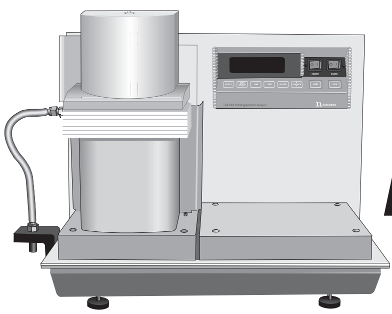

DMA 2980

Dynamic Mechanical Analyzer

Operator's Manual

PN 984004.001 Rev. J (Text and Binder)
PN 984004.002 Rev. J (Text Only)
Issued March 2002

Notice

The material contained in this manual is believed adequate for the intended use of this instrument. If the instrument or procedures are used for purposes other than those specified herein, confirmation of their validity and suitability must be obtained from TA Instruments, Inc. Otherwise, TA Instruments does not guarantee results and assumes no obligation or liability. This publication is not a license to operate under nor a recommendation to infringe upon any process patents.

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Notes, Cautions, and Warnings

This manual uses NOTES, CAUTIONS, and WARNINGS to emphasize important and critical instructions under the guidelines described below:

NOTE: Highlights important information about equipment or procedures.

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

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

Safety

This equipment has been designed to comply with the following standards on safety:

- IEC 1010-1/1990 and A1/1992 and A2/1995
- IEC 1010-2-010/1992 and A1/1996
- EN 61010-1/1993 and A2/1995
- EN 61010-2-010/1994
- UL 3101-1, First Edition.

CE Compliance

In order to comply with the Electromagnetic Compatibility standards of the European Council Directive 89/336/EEC (EMC Directive) and Directive 73/23/EEC on safety as amended by 93/68/EEC, the following specifications apply to the DMA 2980 instrument:

- *Safety:*
  EN 60110-1/1993 and A2/1995 Installation Category II
  EN 61010-2-010/1994
- *EMC*

  EN61326-1: 1997 Electrical equipment for measurement, control, and laboratory use-EMC requirements-Part 1: General Requirements + Amendments. Emissions: Meets Class A requirements (Table 3). Immunity: Meets performance criteria B for non-continuous operation, minimum requirements (Table 1).

Instrument Symbols

The following labels are displayed on the DMA 2980 instrument for your protection:

Symbol	Explanation
	Indicates the presence of one or more of the following: hazardous voltage or moving parts.
	Indicates that a hot surface may be present. Take care not to touch this area or allow any material that may melt or burn come in contact with this hot surface.

Please heed these labels and take the necessary precautions when dealing with those parts of the instrument. The *DMA 2980 Operator's Manual* contains cautions and warnings that must be followed for your own safety.

Electrical Safety

Voltages exceeding 110 Vac are present in this system. Always unplug the instrument before performing any maintenance.

!WARNING

Because of the high voltages in this instrument, untrained personnel must not remove the cabinet cover. Maintenance and repair of internal parts must be performed by TA Instruments, Inc. or other qualified service personnel only.

!WARNING

After transport or storage in humid conditions, this equipment could fail to meet all the safety requirements of the safety standards indicated. Refer to the NOTE on page 2-5 for the method used to dry out the equipment before use.

Thermal Safety

During an experiment, the furnace, sample, and clamp can become very hot or very cold to the touch.

!WARNING

Allow the clamp to return to room temperature before touching the clamp. Take the proper precautions when removing a hot sample or retorquing a sample.

!WARNING

Do not use your hands to manually move the furnace and do not put your hands up inside the furnace. It may be hot enough to cause burns.

Handling Liquid Nitrogen

The DMA 2980 uses liquid nitrogen, as a source of cold gas, in the Gas Cooling Accessory (GCA). Because of its low temperature (-196°C), liquid nitrogen will burn the skin. Use extreme caution when working with liquid nitrogen and other cryogenic materials to ensure that you do not burn yourself.

Personnel working with liquid nitrogen should take the following precautions.

! WARNING

Liquid nitrogen evaporates rapidly at room temperature. Be certain that areas where liquid nitrogen is used are well ventilated to prevent depletion of oxygen in the air.

1. Wear goggles or a face shield, gloves that are 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 shoe tops.

2. Transfer the liquid slowly to prevent thermal shock.

3. Use containers having adequate low-temperature properties. Ensure that closed containers have vents to prevent pressure buildup; liquid nitrogen evaporates rapidly at room temperature.

4. The purity of liquid nitrogen alters as it evaporates. If much of the liquid in the container has evaporated, check the remaining liquid before using it for any purpose in which high oxygen content is dangerous.

WARNING

Potential Asphyxiant

Liquid nitrogen can cause rapid suffocation without warning.

Store and use in an area with adequate ventilation.

Do not vent GCA container in confined spaces.

Do not enter confined spaces where nitrogen gas may be present unless the area is well ventilated.

The warning above applies to the use of liquid nitrogen. Oxygen depletion sensors are sometimes utilized where liquid nitrogen is in use. Please refer to the *TA Instruments Gas Cooling Accessory* manual for more detailed instructions regarding the use of the GCA.

IF BURNED BY LIQUID NITROGEN...

1. Flood the area (skin or eyes) with large quantities of cool water IMMEDIATELY; then apply cold compresses.

2. See a doctor IMMEDIATELY if the skin is blistered or if there is a chance of eye infection.

Air Pressure Warning

! WARNING

The compressed air required to operate the instrument, which is either a house air supply or supplied by the Air Compressor Accessory (ACA), is at high pressures. This high pressure can be dangerous to both personnel and equipment if not handled properly.

- **If you are installing the DMA 2980 without the ACA, the tubing leading to the air filter regulator must have a pressure rating adequate to handle the source pressure. The pressure going to the air filter regulator must not exceed 150 psi (1000 kPa).**

- **If you are installing the DMA 2980 with the ACA, the tubing supplied by TA Instruments with the accessory must be used to connect it to the air filter regulator. The ACA has a pressure relief valve limiting the pressure supplied by the ACA to 70 psi (500 kPa) maximum.**

The tubing supplied with the DMA must be used to connect the instrument to the air filter regulator. Set the pressure on the air filter regulator to 60 to 65 psi (420 to 455 kPa).

DMA Submersion Clamps Warning

Two DMA clamps are available for evaluation of material viscoelastic properties while the material is submerged in an ambient (room ~ 23°C) temperature fluid. These clamps (compression clamp PN 984448.901 and film/fiber clamp PN 984449.901) have been designed to prevent the DMA furnace from closing around them and heating the submersion fluid.

!WARNING

Do not attempt to close the DMA furnace with these clamps mounted on the instrument. Damage to the furnace, clamps, and/or air bearing drive system could result.

In addition, flammable or volatile fluids should not be used as the submersion fluid, nor should an external source of heating be used to elevate the temperature of the submersion fluid during measurement, since these conditions could result in an unsafe situation.

Lifting the Instrument

The DMA 2980 is a fairly heavy instrument. In order to avoid injury, particularly to the back, please follow this advice:

! WARNING

Close the furnace before moving the instrument, even for a short distance. Use two people to lift and/or carry the instrument. The instrument is too heavy for one person to handle safely.

Sample Decomposition

The DMA 2980 is capable of heating samples to 600°C. Many materials may decompose during the heating, which can generate hazardous byproducts.

! WARNING

If you are using samples that may emit harmful gases, vent the gases by placing the instrument near an exhaust.

Samples should not be heated above their decomposition temperatures to prevent the relase of hazardous materials or contamination of the DMA 2980.

We recommend that you measure the decomposition temperatures to determine the potential for problems by heating the sample materials in a thermogravimetric analyzer (TGA) or similar instrument, before running the sample on the DMA.

Using This Manual

CHAPTER 1	Introduces the DMA 2980 and lists its specifications.
CHAPTER 2	Describes how to install and assemble your DMA 2980.
CHAPTER 3	Provides the basic procedures used to calibrate the instrument.
CHAPTER 4	Explains the steps needed to run experiments on the DMA 2980.
CHAPTER 5	Describes the installation, removal, and use of the optional clamps.
CHAPTER 6	Explains the technical aspects of the DMA and its theory of operation.
CHAPTER 7	Provides maintenance and diagnostic procedures.
Appendix A	Lists TA Instruments offices that you can contact to place orders, receive technical assistance, and request service.
Index	Contains an alphabetical list of topics and page number references.

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Introduction

The TA Instruments Dynamic Mechanical Analyzer (DMA) 2980 is an analytical instrument used to test the physical properties of many different materials. The sample is mounted on a clamp and then subjected to changes in stress/strain while undergoing a change in temperature. The DMA measures the modulus (stiffness) and damping (energy dissipation) of the sample.

The DMA 2980 works in conjunction with a TA Instruments controller and associated software to make up a thermal analysis system.

Your controller is a computer that performs the following functions:

- Provides an interface between you and the analysis instruments
- Enables you to set up experiments and enter constants
- Stores experimental data from the instrument
- Runs data analysis programs.

Components

Your instrument consists of two major parts, the DMA cabinet and the DMA assembly (see Figure 1.1 on the next page). The following components make up the DMA assembly:

- The *mechanical section enclosure* surrounds the air bearings, optical encoder, drive motor, and the associated electronics.

- The *clamp assembly* (called the "clamp") is interchangeable for making mechanical measurements in a variety of deformation modes to accommodate a wide array of sample shapes and materials.

- The *furnace assembly* surrounds the clamp assembly to heat and cool the sample. The furnace temperature is monitored by the control thermocouple.

- The CHROMEL®*/ALUMEL®* *sample thermocouple* senses the temperature of the sample and relays the reading to the instrument.

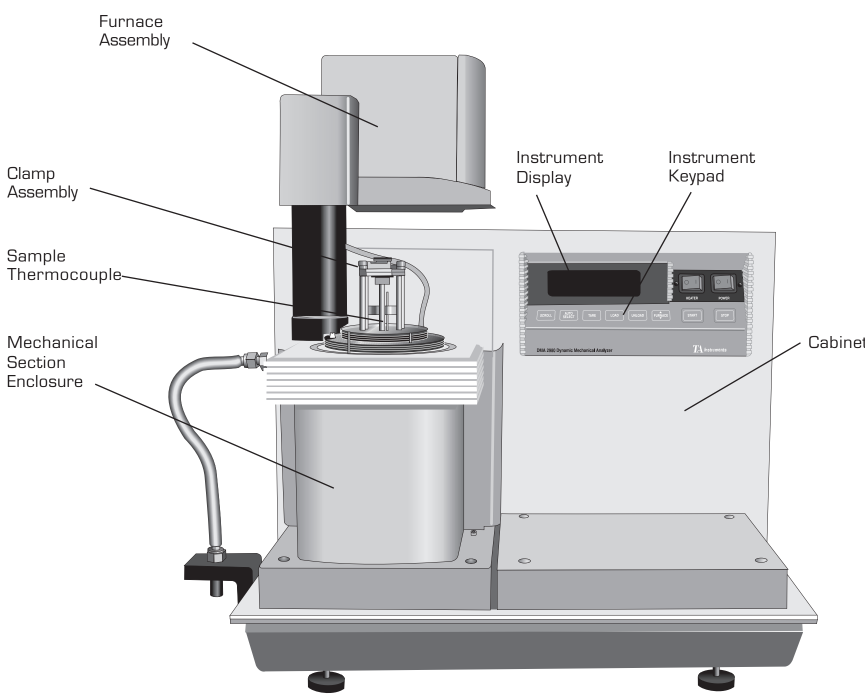

Figure 1.1 The DMA 2980 Instrument

* CHROMEL® and ALUMEL® are registered trademarks of the Hoskins Manufacturing Company.

- The DMA *cabinet* houses the electronics, valves, etc.

- The *keypad* allows you to control and monitor some of the DMA functions from the instrument.

- The *display* is used to monitor the instrument state and the operating parameters.

The DMA Instrument

The DMA 2980 instrument contains the electronics and software needed to perform experiments and store the results. The battery backed-up memory in the cabinet saves parameters vital to system operations if power is interrupted. Also contained in the cabinet is a GPIB interface for communication with the controller and a serial port to communicate with the GCA.

You can set up, start, stop, and reject the experiment using the keypad on the cabinet or the Instrument Control software. The display provides valuable realtime information on the experiment in progress.

The DMA was developed by TA Instruments with the following features:

- operates over a temperature range of -145°C to 600°C, using heating rates up to 50°C/min.

- determines changes in sample properties resulting from changes in five experimental variables: temperature, time, frequency, force, and strain.

- uses samples that can be in bulk solid, film, fiber, gel, or viscous liquid form.

- employs interchangeable clamps allowing you to measure many properties, including: modulus, damping, creep, stress relaxation, glass transitions, and softening points.

DMA 2980 Display

The front of the cabinet contains the *keypad*, used to control the mechanical movements of the instrument, and the *display*, used to observe the status of the instrument.

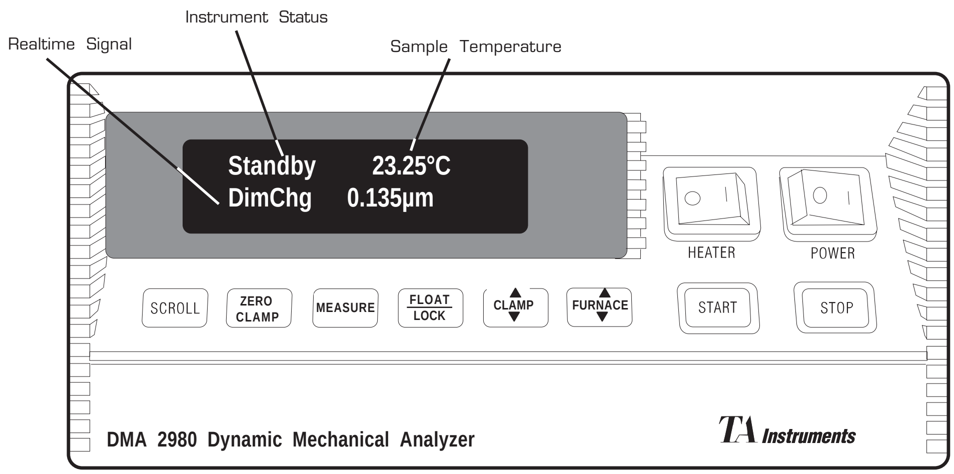

Figure 1.2
DMA 2980
Keypad and Display

The DMA display is the lighted area of the keypad unit (see Figure 1.2). It contains two rows of 20 characters each.

During normal operation, the display is divided into three areas:

- Instrument status display—the eight-character line on the top left
- Sample temperature display—the nine characters on the top right
- Realtime signal display—the whole bottom line.

DMA 2980 Keypad

The instrument keypad (see Figure 1.2) contains the keys found in Table 1.1 and the HEATER and POWER switches.

NOTE: Experiment and instrument parameters are entered from the controller keyboard, not the instrument keypad. See the *Thermal Solutions* User Reference Guide.

Table 1.1 DMA 2980 Keypad Functions

Key/Function	Explanation
SCROLL	Scrolls the realtime signals shown on the bottom line of the display.
ZERO CLAMP	Performs a clamp zero calibration for the tension, compression, and penetration clamps. (See Chapter 3 for more information.)
MEASURE	Used for multiple functions depending upon the instrument mode and the clamp type selected. This key opens the air-bearing gas valve, measures the sample length or thickness (where appropriate) using the selected initial force, and turns the motor drive on according to the instrument mode and conditions.

(table continued)

Table 1.1 DMA 2980 Keypad Functions (continued)

Key/Function	Explanation
▲ CLAMP ▼	Used to raise and lower the moveable clamp by applying the initial force. If zero initial force is selected, then 0.05 N is used. The direction of the clamp motion changes each time the key is pushed. You can stop the clamp motion by pressing the STOP key or the FLOAT/LOCK key.
FLOAT / LOCK	Toggles between floating and locking the moveable clamp. *Float*—turns on the air-bearing gas and applies zero force. *Lock*—turns off the air-bearing gas, locking the clamp in its current position. If the motor drive is inactive, then this key does a float. If the motor drive is active, then this key does a lock.

(table continued)

Table 1.1 DMA 2980 Keypad Functions (continued)

Key/Function	Explanation
FURNACE ▲▼	Raises or lowers the furnace. Press this key a second time to stop the furnace movement. Press the key twice, while the furnace is moving, to halt and reverse the movement. This key can be used while a method is running to open the furnace and adjust the sample clamping. The method will continue running, although the heater and GCA will be inactive while the furnace is open.
START	Initiates the experiment after checking the program method against the mode type. This is the same function as **Start** on the Instrument Control software.
STOP	If an experiment is running, this key ends the method normally, as though it had run to completion; *i.e.*, the method-end conditions go into effect and the data that has been generated is saved. This is the same function as **Stop** on the Instrument Control software.

(table continued)

Table 1.1 DMA 2980 Keypad Functions (continued)

Key/Function	Explanation
STOP	If an experiment is not running (the instrument is in a standby or method-end state), the STOP key halts any activity (air cool, all mechanical motion, etc.). The STOP key can also be used to stop the drive and lock the slide.
REJECT (Hold down SCROLL and press STOP)	If an experiment is running, SCROLL-STOP ends the method normally, as though it had run to completion; *i.e.*, the method-end conditions go into effect and the data that has been generated is *discarded*. This is the same function as **Reject** on the controller.
NOTE:	SCROLL operates normally (scrolls the text) until the STOP key is pressed. Then the display returns to the signal that was displayed before SCROLL was pressed. If an experiment is not running, SCROLL-STOP halts any activity as described for STOP.

HEATER Switch

The HEATER on/off switch turns the power to the instrument heater on and off (see Figure 1.2). The switch should be in the ON position before you start an experiment. If the HEATER switch is off, the method will not start.

NOTE: The light in the HEATER switch will glow only when an experiment is in progress or when the temperature is being controlled by a method-end condition.

POWER Switch

The POWER on/off switch turns the power to the instrument on and off (see Figure 1.2).

Accessories

The DMA 2980 can perform experiments on different types of samples using both standard and optional accessories.

Standard Accessories

The accessory kit supplied with the DMA 2980 contains the following:

- Torque driver wrench
- Hex keys (Allen wrenches)
- Hex driver (Allen driver)
- Calibration samples
- 100 gram weight
- Brass tweezers
- Digital calipers
- Polycarbonate and ABS samples.

Clamps

The DMA 2980 utilizes several different types of clamps. These clamps can be classified as either *tensioning* or *nontensioning* clamps. Tensioning clamps require that a positive force (preload force) be placed on the sample at all times.

Tensioning clamps are:

- 3-point bending
- film tension
- fiber tension
- compression
- penetration
- submersion compression
- submersion film/fiber.

Nontensioning clamps are:

- single cantilever
- dual cantilever
- shear sandwich.

Most of the clamps have two basic parts—a *moveable* clamp and a *fixed* clamp (also sometimes called a stage). The figure below shows the two parts of the standard dual cantilever clamp.

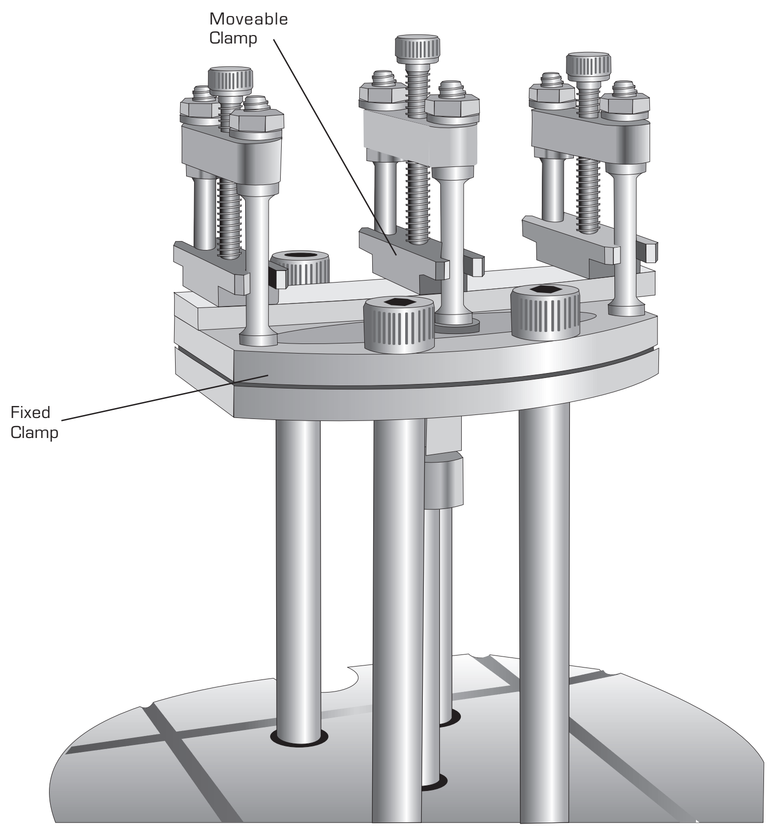

Figure 1.3 DMA Dual Cantilever Clamp

For the directions on installing and removing this clamp, turn to Chapter 2.

Optional Accessories

You can purchase extra items to use with your TA Instruments DMA. Some of the additional options available for use with the DMA are:

- The Gas Cooling Accessory (GCA), which is used as a way to obtain cooling gas to perform subambient experiments. The GCA utilizes liquid nitrogen, stored in a holding tank, to provide cooling. For more information about the GCA, see its operator's manual.

- The Air Compressor Accessory (ACA), which is used as an air supply source. See the next section for details.

- Additional clamps to use with various types of samples. See page 1-17 for a list of optional clamps, and see Chapter 5 for more details on the use of these clamps.

Air Compressor Accessory (ACA)

The Air Compressor Accessory (ACA) is a compact unit that connects directly to the air filter regulator. It supplies compressed air, to the air filter regulator for the DMA air bearing gas supply.

When installing the ACA for use with the DMA 2980, follow these guidelines:

- Do not locate the ACA on the same benchtop or tabletop as the DMA. The vibrations from the ACA will affect the DMA's performance.

- Position the ACA, leaving approximately 5 to 6 inches (12 to 15 cm) clear around the fan vents to allow air to circulate freely.

- Place the four rubber feet flat on the benchtop. Do not stand the ACA on end.

- Connect the power cable to the connection shown in the figure below.

- Connect the air tubing by pushing it into the connection shown in the figure below. Then connect the other end of the tubing to the air filter regulator.

Figure 1.4
Air Compressor Accessory

♦ ***CAUTION:***

Do not attempt to open the ACA; there are no customer-serviceable parts. Contact TA Instruments for service.

When turning the power on to the ACA, follow these steps to prevent pressure buildup:

1. Bleed off any pressure in the air filter regulator by opening the stopcock at the bottom of the coalescer filter. See Chapter 7 for more details.

2. Press the ACA power switch to the ON position.

See page 1-20 for the ACA specifications.

Optional Clamps

These optional clamps will allow you to perform experiments on different types of samples:

- 3-point bending clamp
- film/fiber clamp
- fiber clamp
- various size dual/single cantilever clamps
- small sample 3-point bending clamp
- compression clamp
- shear sandwich clamp
- penetration clamp
- submersion compression clamp
- submersion film/fiber clamp.

Turn to Chapter 5 for details on the installation and operation of these clamps.

Specifications

Tables 1.2, 1.3 and 1.4 contain information about the DMA's specifications and temperature control. Table 1.5 contains specifications for the ACA. Only values with tolerances or limits are guaranteed data. Values without tolerances are for information only.

Table 1.2
DMA 2980 Instrument Specifications

Dimensions	Depth: 17 in. (43.2 cm) Width: 26 in. (66 cm) Height: (furnace open) 28 in. (71 cm) (furnace closed) 22 in. (56 cm)
Weight (approx.)	85 lbs (38.6 kg)
Power	120 Vac 50/60 Hz 7 amps
Fuses	4 amps Slow Blow (M) 8 amps Slow Blow (M)
Temperature Range	-145 to 600°C
Sample Length	2 in. (50 mm) maximum
Sample Width	0.6 in. (15 mm) maximum
Sample Thickness	5 to 10 mm
Displacement Range	1.0 in. (25 mm)
Loading	0.001 to 18 N
Atmosphere	Controlled flow with inert gases or air

Table 1.3 Temperature Control Specifications

Temperature Range	-145 to 600°C
Programmed Heating Rate	0.1 to 50°C/min
Cooling Rate	0.1 to 10°C/min
Temperature Reproducibility	+/-2°C
Isothermal Stability	+/-0.1°C above 50°C +/-1.0°C below 50°C

Table 1.4 Experimental Specifications

Modulus Range	10^3 Pa to 3 x 10^{12} Pa
Modulus Precision	+/- 1%
Frequency Range	0.01 to 200 Hz
Maximum Force	18 N
Minimum Preload Force	0.001 N
Tan Delta Range	0.0001 to 10

(table continued)

Table 1.4 Experimental Specifications (cont'd)

Tan Delta Resolution	0.00001
Tan Delta Sensitivity	0.0001
Dynamic Deformation	+/- 0.5 to 10,000 micrometers
Strain Resolution	1 nanometer

Table 1.5 ACA Specifications

Dimensions ACA	Depth: 15 in. (38 cm) Width: 15 in. (38 cm) Height: 9 in. (23 cm)
Weight (approx.)	22 lbs (10 kg)
Power	120 Vac 50/60 Hz
Fuse	2 amps Slow Blow (M)

DMA 2980 Output Values

You can obtain the following output data from running experiments on the DMA 2980.

Multifrequency and Multistrain Modes:

- Storage modulus
- Loss modulus
- Tan delta
- Complex viscosity
- Dynamic viscosity
- Storage compliance
- Loss compliance
- Stress
- % Strain
- Amplitude
- Frequency
- Temperature
- Position
- Static force (preload force)
- Time.

Creep Mode:

- Creep compliance
- % Strain
- % Strain Recovery
- Stress
- Static force (preload force)
- Temperature
- Time
- Decay time
- Displacement
- Recoverable compliance.

Stress Relaxation

- Stress relaxation modulus
- % Strain
- % Strain Recovery
- Stress
- Static force (preload force)
- Temperature
- Time
- Decay time
- Displacement.

DMA Controlled Force

- Dimension change
- Static force (preload force)
- Position
- Stress
- % Strain
- Temperature
- Time.

DMA Isostrain

- Dimension change
- Position
- Static force (preload force)
- Strain
- Stress
- Temperature
- Time.

Chapter 2: Installing the DMA 2980

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Installing the Instrument

Before shipment, the DMA 2980 instrument is tested and calibrated both electrically and mechanically, so that it is ready for operation after it has been installed. Installation involves the procedures described in this chapter:

- Unpacking the instrument components and accessory kit.

NOTE: The instrument's cables and lines must be connected before the shipping material is removed. The procedures used to remove this material require that the instrument be powered up.

- Inspecting the system for shipping damage and missing parts
- Choosing a location for the DMA
- Connecting the air bearing gas and air cool gas lines, accessories, and power cable
- Assembling the DMA
- Connecting the DMA to the TA Instruments controller
- Removing the shipping material.

If you wish to have your instrument installed by a TA Instruments Service Representative, contact your local representative (see Appendix A) for an installation appointment when you receive your instrument.

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

Choosing a Location

Because of the sensitivity of DMA experiments, it is important to choose a location for the instrument using the following guidelines:

In . . . a temperature-controlled area.
. . . a clean environment.
. . . an area with ample working and ventilation space around the instrument. (Refer to the technical specifications in Chapter 1 for the instrument's dimensions.)

On . . . a **stable, vibration-free,** heat and flame resistant work surface.

NOTE: Placing the DMA on a **stable, vibration-free** work surface is <u>very</u> important to instrument performance.

Near . . . a power outlet (120 Vac, 50 or 60 Hz, 10 amps). A step up/down line transformer may be required if the unit is operated from a higher or lower line voltage.
. . . your TA Instruments thermal analysis controller.
. . . sources of compressed lab air and purge gas supply for use during cooling and subambient experiments.

♦ CAUTION: **Your air source must be clean, dry, and oil-free to ensure the proper operation of the DMA 2980.**

Away
from. . . dusty environments.
. . . exposure to direct sunlight.
. . . direct air drafts (fans, room air ducts).
. . . poorly ventilated areas
. . . flammable material that may come in contact with the furnace assembly.

After you have decided on the location for your instrument, refer to the next several sections to unpack and install the DMA.

♦ ***CAUTION:***

Drying out the instrument may be needed, if it has been exposed to humid conditions. Certain ceramic materials used in this equipment may absorb moisture, causing leakage currents to exceed those specified in the applicable standards until moisture is eliminated. It is important to be certain that the instrument ground is adequately connected to the facilities ground for safe operation.

Run the following method to dry out the instrument (refer to Chapter 4 for further information):

1 Ramp at 10°C/min to 400°C
2 Isothermal for 30 min.

Unpacking/Repacking the DMA

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

Inspecting the System

When you receive your DMA, look over the instrument and shipping container carefully for signs of shipping damage. 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.

A list of TA Instruments offices can be found in Appendix A of this manual.

Unpacking the DMA

Refer to Figures 2.1 to 2.3 while unpacking your instrument.

!WARNING

Have an assistant help you unpack this unit. Do not attempt to do this alone.

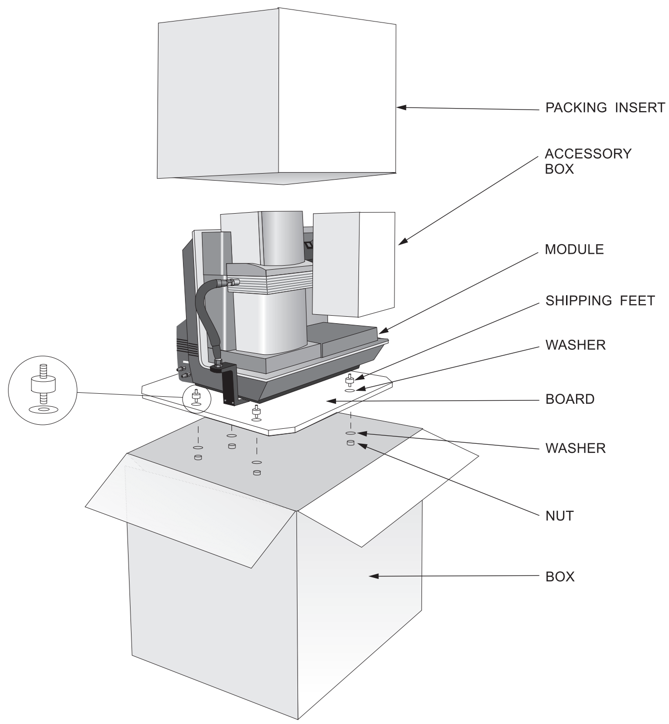

Figure 2.1 Shipping Boxes

1. Open the shipping carton and remove the accessory box.

2. Remove the cardboard packing insert.

3. Stand at one end of the box with your assistant facing you at the other end. Lift your end of the unit out of the box as your assistant lifts his/her end.

4. Place the unit on a lab bench with one side hanging over the edge of the bench (see Figure 2.2). **Someone must be holding onto the unit at all times while it is in this position.**

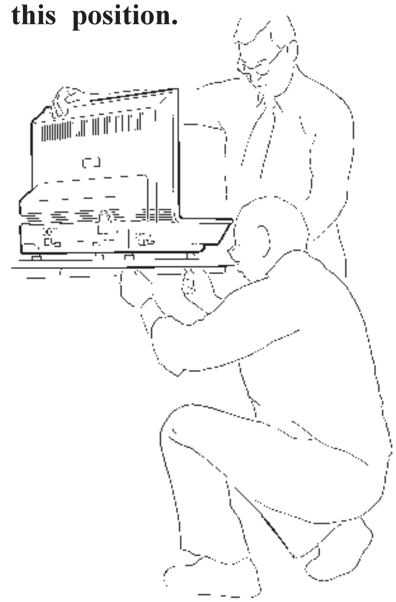

Figure 2.2 Removing the Plywood Board

5. While your assistant holds the unit, use a wrench to remove the nuts and washers from the bottom. Then lift and rotate the unit so that the other end hangs over the edge of the bench. **Someone must hold onto the unit at all times while it is in this position.** While your assistant holds the unit, remove the nuts and washers from the other side. A total of five nuts should be removed.

6. Slide the unit completely onto the lab bench. Have your assistant hold one side up while you unscrew and remove the black rubber vibration mounts from the bottom. Then rotate the unit and remove the vibration mounts from the other side in the same manner.

7. Have your assistant lift the entire unit while you slide the plywood board out from under it.

8. Remove the mounting feet from the accessory kit.

9. Have your assistant lift one side of the unit while you install the mounting feet (see Figure 2.3). Rotate the unit and install the remaining mounting feet in the same manner. A total of four mounting feet are needed.

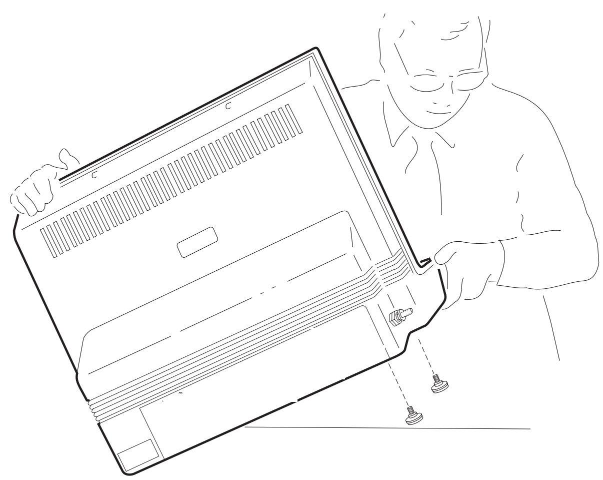

Figure 2.3 Installing the Mounting Feet

Connecting Cables and Gas Lines

In order to connect the cables and gas lines, you will need to have access to the instrument's rear panel.*

NOTE:

* All directional descriptions for this section are written on the assumption that you are facing the back of the instrument.

NOTE:

Connect all cables before connecting the power cords to outlets. Tighten the screws on all computer cables.

♦ ***CAUTION:***

When plugging or unplugging power cords, always handle them by the plugs, not by the cords.

! WARNING

Protect power and communications cable paths. Do not create tripping hazards by laying them across accessways.

GPIB Cable

1. Locate the GPIB connector on the right rear of the instrument (see Figure 2.4).

2. Connect the GPIB cable to the GPIB connector. The GPIB cable is the only cable that fits into this connector.

3. Tighten the hold-down screws on the connector.

4. Connect the other end of the GPIB cable to the controller or to the GPIB cable of another TA Instruments instrument connected to the controller.

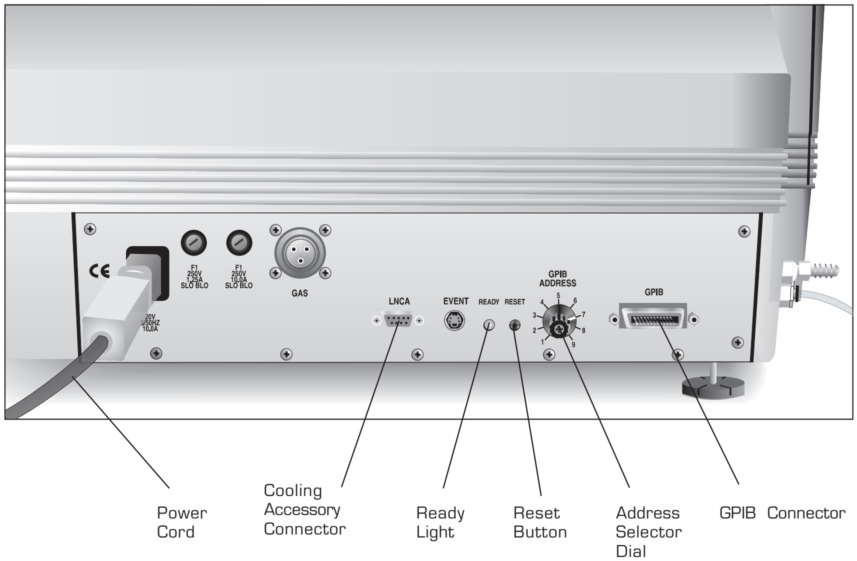

Figure 2.4
DMA Connector Panel

5. Use the GPIB address selector dial to set an address for the DMA. Choose an address that is not being used by other modules connected to the same controller. The figure seen on the next page shows an instrument address of 7.

NOTE:

If you have a multiple instrument system, each instrument must have a different address.

If you change the address after the DMA is powered on, you must press the DMA Reset button to enter the new address. Wait 30 seconds after releasing the Reset button; the green Ready light should begin to glow steadily. Reconfigure the instrument with the controller to bring the instrument back online.

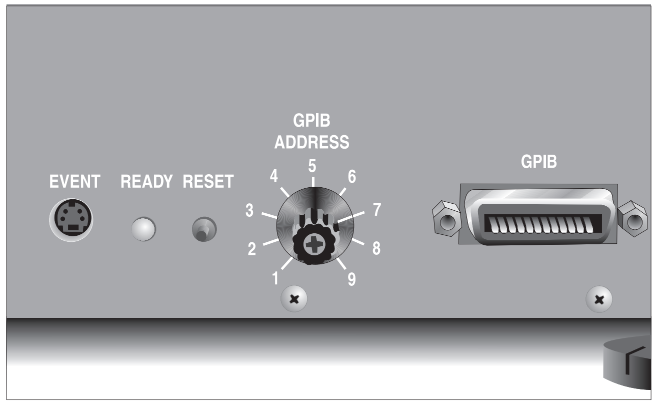

Figure 2.5
Address Selector Dial

Air Filter Regulator Assembly

♦ **CAUTION:** **Your air source must be clean, dry, and oil-free to ensure the proper operation of the DMA 2980. See page 2-18 for details.**

The air filter regulator assembly, shown in the figure below, is used with the instrument to help supply clean air bearing gas to the DMA. The air filter regulator helps to filter any oil, water, and particulates from the air bearing gas.

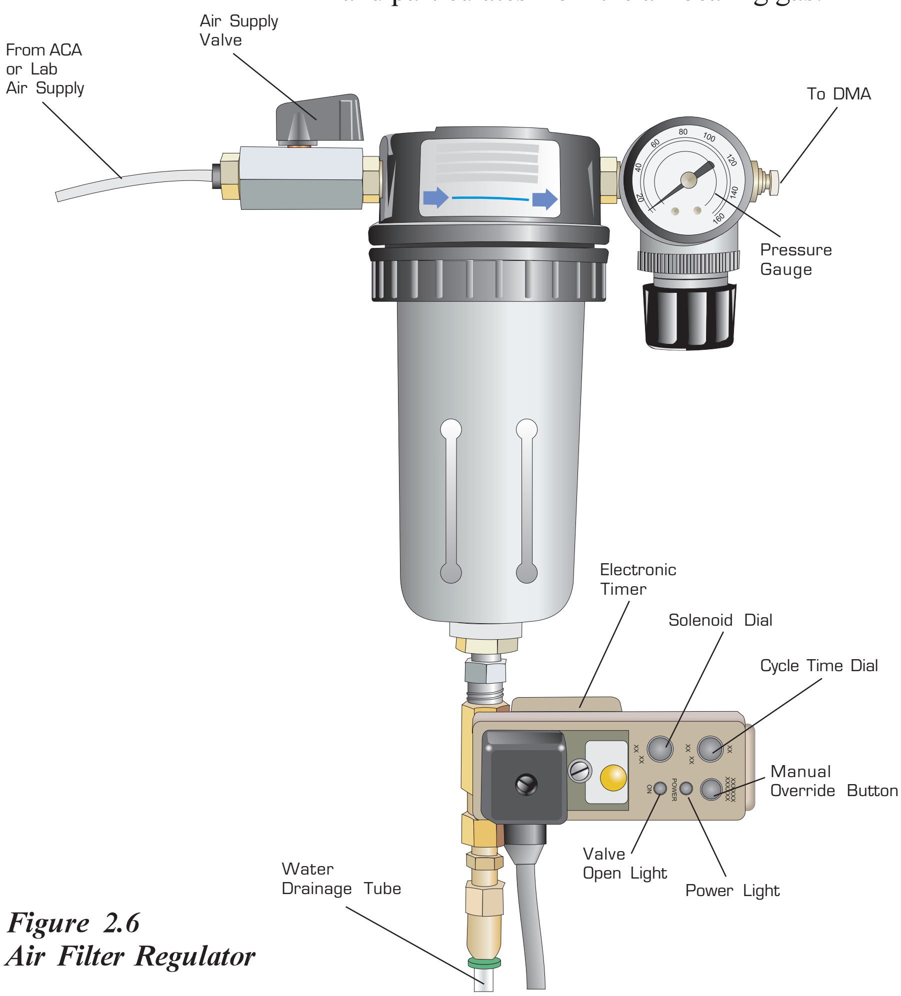

Figure 2.6
Air Filter Regulator

The air source supplying the air filter regulator can come from a central laboratory supply or from the TA Instruments Air Compressor Accessory (ACA). See pages 1-15 to 1-17 for more information about the ACA, which is an oil-less compressor.

The air filter regulator comes with an electronic timer (see Figure 2.6) that automatically drains any accumulated water from the regulator. The timer has two adjustable dials, a manual override button, a power indicator light, and a valve open light.

- The *cycle time dial* is used to set the amount of time between drain cycles. The recommended setting is one (1) minute between drain cycles.
- The *solenoid dial* is used to set the amount of time that the solenoid valve stays open during a drain cycle. The recommended setting is one (1) second.
- The *manual override button* is used to open the solenoid valve any time that it is needed.
- The *power indicator light* shows that power is being supplied to the electronic timer.
- The *valve open light* comes on when the solenoid valve opens to drain the filter.

Follow the manufacturer's recommendation for filter inlet pressure. The filter regulator should be mounted on a vertical surface such as the wall near the instrument or mounted on the side of a lab bench, as desired.

♦ ***CAUTION:***

For optimum performance, do not place the ACA on the same laboratory surface as the DMA. The vibration from the ACA may affect the DMA's performance.

To connect the air filter regulator to the instrument, follow these instructions:

1. Determine the air source that you will be using for the DMA. The fitting that you need on the filter depends upon which air source you intend to use as follows:

   ACA air source: The air filter regulator is preassembled with a Parker quick-connect fitting in the valve on the left side of the regulator (see Figure 2.6). This fitting is used with the tubing supplied with the ACA, therefore, the fitting will not need to be removed.

   Lab air source: You will need to first remove the Parker quick-connect fitting in the valve on the left side of the regulator as seen in Figure 2.6. Then install the Swagelok fitting that is included in the accessory kit.

2. Place the air supply valve in the OFF position.

3. Connect the appropriate tubing, depending upon your air source, to the left side of the air filter regulator as follows:

   ACA air source: Push the 1/8-inch (3.1 mm) tubing from the ACA (see the figure on next page), into the Parker fitting. Insert the tubing into the fitting until it cannot go in any further.

Lab air source: Connect 1/4-inch (1.5 mm) tubing from the air supply source to the Swagelok fitting on the air filter regulator.

4. Push one end of the thin 1/8-inch (3.1 mm) tubing into the air filter regulator fitting. Insert the tubing into the fitting until it cannot go in any further.

Figure 2.7 Air Compressor Accessory

5. Locate the air bearing gas fitting on the right rear of the DMA instrument.

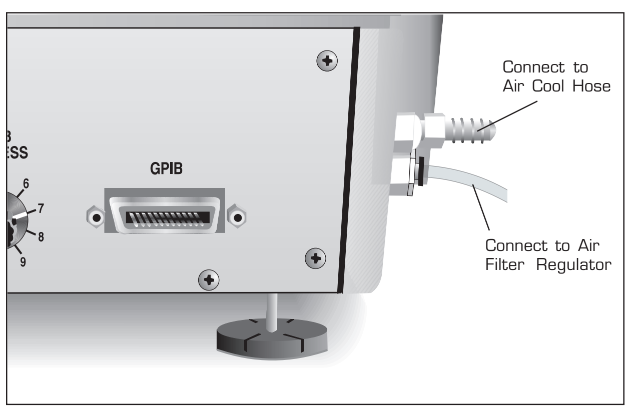

Figure 2.8 Fitting on DMA for Air Bearing Gas

! WARNING

Use of an explosive or corrosive gas as an air bearing gas is dangerous and will damage the DMA instrument. Use air or an inert gas (such as nitrogen) only, for the air bearing gas.

6. Push the opposite end of the thin 1/8-inch (3.1 mm) tubing, which is connected to the air filter regulator, into the Legris fitting on the right side of the back of the DMA. Insert the tubing into the fitting until it cannot go in any further.

7. Plug the electronic timer into a 110V power source to operate.

8. Set the dials on the electronic timer to the desired time between drain cycles and the desired time the solenoid valve remains open during the cycle. See page 2-14 for recommended settings.

9. Ensure that the filter outlet pressure is set to 420 to 455 kPa (60 to 65 psi).

   The air bearing gas also serves as the furnace purge. If an inert atmosphere is needed, you must use inert gas for the air bearings. The air bearings use gas at the rate of approximately 2 liters per minute.

   The purge gas flows through the instrument and is channeled internally to the sample.

10. Turn the air supply valve on the air filter regulator to the ON position. A solenoid valve inside the DMA 2980 controls the flow to the air bearings.

11. *If you are using the Air Compressor Accessory connected to your air filter regulator*:

    a. Check the pressure gauge on the regulator before turning on the ACA.

       If the pressure gauge on the air filter regulator reads more than 10 psig, release the pressure by pressing the manual override button on the electronic timer.

       If you try to turn on the ACA with more than 10 psig pressure in the system, the ACA will draw an excessive amount of current and may overload its fuse.

    b. Plug in the power cord on the ACA and turn it on, after you have performed step 11a.

Proper installation and maintenance of the air filter regulator is important for the performance and life of the DMA air bearings. (See Chapter 7 for information on maintaining the air filter regulator.) An efficient system ensures minimum pressure loss and removal of contaminants such as water, oil, dirt, rust, and other foreign materials. TA Instruments recommends the following minimum criteria for the air being supplied to the air filter regulator:

- Oil and liquid water = < 2 mg per cubic meter
- Water vapor dew point at 100 PSIG = 32°F

NOTE: If you are using a desiccant dryer, it is best to install it after the air filter regulator.

Air Cool Line

One way to rapidly cool the DMA furnace to room temperature is by using air cooling or the GCA. Air cooling is used only when the Gas Cooling Accessory (GCA) is not connected to the DMA 2980. (See the GCA manual for further information on the GCA.)

Follow the procedure below to install the air cool line:

1. Locate the cooling gas fitting, a 6.2 mm (1/4-inch) compression fitting on the left side of the DMA cabinet back, marked with a 125 psi (860 kPa) maximum warning label (see the figure below).

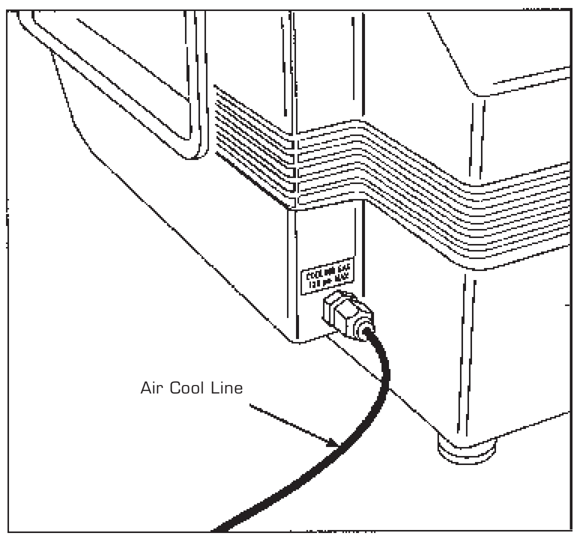

Figure 2.9 DMA Air Cool Fitting

2. Make sure your compressed air source is dry, filtered, and regulated to between 170 and 830 kPa (25 and 120 psi).

3. Connect a compressed lab air line to the air cool fitting.

4. Attach the Cooling Hose Accessory (shown in Figure 2.10) as follows:

   a. Note the shape of the cooling hose shown in Figure 2.10. Before you mount the hose, bend it to the shape shown while holding it up to the instrument to line it up with and obtain the proper distance between the fittings. The hose will retain the shape. (The purpose of this step is to eliminate any stress on the instrument frame that can be caused by bending the hose after installation.)

   b. Use a wrench to connect the cooling hose to the fitting on the top of the bracket and to the fitting going into the instrument.

5. Attach a barbed fitting to the cooling hose accessory inlet shown in Figure 2.10.

6. Locate the air cool outlet fitting, which is next to the air regulator fitting on the side of the DMA 2980 (see Figure 2.8).

7. Connect 3/8-inch (9.5 mm) ID tubing from the air cool outlet to the barbed fitting on the cooling hose accessory inlet (installed in step 5). If the Gas Cooling Accessory (GCA) is connected to the DMA, you must remove the GCA feed hose before connecting the tubing (see the GCA manual).

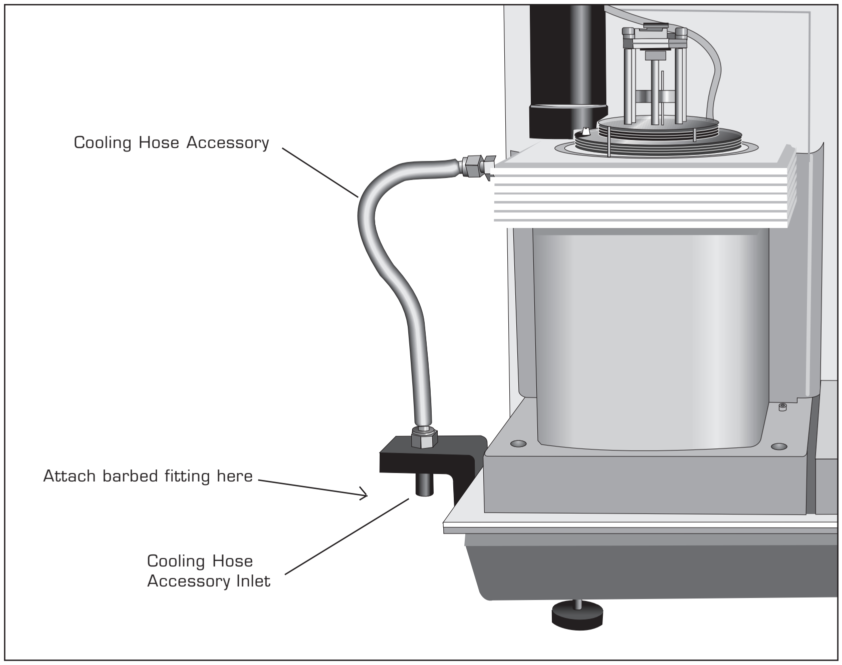

Figure 2.10
Cooling Hose
Accessory Inlet

Power Cable

Use the following steps to connect the power cable to the instrument.

NOTE: Connect all other cables and gas lines before connecting the power cable to a wall outlet.

1. Turn the instrument POWER switch (see the figure below) to the OFF (O) position.

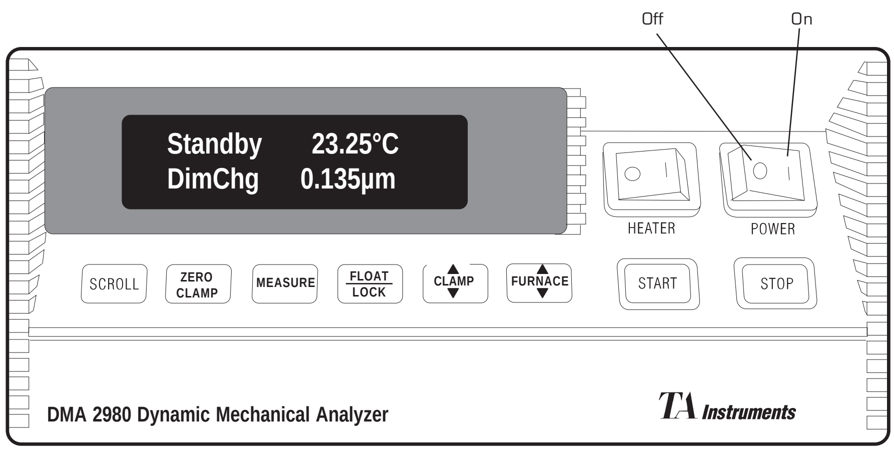

Figure 2.11
Front Panel of DMA
Showing POWER Switch

2. Plug the power cable into the DMA.

♦ ***CAUTION:*** **Before plugging the DMA power cable into the wall outlet, make sure the instrument is compatible with the line voltage. Check the label on the back of the unit to verify the voltage.**

3. Plug the power cable into the wall outlet.

Starting the DMA

The DMA will need to be powered up in order to perform several of the steps used in removing some of the shipping material. Follow these instructions to properly start the DMA.

1. Check all connections between the DMA and the controller. Make sure each component is plugged into the correct connector.

2. Turn the instrument POWER switch to the ON (1) position. The instrument will run an internal confidence test each time you turn on the power.

3. Watch the instrument display during the confidence test for any messages that may be indicated.

After the confidence test has finished, the screen will briefly display the system status, indicating the amount of data storage memory available and the GPIB address. Next follows the copyright display, and then the standby display.

4. Bring the instrument online with the TA controller.

NOTE: Allow the DMA to warm up for at least 15 minutes before performing an experiment. The Signal Display window on the controller should display "Frame Temperature: OK" and "Air Pressure: OK."

Shutting Down the DMA

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

- All of the components of your thermal analysis system are designed to be powered up and left on for long periods.
- The electronics of the DMA and the controller perform more reliably if power fluctuations are minimized.

For these reasons, turning the system components on and off frequently is discouraged. However, if you need to power down the DMA, follow the instructions listed below. When the DMA is powered off, the drive shaft should not move. As a precaution, you can:

(a) Move the drive shaft to the bottom (25 mm) position and press STOP or (b) Install a stiff sample or the shipping clamp and press STOP.

To power down your DMA for any reason, always press the STOP key first (this will save the position calibration). If the drive shaft was active, wait five seconds for the drive to completely stop. Then press both the HEATER and POWER switches to the OFF (O) position and turn off the air bearing gas.

Removing the Shipping Material

The DMA 2980 is shipped with several screws and a bracket to hold the instrument firmly in place and prevent damage. This shipping material must be removed as follows before the instrument can be used.

NOTE: The instrument's cables and lines must be connected before the shipping material is removed. The procedures used to remove this material require that the instrument be powered up.

1. Loosen the four 1/4-20 jackscrews (stand-offs), located under the dress cover, using a 3/16 Allen wrench. Turn the screws counterclockwise until they just touch the underside of the plastic cover. (When packing up the 2980, tighten all four screws evenly until they reach the bottom.)

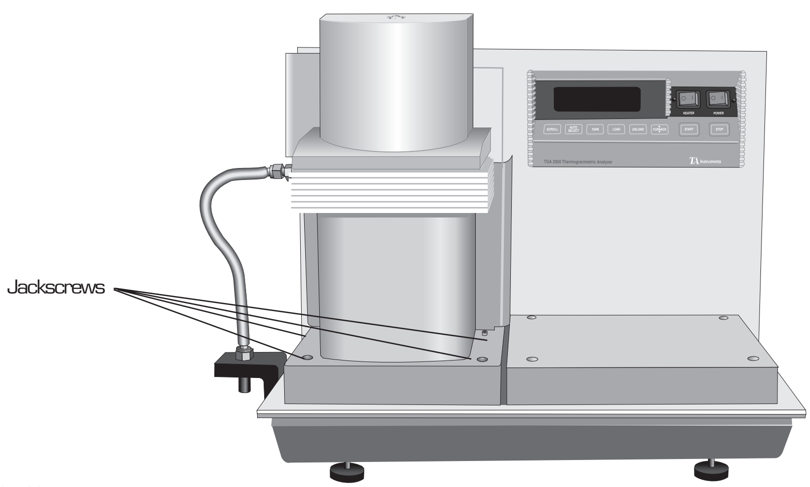

Figure 2.12
Location of Screws

2. Remove the large hex bolt holding the furnace in place. See the figure below.

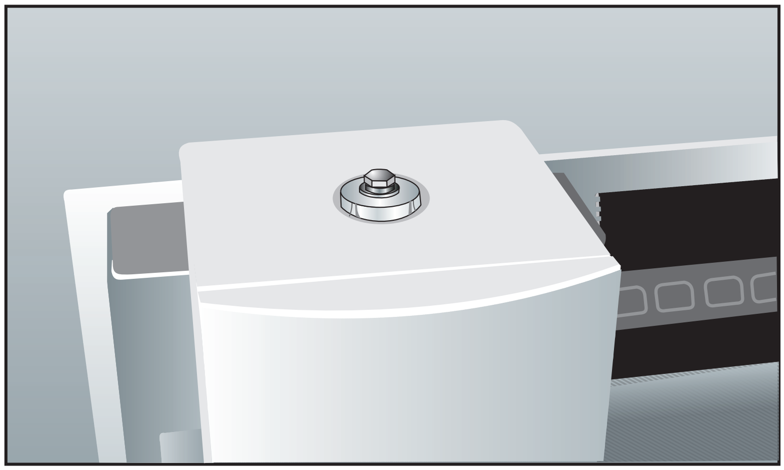

Figure 2.13 Furnace Shipping Nut and Bolt

3. Remove any packing material between the rear of the furnace and the back cabinet of the instrument.

4. Press the FURNACE key to raise the furnace.

5. Remove the blue shipping foam from the handrest.

6. Turn on the air filter regulator valve, which should already be connected, and confirm that the air pressure builds to 60 psi.

7. Check the **Signal Display** window on the controller and verify that the air pressure and frame heaters say “OK.” (The heaters take about 20 to 30 minutes to warm up after the POWER switch is turned on.)

8. Press the FLOAT/LOCK key on the keypad. You should hear the click of the air bearing solenoid.

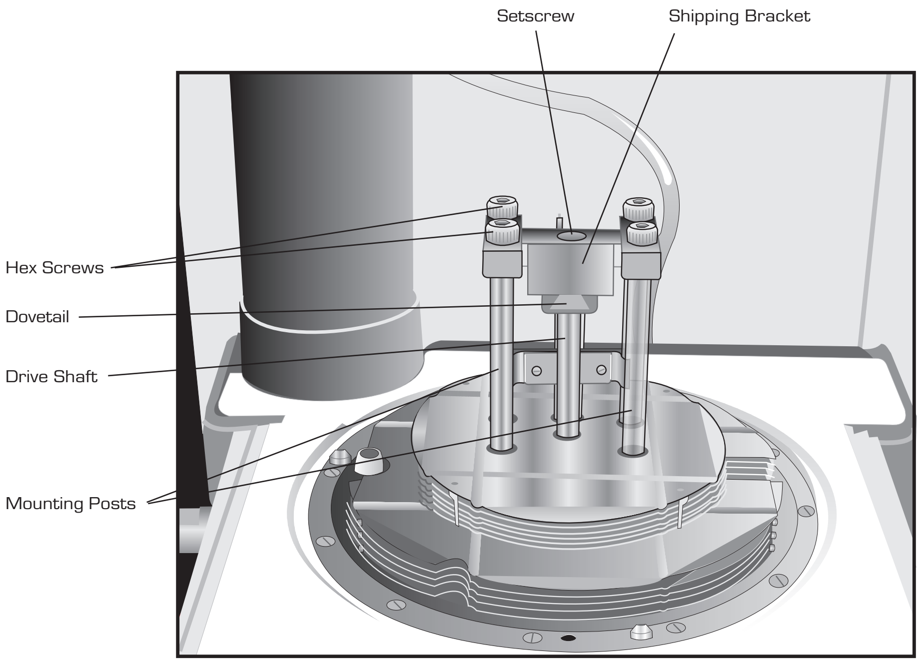

Figure 2.14 The Shipping Bracket

9. Loosen the four hex screws holding the shipping bracket in place (see the figure above).

10. Lift upwards on the shipping bracket to free it from the mounting posts, as shown in the figure on the next page. The drive shaft (slide) should move easily, DO NOT FORCE IT.

11. Press the STOP key after the bracket is off the mounting posts.

12. Loosen the setscrew located in the center of the shipping bracket and slide the dovetail out of the dovetail holder.

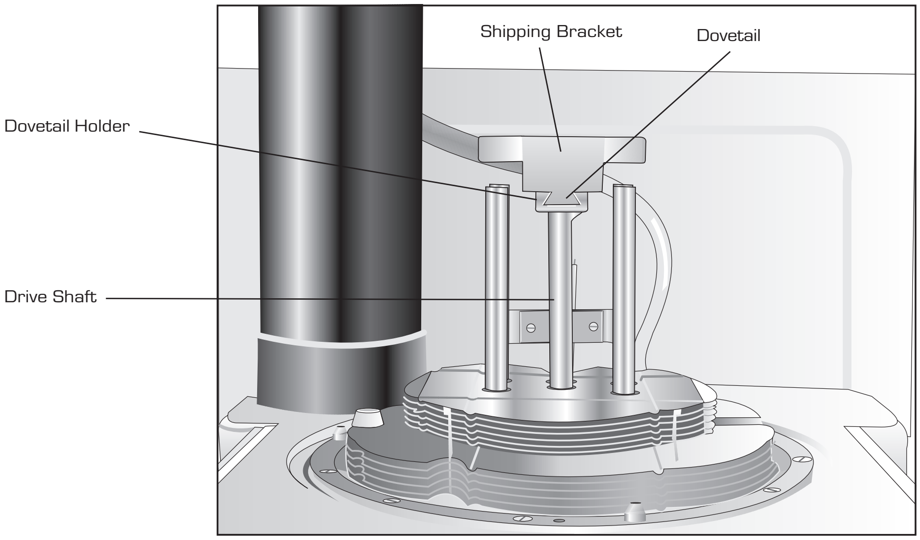

Figure 2.15 Lifting Off the Shipping Bracket

13. Install the desired clamp. See page 2-29 for installation of the standard dual cantilever clamp.

Repacking the DMA

To pack and ship your instrument, use the hardware retained during unpacking and reverse the previous instructions. Make sure that you press STOP after installing the shipping bracket to avoid losing the position calibration.

Installing the Single/Dual Cantilever Clamp

When you first receive the DMA 2980, a clamp will need to be installed. The procedures that follow explain the installation and removal of the single/dual cantilever clamp, which is the standard clamp used on the DMA. Later, if a different sample form is used, you can change to the appropriate clamp for the experiment. (Refer to Chapter 4 for details on clamp selection and Chapter 5 for installation of optional clamps.)

The single/dual cantilever clamps are used to analyze weak to moderately stiff samples. The samples are rigidly clamped using the cantilever clamps.

To install the single/dual cantilever clamp on the DMA 2980, follow these steps (refer to the figure on the next page for identification of parts):

1. Slide the dovetail of the moveable clamp into the dovetail holder of the drive shaft. Align the dovetail with the edge of the holder.

2. Insert the 1/16 hex key on an angle as shown in the figure on the next page to tighten the setscrew in the center of the moveable clamp. (Or use the hex key that has been shortened to allow the key to fit into the opening on the moveable clamp.) Do not overtighten the setscrew.

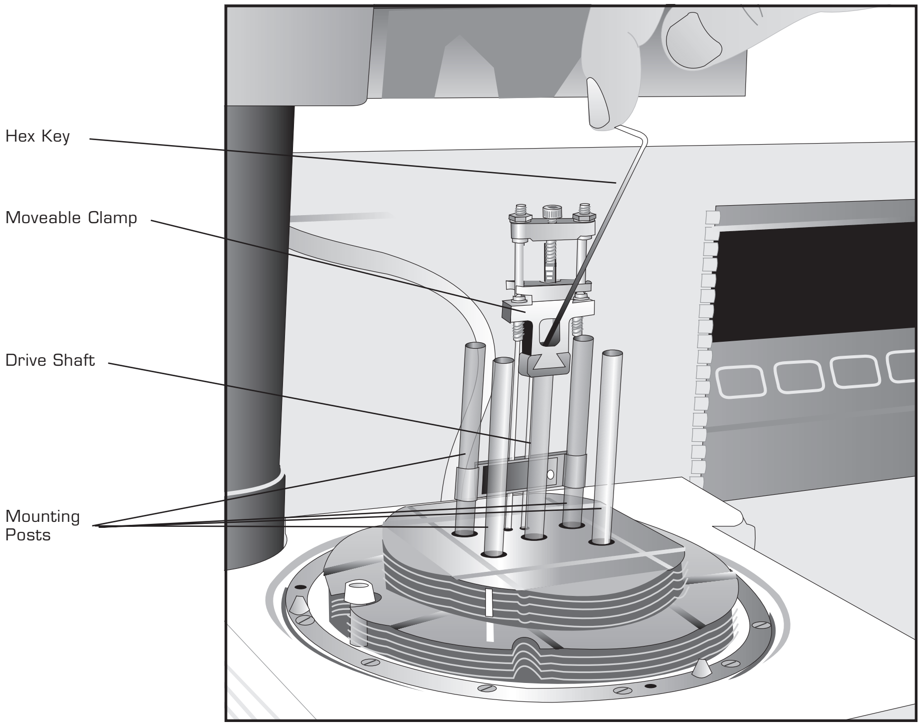

Figure 2.16
Installing the Single/Dual Cantilever Moveable Clamp

3. Lower the fixed clamp carefully over the moveable clamp. (You may need to reposition the thermocouple.)

4. Line up the fixed clamp with the mounting posts and tighten the four hex screws as shown in the figure on the next page.

5. Ensure that the moveable clamp is aligned so that it is parallel to and equally spaced between the fixed clamps. You may need to loosen the setscrew again to adjust the moveable clamp's position. Be sure to retighten the setscrew again, if you have loosened it.

6. Make sure that the appropriate clamp type and mode are selected on the controller.

7. Calibrate the clamp for clamp mass and clamp compliance (see Chapter 3 and the *Thermal Solutions User Reference Guide*).

8. Position the thermocouple so that it is close to, but not touching, the sample.

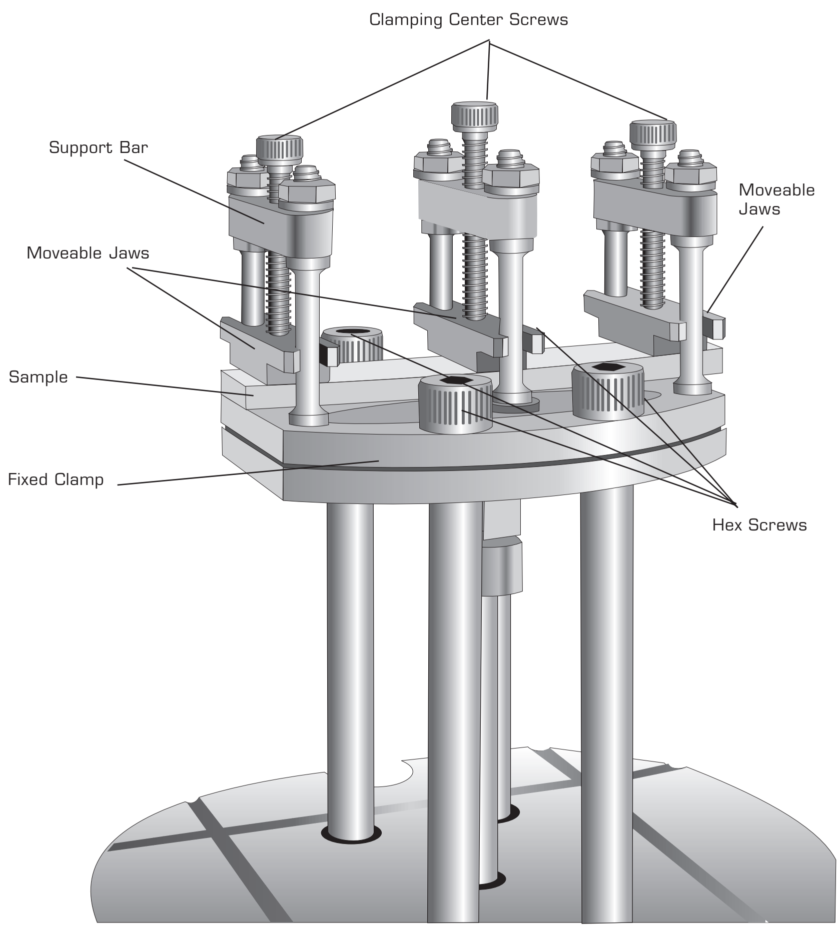

Figure 2.17 Single/Dual Cantilever Clamp Installed (Shown with Sample Mounted)

Removing the Single/Dual Cantilever Clamp

The following procedure is used to remove the clamp.

1. Press the FLOAT/LOCK key to lock the clamp in place.

2. Loosen, but do not remove, the four hex screws holding the fixed clamp on the mounting posts.

3. Lift the fixed clamp off the four supports.

4. Loosen the setscrew on the moveable clamp and then remove the clamp by sliding it out of the dovetail holder.

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Introduction

Calibration of the DMA 2980 is accomplished through the use of the *Thermal Solutions/ Advantage* Instrument Control software.

The types of calibration that are available for the DMA are:

- **Clamp calibration** calibrates the properties of a DMA sample clamp.
- **Position calibration** calibrates the absolute position of the drive shaft (and slide) as read by the optical encoder.
- **Instrument calibration** calibrates the instrument electronics, drive force, and dynamic performance.
- **Temperature calibration** calibrates the temperature of the DMA furnace.

This chapter explains the purpose and frequency of each type of DMA 2980 calibration. Consult the available documentation—the *Thermal Solutions/Advantage User Reference Guide* or the online help or online manual—for the software procedures used for calibration.

Determining When to Calibrate

Each DMA must be calibrated for accuracy, using the following basic guidelines.

- You need to perform the various *Clamp Calibrations* under the following circumstances:

  — when you change from one clamp type to another.

  — if the clamp does not properly float. This can be caused by residue on the clamps or by changes in ambient conditions. If you do not properly calibrate the clamp, it may affect the ability to make measurements as well as modulus values, particularly for very weak samples.

  — if you suspect incorrect sample length measurements.

  — if you have improper clamping or have entered an incorrect sample length, it may result in a high value for the compliance calculation. (*e.g.*, High compliance calculation can lead to modulus values that are too high for stiff materials).

  — after loading new instrument software.

- You need to perform the *Position Calibration* under the following circumstances:

  — when the DMA is moved or at least once a month. The purpose of this calibration is to calibrate the absolute position of the drive shaft (and slide) as read by the optical encoder.

  — if the probe will not float after you have performed a *Clamp Calibration*.

- You need to perform the *Instrument Calibrations* under the following circumstances:

  — when the instrument is moved.

  — if the force is not linear over the entire traveling distance of the moveable clamp, you may need to perform an *Instrument Force Calibration* and a *Clamp Calibration*. For example, the clamp floats normally in some positions, but not in other positions.

  — if the modulus and tan delta have unusual responses to changes in frequency (*e.g.*, there is a spread in the modulus in a non-transition zone), you may need to perform the *Instrument Dynamic* and *Electronics Calibrations*.

  — if the feed hose for the air cool or GCA is either removed or installed.

  — on a regular (monthly) basis. You will need to perform all of the *Instrument Calibrations*.

The calibration reports show the current calibration status and when the calibrations were last performed.

Clamp Calibration

When you install a clamp for the first time or change from one clamp to another on the DMA 2980, you will need to perform the following steps:

1. Install the desired clamp.

♦ ***CAUTION:*** **Make sure that you have installed both the fixed and moveable clamps before performing clamp mass calibration.**

2. Select the appropriate clamp type on the *Thermal Solutions/Advantage* **Mode Parameters** window.

3. Perform a clamp calibration. Clamp calibration may involve up to three steps (mass, zero, and compliance), depending on the clamp installed. Each of the three steps is briefly described on the following pages. Consult the *Thermal Solutions/Advantage* documentation for details.

NOTE: See the appropriate section in the *Thermal Solutions/Advantage* documentation for the complete calibration instructions. Be sure to perform all of the calibration steps.

Clamp Mass Calibration (Step 1)

This type of calibration is performed to allow the instrument to compensate for the mass of a specific clamp to ensure accurate force measurements. Clamp Mass Calibration is Step 1 in the *Thermal Solutions/Advantage* DMA clamp calibration procedure and is performed for all clamps.

NOTE: The air bearings should be energized (*e.g.*, probe floating) for 20 minutes before performing a Clamp Mass Calibration.

You can run a check to see whether your clamp mass needs calibration by performing a small test. Press the FLOAT key to release (float) the clamp. Manually position the clamp in the middle of its range and release it. The clamp should maintain its position or drift slowly up or down. If the clamp rapidly sinks or rises, the clamp mass requires calibration as described in the *Thermal Solutions/Advantage* documentation.

Clamp Zero Calibration (Step 2)

Clamp Zero calibration, which is Step 2 in the DMA clamp calibration procedure, is needed to determine the point of zero sample length for automated sample length (tension) or thickness (compression and penetration) measurement. Clamp Zero calibration is performed when you install a film or fiber tension clamp, a penetration clamp, or a compression clamp. It is not performed when you install a single or dual cantilever clamp, a shear sandwich clamp, or a 3-point bending clamp.

- An offset gauge is used with the film or fiber tension clamps. Measure the length of the gauge, then mount it, and enter the length into the *Thermal Solutions/ Advantage* Clamp Calibration (Step 2) window.

- No offset gauge is used for calibrating the compression or penetration clamps. In this case, enter a zero (0) for this parameter.

When you are recalibrating clamp zero after the offset gauge length is entered (if applicable), you can use the CLAMP ZERO key on the instrument keypad. It is important to note that the gauge length will be reset to zero any time the instrument mode is changed. To perform the Clamp Zero calibration after an initial calibration has been performed, mount the desired clamp and press the CLAMP ZERO key on the instrument keypad.

Compliance Calibration (Step 3)

Compliance calibration used to measure the flexibility of a clamp and calibrates the instrument to that flexibility. There are three different situations that occur, depending upon the type of clamp you have selected:

- Compliance calibration is performed when you install a single or dual cantilever clamp, 3-point bending clamp, or film tension clamp using a rigid steel sample with known dimensions.

- Compliance calibration is also performed, but without a sample, when you install a penetration or compression clamp. This is because the moving and fixed portions of the clamp directly contact each other.

- It is not performed when you install a shear sandwich or fiber tension clamp.

See the *Thermal Solutions/Advantage* documentation for instructions.

Position Calibration

Position calibration must be done when the DMA is moved or at least once a month. The purpose of this calibration is to calibrate the absolute position of the drive shaft (and slide) as read by the optical encoder.

Clamp Check

You can run the following check to see whether your clamp position needs calibration by performing a small test. Press the FLOAT key to release (float) the clamp.

- *For all clamps except penetration, submersion film/fiber, and compression:* Manually move the clamp to the top of the travel. The position signal on the instrument display should read 0 ± 0.5 mm. Move the clamp to the bottom of the travel. The position signal should read 25.0 ± 0.5 mm. If this is not the case, recalibrate the position as directed in the *Thermal Solutions/Advantage* documentation.

- *For all clamps:* Manually position the clamp in the middle of its range of travel and release it. The clamp should maintain its position or *slowly* drift up or down. If the clamp rapidly sinks or rises, and the clamp mass has already been calibrated, the clamp position requires calibration as directed in the *Thermal Solutions/Advantage* documentation.

The instrument stores the position in memory until the next calibration. In the event that the instrument loses the stored position, you will see one of these errors: Error 255, 274, 300, or 322.

NOTE: To retain the Position calibration, make sure that you press the STOP key and wait several seconds before you reset or turn off the instrument.

Instrument Calibrations

The following types of calibrations need to be done when the DMA is moved or at least once a month:

- Electronics calibration
- Force calibration (balance and weight)
- Dynamic calibration.

Electronics Calibration

This procedure calibrates the instrument's electronics and drive motor over the entire frequency range of the instrument. It automatically removes the air to lock the slide (drive shaft) and applies a static force (preload force) to the motor as a calibration reference. The instrument then uses a series of frequencies to perform the calibration.

Before you perform the electronics calibration, you will need to reinstall the shipping bracket on the instrument following these steps:

NOTE: These procedures are the reverse of those used to remove the shipping bracket, which are found on pages 2-25 to 2-27.

1. Remove any fixed and moveable clamps from the instrument.

2. Press the **FLOAT/LOCK** key until the drive shaft can move up and down freely. Slide the shipping bracket dovetail into the dovetail holder. Do not tighten the setscrew yet.

3. Lower the bracket down onto the mounting posts. See the figure below.

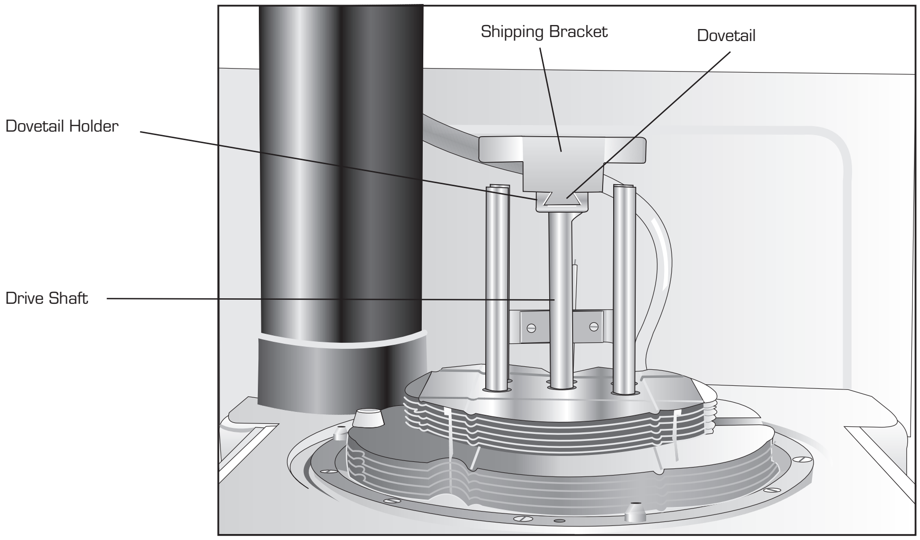

Figure 3.1
Positioning the Shipping Bracket

4. Press downwards on the shipping bracket to position it against the mounting posts, as shown in the figure above. The drive shaft (slide) should move easily, DO NOT FORCE IT. (You may need to reposition the dovetail in the dovetail holder in order to align the shipping bracket with the posts.)

5. Tighten the four hex screws to hold the shipping bracket in place (see the figure on the next page).

6. Tighten the setscrew, located in the center of the shipping bracket, to lock the drive shaft into position.

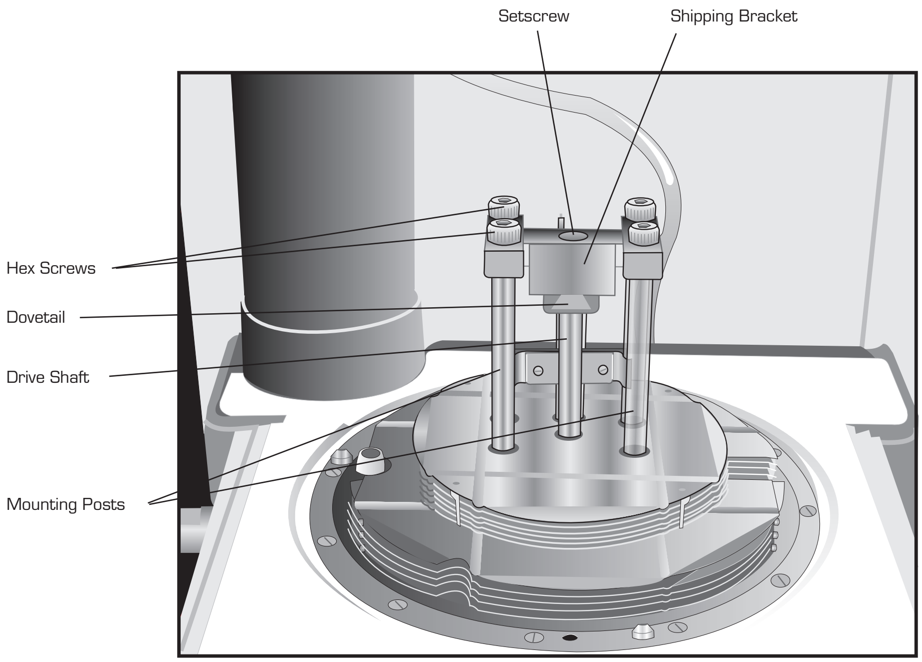

Figure 3.2
Mounting the
Shipping Bracket

7. Follow the instructions found in the *Thermal Solutions/Advantage* documentation to calibrate the DMA electronics.

Force Calibration (Balance and Weight)

Force calibration is used to adjust the force exerted by the clamp on the sample and the force registered by the instrument as the experiment proceeds. There are two steps in the force calibration—**balance (Step 1)** and **weight (Step 2)** calibration. This calibration must be done in the following situations:

- When the DMA is moved

  or

- At least once a month.

See the appropriate section in the *Thermal Solutions/Advantage* documentation for the calibration instructions.

Dynamic Calibration

Dynamic calibration is used to measure several samples of known stiffness and loss to characterize the dynamic performance of the instrument. Dynamic calibration must be done in the following situations:

- When the DMA is moved

- When the feed hose for the GCA or Air Cool is either removed or installed

  or

- At least once a month.

NOTE: Depending on the amount of compliance samples that you use, Dynamic calibration can take from one to two hours for completion.

See the appropriate section in the *Thermal Solutions/Advantage* documentation for the calibration instructions.

Temperature Transition

DMA is a very popular and powerful technique for measuring transitions in materials because it is sensitive to side-chain or main-chain motions and local mode relaxations in polymers that can not be detected in DSC (differential scanning calorimetry). Transitions in materials can be defined by the extrapolated onset temperature of E', or as the peak temperatures in E" or tan δ curves (see Figure 3.3). Therefore, DMA experiments primarily involve monitoring the changes in the viscoelastic parameters of E', E", and/or tan δ, with changing temperature. Because transitions in materials are defined by the temperature at which they are observed, it is important to understand the dynamics of temperature in the DMA 2980 when conducting dynamic mechanical measurements.

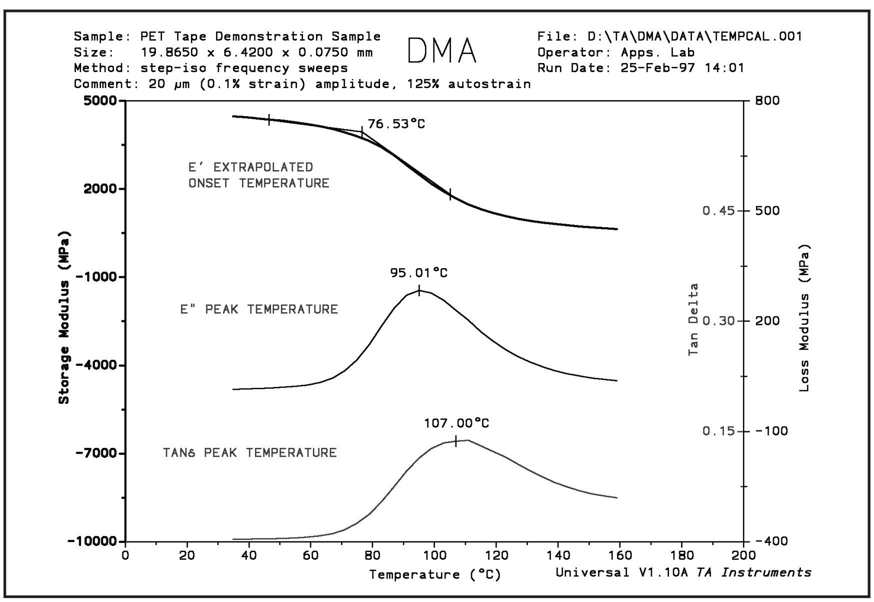

Figure 3.3
DMA Transitions

When you analyze samples on the DMA, the samples are held in position using the clamps explained earlier in this manual. These clamps contribute additional mass to the system and cause temperature lags between the furnace temperature and the sample temperature. The extent of these lags can be determined and compensated depending on the temperature profile employed. Temperature profiles, or methods, in the DMA can programmed in two ways: (1) step-and-hold test, or (2) temperature ramp test.

Step-and-Hold Test

To perform a step-and-hold test, you should set up a method with a start temperature, end temperature, and temperature step size. Hold the sample isothermally at each step for a defined length of time, prior to conducting the measurement.

The step-and-hold test has several advatages including elimination of temperature lag between the furnace and sample, and the ability to use a wider selection of frequencies without the temperature changing during a frequency sweep.

The disadvantages of step-and-hold are longer analysis times as the sample size increases (requiring longer equilibration ("soak") times) or as the number of steps across a temperature region increases (either because the region is large or because an improved resolution of events is desired). It may be better to run low frequencies with the step-and-hold test because, when you are using ramps, the temperature can change a lot over the duration of the measurement.

NOTE: Step-and-hold testing is recommended for time–temperature superposition (TTS) studies.

Temperature Ramp Test

To perform a temperature ramp test, you should set up a method with a start temperature, end temperature, and rate of temperature change (e.g. 2°C/min). The instrument simultaneously ramps the temperature and sweeps the frequency(ies).

The advantage of this method is that testing times are shorter and data can be acquired continuously, which leads to well-defined transition peaks.

The disadvantage of this method is that the temperature lags between the sample and furnace will cause shifts in the observed transition temperatures.

When you perform a temperature ramp test, less than five frequencies are recommended. Those frequencies should be spaced close together and should be from the highest to the lowest (*e.g.*, 10, 5, 1 Hz). In addition, frequencies below 0.5 Hz are not recommended when you are ramping at the heating rates above 2°C/minute.

Testing Considerations

When conducting dynamic mechanical measurements using either temperature profile test already described, the following factors should be considered:

- *Clamping arrangement* —More massive clamps will cause larger temperature lags.
- *Heating/cooling rate*—In temperature ramp tests, the faster the heating/cooling rate, the larger the thermal gradient.
- *Sample size*—The larger the sample, the larger the thermal gradient.
- *Thermocouple position*—the further the thermocouple is from the sample, the larger the thermal lag.
- *Air bearing gas used*—the most common gases used are air and nitrogen. The thermal conductivity of air is slightly higher than nitrogen.
- *Frequency*—consider the oscillation frequency when designing a test, since transitions are frequency-dependent. Higher frequencies cause transitions to shift to higher temperatures (see Figure 3.4 below). Avoid using fast temperature ramp (5°C/min) rates and low frequencies (0.1 Hz) in a single test.

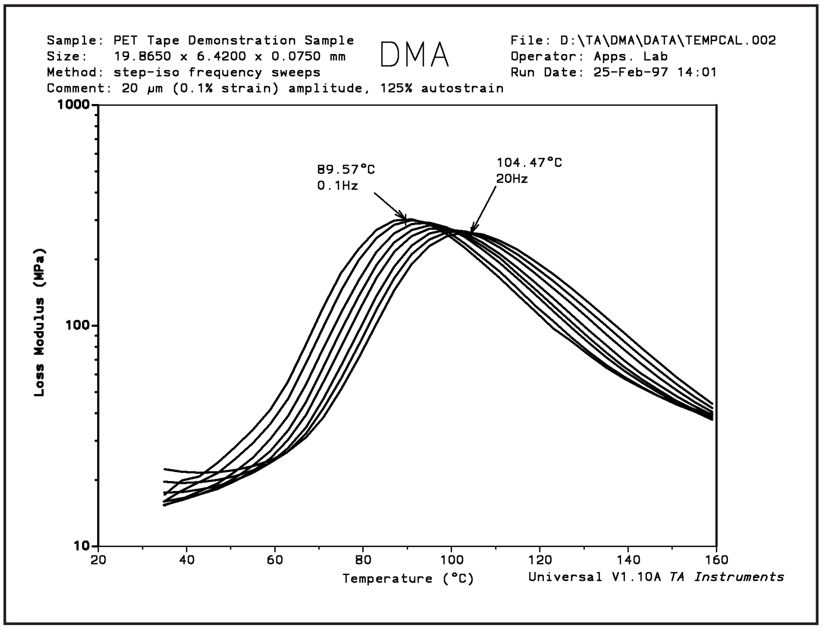

Figure 3.4
Effect of Frequency

DMA Temperature Calibration

When you analyze samples using the DMA, it is important that the measured sample temperature be as accurate as possible. Like any other thermal analyzer, the accuracy of the temperature measurement relies heavily on the temperature calibration. Temperature calibration must be performed routinely to ensure reproducible results.

Perform temperature calibration at least once a month. Calibrations should also be performed when changing clamps, temperature range of interest, or heating rates.

Studies indicate that the temperature difference between the sample and furnace is a function of heating rate (*i.e.*, faster heating rates cause larger temperature lags). In addition, under most conditions, the sample temperature equilibrates at the furnace temperature after it is held isothermal for several minutes. Therefore, you should correct the sample thermocouple two ways: for absolute temperature and for temperature lags encountered upon heating.

The procedure for temperature calibration of the DMA 2980 is detailed beginning on the next page.

Performing Temperature Calibration

Perform these steps for absolute temperature calibration of the DMA sample thermocouple:

1. Open the **Temperature calibration** window using *Thermal Solutions/Advantage* Instrument Control. (See the *Thermal Solutions/ Advantage* documentation for details.) Reset any previous calibrations from the instrument and select OK.

2. Install the desired clamp, then position the sample thermocouple so that the tip of the thermocouple is near the sample, but does not interfere with the motion of the clamps or slide.

3. Make sure that the DMA 2980 is held at ambient temperature, with the furnace open, for at least one hour before beginning the routine.

4. Measure and record the ambient temperature in the general area of the DMA. You can do this using a reliable external thermometer.

5. Note the ambient temperature reading of the DMA 2980.

6. Open the **Temperature calibration** window using *Thermal Solutions/Advantage* Instrument Control. (See the *Thermal Solutions/ Advantage* documentation for details.) Under Point 1, enter the temperature reading from the external thermometer as the correct temperature, and the reading from the DMA as the observed temperature. Then select **OK.**

The sample thermocouple has now been corrected for absolute temperature.

Dynamic Temperature Calibration Procedure

If you are going to perform temperature ramp experiments on the DMA 2980, with the single/dual cantilever or 3-point bending clamps, you can use the following steps to compensate for temperature lag. However, similar temperature calibration standards are not currently available for the other DMA clamps. (NOTE: ASTM Standard Test Method E1867–97, "Standard Test Method for Temperature Calibration of Dynamic Mechanical Analyzer"[1] describes another method for dynamic temperature calibration based on a polymer encapsulated metal standard.)

1. Perform steps 1 through 6 on page 3-22 to correct for absolute temperature.

2. Mount the thin polycarbonate test bar (provided in the accessory kit) on the clamp.

3. Measure the glass transition (T_g) of the polycarbonate sample under stepwise-isothermal conditions. (See Chapter 4 for information on running experiments.)

4. Set up the same instrument parameters you intend to use in your experiment.

[1] You may obtain ASTM E1867–97 from: ASTM, 100 Bar Harbor Drive, West Conshohocken, PA 19428-2959, (610) 832-9500.

5. Measure the T_g of the polycarbonate sample at the underlying heating (or cooling) rate you intend to use in your experiment.

6. Using the Universal Analysis program, analyze both data files, and note the temperature of the glass transition. (See the *Universal Analysis* online help or online manual for information on using the program.)

7. Open the **Temperature calibration** window using *Thermal Solutions/Advantage* Instrument Control. (See the *Thermal Solutions/ Advantage* documentation for details.) Under Point 1, enter the T_g measured by the stepwise-isothermal method as the correct temperature, and the T_g measured from the dynamic experiment as the observed temperature. Then select **OK.**

The instrument is now compensated for thermal lag at the specific underlying heating rate chosen. If a different heating rate is required, the dynamic run should be repeated at the required rate. The compensation for temperature lag can be significant. After completion, the ambient temperature reading may be unusually high or low, which is normal, and should be expected.

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Introduction

Most DMA 2980 experiments follow the same basic set of steps. Use the following instructions as a guide when you perform DMA experiments:

Basic Experimental Steps

1. Choose, install, and calibrate the clamp appropriate for the sample shape and modulus range.

2. Position the thermocouple near the sample.

3. Select the mode of operation (DMA multifrequency, DMA multistrain, DMA controlled force, etc.) needed to perform the desired type of experiment.

4. Select the instrument parameters that are specific to the mode chosen. (Include frequency or amplitude tables when appropriate.)

5. Create the method that is appropriate to the operating mode, including force, frequency, heating rate, etc., as defined by the mode and the clamp type.

6. Mount the properly prepared sample on the DMA 2980. Then start the motor to preview the desired measurement and confirm that conditions are acceptable before continuing with the experiment.

7. Start the thermal method and perform the experiment.

These steps are explained in detail in the remaining part of this chapter.

Choosing a Clamp Set

The shape and the modulus of most samples determine the clamp to be used for your experiment. With some samples, choosing the clamp set is obvious (films, for example). For others, however, there are several possible clamp sets (filled thermoplastic bars, for example). Sometimes more than one set of conditions are necessary to make measurements on a single material over a broad temperature range. The clamping fixtures with usual material types are shown in the table on the next page.

Once the appropriate clamp set is chosen, the DMA must be programmed to reflect the choice of clamps so that the instrument can properly control the experiment and report the correct sample moduli. The clamp type is selected from the Instrument Control software. Consult the *Thermal Solutions/Advantage User Reference Guide* for details.

This chapter provides information regarding the single/dual cantilever clamp that is the standard clamp set provided with the DMA 2980. If you select one of the optional clamps listed in the table on the next page, refer to Chapter 5 for details on using them.

Table 4.1
Clamps and Samples

Clamps	Type of Sample	Examples
3-Point Bending (small and large size)	stiff, low damping	metals, ceramics, highly filled thermo-setting polymers, highly filled crystalline thermoplastic polymers
Cantilever (single/dual)	weak to moderately stiff	thermosetting resins, elastomers, amorphous or lightly filled thermoplastic materials
Shear	unsupported viscous liquids to elastomers above glass transition	uncured resins, b-staged material, tire rubber
Compression	gels and weak elastomers	personal care products, toothpaste, hydrogels
Film Tension	thin films and fibers	various types of films
Fiber Tension	single and bundled fibers	various types of fibers
Penetration	any material	various samples for DMA penetration, glass transition, or melting analysis (Not used for quantitative DMA experiments.)

Single/Dual Cantilever Clamps

The single/dual cantilever clamps can be used for relatively weak to moderately stiff materials. The samples can range from supported thermosetting resins, to elastomers, amorphous, or lightly-filled thermoplastic materials. Dual cantilever clamps are good for testing weak elastomers and for curing supported resins. The single cantilever clamps should always be used for measuring the properties of amorphous polymers and elastomers through the glass transition, and for analyzing materials with high thermal expansion.

Sample Size

Length:	Single: 4, 10, and 17.5 mm
	Dual: 8, 20, and 35 mm
Width:	up to 15 mm
Thickness:	up to 5 mm

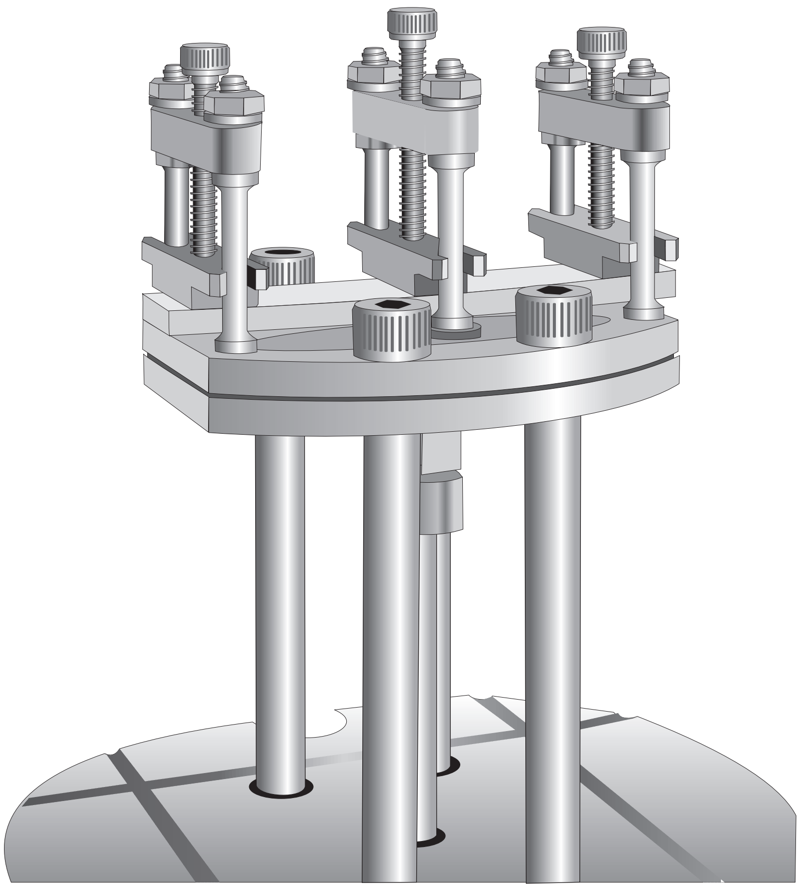

Figure 4.1
Dual Cantilever Clamp

For information on installing the dual cantilever clamp, refer to Chapter 2.

Aligning the Thermocouples

The sample thermocouples should be close to, but not touching the sample when it is loaded on the clamp. (The sample thermocouple is the taller one and is normally located on the right side.) You may find it necessary to realign one or both of the thermocouples periodically, should they become bent or misaligned, or when a new clamp is installed. Follow these steps:

1. Loosen the screw(s) on the thermocouple-mounting bracket(s) shown in the figure below.

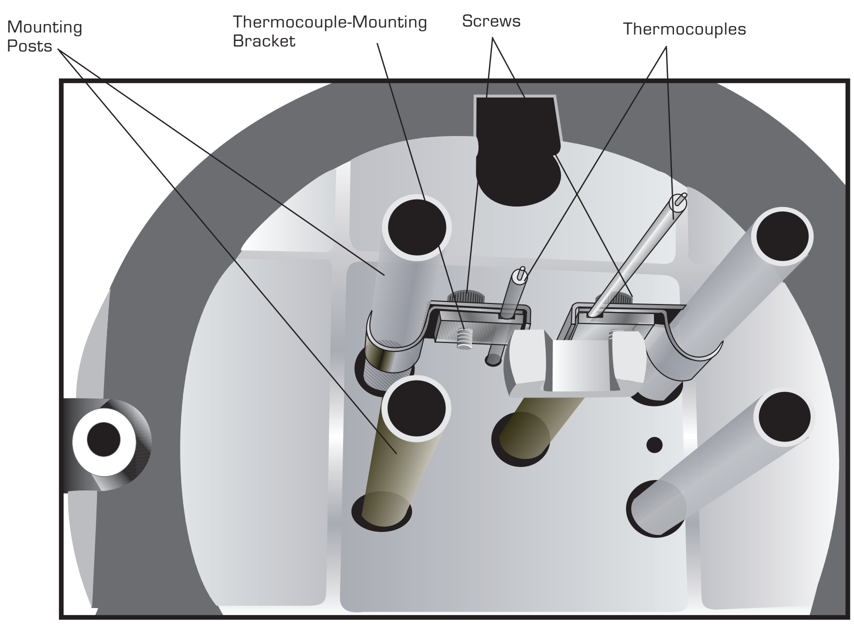

Figure 4.2
View From Top
with Clamp Removed

2. Move the thermocouple(s) up or down, as needed, or bend to the desired angle.

3. Retighten the screw in the bracket(s).

4. Adjust the angle of the thermocouple tip, if needed, so that it is close to, but not touching the sample. You may need to bend the thermocouple in order to get it closer to the sample. Take care when you bend the thermocouple that it does not break.

Selecting the Operating Mode

There are six possible operating modes for the DMA 2980. Each mode reflects a different class of experiment that can be performed. Before you can begin an experiment, you need to select an operating mode.

The following table describes the mode to select for the type of experiment desired.

Table 4.2 Modes and Experiments

Mode	Experiments
DMA Multi-frequency	• single frequency • multifrequency temperature ramps • multifrequency isothermal hold
DMA Multi-strain	• effect of oscillation amplitude on properties
DMA Creep	• effect of constant stress on properties
DMA Stress Relaxation	• effect of constant strain on properties
DMA Controlled Force	• thermal expansion • glass transition • softening point • static modulus
DMA Isostrain	• release of material stresses at constant strain

Once you have selected a mode, you will need to enter your choice using the *Thermal Solutions/ Advantage* Instrument Control program. For further information, see the *Thermal Solutions/ Advantage User Reference Guide*. This chapter contains instructions on how to run an experiment in each mode.

DMA Multifrequency Mode

In the DMA multifrequency mode, constant amplitude DMA (oscillatory stress and strain) experiments are performed on samples as a function of time, temperature, and frequency of oscillation. Storage and loss modulus, tan delta, viscosities, and, with some clamps, displacement and static force (preload force) are the results of experiments in this mode. Using the DMA multifrequency mode, you can perform three types of experiments: single frequency (with temperature ramps, holds, and combinations of the two), multifrequency temperature ramps, and multifrequency isothermal holds. This is a general purpose DMA mode, which is most often used to measure transition temperatures and the effect of frequency on transitions.

Selecting Instrument Parameters

After you have decided on the clamp, mode, and type of experiment, you need to select the instrument parameters to be used. This section provides some guidelines for selecting the instrument parameters to use with the single/dual cantilever clamp (for information on the other types of clamps, see Chapter 5). The instrument parameters are selected using the *Thermal Solutions/Advantage* DMA Instrument Control software. See the *User Reference Guide* for information.

General guidelines to use when you set up your instrument parameters for the DMA 2980, with the single/dual cantilever clamp installed and the DMA multifrequency mode selected, are provided in this section.

Data Sampling Interval

The **data sampling interval** parameter represents the minimum time for data collection at each frequency.

If the natural collection time for a particular frequency is longer than the data sampling interval, then data points will be collected at the natural collection rate. As three oscillations are needed to ensure thermal and mechanical equilibration in the sample (**during single frequency operation**), the natural collection time of a frequency is:

$t = 3/F$

where t is in seconds per data point and F is in Hz.

Recommended values for the data sampling interval are 3 to 10 seconds for temperature ramp experiments. For long (more than one hour) isothermal experiments, 30 seconds (or more) should be used as the sampling interval. For frequencies above 1 Hz, the data collection time is 3 seconds.

Oscillation Amplitude

The range of acceptable oscillation amplitudes is ± 0.5 to 10,000 µm. The actual usable range depends on the geometry (clamp type), sample stiffness, and frequency. The DMA can generate from 0.001 to 18 N of force. The force required to sustain the frequency and amplitude requested must be within that range. To approximate the best amplitude for a specific sample, set an initial amplitude of 10 to 20 µm and an initial (single) frequency of 1 Hz. Monitor the drive force across the temperature range of interest to ensure that it falls with that range. If it does not, adjust the amplitude, change the clamp, or change the sample size. For most samples, 10 to 50 µm should yield good results. Amplitudes less than 5 µm are not recommended. However, for extremely stiff materials, where the force limit is reached during oscillation, even after trying different clamp arrangements, amplitudes as low as 1 µm should still provide useful results.

The dynamic force depends on the oscillation, amplitude, oscillation frequency, and sample modulus. Autostrain is used to automatically adjust the static (preload) force to be a specified percentage greater than the force required to drive the amplitude. Autostrain reduces the change in permanent sample deformation (*e.g.*, stretching or creeping of a film) as the sample modulus decreases with increasing temperature. 125% is a typical autostrain value for tensioning clamps. 0% is used for nontensioning clamps.

When choosing the oscillation ampltiude, the maximum and minimum modulus (stiffness) must be considered. These modulus extremes generally occur at the lowest and highest temperatures of measurement respectively. [These modulus values must be within the DMA's operating range (10^7 to 10^2 N/m) or you may need to use a different sample shape and/or different clamping mode. See "Preparing and Mounting Samples" in this chapter.]

NOTE: If the amplitude is set too high, the oscillation amplitude will be automatically adjusted to keep the static (preload) and dynamic force within the 18 N limit.

See the *Thermal Solutions/Advantage User Reference Guide* for information on the remaining instrument parameters.

When you perform multifrequency experiments using the DMA, you need to either create a frequency table or use an existing frequency table. The instrument will apply the frequencies listed in the table to the sample as the experiment proceeds. The next section provides information needed to create a frequency table.

Creating Frequency Tables

Frequency tables contain a listing of one or more frequencies (up to 28) over the allowable range of 0.01 to 200 Hz. Follow these guidelines to create a frequency table. (See the *Thermal Solutions/Advantage User Reference Guide* for specific information on creating a frequency table.)

- For temperature ramp experiments, one frequency is normally used. You can use more than one frequency, but they should be higher frequencies (above 1 Hz). To measure the time required to sweep through all of the frequencies in a table, observe the sweeping process using the **Signal Control** window, when the drive motor is turned on. Set the heating rate so that the sweep time occurs in about 2 to 3 degrees.

- When you create a frequency table with more than one frequency, you can begin with the highest frequency and end with the lowest to shorten the sweep time.

- If you want your data to span several decades of frequency, then enter frequencies that are spaced "logarithmically" (*e.g*., 1, 2, 5, 10) rather than linearly spaced (*e.g*., 1, 3, 5, 7, 9, 11). The frequency list will repeat when the first zero in the table is encountered.

- You can also estimate the time needed to measure properties over the selected range of frequencies.

  — Seven oscillation cycles are needed to ensure that the specimen is oscillating properly at each selected frequency.

  — For each frequency, the time for one oscillation cycle is 1/F (F in Hz); therefore, the time to make a measurement at the individual frequency is (7)/F.

  — The minimum time to measure a frequency is 7 sec.

  — The estimated time needed to measure over the entire frequency table is :

  $t = (7/F1 + 7/F2 + 7/F3 +....7/Fn)$

NOTE: The recommended frequencies should have one-tenth (0.1) Hz resolution. If frequencies with one-hundredth (0.01) Hz resolution are needed, use frequencies that are divisible by 2 or 5. The frequency generator requires that an integral number of cycles be performed in an integral number of seconds. As you can see from the table on the next page, as the frequency decreases, the amount of time can increase, depending upon the number of cycles needed.

Table 4.2 Recommended Frequencies and Time

Frequency	Number of Cycles	Time Needed
0.1	7	70 sec
0.11	77	12 min
0.12	42	6 min
0.3	21	70 sec
0.32	112	6 min
0.33	231	12 min
1	7	7 sec
1.1	77	70 sec
1.2	42	35 sec
1.5	21	14 sec
10	70	7 sec
100	700	7 sec

Creating a DMA Multifrequency Method

After you have entered the instrument parameters based on the type of clamp and experiment desired, you need to create and edit a preprogrammed series of instrument instructions for your experiment. These instrument instructions are called *segments*. A series of segments make up a *method*. This section provides basic guidelines to use when setting up your methods. For details on methods, refer to the *Thermal Solutions/Advantage User Reference Guide.*

You can select varying numbers of frequencies to use in your experiments. For frequency tables that take less than approximately one minute to measure, select either temperature ramp or isothermal hold experiments. If measurements over more frequencies are desired, use isothermal hold experiments. (If the data is going to be used for Time-Temperature Superpositioning, the isothermal hold experiments are recommended.)

Examples of typical multifrequency methods are listed beginning on the next page.

NOTE: For all heating ramp experiments, use heating rates of 5°C/min or less, because using higher heating rates will create thermal gradients within the specimens. Using a rate of 3°C/min is a good compromise between experimental speed and temperature accuracy and precision. Slower rates are needed for low-frequency or multifrequency experiments.

- Constant heating ramp method from room temperature (this is a one-segment method):

  1 Ramp 3°C/min to 180°C

- Constant heating rate method from subambient temperatures:

  1 Equilibrate at -145°C
  2 Data Storage Off
  3 Isothermal 5 minutes (The instrument can cool faster than the specimen; time is needed for equilibration.)
  4 Data Storage On
  5 Ramp 3°C/min to 150°C

NOTE: The methods above will automatically collect multifrequency data during the temperature ramp. Do not use the frequency sweep segment in either of the methods above.

- Isothermal step frequency sweep method:

  1 Equilibrate 35°C
  2 Data Storage Off (Do not collect data during the initial isothermal segment.)
  3 Isothermal 3 min (Hold at temperature to ensure sample equilibrium.)
  4 Frequency Sweep (This segment turns the data storage on, steps through the frequency table, and turns off data storage after the final frequency.)
  5 Increment 3°C
  6 Repeat from segment 3 until 151°C (This segment creates a loop that will end when the temperature is met or exceeded.)

DMA Multistrain Mode

In the DMA multistrain mode, DMA experiments are performed over a range of amplitudes (strains) at constant frequency and isothermal temperature holds. This mode is used to study the effect of amplitude on material properties, most often to explore the linear viscoelastic limit.

Selecting Instrument Parameters

After you have decided on the clamp, mode, and type of experiment, you need to select the instrument parameters to be used. This section provides some guidelines for selecting the instrument parameters to use with the single/dual cantilever clamp (for information on the other types of clamps, see Chapter 5). The instrument parameters are selected using the *Thermal Solutions/Advantage* DMA Instrument Control software. See the *User Reference Guide* for information.

General guidelines to use when you set up your instrument parameters for the DMA 2980, with the single/dual cantilever clamp installed and the multistrain mode selected, are provided in this section.

Data Sampling Interval

The **data sampling interval** parameter represents the minimum time for data collection at each amplitude.

If the natural collection time for a particular amplitude is longer than the data sampling interval, then data points will be collected at the natural collection rate. As seven oscillations are needed to ensure thermal and mechanical equilibration in the sample at each amplitude, the natural collection time of a frequency is:

$$t = 7/F$$

where t is in seconds per data point and F is in Hz.

Recommended values for the data sampling interval are 7 to 14 seconds for temperature ramp experiments. For long isothermal experiments (over one hour), 30 seconds (or more) should be used for the sampling interval.

Frequency

All experiments are performed at a single frequency.

See the *Thermal Solutions/Advantage User Reference Guide* for information on the remaining instrument parameters.

Creating Amplitude Tables

When you perform multistrain experiments using the DMA, you need to either create an amplitude table or use an existing amplitude table. The instrument will apply the amplitudes listed in the table to the sample as the experiment proceeds. The next section provides information needed to create an amplitude table.

Amplitude tables contain a listing of up to 28 amplitudes (1/2 peak-to-peak motion) over which the material modulus and damping will be measured. Follow these guidelines to create an amplitude table. (See the *Thermal Solutions/ Advantage User Reference Guide* for specific information on creating an amplitude table.)

- For tensioning clamps, use this method to examine the limits of viscoelastic response of a material under isothermal conditions.
- The DMA can be set at an amplitude range of 0.5 to 10,000 µm; however, the actual *usable* amplitude range is determined by the sample stiffness and drive force. Usable amplitudes result in a drive force (dynamic force) within the 0.0001 to 18 N force range of the instrument. For tensioning clamps, usable amplitudes result in a drive force (static plus dynamic) of 0.0011 to 18 N.
- When sweeping amplitudes, sweep from the lowest to the highest values.

Creating a DMA Multistrain Method

After you have entered the instrument parameters based on the type of clamp and experiment desired, you need to create and edit a preprogrammed series of instrument instructions for your experiment. These instrument instructions are called *segments*. A series of segments make up a *method*. This section provides basic guidelines to use when setting up your methods. For details on methods, refer to the *Thermal Solutions/Advantage User Reference Guide.*

When you use the single/dual cantilever clamp in the DMA multistrain mode, experiments are usually performed using an isothermal hold method.

Examples of typical multistrain methods are listed below.

- Amplitude sweep method at room temperature (this is a two-segment method):

    1 Jump 0°C (With the set point below room temperature, the heater does not come on. Turn off the GCA if it is attached.)

    2 Amplitude Sweep (This segment turns the data storage on, steps through the amplitude table, and turns off data storage after the final amplitude.)

- Isothermal step amplitude sweep method:

  1. Equilibrate 35°C
  2. Data Storage Off (Do not collect data during the initial isothermal segment.)
  3. Isothermal 3 min (Hold at temperature to ensure sample equilibrium.)
  4. Amplitude Sweep (This segment turns the data storage on, steps through the amplitude table, and turns off data storage after the final amplitude.)
  5. Increment 3°C
  6. Repeat from segment 3 until 151°C (This segment creates a loop that will end when the temperature is met or exceeded.)

DMA Creep Mode

In DMA creep mode, an instantaneous stress is applied to the sample and its position is measured as a function of time (stress step). The sample is then released to an unstressed state, and its recovery is measured as a function of time. When you perform an experiment, the primary results in this mode are:

- compliance during both steps
- percent strain recovery during the recovery step
- percent strain during both steps.

The strain for the stress and recovery steps is calculated using the sample length at the start of the stress step. This length is the original sample length, plus any cumulative changes since the sample length was measured.

Selecting Instrument Parameters

After you have decided on the clamp, mode, and type of experiment, you need to select the instrument parameters to be used. This section provides some guidelines for selecting the instrument parameters to use with the single/dual cantilever clamp. The instrument parameters are selected using the *Thermal Solutions/ Advantage* DMA Instrument Control software. See the *User Reference Guide* for information.

General guidelines to use when you set up your instrument parameters for the DMA 2980, with the single/dual cantilever clamp installed and the creep mode selected, are provided in this section.

Data Sampling Interval

The **data sampling interval** parameter represents the minimum time for data collection at the start of each retardation and recovery step.

In the DMA creep mode, data collection is performed on a logarithmic-time basis, with a factor of 1.2. If a data sampling interval of 1 second is selected, data will be collected at 1, 2, 3, 4, 5, 6, 8, 10, 12, 15, 18, etc.

Recommended values for the data sampling interval are 2 to 10 seconds, if data is to be plotted on a linear scale (time or decay time), or 0.2 to 1 second, if data is to be plotted on a logarithmic scale.

Static Force (Preload Force)

(For Tensioning Clamps Only)

A preliminary static force (preload force) is available for creep measurements using the film and fiber tensioning clamps. This static force (preload force) ensures that the sample material is "fully elongated" (has no slack). A preliminary static force (preload force) is also available when using the compression and 3-point bending clamps for creep measurement. In those two clamps, the static force (preload force) ensures that the moveable portion of the clamp is in contact with the material prior to the start of the experiment. In all cases, the static force (preload force) should be limited to the minimum force required to achieve the desired starting condition. Otherwise, it may affect the creep results. (0.001 N is the lowest force that can be applied.)

The static force (preload force) is the force applied to the sample when the CLAMP ▼ key is pressed on the DMA 2980 instrument. The static force (preload force) is maintained after the motor is turned on, and throughout the experiment. This static force (preload force) is in addition to the stress applied during the creep test.

The nontensioning clamps, dual/single cantilever and shear sandwich clamps, do not require static force (preload force) in the creep mode.

Stress

The stress represents the amount of force applied to the sample. Select the stress value as follows, based on the type of experiment that you want to run:

- If you want to simulate real-life conditions, you need to calculate the stress that would be experienced by the material while it is in use, and then use that value as the instrument parameter.

- If you want to look at the structure of a material, such as its long-term behavior using the Time-Temperature Superposition software, the stress applied should keep the material in its linear viscoelastic region. To do this, perform your initial creep experiments at ambient temperature, with increasing levels of stress. In the linear viscoelastic region, compliances should superimpose on a creep compliance versus decay time plot. The limit of the linear viscoelastic region is reached when the compliance starts to drift away from the superimposed curves.

Equilibrium Criteria

Creep experiments are run isothermally. To ensure that the sample has reached thermal equilibrium before the stress (force) is applied, a “soak time” (typically 5 to 10 minutes) is recommended (unless the temperature of interest is ambient). Data collection should be initiated immediately prior to adding the stress. (See method on the next page.)

See the *Thermal Solutions/Advantage User Reference Guide* for information on the remaining instrument parameters.

Creating a DMA Creep Method

After you have entered the instrument parameters based on the type of clamp and experiment desired, you need to create and edit a preprogrammed series of instrument instructions for your experiment. These instrument instructions are called *segments*. A series of segments make up a *method*. This section provides basic guidelines to use when setting up your methods. For details on methods, refer to the *Thermal Solutions/Advantage User Reference Guide*.

When you use the single/dual cantilever clamp in the DMA creep mode, experiments are performed isothermally using the Displace/Recover method segment.

Examples of typical creep methods are listed on the next page.

- Creep method at room temperature (this is a two-segment method):

  1. Jump 0°C (With the set point below room temperature, the heater does not come on. Turn off the GCA if it is attached.)
  2. Displace 10 min, Recover 10 min (This segment lets the sample equilibrate, turns the data storage on, applies the selected stress for 10 minutes, then removes the stress and lets the sample recover for 10 minutes.)

NOTE: When you want to simulate real-life conditions, the duration of the displace and recover segments should reflect those times experienced by the material as it is actually used.

- Isothermal creep method (this is a six-segment method):

  1. Equilibrate 50°C
  2. Data Storage OFF
  3. Isothermal 5 minutes
  4. Displace 10 min, Recover 10 min (This segment lets the sample equilibrate, turns the data storage on, applies the selected stress for 10 minutes, then removes the stress and lets the sample recover for 10 minutes.
  5. Increment 5°C
  6. Repeat from segment 2 until 152°C (This segment creates a loop that will end when the temperature is met or exceeded.)

DMA Stress Relaxation Mode

In DMA stress relaxation mode, an instantaneous strain is applied to the sample and the force required to maintain the strain is measured as a function of time (strain step). When you perform an experiment, the primary result in this mode is stress relaxation modulus during the strain step.

In this mode, the strain is calculated using the sample length of the start of the strain step. This length is the original sample length, plus any cumulative changes in the length since the sample length was measured.

Selecting Instrument Parameters

After you have decided on the clamp, mode, and type of experiment, you need to select the instrument parameters to be used. This section provides some guidelines for selecting the instrument parameters to use with the single/dual cantilever clamp. The instrument parameters are selected using the DMA Instrument Control software. See the *User Reference Guide* for information.

General guidelines to use when you set up your instrument parameters for the DMA 2980, with the single/dual cantilever clamp installed and the stress relaxation mode selected, are provided in this section.

Data Sampling Interval

The **data sampling interval** parameter represents the minimum time for data collection at the start of each relaxation and recovery step.

In the DMA stress relaxation mode, data collection is performed on a logarithmic-time basis, with a factor of 1.2. If a data sampling interval of 1 second is selected, data will be collected at 1, 2, 3, 4, 5, 6, 8, 10, 12, 15, 18, etc.

Recommended values for the data sampling interval are 2 to 10 seconds, if data is to be plotted on a linear scale (time or decay time); or 0.2 to 1 second, if data is to be plotted on a logarithmic scale.

Static Force (Preload Force)

(For Tensioning Clamps Only)

A preliminary static force (preload force) is available for stress relaxation measurements using the film and fiber tensioning clamps. This static force (preload force) ensures that the sample material is “fully elongated” (has no slack). A preliminary static force (preload force) is also available when using the compression and 3-point bending clamps for stress relaxation measurement. In those two clamps, the static force (preload force) ensures that the moveable portion of the clamp is in contact with the material prior to the start of the experiment. In all cases, the staic force should be limited to the minimum force required to achieve the desired starting condition. Otherwise, it may affect the stress relaxation results. (0.001 N is the lowest force that can be applied.)

The static force (preload force) is the force applied to the sample when the CLAMP ▼ key is pressed on the DMA 2980 instrument. The static force (preload force) is maintained after the motor is turned on, and throughout the experiment. This static force (preload force) is in addition to the stress applied during the stress relaxation test.

The nontensioning clamps, dual/single cantilever and shear sandwich clamps, do not require static force (preload force) in the stress relaxation mode.

Strain %

The percent **strain** represents the amount of deformation applied to the sample. Select the strain value as follows, based on the type of experiment that you want to run:

- If you want to simulate real-life conditions, you need to calculate the strain that would be experienced by the material while it is in use, and then use that value as the instrument parameter. See Chapter 6 for details on calculation of the strain.

- If you want to look at the structure of a material, such as its long-term behavior using the Time-Temperature Superposition software, the strain applied should keep the material in its linear viscoelastic region. To do this, perform your initial stress relaxation experiments at ambient temperature, with increasing levels of strains. In the linear viscoelastic region, moduli should superimpose on a stress relaxation modulus versus decay time plot. The limit of the linear viscoelastic region is reached when the modulus starts to drift away from the superimposed curves.

Equilibrium Criteria

Stress relaxation experiments are run isothermally. To ensure that the sample has reached thermal equilibrium before the strain is applied, a "soak time" (typically 5 to 10 minutes) is recommended (unless the temperature of interest is ambient). Data collection should be initiated immediately prior to straining the material. (See method on page 4-37.)

See the *Thermal Solutions/Advantage User Reference Guide* for information on the remaining instrument parameters.

Creating a DMA Stress Relaxation Method

After you have entered the instrument parameters based on the type of clamp and experiment desired, you need to create and edit a preprogrammed series of instrument instructions for your experiment. These instrument instructions are called *segments*. A series of segments make up a *method*. This section provides basic guidelines to use when setting up your methods. For details on methods, refer to the *Thermal Solutions/Advantage User Reference Guide*.

When you use the single/dual cantilever clamp in the DMA stress relaxation mode, experiments are performed isothermally using the Displace/Recover method segment.

Examples of typical stress relaxation methods are listed below.

- Stress relaxation method at room temperature (this is a two-segment method):

    1 Jump 0°C (With the set point below room temperature, the heater does not come on. Turn off the GCA if it is attached.)

    2 Displace 10 min, Recover 0 min (This segment lets the sample equilibrate, turns the data storage on, applies the selected percent strain for 10 minutes, then releases the sample.)

NOTE: When you want to simulate real-life conditions, the duration of the retardation segment should reflect that experienced by the material as it is actually used.

- Isothermal stress relaxation method (this is a six-segment method):

  1 Equilibrate 50°C

  2 Data Storage OFF

  3 Isothermal 5 minutes

  4 Displace 10 min, Recover 0 min (This segment lets the sample equilibrate, turns the data storage on, applies the selected strain for 10 minutes, then releases the sample.)

  5 Increment 5°C

  6 Repeat from segment 2 until 152°C (This segment creates a loop that will end when the temperature is met or exceeded.)

DMA Isostrain Mode

The DMA isostrain mode is a special case of stress relaxation, which is used only with the film or fiber tension clamps. In DMA isostrain mode, a specific known strain is applied to the sample. The temperature of the sample is then ramped and the amount of stress (force) required to maintain that strain is measured. Results obtained provide insights into shrinkage stresses and other built-in processing effects in films and fibers.

Isostrain is also known as a shrink force measurement and is typically used in the film and fiber industry to measure orientation in the amorphous phase. Orientation in the amorphous phase will cause a sample under strain to shrink. Since strain is held constant on the sample, the force required to maintain the strain increases as the fiber attempts to shrink on heating. The more the fiber shrinks, the more orientation is in the fiber.

NOTE: This test is intended only for shrinkage force measurements where the amount of shrinkage that occurs when the sample is heated is very large relative to the thermal expansion of the system (sample, clamps, and posts). If the sample does not shrink, do not use this test.

Selecting Instrument Parameters

This section provides some guidelines for selecting the instrument parameters to use with the film or fiber clamps when operating in the DMA isostrain mode. The instrument parameters are selected using the *Thermal Solutions/ Advantage* DMA Instrument Control software. See the *User Reference Guide* for information.

NOTE: You should not use the Auto Zero function when programming the strain in the instrument parameters. When you want to use the Auto Zero function, you need to program the strain as a method segment.

Data Sampling Interval

The **data sampling interval** parameter represents the minimum time for data collection.

Recommended value for the data sampling interval is 2 seconds.

Static Force (Preload Force)

A preliminary static force (preload force) is available for DMA isostrain measurements. This static force (preload force) ensures that the film or fiber sample material is "fully elongated" (has no slack) and should be limited to the minimum force required to achieve that desired starting condition. (0.001 N is the lowest force that can be applied.)

NOTE: The sample must be fully elongated so that the initial sample length, on which the % strain is based, is accurate.

Strain %

The percent strain represents the amount of deformation applied to the sample. The strain chosen should be small enough to ensure that the measurement is occuring within the linear viscoelastic region. Typical values are 0.01 to 0.1%. (To determine the actual linear viscoelastic region, use the procedure described for stress relaxation.)

See the *Thermal Solutions/Advantage User Reference Guide* for information on the remaining instrument parameters.

Creating an Isostrain Method

After you have entered the instrument parameters, you need to create and edit a preprogrammed series of instrument instructions for your experiment. These instrument instructions are called *segments*. A series of segments make up a *method*. This section provides basic guidelines to use when setting up your methods. For details on methods, refer to the *Thermal Solutions/Advantage User Reference Guide*.

When you use the film or fiber clamp in the DMA isostrain mode, experiments are performed using a constant heating rate (ramp) method. Typical heating rate methods are as follows:

NOTE: For isostrain experiments, heating rates should not exceed 5°C/min. Higher heating rates will create thermal gradients within the specimens. Using a rate of 3°C/min is a good compromise between experimental speed and temperature accuracy and precision.

- Constant heating ramp method from room temperature:

  1 Isostrain at 1%

  2 Ramp 3°C/min to 250°C

- Constant heating rate method from subambient temperatures:

  1 Equilibrate at -145°C

2 Isothermal 5 minutes (The instrument can cool faster than the specimen; time is needed for equilibration.)

3 Isostrain at 1%

4 Ramp 3°C/min to 150°C.

DMA Controlled Force Mode

The DMA controlled force mode is used to measure the displacement of a sample as a function of time, temperature, and applied force. Force can be applied in one of three manners: constant force, stepped force, or continuous force ramp. DMA controlled force can be used to measure glass transition, softening point, "static" modulus, etc. Creep measurements can also be made in this mode, although time or position is not reset between the applications of force.

Selecting Instrument Parameters

After you have decided on the clamp, mode, and type of experiment, you need to select the instrument parameters to be used. This section provides some guidelines for selecting the instrument parameters to use with the single/dual cantilever clamp (for information on the other types of clamps, see Chapter 5). The instrument parameters are selected using the *Thermal Solutions/Advantage* DMA Instrument Control software. See the *User Reference Guide* for information.

General guidelines to use when you set up your instrument parameters for the DMA 2980, with the dual cantilever clamp installed and the DMA controlled force mode selected, are provided in this section. See the *Thermal Solutions/ Advantage User Reference Guide* for details on the Instrument Parameters functions.

Data Sampling Interval

The **data sampling interval** parameter represents the minimum time for data collection.

Recommended value is 2 seconds.

Static Force (Preload Force)

(For Tensioning Clamps Only)

A preliminary static force (preload force) is available for DMA controlled force measurements using the film and fiber tensioning clamps. This static force (preload force) ensures that the sample material is “fully elongated” (has no slack) and should be limited to the minimum force required to achieve the desired starting conditions. (0.001 N is the lowest force that can be applied.)

The static force (preload force) is initiated when the CLAMP ▼ key is pressed on the DMA 2980 instrument. This value is altered by Force, Increment Force, Ramp Force, or Motor Drive Off segments.

The nontensioning clamps, dual/single cantilever and shear sandwich clamps, do not require static force (preload force) in the controlled force mode.

See the *Thermal Solutions/Advantage User Reference Guide* for information on the remaining instrument parameters.

Creating a DMA Controlled Force Method

After you have entered the instrument parameters based on the type of clamp and experiment desired, you need to create and edit a preprogrammed series of instrument instructions for your experiment. These instrument instructions are called *segments*. A series of segments make up a *method*. This section provides basic guidelines to use when setting up your methods. For details on methods, refer to the *Thermal Solutions/Advantage User Reference Guide.*

When you use the dual cantilever clamp in the DMA controlled force mode, the following experiments can be performed.

Temperature-Based Experiments

You can use temperature ramps to measure thermal expansion, glass transitions, and softening points of materials, depending on the type of clamping accessory used. The penetration clamps are recommended for temperature-based experiments since these clamps provide the best results (low noise and relatively featureless, low drift baseline).

Typical temperature scan methods are as follows:

NOTE: For all heating ramp experiments, use heating rates of 5°C/min or less, because using higher heating rates will create thermal gradients within the specimens. Using a rate of 3°C/min is a good compromise between experimental speed and temperature accuracy and precision.

- Constant heating ramp method from room temperature (this is a one-segment method):

  1 Ramp 3°C/min to 250°C

- Constant heating rate method from subambient temperatures:

  1 Equilibrate at -145°C

  2 Isothermal 5 minutes (The instrument can cool faster than the specimen; time is needed for equilibration.)

  3 Ramp 3°C/min to 150°C.

Time-Based Experiments (Manual Creep Measurements)

Time-based experiments most often involve stepping the static force (preload force) level up and down to view creep response and recovery. Usually, creep experiments are better performed in the DMA creep mode. (Creep Time-Temperature Superposition [TTS] experiments should always be performed in the DMA creep mode.) An example experiment in the DMA controlled force mode is shown on the next page.

- Room temperature creep/recovery method:

  1. Jump 0°C (No power is applied to furnace. Make certain the GCA is turned off.)
  2. Force 1.7 N (Approximately 1 MPa stress level on standard 50 x 12.5 x 3.2 mm 3-point bending sample.)
  3. Isothermal 5 min
  4. Force 0.01 N (Return to minimal force to see recovery.)
  5. Isothermal 5 min.

Force Ramp Experiments

If you are using the Force Ramp method segment with the DMA Controlled Force mode, the DMA 2980 becomes an instrument capable of performing Controlled Rate of Load (CRL) stress-strain mechanical tests. This capability can be used to explore material properties within and beyond the linear viscoelastic region and to view material parameters such as yield and ultimate strengths. This test can be used to determine the linear viscoelastic region for tensioning clamps. The method for the force ramp is shown here:

1. Equilibrate 30°C
2. Ramp Force 2 N/min to 18 N

After you have created an experiment to perform on the DMA, you need to prepare and mount the samples. See the next section for instructions.

Preparing and Mounting Samples

Sample preparation is one of the most important factors in achieving accurate and reproducible modulus values. Each type of clamp requires a specific method for sample preparation.

The procedures for the standard single/dual cantilever clamp are listed below. See Chapter 5 for information on the other clamping configurations.

Single/Dual Cantilever Clamp Sample Preparation

Ideally, samples should be molded, machined, or otherwise fashioned into a rectangular shape for use with the dual cantilever clamp.

- **Thickness**: The minimum length-to-thickness ratio should be 10 to 1. The thickness of the rectangle should be 1/10 to 1/32 of the span of the dual cantilever clamp. The maximum thickness is 5 mm.

NOTE: It is very important that the sample have a uniform thickness, and that the thickness is accurately measured. The cube of the sample thickness is used in the modulus calculation; therefore, a 3% error in thickness becomes a 10% error in the calculated modulus.

- **Width**: The width of the rectangle should be 5 to 15 mm. The width and thickness dimensions should be uniform across the sample to within 0.02 mm.

- **Length:** Cut the sample 5 mm longer than the distance between the dual cantilever supports, so that the sample will lie across the supports without touching the furnace. This length is approximately 55 to 60 mm for the dual cantilever clamp and approximately 30 mm for the single cantilever clamp.

♦ ***CAUTION:*** **Make sure that the sample does not touch the furnace or the heating element.**

Other sample shapes such as cylinders and tubes can be used in the dual cantilever clamps. However, clamping efficiency is reduced with these shapes, and the uncertainty in modulus measurements increases as a result.

NOTE: The sample size must be chosen so that the sample stiffness is within the instrument's limits. If a sample physically fits in the clamp, it does not mean that the sample will have high or low enough stiffness for accurate measurements.

Operating Range for Single/Dual Cantilever Clamps

The two equations below can help you determine which sample clamps to use and which sample sizes to choose. These equations can also help determine if the properties of a sample of a particular size can be measured or if the sample will have to be resized.

Shown on the next several pages are the modulus range versus the possible sample size range for the sample clamps. The possible sample sizes are calculated as geometry factors (GF) in the equations below. The modulus range is based on the range of stiffness over which the DMA 2980 can operate (10^2 to 10^7 N/m).

Geometry Factor Equations

Single Cantilever:

$$GF = \frac{1}{F}\left[\frac{L^3}{12I} + 2S(1+\nu)\frac{L}{A}\right]$$

Dual Cantilever:

$$GF = \frac{1}{F}\left[\frac{L^3}{192I} + S(1+\nu)\frac{L}{2A}\right]$$

where:

L = sample length (mm)
A = sample cross sectional area (mm^2)
I = geometric moment (mm^4) = 1/12 T^3W for rectangular samples
T = sample thickness (mm)

W = sample width (mm)
F = clamping factor (nominally 0.9)
S = shearing factor (nominally 1.5)
ν = Poisson's ratio (nominally 0.44).

NOTE: Refer to the "Clamping Factors" section in Chapter 6 to determine the value used for the clamping factor, F.

These equations are explained in greater detail in Chapter 6.

The next two figures illustrate some typical sample geometries.

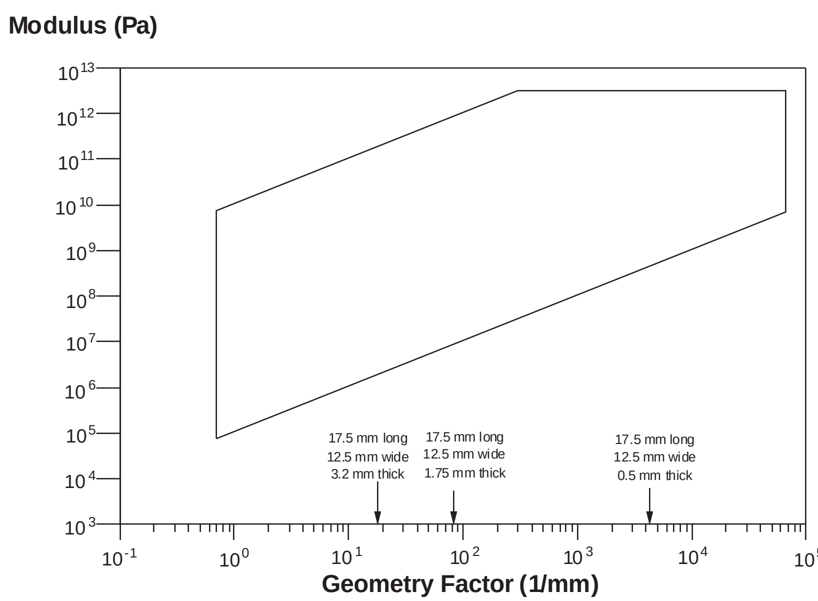

Figure 4.3
Operating Range of the Single Cantilever Clamps

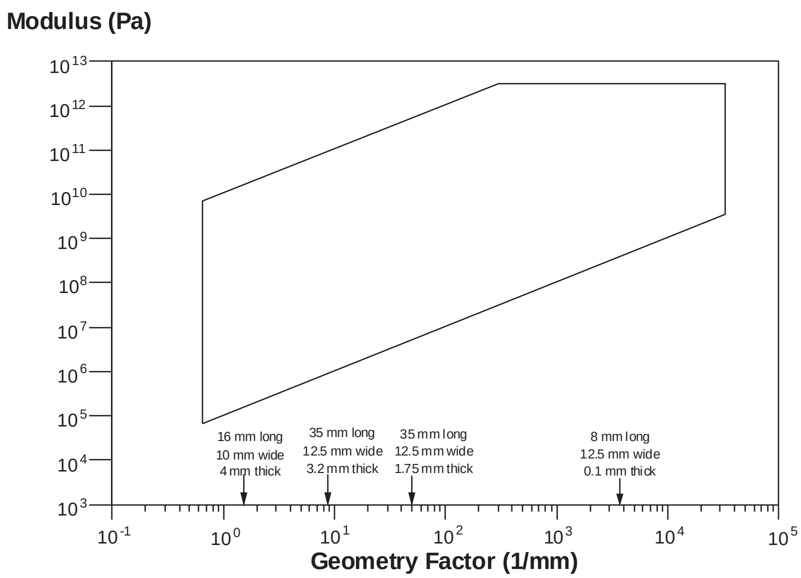

Figure 4.4
Operating Range of the Dual Cantilever Clamps

Use of Operating Range Figures

Figures 4.3 and 4.4 can help to determine the proper sample shape for a given material, or to determine if a sample of a particular size can be measured as is. To determine the proper sample size for a specific material requires some knowledge of the approximate behavior (modulus) of the material to be tested. For example, a given material may have a modulus approximately $3x10^{9}$ Pa at room temperature and will go to 10^{7} Pa above Tg. From Figures 4.3 and 4.4, it can be seen that most geometry factors can accommodate the material at room temperature, but only a limited range of geometries (10^{0} to 10^{2} 1/mm) can accommodate the sample above glass transition.

The second use for Figures 4.3 and 4.4 is to determine whether a sample of a particular size can be used. For example, you may have a sample that is 12.5 mm wide by 3.2 mm thick, with the standard (35 mm) cantilever clamps. The geometry factors for both the single cantilever (17.5 mm) and dual cantilever (35 mm) clamps are displayed on the above figures. If the material has relatively low modulus (*i.e.,* 10^6 Pa), at room temperature, then the dual cantilever clamps can easily accommodate this sample, but this sample is at the lower edge of the operating range of the single cantilever clamps. If accurate information above room temperature is desired, then the dual cantilever clamps would be preferred, or a smaller (10 mm or 4 mm) single cantilever clamp would be needed.

Single/Dual Cantilever Clamp Sample Mounting

Two procedures are provided in this section—one to mount stiff samples and one to mount weak samples. Refer to the section appropriate to the type of sample you are using.

Mounting a Stiff Sample

To mount a stiff sample on the single or dual cantilever clamps, follow these steps:

1. Loosen the three clamping center screws (two for single cantilever clamp), shown in the figure below.

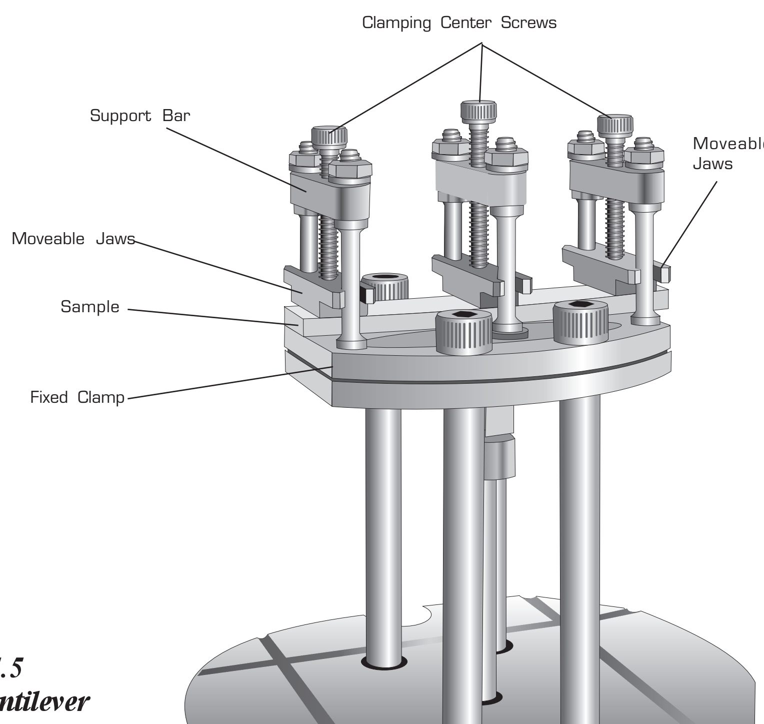

Figure 4.5 Dual Cantilever Clamp with Sample

2. Press the FLOAT/LOCK key to release (float) the moveable clamp.

3. Lifting each one of the moveable jaws in turn, slide the sample in from one side between the clamp faces.

NOTE: If the thermal expansion of a material is high, use the single cantilever clamp, rather than the dual cantilever clamp, to obtain the most accurate results. Use the rear fixed clamp for single cantilever operation to place the sample closer to the thermocouple.

4. Tighten the clamping screws on the fixed clamp until they are fingertight.

5. Tighten the clamping screw on the moveable clamp until it is fingertight.

6. Press the FLOAT/LOCK key again to lock the clamp in position.

7. Use the torque wrench to tighten each of the clamping screws to maximize clamping but minimize sample deformation. Suggested clamping torques are 1.1 N-m (10 in-lb) for high modulus materials (E' > 5 GPa), 0.6 to 0.9 N-m (5 to 8 in-lb) for thermoplastic samples (E' ~ 1 GPa) and fingertight for most elastomers above Tg.

8. Adjust the thermocouple so that it is approximately 1 mm below and 1 mm to the side of the sample.

♦ ***CAUTION:*** **Make sure that the sample does not touch the furnace or the heating element.**

Mounting Weak Materials, Including Resins or Other Materials, on the Fiberglass Braid

If you are using the single or dual cantilever clamps to analyze resins or some other sample on the fiberglass braid, we recommend using the 8-mm dual cantilever clamps. Follow the steps below to mount samples on the clamp:

1. Perform steps 1 through 6 in the previous section using a rigid sample (such as one of the steel compliance samples).

2. Loosen the clamping center screws and remove the rigid sample.

3. Lifting each one of the moveable jaws in turn, slide paperclips or other small items between the jaws to prop the clamp faces open. We recommend that aluminum foil be placed over each clamp face to prevent the resin from sticking the jaws shut.

4. Prepare the braid sample with the resin, spreading the resin thinly and uniformly onto the braid.

5. Carefully slide the prepared braid in from one side of the clamp.

6. Remove the paperclips or other propping devices from between the clamp faces.

7. Tighten the clamping center screws to finger tightness. Use a hex key to tighten the screws an additional 1/4 turn.

8. Adjust the thermocouple so that it is approximately 1 mm below and 1 mm to the side of the sample.

Measuring Sample Length

To obtain accurate modulus values, it is important that the sample dimensions be measured accurately.

- When using the dual cantilever clamp, the sample length is defined by the length between the two fixed clamps, minus the moveable clamp's thickness. This measurement becomes fixed (see page 4-8 for the default values) and can be used without the need for verification.

- When using the single cantilever clamp, the sample length is defined by the length between the fixed and moveable clamps. However, due to the flexibility of the drive shaft, after the sample is tightened in the clamps, the actual sample length may be slightly different from the default values. To accurately measure the sample length, a telescoping gauge is provided in the accessory box. The next section provides the instructions needed to use the telescoping gauge.

Using the Telescoping Gauge

To accurately measure sample length using the telescoping gauge on the single cantilever clamp, follow these steps:

1. Prepare and mount the sample as directed in this chapter.

2. Loosen the gauge tips by turning the knurled knob counterclockwise approximately one-quarter turn. See Figure 4.6.

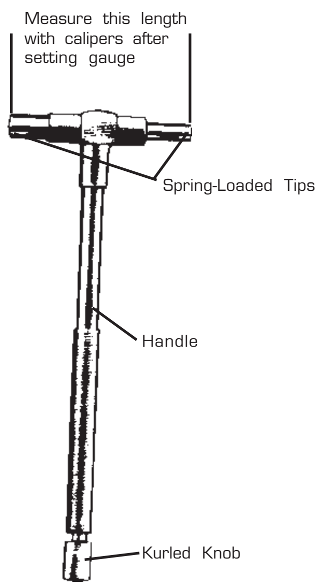

Figure 4.6
Telescoping Gauge

3. Insert the gauge sideways between the clamps, just above the sample.

4. Gently rotate the gauge so that the spring-loaded gauge tips are exactly perpendicular to the clamp jaws and at the middle of the sample. Make sure that you keep the handle vertical.

5. Set the gauge by turning the knurled knobs clockwise to lock the tips in position.

6. Rotate the gauge sideways and remove it from the clamps.

7. Measure the gauge length, which is equal to the actual sample length, with calipers. Use this value as your sample length when entering the instrument parameters.

Performing Experiments

Once you have set the appropriate instrument and experimental parameters and have mounted a sample on the DMA 2980, you are ready to run the experiment.

When you run experiments using the single or dual cantilever clamps, follow the instructions given in the next section. Please note the following conditions that pertain to single/dual cantilever clamp experiments:

- If the thermal expansion of a material is high—such as for thermoplastics and rigid elastomers—use the single cantilever clamp, rather than the dual cantilever clamp, to obtain the most accurate results.

- You may need to tighten the clamps at the minimum temperature, when you run elastomer samples at subambient temperatures. To do this, follow the suggested method below:

  1. Program a method with Drive Off as the first segment and Initial temperature XX°C as the second segment. Then follow with the other segments needed for your particular experiment. The first two segments will bring your sample to the starting temperature without applying forces to it that might distort it.

  2. When the instrument status line reads Ready, press the FURNACE key on the instrument to raise the furnace. The GCA will automatically stop and switch

to a vent state and the furnace heaters will be turned off while the furnace is open.

3. Quickly and carefully tighten the clamping screws.

!WARNING

Use the appropriate tools and safety precautions if you need to handle the sample or clamps. They can be hot or cold enough to cause injury.

4. Press the FURNACE key on the instrument to lower the furnace. As the furnace is lowering, turn the motor on using the **Control**/**Motor**/**On** function on the DMA Instrument Control software.

   The method and temperature control will automatically resume when the furnace is closed.

5. Allow the sample and furnace to return to the initial temperature and equilibrium. Then use the **Control**/**Resume** function (or the START key on the instrument) to continue the experiment.

Starting an Experiment

To start an experiment on the DMA 2980, follow these steps:

1. Use the *Thermal Solutions/Advantage* DMA Instrument Control program to select the clamp, mode, and instrument parameters. When appropriate, you will also need to create an amplitude or frequency table.

2. Create and load an experimental method using the *Thermal Solutions/Advantage* software.

3. Prepare and mount the desired sample on the clamp installed.

4. Press the FURNACE key to close the furnace. Then press the MEASURE key on the instrument keypad.

5. Observe the *Thermal Solutions/Advantage* **Signal Display** window to ensure good starting conditions (*e.g.*, smooth oscillation and relatively constant position).

6. Press the START key on the instrument keypad or select **Start** from the menu or tool bar of the *Thermal Solutions/Advantage* DMA Instrument Control program. The experiment will begin.

Stopping an Experiment

You can stop an experiment before the end of the selected method when necessary. Use one of the following actions to stop an experiment in progress:

- Select **Stop** on the *Thermal Solutions/ Advantage* Instrument Control program or STOP on the 2980 keypad to stop an experiment. The "stop" command ends the method, saves the data, then begins the method-end conditions.

- Select **Reject** on the *Thermal Solutions/ Advantage* Instrument Control program or SCROLL-STOP on the 2980 keypad to reject an experiment. The "reject" command ends the method normally, as though it had run to completion. The method-end conditions go into effect, but the data that has been generated is discarded.

Removing Samples

When the experiment has run to completion, remove the sample from the single/dual cantilever clamp as follows:

1. Wait for the sample to return to room temperature before you attempt to remove it.

2. Press the FURNACE key to raise the furnace.

3. Press the FLOAT/LOCK key or STOP to lock the moveable clamp in position.

4. Loosen the three clamping center screws that are holding the sample between the moveable jaws and remove the sample. If any sample residue remains stuck to the clamp, remove it by scraping it off with a razor blade or similar tool.

If further cleaning is needed, turn to Chapter 7 for further information.

See Chapter 5 to remove the samples from the optional clamps.

Removing the Clamp

If you want to remove the single/dual cantilever clamp after an experiment, refer to Chapter 2 for the steps needed.

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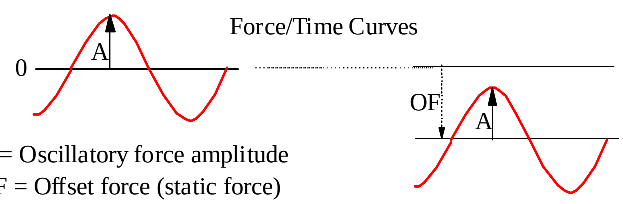
Introduction

The TA Instruments DMA 2980 can be used with various accessory clamps. The 35-mm dual cantilever clamp is the standard clamp that comes with the instrument. This chapter provides details on the optional clamps (clamps other than the dual cantilever clamp). Information such as how to install the optional clamps, mount a sample, and remove the optional clamps is provided on the next several sections. To decide which clamp to use for your samples, turn to Chapter 4 in this manual to learn the differences between the clamps and choose the appropriate type of clamp.

Tensioning/Nontensioning Clamps

There are two classes of clamps for the DMA 2980—tensioning and nontensioning. The 3-point bend, tension/film, tension/fiber, compression and penetration clamps are *tensioning* clamps, while the single/dual cantilever and shear sandwich clamps are *nontensioning*.

- When you use the *tensioning* clamps, such as the 3-point bending clamp, a static (or preload) force must be applied to pretension the sample before the oscillation can occur. (See the figure to the left.) The oscillating force increases and reduces stress in the specimen, but stress must always be positive. Otherwise, the moveable clamp will lift off the sample or the sample will buckle and oscillation will be lost. For more information regarding static (preload) force, see the next section, beginning on page 5-8.



A = Oscillatory force amplitude
OF = Offset force (static force)

Clamps without offset force:	Clamps with offset force:
Single Cantilever	Tension Film
Dual Cantilever	Tension: Fiber
Shear Sandwich	3-Point Bend
	Compression
	Penetration

- When using *nontensioning* clamps, the specimen oscillates about the zero stress point and no static (preload) force is required.

Two methods are available for maintaining a positive force on the sample: *constant force* and *autostrain (force track)*.

- When you use *constant force*, the static force (preload force) on the sample remains steady throughout the experiment.

- When you select *autostrain (force track)*, the total force (preload and dynamic) that is applied to the sample during multifrequency and multistrain experiments, using tensioning clamps, is automatically adjusted. This ensures that the specified amplitude and frequency are sustained without the occurence of permanent sample deformation as the temperature increases (modulus decreases). Recommended autostrain (force track) values are 120% for thin films and low denier fibers (using the film and fiber tension clamps), 150% for thermoplastics (using 3-point bend clamps), and 125% for general applications.

Autostrain (force track) is typically preferred over constant force, since the likelihood of permanently deforming (*e.g.*, stretching) the specimen is greatly reduced by reducing the force as the specimen modulus decreases.

You can select both parameters using the DMA Instrument Control software.

Static Force (Preload Force)

If autostrain (force track) is used, the static force (preload force) will automatically be adjusted to remain a set percentage greater than the force required to drive the sample at the programmed amplitude. The following formula is used to maintain the ratio between the static (preload) force and dynamic force (force to drive amplitude).

Static (Preload) Force =
Autostrain (Force Track) x
Stiffness x Amplitude

For example, if you program an autostrain (force track) of 120% and an amplitude of 10 microns, and the measured sample stiffness is 50,000 N/m, then the static force will be as follows:

Static Force = (1.2) x (10 x 10^{-6}) x (50,000 N/m) = 0.6 N

If autostrain (force track) is not used, the static force (preload force) should read the value set in the instrument parameters.

- *For Creep, Stress Relaxation, Stress/ Strain Experiments (film/fiber clamps)*: Use static (preload) force to elongate samples prior to measuring length and initiating experiments. Use the minimum possible.

- *For Oscillation Experiments (all tensioning clamps)*: Use static (preload) force to ensure that oscillation can be sustained. Use autostrain (force track) to automatically adjust the combined static (preload) and dynamic force to avoid undue elongation of the sample as modulus decreases.

Constant Force

To apply a constant force to the sample, set up your instrument parameters as follows:

1. Clear the Autostrain (Force Track) option.

2. Enter the value desired for the constant force into the Static Force (Preload Force) field. The static force (preload force) value should exceed the dynamic force needed for oscillation over the entire experiment. The force is applied to the specimen throughout the entire experiment.

Autostrain (Force Track)

To apply an adjustable force to the sample, set up your instrument parameters as follows:

1. Select the Autostrain (Force Track) option.

2. Enter the autostrain (force track) value. This value is a constant multiplier used to adjust static force (preload force) during the oscillation according to the equation on the previous page.

   The autostrain (force track) value is a constant multiple of sample stiffness, which is calculated from the drive force, oscillation amplitude, and frequency of oscillation.

3. Enter the static force (preload force) value. When used with autostrain (force track), the static force (preload force) is applied to the sample prior to initiating the experiment. Static (preload) force is never used for nontensioning clamps. It is required with tensioning clamps when running multifrequency and multistrain experiments in order to sustain oscillation (ensure that the force is applied to the sample throughout the complete oscillation). Typical static (preload) forces for those experiments are 0.01 to 0.05 N.

3-Point Bending Clamps

This type of clamp is most useful for stiff, highly elastic materials such as metals, ceramics, highly filled thermosetting polymers, and highly filled and crystalline thermoplastic polymers. When used for oscillatory experiments, 3-point bending clamps require a static (preload) force. The 3-point bending clamp is therefore not useful for DMA samples that will not support a preload force, such as elastomers.

Sample Sizes

Length:	5, 10, 15, 20, and 50 mm
Width:	up to 15 mm
Thickness:	up to 7 mm

♦ ***CAUTION:*** **When the small 3-point bending clamp is used, you must make sure that both the fixed and moveable clamps are installed. The furnace may hit the clamp when it is closed, unless both parts are installed.**

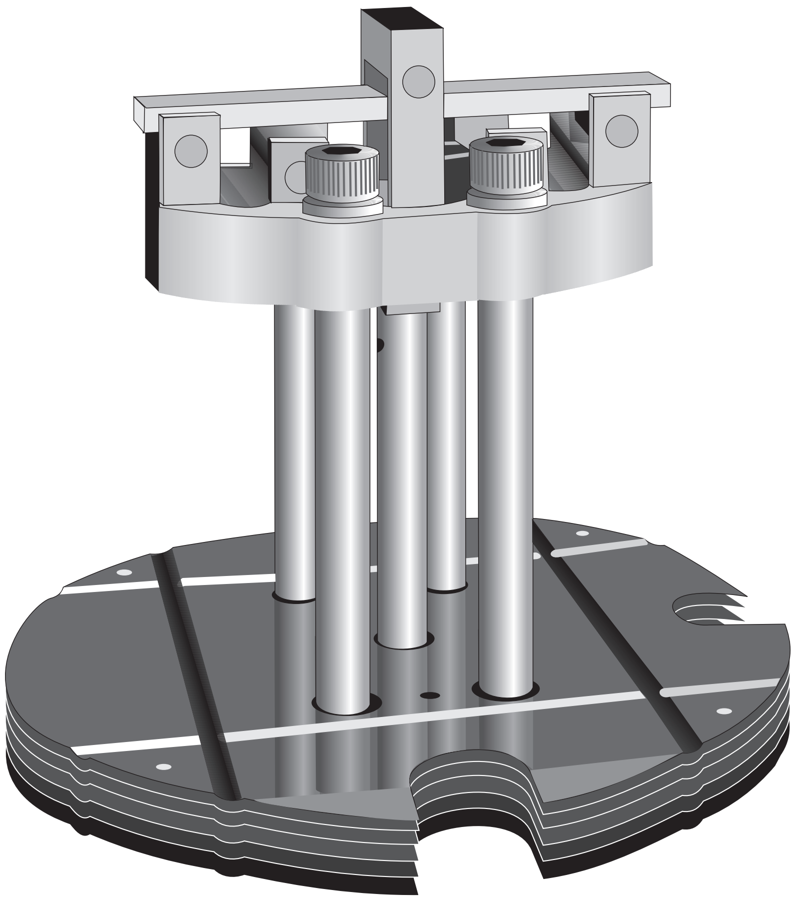


Figure 5.1
Large 3-Point
Bending Clamp

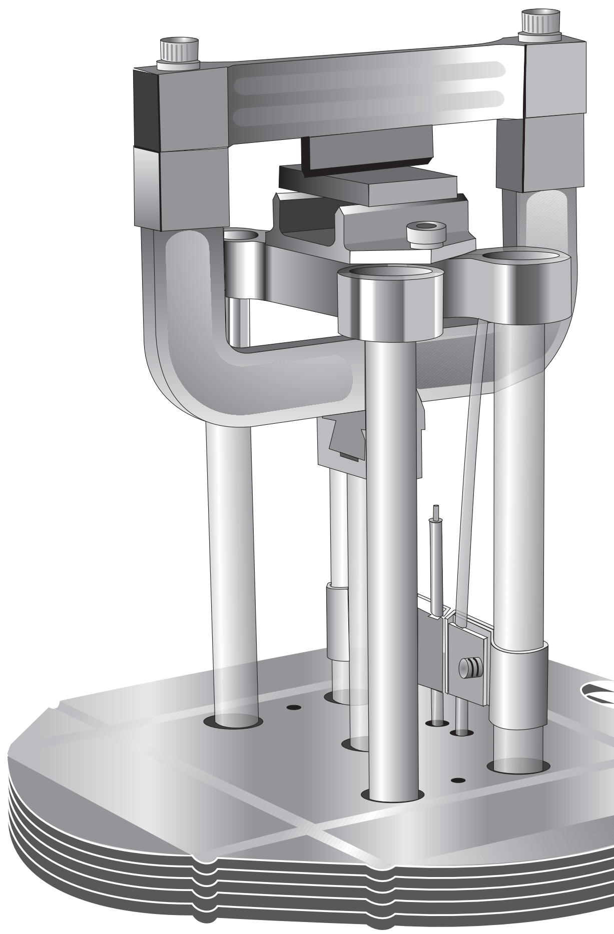


Figure 5.2
Small 3-Point Bending Clamp

To learn how to install the 3-point bending clamps, mount the samples, and remove the clamps, read the next several pages.

Installing the Large Clamps

To install the 20 or 50 mm 3-point bending clamp on the DMA 2980, follow these steps (refer to Figure 5.3 on the next page for identification of the parts):

1. Insert the 3-point bending moveable clamp into the dovetail holder and align the clamp with the holder.

2. Tighten the setscrew with a hex wrench. Do not over tighten the setscrew.

3. Install the large 3-point bending fixed clamp by lowering it onto the mounting posts, with the four screw holes centered over the four mounting posts. Take care not to damage the thermocouple.

4. Seat the fixed clamp firmly onto all four of the mounting posts. Then tighten the screws with a hex wrench. Do not over tighten the attachment screws.

5. Select the clamp type and mode using the *Thermal Solutions/Advantage* Instrument Control program. See Chapter 3 of this manual and the *Thermal Solutions/Advantage User Reference Guide* for information.

6. Calibrate the clamp for mass and compliance as directed in Chapter 3.

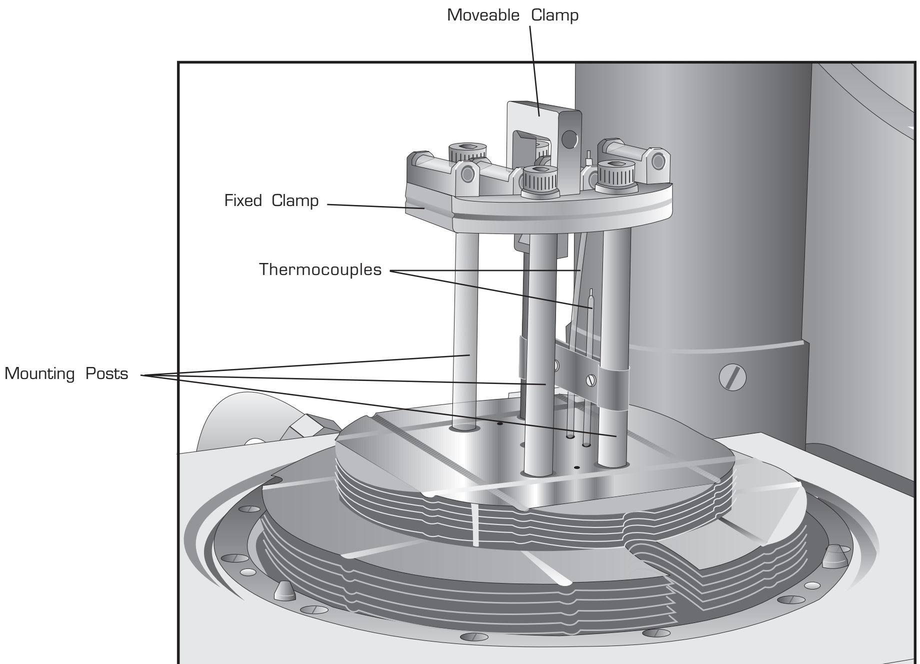


Figure 5.3
Large 3-Point Bending Clamp Installed

Installing the Small Clamps

To install the 5, 10, or 15 mm 3-point bending clamps on the DMA 2980, follow these steps (refer to Figure 5.4 on the next page for identification of the parts):

1. Raise the furnace.

2. Insert the 3-point bending moveable clamp into the dovetail holder and align the clamp with the holder.

3. Tighten the setscrew with a hex wrench. Do not over tighten the setscrew.

4. Install the 5, 10, or 15 mm base onto the 3-point bending fixed clamp. (This step can be done before or after mounting the fixed clamp.)

5. Slide the fixed clamp into position, then lower it onto the mounting posts with the four screw holes centered over the four mounting posts. Take care not to damage the thermocouple.

6. Seat the fixed clamp firmly onto all four of the mounting posts. Then tighten the screws with a hex wrench. Do not over tighten the attachment screws.

7. Select the clamp type and mode using the Instrument Control program. See Chapter 3 of this manual and the *User Reference Guide* for information.

8. Calibrate the clamp for mass and compliance as directed in Chapter 3.

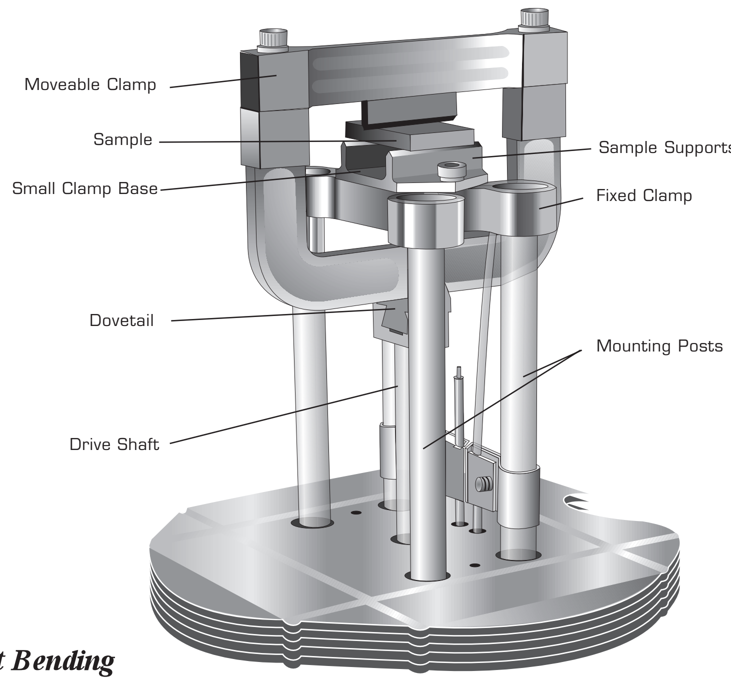


Figure 5.4
Small 3-Point Bending Clamp Installed

3-Point Bending Sample Preparation

Sample preparation is one of the most important factors in achieving accurate and reproducible modulus values. Each type of clamp requires a specific method for sample preparation.

This section contains the procedures for the 3-point bending clamps.

Ideally, samples should be molded, machined, or otherwise fashioned into a rectangular shape for use with either of the 3-point bending clamps. The 3-point bending clamps are very sensitive to any sample malformation, therefore, the rectangle must be well formed, without any twists or buckles.

- **Thickness**: The thickness of the rectangle should be 1/10 to 1/32 of the span (length) of the chosen 3-point bending sample supports. The maximum thickness is 7 mm. The thickness should be as uniform as possible across the sample, to within 0.02 mm or less.

NOTE: It is very important that the sample have a uniform thickness, and that the thickness is accurately measured. The cube of the sample thickness is used in the modulus calculation; therefore, a 3% error in thickness becomes a 10% error in the calculated modulus.

- **Width**: The width of the rectangle should be 5 to 15 mm. The width and thickness dimensions should be uniform across the sample to within 0.02 mm.

- **Length:** Cut the sample 5 mm longer than the span of the chosen 3-point bending clamp sample supports (50, 20, 15, 10 or 5 mm). Make certain the sample does not touch the furnace.

Other sample shapes such as cylinders and tubes can be used in the 3-point bending clamps.

Operating Range of the 3-Point Bending Clamps

The equation below can help you determine which sample clamps to use and the appropriate sample size. This equation can also help determine if the properties of a sample of a particular size can be measured or if the sample dimensions will have to be changed.

Shown in this section is a figure displaying the modulus range versus the possible sample size range for the sample clamps. The possible sample sizes are calculated as geometry factors (GF) in the equation below. The modulus range is based on the range of stiffness over which the DMA 2980 can operate (10^2 to 10^7 N/m).

Geometry Factor Equation for 3-Point Bending Clamps with Rectangular Samples:

$$GF = \frac{L^3}{48I}\left[1 + \frac{12}{5}(1+\nu)\left(\frac{t}{L}\right)^2\right]$$

where:

L = sample length (mm)

I = geometric moment (mm^4) = 1/12 t^3W for rectangular samples

t = sample thickness (mm)

W = sample width (mm)

ν = Poisson's ratio (nominally 0.44).

These equations are explained in greater detail in Chapter 6.

Also shown on the figure below are some typical sample geometries.

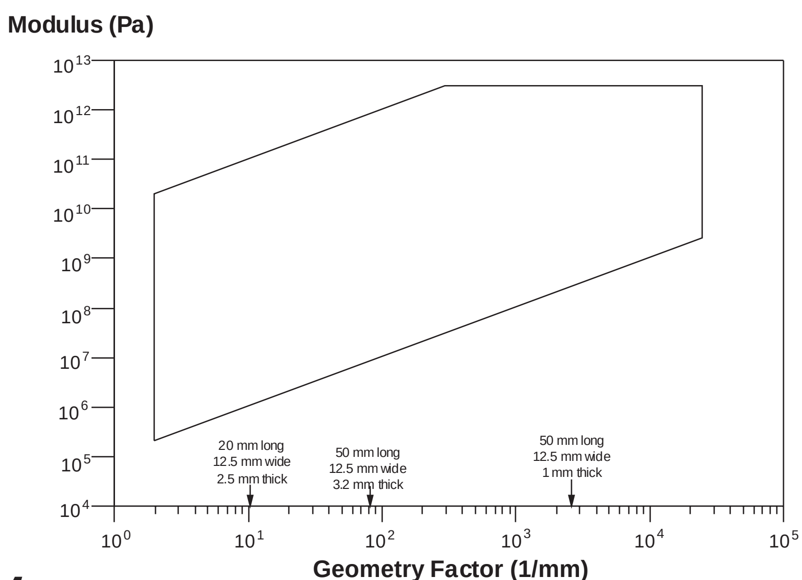


Figure 5.5
Operating Range of the 3-Point Bending Clamps

Mounting a Sample on the 3-Point Bending Clamps

To mount the rectangular sample on the 3-point bending clamps, follow these steps:

1. Press the FLOAT key on the DMA 2980 to allow the clamp to float freely.

2. Manually lift the moveable clamp and slide the sample in from the side onto the two supports.

♦ ***CAUTION:*** **Make sure that the sample does not touch either side of the moveable clamp, the furnace, or the heating elements. See Figure 5.4 on page 5-17.**

3. Align the sample so that it lays perpendicular to the cross member of the support.

4. Adjust the thermocouple so that it is 1 mm below and 1 mm to the side of the sample. When using the small 3-point bending clamps, the thermocouple should be as close as possible to the sample.

Running an Experiment

When you use any of the 3-point bending clamps for experiments, you will need to set up the experimental parameters using the Instrument Control software.

♦ ***CAUTION:*** **When one of the small 3-point bending clamps is used, you must make sure that both the fixed and moveable clamps are installed. The furnace will hit the clamp when it is closed, unless both parts are installed.**

Following these general guidelines when using a 3-point bending clamp:

1. Set up the instrument parameters as directed in Chapter 4. Note that the 3-point bending clamps are tensioning clamps; therefore, autostrain and static force parameters must be entered. The recommended values are 150 to 200 % for autostrain and 0.005 to 1 N for static force.

NOTE: If Autostrain is not selected, then the constant static force is used throughout the experiment. Otherwise the static force is adjusted to maintain the proper autostrain force ratio.

2. Press the CLAMP ▼ key on the DMA 2980 instrument. This will apply the static force selected in step 1.

3. Press the MEASURE key on the DMA. This will start the motor in the selected operation mode and activate the autostrain parameter (if selected in step 1).

4. View the signals. Pay special attention to these signals:

   Amplitude: This signal should achieve and maintain the value programmed. If running a multistrain experiment, the amplitude will cycle through the values programmed.

   Stiffness: The stiffness should be within the instrument's measurable range of 100 N/m to 10,000,000 N/m.

   Drive Force: The drive force should be between 0.0001 and 18 N. If the drive force is not within this range, either increase the programmed amplitude to increase the drive force, or increase the stiffness of the sample by changing the physical dimensions of the sample.

   Static Force (Preload Force): If autostrain (force track) is used, the static force (preload force) will automatically be adjusted to remain a set percentage greater than the force required to drive the sample at the programmed amplitude. If autostrain (force track) is not used, the static force (preload force) should read the value set in the instrument parameters. For more information regarding static (preload) force, refer to page 5-8.

If the initial conditions are acceptable, (*i.e.*, smooth oscillation and good modulus values), start the experiment.

If the values are not acceptable, then change conditions appropriately.

Heat Deflection Temperature

When you use the 3-point bending clamps, experiments with temperature ramps are useful for examining softening points. The large (50 mm) 3-point bending clamp can be used to measure heat deflection temperature (HDT) according to ASTM D648. The procedure for this experiment is summarized below (it contains some minor modifications to accommodate the DMA 2980 dimensions):

1. Form a sample 55 mm long (2.2 in. long) x 12.5 mm (1/2 in.) wide x 3.1 mm (1/8 in.) thick.

2. Select the DMA Controlled Force mode on the DMA Instrument Control program.

3. Calculate the force necessary to achieve the desired stress level, 0.455 MPa (66 psi) or 1.82 MPa (264 psi) according to the equation:

   $F = 2/3 * \sigma (T^2W/L)$

   where:

   F = force (N)
   σ = stress (MPa)
   T = thickness (mm)
   W = width (mm)
   L = length (mm) = 50 mm

4. Place the sample on the clamp and heat at 2°C/min.

   The heat deflection temperature (HDT) is the temperature at which 0.566-mm deflection (0.2 % strain) occurs.

Removing Samples

When the experiment has run to completion, remove the sample from the 3-point bending clamp as follows:

1. Wait for the sample to return to room temperature before you attempt to remove it.
2. Press the FURNACE key to raise the furnace.
3. Press the CLAMP (UP) key twice to raise the moveable clamp.
4. Press the FLOAT/LOCK key to lock the moveable clamp in the upper position.

If further cleaning is needed, turn to Chapter 7 for further information.

♦ ***CAUTION:*** **Be careful not to scratch the contact surface of the clamps.**

Removing the Clamp

The following procedure is used to remove the 3-point bending clamp.

1. Press FLOAT/LOCK to lock the clamp in place.

2. Loosen, but do not remove, the four fixed clamp attachment bolts using a hex wrench.

3. Lift the fixed clamp off the four supports. It may be necessary to raise the moveable clamp to its upper position before the fixed clamp can be removed.

4. Loosen the setscrew on the moveable clamp and then remove the clamp by sliding it out of the dovetail holder.

Shear Sandwich Clamp

The shear sandwich clamp is used to measure samples ranging from unsupported viscous liquids to elastomers above the glass transition point. Much like cantilever clamps, the shear clamp does not require static (preload) force during testing. It should be noted that results are strongly dependent on clamping conditions: Stiff samples tend to slip, and the apparent modulus of an elastomeric sample can change dramatically with clamping force.

Sample Size

Plates: 10 mm x 10 mm
Thickness: up to 4 mm each side

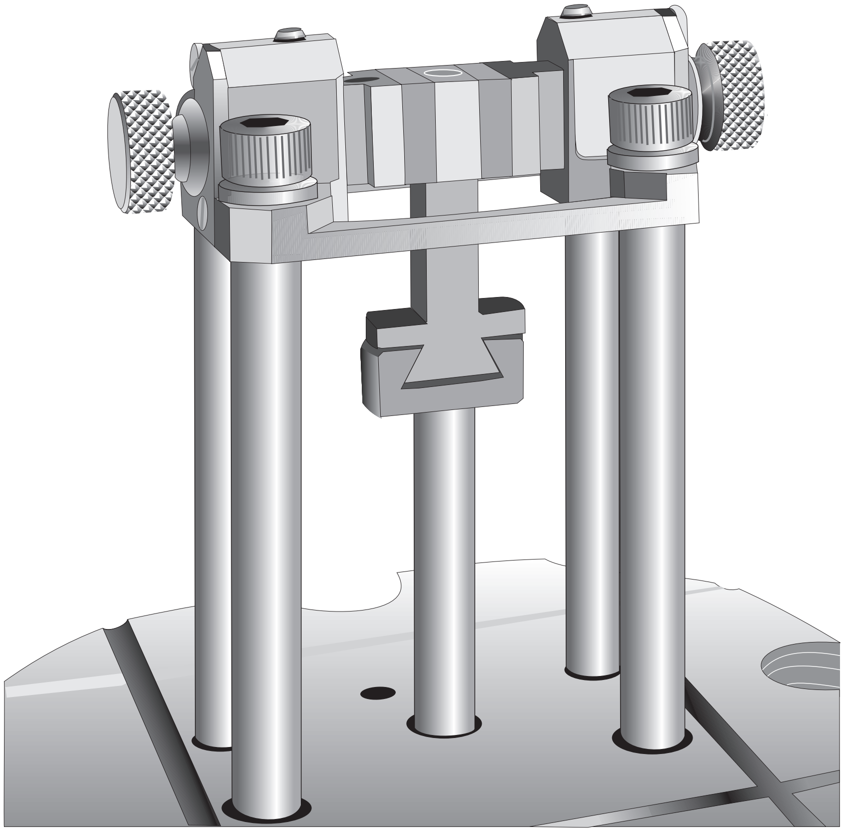


Figure 5.6
Shear Sandwich Clamp

To learn how to install the shear sandwich clamp, mount the samples, and remove the clamp, read the next several pages.

Installing the Clamp

To install the shear sandwich clamp on the DMA 2980 follow these steps (refer to the figure on the next page for identification of the parts):

1. Insert the dovetail of the moveable clamp into the dovetail holder, and align the clamp with the holder.

2. Use the hex key to tighten the setscrew in the center of the moveable clamp.

3. Lower the fixed clamp carefully over the moveable clamp. (You may need to reposition the thermocouple.)

4. Seat the fixed clamp firmly onto all four of the mounting posts. Then tighten the screws with a hex wrench. Do not over tighten the attachment screws.

NOTE: You may need to readjust the front-to-back position of the moveable clamp to ensure that the sides of the moveable clamp are flush with the sides of the fixed clamp.

5. Select the clamp type and mode using the Instrument Control program. See Chapter 3 of this manual and the *User Reference Guide* for information.

6. Calibrate the clamp mass as directed in Chapter 3.

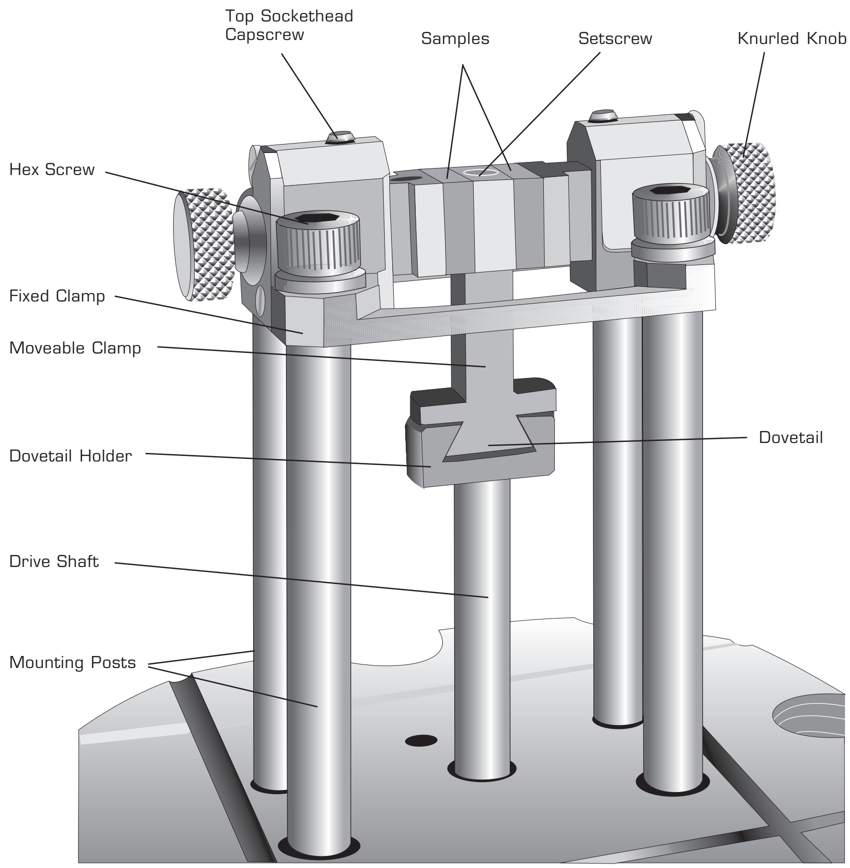


Figure 5.7
Shear Sandwich Clamp
(Shown with Sample Mounted)

Operating Range of the Shear Sandwich Clamp

The equation below can help you determine which sample clamps to use and the appropriate sample size. This equation can also help determine if the properties of a sample of a particular size can be measured or if the sample dimensions will have to be changed.

Shown in this section is a figure displaying the modulus range versus the possible sample size range for the sample clamps. The possible sample sizes are calculated as geometry factors (GF) in the equation below. The modulus range is based on the range of stiffness over which the DMA 2980 can operate (10^2 to 10^7 N/m).

Geometry Factor Equation for Shear Sandwich Clamp:

$$GF = \frac{3}{10}\frac{T}{A}$$

where:

T = sample thickness (mm) (= side A+side B)

A = sample cross sectional area (mm^2).

These equations are explained in greater detail in Chapter 6. The sample should never be larger than the area of the clamp faces.

Also shown on the figure, seen on the next page, are some typical sample geometries.

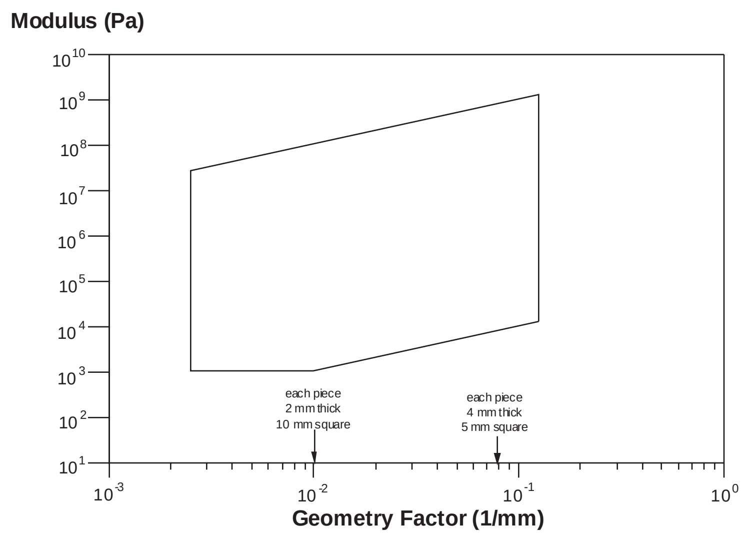


Figure 5.8
Operating Range of
the Shear Sandwich Clamp

NOTE:

The shear sandwich clamp will not work very well with high modulus materials such as polymers below Tg.

Mounting a Solid Sample

You will need to use two samples of equal size and shape for the shear sandwich clamp. Each sample can be up to 10 mm square and up to 4 mm thick. To mount the samples on the shear sandwich clamp, follow these steps:

1. Press FLOAT/LOCK to release (float) the clamp.

2. Manually adjust the moveable clamp until it is aligned with the clamp faces of the fixed clamp, then press LOCK to hold it in place.

3. Tighten the knurled knobs evenly to press the two clamp faces against the moveable clamp. Check to make sure that the moveable clamp is not being pushed away from its centered position by either clamp face. If the moveable clamp is pushed to one side, loosen the knurled knob on the opposite side until the clamp is centered once again.

4. Turn the knurled knob on one side of the fixed clamp until there is enough room between the clamp faces to insert the sample. (The sample should not be larger than the clamp faces.)

5. Hold the sample in place while you tighten the knurled knob. Be careful that you do not tighten the knob too much and over compress the sample. To ensure that the sample is not overcompressed, tighten the knurled knob until the clamp face contact the sample (giving you a slight resistance on the knob). Then tighten the knob an additional one-quarter turn to compress the sample slightly.

6. Repeat steps 4 and 5 for the remaining sample.

7. Tighten the two top sockethead capscrews once the samples have been mounted.

8. Adjust the thermocouple so that it is as close as possible to the sample.

Mounting a Liquid or Gel Sample

You can also analyze certain types of liquid or gel samples on the DMA 2980.

To mount the liquid or gel samples on the shear sandwich clamp, follow these steps:

1. Press FLOAT/LOCK to release (float) the clamp.

2. Manually adjust the moveable clamp until it is aligned with the clamp faces of the fixed clamp, then press LOCK to hold it in place.

3. Tighten the knurled knobs evenly to press the two clamp faces against the moveable clamp. Check to make sure that the moveable clamp is not being pushed away from its centered position by either clamp face. If the moveable clamp is pushed to one side, loosen the knurled knob on the opposite side until the clamp is centered once again.

4. Open one of the fixed clamps to an opening that is larger than the desired sample gap. Take note of the number of turns that you have used to open the clamp.

NOTE: The knurled knobs are not calibrated micrometer screws, but they are reasonably able to set equal distances on both sides of the clamps. This is important for obtaining accurate results.

5. Load the sample into the gap between the clamp faces. Try to load the sample into the center of the clamp face. Use enough sample so that when the clamps are compressed, a small, excess amount will be squeezed out of the clamp. Do not overfill the clamps.

6. Turn the knurled knob to close the gap to the desired value—up to 2 mm for heavier gels.

7. Tighten the top sockethead capscrew and measure the gap (containing the sample) with calipers.

8. Trim any excess material from around the clamp using a spatula or similar implement.

9. Repeat steps 4 through 8 for the remaining side. When repeating step 4, use the same number of turns to set the second clamp gap as you did for the first. Be careful when measuring the second gap so that the moveable clamp is not disturbed. Remember that the sample length is equal to the total of side A + side B. The dimension of the clamp face is 10 mm by 10 mm.

10. Adjust the thermocouple so that it is as close as possible to the sample.

Running an Experiment

When you use the shear sandwich clamp for experiments, you will need to set up the experimental parameters using the Instrument Control software. Follow these general guidelines when using the shear sandwich clamp:

1. Set up the experimental parameters as directed in Chapter 4.

2. Press the MEASURE key on the DMA. This will start the motor according to the operation mode, frequency range, etc.

3. View the signals. Pay special attention to these signals:

   Amplitude: This signal should achieve and maintain the value programmed. If running a multistrain experiment, the amplitude will cycle through the values programmed.

   Stiffness: The stiffness should be within the instrument's measurable range of 100 N/m to 10,000,000 N/m.

   Drive Force: The drive force should be between 0.0001 and 18 N. If the drive force is not within this range, either increase the programmed amplitude to increase the drive force, or increase the stiffness of the sample by changing the physical dimensions of the sample.

If the initial conditions are acceptable, (*i.e.*, smooth oscillation and good modulus values), start the experiment.

If the values are not acceptable, then change conditions appropriately.

Removing Samples

To remove the samples from the shear sandwich clamp, loosen the two top setscrews and reverse the steps used to mount the samples.

If the clamp becomes stuck together by the sample (such as with some resin materials), you may need to remove the clamp and samples as one piece and clean the clamps at a location away from the instrument. Follow these steps to remove samples:

1. Loosen, but do not remove, the four hex screws that hold the fixed clamp in place on the mounting posts.

2. Use the hex key to loosen the setscrew in the center of the moveable clamp.

3. Press the FLOAT/LOCK key to release (float) the clamp.

4. Manually raise the clamp and samples off the mounting posts.

5. Press the FLOAT/LOCK key to lock the drive shaft.

6. Slide the dovetail of the moveable clamp out of the dovetail holder of the drive shaft.

The whole clamp assembly and samples are now free of the instrument and can be cleaned.

Removing the Clamp

To remove the shear sandwich clamp, follow these steps:

1. Loosen, but do not remove, the four hex screws that hold the fixed clamp in place on the mounting posts.

2. Lift off the fixed clamp.

3. Use the hex key to loosen the setscrew in the center of the moveable clamp.

4. Slide the dovetail of the moveable clamp out of the dovetail holder of the drive shaft.

Compression Clamp

Compression is used to measure the properties of low to medium modulus materials, including gels and weak elastomers. The sample must support a static (preload) force during testing. The sample should have as high a thickness-to-diameter ratio as is possible, depending on the sample preparation and instrument limits.

Sample Size

Diameter: up to 15 mm and 40 mm
Thickness: up to 10 mm

♦ ***CAUTION:***

When the compression clamp is used, you must make sure that both the fixed and moveable clamps are installed. The furnace may hit the clamp when it is closed, unless both parts are installed.

Figure 5.9
Compression Clamp

To learn how to install and align the compression clamp, mount the samples, and remove the clamp, read the next several pages.

Installing the Clamp

To install the compression clamp on the DMA 2980, follow these steps:

1. Slide the lower section of the moveable clamp onto the dovertail holder and align the dovetail with the holder. Lightly tighten the center setscrew only.

2. Press the CLAMP ▼ key on the instrument keypad to lower the moveable clamp so that the four mounting posts are easily accessible.

3. Seat the fixed clamp firmly onto all four of the mountings posts and tighten the screws with a hex wrench. Do not over tighten the attachment screws. Be careful not to damage the thermocouple.

4. Screw the lower compression disk (15 mm or 40 mm) onto the fixed clamp. See Figure 5.10.

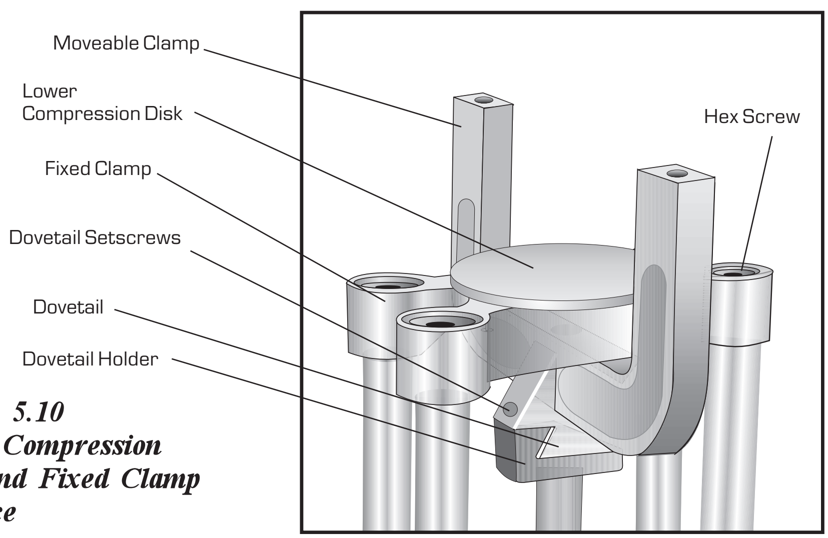


Figure 5.10 Lower Compression Disk and Fixed Clamp in Place

5. Attach the upper compression disk (15 mm or 40 mm) onto the upper section of the moveable clamp.

6. Press the CLAMP ▲ key on the instrument keypad to raise the clamp.

7. Attach the upper moveable clamp section (with the upper compression disk attached) to the U-shaped lower section of the moveable clamp with the two Allen screws and washers provided. The clamp should now be fully assembled as seen in Figure 5.9.

8. Check the moveable clamp for alignment (front-to-rear parallelism, left-to-right parallelism, and concentricity). Parallelism can be checked either visually or by pulling a piece of paper between the compression disks. If they are properly aligned, the paper should drag evenly when pulled. If the clamp needs to be realigned, use the procedures in the next section.

Aligning the Clamp

To align the compression clamp follow these steps:

NOTE: The clamp in Figure 5.11 is shown rotated 90 degrees from the acutal mounting configuration on the instrument; this is to allow you to more easily compare the adjustments to the instructions.

1. Adjust the left-to-right parallelism as follows:

   a. Loosen the center setscrew. If the upper compression disk is high on the left side, turn the right dovetail setscrew clockwise to make the adjustment. If the disk is high on the right side, turn the left dovetail setscrew clockwise to make the adjustment.

b. Tighten the center setscrew again and recheck the left-to-right parallelism either visually or by pulling a piece of paper between the compression disks. If they are properly aligned, the paper should drag evenly when pulled. Repeat steps 1a and 1b, if needed, or adjust the front-to-rear parallelism (see step 2).

2. Adjust the front-to-rear parallelism as follows:

   a. Remove the Allen screws from the upper section of the moveable clamp.

   b. Insert the appropriate number of shims needed to adjust the parallelism correctly. If the upper clamp is high in the back, place shims in the front, and vice versa. (See Figure 5.11 on the next page.)

3. Adjust the upper disk for concentricity as follows: Loosen the upper moveable clamp Allen screws. Manually move the disks into alignment. The screw holes in the upper part of the moveable clamp are "oversized" to allow this adjustment.

4. Tighten all Allen screws and setscrews and recheck the parallelism and concentricity. Repeat any of the steps above, if needed.

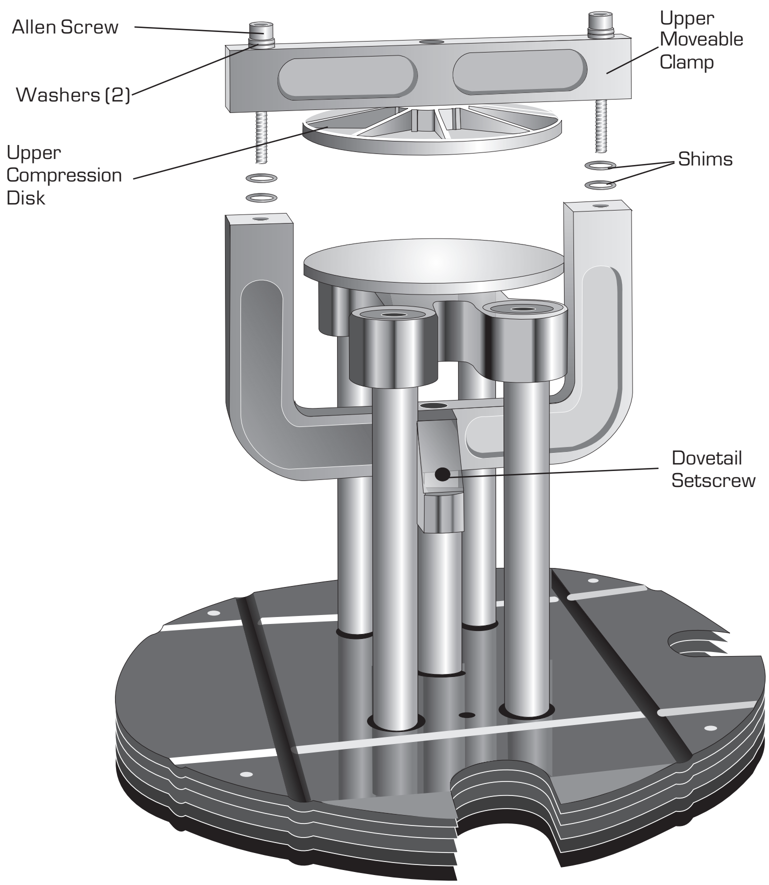


Figure 5.11
Adding Shims to the Compression Clamp for Alignment

5. Select the clamp type and mode using the Instrument Control program. See Chapter 3 of this manual and the online documentation for further information.

6. Calibrate the clamp mass, offset, and compliance as directed in Chapter 3.

Operating Range of the Compression Clamp

The equation below can help you determine which sample clamps to use and the appropriate sample size. This equation can also help determine if the properties of a sample of a particular size can be measured or if the sample dimensions will have to be changed.

Shown in this section is a figure displaying the modulus range versus the possible sample size range for the sample clamps. The possible sample sizes are calculated as geometry factors (GF) in the equation below. The modulus range is based on the range of stiffness over which the DMA 2980 can operate (10^2 to 10^7 N/m).

Geometry Factor Equation for Compression:

$$GF = Fe\frac{T}{A}$$

where:

T = Sample thickness (height) (mm)
A = Sample cross sectional area (mm^2)
Fe = Sample shear factor. This factor is a strong function of Poisson's ratio and the thickness to area ratio of the sample. Range: 1.0 to 0.1. See "Clamping Factors" in Chapter 6 to determine the value of Fe.

Samples which are tall relative to the diameter tend to work better in the compression clamps. The sample should never be larger than the area of the clamp faces.

Also shown on the figure below are some typical sample geometries.

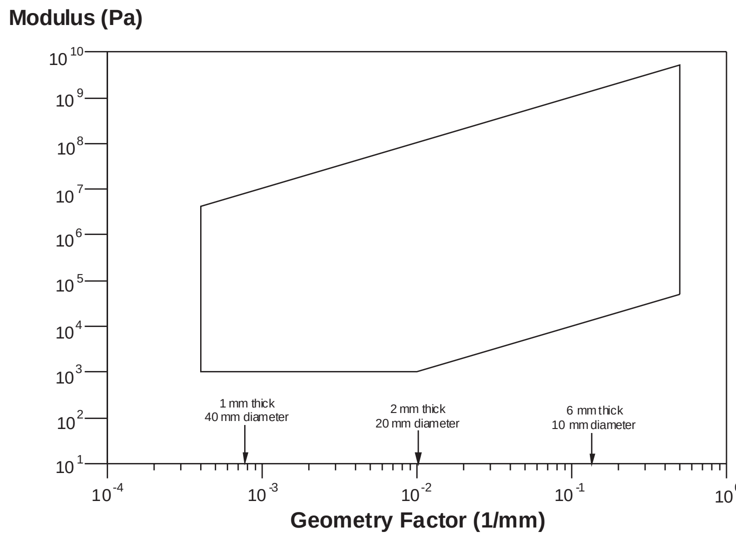


Figure 5.12
Operating Range of the Compression Clamps

NOTE: The compression clamps will not work very well with high modulus materials such as polymers below Tg.

Mounting a Sample

To mount a sample on the compression clamp, follow these directions:

NOTE: If the sample is expected to crosslink or cure, we recommend that you protect the clamp faces with thin metal foil.

1. Press the FLOAT/LOCK key to release (float) the clamp.

2. Manually raise the moveable clamp and place the sample on the bottom disk, making sure that it does not extend beyond the edges of the disk.

3. Apply the desired static force using the Instrument Control program. (See the *User Reference Guide* for details.)

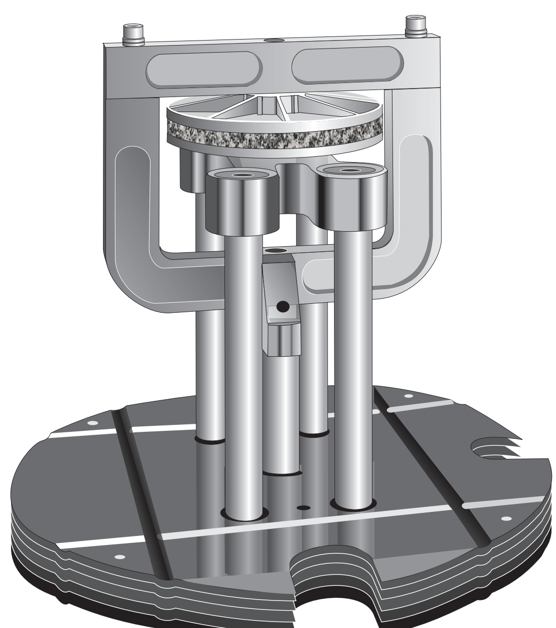


Figure 5.13 Compression Clamp (Shown with Sample Mounted)

4. Adjust the thermocouple so that it is as close as possible to the sample.

Running an Experiment

When you use the compression clamp for experiments, you will need to set up the experimental parameters using the instrument control software.

♦ ***CAUTION:*** **When the compression clamp is used, you must make sure that both the fixed and moveable clamps are installed. The furnace will hit the clamp when it is closed, unless both parts are installed.**

Follow these general guidelines when using the compression clamp:

1. Set up the experimental parameters as directed in Chapter 4. Note that these clamps are tensioning clamps; therefore, autostrain (force track) and static force (preload force) values must be selected.

   The recommended values are 0.005 to 1 N for static force (preload force) and 115 to 200 percent for autostrain (force track), if appropriate.

NOTE: When you use weak samples such as gel foams and uncured elastomers, low amplitude (1-5 µm) and low autostrain (115 to 125%) values are recommended for multistrain and multifrequency DMA experiments.

2. Press the CLAMP ▼ key on the instrument keypad to move the clamp faces together and compress the sample under the static force (preload force) selected in step 1.

3. Press the MEASURE key on the DMA. This will measure the sample thickness and automatically enter the thickness into the experimental parameters. Then the motor will start in the desired operation mode.

4. View the signals. Pay special attention to these signals:

   Amplitude: This signal should achieve and maintain the value programmed. If running a multistrain experiment, the amplitude will cycle through the values programmed.

   Stiffness: The stiffness should be within the instrument's measurable range of 100 N/m to 10,000,000 N/m.

   Drive Force: The drive force should be between 0.0001 and 18 N. If the drive force is not within this range, either increase the programmed amplitude to increase the drive force, or increase the stiffness of the sample by changing the physical dimensions of the sample.

   Static Force (Preload Force): If autostrain (force track) is used, the static force (preload force) will automatically be adjusted to remain a set percentage greater than the force required to drive the sample at the programmed amplitude. If autostrain (force track) is not used, the static force (preload force) should read the value set in the instrument parameters. For more information regarding static (preload) force, refer to page 5-8.

If the initial conditions are acceptable, (*i.e.*, smooth oscillation and good modulus values), start the experiment.

If the values are not acceptable, then change conditions appropriately.

Removing Samples

To remove a sample from the compression clamp follow these guidelines.

If the sample is not stuck to the clamps:

1. Press the CLAMP ▲ key on the instrument keypad to raise the moveable clamp.

2. Remove the sample and clean the compression disk face thoroughly. The disks can be removed, if necessary. Be careful not to scratch their surfaces.

If the sample has cured, or crosslinked, or otherwise bonded the disks together:

1. Use a hex wrench to remove the screws on top of the moveable clamp.

2. Press the CLAMP ▼ key on the instrument keypad to lower the moveable clamp.

3. Unscrew the compression disks from the fixed clamp. The disks can now be taken away from the instrument to be cleaned. Be careful not to scratch their surfaces.

Removing the Clamp

To remove the compression clamp from the DMA 2980, follow these steps:

1. Unscrew and remove the lower compression disk from the fixed clamp.

2. Press the CLAMP ▲ key on the instrument keypad to raise the moveable clamp.

3. Loosen, but do not remove, the four hex screws holding the fixed clamp, then remove the fixed clamp.

4. Loosen the setscrew holding the moveable clamp on the drive shaft.

5. Slide the moveable clamp off the dovetail holder.

Penetration Clamp

The penetration clamp is only used for TMA penetration analysis on the DMA 2980. Information such as glass transition or melting points can be obtained. Almost any type of material can be used as a sample with the penetration clamp installed.

Sample Size

Diameter: up to 15 mm and 40 mm
Thickness: up to 10 mm

♦ ***CAUTION:***

When the penetration clamp is used, you must make sure that both the fixed and moveable clamps are installed. The furnace may hit the clamp when it is closed, unless both parts are installed.

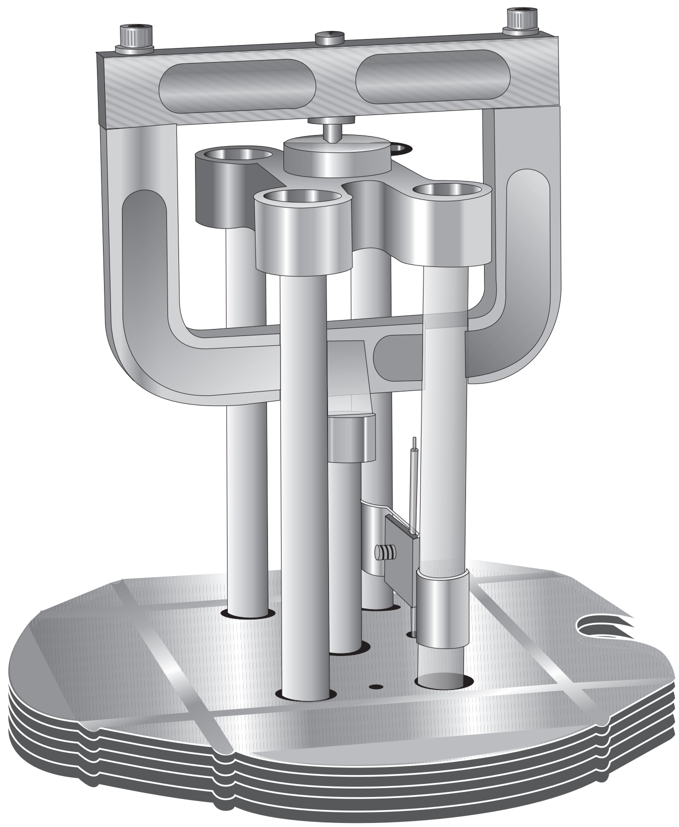


Figure 5.14 Penetration Clamp

To learn how to install the penetration clamp, mount the samples, and remove the clamp, read the next several pages.

Installing the Clamp

To install the penetration clamp on the DMA 2980 instrument, follow these steps:

1. Screw the penetration piece into the moveable clamp before installing it onto the instrument.

2. Slide the moveable clamp (with the penetration piece in place) onto the instrument. Align the dovetail with the holder and tighten the setscrew.

3. Press CLAMP ▲ key on the instrument keypad to raise the moveable clamp.

4. Seat the fixed clamp firmly onto all four of the mounting posts. Then tighten the screws with a hex wrench. Do not over tighten the attachment screws. Be careful not to damage the thermocouple.

5. Select the clamp type and mode using the Instrument Control program. See Chapter 3 of this manual and the *User Reference Guide* for information.

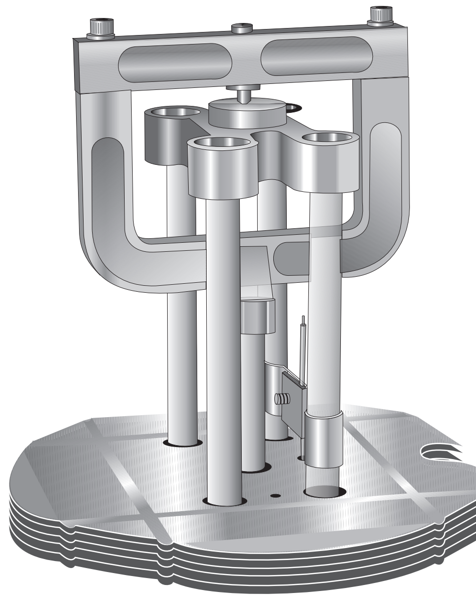


Figure 5.15 Penetration Clamp in Place

6. Calibrate the clamp mass, zero, and compliance as directed in Chapter 3.

Mounting a Sample

After the penetration clamp has been installed on the DMA, you can mount the sample using the following steps:

1. Press the FLOAT/LOCK key to release (float) the clamp.

2. Manually raise the moveable clamp and place the sample on the fixed clamp, making sure that it does not extend beyond the edges of the fixed clamp area.

3. Press the CLAMP ▼ key on the DMA 2980 keypad to move the clamp down until it just touches the sample.

Running an Experiment

When you use the penetration clamp for experiments, you will need to set up the experimental parameters using the *Thermal Solutions/ Advantage* software.

♦ ***CAUTION:***

When the penetration clamp is used, you must make sure that both the fixed and moveable clamps are installed. The furnace will hit the clamp when it is closed, unless both parts are installed.

Follow these general guidelines when using the penetration clamp:

1. Set up the experimental parameters as directed in Chapter 4. Note that these clamps are tensioning clamps; therefore, autostrain and static force values must be selected.

   The recommended values are 0.005 to 1 N for static force and 115 to 200 percent for autostrain (if appropriate).

2. Press the MEASURE key on the DMA. This will apply the static force, measure the sample thickness, and automatically enter the thickness into the experimental parameters. Then the motor will start in the desired operation mode.

3. View the signals. Pay special attention to these signals:

   Amplitude: This signal should achieve and maintain the value programmed. If running a multistrain experiment, the amplitude will cycle through the values programmed.

   Stiffness: The stiffness should be within the instrument's measurable range of 100 N/m to 10,000,000 N/m.

   Drive Force: The drive force should be between 0.0001 and 18 N. If the drive force is not within this range, either increase the programmed amplitude to increase the drive force, or increase the stiffness of the sample by changing the physical dimensions of the sample.

If the initial conditions are acceptable, (*i.e.*, smooth oscillation and good modulus values), start the experiment.

If the values are not acceptable, then change conditions appropriately.

Removing the Clamp

To remove the penetration clamp from the DMA 2980, follow these steps:

1. Press the CLAMP ▲ key on the DMA 2980 instrument keypad to raise the moveable clamp.

2. Loosen the four hex screws holding the fixed clamp, then remove the fixed clamp.

3. Loosen the setscrew holding the moveable clamp on the drive shaft.

4. Slide the moveable clamp off the dovetail holder.

Film Tension Clamp

The film tension clamp can be used for film samples that are up to 2 mm thick.

Sample Size

Length:	5 to 30 mm
Width:	up to 6.5 mm
Thickness:	up to 2 mm

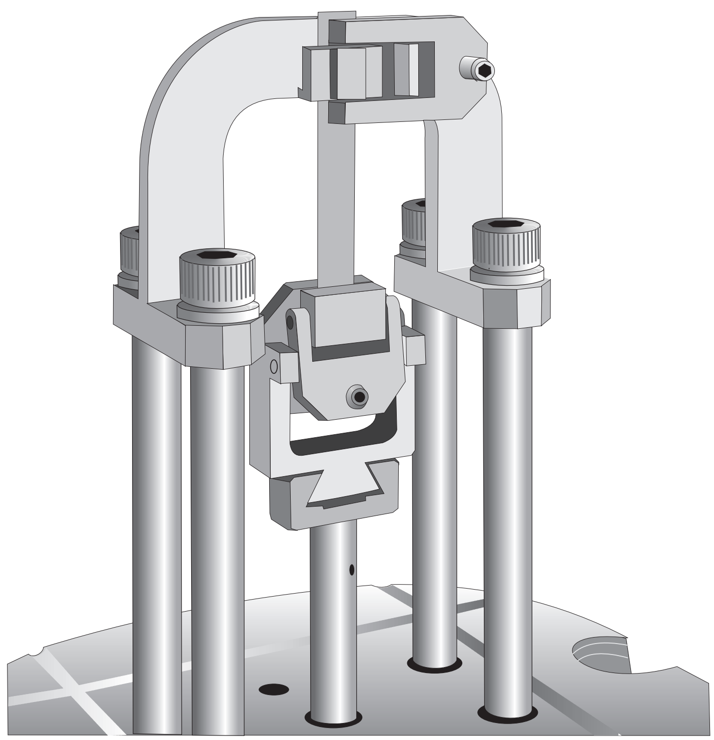


Figure 5.16 Film Tension Clamp

To learn how to install the film tension clamp, mount the samples, and remove the clamp, read the next several pages.

Installing the Clamp

To install the film tension clamp on the DMA 2980 instrument, follow these steps:

1. Place the fixed clamp on the instrument first, aligning the screw holes with the mounting posts.

2. Seat the fixed clamp firmly onto all four of the mounting posts. Then tighten the screws with a hex wrench. Do not over tighten the attachment screws.

3. Raise the drive shaft all the way up using the CLAMP ▲ key on the instrument keypad.

4. Loosen the clamping screw on the moveable clamp and open the clamp face.

5. Slide the hex wrench through the open clamp face of the moveable clamp and loosen the setscrew. Slide the dovetail of the moveable clamp into the dovetail holder on the drive shaft.

6. Loosen the clamping screw on the fixed clamp and open the clamp face.

7. Carefully align the fixed and moveable clamps so that the front face of the horse-shoe frame is parallel to the front face of the moveable clamp. See the figure on the next page for guidance.

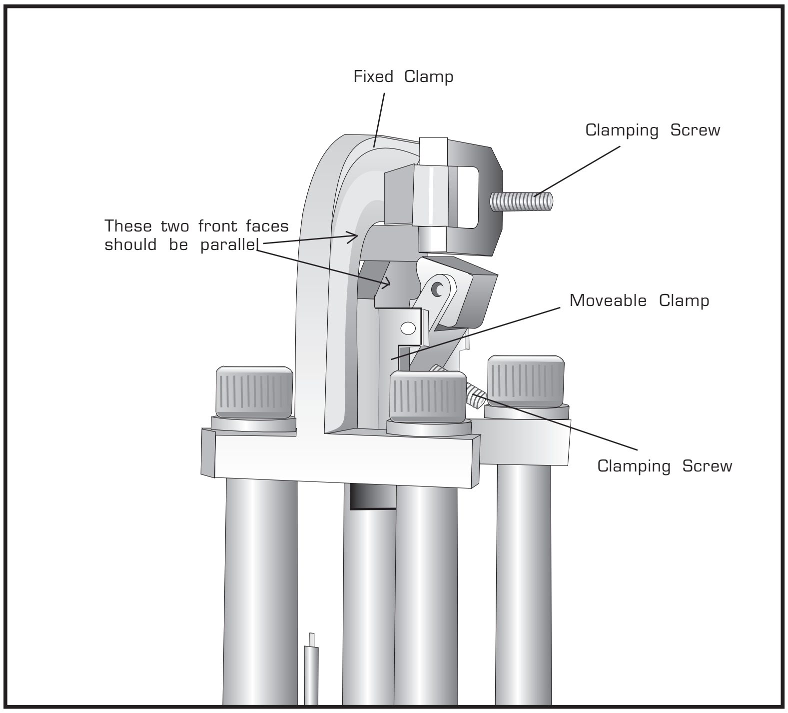


Figure 5.17
Aligning the Fixed and Moveable Clamps of the Film Tension Clamp

8. Tighten the setscrew while holding the moveable clamp in place.

9. Tighten the clamping screws on the moveable and fixed clamps.

10. Select the clamp type and mode using the instrument control program. See Chapter 3 of this manual and the *User Reference Guide* for information.

11. Calibrate the clamp mass and clamp zero as directed in Chapter 3.

Operating Range of the Tension Clamps

The equation below can help you determine which sample clamps to use and the appropriate sample size. This equation can also help determine if the properties of a sample of a particular size can be measured or if the sample dimensions will have to be changed.

Shown in this section is a figure displaying the modulus range versus the possible sample size range for the sample clamps. The possible sample sizes are calculated as geometry factors (GF) in the equation below. The modulus range is based on the range of stiffness over which the DMA 2980 can operate (10^2 to 10^7 N/m).

Geometry Factor Equation for Tension:

$$GF = \frac{L}{A}$$

where:

L = sample length (mm)
A = sample cross sectional area (mm^2).

These equations are explained in greater detail in Chapter 6.

Also shown on the figure below are some typical sample geometries.

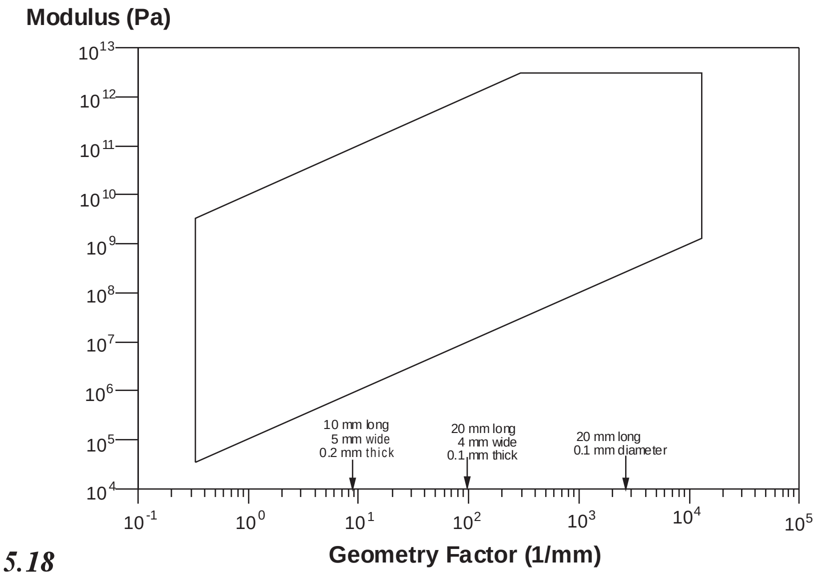


Figure 5.18
Operating Range
of the Tension Clamps

Samples which are 15 to 20 mm long and 3 to 6 mm wide (films 0.5 mm thick and below) or 2 to 3 mm wide (0.5 to 2 mm thick), should yield good results for most materials.

Mounting a Sample

After the film tension clamp has been installed on the DMA, you can mount the sample using the following steps:

NOTE: The clamping of film samples is critical to achieving accurate and reproducible results.

1. Press the FLOAT/LOCK key on the instrument keypad to release (float) the clamp.

2. Move the moveable clamp to the approximate position of the desired specimen length. The sample length can be directly observed as a signal output on the instrument display.

3. Press the FLOAT/LOCK key to lock the clamp in position. (The actual specimen length can be measured by the DMA by using the MEASURE key after the specimen is mounted.)

4. Loosen the clamping screws on the moveable and fixed clamps (see Figure 5.17).

5. Slide the sample in from the side of the fixed clamp, then lower it down into the moveable clamp.

6. Push the moveable clamp shut using your finger, then adjust the sample so that it is centered and aligned vertically on the clamp. Make sure that there is no sample underneath the clamping screw. Finger tighten the clamping screw.

7. Using the torque wrench, tighten the bottom clamp screw on the moveable clamp to the appropriate clamping torque [2 to 3 in-lbs (20 to 40 cm-N) for rigid materials].

!WARNING

Use of excessive torque will bend the stage, causing permanent damage. Use the torque wrench as directed and do not exceed specifications.

8. Carefully align the film so that it is vertical and evenly tensioned across the sample width. Tighten the clamping screw on the fixed clamp. Trim any excess sample.

   See the figure below for an example of a sample mounted on the film tension clamp.

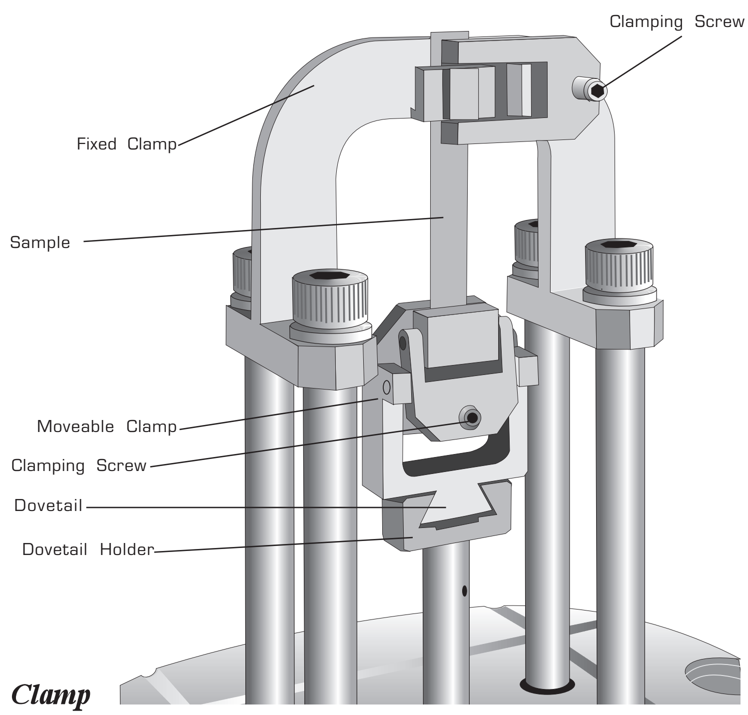


Figure 5.19
Film Tension Clamp (Shown with Sample Mounted)

9. Position the thermocouple so that it is approximately halfway between the two clamps and close to, but not touching, the sample.

10. Place the thermal shield over the clamps, if you are using a heating rate of 2°C/min or higher. NOTE: Do not use the thermal shield when using heating rates less than 2°C/min or when performing a step and hold experiment.

Running an Experiment

When you use the film tension clamp for experiments, you will need to set up the experimental parameters using the instrument control software.

Follow these general guidelines when using the film clamp:

1. Set up the experimental parameters as directed in Chapter 4. Note that these clamps are tensioning clamps; therefore, autostrain (force track) and static force (preload force) values must be selected.

   The recommended values are 0.005 to 1 N for static force (preload force)and 115 to 200 percent for autostrain (force track), if appropriate.

2. Press the MEASURE key on the DMA. This will apply the static force (preload force), measure the sample length, enter the length into the experimental parameters, and then start the motor in the desired operation mode.

3. View the signals. Pay special attention to these signals:

   Amplitude: This signal should achieve and maintain the value programmed. If running a multistrain experiment, the amplitude will cycle through the values programmed.

   Stiffness: The stiffness should be within the instrument's measurable range of 100 N/m to 10,000,000 N/m.

Drive Force: The drive force should be between 0.0001 and 18 N. If the drive force is not within this range, either increase the programmed amplitude to increase the drive force, or increase the stiffness of the sample by changing the physical dimensions of the sample. Since the length is fixed in the film tension clamp, the width and thickness will need to be increased in order to increase the sample stiffness.

Static Force (Preload Force): If auto-strain (force track) is used, the static force (preload force) will automatically be adjusted to remain a set percentage greater than the force required to drive the sample at the programmed amplitude. If autostrain (force track) is not used, the static force (preload force) should read the value set in the instrument parameters. For more information regarding static (preload) force, refer to page 5-8.

If the initial conditions are acceptable, (*i.e.*, smooth oscillation and good modulus values), start the experiment.

If the values are not acceptable, then change conditions appropriately.

Removing a Sample

After you have finished your experiment, you can remove the sample from the film tension clamp as follows:

1. Loosen the two clamping screws and open the clamp faces.

2. Remove the sample.

3. Use a knife or razor blade to gently scrape all clamp faces to remove any residue. Be careful not to scratch the clamp faces.

Removing the Clamp

To remove the film tension clamp from the DMA 2980, follow these steps:

1. Loosen, but do not remove, the four hex screws holding the fixed clamp on the clamp mounting posts.

2. Loosen the setscrew holding the moveable clamp to the drive shaft.

3. Slide the dovetail out to remove the moveable clamp.

4. Remove the fixed clamp.

Fiber Tension Clamp

The fiber tension clamp can be used for individual thin fiber samples or bundles of fibers.

Sample Size	
Length:	5 to 30 mm
Diameter:	up to 1.25 mm

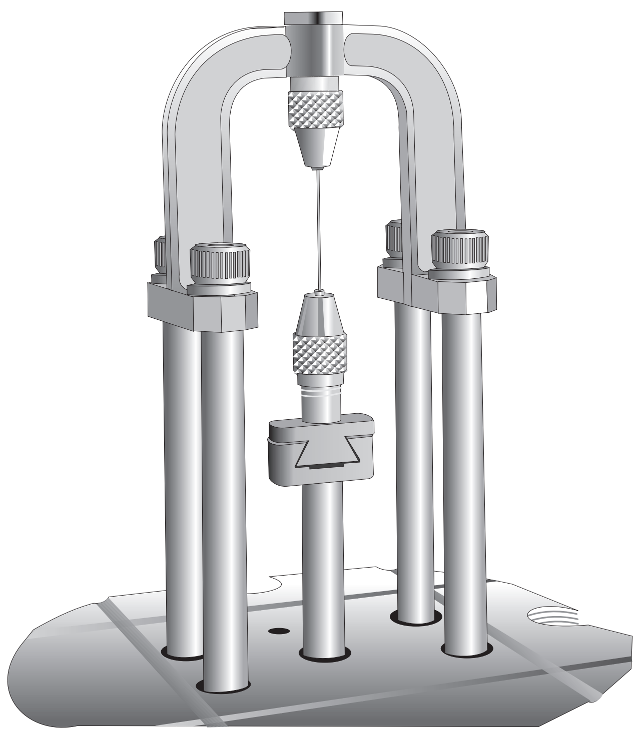


Figure 5.20
Fiber Tension Clamp

To learn how to install the fiber tension clamp, mount the samples, and remove the clamp, read the next several pages.

Installing the Clamp

To install the fiber tension clamp on the DMA instrument, follow these steps (see the figure on the next page for identification of parts):

1. Remove the pin vises from both the fixed and moveable clamps by unscrewing them.

2. Press the CLAMP ▲ key to raise the driveshaft.

3. Press the FLOAT/LOCK key to lock the driveshaft in the upper position.

4. Slide the dovetail of the moveable clamp into the dovetail holder of the drive shaft. Align the dovetail so that it is flush with the dovetail holder.

5. Use the hex key to tighten the setscrew in the center of the moveable clamp.

6. Seat the fixed clamp firmly onto all four of the mounting posts. Then tighten the screws with a hex wrench. Do not over tighten the attachment screws.

7. Place the pin vises into the fixed and moveable clamps.

8. Select the clamp type and mode using the Instrument Control program. See Chapter 3 of this manual and the *User Reference Guide* for information.

9. Calibrate the clamp mass and clamp zero as directed in Chapter 3.

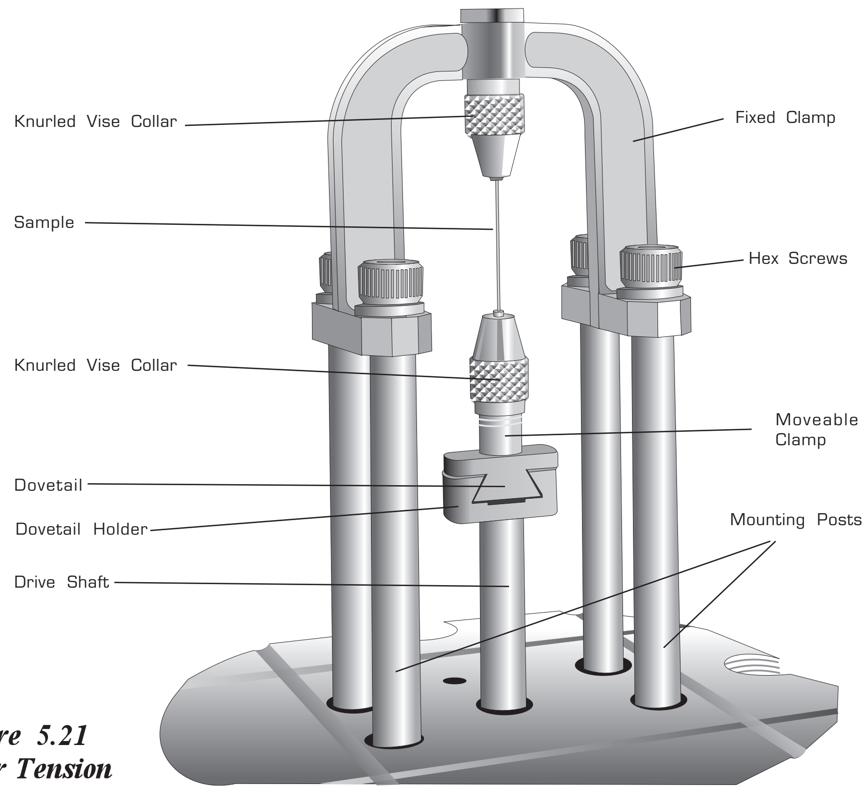


Figure 5.21 Fiber Tension Clamp (Shown with Sample Mounted)

Mounting a Sample

After the fiber tension clamp has been installed on the DMA, you can mount the samples according to the diameter of the fiber: large diameter (high denier) monofilaments or small diameter filaments and fiber bundles. The fiber clamp can hold samples from 5 denier (20 µm) up to 1/32 in. (0.8 mm) in diameter. See the following sections for instructions.

Large Diameter (High Denier) Monofilaments

Follow these steps to mount large diameter monofilaments:

1. Press the FLOAT/LOCK key to release (float) the moveable clamp.

2. Move the moveable clamp to the approximate position of the desired specimen length. The sample length can be directly observed as a signal output on the instrument display.

3. Press the FLOAT/LOCK key to lock the clamp in position. (The actual specimen length can be measured by the DMA by using the MEASURE key after the specimen is mounted.)

4. Loosen both of the knurled vise collars.

5. Feed one end of the monofilament into the moveable clamp and tighten the knurled vise collar.

6. Feed the other end of the monofilament into the fixed clamp. Be sure to use a sample that is long enough to extend beyond the end of the fixed clamp.

7. Pull up on the sample gently and tighten the knurled vise collar on the fixed clamp. Trim off any excess sample.

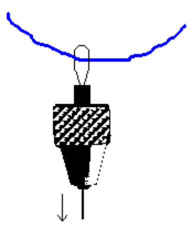
8. Adjust the thermocouple so that it is approximately halfway between the two clamps and close to, but not touching, the sample.

9. Place thermal shield over the clamps, if you are using a heating rate of 2°C/min or higher. Do not use the thermal shield when using heating rates less than 2°C/min.

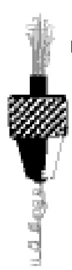
Small Diameter Filaments and Fiber Bundles

1. Press the CLAMP ▼ key to lower the moveable clamp.

2. Remove the knurled vise collar from the moveable clamp.



3. Make a loop of thick monofilament or metal wire and push that loop through the back of the vise collar center and then through the screw. Feed a length of the specimen through the loop of monofilament. Use a specimen that will be long enough to extend beyond the back of the fixed clamp (at least 75 mm, or 3 inches).



4. Carefully pull the monofilament loop back through the center of the pin vise inside the vise collar. Make certain to keep the fibers within the center of the pin vise and vise collar. Adjust or trim the fibers so that they do not extend beyond the back of the vise collar.

5. Carefully replace the vise collar with the fibers onto the moveable clamp. Tighten the knurled vise collar.

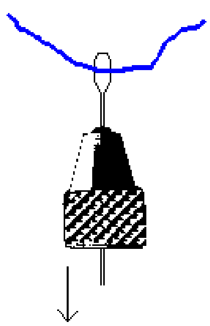
6. Press the FLOAT/LOCK key to release the moveable clamp.

7. Move the moveable clamp to the approximate position of the desired specimen length. The sample length can be directly observed as a signal output on the instrument display.

8. Press the FLOAT/LOCK key to lock the clamp in position. (The actual specimen length can be measured by the DMA by using the MEASURE key after the specimen is mounted.)

9. Loosen the knurled vise collar on the fixed clamp.

10. Feed a loop of monofilament into the *top* of fixed clamp and through the pin vise inside the vise collar.



11. Thread the fiber bundle through the loop.

12. Carefully pull the loop back through the fixed pin vise and vise collar. Be sure to keep the fibers within the center of the pin vise and collar. It is best to minimize twisting the fiber bundles.

13. Pull up on the sample gently, to equally tension all fibers.

14. Tighten the knurled vise collar on the fixed clamp. Trim off any excess sample.

15. Adjust the thermocouple so that it is approximately halfway between the two clamps and close to, but not touching, the sample.

16. Place thermal shield over the clamps, if you are using a heating rate of 2°C/min or higher. NOTE: Do not use the thermal shield when using heating rates less than 2°C/min or when performing a step and hold experiment.

Running an Experiment

When you use the fiber tension clamp for experiments, you will need to set up the experimental parameters using the *Thermal Solutions* software.

Follow these general guidelines when using the fiber clamp:

1. Set up the experimental parameters as directed in Chapter 4. Note that these clamps are tensioning clamps; therefore, autostrain (force track) and static force (preload force) values must be selected.

   The recommended values are 0.005 to 1 N for static force (preload force) and 125 to 200 percent for autostrain (force track), if appropriate.

NOTE: For crimped fibers, higher autotensions (200 to 250 percent) may be needed to uncrimp the fibers.

2. Press the MEASURE key on the DMA. This will apply the static force (preload force), measure the sample length, enter the length into the experimental parameters, and then start the motor in the desired operation mode.

3. View the signals. Pay special attention to these signals:

   Amplitude: This signal should achieve and maintain the value programmed. If running a multistrain experiment, the amplitude will cycle through the values programmed.

   Stiffness: The stiffness should be within the instrument's measurable range of 100 N/m to 10,000,000 N/m.

   Drive Force: The drive force should be between 0.0001 and 18 N. If the drive force is not within this range, either increase the programmed amplitude to increase the drive force, or increase the stiffness of the sample by changing the physical dimensions of the sample. Since the length is fixed in the fiber clamp, the width and thickness will need to be increased in order to increase the sample stiffness.

   Static Force (Preload Force): If autostrain (force track) is used, the static force (preload force) will automatically be adjusted to remain a set percentage greater than the force required to drive the sample at the programmed amplitude. If autostrain (force track) is not used, the static force (preload force) should read the value set in the instrument parameters. For more information regarding static (preload) force, refer to page 5-8.

If the initial conditions are acceptable, (*i.e.*, smooth oscillation and good modulus values), start the experiment.

If the values are not acceptable, then change conditions appropriately.

Removing Samples

To remove the fiber samples from the fiber tension clamp after an experiment, take off the knurled vise collars and then remove the sample from clamps. If necessary, scrape sample out of pin vise in the center of the vise collar.

Removing the Clamp

To remove the fiber tension clamp, follow these steps:

1. Loosen, but do not remove, the four hex screws that hold the fixed clamp in place on the mounting posts.

2. Lift off the fixed clamp.

3. Remove the knurled vise collar and pin vise inside from the moveable clamp.

4. Use the hex key to loosen the setscrew in the center of the moveable clamp.

5. Slide the dovetail of the moveable clamp out of the dovetail holder of the drive shaft.

Submersion Film/Fiber Clamp

This clamp is used to evaluate thin films or fibers while they are submerged in an ambient temperature fluid.

♦ ***CAUTION:*** **The Submersion Film/Fiber Tension clamp is designed for room temperature operation only. The clamp will not physically fit in the oven of the DMA 2980. When you use this clamp, you must select the "Tension Film Submersion" clamp when setting the mode in the *Thermal Solutions* software. This selection will not allow the oven to close when conducting tests and will avoid damaging the instrument. ANY ATTEMPT TO CLOSE THE OVEN OVER THESE CLAMPS MAY RESULT IN SERIOUS DAMAGE TO THE INSTRUMENT and MAY VOID ANY WARRANTY ON THE INSTRUMENT.**

The submersion film/fiber tension clamp can be used for testing either film or fiber samples immersed in a fluid at room temperature. This section shows the use of the clamp with film samples, but fiber samples can also be tested with this clamp. You can follow the same procedures when using fiber samples. Samples that are in the stiffness range of 100 N/m to 10,000,000 N/m can be tested.

Sample Size

Length:	15 mm fixed length
Width:	up to 6.5 mm
Thickness:	up to 2 mm

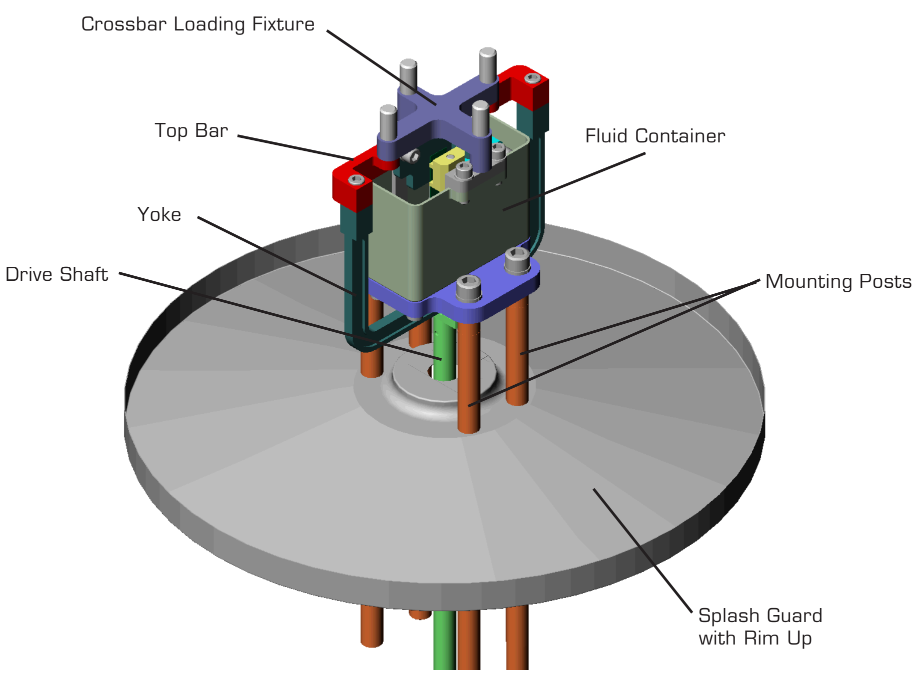


Figure 5.22
Submersion Film/Fiber Tension
Clamp (with Sample Loading Fixture in Place)

To learn how to install and calibrate the submersion film tension clamp, mount the samples, and remove the clamp, read the next several pages.

Installing and Calibrating the Clamp

NOTE: The submersion film/fiber tension clamp requires the clamp mass calibration to be performed prior to complete installation of the clamp. The following instructions will outline the installation, calibration, and sample loading of the submersion film/fiber fixture.

To install and calibrate the submersion film/fiber tension clamp on the DMA instrument, follow these steps:

1. Select the "Tension Film Submersion" clamp type and the desired mode using the Instrument Control program. (See the online help or the *User Reference Guide* for instructions.)

2. Remove any existing clamp assembly as instructed in this manual.

3. Press the CLAMP ▲ key to raise the driveshaft.

4. Press the FLOAT/LOCK key to lock the driveshaft in the upper position.

5. Loosen the screws on the thermocouple-mounting brackets, shown in the figure on the next page. Then lower the brackets to their lowest possible position.

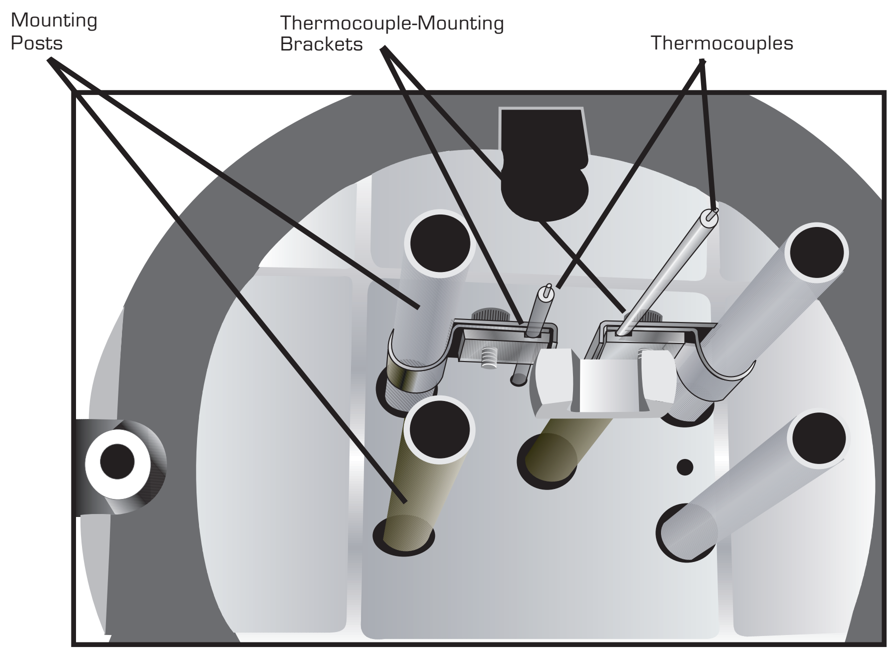


Figure 5.23
View from the Top

6. Straighten the thermocouples, then push them down to their lowest position.

7. Orient the splash guard so that the rim is facing up (see Figure 5.22). Install the splash guard over the four mounting posts, the drive shaft, and the thermocouples by inserting these components through the pre-cut holes. Lower the splash guard down until it rests on the thermocouple mounting brackets. The splash guard helps keep fluid from getting down into the air bearing assembly. **Even with the splash guard installed, be careful not to splash fluid out of the clamp assembly. By necessity, there is still a small gap around the driveshaft that even the splash guard can not protect.**

8. Move the drive shaft all the way down using the CLAMP ▼ key on the instrument.

9. Using the hex wrench, loosen the setscrew and slide the dovetail of the yoke into the dovetail holder on the drive shaft. Adjust the thermocouple position so that it does not touch the yoke. Tighten the setscrew. (See the figure below.)

10. Install the top bar onto the yoke and tighten the two hex screws. (See the figure below.)

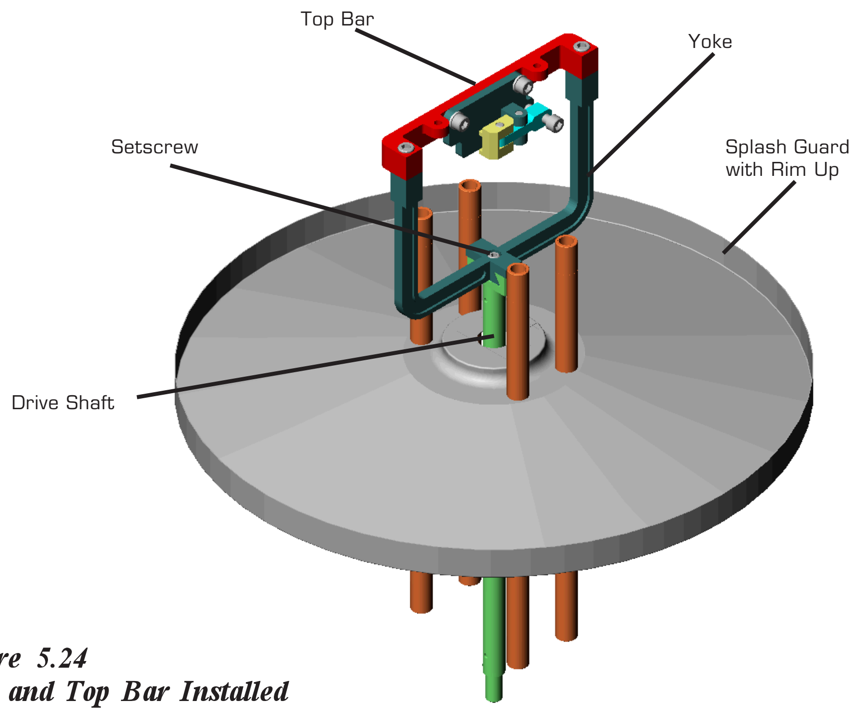


Figure 5.24
Yoke and Top Bar Installed on Drive Shaft

11. Calibrate the clamp as instructed in the Instrument Control program. (See the online help or the *User Reference Guide* for instructions.) Clamp mass calibration will take 5 minutes to complete.

    When clamp calibration step 1 (clamp mass) has been completed successfully, you will need to install remainder of the clamp before completing the clamp calibration. Follow steps 12 to 19 to install the remainder of the clamp.

12. Press the FLOAT/LOCK key to lock the driveshaft. Remove the top bar from the yoke.

13. Place the fluid container on the four mounting posts, aligning the screw holes. Seat the fluid container firmly onto all four of the mounting posts and tighten the screws with a hex wrench. Do not over tighten the attachment screws. (See the figure below.)

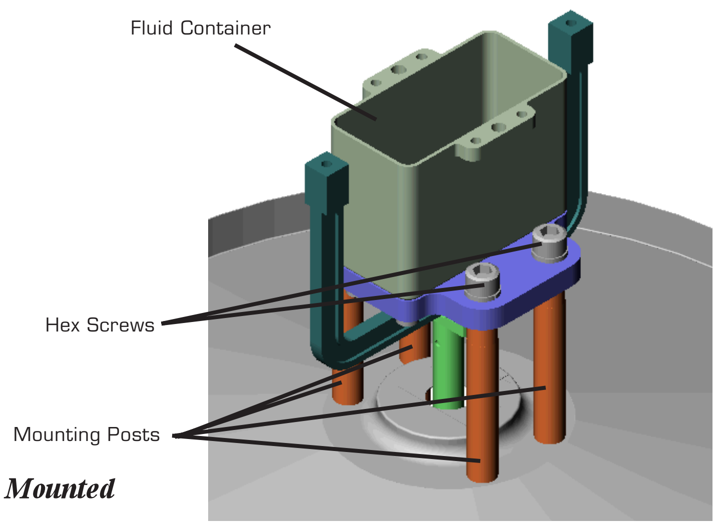


Figure 5.25
Fluid Container Mounted

14. Put together the *Sample-Loading Assembly* as follows (see the figure below):

    a. Attach the crossbar loading fixture onto the top bar using two thumbscrews. The long side of the crossbar should be positioned parallel with the top bar.

    b. Attach the remaining two crossbar loading fixture thumbscrews to the lower U-Clamp.

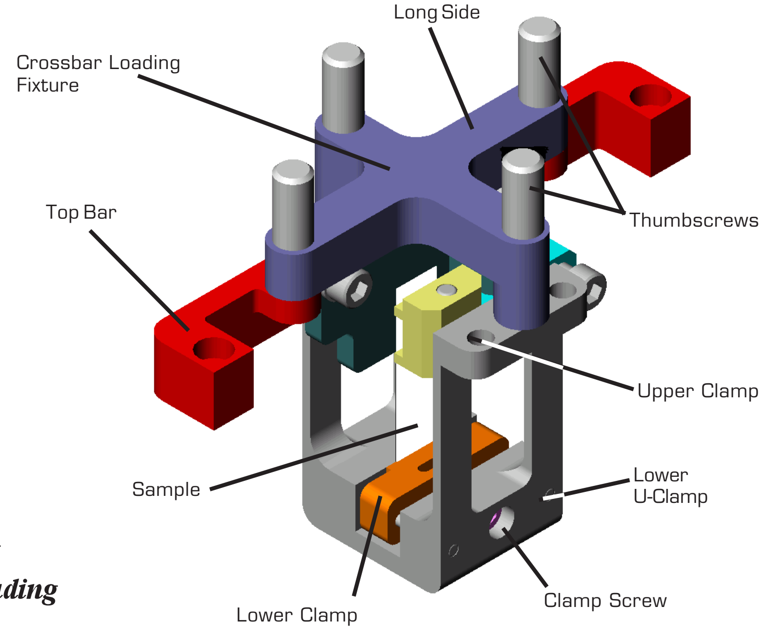


Figure 5.26 Sample-Loading Assembly

15. Measure the width and thickness dimensions of the steel compliance calibration sample. Make note of the measurements.

16. Load the steel compliance calibration sample into the upper and lower clamps of the Sample-Loading Assembly. Using the torque wrench, tighten the top and bottom sample clamp screws to the appropriate clamping torque [1 to 2 in-lbs.].

17. Press the FLOAT/LOCK key to float the driveshaft.

18. Insert the Sample-Loading Assembly into the cup of the fluid container. Fix the Sample-Loading Assembly in place by first screwing the two top bar screws to the yoke and subsequently screwing the four lower U-clamp screws to the fluid container tub of the fixed clamp. See the figure below.

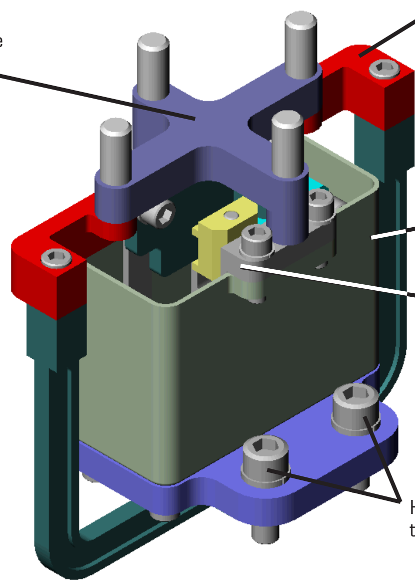


Figure 5.27 Sample Loading Assembly in Place

19. Unscrew the four thumbscrews to remove the crossbar loading fixture from the lower U-Clamp and top bar. This is the way the clamp will be assembled while the experiment is run. See the figure on the next page.

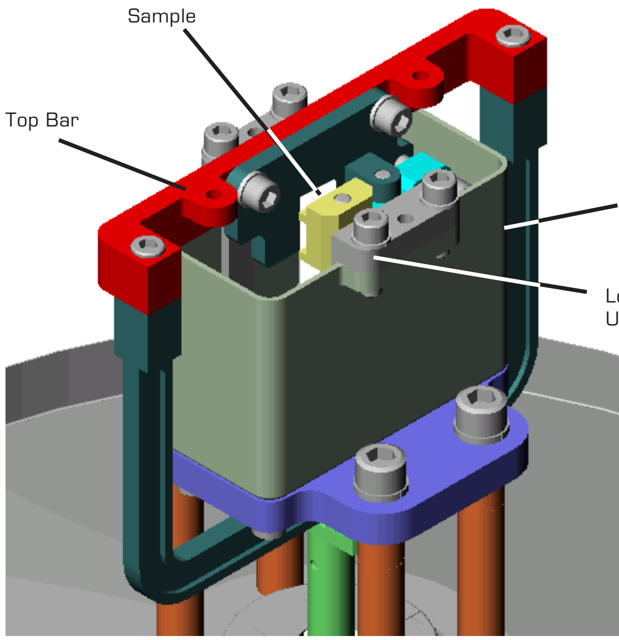


Figure 5.28
Sample in Place on
Submersion Film Clamp

20. Use the Instrument Control software to complete the DMA clamp calibration procedure (left off at step 9) by entering the dimensions of the steel compliance sample on the clamp compliance window. The sample length is fixed at 15 mm.

21. Remove the steel compliance calibration sample as follows:

    a. Press the FLOAT/LOCK key to lock the driveshaft.

    b. Install the crossbar loading fixture onto the upper top bar and lower U-Clamp.

c. Loosen the lower U-clamp screws from the tub of the lower fixed clamp and the top bar screws from the yoke.

d. Remove the Sample-Loading Assembly.

22. Remove the steel compliance calibration sample from the upper and lower clamps.

Once the clamp calibration procedure is complete a test sample can be mounted. See "Mounting a Sample" later in this section.

Operating Range of the Submersion Tension Film/Fiber Clamp

The geometry factor, GF, in the equation below can help you determine the optimum size for the sample material or determine if a sample of a particular size can be measured.

$$GF = L/A$$

where:

L = sample length, which is fixed to 15 mm, so...

$$GF \quad = \quad 15/A$$

for the Submersion Film Tension Clamp

The sample modulus is calculated by multiplying the measured sample stiffness, Ks, by the geometry factor, GF.

$$\text{Modulus} = \text{Ks} \times \text{GF, or}$$
$$\text{Modulus} = \text{Ks} \times (15/\text{A})$$

where the measurable stiffness range of the instrument is a fixed quantity between 100 to 10,000,000 N/m.

Note we can solve the modulus equation above for stiffness as follows

$$\text{Ks} = \text{Modulus/GF, or}$$
$$\text{Ks} = (\text{Modulus} \times \text{A})/15$$

Since the modulus of the material is an intrinsic property—*i.e.,* independent of the sample dimensions—it can be seen that the sample dimensions must be selected to yield stiffness values within the allowable measurable range of the instrument.

Mounting a Sample

After the submersion film/fiber tension clamp has been installed and calibrated, you can mount the sample using the following steps:

NOTE: The mounting of film samples is critical to achieving accurate and reproducible results.

1. Measure and note the width and thickness dimensions of test sample (or appropriate dimensions if other geometries are used) and install into upper and lower clamps.

2. Load sample into the upper and lower clamps of the Sample-Loading Assembly as shown in the figure below.

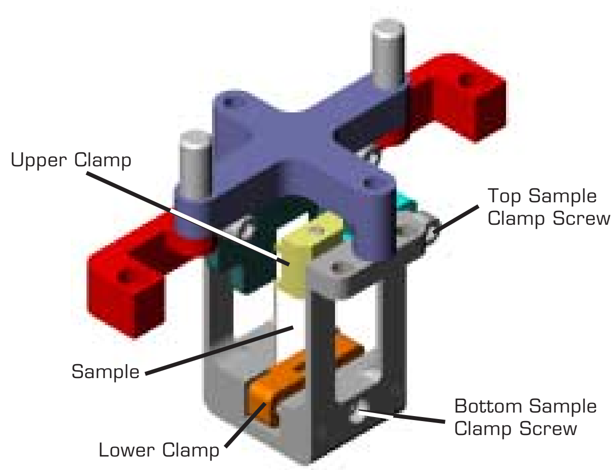


Figure 5.29
Loading the Sample

3. Using the torque wrench, tighten the top and bottom sample clamp screws to the appropriate clamping torque [1 to 2 in-lbs. (10 to 20 cm-N) for a rigid sample].

4. Press the FLOAT/LOCK key on the instrument keypad to release (float) the clamp.

5. Insert the Sample-Loading Assembly into the cup of the fluid container.

6. Fix the Sample-Loading Assembly in place by first screwing the four lower U-clamp screws to the fluid container tub of the fixed clamp and subsequently screwing the two top bar screws to the yoke. See Figure 5.28 on page 5-87.

7. Press the FLOAT/LOCK key on the instrument keypad to lock the clamp into position.

8. Unscrew the four thumbscrews to remove the crossbar loading fixture from the lower U-Clamp and top bar. This is the way the clamp will be assembled while the experiment is run. See the Figure 5.29 on page 5-90.

   The sample is now loaded and ready for testing. The sample length will be set to 15 mm after loading.

9. Carefully fill the fluid container with the fluid of interest. DO NOT OVERFILL CUP. It is recommended that the fluid be loaded into cup using a squeeze bottle, small funnel, pipette, or other device that will direct flow of fluid directly into cup thereby minimizing spilling or splashing of fluid.

Running an Experiment

When you use the submersion film tension clamp for experiments, you will need to set up the experimental parameters using the Instrument Control software.

Follow these general guidelines when using the submersion film clamp:

1. Use the Instrument Control program to select the clamp type, mode, and experimental parameters. (See the online help or the *User Reference Guide* for instructions.)

2. Set up the instrument parameters as directed in Chapter 4. Note that the amplitude range of the DMA 2980 is 0.5 to 10,000 microns. The upper amplitude range is limited to 1,000 microns when using the submersion film clamp. This amplitude range is a physical limit that is limited by distance between the yoke and the top of the fluid container.

   This clamp is a tensioning clamp; therefore, static force (preload force) and autostrain (force track) values must be selected.

   The recommended values for static force (preload force) values are 0.005 to 1 N. The recommended values for autostrain (force track) are 120 to 150% (when appropriate).

3. Press the MEASURE key on the DMA. This will apply the static force (preload force) and then start the motor in the desired operation mode.

4. View the signals. When running a dynamic experiment (multifrequency and multistrain mode) pay special attention to these signals:

   Amplitude: This signal should achieve and maintain the value programmed. If running a multistrain experiment, the amplitude will cycle through the values programmed.

   Stiffness: The stiffness should be within the instrument's measurable range of 100 N/m to 10,000,000 N/m. If it is not, refer to section titled "Operating Range of Submersion Tension Film Clamp" to make appropriate adjustments.

   Drive Force: The drive force should be between 0.0001 and 18 N. If the drive force is not within this range, either increase the programmed amplitude to increase the drive force, or increase the stiffness of the sample by changing the physical dimensions of the sample. Since the length is fixed in the submersion film clamp, the width and thickness will need to be increased in order to increase the sample stiffness.

   Static Force (Preload Force): If autostrain (force track) is used, the static force (preload force) will automatically be adjusted to remain a set percentage greater than the force required to drive the sample at the programmed amplitude. If autostrain (force track) is not used, the static force (preload force) should read the value set in the instrument parameters. For more information regarding static (preload) force, refer to page 5-8.

If the initial conditions are acceptable, (*i.e.*, smooth oscillation and good modulus values), start the experiment.

If the values are not acceptable, then change conditions appropriately.

Removing a Sample and Clamp

After you have finished your experiment, you can remove the sample from the submersion film clamp as follows:

1. Attach the Sample-Loading Assembly as follows:

   a. Press the FLOAT/LOCK key on the instrument keypad to release (float) the clamp.

   b. Attach the crossbar loading fixture onto the top bar using two thumbscrews (see Figure 5.28 on page 5-87).

   c. Attach the remaining two crossbar loading fixture thumbscrews to the lower U-Clamp.

   d. Press the FLOAT/LOCK key on the instrument keypad to lock the drive-shaft.

2. Loosen the two top bar screws and the four U-clamp screws. Carefully remove the Sample-Loading Assembly, letting excess fluid drip into the cup.

3. Wipe excess fluid from Sample-Loading Assembly and load a new sample, if desired.

   If another sample is not to be tested, pipette the fluid from the fluid container to avoid spilling fluid on the instrument. After removing fluid from the cup, loosen the four mounting post screws and remove the lower clamp. Then, loosen the dovetail screw and remove the yoke.

Submersion Compression Clamp

Compression is used to measure the properties of low to medium modulus materials, including gels and weak elastomers. These clamps are used to to test low to medium modulus samples while they are submerged in an ambient temperature fluid. The sample must support a preload force, so the samples should not flow. The sample should have as high a thickness-to-diameter ratio as is possible, depending on the sample preparation and instrument limits. Samples that are in the stiffness range of 100 N/m to 10,000,000 N/m can be tested.

♦ ***CAUTION:***

The Submersion Compression clamps are designed for room temperature operation only. The clamps will not physically fit in the oven of the DMA 2980. When you use these clamps, you must select the Submersion Compression clamp when setting the mode in the *Thermal Solutions* software. This selection will not allow the oven to close when conducting tests and will avoid damaging the instrument. ANY ATTEMPT TO CLOSE THE OVEN OVER THESE CLAMPS MAY RESULT IN SERIOUS DAMAGE TO THE INSTRUMENT and MAY VOID ANY WARRANTY ON THE INSTRUMENT.

Sample Size

Diameter: up to 15 mm and 30 mm
Thickness: up to 5 mm

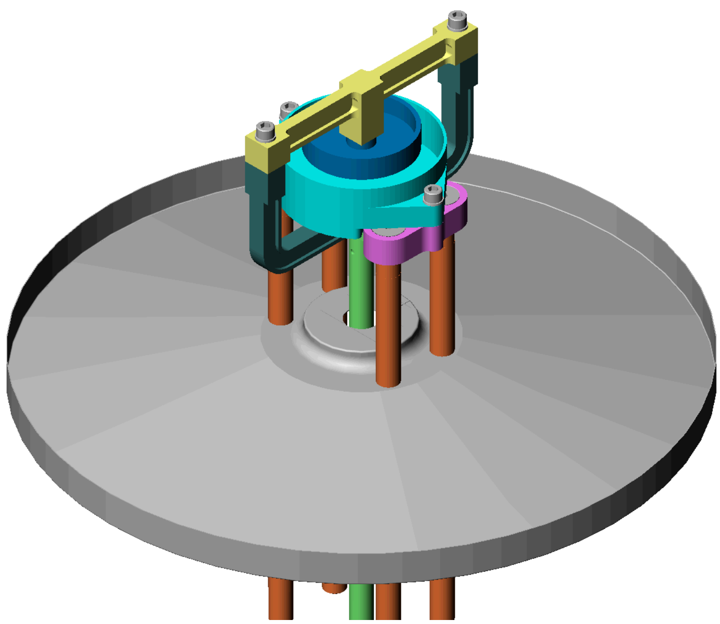


Figure 5.30
Submersion Compression Clamp

To learn how to install the submersion compression clamp, mount the samples, and remove the clamp, read the next several pages.

Installing the Clamp

To install the submersion compression clamp on the DMA instrument, follow these steps:

1. Remove any existing clamp assembly as instructed in this manual.

2. Select the clamp type and mode using the Instrument Control program. (See the online help or the *User Reference Guide* for instructions.)

3. Press the CLAMP ▲ key to raise the driveshaft.

4. Press the FLOAT/LOCK key to lock the driveshaft in the upper position.

5. Loosen the screws on the thermocouple-mounting brackets, shown in the figure on the next page. Then lower the brackets to their lowest possible position.

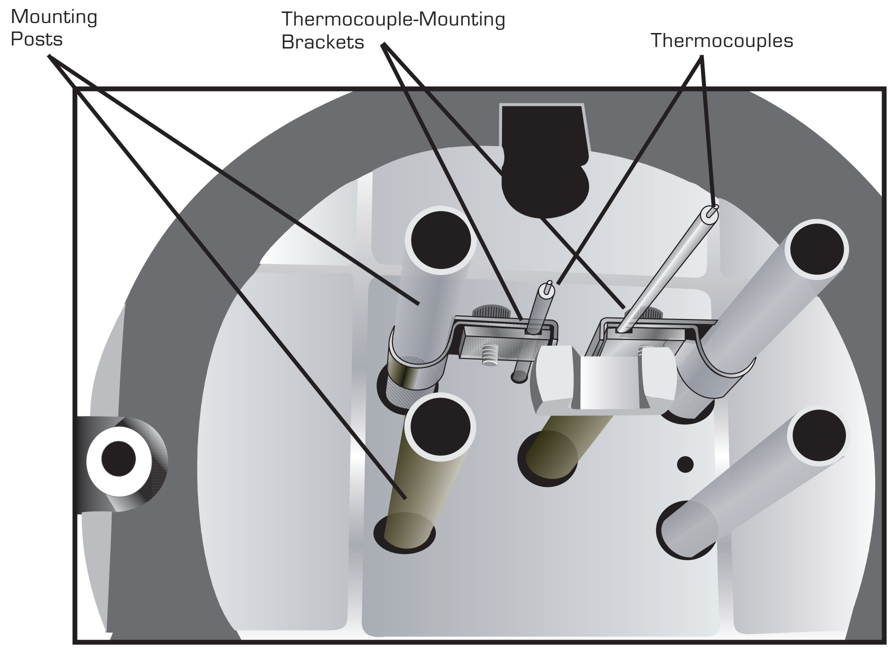


Figure 5.31
View from the Top

6. Straighten the thermocouples, then push them down to their lowest position.

7. Orient the splash guard so that the rim is facing up. Install the splash guard over the four mounting posts, the drive shaft, and the thermocouples by inserting these components through the pre-cut holes. Lower the splash guard down until it rests on the thermocouple mounting brackets. The splash guard helps keep fluid from getting down into the air bearing assembly. **Even with the splash guard installed, be careful not to splash fluid out of the clamp assembly. By necessity, there is still a small gap around the driveshaft that even the splash guard can not protect.**

8. Move the drive shaft all the way down using the CLAMP ▼ key on the instrument. If the clamp does not move down, increase the static force (preload force) to 1 to 2 N using the Instrument Control software.

9. Using the hex wrench, loosen the setscrew and slide the dovetail of the yoke into the dovetail holder on the drive shaft. Adjust the thermocouple position so that it does not touch the yoke. Tighten the setscrew. (See the figure below.)

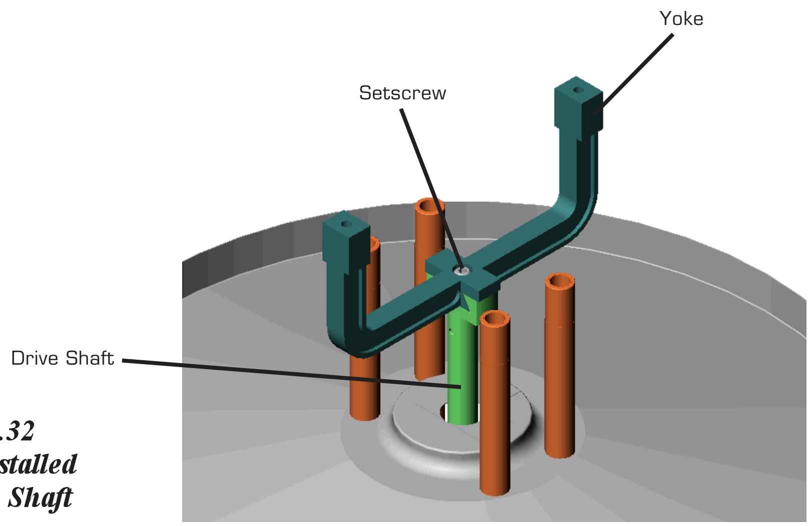


Figure 5.32 Yoke Installed on Drive Shaft

10. Mount the fixed clamp, which consists of two stages and a fluid cup, as follows:

    a. Seat one of the two stages firmly onto two of the mounting posts and tighten the captive screws with a hex wrench. Repeat this for the second stage as shown in the figure on the next page. Do not over tighten the screws.

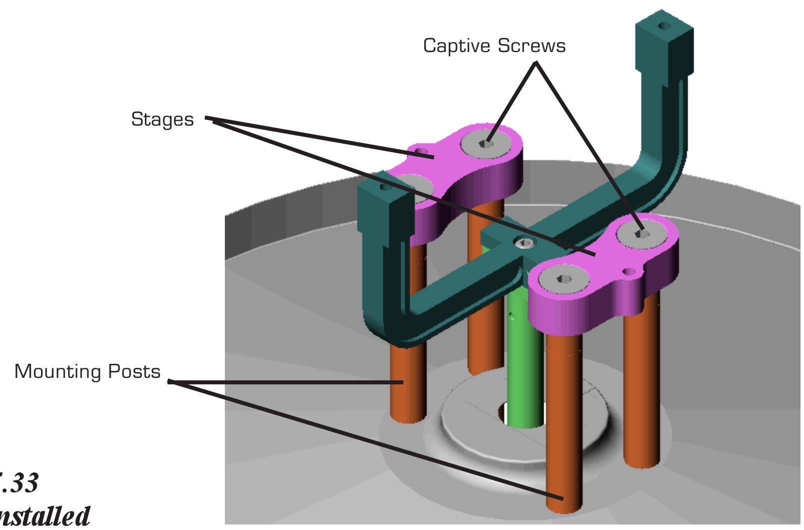


Figure 5.33
Stages Installed

b. Mount the fluid cup onto the stages using the two hex screws and washers found in the plastic bag. The fixed clamp is now fully installed as shown in the figure below. Position the thermocouple so that it is near, but not touching, the fluid cup.

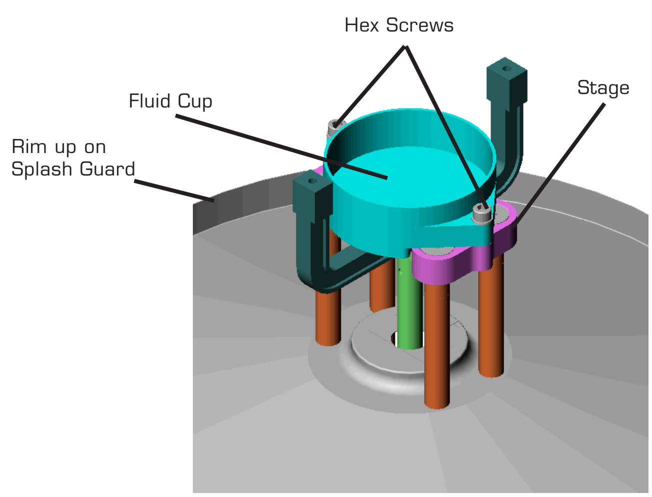


Figure 5.34
Fixed Clamp
Fully Installed

11. Screw the 15 mm or 30 mm compression disk into the top bar.

12. Press the FLOAT/LOCK key to release (float) the clamp.

13. Press the CLAMP ▲ key to raise the driveshaft to the top position.

14. Press the FLOAT/LOCK key to lock the driveshaft in the upper position.

15. Attach the top bar and compression disk onto the yoke using the two hex screws and washers. The yoke, top bar, and compression disk make up the moveable part of the clamp. The clamp is now fully assembled and ready for calibration as seen in the figure below.

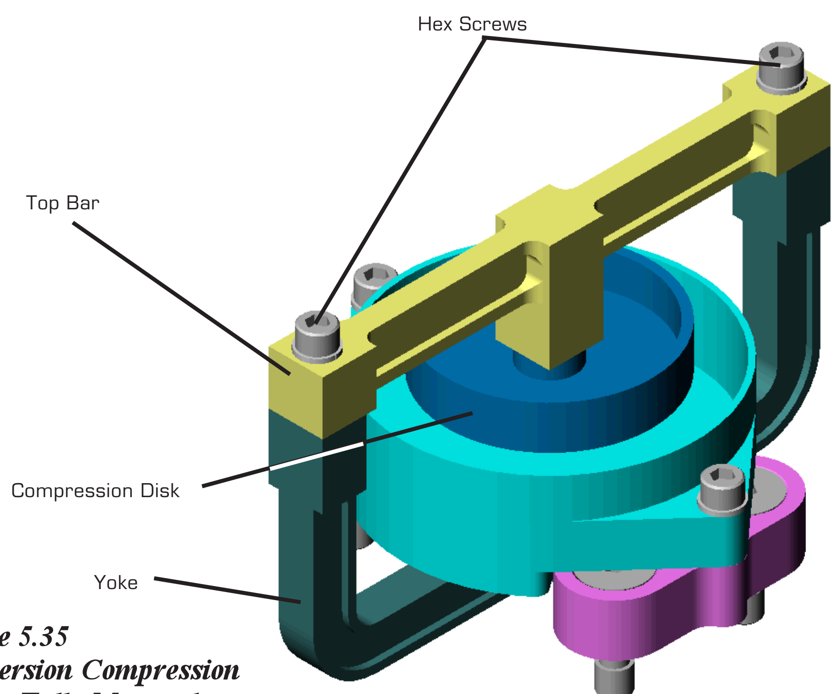


Figure 5.35
Submersion Compression Clamp Fully Mounted

16. Calibrate the clamp as instructed in the Instrument Control program, following the instructions displayed. (See the online help or the *User Reference Guide* for instructions.)

After the clamp has been calibrated, you can mount a sample as directed on page 5-105.

Operating Range of the Submersion Compression Clamp

The geometry factor, GF, in the equation below can help you determine the optimum sample size for the sample material or determine if a sample of a particular size can be measured.

$$GF = Fe(T/A)$$

where:

T = Sample Thickness (height) in mm
A = Sample Cross Sectional Area in mm^2
Fe = Sample Shear Factor.

The sample modulus is calculated by multiplying the measured sample stiffness, Ks, by the geometry factor, GF.

$$\text{Modulus} = Ks \times GF \text{ or}$$
$$\text{Modulus} = Ks \times Fe(L/A)$$

where the measurable stiffness range of the instrument is a fixed quantity between 100 to 10,000,000 N/m.

Note we can solve the modulus equation above for stiffness as follows

$$Ks = Modulus/GF \text{ or}$$
$$Ks = (Modulus \times A)/Fe \times L$$

Since the modulus of the material is an intrinsic property—*i.e.*, independent of the sample dimensions—it can be seen that the sample dimensions must be selected to yield stiffness values within the allowable measurable range of the instrument.

NOTE: Refer to the "Clamping Factors" section in Chapter 6 to determine the value used for the sample shear factor, Fe.

Mounting a Sample

After the submersion compression clamp has been installed and calibrated on the DMA, you can mount the sample using the following steps:

1. Press the FLOAT/LOCK key to release (float) the clamp.

2. Manually raise the moveable clamp and place the sample in the bottom cup, making sure that it does not extend beyond the edges of the disk.

3. Enter the static force (preload force) using the Instrument Control program. (See the *User Reference Guide* or online documentation for details.) The recommended values for static force (preload force) values are 0.1 to 1 N.

4. Move the moveable clamp all the way down using the CLAMP ▼ key on the instrument.

5. Carefully fill the fluid cup so that the fluid covers the sample, but does not go over the edge of the compression disk. It is recommended that the fluid be loaded into the cup using a squeeze bottle, small funnel, pipette, or other device that will direct the flow of the fluid directly into the cup, thereby mimimizing the spilling or splashing of the fluid.

You are now ready to run an experiment as directed in the next section.

Running an Experiment

When you use the submersion compression clamp for experiments, you will need to set up the experimental parameters using the Instrument Control software.

Follow these general guidelines when using the submersion compression clamp:

1. Use the Instrument Control program to slect the clamp type, mode, and experimental parameters. (See the online help or the *User Reference Guide* for instructions.)

2. Set up the instrument parameters as directed in Chapter 4. Note that the amplitude range of the DMA 2980 is 0.5 to 10,000 microns. The upper amplitude range is limited to 100 microns when using a maximum sample thickness of 5 mm with the submersion compression clamp. This amplitude range is a physical limit that is limited by distance between the bottom of the top plate and the top edge of the cup. However, because of the possibility of splashing during oscillations when making measurements in the submersion compression clamp, amplitudes should not exceed 100 microns regardless of the sample thickness.

   This clamp is a tensioning clamps; therefore, static force (preload force) and autostrain (force track) values must be selected.

   The recommended values for static force (preload force) values are 0.1 to 1 N. (The static force (preload force) may be the same value that you entered while mounting the sample, if desired.) The recommended values for autostrain (force track) are 120 to 150%.

3. Press the CLAMP ▼ key on the instrument keypad to move the clamp faces together and compress the sample using the static force (preload force) programmed in step 1.

4. Press the MEASURE key on the DMA. This will apply the static force (preload force), measure the sample thickness and automatically enter the thickness into the experimental parameters. Then the motor will start in the programmed operation mode.

5. View the signals. When running a dynamic experiment (multifrequency and multistrain mode) pay special attention to the following signals:

   Amplitude: This signal should achieve and maintain the value programmed. If you are running a multistrain experiment, the amplitude will cycle through the values programmed.

   Stiffness: The stiffness should be within the instrument's measurable range of 100 N/m to 10,000,000 N/m. If it is not, refer to section called the "Operating Range of Submersion Compression Clamp" to make the appropriate adjustments.

   Drive Force: The drive force should between 0.0001 N and 18 N. If the drive force is not within this range, either increase the programmed amplitude to increase the drive force, or increase the stiffness of the sample by changing the physical dimensions of the sample. To increase the sample stiffness, increase the diameter and decrease the thickness of the sample.

Static Force (Preload Force): If autostrain (force track) is used, the static force (preload force) will automatically be adjusted to remain a set percentage greater than the force required to drive the sample at the programmed amplitude. If autostrain (force track) is not used, the static force (preload force) should read the value set in the instrument parameters. For more information regarding static (preload) force, refer to page 5-8.

If the initial conditions are acceptable (*i.e.*, smooth oscillation and good modulus values), start the experiment.

If the values are not acceptable, then change the conditions appropriately.

NOTE: Pay special attention to the sample during the measurement. If all conditions are acceptable, as observed on the signal display, splashing may still be occurring with the selected parameters. If splashing is occurring, then change the conditions should be changed until you have eliminated the problem. This may require changing the amplitude, the frequency, or both.

Removing a Sample and Clamp

After you have finished your experiment, you can remove the sample from the submersion compression clamp as follows:

1. Press the FLOAT/LOCK key to lock the driveshaft.

2. Use a hex wrench to remove the screws from the top bar.

3. Carefully remove the top bar with the compression disk still connected and wipe excess fluid from the compression disk. (It is important to wipe the fluid from the compression disk so that it will not interfere with subsequent tests.)

4. Using a pipette or syringe to avoid spilling fluid on the instrument, remove the remaining fluid from fluid cup and remove the sample.

5. Load a new sample, if desired.

   If another sample is not to be tested, continue to remove the clamp using the remaining steps.

6. Loosen the two hex screws and remove the fluid cup from the stages.

7. Loosen the four mounting post screws and remove the two stages.

8. Loosen the dovetail screw and remove the yoke.

9. Clean and dry the fluid cup and compression disk before storing them.

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Introduction

This chapter provides information regarding the theory and applications of the DMA 2980 instrument and the Dynamic Mechanical Analysis technique.

The following are explained:

- theory of operation
- comparison to other techniques
- viscoelasticity
- modes of operation
- sample stiffness and modulus calculations
- calculations based on clamp type.

Theory of Operation

The Dynamic Mechanical Analyzer 2980 is a precision instrument designed to measure viscoelastic properties, such as modulus (stiffness) and damping (energy dissipation), of solid and soft solid materials. Samples of various shapes and sizes are fixed into position using a variety of stainless steel clamping arrangements. A mechanical deformation is applied to the sample via the instrument's drive motor. The deformation can be applied sinusoidally, in a constant (or step) fashion, or under a fixed rate. The combination of temperature and deformation profile are programmed using the *Thermal Solutions* Instrument Control software.

Comparison to Other Techniques

DMA and DEA (Dielectric Analysis) are two of the techniques that can bridge the disciplines of thermal analysis and rheology as seen in the figure below.

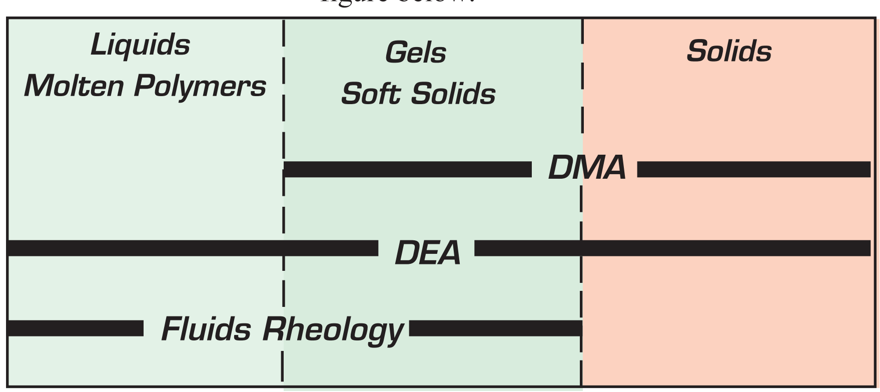

Figure 6.1
Correlation to Other Techniques

Defining Viscoelasticity

The concept of viscoelasticity comes from the fact that most materials do not exhibit purely elastic (ideal solids) or purely viscous (ideal liquids) behavior but a combination of both. By definition, *viscoelasticity* simply means having both viscous and elastic properties. This section presents a discussion of solids and liquids and their behavior as it applies to the DMA.

Solids

Materials are often referred to as solids or liquids, depending on whether or not they retain their shape under the force of gravity. An ideal solid is a material that is purely elastic. *Elasticity* is simply a materials ability to store deformational energy. A purely elastic material (an ideal solid) will change its shape when deformed and regain its original shape when the deformation is removed. All the energy (stress) applied to the material is stored by the material during the deformation.

An example of a material that demonstrates purely elastic deformation is a steel spring (see Figure 6.2 on the next page). When a stress is applied to an ideal solid, it deforms immediately to a constant value of strain and recovers immediately when the stress is removed. There is no time dependence in the behavior of the material. This ideal mechanical behavior is described by Hooke's law in which stress and strain are related through a proportional constant called the *modulus* (E or G):

Hooke's Law:

$\sigma \quad = \quad E\varepsilon$ (Tension, Compression or Bending)
$\tau \quad = \quad G\gamma$ (Shear)

Where:

σ and τ are stress terms
ε and γ are strain terms.

The modulus is a measure of the stiffness of a material. *Stiffness* is the ability of a material to resist deformation. A material is called Hookean, if the modulus does not change when the deformation is changed. This is also called the linear region.

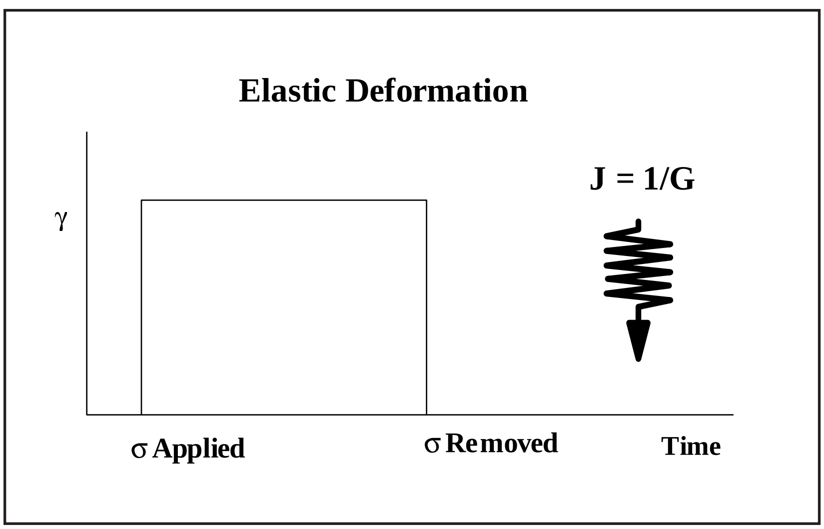

Figure 6.2
Elastic Deformation

Liquids

An ideal liquid, in contrast, is a material that has no elasticity. An ideal fluid stores no deformational energy. An ideal liquid is not rigid but conforms to the shape of its container and finds its own level under gravity. When an ideal fluid is deformed, it changes its shape and, when the deformation is removed, it does not regain its original shape but remains in its new deformed shape. An example of an ideal fluid (ideal viscous fluid) is water. Consider a glass of water sitting on your table. The water takes on the shape of glass. If you spill the glass of water, when you pick up the glass the water does not find its way back into the glass. This behavior is referred to as *viscous flow* and is described by Newton's Law which linearly relates the stress to the rate of shear:

Newton's Law:

$$\tau = \eta d\gamma/dt$$

The proportionality factor η is called the *coefficient of viscosity*. A material is referred to as Newtonian, if the viscosity is independent of the applied shear rate. A simple graphical representation of the behavior of an ideal fluid is shown in Figure 6.3. An ideal fluid will deform continuously under the application of a stress but will not recover when the stress is removed. The strain developed under the application of the stress is a function of time until the stress is removed.

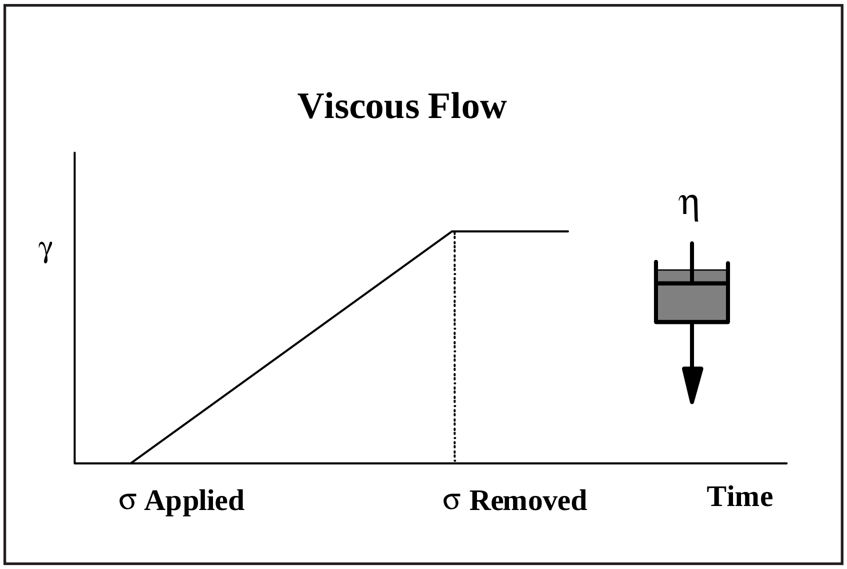

Figure 6.3
Viscous Flow

Viscoelastic Behavior

As stated earlier, the concept of viscoelasticity comes from the fact that most materials do not exhibit purely elastic (ideal solids) or purely viscous (ideal liquids) behavior but a combination of both. When as stress is applied to a viscoelastic material, it will show time-dependent deformation. Any viscoelastic material, given enough time, will flow under an applied stress. When the stress is removed the material will not fully recover. The portion of strain that is recovered represents the energy stored or the elastic portion of the material's response. The portion of the strain that is not recovered represents the energy dissipated or viscous portion of the material's response. Viscoelastic behavior of a material is shown graphically in Figure 6.4 below.

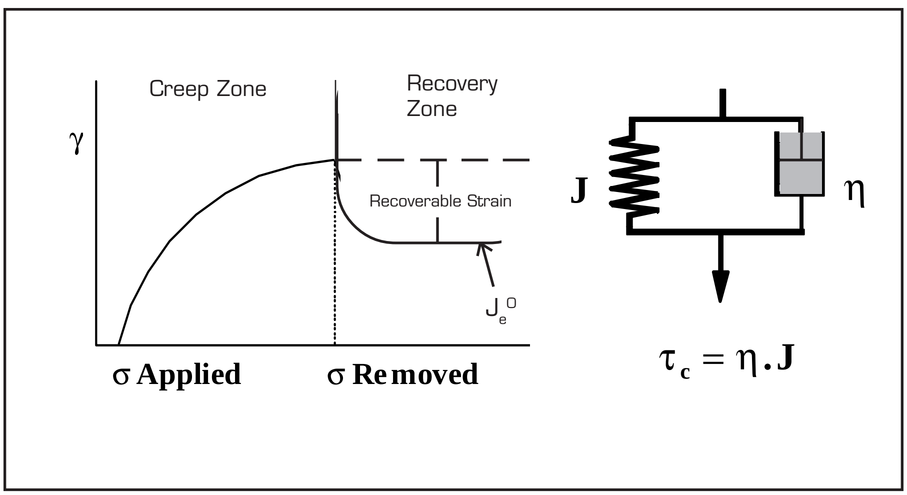

Figure 6.4
Viscoelastic Response
(The Voigt Model)

As an example of a viscoelastic response, consider holding a tennis ball in you hand with your arm straight out. If you drop the tennis ball on the ground it will not bounce back to the same height from which you dropped it. The height to which the ball bounced back represents the elastic response of the material and the remainder represents the viscous portion (shown in Figure 6.5). The more elastic the material. the more it will bounce back to the original height from which it was dropped.

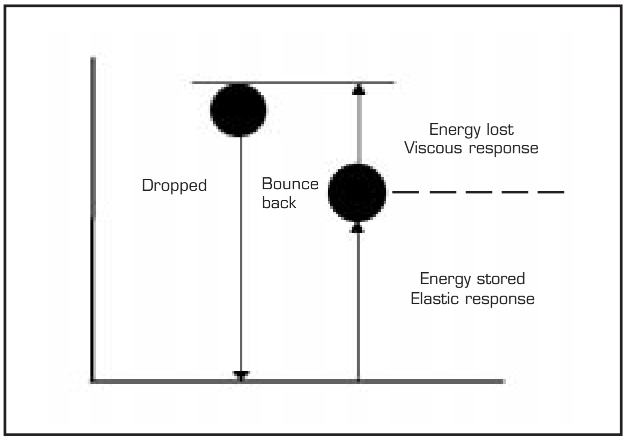

Figure 6.5
Viscoelastic Behavior

Modes of Operation

The viscoelastic properties of a material can be characterized on the DMA 2980 using three experimental testing modes. The next sections discuss these modes:

- Dynamic Mechanical Analysis (DMA) tests (also known as oscillatory measurements)
- Creep tests (also known as a transient test mode)
- Stress relaxation tests (also known as a transient test mode).

Dynamic Mechanical Analysis Testing

During dynamic testing, an oscillatory (sinusoidal) strain (or stress) is applied to the material and the resulting stress (or strain) developed in the material is measured. For an ideal solid material, which obeys Hooke's law, the resulting stress will be proportional to the amplitude of the applied strain. The stress and strain waves will be in phase or, put another way, the phase shift (phase angle δ) between the stress and strain is 0° (see Figure 6.6A on the next page). For an ideal fluid which obeys Newton's law, the stress will be proportional to the strain rate. The stress signal will lead the strain signal by 90° (see Figure 6.6B on the next page).

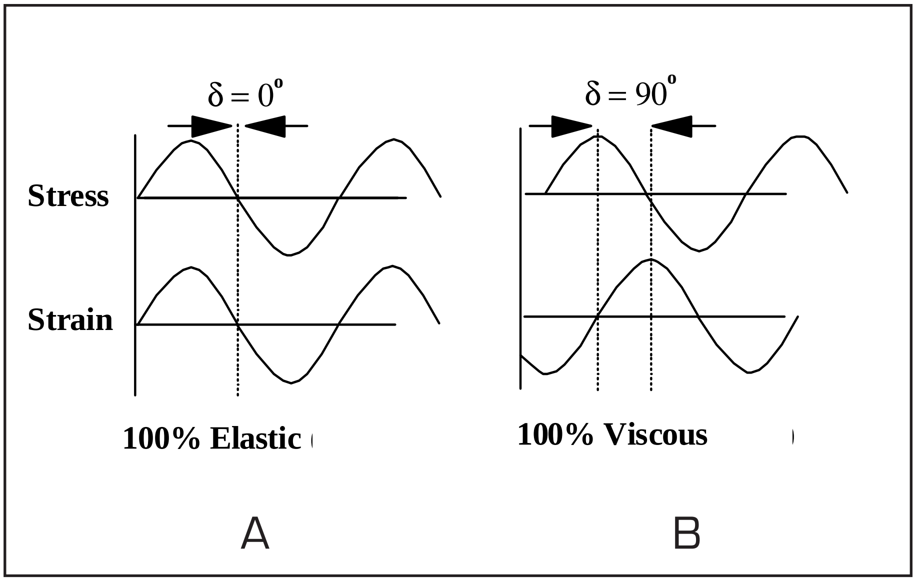

Figure 6.6
Ideal Solid and Fluid
DMA Stress and Strain

For a viscoelastic material, the phase angle will lie somewhere between 0° and 90° (Figure 6.7).

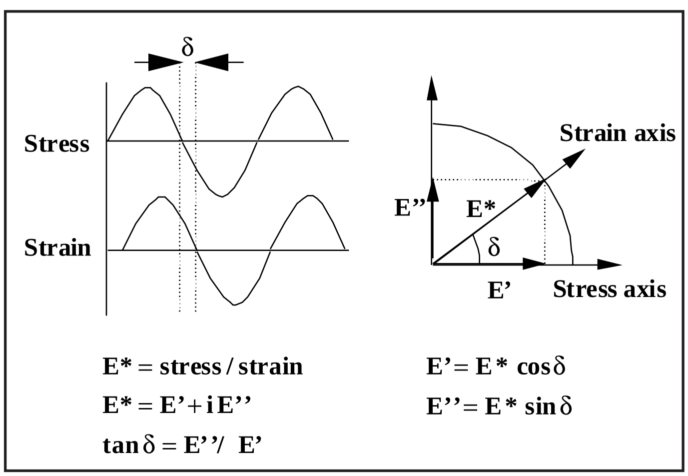

Figure 6.7
Viscoelastic Materials
DMA Stress and Strain

As the *modulus* is defined as the ratio of stress to strain (stress/strain), the resultant stress generated in a viscoelastic material—also referred to as the *complex stress* (σ* or τ*)—can be used to calculate the *complex modulus* E* or G*. The complex modulus is a measure of the materials resistance to deformation. It encompasses both elastic and viscous responses. The power of dynamic testing is that, by using the measured phase angle, the stress in turn can be deconvoluted into two parts:

- an elastic stress (σ' or τ'), that is in phase with the strain, and

- a viscous stress (σ" or τ"), that is in phase with the strain rate (90° out of phase with the strain).

The elastic modulus, or *storage modulus* (E' or G') and the viscous modulus, or *loss modulus* (E" or G"), can then be calculated directly from the elastic and viscous stress respectively. A summary of the calculations are as follows:

Complex Modulus:

$E^* = \sigma^*/\varepsilon$

or

$E^* = E' + iE''$

(Tension, Compression or Bending)

$G^* = \tau^*/\gamma$

or

$G^* = G' + iG''$

(Shear)

Storage Modulus:

$E' = \sigma'/\varepsilon$

or

$E' = E^* \cos \delta$

(Tension, Compression or Bending)

$G' = \tau'/\gamma$

or

$G' = G^* \cos \delta$

(Shear)

Loss Modulus:

$E'' = \sigma''/\varepsilon$

or

$E' = E^* \sin \delta$

(Tension, Compression or Bending)

$G'' = \tau''/\gamma$

or

$G'' = G^* \sin \delta$

(Shear)

Figure 6.7 on page 6-12 shows a vector depiction of these calculations. It can be seen from this figure that the tangent of the phase angle is the ratio of the loss modulus to storage modulus ($\tan \delta = E''/E'$ or $\tan \delta = G''/G'$). This ratio is a measure of the damping ability of a material.

Creep (or Step Stress) Testing
(Transient Experiment)

In a creep test, a constant stress is applied to the sample and the resulting strain is measured as a function of time.

- The creep compliance [J(t)] is calculated by dividing the time-dependent strain $\gamma(t)$ by the applied stress τ. Once the creep compliance has reached a steady state, the sample recovery can be measured by instantaneously removing the stress (stress = 0), and monitoring the recovered strain as a function of time.

- The recoverable compliance can be calculated by dividing the time-dependent recoverable strain $\gamma_r(t)$ by the stress applied in the creep zone. The recoverable compliance will eventually stabilize to a constant value. If the response is linear, the stabilized value of compliance is referred to as the equilibrium recoverable compliance, J_e^0.

Creep/recovery behavior is shown graphically in Figure 6.4 on page 6-9. Viscoelastic behavior in creep can be represented by the Voigt model. This model consists of a spring and a dashpot combined in parallel. Upon the application of stress, the dashpot acts to exponentially retard the growth of strain with time. When the stress is removed, the dashpot again exponentially retards the recovery of strain. The time constant (λ) is known as the retardation time and represents the characteristic time dependence of the system.

Stress Relaxation (or Step Strain) Testing
(Transient Experiment)

In a stress relaxation test, a strain is instantaneously applied to the sample, and the stress required to maintain that strain is measured as a function of time. The stress relaxation modulus [G(t) using shear sandwich clamps or E(t) using tension, compression, or bending clamps] is calculated as the time-dependent stress divided by the constant strain. The sample recovery can also be monitored with time upon release of the strain. Stress relaxation behavior is shown graphically in Figure 6.8 below.

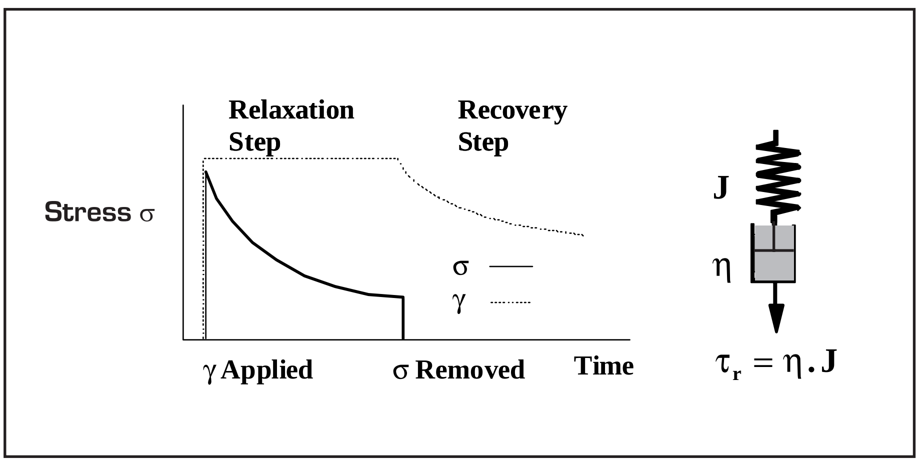

Figure 6.8
Viscoelastic Response of the Maxwell Model

Viscoelastic behavior in stress relaxation can be represented by the Maxwell model. This model consists of a spring and a dashpot combined in series. Since the two elements are in series, when the strain is applied, a step change in strain results in the spring because the dashpot cannot respond instantaneously to the step change. Once the spring has responded, the stress of the system is at a maximum, but the gradual response of the dashpot is to extend itself, allowing the spring (and the stress) to relax.

Sample Stiffness and Modulus Calculations

The fundamental measurement of the DMA 2980 is *sample stiffness* (K). Sample stiffness is defined as the force applied to the sample divided by the amplitude of deformation. The stiffness of a material is dependent on its geometry (physical dimensions). The modulus of a material however is independent of its geometry. As an example, consider a piece of aluminum foil. It is very easy to bend the aluminum foil. Now take a bar of aluminum one-inch thick. This piece of aluminum is not easily bent. Both the aluminum foil and bar are made of the same material but, simply changing the physical dimensions of the material changes the amount of force required to deform the material. If we were to measure the modulus of both the foil and bar, we would get the same number. A good understanding of sample stiffness is important for understanding geometry selection when conducting DMA measurements.

Dynamic Measurements

In dynamic experiments, the DMA 2980 measures the raw signals of force, amplitude of deformation, and phase angle. Instrument calibration constants are applied to the raw signals. Force and amplitude are used with the phase angle to calculate the storage and loss stiffness (K' and K"). Tan δ is calculated as the ratio of K” to K’. Storage and loss moduli are then calculated by multiplying the raw stiffness measurements by the appropriate geometry factors.

Transient Measurements

In transient measurements, the DMA 2980 measures the raw signals of force and amplitude of deformation. Stress and strain are derived from the raw force and amplitude, normalized for the geometry factor.

Calculations Based on Clamp Type

Since a material's modulus is independent of its geometry, equations relating the sample stiffness to the modulus depend on the type of clamps used, the sample shape, and the mode of deformation. This section contains stiffness calculations for the different clamp types along with comments on appropriate correction factors. Also included are stress and strain equations, which can be used as a general guideline for calculations made from the force and amplitude of deformation. The equations for stress and strain assume linear viscoelastic behavior. Modulus calculations based on the equations may not agree with values computed by the DMA 2980 program, especially if the sample stretches or shrinks significantly during an experiment.

The DMA 2980 determines the modulus (stiffness) of a material differently depending upon the clamp type installed on the instrument and selected using the *Thermal Solutions* Instrument Control software. This section provides the equations used for calculations, based on the type of clamp.

Dual Cantilever

When running experiments using the dual cantilever clamp, the equations found in this section are applied to obtain your results.

Modulus Equation

The stiffness model equation for a rectangular cross section sample, analyzed on the dual cantilever clamp, is as follows. (Similar calculations were performed for cylindrical samples but are not detailed here.)

$$K = \frac{24 \cdot E \cdot I}{L^3 \cdot \left[1 + \frac{12}{5} \cdot (1 + \nu) \cdot \left(\frac{t}{L} \right)^2 \right]}$$

Where:
K = stiffness or spring constant
E = elastic modulus
L = sample length (one side)
t = sample thickness
I = sample moment of inertia
ν = Poison's ratio

The sample moment of inertia is

$$I = \frac{w \cdot t^3}{12}$$

Where:
t = sample thickness
I = sample moment of inertia
w = sample width

The stiffness model equation assumes that the ends of the sample are fixed, or that there is no deformation of the sample beyond where the sample enters the clamps. This is never achieved in practice, to do so would require a discontinuity in the strains within the sample at the clamp face. To account for this error, a sample stiffness correction factor can be defined as:

$$F_c = \frac{Ks}{K}$$

Where:
K = stiffness or spring constant
Ks = measured stiffness
F_c = clamping correction factor

Substituting for K in the model equation and solving for the modulus:

$$E = \frac{K_s}{F_c} \cdot \frac{L^3}{24 \cdot I} \cdot \left[1 + \frac{12}{5} \cdot (1 + \nu) \cdot \left(\frac{t}{L} \right)^2 \right]$$

$$Fc = 0.7616 - 0.02713 \text{ x } \sqrt{\frac{L}{t}} + 0.1083 \ln \left(\frac{L}{t} \right)$$

Where:
E = elastic modulus
L = sample length (one side)
ln = natural log
K_s = measured stiffness
t = sample thickness
I = sample moment of inertia
ν = Poison's ratio
F_c = clamping correction factor

The clamping correction factor, F_c, was determined by finding the sample stiffness using *Finite Element Analysis* of the sample deformation and calculating F_c using the sample stiffness equation and the FEA stiffness. This was done by studying many cases including a variety of materials and geometries with corresponding fit applied to the result.

NOTE: Poisson's ratio accounts for the shear deformation taking place in flexure, when using samples of relatively small length-to-thickness ratios. It is introduced in the equation using the standard equation relating E and G:

$$E = 2 (1 + \nu) G$$

Stress and Strain

When using the dual cantilever clamp, stress and strain are not constant throughout the sample thickness. The maximum level of strain occurs at the sample surface, while the center experiences no strain at all. This also means that both stress and strain can have positive or negative sense, depending on whether it is on the top or bottom surface of the sample. The following equation expresses the maximum stress and strain levels and does not include any contribution from the clamp:

$$\sigma_x = \frac{3 \cdot P \cdot L}{w \cdot t^2}$$

$$\varepsilon_x = \frac{3 . \delta \cdot t \cdot F_c}{L^2 \cdot \left[1 + \frac{12}{5} \cdot (1 + \nu) \cdot \left(\frac{t}{L} \right)^2 \right]}$$

Where:

σ_x = stress

ε_x = strain

P = 1/2 applied force

δ = amplitude of deformation

L = sample length (one side)

t = sample thickness

w = sample width

F_c = clamping correction factor

ν = Poison's ratio

Single Cantilever

When running experiments using the single cantilever clamp, the equations found in this section are applied to obtain your results.

Modulus Equation

The stiffness model equation for a rectangular cross section sample, analyzed on the single cantilever clamp, is as follows. (Similar calculations were performed for cylindrical samples but are not detailed here.)

$$E = \frac{K_S}{F_c} \cdot \frac{L^3}{12 \cdot I} \cdot \left[1 + \frac{12}{5} \cdot (1 + \nu) \cdot \left(\frac{t}{L} \right)^2 \right]$$

$$Fc = 0.7616 - 0.02713 \text{ x } \sqrt{\frac{L}{t}} + 0.1083 1n \left(\frac{L}{t} \right)$$

Where:

E = elastic modulus
L = sample length
t = sample thickness
I = sample moment of inertia
ν = Poison's ratio
K_s = measured stiffness
F_c = clamping correction factor

Stress and Strain

When using the single cantilever clamp, the equations for stress and strain are the same as for the dual cantilever clamp (see page 6-24), except that P in the stress equation is the full applied force.

3-Point Bending

When running experiments using the 3-point bending clamp, the equations found in this section are applied to obtain your results.

Modulus Equation

The stiffness model equation for a rectangular cross section sample, analyzed on the 3-point bending clamp, is as follows. (Similar calculations were performed for cylindrical samples but are not detailed here.)

$$E = K_s \cdot \frac{L^3}{6 \cdot I} \cdot \left[1 + \frac{6}{10} \cdot (1 + \nu) \cdot \left(\frac{t}{L} \right)^2 \right]$$

Where:
E = elastic modulus
L = sample length (one side)
t = sample thickness
I = sample moment of inertia
ν = Poison's ratio
K_s = measured stiffness

This equation assumes that local deformation of the sample in the region of the supports are negligible.

Stress and Strain

When using the 3-point bending clamp, the maximum stress and strain occur at the midspan of the sample where the drive applies the load, so both stress and strain can have positive or negative sense depending on whether it is on the top or the bottom surface of the sample.

$$\sigma_x = \frac{P \cdot L \cdot t}{4 \cdot I} = \frac{3 \cdot P \cdot L}{w \cdot t^2}$$

$$\varepsilon_x = \frac{3 \cdot \delta \cdot t}{2 \cdot L^2 \cdot \left[1 + \frac{6}{10} \cdot (1 + \nu) \cdot \left(\frac{t}{L}\right)^2\right]}$$

Where:

σ_x = stress
ε_x = strain
P = applied force
δ = amplitude of deformation
L = 1/2 sample length (span)
t = sample thickness
w = sample width
ν = Poison's ratio
I = moment of inertia

Shear Sandwich

When running experiments using the shear sandwich clamp, the equations found in this section are applied to obtain your results.

Modulus Equation

The stiffness model equation for a rectangular cross section sample, analyzed on the shear sandwich clamp, is as follows. (Similar calculations were performed for cylindrical samples but are not detailed here.)

$$K = \frac{5 \cdot G \cdot w \cdot h}{3 \cdot t}$$

Where:

K	=	stiffness or spring constant
G	=	shear modulus
w	=	sample width, *i.e*. horizontal dimension
h	=	sample height, *i.e*. vertical dimension
t	=	sample thickness, between clamp faces

Dilatation of the sample due to compression when the screws are tightened, causes the measured stiffness to differ from the model equation. The amount of dilatation is dependent upon the degree of compression of the sample which cannot be quantified so, a correction factor for this effect is ignored. The equation for the shear modulus is:

$$G = K_s \cdot \frac{3 \cdot t}{5 \cdot w \cdot h}$$

Where:

G = shear modulus

w = sample width, *i.e*. horizontal dimension

h = sample height, *i.e*. vertical dimension

t = sample thickness, between clamp faces

K_s = measured stiffness

Stress and Strain

$$\tau_0 = \frac{P}{2 \cdot A}$$

$$\gamma_0 = \frac{\delta}{T}$$

Where:

σ_0 = stress

γ_0 = strain

P = applied force

δ = amplitude of deformation

A = sample cross-sectional area

T = separation of clamp surfaces

Compression

When running experiments using the compression clamp, the equations found in this section are applied to obtain your results.

Modulus Equation

The stiffness model equation for a sample, analyzed on the compression clamp, is as follows.

$$K = \frac{A \cdot E}{t}$$

Where:

K = stiffness or spring constant
E = elastic modulus
A = sample cross-sectional area
t = sample thickness

The model equation assumes that transverse strains are negligible. In fact, due to dilatation effects, transverse strains in the sample are significant, therefore, the measured stiffness will be lower than the sample stiffness. A stiffness correction factor can be defined as:

$$F_e = \frac{K_S}{K}$$

Where:

K = stiffness or spring constant
K_s = measured stiffness
F_e = correction factor. See “Clamping Factors” in Chapter 6 to determine the value of Fe.

Substituting for K in the model equation and solving for the modulus,

$$E = \frac{K_s}{F_e} \cdot \frac{t}{A}$$

Where:
E = elastic modulus
t = sample thickness
K_s = measured stiffness
F_e = correction factor. See “Clamping Factors” in Chapter 6 to determine the value of Fe.
A = sample cross-sectional area

The clamping correction factor, F_e , was determined by finding the sample stiffness using *Finite Element Analysis* of the sample deformation. F_e was calculated using the sample stiffness equation and the FEA stiffness. This was done by studying many cases, including a variety of materials and geometries with corresponding fit, applied to the result.

Stress and Strain

$$\sigma_0 = Fe \cdot \frac{P}{A_0}$$

$$\gamma_0 = \frac{\delta}{T}$$

Where:

σ_0 = stress
γ_0 = strain
P = applied force
δ = amplitude of deformation
A = sample cross-sectional area
T = separation of clamp surfaces
Fe = correction factor that accounts for sample dilatation effects. See "Clamping Factors" in Chapter 6 to determine the value of Fe.

The DMA 2980 uses the nominal or engineering stress and strain, which are based on the assumption that the cross-sectional area of the sample maintains the initial value for the duration of the experiment.

Tension: Film and Fiber

When you are running experiments using the film or fiber tension clamps, the equations found in this section are applied to obtain your results.

Modulus Equation

The stiffness model equation for a sample, analyzed on the film or fiber tension clamp, is as follows.

$$K = \frac{A \cdot E}{L}$$

Where:

K = stiffness or spring constant
E = elastic modulus
A = sample cross-sectional area
L = sample length

Because the sample will have a very small area as compared to it's length, no end-effects correction is needed, the modulus equation is,

$$E = K_s \cdot \frac{L}{A}$$

Where:

E = elastic modulus
A = sample cross-sectional area
L = sample length
K_s = measured stiffness

Stress and Strain

$$\sigma_0 = \frac{P}{A_0}$$

$$\varepsilon_0 = \frac{\Delta L}{L_0}$$

Where:

σ_0 = stress
γ_0 = strain
P = applied force
ΔL = cumulative change in sample length
L_0 = initial sample length
A_0 = initial sample cross-sectional area

The DMA 2980 uses the nominal or engineering stress and strain, which are based on the assumption that the change in sample length is small for the duration of the experiment.

Clamping Factors

(Compression Clamps Only)

This section provides clamping correction factors for compression clamps that can be used to solve the equations found in the previous sections.

Table 6.1 Clamping Factors (Fe) for 1 mm Ring Sample

Thickness (mm)	OD (mm)	ID (mm)	Fe
1	5	4	0.7669
1.5	5	4	0.8026
2	5	4	0.8207
2.5	5	4	0.8314
3	5	4	0.8383
3.5	5	4	0.8430
4	5	4	0.8464
4.5	5	4	0.8488
5	5	4	0.8507
1	10	9	0.7669
1.5	10	9	0.8026
2	10	9	0.8207
2.5	10	9	0.8314
3	10	9	0.8383
3.5	10	9	0.8430
4	10	9	0.8464
4.5	10	9	0.8488
5	10	9	0.8507
1	15	14	0.7669
1.5	15	14	0.8026
2	15	14	0.8207
2.5	15	14	0.8314
3	15	14	0.8383
3.5	15	14	0.8430

(table continued)

Table 6.1
Clamping Factors (Fe)
for 1 mm Ring Sample
(continued)

Thickness (mm)	OD (mm)	ID (mm)	Fe
4	15	14	0.8464
4.5	15	14	0.8488
5	15	14	0.8507
1	20	19	0.7669
1.5	20	19	0.8026
2	20	19	0.8207
2.5	20	19	0.8314
3	20	19	0.8383
3.5	20	19	0.8430
4	20	19	0.8464
4.5	20	19	0.8488
5	20	19	0.8507
1	25	24	0.7669
1.5	25	24	0.8026
2	25	24	0.8207
2.5	25	24	0.8314
3	25	24	0.8383
3.5	25	24	0.8430
4	25	24	0.8464
4.5	25	24	0.8488
5	25	24	0.8507
1	30	29	0.7669
1.5	30	29	0.8026
2	30	29	0.8207
2.5	30	29	0.8314
3	30	29	0.8383
3.5	30	29	0.8430
4	30	29	0.8464
4.5	30	29	0.8488

(table continued)

Table 6.1
Clamping Factors (Fe) for 1 mm Ring Sample (continued)

Thickness (mm)	OD (mm)	ID (mm)	Fe
5	30	29	0.8507
1	35	34	0.7669
1.5	35	34	0.8026
2	35	34	0.8207
2.5	35	34	0.8314
3	35	34	0.8383
3.5	35	34	0.8430
4	35	34	0.8464
4.5	35	34	0.8488
5	35	34	0.8507
1	40	39	0.7669
1.5	40	39	0.8026
2	40	39	0.8207
2.5	40	39	0.8314
3	40	39	0.8383
3.5	40	39	0.8430
4	40	39	0.8464
4.5	40	39	0.8488
5	40	39	0.8507

Table 6.2
Clamping Factors (Fe)
for 2 mm Ring Sample

Thickness (mm)	OD (mm)	ID (mm)	Fe
1	5	3	0.67337
1.5	5	3	0.73322
2	5	3	0.76688
2.5	5	3	0.78811
3	5	3	0.80256
3.5	5	3	0.81293
4	5	3	0.82069
4.5	5	3	0.82666
5	5	3	0.83137
1	10	8	0.67337
1.5	10	8	0.73322
2	10	8	0.76688
2.5	10	8	0.78811
3	10	8	0.80256
3.5	10	8	0.81293
4	10	8	0.82069
4.5	10	8	0.82666
5	10	8	0.83137
1	15	13	0.67337
1.5	15	13	0.73322
2	15	13	0.76688
2.5	15	13	0.78811
3	15	13	0.80256
3.5	15	13	0.81293
4	15	13	0.82069
4.5	15	13	0.82666
5	15	13	0.83137
1	20	18	0.67337
1.5	20	18	0.73322

(table continued)

Table 6.2
Clamping Factors (Fe)
for 2 mm Ring Sample
(continued)

Thickness (mm)	OD (mm)	ID (mm)	Fe
2	20	18	0.76688
2.5	20	18	0.78811
3	20	18	0.80256
3.5	20	18	0.81293
4	20	18	0.82069
4.5	20	18	0.82666
5	20	18	0.83137
1	25	23	0.67337
1.5	25	23	0.73322
2	25	23	0.76688
2.5	25	23	0.78811
3	25	23	0.80256
3.5	25	23	0.81293
4	25	23	0.82069
4.5	25	23	0.82666
5	25	23	0.83137
1	30	28	0.67337
1.5	30	28	0.73322
2	30	28	0.76688
2.5	30	28	0.78811
3	30	28	0.80256
3.5	30	28	0.81293
4	30	28	0.82069
4.5	30	28	0.82666
5	30	28	0.83137
1	35	33	0.67337
1.5	35	33	0.73322
2	35	33	0.76688
2.5	35	33	0.78811
3	35	33	0.80256

(table continued)

Table 6.2 Clamping Factors (Fe) for 2 mm Ring Sample (continued)

Thickness (mm)	OD (mm)	ID (mm)	Fe
3.5	35	33	0.81293
4	35	33	0.82069
4.5	35	33	0.82666
5	35	33	0.83137
1	40	38	0.67337
1.5	40	38	0.73322
2	40	38	0.76688
2.5	40	38	0.78811
3	40	38	0.80256
3.5	40	38	0.81293
4	40	38	0.82069
4.5	40	38	0.82666
5	40	38	0.83137

Table 6.3
Clamping Factors (Fe)
for 3 mm Ring Sample

Thickness (mm)	OD (mm)	ID (mm)	Fe
1	5	2	0.6013
1.5	5	2	0.6734
2	5	2	0.7173
2.5	5	2	0.7464
3	5	2	0.7669
3.5	5	2	0.7820
4	5	2	0.7935
4.5	5	2	0.8026
5	5	2	0.8098
1	10	7	0.6013
1.5	10	7	0.6734
2	10	7	0.7173
2.5	10	7	0.7464
3	10	7	0.7669
3.5	10	7	0.7820
4	10	7	0.7935
4.5	10	7	0.8026
5	10	7	0.8098
1	15	12	0.6013
1.5	15	12	0.6734
2	15	12	0.7173
2.5	15	12	0.7464
3	15	12	0.7669
3.5	15	12	0.7820
4	15	12	0.7935
4.5	15	12	0.8026
5	15	12	0.8098
1	20	17	0.6013
1.5	20	17	0.6734
2	20	17	0.7173

(table continued)

Table 6.3 Clamping Factors (Fe) for 3 mm Ring Sample (continued)

Thickness (mm)	OD (mm)	ID (mm)	Fe
2.5	20	17	0.7464
3	20	17	0.7669
3.5	20	17	0.7820
4	20	17	0.7935
4.5	20	17	0.8026
5	20	17	0.8098
1	25	22	0.6013
1.5	25	22	0.6734
2	25	22	0.7173
2.5	25	22	0.7464
3	25	22	0.7669
3.5	25	22	0.7820
4	25	22	0.7935
4.5	25	22	0.8026
5	25	22	0.8098
1	30	27	0.6013
1.5	30	27	0.6734
2	30	27	0.7173
2.5	30	27	0.7464
3	30	27	0.7669
3.5	30	27	0.7820
4	30	27	0.7935
4.5	30	27	0.8026
5	30	27	0.8098
1	35	32	0.6013
1.5	35	32	0.6734
2	35	32	0.7173
2.5	35	32	0.7464
3	35	32	0.7669
3.5	35	32	0.7820

(table continued)

Table 6.3
Clamping Factors (Fe)
for 3 mm Ring Sample
(continued)

Thickness (mm)	OD (mm)	ID (mm)	Fe
4	35	32	0.7935
4.5	35	32	0.8026
5	35	32	0.8098
1	40	37	0.6013
1.5	40	37	0.6734
2	40	37	0.7173
2.5	40	37	0.7464
3	40	37	0.7669
3.5	40	37	0.7820
4	40	37	0.7935
4.5	40	37	0.8026
5	40	37	0.8098

Table 6.4
Clamping Factors (Fe)
for 4 mm Ring Sample

Thickness (mm)	OD (mm)	ID (mm)	Fe
1	5	1	0.7752
1.5	5	1	0.8539
2	5	1	0.8572
2.5	5	1	0.8390
3	5	1	0.8150
3.5	5	1	0.7903
4	5	1	0.7669
4.5	5	1	0.7452
5	5	1	0.7254
1	10	6	0.7752
1.5	10	6	0.8539
2	10	6	0.8572
2.5	10	6	0.8390
3	10	6	0.8150
3.5	10	6	0.7903
4	10	6	0.7669
4.5	10	6	0.7452
5	10	6	0.7254
1	15	11	0.7752
1.5	15	11	0.8539
2	15	11	0.8572
2.5	15	11	0.8390
3	15	11	0.8150
3.5	15	11	0.7903
4	15	11	0.7669
4.5	15	11	0.7452
5	15	11	0.7254
1	20	16	0.7752
1.5	20	16	0.8539
2	20	16	0.8572

(table continued)

Table 6.4
Clamping Factors (Fe)
for 4 mm Ring Sample
(continued)

Thickness (mm)	OD (mm)	ID (mm)	Fe
2.5	20	16	0.8390
3	20	16	0.8150
3.5	20	16	0.7903
4	20	16	0.7669
4.5	20	16	0.7452
5	20	16	0.7254
1	25	21	0.7752
1.5	25	21	0.8539
2	25	21	0.8572
2.5	25	21	0.8390
3	25	21	0.8150
3.5	25	21	0.7903
4	25	21	0.7669
4.5	25	21	0.7452
5	25	21	0.7254
1	30	26	0.7752
1.5	30	26	0.8539
2	30	26	0.8572
2.5	30	26	0.8390
3	30	26	0.8150
3.5	30	26	0.7903
4	30	26	0.7669
4.5	30	26	0.7452
5	30	26	0.7254
1	35	31	0.7752
1.5	35	31	0.8539
2	35	31	0.8572
2.5	35	31	0.8390
3	35	31	0.8150
3.5	35	31	0.7903

(table continued)

Table 6.4
Clamping Factors (Fe)
for 4 mm Ring Sample
(continued)

Thickness (mm)	OD (mm)	ID (mm)	Fe
4	35	31	0.7669
4.5	35	31	0.7452
5	35	31	0.7254
1	40	36	0.7752
1.5	40	36	0.8539
2	40	36	0.8572
2.5	40	36	0.8390
3	40	36	0.8150
3.5	40	36	0.7903
4	40	36	0.7669
4.5	40	36	0.7452
5	40	36	0.7254

Table 6.5
Clamping Factors (Fe)
for Square Sample

Thickness (mm)	Length (mm)	Fe
1	5	0.4647
1.5	5	0.5550
2	5	0.6326
2.5	5	0.6937
3	5	0.7404
3.5	5	0.7759
4	5	0.8032
4.5	5	0.8246
5	5	0.8417
1	10	0.3784
1.5	10	0.4193
2	10	0.4647
2.5	10	0.5108
3	10	0.5550
3.5	10	0.5958
4	10	0.6326
4.5	10	0.6651
5	10	0.6937
1	15	0.3558
1.5	15	0.3784
2	15	0.4050
2.5	15	0.4342
3	15	0.4647
3.5	15	0.4956
4	15	0.5259
4.5	15	0.5550
5	15	0.5827
1	20	0.3465
1.5	20	0.3610
2	20	0.3784

(table continued)

Table 6.5
Clamping Factors (Fe)
for Square Sample
(continued)

Thickness (mm)	Length (mm)	Fe
2.5	20	0.3980
3	20	0.4193
3.5	20	0.4417
4	20	0.4647
4.5	20	0.4879
5	20	0.5108
1	25	0.3415
1.5	25	0.3519
2	25	0.3643
2.5	25	0.3784
3	25	0.3940
3.5	25	0.4107
4	25	0.4282
4.5	25	0.4463
5	25	0.4647
1	30	0.3386
1.5	30	0.3465
2	30	0.3558
2.5	30	0.3665
3	30	0.3784
3.5	30	0.3913
4	30	0.4050
4.5	30	0.4193
5	30	0.4342
1	35	0.3366
1.5	35	0.3429
2	35	0.3503
2.5	35	0.3588
3	35	0.3682
3.5	35	0.3784

(table continued)

Table 6.5
Clamping Factors (Fe)
for Square Sample
(continued)

Thickness (mm)	Length (mm)	Fe
4	35	0.3894
4.5	35	0.4010
5	35	0.4131
1	40	0.3352
1.5	40	0.3404
2	40	0.3465
2.5	40	0.3534
3	40	0.3610
3.5	40	0.3694
4	40	0.3784
4.5	40	0.3880
5	40	0.3980

Table 6.6
Clamping Factors (Fe)
for Solid Circular Sample

Thickness (mm)	Diameter (mm)	Fe
1	5	0.4871
1.5	5	0.5890
2	5	0.6708
2.5	5	0.7319
3	5	0.7771
3.5	5	0.8114
4	5	0.8385
4.5	5	0.8612
5	5	0.8814
1	10	0.3842
1.5	10	0.4334
2	10	0.4871
2.5	10	0.5400
3	10	0.5890
3.5	10	0.6327
4	10	0.6708
4.5	10	0.7037
5	10	0.7319
1	15	0.3570
1.5	15	0.3842
2	15	0.4162
2.5	15	0.4511
3	15	0.4871
3.5	15	0.5226
4	15	0.5568
4.5	15	0.5890
5	15	0.6187
1	20	0.3459
1.5	20	0.3632
2	20	0.3842

(table continued)

Table 6.6
Clamping Factors (Fe)
for Solid Circular Sample
(continued)

Thickness (mm)	Diameter (mm)	Fe
2.5	20	0.4079
3	20	0.4334
3.5	20	0.4601
4	20	0.4871
4.5	20	0.5139
5	20	0.5400
1	25	0.3401
1.5	25	0.3523
2	25	0.3672
2.5	25	0.3842
3	25	0.4030
3.5	25	0.4230
4	25	0.4440
4.5	25	0.4655
5	25	0.4871
1	30	0.3367
1.5	30	0.3459
2	30	0.3570
2.5	30	0.3699
3	30	0.3842
3.5	30	0.3997
4	30	0.4162
4.5	30	0.4334
5	30	0.4511
1	35	0.3345
1.5	35	0.3417
2	35	0.3504
2.5	35	0.3605
3	35	0.3718
3.5	35	0.3842

(table continued)

Table 6.6
Clamping Factors (Fe)
for Solid Circular Sample
(continued)

Thickness (mm)	Diameter (mm)	Fe
4	35	0.3974
4.5	35	0.4114
5	35	0.4260
1	40	0.3329
1.5	40	0.3388
2	40	0.3459
2.5	40	0.3541
3	40	0.3632
3.5	40	0.3733
4	40	0.3842
4.5	40	0.3957
5	40	0.4079

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Introduction

When you have been using the TA Instruments DMA 2980 for a while, some maintenance procedures that need to be done in order to keep your instrument running as it should. Several steps are recommended:

- Periodic inspection and cleaning of the instrument.

- Calibration as required—see Chapter 3.

- Replacement of the thermocouples, as required—see the instructions enclosed with your new thermocouples.

Inspection

Examine the instrument for good condition as follows:

- Make sure the furnace area is clean and remove any residue before starting the next experiment.

- Clean the clamp and sample thermocouple of any remaining sample material before loading the next sample.

Cleaning

Cleaning your instrument regularly helps to increase its longevity and maintain its efficiency.

Cleaning the Keypad

You can clean the instrument keypad as often as desired. The keypad is covered with a silk-screen Mylar® overlay that is reasonably water resistant, but not waterproof or resistant to strong solvents or abrasives.

Use a regular household liquid glass cleaner and a paper towel to clean the keypad. Wet the towel—not the keypad—with the glass cleaner, then wipe off the keypad and display.

Cleaning the Clamps

All of the clamp assemblies are made of stainless steel and should be cleaned with that in mind. Use one or more of the following suggestions to clean any sample residue from the clamps:

- Remove the clamp from the instrument. Then, carefully scrape off residue using a razor blade or similar tool.

♦ ***CAUTION:*** **Do not use any abrasive cleanser or acidic solvents to clean the clamps. Damage to the clamp assembly could result.**

®Mylar is a registered trademark of the DuPont Company.

- Use a mild household or laboratory solvent (such a methanol or acetone) on a paper towel to wipe off excess residue.

- Bake the clamp in an oven up to 600°C to bake off residue.

Maintaining the Air Filter Regulator

The air filter regulator supplies clean air bearing gas to the DMA. It must be checked regularly to maintain optimum air-cleaning capability.

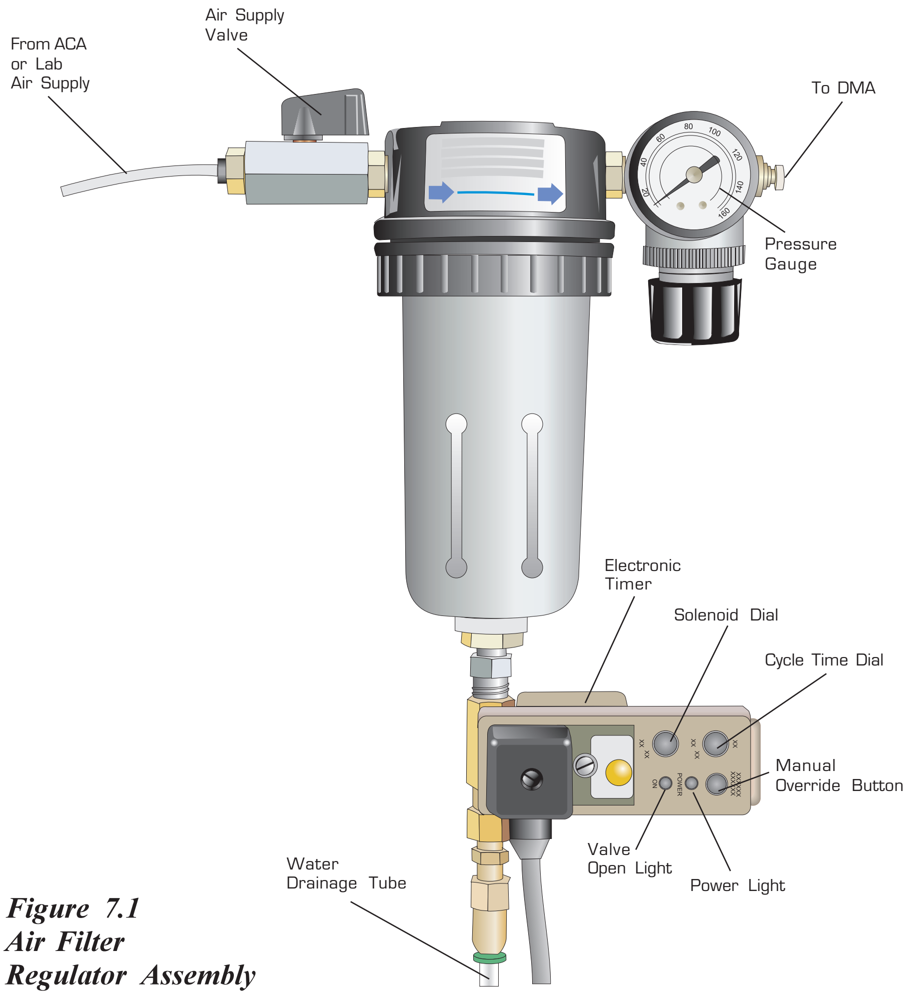

Figure 7.1
Air Filter Regulator Assembly

- *If you are using the Air Compressor Accessory connected to your air filter regulator*:

    — Check the pressure gauge on the regulator before turning on the ACA.

    — If the pressure gauge on the air filter regulator reads more than 10 psig, release the pressure by pressing the manual override button on the electronic time (see Figure 7.1) until the air stops escaping.

    — If you try to turn on the ACA with more than 10 psig pressure in the system, the ACA will draw an excessive amount of current and may overload its fuse.

- The filter should be changed once a year. See the next section for instructions.

Changing a Filter

Follow these instructions to change a filter:

1. Turn the air supply valve to the off position.

2. Press the manual override button on the electronic timer and bleed off all pressure until the pressure gauge reads zero.

3. Loosen the locking ring at the top of the bowl on the air filter regulator. Then pull straight down on the bowl to remove it.

4. Unscrew and remove the old filter.

5. Screw a new filter in place.

6. Replace the bowl and tighten the locking ring.

Error Messages

As you perform experiments using your DMA 2980, you may see error messages appearing on the display or on the controller.

- If the error is *fatal*, the error message is posted and the system halts.
- If the error is *nonfatal*, the system can continue operating.

The error messages are stored in the error log. To obtain help for an error condition, as the error is displayed on the controller, press the F1 key for online help. To obtain information about the errors contained in the error log, use the *Thermal Solutions* Instrument Control menu to access the log. Online help is available for each error in the error log. See the *Thermal Solutions User Reference Guide* for details.

Diagnosing Power Problems

Fuses

The DMA contains internal fuses that are not user serviceable. If any of the internal fuses blows, a hazard may exist. Call your TA Instruments service representative.

The only fuses that you should service yourself are the external fuses, located on the DMA's rear panel. Both are housed in safety-approved fuse carriers, labeled F1 and F2 (Figure 7.2).

!WARNING

Always unplug the instrument before you examine or replace the fuses.

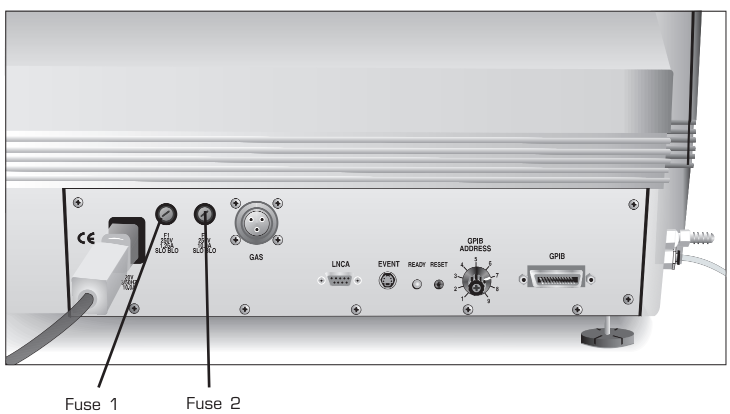

Figure 7.2
Fuse Locations

Fuse 1 is in the circuit between the main electrical input and the POWER switch. All power for internal operations and instrument functions, except heater power, passes through this fuse. If this fuse blows, you will get no response from the instrument when you attempt to turn it on.

Fuse F2 is in the circuit between the main electrical input and the POWER switch. This fuse protects all internal components, including the furnace. If this fuse blows, you will get no response from the instrument when you attempt to turn it on.

Furnace Power Check

Furnace power is always checked at the beginning of a method. Power supplied to the furnace is switched by a computer-controlled relay as well as the HEATER switch located on the instrument's front panel. The HEATER switch must be on (1) to start a method.

NOTE: The light in the HEATER switch will glow only after an experiment is initiated. The HEATER switch will continue to glow, even if it is switched to the OFF (O) position, until the "Stand By" status code is displayed.

Heater Indicator Light

The indicator light in the HEATER switch on the front panel of the DMA 2980 glows whenever the power control circuitry is enabled. If the light does not come on when the method is started, the indicator light may be defective or a hardware problem may exist in the instrument (call your TA Instruments Service Representative).

The heater light may also remain on after a method has terminated. This can happen if the method-end condition "Return to temperature range" function is chosen; see the *Thermal Solutions User Reference Guide* for further details.

Pressing the instrument **STOP** key after the completion of a method manually overrides the post-experiment heater power conditions.

Power Failures

A power failure caused by a temporary drop in line voltage results in one of two responses by the DMA instrument:

If the drop is fairly large and of long duration (20 milliseconds or more), the system will reset and go into its power-up sequence when power resumes.

If the drop is small or of short duration, the system may halt, and you may see “Err F02” on the display. This message means that the system has detected a power failure and has shut down. The instrument will not restart until it is reset. To reset, press the Reset button on the DMA’s back panel or turn the POWER switch off for a few seconds and then turn it back on.

If “Err F02” appears at start-up and remains even after you have tried to restart the instrument, the detection circuitry itself is probably at fault. Do not try to repair it yourself; call your TA Instruments service representative.

The DMA is designed for a nominal line voltage of 120 Vac (+ 10%), 50 or 60 Hz. It should not be operated outside this range. Low line voltage may result in poor instrument operation; high line voltage may damage the instrument.

DMA 2980 Test Functions

The DMA 2980 has three levels of test and diagnostic functions:

- The confidence test that is run every time the instrument is started.
- Cycling test functions that continuously test specific functions.
- A manufacturing verifier test mode that coordinates and logs the results of a sequence of confidence tests and drift runs.

These test functions are always present in the instrument. They are designed to aid manufacturing and service personnel when checking and repairing the instrument.

The Confidence Test

The DMA confidence test is run each time the instrument is turned on or reset. The confidence test checks most of the computer and interface components in the system.

When the confidence test is running, the number of the test currently being performed is shown on the display. The test number appears as a two-digit hex number on the lower right of the display. This number is changed as each new test is started. Most of the tests are very brief, so their test numbers may not be apparent.

The length of time required to run the confidence test depends on the options installed. A standard DMA 2980 system takes about 4 seconds. The longest tests are the DRAM tests, which take about 2 seconds.

After the tests are completed, a series of sign-on messages are displayed. The system then starts running, and the ready light on the back of the instrument glows.

If an error is detected, an error message is posted on the bottom line of the display. Nonfatal errors are displayed for 3 seconds, and then the confidence test continues. A fatal error occurs when a circuit essential to the operation of the instrument has failed the confidence test; the instrument cannot reliably perform any further functions. The system stops when the fatal error is posted, and the ready light remains off.

Table 7.1, on the next page, summarizes the primary confidence tests for the DMA. If any errors occur during the confidence test, call your TA Instruments service representative.

Table 7.1
DMA Confidence Test

Test Number	Area Being Tested
—	CPU logic
30	CMOS RAM
4*n*	Program memory
5*n*	CPU board I/O functions
6*n*	DRAM data storage memory
70	GPIB test
82	Keypad test
90	RS-232 test
A*n*	Analog board tests
B*n*	Drive board tests
D0	Saved memory checksum

Parts List

The following parts may be ordered for use with the DMA 2980.

Table 7.2
DMA 2980 Parts List

Part Number	Description
984286.901	Sample thermocouple
984287.901	Control thermocouple
984054.001	Drive shaft
984003.982	DMA furnace
980228.901	Glass cloth, 0.086 mm
980228.902	Glass cloth, 0.205 mm
984370.901	Furnace inlet ferrule/ spring kit
984309.901	PET film samples 1.5" long (10)
984310.901	Indium wire samples 1.5" long (10)
984313.901	PET string sample 36" long
270975.001	Filter element
890035.901	Power cord
251468.002	GPIB cable, 2 meters long
270976.001	Filter/Regulator with Auto-Drain
984011.901	**DMA Accessory Kit, which includes**:
280037.000	Tool wrench torque drive
982161.902	Sample, ABS (Acrylonitrile Butadiene Styrene) 2.35" (5)
982161.903	Sample, ABS (Acrylonitrile Butadiene Styrene) 1.4" (5)

(table continued)

Table 7.2
DMA 2980 Parts List
(cont'd)

Part Number	Description
	DMA Accessory Kit *(continued)*, which includes:
982165.902	Sample, polycarbonate 2.35" (5)
982165.903	Sample, polycarbonate 1.4" (5)
984308.001	Sample, .005" calibration
984308.002	Sample, .010" calibration
984308.003	Sample, .020" calibration
984308.004	Sample, .030" calibration
984308.005	Sample, .030" x .75 calibration
982166.003	Sample, .125 compliance, 2.35" long
982166.004	Sample, .125 compliance, 1.4" long
205220.030	Fuse, 4.0 Amp Slow Blow (M)
205220.038	Fuse, 8.0 Amp Slow Blow (M)
900902.901	Indium sample, temperature calibration
900907.901	Zinc, sample, temperature calibration
983169.001	Digital caliper B&S
270339.002	100 gram weight
259508.000	Brass tweezers
270962.002	Gauge, telescoping
984014.901	3-point bending clamp kit
984015.901	Dual cantilever clamp kit

(table continued)

Table 7.2 DMA 2980 Parts List (cont'd)

Part Number	Description
984016.901	Film\fiber tension clamp kit
984017.901	Shear sandwich clamp kit
984018.901	Compression clamp kit
984075.001	15-mm disk for compression clamp
984074.001	40-mm disk for compression clamp
984022.901	Penetration clamp kit
984023.901	Specialized fiber clamp kit
984094.001	Pin vise
984026.901	3-point bending clamp kit, for small samples
984047.901	20-mm Dual cantilever clamp kit
984048.901	8-mm Dual cantilever clamp kit
984448.901	Sumbersion compression clamp kit
984449.901	Submersion film/fiber clamp kit

Appendix A: Ordering Information

TA Instruments, Inc.
109 Lukens Drive
New Castle, DE 19720
Telephone: 1-302-427-4000 or 1-302-427-4040
Fax: 1-302-427-4001

HELPLINE—U.S.A.
For technical assistance with current or potential thermal analysis applications, please call the Thermal Analysis Help Desk at 1-302-427-4070.

SERVICE—U.S.A.
For instrument service and repairs, please call 1-302-427-4050.

TA Instruments Ltd.
Europe House, Bilton Centre
Cleeve Road
Leatherhead, Surrey KT22 7UQ
England
Telephone: 44-1372-360363
Fax: 44-1372-360135

TA Instruments GmbH
Max-Planck-Strasse 11
D-63755 Alzenau
Germany
Telephone: 49-6023-9647-0
Fax: 49-6023-9647-77

TA Instruments Benelux
Ottergemsesteenweg 461
B-9000 Gent
Belgium
Telephone: 32-9-220-79-89
Fax: 32-9-220-83-21

TA Instruments Japan
No. 5 Koike Bldg.
1-3-12 Kitashinagawa
Shinagawa-Ku, Tokyo 140
Japan
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Fax: 813/3450-1322

TA Instruments France
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78056 Saint-Quentin-Yvelines
Cedex
France
Telephone: 33-1-30-48 94 60
Fax: 33-1-30-48 94 51

TA Instruments Spain
Waters Cromatografía, S.A.
División TA Instruments
Avda. Europa, 21. Pta. Baja
28108 Alcobendas
Madrid, Spain
Telephone: 34-91-661-8448
Fax: 34-91-661-0855

TA Instruments Australia
Unit 3
38-46 South Street
Rydalmere NSW 2116
Autstralia
Telephone: 61-29-9331-705
Fax: 61-29-8981-455

TA Instruments Italy
Division of Waters SpA
via Achille Grandi 27
20090 Vimodrone (MI), Italy
Telephone: 39-02-27421-1
Fax: 39-02-250-1827

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