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MAZURSKI W OLSZTYNIE

Development of a parallelizable QCM-D array for the mass spectrometric analysis of proteins

Master thesis

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Abstract

Quartz crystal microbalances allow the monitoring of the adsorption process of mass from a liquid to their surface. The adsorbed mass can be analysed regarding to its protein content using mass spectrometry. To ensure the protein identification the results of several measurements can be combined. A high content QCM-D array was developed to allow up to ten measurements parallel. The samples can be routed inside the array distributing one sample to several chips. The fluidic parts were prototyped using 3D printing. The assembled array was tight and the sample routing function could be demonstrated. A temperature controller was developed and implemented. The parameters for the PID controller were determined and the controller was shown to be able to keep the temperature constant over long time with high accuracy.

KEYWORDS: QCM-D, Protein, Mass spectrometry, High content measurement, QCM-D Array, 3D printing, Temperature control, Arduino

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1 Introduction

1.1 Motivation

The amount of substance adsorbing from a liquid phase to a defined solid surface can be monitored using quartz crystal microbalances. They consist of a cylindrical quartz crystal with electrodes on each side. The inverse piezoelectric effect causes the crystal to be deformed, if a voltage is applied to it. The resonance frequency depends on the mass of the crystal and changes if mass from the liquid phase adsorbs onto the crystal surface. Additional information about the viscoelastic properties of the adsorbed layer can be obtained by monitoring the decay of the oscillation of the crystal, after its excitation is stopped. This is called quartz crystal microbalance with dissipation monitoring (QCM-D) (O , 1999; Voinova et al., 1999).

If a complex sample is adsorbed onto the chip surface the composition of the adsorbed layer also depends on the chip surface. To determine the composition of the layer on the surface regarding to its protein content, QCM-D coupled with mass spectrometry can be used. The adsorbed proteins are digested on the chip and the resulting peptides are enriched chromatographically. Matrix assisted laser desorption ionization time of flight mass spectrometry (MALDI-ToF MS) allows the detection of the peptide masses with an accuracy allowing to identify the proteins the peptides result from. The sample is mixed with a matrix, that absorbs the energy at the wavelength of the laser. In a vacuum this allows the desorption of the peptides into the gas phase without their destruction. Adding tetrafluoroacetic acid (TFA) to the sample promotes the ionization of the peptides by the addition of a proton. The ionized peptides are accelerated into a flight tube and the time, needed to pass it, is determined. The flight time depends on the mass of the peptide and allows to determine it (Perkins et al., 1999; Kirschhöfer et al., 2013).

By comparing the measured masses with the masses of theoretical tryptic digests, the proteins the peptides originate from can be determined. To ensure the correct identification the peptide masses can be further fragmented using a higher laser energy or a collision cell. The comparison of the resulting spectra with theoretical fragmentations of the expected peptide sequences allows to verify their correct identification. To reduce the complexity of the mass spectra sets of at least three chips sampled with independent samples are used in this method (Hohmann et al., 2014, 2015).

1.2 Aim of the work

Commercially available QCM-D systems only allow up to six parallel measurements. To compare e.g. the adsorption of one sample onto three different chip surfaces three measurements have to be done after another. A high content QCM-D array would allow to perform all measurements parallel. To measure the same sample on three chips with the same surface the possibility to connect them serially would also be usefull.

Aim of the work was to develop a parallelizable QCM-D array, that allows to measure the adsorption of substances from up to ten liquid samples to up to ten QCM-D chips. Also a flexible sample routing allowing the distribution of one sample to more than one chip should be possible.

1.3 Structure of the thesis

A 3D model of a parallelizable QCM-D array was developed. The fluidic parts were prototyped using a 3D printer. Copper wires were plated with gold to providing the electrical contacts to the chip. Printed three way valves were developed to route the samples inside the array. The sample routing and tightness of the fluidic was tested using different colored samples. Cooling channels at the bottom of the array allow to keep the sample at a constant temperature. To control the temperature a digital temperature sensor with a PID controller was developed. After the circuit was tested on a breadboard a printed circuit board was developed and manufactured. The parameters of the PID controller were determined using empirical rules applied to the jump response of the control system. The response of the system to jumps in the setpoint were recorded and the long time stability of the control process was examined.

2 Materials and Methods

2.1 Chemicals, consumables and devices

A list of the used chemicals and their manufacturers can be found in supplement A. The used consumables are listed in supplement B. Supplement D contains a list of the used devices.

2.2 Components

A complete list of all used components can be found in supplement C. In the following sections selected non standard components are described in more detail.

2.2.1 QCM-D Sensors

Ion crystals consist of positive and negative charged ions. These are arranged in a regular three dimensional pattern. The pattern can be reduced to a single minimal cell, that describes the whole crystal structure, called elementary cell. Each elementary cell is inert to at least one symmetric operation, resulting in a net charge of zero at the macroscopic crystal surfaces. An axis in the elementary cell, that is not identical with its rotation about 180 degree around any axis perpendicular to itself, is called a polar axis. Figure 1 shows the elementary cell of a crystal with three polar axes and without a centre of symmetry. Applying a force to a crystal deforms it reversibly unless the force exceeds its structural limits. Applying the force nonperpendicular to one of the polar axes of the crystal in figure 1 **A** the elastic deformation will also cause a dislocation of the mass centers of the positive and negative charged ions (**B**). Summed up over the elementary cells of a macroscopic crystal, that dislocation can be measured as voltage. This is called the piezoelectric effect. The inverse piezoelectric effect describes a deformation of the crystal, when a voltage is applied to its surface (Auld, 1973; Reichl and Ahlers, 1989).

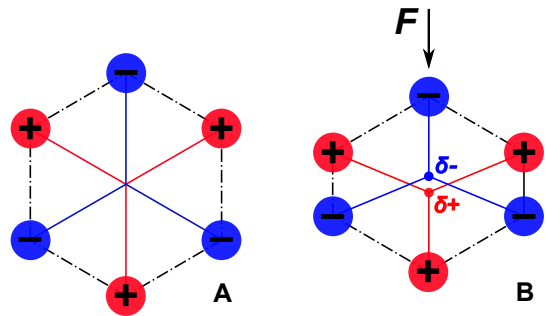


Figure 1: Piezo effect

To deform a crystal using the inverse piezoelectric effect and to record the effects of the piezoelectric effect while the crystal is swinging back into its undeformed state is the principle of the quartz crystal microbalance with dissipation monitoring (QCM-D). The resonance frequency of a crystal is dependent to its mass. By measuring the resonance frequency (QCM) or the response to a pulse (QCM-D) of a crystal, changes in its mass can be determined. If one side of the crystal is in contact with a liquid flow, adsorptions onto its surface can be detected. Due to the mass of the adsorbed substances leading to the change in the resonance frequency these types of sensors are called mass sensitive sensors (O , 1999; Voinova et al., 1999).

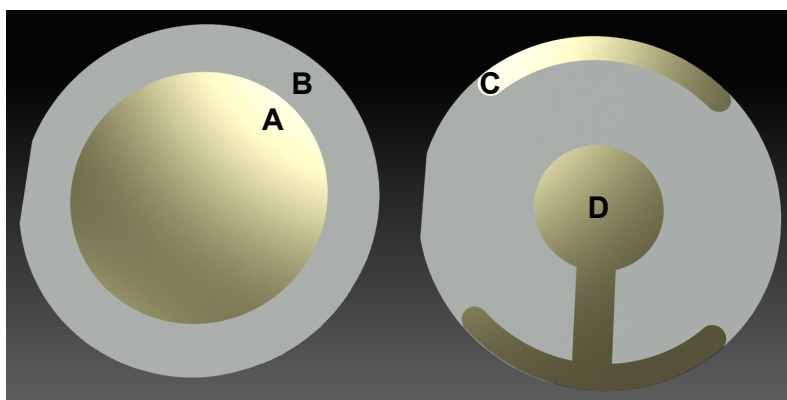


Figure 2: QCM-D chip; A: Sample electrode, B: AT cut quartz substrate, C: Wrap around of the sample electrode, D: Signal electrode

Figure 2 shows a QCM-D sensor based on an AT-cut quartz crystal substrate (B). The large circular electrode on one side of the chip (A) is in contact with the sample whilst a measurement. To simplify the handling of fluidic probes and electrical connections, the sample electrode is wrapped around to the other side of the chip (C). The other electrode (D) carries the electrical signal

as it is suitable to share ground with the liquids and all sensors. The sample electrode can be modified in a variety of ways allowing it to analyse the adsorption onto different surfaces.

The used QCM-D sensors have a diameter of 14 mm and a thickness of 0.3 mm. The active surface in contact with the sample is about 80 mm². Their base frequency is 4.95 MHz.

2.2.2 Peltier elements

The diffusion velocity of the main carriers of charge in a metal or semiconductor depends on temperature. If a semiconductor is heated on one side, the charge carriers diffuse to the cold side. In case of a negative doped semiconductor electrons are the main carriers of charge and the cold side becomes negatively charged. Defect electrons, like the main carriers of charge in positive doped semiconductors, lead to a positive charge at the cold side. This is called the Seebeck effect. Flows a current through a semiconductor the reverse effect leads to a temperature gradient in direction of the current flow. This is called the peltier effect.

A peltier element consists of negative and positive charged semiconductors connected serial between two ceramic plates as shown in figure 3. The direction of the current flow through the element determines the direction of the heat transport. This allows to use peltier elements as well for heating as for cooling an object connected to one of its sides. Connecting a heat sink and a fan to the other side helps to increase the heat transport. As the semiconductors heat up by thermal losses the efficiency of the cooling process is lower than that of the heating process (Riffat and Ma, 2003).

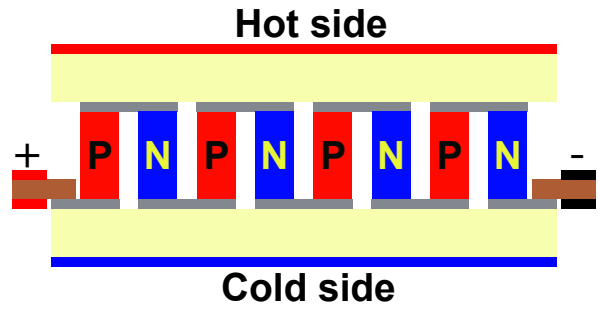


Figure 3: Peltier element

The peltier elements used in this work can be supplied with a voltage up to 15.4 V drawing a current of 4.6 A. It consists of 127 semiconductor elements between two quadratic ceramic plates with a size of 39.5 x 39.5 mm. The possible temperature difference between the hot and the cold side is 60 K and their heat pump power is up to 41 W (NTS electronic and components GmbH, 2014).

2.2.3 Motor driver

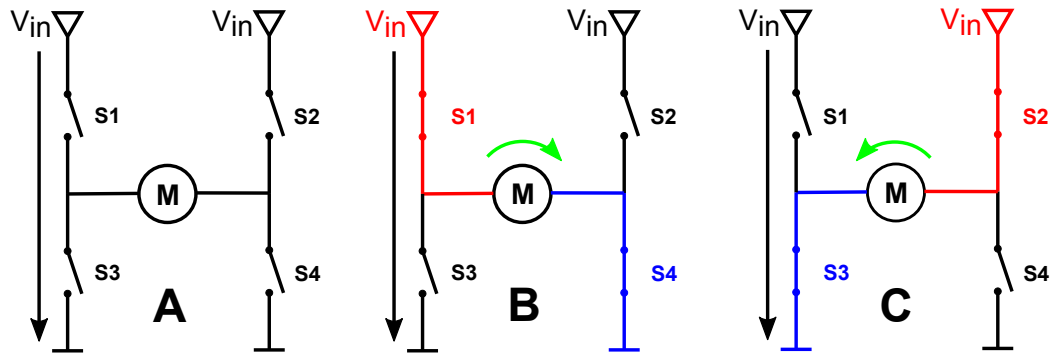


Figure 4: Function of a H bridge

To control the rotation of a motor its speed and its direction is important. The direction can be changed by reversing the polarity of the voltage applied to the motor. To archive that a H bridge as shown in figure 4 can be used. It consists of four switches S1 to S4. S1 and S2 are connected to the positive supply voltage V_{in} , S3 and S4 to ground. The motor is connected with S1 and S3 on its one junction and with S2 and S4 on its other one (A). The resulting circuit

looks like the capital letter H and is therefore called H bridge. By activating the switches S1 and S4, the left connector of the motor is connected to V_{in} and the right one to ground, resulting in a clockwise rotation (**B**). By activating the switches S2 and S3, the left terminal is connected to ground and the right one to V_{in} , resulting in a counterclockwise rotation (**C**). Activating S1 and S2 or S3 and S4 can be used to break the motor as its coils are shorted. Activating S1 and S3 or S2 and S4 should be avoided as it shorts the supply voltage to ground (Tieze and Schenk, 1990).

In this work the integrated motor driver L6203 from SGS-Thomson is used to control the amount and the direction of the current flowing through peltier elements. It contains a H bridge build up by DMOS power transistors as switches combined with a logit circuit. It has two TTL compatible digital inputs. One toggles the transistors used as switches S1 and S3, the other one these of S2 and S4 in a way that either S1 or S3 are activated by the one and either S2 or S4 by the other input. This avoids the possibility of shortening the supply voltage to ground as described above. It allows a supply voltage of up to 48 V, can drive a current of up to 4 A and can be operated at frequencys up to 100 kHz (SGS-THOMSON Microelectronics, 1997).

2.2.4 Digital temperature sensor

To measure the array temperature the digital temperature sensor TSICTM 506 is used. It measures the temperature in a range from $-10\text{ }^{\circ}\text{C}$ to $+60\text{ }^{\circ}\text{C}$ with an accuracy of 0.1 K and a resolution of 0.034 K and is calibrated by the manufacor. The 11 bit temperature value is transmitted serial and can easily be read by a microprocessor (Thermo Technik GmbH, 2014).

2.2.5 Seven segment driver

A seven segment display unit consists of 8 elements, which can be turned on and off separately and a common connection for the current backflow. The numbers from 0 to 9 and a decimal point can be displayed in a good readability using the seven number and the decimal point segment. To control each segment of a unit, 8 digital signal lines are required without a driver. Using 3 digits it would be even 24 one. The human eye is based on photochemical reactions and their processing making it slow compared to the timescales of a microprocessor. The common connections of the displays are used for a time sharing of the complete information between the digits. If the frequency of the next digit position being allowed to display its content is fast enough, the human eye can see no difference between the digits being all steady on except of a loss of brightness. The state of a display unit fits into one byte. Wired up the state of a display unit is transferred parallel with eight lines. This allows the defined switching between the data for each display unit while iterating over the common connectors. Because digital output lines

are mostly rare there are 7 segment drivers managing all the multiplexing and communicating serial to the user. The wanted values for the digits of the display are transferred serially to the driver. The input is read into an internal memory, which is used as value for the digits until changes are received. To be able to cascade these drivers to display more numbers than one alone could handle an additional carriage select signal line can be used. Using it, the master device can select by that line, on which display driver to update the values next (Tieze and Schenk, 1990). The MAX7221 7 segment driver from MAXIM was used in this work. It can drive up to eight digits and allows further cascading. The data is transferred serial using the SPI protocol. It allows brightness control by pulse width modulation. Only one resistor is needed to set the current for the segments (MAXIM, 1997).

2.2.6 Microcontroller board

The arduino Micro microcontroller board was used for the PID controller. It is based on an ATmega32u4 microcontroller with 16 MHz clock and 2.5 kB SRAM. It provides 20 digital IO pins of which 7 can be used for pulse width modulation (PWM) and 12 as analog input channels. It provides 32 kB flash memory for the executable code of which 4 kB are used for the boot loader. 1 kB EEPROM can be used for the nonvolatile storage of data. It has an integrated USB 2.0 port, that is used to program the controller. It can also be used to communicate with a PC to transmit data (Arduino LLC, 2015; Atmel Corporation, 2014).

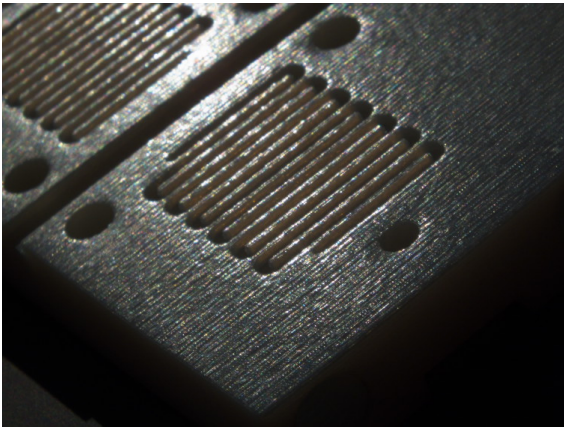
2.3 Software

This document was typesetted with L^AT_EX using T_EXnicCenter v. 1.0. References were included using B_IB_TE_X. Text was edited using Notepad++ v. 6.7.5. Calculations were performed with Matlab R2014a and Octave v. 8.3.2-5. To plot diagrams Microsoft Excel v. 14.0.6129.5000 was used. Technical drawings were made with AutoCAD 2015 SP2. 3D models were produced using Inventor 2015 and AutoCAD 2015 SP2. Arduino IDE v. 1.6.1 was used to program the microcontroller. Code::Blocks v. 13.12 was used for C programming on the PC. KiCAD 2013-07-07 BZR 4022 stable was used to draw circuit diagrams to create component footprints and to develop PCB layouts. Pictures were rendered using Silkipix RAW File Converter v. 3.2.2.0 and edited using Paint Shop Pro v. 5.0 and Gimp v. 2.8.6. Vector graphics were produced and edited using Inkscape 0.91.

2.4 3D Printing

The fluidic parts and ventiles were produced using the Object Eden260V 3D printer from Stratasys and the polymer Vero White. The printer allows object sizes up to 255 x 252 x 200 mm with a horizontal layer size of 16 μm . The printing head moves with a resolution of 600 dpi at the X and Y axis and with 1600 dpi at the Z axis. To print hollow structures a supporting material is used. It can be removed after the printing process using sodiumhydroxide solution. The printer has eight printing heads for the object and the supporting structure. The basic material is a solution from acrylate monomers and oligomers and is applied in small droplets, like in an inkjet printer. The liquid is then polymerized using UV light (Stratasys Ltd., 2015).

2.5 Modifications of printed parts



(a) Surface roughness of the printed fluidic part



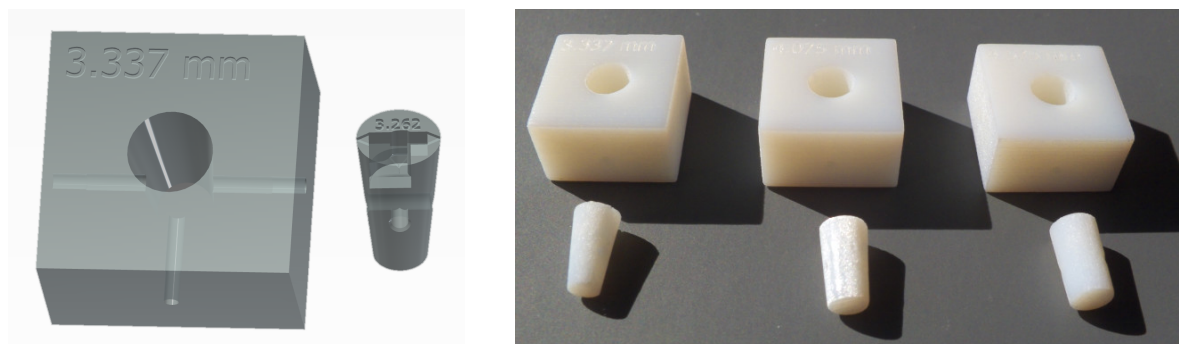
(b) Wet sanding process

Figure 5: Surface roughness caused by the printing process

The surface of 3D printed parts shows a rough structure as shown in figure 5 (a). To be able to bond plane glass sheets onto its side containing the cooling channels, the surface was wet sanded. To archive a flat surface sand paper was glued onto a glass sheet and the printed part was moved on its wetted surface in an eight-shaped path.

In two steps threads for the fittings were cutted into the printed material. Therefore a hollow structure in the diameter of the core drill was printed. To correct tolerance errors in the position of the threads in the cooling plate, a round file and a scalpel were used. To correct the deepnes of the structures to insert the temperature sensor a sperical cutter mounted onto a Dremel rotary tool was used.

2.6 Valve development using test bodys



(a) 3D model of the test body and the ventile

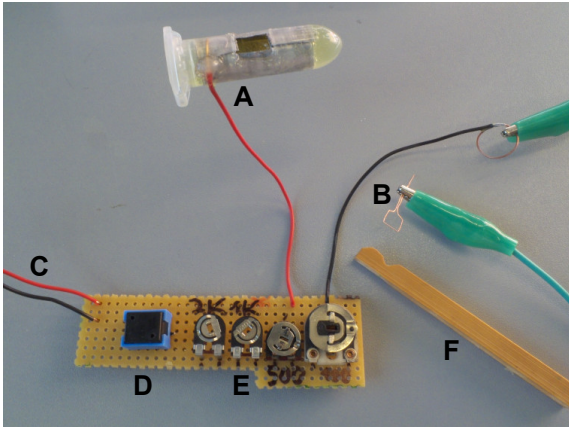
(b) Test bodys with different conical angels

Figure 6: 3D model of the test body, the ventiles and test bodys with different conical angles

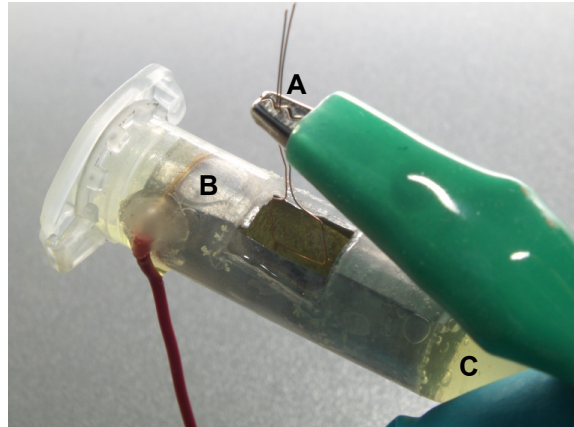
The designed three way valves are conically shaped and can be removed by a hook, that is inserted and turned about 90° . To determine the best angle of the conical shape three versions with different angles were printed together with corresponding test bodys. The test bodys contain three channels and a hollow shape to put in the ventile as shown in figure 6 (a). The ventiles were pressed into the test bodys and a needle with a syringe was put into the channels of the test body. Water was pressed through every channel of the ventile to test its function. To determine if they close tight against the environment the test body with the ventiles were submerged in water and air was pressed into the channel while the ventile was in a position blocking it.

2.7 Galvanization

The galvanization setup developed to plate the wires, allowing the electrical connection to the QCM-D chips by being wrapped around the sealing rings is shown in Fig. 7 (a). The galvanization chamber (A) is filled with a manufacturers electrolyte solution containing gold ions. The used wire is shaped by a wire shaping tool (F) and then submerged into the electrolyte in the galvanization chamber as shown in 7 (b). The circuit board is connected to a 3 V DC power supply by the wires (C). The potentiometers (E) allow the adjustment of the current while the plating process in a wide range of intensitys and current per area. The pushbutton (B) can be used to define the plating duration more exactly than disassembling a hole plating setup.



(a) Galvanization setup; A: Galvanization chamber, B: Target, C: Power supply, D: Pushbutton, E: Current control, F: Wire shaping tool

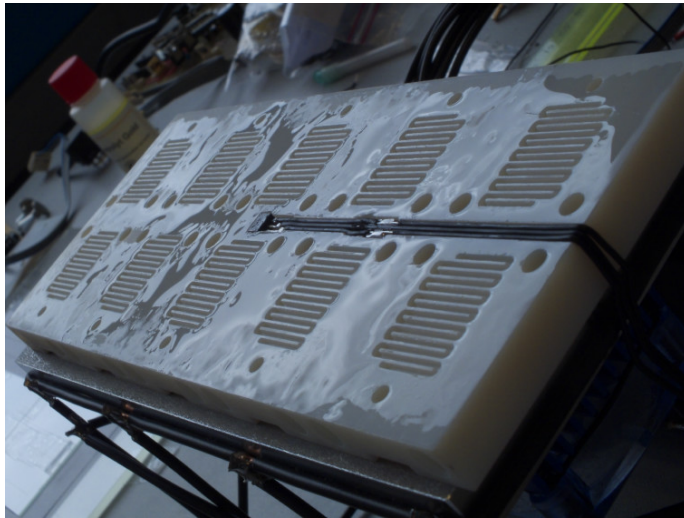


(b) Galvanization chamber; A: Wire as cathode, B: Round shaped metal sheet anode, C: Electrolyte solution

Figure 7: Galvanization circuit and chamber

2.8 UV bonding

After the sanding process the printed fluidic part was sonicated in water several times. Remaining water was removed using nitrogen and the part was allowed to dry for an hour. The UV bonder was allowed to reach the room temperature in 30 minutes. It was applied to the printed part using a 1 mL syringe with a needle. The needle allowed to apply the bonder to the dividers of the cooling channels. The printed fluidic part with the applied bonder can be seen in figure 8 (a). Glass sheets were cut to cover 2 or 1 of the cells. The sheets were sanded to remove sharp edges and fixed together using transparent tape. Then they were put onto the prepared fluidic part and exposed to the radiation of a UV lamp in the wavelength range the bonder adsorbs UV light best for about half an hour as seen in figure 8 (b). Remaining bonder on the sides was removed using acetone (DELO Industrie Klebstoffe, 2014).



(a) Application of the UV bonder



(b) UV gluing

Figure 8: Bonding between the wet sanded fluidic part and glass plates using UV light

2.9 Printed circuit board etching

The designed layout of the printed circuit board for the temperature controller was printed onto transparency film, using an inkjet printer. The foil was fixed onto the copper side of a circuit board coated with photoresist and exposed to sunlight and a UV lamp. The photoresist exposed to the light was removed using sodiumhydroxide solution with a concentraion of 10 g/L. The board was etched using sodium persulfate solution in a concentration of 120 g/L at a temperature of about 60°C. The glass plate used to keep the foil plane adsorbed most of the UV light leading to bad results. Finally the PCB was produced at the Institute for Data Processing and Electronics.

2.10 Determination of controller parameters

The principle of a control loop is shown in figure 9 (a). Changes in the system input lead to changes in the system output. The system input could e.g. be the position of a valve. Changes in its position cause changes in the flow though the valve, which is the system output. The system output is measured by a sensor. The difference between the measured system output and the wanted setpoint is built and fed as input into the controller. Depending on the error and its behaviour over the time the controller output changes. The controller output is coupled to the system input, closing the control loop.

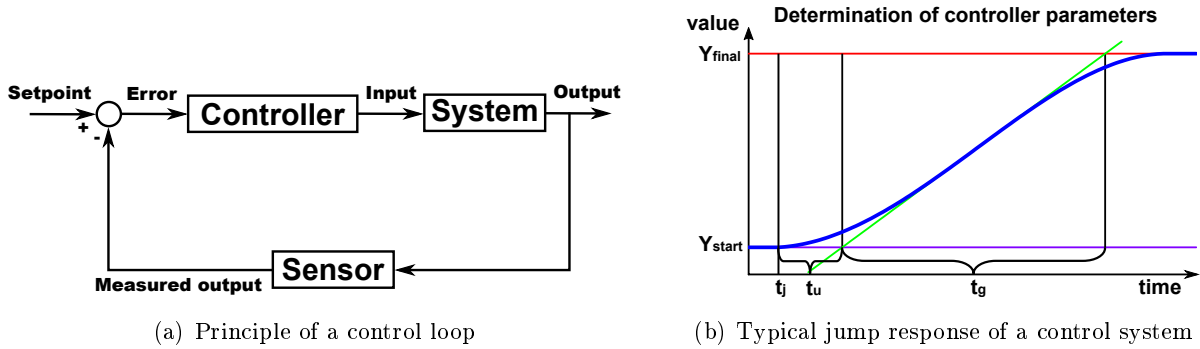


Figure 9: Principle of a control loop and typical jump response

The most commonly used controller type is the PID controller. PID is an acronym for proportional, integral and derivative. Its behaviour is determined by three values: (i) its proportional gain K_p , its integral constant K_i and its derivative constant K_d . The proportional gain determines the amount of the controller output, that directly depends on the input. The integral constant determines the amount of the error, that is summed up over the time and added to the controller output. The derivative constant determines the amount the controller reacts to the speed of changes in the error (Unbehauen, 1989a).

The controller parameters can be determined by a mathematical model of the control system. This is mostly done for easy control systems. If the number of influencing energy reservoirs and their interaction is too complex for a mathematical modelling, empirical rules can be used to determine the parameters. Figure 9 (b) shows the typical response of a control system to a jump in its input at the time t_j over the time. Y_{start} is the value of the system to be controlled in its stable state at the time of the input jump and Y_{final} is the final state the value approaches after the input jump. The difference between the time of the input jump and the section between the first inflexion tangent and the start value is called t_u . The difference between t_u and the intersection of the tangent with the final value Y_{final} is called t_g .

The empirical rules by Chien, Hrones and Reswick shown in table 1 were used to determine the controller parameters. The jump response of the system was recorded three times for heating and for cooling. For heating an input jump of 50 PWM was used, because it is very efficient. For cooling its complete intensity of 255 PWM was used.

The jump responses were recorded via the USB interface and the comma separated files were imported into Matlab to determine the inflexion point. Listing 1 shows the performed calculations. The variable $maxC1$ contains the start temperature, $minC1$ the final temperature in steady state, $jumpTimeC$ the time point at which the input jump was applied and $jumpC$ the amount the input was changed at $jumpTimeC$. The system gain is the ratio between the

Parameter	Control response	Disturbance response
T_n	$2.4 \cdot T_u$	T_g
T_v	$0.42 \cdot T_u$	$0.5 \cdot T_u$
K_p	$0.95 \cdot \frac{T_g}{T_u \cdot K_s}$	$0.6 \cdot \frac{T_g}{T_u \cdot K_s}$
K_i	$\frac{K_p}{T_v}$	$\frac{K_p}{T_v}$
K_d	$K_p \cdot T_v$	$K_p \cdot T_v$

Table 1: Rules by Chien, Hrones and Reswick for the determination of PID controller parameters without overshoot

amount the temperature changed *diffC1* and the amount the input was changed. To determine the inflexion point a polynom *polyC1* with order 10 was fitted to the measured data imported into the variables *TimeC1* and *TempC1*. Its first and second derivative *polyC1d* and *polyC1dd* were determined. The zero points *zeroC1dd* of the second derivative were determined and the real one at the fitting time point *xC1* was selected manually. The gain of the inflexion tangent *mC1* is the value of the first derivative of the polynom at the time *xC1*. The time points *TuC1* and *TgC1* result from the intersections between the tangent and the start and final temperature as shown in figure 32. The rules by Chien, Hrones and Reswick for control response without overshoot were finally applied to determine the controller gain *KpC1*, the integral constant *KiC1* and the derivative constant *KdC1* (Unbehauen, 1989a,b; Korsane et al., 2014). The jump response was recorded for heating and cooling three times and the arithmetic average of the resulting constants were used for the controller.

```

1 KsC1 = diffC1 / jumpC
polyC1 = polyfit (TimeC1, TempC1, 10)
3 polyC1d = polyder (polyC1)
polyC1dd = polyder (polyC1d)
5 zeroC1dd = roots (polyC1dd)
xC1 = zeroC1dd (7)
7 yC1 = polyval (polyC1, xC1)
mC1 = polyval (polyC1d, zeroC1dd (7))
9 TuC1 = ((maxC1 - yC1) / mC1) + xC1 - jumpTimeC
TgC1 = ((minC1 - yC1) / mC1) + xC1 - jumpTimeC - TuC1
11 TnC1 = TgC1
TvC1 = 0.5 * TuC1
13 KpC1 = (0.6 * TgC1) / (TuC1 * KsC1)
KiC1 = KpC1 / TnC1
15 KdC1 = KpC1 * TvC1

```

Listing 1: Matlab code used to determine the controller parameters

2.11 Calibration of the current measurement

The shunt resistors were disconnected from ground to calibrate the current measurement. A defined current in the range from 0.5 A to 3.5 A was driven through the resistors using the current limitation function of the laboratory power supply. The current was additionally measured with a multimeter. The readings from the analog to digital converters (ADCs) were recorded. Assuming a linear behavior between the current and the measured voltage at the analog input a linear regression was performed. The gain was used to calculate the current flowing through the resistor and thereby through the peltier elements (Felderhoff, 1990).

2.12 Calibration of the room temperature sensor

A negative temperature coefficient resistor (NTC) was used to estimate the room temperature and thereby adjust the controller parameters to whether the main task is heating or cooling. To calibrate the sensor it was fixed on the cooling plate next to the digital temperature sensor. The temperature was set to values between 20 °C and 40 °C in steps of 1 K and the voltage resulting in the voltage divider between the NTC and a resistor was determined. As the readings showed a highly linear response to the temperature the slope of the linear regression through the measured points was determined and used to calculate the room temperature from the readings of the ADC (Herold, 1993; Schröder, 1984).

3 Results

3.1 QCM-D Array

3.1.1 Concept

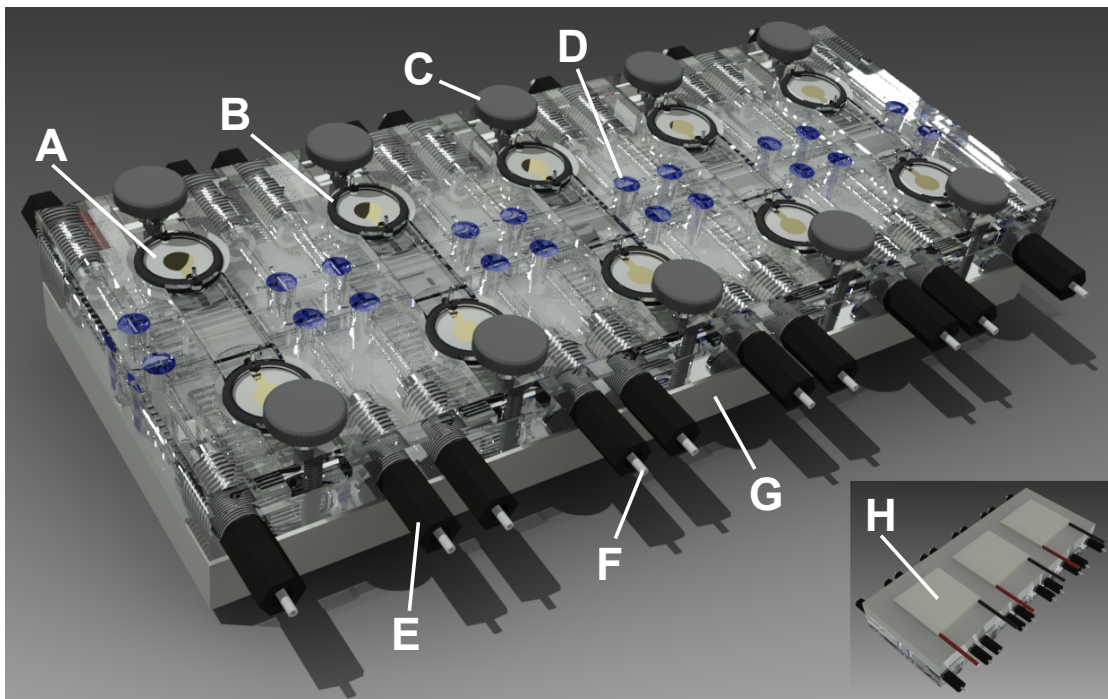


Figure 10: 3D Model of the QCM-D Array; A: QCM-D chip, B: Sealing ring, C: Fixing screw, D: Ventile, E: Tube fitting, F: Teflon tubing, G: Steel plate, H: Peltier element

The 3D model of the complete fluidic array is shown in figure 10. The array consists of a lower part containing the fluidic structures and an upper part providing the electrical connections and the fixation of the QCM-D chips. The array can be loaded with up to ten QCM-D chips (A). The chips are fixed between two sealing rings (B). One fixing screw (C) per chip allows a position independent defined pressure over the chip. Samples enter the array through teflon tubings (F)

with fittings (**E**). Below the fluidic part a steel plate (**G**) with peltier elements (**H**) allows to control the temperature of the samples and the array.

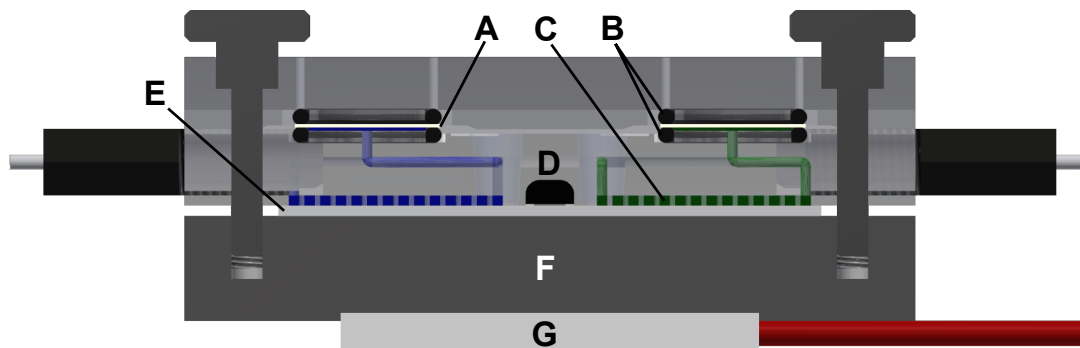


Figure 11: Section through the fluidic array; A: QCM-D chip, B: Sealing rings, C: Cooling channels, D: Temperature sensor, E: Glass sheets, F: Steel plate, G: Peltier element

Figure 11 shows a section through the middle of the array. The QCM-D chips (**A**) are fixed between two sealing rings (**B**). Before the sample reaches the chip, it flows through cooling channels at the bottom of the array (**C**). The temperature sensor (**D**) is located at the same level as the cooling channels, allowing to control the temperature of the sample flowing through the channels. The cooling channels are sealed with glass sheets (**E**) and fixed to the steel plate (**F**) with screws.

On one side the steel plate has ten threads for the fixing screws and three indentations for the peltier elements on the other side. Figure 19 shows the technical drawing given to the workshop for the production of the plate. Steel was chosen as material for the plate because of its high thermal capacity and its quite low thermal conductance. Caused by the low thermal conductivity the plate needs about half an hour to reach its target temperature, but once reached it also holds its temperature some time. This helps to keep the temperature of the samples and the chips constant against external disturbances.

The internal hollow structure of two cells of the array are shown in figure 12. The sample enters the array through the first fitting (**A**) and flows through the first ventile (**B**) into the cooling channels (**C**). The channels are used to bring the sample to the measurement temperature by contact with the temperature controlled glass plate that covers it below. The sample flows from the cooling channels over the QCM-D chip (**D**) to the next ventile (**E**). There it can either be routed to the first exit fitting (**F**) to leave the array or to the next cell (**G**). At the edges of the array the sample can be routed to the other row (**H**) allowing every possible sample distribution between the ten cells.

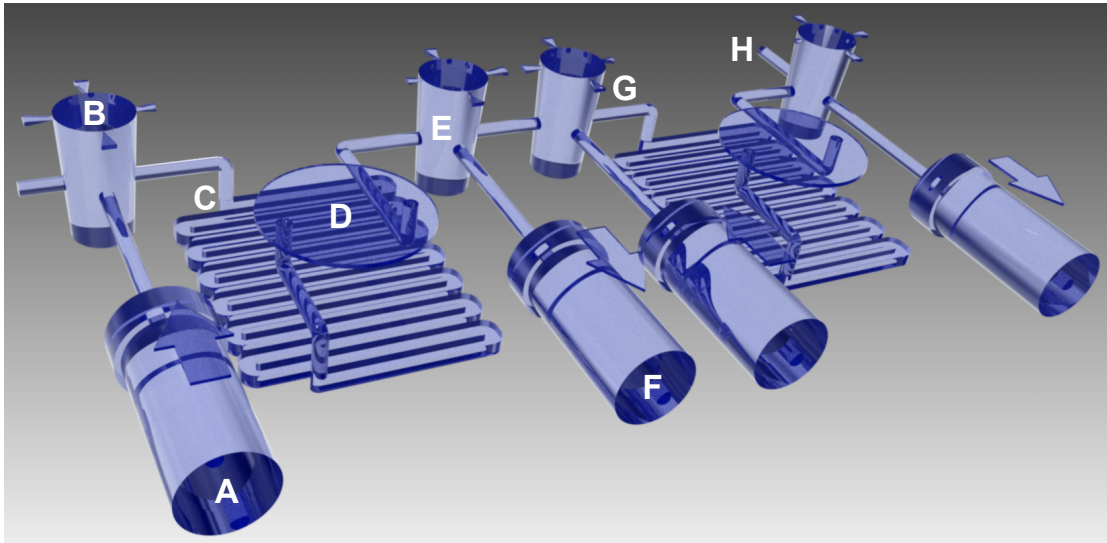


Figure 12: Structure of the channels inside the fluidic array; A: Sample inflow through first fitting, B: Input ventile, C: Cooling channels, D: Sample volume below the QCM-D chip, E: Exit ventile, F: Exit through fitting, G: Exit to the input ventile of the next chamber, H: Routing of the sample over the edge

3.1.2 Ventiles

The three way ventiles used to route the sample are shown in figure 13. The best results for removability and tightness were resulted with the diameter of the lower part of the conical shape of 4.075 mm. The removal tool shown in figure 13 (b) is used to remove the ventiles from the array to rearrange the sample distribution. It consists of copper wire, shaped to a hook at one side and soldered to a loop at the other side (figure 13 (c)). To be able to remove the ventiles they contain a hollow structure at their top shown in figure 13 (a). It allows the tip of the removal tool to be inserted and rotated by 90°. By throwing at the removal tool the ventile can be removed from the array.

3.1.3 Printed fluidic parts

Figure 14 shows the printed fluidic parts. In the top part of the array the gold plated electrical contacts (**A**) are wrapped around the sealing rings (**B**). The sample reaches the chips through the channels (**C**). The ventiles (**D**) determine the way of the samples through the array. The chip holding mechanism was adopted from the commercial available flow cell QFM 401 contained in the Qsense 4 QCM-D system. The used chips, sealing rings and fittings were original spare parts available for the system (LOT-QuantumDesign GmbH, 2015).

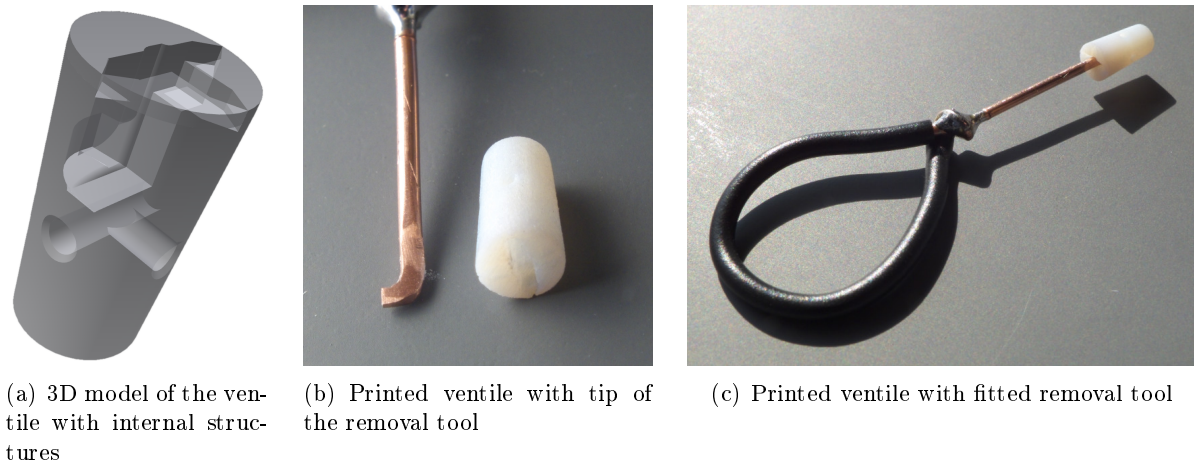


Figure 13: Internal structure of the ventiles and the ventile removal tool

3.1.4 Flexible sample routing

Figure 15 shows the printed fluidic part equipped with the sealing rings and the glass sheets loaded with ten QCM-D sensors. The ventiles are inserted in the positions allowing a routing of four samples over three groups of three chips and the remaining single chip as shown in figure 16. After closing the array four pump tubes and four tubes for the sample inflow were connected to the array.

The result of using four samples differently colored using food color is shown in figure ???. To be able to see the samples in the cooling channels the upper part of the array was fixed with nuts and the array was placed upside down. The channels are all tight and the ventiles are able to route the samples over the chips as intended (compare to figure 16).

3.1.5 Cooling plate holder with fans

The peltier elements were fixed into the hollow structures of the cooling plate using thermal compound pads. Chipset coolers were used to archive a better heat flow. They consist of a heat sink with thermal compound and a fan mounted onto it. The direction of the airflow of the fans was initially from the ambient to the cooler. As this leads to a heating up of the plate in the cooling mode by the warm air flowing over the plate the fans were removed and mounted in the opposite direction using wire as spacer. The cooling plate is fixed in a holder made of insulated copper wire shown in figure 18. At its back side additional fans suck in ambient air and remove the hot or cold air from the chipset coolers to the back side.

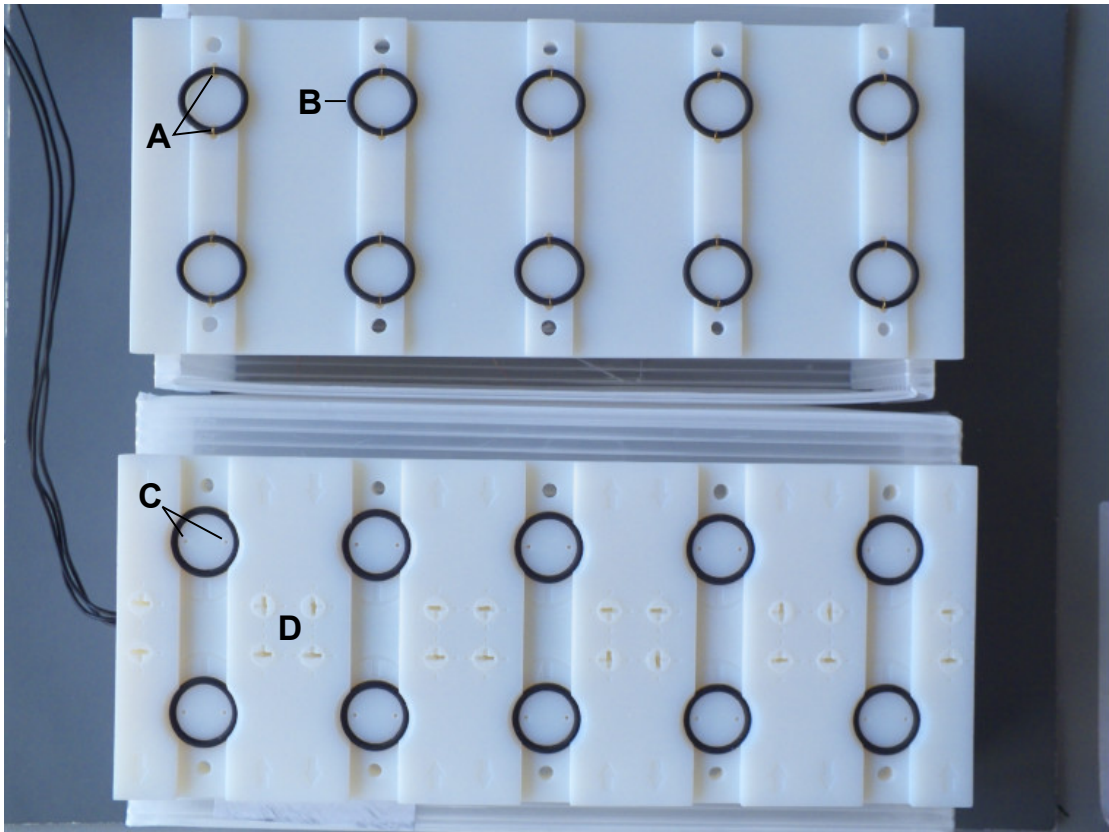


Figure 14: Printed fluidic parts; A: Gold plated electrical contacts, B: Sealing ring, C: Sample channels for the QCM-D chip, D: Ventiles

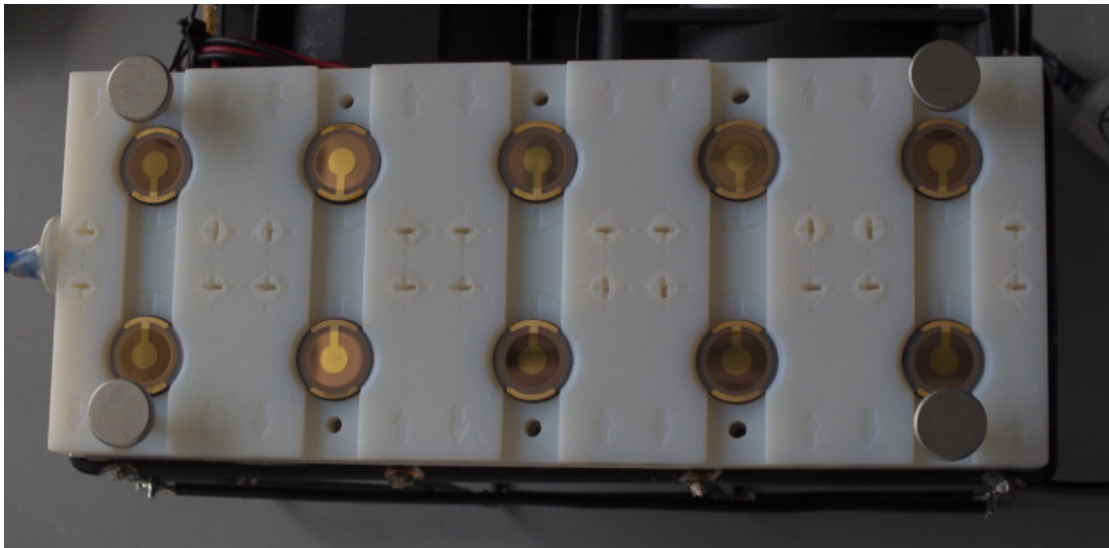


Figure 15: Fluidic loaded with QCM-D chips

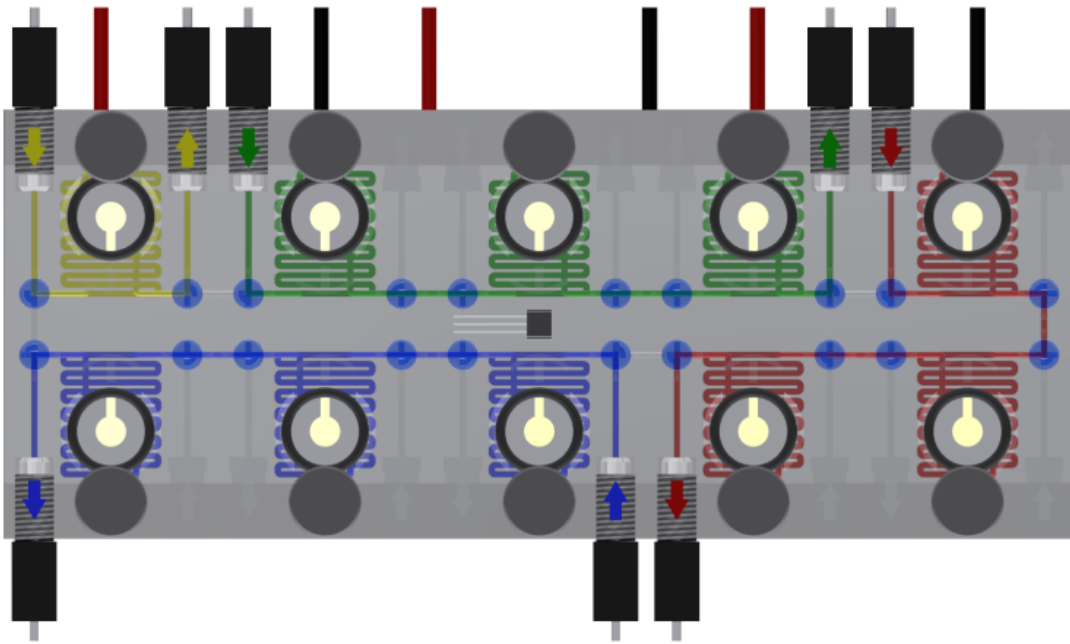


Figure 16: Flexible sample routing

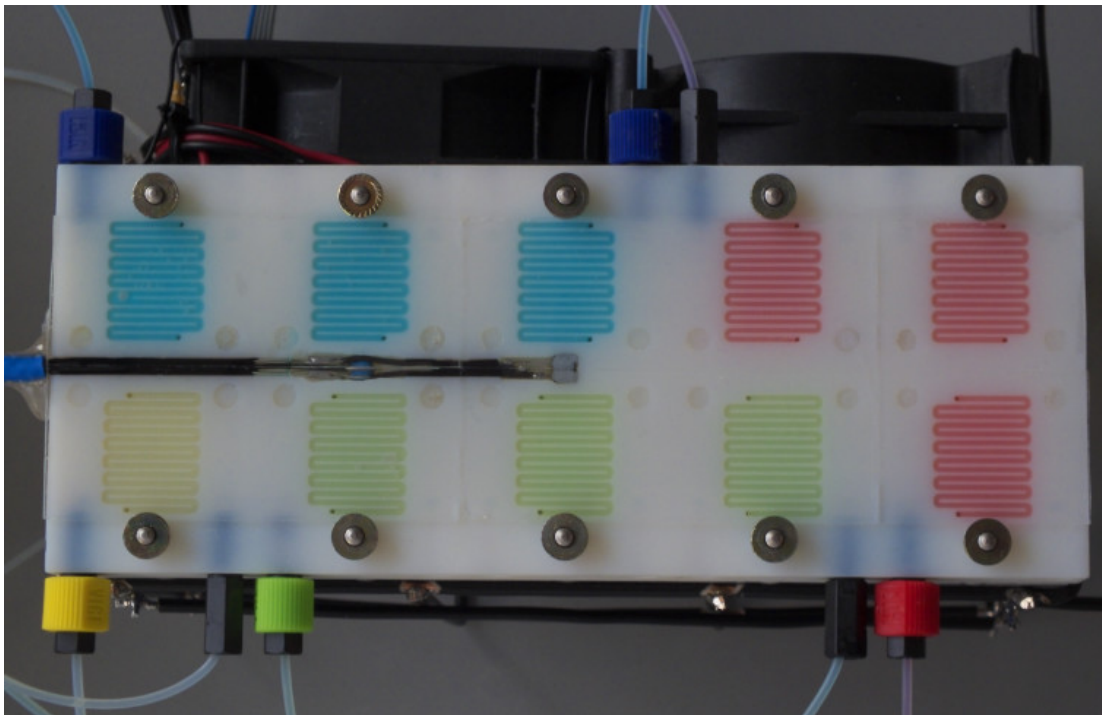


Figure 17: Routing of four samples in the printed array



Figure 18: Cooling plate holder with fans

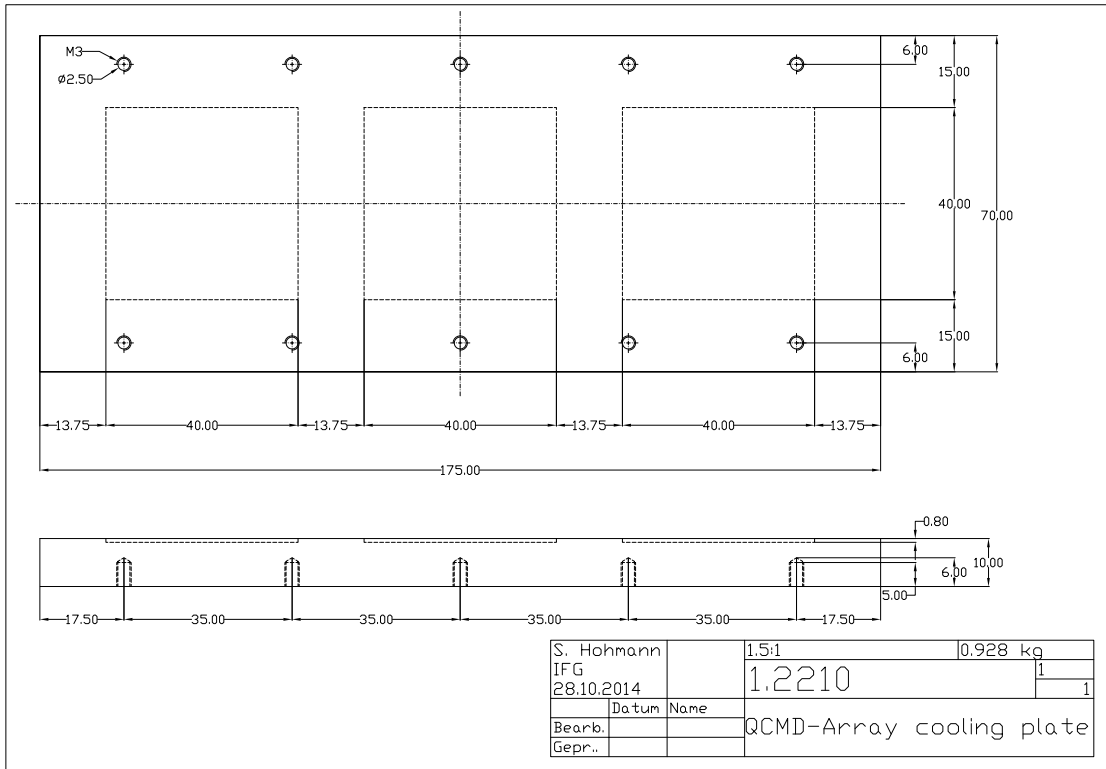


Figure 19: Temperature control steel plate

3.2 Temperature control

3.2.1 Concept

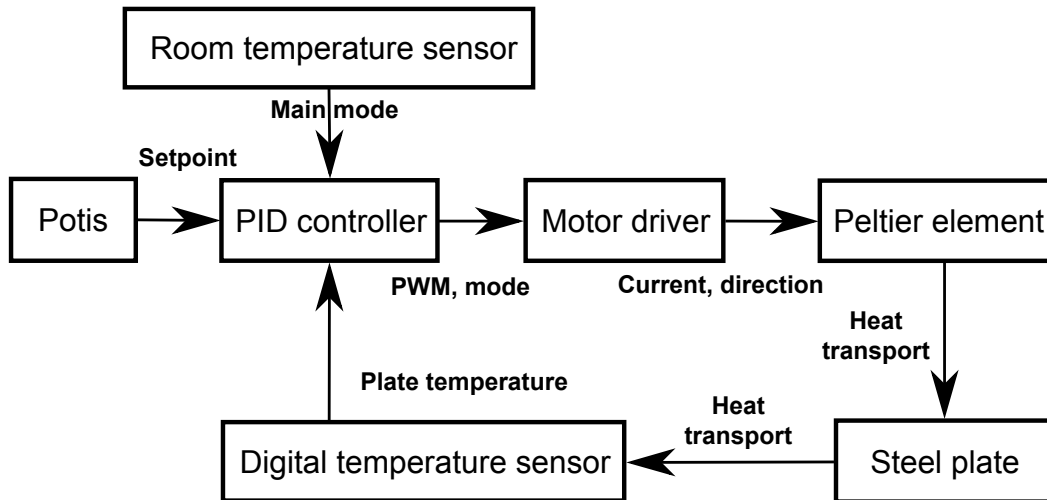


Figure 20: Control loop

The conception of the temperature controller is shown in figure 20. The wanted temperature is set using two potentiometers. They are connected as voltage dividers between ground and the stabilized voltage of the arduino board. They divide the voltage on their outer contacts to a fraction of it on their sliding contacts depending on the angle their axis is moved to. The voltage is measured using two of the analog to digital converter channels contained in the microcontroller board. One potentiometer is used to set the coarse temperature. Its ADC reading is mapped to an output between 20 and 40. The output of the one used to set the fine part of the temperature is mapped to values from 0 to 9. Combining their outputs the temperature can be set between 20.0 and 40.0 degree celsius. Readings of the fine potentiometer are ignored, if the coarse setting is 40 °C. The room temperature sensor is used to estimate the room temperature and thereby to determine the main operation the controller has to fulfill. If the setpoint is below the room temperature the main mode is cooling and the determined controller parameters for cooling are used. If it is above the room temperature the heating mode and parameters are used. The outputs of the PID controller are the mode signal changing the direction of the current supplied to the peltier elements and a pulse width modulated signal, controlling the amount of current. The peltier elements mounted onto the cooling plate heat or cool the plate depending on the direction of the current flow through it. The plate temperature sensor is mounted inside the

fluidics at the same height, the sample flows through the cooling channels. Its output is read by the PID controller closing the control loop.

3.2.2 Circuit

3.2.2.1 Power supply

Figure 21 shows the circuit diagram of the power supply unit of the temperature controller. An external laboratory power supply is connected between $12VC$ and GND . The electrolyte capacitor $C1$ buffers the voltage against fast changes in the drawn total current. The $12V$ voltage is used to drive the peltier elements and the fans. $U3$ is a linear voltage regulator of the type 7809, which stabilizes its input voltage down to 9 V. The 9V voltage is used as input for the microcontroller board, which stabilizes down to 5 V internally. This 5V *Arduino* is used as reference voltage for the ADC channels of the board. To receive accurate readings it is also used for the potentiometers, the voltage divider of the room temperature sensor and the current measurement. The 9V voltage is further stabilized down by the second linear voltage regulator $U6$ of the type 7905. Its stabilized 5 V voltage is used for the 7 segment display and the bus driver between the microprocessor board and the peltier driver units.

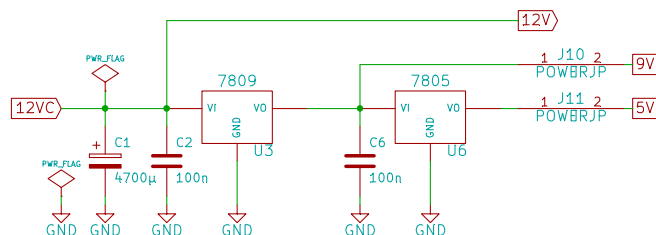


Figure 21: Circuit of the power supply for the temperature controller

3.2.2.2 Peltier driver unit

One of the three used peltier driver units is shown in figure 22. The used motor driver $U7$ of the type L6203 is supplied with voltage from the 12V line. Its input voltage and its internal voltage reference output are buffered using a 100 μ F electrolyte and a 100 nF ceramic capacitor. They were located as close as possible to the driver on the circuit board. The bootstrap capacitors $C11$ and $C12$ and the fast switching diodes $D5$ and $D6$ of the type UF 4508 help to supply enough charge for the transistors in the driver allowing a fast switching. The capacitors $C13$ and $C14$ are used to smooth the PWM current for the peltier elements. They are connected between the two outputs of the driver. The shunt resistor $R19$ is connected to the sense output of the driver. The voltage drop on it can be used to measure amount of current, because all current flowing through the peltier element is also flowing through it.

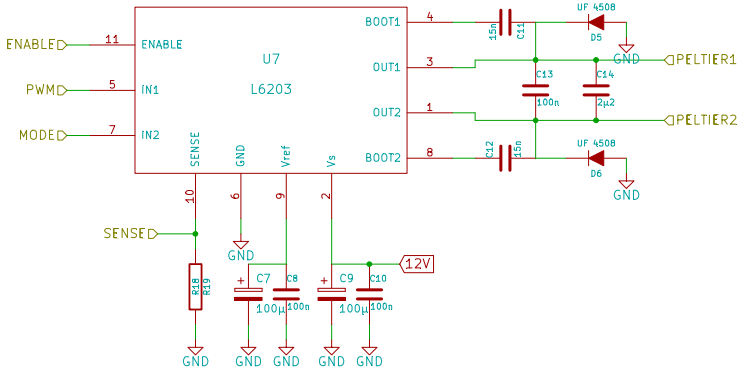


Figure 22: Circuit of the peltier driver unit of the temperature controller

of its states while the total interval of the pulse width allows to control how long the peltier element is supplied with current (Pang et al., 2015).

3.2.2.3 Current measurement

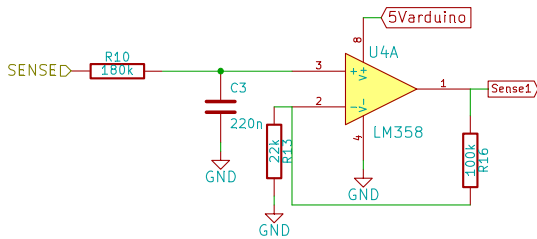


Figure 23: Circuit of the current measurement in the temperature controller

is determined by the ratio of the resistors $R13$ and $R16$. It was set to result in the maximum output voltage of the amplifier for a voltage of 0.9 V at the shunt resistor. Its output is connected to a ADC channel of the microcontroller board (Tieze and Schenk, 1990).

3.2.2.4 Fan driver units

The fan driver units of one of the chip driver fans and the back fans are shown in figure 24. To switch the fans on the peltier elements the general purpose small signal transistor $Q2$ of the type BC547 is used. To switch the back fans the transistor $Q4$ of the type BC639 is used. It allows a higher collector emitter current needed to drive the 80 mm fans on the back. The transistors are

The inputs of the driver are $ENABLE$, PWM , and $MODE$ connected to the enable and switching inputs of the driver. The enable function is used to turn on the peltier driver units after the init process to allow the control. One of the inputs is used to determine the direction of the current flow. The other one is supplied with the PWM output of the PID controller. By changing the duration it is switched to one

One unit of the current measurement is shown in figure 23. The voltage from the shunt resistor is connected the RC pass built by $R10$ and $C3$. It converts the pulse width modulated input into a direct voltage. This voltage is connected to the positive input of the operational amplifier $U4A$ of the type LM358. The amplifier is connected as non inverting amplifier and supplied by the 5V *arduino* voltage. Its gain is

connected with common emitter.

Between their collector and the 12V line the fans and the diodes $D3$ and $D4$ of the type 1N4148 are connected. Applying a voltage to the resistors $R2$ or $R4$ by the microcontroller switches the transistors and connects the terminal of the fan to ground. The diode is used to protect the circuit from induced voltages in the coils of the fans by shorting them. The values of the basis transistors were calculated depending on the amount of collector emitter current needed for the fans and the gain of the transistor. The transistors are saturated at these working points to allow a fast switching.

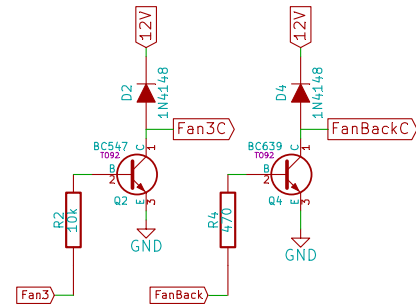
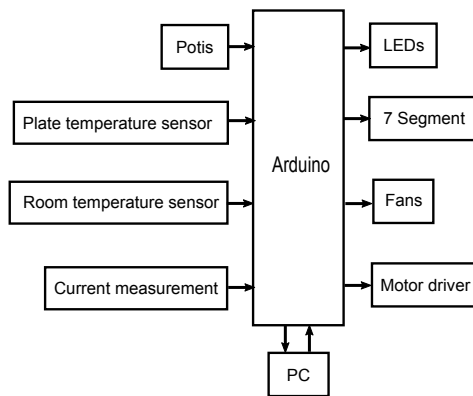
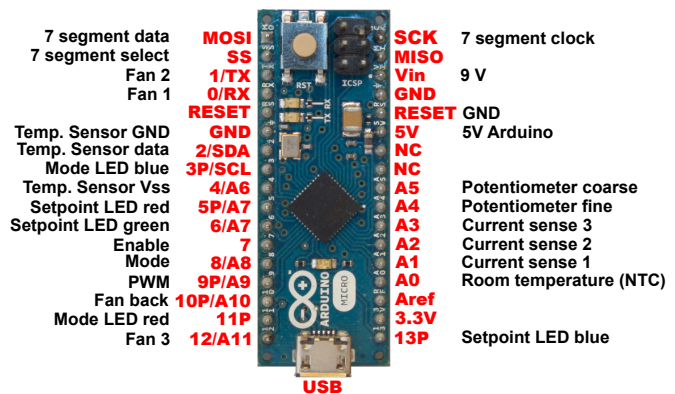


Figure 24: Circuit of the fan driver unit of temperature controller

3.2.2.5 Usage of the microcontroller board



(a) Overview of the connections



(b) Detailed pin usage of the arduino board

Figure 25: Usage of the arduino microcontroller board as PID controller

Figure 25 (a) gives an overview of the connections to the microcontroller board. The potentiometers to set the wanted temperature, the plate with the room temperature sensor and the current measurement units are connected to input lines. The setpoint and the mode LEDs, the 7 segment display, the fans and the peltier driver units are connected to its outputs. The USB connection allows the programming of the controller and transmits the control parameters to a PC.

The detailed usage of the Arduino Micro pins is shown in figure 25 (b). The usage of the pins is shown in black, the function of the pins in red. The room temperature sensor, the three current

measurement units and the potentiometers are connected to five of its analog inputs *A0* to *A5*. The digital plate temperature sensor is connected to ground, Pin 4 and Pin 2. Pin 4 supplies the voltage for the sensor allowing to turn it on for the measurement. Its transmitted data is read from Pin 2. The 7 segment display is connected to the ICSP pins *SS*, *MOSI* and *SCK*. *SS* selects the display driver for data transmission. *MOSI* is used to sent the values to display to it serially. *SCK* provides the clock for the serial transmission. The fan drivers are connected to the digital pins 0, 1, 12 and 10. The setpoint LED is connected to the PWM capable pins 5, 6 and 13 for its red, green and blue input respectively. The mode LED is connected to pin 3 and 11 with its inputs for blue and red respectively. The green input is not connected as the LED is used to indicate the mode of heating or cooling by its intensity in red or blue only. The enable line of the peltier driver units is connected to pin 7. Whether it is heating or cooling is set by pin 8. Pin 9 is used to supply the PWM signal for the peltier drivers. The board is supplied with power by the 9V line. Its on board stabilized 5 V voltage is supplied to the potentiometer, room temperature sensor and current measurement circuits over the 5V *arduino* line (Wheat, 2011).

3.2.3 Printed circuit board

The copper side of the designed printed circuit board (PCB) is shown in figure 26. The common ground of the circuit is supplied by a ground plane over the entire PCB. This allows to reduce influences between the subcircuits. Where the motor drivers are mounted the ground plane was removed to reduce the risk of short circuits and to allow a better dissipation of the heat caused by the current through the copper lines.

Figure 27 shows the component mounting diagram of the PCB. The power is supplied through a connector at the right side in the front of the PCB. Here it has the highest distance to the other parts of the circuit. The three identical peltier driver units are located in the back half of PCB allowing a better heat dissipation of the heat sinks at the back of the planned casing. The microprocessor board is located at the left side of the front of the PCB allowing to access its USB connection from outside of the casing. The connections between the microprocessor board and the peripherals are located as close as possible to the board.

The assembled PCB is shown in figure 28. The Arduino Micro microcontroller board is plugged into a socket (**A**). The bus driver (**B**) is used to protect the microcontroller from possible errors in the peltier driver units and to supply enough current to them. The motor drivers (**D**) are mounted onto heat sinks (**C**). Thermal conductive compound was applied between the back of the drivers and the heat sinks to archive a better thermal conductance. The shunt resistors (**E**) are not mounted directly onto the surface of the PCB to allow a better convection for the heat transport and to protect the insulation of the jumper wires below it. The current measurement

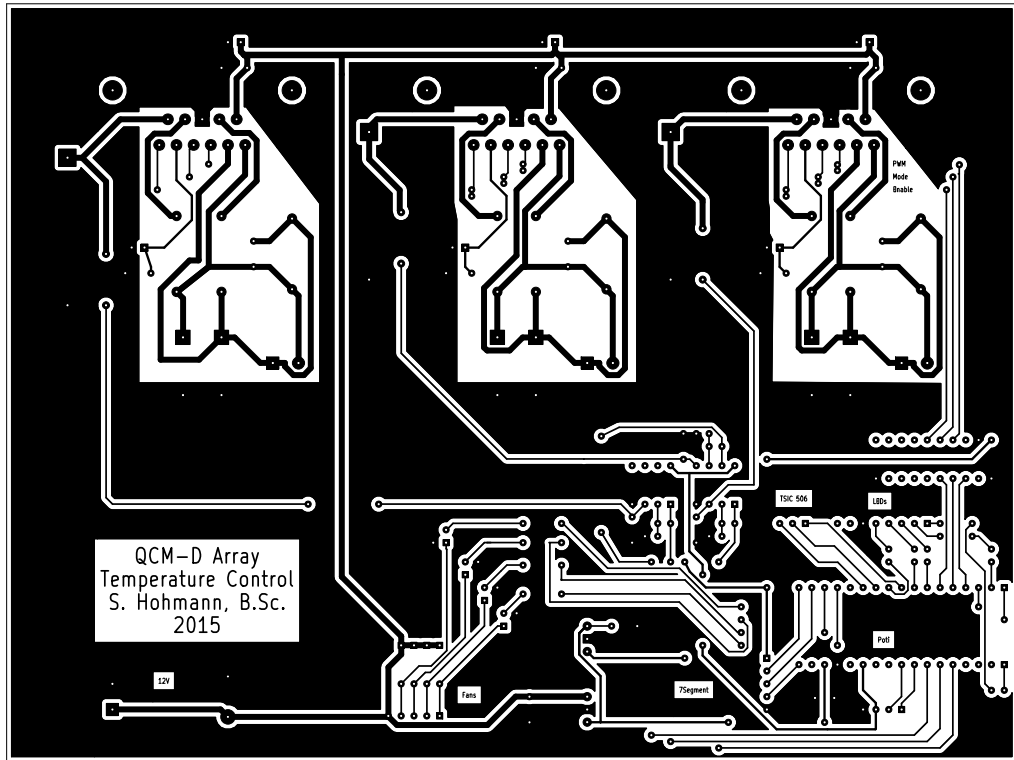


Figure 26: Layout of the printed circuit board

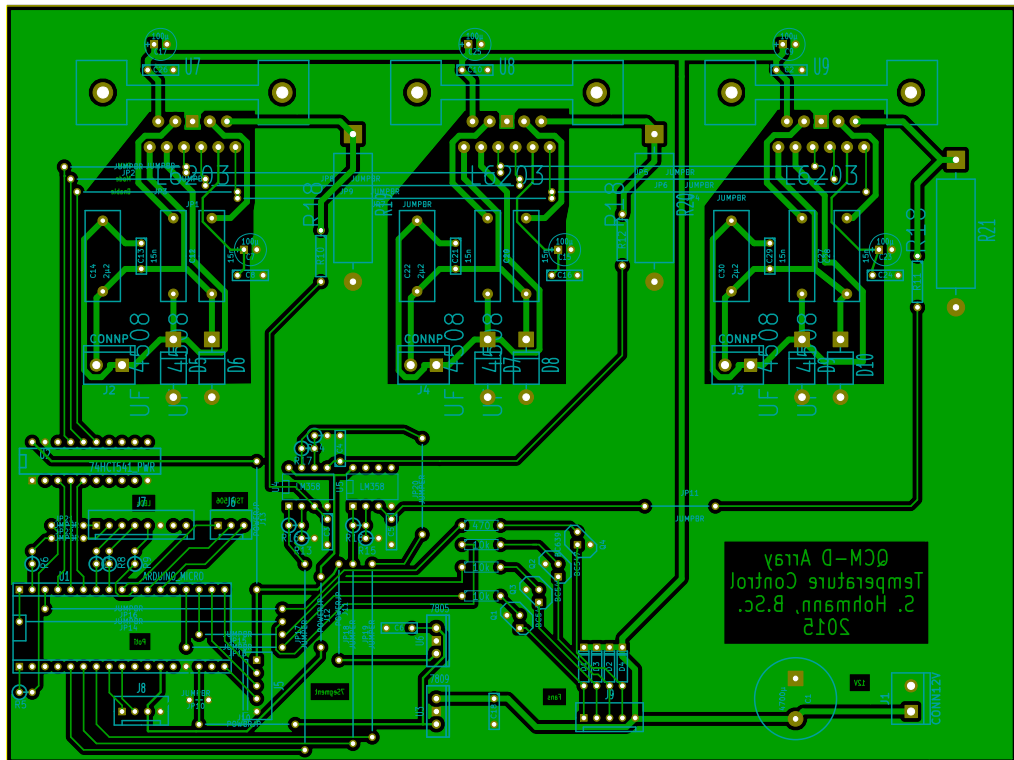


Figure 27: Component mounting diagram of the printed circuit board

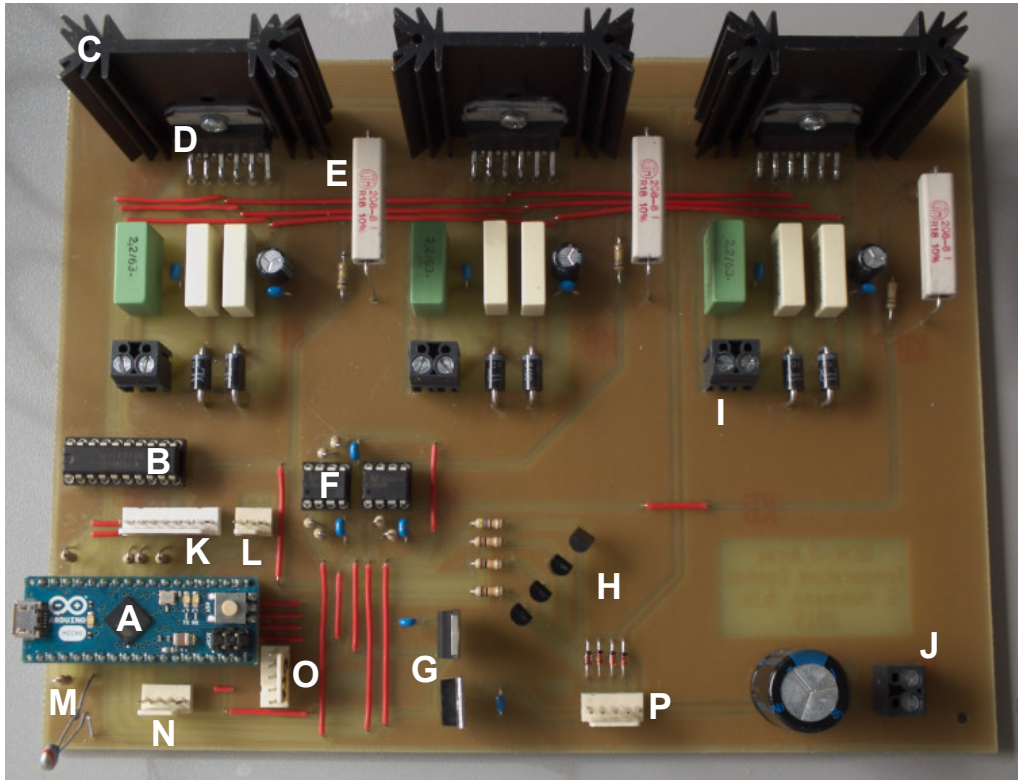


Figure 28: Assembled printed circuit board; A: Arduino Micro, B: Bus driver, C: Heat sink, D: Motor driver, E: Shunt resistor, F: Current measurement, G: Voltage regulators, H: Fan drivers, I: Peltier connector, J: Power supply connector, K: LED connector, L: Temperature sensor connector, M: Room temperature sensor, N: Potentiometer connector, O: 7 Segment display connector, P: Fan connector

subcircuits (**F**) are located near the microprocessor. Because the used LM358 contains two operational amplifiers circuits, only two of them are needed. The unused one is not connected. The linear voltage regulators (**G**) are located left of the fan driver units (**H**). This allows a short distance between the generation of the used voltages and the regulators. The fan drivers use the 12V voltage and are thereby located next to its input (**J**). The peltier elements are connected through the connectors (**I**). They allow to turn in the cables and fix them with the contained screws. This allows them to be easily connected. The PCB connectors (**K**), (**L**), (**N**), (**O**) and (**P**) are used to connect the LEDs, the plate temperature sensor, the potentiometers, the 7 segment display and the fans to the circuit. The room temperature sensor (**M**) was initially mounted directly onto the PCB. Because it did heat up by the heat dissipated from the motor drivers, the distance was increased using wires later.

3.2.4 User interface

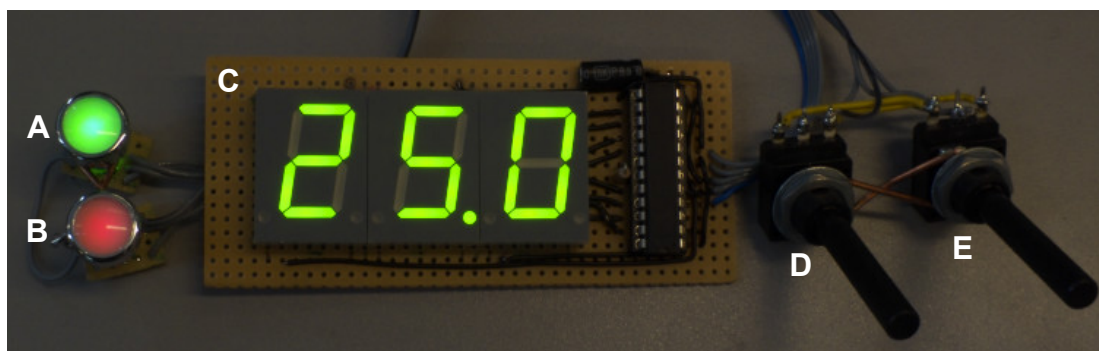


Figure 29: User interface of the temperature controller; A: Status LED, B: Mode LED, C: 7 segment display, D: Coarse setpoint potentiometer, E: Fine setpoint potentiometer

The user interface of the temperature controller is shown in figure 29. It consists of two LEDs (A) and (B), a 7 segment display (C) and two potentiometers (D) and (E). Their usage is described in the following sections.

Setting the wanted temperature

Two potentiometers are used to set the wanted temperature. The left one (D) is used to set the coarse part of the temperature between 20 °C and 40 °C. Is the temperature set with the left potentiometer to a value below 40 °C, the right potentiometer (E) can be used to set the fine part of the temperature between 0.0 °C and 0.9 °C. Is the coarse potentiometer set to 40 °C, the value of the fine potentiometer is discarded.

7 Segment display

The 7 segment display (C) is used to display the actual plate temperature with reduced intensity while the controlling process. Is one of the potentiometers turned it starts to display the setpoint with maximum intensity. The status LED also indicates the setting of the setpoint by its color.

Status and mode leds

Table 2 shows the different colors used to signal different states of the controller and the controlling process. While the setpoint is set and the following 700 ms the color of the setpoint LED (A) color is purple. If the setpoint defined its color changes depending on the difference between the plate temperature and the setpoint. Is the difference 5 K or higher, it is red. Below 5 K but







LED color						
Setpoint difference	Setting	≥ 5.0 K	≥ 2.5 K	≥ 1.0 K	> 0.6 K	< 0.6 K

Table 2: Signal colors of the setpoint LED

above 2.5 K it is orange followed by dark yellow for the range from 2.5 K to 1.0 K, light yellow for above 0.6 K and green for below 0.6 K. The highest accuracy of the used temperature sensor is used as the difference below the LED begins to glow green. The green color indicates, that the setpoint is reached. The mode LED (B) signals the amount of current set through the peltier drivers by its intensity and the operation of the peltier elements by its color. For the cooling process it is blue and for the heating process red.

Init sequence

DCNd-ARRAY tEMPERAturE Control 5. HohNArn 2015

Figure 30: Init message scrolled over the 7 segment display

When the controller is turned on an init sequence is executed. The status LED shows all its possible signal color while the mode LED shows the intensity of the heating or cooling current applied to the peltier element by the intensity of its red or blue light in as many steps as the status LED has signal colors. The fans are turned on in sequence, allowing to verify their function by the user. While the init sequence the message shown in figure 30 is scrolled from right to left over the three digits of the 7 segment display.

USB communication with PC

The output of the programm written in C to read the control process data from the arduino micro to a personal computer is shown in figure 31. When executed from the command line, the program shows its menu (a). By pressing the keys for the numbers 1 to 4, the user can select between the given options. To select the serial port 1 is entered in the menu. The program forks to ListSerialPorts.exe by Tod E. Kurt piping its output into a file. The file is openend and the available serial ports and their descriptions are extracted from it. The serial ports available in the system are listed as shown in figure 31 (b). By pressing the number displayed left of the serial port, the serial port is selected and opened for writing. In this case 2 would have been selected, as it is the virtual serial port *COM7* supplied by the driver for the Arduino

```

=====
# QCM-Array Temperature Control #
#                               #
#   programmed by               #
#                               #
# Siegfried Hohmann, B.Sc. 2015 #
=====

1. Select serial port
2. Monitor control process
3. Monitor and write data to file
4. Exit

```

(a) Menu

```

1: COM1 (Standardanschlusstypen)
2: COM7 Arduino LLC (www.arduino.cc)
-

```

(b) Selection of the serial port

```

Enter file name: setpointjumping-overnight-new.txt
Writing to file setpointjumping-overnight-new.txt
Time: 0.0s Temp: 29.736 SP: 30.00 Diff: 0.264 heating PWM: 106 I: 5.13A
Time: 0.5s Temp: 29.736 SP: 30.00 Diff: 0.264 heating PWM: 242 I: 0.82A
Time: 1.0s Temp: 29.736 SP: 30.00 Diff: 0.264 heating PWM: 242 I: 0.77A
Time: 1.5s Temp: 29.702 SP: 30.00 Diff: 0.298 heating PWM: 223 I: 1.42A
Time: 2.0s Temp: 29.702 SP: 30.00 Diff: 0.298 heating PWM: 240 I: 0.87A
Time: 2.5s Temp: 29.702 SP: 30.00 Diff: 0.298 heating PWM: 240 I: 0.84A
Time: 3.0s Temp: 29.667 SP: 30.00 Diff: 0.333 heating PWM: 220 I: 1.46A
Time: 3.5s Temp: 29.667 SP: 30.00 Diff: 0.333 heating PWM: 238 I: 0.91A
Time: 4.0s Temp: 29.667 SP: 30.00 Diff: 0.333 heating PWM: 238 I: 0.89A
Time: 4.5s Temp: 29.667 SP: 30.00 Diff: 0.333 heating PWM: 238 I: 0.90A
Time: 5.0s Temp: 29.633 SP: 30.00 Diff: 0.367 heating PWM: 219 I: 1.51A
Time: 5.5s Temp: 29.633 SP: 30.00 Diff: 0.367 heating PWM: 236 I: 0.98A
Time: 6.0s Temp: 29.633 SP: 30.00 Diff: 0.367 heating PWM: 236 I: 0.95A
Time: 6.5s Temp: 29.599 SP: 30.00 Diff: 0.401 heating PWM: 217 I: 1.59A
Time: 7.0s Temp: 29.599 SP: 30.00 Diff: 0.401 heating PWM: 234 I: 1.04A
Time: 7.5s Temp: 29.599 SP: 30.00 Diff: 0.401 heating PWM: 234 I: 1.02A
Time: 8.0s Temp: 29.599 SP: 30.00 Diff: 0.401 heating PWM: 234 I: 0.99A
Time: 8.5s Temp: 29.599 SP: 30.00 Diff: 0.401 heating PWM: 234 I: 1.02A
Time: 9.0s Temp: 29.599 SP: 30.00 Diff: 0.401 heating PWM: 234 I: 1.02A
Time: 9.5s Temp: 29.565 SP: 30.00 Diff: 0.435 heating PWM: 215 I: 1.62A
Time: 10.0s Temp: 29.599 SP: 30.00 Diff: 0.401 heating PWM: 252 I: 0.26A
Time: 10.5s Temp: 29.599 SP: 30.00 Diff: 0.401 heating PWM: 234 I: 1.00A

```

(c) Writing controller data to a file

Figure 31: Program to read to read data from the temperature controller to a PC

Micro microcontroller board. Option 2 in the menu allows to monitor the actual control process. Option 3 shows the same output as option 2 on the screen and additionally writes the recieved values into a comma seperated file. If selecting 3 the user is prompted to enter a filename. If the file could be opened for reading this is acknowledged on the next screen line as seen in figure 31 (c). Then the process data recieved from the temperature controller board is displayed in seven columns. At each system hartbeat a new packed is recieved and displayed in the next line. The first column contains the time in seconds since the control process was started. The offset to the higher actual system time was already removed in the Arduino. The next column shows the actual plate temperature in °C with 5 digit resolution. This is the highest resolution containing information, as the digitizing steps in the digital temperature sensor is 0.034 K. To archive this higher resolution the TSIC 506 libary by Wagner (2014) was modified. The next

columns show the selected setpoint and the difference between the actual plate temperature and the setpoint used as input for the PID controller. The next column either shows "heating" or "cooling" depending on the value of the mode input of the peltier drivers. The next value shows the actual pulse width of the PWM input signal of the peltier drivers. Caused by the fact, that the direction selection and the PWM signal are applied to the inputs of the motor driver, the same pulse width has different effects on the resulting current. In the cooling mode a PWM value of 0 leads to no current, a value of 255 to the maximum current. In heating mode the behaviour is reversed. This can also be seen in the first two process data lines in figure 31 (c). The PWM input of 106 in the first line leads to a current of 5.13 A through the peltier elements. The following value of 242 reduces this current to 0.83 A. The current is displayed in the last column of the output. The currents of the three shunt resistors in the temperature controller are determined after the RC pass had time to settle. To minimize the influence of the residual ripple after the filter the voltage on the capacitor is read with oversampling. The arithmetic averages of the measurements of the three peltier drivers are added and sent to the PC as current value (Prinz and Kirch-Prinz, 2002; Wheat, 2011).

3.2.5 Determination of the controller parameters

The determination of the controller parameters is shown exemplarily for on one of the jump responses recorded for the cooling process in figure 32. The measurement starts at the room temperature Y_0 . The controller was used with the reading of the room temperature sensor as setpoint first followed by 10 minutes without peltier operation. After one hour the plate reached the final temperature Y_B . The inflexion tangent was determined by the inflexion point of the fitted polynom as described above. The temperature changes from the stable start value not directly after the input jump was applied. The moment it starts to change was used as start point for the fitting of the polynom.

Table 3 shows the determined controller parameters for the three measurements for the heating and cooling mode, their arithmetic averages, their standard deviations and their procentual error. The procentual error for the time T_u has a quite high value of over 16 %. This error spreads to the values for K_p and K_i because they depend on it.

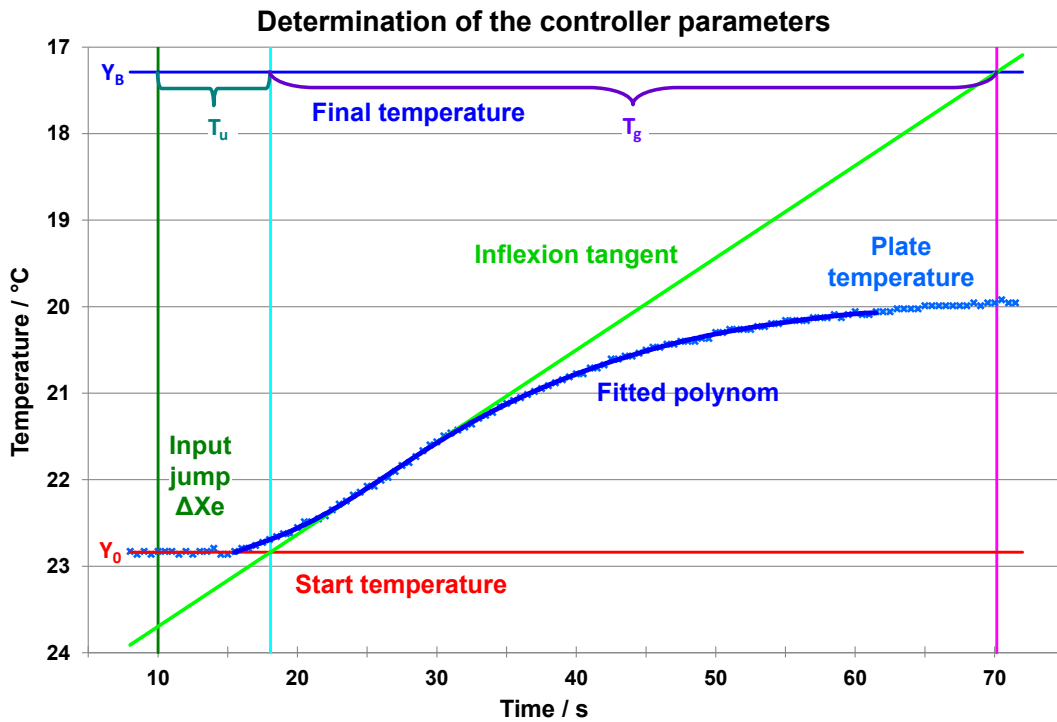


Figure 32: Determination of the controller parameters using the inflexion tangent of an input jump in the openend control loop

Mode	M. No.	$K_s / \frac{kK}{PWM}$	T_u / s	T_g / s	$K_p / \frac{PWM}{kK}$	$K_i / \frac{PWM}{kK \cdot s}$	$K_d / \frac{PWM \cdot s}{kK}$
cooling	1	88.63	7.85	140.9	121.5	862.8	476.8
	2	82.68	8.12	131.5	117.5	894.2	477.0
	3	90.13	8.07	142.0	117.2	824.8	472.8
	average	87.15	8.01	138.1	118.7	860.6	475.5
	error	3.9	0.14	5.8	2.4	34.7	2.4
	error %	4.5	1.8	4.2	2.0	4.0	0.5
heating	1	321.1	9.82	288.4	54.8	190.2	269.4
	2	318.3	11.27	266.3	44.6	167.3	251.0
	3	317.1	8.14	268.3	62.4	232.4	253.9
	average	318.9	9.74	274.3	53.9	196.6	258.1
	error	2.1	1.56	12.2	8.9	33.0	9.9
	error %	0.6	16.1	4.4	16.6	16.8	3.8

Table 3: Determination of the controller parameters

3.2.6 Response of the controller to setpoint jumps

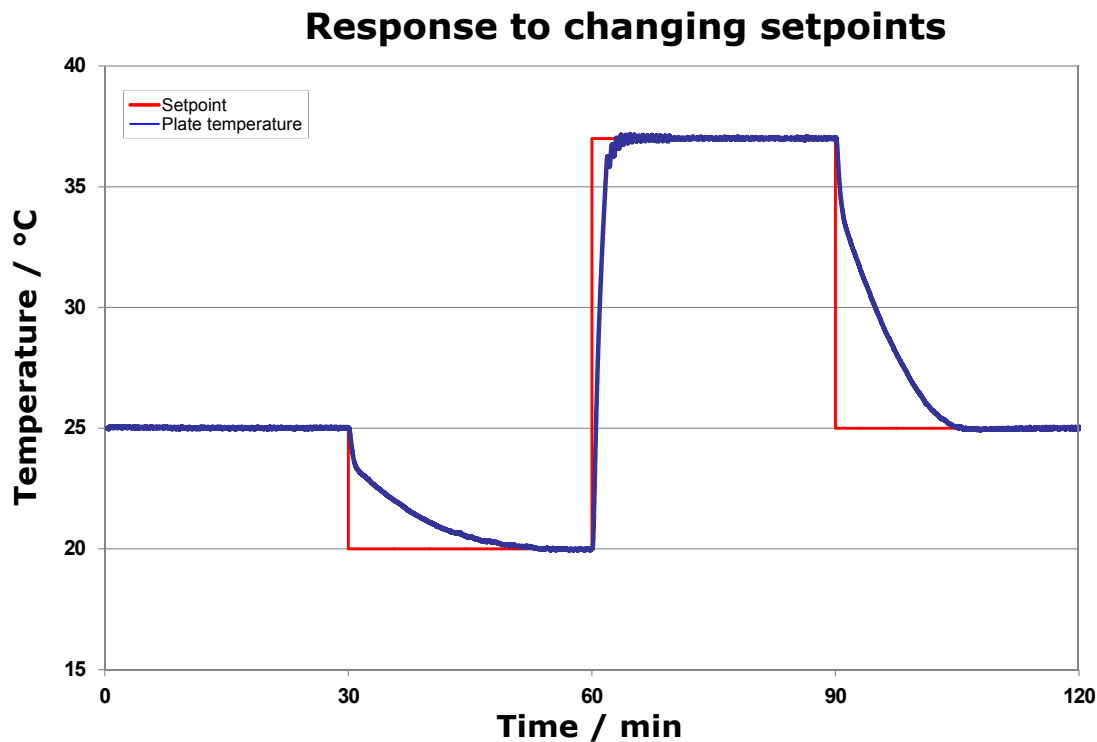


Figure 33: Respond of the control system to changes in the setpoint

Figure 33 shows the response of the control system to changes in the setpoint. Starting at a temperature of 25 °C the setpoint is changed to 20 °C. The plate needs nearly 30 minutes to cool down to this temperature, as the cooling process is the less efficient ones using peltier elements. After the setpoint is raised to 37 °C the plate temperature follows it in about 10 minutes. Here the respond shows oscillations which decay over the time. To cool down to 25 °C again needs about 15 minutes. Except of the oscillations before reaching 37 °C the system responds as wanted showing no over- or undershoots.

3.2.7 Long term stability

The long term stability of the control process was determined by cooling the plate down to 20 °C and then rising it to 40 °C in steps of 2.5 K. Each temperature was hold for 90 minutes. The controller response shows initial overshoots when heating up. Their amount is the highest at low temperatures, declining with rising temperature. Because the plate needs a maximum time of 30 minutes to stabilize, the first 30 minutes of every temperature setting were discarded.

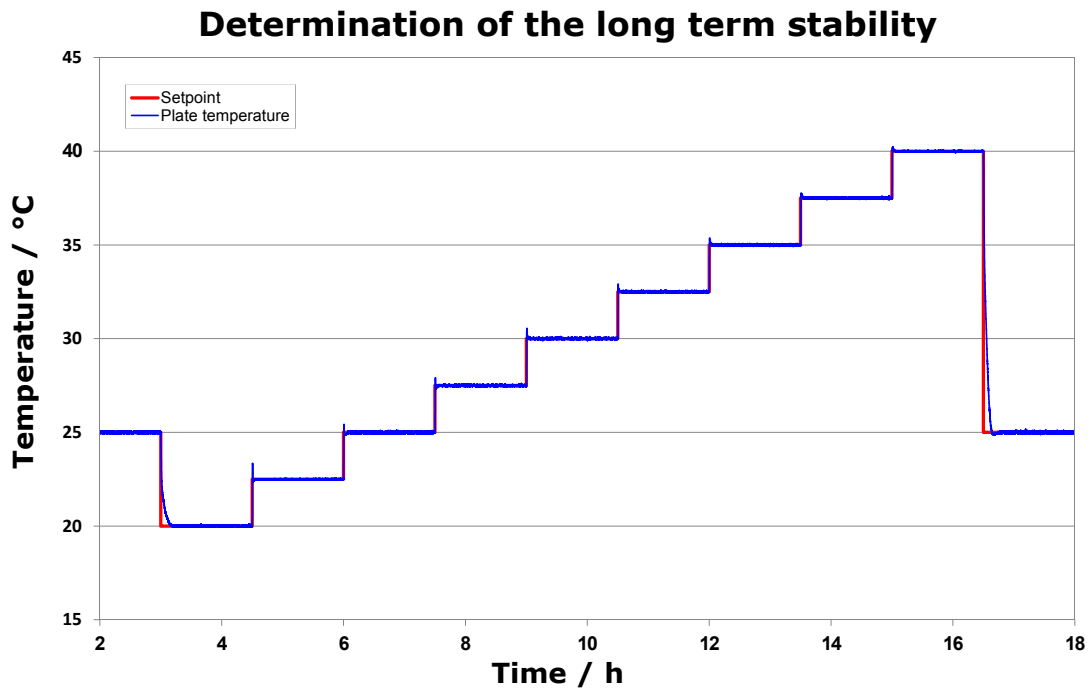


Figure 34: Determination of the long term stability

Setpoint	Mean temp.	Std. dev.	Max. neg. dev.	Max. pos. dev.	Min. value	Max. value
20.00	20.001	0.021	0.050	0.090	19.95	20.09
22.50	22.500	0.021	0.050	0.050	22.45	22.55
25.00	25.000	0.027	0.050	0.080	24.95	25.08
27.50	27.500	0.032	0.090	0.110	27.41	27.61
30.00	30.000	0.029	0.100	0.110	29.90	30.11
32.50	32.500	0.026	0.070	0.100	32.43	32.60
35.00	35.000	0.025	0.070	0.070	34.93	35.07
37.50	37.500	0.026	0.070	0.130	37.43	37.63
40.00	40.000	0.018	0.080	0.060	39.92	40.06
Average		0.025	0.070	0.089		

Table 4: Long term stability of the temperature control

Table 3.2.7 shows the average temperatures, their standard deviations, their maximum negative and positive read deviations and the maximum and minimum measured values for each temperature. Except of the value of 20.001 °C all mean temperatures are equal to the setpoint. The maximum standard deviation of 0.032 K resulted at a setpoint of 27.5 °C. The maximum negative deviation was 0.100 K with a temperature of 29.90 °C at the setpoint 30.00 °C. The maximum positive deviation was 0.130 °C with a value of 37.63 °C at the setpoint 37.50 °C. The average values of the standard deviation and the maximum negative and positive deviation were 0.025 K, 0.070 K and 0.089 K respectively. As the accuracy of the digital temperature sensor is 0.06 K in the used range, this values show, that the temperature controller is able to control the temperature with a high enough accuracy over long time.

4 Discussion

4.1 Fluidic design

4.1.1 Integration of the temperature sensor

The tolerance of the hollow structures at the bottom of the array, taking up the temperature sensor and its blocking capacitor, was chosen to be low. To insert the sensor the structures were deepened using a rotary tool and a scalpel. Figure 35 shows the result of the UV bonding. The bright structures below the temperature sensor consist of air. The cables, connecting the temperature sensor with the temperature controller, did not flush plane with the outer structure. So the glass plates did not get in contact with the glue and the bonding remained incomplete. The structures connect the lower cooling channels with the channel containing the temperature sensor. This would lead to sample reaching the unisolated parts of the sensor and disturbing its function. The glass plates were scraped off the printed structure with acetone and a knife. This caused partly destructions of the cooling channel structures, leading to the formation of air bubbles when the channels are probed. The hollow structures, taking up the temperature sensor, should be deeper in the next version to avoid such bonding errors.

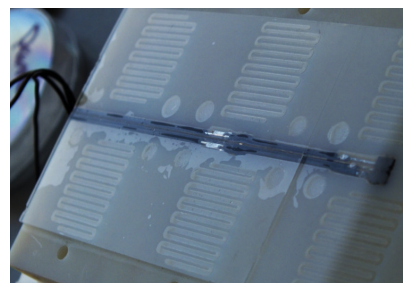


Figure 35: Bonding failure caused by too low tolerances for the included parts

4.1.2 Diffusion inside the cooling channels

The diffusion occurring inside the cooling channels while probing three sensors in a row is shown in figure 36. The effect is only visible after the second chip chamber. If it has an influence on the obtained QCM-D signals has to be shown in further experiments. At the left side of figure 36 the destruction of one channel divider and the air bubbles resulting from smaller destructions of the structure can be seen.

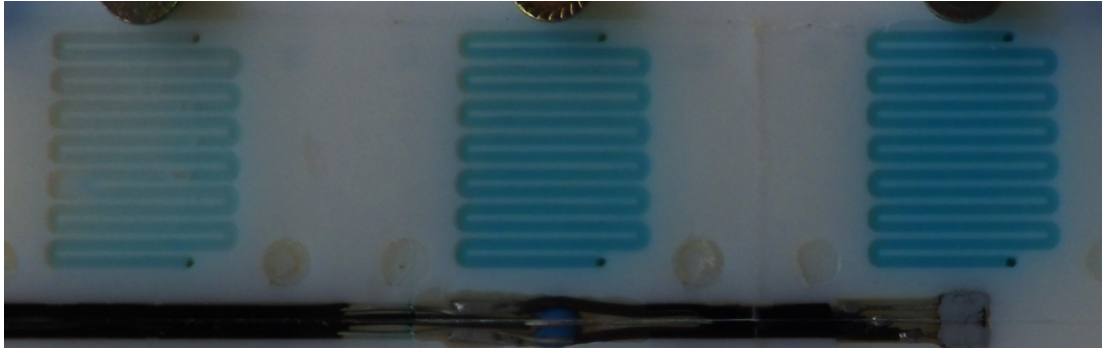


Figure 36: Diffusion inside the cooling channels

4.1.3 Inserting of the ventiles



Figure 37: Venting channels for the ventiles

The ventiles were developed and tested using the test bodys shown in figure 6. Their bottom part is open, allowing the air, replaced by the ventile, to escape. In the assembled array the bottom part of the ventiles is closed by the glass sheets glued to the cooling channels. The closed structure did not allow the air to escape and the ventiles could not be inserted completely. Venting channels were cut into the hollow structures taking up the ventiles to allow the air to escape from the ventile. Through this channels air can escape and the ventiles can be inserted completely. The channels should be included in the next version of the array to avoid that problem. Instead of locating them inside the hollow parts of the bottom array a

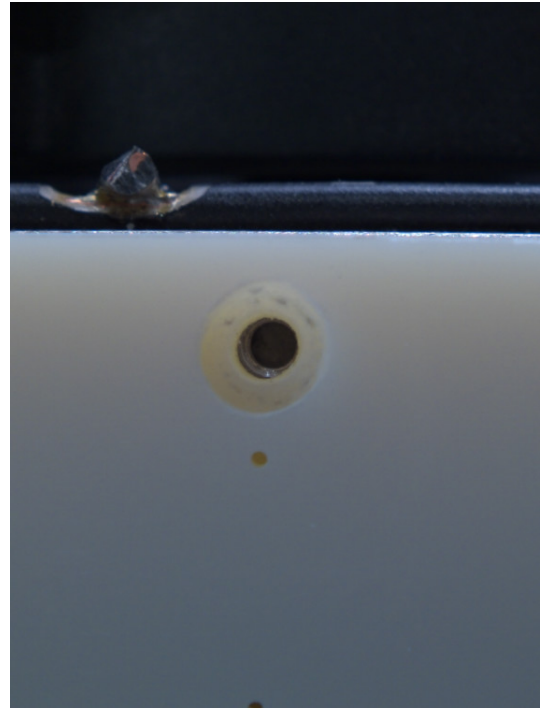
venting channel could be included in the ventile itself. This would not affect the outer surface of the ventile, which is important for the tightness.

4.1.4 Manufacturing of the cooling plate

The correction of tolerance effect while the manufacturing of the cooling plate is shown in figure 38. The positions of the threads, cutted into the plate, are not all accurate (a). To be able to fix the array with all its screws the printed parts were modified using a round file and a scalpel until all screws did fit in without mechanical stress. The plate is much more difficult to manufacture than the printed parts. In further versions of the array the exact position of the threads in the cooling plate should be determined and the position of the holes for the fixing screws in the 3D model of the array should be aligned to the positions in the plate.



(a) Tolerance error of the threads



(b) Corrected printed part

Figure 38: Tolerance effects of the steel plate and correction by modification of the printed parts

4.2 PCB design

The amount the Arduino microcontroller exceeds its pins at the opposite side of the USB connector was not correctly factored in. The connector for the 7 segment display located next to the microcontroller board does thereby overlap with the board. To be able to plug the connector in, parts of it were removed using a cutting disk mounted onto a rotary tool as shown in figure 39. The connector should be placed considering to the dimensions of the arduino board in further versions of the PCB design to avoid the need for this modification.

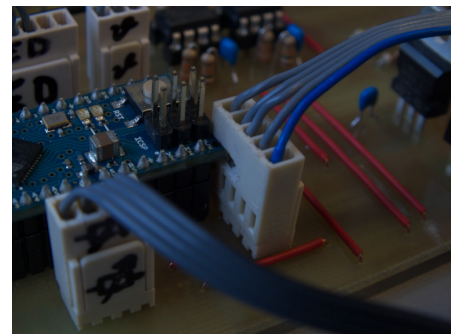


Figure 39: Modification of a PCB connector

5 Concluding Remarks

5.1 Conclusion

A functional fluidic array for up to ten QCM-D chips was designed. Ventiles allowing a flexible routing through the array were developed. The array was shown to be tight and the sample routing was visualized using colored samples. A temperature controller with an easy to use interface for the user and a USB connection to a PC was developed and build up. The controller parameters were determined. The controller was able to keep the temperature stable in a range below the accuracy of the used temperature sensor over long time.

5.2 Outlook

A second version of the fluidic array should be printed considering the design issues discussed above. The temperature controller should be put into a casing to protect it from outside influences and to facilitate its handling. If a second temperature controller would be build, the PCB design should be adopted to the actual size of the microcontroller board. The QCM-D chips should be connected to the existing commercial QCM-D electronics to determine whether the chips are able to oscillate in it. To actually measure the mass adsorbing onto the QCM-D chips in the array their electrical connections have to be included in an oscillator or vector analyzer circuit.

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List of supplementary data

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Supplement B: List of consumables

Supplement C: List of components

Supplement D: List of devices

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Supplement I: Source codes

Supplement A: List of chemicals

Substance	Manufacturer	Order number	Charge number
Water	Millipore Q-Gard 2, Membran Pure Q2	194-0009	304-1-6-4890R
Sodium hydroxide	Merk	1-06498-1000	B0127298 780
Ethanol	Merk	1.000983.1000	K45251683 405
Ethanol	Laboratory stock	Barrel in storage	n.a.
Acetone	Laboratory stock	Barrel in storage	n.a.
2-Propanole	Laboratory stock	Barrel in storage	n.a.

Supplement B: List of consumables

Component	Description	Manufacturer	Order number
Membrane filter	Aerodisk Premium 25 mm Syringe Filter with GxF 1,2 µm GHP Membrane	Pall Life Sciences	A4-4307T
Pump tubings	LFL Longlife ID 0,38 mm	TYGON	070703-04
Water filter	Quantum Ultrapute Organex Cartridge	Millipore	QTUM000EX

Supplement C: List of components

Component	Description	Manufacturer	Order number
7 segment display	SC08-11CGKWA	Kingbright	1050568-62
7 segment driver	MAX 7221	MAXIM	MAX 7221 CNG
Thermal compound	Arctic MX-4	ARCTIC	ARCTIC MX-4-20
Potentiometer	lin. 6mm 220k	Omeg	PO6M-LIN 220K
Bus driver	74HC541	SGS Thomson	74HC 541
Voltage regulator	7809	TS	TS 7809 CZ
Voltage regulator	7805	TS	TS 7805 CZ
Galvanisation set	Galvanisier-Zubehör-Set	Conrad	527983-62
Galvanisation set	Handgalvanisier-Set	Conrad	530506-62
Copper wire	1 x 0.20 mm ²	Conrad	606397-62
Copper wire	1 x 1.50 mm ²	Lapp Kabel	607051-62
Copper wire	1 x 1.50 mm ²	Conrad	549236-62
Microcontroller board	Arduino Micro 65192	Arduino	323485-62
USB cable	2m A B	Delock	1007855-62
Circuit board	Proma photoresist	Proma	528579-62
Matrix hole board	SU527769	Conrad	530753-62
Chipset cooler	28513C60	Akasa	999026-62
Diode	BYW98-200	STMicroelectronics	155472-62
Peltier element	CP12705	TEC	193569-62
Heat sink	CTX/409/50	CTX Thermal Solutions	188041-62
Shunt resistor	0.18 75 W	Vitrohm	427970-62
Temperature sensor	TSIC506-T092	B+B Sensors	506360-62
Thermal compound pad	0.3 mm 1.4 W/mK (L x B)	Keratherm	181133-62

Component	Description	Manufacturer	Order number
Electrolyte capacitors	Different values	velleman	K/CAP2
Ceramic capacitors	Different values	velleman	K/CAP1
Resistors	E12 different values	velleman	K/RES-E12
Motor driver	L6203	STMicroelectronics	189-1217
Sealing rings	QS-QCS 002	Lot	QS-QCS 002
Black nuts	QS-QCS 013	Lot	QS-QCS 013
Screws	464-M3-16-NI	Ganter Griff	464-M3-16-NI
Fittings	Gripper Fitting, Ferrules Flat Bottom	Diba	002310
Teflon tubing	PTFE tubing	Bohlender	271730005

Supplement D: List of devices

Device	Description	Manufacturer
Analytic balance	Analytic AC220S	Satorius
Digital camera	Finepix HS20EXR	Fujifilm
Piston pipettors	Reference 5000 / 1000 / 100 / 10	Eppendorf
Magnetic stirrer	MR 3001	Heidolph
Magnetic stirring plate	Multipoint HP 15	Variomag
Voltage supply	PPS 13610	Voltcraft
Peristaltic pump	Reglo-Analog ISM796B	Ismatec
QCM-D flowcell	QFM 401	Lot Oriel Group Europe
Caliper rule	10059953	Mitutoyo
Water filter	Millipore Q-Gard 2	Millipore
Soldering station	i-CON 1	Ersa
Multimeter	87 V True RMS Multimeter	Fluke

Supplement E: List of abbreviations

Abkürzung	Bedeutung
ADC	Analog to Digital Converter
DMOS	Double-Diffused Metal Oxide Semiconductor
dpi	dots per inch
EEPROM	Electrically Erasable Programmable Read-Only Memory
GND	GrouND
ICSP	In Circuit Serial Programming
IO	Input Output
LED	Light Emmiting Diode
MALDI-ToF MS	Matrix Assisted Laser Desorption Ionization Time of Flight Mass Spectrometry
NTC	Negative temperature coefficient
PC	Personal Computer
PCB	Printed Circuit Board
PID	Proportional, Integral, Differential
PWM	Pulse Width Modulation
QCM	quarz crystal microbalance
QCM-D	quarz crystal microbalance with dissipation monitoring
RC	Resistor Capacitor
SRAM	Short-Range Attack Missile Static Random-Access Memory
TFA	TriFluoroacetic Acid
TTL	Transistor Transistor Logic
USB	Universal Serial Bus
UV	UltraViolet

Supplement F: List of figures

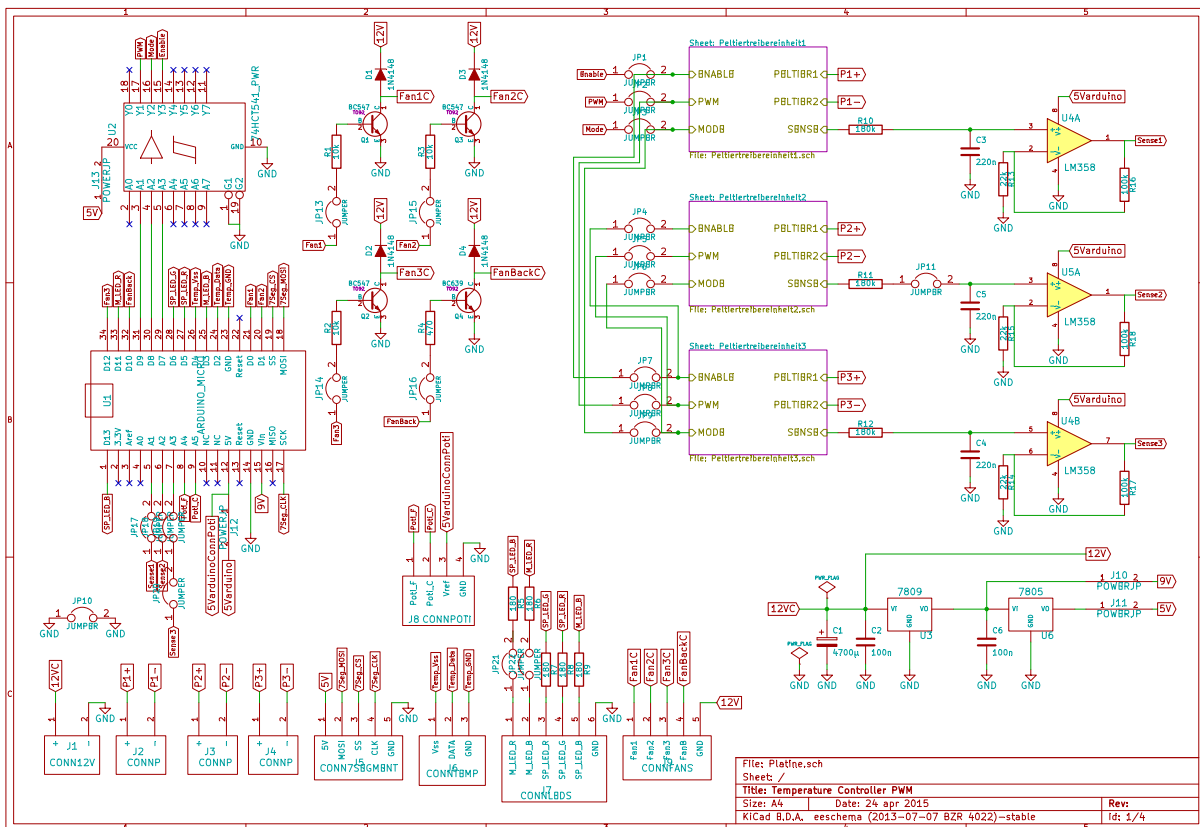
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Supplement H: Complete circuit diagram of the temperature controller



Supplement I: Source codes

Source code of the PID controller on the Arduino board

```
2 // ~~~~~  
4 //                               include libarys  
6 // ~~~~~  
8 #include <math.h>  
10 #include <tsic.h>  
12 #include <SoftwareSerial.h>  
14 #include <LedControl.h>  
16  
18 extern "C" {  
20     #include <inttypes.h>  
22 }  
24  
26 // ~~~~~  
28 //                               define constants  
30 // ~~~~~  
32 const int potiPinFine = A5;  
34 const int potiPinCoarse = A4;  
36  
38 const int roomTemperaturePin = A0;  
40  
42 const int Sense1Pin = A1;  
44 const int Sense2Pin = A2;  
46 const int Sense3Pin = A3;  
48  
50 const int PWM1Pin = 9; // PWM for Peltier  
52  
54 const int directionPin = 8;  
56 const int enablePin = 7;  
58  
60 const int Fan1Pin = 0;
```

```

36 const int Fan2Pin = 1;
const int Fan3Pin = 12;
const int FanBackPin = 10;
38
const int ledPinR = 5;
40 const int ledPinG = 6;
const int ledPinB = 13;
42 const int led2PinR = 11;
const int led2PinB = 3;
44
const int TempVssPin = 4;
46 const int TempSignalPin = 2;

48 // ~~~~~
//                               define variables
50 // ~~~~~

52 boolean dir = false;
boolean mainMode = false;

54 int error; // 1 = OK, 0 = parity error    return value of getTSicTemp()
56 int temperatur; // "return" of temperature in degrees Celsius * 100

58 uint32_t time, timesense, timeOffset, timeLast, timeSend, settingSinceTime,
    roomTemperatureSinceTime;
uint32_t Ta = 500; // System heartbeat
60 uint16_t temperature;
uint16_t temperaturewanted = 30000;
62 uint16_t temperaturewantedLast;
uint16_t temperaturewantedSend = 30000;
64 int16_t temperaturedifference;
uint16_t sensorValue = 0;
66 uint16_t sensorValueSend = 0;
uint16_t outputValue = 0;
68 uint16_t outputValueSend = 0;

70 int potiValueFine;
int potiValueCoarse;

72 unsigned long potiValueFine_Sum;
74 unsigned long potiValueCoarse_Sum;

76 const int potiValueSum_Count = 50;
int potiValueSum_Counter;

```



```

78 int roomTemperature; // PTC temperatur intern
80 double roomTemperature_Sum;
82 const byte roomTemperatureSum_Count = 200;
84 int roomTemperatureSum_Counter;
   boolean roomTemperatureInit = true;

86 const double roomTemperatureCALslope = -0.099964;
88 const double roomTemperatureCALoffset = 83.480492;
   double roomTemperatureCelsius;
   unsigned int roomTemperatureCelsiusInt;
90 unsigned int roomTemperatureSetpointDiff;
   unsigned int roomTemperaturePlateDiff;
92 unsigned int roomTemperatureSetpointDiffAbs;
   unsigned int roomTemperaturePlateDiffAbs;
94 unsigned int roomTemperatureUsedDiff;

96 const byte tempDisplayNormalIntensity = 9;

98 int fine;
   int coarse;
100 unsigned int temperaturePoti;
102 int temperatureDiffAbs;
   int tempSend;
104
   int currentPeltier1;
106 int currentPeltier2;
   int currentPeltier3;
108 long currentPeltier1Sum;
   long currentPeltier2Sum;
110 long currentPeltier3Sum;
   int currentPeltierSumTotal;
112 int currentPeltierSum_Count = 100;
   byte currentPeltierSum_Counter;
114
   const double currentPeltier1CAL = 185.8280;
116 const double currentPeltier2CAL = 168.7825;
   const double currentPeltier3CAL = 174.3662;
118
   double currentPeltier1Ampere;
120 double currentPeltier2Ampere;
   double currentPeltier3Ampere;

```

```

122 double currentPeltierTotalAmpere;
123 int currentPeltierTotalAmpereSend;
124
125 volatile byte Last_Digit;
126 volatile byte Digit_0_value;
127 volatile byte Digit_1_value;
128 volatile byte Digit_2_value;
129
130 volatile boolean Digit_0_DP = false;
131 volatile boolean Digit_1_DP = true;
132 volatile boolean Digit_2_DP = false;
133 volatile boolean Digit_Round;
134
135 byte ledValue = 0;
136 int counter = 0;
137 byte dirbyte = 0;
138
139 byte Outputarray[15];
140 byte tempBuffer[3];
141
142 double e, esum, ealt, y;
143 double AS = 0;
144
145 double Kp;
146 double Ki;
147 double Kd;
148
149 double KpC = 1.187451E-01;
150 double KiC = 8.605826E-07;
151 double KdC = 4.755258E+02;
152
153 double KpH = 5.391898E-02;
154 double KiH = 1.966363E-07;
155 double KdH = 2.580668E+02;
156
157 unsigned long delaytime=220;
158
159 byte textCounter;
160
161 byte initMessage[] =
162     {0,0,254,78,118,61,1,119,5,5,119,59,0,15,79,118,103,79,5,119,15,28,5,79,
        // Q C M D - A r r a y T e m p e r a t u r e
        0,78,29,21,15,5,29,14,0,219,0,55,29,23,118,119,21,21,0,109,126,48,91,0,0,0};

```

```

164 // C o n t r o l   S .   H o h m a n n   2   0   1   5
166 // ~~~~~
168 // ~~~~~
170 tsic Sensor1(TempVssPin, TempSignalPin);
172 LedControl tempDisplay = LedControl(MOSI,SCK,SS,1);
174 // ~~~~~
176 // ~~~~~
178 void initLEDstep(byte step);
180 void initFANstep(byte step);
182 void scrollInitMessage();
184 void parseTemp(unsigned int temp);
186 void getRoomTemperature();
188 // ~~~~~
190 // ~~~~~
192 // ~~~~~
194 // ~~~~~
196 // ~~~~~
198 // ~~~~~
200 // ~~~~~
202 // ~~~~~
204 // ~~~~~
206 // ~~~~~

void setup() {
    TCCR1B = TCCR1B & 0b11111000 | 0b00000001; // set timer 1 divisor to 1
                                                // for PWM frequency of 31372.55 Hz

    Serial.begin(9600);

    pinMode(directionPin, OUTPUT);
    pinMode(enablePin, OUTPUT);
    pinMode(Fan1Pin, OUTPUT);
    pinMode(Fan2Pin, OUTPUT);
    pinMode(Fan3Pin, OUTPUT);
    pinMode(FanBackPin, OUTPUT);

    tempDisplay.setScanLimit(0,2);
    tempDisplay.shutdown(0,false);
    tempDisplay.setIntensity(0,tempDisplayNormalIntensity);
    tempDisplay.clearDisplay(0);

    scrollInitMessage();
}

```

```

208     delay(300);
210
212     digitalWrite(Fan1Pin, HIGH);           // all on
214     digitalWrite(Fan2Pin, HIGH);
216     digitalWrite(Fan3Pin, HIGH);
218     digitalWrite(FanBackPin, HIGH);
220     analogWrite(ledPinR, 255);
222     analogWrite(ledPinG, 255);
224     analogWrite(ledPinB, 255);
226     analogWrite(led2PinR, 255);
228     analogWrite(led2PinB, 255);
230     tempDisplay.setIntensity(0,15);
232     tempDisplay.setRow(0,2,255);
234     tempDisplay.setRow(0,1,255);
236     tempDisplay.setRow(0,0,255);
238     delay(1000);
240
242     digitalWrite(Fan1Pin, LOW);           // all off
244     digitalWrite(Fan2Pin, LOW);
246     digitalWrite(Fan3Pin, LOW);
248     digitalWrite(FanBackPin, LOW);
250     analogWrite(ledPinR, 0);
252     analogWrite(ledPinG, 0);
254     analogWrite(ledPinB, 0);
256     analogWrite(led2PinR, 0);
258     analogWrite(led2PinB, 0);
260     tempDisplay.setIntensity(0,tempDisplayNormalIntensity);
262     tempDisplay.clearDisplay(0);
264
266     digitalWrite(Fan1Pin, HIGH);
268     digitalWrite(Fan2Pin, HIGH);
270     digitalWrite(Fan3Pin, HIGH);
272     digitalWrite(FanBackPin, HIGH);
274
276     digitalWrite(enablePin, HIGH);
278
280     getRoomTemperature();           // discard first measurement
282     delay(500);
284     getRoomTemperature();
286
288     timeOffset = millis();
290     time = timeOffset;
292     roomTemperatureSinceTime = time;

```

```

252     timeLast = timeOffset - Ta;
253     timeSend = 0;
254 }
255
256 // ~~~~~
257 //                               loop fuction
258 // ~~~~~
259
260 void loop() {
261
262     while (time < timeLast + Ta) {
263
264         time = millis();
265
266     }
267
268     timeLast = time;
269     timeSend = time - timeOffset;
270
271     error = Sensor1.getTSicTemp(&temperatur);
272     temperature = temperatur;
273
274     potiValueFine_Sum = 0;
275     potiValueCoarse_Sum = 0;
276
277     for (potiValueSum_Counter = 0; potiValueSum_Counter < potiValueSum_Count;
278         potiValueSum_Counter++) {
279
280         potiValueFine_Sum += analogRead(potiPinFine);
281         potiValueCoarse_Sum += analogRead(potiPinCoarse);
282
283     }
284
285     potiValueFine = (int)(potiValueFine_Sum / potiValueSum_Count);
286     potiValueCoarse = (int)(potiValueCoarse_Sum / potiValueSum_Count);
287
288     fine = map(potiValueFine, 0, 1000, 0, 9);
289     coarse = map(potiValueCoarse, 0, 1000, 20, 40);
290
291     temperaturePoti = 10 * fine + 100 * coarse;
292
293     temperaturePoti *= 10;
294

```

```

296  if (temperaturePoti > 40000) {
      temperaturePoti = 40000;
    }
298  if (temperaturePoti < 20000) {
      temperaturePoti = 20000;
300  }

302  if (temperaturePoti != temperaturewanted) {

304  settingSinceTime = millis();
      temperaturewanted = temperaturePoti;
306  roomTemperatureInit = true;

308  tempDisplay.setIntensity(0,15);    // full intensity

310  parseTemp(temperaturewanted);

312  tempDisplay.setDigit(0,0, Digit_0_value, Digit_0_DP);
      tempDisplay.setDigit(0,1, Digit_1_value, Digit_1_DP);
314  tempDisplay.setDigit(0,2, Digit_2_value, Digit_2_DP);

316  }

318

320  temperaturedifference = temperaturewanted - temperature;

if (temperaturedifference < 0) { temperatureDiffAbs = 0 - temperaturedifference;
    } else { temperatureDiffAbs = temperaturedifference; }

322

if (roomTemperatureInit == true) {

324

      roomTemperatureInit = false;

326

      roomTemperatureSetpointDiff = temperaturewanted - roomTemperatureCelsiusInt;
328  roomTemperaturePlateDiff = temperaturewanted - temperature;

330  if (roomTemperatureSetpointDiff < 0) { roomTemperatureSetpointDiffAbs = 0 -
      roomTemperatureSetpointDiff; } else { roomTemperatureSetpointDiffAbs =
      roomTemperatureSetpointDiff; }
      if (roomTemperaturePlateDiff < 0) { roomTemperaturePlateDiffAbs = 0 -
      roomTemperaturePlateDiff; } else { roomTemperaturePlateDiffAbs =
      roomTemperaturePlateDiff; }
332

```

```

    if (roomTemperatureSetpointDiffAbs > roomTemperaturePlateDiffAbs) {
roomTemperatureUsedDiff = roomTemperatureSetpointDiff; } else {
roomTemperatureUsedDiff = roomTemperaturePlateDiff; }
334
    if (roomTemperatureUsedDiff < 0) {
336
        mainMode = false;    // cooling
338
        Kp = KpC;
340        Ki = KiC;
        Kd = KdC;
342
    } else {
344
        mainMode = true;    // heating
346
        Kp = KpH;
348        Ki = KiH;
        Kd = KdH;
350
    }
352 }
354 e = temperatureDifference;
356 if ((e >= AS) || (e <= (AS*(-1))))    //
{
358     if ((y < 255)&&(y > -255))        // stop integration on overflow
        {                               // anti windup
360             esum = esum + e;        // integrate
        }
362
    y = (Kp*e)+(Ki*Ta*esum)+(Kd*((e-ealt)/Ta));    // PID
364
    ealt = e;                            // store last error for next heartbeat
366 }
368 if (y > 255)                            // limit output value into range from
    -255 to 255 (9 bit PWM)
    {
370     y = 255;
    }
372 if (y < -255)
    {

```

```

374     y = -255;
375     }
376     sensorValue = y + 255;
377
378     if (sensorValue < 256) {
379
380         dir = false;                // cooling
381         outputValue = 255 - sensorValue;
382         ledValue = outputValue;
383     } else {
384
385         dir = true;                // heating
386         outputValue = 255 - (sensorValue - 256);
387         ledValue = sensorValue - 256;
388
389     }
390
391     analogWrite(PWM1Pin, outputValue);    // adjust PWM for motor controllers
392
393     if(dir == true) {
394
395         digitalWrite(directionPin , HIGH);
396
397         analogWrite(led2PinR , ledValue);
398         analogWrite(led2PinB , 0);
399
400         dirbyte = 1;
401     } else {
402
403         digitalWrite(directionPin , LOW);
404
405         analogWrite(led2PinB , ledValue);
406         analogWrite(led2PinR , 0);
407
408         dirbyte = 0;
409     }
410
411     if (time > roomTemperatureSinceTime + 60000) {

```



```

418     roomTemperatureSinceTime = millis();
419     getRoomTemperature();
420     roomTemperatureInit = true;
421
422 }
423
424 if (settingSinceTime + 700 > time) {
425
426     analogWrite(ledPinR, 255);           // pink
427     analogWrite(ledPinG, 0);
428     analogWrite(ledPinB, 180);
429
430     tempDisplay.setIntensity(0,15);
431
432     parseTemp(temperaturewanted);
433
434     tempDisplay.setDigit(0,0,Digit_0_value,Digit_0_DP);
435     tempDisplay.setDigit(0,1,Digit_1_value,Digit_1_DP);
436     tempDisplay.setDigit(0,2,Digit_2_value,Digit_2_DP);
437
438 } else {
439
440     tempDisplay.setIntensity(0,tempDisplayNormalIntensity);
441
442     tempSend = temperature;
443
444     if (temperatureDiffAbs > 4999) {
445
446         analogWrite(ledPinR, 255);       // rot
447         analogWrite(ledPinG, 0);
448         analogWrite(ledPinB, 0);
449
450     } else if (temperatureDiffAbs > 2499) {
451
452         analogWrite(ledPinR, 224);       // orange
453         analogWrite(ledPinG, 30);
454         analogWrite(ledPinB, 0);
455
456     } else if (temperatureDiffAbs > 999) {
457
458         analogWrite(ledPinR, 174);       // dunkelgelb
459         analogWrite(ledPinG, 109);
460         analogWrite(ledPinB, 0);

```

```

462     } else if (temperatureDiffAbs > 60) {
analogWrite(ledPinR, 108);           // gruengelb
464 analogWrite(ledPinG, 146);
analogWrite(ledPinB, 0);
466     } else {
analogWrite(ledPinR, 0);           // gruen
468 analogWrite(ledPinG, 255);
analogWrite(ledPinB, 0);
470     tempSend = temperaturewanted;
    }
472
    parseTemp(tempSend);
474
    tempDisplay.setDigit(0,0, Digit_0_value, Digit_0_DP);
476    tempDisplay.setDigit(0,1, Digit_1_value, Digit_1_DP);
    tempDisplay.setDigit(0,2, Digit_2_value, Digit_2_DP);
478
}
480
delay(100); // wait for RC pass to settle
482
currentPeltier1Sum = 0;
484 currentPeltier2Sum = 0;
currentPeltier3Sum = 0;
486
for (currentPeltierSum_Counter = 0; currentPeltierSum_Counter <
    currentPeltierSum_Count; currentPeltierSum_Counter++) {
488
currentPeltier1Sum += analogRead(Sense1Pin);
490 currentPeltier2Sum += analogRead(Sense2Pin);
currentPeltier3Sum += analogRead(Sense3Pin);
492
}
494
currentPeltier1 = (int)(currentPeltier1Sum / currentPeltierSum_Count);
496 currentPeltier2 = (int)(currentPeltier2Sum / currentPeltierSum_Count);
currentPeltier3 = (int)(currentPeltier3Sum / currentPeltierSum_Count);
498
currentPeltier1Ampere = currentPeltier1 / currentPeltier1CAL;
500 currentPeltier2Ampere = currentPeltier2 / currentPeltier2CAL;
currentPeltier3Ampere = currentPeltier3 / currentPeltier3CAL;;
502
currentPeltierTotalAmpere = currentPeltier1Ampere + currentPeltier2Ampere +
    currentPeltier3Ampere;

```

```

504     currentPeltierTotalAmpereSend = (int)(currentPeltierTotalAmpere*1000);
506
508     Outputarray [0] = lowByte(timeSend);
510     timeSend = timeSend>>8;
512     Outputarray [1] = lowByte(timeSend);
514     timeSend = timeSend>>8;
516     Outputarray [2] = lowByte(timeSend);
518     timeSend = timeSend>>8;
520     Outputarray [3] = lowByte(timeSend);
522
524     Outputarray [4] = lowByte(temperature);
526     temperature = temperature>>8;
528     Outputarray [5] = lowByte(temperature);
530
532     temperaturewantedSend = temperaturewanted;
534
536     Outputarray [6] = lowByte(temperaturewantedSend);
538     temperaturewantedSend = temperaturewantedSend>>8;
540     Outputarray [7] = lowByte(temperaturewantedSend);
542
544     Outputarray [8] = lowByte(temperaturedifference);
546     temperaturedifference = temperaturedifference>>8;
548     Outputarray [9] = lowByte(temperaturedifference);
550
552     sensorValueSend = currentPeltierTotalAmpereSend;
554     Outputarray [10] = lowByte(sensorValueSend);
556     sensorValueSend = sensorValueSend>>8;
558     Outputarray [11] = lowByte(sensorValueSend);
560
562     Outputarray [12] = dirbyte;
564
566     outputValueSend = outputValue;
568     Outputarray [13] = lowByte(outputValueSend);
570     outputValueSend = outputValueSend>>8;
572     Outputarray [14] = lowByte(outputValueSend);
574
576     Serial.write(Outputarray, sizeof(Outputarray));
578 }
580
582 //~~~~~
584 //          fuction to determine the ambient temperature via the ntc
586 //~~~~~

```

```

548 void getRoomTemperature() {
550     roomTemperature_Sum = 0;
552     for (roomTemperatureSum_Counter = 0; roomTemperatureSum_Counter <
        roomTemperatureSum_Count; roomTemperatureSum_Counter++) {
554         roomTemperature_Sum += analogRead(roomTemperaturePin);
556         delay(1);
558     }
560     roomTemperature = (int)(roomTemperature_Sum / roomTemperatureSum_Count);
562     roomTemperatureCelsius = roomTemperature * roomTemperatureCALslope +
        roomTemperatureCALoffset;
564     roomTemperatureCelsiusInt = (int)(round(roomTemperatureCelsius*10)*100);
566 }
568 void initLEDstep(byte step){
570     switch (step) {
572         case 4:
574             analogWrite(ledPinR, 255);           // rot
576             analogWrite(ledPinG, 0);
578             analogWrite(ledPinB, 0);
580             analogWrite(led2PinR, 0);           // blau stark
582             analogWrite(led2PinB, 255);
584             break;
586         case 9:
588             analogWrite(ledPinR, 224);           // organge
590             analogWrite(ledPinG, 30);
592             analogWrite(ledPinB, 0);
594             analogWrite(led2PinR, 0);           // blau mittel
596             analogWrite(led2PinB, 120);
598             break;
600         case 14:
602             analogWrite(ledPinR, 174);           // dunkelgelb
604             analogWrite(ledPinG, 109);
606             analogWrite(ledPinB, 0);
608             analogWrite(led2PinR, 0);           // blau schwach
610             analogWrite(led2PinB, 23);

```

```

590     break;
591     case 19:
592         analogWrite(ledPinR , 108);           // gruengelb
593         analogWrite(ledPinG , 146);
594         analogWrite(ledPinB , 0);
595         analogWrite(led2PinR , 17);           // rot schwach
596         analogWrite(led2PinB , 0);
597         break;
598     case 24:
599         analogWrite(ledPinR , 0);             // gruen
600         analogWrite(ledPinG , 255);
601         analogWrite(ledPinB , 0);
602         analogWrite(led2PinR , 90);           // rot mittel
603         analogWrite(led2PinB , 0);
604         break;
605     case 29:
606         analogWrite(ledPinR , 255);           // pink
607         analogWrite(ledPinG , 0);
608         analogWrite(ledPinB , 180);
609         analogWrite(led2PinR , 255);           // rot stark
610         analogWrite(led2PinB , 0);
611         break;
612     case 34:
613         analogWrite(ledPinR , 0);             // aus
614         analogWrite(ledPinG , 0);
615         analogWrite(ledPinB , 0);
616         analogWrite(led2PinR , 0);
617         analogWrite(led2PinB , 0);
618         break;
619
620     default :
621
622     break;
623 }
624
625 }
626
627 // ~~~~~
628 // ~~~~~ fuction for fan control in init ~~~~~
629 // ~~~~~
630
631 void initFANstep(byte step){
632
633     switch (step) {

```

```

634
        case 30:
636     digitalWrite(FanBackPin, HIGH);
        break;
638     case 33:
        digitalWrite(FanBackPin, LOW);
640     break;
        case 35:
642     digitalWrite(Fan1Pin, HIGH);
        break;
644     case 37:
        digitalWrite(Fan1Pin, LOW);
646     digitalWrite(Fan2Pin, HIGH);
        break;
648     case 39:
        digitalWrite(Fan2Pin, LOW);
650     digitalWrite(Fan3Pin, HIGH);
        break;
652     case 41:
        digitalWrite(Fan3Pin, LOW);
654     digitalWrite(Fan2Pin, HIGH);
        break;
656     case 43:
        digitalWrite(Fan2Pin, LOW);
658     digitalWrite(Fan1Pin, HIGH);
        break;
660     case 45:
        digitalWrite(Fan1Pin, LOW);
662     digitalWrite(Fan2Pin, LOW);
        digitalWrite(Fan3Pin, LOW);
664     break;
666     default:
        break;
668 }
670 }
672 // ~~~~~
673 //          fuction to print welcome message on 7 segment display
674 // ~~~~~
676 void scrollInitMessage() {

```

```

678 tempDisplay.setIntensity(0,15);
680 for(textCounter = 0; textCounter < 48; textCounter++) {
682     tempDisplay.setRow(0,2,initMessage[textCounter]);
        tempDisplay.setRow(0,1,initMessage[textCounter+1]);
684     tempDisplay.setRow(0,0,initMessage[textCounter+2]);
        initFANstep(textCounter);
686     initLEDstep(textCounter);
        delay(delaytime);
688 }
690 tempDisplay.setIntensity(0,tempDisplayNormalIntensity);
}
692 //~~~~~
694 //          fuction to parse a number to display it on 7 segments
//~~~~~
696 void parseTemp(unsigned int temp) {
698     Digit_0_DP = false;
700     Digit_1_DP = true;
        Digit_2_DP = false;
702
        temp = temp / 10;
704
        Last_Digit = temp % 10;
706     if (Last_Digit < 5) Digit_Round = false; else Digit_Round = true;
        temp = temp / 10;
708
        Digit_0_value = temp % 10;
710
        if (Digit_Round) {
712             if (Digit_0_value == 9) {
                    Digit_0_value = 0;
714             } else {
                    Digit_0_value++;
716             Digit_Round = false;
                }
718         }
        temp = temp / 10;
720
        Digit_1_value = temp % 10;

```

```
722  if (Digit_Round) {
724      if (Digit_1_value == 9) {
726          Digit_1_value = 0;
728      } else {
730          Digit_1_value++;
732          Digit_Round = false;
734      }
736  }
738  temp = temp / 10;
740  Digit_2_value = temp % 10;
742  if (Digit_Round) {
744      Digit_2_value++;
746      Digit_Round = false;
748  }
750  Digit_Round = false;
752 }
```

tempcontrol56.ino

Source code for the programm to read the control parameters via the USB interface

```
2 // ~~~~~  
3 //  
4 // ~~~~~  
5  
6 #include <stdio.h>  
7 #include <stdlib.h>  
8 #include <windows.h>  
9 #include <string.h>  
10 #include <conio.h>  
11 #include <stdint.h>  
12 #include <stdarg.h>  
13 #include <string.h>  
14 #include <inttypes.h>  
15  
16 // ~~~~~  
17 //  
18 // ~~~~~  
19  
20 #define ESC 27  
21 #define MAXLINE 100;  
22  
23  
24 // ~~~~~  
25 //  
26 // ~~~~~  
27  
28 int verbose = 0;  
29 char* VIDstr;  
30 char* PIDstr;  
31 CHAR FileFullPath [] = {"COM7"} ;  
32 DWORD dwError ,mode;  
33 BOOL fSuccess ;  
34 DCB dcb;  
35 HANDLE hCom;  
36 FILE* comPortFileHandle ;  
37  
38 // ~~~~~  
39 //  
40 // ~~~~~  
41  
42 #include "mainmenu.h"  
43  
44 unsigned mainmenu (void);
```

```

42 void monitor (HANDLE hCom);
43 void warten(void);
44 void stremove(char* source, char ch);

46 // ~~~~~
47 //                                     main fuction
48 // ~~~~~

50 int main(int argc, char *argv[]) {

52
53 HANDLE keyboard = GetStdHandle(STD_INPUT_HANDLE);
54
55 CHAR ComPortFile[] = {"comports.list"};
56 CHAR puffer[100];
57
58 CHAR *comPortListe[9];
59
60 int i=1;
61 char *ptr;
62 unsigned c,d,e,f;
63 unsigned inputbuffer[10];

64
65 // set keyboard to raw reading.
66 if (!GetConsoleMode(keyboard, &mode))
67     printf("getting keyboard mode");
68 mode &= ~ ENABLE_PROCESSED_INPUT;
69 if (!SetConsoleMode(keyboard, mode))
70     printf("setting keyboard mode");
71
72
73
74 hCom = CreateFile( (LPCTSTR) FileFullPath,
75     GENERIC_READ | GENERIC_WRITE,
76     0, // comm devices must be opened w/exclusive-access
77     NULL, // no security attributes
78     OPEN_EXISTING, // comm devices must use OPEN_EXISTING
79     0, // not overlapped I/O
80     NULL // hTemplate must be NULL for comm devices
81 );

82
83 if (hCom == INVALID_HANDLE_VALUE)
84 {

```

```

86     dwError = GetLastError();
      printf("Invalid value: %d\r\n", dwError);
88     // handle error
    }
90
    fSuccess = GetCommState(hCom, &dcb);
92
    if (!fSuccess)
94    {
96        printf("Error 1\n");
      // Handle the error.
98    }
100 // Fill in the DCB: baud=9600, 8 data bits, no parity, 1 stop bit.
102 dcb.BaudRate = 9600;
    dcb.ByteSize = 8;
104 dcb.Parity = NOPARITY;
    dcb.StopBits = ONESTOPBIT;
106
    fSuccess = SetCommState(hCom, &dcb);
108
    if (!fSuccess)
110    {
112        printf("Error 2\n");
      // Handle the error.
    }
114
    while (c!=27) {
116
    c = mainmenu();
118
    switch (c)
120
    {
122
        case '1' :
124
            system("listComPorts.exe > comports.list");
126
            comPortFileHandle = fopen(ComPortFile, "r");
128
            i = 1;

```

```

130     system("Cls");
132     printf("\n\n");

134     while(fgets(puffer , 100, comPortFileHandle))
136     {

138         ptr = strtok(puffer , "-");
            while(ptr != NULL) {

140             printf("%d: ", i);
                comPortListe[i]=ptr;
142                 strremove(comPortListe[i], ' ');
                printf("%s", comPortListe[i]);
144                 ptr = strtok(NULL, "-");
                printf("%s\n", ptr);
146                 ptr = strtok(NULL, "-");
                ptr = strtok(NULL, "-");
148                 i++;
            }
        }

150     }

152     label:

154     do f = getch(); while (!(isdigit(f) || (f!='ESC')));

156

158     if (f-48<i) {
159     CloseHandle(hCom);
160         printf("%d\n", f-48);
161         printf("%s", comPortListe[f-48]);

162         hCom = CreateFile( (LPCTSTR)comPortListe[f-48] ,
163             GENERIC_READ | GENERIC_WRITE,
164             0, // comm devices must be opened w/exclusive-access
165             NULL, // no security attributes
166             OPEN_EXISTING, // comm devices must use OPEN_EXISTING
167             0, // not overlapped I/O
168             NULL // hTemplate must be NULL for comm devices
169         );

170

172         if (hCom == INVALID_HANDLE_VALUE)
            {
                dwError = GetLastError();

```

```

174         printf("Invalid value: %d\r\n", dwError);
175         // handle error
176     }
177
178
179     fSuccess = GetCommState(hCom, &dcb);
180
181     if (!fSuccess)
182     {
183
184         printf("Error 1\n");
185         // Handle the error.
186     }
187
188     // Fill in the DCB: baud=9600, 8 data bits, no parity, 1 stop bit.
189
190     dcb.BaudRate = 9600;
191     dcb.ByteSize = 8;
192     dcb.Parity = NOPARITY;
193     dcb.StopBits = ONESTOPBIT;
194
195     fSuccess = SetCommState(hCom, &dcb);
196
197     if (!fSuccess)
198     {
199         printf("Error 2\n");
200         // Handle the error.
201     }
202
203     } else goto label;
204
205     break;
206 case '2' :    monitor(hCom);
207
208     break;
209 case '3' :    monitorwrite(hCom);
210
211     break;
212 case '4' :    return(0);
213     break;
214 case 'ESC' :  return(0);
215     break;
216 default :    break;

```

```

218 }
219 }
220
221     CloseHandle ( keyboard );
222     CloseHandle ( hCom );
223
224     return 0;
225 }
226
227 void warten ( void ) {
228     while ( !( kbhit ( ) ) );
229 }
230
231 }
232
233 unsigned mainmenu ( void ) {
234     unsigned c;
235
236     system ( "Cls" );
237
238     printf ( " ===== \n" );
239     printf ( " # QCM-Array Temperature Control # \n" );
240     printf ( " #                               # \n" );
241     printf ( " #           programmed by           # \n" );
242     printf ( " #                               # \n" );
243     printf ( " # Siegfried Hohmann, B.Sc. 2015 # \n" );
244     printf ( " ===== \n" );
245     printf ( "\n\n" );
246     printf ( " 1. Select serial port\n" );
247     printf ( " 2. Monitor control process\n" );
248     printf ( " 3. Monitor and write data to file\n" );
249     printf ( " 4. Exit\n" );
250
251     do c = getch ( ); while ( ! ( ( isdigit ( c ) || ( c != 'ESC' ) ) ) );
252
253     return c;
254 }
255
256 // ~~~~~
257 //                               function to monitor
258 // ~~~~~

```

```

262 void monitor (HANDLE hCom) {
264     unsigned c;
266     unsigned d;
    byte inputbuffer [15];
268
        uint32_t time;
270        uint16_t temperature;
        uint16_t temperaturewanted;
272        int16_t temperaturedifference;
        uint16_t sensorValue;
274        byte dirbyte;
        uint16_t outputValue;
276
278    DWORD read, written;
280
        if (kbhit()) {
282            c = getch();
        }
284
    do {
        // check for data on port and display it on screen.
286        ReadFile(hCom, inputbuffer, sizeof(inputbuffer), &read, NULL);
        if ( read ) {
288
            time = inputbuffer [3];
290            time = time<<8;
            time += inputbuffer [2];
292            time = time<<8;
            time += inputbuffer [1];
294            time = time<<8;
            time += inputbuffer [0];
296
            temperature = inputbuffer [5];
298            temperature = temperature<<8;
            temperature += inputbuffer [4];
300
            temperaturewanted = inputbuffer [7];
302            temperaturewanted = temperaturewanted<<8;
            temperaturewanted += inputbuffer [6];
304
            temperaturedifference = inputbuffer [9];

```

```

306     temperaturedifference = temperaturedifference <<8;
307     temperaturedifference += inputbuffer [8];
308
309     sensorValue = inputbuffer [11];
310     sensorValue = sensorValue <<8;
311     sensorValue += inputbuffer [10];
312
313     dirbyte = inputbuffer [12];
314
315     outputValue = inputbuffer [14];
316     outputValue = outputValue <<8;
317     outputValue += inputbuffer [13];
318
319     printf("Time: %.1fs Temp: %.3f SP: %.2f Diff: ",time/1000.0,
temperature/1000.0,temperaturewanted/1000.0);
320     if (temperaturedifference >= 0) printf(" ");
321     printf("%.3f ",temperaturedifference/1000.0);
322     if (dirbyte == 0) { printf("cooling"); } else { printf("heating"); }
323     printf(" PWM:");
324     if (outputValue < 10) printf(" ");
325     if (outputValue < 100) printf(" ");
326     printf(" %d ",outputValue);
327     printf(" I: %.2fA\n",sensorValue/1000.0);
328 }
329
330 if (kbhit()) {
331     c = getch();
332 }
333
334 } while (c!=27);
335 }
336
337
338 //-----
339 //           function to monitor and write
340 //-----
341
342 void monitorwrite (HANDLE hCom) {
343
344     unsigned c;
345     unsigned d;
346     byte inputbuffer [15];
347     FILE *fp;

```



```

char filename[50] = {0};
350
system("Cls");
352
printf("Enter file name: ");
354
scanf("%s", filename); //
356
fp = fopen(filename, "w");
358
if (fp == NULL) {
printf("Error opening %s for writing.\n", filename);
360
}
362
printf("Writing to file %s\n", filename);
364

uint32_t time;
uint16_t temperature;
368
uint16_t temperaturewanted;
int16_t temperaturedifference;
370
uint16_t sensorValue;
byte dirbyte;
372
uint16_t outputValue;

374
DWORD read, written;

376
do {
378
ReadFile(hCom, inputbuffer, sizeof(inputbuffer), &read, NULL);
380
if ( read ) {

382
time = inputbuffer[3];
time = time<<8;
384
time += inputbuffer[2];
time = time<<8;
386
time += inputbuffer[1];
time = time<<8;
388
time += inputbuffer[0];

390
temperature = inputbuffer[5];
temperature = temperature<<8;
392
temperature += inputbuffer[4];

```

```

394     temperaturewanted = inputbuffer [7];
396     temperaturewanted = temperaturewanted<<8;
398     temperaturewanted += inputbuffer [6];

400     temperaturedifference = inputbuffer [9];
402     temperaturedifference = temperaturedifference<<8;
404     temperaturedifference += inputbuffer [8];

406     sensorValue = inputbuffer [11];
408     sensorValue = sensorValue<<8;
410     sensorValue += inputbuffer [10];

412     dirbyte = inputbuffer [12];

414     outputValue = inputbuffer [14];
416     outputValue = outputValue<<8;
418     outputValue += inputbuffer [13];

420     printf("Time: %.1fs Temp: %.3f SP: %.2f Diff: ",time/1000.0,
temperature/1000.0,temperaturewanted/1000.0);
422     if (temperaturedifference >= 0) printf(" ");
424     printf("%.3f ",temperaturedifference/1000.0);
426     if (dirbyte == 0) { printf("cooling"); } else { printf("heating"); }
428     printf(" PWM: ");
430     if (outputValue < 10) printf(" ");
432     if (outputValue < 100) printf(" ");
434     printf(" %d ",outputValue);
436     printf(" I: %.2fA\n",sensorValue/1000.0);

438     fprintf(fp, "%.1f,%.3f,%.2f,%.3f,",time/1000.0,temperature/1000.0,
temperaturewanted/1000.0,temperaturedifference/1000.0);
440     if (dirbyte == 0) { fprintf(fp, "cooling,"); } else { fprintf(fp, "
heating,"); }
442     fprintf(fp, "%d,%.2f\n",outputValue ,sensorValue/1000.0);

444 }

446     if (kbhit()) {
448         c = getch();
450     }

452 } while (c!=27);

```

```
434 fclose(fp);
436 }
438 //~~~~~
440 //          function to remove a string
442 void stremove(char* source, char ch) {
444     char* target=source;
446     for (;(*target!=0); target++)
        if (*target!=ch) target++;
}
```

main.c

Declaration of authorship

I declare in lieu of an oath that the Master Thesis submitted has been produced by me without illegal help from other persons. I state that all passages which have been taken out of publications of all means or un-published material either whole or in part, in words or ideas, have been marked as quotations in the relevant passage. I also confirm that the quotes included show the extent of the original quotes and are marked as such. I know that a false declaration will have legal consequences.

Date

Siegfried Hohmann