TESTING AN UNKNOWN MATERIAL USING A RHEOMETER

Fred Mazzeo, Ph.D.
TA Instruments, 109 Lukens Drive, New Castle, DE-19720, USA

This guide is used for testing a material that has not yet been rheologically evaluated. This guide will focus on evaluation of a material, being classified as either a dispersion or a polymer. Not all procedures listed in this guide are suited for all materials, and other procedures, not discussed, may be more appropriate with certain materials. The suggested conditions for any of the procedures that are listed can be used independently to further evaluate a materials rheological response. Please note that any compositional modification made to a material or change in evaluation temperature will influence the material's rheological behavior. Therefore, the material should be rheologically re-evaluated to ensure proper conditions are applied during measurement. It is recommended that a new sample be used with each test.

METHOD OF ATTACK

Preliminary Testing

This section will discuss how to determine certain preliminary procedural conditions to use to further evaluate an unknown material.

1. Determine Pseudo-Linear Viscoelastic Region (LVR) [Figure 1]

This procedure will give a general range of where the LVR is located.

Use an Oscillatory Stress Sweep (OSS)

Conditioning step

- Set temperature
- Equilibration time = ~ 5 minutes
    - This is an approximate time in order for any structure to build and/or sample and geometry to come to thermal equilibration before data acquisition begins

Stress Sweep step

- Control variable: Broad torque range (1 – 10,000 μN.m)

Since this is an unknown sample, a good rule of thumb is to test over the allowable shear stress (torque) range of the instrument. In subsequent testing, this shear stress (torque) range can be adjusted appropriately to collect only reliable data. Because the allowable shear stress range is dependant on the geometry used, torque will be used as the controlled variable. The SI units for torque are N.m. After the data is generated, the x-axis can then be converted to shear stress or %strain.

- Frequency = 1 Hz
- 10 pts per decade (log mode)

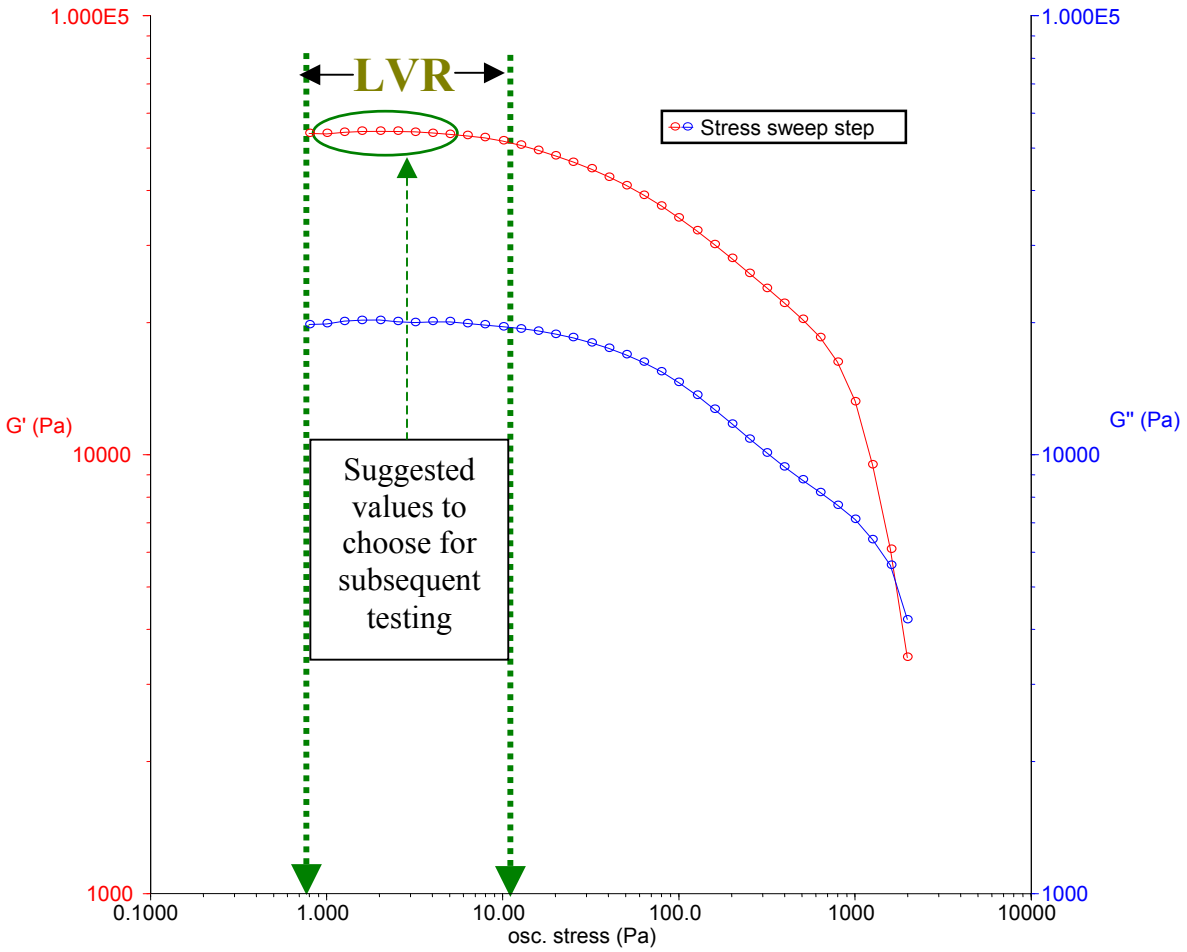

Figure 1

2. Determine Pseudo-Viscosity profile [Figure 2] if a material requires being pre-sheared before collecting data (i.e. dispersion or filled polymer systems). Pre-shearing will determine a zero-time of shear, effectively eliminating any structure history prior to loading.

Use a Continuous Flow test (**CF**)

Conditioning step

- Set temperature
- Equilibration time = Same time in **Step 1**

Continuous Flow step

- Broad torque range
- 10 pts per decade (log mode)

* Data then can be viewed as viscosity vs. torque/stress and converted to viscosity vs. shear rate (Figure 2)

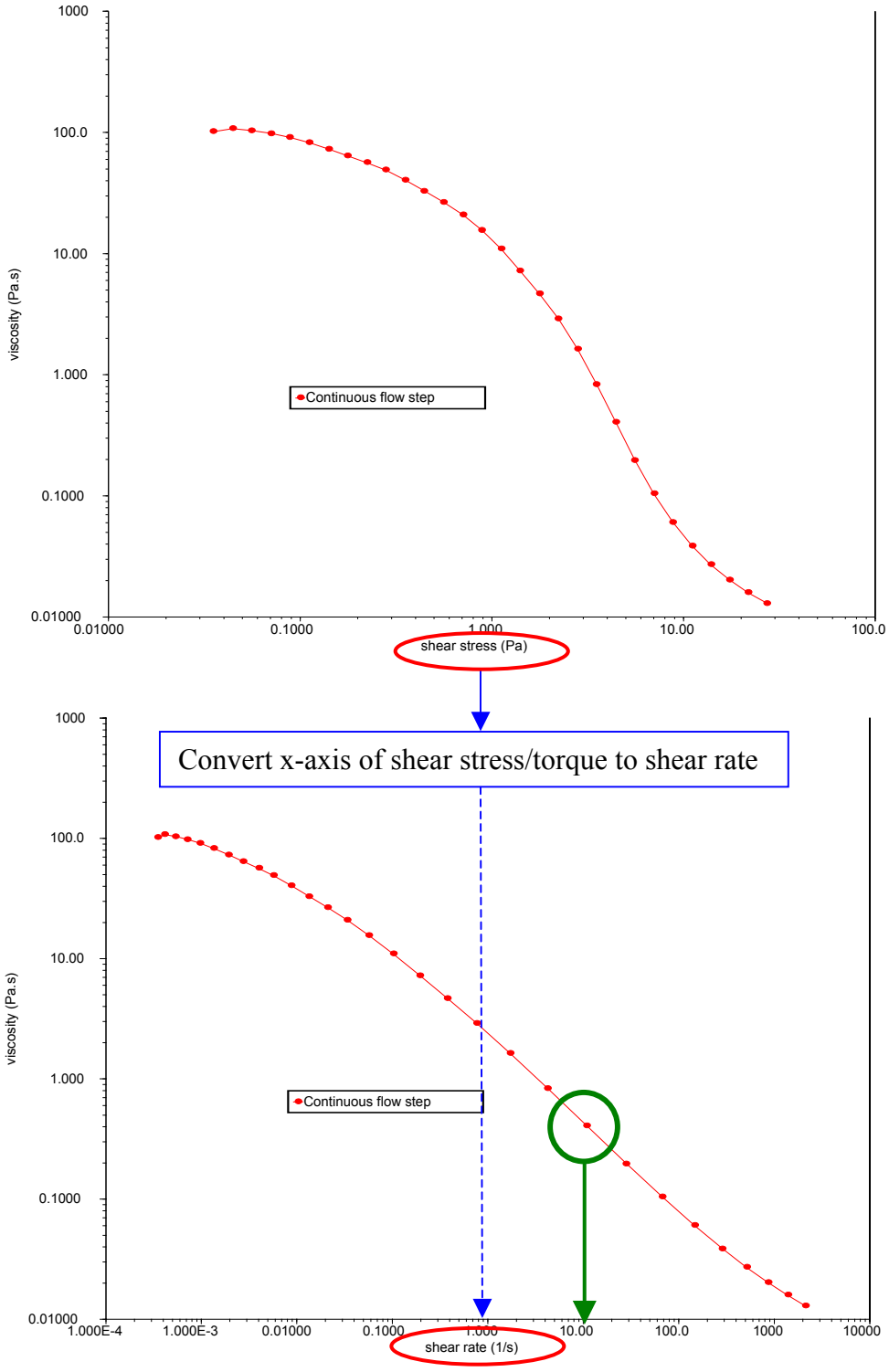
1000
100.0
10.00
1.000
0.1000
0.01000
viscosity (Pa.s)
Continuous flow step
0.01000
0.1000
1.000
10.00
100.0
shear stress (Pa)
Convert x-axis of shear stress/torque to shear rate
1000
100.0
10.00
1.000
0.1000
0.01000
viscosity (Pa.s)
Continuous flow step
1.000E-4
1.000E-3
0.01000
0.1000
1.000
10.00
100.0
1000
10000
shear rate (1/s)

Figure 2

3. Determine if the material properties are changing over the time of testing, i.e. the necessary amount of time to form a stable structure or for a polymer to degrade. [Figure 3]

Use an Oscillatory Time Sweep (**OTS**)

Conditioning step

- Set temperature
- Pre-shear = value of shear rate beyond the 1st Newtonian plateau from **CF** in **Step 2**
- Equilibration time = 0

Time Sweep step

- Choose time duration of experiment ~ 15 minutes
- Frequency = 1 Hz
- Control Variable: Choose a value of oscillation shear stress/torque within the **LVR** from **Step 1**
- Sampling time= 5 seconds

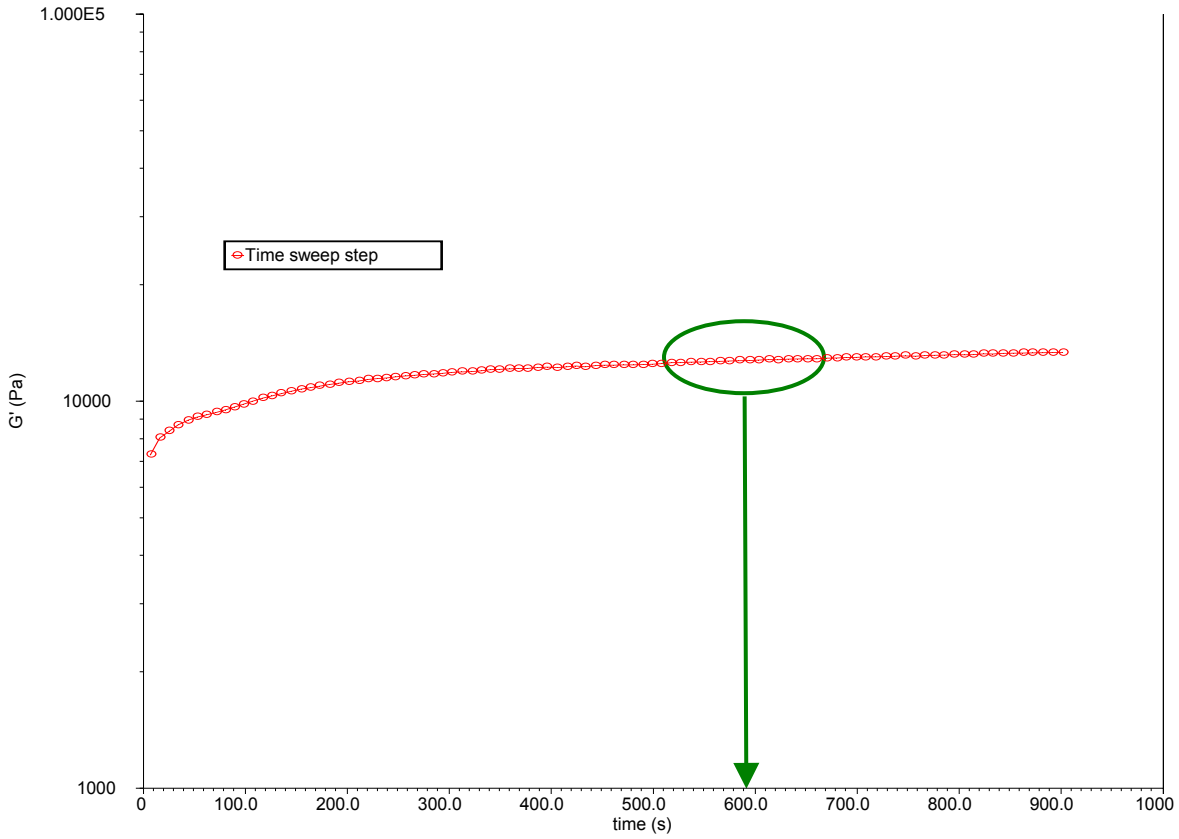

Figure 3

4. Determine True LVR (*Perform test if value of time to form a stable structure in **Step 3** is greater than equilibration time chosen in **Step 1**)

Use an Oscillatory Stress Sweep (**OSS**)

Conditioning step

- Set temperature
- Pre-shear = value of shear rate beyond the 1st Newtonian plateau from **CF** in **Step 2**
- Equilibration time = the amount of time necessary to obtain a stable profile from **OTS** in **Step 3**, by determining when the elastic modulus G' becomes relatively constant or stable

Stress Sweep step

- Adjusted shear stress/torque range from **Step 1** to collect only reliable data
- Frequency = 1 Hz
- 10 pts per decade (log mode)

Additional Testing

The following additional procedures can be used in conjunction with the above preliminary testing for further material characterization:

A. Oscillatory Frequency Sweep (**OFS**)

Conditioning step

- Set temperature
- Pre-shear = value of shear rate beyond the 1st Newtonian plateau from **CF** in **Step 2**
- Equilibration time = the amount of time necessary to obtain a stable profile from **OTS** in **Step 3**, by determining when the elastic modulus G' is relatively constant

Frequency sweep step

- Frequency range = 100Hz - 0.1Hz
- 5 pts per decade (log mode)
- Controlled variable
  - %Strain = value within **LVR** found from the **OSS** in **Step 4** by plotting G' vs. % strain
  - Or Shear stress = value within the **LVR** from **OSS** in **Step 4**

B. Steady State Flow (**SSF**)

Conditioning step

- Set temperature
- Pre-shear = value of shear rate beyond the 1st Newtonian plateau from **CF** in **Step 2**
- Equilibration time = the amount of time necessary to obtain a stable profile from **OTS** in **Step 3**, by determining when the elastic modulus G' is relatively constant

Steady state flow step

- Broad or adjusted shear stress / shear rate range from **Step 2** to collect only reliable data
- 10 pts per decade
- Percent tolerance= 10%
- Consecutive within tolerance = 3
- Maximum point time = 1:30

* Data can then be viewed as viscosity vs. torque/stress and converted to viscosity vs. shear rate

C. Creep / Recovery test (**C/R**)

Conditioning step

- Set temperature
- Pre-shear = value of shear rate beyond the 1st Newtonian plateau from **CF** in **Step 2**
- Equilibration time = the amount of time necessary to obtain a stable structure from **OTS** in **Step 3**, by determining when the elastic modulus G' is relatively constant

Retardation

- Select a shear stress from within the 1st Newtonian plateau of **SSF**
- Duration should be set to ~15 minutes or enough time for slope to be constant (Equilibrium settings can be used to detect steady state conditions)

Recovery

- Shear stress = 0
- Duration should be set to ~10 minutes or enough time for slope to be constant (Equilibrium settings can be used to detect steady state conditions)