Variable Temperature Options for Optical Experiments

MMR TECHNOLOGIES

THE WORLD'S RESOURCE FOR
VARIABLE TEMPERATURE
SOLID STATE CHARACTERIZATION

The Variable Temperature Options for Optical Experiments

MMR Technologies' Joule-Thomson refrigerators have been integrated into several different types of chambers, allowing variable temperature control during experiments where reflection, refraction, transmission and Raman measurement studies are desired.

With optical measurement experiments, there are often several critical factors, including control over the window material or over the distance between the window and the sample surface. Often the size of the sample chamber itself is a factor as to whether the optical control system can fit within the desired optical set up. The MMR optical properties measurement systems come in a wide range of sizes. Different window materials may be customized to meet any experimental requirement, and there are chambers where the distance between the sample and the window can be adjusted to suit the needs of the specific experiment. These systems include the patented Joule-Thomson temperature stages to function as both a cryogenic cooling and heating system to provide automated and high precision control of the temperature of the sample stage over a wide range of temperatures, for noise-free, vibration-free operation.

Optical setups have been created to allow the following types of optical experiments:

- Transmission
- Reflection
- Refraction
- Sideways Propagation
- Raman
- Laser Studies
- Absorption

These are flexible systems and adaptable to a wide variety of setups including optical benches, desktop microscopes, spectroscopy chambers, custom environmental chambers and vacuum chambers.

A Typical Variable Temperature Optical System

A typical variable temperature optical system includes:

- High-purity high-pressure gas (typically nitrogen or argon, for Joule-Thomson Cooling only)
- A filter/dryer apparatus (Joule-Thomson thermal cooling stages only) with high-pressure gas lines
- Thermal stage for heating and/or cooling
- A temperature controller
- Optical Vacuum Chamber
- Vacuum Pump Setup
- Computer
- The Optical Bench or Experiment you are integrating the temperature control option into

Available Temperature Ranges on Thermal Stages

When the vacuum chamber system is held under a vacuum pressure of at least 8 milliTorr, the following temperature ranges are available on the MMR Technologies' instruments:

- Room Temperature
- 70K to 580K*
- 80K to 580K
- 70K to 730K*
- 80K to 730K
- Room temperature to 730K*

* Vacuum assist Joule-Thomson thermal stages require an auxiliary vacuum pump at the thermal stage gas exhaust. These thermal stages are not available on ultra high vacuum or scanning electron microscope systems. Hot stages do not require a filter/dryer setup or high

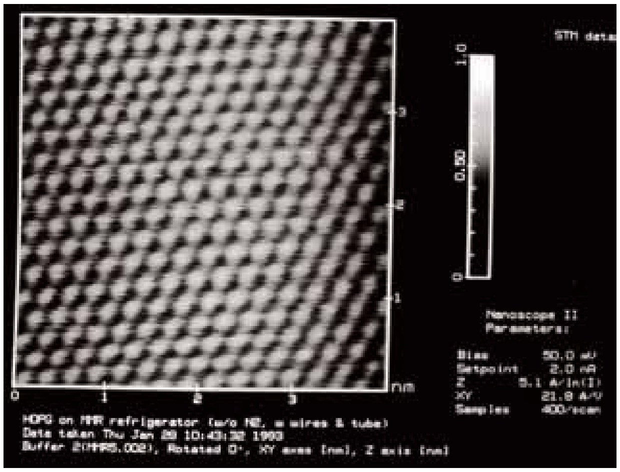
When a thermal stage is used within an ambient pressure setup, with a well controlled atmosphere, the following temperature ranges*** are available using the appropriate thermal stage setup:

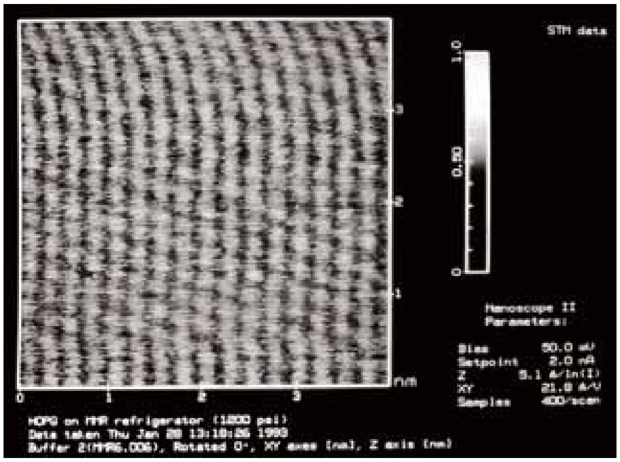
- ♦ -10 °C to 200 °C (using nitrogen gas) or -30 °C to 200 °C (using argon gas)
- ♦ -10 °C to 350 °C (using nitrogen gas) or -30 °C to 350 °C (using argon gas)
- ♦ Room temperature to 350 °C

** These are the maximum temperature ranges under ideal conditions like a glove box where there is no humidity and a dry, clean gas environment.

Noise and Vibration Levels in Thermal Stages and Optical Chambers

Experiments were performed on highly ordered pyrolytic graphite (HOPG) on a scanning tunneling microscope (STM) to evaluate



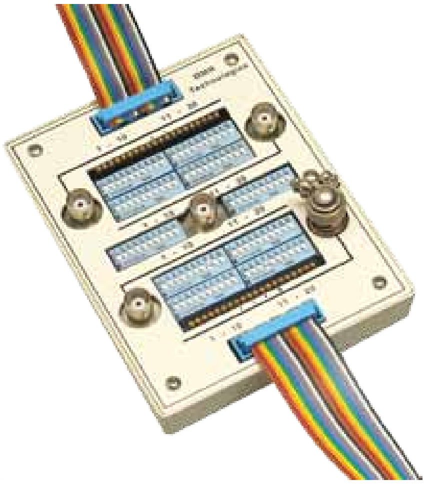


the noise and vibration levels found when using the Joule-Thomson thermal stages both with and without nitrogen gas flowing through the Joule-Thomson stage for cooling. The stages were found to be free of microphonics - providing ultra-stable, ultra-quiet operation. These stages are ideal for any vibration sensitive application.

The image* above on the left was taken with no nitrogen flow moving through the Joule-Thomson thermal stage. The image on the right was taken with high pressure nitrogen gas moving through the Joule-Thomson thermal stage, with active cooling occurring.

* These images were taken at the Department of Physics and Astronomy, Ohio University in the Clippinger Research Laboratories and were provided to MMR Technologies by Dr. Hunt.

MMR Technologies' Unique Temperature Controller



MMR Technologies' offers a unique Programmable Temperature Controller that is exclusively intended for use with our patented cryogenic cooling and thermal stage systems. This controller provides accurate temperature measurement, precise and very stable temperature control and easy-to-use data acquisition functions over the temperature range from 70K to 730K. Controlled cycling, temperature ramping and changing temperature operation under software control gives the user a valuable tool for solid state characterization studies.

The Circuit Breakout Box

The temperature controller and the unique temperature stages can work together with a Circuit Breakout Box - enabling direct electrical connections between the sample surface and extra pins available on the thermal stage.

The Filter/Dryer Setup

A filter-dryer system is necessary to remove both the water and the other condensable contaminants to ensure optimal performance of the Joule Thompson refrigerators. Failure to use one of these systems in conjunction with your cooling system will ultimately result in a loss of cooling capacity, reduction in the temperature range the microminiature refrigerator can obtain, clogging in the channels of the glass refrigerator, and ultimately damage to these channels that may not be repairable.

There are two types of filter/dryer systems available from MMR Technologies: Model F2115 and Model F2105. These systems are designed to work with the company's line of microminiature Joule-Thomson refrigerators.

There are a number of applications for the filter-dryers from MMR Technologies, including:

- ♦ Suitable for drying argon, nitrogen, hydrogen, helium, and many other gases to a Dew Point below -75 °C.
- ♦ Use of this dryer with the MMR line of microminiature refrigerators allows continuous operation of the refrigerators for up to hundreds of hours without clogging.
- ♦ The dryer may be used to provide point-of-use, dry gas at a purity level, previously attainable only with dryers of much greater cost.

*** For more detailed information on the two types of filter dryer systems, please refer to the data sheets or the technical support bulletin TSB003. These are available by contacting sales@mmr-tech.com.

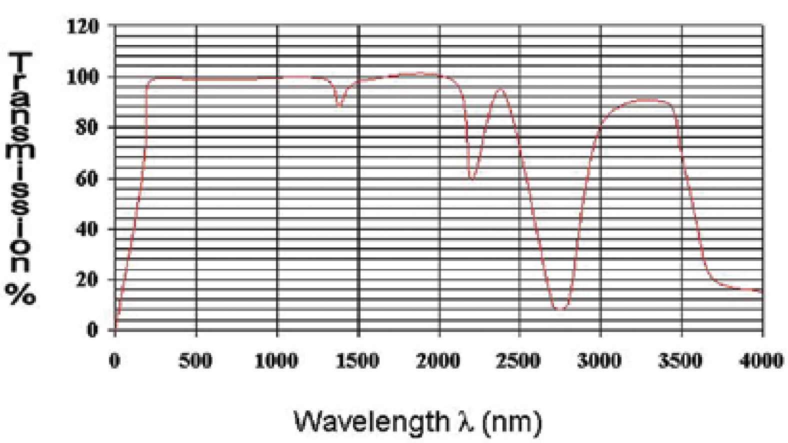
The Vacuum Chamber

There are several systems provided by MMR Technologies that allow optical property measurements to be made.

- ♦ System I Optical Properties Research Systems
- ♦ System IT Optical Properties Research Systems
- ♦ System IIB Optical Properties Research Systems
- ♦ System IIB Raman Optical Properties Research Systems
- ♦ System IIT Optical Transmission Properties Research Systems

These systems have different vacuum chambers - varying in dimensions, the number of windows, and the working distance. Standard window material is fused silica - but a limited number of alternate window materials are available as a custom order.

The transmission spectrum for fused silica 7980 can be seen to the left.



Variable Temperature Additions for Optical Experiments

Determining the correct Optical Properties Measurement System is the challenge. There are several factors that influence this, including the working distance, chamber size, and number of windows available on a chamber.

Instrument Type	Working Distance	Reflection Experiments	Refraction Experiments	Transmission Experiments	Raman Experiments
System I	6 mm or 12 mm (with inter leaf - 25 mm or 31 mm)	yes	yes	no	no
System IT		yes	yes	yes	no
System IIB	12 mm	yes	yes	no	no
System IIB Raman		yes	yes	possible*	yes
System IIT	3 or 6 mm	yes	yes	yes	possible**

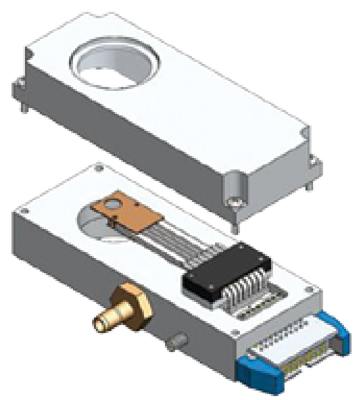
* A modification on the standard Raman chamber can give a chamber with four windows instead of three.

** A special System IIT chamber can be made with two removable windows on the top and the bottom, and two fixed-side windows.

System I and IT Optical Setups

The model K2001 and K2001T optical systems have a clamshell vacuum chamber providing the user with easy access to the sample mounted on the Joule-Thomson thermal stage. This setup facilitates making electrical connections to the sample surface. The quick release vacuum chamber lid provides fast access to the refrigerator and allows fast sample turnaround.

Both chambers have the option of a lid with a 6 mm or 12 mm working distance. An additional inter leaf can be installed between the base and the lid to add 19 mm of working distance. This can be particularly useful when electrical connections are made to the sample.



The clamshell vacuum chamber design occupies very little space and ideally mounts to a microscope translation stage. This setup has been used, without the lid, within environmental chambers, when open access to the sample surface is required, such as in atomic force microscopy experiments (AFM).

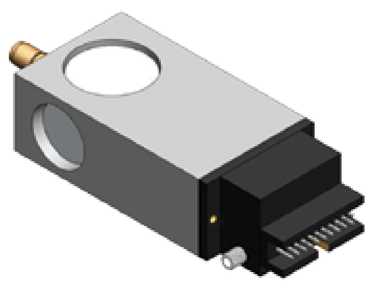
System IIB and System IIB Raman Optical Setups

The System IIB and IIB Raman vacuum chambers are of a sleeve like design, sliding over the Joule-Thomson thermal stages. The quick release vacuum chamber lid provides fast access to the refrigerator and allows fast sample turnaround. These chambers were designed for integration into an optical bench, or into a spectrometer.

The System IIB setup has a single topside window for basic experiments. The System IIB Raman setup has three windows, enabling experiments including Raman Spectroscopy, fluorescence excitation, transmission and absorption studies utilizing optical access to the sample on two axes via the three windows.

System IIT Optical Setups

The System IIB and IIB Raman vacuum chambers are of a sleeve like design, sliding over the Joule-Thomson thermal stages. The quick release vacuum chamber lid provides fast access to the refrigerator and allows fast sample turnaround. These chambers were designed for integration into an optical bench, or into a spectrometer.



The System IIT vacuum chamber provides the smallest possible working distance of all the available setups, and provides optical access to the sample via two windows on a single optical access.

An optional version of this chamber has 4 windows, providing access to the sample from any direction. There are also two removable windows on the top and bottom side of the optical four-window vacuum chamber. This enables easy exchange of windows when different material are required for different experiments. Empty removable window fixtures are available as well for you to mount your own special window material if the material you want is not available from MMR Technologies.

The System IIT optical setup is the most versatile and adaptable setup available for optical experiments.

Specifications for the Variable Temperature Optical Systems

The following are specifications for all models of the optical systems:

Operating Temperature Range:	Available between 70K and 730K (Joule-Thomson Thermal Stage)*
Sample Mounting Surface Size:	10 mm x 12 mm
Maximum Sample Weight Allowed:	No more than 5 grams
Optical Window Material	Fused Silica - other materials are possible as a special order
Electrical Connections:	Extra connection points on the thermal stage on the inside of the vacuum chamber can be used to wire bond connections within the vacuum chamber, and be accessed externally through the circuit breakout box via BNC Connectors.
Temperature Controller Requirements:	MMR's Programmable Temperature Controller
Temperature Accuracy:	< 0.5K at 80K; +/- 0.5K between 80K and 400K; < 1.5K from 400K to 730K
Temperature Stability:	+/- 0.05K
Temperature Resolution:	0.01 K
Filter/Dryer Requirements:	Either the standard filter dryer or the reversible filter dryer system if operation below room temperature is required.
Vacuum Requirement:	For operation outside of room temperature, 8 milliTorr of vacuum pressure is a minimum requirement.

System I

Type of Vacuum Chamber:	Clamshell
Dimensions of Vacuum Chamber:	1.75 in wide x 4.5 in long x 1.0 in high 4.45 cm x 11.43 cm x 2.54 cm
Weight of Vacuum Chamber with Thermal Stage:	312 grams
Working Distance:	6 mm or 12 mm*** - select one at purchase. or with optional inter leaf can have 25 mm or 31 mm
Number of Windows:	One - fused silica
Window Dimensions:	1.0 inch in diameter, 0.04 inches thick 254. mm in diameter, 1 mm thick
Types of Experiments:	Reflection, Refraction

System IT

Type of Vacuum Chamber:	Clamshell
Dimensions of Vacuum Chamber:	1.75 in wide x 4.5 in long x 1.0 in high 4.45 cm x 11.43 cm x 2.54 cm

Weight of Vacuum Chamber with Thermal Stage:	312 grams
Working Distance:	6 mm or 12 mm*** - select one at purchase.
	or with optional inter leaf can have 25 mm or 31 mm
Number of Windows:	Two - fused silica
Window Dimensions:	1.0 inch in diameter, 0.04 inches thick
	254. mm in diameter, 1 mm thick
Types of Experiments:	Reflection, Refraction, Transmission

System IIB

Type of Vacuum Chamber:	Sleeve
Dimensions of Vacuum Chamber:	1.6 in wide x 4.5 in long x 1.0 in high
	4.1 cm x 11.4 cm x 2.5 cm
Weight of Vacuum Chamber with Thermal Stage:	170 grams
Working Distance:	12 mm
Number of Windows:	One - fused silica
Window Dimensions:	1.0 inch in diameter, 0.04 inches thick
	254. mm in diameter, 1 mm thick
Types of Experiments:	Reflection, Refraction

System IIB Raman

Type of Vacuum Chamber:	Sleeve
Dimensions of Vacuum Chamber:	1.6 in wide x 4.5 in long x 1.0 in high
	4.1 cm x 11.4 cm x 2.5 cm
Weight of Vacuum Chamber with Thermal Stage:	170 grams
Working Distance:	12 mm
Number of Windows:	Three - fused silica
Window Dimensions:	1.0 inch in diameter, 0.04 inches thick - Top Window
	254. mm in diameter, 1 mm thick - Top Window
	0.75 inch in diameter, 0.04 inches thick - Side Windows
	254. mm in diameter, 1 mm thick - Side Windows
Types of Experiments:	Reflection, Refraction, Raman

System IIT

Type of Vacuum Chamber:	Sleeve
Dimensions of Vacuum Chamber:	1.2 in wide x 3.5 in long x 0.75 in high
	3.0 cm x 8.9 cm x 1.9 cm
Weight of Vacuum Chamber with Thermal Stage:	114 grams
Working Distance:	3 mm or 6 mm - depending on how the thermal stage is installed
Number of Windows:	Two - fused silica
Window Dimensions:	1.0 inch in diameter, 0.04 inches thick
	254. mm in diameter, 1 mm thick
Optional 4 window chamber:	All windows 1.0 inch in diameter, 0.04 inches thick
	254. mm in diameter, 1 mm thick
Types of Experiments:	Reflection, Refraction, Transmission

* For more information, please refer to the product data sheets for the Joule-Thomson thermal stages.

**For more detailed information on the Leakage Current, please contact sales@mmr-tech.com.

*** The default working distance shipped with the System I and IT chambers is 6 mm.

Features and Benefits

The variable temperature optical systems are noted for their unique benefits and features, making these systems easy to use and inexpensive additions to research facilities:

- Modular - you can build the systems up over time to meet your budget and experimental needs.
- Bench-top Configuration: small and compact in size
- Excellent temperature setability, stability, and reproducibility.
- Absence of mechanical, acoustic, or electrical noise.
- Fast cool down and warm up times, with frost free operation.
- Wide range of operation: 70K to 730K
- Non-magnetic electrical feedthroughs facilitate electrical connections directly to samples on the thermal stage.
- Low cost of operation: $0.50/hour
- On the Joule-Thomson stages there are no liquid cryogens to handle.
- Very low power consumption - less than 12 watts on any stage.
- Window Selection - windows provide optical access to the cooled sample. A variety of window materials are available to match operating wavelengths for a range of materials and detectors.
- Ability to have electrical connections to the sample and access these through an external circuit breakout box.

Applications

MMR Technologies Variable Temperature Optical systems are valueable addtions to optical experiments for solid state characterization. There are many possible applications for these systems, including but not limited to:

- Integration into an optics system
    - Raman Spectroscopy
    - Fluorescence Imaging
    - UV-Vis Experiments
    - IR Detectors
    - Transmission and Absortion studies
- APD's, FET's and Diode Lasers
- Biological Samples
- Material studies
- Nanotechnology
- Environmental Control Chamber Studies

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