ARES-2 Rheometer Manual:
Torsion Testing

-McCoy Group, NMT-
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Introduction

The ARES rheometer measures the materials response under certain stresses and/or temperatures. There are 2 ARES Rheometers: an older, tan one in Jones 130C referred to as ARES-1, and a newer, blue one in Jones 128 referred to as ARES-2. Both ARES instruments have parallel plate and torsion testing functions; ARES-2 is the easiest to add a LN2 tank to for sub-ambient experiments. This manual regards ARES-2 specifically, though there is abundant procedural overlap with the operation of ARES-1.

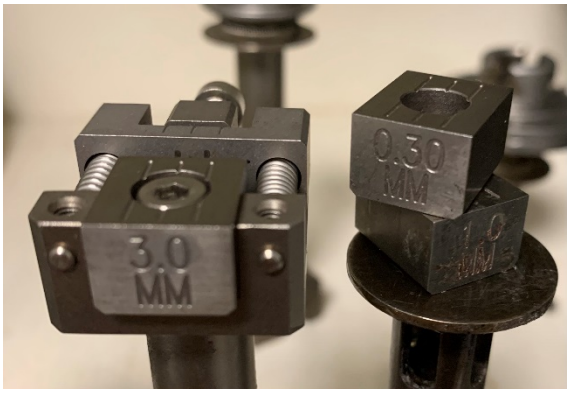
Torsion samples are solid-phase rectangular prisms. These samples are clamped on each end, and a strain is applied from the motor below. The upper transducer senses the torque response from the sample, thus characterizing various moduli and flow behaviors. This manual will detail how to perform a torsion test on the ARES-2.

Torsion Test Setup:

1. Complete the ARES startup procedure stipulated in the "Apparatus, Startup, Shutdown, and LN2" manual. **Instrument MUST NOT be used without the compressed house-air being turned on.**

2. Measure the width and thickness of the torsion sample. Each measurement should be made in three places, and the average should be taken. Ensure that the sample has straight corners and edges and has been cut without an angle.
    a. Test with appropriately sized samples:
        - Maximum sample width: 1/2 inch, 12.7 mm.
        - Maximum sample length (if oven is used: 55mm). Check to be sure the loaded sample fits in oven before attempting to close the oven to avoid damaging the ceramic of the oven. If sample is too long, remove, cut to size with diamond saw, and reload.

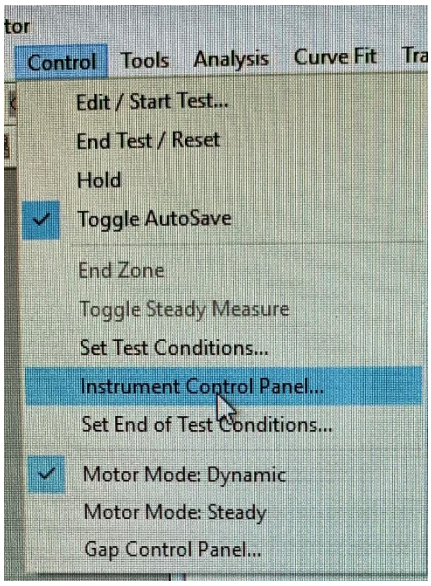
3. The blocks on torsion fixture should be changed to reflect the sample's width. For example: a sample with a thickness of 1.3mm should be clamped into a torsion fixture with 1mm blocks. (Each block has two measurements, and should be put in the fixture with the desired measurement facing out.)

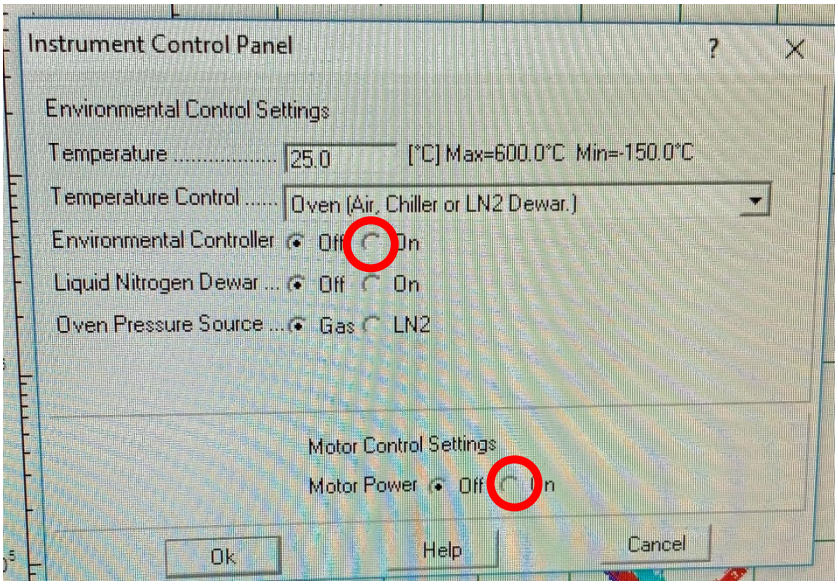


4. Attach the torsion fixtures to main machine. Hand-tighten the screws. Make sure the shorter fixture is on bottom, and make sure the upper fixture is flush with the machine. **Do not install the fixtures if the ARES is not turned on and/or does not have air.**

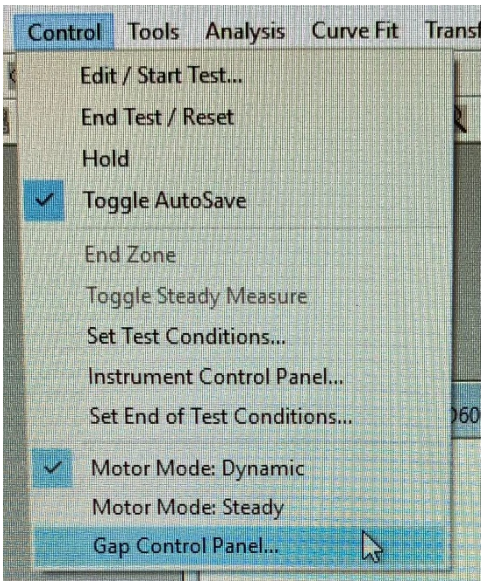
5. Go to **Utilities** in the RSI software, hover over the **Service** section, and **select Instrument Configuration**. Go to the **Temperature Control Loop** menu. Select Mode 3 (“RAA Oven Air”) . **Keep Temp Calibration Table** as **Default**.

6. Under the **Control** menu, open **Instrument Control Panel** and set the motor (and environmental controller, if oven is to be used) to **On**. Set the desired oven temperature.

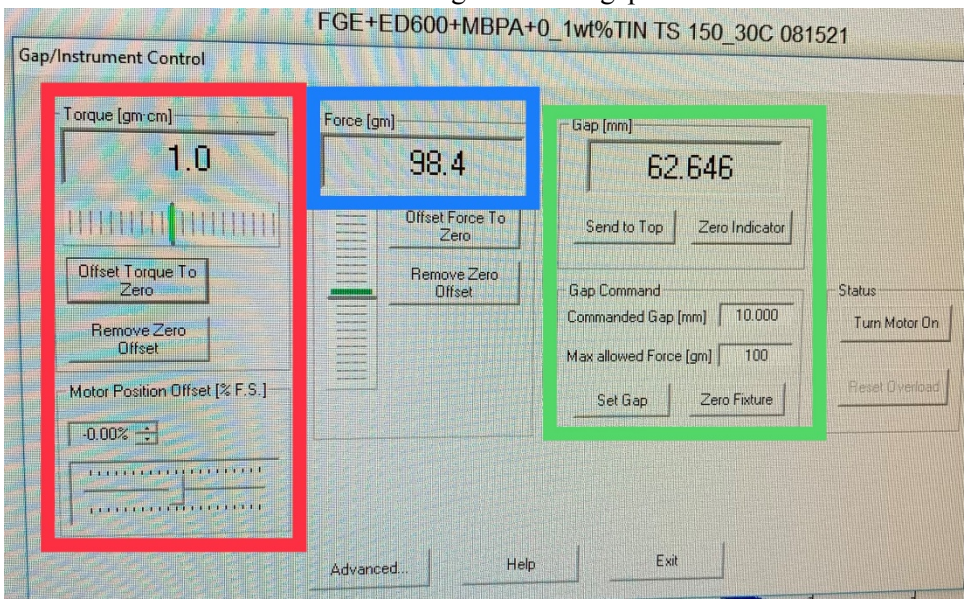




7. Lower the top fixture to a near-zero gap using the buttons on the side of the main machine. Then, zero the gap in the RSI software. Go to **Control** and select **Gap Control panel**.

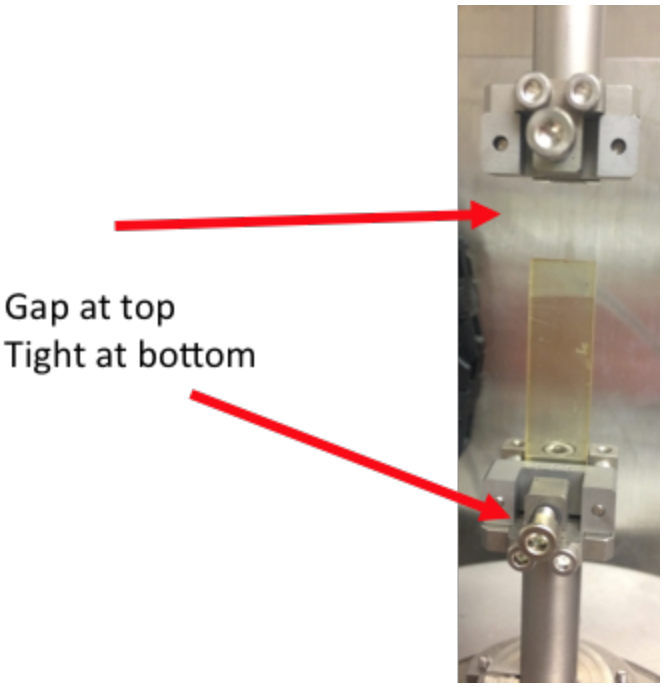


Select "Offset Torque to Zero" and "Offset Force to zero" The section of the window (pictured below) boxed in green has to do with the fixture gap. Select **Zero Fixture**, and wait for the gage to read 0.00 mm and then the value in "Commanded gap". Make sure the normal force, boxed in blue, did not spike as the instrument goes to zero gap.



8. Now the sample may be installed. Please use the appropriate precision wrench (pictured below) to load torsion samples, and not a simple Allen wrench.
    b. For 828DEA epoxy, 40 cN*m is usually employed. To change the force, pull the upper knob up, twist, and push down again.

9. **Confirm that the motor is On in the Instrument Control Panel before sample installation.** Then, proceed as such:
    a. Raise the upper fixture.
    b. Slip the sample in place in the bottom fixture. Center it, and tighten with a precision wrench set to an appropriate force limit. (This limit may be tested for in advance; the pressure on the sample should be great enough to hold it firmly, but moderate enough to avoid any deformation or splintering.)



c. Make sure the upper grip is open, and lower the top fixture over the sample. The normal force should be slightly positive, about 10-20g. The sample might need to be gently pushed forward a little at the top as it enters the upper fixture.

d. Carefully tighten the screw on the top fixture with the precision wrench. Make sure the torque (boxed in red) stays in range and adjust with the Motor Position Offset slider bar as the sample is tightened if necessary. If the motor is overloaded, the sample must be released from the upper fixture before the motor is turned on again.

Do not turn on the motor while the sample is clamped in the upper fixture. It should be on already. The motor will automatically twist into place upon startup, and if it is restrained by a clamped sample, it will be damaged. If the motor shuts off during sample loading, loosen the upper fixture, lift upper fixture and then turn the motor back on.

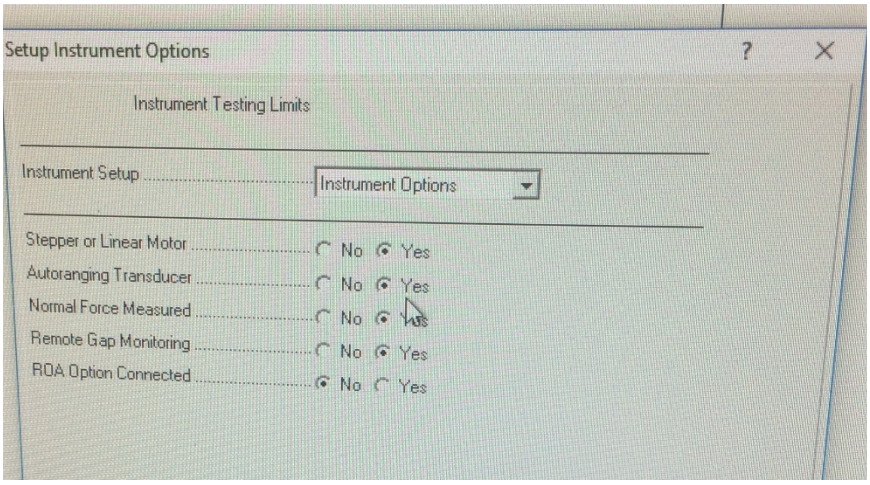
10. Zero the torque on the transducer. Within the section boxed in red, use the slider at the bottom to rotate the motor clockwise or counterclockwise. For more precise adjustment, use the up/down arrows next percent-indicator. Adjust the torque to near-zero, but **Please do not click Offset Torque to Zero. (You should have done this before loading the sample)**

11. If applicable, push the environmental chamber to the right, and close it around the sample. Let the sample equilibrate for about 10 to 30 min before starting test.

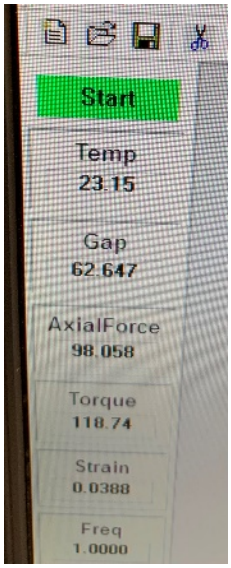
12. After equilibration, adjust the fixture position to give the sample -10g (negative, indicating tension) of normal force. Center the torque to near-zero, and zero the transducer.

Torsion Test:

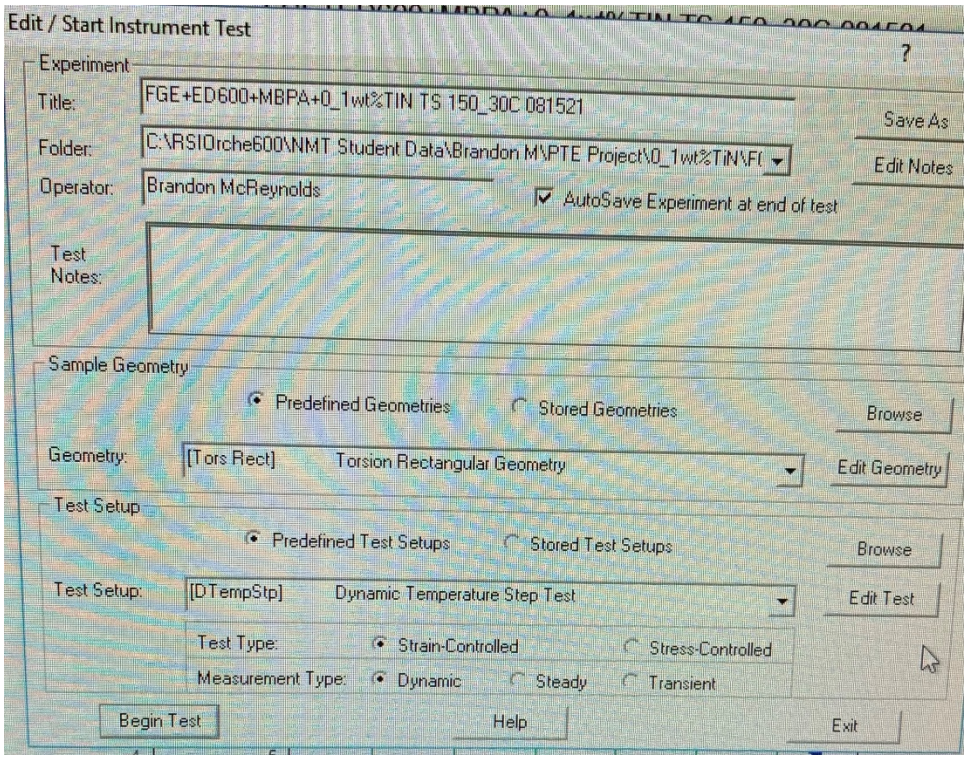
1. Before test is set up, consider whether you would like to implement multiple ranges for the transducer. If so, go to **Utilities** in the RSI software, hover over the **Service** section, and **select Instrument Configuration**. Under the Instrument Setup menu, select **Instrument Options**. Check "Yes" to **Autoranging Transducer**. This will permit both high and low moduli to be measured during a single test (e.g., as would occur in a temperature ramp for a glassy polymer)



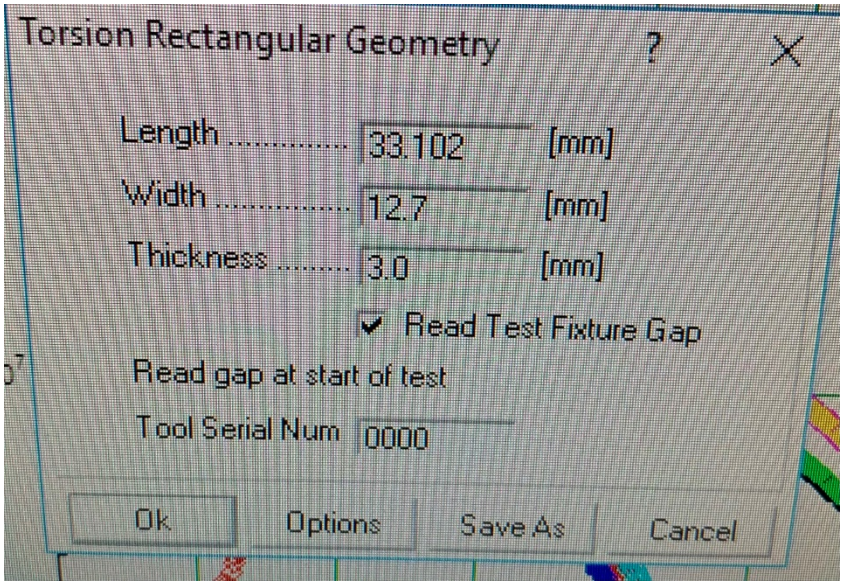
2. Click the green **Start** button, pictured below.



3. Name and save the file to the correct location. It is recommended to include sample width and thickness in the *Notes* segment.



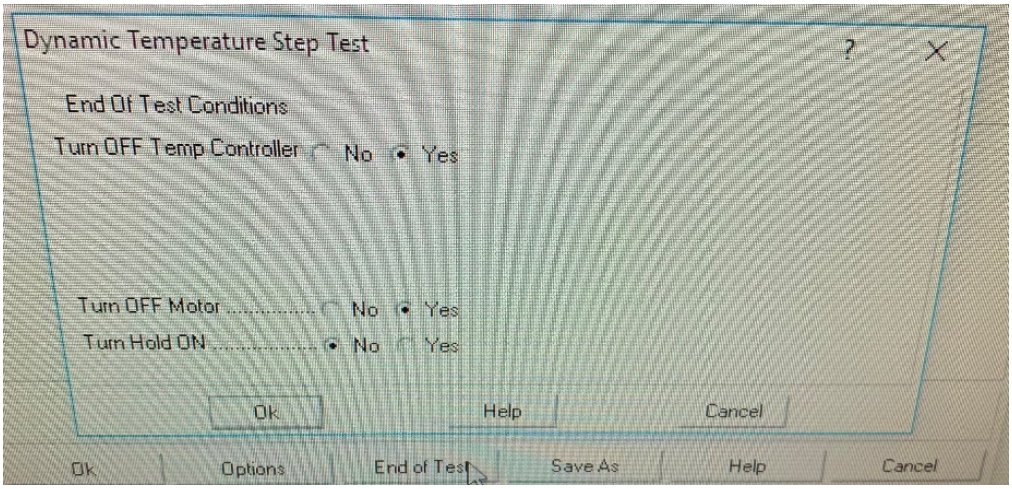
4. In the *Sample Geometry* section, select **Torsion Rectangular Geometry**. Click **Edit Geometry**, and input the sample width and thickness. The sample length is the same as the instrument gap.
    a. Click **Options**, and enter the **Tool Thermal Expansion Coefficient** as 1.81 um/C. Check the **Change Gap to Match** option. Notice that the length is the distance between the fixtures and not the total sample length



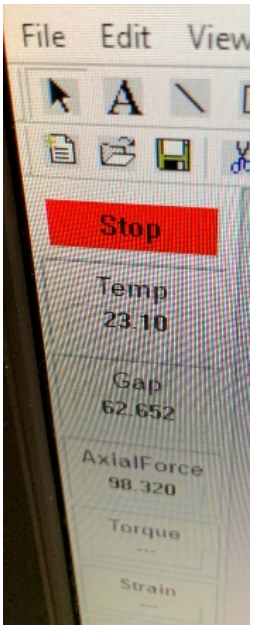
5. In the *Test Setup* section, choose your test type. Click **Edit Test**, and set the test parameters. Under **Options**, many features can be implemented if desired:

a. There can be a **Delay Before Test**, if you would like to start once the chamber is a certain temperature, or if you would have the sample soak for a certain amount of time.
b. **AutoTension** and/or **AutoStrain** can be set to keep a constant normal or torque force on the sample. For AutoTension, the usual parameters are 10g Initial Force and 2g Sensitivity. Use "Tension" for parallel plate testing. AutoStrain is not typically used, and may be set to 0 g/cm.
c. The time per measurement can be adjusted. This may be set to a higher interval for long experiments. Note that measurements cannot be taken more often than the motor frequency dictates (i.e. a 1Hz test cannot take more than 1 measurement per 1 second).

6. Once the Options have been set, open the **End of Test** menu:
   a. It is recommended to check **Turn OFF Motor**.
   b. If **Turn OFF Temp Controller** is selected, please also select **Turn Hold ON**. This will prevent motor damage as the sample changes length with temperature.



7. After all test parameters have been set, click **Begin Test**. If the test must be stopped for whatever reason, click the **Stop Test** button (pictured below)



8. **Log the run in the ARES logbook**. Include the date, the project for which the test is performed, and the duration of the run in hours.

9. Wait for the test to complete, and then perform the shutdown procedure stipulated in the "Apparatus, Startup, Shutdown, and LN2" manual.

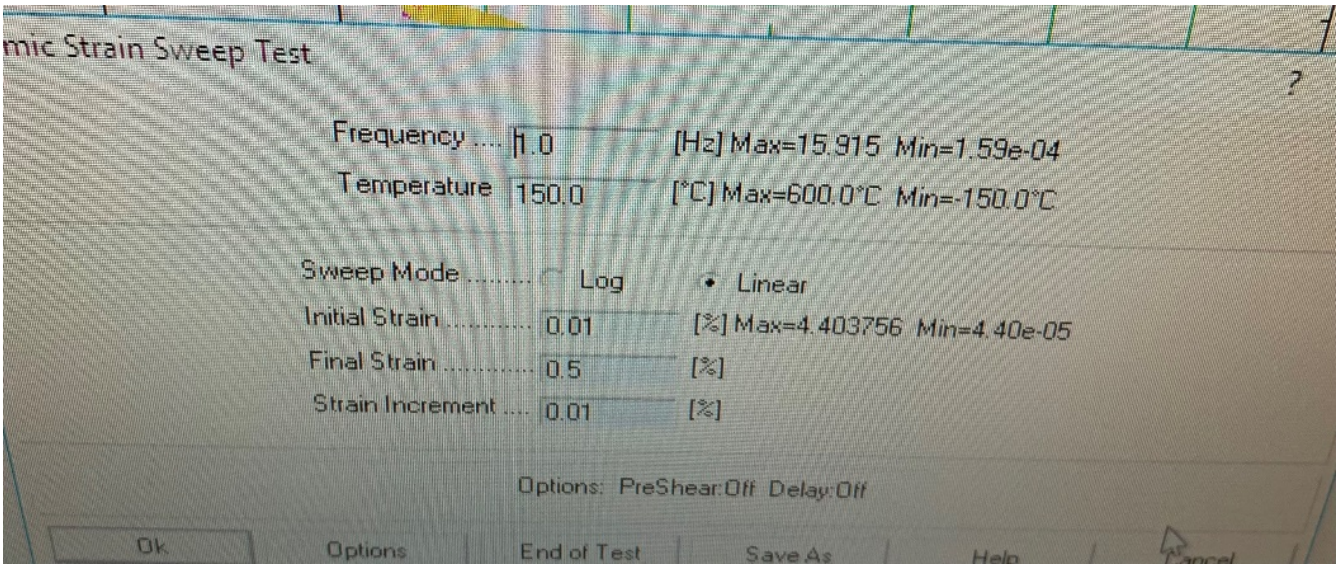
Common Torsion Tests:

Dynamic Strain Sweep

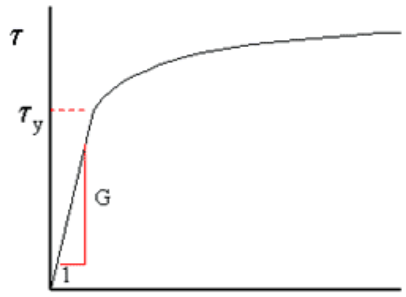
A strain sweep should be performed every time a new material, temperature, or new sample thicknesses are being tested. The purpose of this is to determine the maximum Strain % to test at.

1. The Frequency remains constant throughout the test. Usually, this is set at 1 Hz.
2. The Temperature is also remains constant throughout the test.
3. A Log or Linear sweep may be applied. Typically, we implement a linear sweep.
4. The Initial and Final Strains should be conservative. If you are uncertain where the maximum strain % may lie, start with a low Final Strain (0.1% or 0.5%).
5. The Strain Increment can be moderate. We usually use 0.01% for smaller ranges, and 0.5% for larger ranges.

Below is an example of a Dynamic Strain Sweep at ambient conditions, where the sample was brought from 0.01% strain to 0.5% strain.



6. The test should generate a Torque vs Strain plot that goes through (0,0). Make sure both axes are linearly scaled by double-clicking into the plot.



7. You are looking for a strain at which the curve loses linearity and begins to plateau.
    - If this does not occur, test again at a higher Final Strain%
    - If motor overload happens before a plateau forms, choose a strain moderately below the point of motor failure to test at.

Dynamic Temperature Ramp

This test measures the behavior of a sample as temperature increases or decreases. Please determine the appropriate maximum Strain % for the conditions before proceeding with this test.

1. The Frequency remains constant throughout the test. Usually, this is set at 1 Hz.
2. The Initial Temp is the temperature preceding Zone 1, and no other zones.
3. Each zone represents another ramp in temperature. The zones occur in order. You may use as many zones as necessary, up to 8. Ensure that all parameters of all unused zones are set to 0.
4. The Final Temp stipulates the temperature at which that zone's ramp ends. This is the next zone's initial temperature.

5. Ramp Rate determines how fast the temperature changes. 1C/min Can be used for very slow, thorough ramps, while 10C/min can be used for shorter runs.
6. Soak Time determines how much time the sample remains at the final temperature before the next zone begins (or the test ends).
7. % Strain is determined for each zone depending on the material being tested and the temperature.

Below is an example of a Dynamic Temperature Ramp test where a sample is brought from room temperature to 100C and back down again.

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