DMA Manual

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Sample preparation is one of the most important factors in achieving accurate and reproducible modulus values.

Basic Experimental Steps

1. Choose, install, and calibrate the clamp appropriate for the sample shape and modulus range (see the Table).
2. Position the thermocouple near the sample.
3. Select the mode of operation (DMA multi-frequency, DMA multi-strain, 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. defined by the mode and the clamp type.
6. Mount the properly prepared sample on the DMA. 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.

FILM TENSION CLAMP

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.

Installing the Clamp

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 attached 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 face of the moveable clamp and loosen the screw. 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 frame is parallel to the front face of the moveable clamp.
8. Tighten the screw 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.
11. Calibrate the clamp mass and clamp zero.

Mounting a Sample

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 on the instrument keypad 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.
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 is centered and aligned vertically on the clamp. Make sure that there is no sample under 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-lb (20 to 40 cm-N) for rigid materials].
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.
9. Position the thermocouple so that is approximately halfway between the two clamps and close to, but not touching, the sample.

Running an Experiment

You will need to set up the experimental parameters using the instrument control software.

1. These clamps are tensioning clamps; therefore, auto-strain (force track) and static force (pre-load force) values must be selected. The recommended values are 0.005 to 1N for static force (pre-load force) and 115 to 200 % for auto-strain (force track).
2. Press the MEASURE key on the DMA. This will apply the static force (pre-load 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. 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 to 18N. If the drive force is not within this range, either to 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 (Pre-load Force)- If auto-strain (force track) is used, the static force (pre-load force) will automatically be adjusted to remain a set percentage greater than the force required to drive the sample at the programmed amplitude. If auto-strain (force track) is not used, the static force (pre-load force) should read the values set in the instrument parameters.

If the initial conditions are acceptable (smooth oscillation and good modulus values), start experiment. If the values are not acceptable, then change conditions appropriately.

Removing a Sample

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

1. Loosen, but do not remove, the four hex screws holding the fixed clamp on the clamp mounting posts.
2. Loosen the screw holding the moveable clamp to the drive shaft.
3. Slide the dovetail out to remove the moveable clamp.
4. Remove the fixed clamp.

SINGLE/DUAL CANTILEVER CLAMP

The single/dual clamps are used to analyze weak to moderately stiff samples. The samples are rigidly clamped using the cantilever clamps. 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. It is very important that the sample has a uniform thickness, and that the thickness is accurately measured. 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. 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.

The sample thermocouples should be close to, but not touching the sample. The sample thermocouple is the taller one and is normally located on the right side.

Installing the Clamp

To install the film tension clamp on the DMA, follow these steps:

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 to tighten the screw in the center of the moveable clamp. Do not overtighten the screw.
3. Lower the fixed clamp carefully over the moveable clamp.
4. Line up the fixed clamp with the mounting posts and tighten the four hex screws.
5. Ensure that the moveable clamp is aligned so that is parallel to and equally spaced between the fixed clamps.
6. Make sure that the appropriate clamp type and mode are selected.
7. Position the thermocouple so that is close to, but not touching, the sample.
8. Tighten the clamping screws on the moveable and fixed clamps.
9. Calibrate the clamp.

Mounting a Sample

1. Loosen the three clamping center screws.
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.
4. Tighten the clamping screws on the fixed clamp util they are finger-tight.
5. Press the FLOAT/LOCK key again to lock the clamp in position.
6. Use the torque wrench to tighten each of the clamping screws to maximize clamping but minimize the sample deformation. Suggested clamping torque is 1.1 N-m (10 in-lb) for high modulus materials (E'>5Gpa), 0.6 to 0.9 N-m (5 to 8 in-lb) for thermoplastic samples (E' ~ 1GPa) and finger-tight for most elastomers above T_g.
7. Adjust the thermocouple so that is approximately 1 mm below and 1 mm to the side of the sample.

To obtain accurate modulus values, it is important that the sample dimensions be measured accurately. When using the dual cantilever clamp the sample length between the two fixed clamps, minus the moveable clamp's thickness. The sample length is defined by the length between the fixed and moveable clamp.

Running an Experiment

You will need to set up the experimental parameters using the instrument control software.

1. Press the Furnace key to close the furnace. Then press the MEASURE key on the instrument keypad. Observe the Advantage Signal Display window to ensure good starting conditions (smooth ocsillation and relatively constant position)
2. Press the START key on the instrument or select Start from the software menu.

Removing the Single/Dual Cantilever Clamp

1. Press the FLOAT/LOCK key to lock the clamp in place.
2. Loosen the three clamping center screws and remove sample.

Compression Clamp

Compression is used to measure the properties of low to medium modulus materials, including gels and weak elastomers. Sample size: Diameter up to 15 mm and 40 mm. Thickness: up to 10 mm.

Installing the Clamp

1. Slide the lower section of the moveable clamp onto the dovetail holder and align the d dovetail with the holder. Lightly tighten the center screw 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 mounting posts and tighten the screws.
4. Screw the lower compression disk (15 mm or 40 mm) onto the fixed clamp.
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 washer provided.
8. Check the moveable clamp for alignment.
9. Tighten all Allen screws and recheck the parallelism.
10. Select the clamp type and mode using software.
11. Calibrate the clamp mass, offset and compliance.

Running an Experiment

1. Set up the experimental parameters. Note that these clamps are tensioning clamps; therefore, auto-strain (force track) and static force (pre-load force) values must be selected. The recommended values are 0.005 to 1 N for static force (pre-load force) and 115 to 200 Percent for auto-strain (force track).
2. When you use weak samples such as gel foams and uncured elastomers, low amplitude (1-5 μm) and low auto-strain (115 to 125%) values are recommended for multi-strain and multi-frequency.
3. Press the CLAMP ▼ key on the instrument keypad to move the clamp faces together and compress the sample under the static force (pre-load force).
4. Press the MEASURE key on the DMA. This will measure the sample thickness into the experimental parameters. Then the motor will start in the desired operation mode.
5. View signals:

Amplitude- the signal should achieve and maintain the value programmed. If running a multi-strain experiment, the amplitude will cycle through the values programmed.
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 stiffness of the sample by changing the physical dimensions of the sample.
Static Force (Pre-load Force)- if auto-strain (force track) is used, the static force (pre-load force) will automatically be adjusted to remain a set % greater than the force required to drive the sample at the programmed amplitude. If auto-strain is not used, the static force should read the value set in the instrument parameters. If the initial conditions are acceptable (smooth oscillation and good modulus values) start the experiment.

Removing Sample

If the sample is not stuck to the clamps:
1. Press the clamp ▲ key to raise the moveable clamp.
2. Remove the sample and clean the compression disk face. The disks can be removed, if necessary. Be careful not to scratch their surfaces.

If the sample was cured:

1. Use a hex wrench to remove the screws on top of the moveable clamp. Press CLAMP ▼ key to lower the moveable clamp.
2. Unscrew the compression disks from the fixed clamp. The disks can be taken away from the instrument to be cleaned.

Thermal Advantage Universal Analysis

1. Universal Analysis Program: Select Start / Programs / TA Thermal Advantage / TA Universal Analysis or double click on shortcut icon. Open a data file. Locate Data / DSC / Users / Professor Name or Your Name. Make any corrections needed to the information displayed on the screen.
2. Click on the **Signal Button**. The Signal Selection is used to choose signals that are used to plot the analysis data. Select the desired y-axis and x-axis signals (Storage Modulus, Tan Delta, Storage Modulus etc). Use the **Analyze menu** to choose the type of analysis that you want perform on the current data file. Use the **Tools menu** to select from the list of different types of functions that can be performed on the graph. You can smooth, shift, and/or rotate the curve.
3. At the top of the Universal Analysis main window is a tool bar that allows you to perform the same operations found in the menus.