The Challenges of Low Temperature Research

Luke Mauritsen, Brian Smithgall
Montana Instruments Corporation, USA

Introduction

The study of material science at cryogenic temperatures is full of challenges. The nature of work at near absolute zero requires a thorough understanding of the interactions of the room temperature environment on the experimental space. A researcher and colleague once said to me, "The only thing worse than a high vacuum experiment is a cryogenic high vacuum experiment; If a cryogenic experiment is anticipated to take 3 hours, it will take three days, if anticipated to take 3 days, it will take 3 weeks." Most often, the pain of low temperature research results in lost time and lack of complete results. Both of which are vital to a researcher who wants to be first to publish great data.

Although closed-cycle low temperature systems have progressed significantly over the years, many applications require much greater performance than what is commercially available on the market. The state of the art technology available for optical cryogenic research still lacks in performance capabilities and makes low temperature research difficult and time-consuming for the scientist. Many issues remain unsolved.
We contacted several scientists active in the field and collected their thoughts on why low temperature research is so difficult.

Many people who are familiar with low temperature work will find the information in this article very familiar. This paper was written with the researcher in mind who is an expert in materials science or optics, but less familiar with the challenges of research at low temperatures. The author identified and studied these issues as he developed a new cryogenic optical platform, the Cryostation. We hope you find value in becoming more aware of the issues involved in low temperature research. This awareness may help you anticipate and avoid surprises in your low temperature work, and also appreciate the technology that has been developed to solve the issues.

Cryogenic research is valuable…and challenging

Researchers are doing material science investigations at low temperatures for many applications including spectroscopy, microscopy, nanotechnology, high pressure studies, and magnetic studies. There are many reasons for studying materials at low temperatures. Temperature can be viewed as a measure of the kinetic energy of particles, therefore removing the thermal noise floor can be critical to discovering subtle phenomena. In some cases the desire is to achieve an improved signal to noise ratio and find signals buried in the thermal noise floor, while in other cases, the absence of thermal energy creates an environment where physical changes to the material occur. Regardless of the purpose for achieving low temperatures, the successful practical application of experiments at low temperatures is dependent upon how well many of the associated challenges are navigated.

The challenges

Cryogenic research poses many challenges:

Vibrations

The demand for cryogenic systems based on closed-cycle technology is increasing and there is a significant shift away from using liquid cryogens, especially Helium. This shift has many similarities to the historical shift from ice blocks delivered door to door, to the electric refrigerator with the high upfront cost and need for power. Except this time, the major challenge to the acceptance and use of the closed-cycle cryogenic refrigerator is the unstable sample environment it creates. Vibrations and thermal fluctuations limit many types of research in closed-cycle refrigerator systems. Current 4K cryocoolers (both Gifford McMahon "GM" and Pulse Tube "PT") suffer from vibrations at the cooled stage of as much as 60 microns and thermal fluctuations of about 0.25- 0.5 Kelvin. Thermal fluctuations will be discussed in the next sections so we'll focus on vibrations here. The most significant vibration in a cryocooler is due to the low frequency oscillating pressure which expands the pressure tubes and causes the cold stages to not only move, but ring at a natural frequency of one hundred hertz to a few hundred hertz each time the pressure pulse excites the tubes. Pressure tubes in both GM and PT cryocoolers tend to be very stiff structures with very little damping and ring like tuning forks. Pulse Tubes tend to have larger pressure tube vibrations than GMs because rather than having a concentric tube construction they often have separate tubes which carry the gas in and out of the system. This causes an out-of-phase push-pull fight between tubes resulting in X, Y, and Z vibrations on the order of 30 to 60 microns. A GM system in contrast only experiences a comparable vibration in the Z direction, while X and Y vibrations tend to be on the order of a few microns. With either cryocooler, vibrations need to be isolated from the sample platform. Another source for vibrations in cryocoolers is from the internal mechanical components, such as the rotary valves, scotch yoke, bearings, bushings and electric motors. GM's have higher internal component vibrations than Pulse Tubes, mainly due to their need to convert rotary motion into linear motion of the displacers. These internal vibrations need to be isolated from both the sample space as well as the optical table because noise on the laser or optics is just as troublesome as noise at the sample.

So how much vibration is acceptable? Every application has different requirements. In many microscopy and spectroscopy applications, less than 10 nanometers is required, while others have thresholds of hundreds of nanometers. In many cases the level of vibrations required are not yet known by the researcher. If the sample moves with respect to the beam, the signal is effectively smeared, causing laser line broadening and lower resolution results. Vibrations axial to the beam path can introduce Doppler effects on measurements. Closed-cycle optical cryostat products currently offered either isolate from vibrations at the expense of operating at higher temperatures or they achieve low temperatures (4K) but do not isolate from vibrations. Customers have to choose one or the other, or are forced to go back to using an open-cycle Helium consuming solution. Additionally, some current solutions require the customer to do elaborate support installations in order to isolate the sample from vibrations. The fact that researchers have to decide whether or not a low-vibration cryogenic system is necessary is indicative of the early stage that current commercial cryogenic technology is at. By analogy, when shopping for a new car it is an irrelevant to consider whether the car will go 80mph on the freeway or not. Auto technology has advanced such that it is expected that if acceleration to 80mph is needed to pass a truck on the freeway, the performance is there. It seems appropriate that the researcher should be able purchase a cryogenic system knowing that a fundamental need such as a stable sample area that is adequate for today's and likely tomorrow's requirements is as standard as freeway speeds in a car. Closed-cycle cryostat technology is still a field that is ripe for innovative solutions.

Thermal Fluctuations

All cryogenic systems, both liquid helium-based, and closed-cycle have thermal fluctuations on the long and/or short timescale. Liquid helium-based systems tend to have long-term temperature drift fluctuations, and some systems require some type of external PID control loop to stabilize. Closed-cycle cryocooler systems tend to suffer from short term fluctuations of 0.25 to 0.5 degrees at the mechanical frequency of the system which is approximately 1 Hz. If the cryocooler sample stage has a heat load on it, the fluctuations quickly go up because between each gas expansion cycle where the cooling occurs, the input heat raises the temperature of the sample stage. Some vibration isolation stages reduce thermal fluctuations because they add thermal resistance between the sample and the oscillating cryocooler stage. This is not an ideal method for decreasing thermal fluctuations because the added thermal resistance directly affects the base temperature and cooling capacity of the system at the sample. An ideal way to reduce thermal fluctuations is to increase the thermal capacitance without increasing the thermal resistance. In practice it is possible have a large degree of thermal damping (effective thermal capacitance) with effectively no thermal resistance added to the system. An increasing number of applications are limited by thermal fluctuations. For example, some cryogenically cooled optical materials can be used as a frequency reference for actively stabilizing lasers, and have a seventh order temperature dependence at 4K. In this case, fluctuations of more than 10-20mK are limiting.

Positional Drift

For many microscopy and spectroscopy applications the position of the sample needs to remain fixed over large temperature ranges. In this case, sample temperature directly affects the thermal contraction of the materials supporting the sample. Some cryostat sample stages which are supported by long stainless steel tubing can move hundreds of microns as the temperature changes from 300K to 4K. Large drifts like this necessitate frequent re-alignment of optics, and make automation experiments over any significant temperature range impractical. Most materials undergo most of their length change from room temperature to about 50 K and contract much less below this temperature. However, even below 10K materials undergo some temperature dependent length change which can be significant for applications with nanometer-level sensitivities.

Maintenance

Cryocoolers are mechanical systems with moving parts and need maintenance both on the compressor and the cryocooler head. However, this is one area that may have some generally accepted misconceptions. Some cryocooler manufacturers have made nice recurring revenues from the rebuild of cryocooler heads every 10,000 hours. What is more painful is the time necessary to wait for the system to come back from the factory. This has been generally understood by the customer as a black-box system that can only be rebuilt at the factory. In truth, a cryocooler rebuild involves a total of a few hundred dollars in parts that can be replaced in about the same time it takes to fill a bath cryostat with liquid cryogens. This can be done on-site, by a service technician or, in principle it could even be done by the customer themselves if equipped with some parts, tools and a little guidance. Instead of sending an entire system back to the factory, an infrequent rebuild for both the compressor and cryocooler head can be inexpensive and require almost no downtime to keep a system running for over 25 years.

Sample area access and wiring

Anyone with some level of cryocooler experience can probably remember days (and nights before a deadline) of lying on your back or sitting on the floor staring up into the dark sample space of a cryostat with a flashlight in one hand, allen key in the other, being careful not to snag the small wires dangling down. Alternatively, if possible, the whole cryocooler gets flipped upside down until the sample is installed and wiring thermally lagged. Lagging wires, or thermally grounding wires that run into the sample space can be an unexpected challenge, and every wire that runs from 300K to the sample area should be thermally attached to an intermediate temperature stage to avoid excessive heat loads on the sample stage. The challenge is where and how to attach the wires. The location most accessible is the radiation shield surrounding the sample, however, attaching wires to this often means you no longer have access to the sample area because the radiation shield must be in place. Wrapping wires down the pressure tubes of the cryocooler and lagging at the first stage works, but then the entire cryostat must be dismantled (probably upside-down again) in order to make a simple change and if there is any vibration isolation in place the job becomes more difficult. Once a location is found for lagging wires, a method is needed for holding them in place without damaging them but with enough force to effectively pull heat out. Most tapes with adhesive should be avoided because under vacuum the volatiles in the adhesive will just fly away and freeze onto every surface, including the sample and cold windows. Other methods include dental floss, varnish, and cotton pads. A commercial cryogenic system should have a well thought-out method for the average user to run wiring into the sample space. This can be one of the most time-consuming parts of experimental setup without a good method and system.

High utility consumption

Laboratory managers are constantly faced with high operating costs of their equipment. The world's finite supply of helium is concentrated primarily in inclusions in natural gas fields and costs have skyrocketed lately in some nations. In some countries the demand for military or industrial Helium has left researchers with high costs or no availability. History will look back one day, and ponder the days of shipping around liquid helium dewars, and sometimes even venting every drop of boiled-off helium to the atmosphere.

Many labs maintain costly helium recovery systems or cryocooled recondensing units which extend the useful life of the liquid helium systems already in use. With closed-cycle technology making its way into the labs, the long-term utility trade-off is helium consumption costs for electricity costs. The bigger obstacles for labs are that many of the larger cryocooler compressors need 5-7kW or more input power and water cooling.

Condensation, frost and contaminates.

All materials and every interface leaks at some rate. What this means for a cryogenic system, is that the air that leaks in, has to end up on some surface as a frozen solid. Some applications are sensitive to tiny ice crystal formations on the sample. Many cryogenic systems during cooldown will have the sample stage colder than the rest of the cryostat, which causes the surfaces at the sample stage to be the primary surfaces that will cryo-pump or freeze particles inside the vacuum. Ideally, the sample space will lag the other temperatures stages of the cryostat and be the last to reach the base temperature. Radiation shield enclosures and activated charcoal adsorbers can be designed and strategically placed to protect the sample from foreign contaminates. Also helpful, is to have a sample stage that can be heated up above 270K and cooled back down to temperature while keeping the cryocooler below 77K. This provides the ability to clean a sample in-situ. It is often overlooked that the cleanliness of the optical cryostat can be controlled by venting up with clean dry gas, rather than air. Air can contain many contaminates that should be kept out of cryostat systems, but two at the top of the list are water, and helium. Water is a culprit because of its strong adhesion to all the surfaces which soak up everything they can when a system is vented up from high vacuum. Helium can exist in high concentrations in the air where liquid helium is in use. In both cases, these contaminates can be difficult to get out of the system once in, and it is much better to not introduce them to the clean cryostat and optical surfaces at all. Nitrogen is good to vent up with because it is dry, and any contaminates that evaporate when the system warms up, readily attach to nitrogen molecules and will be completely vented out of the system if some positive flow is maintained for brief time. Keeping a little positive flow of nitrogen gas during a sample change and otherwise keeping the cryostat closed up can help keep surfaces clean between cooldowns.

Limited flexibility

Current optical cryogenic systems have limited flexibility to perform the wide range of applications that exist. This forces the researcher to decide between doing major custom modifications to a current system or purchasing a new system built for each specific technique. Presently the researcher is unable to add a variable magnetic field, high pressure, positioning stages, have the sample cooled by gas or by vacuum, and have side and top and bottom optical access for simultaneous microscopy and spectroscopy without buying multiple cryogenic systems for each specific requirement or doing multiple custom modifications. Ideally, an optical cryogenic system would utilize modular options to allow a flexible sample environment that can be adapted for a variety of experiments and grow as a researcher's needs change over time.

In summary, cryogenics adds an exciting dimension to research that can open new doors to valuable material science knowledge and also is a minefield of time-consuming challenges. The main reasons for these losses in time are:

1. Handling and scheduling the use of liquid cryogens
2. Consistent monitoring by the researcher of pump-down rate, vacuum levels, fill rates, cooling rates, temperature ramping and stabilization, and liquid cryogen levels. (aka "nursing the cryo")
3. Vacuum leaks
4. Poor thermal contact and thermal shorts
5. Clouding of cold optical surfaces or sample due to contaminates
6. Vibrations and thermal fluctuations limiting results
7. Customization necessary for a wide range of applications

The challenges listed here are a general collection, and hopefully provide a useful background in the area of low temperature research. Certainly there are more challenges than listed here, such as challenges and techniques for sample mounting, which sounds like a useful topic for another paper.

Our passion:

Montana Instruments had these challenges in mind when they designed the Cryostation optical platform. Issues including vibration, thermal stability, sample access, multiple-application flexibility and automation were addressed, and we believe we've created some tremendous new value in the field of low temperature research. Our team is passionate about innovation, and excited to be changing the rules for low temperature experiments, all so that we can create value for researchers worldwide by making them more effective at getting results.

“That’s why we exist – to make cold science simple. This is our passion.”

Luke Mauritsen, President
Montana Instruments

For more information or to make comments on this article, contact us at:
info@montanainstruments.com
(406) 551-2796