Applying dynamic mechanical analysis to research & development for viscoelastic damping materials

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ABSTRACT

Dynamic mechanical analysis (DMA) is a versatile method that can provide results for a wide array of parameters relating to material behaviour. In this paper it is used as a method to investigate and characterise the damping properties of viscoelastic materials. Investigations on polymer based viscoelastic damping material samples with varying formulations have been conducted using DMA. Results have been presented and discussed, highlighting the efficiency, accuracy and resolution of the DMA method. The differences in product formulation across the samples and the effect on material characteristics has also been discussed. The results supplied using the DMA method have been shown to be beneficial to research and development for viscoelastic damping materials.

Keywords: Damping, Viscoelastic, Vibration I-INCE Classification of Subjects Numbers: 47, 72.7

1. INTRODUCTION

Reducing vibration in a system has many benefits ranging from decreasing noise output to increasing component life. If vibrations cannot be controlled through isolation or by modifying the mass and/or stiffness characteristics, then damping is a viable solution. System damping can be effectively improved by applying a material with high damping characteristics in contact with the vibrating components. These materials primarily achieve damping through their viscoelastic characteristics, allowing the mechanical energy to be dissipated into heat during deformation of the material (1). Hence, it is of interest for researchers to understand how the viscoelastic properties of different materials compare.

Commonly, damping is measured as loss factor (η) which is a measure of energy loss per cycle of deformation. It can be useful to measure the loss factor at a range of temperatures and frequencies, not only as the loss factor of the material is dependent on these variables, but also because these conditions can vary substantially depending on the vibration problem. Loss factor can be determined for viscoelastic materials through a multitude of test methods. One such test is the Oberst beam method, which involves applying a damping system to a beam, providing excitation with an electromagnet, and measuring the response with an accelerometer (2,3). The distinct difference between the Oberst and DMA method is that DMA will directly test the material properties while the Oberst beam method can only test the system properties. Whilst methods such as the Oberst beam can provide useful data, they can require considerable time and labour to collect data over a wide temperature range. Furthermore, measurement is limited to the resonance frequencies of the test beam. Collecting data on viscoelastic damping materials is where DMA can be a fast, efficient and accurate tool.

A selection of sample viscoelastic damping materials were chosen to analyse using DMA. All materials were polymer based with various fillers. The test procedure and results were analysed to evaluate the use of DMA for research and development purposes.

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2. BACKGROUND

2.1 Viscoelastic Damping Materials

Viscoelasticity is when a material exhibits both viscous and elastic characteristics during deformation. Therefore it will have a time-dependent strain, as the behaviour of the material not only depends on the load, but also on the history of the load (4,5).

The damping mechanism behind viscoelastic materials is through the dissipation of mechanical energy into heat due to the internal interactions among the molecules during deformation, or vibration in particular (1).

Different polymers have a different range of useful temperatures. These can be broken into three regions: the glassy region, the transition region and the rubbery region as illustrated in Figure 1, below. The low end of the temperature range is the glassy region, where the polymer will exhibit glasslike properties. The modulus (E') is higher here and the loss factor (tan δ) is low. At higher temperatures is the rubbery region, where the polymer decreases in modulus to the point of becoming rubbery and loss factor lessens. The transition region is the region in between; within this region is where the loss factor peaks at the glass transition (T_g). To achieve the desired transition region a lot of research and development time is spent in order to maximise loss factor within the required temperature range, and achieve adequate damping for the given application (6,7). These viscoelastic materials can be designed to be effective at particular temperature ranges through the way the product has been formulated and processed.

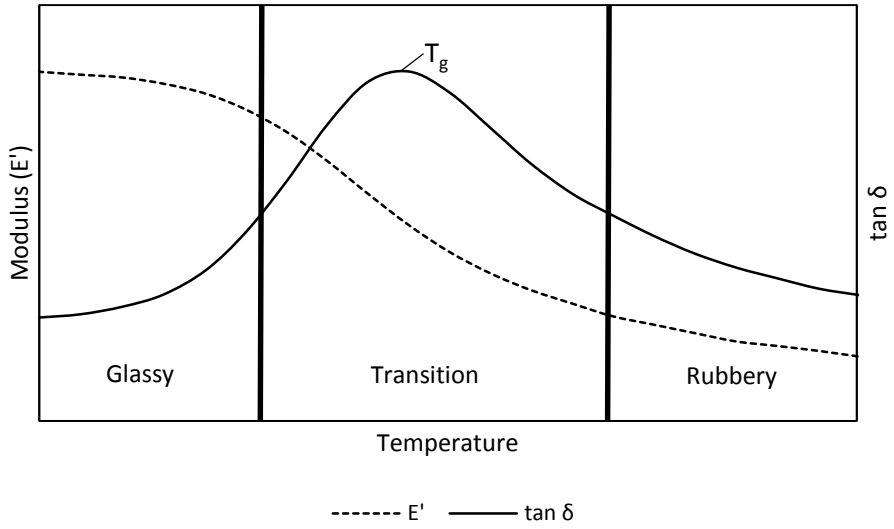

Figure 1 – Damping (tan δ) and modulus (E') as a function of temperature with the three regions marked

Vibrational frequency also has an effect on the behaviour of viscoelastic materials and the response has similarities to temperature. At lower frequencies the material has time to respond and the viscous characteristic dominates the material response. High frequencies have the opposite effect and the elastic characteristic dominates (8). Like temperature, there is an optimal frequency range where loss factor peaks. As the formulation of the viscoelastic material can change the viscoelastic response, materials can be designed to offer maximum loss factor given specific parameters for temperature and frequency.

2.2 DMA Application

DMA (dynamic mechanical analysis) or DMTA (dynamic mechanical thermal analysis) is a powerful tool used to study material phase transitions and the response to mechanical and thermal stress. This is particularly useful for polymers and, in this case, viscoelastic materials.

DMA machines work under the concept of applying a force to a material and analysing the material's response to that force (a non-resonance method). The force used in this case is sinusoidal and can be oscillated at a range of frequencies, typically 0.1 Hz – 200 Hz, and across a range of temperatures, typically -150 °C – 600 °C (-238 °F – 1112 °F). From analysing this response, the DMA

can calculate various properties from the recorded dynamic modulus. In research and development of viscoelastic materials we are primarily interested in measuring and comparing the elastic modulus (E′ - the ability to return energy) and the loss modulus (E″ - the ability to lose energy). The ratio of these two parameters – the tan delta (tan δ) - is a useful result for damping material evaluation when used as a function of temperature (8):

$$\tan\delta = \frac{E''}{E'}$$

The sensitivity, resolution and precision of the DMA method is a primary benefit, allowing data on material behaviour to be collected with high detail. Furthermore, the ability to analyse a material across a temperature and frequency range without any user input allows a material to be tested to a defined program without supervision. A huge number of data points can be generated with a high degree of sensitivity as measurements can be made at increments of less than a degree. The speed at which this data can be recorded can be highly efficient, especially in comparison to methods such as the Oberst beam. A major contributor to the efficiency is through the ability to modulate the temperature rapidly. The small sample size reduces the thermal mass and therefore reaches thermal equilibrium faster than larger samples used in other methods. In comparison, the Oberst beam requires a minimum soak time of 30 minutes when testing according to ASTM E756-05(10) (3).

3. EXPERIMENTAL

3.1 DMA Setup

The DMA used to collect this data was a Rheometric Scientific DMTA-3E. The testing procedure was based on ISO 6721-5:1996 (9). Calibration was conducted before beginning the test batch and the surrounding ambient conditions were kept stable and consistent, including isolation from external vibration. Liquid nitrogen fed from a pressurised dewar and the internal oven were used to modulate the test chamber temperature, with a filtered pressure and flow controlled air system for convection. Samples were cooled to 0 °C (32 °F) before clamping to minimise deformation. Samples were mounted in a dual cantilever configuration for suitability with the modulus range.

Table 1 – DMA parameters

Parameter	Value
Test type	Dynamic temperature step
Sample mounting	Dual cantilever
Clamping torque	20 cNm (0.1 ft-lb)
Sample length	20.0 mm (787 mil)
Sample width	12.0 mm (472 mil)
Sample thickness	2.9 mm +/- 0.1 mm (114 +/- 4 mil)
Frequency	1 Hz
Temperature range	5 °C – 55 °C (41 °F – 131 °F)
Temperature increment	2.5 °C
Soak time	5 minutes
Air convection flow	50 cm^3/min (0.002 ft^3/min)
Air convection pressure	10 kPa (1.5 psi)

Stress (σ) and strain (ε) are determined according to force and displacement measurements by the machine, multiplied by constants that reflect the sample geometry and mounting method. In this example, the stress constant (K_σ) for dual cantilever bending on the Rheometric Scientific DMTA-3E is calculated by:

$$K_\sigma = \frac{3l}{4wt^2} G_C$$

While the strain constant (K_ε) is calculated by:

$$K_\varepsilon = \frac{12t}{l^2}$$

Where $w =$ sample width (m)
$t =$ sample thickness (m)
$l =$ sample length (m)
$G_C = 98.07$ (gravitational constant) (10)

Figure 2 & 3 – The DMA test chamber and system during the calibration process

3.2 Samples

A variety of polymer based prototype viscoelastic materials were chosen to test. In order to see how the DMA can detect changes due to formulation and/or processing, the samples varied from each other only in one of:

- Type and particle size of the filler
- Polymer content
- Plasticiser content
- Processing method (laboratory and production samples).

Samples were cut with identical dimensions taken randomly from large sample pieces. Slight variations in thickness between individual samples are accounted for by the DMA calculation as all dimensions are entered into the unit's computer program. Thickness was measured with a micrometre accurate to 0.001 mm (0.04 mil) and other dimensions with callipers accurate to 0.05 mm (2 mil).

4. RESULTS

4.1 Filler Type

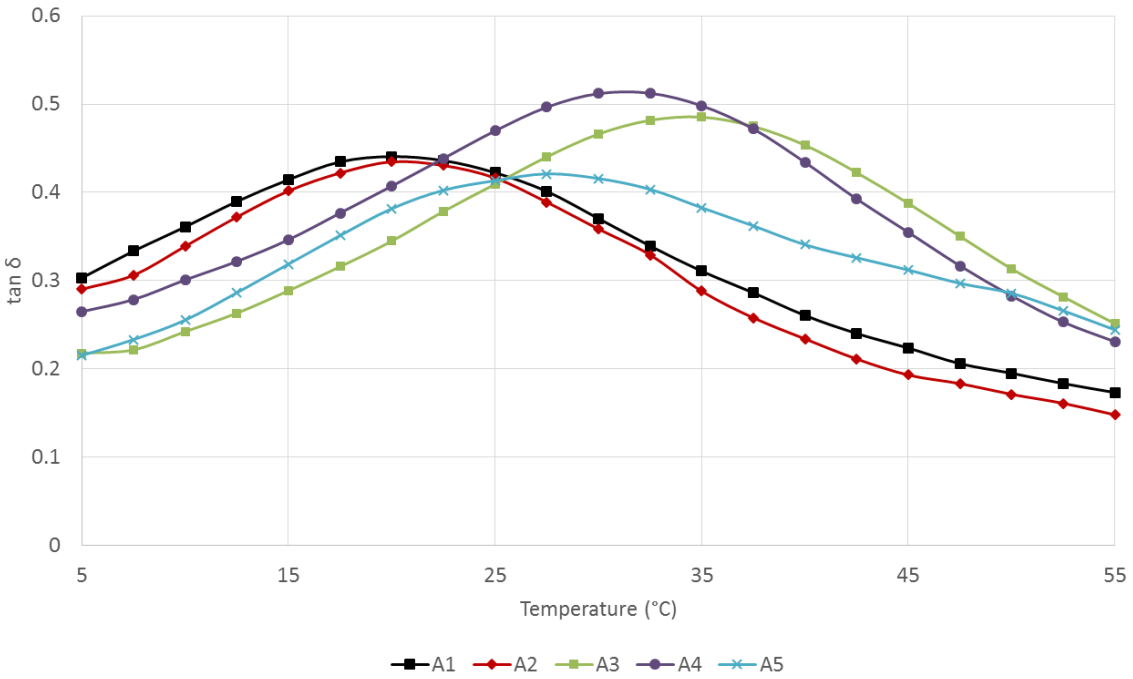

Figure 4 – DMA results for viscoelastic materials with different fillers

Table 2 – 'A' Samples

Sample	Change
A1	Control prototype
A2	Change of filler (small particle size)
A3	Change of filler (intermediate particle size)
A4	Change of filler (large particle size)
A5	Blended fillers

Investigation 4.1 applied no changes to the formulation other than completely changing the type of filler. A2 substituted the type of filler with a small particle size, whilst A3 substituted an intermediate filler and A4 substituted a larger particle size filler. A5 substituted multiple fillers which were blended together.

The DMA machine recorded clear differences in viscoelastic response between the five samples. Sample A1 and A2 favourably held their transition region around ambient temperatures, with similar tan δ. Changing the filler type achieved a slight decrease in this case. Sample A3 and A4 had their transition regions at higher than ambient temperatures and with a relatively high tan δ. Larger particle size had a noticeable effect in this case. Sample A5 showed a significant change due to the combination of fillers, the transition region widened and the T_g had a middling value.

4.2 Polymer Content

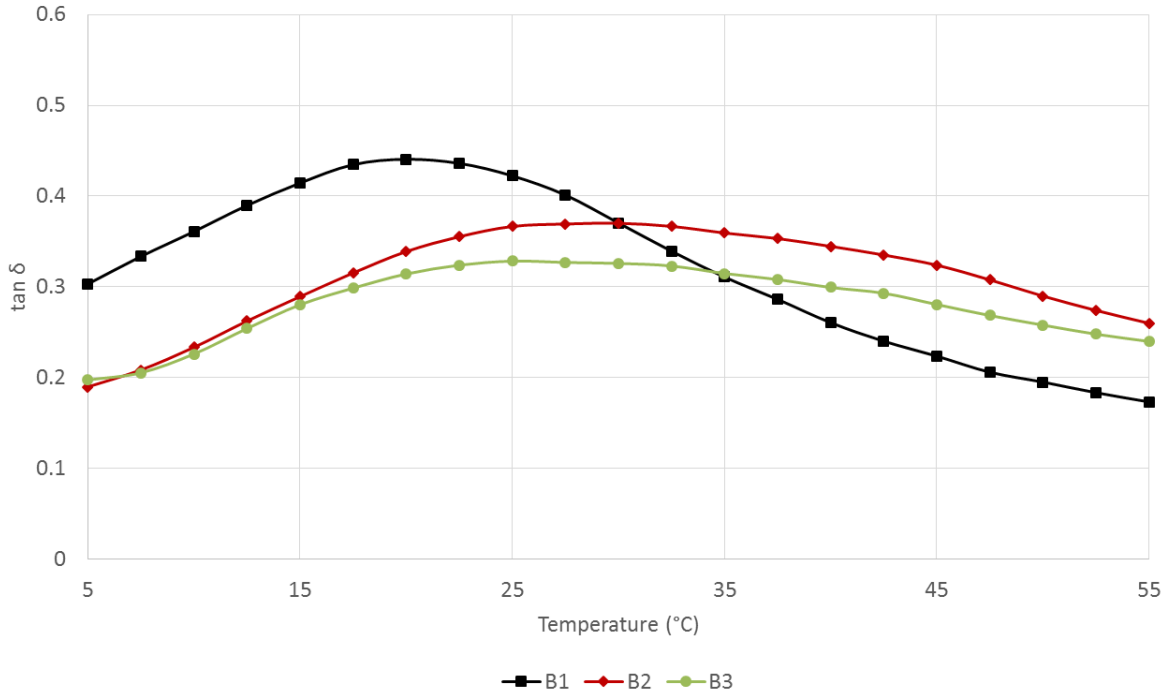

Figure 5 – DMA results for viscoelastic materials with different polymer content

Table 3 – 'B' Samples

Sample	Change
B1	Control prototype
B2	5% less polymer
B3	10% less polymer

Investigation 4.2 applied no formulation changes apart from a reduction in polymer. Sample B2 had a reduced polymer content by 5% and B3 had a reduced polymer content by 10%.

The DMA machine recorded shifts in tan δ between the three samples. Control sample B1 had a peak tan δ value at ambient temperatures. Sample B2 showed a substantial reduction in tan δ, as well as a shift of the transition region to higher temperatures. Sample B3 continued to show a reduction in tan δ as the polymer content was further reduced. This could be explained in terms of the increasing elastic modulus of samples with a lower polymer content. Warmer temperatures are required to soften the material to its transitional phase and the reduction of polymer may also reduce the overall loss modulus as well, hence the lower peak.

4.3 Plasticiser Content

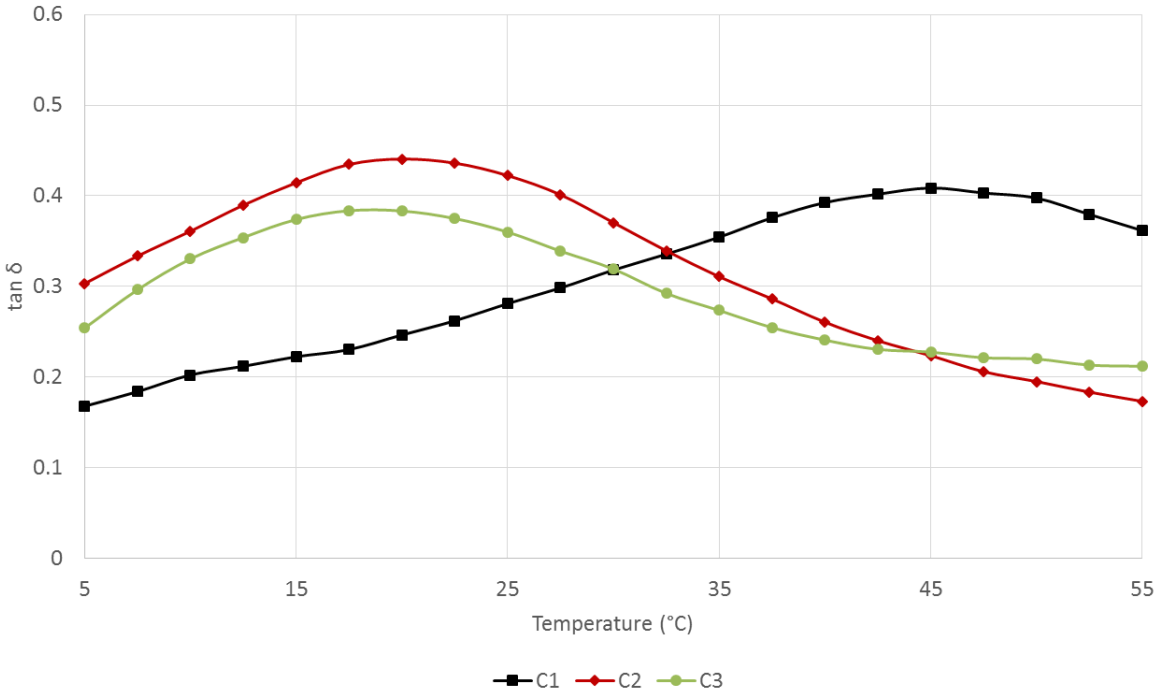

Figure 6 – DMA results for viscoelastic materials with different plasticiser content

Table 4 – 'C' Samples

Sample	Change
C1	Control prototype
C2	5% more plasticiser
C3	10% more plasticiser

Investigation 4.3 applied no formulation changes apart from the increased plasticiser content. Sample C2 increased plasticiser content by 5% and sample C3 increased plasticiser content by 10%.

As with investigation 4.2, changes to the formula which affect the modulus of the material result in substantial shifts in the optimal temperature and overall damping properties. Control sample C1 had a tan δ value that peaked at a relatively elevated temperature. The addition of extra plasticiser in sample C2 softened the damping material and had the desired effect of shifting the transition region down to ambient temperatures, with no major change in tan δ. Sample C3 further reduced the transition temperature with the additional plasticiser, however tan δ was noticeably reduced reflecting that the material had become too soft.

4.4 Laboratory and Production Sample Processing

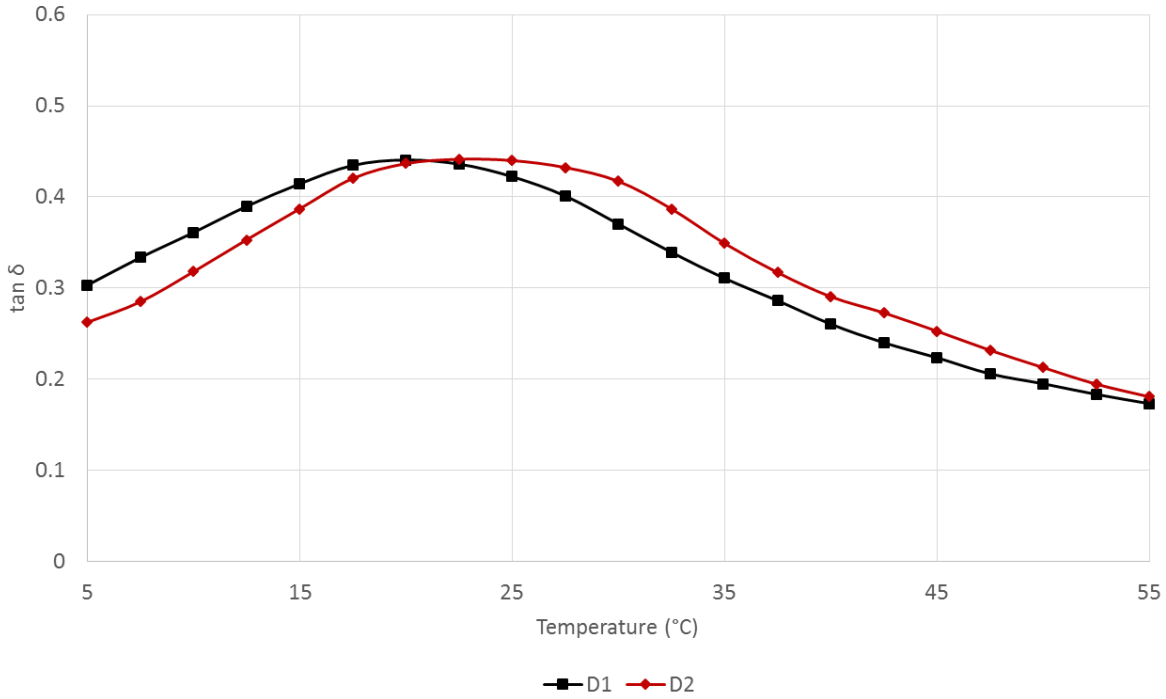

Figure 7 – DMA results for viscoelastic materials with different sample processing methods

Table 5 – 'D' Samples

Sample	Change
D1	Lab sample
D2	Production sample

Investigation 4.4 applied no formulation changes, only a change in the processing method. Sample D1 was processed in a laboratory and sample D2 was processed in a full scale production environment.

The DMA machine detected minor differences between the two samples. The production sample recorded a slight shift in the transition region to higher temperatures. No significant difference was recorded in tan δ. These minor differences may be due to a multitude of factors such as: mixing efficiency and packing efficiency of filler particles, crushing of particulate fillers under mixing stress, or product cooling profile. This relates to the range of challenges of upscaling from product development to production and the DMA's potential in evaluating the final product throughout this transition.

5. DISCUSSION

DMA was able to return precise results to distinguish samples with changes to the filler, polymer content, plasticiser content and differences in sample processing. The DMA can also be useful for quality assurance or quality control purposes because of its ability to identify minor variations in material composition and processing. Test time per sample investigation was approximately 100 minutes across the full temperature range, showing the efficiency of the DMA method. To test these samples using the Oberst beam method would take extensively longer, not only in testing but in sample preparation time. Considering a 30 minute soak time per temperature step when testing to ASTM E756-05(2010) (3), testing would take approximately 600 minutes per sample. Sample preparation time under the Oberst beam method is also slower due to the need to laminate or adhere the product to a test beam. When comparing multiple materials, this is a vast improvement in test efficiency.

Over the course of testing, it was found that significant differences in results could be achieved by seemingly minor variations in the sample preparation or testing procedure. There are many variables to consider. Sample dimensions must be precise and consistent across the entire sample piece, as any inaccuracies are exaggerated by the calculations used by the DMA machine. The geometry of the sample should be carefully chosen to suit the fixture and clamps, without deviating from the recommended dimension ratios generally supplied in the manufacturer's literature. One concern with filled damping products is that the fillers – inert particles that can reduce cost and add weight – can be relatively large compared to the sample thickness. Particles sized >100 µm represent enough of a proportion of the overall sample thickness that it is foreseeable that this might contribute a degree of error. Likewise the production method used (in this case a comparison between laboratory and production samples) may lead to physical inconsistencies in the sample. One issue then for DMA is the limitation of only testing small sample pieces. Conversely, small samples are easier and quicker to heat, allowing heating rates of 20 °C per minute or more.

The sample mounting process, in particular the clamping system, technique and torque, must be carefully selected and carried out. Otherwise undue stress may be placed upon the sample and cause inaccuracies in results. Other problems can arise from tension changes on the sample as the temperature affects the material softness, therefore it can be best to clamp the sample at the beginning test temperature. The temperature range must not exceed the working range to avoid issues such as melting. The temperature ramp rate must be adequate for all within the test chamber to reach thermal equilibrium, including the fixture and clamps. Furthermore, the strain exerted onto the sample must not be high enough to damage the sample or push the response into the nonlinear stress-strain region. These issues can be addressed by careful selection of test parameters that are relevant for the material under investigation.

Although a dual cantilever fixture was selected for this test, samples can also be mounted in single cantilever, three-point bend, shear sandwich, compression, tension or submersible fixtures. Selecting the fixture depends on the material properties and the intended material application under investigation. Different classes of damping materials are better suited to particular test fixtures, and it is intended to broaden the scope of this research by investigating as wide a range of damping products as possible. For example, constrained layer damping systems are ideally tested in the shear sandwich fixture. Furthermore, depending on the DMA system, humidity can also be modulated. The effect of humidity on a material can be valuable to know for particular applications as it can affect modulus similarly to temperature (8). Further testing would be required to understand how humidity affects particular formulations. Also, future investigations could test a larger sample size to obtain a greater resolution between formulation changes.

One advanced use of DMA data is in combining test data across a temperature range at various frequencies. This can be done using a technique known as time-temperature superposition (TTS) which uses the proportionality of tan δ results at different temperatures to compile data onto a merged frequency axis. This allows a theoretical extension of the tan δ curve to be read at any frequency. Loss factor can be predicted for any given value for temperature and frequency including frequencies beyond the testing limits of the DMA. Modern DMA systems can perform a multi-frequency, multi-temperature test in a single operation. The results of these ongoing investigations will provide even further ability to assess damping material performance. (11)

Due to the vast array of variables and the variety of techniques different DMA systems can use to calculate material properties, it can be hypothesised that there is a level of ambiguity when comparing results from different DMA systems. This may mean that an identical material may return a different result from one DMA system to another. This does not detract from the ability to compare multiple

samples using one DMA system, but from the reproducibility among separate DMA systems. Further testing is required between separate DMA systems with identical materials to further investigate this hypothesis.

6. CONCLUSIONS

An example of applying dynamic mechanical analysis to research and development for viscoelastic damping materials has been presented. A set of viscoelastic damping material samples have been tested using DMA, and the results have been presented and discussed. The results show how DMA can assist in the optimisation of viscoelastic damping material formulation in terms of filler, polymer and plasticiser ratios for damping performance at a given temperature range. Testing a wider range of formulations, fixtures and parameters would lead to a stronger understanding of the DMA method and of the way in which material formulation and processing can influence the performance of a sample. In this regard, DMA can be seen as a useful member of the research and development toolkit for viscoelastic damping material characterisation.

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