Advanced Research Systems

Basics of Closed Cycle Cryostats for Optical Spectroscopy

Optical, electrical, and magnetic property testing of materials can be done at low temperature using a Closed Cycle Refrigerator (CCR). Liquid Helium has become increasingly expensive over the past few years making the CCR's or "Dry Cryostats" a more popular choice. The new high performance CCR's are easy to set up and sub 3K performance can be achieved with the flip of a switch.

Experimental Setup

A typical Closed Cycle Cryostat setup for an optical experiment consists of the following parts: cold head, compressor, hoses, vacuum shroud, radiation shield, sample holder, windows, experimental wiring, temperature controller, temperature sensors, and vacuum system (vacuum pump, hose, filters, valves, gauge, etc).

Fig 1: Typical closed cycle cryostat

- **Cryocooler**:

The cryocooler has two main components: the cold head and the compressor. In addition, flexible high pressure helium hoses and power cables are supplied to connect the two. This is the heart of the system and is available in a range of sizes and cooling powers to suit the experiment, sample size, temperature range, sensitivity to vibrations, etc. The basic cooler, the model CS202A, will provide a temperature range of 9K to 350K. Lower temperature down to 2.8K, and high temperature up to 800K are available. The basic cooler has vibrations in the range of 10 - 15 μm at 2 or 2.4 Hz. This can easily be reduced to 3 - 5 nm using an Ultra Low Vibration Interface, Model DMX-20, available as an option and retrofitable in some cases.

Fig 2: Cold head with sample holder, radiation shield, and vacuum shroud

- **Vacuum Shroud**:

Vacuum shrouds for optical experiments are constructed from Stainless Steel or Aluminum. Stainless is more durable and less susceptible to adsorbing water vapor making it more high vacuum compatible which results in a cleaner sample environment. Aluminum is more cost effective. These shrouds are available for a wide variety of sample configurations. The standard shroud, series DMX-1 has a sample space of 1.4 inch diameter by 1.5 inches long.

A typical vacuum shroud will be mounted on the cryocooler using double O-Rings for vacuum seal. This allows the user to rotate the shroud (window with respect to the sample) without losing vacuum. The size of the optical block depends on the size of the sample space required by the sample holder (sample space inside the radiation shield). The vacuum shroud can be modified or customized for commonly available spectrometers, including a mounting arrangement to seal the spectrometer sample compartment when it is nitrogen purged.

- **Radiation Shield**:

Constructed from high purity copper (nickel plated for durability and low emissivity) or aluminum. ARS typically plates the OFHC (Oxygen Free High Conductivity) copper radiation shield with nickel or gold; this gives the surface a low thermal emissivity for better thermal performance. Nickel plating is more durable than gold and has about the same emissivity. Gold plating, without a nickel base, will diffuse into the copper over time, leaving the surface tarnished and blotchy, deteriorating the surface emissivity and its effectiveness.

- **Sample Holder**:

Sample holders are designed for sample size (large or small), thin film, bulk, or liquid samples. Sample holders can also have electrical pins for easy wiring to the sample when performing electro-optical research. Sample holders are constructed of OFHC copper for high thermal conductivity and nickel plated.

Fig 3: Optical, electrical sample holder

- **Optical Windows**:

Good window port design allows a maximum optical cone of light to the sample. This is defined by the f/# which is explained in Fig 4.

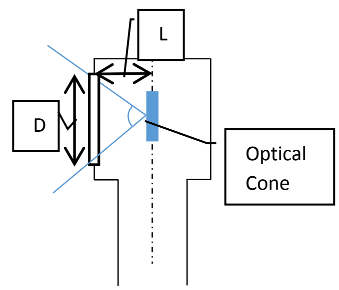

Fig 4: **f/#=L/D**. Preferably smaller than 1

Depending on the type of experiment and the wavelength of the optical beams the window material can be for Visible, UV or IR experiments. Common materials are Quartz, Sapphire, CsI, KBr, ZnSe. Several materials are used for IR or UV experiments. Transmission curves (Fig 5) are available for common windows used for Optical Spectroscopy.

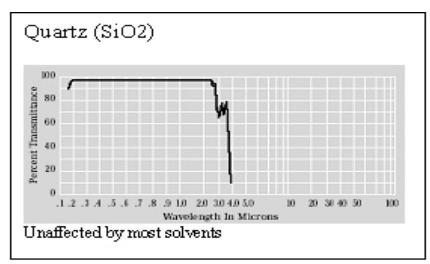

Fig 5: Quartz Transmission curve

Windows for the vacuum shroud are at room temperature and sealed with an O-Ring (Fig 6). The thickness of the window is a function of the window diameter and the material. The larger the window, the thicker it has to be for mechanical strength to cope with the vacuum forces. Thin windows are advisable for collecting weak optical signals. The windows are mounted using a stress-free mounting arrangement for minimal birefringence.

Coated windows for low reflection, brewster windows and wedge windows are available for specific experiments.

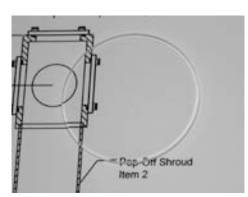

Fig 6: Quartz window

Cold windows for the radiation shield is advisable when the minimum temperature at the sample is critical. Cold windows will minimize the radiant heat through the optical hole of the shield falling on the sample. The sample temperature will be 1K lower with 4 cold windows instead of holes in the radiation shield. The drawback is the added thickness of the window material in the beam path. Cold windows will cut off IR wavelength so it is common in the UV and Vis region.

When doing transmission spectroscopy, the sample is generally mounted on the window installed on the sample holder. Indium gaskets ensure cold window temperature. The goal of this design is to have the lowest sample temperature capability which means good thermal conductivity of the window material. Use sapphire or quartz window if possible, they have the highest thermal conductivity.

Sample Mounting

Getting to the lowest possible sample temperature and measuring it correctly will depend on several factors, including:

- Proper sample holder mounting to the cryocooler:

  This is a solid-to-solid surface conductive cooling. The typical area in actual contact varies as the normal pressure, but expect to have only a 10% (A. M. Khounsary, APS. ANL) surface contact in normal cases. Using soft material between the two surfaces will help a lot. Indium is a good choice where the system is not UHV or the sample will not be heated above room temperature. If the system is UHV or has a high temperature interface, use annealed silver (annealed for softness so it can flow).

Fig 7: Sample holder with window for sample mount

- Sample mounting to sample holder:

  Good heat transfer between the sample holder and the sample is crucial for good performance. Conductive heat transfer between two surfaces is a function of pressure between the two surfaces. As the sample is generally a delicate material, high forces are not possible. The best available option is to use a grease or epoxy to glue the sample to the holder. The advantage of grease is that it is easy to remove with alcohol or acetone. Recommended greases are CryconTM grease which is a copper loaded grease (Vacuum compatible) for good thermal conductivity, or Apiezon grease. Both should be tested for optical background before they are used in the final experiment.

Fig 8: Sample mounting clips

It is also advisable to install the sensor on the sample to see what the actual temperature of the sample is. Information on how long it takes the temperature to level out and the "ΔT" with respect to the control sensor are useful points to understand. This is even more important when the user plans to control the temperature with a fast ramp.

Other materials which should be considered for sample mounting are varnish (IMI-7031 thinned with ethanol) and epoxy (Stycast).

If all this fails, use a clamp to hold the sample, but thermal characterization is even more important when the sample is dry mounted. Spring washers and clips are available with most ARS sample holders.

Other factors to consider:

- Material of the sample holder window
- Material of the sample (thermal conductivity and emissivity of the sample)
- Accuracy of the temperature sensor
- Proper installation of the sensors

Temperature Control

The cryostat must be configured with a temperature controller, temperature sensors and a heater for precise temperature control.

The controller has dual channels, one sensor is for control and the second sensor is for precise sample temperature monitoring.

The control sensor (Sensor A) is factory installed in close thermal proximity to a control heater. This allows temperature to stabilize quickly without cycling.

A second sensor (Sensor B) is provided with 4 inches of free length. This sensor should be installed on the sample holder using a spring washer to hold it down (or use grease or varnish if possible).

Accuracy of reading the sample temperature will depend on how well and where the temperature sensor is mounted and the accuracy of the sensor. For best results, to minimize experimental (user) error the sensor should be mounted on the sample. This is generally not possible, so the next best thing is to locate the sensor as close to the sample and characterize the actual sample temperature and the senor reading.

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Vacuum in Cryostat

Having a good vacuum in the cryostat is important for multiple reasons. The better the vacuum, the lower the sample temperature. The relationship between absolute pressure and conductive cooling is fairly linear at low pressure. As the surfaces are very close to the warm shroud, the heat energy conducted to the cold surfaces is proportional to the number of molecules inside the cryostat.

Fig 9: High vacuum pumping system including vacuum gauge, metal bellows hose, filters, vapor trap, manifold, and fittings

Create good vacuum by using a vacuum pump, mechanical (High Vacuum) or turbopump (Ultra High Vacuum). The radiation shield at 30K freezes all the remaining molecules on its surface, providing a localized UHV region inside the radiation shield where the sample is located.

Sample Vibrations

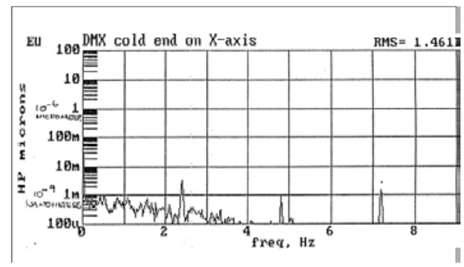


Fig 10: Ultra Low Vibration Interface (ULV) will lower the sample vibrations to 3-5 nanometers.

The vibrations coming from the cryocooler to the sample depends on the size of the cooler selected. The ARS Pneumatic drive cryocooler has very low vibrations in the range of 10 to 15 microns in amplitude. If a lower vibration is required at the sample, an Ultra Low Vibration interface can be added. The sample vibration with respect to the optical table when proper mounting techniques are followed are 3-5 nanometers of vibrations at 2 or 2.4Hz (50/60 Cycles).

Small vibrations of the sample is not always a problem. Some experimental scientists prefer the heat dissipation, from beam heating, over a larger area on the sample.

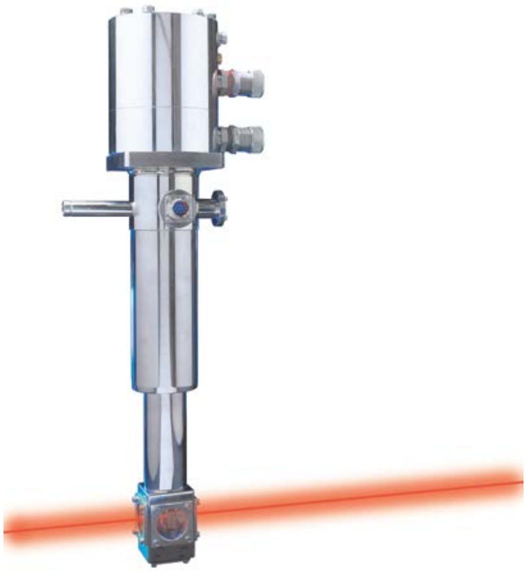
Variable (Small) sample distance from the window

Available Options and Features

- Special and custom sample holders for your application and sample geometry
- Optical vacuum shrouds are constructed from polished stainless steel or brushed aluminum
- Low Vibration – DMX-20 will reduce sample vibrations down to few nanometers
- Wide temperature range - Typically from 4K to 800K depending on the cryocooler
- Variable (Small) sample distance from the window. The sample can be brought within a few mm from the window. This is important during microscopy experiments or when a large cone of emitted light from then sample needs to be captured by the detector.
- High cooling power for large samples and fast cool down
- Sample in Vacuum or Sample in Vapor (Omniplex)
- Vacuum Pumps are available as High Vacuum or Ultra High Vacuum

Hall Probe sample holder

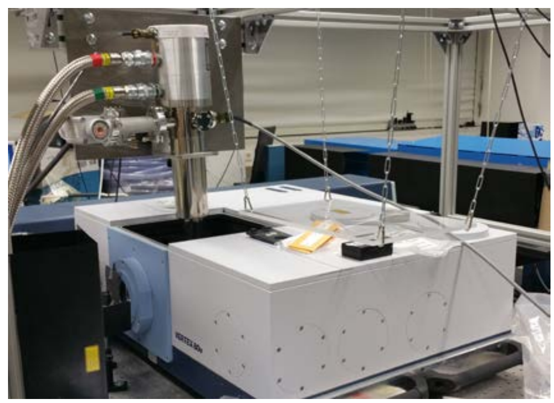
Picture Gallery



Geometry of the cryostat which holds the sample at cryogenic temperature

Temperature Range: 4K to 700K

Cryostat installed in a Horiba Spectrometer



Cryostat installed in a Bruker FTIR Spectrometer