SII NanoTechnology Inc.

PREDICTION OF POLYMER DAMPING PROPERTIES BY DMA AND MASTER CURVES

Introduction

Dynamic mechanical analysis (DMA) is a powerful technique for the characterization of the viscoelastic properties of materials. DMA measures the modulus (stiffness) and damping (energy dissipation) properties of materials as they are deformed under periodic stress. These measurements provide quantitative information about the performance of materials. The technique can be used to evaluate a wide variety of materials such as thermoplastics, composites, thermosets, elastomers, films, fibers, coatings and adhesives.

Polymeric materials exhibit viscoelastic behavior which means that they simultaneously possess both solid-like as well as liquid-like characteristics. The degree to which the polymer exhibits more solid-like or liquid-like properties is dependent upon temperature as well as time or frequency.

With dynamic mechanical analysis, a sinusoidal force or stress is applied to a sample and the resulting sinusoidal deformation or strain is monitored. The sample strain response lags behind the input stress wave with respect to time and this lag is known as the phase angle, δ. The ratio of the dynamic stress to the dynamic strain yields the complex modulus, E*, which can be further broken down to yield the storage modulus, E', and the loss modulus, E".The storage modulus refers to the ability of a material to store energy and it is related to the stiffness of the material. The loss modulus represents the heat dissipated by the sample as a result of the material's given molecular motions and this reflects the damping characteristics of the polymer. Because of the viscoelastic nature of many materials, which includes all polymers, these mechanical properties are functions of temperature as well as time (frequency).

Benefits of Frequency Multiplexing DMA

One powerful means of analyzing polymers by DMA is through the use of frequency multiplexing experiments. In this approach, a sample is subjected to a number of different frequencies (generally 5 or more) in order to clearly define the effects of frequency, or time, on the mechanical responses exhibited by the polymer. In the past, the problems associated with data collection have necessitated that most DMA instruments operate in a slow isothermal step mode when performing frequency multiplexing experiments.

With the state of the art Seiko DMS6100, real time patented Fourier transform technology (U.S. patents 5287749 and 5452614) has been implemented which now makes it possible to perform frequency multiplexing experiments while dynamically heating even at relatively fast rates (e.g., 5°C/min). The Seiko DMS6100 has added a further technological advance with the introduction of Synthetic Oscillation (SO) measurements. In the SO mode, a complex stress sine wave is applied to the sample and this complex stress wave contains 5 simultaneous frequencies. The

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resulting complex strain and stress sine waves are deconvoluted using Fourier transform technology and compared to compute the quantitative viscoelastic properties. The SO different is different from standard frequency multiplexing in that, with the latter, one single discrete frequency is sequentially applied to the sample at a time. With the SO mode, the sample is exposed to all 5 frequencies in one instant.

The advantage of performing frequency multiplexing and Synthetic Oscillation DMA experiments is that much more informative sample characterization information can be generated. One example is the development and assessment of polymers for acoustical or mechanical dampening purposes. From DMA frequency multiplexing results, the effects of time as well as temperature for a material can be established through the well-known time - temperature superposition principle. This permits the generation of master curves by which the acoustical or vibrational dampening properties of a polymer can be estimated. This feature has been effectively utilized by the maker of a Japanese line of luxury automobiles where specific polymers and elastomers were selected using DMA and master curves in order to optimize the acoustical and vibrational dampening characteristics of the materials in different locations throughout the automobile.

The Seiko DMS6100 system features Rheo Master Curve software for the generation of these valuable and informative master curves. The software offers an automated best-fit procedure to determine the values of the WLF constants, C1 and C2, used to generate the master curves.

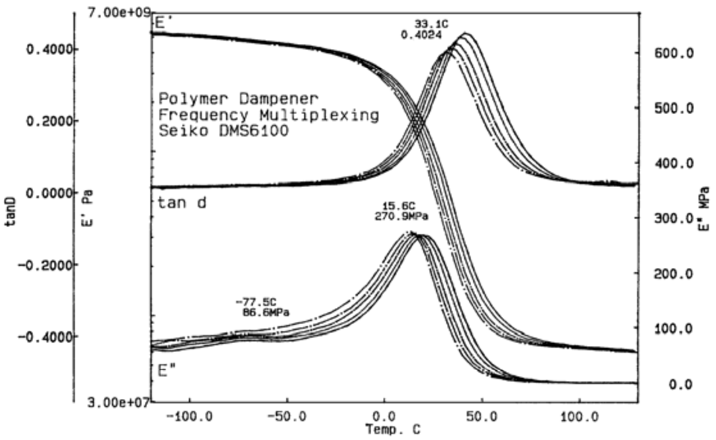

Figure 1

DMA Results on Polymer Vibrational Dampening Sample

An polymeric material, specially developed to dampen out vibrational energy, was evaluated using the Seiko DMS6100. The material was analyzed using the frequency multiplexing mode (0.5, 1, 2, 5 and 10 Hz) at a heating rate of 5°C/min between -120°C and 100°C. The polymer was handled using the single cantilever bending mode of deformation. The results generated by the Seiko DMS6100 on the polymeric dampening sample are displayed in Figure 1. This plot shows the log of the flexural storage modulus, E', the loss modulus, E", and tan delta (E"/E') as a

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function of temperature at the 5 different frequencies. At the glass transition event, in the region between -20 and 40°C, the polymer exhibits the classic shift in the E" and tan delta peak temperatures with respect to increasing frequency. For this polymer, the Tg occurs at 15.6°C based on the E" peak temperature at a frequency of 1.00 Hz. A very small secondary loss transition is observed at -78°C. The Seiko DMS6100 has the high degree of sensitivity required to detect this low energy, subambient transition.

This increase in the loss peak temperature with respect to the applied DMA frequency can be used to assess the apparent activation energy associated with the polymer. An Arrhenius plot can be produced in which the log of thefrequency is plotted against the inverse of the peak temperature. Over a limited frequency range, the response is linear and the slope of the best-fit line yields the apparent activation energy, Ea. The value of the activation energy provides information on crosslink densities of thermosets or elastomers and on the particular nature of the relaxation transition in terms of its assignment based on molecular rotations (α, β, or γ).

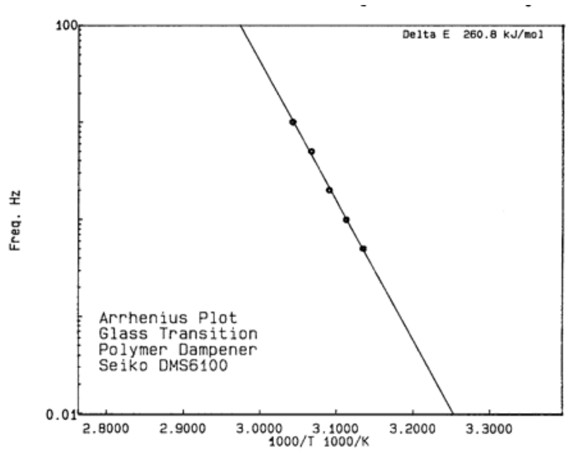

Figure 2

The Seiko DMS offers RheoDeltaE software which automatically computes the activation energy based on the Arrhenius approach. Displayed in Figure 2 is the Arrhenius plot for the glass transition event of the polymer dampening material. An activation energy of 261 kJ/mole is obtained for this sample. This activation energy assessment also permits the estimation of the material's peak temperature at a frequency which is outside the range of the DMA.

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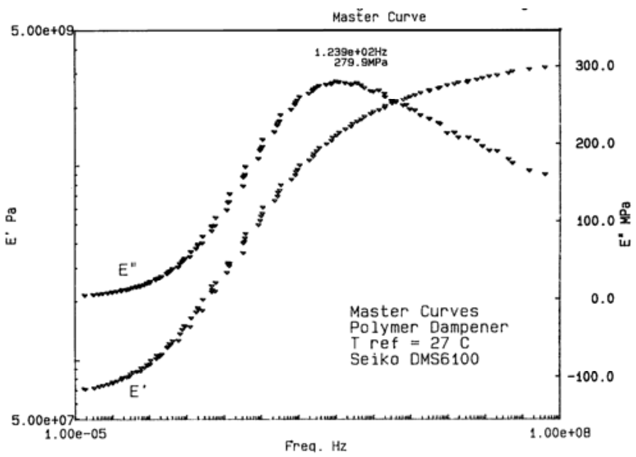

Figure 3

For acoustical or vibrational dampening purposes, master curves are very informative in estimating the frequency interval over which maximum energy absorption occurs. Displayed in Figure 3 are the E′ and E″ master curves generated for the polymer dampening sample. The curves were generated using an isothermal reference temperature of 27°C. The E″ master curve shows that the polymer dampening material is effective at absorbing energy over the desired frequency range (1 to 1000 Hz). The curve shows that maximum energy absorption and damping would occur at a frequency of 124 Hz at 27°C for this particular polymer. In this way, master curves can be used to evaluate different formulations or different polymers to determine the frequency range where maximum energy absorption takes place and thus 'tune' polymers for the effective damping of specific frequencies for specific engineering applications.

Summary

Dynamic mechanical analysis (DMA) is a valuable technique for the measurement of the viscoelastic properties of polymers. The information obtained from DMA can be significantly enhanced using frequency multiplexing experiments. Such experiments, easily performed with the Fourier transform technology associated with the Seiko DMS6100, permit the assessment of activation energy as well as the generation of master curves. From master curves, the frequency range over which polymers can effectively dampen acoustical or vibrational energy, can be assessed.

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