

THERMAL APPLICATIONS NOTE

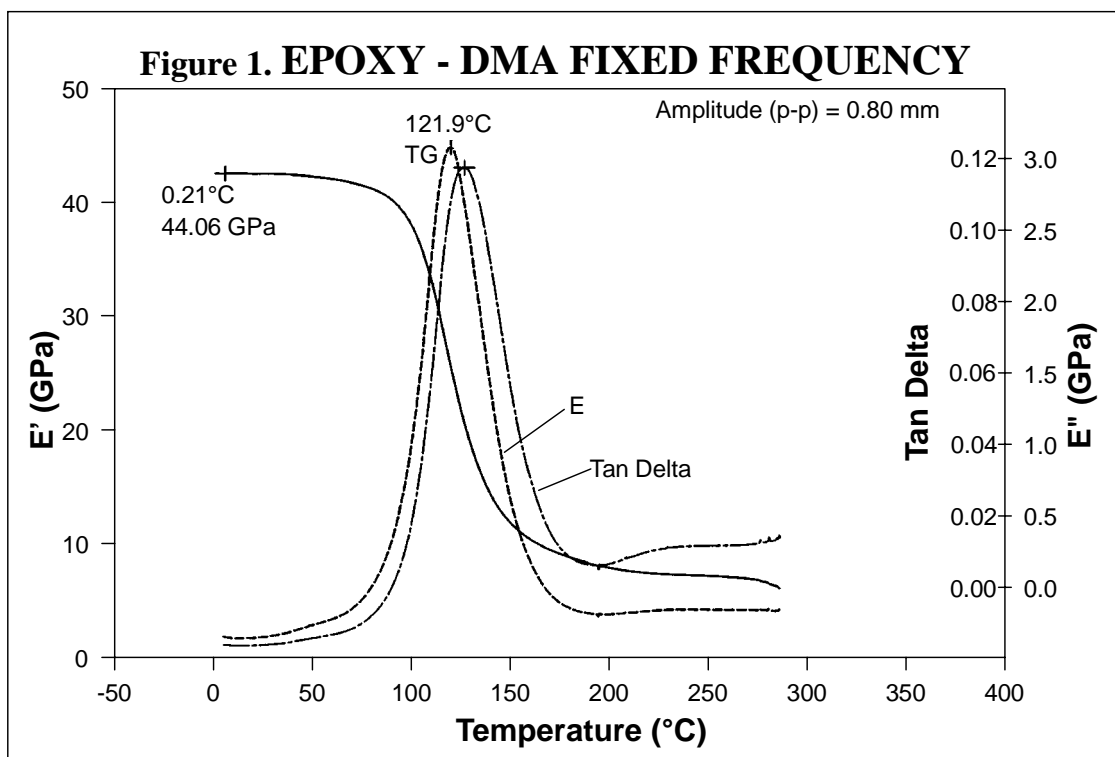
GUIDELINES FOR PERFORMING DMA CREEP EXPERIMENTS*

The TA Instruments DMA 983 Dynamic Mechanical Analyzer features four modes of operation (resonant frequency oscillation, fixed frequency oscillation, stress relaxation, and creep) providing versatility for complete characterization of materials. Although not as widely used as fixed frequency oscillation, creep, which measures material deformation with time under constant stress (load), is a valuable tool for rapidly accessing a material's long-term properties. Several recent papers are available which illustrate this point (1,2).

This Note describes the technique for running a creep experiment and for using time/temperature superposition software to project long-term material performance. The technique described can be used for amorphous or semi-crystalline thermoplastics as well as thermosets.

EXPERIMENTAL PROCEDURE

Begin by first running a "scouting" experiment on the material in the fixed frequency mode to establish the optimum temperature range to conduct the creep experiment. In addition, this "scouting" experiment provides an understanding of the material's ambient temperature modulus as well as its glass transition temperature [appears as a peak in the loss modulus (E'') curve]. The sample material should be cut to yield a test specimen that is up to 3 mm (0.125 inch) thick by up to 12.5 mm (0.5 inch) wide by up to 75 mm (3 inches) long. A clamping distance of 10:1 length:thickness ratio should be used for thermoplastics or unfilled thermosets. A 20:1 ratio should be used for stiffer composites. A fixed frequency of 1 Hz at an amplitude of 0.5 - 1.0 mm peak to peak is recommended. The typical DMA plot obtained is shown in Figure 1.



The DMA 983 is then converted to the creep mode by choosing [F12] - INSTRUMENT CONTROL, [F4] - EXPERIMENTAL PARAMETERS, and [F4] - SELECT MODE. After selecting the creep mode, access the MODULE PARAMETERS [F5] screen and set the following conditions:

- **Displacement** - Most materials can be evaluated at 0.1 - 0.4 mm. Choose 0.2 mm initially.
- **Temperature Dependent Load** - Choose "No".
- **Data Factor** - Enter 4.
- **Log Factor** - Enter 1.1 or 1.2.
- **Equilibrate Criteria** - Choose "Time".
- **Equilibrate Time** - Enter 15 - 30 minutes. This example used 15 minutes.
- **Data Collection Threshold** - Enter 0 in both fields.
- Leave other variables at their default values (i.e. unchanged).

Access the METHOD EDITOR [F2] and create a creep method using the following segments:

1. **Displacement Time:** Displace 1 minute; Recover 1 minute. This initial Displacement Time segment sets the force at room temperature and allows any clamping relaxation to occur before the true experimental data (segment #3) is collected.
2. **Equilibrate:** Equilibrate at approximately 40°C below the glass transition temperature as determined from the "scouting run." The starting temperature was 80°C in the example shown in Figure 1. Note that the projected use temperature should be included in the creep experiments, so the starting temperature may need to be more than 40°C lower than the T_g.
3. **Displacement Time:** Displace 15-30 minutes; Recover 15-30 minutes. The recover time should be at least as long as the displacement time.
4. **Increment Temperature:** Increment temperature 2 -10°C. 5°C is often very useful. The increment 2.5°C was chosen in this example.
5. **Repeat Until Final Temperature:** Repeat from segment 3 until some temperature above the final experiment temperature, which is usually T_g. This segment is like a "do loop." It tells the DMA to repeat segments 3 and 4 until the final temperature is at or above the programmed temperature. In this experiment the last creep experiment was performed at the T_g (120°C), and the value for the final temperature listed in the segment was 121°C.

Clamp the sample in the DMA. Use a clamping length of 15-20 mm for thermoplastics or unfilled thermosets. Use 25-35 mm for high modulus thermoset composites. The goal is to select a clamping distance and displacement which yields a creep drive signal in the range 1000 - 8000 mV. [Note: Maximum system drive signal is 10,000 mV.] Tighten the sample in the vertical serrated clamps with a torque of 0.7 - 1.4 Nm (6 - 12 in lb). Zero the arms as usual and start the experiment.

After the 15 minute equilibration time, the DMA performs the first displacement. Observe the drive signal (on the Signal Control screen) to ensure the value is 1000 - 8000 mV. If it is not, reject the run and adjust sample length and or displacement to bring the drive signal into the optimum range. Decreasing sample length or increasing displacement increases the drive signal. Alternatively, you may choose to adjust the sample length and displacement to yield a particular stress level. The stress level (σ) is related to the drive signal and the sample geometry and can be calculated from:

$$\sigma = \frac{24\pi^2 J C' V L}{B R T^2 W}$$

where σ = stress level (MPa)
 J = moment of inertia (g-m²)
 C' = drive signal constant (mm/mV-sec²)
 V = drive signal (mV)
 L, T, W = sample length, thickness and width (mm)
 B = pivot center distance = L + 16mm
 R = arm length = 128 mm

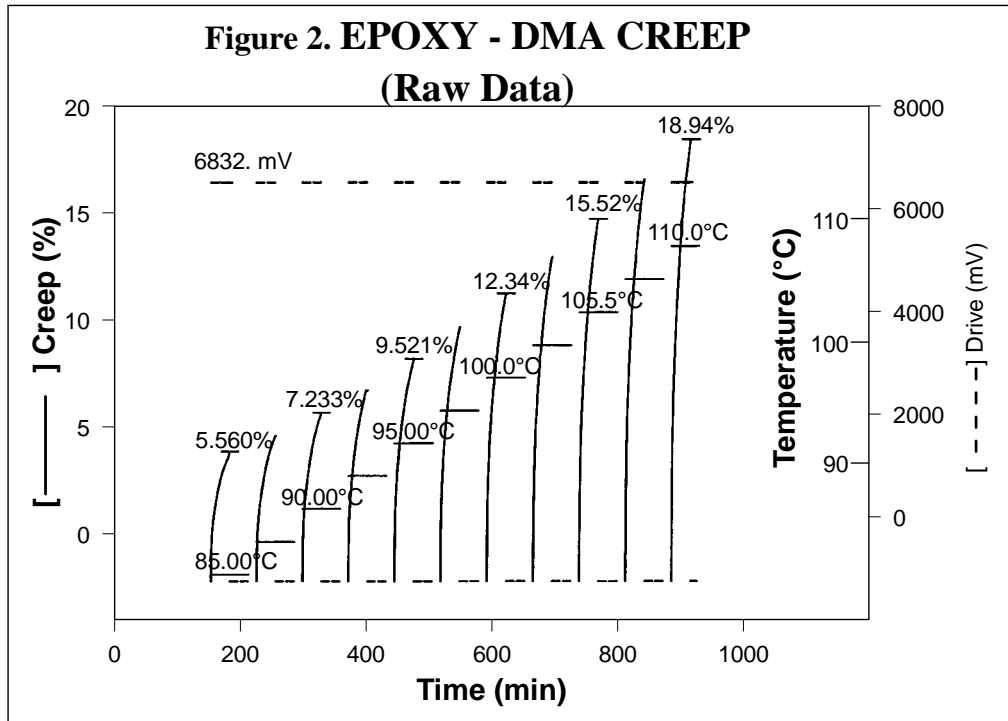
Creep experiments are long and hence should be run overnight. For example, the study here took 16 hours.

DATA ANALYSIS

Use the DMA Standard Data Analysis Software to playback the % Creep information (see Figure 2). Select the following plot curves:

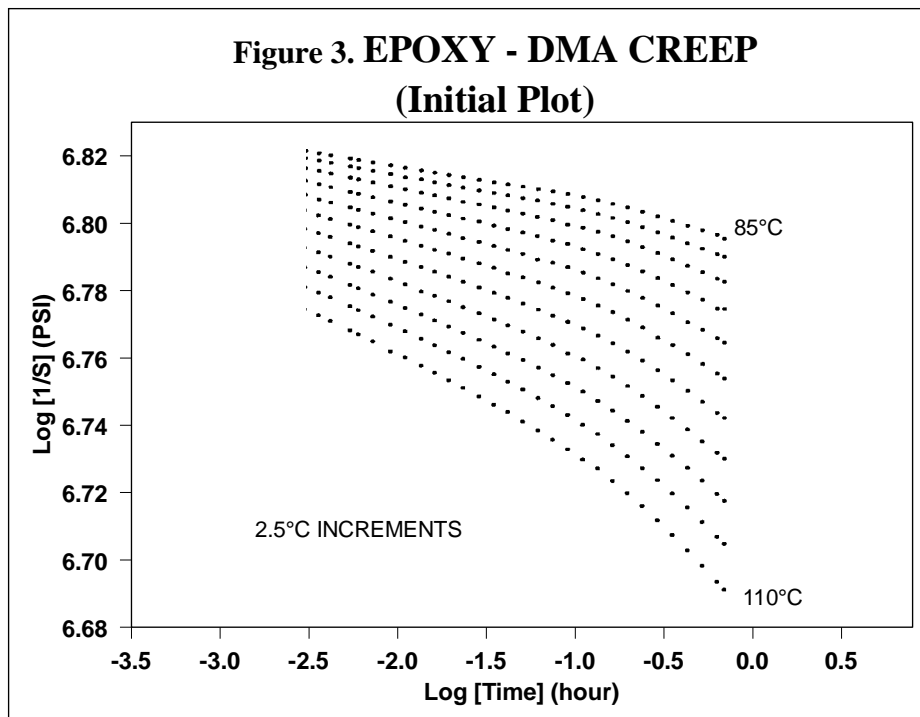
Y-axis Curve 1	% Creep
Y-axis Curve 2	Drive Signal
Y-axis Curve 3	Temperature
X-axis	Time

Use CUSTOMIZE PLOT [F2] and CURVE VALUE [F5] to determine the % Creep at each temperature increment. These % Creep values should increase as temperature increases. This data helps determine the temperature limits for superpositioning.

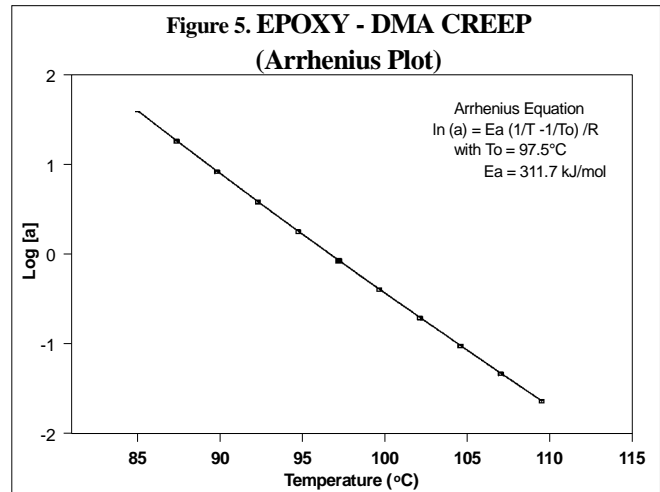
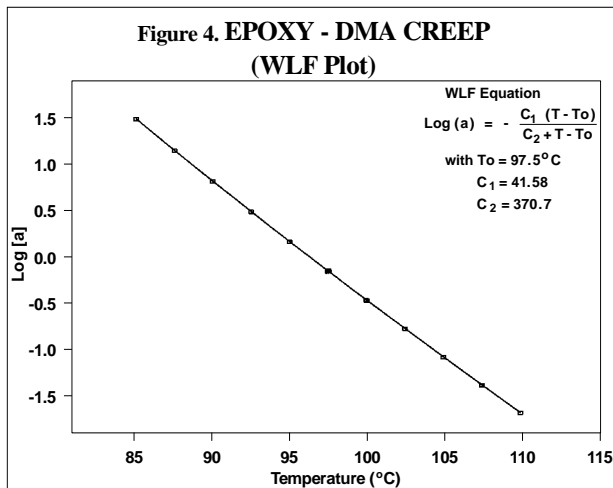


Change to the DMA Superpositioning Software. Develop a master curve as follows:

1. Enter the filename.
2. Accept sample ID, size, and instrument constants.
3. Select "NONE" for Theoretical Vertical Shift.
4. Select TEMPERATURE LIMITS [F2] and enter in the desired start and stop limits (using Figure 2). Note: Make sure to exclude the initial creep segment.
5. Plot "Flexure Creep Modulus (1/S) versus Time". Select time units as hours, 1/time as hertz, and modulus as either pascals or psi.
6. Check the raw creep data (Figure 3) to assure there are no increment segments that cross each other.
7. Press [F5]-GO TO SUPERPOSITION.
8. Press [F1]-PERFORM SHIFTING.
9. Press [F7]- SWITCH TO TABLE. Select a reference temperature.
10. Press [F2]-CHANGE SCALING.
 - X-Axis - Change the start value by 2X and increase the stop value to 2.
 - Y-Axis - Do not change.
11. For each segment (temperature increment)
 - Press the right arrow to shift the segment (red curve) to lie on top of the reference (blue) segment.
 - Use [F1] - ZOOM IN to fine tune the overlap of the curves, then press ENTER.
 - Next, press the left arrow to shift the second segment to lie on top of the reference (blue) segment.
 - Use [F1] - ZOOM IN to fine tune the overlap of the curves, then press ENTER.
 - Repeat the procedure, alternating between sides until all the curves are overlaid.

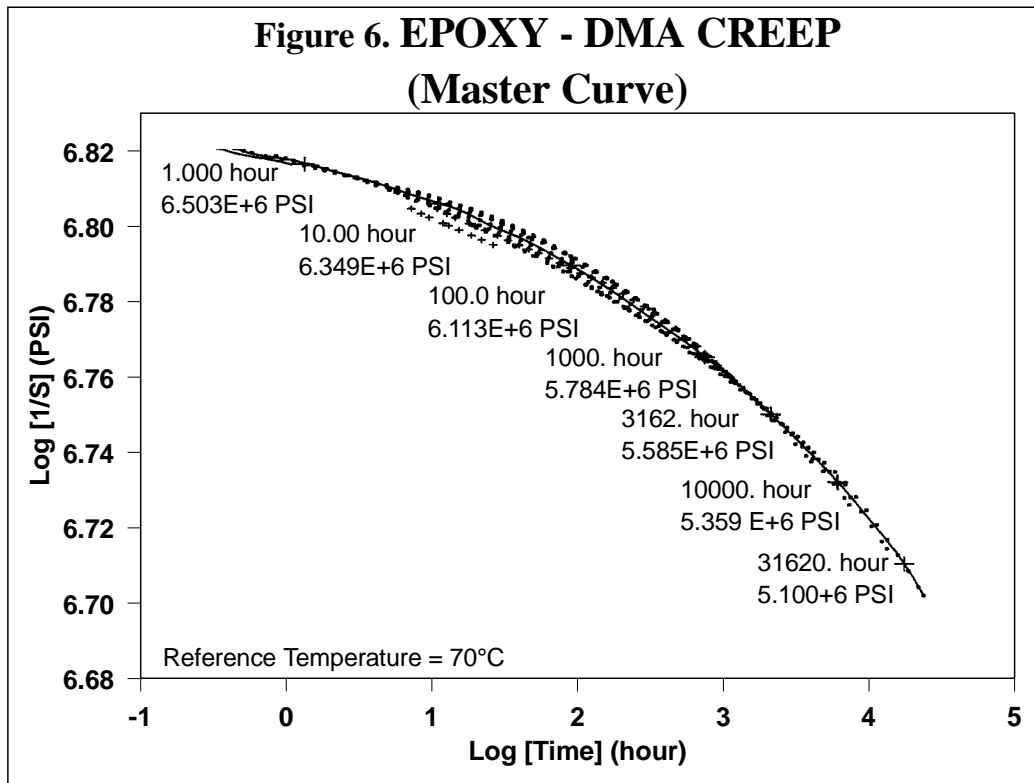


12. Press [ESC].
13. Chose [F2] - FIT HORIZONTAL SHIFTS.
14. Select a model - either [F3] - WLF EQUATION or [F4] - ARRHENIUS EQUATION. The WLF model (Figure 4) is generally better for thermoplastic materials, where as the Arrhenius model (Figure 5) is better for thermosets.



15. You may want to STORE COMPUTED SHIFTS [F5]. This force-fits the creep data to the model chosen in step 14. To obtain a hard copy of this curve (with or without storing the computed shifts) press [F7] - PLOT OPTIONS, [F4] - HARD COPY. It's also a good idea to print out a report at this time. When the menu returns, press [F6] - GO TO PRINT REPORT. After printing, press [ESC] twice.
16. Press [F4] - COMPUTE MASTER CURVE.
 - Enter the desired reference temperature if other than default. Temperatures 5-10°C just outside the experimental temperature range may be choosen.
 - Use 25 as the number of knots in the smoothed master curve.
17. Press [ESC] to return to the main menu.

18. To label the curve, press [F2] - CUSTOMIZE PLOT, [F5] - CURVE VALUE and position the cursor at the desired time values to generate the corresponding X/Y (modulus/time) values.
19. To plot the completed Master Curve, press [F4] - HARD COPY, and [F1] - PLOTTER. An example curve is shown in Figure 6.
20. If another Master Curve is desired using a different reference curve, press [F5] - GO TO SUPERPOSITION, and repeat steps 16 through 19 as described above.



References:

1. M. Sepe, Material Selection for Elevated Temperature Applications: An Alternative to DTUL, TA Instruments White Paper, TA-133.
2. TA Instruments Hotline Newsletter, Vol 4, (1993).

*These guidelines are based on a system with RMX software and the DMA 983. They do not apply directly to the DMA 2980 system with the OS/2[™]-based *Thermal Solutions* software.

Acknowledgment: This applications note was submitted by Marvin Grzys, Technical Sales Manager (US).

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