

# **MEMS pressure sensor testing at liquid helium temperatures**

John Spear

Advisor: Dr. Gary Ihas

University of Florida Physics REU

August 4, 2010

## **Abstract**

MEMS pressure sensors ranging from 130-170  $\mu\text{m}$  in diameter were tested in a suckstick with temperatures ranging from 4.2 K to 30 K to investigate their pressure sensitivity and temperature dependence. A wheatstone bridge was constructed using a pair of capacitors and a ratio transformer in an attempt to diminish the capacitors' temperature dependence. Unexpected behavior of the voltage curves from this circuit led to the assumption that both temperature and pressure were dominating the voltage readings in different temperature regions. Temperature is thought to dominate initially at 4.2 K with the vast majority of the gaseous helium atoms adsorbed onto the cell wall and therefore unable to contribute to the pressure. As the cell warms, pressure dependence is thought to become more significant with a larger fraction of helium atoms breaking free from the cell wall and contributing pressure. The entire apparatus used to test the sensors was hand-made in lab and the machine shop at the University of Florida and is described in further detail.

## Introduction

A vast number of experiments and applications require the use of pressure sensors in the field of low temperature physics. These experiments often involve liquid helium and require very high spatial resolution. The purpose of this experiment was to test, at liquid helium temperatures (around 4.2 K), lab designed micro-electro-mechanical system (MEMS) pressure sensors. The sensors were constructed from a multi-layer polysilicon surface which used hydrofluoric acid to etch holes for capacitors [1]. The capacitors on the polysilicon surface were the physical pressure detectors and ranged from about 130-170  $\mu\text{m}$  in diameter. A total of 16 capacitors were deposited onto the surface; 12 were electrically connected in pairs and four were wired to be lone capacitors. The pairs were designed such that only one of the two capacitors was etched by the hydrofluoric acid (HF). Only the capacitors wired in pairs were of interest for this experiment because lone capacitors are temperature dependent, while the circuit used was set up to diminish the temperature dependence of a pair of capacitors. One of the capacitors in the pair was sealed so no HF could flow in between the plates; the other capacitor was open with a miniature hole to allow the HF to eat away the material between the membranes. The cell and capacitors were cooled to 4.2 K, and the gas inside was heated according to the ideal gas law by a heater wire



**Figure 1: Pressure sensors next to the copper cell which has resistance wire wrapped around and coated in varnish.**

wrapped around the sealed copper cell. Temperature fluctuations allowed the effective measure of pressure as a function of temperature. Figure 1 shows the pressure sensors plugged into a pinboard next to the copper cell wrapped in the heater wire. Two pairs of capacitors were tested in the experiment, the pair

described above with a closed and an open capacitor and one pair with both capacitors closed for a control measurement.

The entire apparatus that the sensors were mounted on was built by hand. A copper cell was built for the sensors to sit inside while under a helium atmosphere, multiple miniature coax cables were constructed by hand, all electrical wiring was soldered by hand, and the physical mounting device was created in the lab as well. Everything was assembled to fit inside a suckstick 42 mm in diameter. Calculations took into account the time constant for the capacitors to equalize pressure, the shape of the capacitors as the top plate bent, the heat transported through all electrical wires, the amount of helium boiled off, and the amount of energy needed to heat up the copper cell to a specific temperature for pressure readings.

## **Experimental**

The apparatus was completely assembled in the lab and the machine shop at the University of Florida. Before anything could be assembled, multiple calculations were conducted to find the time constant to equalize pressure to ensure the sensors would not break, the capacitance for the specific geometry of the sensors, the heat transported through all wires, and the optimal amount of current to run through the heater wire to heat the copper cell for pressure measurements.

Capacitor pairs were of interest and, as previously mentioned, one of the capacitors was sealed while the other capacitor in the pair was open with a miniature hole and allowed to deflect. The time constant for the open capacitor to equalize pressure was of interest to ensure too much force was not applied and to understand the sensitivity time rate. The time constant turned out to be on the order of milliseconds, which was important for the purposes of this

experiment. With such a short time response, measuring pressure changes would be difficult. Even with such a short response, precautions for not altering pressure too quickly were taken regardless. Since the pressure change was not a critical issue, the most important calculation concerning the capacitors was finding the capacitance for the odd geometry they created.

The capacitors were circular parallel plate capacitors when no pressure difference was present, but rather than staying flat, the top plate bent into a bowl shape when pressure was applied. The distance the top plate deflected from its flat position can be found by solving a differential equation for a circular membrane under uniform pressure with fixed boundary conditions for both position and its derivative to obtain

$$D = \frac{3P(1-\nu^2)}{16Et^2} (R^2 - r^2) \quad (1)$$

where P is the applied pressure difference (Pa),  $\nu$  is Poisson's ratio, E is the elasticity of the plate (Pa), t is the thickness of the plate (m), R is the total radius of the flat plate (m), and r is the radial position from the center of the plate (m). The capacitance was then calculated for various pressures using the standard equation for capacitance [2]

$$C = \epsilon_0 \frac{A}{d} = \epsilon_0 \frac{A(r)}{D_i - D(r)} \quad (2)$$

where  $\epsilon_0$  is the permittivity of free space, A(r) is the area of the plate as a function of r,  $D_i$  is the distance from plate to plate when they are flat, and D(r) is the deflection stated in Eq. 1 as a function of r. Eq. 2 was integrated over r from zero to R to get the entire capacitance of the sensor.

Managing the amount of heat in the copper cell where the sensors were placed was critical to the success of the experiment. Wires connected to the copper cell ran all the way up

and out of the suckstick which produced a large temperature gradient and would allow the transport of heat into and out of the system. A total of nine miniature coax cables and four fine copper wires could transport heat, so all 13 had to be taken into account. The power, in watts, each wire carried can be represented by [3]

$$\frac{dQ}{d\phi} = kA \frac{dT}{dx} \quad (3)$$

where Q is heat (J),  $\phi$  is time (s), k is the thermal conductivity (W/m\*K), A is the cross sectional surface area of the wire (m<sup>2</sup>), T is the temperature (K), and x is the length of the wire (m). The wires carried heat calculated to boil off about 0.12 L/hour since helium has a heat of vaporization of 20.9 J/g at 4.2 K [3]. The other temperature control feature on the apparatus was a heater wire which wrapped around the copper cell shown in Figure 1. The heater wire was nothing more than a 4.7  $\Omega$ /ft resistance wire which was cut to 4.25 ft to provide 20  $\Omega$  of resistance. The power put into the system was calculated using [2]

$$P = I^2R \quad (4)$$

where P is the power (W), I is the current (A), and R is the resistance ( $\Omega$ ). The volume of the copper cell and copper's heat capacity of 24.47 J/mol\*K were taken into account to find out how much energy was needed to raise the cell to a specific temperature [3]. The experiment called for about 815 J to raise the temperature to 30 K.

The copper cell was a cylindrical shell with a flange-like cap at one end and a small opening at the other end, out of which ran a capillary tube. The pressure sensors were assembled in collaboration with Professor Chan's group at the University of Florida and were plugged into a pin-board along with a miniature Scientific Instruments Ruthenium Oxide thermometer to monitor the temperature inside the cell. The cap of the cell was closed with an indium seal to

ensure a vacuum tight seal. The wires from the pin-board ran through a hole in the cap which was sealed with Stycast 2850 epoxy to keep the vacuum seal. The 4.25 ft long resistance wire of  $4.7 \Omega/\text{ft}$  mentioned earlier was wrapped around the outside of the cell to heat it. Wires attached to the capacitor leads were connected to hand-made miniature coax cables to decrease the electrical noise of capacitance measurements. The thermometer leads and heater wire were attached to fine copper wire and were run up to the top of the suckstick and out of the flange on top. The copper cell which encased the sensors was mounted such that it would hang as close to the bottom of the suckstick as possible to ensure the liquid helium level was above the cell. Three screws were threaded into the cell cap and attached to an aluminum circular disk above the cell for stabilization. The disk was attached to a long hollow stainless steel tube with 0.25" outer diameter and 0.02" thickness by swage locks. The stainless steel tube extended up and out of the suckstick through the flange on top. Small holes were drilled into the tube at the bottom and the top right below the cap of the suckstick to prevent Taconis oscillations. The cell and mounting device are shown in Figure 2.

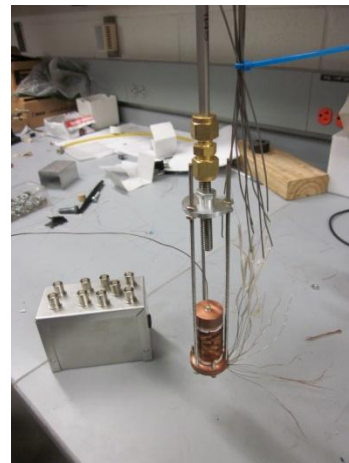


Figure 2: Copper cell attached to mount with coax cables connected.

Once the cell was sealed with the sensors inside, the air was pumped out through the capillary because the sensors could corrode in an oxygen rich atmosphere if exposed for extended periods of time. The seals were leak tested with a helium leak detector at room temperature (293 K) and at liquid nitrogen temperature (77 K). Once the cell was known to have no leaks, one atmosphere of helium gas was released into the cell at room temperature and the capillary was shut with a valve. Next, each wire was tested before and after the apparatus was inserted into the suckstick to ensure it was not shorted to ground. When the apparatus was slid

into the suckstick and no electrical shorts were present, the suckstick was sealed and pumped out. The coax cables were soldered to a grounded shield box so commercial coax cables could



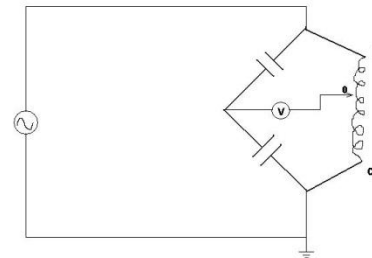
**Figure 3: The suckstick inside the liquid helium dewar attached to electronics.**

easily be plugged in for measurements. The thermometer leads were then attached to a power source supplying 10  $\mu\text{A}$  and a voltmeter to track the temperature in the cell.

The calibration data to look up temperatures for the Ruthenium Oxide thermometer were available on the Scientific Instruments website under the model number RO105 [4]. The heater wire was connected to a variable

power source and an ammeter to supply the energy needed to heat the cell. Finally, the entire suckstick was slowly lowered into the 110 L liquid helium dewar and cooled to 4.2 K for the data collection as shown in Figure 3.

Since materials are known to have significant temperature dependence, a voltage difference between the pair of capacitors rather than individual capacitance measurements alone were used to reduce the temperature dependence of the measurements. This was accomplished with a form of a wheatstone bridge and a ratio transformer. The circuit, shown in Figure 4, uses the two capacitors in a pair and connects them to a ratio transformer which acts as a variable inductor to adjust to zero the measured voltage and subsequently balance the bridge. The voltmeter in the wheatstone bridge and the voltmeter attached to the thermometer were hooked up to a LabVIEW interface on a computer to automatically record



**Figure 4: Wheatstone bridge with two capacitors and a ratio transformer acting as a variable inductor.**

data. After the circuit was built and the bridge was balanced, the LabVIEW program was turned

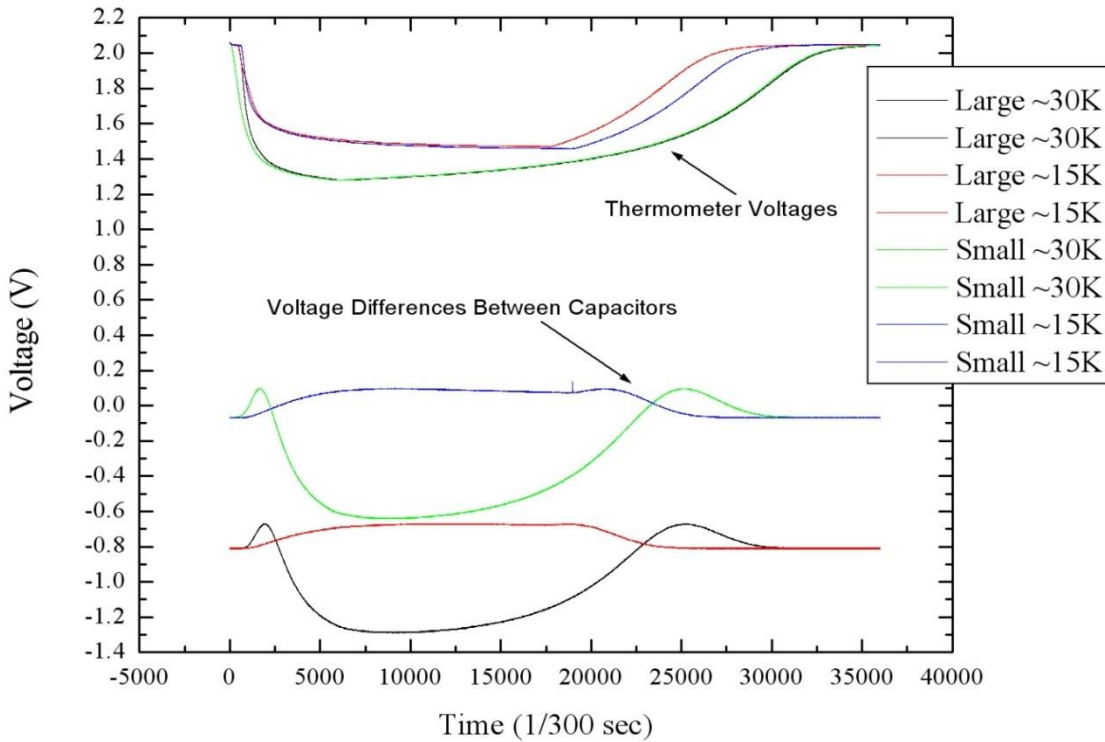
on to collect data at a rate of 300 Hz for 2 min from both the thermometer and the wheatstone bridge. Once the program began collecting data, the power source connected to the heater wire was turned on to supply a current and raise the cell to the desired temperature. Two runs were conducted for each pair of capacitors, and all the data are present on Figure 5 in the Results and Conclusions section. The large capacitors (170  $\mu\text{m}$  diameters) were the control measurements where both capacitor membranes were assumed to be immobile while the small capacitors (130  $\mu\text{m}$  diameters) were described above with one capacitor having a fixed membrane and the other a flexible membrane. All runs started at 4.2 K and increased to either 15 K or 30 K which Figure 5 indicates. The individual capacitance of each capacitor was also measured for reference as the temperature increased from 4.2 K to 30 K. These four graphs are shown in Appendix I.

## **Results and Conclusions**

Figure 5 shows the voltage readings from the thermometer voltmeter and the wheatstone bridge voltmeter as a function of time. Data points were taken every 1/300 seconds for 2 min each run. Four data runs were taken throughout the entire experiment, each run producing 2 voltage curves. The legend indicates whether the run measured the small capacitor pair or the large capacitor pair and to what temperature the cell was heated. The large capacitor pair consisted of 2 closed capacitors which were plugged and therefore not etched out by hydrofluoric acid. The small capacitor pair consisted of 1 closed capacitor and 1 open capacitor that had been etched out by hydrofluoric acid so the top plate could deflect. Both capacitor pairs were run in temperature ranges from 4.2 – 15 K and from 4.2 – 30 K.



### Voltage vs. Time for Thermometer and Voltage Difference Between Capacitors



**Figure 5: Voltage vs. Time graph for all four data runs. The top four curves are the voltage readings of the thermometer while the bottom four curves represent the difference in voltage between the two capacitors measured by the wheatstone bridge in Figure 4.**

Each trial started at 4.2 K and increased rapidly to either 15 K or 30 K illustrated by the quick drop in voltage of the thermometer curves. After 1 min of heating, the current through the heater wire was stopped and the cell began to cool which is shown by the slower increase in voltage of the thermometer curves. These data exhibit normal behavior which was expected. The interesting data are illustrated in the voltage differences between the paired capacitors shown in the lower four curves of the graph. The curves representing temperature increases up to 15 K rise in voltage and level off before dropping again during their cooling phase. The curves representing temperature increases up to 30 K indicate unexpected behavior of a rapid voltage increase followed by a large drop in voltage shown in Figure 5. During the phase in which the cell cools back down, the voltage increases to the same value as the previous maximum before

dropping back to the original voltage at 4.2 K. This indicates that the measurements are not purely electrical but represent a real physical effect on the sensors. Though the behavior is not completely understood, it is thought to show the temperature and pressure dependence of the sensors each dominating in different temperature regions. Temperature dependence is thought to dominate initially in the temperature range from 4.2 K to just over 15 K because at temperatures that low, the helium atoms inside the copper cell are adsorbed onto the walls so the sensors cannot be affected by them. Helium atoms will break free from the wall as the heater wire raises the temperature of the wall above the threshold and the pressure dependence will begin to dominate as shown in Figure 5. Essentially, the system acts according to the ideal gas law with the number of moles of gas fluctuating. This behavior of the atoms being adsorbed back onto the cell wall may explain why the voltage jumps back up to the maximum shown in Figure 5 before dropping again to the starting voltage. To verify the data shown truly exhibit pressure dependence, further experimentation must be performed with all the same parameters except different pressures, such as 0.5 atm or 2 atm of helium, to see if and when the same behavior occurs.

## **Acknowledgments**

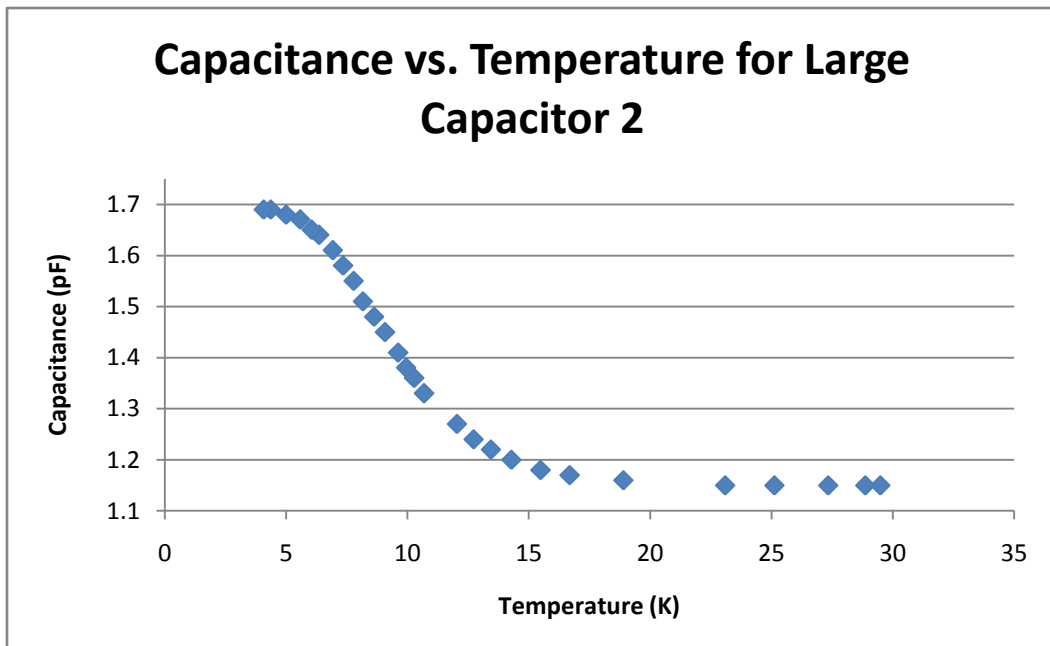
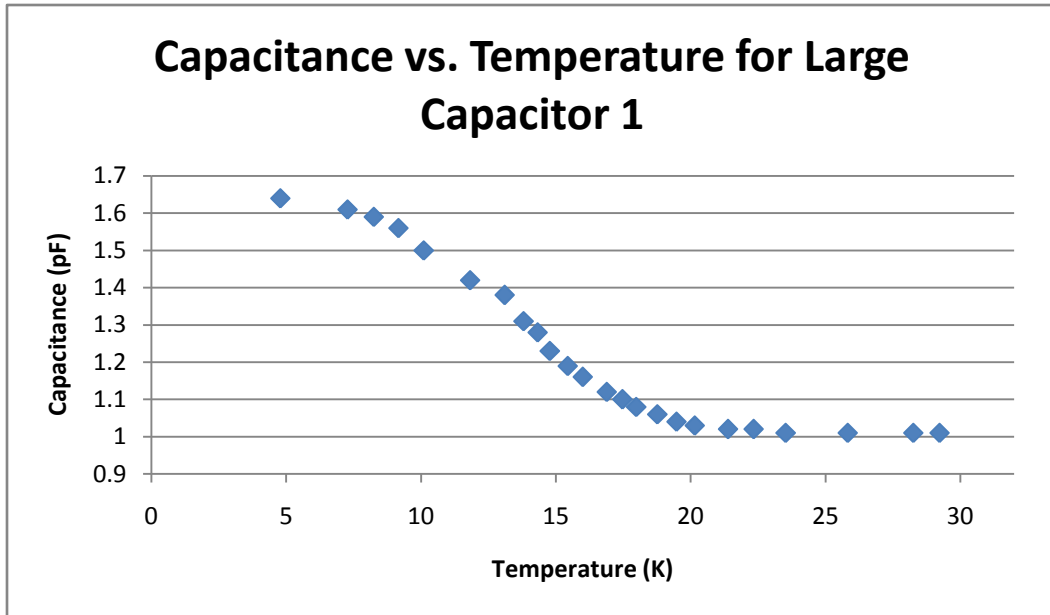
I would like to thank the University of Florida for accepting me into their REU program and NSF for funding their program. I would also like to thank Kyle Thompson, Dr. Gary Ihas, Travis Nelson, and Milap Patel for all their help on this project. This research was supported in part by US NSF grant DMR 1007937.

## References

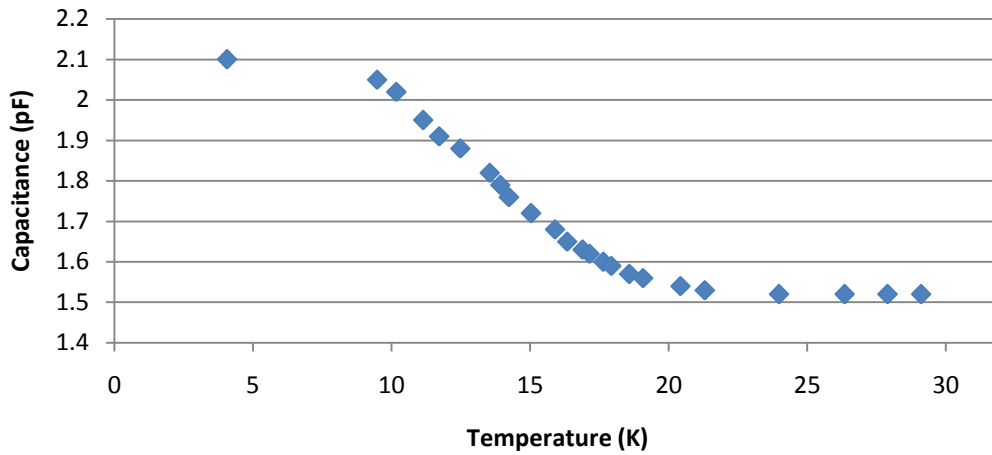
- [1] J. Carter, A. Cowen, and B. Hardy, *PolyMUMPs Design Handbook*. Rev. **9**, 1-3 (1992-2005).
- [2] D. Griffiths, *Introduction to Electrodynamics, Third Ed.* (Prentice Hall, Inc., Upper Saddle River, NJ, 1999).
- [3] T. Flynn, *Cryogenic Engineering: Second Edition Revised & Expanded* (Marcel Dekker, New York, NY, 2005).
- [4] *Report of Calibration for Ruthenium Oxide Thermometer.* (Scientific Instruments, Inc., West Palm Beach, FL, 2005).

## Appendix I

The four graphs below indicate the individual capacitance measurements as a function of temperature for each capacitor on the temperature range from ~4.2 K to ~30 K. Both large capacitors were closed while one of the small capacitors was open.



### Capacitance vs. Temperature for Small Open Capacitor



### Capacitance vs. Temperature for Small Closed Capacitor

