Improvements in DMA Measurements Using Low-Friction 3-Point Bending Clamps

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ABSTRACT

For modulus measurements, the purest form of evaluation occurs when using 3-point bending and rectangular samples. If, however, the sample has a twist along its length, then there is only partial contact between the clamp and the sample across its width at the middle. This creates an artifact in the thermal curves of an apparent increase in the storage modulus just before the onset of the glass transition. New low-friction self-adjusting 3-point bending clamps, offered by TA instruments, eliminate this artifact.

INTRODUCTION

One of the most common experiments performed in dynamic mechanical analysis is heating with continuous mechanical oscillation of the test specimen at constant frequency and amplitude. This experiment provides valuable information about the glass transition temperature (T_g), the change in modulus and stiffness of the material with temperature, as well as qualitative information about cross-linking, molecular weight, etc. When using the 3-point bending clamps, it is assumed that the sample has good contact with the clamps. However, if there is a twist in the sample, no matter how subtle, the contact is less than adequate. Figure 1 illustrates this effect in an exaggerated form.

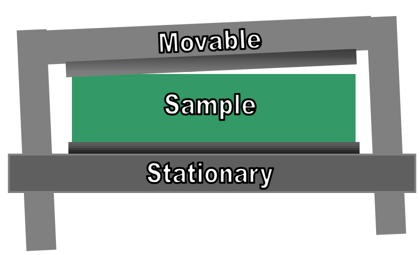

Figure 1 – Poor Sample Clamping

For very rigid, high modulus materials, such as high-performance composites, the inadequate contact leads to erroneous evaluations of modulus. Even very small twists in the sample cause the effects shown in Figure 2. The storage modulus seems to increase near 135 °C just before the glass transition. This occurs as the material softens and conforms to the clamp geometry. The clamp then experiences the full width of the sample responding to the oscillations, causing higher modulus readings.

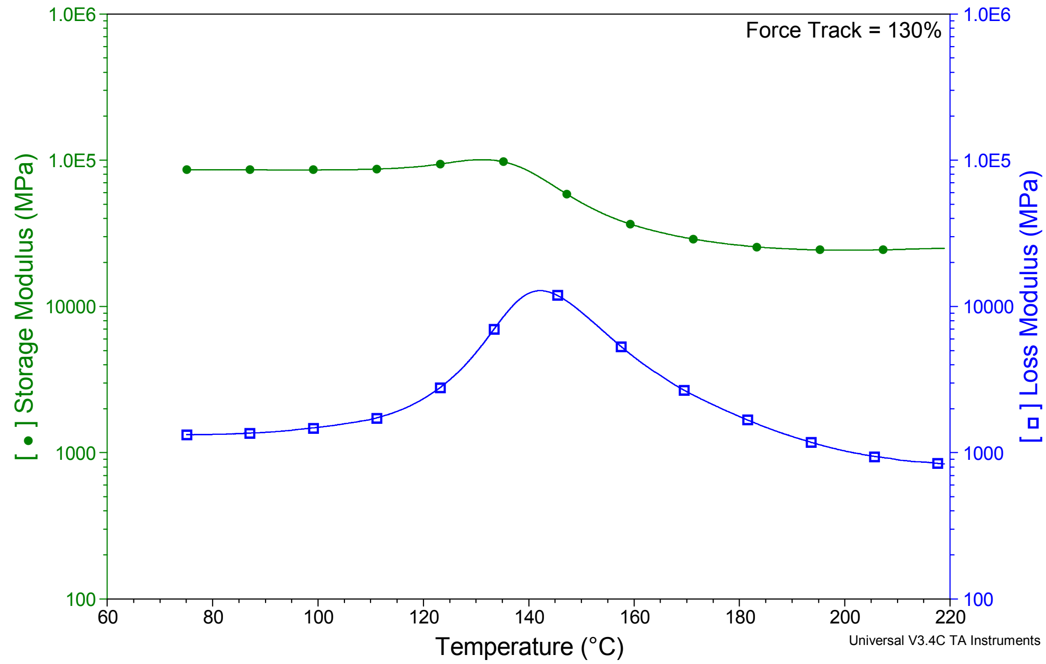

Figure 2 - Glass Transition with Poor Clamp Contact and low Force-Track

This artifactual effect may be overcome by applying a larger overall force to pre-bend the sample. That is, if the Force-Track value is increased, the overall load on the sample is greater. This may 'straighten' the sample to conform to the clamps. For example, Figure 3 shows the response of the same material, run using 250 % Force-Track compared to the 130 % used in Figure 2.

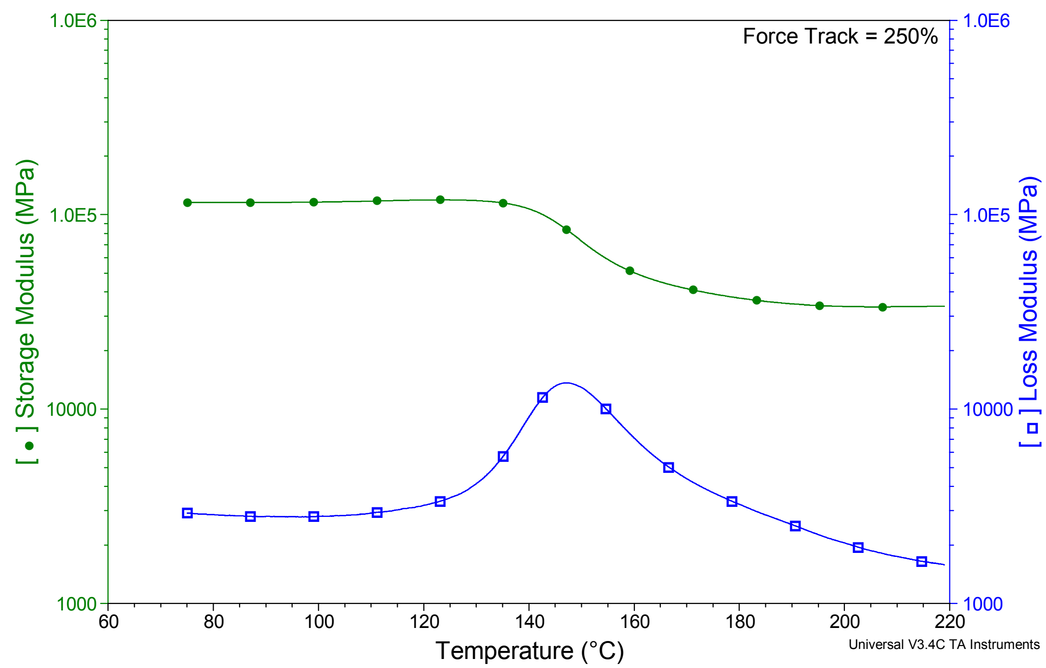

Figure 3 – Glass Transition with Poor Clamp Contacting and Large Force-Track

Based on the clamping and the variation in the degree of twist from sample to sample, several experiments may have to be performed to properly minimize the errors.

TA Instruments now offers low-friction 3-point bending clamp that eliminates these artifacts shown in Figure 4.

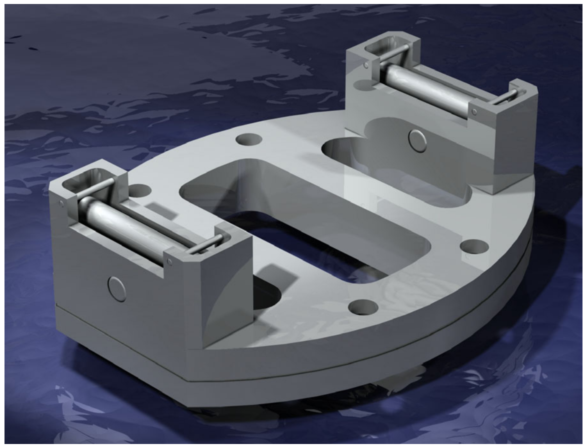

Figure 4 – Low Friction 3-Point Bending Clamp

This clamp does not require the movable shaft to be aligned to suit the twists in the sample. It automatically conforms to the shape of the sample, whether straight or with some degree of twist along its length because the support pivots along the sample width. Additionally, the supports are cylindrical rollers that provide friction free support of the sample.

EXPERIMENTAL

A stiff composite test specimen, 12.6 mm in width and 2.1 mm in thickness, is placed on the clamp taking care to ensure that it is parallel to the length axis of the clamp. The sample is heated at 1 °C/min to ensure that the 50 mm long sample achieves temperature equilibrium. As usual, the clamps are compliance calibrated. A low Force-Track value of 130 % is used along with the low friction, 3-point bending clamps.

RESULTS AND DISCUSSION

The results in Figure 5 show the absence of the increase in storage modulus when the new clamps are used. The modulus below the glass transition is higher than that when the regular clamp is used. This can be reasoned by the clamp's proper contact with the sample along its width. Figure 5 shows the comparison of the storage and loss moduli versus temperature. As expected, the storage modulus is higher for the sample run using the new low-friction clamps.

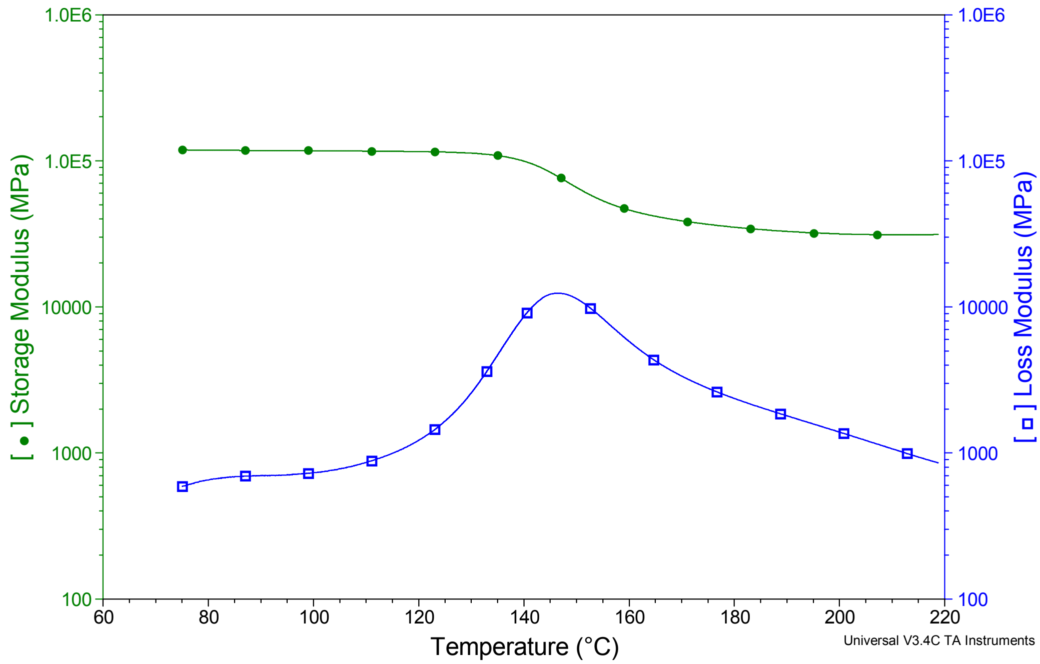

Figure 5 – Glass Transition with Low Friction Clamp and Low Force Track

Due to the artifact associated with inadequate contact, the glass transition temperature, taken here at the peak of the loss modulus curve, seems to be different in Figure 1 and 5. The T_g of the sample run with the regular clamp is artificially lower as seen in Figure 4 and overlain in Figure 6.

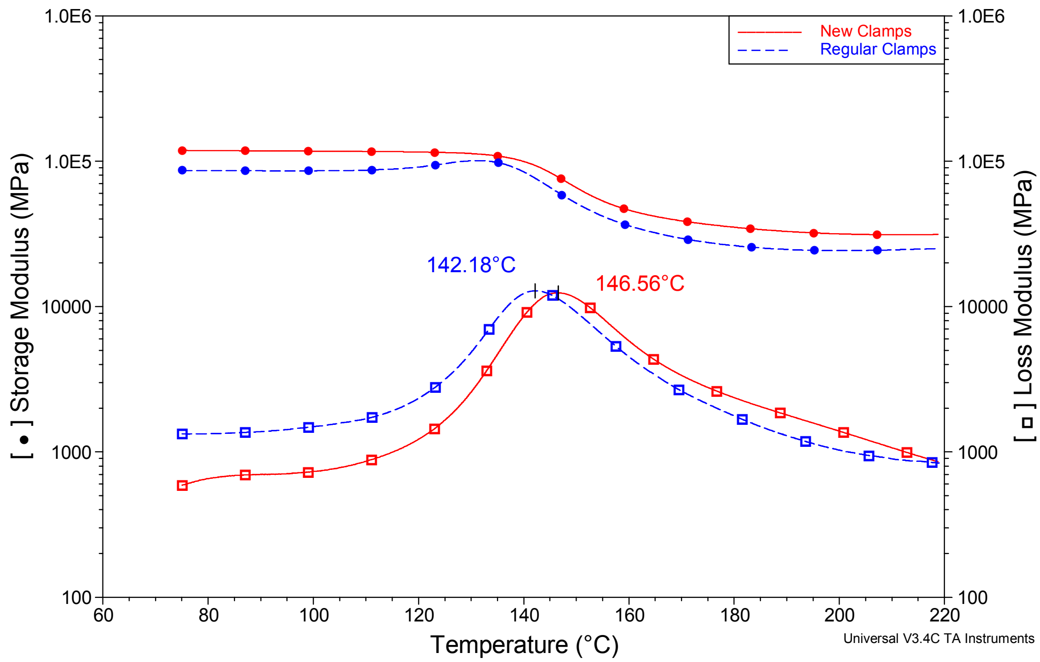

Figure 6 – Glass Transition Differences Between Traditional and Low Friction 3-Point Bending Clamps.

SUMMARY

The low-friction 3-point bending clamp is well suited for rigid samples and reduces the effects of poor contact between sample and support compensating for any small, subtle twists in the sample. This prevents incorrect storage modulus and glass transition temperature assignment to the sample. Nevertheless, the phenomenon of an increasing storage modulus before T_g makes the presence of twist known. Since the new clamps adjust automatically to the sample, the data obtained is free from the artifact that is usually observed.

KEYWORDS

dynamic mechanical analysis, glass transition, modulus