

**MEMS Pressure Sensor Testing at Liquid Helium Temperatures Using
a Pressure Bomb**

Carley Nicoletti

Advisor: Dr. Gary Ihas

University of Physics 2012 REU Program

August 2012

Abstract

MEMS (micro-electro-mechanical system) pressure sensors were tested at 4.2 Kelvin to measure their pressure sensitivity, to evaluate their suitability for the detection of quantum turbulence. A pressure bomb would be utilized to ensure sensitivity solely to pressure, rather than pressure and temperature. An apparatus originally developed in a former project was modified and cooled in a suckstick refrigerator, allowing quick turn-around from room temperature to 4.2 K. Many changes were made to the original apparatus to make the cool down as successful as possible. Since the ultimate goal is the use the pressure transducers at millikelvin temperature, familiarity with the dilution refrigerator was established

Introduction

Turbulence is not completely understood and is still one of the great mysteries of physics today, even though it is present in almost every fluid. Quantum turbulence occurs when a quantum fluid undergoes turbulent flow¹. Since classical turbulence is closely related to quantum turbulence¹, it is an extremely important topic to study to determine how turbulence develops and decays. Quantum turbulence is broken into two parts in the University of Florida's research: visualization and decay. Pressure sensors are invaluable when investigating the decay of quantum turbulence, enabling the measurement of pressure fluctuations and therefore energy content in the fluid.

One of the main ways the turbulence is being produced is through the use of a frictionless linear motor. A stainless steel grid is attached to a superconducting niobium rod. With the use of magnetic solenoids, the rod, and therefore the grid, move up and down through magnetic levitation. When quantum turbulence decays, there is a slight temperature increase. Since the temperature increase is so miniscule (order of microKelvin), it is extremely important to make the motor frictionless, since friction generates heat. Any occurrence of quantum turbulence decay can be detected via this slight change. Another way of detecting turbulence is through the pressure fluctuations generated in the fluid by the turbulence. MEMS pressure sensors may be used to identify these small pressure changes once their sensitivity is known². Therefore, the purpose of this research project is to test the sensitivity of the pressure sensors so that they are able to be used in future experiments.

Future experiments are to be performed at millikelvin temperatures using a dilution refrigerator. Therefore, a familiarity with dilution refrigeration was developed³, and then a dilution refrigerator was partially disassembled and prepared for mounting of the transducers.

A previous REU student developed an apparatus that could be placed into a suckstick⁴ apparatus. A suckstick is designed to hold a small cell in a helium dewar. The suckstick is able to get to temperatures as low as 1K, which is sufficient for this specific project. The purpose of his experiment was to bring the suckstick to temperatures ranging from 4.2K to 30K in order to characterize the effect of pressure and temperature on the MEMS pressure sensors⁵. A Wheatstone bridge was built to compensate for the voltage dependence of the capacitive pressure transducers on temperature. Despite his

efforts, this approach did not succeed, and he was unable to distinguish between temperature and pressure changes. His experiment was not able to truly show the pressure dependence and as a result, he was not able to test the sensitivity of the pressure sensors based solely on the pressure changes⁵. It was determined that the use of a pressure bomb would help in accomplishing this task.

The pressure bomb uses another volume that will be placed on top of the cell on the original apparatus. The cell contains the pressure sensors as well as a thermometer, while the pressure bomb contains a heater that increases the temperature and pressure in its volume. Due to the ideal gas law, when the temperature increases inside the pressure bomb, the pressure will increase as well. Through a tube connecting the pressure bomb to the cell, when the pressure increases in the pressure bomb, the pressure will increase inside of the cell. But since the cell is immersed in liquid helium and the bomb is in helium vapor, the thermal connection between them is very poor. Hence, the cell pressure rises without increasing the temperature. This will enable us to determine the sensitivity of the MEMS pressure sensors. A diagram of this set-up is shown below in Figure 1.

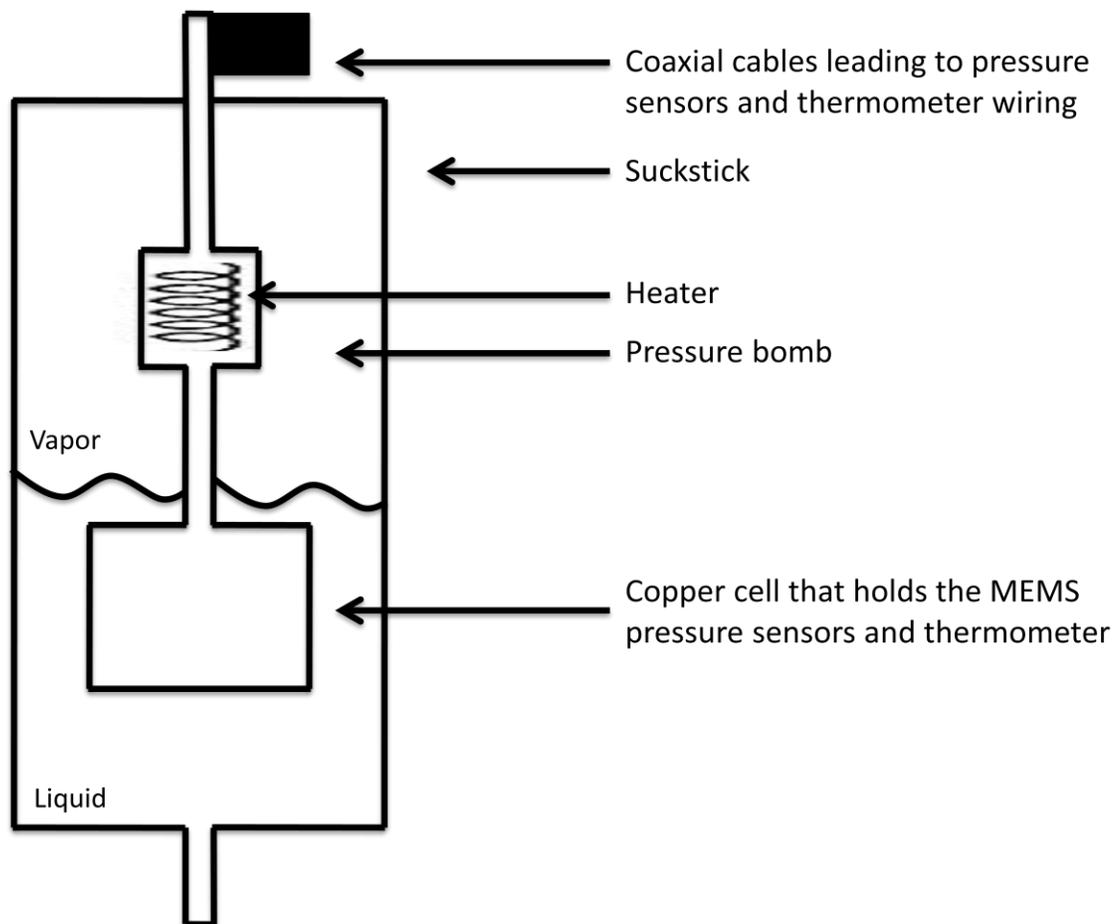


Figure 1: A diagram of the suckstick with the apparatus inside it. The pressure bomb is shown with the heater inside. The pressure bomb is attached to the copper cell by a capillary. When the temperature rises inside of the pressure bomb, the pressure will rise inside of the copper cell. The copper cell is in liquid helium, while the pressure bomb is in vapor, which will allow the temperature, and hence pressure, to rise quickly in the pressure bomb without raising the temperature of the cell.

Method

Before it was possible to begin developing the pressure bomb, some corrections needed to be made to the original apparatus. The capacitances read along the pressure sensors, which are shown in the Table 1, were off by a factor of 20. It was apparent that there were some serious problems throughout the apparatus created by the former REU student because these high capacitances meant that something was not working correctly

in the set up. It was believed that the connections between the coaxial cables and the wires to the sensors were the issue. As a result, all of the connection pieces needed to be removed and fixed. The home-made rigid coaxial cables were cut down and the wires were stripped back, the connector pieces were then reapplied.

Sensor	Capacitance (pF)
5-7	243.96
6-7	254.20
11-12	266.59
12-13	246.81
14-16	405.12
15-16	415.27

Table 1: Table of the first capacitance values taken across the MEMS pressure sensors

Once this was accomplished, the capacitances across the pressure sensors were measured again. They all appeared to be stable, though they were still off from the expected value of about 5 pF. In another attempt to fix the capacitance across the pressure sensors, the other side of the connecting piece that attaches to the pressure sensors was removed and made into a plug. This was done to try to prevent any possibilities of shorts. The capacitances were measured to ground; the values displayed in Table 2 were reasonable and allowed the experiment to move on to the next step.

Sensor	Capacitance (pF)
5-7	3.01
6-7	3.19
11-12	3.13
12-13	4.25
14-16	2.61
15-16	2.70

Table 2: Table of the capacitance values taken across the MEMS pressure sensors after corrections were made. These capacitors were grounded when the values were collected.

It was now necessary to create a Wheatstone bridge detector circuit to use at low temperature to see if the pressure sensors worked. The Wheatstone bridge consists of 4 capacitors. One of these capacitors would be a variable capacitor, another would be the capacitor formed by the pressure sensor, and the final two needed to be the same specific capacitor value. The design for the Wheatstone bridge is shown in Figure 2.

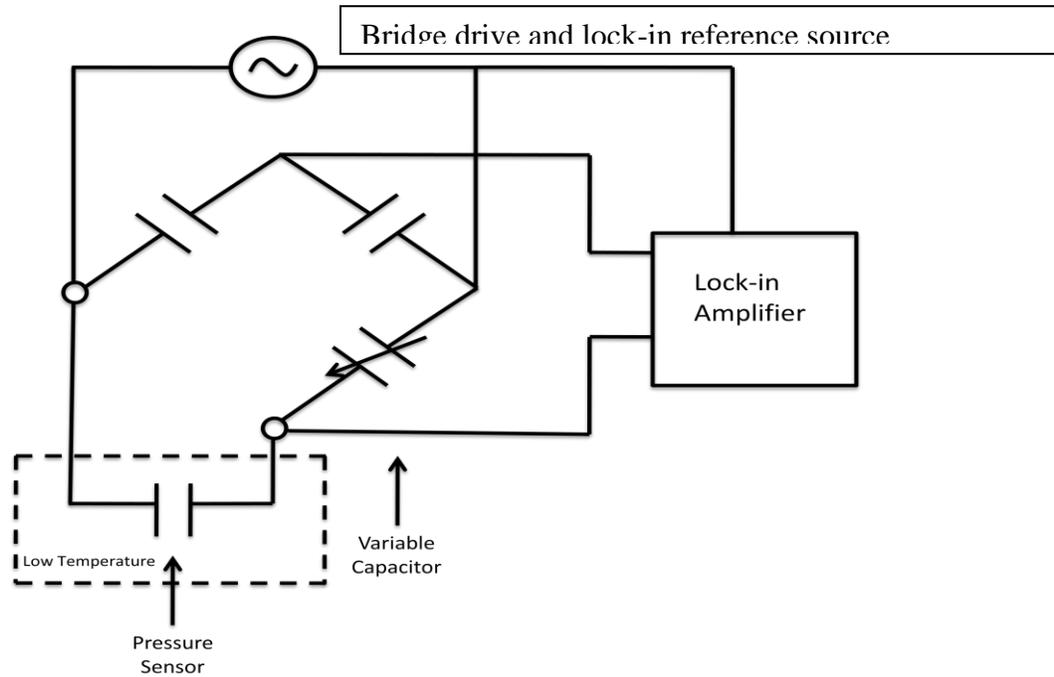


Figure 2: The Wheatstone bridge circuit. It is composed of 4 capacitors. One capacitor is the variable capacitor, another capacitor acts as the pressure sensor and the final two are fixed capacitors each reading 1 pF. The pressure sensor will be cooled down to 4.2 Kelvin.

In order to find the values of the two fixed capacitors, the total capacitance across the six sensors needed to be calculated

$$\frac{1}{C_T} = \frac{1}{C_1} + \frac{1}{C_2} + \frac{1}{C_3} + \dots + \frac{1}{C_n}$$

where C_T is the total capacitance and every other C value is the capacitance of a pressure sensor listed in Table 2. The total capacitance computed was 0.51177pF. This equation

was then used again to finally determine the two fixed capacitor values. C_T was the value received from the first calculation; one of the other C values was the variable capacitor set at 50% of its possible capacitance (xx pF), and the final two C values were the unknown fixed capacitors. It was determined that the two fixed capacitors needed to be 1pF each. The completed Wheatstone bridge is shown in Figure 3.



Figure 3: The completed Wheatstone bridge with the 2 capacitors reading 1 pF and 1 variable capacitor. The final capacitor, the pressure sensor, is inside the cell and the capacitance is able to be read through the electronics connected to the Wheatstone bridge. Connections to the low temperature pressure transducer, the bridge drive source, and the lock-in amplifier can be seen as the cables connected to the BNC connectors on the box.

The cell fill capillary, mounted on the insert for the suckstick, was attached to a vacuum pump. The lowest pressure that it could be pumped down to was 0.10 torr. However, at this time, it was noticed that the capillary attached to the cell had snapped, which created a leak in the system. As a consequence, the cell had to be broken apart. A new capillary was attached with epoxy and the cell was closed with an indium seal to insure that no leakage would occur. The apparatus was then placed back into the suckstick and pumped out to vacuum pressure. It was left for about 17 hours overnight to check for leaks. The pressure reading the following morning was atmospheric pressure

instead of vacuum pressure. Therefore, leak tests needed to be performed in order to locate the problem.

Leak checking was a vital part to ensuring the positive outcome to a cool down in the suckstick. A 'bubble test' is used first in hopes of revealing the leak. Nitrogen is used to pressurize the suckstick, so that the pressure in the suckstick is greater than atmospheric pressure. A mixture of soap and water is applied to the possible leak points around the cell. If a leak is present, bubbles will form. In some cases, the leaks were too small to find with the bubble test. The next procedure necessary is the 'acetone test.' When acetone is placed on the possible leak points when the suckstick is under vacuum, the pressure increases by a large amount, as measured on a thermocouple gauge, if there is a leak detected. This is because the acetone passes through small leaks and produces a highly thermally conducting vapor in the vacuum system, which is detected by the thermocouple gauge.

It was then discovered that the top of the capillary was snapped and that it needed to be replaced. The capillary was fixed once more and tested for leaks in the suckstick. The suckstick was pumped down to vacuum pressure and was able to get down to roughly 0.6 torr. This value decreased because the leaks had been repaired. It was confirmed that the cell was leak tight. The cell was then filled with nitrogen in order to determine its volume. With this measurement, it would be possible to confirm that the entire capillary leading to the cell was fully opened and there was nothing blocking the path to fill and pressurize the cell. We measured the volume to be $2.567 \times 10^{-5} \text{ m}^3$, which is consistent with the measurement done by the previous REU student. With this

information, the time Δt necessary to pump out the cell to vacuum pressure could be calculated

$$z = \frac{\Delta P(P_2)}{V_1 P \dot{p} \eta}$$

where z is the impedance, $\Delta P / \dot{p}$ is Δt (\dot{p} is the standard time derivative of pressure), P_2 is atmospheric pressure, V_1 is the volume of the cell and capillary and η is the viscosity of nitrogen. Δt is calculated to be 91.241 seconds. Based on this conclusion, the capillary was open and clear and the apparatus was ready to be cooled down since after 30 minutes the cell was pumped down to 0.60 torr. The suckstick was flushed with helium before placing the suckstick in the dewar. The Wheatstone bridge was attached to the apparatus with a lock-in amplifier, shown in Figure 4, and tested to make sure the electronics are prepared for cool down. During this check,



Figure 4: On the left is the lock-in amplifier. When attached to the wheatstone bridge, the voltage difference is able to get to zero with the help of the variable capacitor. The capacitance across the variable capacitor is important when the voltage difference is zero. On the right is the LCR bridge which is used to read the capacitance across the variable capacitor.

sensors 5-7, 14-16 and 15-16 were found not to be working. The reading on the lock-in amplifier did not vary when the variable capacitor was altered. The decision was made to proceed with the cool down since the majority of the sensors were working.

The suckstick was placed inside of a helium dewar and was cooled down to 4.2 Kelvin. Capacitance readings were taken at 4.2 K to make sure that all of the electronics were still working properly at this temperature. The capacitances were taken when the voltage difference read on the lock-in amplifier was 0 mV. When the voltage difference is zero, the bridge is balanced, so the setting of the variable capacitor is equal to the unknown capacitance of the pressure transducer being tested. When the voltage difference changes the capacitance across the pressure sensors is changing since the four capacitors are no longer in balance. It was observed that sensors 5-7, 14-16 and 15-16 began working correctly at low temperatures as seen in Table 3. A RED-I (Roving

Sensor	Capacitance (pF)
5-7	36.189
6-7	36.548
11-12	41.626
12-13	37.145
14-16	39.674
15-16	38.218

Table 3: Table of the capacitance values taken across the variable capacitor when the voltage difference on the lock-in amplifier was zero. This occurred once the pressure sensors were cooled down to 4.2 Kelvin. Sensors 5-7, 14-16 and 15-16 began working at this temperature.

Experimental Device Investigator) was connected for leak checking and providing the ability to pump through a liquid nitrogen (LN2) trap. The RED-I is shown in the middle of Figure 5.

RED-I

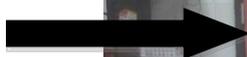


Figure 5: The entire set up for the cool down process is shown. The RED-I can be seen as well as a helium gas tank, the electronics and the suckstick inside of the transport dewar.

This device became very useful once the cool down progressed. There was a major leak in the tubing somewhere between the suckstick and the RED-I. The leak was known to exist because the pressure would increase 1 torr every 9 seconds. The leak checking began by pumping down through the RED-I and using clamps to prevent flow through certain connections. If the pressure stopped increasing, then the area within the clamps was leak tight. The area of tubing and connections were expanded an amount each time until the leak was detected. The leak was found in the yellow tubing, which can be seen in Figure 4. After the yellow tubing was replaced, the pressure would go up 1 torr every 50 seconds, this was a significant improvement.

Using the RED-I, the cell was pumped out. Unfortunately, after attempting the evacuation, the pressure was reading atmospheric pressure instead of vacuum pressure. This meant that there was helium present in the cell and therefore the cell had a leak. It was then concluded that there was a leak in the cell through the epoxy seal or the indium seal. Fortunately, data was collected for sensor 12-13 at two various pressures which is

shown in Figure 8. This meant that the pressure sensors work as they were reading different capacitances at different pressures.

Sensor 12-13		
Pressure in cell	176.91 torr	804.91 torr
Capacitance when Voltage Difference = 0	23.186 pF	22.228pF

Table 4: The data collected for sensor 12-13 at 4.2 Kelvin at two different pressures. It is shown that as pressure increases, the capacitance across the sensor decreases.

At that point the pressure sensor readings stopped changing when pressure was increased in the cell, which indicates that the sensors were being pushed to their physical limit. Since no more data could be collected, the warming up process began. This was a very careful process because if the suckstick was removed from the dewar too quickly, the cell had the potential to blow up. As temperature increases, pressure increases so a pressurized liquid inside of a container that is warming up is a bomb. Therefore the temperature was slowly increased over two days so that the helium had time to escape to the recovery system.

With the suckstick safely removed, leak checking the cell was possible. First, in order to prepare for another cool down, it was important to check that the thermometer was working correctly. The cell was opened up and the resistance across the thermometer was confirmed at 100.1 k Ω at room temperature. The copper cell was closed using an indium seal. Even though the indium seal could have been the problem during the first cool down, it was important to leak check again in case that the leak occurred at one of the two epoxy joints at the top and bottom of the cell. The bubble test and acetone test failed to find a leak, so using the mass spectrometer was the next step. A

helium mass spectrometer is used for small leak detection. The leaks are detected to sizes as small as 1×10^{-9} moles per second. A closed container, like the copper cell at the bottom of the apparatus, is pumped down using a vacuum pump to 10^{-6} torr. Helium gas is then sprayed near the possible leak points. The mass spectrometer will detect the rate that the helium leaks through the closed container if there is a leak. After running the mass spectrometer once it was connected to the copper cell, it was shown that the helium was leaving the cell at a very slow rate. It was interpreted that this could be helium diffusing through the epoxy, which is expected at room temperature. To confirm this idea, the mass spectrometer was used to leak check again but this time a plastic bag was placed over the cell and then sealed. The bag was then filled with helium and the mass spectrometer will detect the rate that the helium leaks through the closed cell. This test showed that the copper cell was leak tight. It was decided that a third test with the bag over the cell would be conducted to ensure that the next cool down would have a positive outcome. The third test using the mass spectrometer showed that a large leak was present. This was not the result anticipated, therefore another form of leak checking, the helium sniffer, was used. The cell is filled with helium, then the sniffer probe, detecting helium by measuring the thermal conductivity of the gas it sniffs, is guided over the areas of possible leaks. This device allows one to find the exact location of the leak when the probe is passing over the faulty area. The outcome of this test was that the cell was leak tight. As a precaution, the epoxy seals at the top and the bottom of the cell were redone.

The apparatus was attached once more to the mass spectrometer. It was checked three times again. The first time was by spraying helium around the possible leak points and the final two were with the bag method. All three times proved that the cell was leak

tight. The apparatus was placed back into the suckstick and put through a helium flush to prepare for cool down. The suckstick was placed into a helium storage dewar and cooled down to 4.2K. It was confirmed that the suckstick was at this temperature when the resistance on the thermometer read 356 k Ω , which is equivalent to 4.2K when looking at the thermometer calibration chart. The capacitances were read across the variable capacitor once the voltage difference was zero to check and see which sensors were working. As shown in Table 5, sensors 14-16 and 15-16 were not working at this low

Sensor	Capacitance (pF)
5-7	25.32
6-7	24.75
11-12	46.38
12-13	48.11
14-16	Did Not Work
15-16	Did Not Work

Table 5: Table of the capacitance values taken across the variable capacitor when the voltage difference on the lock-in amplifier was zero in the second cool down. This occurred once the pressure sensors were cooled down to 4.2 Kelvin. Sensors 14-16 and 15-16 were not working at this temperature.

temperature. By adding helium gas, we were able to change the pressure that was inside of the cell. When this occurred, we expected to see the voltage difference change across the pressure sensor at that time. We began with sensor 12-13 and when pressure was added, no change occurred. Unfortunately, when checking other sensors, they also exhibited no change. It was decided that the capillary leading the helium gas to the cell was blocked with frozen air, and as a result the pressure sensors could not be tested. The suckstick was taken out of the helium storage dewar and left to warm up for two days (the weekend).

Conclusion

Even though the pressure bomb was not developed, there were positive outcomes as a result of this research opportunity. After some corrections to the original apparatus, it was confirmed that the MEMS pressure sensors do work and are sensitive at low temperatures near 4.2K. It is important to understand that without the pressure bomb, it is impossible to differentiate whether the sensitivity on the pressure sensors are changing due to temperature or pressure changes. In this specific area of low temperature physics, there is more that can be done with MEMS pressure sensor sensitivity. It is important to continue and proceed with building the pressure bomb so that these pressure sensors can be used in quantum turbulence decay. One would also be able to test the sensitivity at even lower temperatures. In order for this to occur, another cell would be placed on top of the original copper cell which would raise the temperature causing the pressure to increase as well. With this growth in the knowledge of MEMS pressure sensors, they could be used to measure the small pressure fluctuations caused by the decay of quantum turbulence.².

Acknowledgements

I would like to thank the University of Florida for this opportunity to participate in the 2012 Summer REU Program as well as NSF for providing the funding for this grant. I also would like to thank Dr. Gary Ihas, Dr. Kyle Thompson, Lydia Munday, Alex Marakov, Roman Chapurin, Mannish Amin, Jihee Yang and Andrew Carnes for their help throughout my stay in Gainesville. I would like to give a special thanks to Dr. Gary Ihas, Dr. Kyle Thompson and Lydia Munday for working so closely with me day in and day out, helping me develop a greater understanding for low temperature physics, specifically, quantum turbulence. The research completed over the ten weeks was supported by the US NSF MWN Quantum Turbulence grant DMR 1007937, as well as NSF grant DMR-1156737.

References

- [1] **W. F. Vinen, J. J. Niemela**, *Quantum Turbulence*, *Journal of Low Temperature Physics* **128**, (2002). Available from: <http://dx.doi.org/10.1023/A:1019695418590>.
- [2] **K. J. Thompson**, *Generation and Characterization of Quantum Turbulence*, Doctorate Thesis at The University of Florida, (2012).
- [3] **Frank Pobell**, *Matter and Methods at Low Temperature*, **Ch. 7 p. 105 ff**, Springer-Verlag Berlin (1992). ISBN 3-540-53751.
- [4] **B. N. Engel, G. G. Ihas, E. D. Adams, C. Fombarlet**, *Insert for Rapidly Producing Temperatures Between 300 and 1K in a Helium Storage Dewar*, *Review of Scientific Instruments* **55 (9)**, (1984).
- [5] **J. Spear**, *MEMS Pressure Sensor Testing at Liquid Helium Temperatures*, REU Paper at The University of Florida, (2010). Available from: <http://www.phys.ufl.edu/REU/2010/projects.html>.