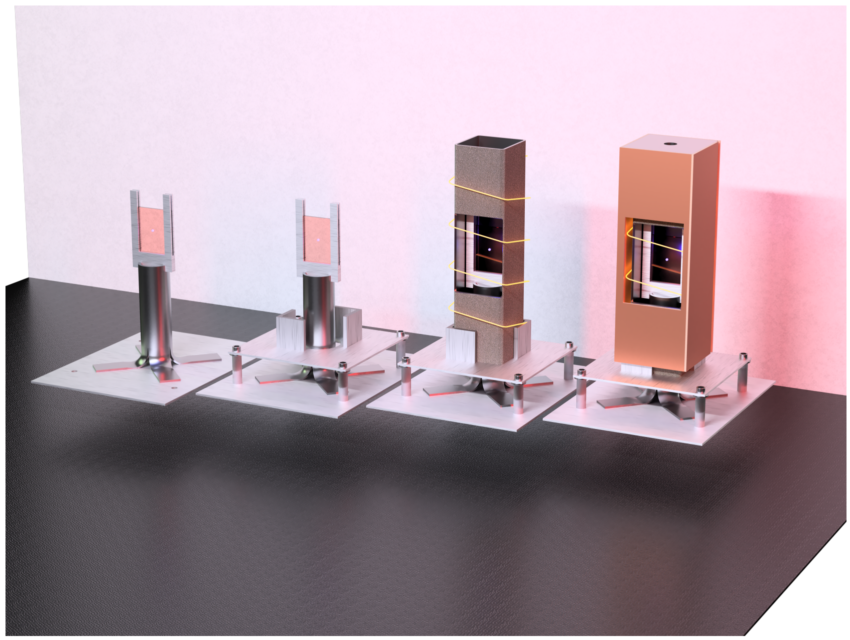
0.2 Vertical Sample Holder/Heater



0.2.1 Fabrication

A heating system was designed with the goal of evenly heating film samples held vertically, while maintaining an optical path to allow the film to be excited by laser light and emission to be detected. The design requirements called for the ability to heat up to 200 °C with ± 2 °C accuracy, with sufficiently low spatial variability in temperature, and an electronic interface for control and monitoring.

The final design consisted of three major components, a power source and heater element, a sample oven, and a feedback and control circuit. The power source used was a repurposed power supply unit taken from a computer capable of delivering 120 W of power at +12V. This low-cost and well tested unit met the power requirements of the heater design, and saved money and time over a bespoke design, while simultaneously providing +5V and +3.3V for any control circuitry. To control the amount of power drawn, the 12V output is fed into a step-down converter (XLSEMI XL4016) which could vary the output voltage from 0-12V based on a feedback measurement. To form the heating element, 22 gauge nichrome wire was cut to length to measure 1.2 Ω, a value chosen to draw the full 120W at the maximum 12V.

The sample oven was to interface with the pre-existing sample holder in a way that did not disturb the established optical path and was easily removable. To increase reliability and stand up to repeated installation and removal, the heating element was securely wound around a length of square metal tubing, which acted as the key structural element. The 1" square tubing had a 1.5" section of one face removed, to maintain the optical path to the sample, and was coated in a thick layer of sodium silicate bonded ceramic powder to electrically insulate it from the heater wire. The heating element wrapped tube acted as a heat spreader, since the direct contact would quickly heat the entire tube, whose large surface area would more evenly radiate heat towards the sample. Though this came at the cost of a significant time lag in heating due to the additional thermal mass. The ends of the tube were initially left unsealed to increase cooling rates. Upon testing, this design proved wasteful, as the open-top sample volume and externally exposed heating element led to a large amount of energy used to heat air that quickly left the heater vicinity. As well, the open air-flow nature of the sample volume made the sample temperature too susceptible to variation in air currents within the room. To remedy these shortcomings, an insulating layer was constructed to reduce airflow. FR4 grade glass-reinforced epoxy laminate was chosen for its strength, insulating, and flame retardant properties as the insulating material. A 2" square tube of the laminate constructed with an additional piece closing off the top. A 1.5" window cut was into one side, again to maintain the optical path. This additional insulation decreased both the required power to reach a certain temperature as well as the susceptibility to changing air currents.

The feedback and control circuitry consists of a microcontroller (ATtiny85) to run the feedback loop and communicate with other electronic devices, the step-down converter integrated into the power circuit, and a digital thermocouple amplifier. The step-down converter is based on an XLSEMI XL4016 buck converter and its' datasheet's example circuit (fig. 3), with the feedback pin not connected to the output voltage, but rather directly to the microcontroller which drives the pin according to the measured temperature. Temperature information is collected from a thermocouple embedded in the sample holder by means of a

MAX31850 1-Wire thermocouple-to-digital converter. The converter is set up according to the example circuit given in Fig 4, based on the example circuit given in the datasheet. The temperature data was communicated to the microcontroller over the 1-Wire bus to reduce pin usage, but required significant correction before use (see code comments). The microcontroller acts as PID controller, using I2C communication to interact with an external device to report temperatures and accept changing set points. The thermocouple reading is the measured process variable, while the voltage of the pin connected to the buck converter's feedback pin is the control variable.

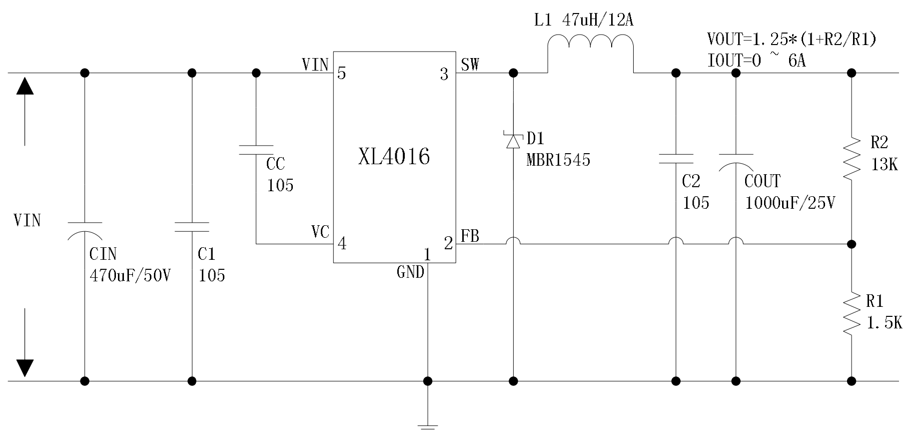


Figure 3: Example circuit diagram of the XL4016 buck converter which drives the heater circuit.

0.2.2 Evaluation of Performance

The PID loop was tuned to minimize overshoot foremost, and displayed a maximum heating rate of 22 K/min and a cooling time constant of 530 seconds. The maximum undershoot observed was 5 K and the maximum overshoot was 2 K. The average steady state temperature at the thermocouple was 0.32 K below setpoint, with standard deviation of 0.45 K, as shown in figure 5.

However, this measurement at a single point does not represent the overall accuracy of the apparatus, and the variation at the sample. To estimate the spatial distribution, a second thermometer measured the temperature at several points at the sample front and back

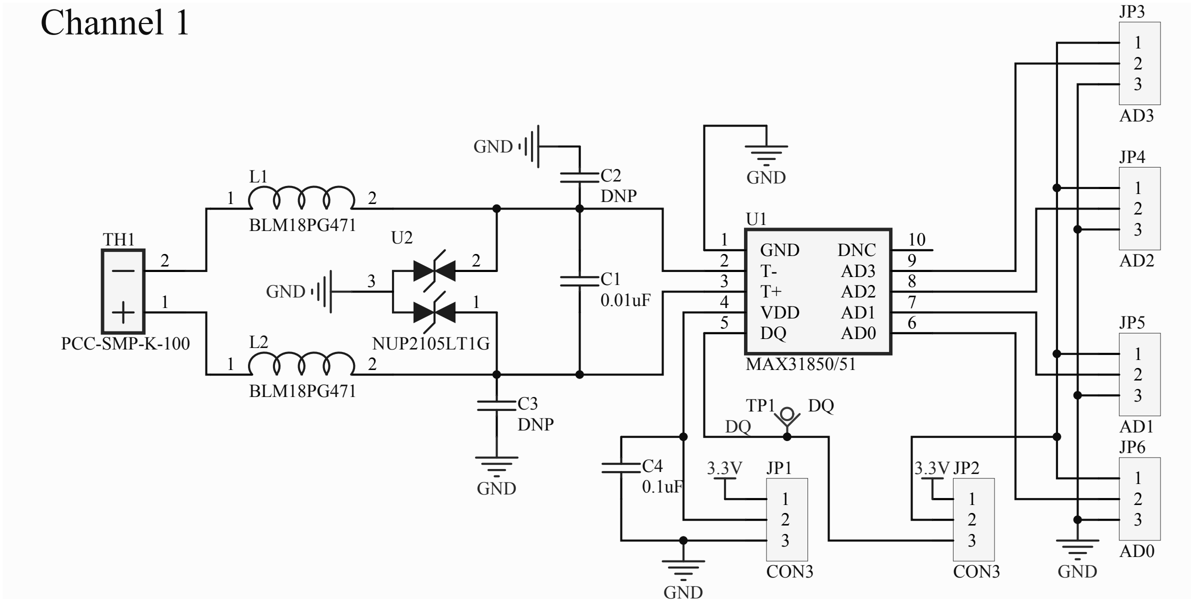


Figure 4: Example circuit diagram of the MAX31850 temperature sensor.

surface, as well as the air temperature 1 cm away from each surface. Temperature measurements at the sample surface were measured within ± 1.5 K of the setpoint, while the air temperature in front of the sample (nearer the opening for the optical path) was on average 1 K cooler than the setpoint, while the air behind the sample was ± 2.5 K, showing a larger susceptibility to the current heater state.

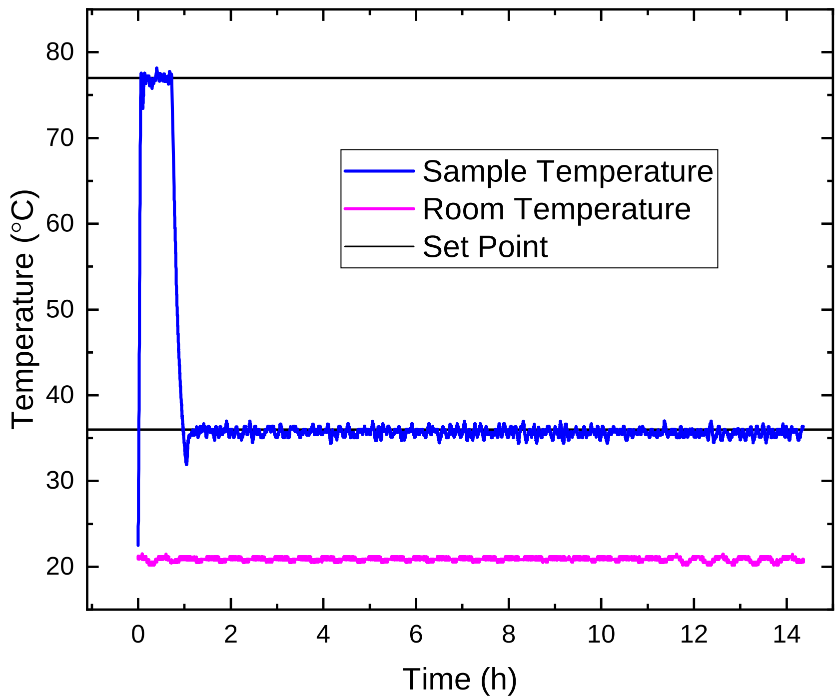


Figure 5: Sample data of PID loop heater performance. Overshoot is minimized to 2 K while undershoot is allowable, and measured at 5 K.