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**_ARS Technical Note_**

# _DMX-20 OPERATION FOR MAXIMUM PERFORMANCE_

The DMX-20 can be used for Mossbauer Experiments, Optical or Non-Optical experiments where vibrations less than 5 Nanometers of displacement are required. At these vibration levels there should be no line broadening in Mossbauer experiments due to vibrations at the sample.

This Technical Note should be followed in conjunction with the technical manuals supplied with the system. The purpose of this note is to condense the set up information in one informal document. For UHV applications (DMX-20B) avoid using non UHV compatible materials.

**SYSTEM SETUP:**

If the system is new, inspect the boxes from the outside to confirm no damage was done during shipment. There are "Tip N Tell's" installed on (or inside) the compressor box, inspect this to confirm the box was not set on its side. It is important that the compressor not be set on its side during shipment. Compare the packing list with the shipment to confirm all the components ordered with the system are received. Carefully inspect the packing foam for small components which may be stuck to it. Do not discard the boxes, in the unlikely event that some components needs to be returned to ARS it must be shipped in the original boxes and packing materials.

Follow the Technical Manual procedure to unpack and set up the system as follows:

During shipment the oil in the compressor may have migrated outside the compressor sump due to careless handling. It is prudent to operate the compressor by itself for about an hour before it is connected to the Cryocooler. This allows the oil in the compressor to settle back into the sump. This is only necessary the first time.

**TEST SETUP:**

* Install the proper windows on the vacuum shroud. Follow good vacuum practice, grease the O-rings etc.
* Install the sample holder and put the second sensor supplied at the sample holder.
* Install the vacuum Shroud, (grease the O-rings).
* Install the vacuum pumping system (see the tech note on how to install the ARS supplied vacuum system).
* Start the vacuum pump and check for any leaks in the vacuum shroud, around the windows etc. If it is tight, continue to next step.
* Release vacuum and disconnect the vacuum hose from the Cryocooler.
* Place the Cryocooler in the location where the experiment is to be performed. Align the windows with the beam and set up the vibration isolation as desired. See section on "Vibration Isolation".
* In the most common set up the Cryocooler will be bolted to a stand and the DMX-20 interface will rest on the vibration isolated table.
* Install the helium flex hoses and the cables to the cold head. Including the controller cable.
* Care should be taken at this point that the support for the Cryocooler is tight and the DMX-20 is stable.
* Remove the shipping spacers slowly. (Tubing with screws mounted on the square flange). Nothing should move when the shipping spacers are being removed.
* Install a helium cylinder with a low pressure regulator (0-15 psi). (Test the pressure at the outlet. You should be able to shut it by pressing your thumb over the tube).

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Connect the ¼ inch nylon tube (supplied) from the helium cylinder to the Tee on the square flange of the Cryocooler. See Fig 1. the other side of the tee has a 1.5psi relief valve for safety.

* On the opposite side of the square flange is a valve to purge the helium through the interface. Open this valve.
* Start the helium flow from the cylinder and slowly close the valve, <u>watch the bellows</u>. If it inflates open the valve to release the pressure. The bellows should always have the normal look about it. Not inflated or deflated.
* Open and close the valve several times to displace the air in the exchange gas interface with helium. Close this valve.
* Reconnect the vacuum hose and start the vacuum pump. The vacuum level required is at 5E-3 Torr,
* Start the compressor. Make sure the cooling water is on.
* Log the Temperature Vs. Time.
* As the Cryocooler gets colder the helium in the exchange gas gets more dense, continue to supply the helium from the cylinder at 1.5 psi.
* It will take between 2-3 hours to cool the sample down to min temperature.
* Minimum temperature at the sample will be 1-2K higher than cryostat temperature at no load from optical windows or electrical wiring etc.

**NORMAL OPERATION:**

Following are some tips on good cryogenic practice:

1. Most Cryocoolers are sold with two sensors installed: one for temperature control and a second sensor with 4-6 inches free length to install on or near the sample. For test purposes only, mount the second sensor as close to the control sensor as possible to confirm accuracy of both sensors (Make sure to follow good sensor installation procedure or see TECH NOTE 112).
2. Install the sample holder with an indium gasket provided. The orientation of the sample holder can be controlled by adding an indium gasket and turning the holder to the desired position. Mark this orientation for future reference.
3. Install the radiation shield using a little vacuum grease on the threads of the mount. Note the position of the radiation shield openings and mark their position on the Cryocooler for future alignment of the sample holder. If the openings on the radiation shield are not lined up with the optics, loosen the shield until the alignment is correct. A slightly loose radiation shield is not a problem, during operation the grease will harden and keep the shield from moving.
4. When Installing the vacuum shroud over the double o-rings, make sure the o-rings are lightly (vacuum) greased.
5. When connecting the vacuum pumping system to the Cryocooler. Be careful about the backstreaming of the oil, if you purchased the vacuum system from ARS an oil trap is provided between the pump and Cryocooler. Close the pump out port valve as soon as you reach 5*E-3 Torr pressure on the gage provide with the vacuum system. If you do not have a vacuum gauge, close the vacuum valve when the Cryocooler reaches 100K.

**VIBRATION ISOLATION:**

The vibration isolation in the DMX-20 interface is unique and patented. The Cryocooler has a small vibration (10-15ì amplitude) to it. This is separated from the DMX-20 by the helium exchange gas and the soft bellows.

The typical installation is shown in Fig 3.

The only way vibrations can get to the sample is if the Cryocooler is touching the DMX-20 which means it is not centered in the DMX-20.

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After installation touch the vacuum shroud, if you feel the vibrations, move it slightly so it stops touching the cryocooler. When it is correctly centered you can not feel any vibrations with your fingers. At this point it is isolated and in the correct position.

**SAMPLE INSTALLATION**

The Cryocooler must be at room temperature when installing the sample. Remove the radiation shield and vacuum shroud. Install the sample holder using an indium gasket between the sample holder and the Cryocooler. If using an optical sample holder, indium wire should be installed in the groove between the sample window and the sample holder. On other sample holders make sure there is good thermal contact between sample and Cryocooler. In UHV systems substitute silver gasket for Indium.

The function of indium gaskets between two surfaces is to ensure contact and good thermal conductivity over the entire surface. Heat transfer between bare metal surfaces in a vacuum is otherwise very poor. Cryo con grease can also be used but it has a lower thermal conductivity than Indium.

Install the sample and the free length temperature sensor on the sample holder. Make sure that the sensor wires are properly thermally anchored (see technical note #112 for temperature sensor installation).

The sample, for optical experiments and the temperature sensor can be installed using the following methods:

| | |
|---|---|
| 1. Crycon grease (supplied with system) | A high conductivity vacuum grease with copper matrix. Available from ARS. Not UHV compatible. |
| 2. Epoxy | Conductive epoxies are available from suppliers of epoxy. Semi permanent, hard to remove. Not UHV compatible. |
| 3. Mechanical Means (clips, etc.) | This makes the installation and removal of samples easy but good thermal contact is more difficult to achieve.. UHV compatible. |
| 4. Rubber Cement | Users at Lucent Technologies, think it's great. It's easy to put on and take off, but there is insufficient data on its thermal properties. Not UHV compatible. |

Make the electrical contacts on the sample holder for electrical experiments. Minimize heat leak into the sample by properly thermally anchoring the leads at the sample holder.

**Alignment:** The sample holder can be easily aligned with the optical beam (and the position of the radiation shield openings) as noted earlier. Using two indium gaskets instead of one makes this easier. Gently hand tighten the sample holder, then rotate until it is in the proper position.

***CAUTION****:* for 450K or 800K options always support the interface while tightening the holder. These interfaces are mechanically fragile and can easily break if the sample holder is torqued (tightened) with out supporting the interface.

**RADIATION SHIELD INSTALLATION:**

After the sample has been installed and the sensor located near the sample, a radiation shield should be installed over the sample. The radiation shield serves two functions: first it allows the sample to get colder and second it allows more uniform temperature over the entire surface of the sample.

The radiation shield works most effectively when it is clean, inside and out, and cold. Both surfaces, inner and outer, must be highly reflective for minimum emissivity (low emissivity surfaces neither absorb heat nor emit heat so the sample does not have a high radiant heat load). Tarnished surface and fingerprints are black to thermal radiation and should be avoided.

Cover any unused openings on the radiation shield with aluminum foil (aluminum mylar tape is not enough because although it reflects the optical light it is not a good conductor and emits room temperature radiation onto the sample).

In non-UHV applications it may be acceptable to use vacuum grease on the threads of the radiation shield to get better thermal contact. This will also ensure that the radiation shield does not freeze onto the radiation shield mount.

**Alignment:** The radiation shield is easily aligned with the sample and the optical beam in optical experiments. To do this, put vacuum grease (or Crycon grease) on the threads and tighten the radiation shield all the way. Now rotate it back to the proper optical orientation desired. The grease will get hard during cooldown and the shield will not loosen or move during long term experiments.

**VACUUM SHROUD INSTALLATION:**

Install the vacuum shroud making sure the o-rings are lightly greased. Again, make sure the inner and outer surfaces are clean. Alcohol or freon is commonly used to clean these surfaces.

Pump the vacuum shroud to a pressure between 5x10E-2 to 1x10E-3 Torr and start cool down. The vacuum shroud can be rotated with respect to the sample anytime during operation – this feature can be used to an advantage if multiple sample testing is desired.

**WINDOWS:**

Vacuum shroud windows can be made from any material that can withstand one atmospheric pressure difference. Transmission curves are available from ARS for most window materials.

During installation, put a thin film of vacuum grease on the O-Rings and install the window and the low stress frame with the screws. Make it finger tight. The vacuum will pull the windows in during operation. This uniform force prevents the uneven stress inherent in hand tightened windows. The frame is only for support when there is no vacuum in the cryostat. During operation the screws will be loose and the window pressed against the O-Ring. The frame is not designed to touch the shroud there is a gap, the width of which depends on the window thickness provided.

The sample should be installed on a sample holder window made of the same material as the shroud windows for transmission experiments. These windows should not be more than 1 mm thick, since they require no structural integrity.

***CAUTION:*** Avoid possible injury from imploding windows. This can result if the windows are stressed, cracked, or cooled on one side, (A possibility in the event of a helium cold leak inside the shroud). Cold leaks are rare.

**DMX-20B FOR UHV APPLICATIONS:**

This technical note is valid for the UHV application but care must be taken not to have non UHV compatible materials on the UHV side of the cryostat.

**HIGH TEMPERATURE INTERFACE, 450K AND 800K:**

Follow all the guidelines for the high temperature interfaces. This includes thermometry and the mechanical considerations to avoid damaging the interface.

**TROUBLESHOOTING:**

Troubleshooting of problems associated with the mechanical or electrical aspects of the compressor or Cryocooler are covered in the appropriate manual. This note covers troubleshooting problems associated with the experiment itself.

**1. Cryostat will not reach minimum temperature:**

Or, Sample temperature is higher than Cryocooler temperature

Possible Reasons are:

1) T1 or T2 sensor calibration is not correct in the controller
2) Controller circuitry may have malfunctioned
3) Sensor T2 not installed properly
4) Sensor wires not properly thermally anchored to the first stage of the Cryocooler. (This is the most common problem)
5) Thermal boundaries not in good contact, between the Cryocooler and sample holder or between the sample window and holder or between the sample and the sample window.

Thermometry is generally the cause of this problem. Interchange T1 and T2 by switching the plugs at the back of the controller. This will isolate any problems with the sensors or the controller. If both channels read the same temperature difference when the sensors are switched it is probably not a controller problem. If T2 (Sample Temperature) is still higher than T1 and the temperature difference is only a few degrees, look for a problem with contacts at the thermal boundaries between sample holder and Cryocooler (Tighten sample holder and sensor (Visual check will identify a problem)). If sensor wires are not heat sunk properly, a temperature rise of up to 15K is possible.

**2. Shroud sweats during cooldown:**

This can happen if the vacuum in the shroud is not low enough or the humidity in the room is exceptionally high. By itself it is not a problem and dries up once the Cryocooler is cold (Vacuum level inside the shroud is better when the system is cold). If the Cryocooler temperature starts hanging up check all the O-Rings on the skirt and windows; they should be in good condition and lightly greased (the wet look). Check all connections to the vacuum pump.

**3. Windows on the shroud are breaking:**

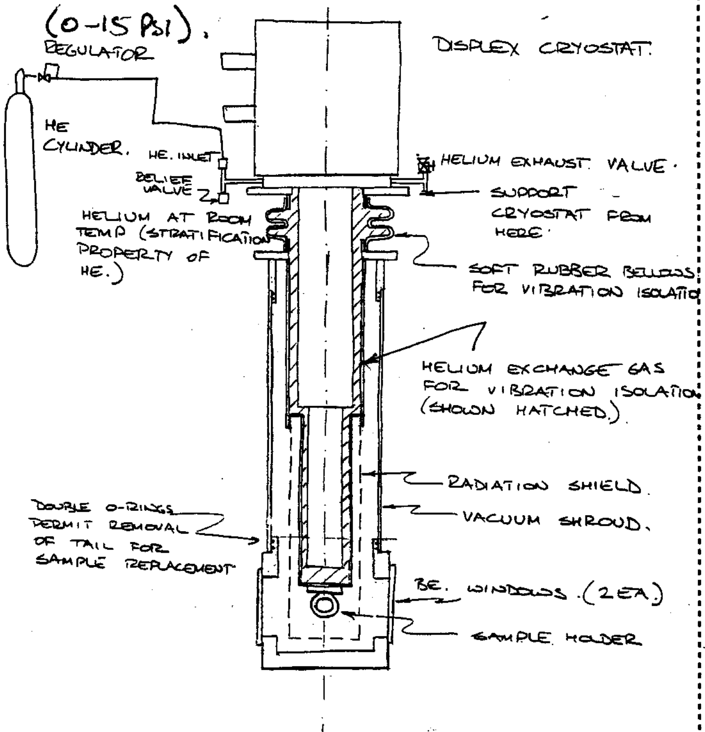
Window breakage is generally due to uneven tightening of the holding screws (these screws are merely to hold the window in place while the system is not under vacuum). When a vacuum is pulled the windows are evenly pressed into place by the pressure difference. As a test, during operation the screws should become loose.

In rare cases a small helium leak in the vacuum shroud can cool the window from the inside while it is warm on the outer surface; this can cause the windows to implode when the system gets cold.

**4. Temperature cycling:**

Check the PID settings on the controller, check the manual for optimization. After some experience these settings can be optimized to achieve better stability and response time.

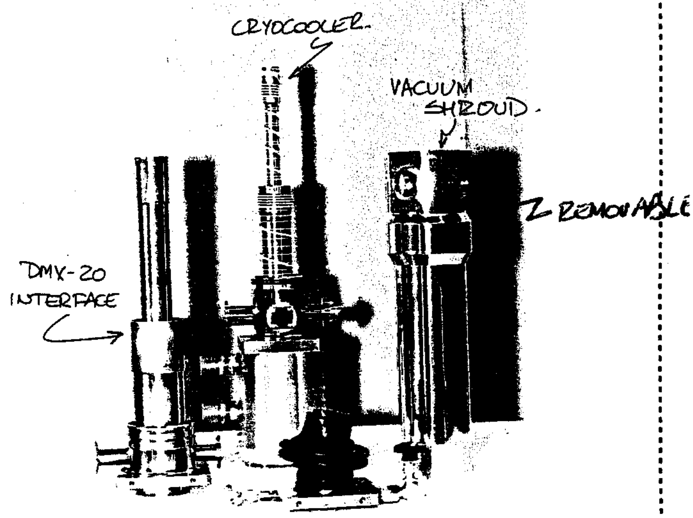
Poor thermal contact between control sensor and heater can also set up temperature cycling. They should be placed thermally adjacent to one another for good thermal stability. It is important to understand that between 4 and 20K the specific heat of all materials is very low. Small amounts of heat input can change the temperature of the sample holder appreciably. The high end temperature controllers will control better at these temperatures.




LOW VIBRATION CLOSED CYCLE SYSTEM
DMX-20 INTERFACE

FIG 1

COMPONENTS OF DMX-20.




FLAT FLANGE WITH BELLOWS.

FIG 2.

ADVANCED RESEARCH SYSTEMS, INC. SK1.0521
SUBJECT CLOSED CYCLE CRYOSTAT - LOW VIBRATION MOUNTING STAND

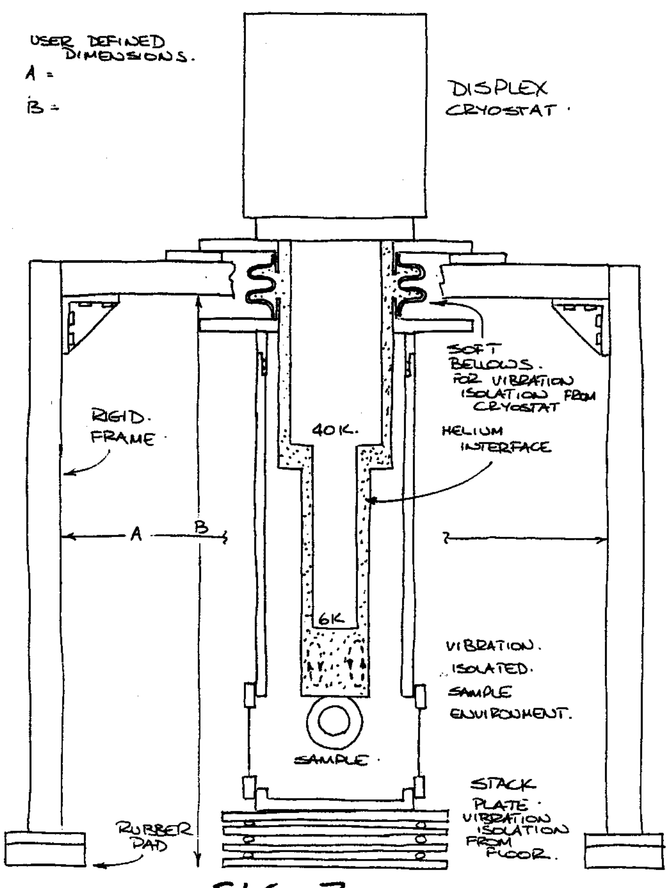



FIG 3.