# DISCOVERY XENON FLASH
DXF-200


# Getting Started Guide

Revision A  Issued September 2020

## Notice

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TA Instruments — Waters LLC
159 Lukens Drive
New Castle, DE 19720

# Introduction

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## Notes, Cautions, and Warnings

This manual uses NOTES, CAUTIONS, and WARNINGS to emphasize important and critical instructions. In the body of the manual these may be found in the shaded box on the outside of the page.

**NOTE:** A NOTE highlights important information about equipment or procedures.

**CAUTION: A CAUTION emphasizes a procedure that may damage equipment or cause loss of data if not followed correctly.**

**MISE EN GARDE: UNE MISE EN GARDE met l'accent sur une procédure susceptible d'endommager l'équipement ou de causer la perte des données si elle n'est pas correctement suivie.**

**A WARNING indicates a procedure that may be hazardous to the operator or to the environment if not followed correctly.**

**Un AVERTISSEMENT indique une procédure qui peut être dangereuse pour l'opérateur ou l'environnement si elle n'est pas correctement suivie.**

Please heed the warning labels and take the necessary precautions when dealing with those parts of the instrument. The *DXF Getting Started Guide* contains cautions and warnings that must be followed for your own safety.

## Safety

### *Instrument Symbols*

The following label is displayed on the instrument for your protection:

| Symbol | Explanation |
|---|---|
|  | This symbol indicates that a hand crush hazard is present. Take care when placing the top module onto the bottom module.<br><br>Ce symbole indique la présence d'un risque de broyage de main. Prenez soin de placer le module supérieur sur le module inférieur. |
|  | This symbol indicates that a hot surface may be present. Take care not to touch this area or allow any material that may melt or burn to come in contact with this hot surface.<br><br>Ce symbole indique la présence possible d'une surface chaude. Prenez soin de ne pas toucher cette zone ou de laisser un matériau susceptible de fondre ou de brûler entrer en contact avec cette surface chaude. |

Please heed the warning labels and take the necessary precautions when dealing with these areas. This *Getting Started Guide* contains cautions and warnings that must be followed for your own safety.

## *Warnings*

**WARNING: The operator of this instrument is advised that if the equipment is used in a manner not specified in this manual, the protection provided by the equipment may be impaired.**

**AVERTISSEMENT: L'utilisateur de cet instrument est prévenu qu'en cas d'utilisation contraire aux indications du manuel, la protection offerte par l'équipement peut être altérée.**

**WARNING: Always unplug the instrument before performing any maintenance.**

**AVERTISSEMENT: Débranchez toujours l'instrument avant de procéder à la maintenance.**

## *Electrical Safety*

**DANGER: This pulse source operates from a high voltage power supply. Do not override the safety interlocks as they prevent contact with potentially lethal voltages.**

**DANGER: Cette source d'impulsion fonctionne à partir d'une alimentation électrique haute tension. N'annulez pas les dispositifs de sécurité car ils empêchent tout contact avec les tensions potentiellement mortelles.**

## *Liquid Nitrogen Safety*

The furnace uses the cryogenic (low-temperature) agent, liquid nitrogen, for cooling. Because of its low temperature (–196°C [–321°F)]), liquid nitrogen may burn the skin. When you work with liquid nitrogen, use the following precautions:

**NOTE**: Please adhere to your company's safety guidelines for handling liquid nitrogen.

**WARNING: Liquid nitrogen boils rapidly when exposed to room temperature. Be certain that areas where liquid nitrogen is used are well ventilated to prevent displacement of oxygen in the air.**

**AVERTISSEMENT: L'azote liquide bout rapidement lorsqu'il est exposé à la température ambiante. Assurez-vous que les zones où l'azote liquide est utilisé sont bien aérées pour éviter le déplacement de l'oxygène dans l'air.**

Wear goggles or a face shield, thermally insulated gloves large enough to be removed easily, and a rubber apron. For extra protection, wear high-topped, sturdy shoes, and leave your pant legs outside the tops.

## *Thermal Safety*

**DANGER: Source instrument surfaces can be hot enough to cause discomfort when in contact with the skin during a sample run. If you are conducting a subambient test, cold could also cause injury. After running any type of experiment, you must allow the DXF system to return to near room temperature before you touch the inner cell surfaces. Always use the available software monitor to display temperature even after the termination of the test cycle.**

**DANGER: Les surfaces de l'instrument source peuvent être assez chaudes pour provoquer un malaise au contact de la peau pendant l'analyse d'un échantillon. Si vous effectuez un essai à basse température, le froid peut également provoquer des blessures. Après avoir effectué un type d'expérience quelconque, vous devez laisser le système DXF revenir à la température quasi ambiante avant de toucher les surfaces internes de la cellule. Utilisez toujours le moniteur logiciel disponible pour afficher la température même après la fin du cycle d'essai.**

**WARNING: In the event of a power failure, do NOT open the furnace.**

**AVERTISSEMENT: En cas de coupure de courant, N'ouvrez PAS le four.**

If the power has not come back on after a power failure, do NOT open the furnace. The temperature controllers will not be displaying a temperature so you will not know what the temperature is inside the furnace.

The test is over once a power failure occurs. This is no recovery method to restart the test at that point, but all data is saved up until the power failure occurs.

Do not press any buttons on the front panel after a power failure, just start a test normally.

## *Chemical Safety*

**WARNING: Do not use hydrogen, oxygen, or any other explosive gas in the DXF system. Only inert gas, such as nitrogen or argon, should be used.**

**AVERTISSEMENT: N'utilisez pas d'hydrogène, d'oxygène ou tout gaz explosif dans le système DXF. Seuls les gaz inertes comme l'azote ou l'argon doivent être utilisés.**

## *Lifting the Instrument*

**WARNING: Use two people to lift and/or carry the instrument. The instrument is too heavy for one person to handle safely.**

**AVERTISSEMENT: Demandez à deux personnes de soulever et/ou de porter l'instrument. L'instrument est trop lourd pour qu'une seule personne le manipule en toute sécurité.**

# Table of Contents

# *Chapter 1:*

# Introducing the Discovery Xenon Flash

## *Overview*

Thermal diffusivity (α) is the thermophysical property defined as a ratio of thermal conductivity and volumetric heat capacity. The thermal diffusivity is measured in $cm^2/s$ (SI units). The most popular method used for measuring thermal diffusivity is the flash method. It has the advantage of being fast while providing values with excellent accuracy and reproducibility. The flash diffusivity method involves uniform irradiation of a small, disc-shaped specimen over its front face with a very short pulse of energy. The time-temperature history of the rear face is recorded through high-speed data acquisition from a solid-state pins sensor with very fast thermal response, and thermal diffusivity is determined from the time-dependent thermogram of the rear face. Thermal conductivity can be calculated as a product of the thermal diffusivity, the specific heat, and the density of the material. A Discovery Xenon Flash (DXF) system automatically determines the thermal conductivity using the measured (or separately entered) specific heat capacity and thermal diffusivity, with separately-entered density data.

The DXF system is comprised of a DXF Pulse Source, Environmental Module, Solid State (Pins) Detector Assembly, and associated software.


**Figure 1** DXF system with EM-200 and Solid State (Pins) Detector Assembly.

Your controller is a computer that performs the following functions:

- Provides an interface between you and the analysis instrument.
- Enables you to set up experiments and enter necessary information.
- Stores experimental data.
- Allows data analysis of results.

# *DXF System Components*

A DXF system has three major components: the DXF Pulse Source module, the Environmental Module with Detector Assembly, and the controller.

## Pulse Source Module

The Discovery Xenon Flash (DXF) covers the most commonly needed cryogenic to 200°C temperatures with a range of Environmental Modules, and employs a High Speed Xenon-Pulse Delivery Source (HSXD). The reflective optic configuration effectively harnesses the power of a Xenon flash tube, and, with the aid of proprietary wave guides, delivers it to the sample inside the Environmental Module. The DXF produces a maximum pulse width of 400 to 600 µs while uniformly concentrating sufficient power from the flash source directly on the entire face of the sample. This optimized energy gathering scheme and the broad light spectrum allows samples as large as 25 mm in diameter to be illuminated with sufficient energy.


**Figure 2** DXF Pulse Source Module.

**Table 1: DXF Front Panel LED Indicators - Flashlamp**

| Indicator | Function |
|---|---|
| BYPASS | This function is not used. |
| INTERNAL INTERLOCK 1 | This internal interlock checks to make sure the metal covers are in place around the flashlamp assembly. When the covers are closed the INTERNAL INTERLOCK 1 LED changes from red to green. When all interlocks are green, the flashlamp is safe to fire. |

**Table 1: DXF Front Panel LED Indicators - Flashlamp**

| Indicator | Function |
|---|---|
| INTERNAL INTERLOCK 2 | This internal interlock checks to make sure the metal covers are in place for the high voltage power section of the instrument. When the covers are closed the INTERNAL INTERLOCK 2 LED changes from red to green. When all interlocks are green, the flashlamp is safe to fire. |
| UNLABELED INTERLOCK 3 | This function is not used. This indicator will remain green. |
| UNLABELED INTERLOCK 4 | When the internal interlocks for the optical path are enabled and the operating software enables it, the UNLABELED INTERLOCK 2 LED changes from red to green. When all interlocks are green, the flashlamp is safe to fire. |
| POWER | When the flashlamp 12 VDC power supply is ON, the green POWER LED lights up. |
| INTERLOCK | This function is not used. |
| CHARGE | The flashlamp controller has 4 control states. The CHARGE indicator shows when the controller is charging the main capacitors with the programmed flash voltage. |
| ARM | The flashlamp controller has 4 control states. The ARM indicator shows when the controller has charged the fire capacitor and is able to fire the flashlamp. |
| FIRE | The flashlamp controller has 4 control states. The FIRE indicator shows when the controller has fired the flashlamp. |
| DUMP | The flashlamp controller has 4 control states. The DUMP indicator shows when the controller has dumped all the charge on the main capacitor and disabled the fire circuit. |

**Table 2: DXF Front Panel LED Indicators - Temperature Displays**

| Indicator | Function |
|---|---|
| FURNACE TEMPERATURE°C | Indicates the current temperature of the furnace. There are several indicators just to the left of the temperature display that may light up during normal operation. |
| SPECIMEN TEMPERATURE °C | Indicates the current temperature of the sample. There are several indicators just to the left of the temperature display that may light up during normal operation. |
| OVER TEMP | When the temperature controllers detect a temperature outside the acceptable range, an alarm is triggered, and the OVER TEMP red LED will light up. If the interlock ON LED is enabled, it will be disabled. |
| OPTICAL | When the internal interlocks for the optical path are enabled and the operating software enables it, the OPTICAL LED lights up. When the OPTICAL LED is ON, the flashlamp is enabled to fire. |
| ALARM | This indicator shows the status of the External Alarm input. This unit does not have an External Alarm Input and this LED will not be on. |
| POWER | When the internal 12 VDC power supply is ON, the yellow POWER LED lights up. |
| DET. POWER | When the operating software is ready to start a test, a separate DC power supply is connected to the Detector Electronics and the DET POWER LED lights up. |

**Table 3: DXF Front Panel Switches**

| Indicator | Function |
|---|---|
| HARDWARE ENABLE | When prompted by the operating software, this switch is pressed to enable the operation of the electronic hardware. If all the electronic checks are correct, the switch's integrated green LED will light up. |
| ON INTERLOCK | When prompted by the operating software, this switch is pressed to enable the temperature control of the furnace. If all the electronic checks are correct, the switch's integrated green LED will light up. |
| OFF INTERLOCK | At any time, this switch is pressed to disable the temperature control of the furnace. The switch's integrated red LED does not light up. When the switch is pressed, if the ON interlock LED is enabled, it will be disabled. |
| INDEX ON | This switch is not used. |

## Environmental Modules and Solid State (Pins) Detector Assembly

The Environmental Module includes a cooling block with heater cartridge-resistance furnace, providing temperature control of the sample operation in air or inert gas. The EM also includes a two-sample holder, supporting a variety of sample sizes. Adapters are available for smaller cylindrical and/or square samples. The module includes a Solid State (Pins) Detector.

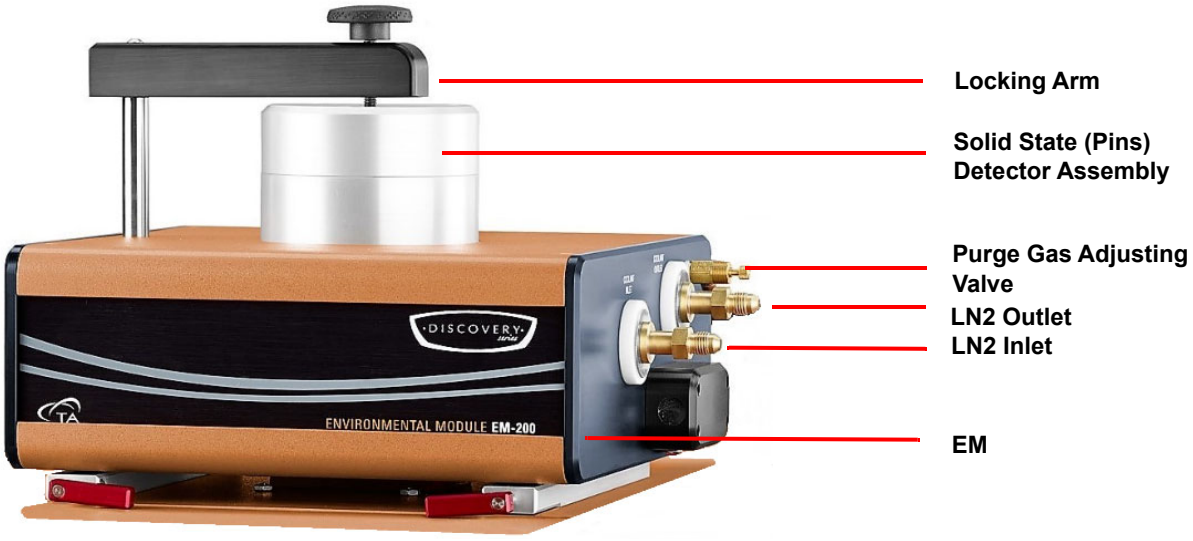


**Figure 3** EM and Solid State (Pins) Detector Assembly.

# *Instrument Specifications*

The tables found below contain the technical specifications for the DXF and EM.

**Table 4: DXF Technical Specifications**

| Item/Area | Specifications |
|---|---|
| Dimensions | Depth 66.04 cm (26 in)<br>Width 53.34 cm (21 in)<br>Height 40.64 cm (16 in) |
| Weight | 44 kg (96 lbs) |
| Power[a] | System supply voltage: 198–242 VAC (rated for 10A)<br>50/60 Hz. |
| Operating environmental conditions | Temperature: 15 to 35°C<br>Relative humidity: 5 to 80% (non-condensing)<br>Installation Category II<br>Pollution Degree 2<br>Maximum altitude: 2000 m |
| Pulse Source | High speed Xenon module |

a. This instrument is supplied with a power cord 2.4 m (7.75 ft) long, rated for 10A and 250V and a USA NEMA 6-15P style plug. The TA Representative installing the instrument will provide the appropriate power cable for the geographical location where the instrument is being installed. Connect the instrument, computer, and monitor to wall outlets on the same circuit and make sure that the mains assigned do not also supply power to noise generating equipment nearby, such as motors, welders, transformers, etc.

**Table 5: EM Technical Specifications**

| Item/Area | EM-200 |
|---|---|
| Thermocouple Measurement | 3 wire PT 100Ω RTD ± 0.15% |
| Thermal Diffusivity | Accuracy** ± 4% (worst case) |
| Measurement | Repeatability** ± 2% (worst case) |
| Temperature Range | -150 to 200°C |
| Atmosphere | Air, inert gas |
| Number of Specimens | 2 |
| Sample Dimension | 12.7/25.4 mm (d) x up to 6 mm (t) |
| Sample Shape | Round disk, square plate (with adapters) |
| Solid State Detector Assembly | 8 and 10 mm diameter Intrinsic thermocouples (8, 10, 12.7 mm length Bismuth telluride [$B_{i2}T_{e3}$] pins) |

**Using ideal, well-defined, well-behaved 12-mm diameter samples of known properties

**CAUTION: Dewar $LN_2$ tank minimum 50 litters capacity. Maximum 22 psi (1.5 bar) supply pressure. Pressure exceeding 22 psi will damage the instrument.**

**MISE EN GARDE: Réservoir Dewar LN2 d'une capacité minimale de 50 litres. Pression d'alimentation maximale de 22 psi (1,5 bar). Une pression supérieure à 22 psi endommagera l'instrument.**

# *Chapter 2:*

# Installing the DXF System

## *Unpacking/Repacking*

You may wish to retain all the shipping hardware, the plywood, and boxes from the instrument in the event you wish to repack and ship your instrument.

## *Installing the System*

Before shipment, the DXF is inspected both electrically and mechanically so that it is ready for operation upon proper installation. Only limited instructions are given in this manual; consult the Help documentation for additional information. Installation involves the following procedures:

- Inspecting the system for shipping damage and missing parts
- Choosing a location for instrument installation
- Connecting the Environmental Module to Pulse Source Module
- Setting up system communication with the controller
- Connecting the utilities

It is strongly recommended that you have your DXF system installed by a TA Instruments Service Representative; call for an installation appointment when you receive your instrument.

**CAUTION: To avoid mistakes, read this entire chapter before you begin installation.**

**MISE EN GARDE: Pour éviter de commettre des erreurs, lisez tout le chapitre avant de commencer l'installation.**

### Inspecting the System

When you receive your DXF system, look over the instrument and shipping container carefully for signs of shipping damage, and check the parts received against the enclosed shipping list.

- If the instrument is damaged, notify the carrier and TA Instruments immediately.
- If the instrument is intact but parts are missing, contact TA Instruments.

## Choosing a Location

Because of the sensitivity of DXF experiments, it is important to choose a location for the instrument using the following guidelines. The DXF system should be:

### *In*

- A temperature and humidity controlled area.
- A clean, vibration-free environment.
- An area with ample working and ventilation space.

### *On*

- A stable, non-flammable work surface.

### *Near*

- A power outlet for the specific voltage of the instrument.
- The controller (computer).
- Compressed lab air and purge gas supplies with suitable regulators and filtering where needed.

### *Away from*

- Dusty environments.
- Exposure to direct sunlight.
- Direct air drafts (fans, room air ducts).
- Poorly ventilated areas.
- Electrically noisy areas or areas prone to mechanical vibrations.

**NOTE**: Do not place equipment against walls or cabinets that might impede air flow. Leave at least 7.5 cm (3 in) clearance between the back of the instrument and any objects

**WARNING: For safety, position the equipment in a manner that allows access to the power cord for emergency disconnection.**

**AVERTISSEMENT: Par mesure de sécurité, placez l'équipement de sorte qu'il permette d'accéder facilement au cordon d'alimentation en cas de débranchement d'urgence.**

**WARNING: Protect power and communication cable paths. Do not create tripping hazards by laying the cables across access ways.**

**AVERTISSEMENT: Protégez les chemins de câble électriques et de câbles de télécommunication. Ne créez pas de risques de déclenchement en posant des câbles sur les voies d'accès.**

**CAUTION: An independent heavy GROUND wire must be provided through the power hook up. Improper grounding may cause severe damage for which the supplier will not accept responsibility.**

**All power strips must be fully grounded and carry the ground through to the sockets into which the computer is plugged.**

**MISE EN GARDE: Un fil de masse lourd et indépendant doit être fourni via le raccordement électrique. Une mise à la terre incorrecte peut causer de graves dommages pour lesquels le fournisseur décline toute responsabilité. Toutes les barrettes d'alimentation doivent être entièrement mises à la terre et acheminer la terre jusqu'aux prises dans lesquelles l'ordinateur est branché.**

## Moving the Instrument

**WARNING: Use two people to lift and/or carry the instrument. The instrument is too heavy for one person to handle safely.**

**AVERTISSEMENT: Demandez à deux personnes de soulever et/ou de porter l'instrument. L'instrument est trop lourd pour qu'une seule personne le manipule en toute sécurité.**

Lifting the DXF should always be done with two people.

1 With each person standing on either side of the DXF, place hands on the outside bottom corners of the DXF and lift upwards, keeping the module in an upright position.

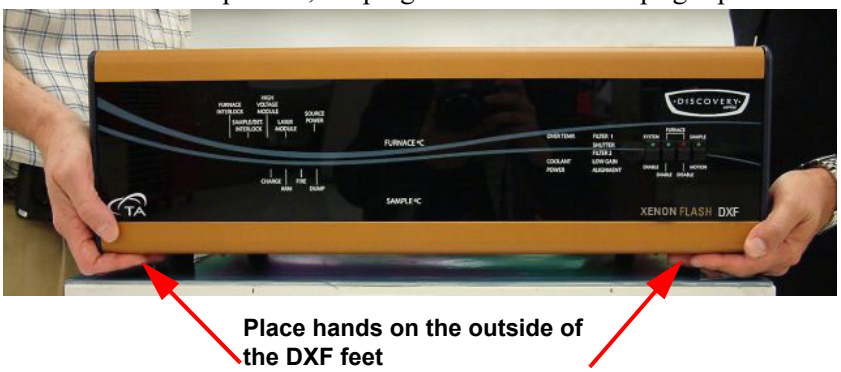


**Figure 4** Lifting the DXF.

## Connecting the Environmental Module to the Pulse Source Module

**1** Before making any connections, make sure the DXF is level.

**2** Place the reflector in the location shown in the image below, and then install the sealing foam.

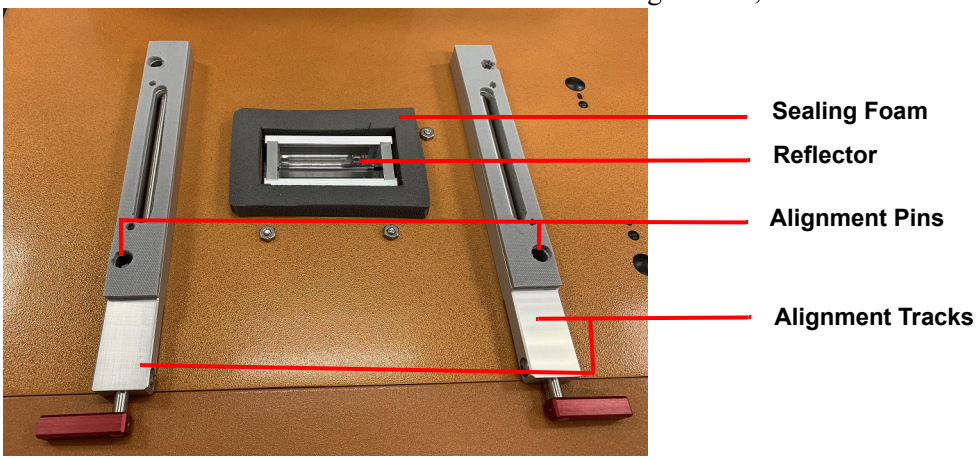


**Figure 5** Reflector and sealing foam.

**3** Place the Environmental Module on top of the DXF so that they are stacked; make sure to align the 4 pins on the bottom of the furnace with the corresponding holes on the alignment tracks on the top of the DXF.


**Figure 6** Pins on the bottom of the Environmental Module.

**4** Lock the EM into place by rotating the red locking mechanisms 180 degrees to face inward.

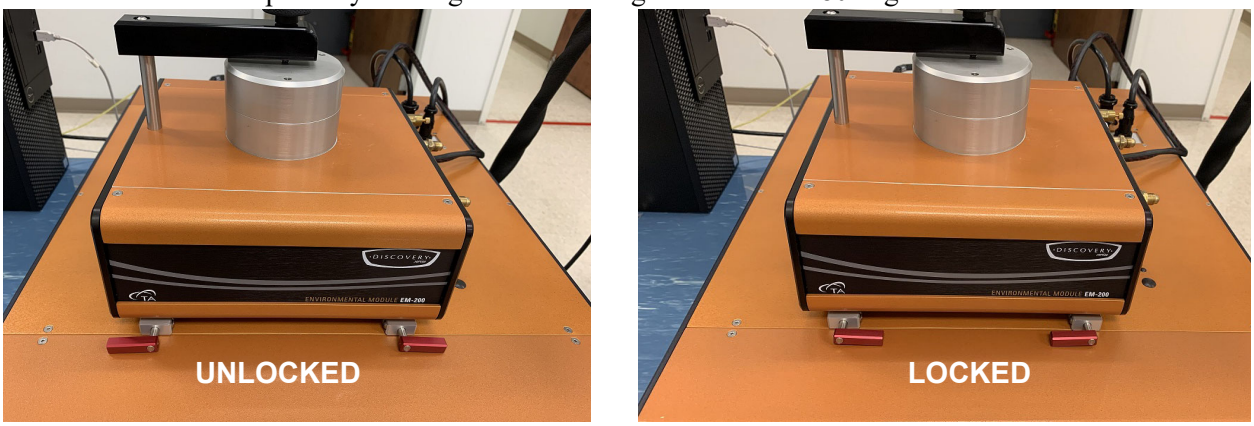


**Figure 7** Lock the EM onto the DXF.

Allow an additional 30 cm (12 in) clearance to the right of the instrument for connecting the $LN_2$ tank.

To connect the Environmental Module, you will need access to the DXF instrument back panel and the Environmental Module back panel.

**NOTE**: Connect all cables before connecting the power cords to outlets. Tighten the screws on all cables.

**CAUTION: Whenever plugging or unplugging power cords, handle them by the plugs, not by the cords.**

**MISE EN GARDE: Chaque fois que vous branchez ou débranchez les cordons d'alimentation, tenez-les par les fiches et non par les cordons.**

**WARNING: Protect power and communications cable paths. Do not create tripping hazards by laying the cables across access ways.**

**AVERTISSEMENT: Protégez les chemins de câble électriques et de câbles de télécommunication. Ne créez pas de risques de déclenchement en posant des câbles sur les voies d'accès.**

**CAUTION: An independent heavy GROUND wire must be provided through the power hook up. Improper grounding may cause severe damage for which the supplier will not accept responsibility. All power strips must be fully grounded and carry the ground through to the sockets into which the computer is plugged.**

**MISE EN GARDE: Un fil de masse lourd et indépendant doit être fourni via le raccordement électrique. Une mise à la terre incorrecte peut causer de graves dommages pour lesquels le fournisseur décline toute responsabilité. Toutes les barrettes d'alimentation doivent être entièrement mises à la terre et acheminer la terre jusqu'aux prises dans lesquelles l'ordinateur est branché.**

## *DXF Back Panel*

The table below provides a description of the function of each port/cable on the DXF back panel. Refer to Figure 8 for an illustration of rear connections. Ports not described and not labeled are not used.

**Table 6: DXF Back Panel Ports and Functions**

| Port | Function |
|---|---|
| J101 Port | Connects the Beam Mapping Board to the Data Acquisition Board |
| J102 Port | Connects the Pins Amplifier Board to the Data Acquisition Board, providing communication between the DXF and the Solid State Detector Assembly |
| USB Cable | Provides flash control |
| USB1 Cable | Provides data acquisition |
| TEMPERATURE CONTROLLER COMMUNICATION Cable | Provides communication between the temperature controllers and the controller |
| SYSTEM POWER | SYSTEM POWER PLUG provides power to the EM and SYSTEM POWER SWITCH powers the DXF off (**O**) / on (**I**) |
| LED indicators | 6 green LED indicators indicate system readiness. From left to right:<br>**1** +12 Volts Analog Power Supply<br>**2** +12 Volts Analog Power Supply<br>**3** +12 Volts Digital Power Supply<br>**4** +12 Volts Communications Power Supply<br>**5** Filter Output 2 – not used on this instrument<br>**6** Filter Output 1 – not used on this instrument |

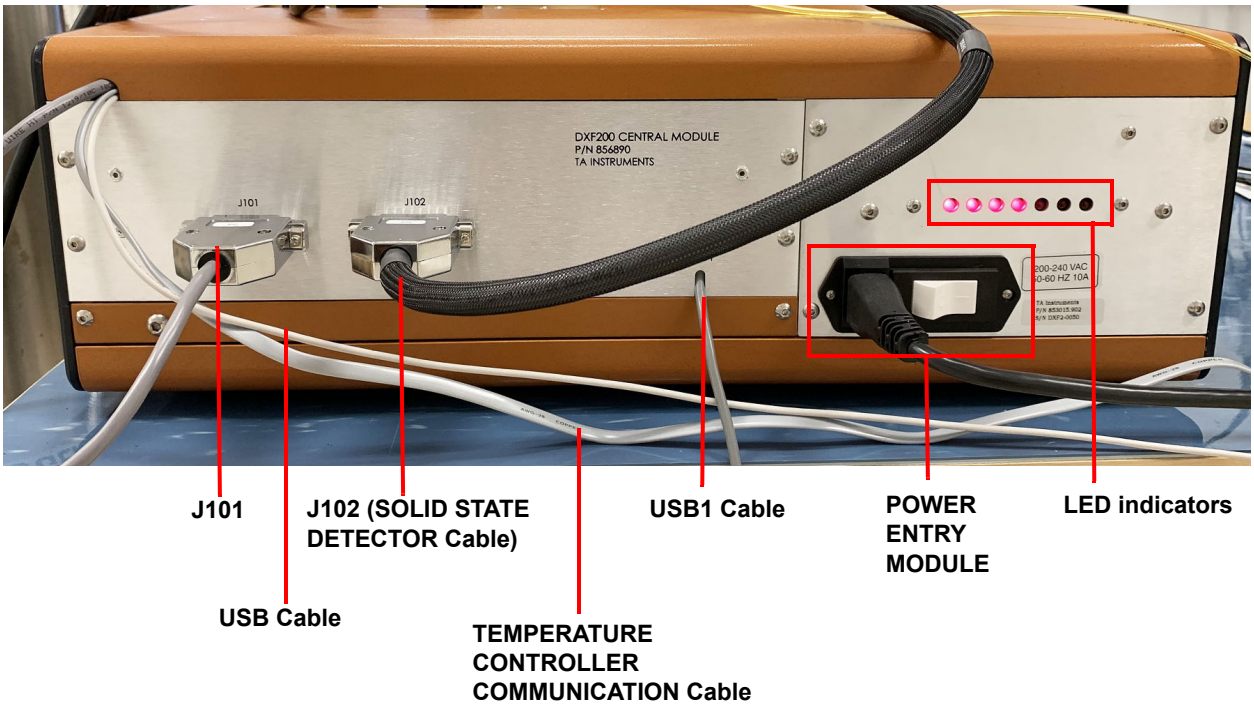


**Figure 8** DXF back panel connections.

## *DXF Top Panel*

The DXF top panel has three ports; the table below provides a description of the function of each port. Refer to Figure 7 for an illustration of rear connections.

| Port | Function |
|---|---|
| TEMPERATURE SENSORS | Connect Furnace and Sample RTD wires to Controls |
| HEATER POWER | Connect Heater Wires to Controls |
| CRYOGENIC VALVE CONNECTION | Connect the Cryogenic Cooling Valve to the Controls |

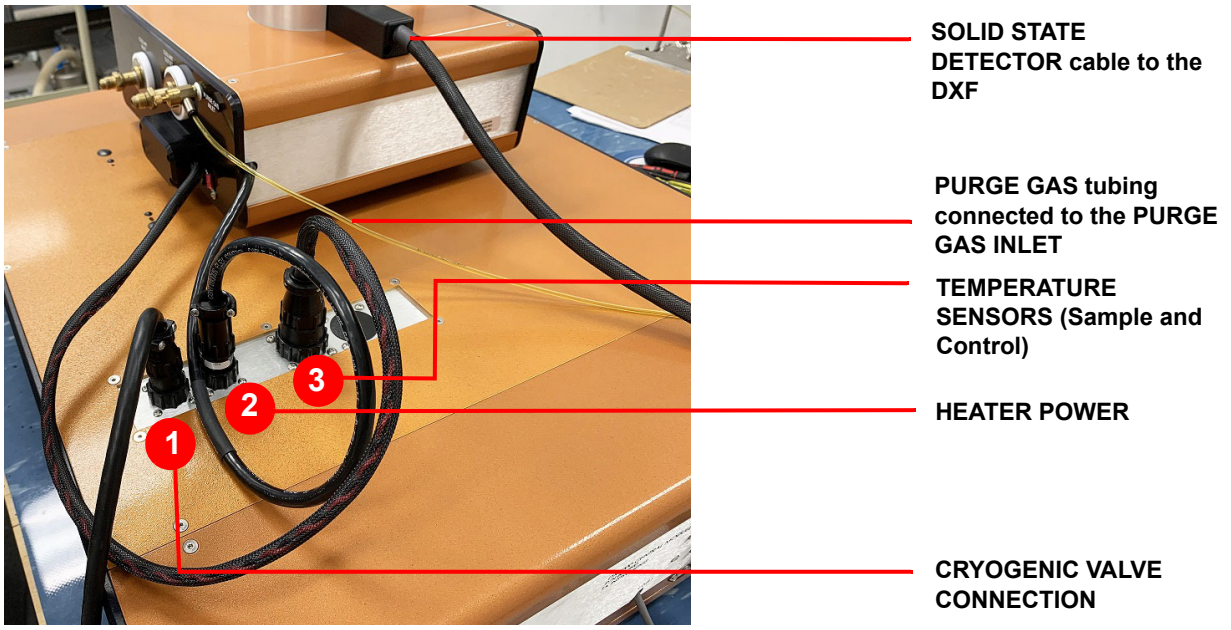


**Figure 9** Environmental Module top panel connections.

## *Connecting the Environmental Module to the Pulse Source*

To connect the DXF to the EM, follow the instructions below. Refer to Figure 8 and Figure 9 above for connection ports, or the diagram located in the Appendix.

1 Connect the **SOLID STATE DETECTOR** cable from the back of the head plate on the EM to the **J102** port on the back of the DXF.

2 Connect the **TEMPERATURE SENSORS** cable from the side of the EM to the 3 port on the top of the DXF.

3 Connect the **HEATER POWER** cable from the back of the EM to the 2 port on top of the DXF.

4 Connect the **CRYGENIC VALVE CONNECTION** from the **LN2 OUTLET** on the tank to the 1 port on top of the DXF.

## Setting Up System Communication with the Controller

1. Place the computer and monitor to the side of the unit and connect the keyboard and mouse.
2. Connect the **USB** cable from the back of the DXF to the controller.
3. Connect the **USB1** cable from the back of the DXF to the controller.
4. Connect the monitor cable to the monitor port on the computer.
5. Connect the temperature controller communication cable.
6. Connect the power cords to the computer and monitor and plug them into the same wall outlets on the same circuit that the DXF is plugged into.
7. Turn on the computer and monitor.

# Connecting the Utilities

## *Connecting the Power*

Plug the **SYSTEM POWER** cord on the back of the DXF to an electrical outlet rated in accordance with the power input information on the back of the instrument.

**CAUTION: Whenever plugging or unplugging power cords, handle them by the plugs, not by the cords.**

**MISE EN GARDE: Chaque fois que vous branchez ou débranchez les cordons d'alimentation, tenez-les par les fiches et non par les cordons.**

**CAUTION: An independent heavy GROUND wire must be provided through the power hook up. Improper grounding may cause severe damage for which the supplier will not accept responsibility. All power strips must be fully grounded and carry the ground through to the sockets into which the computer is plugged.**

**MISE EN GARDE: Un fil de masse lourd et indépendant doit être fourni via le raccordement électrique. Une mise à la terre incorrecte peut causer de graves dommages pour lesquels le fournisseur décline toute responsabilité. Toutes les barrettes d'alimentation doivent être entièrement mises à la terre et acheminer la terre jusqu'aux prises dans lesquelles l'ordinateur est branché.**

# Connecting the LN2 Lines

1 Obtain an LN2 tank that can provide a maximum of 22 psi per the Thermal Diffusivity DXF 200 Site Preparation Guide.

2 Connect the **LN2 LIQUID OUTLET** on the tank to the **LN2 INLET** fitting on the right side of the EM. See Figure 3.

3 Connect the **CRYOGENIC VALVE** cable to the **LN2 OUTLET** on the top of the DXF (see Figure 9).


**Figure 10** LN2 tank connections.

**NOTE**: Use one wrench to keep the fitting on the furnace stationary while tightening the hose end to it. Do the same for the next step.

**4** Connect one end of the second coolant line to the **LN2 OUTLET** (see Figure 3) fitting and connect the other end to a hood or vent.

## Connecting the Gas Lines

**1** Connect 1/8” tubing to the **PURGE GAS INLET**. Optional: tee off this line to a flow gauge to monitor the flow rate.

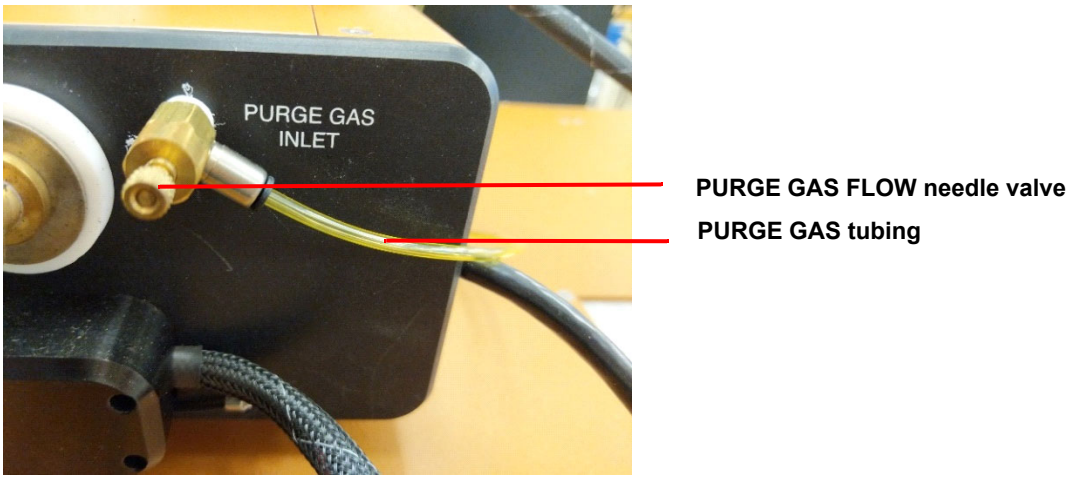


**Figure 11** Gas Line connections.

**2** Close the **PURGE GAS FLOW** needle valve.

**3** Check all air lines and connections for leakage.

# *Chapter 3:*

# Operating the DXF

## *Using the DXF*

All your DXF experiments will have the following general outline. In some cases, not all of these steps will be performed. Most of these steps are performed using the instrument control software. The instructions needed to perform these actions can be found in the online help in the instrument control software; therefore, they will not all be covered in detail here.

- Preparing the sample
- Selecting the appropriate sample adapter and holder
- Loading the sample and closing the EM
- Evacuating the system and setting the purge gas flow rate
- Creating or choosing the test procedure and entering sample and instrument information through the instrument control software
- Starting the experiment

To obtain accurate results, follow these procedures carefully.

### Before You Begin

Before you set up an experiment, ensure that the DXF system and the controller have been installed properly. Make sure you have:

- Made all necessary cable connections from the Environmental Module to the DXF
- Connected the instrument with the controller
- Connected the LN2 and gas lines
- Powered up the unit
- Become familiar with controller operations
- Viewed the software's Demo mode by going to **Setup** > **Select Demo Mode**

# *Startup and Shutdown Procedures*

## Starting the DXF System

The power switch is located on the back panel of the DXF. The power switch is used to turn the DXF system on and off.

To power on the system:

**1** Check all connections between the DXF, the Environmental Module, the Solid State Detector Assembly, and the controller. Make sure each component is plugged into the correct connection port.

**2** Set the DXF **SYSTEM POWER** switch to the ON (**I**) position.

**3** The temperature controllers on the front panel indicate that power is turned on. After the proper power up sequence, the instrument user interface appears and the keypad buttons are lit; this indicates that the instrument is ready for use.

## Shutting Down the DXF System

Before you decide to power down your system, consider the following:

- All the components of your system are designed to be powered on for long periods.
- The electronics of the DXF and the controller perform more reliably if power fluctuations caused by turning units on and off are minimized.

For these reasons, turning the system and its components on and off frequently is discouraged. Therefore, when you finish running an experiment on your instrument and wish to use the system for some other task, it is recommended that you leave the instrument on.

To power down your system, set the DXF **SYSTEM POWER** switch to the OFF (**0**) position.

## Preparing the Sample

The method is applicable for testing homogeneous, solid, and opaque materials. The sample should have an electrically conducive surface. Materials greatly deviating from the above can be tested with special sample preparations–for non-electrical contact surfaces (polymers, ceramics, etc) a layer of silver paint must be applied, or you can sputter the sample with gold, platinum, silver, nickel. It is up to you to determine if a new material can be tested in an "as received form" or if special preparation is needed.

### *Sample Dimensions*

It is extremely important to machine samples to exact dimensions. A sample must clear and fit the holder (or the insert) opening. If the sample diameter is equal or slightly bigger than the opening, then there will be an additional heat transfer from the tightly fitted sample to the holder during the measurement, which may negatively affect the preciseness of the thermal diffusivity measurement. If the sample diameter is much smaller than the opening, then the light beam can bypass the sample and interact directly with the Solid State detector, causing temperature signal distortion.

The thermal diffusivity calculation depends on square of the sample thickness. Uncertainty in the sample thickness value due to rough, or not completely flat surfaces will result in uncertainty (error) in the thermal diffusivity determination. It is therefore very important to machine a sample with the tolerance as high as possible (recommended tolerance ±1 mil). The tolerance on the lateral surfaces can be lower (± 5 mil).

Samples that are too thick are difficult to test due to weak temperature response signal and slow and sluggish response to the flash affected by heat losses from the sample. Samples that are too thin may not represent bulk material, and potentially increase measurement error due to severe finite pulse time effect. The thickness should be designed the way the sample experimental halftime should be within the range from 40 ms to 2 s. (See Theory of the Flash Method in online help for a more detailed explanation of the relationship between the sample thickness and the thermal diffusivity of the material.) More than one iteration of trial and error may be needed to find the optimal sample thickness for a particular material. Examples of thicknesses for different materials:

- Stainless steel = 1.6 mm
- Pyroceram = 2 mm
- Graphite, alumina, and molybdenum = 3.2 mm
- Copper and aluminum = 5 mm

**NOTE**: Testing samples of irregular shape or uneven thickness is not recommended, due to possible errors in the thermal diffusivity and the specific heat determination and/or possible damage to the instrument.

## *Transparent or Translucent Materials*

The method requires that all of the energy from the flash be absorbed on one face of the sample. For materials that are transparent or translucent, the energy from the light pulse will travel to different depths in the sample (depending on opacity) or may completely pass through in a completely transparent sample. To prevent this, a very thin and opaque layer of high thermal diffusivity material must be deposited on the faces of the samples. For best results, one should select a coating that has a high reflectivity (low emissivity), such as gold or platinum. Conversely, this type of coating is not well suited to absorb the energy pulse, and therefore it is customary to put a second, highly absorbing graphite coat on top of it.

It is critical to keep both coatings at minimal thickness. This reduces the contribution of the layers to the total transmission time as compared to the contribution of the sample.

Best results are obtained with vacuum sputtering of a gold, silver, nickel, or platinum layer with < 1 µm thickness. This coating is then over-coated using an aerosol spray coating of graphite.

Please note that:

- In all cases the coating material must be selected to safely withstand any temperature within the test parameters, and the graphite coating must be applied in a thin layer.
- Avoid scratching the coated surfaces after curing; even tiny amounts of porosity will severely affect the data. Prevent any dust from settling on the coated surfaces.
- Some materials that generally require coating for opacity are: glass, quartz, alumina, zirconia, silicon carbide, silicon nitride, calcium fluoride, zinc selenide, etc.

## *High Reflectance Samples*

In instances where the material has a highly reflective surface, it may be difficult to absorb sufficient energy from the flash to produce a good signal on the opposite face. A thin coating of graphite spray usually remedies this situation.

For specific heat capacity testing it is imperative that both the unknown sample and the reference have identical emissivities over the spectral range of the High-Speed Xenon Source. To ensure this, always coat both the sample and the reference with graphite coating.

## Applying Graphite Spray

The following is the procedure for coating samples with graphite spray:

**NOTE**: Only one side (the bottom) of the sample should be coated with graphite spray.

**1** Place the samples onto a plain sheet of paper. Locate them in one line close to each other.

**2** Using a heat gun, gently warm the samples.

**3** Hold the aerosol can of graphite spray approximately 8–10 inches from the samples. Using a single, fluent motion spray the exposed surfaces from left to right.

**4** Allow one minute for the coating to dry on the samples.

**5** Following the same path as in step 3, apply a second coating across first pass.

**6** Allow a minimum of 3 minutes for the samples to dry before coating the other side. Repeat steps 2 through 6.

**7** After drying, store the samples on a soft surface (ex: tissue paper).

## Loading and Unloading the Sample

**1** Turn off the LN2 supply.

**WARNING: Cartridges will be hot. Use caution when loading and unloading samples. Check the furnace temperature on the front panel; 60°C and below is safe to handle.**

**AVERTISSEMENT: Les cartouches sont chaudes. Soyez prudent lors de la mise en place et de la décharge des échantillons. Vérifiez la température du four sur le panneau avant; si elle est égale ou inférieure à 60° C, alors vous pouvez le manipuler en toute sécurité.**

**2** Unlock the Solid State Detector locking arm by turning the knob counter-clockwise until the arm loosens.

**Figure 12** Unlocking the locking arm.

**3** Swing the locking arm away from the Detector.

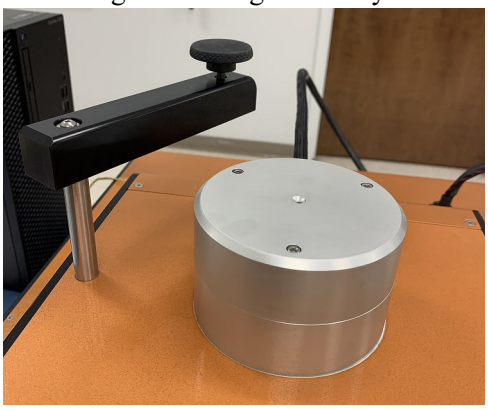

**Figure 13** Swing the locking arm away from the Detector.

**4** Lift the Solid State Detector up and swing it to the right side away from the sample area.

**Figure 14** Swing the Solid State Detector away from the sample area.

**5** Swing the locking arm back so that it rests against the side of the Solid State Detector, holding it in place.

**Figure 15** Locking arm holding the Solid State Detector away from the sample area.

**6** Unscrew the sample holder cover with the provided screwdriver (kit p/n?).


**Figure 16** Removing the sample holder cover.

**7** The sample holder fits 1/2” (12.7 mm) O.D. samples. If there are screws in the four corners of the sample holder, remove them. If using 1” (25.4 mm) O.D. samples, remove the sample holder.


**Figure 17** 1/2” (12.7 mm) O.D. sample holder cover.

**8** With tweezers, carefully place the sample into the sample holder.

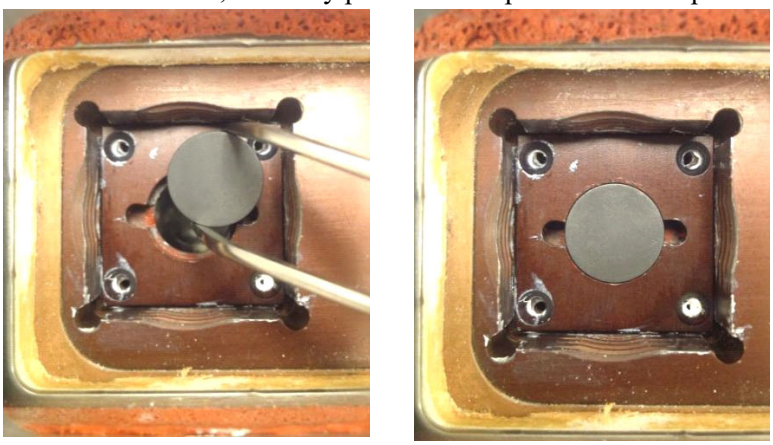

**Figure 18** Loading the sample (left); Sample loaded (right).

**9** Screw the sample holder cover back into place.

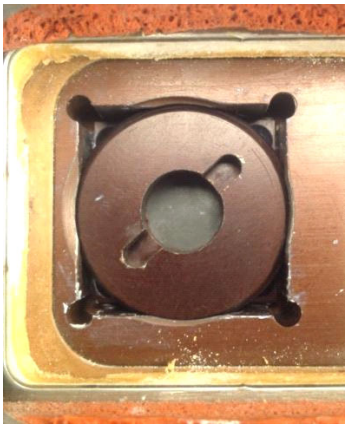

**Figure 19** Cover back in place.

**10** Move the Solid Sample Detector back into place on top of the sample chamber, making sure that the tip of the pins reaches the center of the sample.

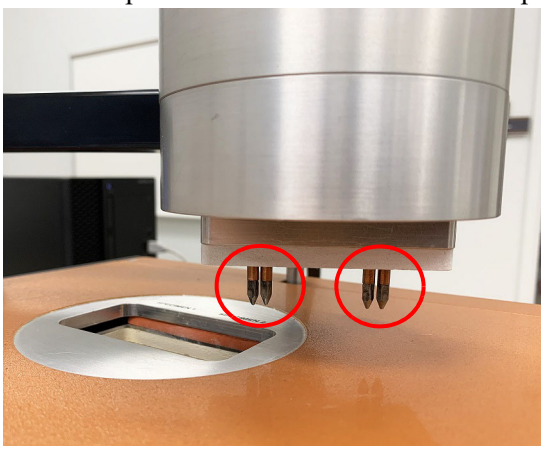

**Figure 20** Pins.

**11** Move the locking arm back into place, aligning the locking arm with the groove in the top center of the Solid State Detector.

**Figure 21** Center groove on the Solid State Detector.

# Preparing the Instrument

## *Evacuating the System*

All are equipped with purge capabilities.

### Back-fill with Purge Gas

Open the needle valve and allow the gas to flow inside of the furnace for approximately ? minutes.

# *Starting an Experiment*

Access the instrument control software to create or choose the test procedure as well as enter sample and instrument information through the instrument control software. Consult online help for experimental instructions.

**1** To open the software, double-click the FlashLine icon on the computer desktop.

**2** Click **Operation** > **Startup Information**.

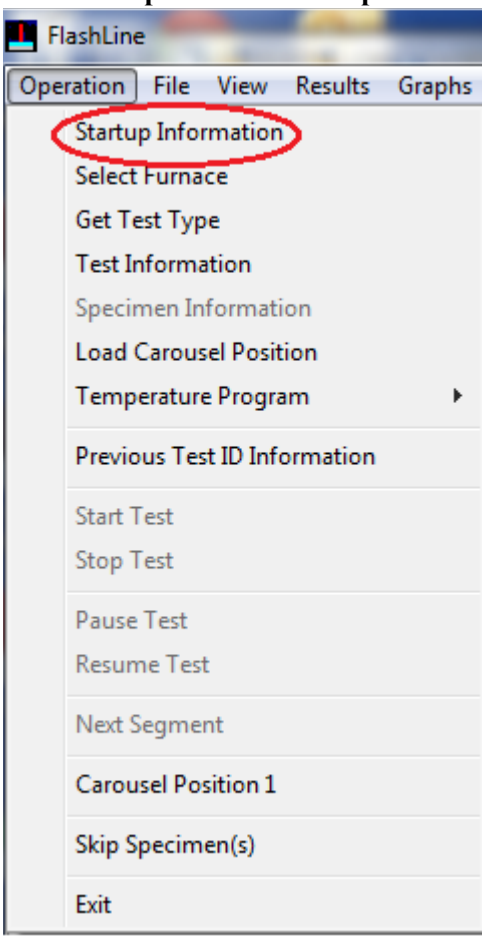


**Figure 22** Operation > Startup Information.

**3** Click **OK** to confirm your selection. The Test Information window displays.

4 Enter the test parameters and then click **Start Test**. Refer to online help for details on entering test parameters in the Test Information window.

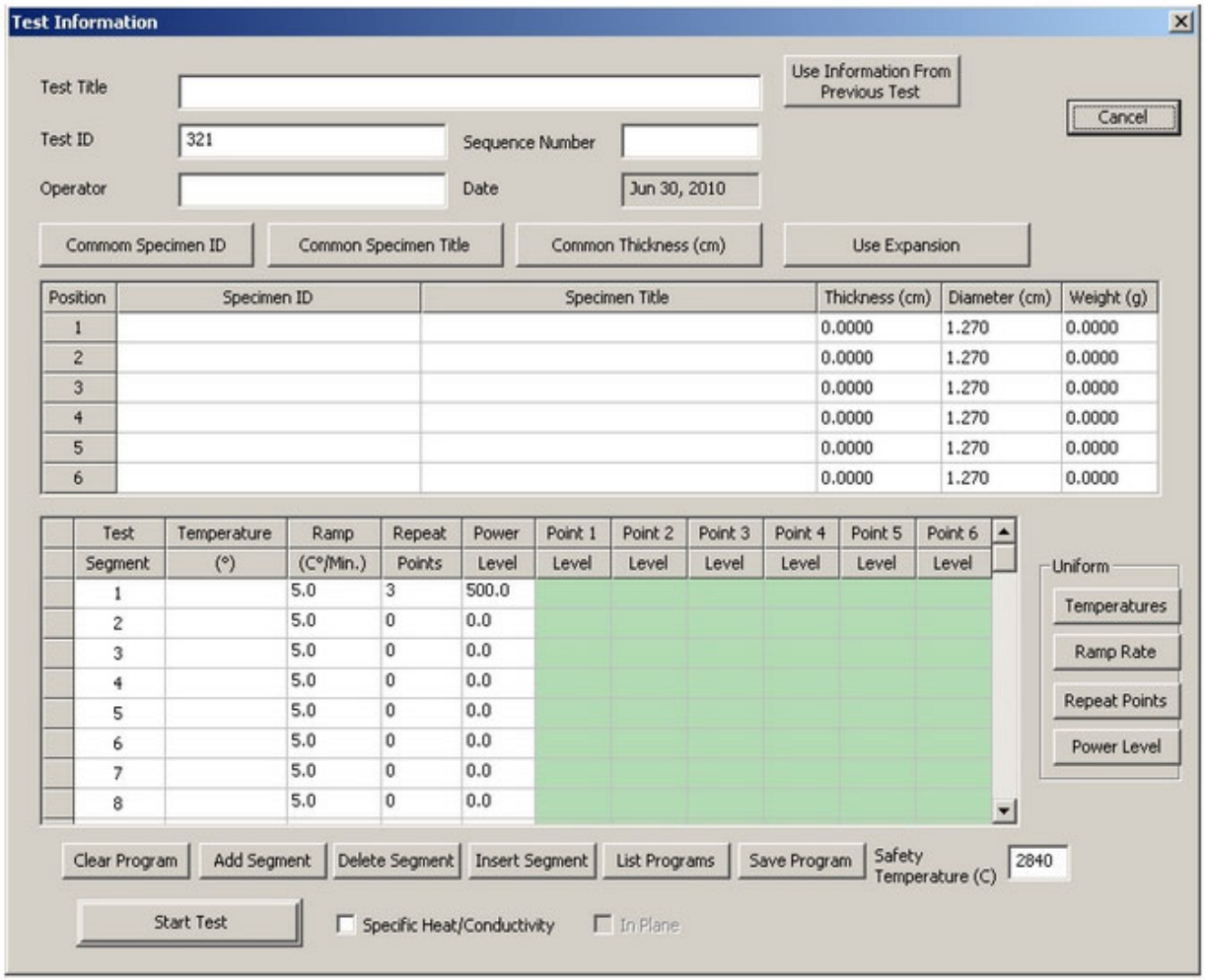


**Figure 23** Test Information window.

# *Stopping an Experiment*

If for some reason you need to discontinue the experiment, you can stop it at any point by clicking the **Operation** menu in the Flashline software and then selecting **Stop Test**.

# *Chapter 4:*

# Maintaining the DXF/EM

The primary maintenance procedures described in this chapter are the customer's responsibility. Any further maintenance should be performed by a representative of TA Instruments or other qualified service personnel. Consult the Help documentation installed with the instrument control software for further information.

**DANGER: Because of the high voltages in this instrument, untrained personnel must not attempt to test or repair any electrical circuits.**

**DANGER: À cause de la présence de tensions élevées dans cet instrument, le personnel non formé ne doit pas essayer de tester ou de réparer les circuits électriques.**

**CAUTION: Before using any cleaning or decontamination method except those recommended by TA Instruments, check with TA Instruments that the proposed method will not damage the instrument.**

**MISE EN GARDE: Avant d'utiliser une méthode de nettoyage ou de décontamination autre que celle recommandée par TA Instruments, vérifiez auprès de TA Instruments que la méthode proposée n'endommagera pas l'instrument.**

## *General Cleaning Practices*

- Use protective eye glasses
- Use finger coats or gloves
- Always hold optical components (mirrors, lenses, windows, etc.) from the edges; never touch the center after cleaning
- If strongly burned films remain on the components after the cleaning process, use weak acid (like vinegar) to try to dissolve the residual layers
- Always use distilled water and acetone at the end of cleaning process
- Use a thin layer of vacuum grease to seal all O-ring parts of the EM module whenever it is necessary to reassemble the block containing O-rings

# *Recommended Cleaning Supplies*

- Compress air duster can or dry nitrogen from the bottle which supply the purge gas for instrument-with flexible plastic, movable/bend nozzle (never use compress air from industrial compressor lines; it contains a large amount of water and residual oil)
- Acetone and propanol-laboratory grade-with droplets pump attach to bottles
- Dust- and lint-free optical tissue or fabric
- Cotton swabs, cleaning brushes, mini vacuum cleaner
- Scouring pads (such as Scotch-Brite™), detergent, acetic acid (vinegar), distilled water

# *Cleaning Maintenance Schedule*

How often cleaning should be performed depends on the following factors: how often the instrument is used, the maximum temperature of the tests, if it is being used with purge gas, and the nature of the samples. Tests that measure only thermal diffusivity are more tolerant with contamination of the optics than tests that measure diffusivity and specific heat. For only diffusivity, multiply with 3 the number of tests below.

The cleaning schedule below is recommended for the best results of the instrument:

- After 10 tests run at max. temperature of 200°C in an inert purge gas atmosphere
- After 20 tests run at max. temperature of 100°C in an inert purge gas atmosphere
- In the case of testing volatile or reactive materials, cleaning is recommended after each test run

Periodically a check test should be run to verify the cleanliness of the optical path of the instrument. Use a thermographite reference sample 0.300 cm thick, room temperature test, 500 volts charging, and 3 shots. The instrument is considered clean if the signal (max. temperature of thermo diagram) is larger than 200 millivolts.

If, after cleaning the instrument and running a check test, the signal of thermo diagram is still smaller than 200 millivolts, contact TA Instruments Service.

## Clean the Solid State Detector Pins

The Solid State Detector pins can be worn down over time after repeated contact. Debris from the sample can also accumulate on the tips. Checking and cleaning the pins periodically is important because they must make good contact with the surface of the sample to obtain accurate results. If the pins are not making good contact or if the tips are not the correct shape (see Figure 27), you will see excess noise on the signal.

**1** Unlock the Solid State Detector locking arm by turning the knob counter-clockwise until the arm loosens.

**Figure 24** Unlocking the locking arm.

**2** Swing the locking arm away from the Detector.

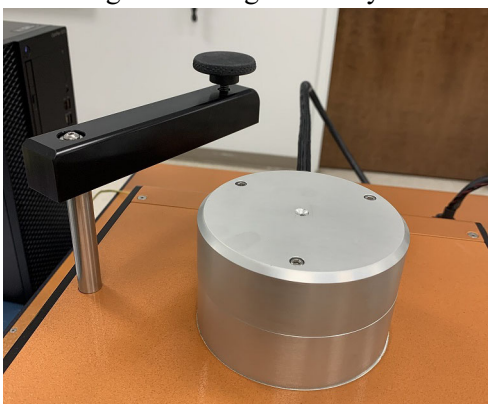

**Figure 25** Swing the locking arm away from the Detector.

3 Lift the Solid State Detector up and swing it slightly to the side away from the sample area. Swing the locking arm back so that it rests against the side of the Solid State Detector, holding it in place.

**Figure 26** Locking arm holding the Solid State Detector in place.

4 Check the pin shape. The slightly pointy shape of the pin on the left in the image below can dent or even penetrate the sample. The pin shown on the right has a flat tip, allowing for good contact with the sample.

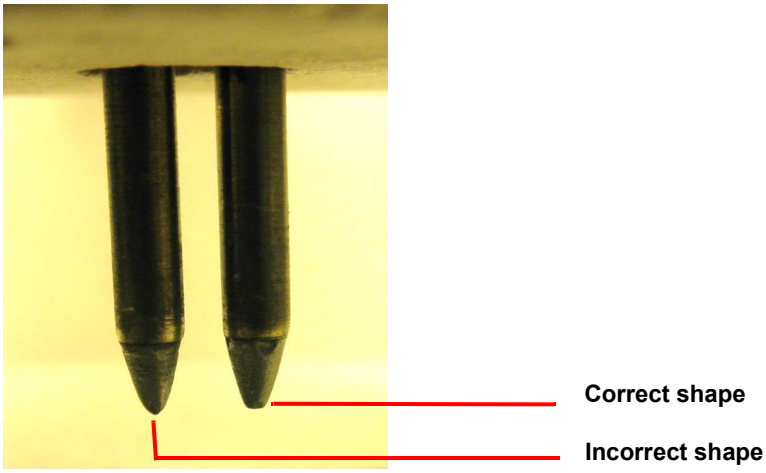


**Figure 27** One pair of Solid Sample Detector pins (for one sample).

5 Use alcohol and very fine sandpaper to clean the tip of each pin. Slowly and carefully smooth out any rough edges or points.

6 Put the Detector back in place, locking the locking arm.

## Clean the Light Pipe Assembly

**NOTE**: The sample is directly above the Xenon pulse distribution light pipe. It is therefore important to protect the light pipe from dirt and possible damage. Periodic cleaning of the top surface of the light pipe is recommended. If dirt or other substances build up on it, it will adversely affect the signal for the temperature rise of the top surface of the sample, and yield erroneous results.

1 Disconnect the LN lines.

2 Lift the EM module from the top of DXF module. The EM can simply be moved a few inches back, remaining on top of the DXF.

3 Tilt the EM module at a 90° angle, allowing it to rest on its front so that you can access the light pipe

assembly located on the bottom of the EM.

**4** Remove the four screws from the Light Pipe Assembly panel.

**Figure 28** Remove the light pipe assembly.

**5** Remove the Light Pipe Assembly from the EM.

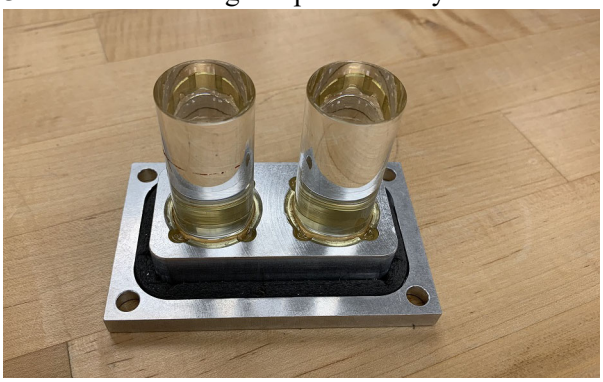

**Figure 29** Light Pipe Assembly.

**6** Inspect the assembly for debris, dust, and smoke. Using a clean, oil-free optical-grade air source, blow out all the dust/debris that is on the surface of the light pipe. Clean all outer surfaces of the light pipe using a lint-free optical-grade cloth and high-purity acetone. A small nozzle vacuum may also be used to clean dust from the surface of the light pipe.

**NOTE**: Take care not to scratch the surface of the light pipe.

**7** Reassemble the parts back into EM module.

## Clean the Xenon Lamp Assembly Optics

**8** Using the compress air duster, clean the exit window to remove any possible dust that may be contaminating the window.

**9** Realign the EM module on top of DXF module in original working position.

# *Replacement Parts*

Replacement parts for the DXF are listed below. Refer to the table below when ordering parts.

| Part Number | Description |
|---|---|
| 853866.901 | In-Plane Test Kit, Stainless Steel, 25.4 mm OD, DXF-200 |
| 202417.001 | Graphite Paint |
| 202419.001 | Silver Paint - Vial - 1 oz. |
| 853189.901 | Pin Detectors SOLID STATE PKG/2 |
| 853135.901 | Thermographite Reference, 12.7 mm OD, 3.175 mm thick |
| 853142.901 | Thermographite Reference, 25.4 mm OD x 3.175 mm thick |
| 853143.901 | Stainless Steel Reference, 12.7 mm OD x 1.58 mm thick |
| 853144.901 | Stainless Steel Reference, 25.4 mm OD x 1.58 mm thick |
| 853145.901 | Electrolytic Iron Reference, 12.7 mm OD x 2.54 mm thick |
| 853146.901 | Electrolytic Iron Reference, 25.4 mm OD x 2.54 mm thick |
| 853147.901 | OFHC Copper Reference, 12.7 mm OD x 5.08 mm thick |
| 853152.901 | Vespel Reference, 12.7 mm OD x 1.27 mm thick |
| 853154.001 | Alumina Reference, 12.7 mm OD x 3.175 mm thick |
| 853157.901 | Molybdenum Reference, 12.7 mm OD x 3.175 mm thick |
| 854144.901 | Solenoid Valve Cryogenic |