick structure, and another one with a 10µm thick structure. Hard baking will also be tried out to strengthen the structures.

Materials, chemicals and equipment:

- The glass mask designed with L-edit
- Isopropanol (IPA))
- Acetone
- Silicon wafer, 4”
- SU-8 100 negative photoresist
- Remover for SU-8 100
- Fume hood 4 for general solvents
- Fume hood 5 for corrosive solvents
- Spinner 1 Semitool 1
- Mask Aligner Karl Suss MA56
- DEKTAK profilometer
- Optical microscope I Leika DM4000M
- Hot plate

Procedure:

20. Cleaning wafer. Always finish with Ni.
    - 20.1. First, try to clean with Ni
    - 20.2. Clean with acetone and rinse with isopropyl (IPA)
    - 20.3. After drying with Ni, bake on 200°C during 10 minutes. Take it to the spinner quickly and spincoat as quick as possible. It might absorb humidity while cooling.
21. Coating the photoresist. We aimed for a layer of 250µm on wafer one and 100µm on wafer two.
    - 21.1. Coat bowl and shield with aluminum foil to protect

21.2. Set up the timer the following way: This can be adjusted somehow. The instructions are on the side of the spincoater.

TABLE XXXIV Spincoating parameters

Step	Speed (rpm)	Accelerate (sec)	Time (s)	Decelerate (s)
Thickness	100µm/250µm	100µm/250µm		
1	0	0	0	-
2	500	5s (500/5=100)	10	-
3	3000/900	10 s/10s	30/60	30

21.3. Try to place the wafer as centered as possible.

21.4. Pour the SU-8 carefully in the center. It is very viscous, so when it gets to the entrance of the bottle, slow the fluid by inclining the bottle. Try to control the flow by turning the bottle. Try to stop when you have a diameter =4cm approx.

21.5. Clean the bottleneck with IPA and place in the box.

21.6. Cover with the lid and start the program.

21.7. Use needle to gently remove the bubbles.

22. Soft bake. To eliminate residual solvent. Use alumina foil on the hot plate. Add 10°C to compensate

TABLE XXXV Soft bake parameters

Temperature (°C)	Time (min) 100µm	Time (min) 250µm
75	10	30
105	55	150

23. Pattern transfer using designed mask for the mask aligner. We measured the light intensity, ≈8,5mW/cm^2. C=8.5, T=6, B=12, R=8.5 and L=9. The table in the instruction manual showed that the exposure energy should be approx. 600 mJ/cm^2. 600:8,5=71 sec. (J=kgm^2/s^2 and W=kgm^2/s^3)

TABLE XXXVI Exposure parameters

Step	Dose (mJ/cm^2)	Light intensity (mW/ cm^2)	Time (s)
Exposure 100µm	600	8,5	71
Exposure 250µm	600	8,5	150

24. Post exposure bake. Still use the alumina foil and augment with 10 °C . Slowly from one temperature to the other, and let cool slowly on the hot plate at least 5 minutes to avoid cracks in the SU-8.

TABLE XXXVII Post exposure bake parameters

Temperature (°C)	Time (min) 100µm	Time (min) 150µm	Time (min) 250µm
75	1	1	1
105	10	12	20

25. Turn off the hot plate and leave the wafer until it reach approx. 75 °C
26. Table for Develop:

TABLE XXXVIII Development time

Thickness	Developement
100µm	10 min
250µm	22 min

Immerse the wafer in the developer. Move it carefully to for quicker results.

27. Hard bake at 200 °C for approximately 10 minutes
28. Clean everything and put the chemicals away. Throw used chemicals in waste bottle.

Results and discussion:

1. The first wafer has 250µm thickness and the second wafer has 100µm thickness. Wafer 1 had some impurity. I cleaned it twice but it seemed as if some residues stayed.

2. I applied the SU-8 as centered as I could, but I did not seem to get it right, hence it tended to spread unevenly as it slid toward the edges. This is something I need to work on. Both the wafers were totally covered with resist. I did not remove any bubbles because I could not see any, but one big bubble appeared on wafer 1 and several smaller on wafer two when I soft baked.

3. I used the big plate for both wafers at it seems to be the plate that gives the best contact area with the wafers. I just calculated the second wafer to be ready for soft baking when it remained 10 minutes of the first temperature (75 ^{o}C). The second wafer only needed 50 minutes on the second temperature (105 ^{o}C) where the first needed 150 minutes. This gave me the time to finish the second wafer, including development before the soft bake of the first wafer was ready.

4. I calculated the light intensity to 8,5mW/ cm^2, the energy required to 600 mJ/cm^2. This gives 71 seconds. I used this on the second wafer but used 150 seconds on wafer number one, being mre than of dobble thickness. At the laboratory exercise dated 16/3, I used 71 seconds on both the wafers measuring 180µm to 410µm, and it also seemed to work. It might not be necessary.

There was no problem with the second wafer, but even though the soft baking time had been increased by 67%, the first wafer still got stuck on the glass mask, but only at a very small spot. Maybe I have to wait longer for the resist to cool down and harden more before I use the mask aligner. If that is not enough I will have to soft bake even more

5. The post exposure bake went fine, with no problems.

6. I turned off the plate and let it cool down to less than 75 ^{o}C and at least 5 minutes, before removing it from the hot plate.

7. The second wafer was developed in exactly the time estimated for 100µm. The first wafer took 2 minutes longer than expected. It seemed to have different thicknesses across the design. This will be measured when I have access to the profilometer. The second wafer looked even at the mere sight.

8. I hard baked both wafers to release stress in the structures. After 10 minutes at 200 ^{o}C, I turned off the plate and waited approximately 5 minutes for the plate to slowly cool down. This went perfect with the thinner resist, but when I took the first wafer off the plate, it started cracking and detach from the wafer. Next time I will try to cool it for 30 minutes, and if that does not work I go back to not hard bake the wafers.

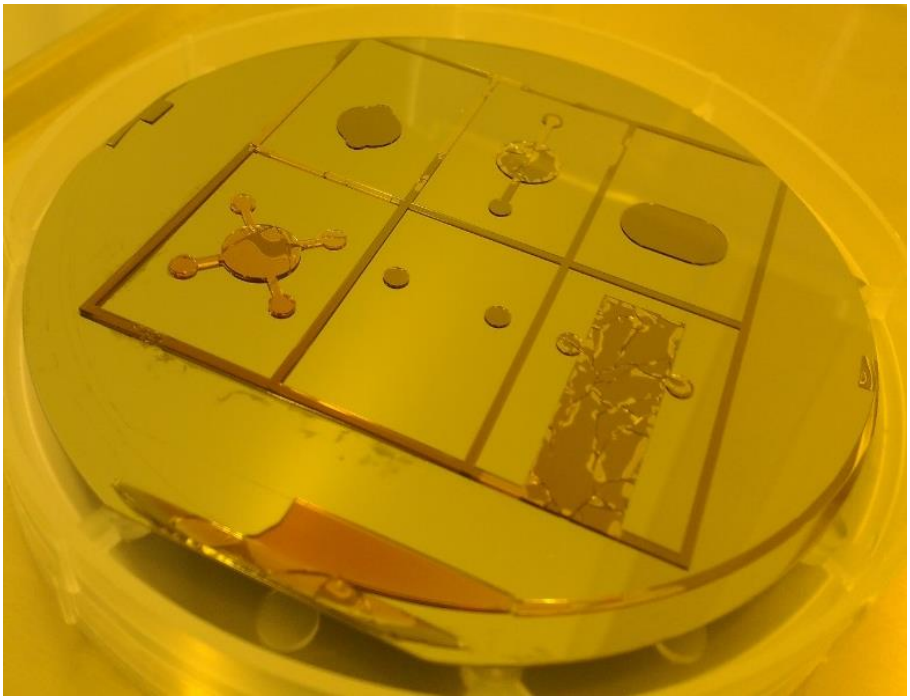
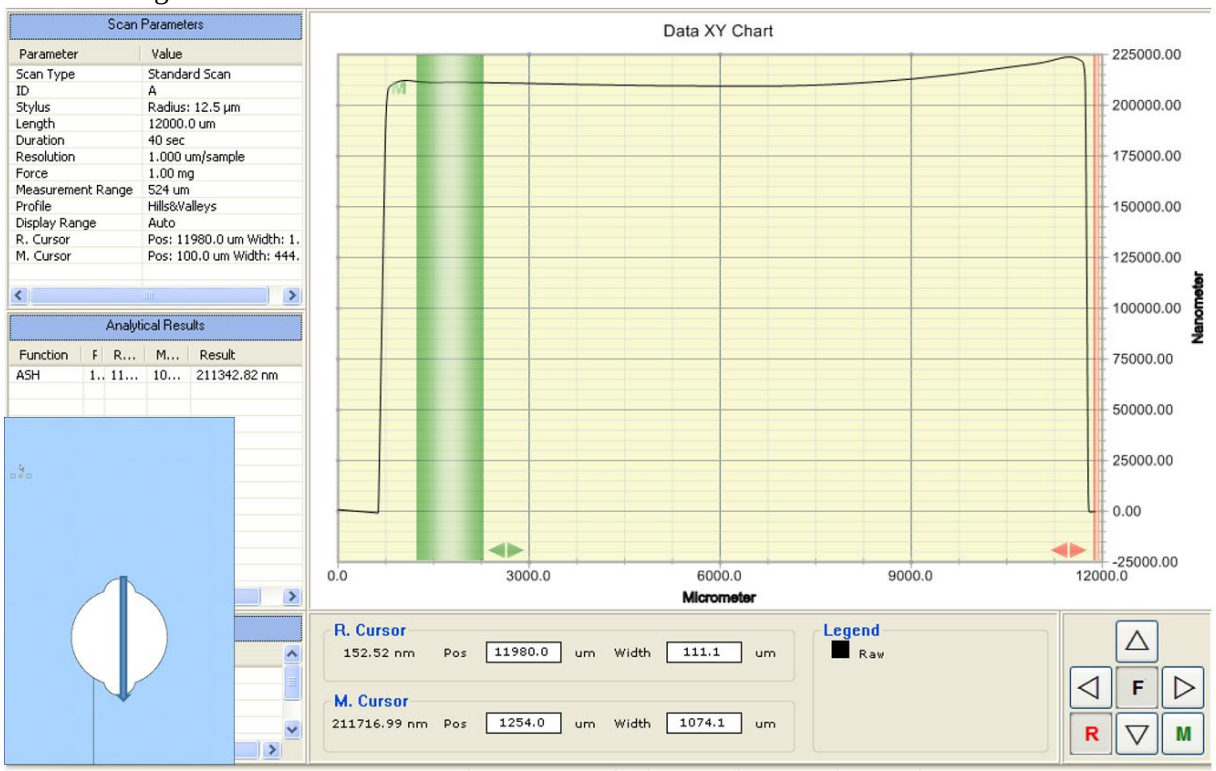


Figure 93 Wafer 1 after hard baking

Measurements:

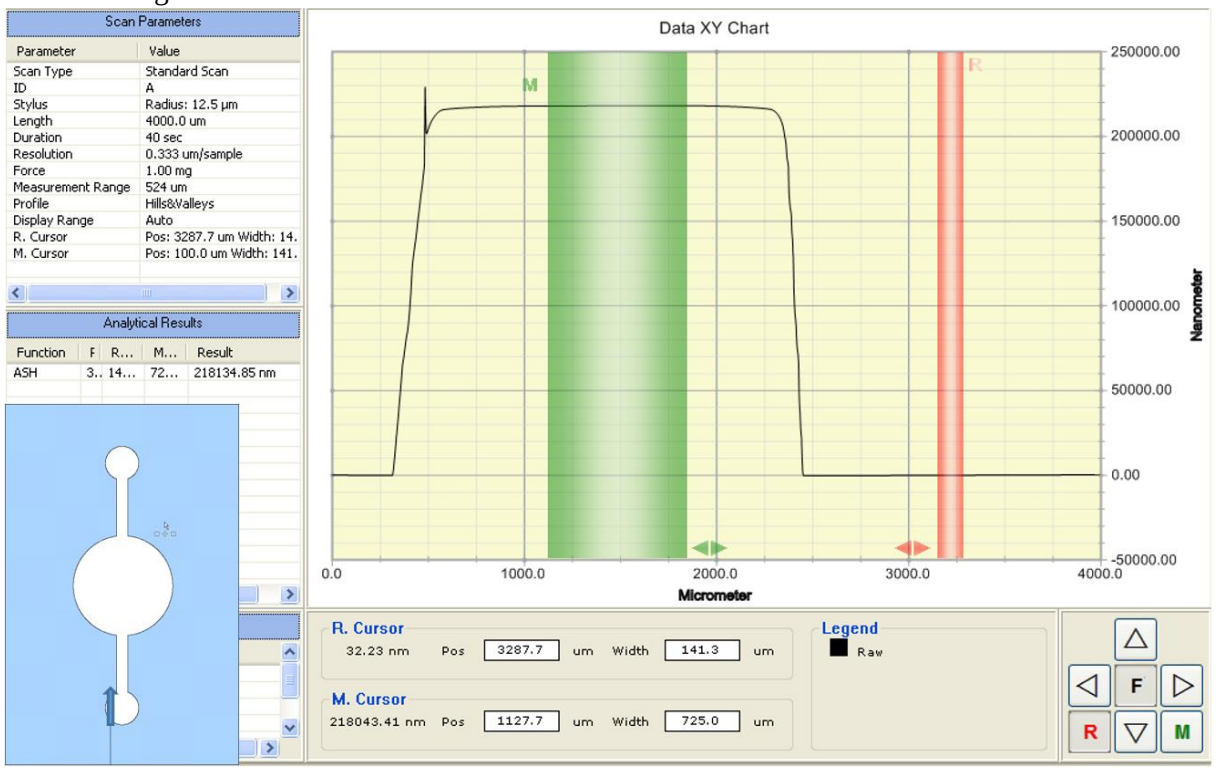
Wafer one 250µm(The measurements I could do were limited due to the breakage):

- Design one:



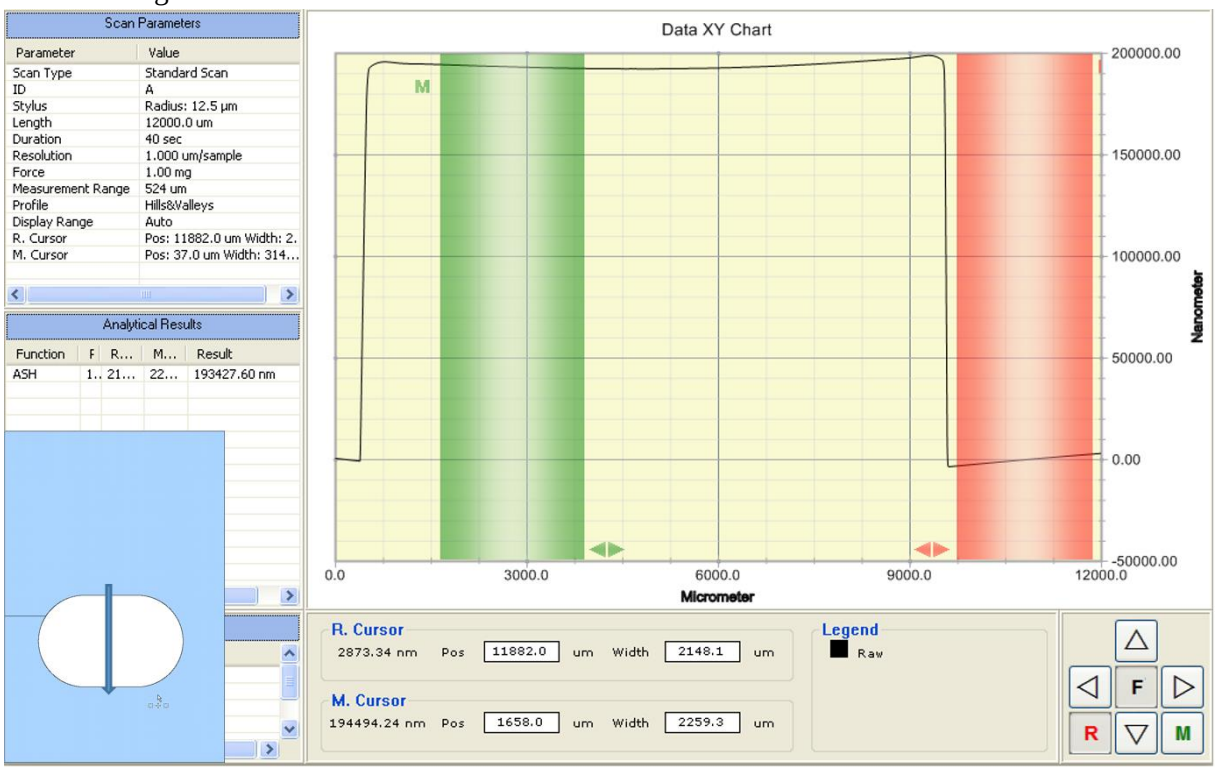
Minimum 210µm and maximum 225µm

- Design two:



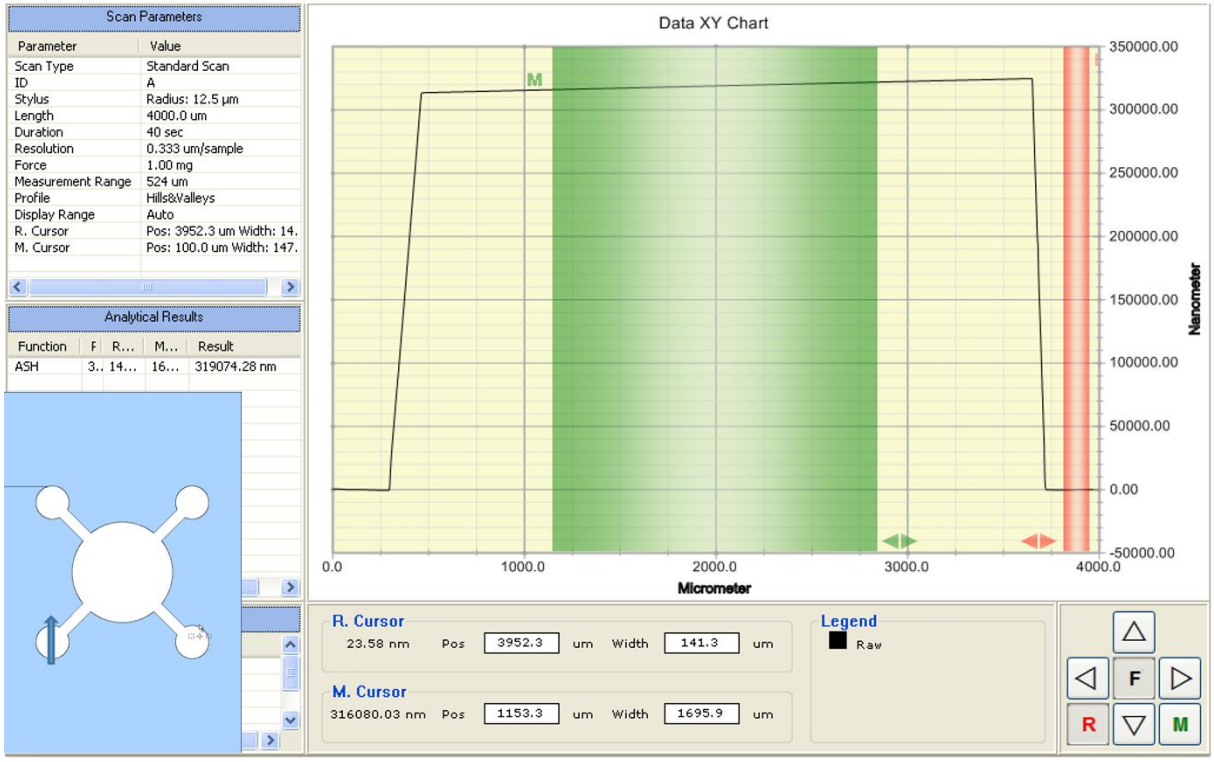
220µm. The peak is just the needle of the profilometer that jumped after

- Design three:



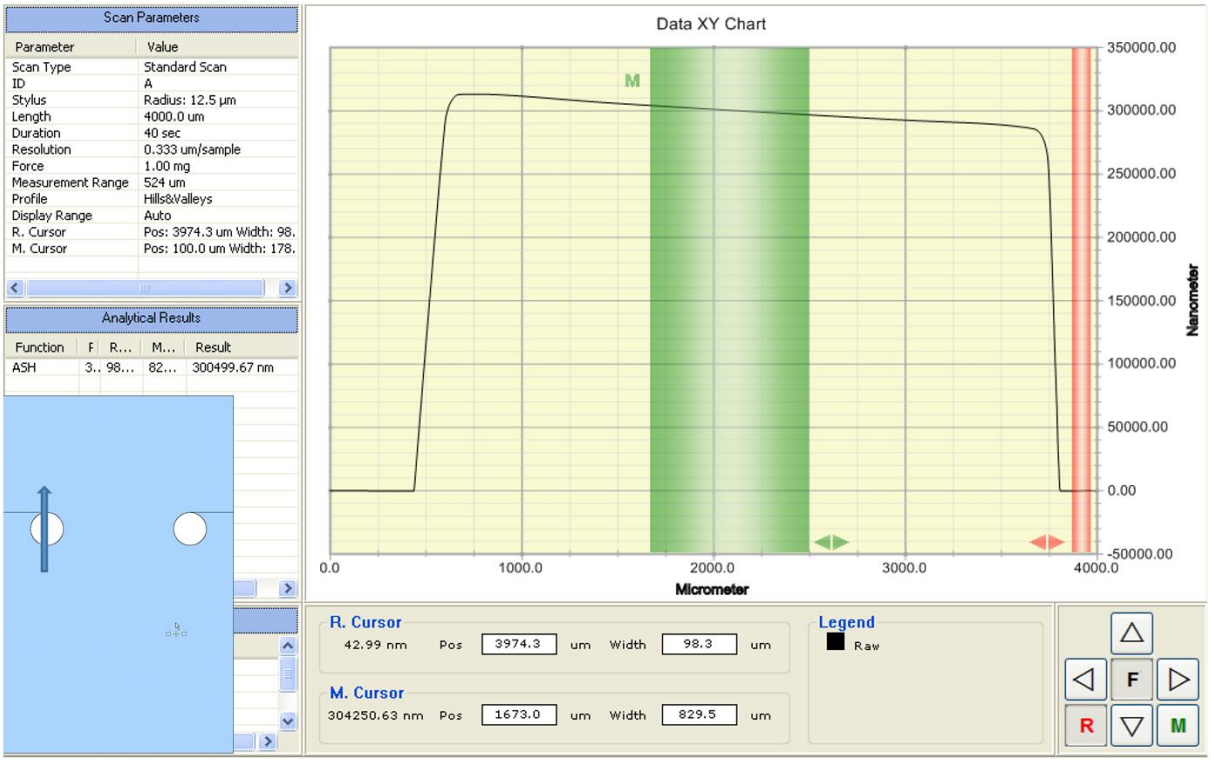
Minimum 190µm and maximum 200µm

- Design four:

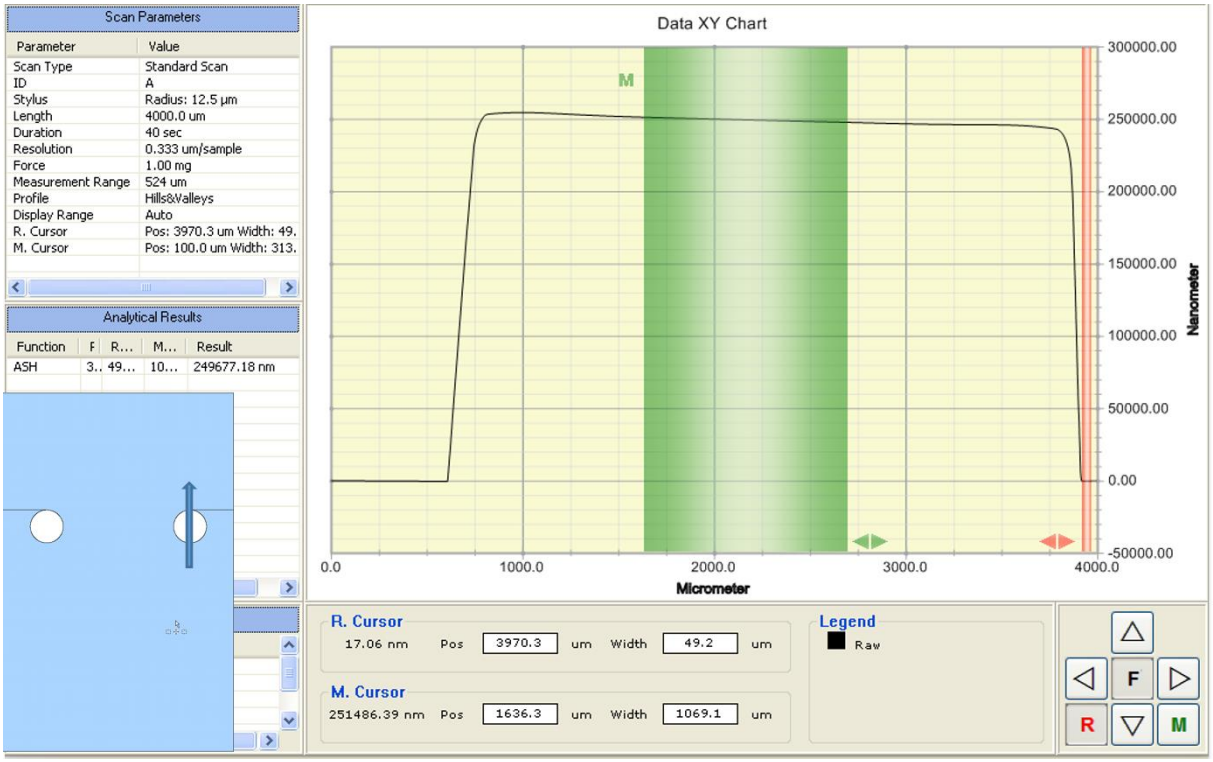


Minimum 310µm and maximum 325µm

- Design five:



Minimum 285µm and maximum 315µm



Minimum 245µm and maximum 255µm

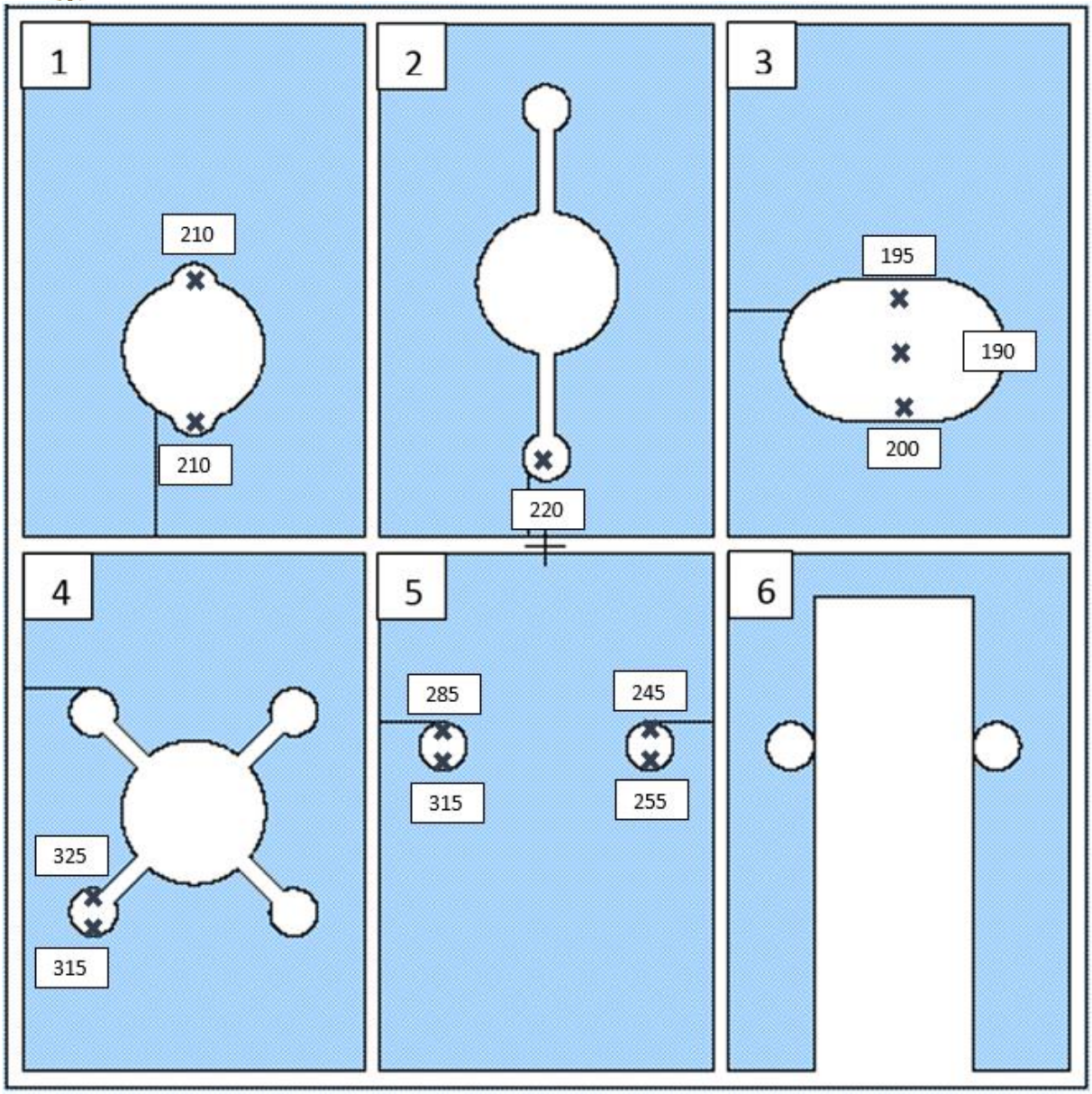
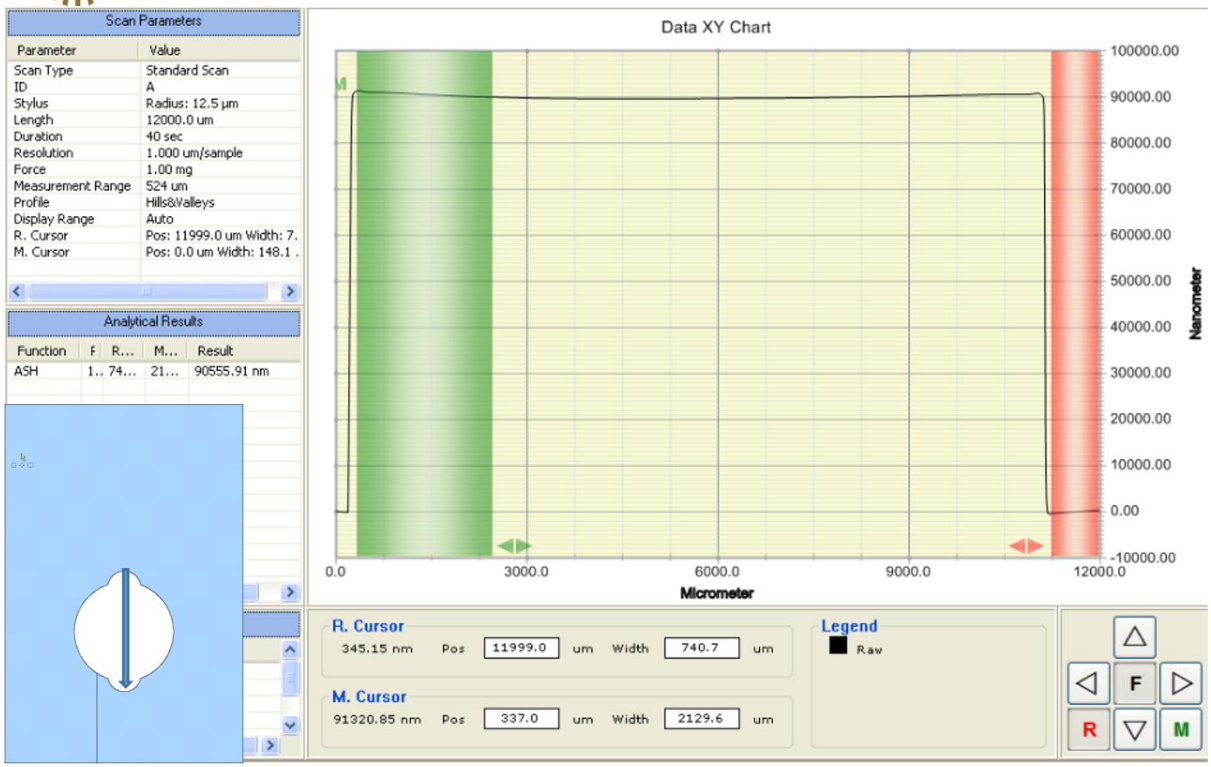


Figure 94. Wafer 1, measurements

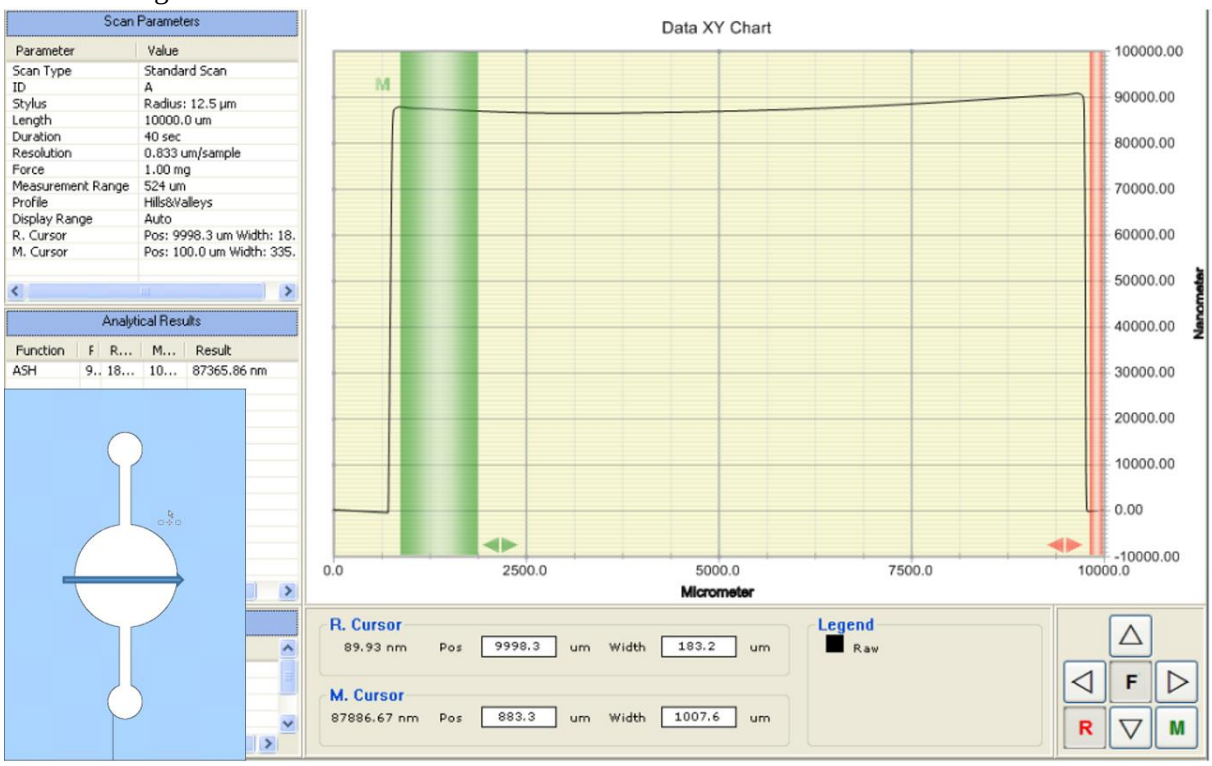
Wafer two 100µm:

- Design one:

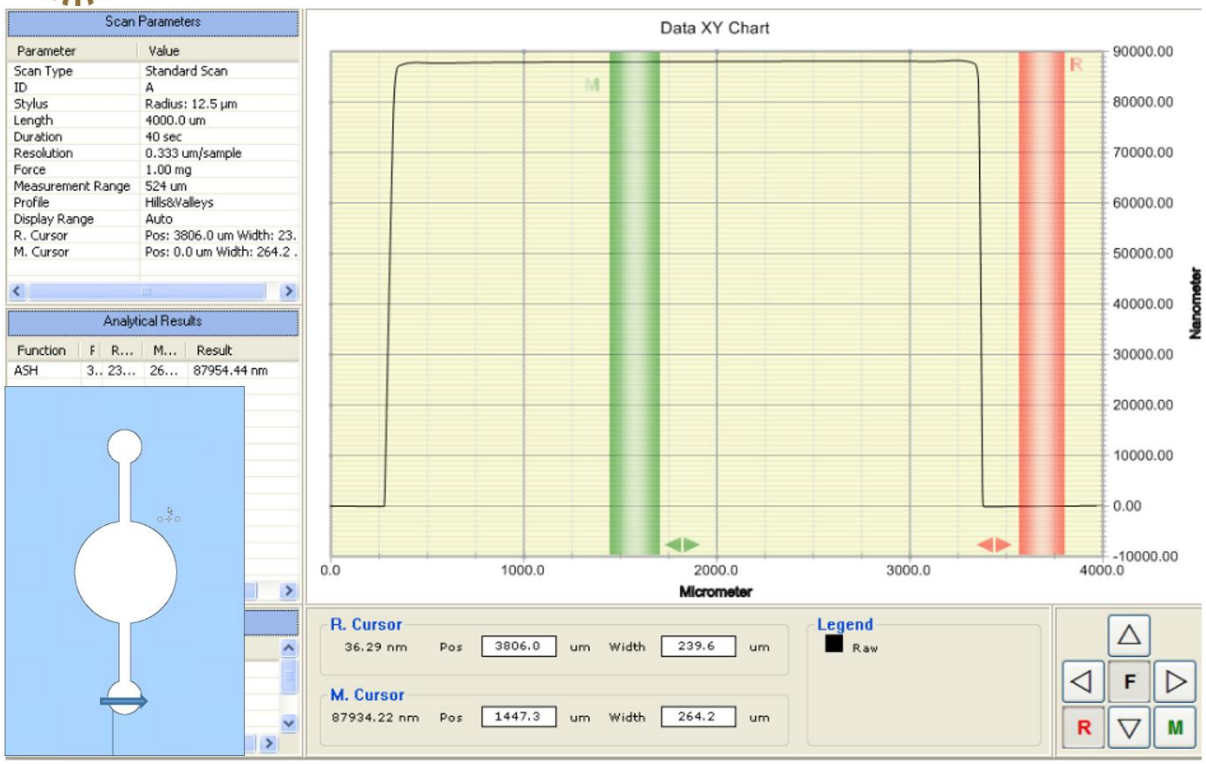


90µm

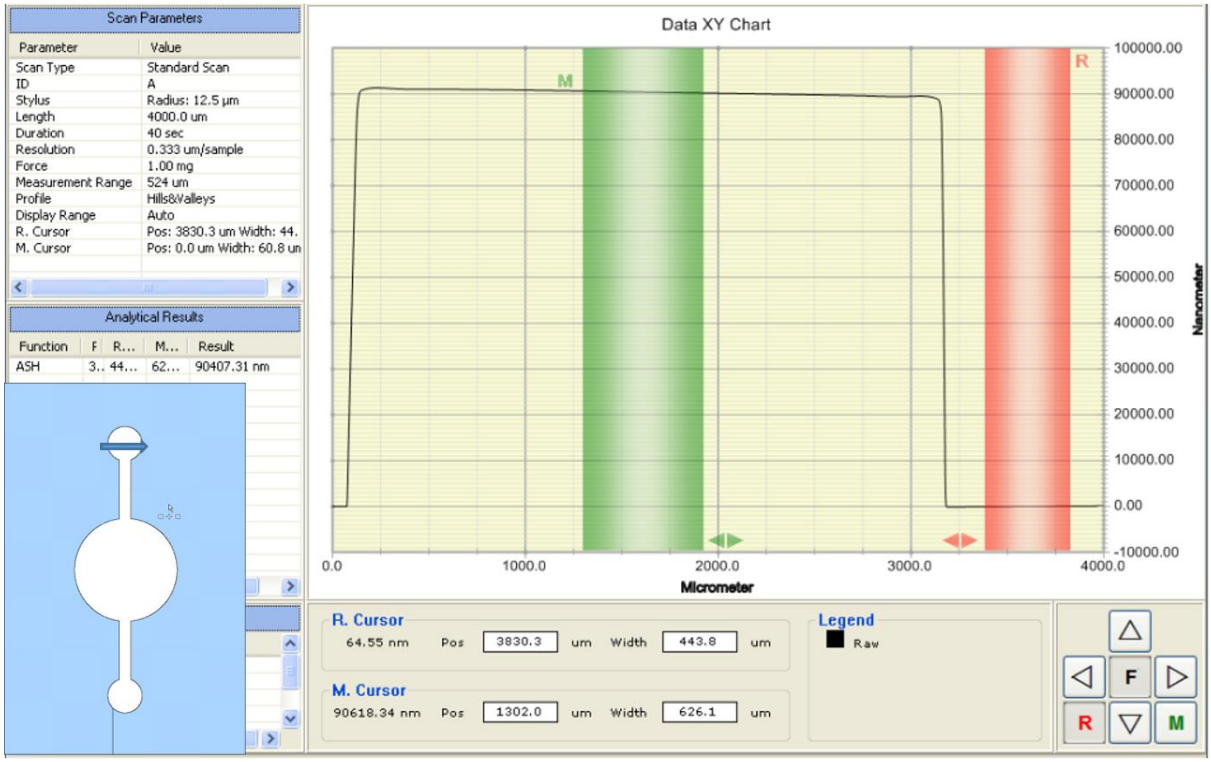
- Design two:



Minimum 88µm and maximum 91µm

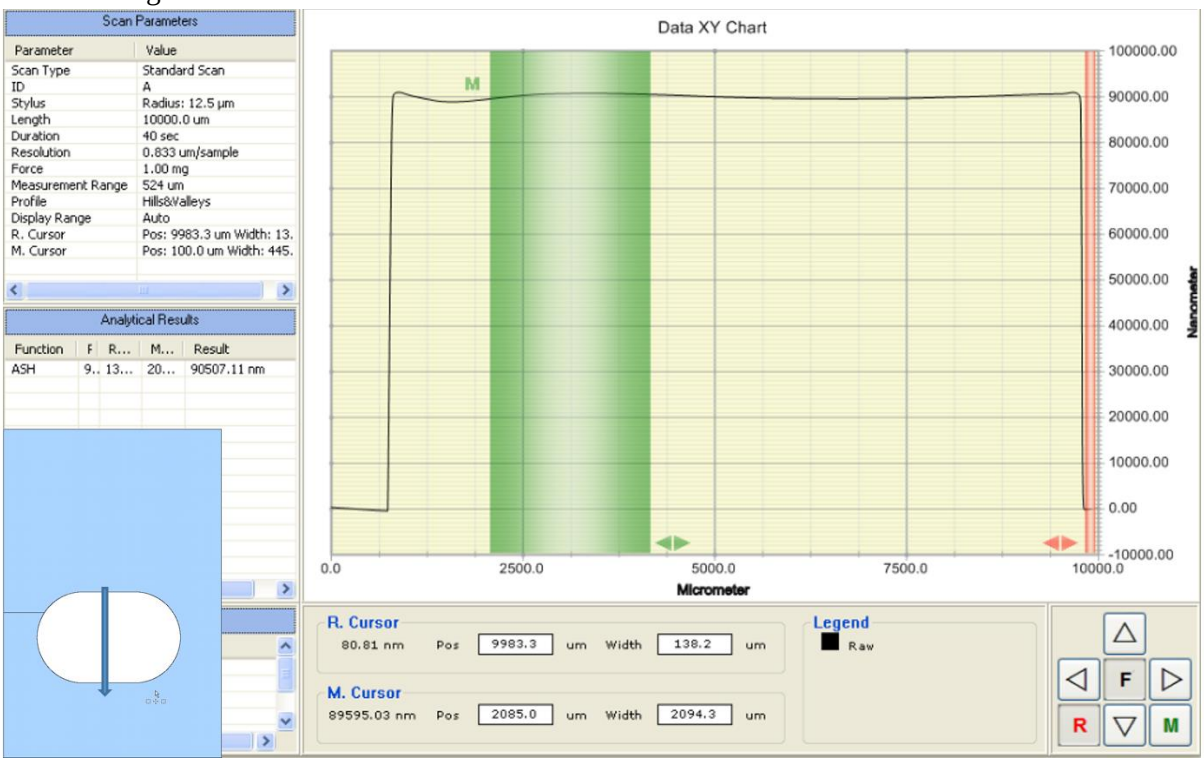


88µm



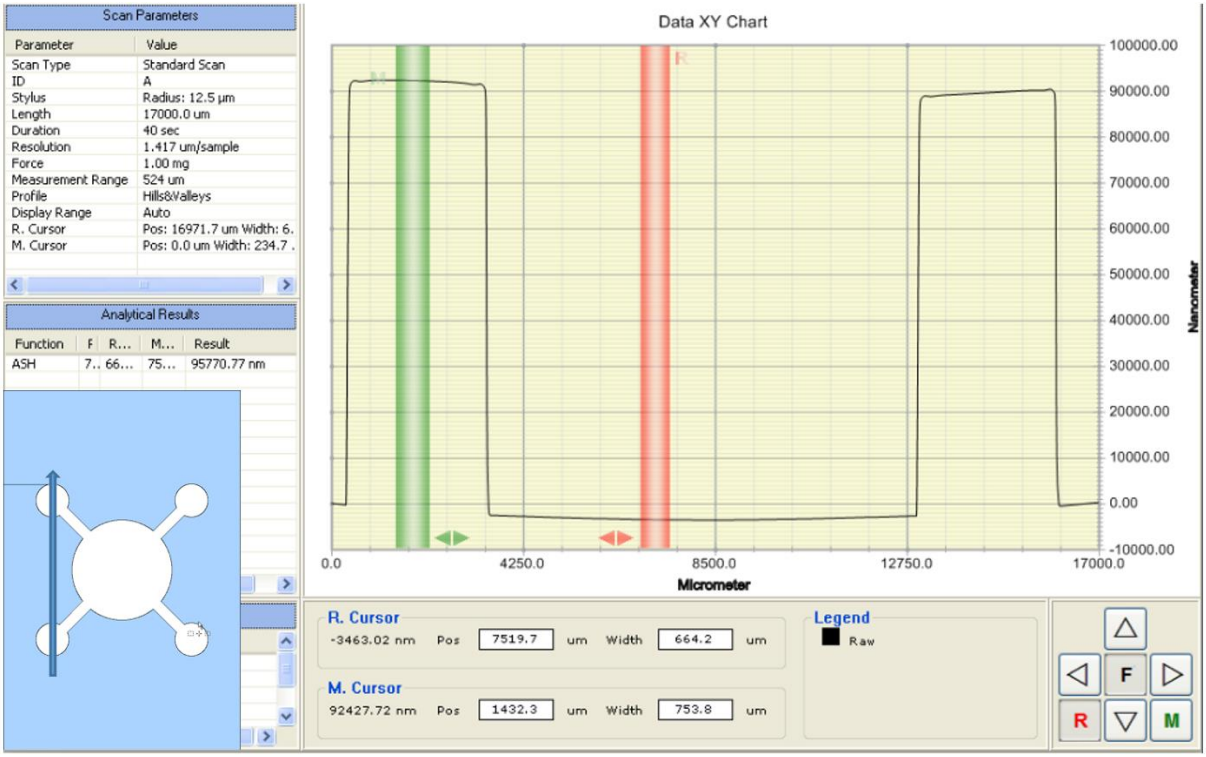
Minimum 90µm and maximum 91µm

- Design three:

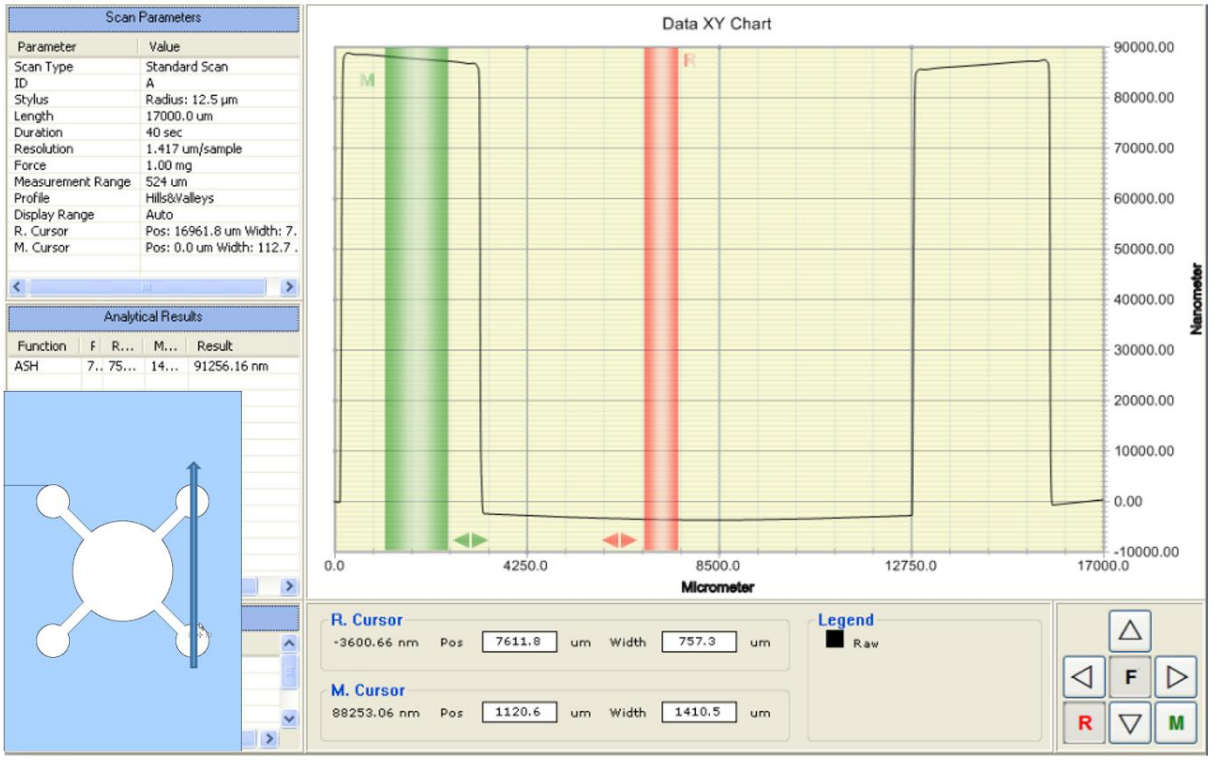


90µm

- Design four:

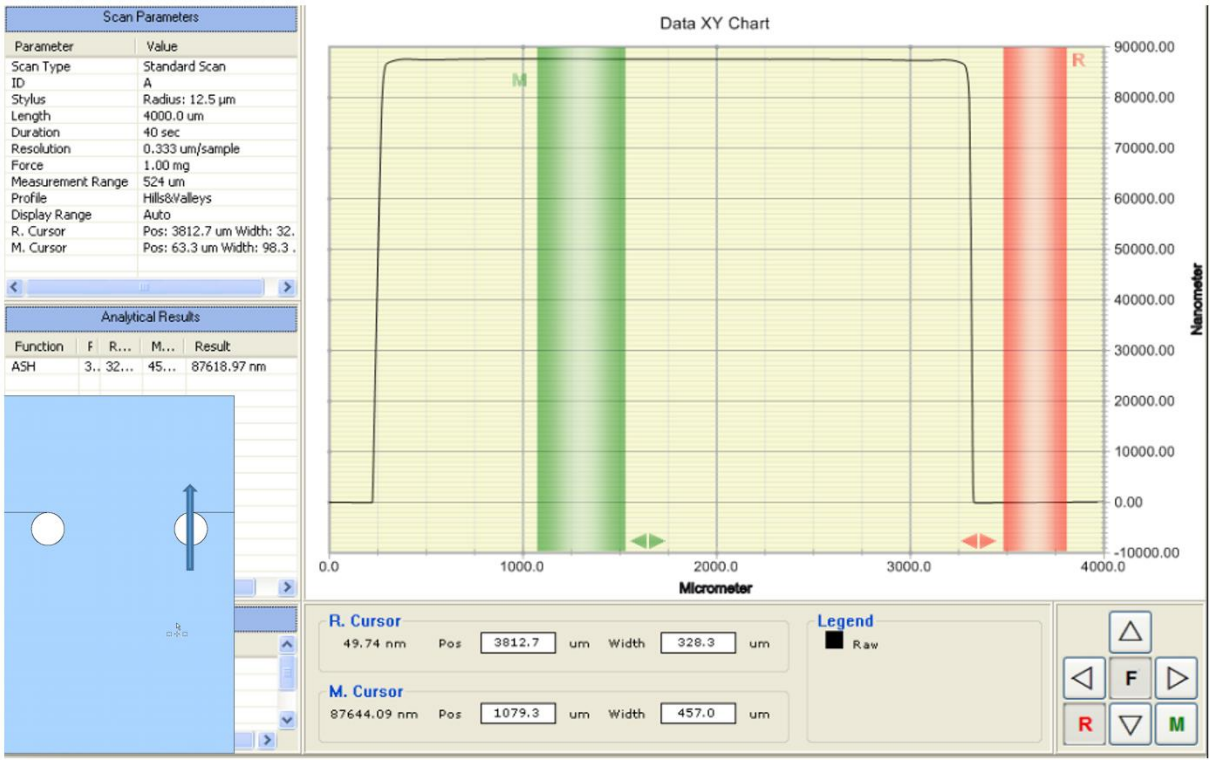


Minimum 89µm and maximum 92µm



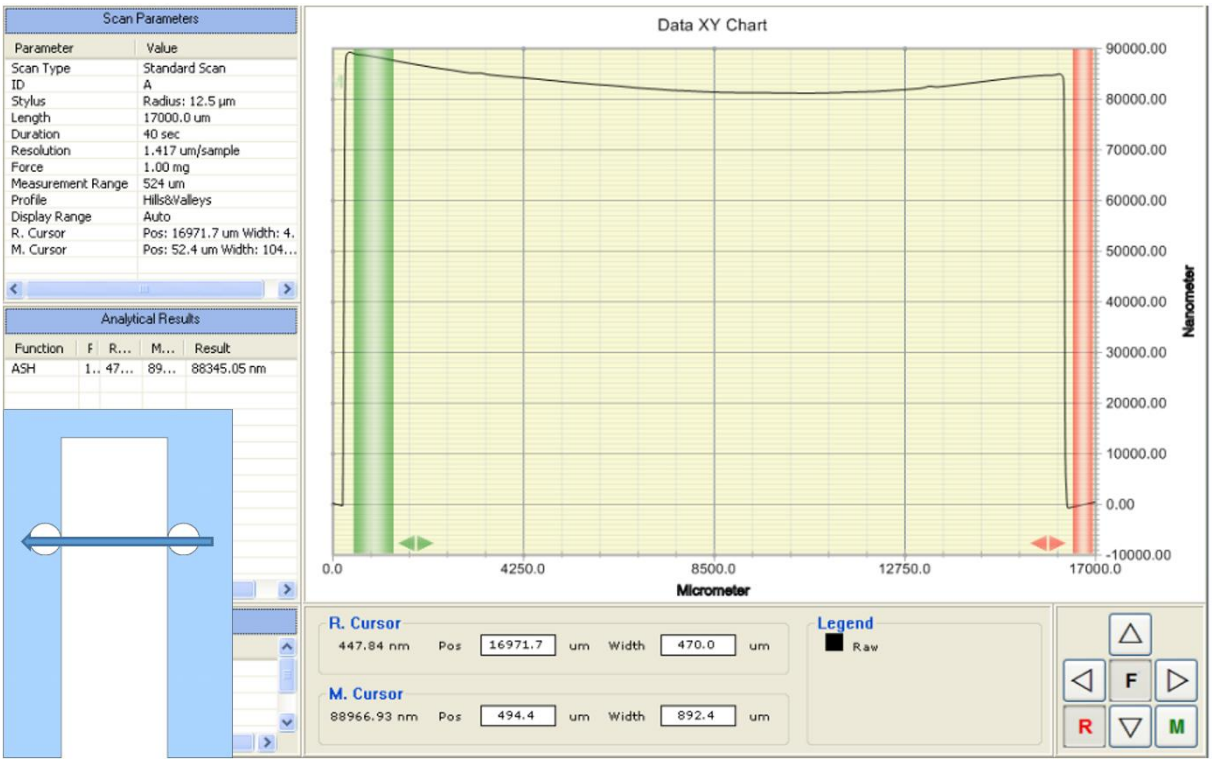
Minimum 88µm and maximum 89µm

- Design five:



88µm

- Design six:



Minimum 85µm and maximum 89µm

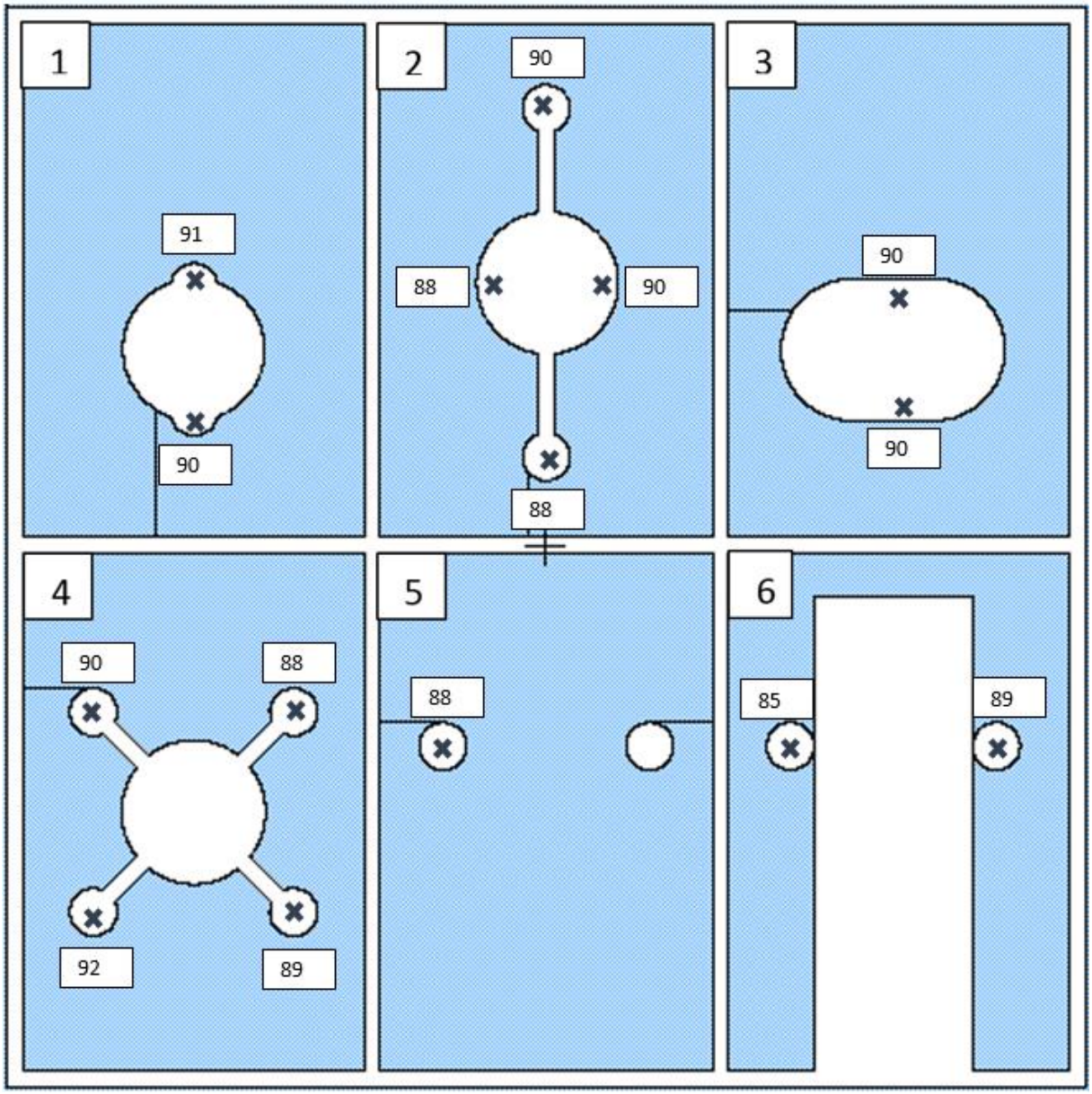


Figure 95. Wafer 2, measuremets

HØGSKOLEN I BUSKERUD OG VESTFOLD

7.9.3 Exercise 3

Laboratory exercise, Mars 23, 2015

Negative photoresist SU-8 100

Procedure. We followed this: http://www.microchem.com/pdf/SU8_50-100.pdf

First, all the machines have to be turned on. VERY IMPORTANT! REMEMBER THE PRESSURE VALVE IN THE CORRIDORE! Always clean all the surroundings. USE PROTECTION GLASES!

Introduction:

The intention with this laboratory exercise is to learn the process of creating a master using SU-8 100 structure on a wafer. The idea is to follow the procedure recommended by MicroChem to create one wafer with 250µm thick structure. Hard baking will be tried again to strengthen the structures.

Materials, chemicals and equipment:

- The glass mask designed with L-edit
- Isopropanol (IPA)
- Acetone
- Silicon wafer, 4”
- SU-8 100 negative photoresist
- Remover for SU-8 100
- Fume hood 4 for general solvents
- Fume hood 5 for corrosive solvents
- Spinner 1 Semitool 1
- Mask Aligner Karl Suss MA56
- Optical microscope I Leika DM4000M
- Hot plate

Procedure:

29. Cleaning wafer. Always finish with Ni.
    29.1. Clean with acetone and rinse with isopropyl (IPA)
    29.2. After drying with Ni, bake on 200ºC during 10 minutes. Take it to the spinner quickly and spincoat as quick as possible. It might absorb humidity while cooling.
30. Coating the photoresist. We aimed for a layer of 250µm.
    30.1. Coat bowl and shield with aluminum foil to protect it from the SU-8.
    30.2. Set up the timer the following way: The instructions are on the side of the spincoater.

TABLE XXXIX Spin coating parameters

Step	Speed (rpm)	Accelerate (sec)	Time (s)	Decelerate (s)
1	0	0	0	-
2	500	5s (500/5=100)	10	-

3	900	10 s	60	30

30.3. Place the wafer as centered as possible.

30.4. Pour the SU-8 carefully in the center, IMPOTANT! It is very viscous, so when it gets to the entrance of the bottle, slow the fluid by inclining the bottle. Try to control the flow by turning the bottle. Try to stop when you have a diameter =4cm approx.

30.5. Clean the bottleneck with IPA and place in the box.

30.6. Cover with the lid and start the program.

30.7. Use needle to gently remove the bubbles.

31. Soft bake. To eliminate residual solvent. Use alumina foil on the hot plate. Add 10ºC to compensate. Let the resist cool down so that it hardens before pattern transfer.

TABLE XL Soft bake parameters

Temperature (ºC)	Time (min) 250µm
75	30
105	150

32. Pattern transfer using designed mask for the mask aligner. We measured the light intensity, ≈9mW/cm^2. C=8.5, T=6, B=12, R=8.5 and L=9. The table in the instruction manual showed that the exposure energy should be approx. 600 mJ/cm^2. 600:9=67 sec. (J=kgm^2/s^2 and W=kgm^2/s^3)

TABLE XLI Exposure parameters

Step	Dose (mJ/cm^2)	Light intensity (mW/ cm^2)	Time (s)
Exposure 250µm	600	8,5	150

33. Post exposure bake. Still use the alumina foil and augment with 10 ºC . Slowly from one temperature to the other, and let cool slowly on the hot plate to avoid cracks in the SU-8.

TABLE XLII Post exposure bake parameters

Temperature (ºC)	Time (min) 250µm
75	1
105	20

34. Turn off the hot plate and leave the wafer until it reach approx. 75 ºC
35. Table for Develop:

TABLE XLIII Development time

Thickness	Development
250µm	20 min

36. Immerse the wafer in the developer. Move the wafer slowly in the developer. The estimated time is 20 minutes, but it took 5 more minutes, leaving a total of 25 minutes.
37. Rinse the wafer with IPA and blow dry with Ni.
38. Hard bake at 200 for approximately 10 minutes. Cool down very slowly. Let the wafer rest on the hot plate for approximately 30 minutes to assure the temperature has reach less than 75 ºC.
39. Clean everything and put the chemicals away. Throw used chemicals in waste bottle.

Results and discussion:

The planned thickness was 250µm. It seems to be a difficult task to manage a uniform thickness, therefor I was very careful to place the SU-8 perfectly in the center.

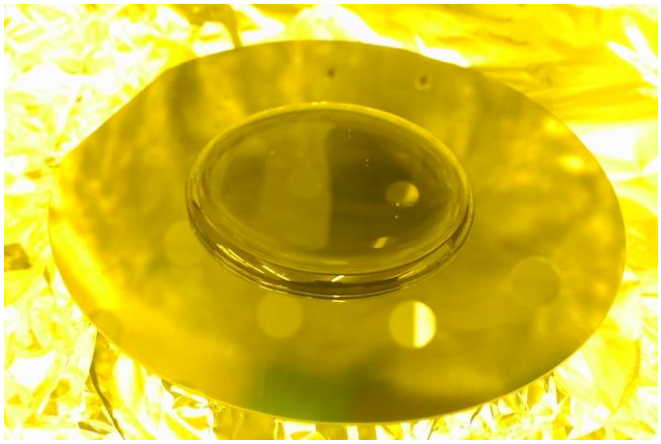


Figure 96 Colocation of the SU8 before spinning

I also managed to place the wafer quite centered in the spin coater.

After I finished the soft baking, I could see that the wafer seemed a bit thicker on one side, and it seemed to have relation with inclination the hot plate. Next time I am going to try with the other hot plate.

Because of the thickness on one of the side, the wafer was a bit stuck to the mask. This did not affect the designs.

It took 25 minutes to develop the master.

Hard bake. Last time I left the wafer to cool on the hot plate about 5 minutes, and it broke when I lifted it off because of the change of temperature. This time I let the wafer cool for 30 minutes on the hot plate. I lifted it off onto a tray, and finally guarded it in a box. When I looked at it a few days later, it was broken.

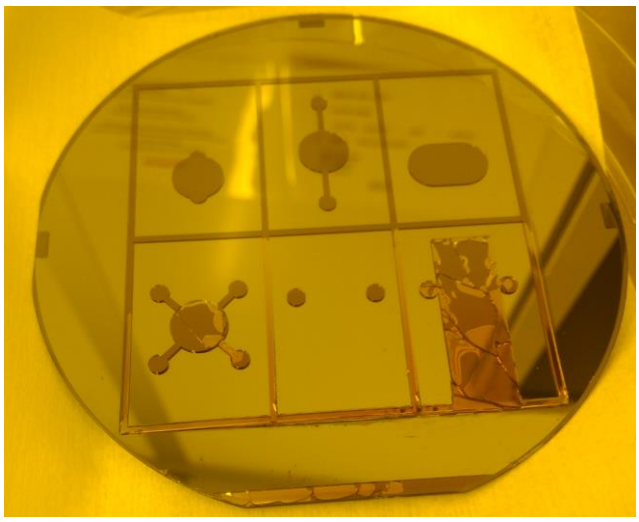


Figure 97 Image of the broken master

Again it had broken where the resist was thickest.

This means that I have to be careful when hard baking thick SU-8 100. From the measurements, the part that had broken seamed to measure more than 300µm. I did not want to use the profilometer on

the broken parts to avoid damaging the needle. Next time I will not hard bake the master. It should not be necessary to hard bake if the master is not meant to be used with acids or other abrasive chemicals.

Visual inspection with the optical microscope:

Pictures of the breakage taken with the optical microscope:

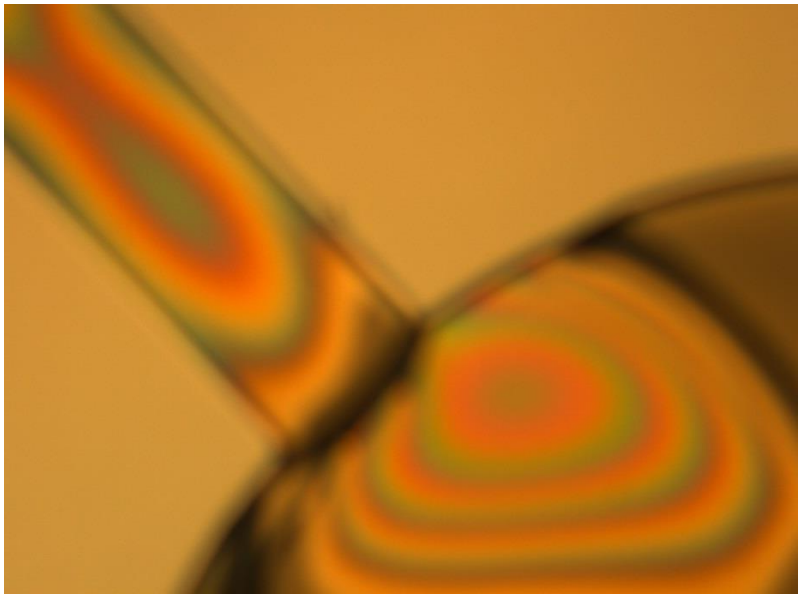


Figure 98 Design 4 Output low right

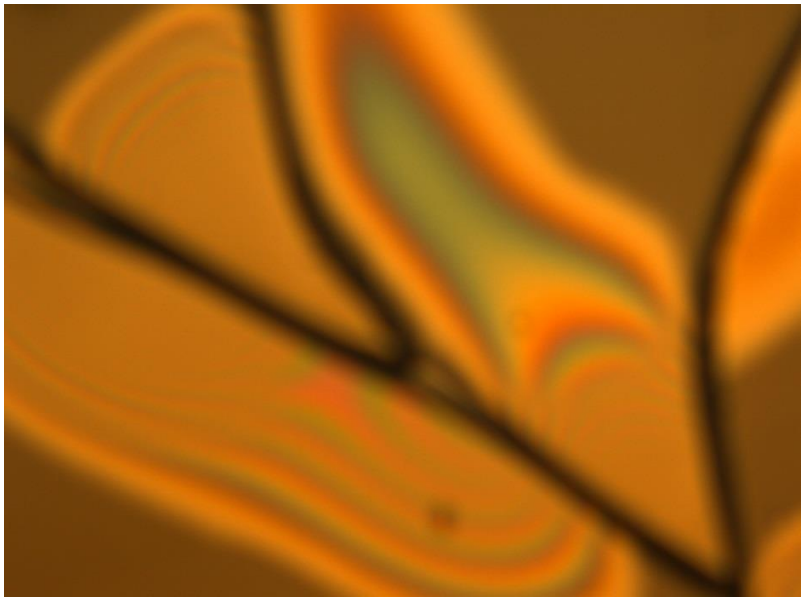


Figure 99 Design 6 Center

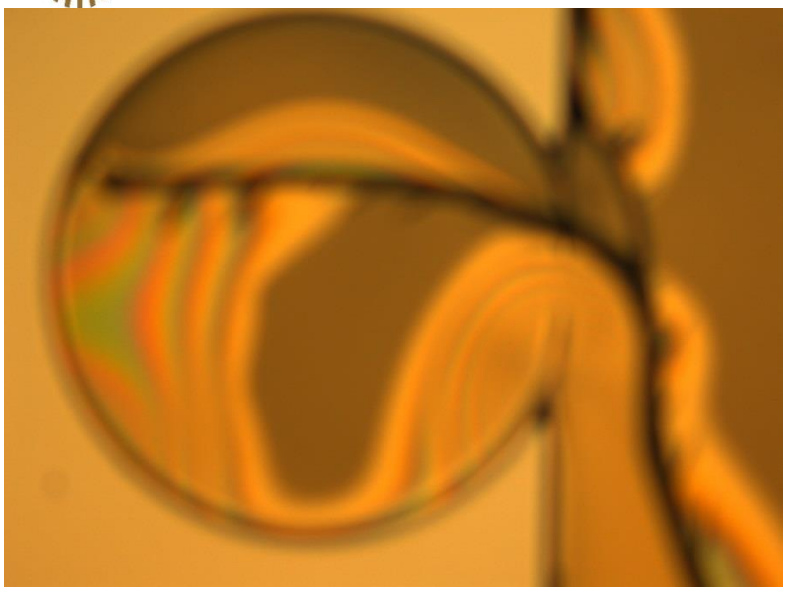


Figure 100 Design 6 Left outlet

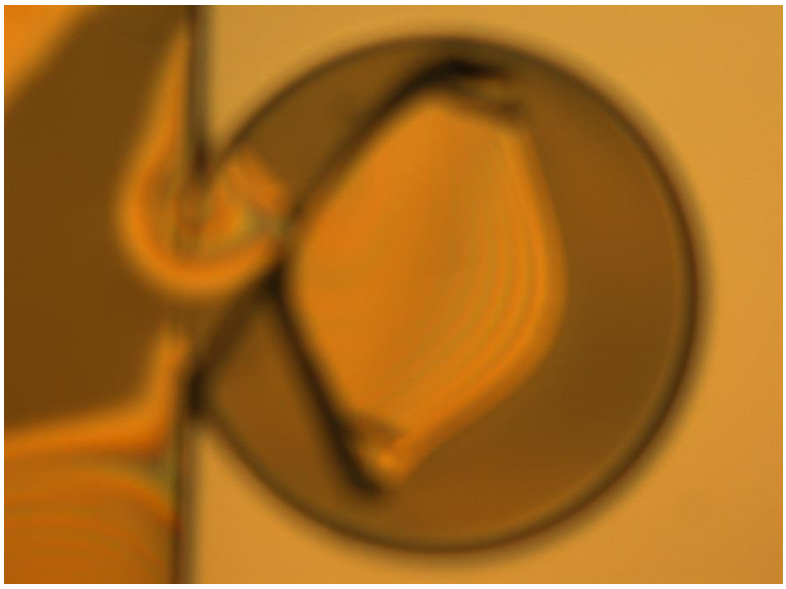
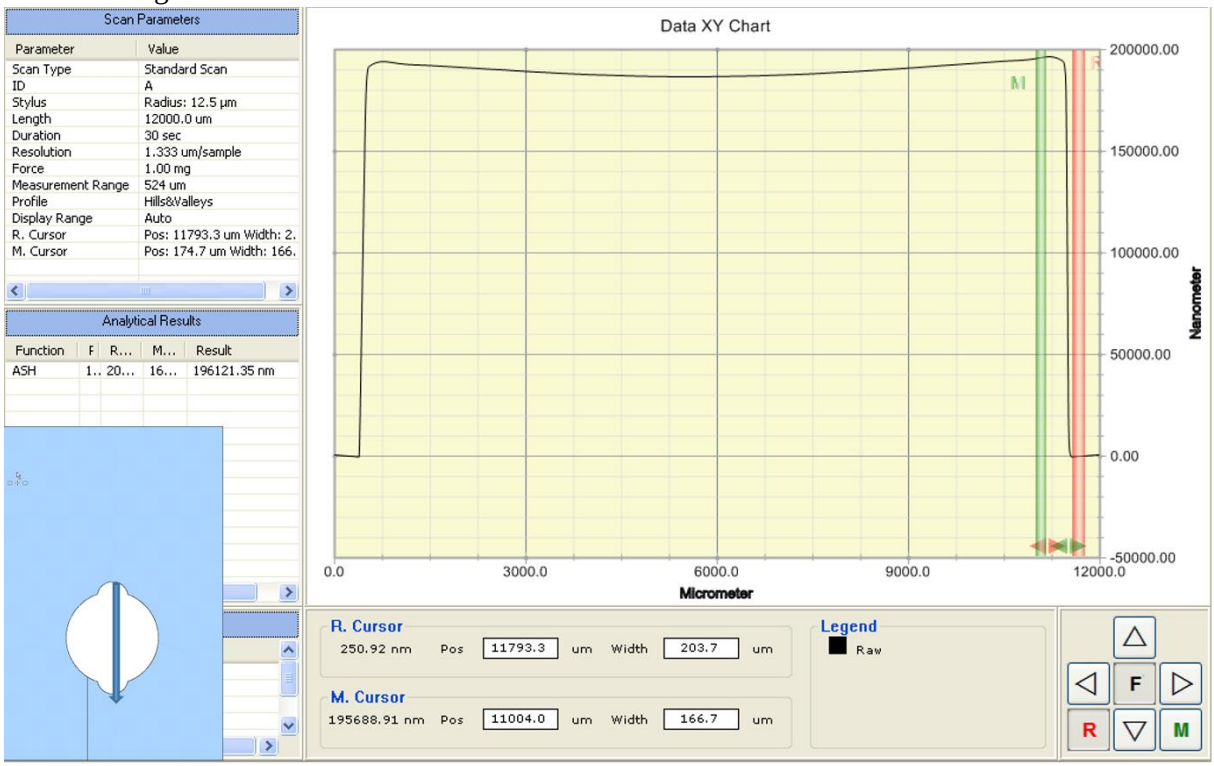


Figure 101Design 6 Right outlet

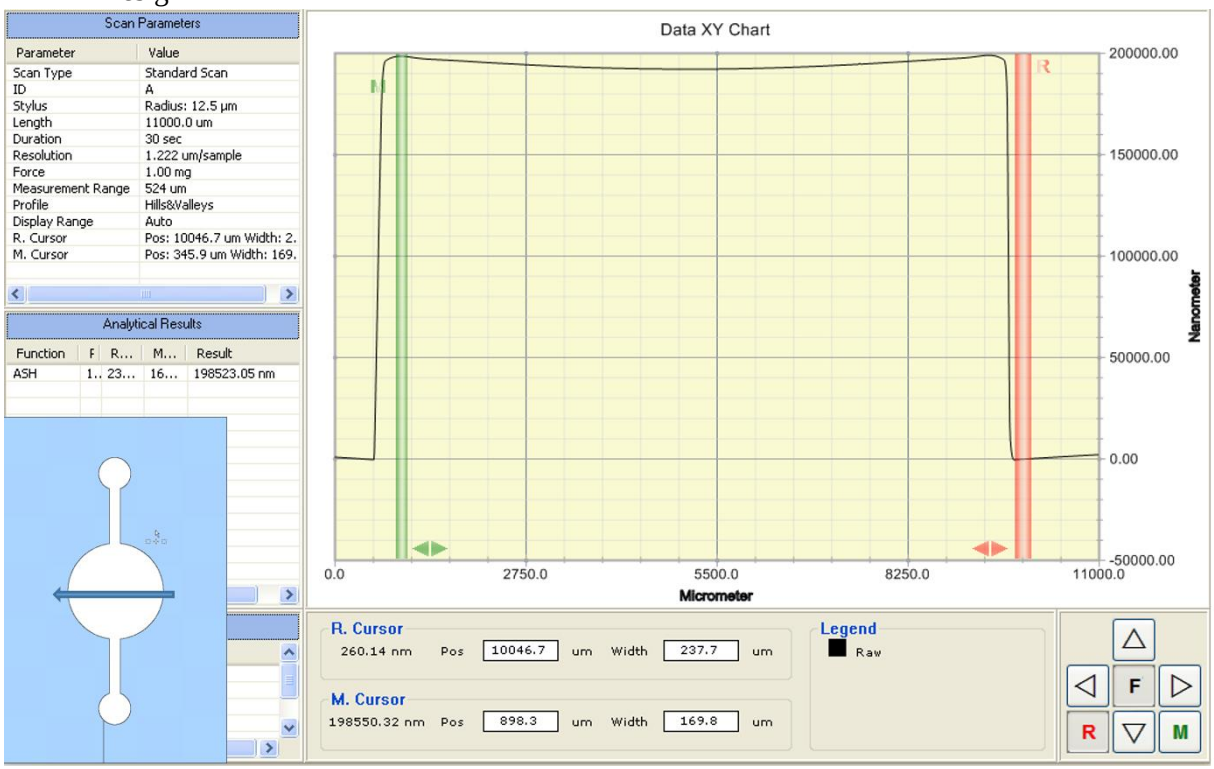
Measurements:

- Design 1

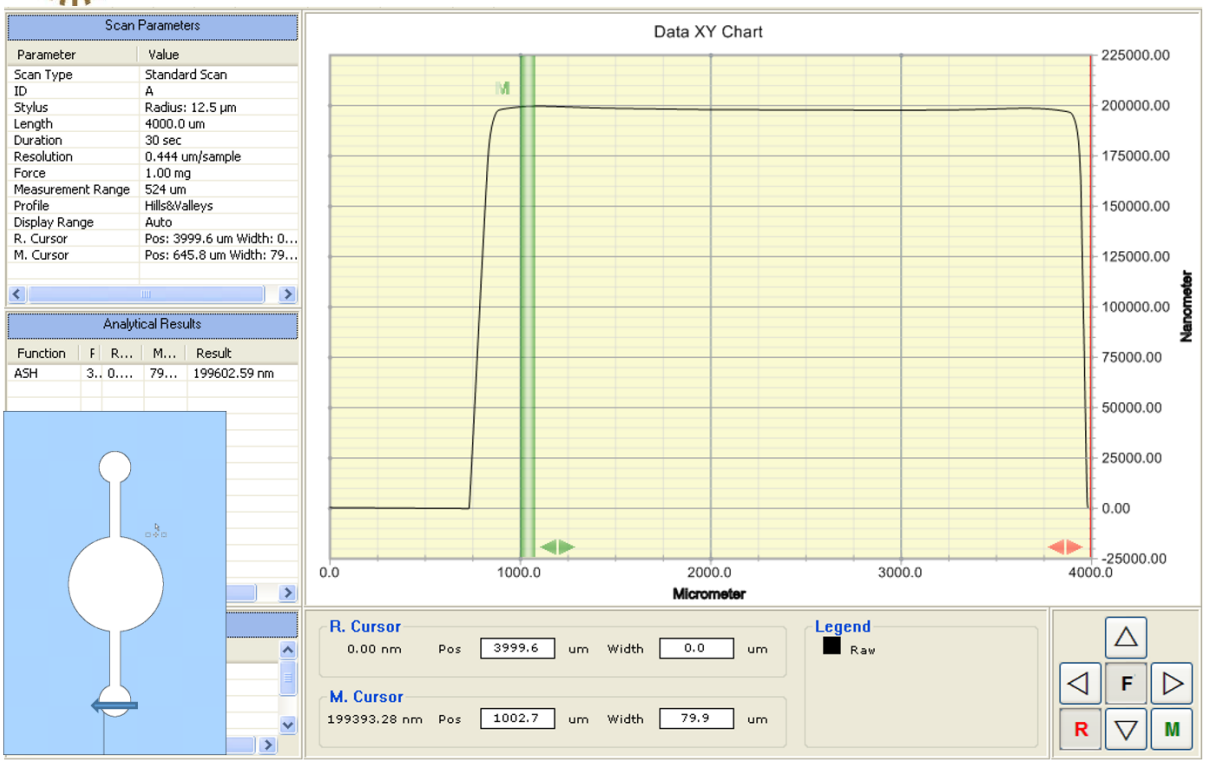


Minimum 185Mm and maximum 195µm

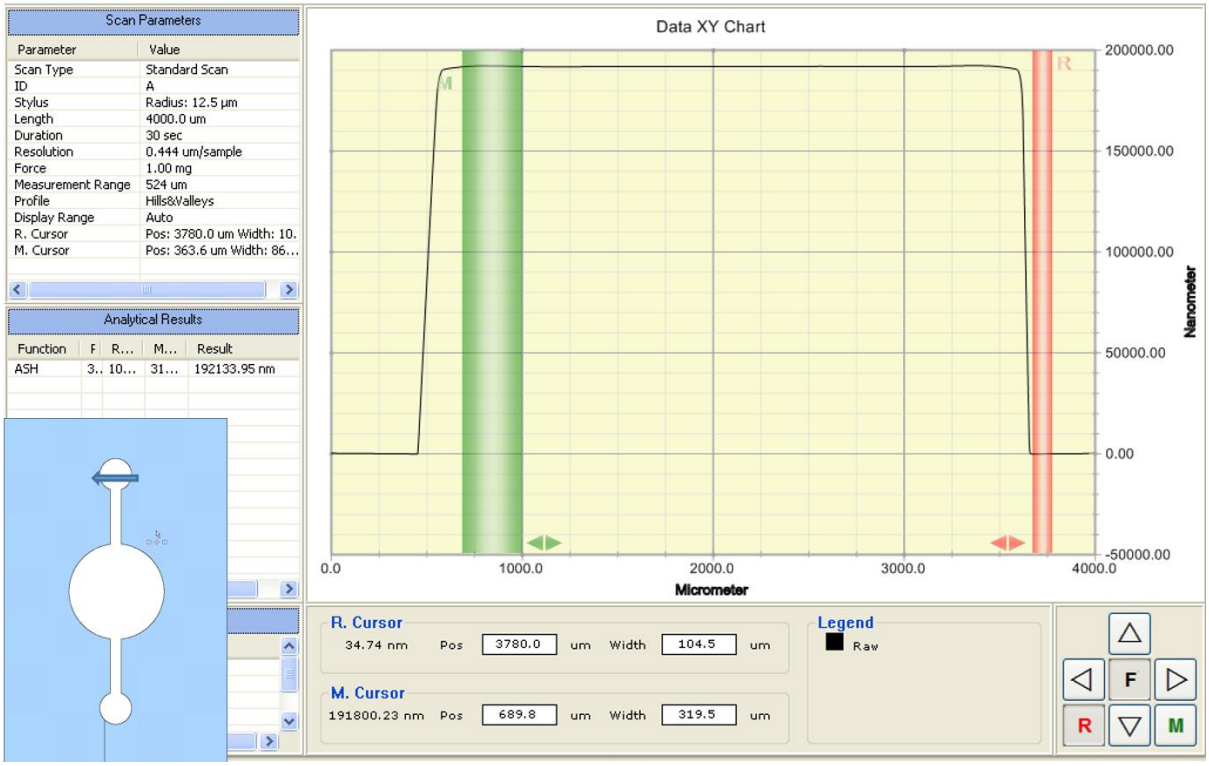
- Design 2



Minimum 190µm and maximum 200µm

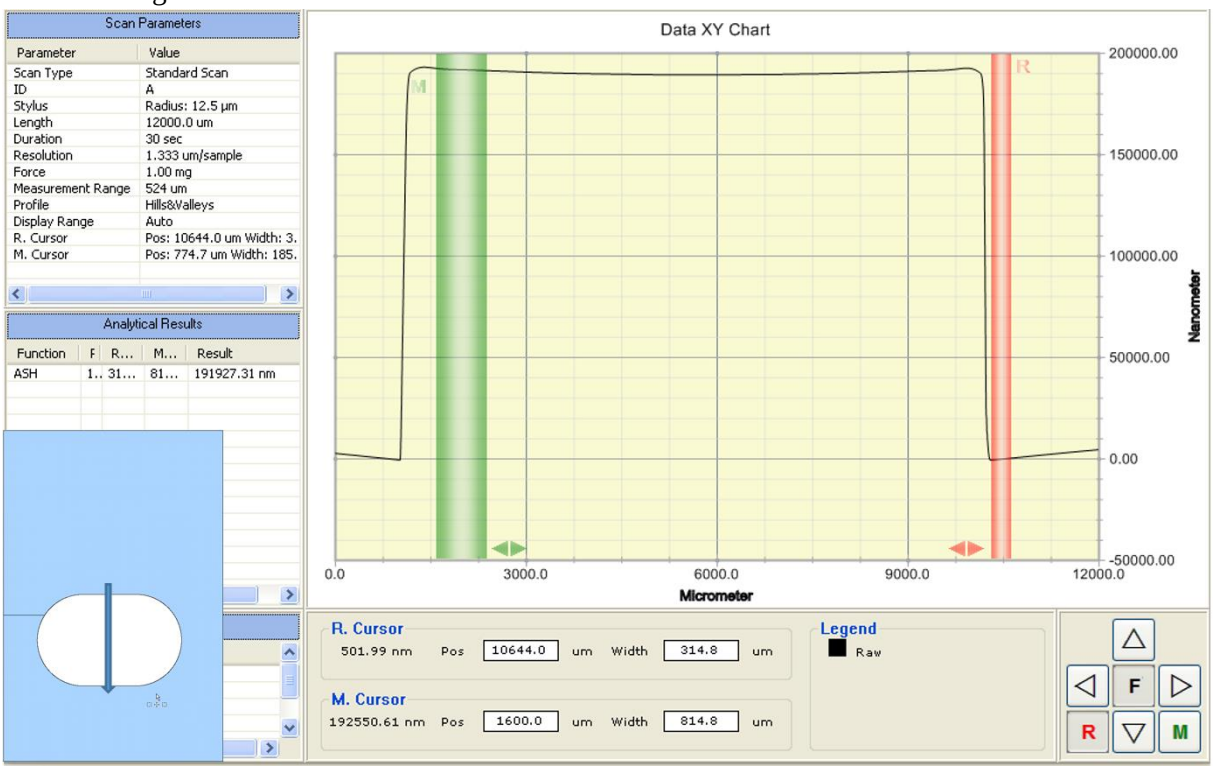

Scan Parameters
Parameter Value
Scan Type Standard Scan
ID A
Stylus Radius: 12.5 µm
Length 4000.0 um
Duration 30 sec
Resolution 0.444 um/sample
Force 1.00 mg
Measurement Range 524 um
Profile Hills&Valleys
Display Range Auto
R. Cursor Pos: 3999.6 um Width: 0...
M. Cursor Pos: 645.8 um Width: 79...
Analytical Results
Function F R... M... Result
ASH 3.. 0.... 79... 199602.59 nm
Data XY Chart
225000.00
200000.00
175000.00
150000.00
125000.00
100000.00
75000.00
50000.00
25000.00
0.00
-25000.00
Nanometer
0.0
1000.0
2000.0
3000.0
4000.0
Micrometer
R. Cursor
0.00 nm Pos 3999.6 um Width 0.0 um
M. Cursor
199393.28 nm Pos 1002.7 um Width 79.9 um
Legend
Raw
F
R
M

200µm


Scan Parameters
Parameter Value
Scan Type Standard Scan
ID A
Stylus Radius: 12.5 µm
Length 4000.0 um
Duration 30 sec
Resolution 0.444 um/sample
Force 1.00 mg
Measurement Range 524 um
Profile Hills&Valleys
Display Range Auto
R. Cursor Pos: 3780.0 um Width: 10.
M. Cursor Pos: 363.6 um Width: 86...
Analytical Results
Function F R... M... Result
ASH 3.. 10... 31... 192133.95 nm
Data XY Chart
200000.00
150000.00
100000.00
50000.00
0.00
-50000.00
Nanometer
0.0
1000.0
2000.0
3000.0
4000.0
Micrometer
R. Cursor
34.74 nm Pos 3780.0 um Width 104.5 um
M. Cursor
191800.23 nm Pos 689.8 um Width 319.5 um
Legend
Raw
F
R
M

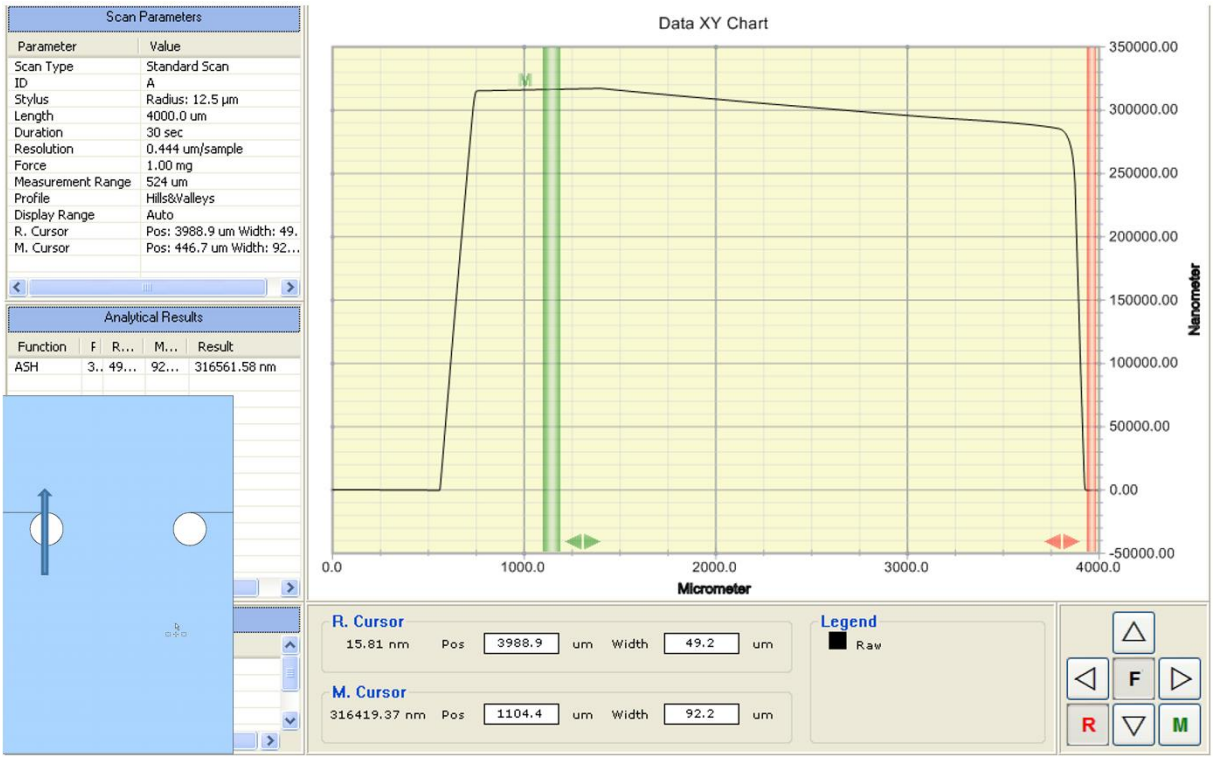
190µm

- Design 3

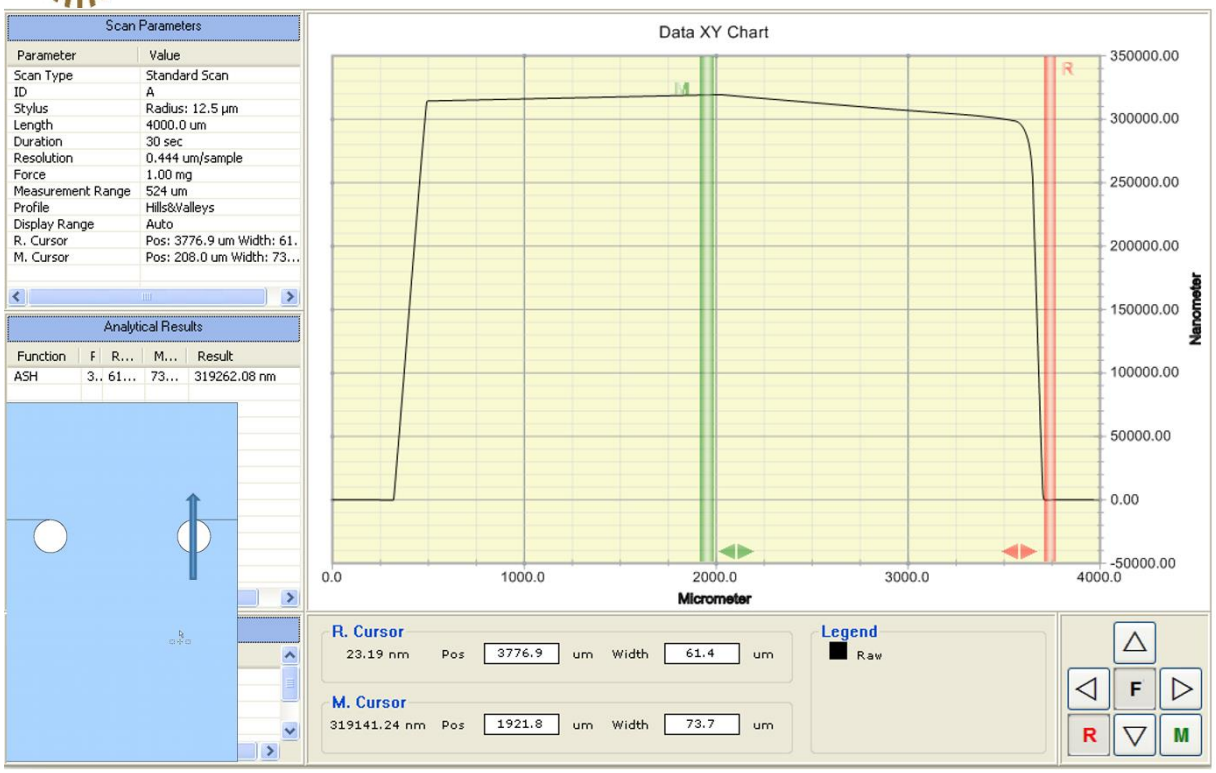


190µm

- Design 5



Minimum 280µm and maximum 320µm



Minimum 300µm and maximum 320µm

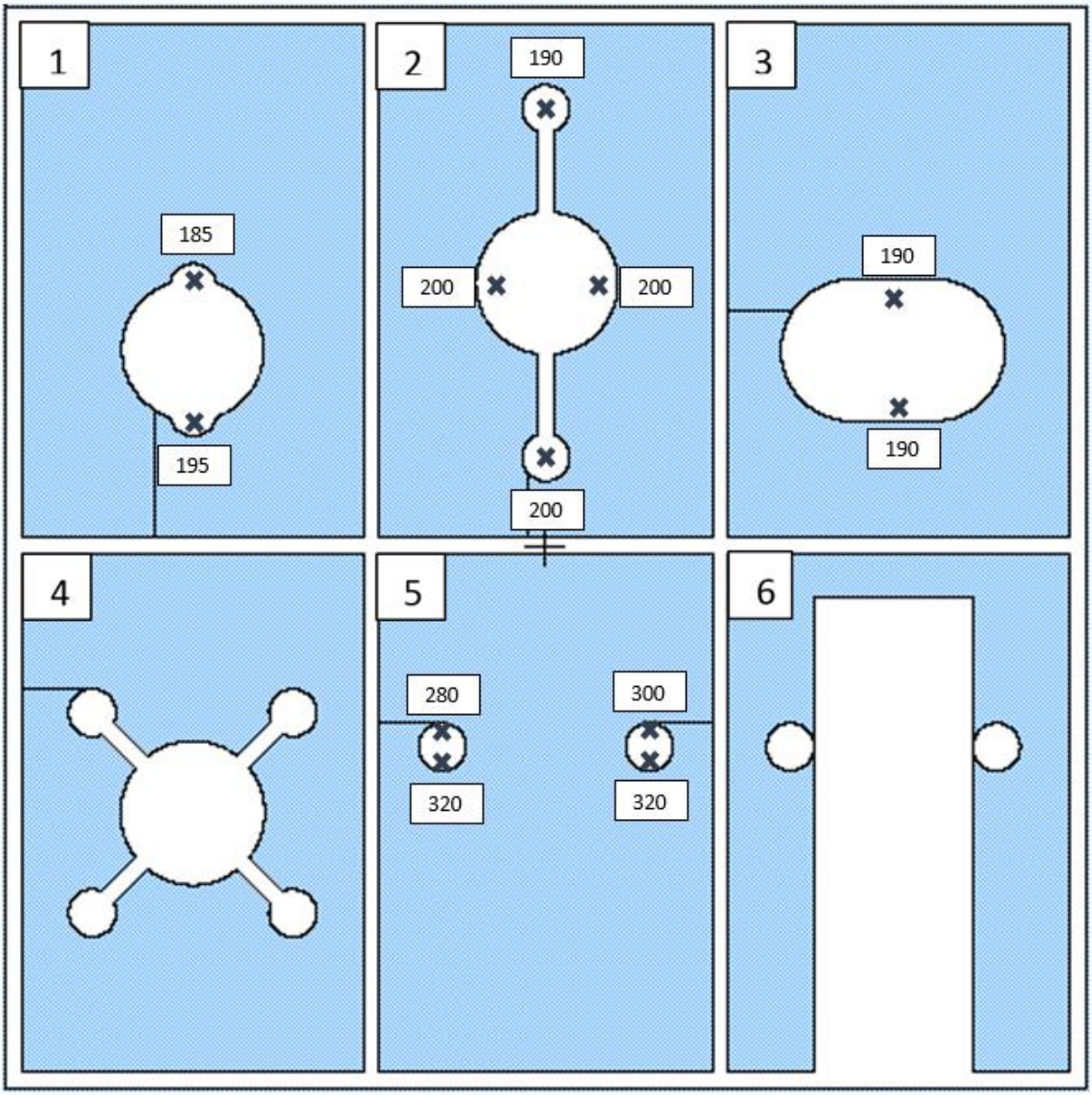


Figure 102. Measurements

HØGSKOLEN I BUSKERUD OG VESTFOLD

7.9.4 Exercise 4

Laboratory exercise, Mars 26, 2015

Negative photoresist SU-8 100

Procedure. We followed this: http://www.microchem.com/pdf/SU8_50-100.pdf

First, all the machines have to be turned on. VERY IMPORTANT! REMEMBER THE PRESSURE VALVE IN THE CORRIDORE! Always clean all the surroundings. USE PROTECTION GLASES!

Introduction:

The intention with this laboratory exercise is to learn the process of creating a master using SU-8 100 structure on a wafer. The idea is to follow the procedure recommended by MicroChem to create one wafer with 250µm thick structure. Hard baking will be tried again to strengthen the structures.

Materials, chemicals and equipment:

- The glass mask designed with L-edit
- Isopropanol (IPA)
- Acetone
- Silicon wafer, 4”
- SU-8 100 negative photoresist
- Remover for SU-8 100
- Fume hood 4 for general solvents
- Fume hood 5 for corrosive solvents
- Spinner 1 Semitool 1
- Mask Aligner Karl Suss MA56
- DEKTAK profilometer
- Optical microscope I Leika DM4000M
- Hot plate

Procedure:

40. Cleaning wafer. Always finish with Ni.
    40.1. Clean with acetone and rinse with isopropyl (IPA)
    40.2. After drying with Ni, bake on 200ºC during 10 minutes. Take it to the spinner quickly and spin coat as quick as possible. It might absorb humidity while cooling.
41. Coating the photoresist. We aimed for a layer of 250µm.
    41.1. Coat bowl and shield with aluminum foil to protect it from the SU-8.
    41.2. Set up the timer the following way: The instructions are on the side of the spin coater.

TABLE XLIV Spincoating parameters

Step	Speed (rpm)	Accelerate (sec)	Time (s)	Decelerate (s)
1	0	0	0	-

2	500	5s (500/5=100)	10	-
3	900	10 s	60	30

41.3. Place the wafer as centered as possible.

41.4. Pour the SU-8 carefully in the center, IMPOTANT! It is very viscous, so when it gets to the entrance of the bottle, slow the fluid by inclining the bottle. Try to control the flow by turning the bottle. Try to stop when you have a diameter =4cm approx.

41.5. Clean the bottleneck with IPA and place in the box.

41.6. Cover with the lid and start the program.

41.7. Use needle to gently remove the bubbles.

42. Soft bake. To eliminate residual solvent. Use alumina foil on the hot plate. Add 10ºC to compensate. Let the resist cool down so that it hardens before pattern transfer.

TABLE XLV Soft bake parameters

Temperature (ºC)	Time (min) 250µm
75	30
105	150

43. Pattern transfer using designed mask for the mask aligner. We measured the light intensity, ≈9mW/cm^2. C=8.5, T=6, B=12, R=8.5 and L=9. The table in the instruction manual showed that the exposure energy should be approx. 600 mJ/cm^2. 600:9=67 sec. (J=kgm^2/s^2 and W=kgm^2/s^3)

TABLE XLVI Exposure parameters

Step	Dose (mJ/cm^2)	Light intensity (mW/ cm^2)	Time (s)
Exposure 250µm	600	8,5	150

44. Post exposure bake. Still use the alumina foil and augment with 10 ºC . Slowly from one temperature to the other, and let cool slowly on the hot plate to avoid cracks in the SU-8.

TABLE XLVII Post exposure bake parameters

Temperature (ºC)	Time (min) 250µm
75	1
105	20

45. Turn off the hot plate and leave the wafer until it reach approx. 75 ºC
46. Table for Develop:

TABLE XLVIII Development time

Thickness	Development
250µm	20 min

47. Immerse the wafer in the developer. Move the wafer slowly in the developer. The estimated time is 20 minutes, but it took 5 more minutes, leaving a total of 25 minutes.
48. Rinse the wafer with IPA and blow dry with Ni.
49. Clean everything and put the chemicals away. Throw used chemicals in waste bottle.

Results and discussion:

The planned thickness was 250µm. It seems to be a difficult task to manage a uniform thickness, therefor I was very careful to place the SU-8 perfectly in the center.

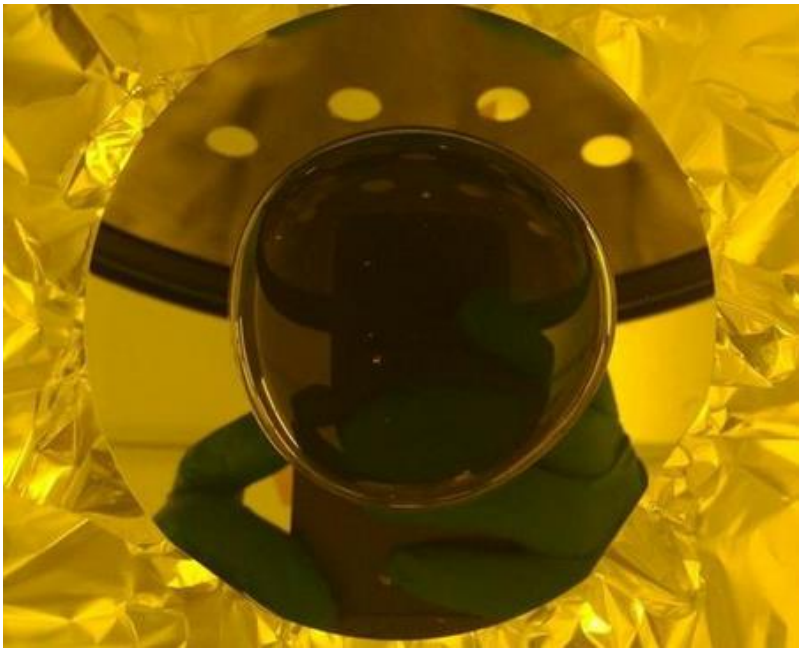


Figure 103 Colocation of the SU8 before spinning

I also managed to place the wafer quite centered in the spin coater.

I used another hot plate for soft baking, but I could still see that the wafer seemed a bit thicker on one side. If I have time to do a new master, I will need to level the hotplate, or move the wafer so the resist does not slide to one of the ends of the wafer.

The measurements done with the profilometer shows that this master is the most uneven by far. Design 4 measured 500µm in one corner and 135 in the opposite corner. That makes the design unsuitable hence the idea of the design was that all the outlets should have the same flow rate. The same problem happens with design 2 as well, going from 120µm on one outlet to 235 on the other outlet. This wafer should not be used as a master

The wafer was very stuck to the mask. I had to remove it carefully and eliminate the rests of the resist on the mask. It did not affect the designs this time either.

I did not hard bake and it seemed to be no need for it.

Visual inspection with the optical microscope:

I used the optical microscope to take some pictures and measure the designs. Due to the size of the designs, I had to use the least amplifying lens. Even so, the largest area I managed to measure was just above 3mm x 3mm. Because of this limitation, I was only able to measure some of the channels and the pads for the outlets.

The results were very similar to the measures used in the drawing of the designs. The measures of the pictures made with the optical microscope are not very precise, but for the use pretended for the designs it seems to be very accurate.

- Design 2

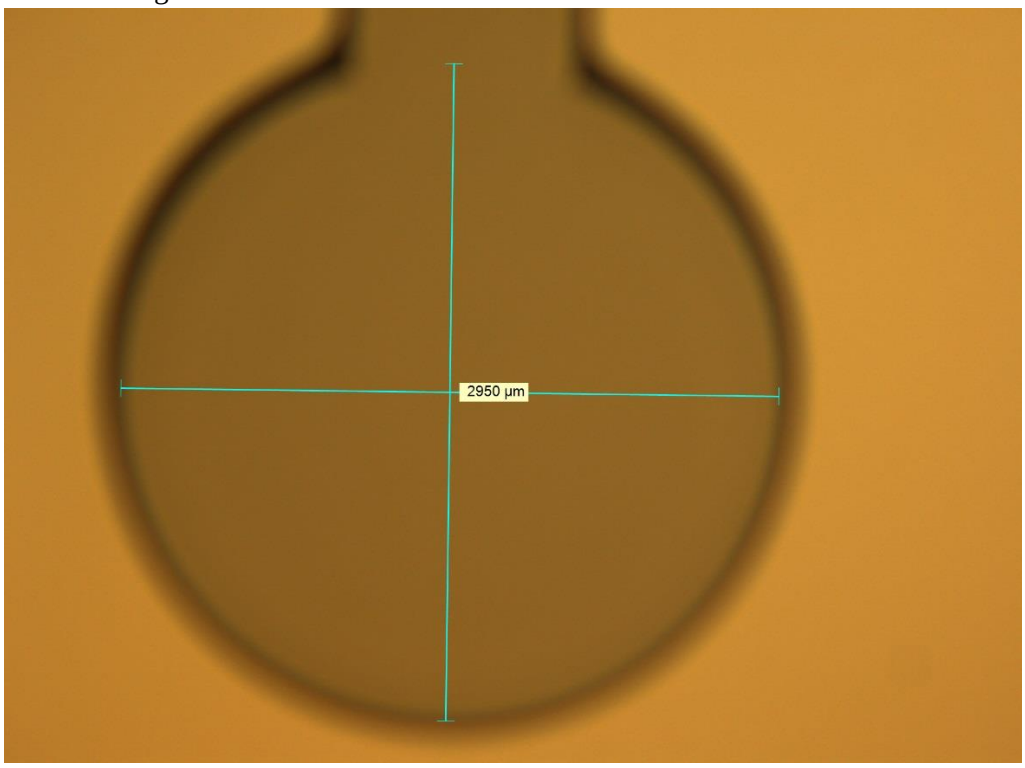


Figure 104 Lower outlet

- Design 4

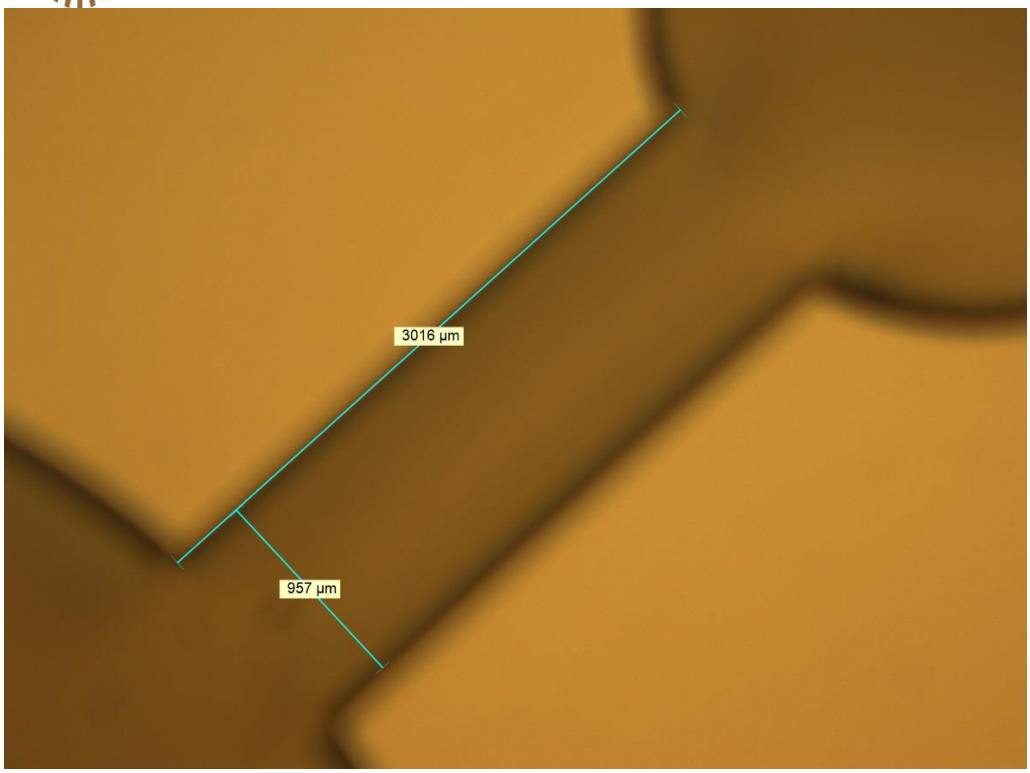


Figure 105 Left lower channel

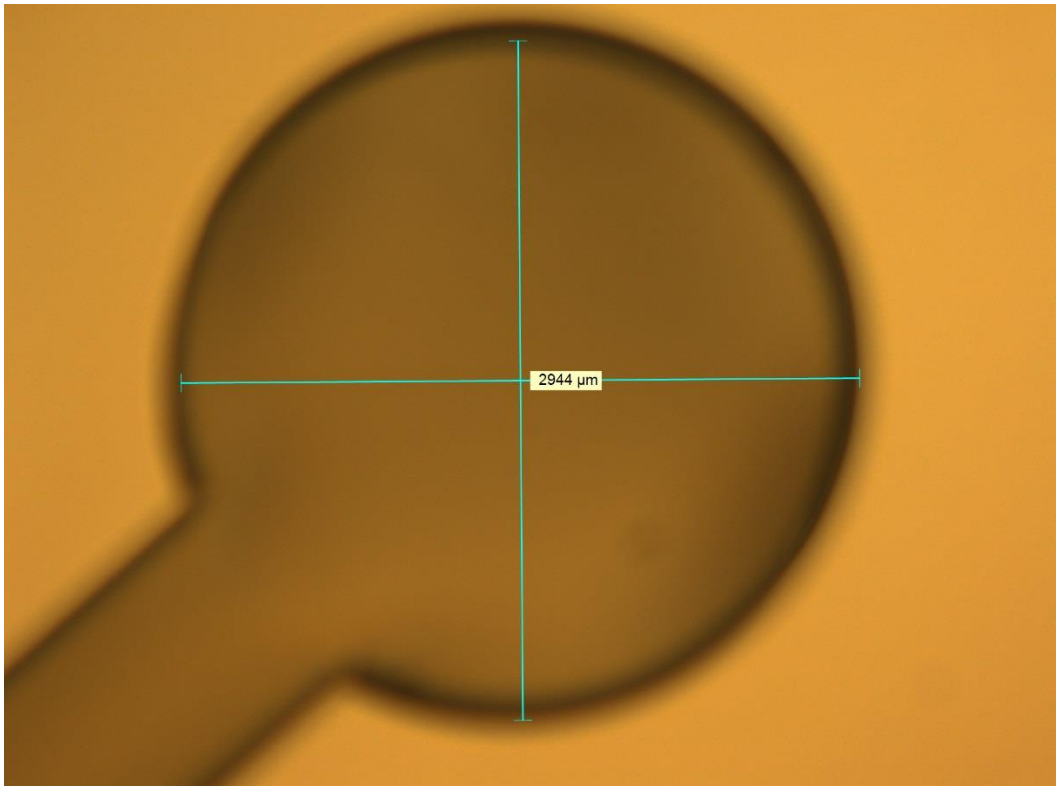


Figure 106 Left lower outlet

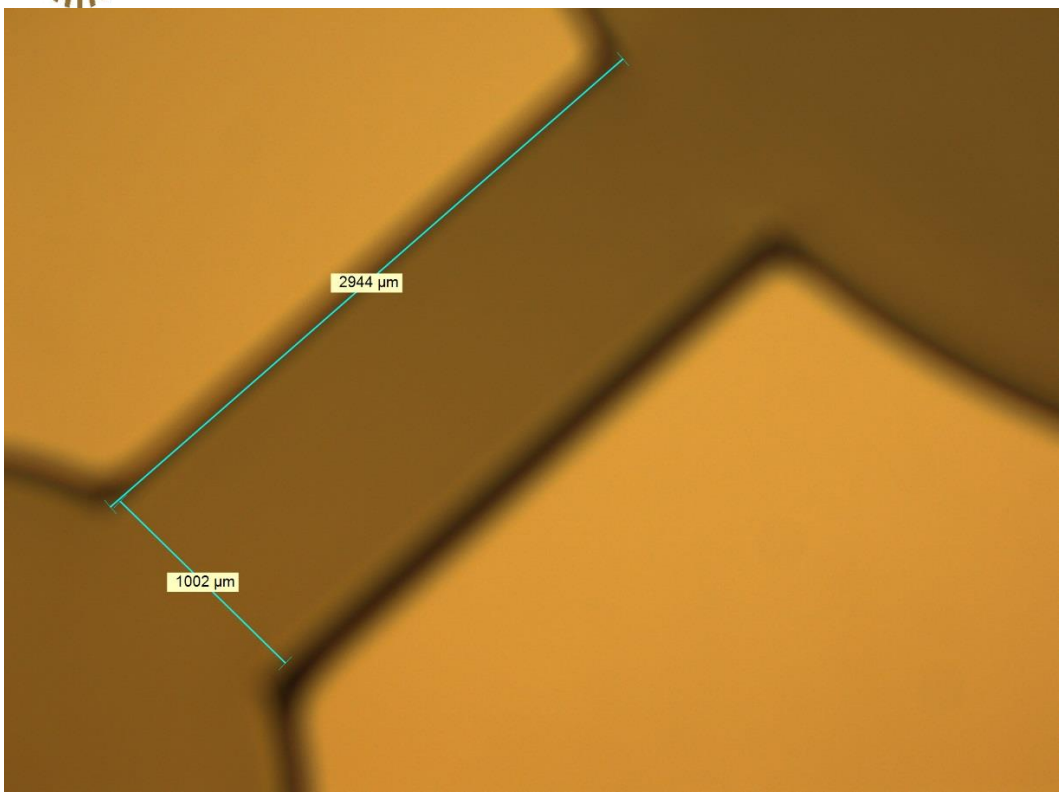


Figure 107 Right upper channel

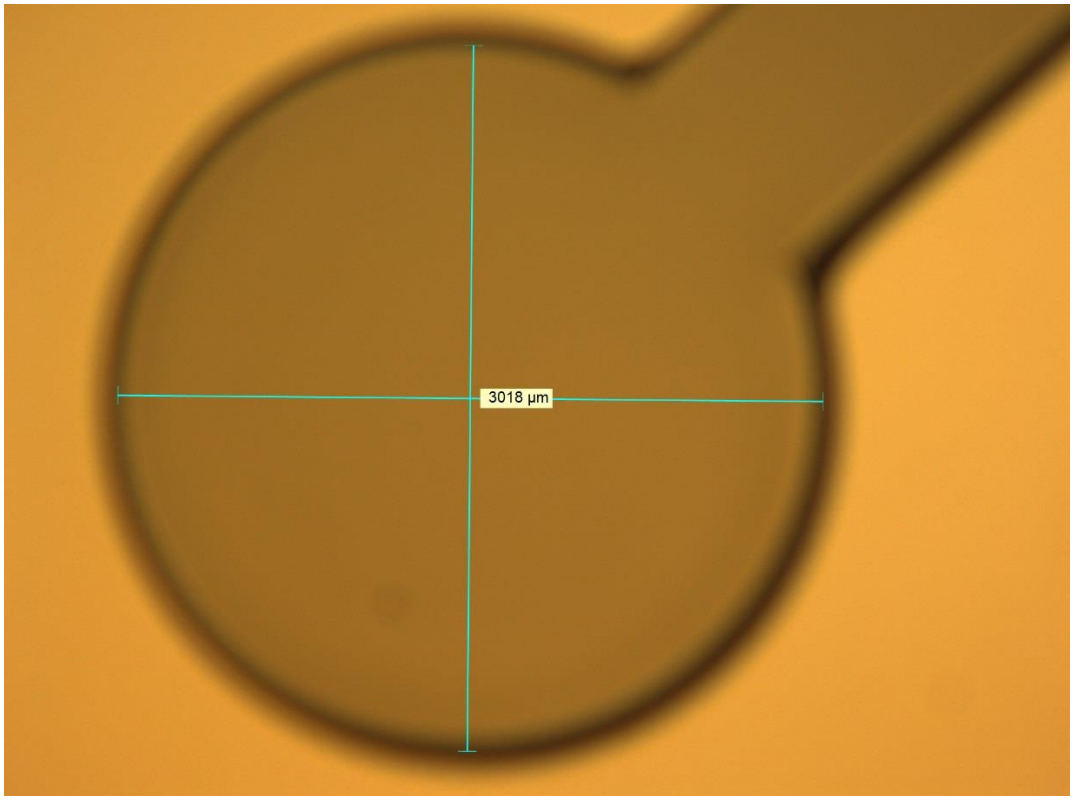


Figure 108 Right upper outlet

- Design 5

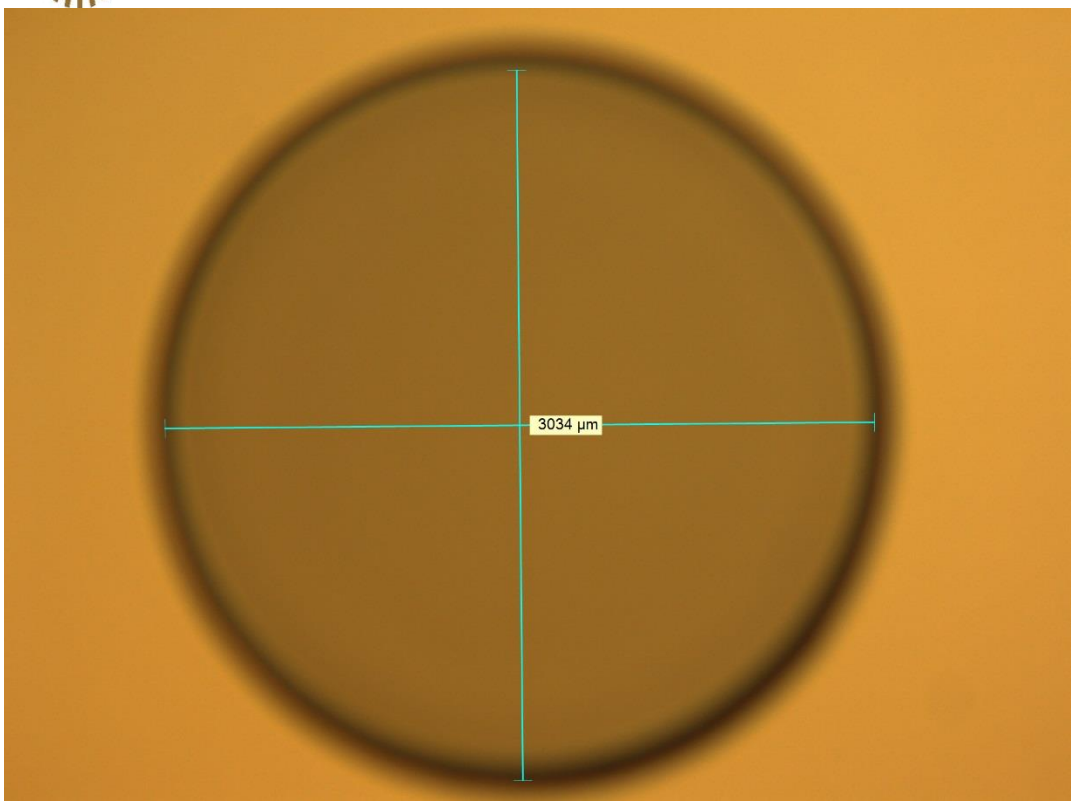


Figure 109 Left outlet

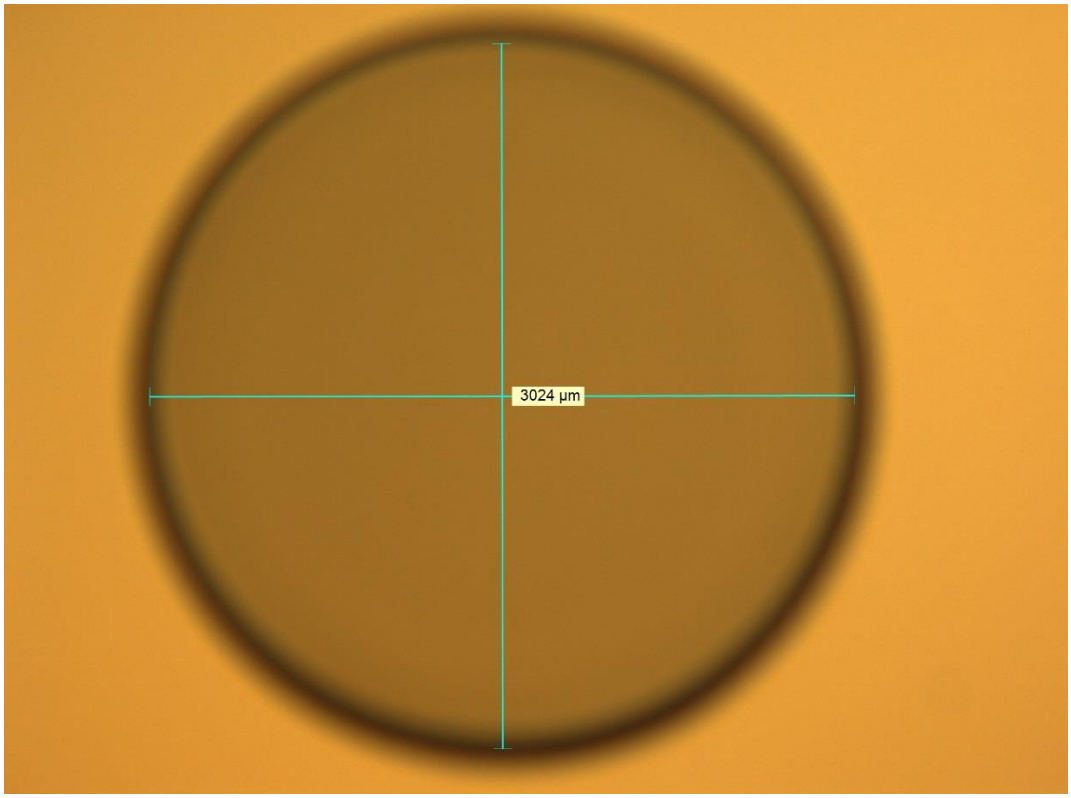


Figure 110 Right outlet

- Design 6

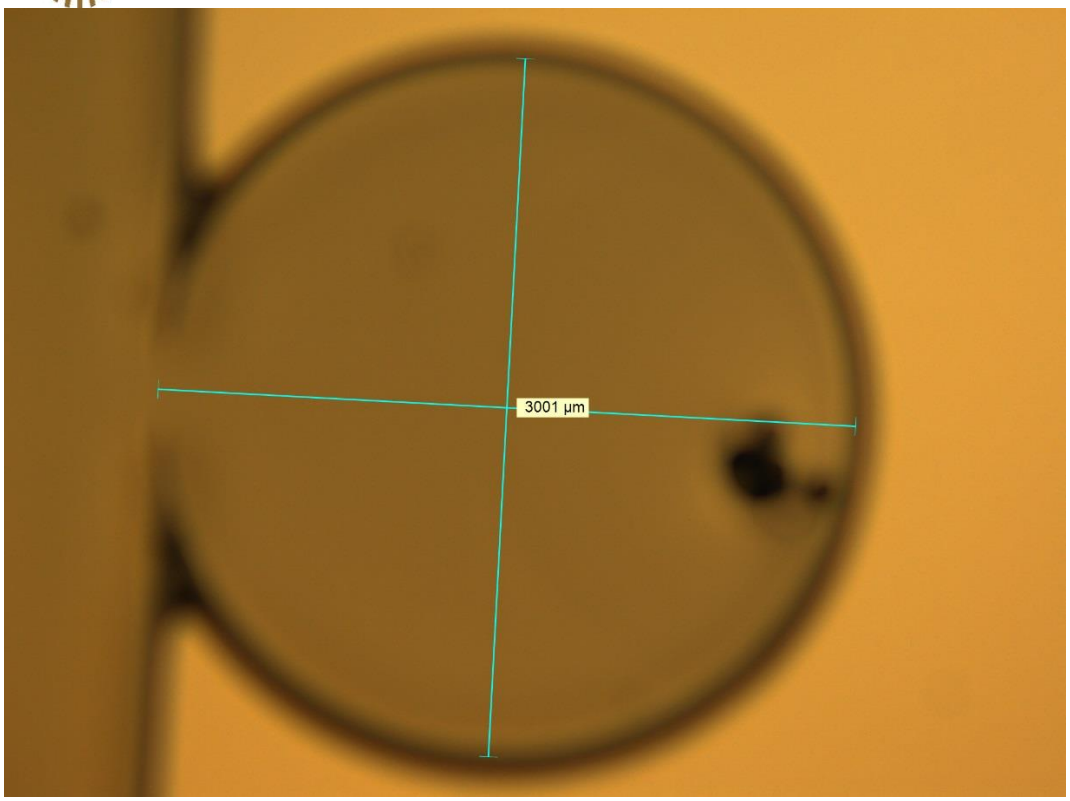


Figure 111 Left outlet with a small flaw

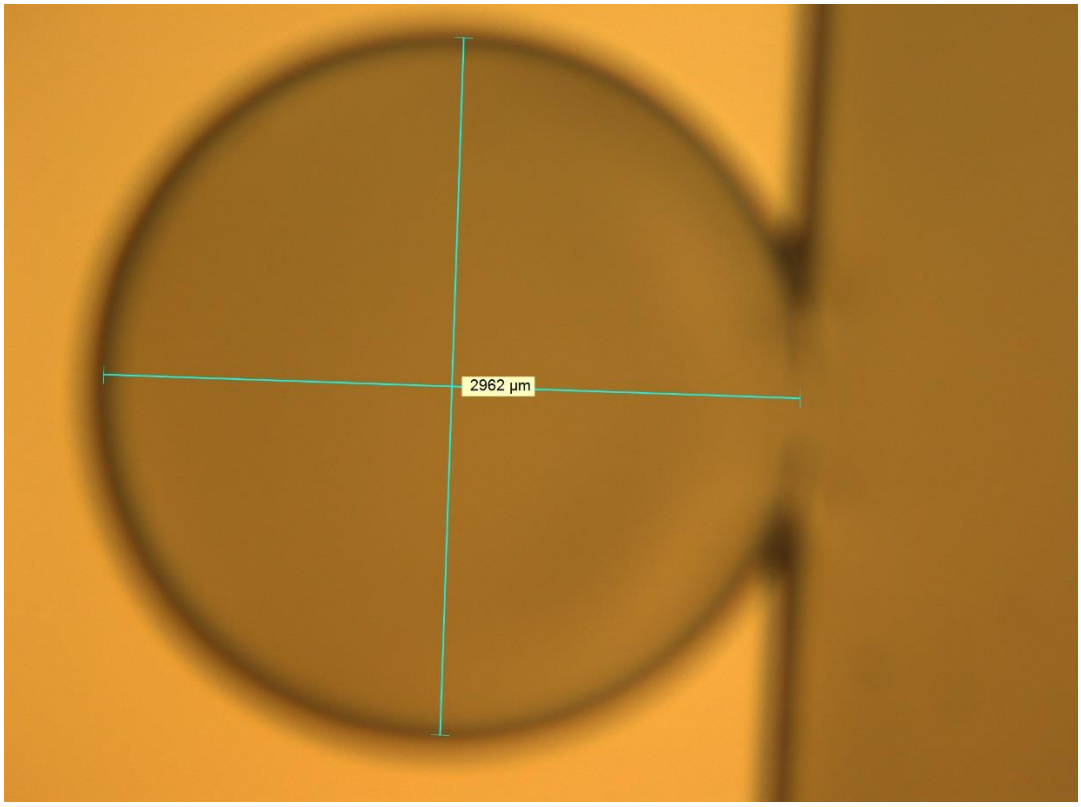
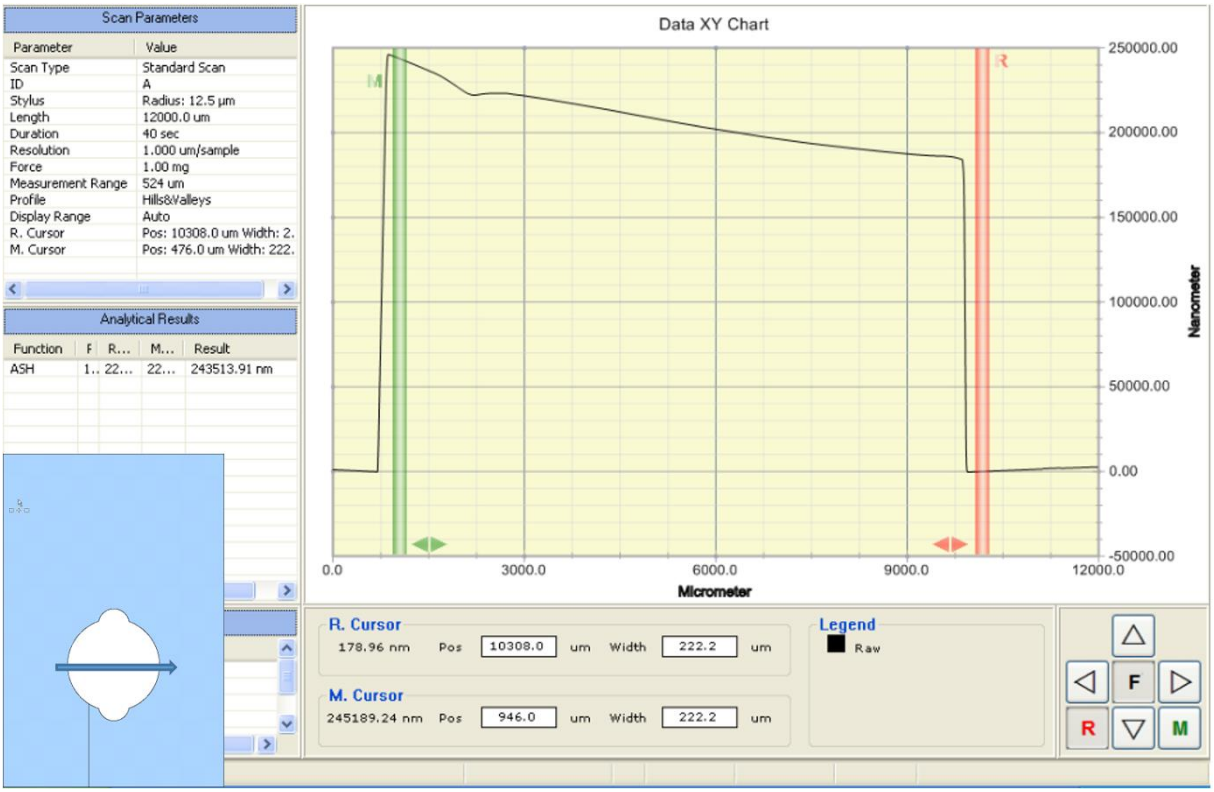


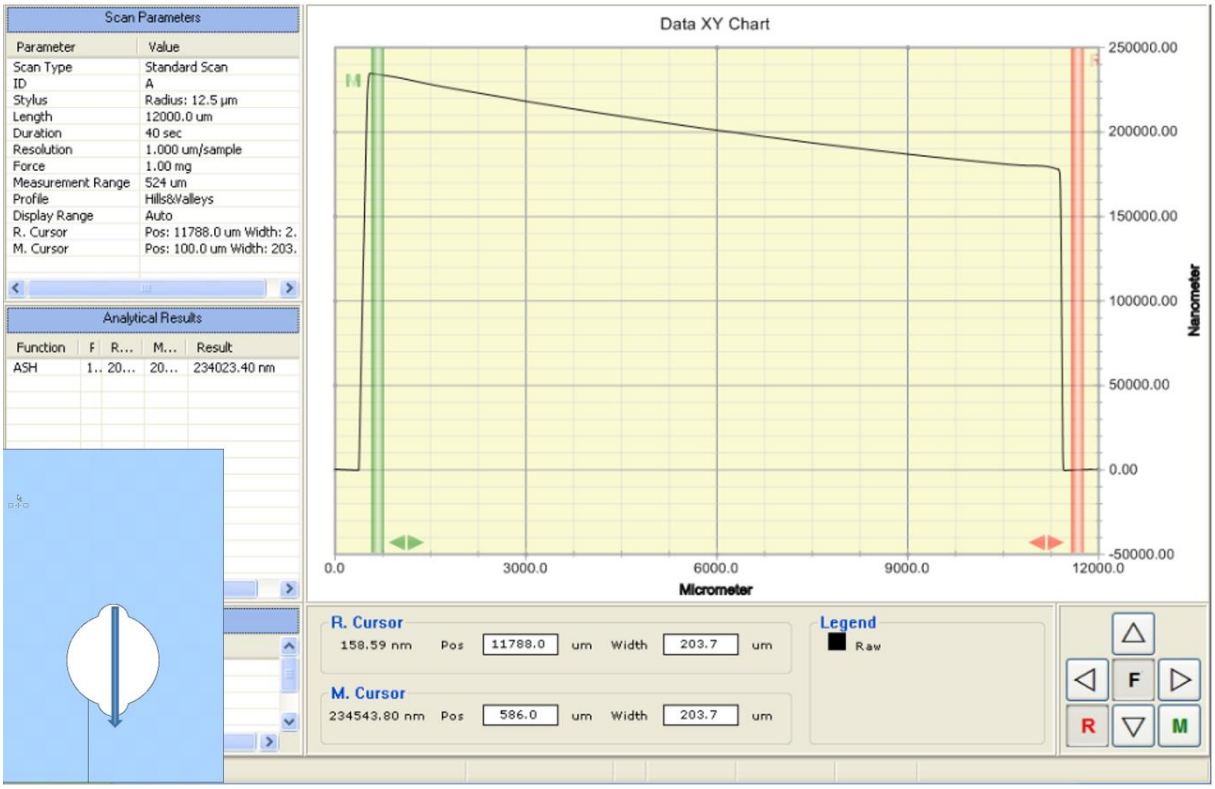
Figure 112 Right outlet

Measurements:

- Design 1

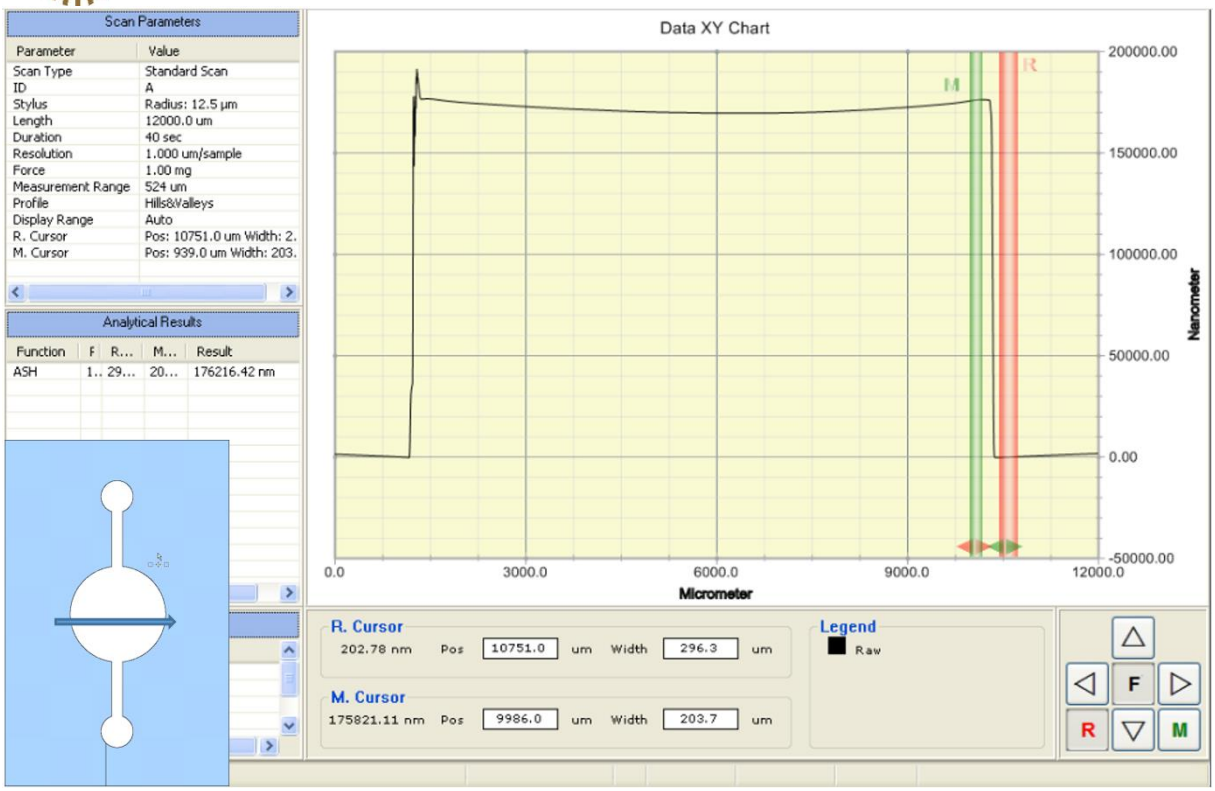


Minimum 185 and maximum 245µm

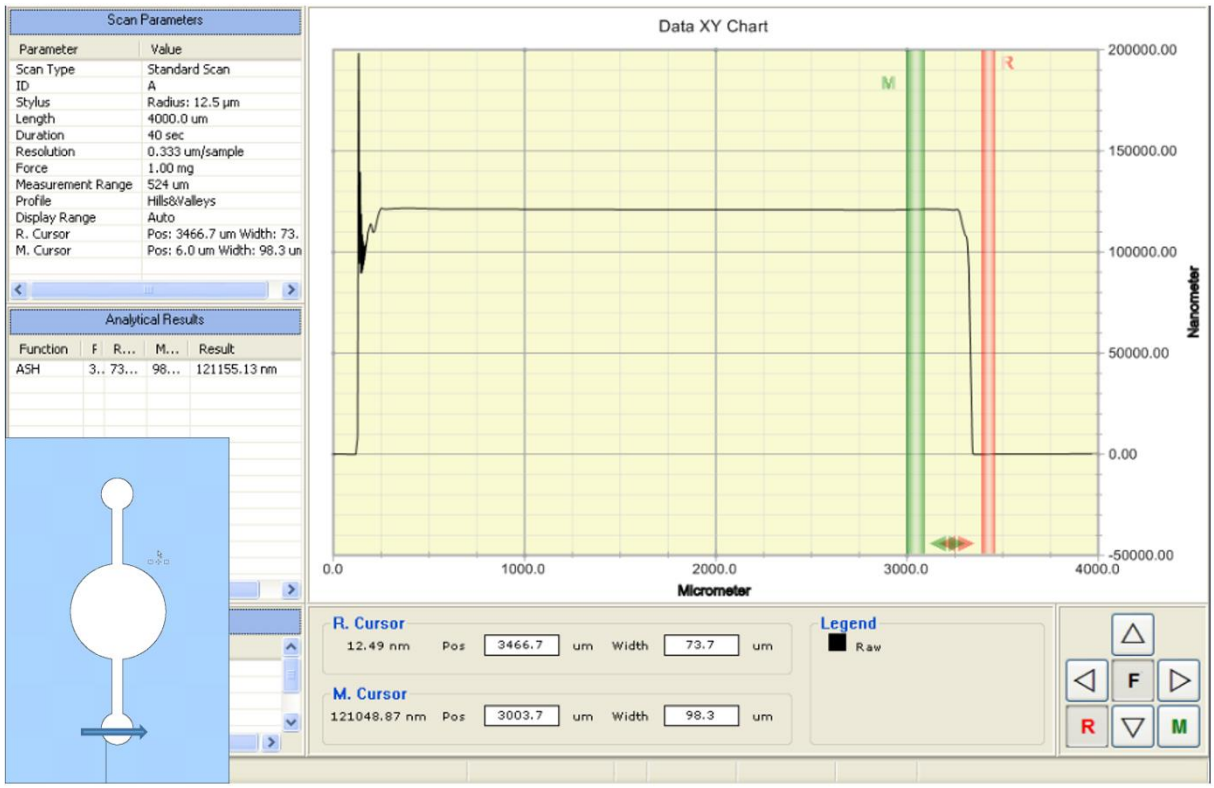


Minimum 180µm and maximum 235µm

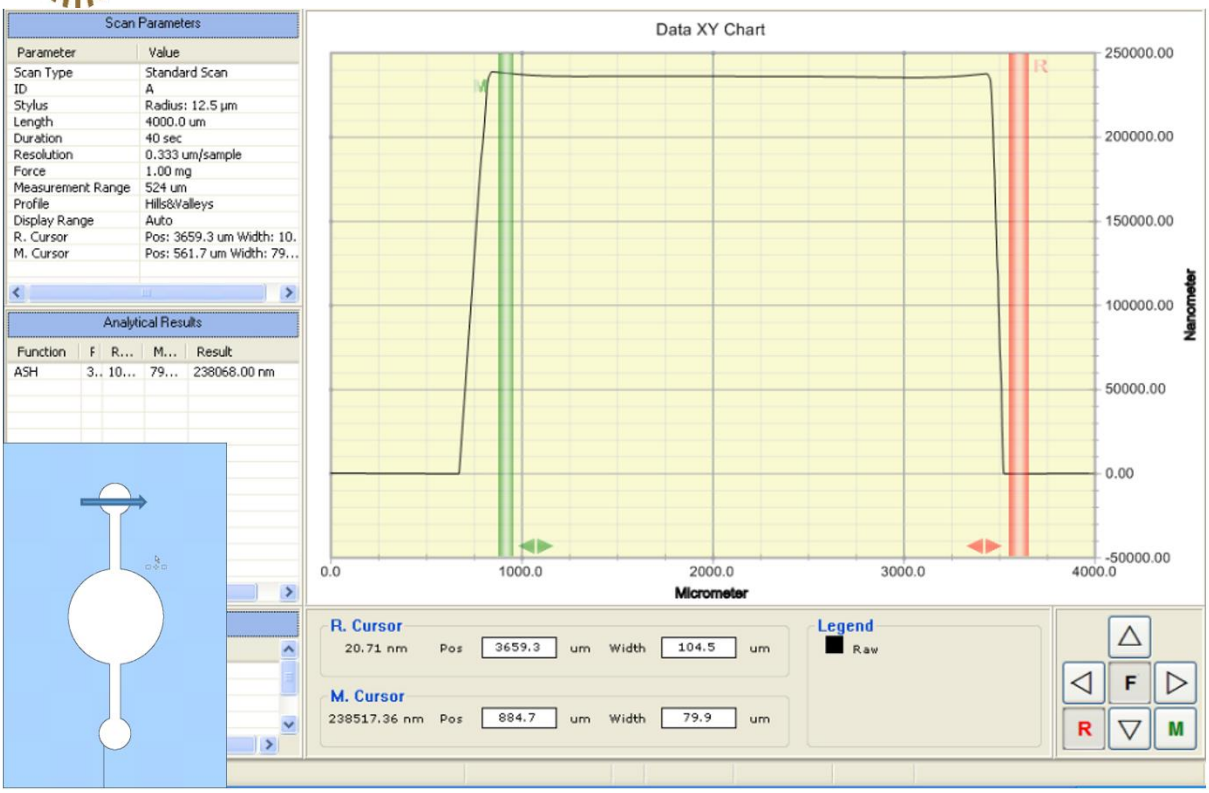
- Design 2



Minimum 170µm and maximum 175µm

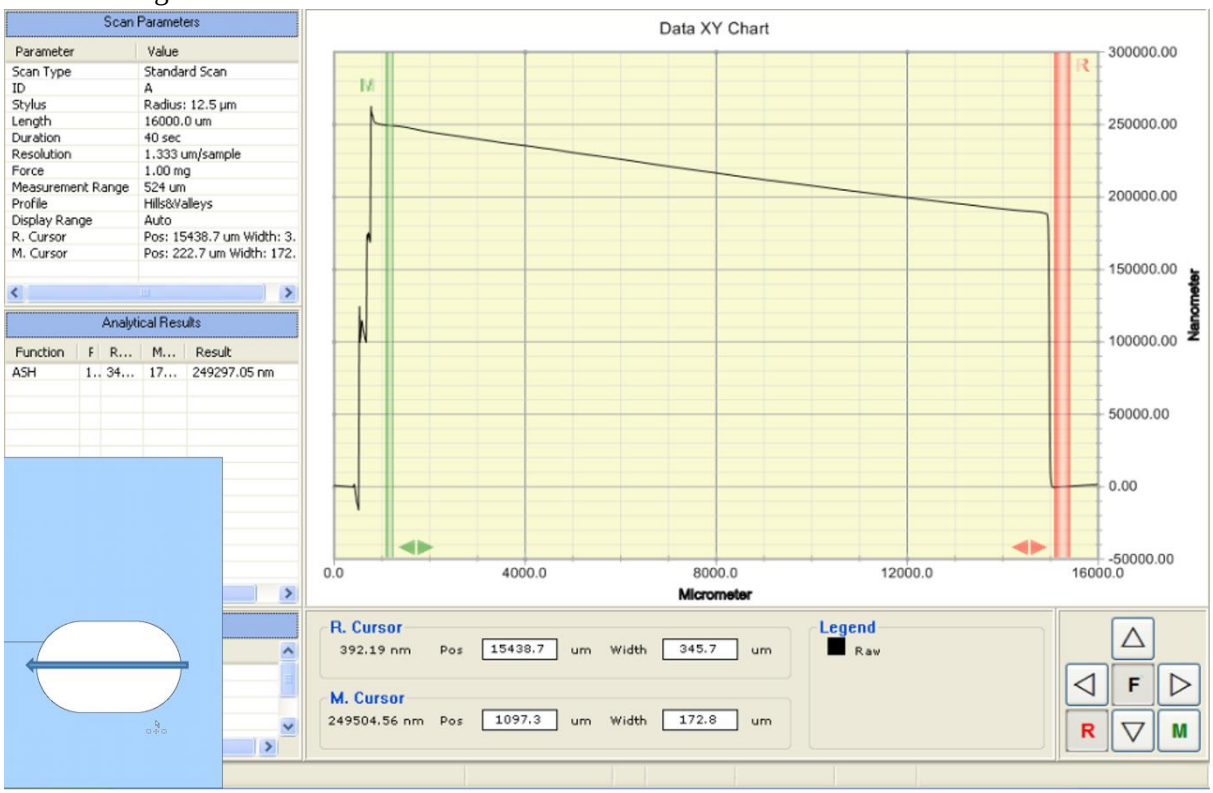


120µm average

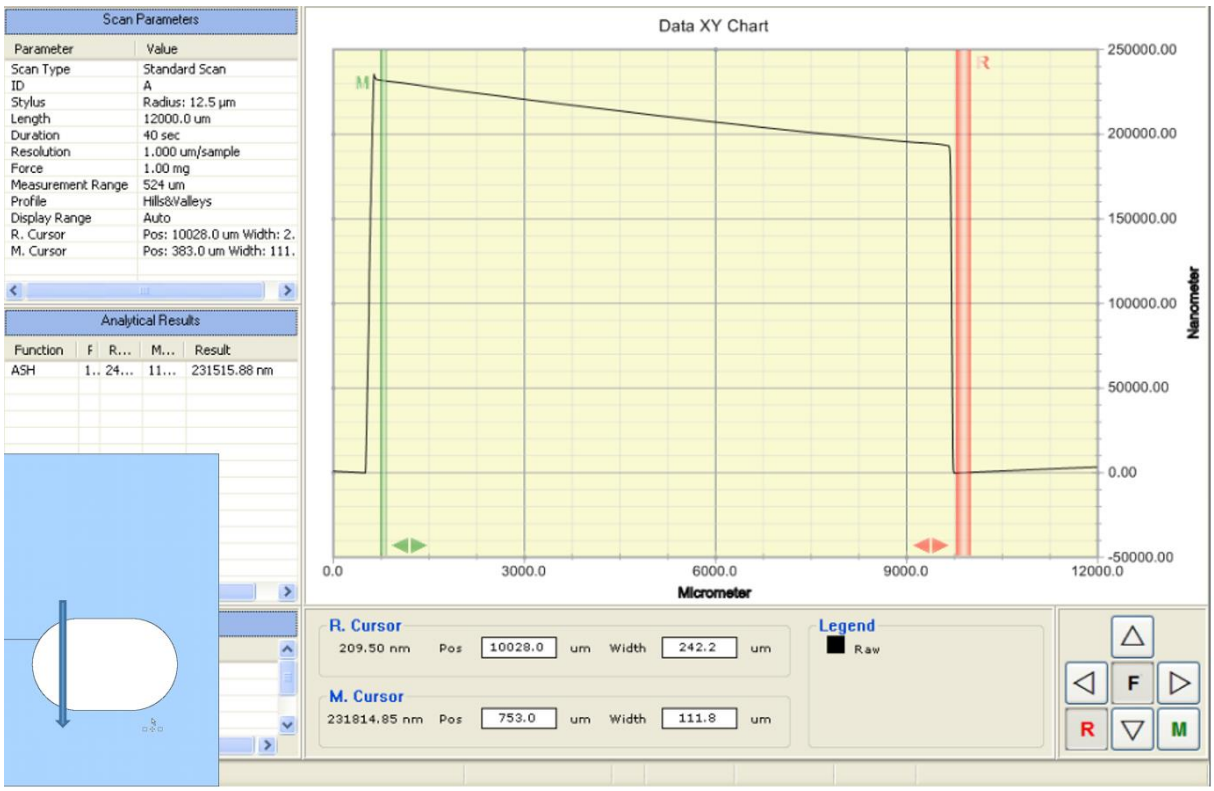


235µm average

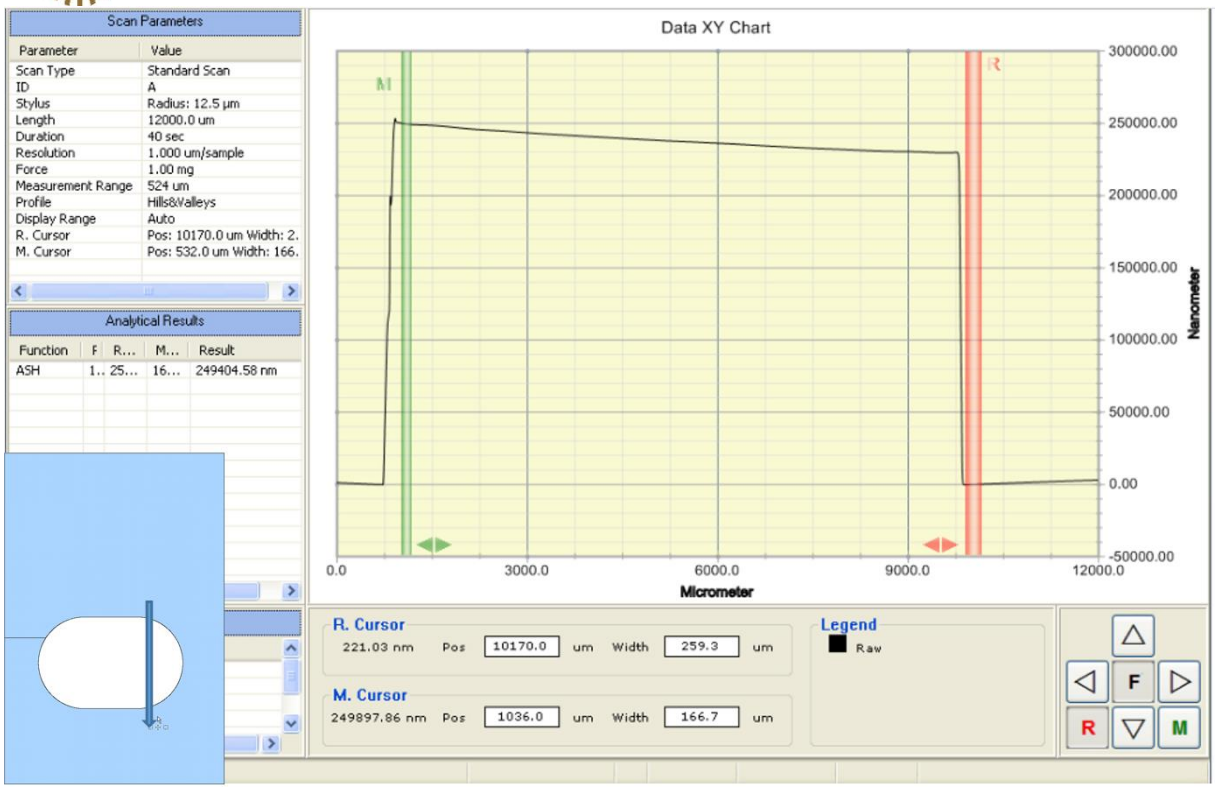
- Design 3



Minimum 190µm and maximum 250µm

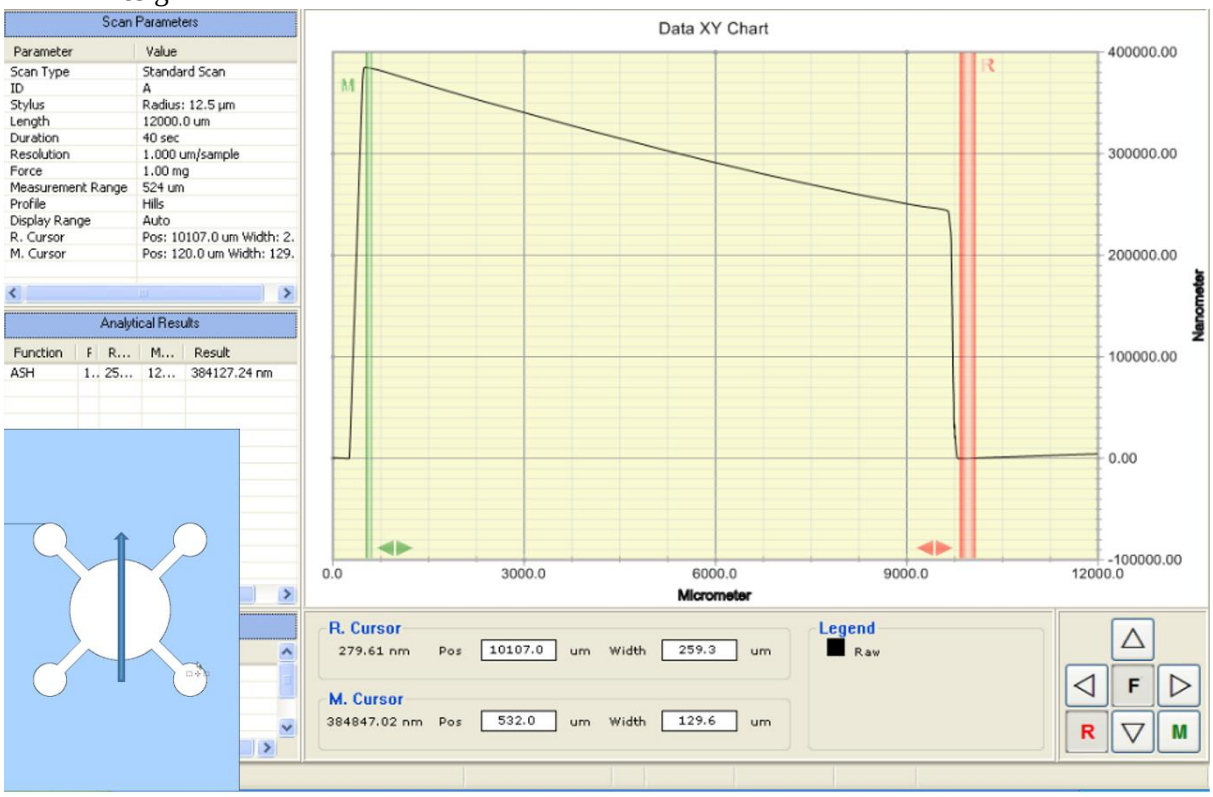


Minimum 195µm and maximum 230µm

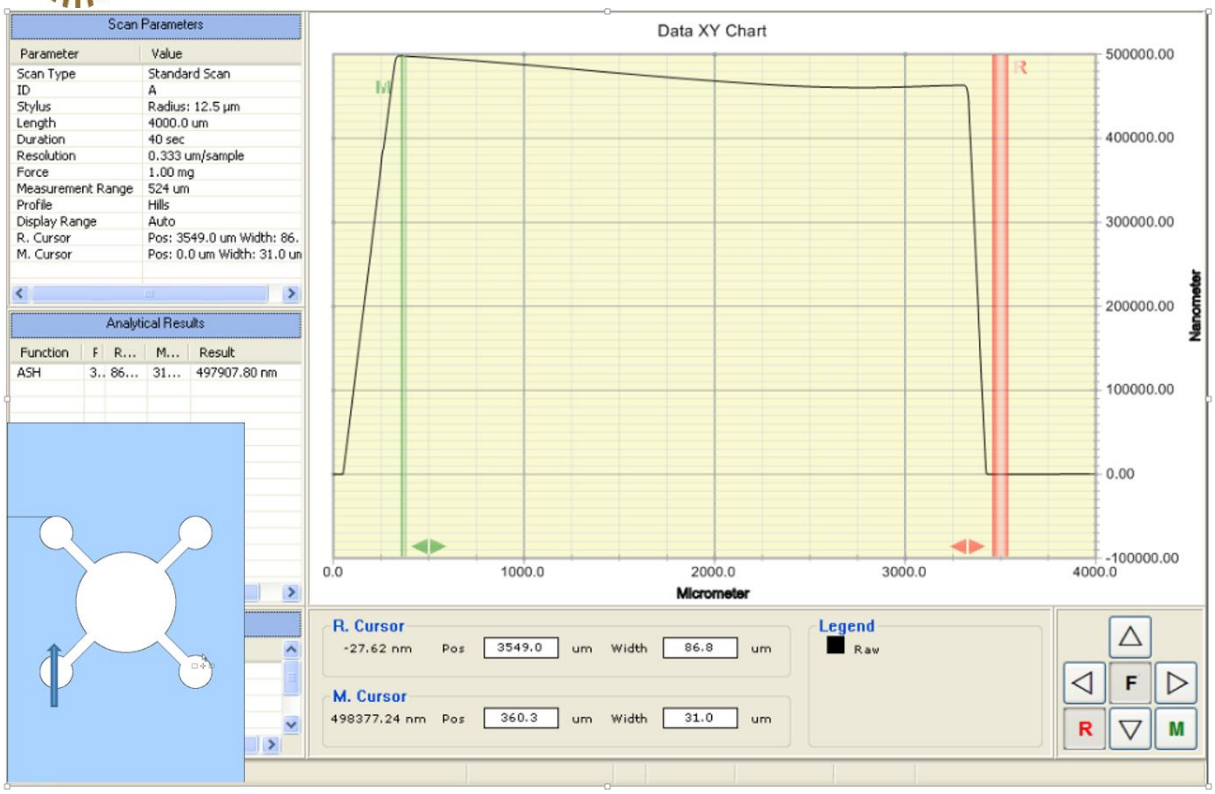


Minimum 230µm and maximum 250µm

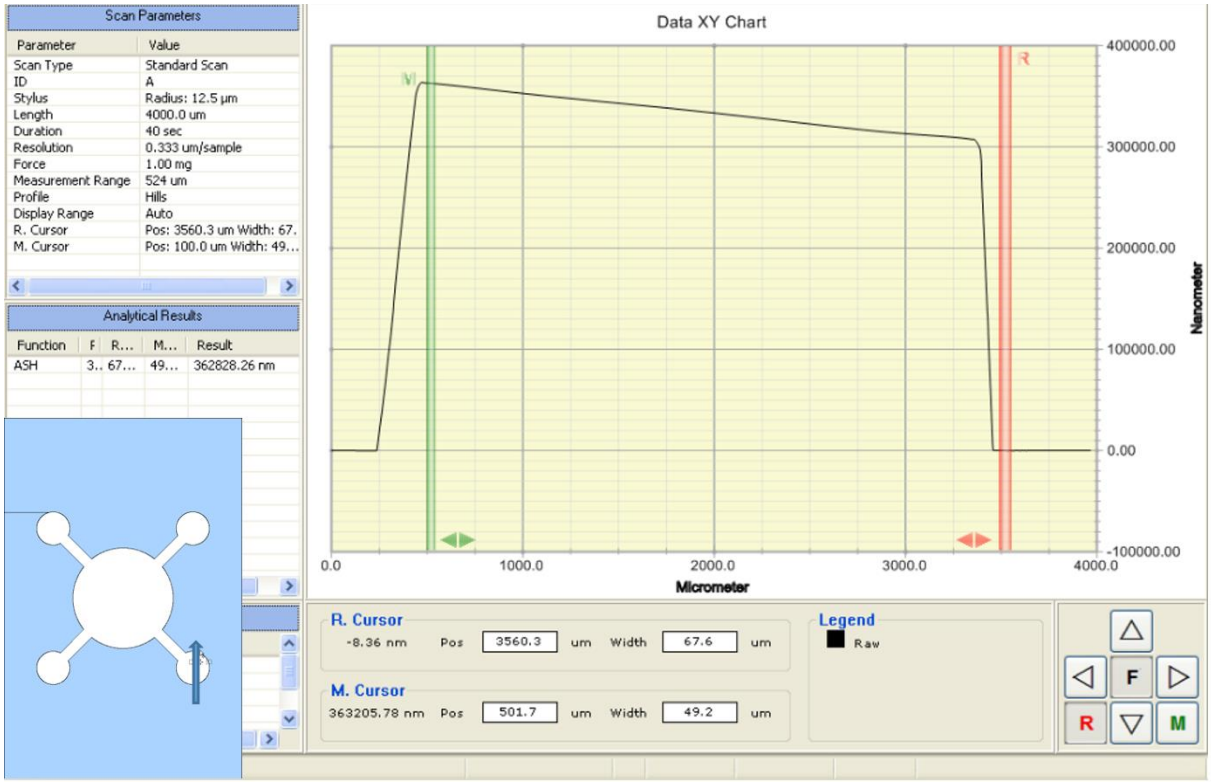
- Design 4



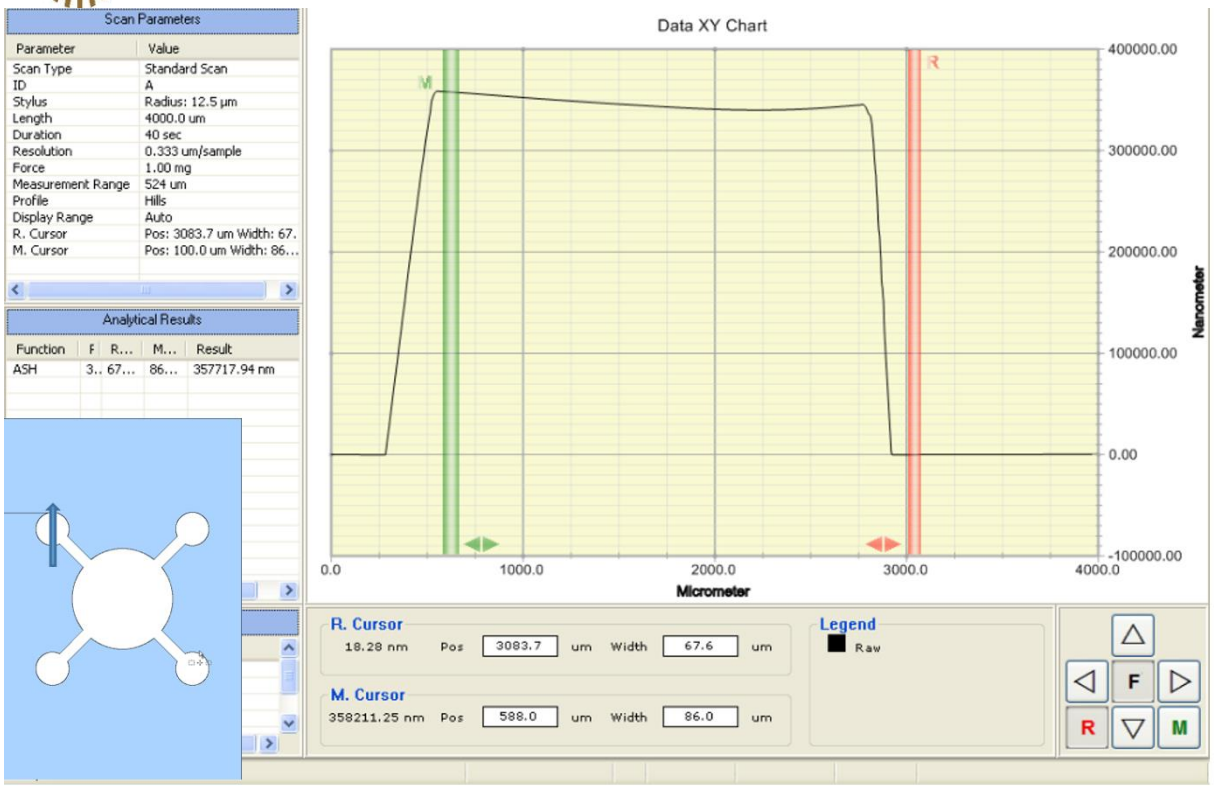
Minimum 245µm and maximum 385µm



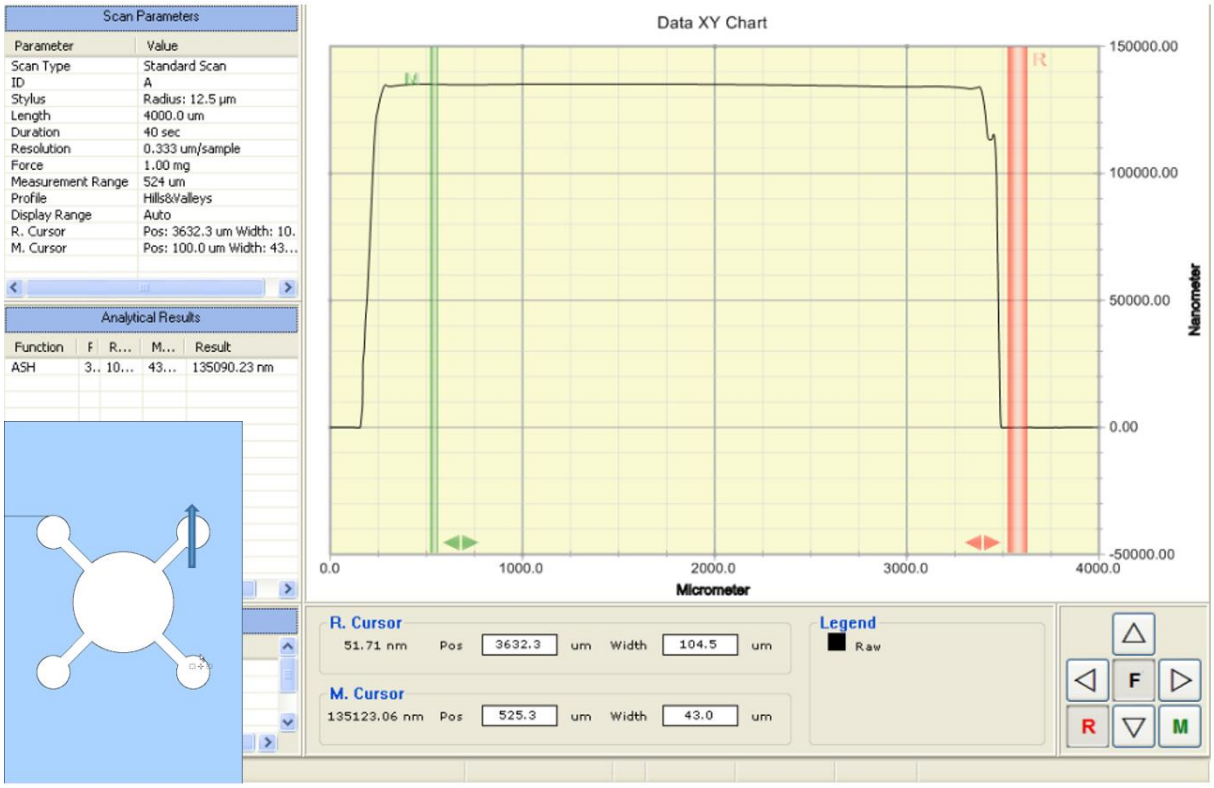
Minimum 465µm and maximum 500µm



Minimum 310µm and maximum 365µm

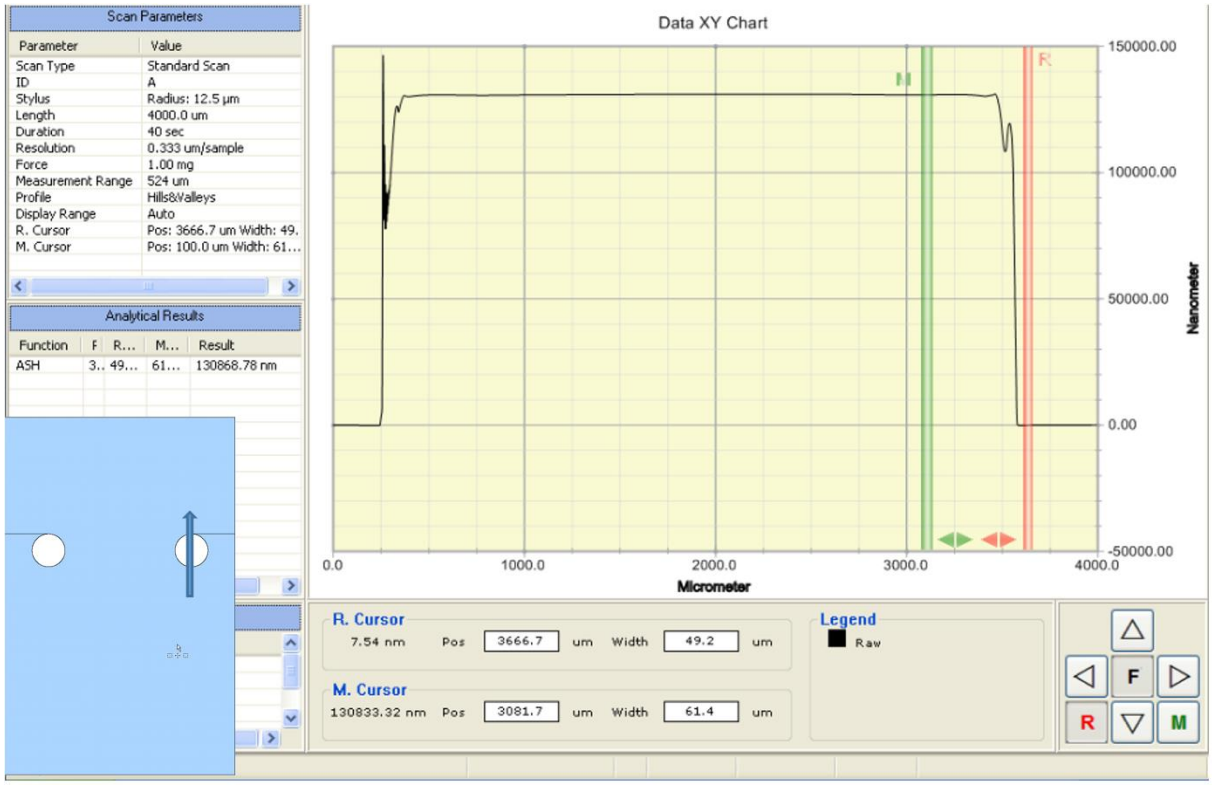


Minimum 345µm and maximum 360µm



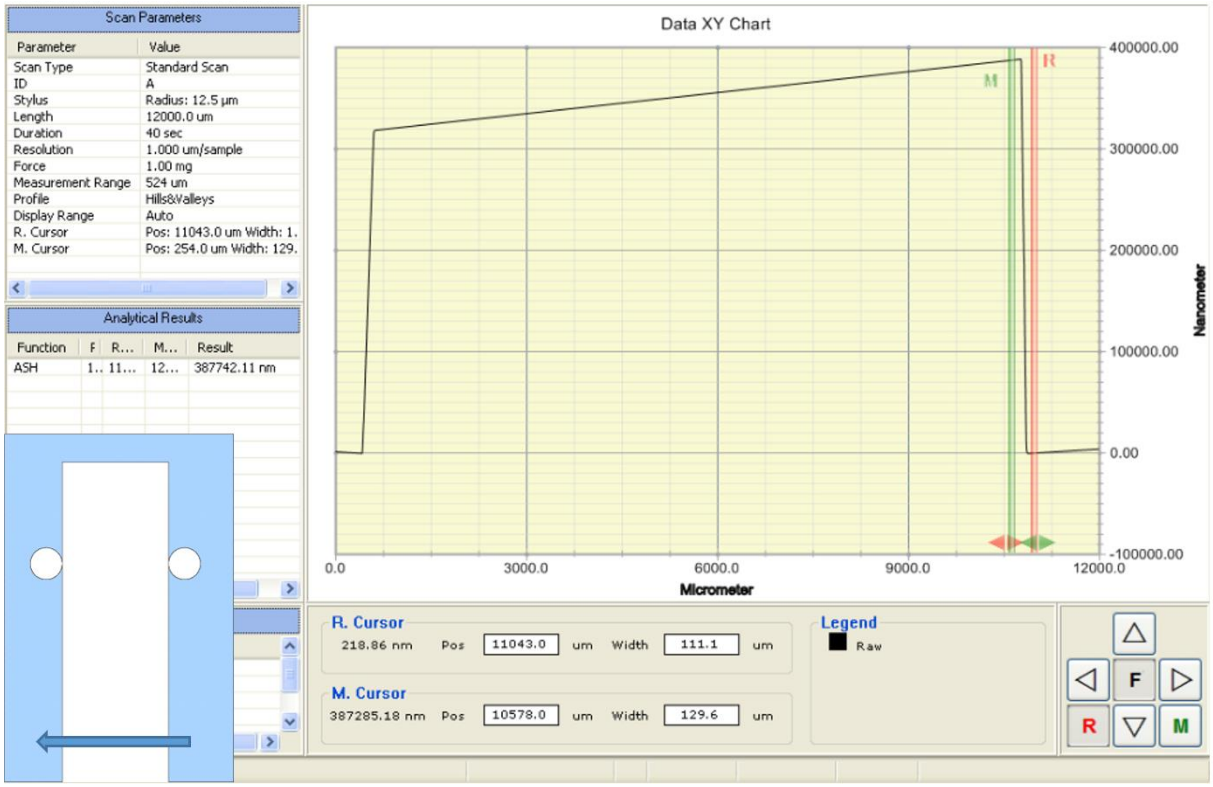
Average 135µm

- Design 5

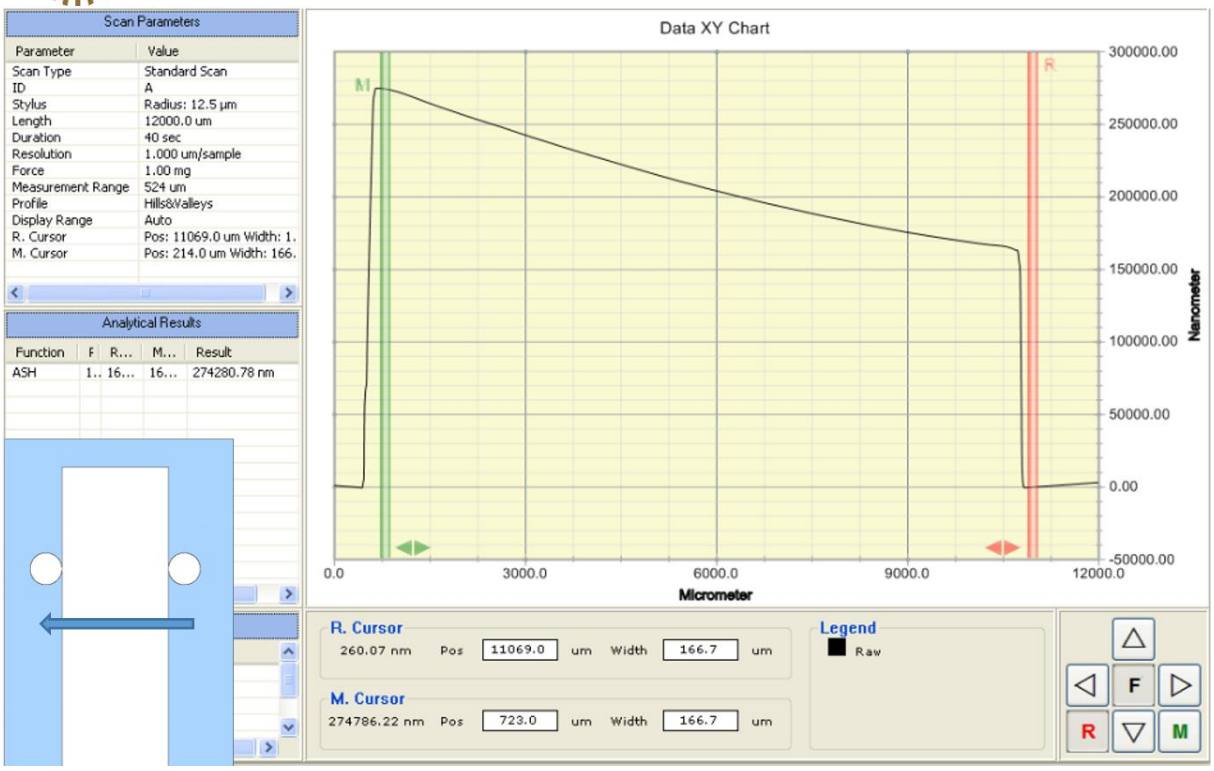


Average 130µm

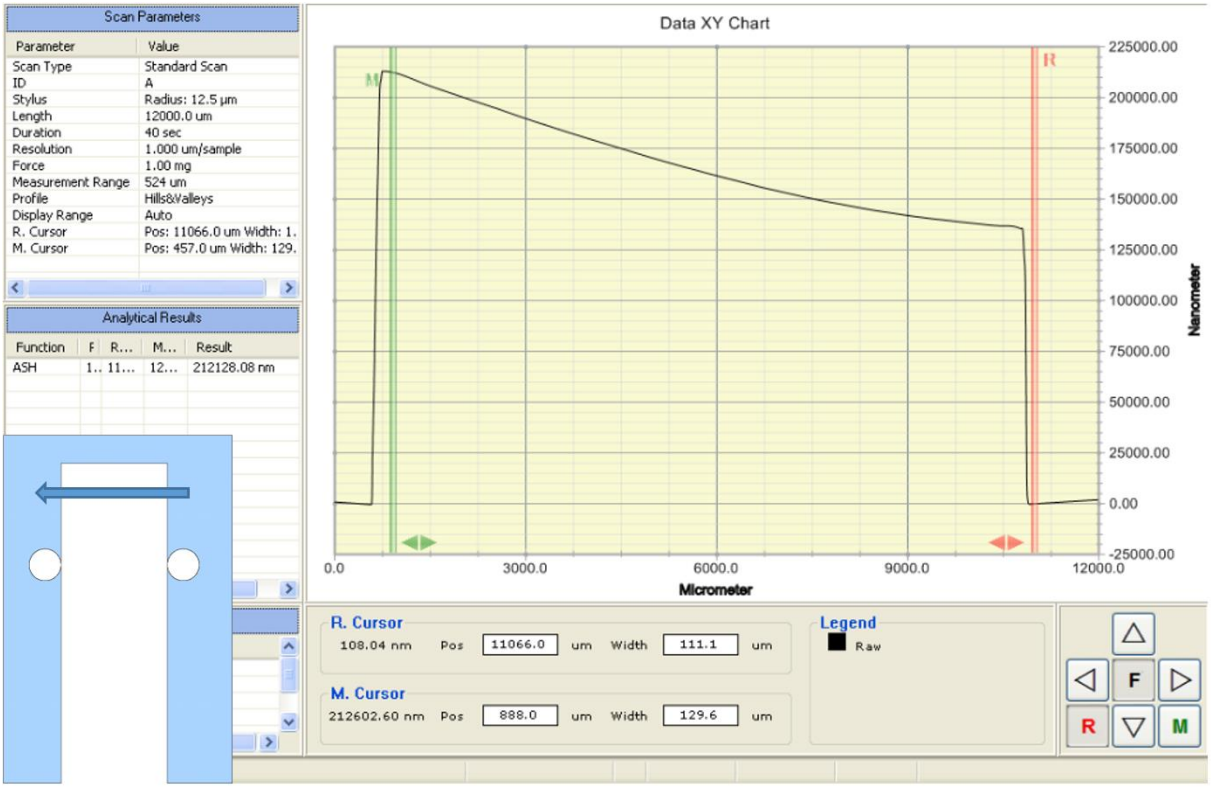
- Design 6



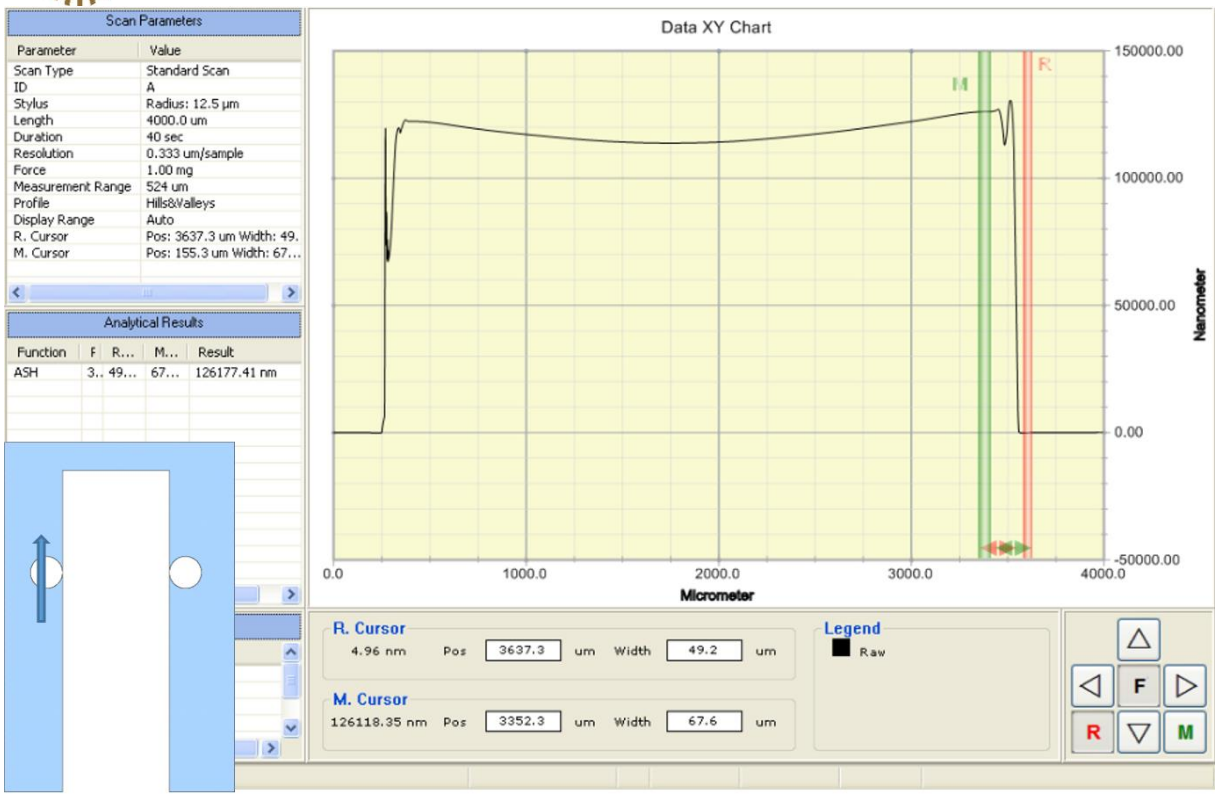
Minimum 320µm and maximum 390µm



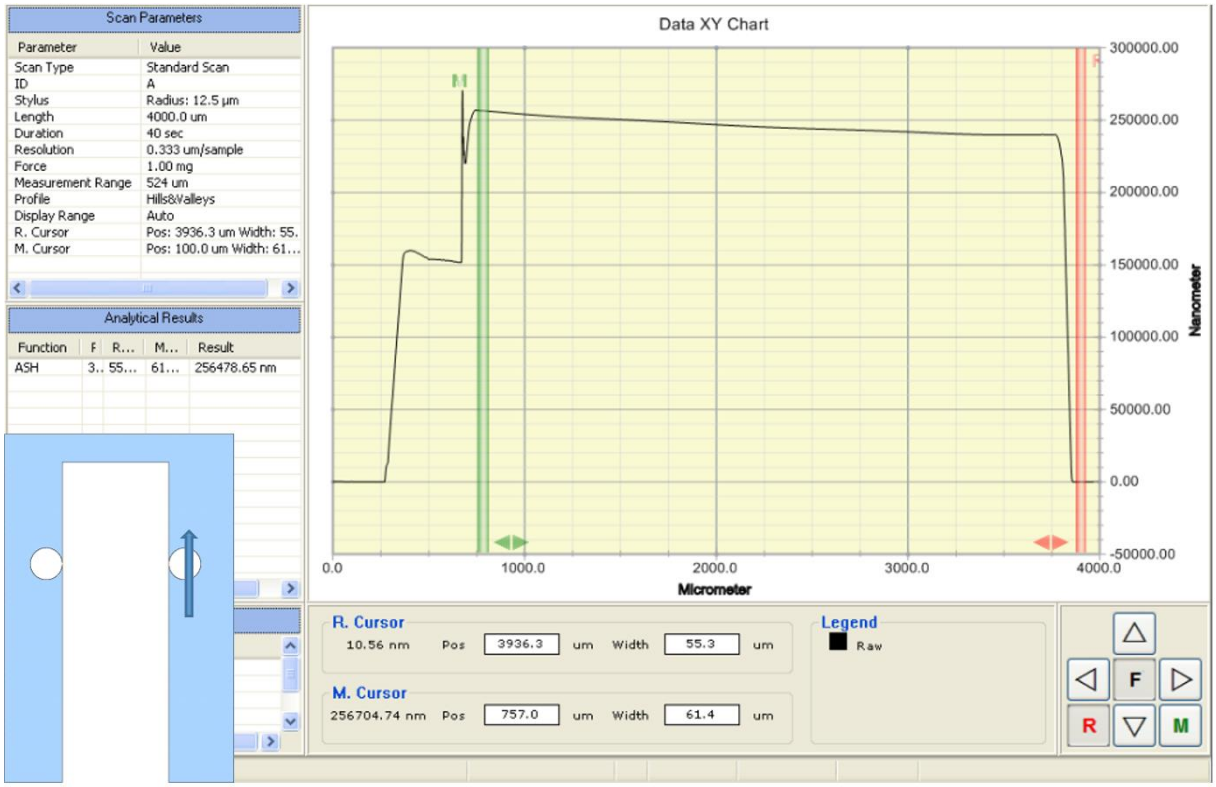
Minimum 165µm and maximum 275µm



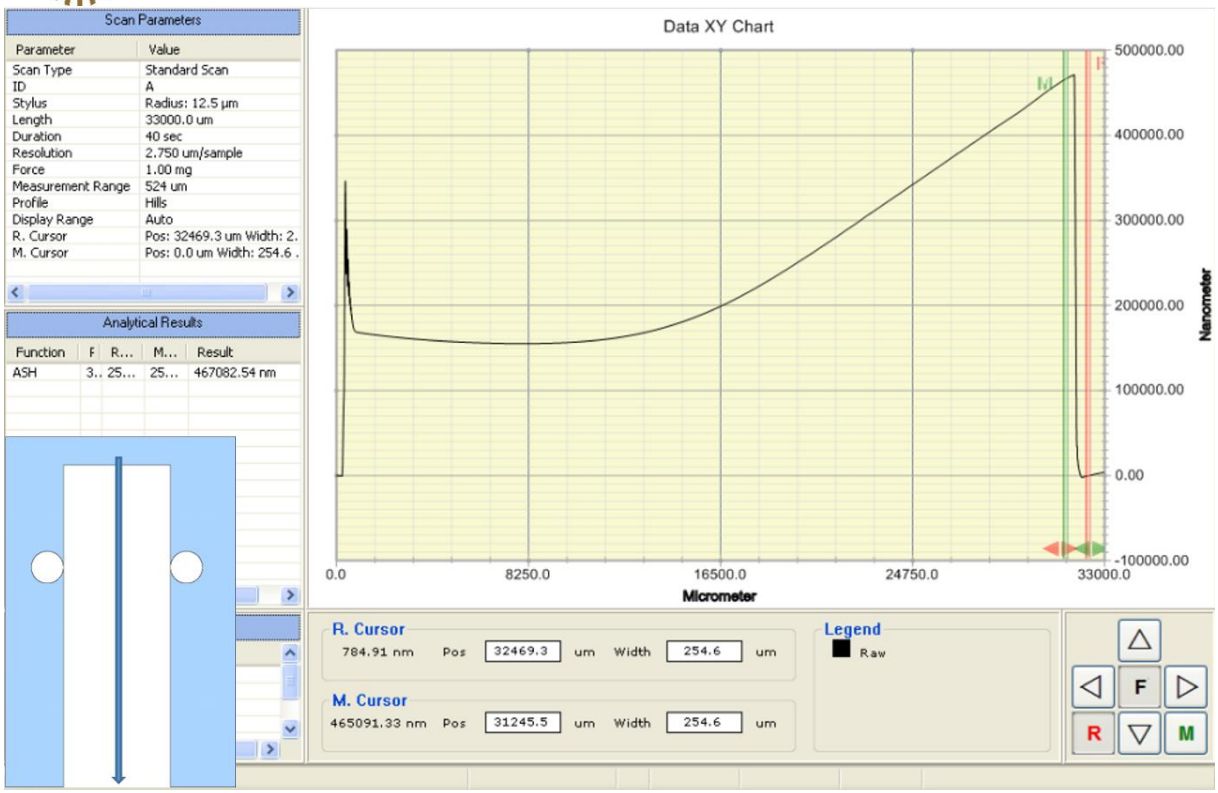
Minimum 135µm and maximum 215µm



Minimum 115µm and maximum 125µm



Minimum 240µm and maximum 260µm



Minimum 160µm and maximum 470µm

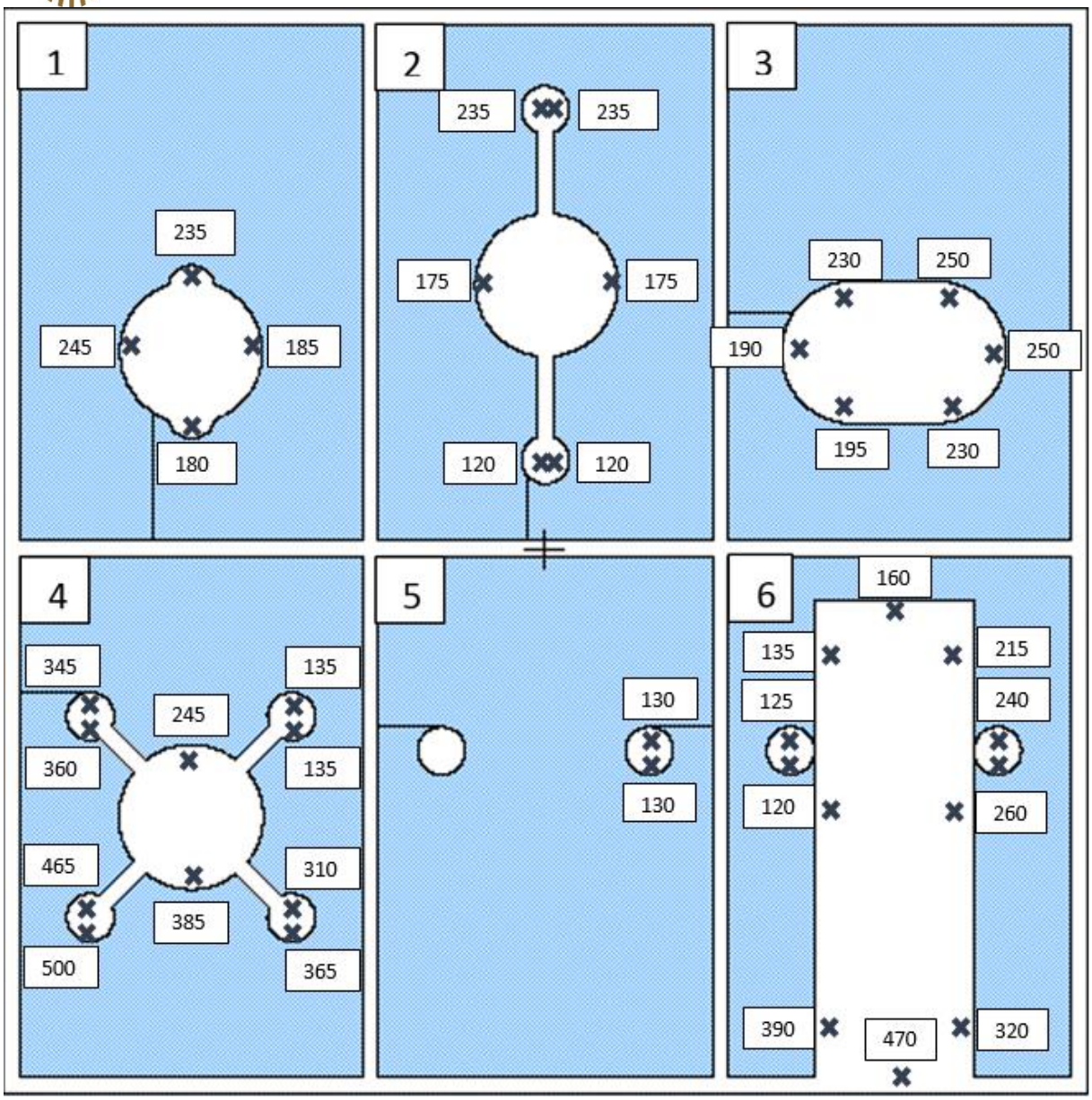


Figure 113. Measurement

HØGSKOLEN I BUSKERUD OG VESTFOLD

7.9.5 Replica molding 1

Laboratory exercise, Mars 19, 2015

PDMS replica molding

I used the procedure of BioMEMS laboratory exercise 3 from the course MN-BIO4600 on HBV-Vestfold:

Equipment and supplies:

- Scale
- Vacuum jar (desiccator) with vacuum pump (or house vacuum)
- Oven
- Tweezers
- Gloves
- Plastic mold
- Disposable syringe (1-2 mL) or transfer pipette
- Stirring spoon / bar
- PDMS Resin, Dow Corning Sylgard 184 Part A, Part B
- SU-8 master (Laboratory exercise 150316, wafer 1)
- Silicone tubing
- Duco cement, household glue or superglue
- Paper cutter
- Ruler

Procedures:

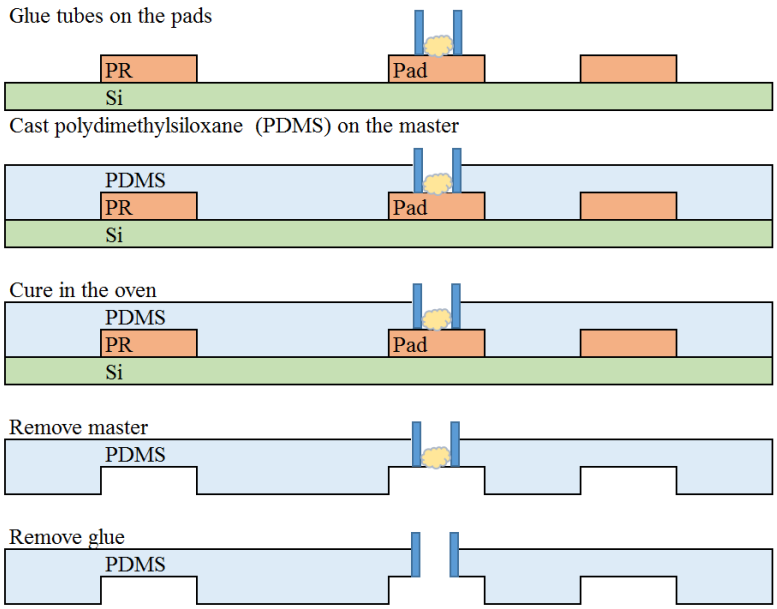


Figure 114. Simple ilustration of the process

1. Set the oven to 65ºC.
2. Cleaning of the master and the mold:

Clean the master of impurities and rests of former PDMS if necessary. Use a mold with border height of 3-4cm and diameter just a bit bigger than a wafer of 4". Remove the rests of PDMS from the mold. Both can be washed with DI-water and Isopropanol. Do not use Acetone.

3. Coat with Trichloro (1H, 1H, 2H, 2H-perfluorooctyl) silane (TFOCS):

Normally the master is coated with TFOCS in the gas phase for 45 minutes, using a vacuum desiccator. This is done to prevent that the PDMS get stuck to the master. It is very important to do this under a fume cabinet, since these chemicals are toxic and corrosive. This part of the procedure was not followed in this exercise, because there were problems with the vacuum desiccator. We decided to use this
as a test to observe if there was any difference between using the coating or not.

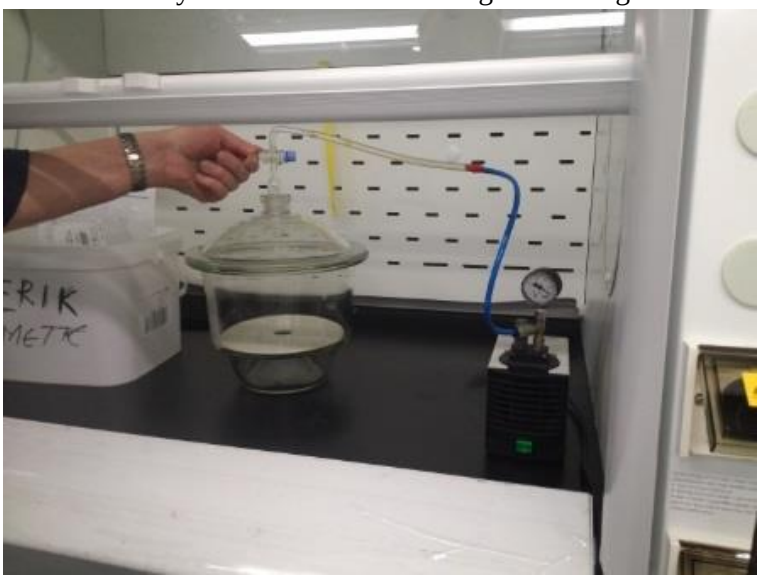


Figure 115. The vacuum desiccator

4. Prepare the in-/outlets:

Use plastic tube with ID of 1 mm and OD of 3 mm. Take the plastic tube and cut in as straight as possible in pieces of 1 cm. This design requires 16 in-/outlets.

5. Glue the in-/outlets to the master:

Use Duco cement to fasten the pieces of tubes in the correct position. It is very important that the glue fills the tubes a bit to prevent the PDMS from entering. Let the glue cure for approximately 5-20 minutes.

6. Prepare the PDMS:

Use a clean beaker and gloves to work with the PDMS. Put the beaker on the scale and press tare to set the scale to zero. Take the beaker out of the scale and add approximately 30g of PDMS base. The best way is using a syringe, and it was filled with approximately 30ml. Remember, PDMS weigh more than water. We ended up using 40g of PDMS.

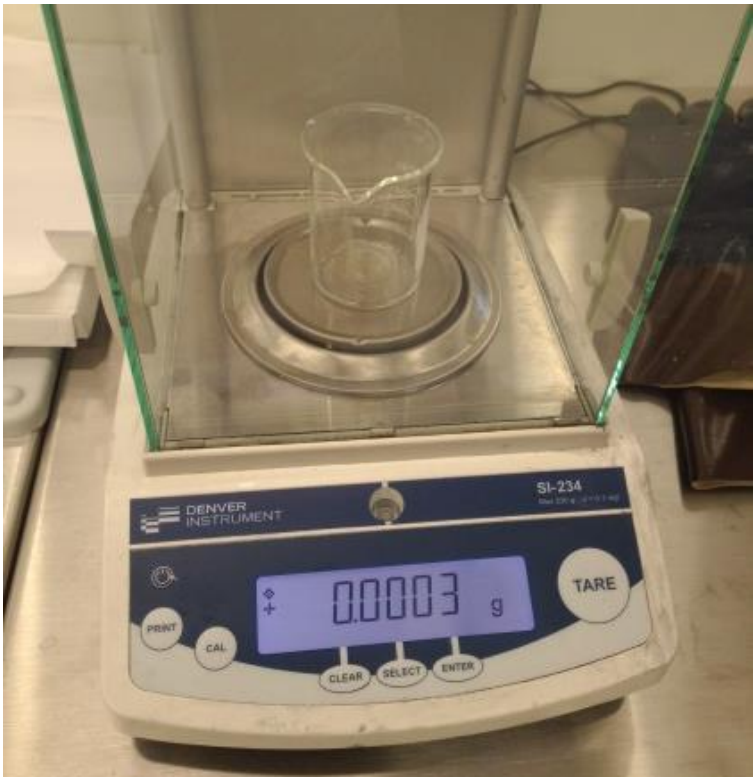


Figure 116. The scale set.

The curing agent is applied in a ratio 1/10 with the PDMS. The scale was set to zero again with the PDMS, and the curing agent was added drop by drop until reaching 4g.
Mix the PDMS with the hardener thoroughly, until the PDMS is white with bubbles.

7. Eliminating the bubbles:

Degas the PDMS in a vacuum chamber. The bubbles rise to the surface where they burst. Vent the chamber occasionally to help bursting the bubbles. If the vacuum is very high, it is possible the foam flows over the edges of the beaker, so it is necessary to keep an eye on it.

Figure 117. The vacum chamber

8. Filling the mold:

When the bubbles are eliminated, pour gently the mix over the master avoiding the in-/outlets. Degas again approximately 15 to 20 minutes, being sure no bubbles remain.

9. Curing the PDMS:

The PDMS is cured in the oven at 65ºC through the night (it cannot be over cured). Without heating, it is supposed to cure in approximately 24 hours in room temperature, but it is better to use the oven and assure that it is perfectly cured.

10. Separating the designs:

Remove the PDMS from the master. It is possible to use a ruler and a paper cutter to divide the 6 designs while in the mold. Use tweezers to lift the designs out of the mold. When the designs are separated, remove the glue from the in-/outlets.

Figure 118. The designs freed from the mold

11. Prepare the mold and the master for reuse:

The master is easily stuck in the mold, but using the cutter and tweezers it should come out. Try to eliminate all the rests of PDMS on the mold and the master. Place the master in a dust-free box.

Results and discussion:

The tubes were not perfectly straight cut, and therefore not all were vertical, but many were leaning to one side or another. It is important to try to cut them as straight as possible in future laboratory exercises.

The amount of PDMS seemed perfect. The thickness of the PDMS was approximately 5mm. I will try to use the same amount in future exercises.

When degasing the PDMS, it almost foamed over when the vacuum was set to maximum. I had to turn it down to ¾ of maximum.

The PDMS was perfect without any bubbles.

I tried to cut the PDMS without the ruler, but it should be cut a bit straighter.

The designs were lifted out without any problems. There seem to be no need for TFOCS.

No damage or flaws were observed.

7.9.6 Replica molding 2

Laboratory exercise, April 7, 2015

PDMS replica molding

I used the procedure of BioMEMS laboratory exercise 3 from the course MN-BIO4600 on HBV-Vestfold:

Equipment and supplies:

- Scale
- Vacuum jar (desiccator) with vacuum pump (or house vacuum)
- Oven
- Tweezers
- Gloves
- Plastic mold
- Disposable syringe (1-2 mL) or transfer pipette
- Stirring spoon / bar
- PDMS Resin - Dow Corning Sylgard 184 Part A, Part B
- SU-8 master (Laboratory exercise 150326)
- Silicone tubing
- Duco cement
- Paper cutter
- Ruler

Procedures:

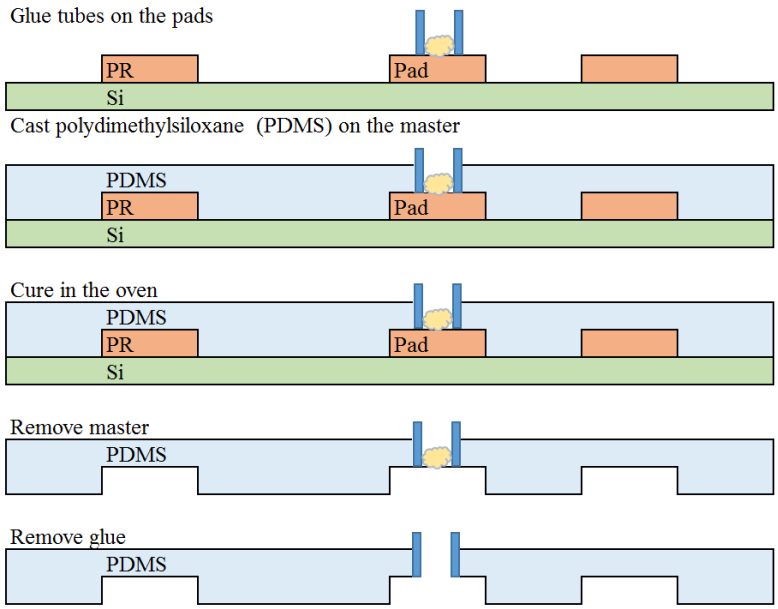


Figure 119. Simple ilustration of the process

12. Set the oven to 65ºC.
13. Cleaning of the master and the mold:

Clean the master of impurities and rests of former PDMS if necessary. Use a mold with border height of 3-4cm and diameter just a bit bigger than a wafer of 4". Remove the rests of PDMS from the mold. Both can be washed with DI-water and Isopropanol. Do not use Acetone.

14. Coat with Trichloro (1H, 1H, 2H, 2H-perfluorooctyl) silane (TFOCS):

Due to the results of the laboratory exercise from 19th of March, it seemed to be no need for coating with TFOCS, because the PDMS casting loosened without problems.

15. Prepare the in-/outlets:

Use a plastic tube with ID of 1 mm and OD of 3 mm. Cut it in pieces of 1 cm as straight as possible. This design requires 16 in-/outlets.

16. Glue a Dummy chip to the master:

One of the designs required a dummy chip to create a socket. It was necessary to use a knife to scratch away some of the photoresist, to give space to the dummy. It was then glued to the master with Duco cement.

17. Glue the in-/outlets to the master:

Use Duco cement to fasten the pieces of tubes in the correct position. It is very important that the some of the glue enters the tubes to prevent the PDMS from entering. Let the glue cure for approximately 5-20 minutes.

18. Prepare the PDMS:

This is done under the fume hood to prevent breathing the fumes. Use a clean beaker and gloves to work with the PDMS. Put the beaker on the scale and press tare to set the scale to zero. Using a syringe, I aimed for 40g and ended up with 39,6g. Then the tare was set to 0 again, and I used another syringe to add 1/10 of the weight in curing agent. I ended up with 3,97g. Then it was mixed thoroughly until it was white with bubbles.

19. Eliminating the bubbles:

Degas the PDMS in a vacuum chamber. The bubbles rise to the surface where they burst. Vent the chamber occasionally to help bursting the bubbles. I used the vacuum at approximately 2/3 of the vacuum. It took 28 minutes to remove the bubbles.

20. Filling the mold:

When the bubbles are eliminated, pour gently the mix over the master avoiding the in-/outlets. I had to degas 30 minutes to eliminate all the bubbles.

21. Curing the PDMS:

The PDMS is cured in the oven at 65ºC through the night (it cannot be over cured). Without heating, it is supposed to cure in approximately 24 hours in room temperature, but it is better to use the oven and assure that it is perfectly cured.

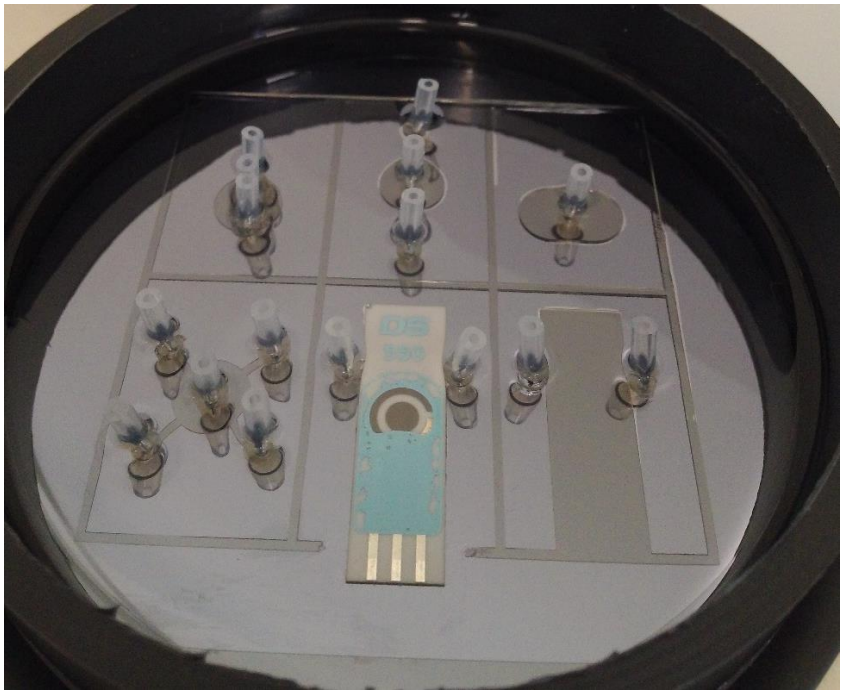


Figure 120. The PDMS cured and prepared to be lifted off the master.

22. Separating the designs:

Remove the PDMS from the master. I found it easier to cut loose the PDMS without using a ruler. I used tweezers to lift the designs out of the mold. When the designs are separated, I removed the glue from the in-/outlets.

23. Prepare the mold and the master for reuse:

The master was very stuck in the mold, and when I used the cutter and tweezers to get it out, it broke.

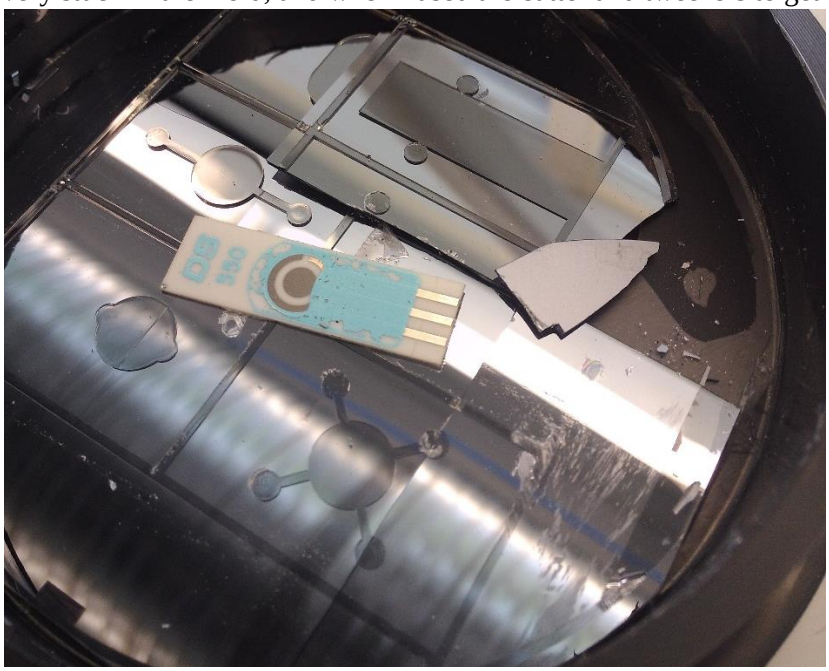


Figure 121. The broken master.

Results and discussion:

The tubes were cut straighter this time, and the dummy chip seemed to work fine to create a socket.

The amount of PDMS was almost the same as the former laboratory exercise and the thickness was still approximately 5mm.

The PDMS was perfect without any bubbles.

I cut the PDMS just following the guidelines made with SU8. I adjusted a bit depending on the requirements of each design.

The designs were lifted out without any problems.

No damage or flaws were observed.

The master broke when I tried to lift it out, caused by a rather big amount of PDMS that had gotten under the wafer. I still have the master created in the laboratory exercise on 16th of March with similar measures, and I also have one master created the 20th of march, but the SU8 is much thinner. For later exercises it would be better to not remove the master from the mold if not necessary.