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# Predicting Practical Properties of Unfilled and Filled Adhesives From Thermomechanical Data

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## Abstract

This paper presents research that was conducted to investigate the interpretation of glass transition temperature in the context of conservation. The results of thermomechanical and bulk mechanical properties of Araldite EA1253 epoxy and Resin W™ poly(vinyl acetate) (PVAc) are reported. Both adhesives are used in painting conservation and represent two generic types used within conservation. The adhesives were also tested with the addition of fillers.

The thermomechanical techniques employed were Dynamic Mechanical Analysis (DMA) and Differential Scanning Calorimetry (DSC). Frequency dependency, sample configuration, and moisture sensitivity were investigated. Bulk tensile testing was performed at temperatures on either side of the glass transition onset, calculated from DSC data. Comparisons were made between data from DMA, DSC, and tensile testing.

The results highlight the inherent ambiguities in the interpretation of thermomechanical data (in particular, a difference of at least 15°C between the onset of the change in DMA storage modulus and the ductile/brittle transition compared to the Tan delta peak value), and the implications this has for conservation treatments. The use of the data to predict the physical properties of the unfilled and filled adhesive is demonstrated. It is suggested that a more consistent and sophisticated use of thermomechanical data would be of benefit to conservation practice.

## Titre et Résumé

### Utilisation de données thermomécaniques des adhésifs chargés et non chargés pour prévoir leurs propriétés pratiques

Le présent article comprend les résultats de travaux de recherche portant sur l'interprétation de la température de transition vitreuse dans le domaine de la restauration. Les résultats de la détermination des propriétés thermomécaniques et de propriétés mécaniques globales de l'adhésif époxyde Araldite EA1253 et de la résine à base de poly(acétate de vinyle) [PVAc] Resin W<sup>MC</sup> sont présentés. Les deux adhésifs sont utilisés en restauration des peintures et ils constituent des types d'adhésifs couramment employés en restauration. Les essais ont aussi été réalisés sur des échantillons d'adhésifs auxquels ont été ajoutées des matières de charge.

Les techniques thermomécaniques utilisées sont l'analyse mécanique dynamique (AMD) et l'analyse calorimétrique différentielle (ACD). Les paramètres étudiés comprennent la dépendance relative à la fréquence, la configuration de l'échantillon et la sensibilité à l'humidité. Des essais de traction totale ont été réalisés à des températures immédiatement inférieures et supérieures à celle du début de la transition vitreuse, à partir de calculs basés sur les données d'ACD. Les données d'AMD et d'ACD et les résultats des essais de traction sont comparés.

Les résultats mettent en évidence les ambiguïtés intrinsèques de l'interprétation des données thermomécaniques (en particulier une différence d'au moins 15 °C entre la température du début de la variation du module de conservation, en AMD, et de la transition ductile-fragile, comparativement à la valeur maximale de « tan-delta »), ainsi que les implications de ce phénomène lors de l'exécution de traitements de restauration. Des résultats particuliers serviront à démontrer que les données peuvent être utilisées pour prévoir les propriétés physiques d'un adhésif chargé et non chargé. Il est recommandé d'adopter une approche plus uniforme et perfectionnée en matière d'utilisation des données thermomécaniques, ce qui aurait des incidences avantageuses sur les pratiques de restauration.

## Introduction

If the glass transition temperature ( $T_g$ ) is known for a material it is possible, to some degree, to predict the physical properties such as stiffness, flexibility and fracture mode, of the material under different conditions. This is important for objects undergoing external forces: vibration and shock, tensioning and handling. It also enables a prediction of internal forces that may affect the structural integrity of an object, including creep and the flexing of joints in response to changing relative humidity (RH).  $T_g$  values also give a good indication of surface related properties such as dirt pick-up, surface texture and degree of gloss. However,  $T_g$  data does not inform a conservation scientist or conservator precisely what physical behaviour to expect and how this might relate to damage of an object. A useful approach, to establish how materials will behave under stress, is prediction of the ductile/brittle fracture transition. A fracture can be said to be ductile when the material under load undergoes some degree of plastic (permanent) deformation before finally breaking. In the case of brittle fracture very little or no plastic deformation occurs. Brittle fracture results in less predictable, high energy failure, because the energy stored in the crack tip is released fast. This also results in rapid crack propagation and usually greater damage. During ductile failure the energy is absorbed, crack propagation is slower, its direction is more predictable, and less damage occurs (Moore 2003). An important property for an adhesive is the degree of fracture toughness which quantifies the ability of a material to prevent crack propagation. An adhesive with low fracture toughness is more likely to lead to damage of the surrounding object, especially if the object itself is brittle and also has low fracture toughness.

This paper presents part of continuing research into the potential and limitations of thermomechanical data (including  $T_g$ ), obtained from Differential Scanning Calorimetry (DSC) and Dynamic Mechanical Analysis (DMA), in the context of conservation. Both techniques are explained in Appendix 1. It is well established within polymer mechanics that the glass transition temperature ( $T_g$ ) is dependent on the thermomechanical technique used and interpretation of the data (Chartoff 1997). The numerical difference this makes has also been demonstrated for a subset of resins used in conservation (Schilling 1989). This is because of the viscoelastic nature of the materials under investigation rather than an error in methodology. However, this can lead to very different predictions of the physical properties of the materials and hence, the correct choice of adhesive for a conservation treatment.

Thermomechanical and static uniaxial tensile data are compared for two adhesives to establish the relationship between  $T_g$  and fracture properties, and the most appropriate method of measurement. Thermomechanical analysis is also used to demonstrate how the data aids the

modification and potential design of adhesives, for conservation applications, with the example of the addition of filler to an adhesive.

## Samples

The adhesive and fillers chosen are used in panel painting conservation and represent two generic types used within the profession (Young 2011). A crystalline two-part epoxy adhesive pre-filled with silica, Ciba Geigy Araldite 1253, and an amorphous poly(vinyl acetate) (PVAC) adhesive, Bostik Findley Interior Wood Glue Resin W. The Resin W is modified with addition of a microballoon and coconut shell flour, this mixture is used as a gap filling adhesive for panel paintings (Young 2001). The Araldite 1253 was modified with the addition of microballoons to investigate the possibility of increasing its fracture toughness (see Table 1).

Free films of the epoxy (test code EA1253), modified epoxy (test code EA1253Mi), PVAC (test code RW), and filled PVAC (test code RWCoMi) were draw down onto flexible polytetrafluoroethene (PTFE) sheets taped to rigid boards (see Table 1). They were made in a controlled environment (relative humidity (RH) 50% RH  $\pm$  2% RH and a temperature of 20°C  $\pm$  2°C) then left uncovered in UV filtered light for one week to allow volatile materials to evaporate. Subsequently, the samples were placed in air tight opaque boxes with silica gel matting (Artsorb) conditioned to 55% RH and stored in ambient conditions (typically 55% RH  $\pm$  10% RH, 20°C  $\pm$  4°C). Once a week the boxes were opened to allow any further volatile components to escape. The films were left for a minimum of 6 weeks to ensure that they had reached their dry film thickness. To prevent any pre-strain, the flexible PTFE was peeled away from the sample, rather than peeling the sample film. All test samples were cut from the same films and were nominally 6mm wide, 0.4 mm thick (PVAC) and 1.0 mm thick (epoxy), with a gauge length of 5mm for both the DMA and static uniaxial tensile testing. Prior to testing, all the dimensions were measured with Vernier calipers and a micrometer.

Table 1 Materials Tested

Adhesive	Code	Composition
Bostik Findley Resin W UK interior wood glue	<b>RW</b>	Poly Vinyl Acetate
2 part (mixed 1:1) Araldite 1253HV: Araldite1253SV Carvable wood	<b>EA1253</b>	Epoxy : phenolic resin.
<b>Filler Material</b>		
Phenolic microballoons.	Mi	Spherical balloons of 50 microns diameter.
Coconut shell flour	Co	Ground and sieved coconut husks.
Filler in Araldite 1253HV	-	Silica/ titanium dioxide/ iron oxide
<b>Modified adhesives</b>		

Filled Resin W	<b>RWCoMi</b>	Filler mix 1:1 (wt:wt) of coconut flour and phenolic microballoons. 1g of filler mix added to 5ml of adhesive.
Modified Epoxy	<b>EA1253Mi</b>	1g of phenolic microballoons added to 5ml of epoxy.

## Static Uniaxial Tensile Testing

Static uniaxial tensile testing was carried out on an Instron 4301 with integrated environmental chamber. The load, extension, RH and temperature were logged throughout the testing. Samples were clamped in the upper grip and aligned, then allowed to come into equilibrium at the desired conditions (typically 20 minutes) before tightening the bottom grip. A crosshead speed of 5mm/min was used because this is between the average speeds of the DMA test runs 1Hz (1.2 mm/min) and 10 Hz (12 mm/min). It also represents a speed at which induced strains might occur on works of art (not including shock). A minimum of three samples were tested at each test temperature and RH (see Table 2).

## DSC (Differential Scanning Calorimetry) and DMA (Dynamic Mechanical Analysis)

Thermomechanical testing was carried out at the Getty Conservation Centre, Los Angeles. Three of each sample type were tested for both DSC and DMA measurements. Measurements with the Mettler Toledo DSC 822 followed a method, developed by Schilling (Schilling 1989), for the measurement of polymers such as Paraloid B72 (see Appendix 1). After baking out the DSC, a blank pan was run to provide a baseline measurement from which the thermal characteristics of the test materials could be extracted. Nitrogen was used as the purge gas, discs were cut from the free films and placed in the sample pan ensuring good thermal contact. The typical weight of the sample was 2-4 mg.

Dynamic thermomechanical analysis was carried out on a Triton 2000 DMA. In dynamic mode the instrument can operate in a variety of configurations - tension, shear, cantilever bend, and three point bend (Appendix 1). Most operating parameters are user defined. Preliminary testing on acrylic and alkyd paint films had allowed pre-selection of the best combination of parameters (configuration/ frequency/heating rate/sample dimension/preload) to obtain meaningful results. All samples were run on the DSC first to obtain a Tg value to ensure that the DMA temperature scan covered this region. Where possible, tests were performed in all modes.

## Results

Table 2 summaries the results for all the testing undertaken.

## Comparison of DMA configurations

Figures 1a&b show the DMA storage modulus  $E'$ , loss modulus  $E''$ , and tan delta curves for EA1253 measured in single cantilever mode and tension mode. Storage modulus is related to the stiffness of the material, while loss modulus reflects the damping capacity (viscous nature) of the material. Tan delta is the ratio  $E''/E'$ , thus a material with a high tan delta is more viscous.

These moduli are simply related to Young's modulus  $E$  (calculated from the initial linear gradient of the stress/strain curve as measured using static uniaxial tensile testing) by  $E^2 = (E')^2 + (E'')^2$ . The peak of the Tan delta ( $\delta$ ) curve is often taken a value for  $T_g$  ( $64.5^\circ\text{C}$ ). However, the storage modulus starts to drop significantly from 388 MPa at  $47^\circ\text{C}$ , falling to 12 MPa at  $77^\circ\text{C}$ . Thus the stiffness starts to fall  $17.5^\circ\text{C}$  below the  $\text{Tan}\delta$  value. The temperature and value of the  $\text{Tan}\delta$  peak was measured as  $64.5^\circ\text{C} / 0.56$ , and  $58.2^\circ\text{C} / 0.49$ , in cantilever and tension mode respectively. In comparison, the DSC  $T_g$  onset value was  $43.8^\circ\text{C}$ . Also, a greater change in storage modulus occurred in cantilever than in tension mode. DMA testing configuration had a significant effect on moduli values and the position of the  $\text{Tan}\delta$  peak for all samples tested.

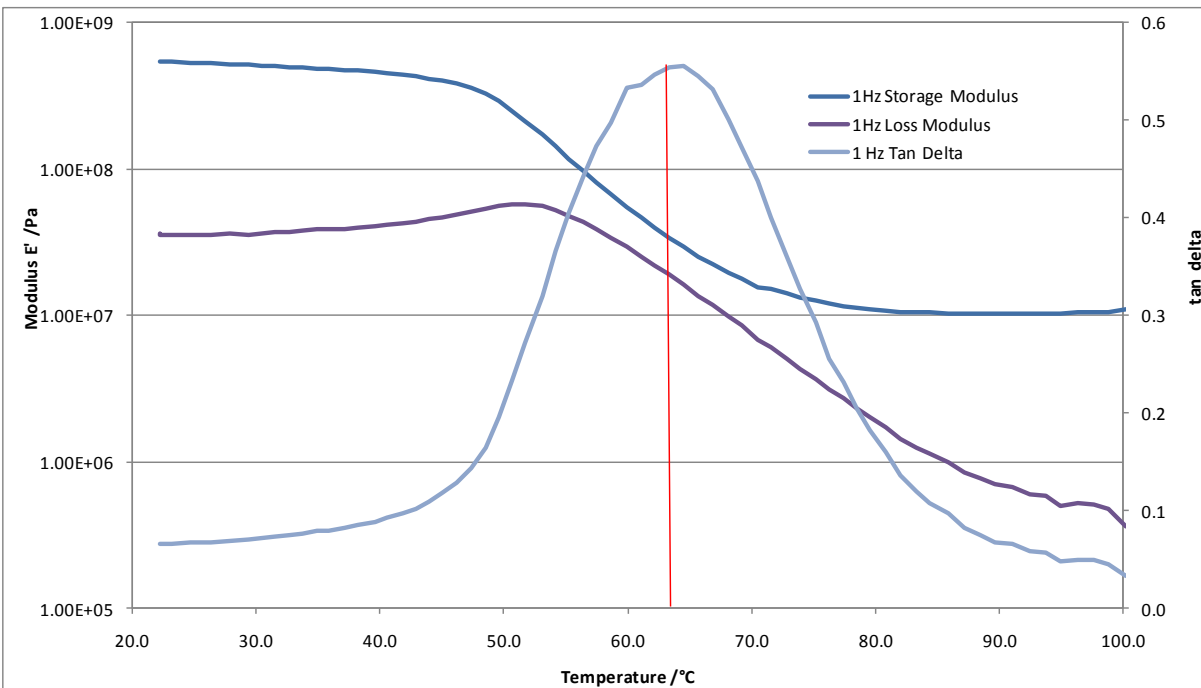


Figure 1a DMA curves for EA1253 at 1Hz in single cantilever mode.

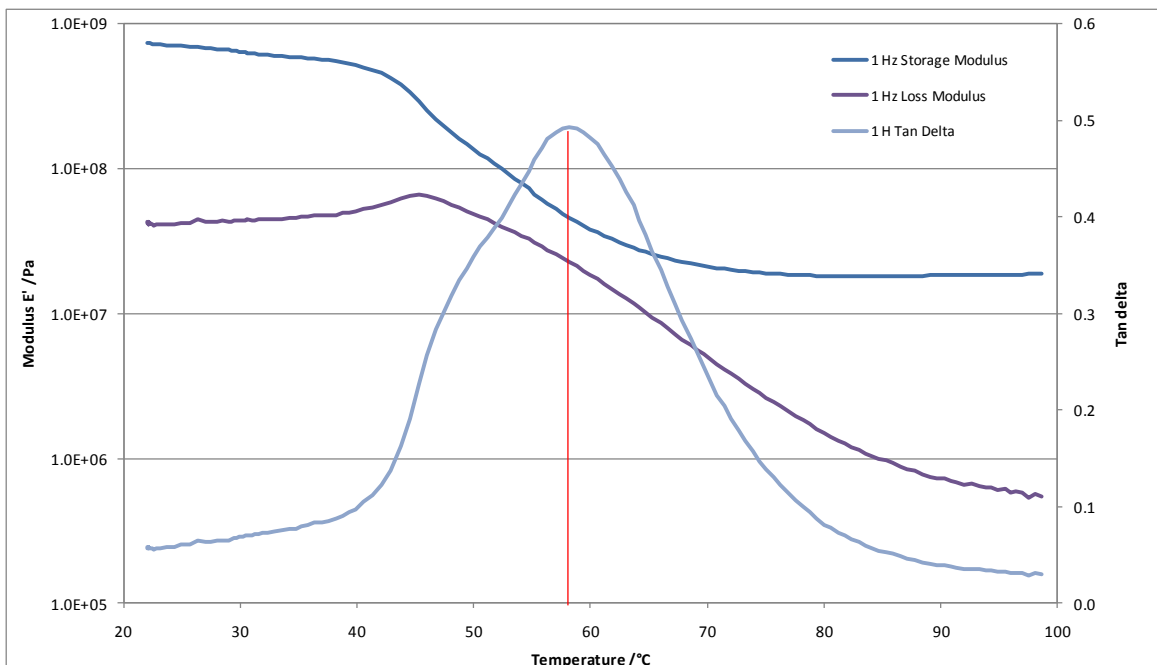


Figure 1b DMA curves for EA1253 at 1Hz in tension mode.

In practice, the stiffness and dimensions of the sample determine the test mode (Duncan 2008). The best mode depends on the relative stiffness of the DMA spring coil and the sample and it is paramount for obtaining reliable and meaningful data. For the typical stiffness of paint and adhesives used for transportable cultural heritage (paintings/paper/objects), stiffness values lie in a region that is most suitable for single cantilever and tension modes (Erlebacher 1992; Grattan 1998; Williams 1978; Young 2001; Young 2008). Single cantilever is probably the most common mode for data published in the conservation literature. This is partly because it is the easiest to perform, especially on brittle samples. However, it is more sensitive to experimental error because small errors in dimensional measurement lead to large errors ( $\pm 40\%$ ) in modulus determination. Tension mode is less sensitive to error as the dimension sensitivity is  $\pm 11\%$  (Young 2011b). If the  $\text{Tan}\delta$  is used for  $T_g$  this dimensional error is not a factor.

### Reproducibility

To establish the experimental reproducibility of DMA data, identical samples of EA1253 were tested in the same mode and conditions and the standard deviation ( $\sigma$ ) calculated (see Table 2). In tension mode the average  $\text{Tan}\delta$  value was  $57.8^\circ\text{C}$  ( $\sigma \pm 0.6^\circ\text{C}$ ) and storage modulus in the glassy region  $715 \text{ MPa}$  ( $\sigma \pm 86 \text{ MPa}$ ).

### Frequency dependency

The shift towards higher temperatures with increasing test frequency for the storage modulus, loss modulus and tan delta curves has been discussed in many publications. For example, Figure 2 shows that for EA1253 the shift in the  $\text{Tan}\delta$  peak is from  $58.2^\circ\text{C}$  to  $63.7^\circ\text{C}$  between 1 Hz and

10 Hz. This is a result of the strain rate dependency of viscoelastic polymers, and thus it is important that comparisons in  $T_g$  between materials are made at the same frequency.

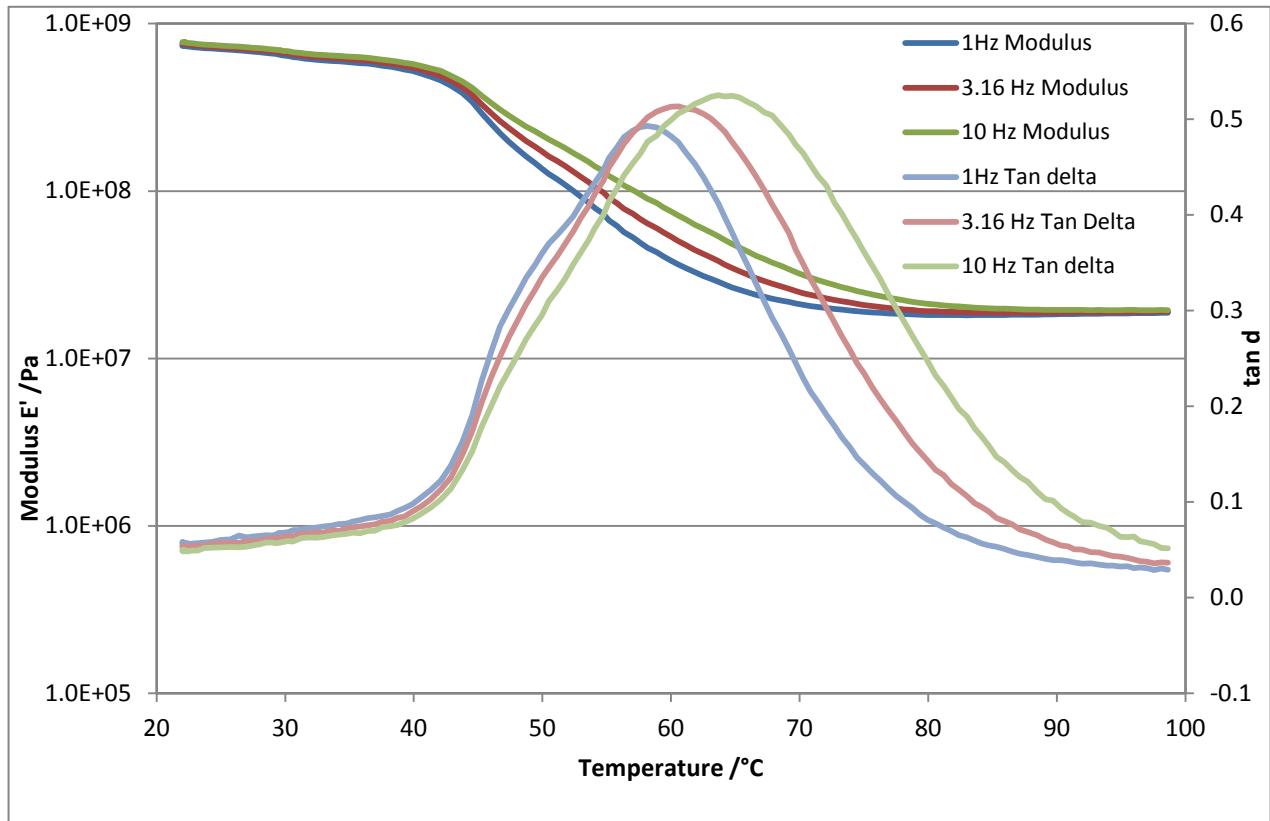


Figure 2 Shift in DMA  $Tan\delta$  and storage modulus for EA1253 in tension mode with test frequency.

### Static uniaxial tensile testing

Figure 3 shows typical stress vs. strain curves for the filled PVAC (RwCoMi) at 10°C 53% RH, 10°C 68% RH and 20°C 55% RH (see Table 2). At 10°C, 53% RH there is no measurable plastic deformation before failure at 3% strain (brittle fracture) whereas at 20°C 55% RH there is a very large plastic deformation, after the linear elastic region, before failure initiates at 26% strain (ductile fracture). Thus the transition between brittle and ductile fracture is occurring between 10°C and 20°C. The failure strain (strain at which fracture occurs) rather than the yield strain (strain at which plastic deformation starts to occur) has been taken for quantitative comparison because, for viscoelastic materials, the yield point is not unequivocally discernible from non-linear elastic behaviour.



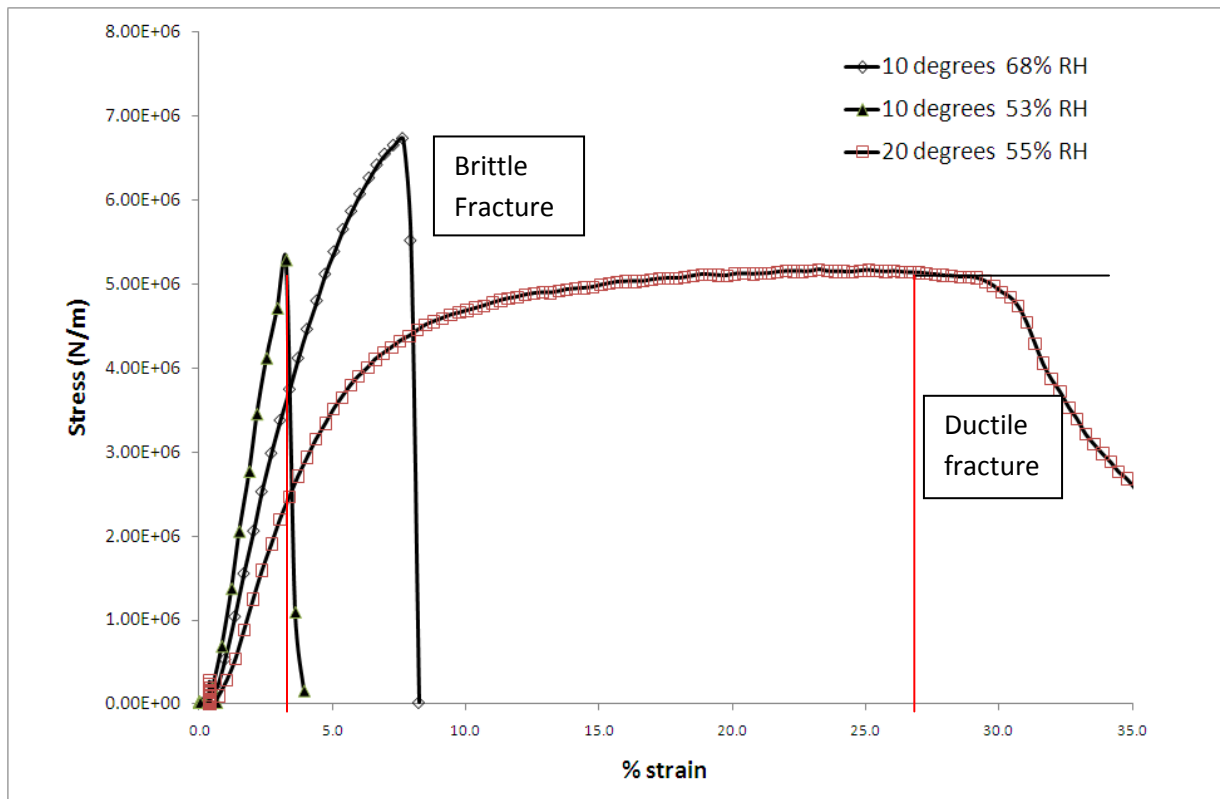


Figure 3 Tensile stress vs. strain curves filled PVAC (RWC0Mi)

The effect of moisture content on the type of fracture can be seen from the 10°C 68% RH curve which shows some plastic deformation before failure at 7.6% strain. The higher moisture content is sufficient to act as a plasticiser in the polymer. However, this 15% difference in RH at 10°C results in a less significant effect compared to the 10°C temperature change which causes much greater mobilisation of the molecular structure. The bar chart in Figure 4 summarises the results for all the samples tested. It can be seen that although there is a large variation within each set of conditions, there is a clear trend in behaviour between the samples at 10°C and 20°C, with an increase in strain to failure (average values went from 3.2% to 21.7%), there is also a corresponding reduction in stress at failure (average values went from 171 MPa to 110 MPa). Without tests at intermediate temperatures one can only predict that the transition from brittle to ductile fracture occurs in the region of 10°C to 20°C. From the DMA data, the onset in the change in modulus from glassy to leathery is 17°C (Tan $\delta$  peak 33.7°C), and from DSC, T $g$  is at 23.2°C. Thus the DSC data correlates closely with the brittle to ductile transition as measured by static tensile testing.

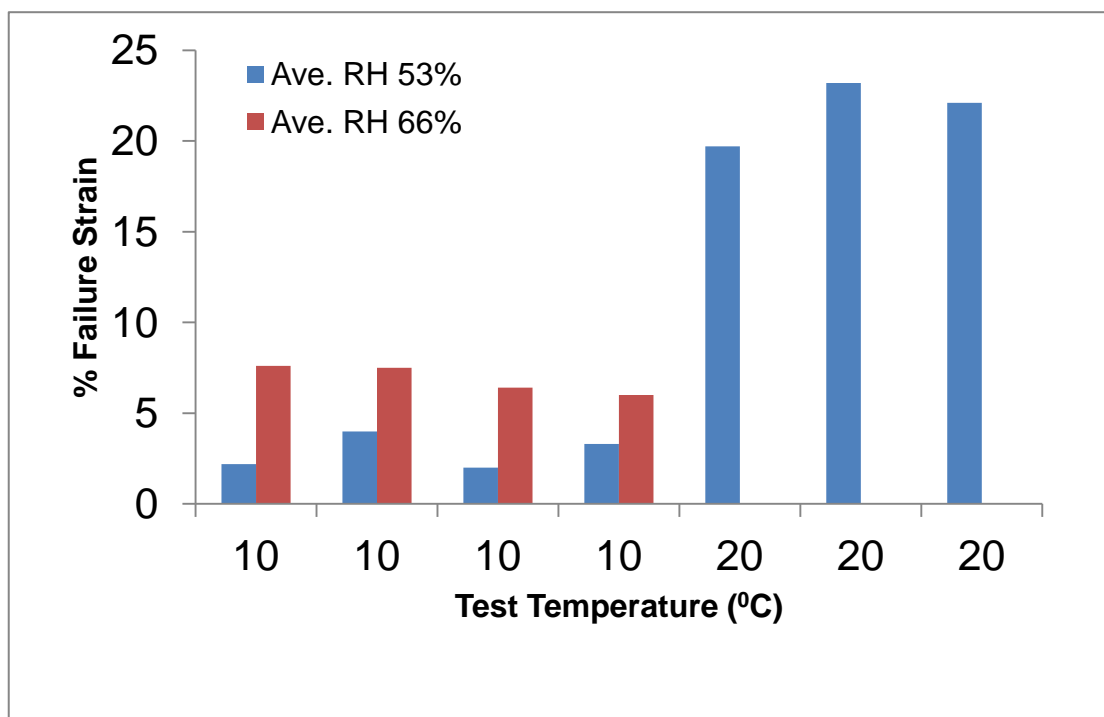


Figure 4 Summary of % Failure Strains for filled PVAC (RwCoMi) with temperature (RH at which the tests were performed has been averaged for each temperature range).

Figure 5 shows the typical stress vs. strain curves for the epoxy (EA1253) at 10°C, 20°C, 30°C, 40°C, 50°C and 60°C (see Table 2 for complete results). From 20°C to 50°C failure occurs at similar strains (7%-11%) although there is evidence of increasing plasticity especially at 50°C, where the changes suggest an alteration in properties, even if the fracture is relatively brittle. At 60°C there is a very large plastic deformation after the linear elastic region before failure at 30% strain (ductile fracture). The tensile data suggests that the transition between brittle and ductile fracture occurs between 40°C to 50°C. From the DMA data, the onset in the change in modulus occurs around 42°C (Tanδ peak 57.8°C), from DSC the T<sub>g</sub> is at 43.8°C. It is worth noting that the manufacturers quoted T<sub>g</sub> is 65°C.

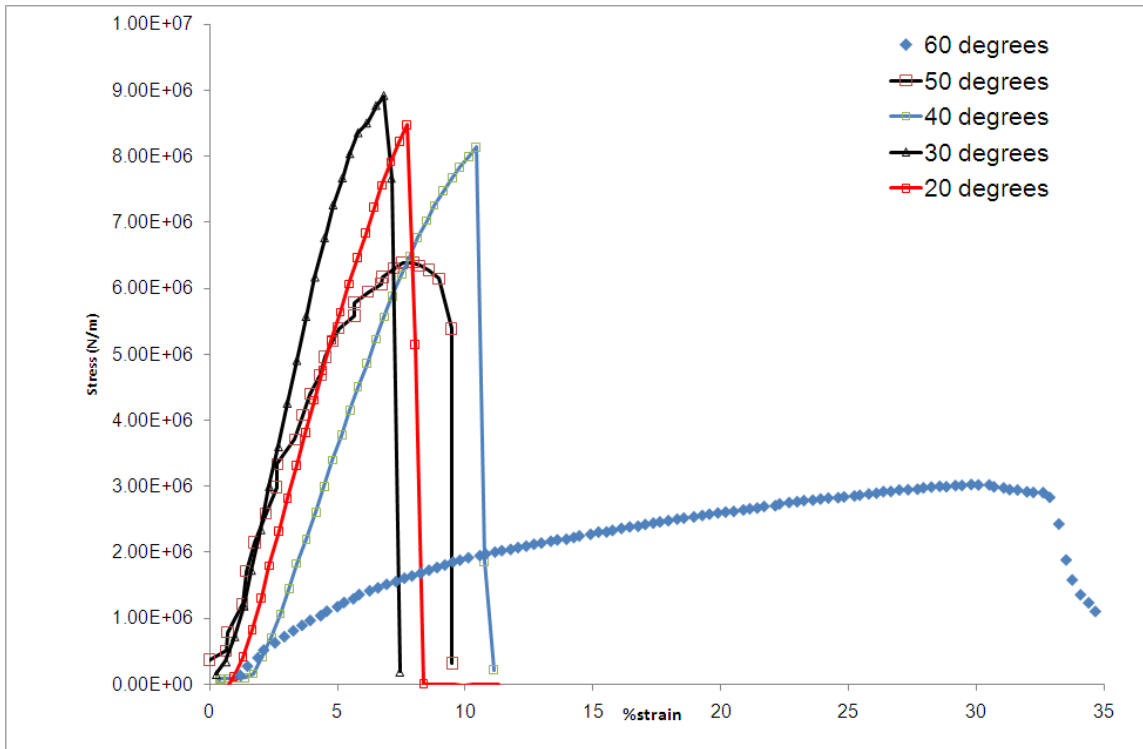


Figure 5 Tensile stress vs. strain curves for filled epoxy (EA1253)

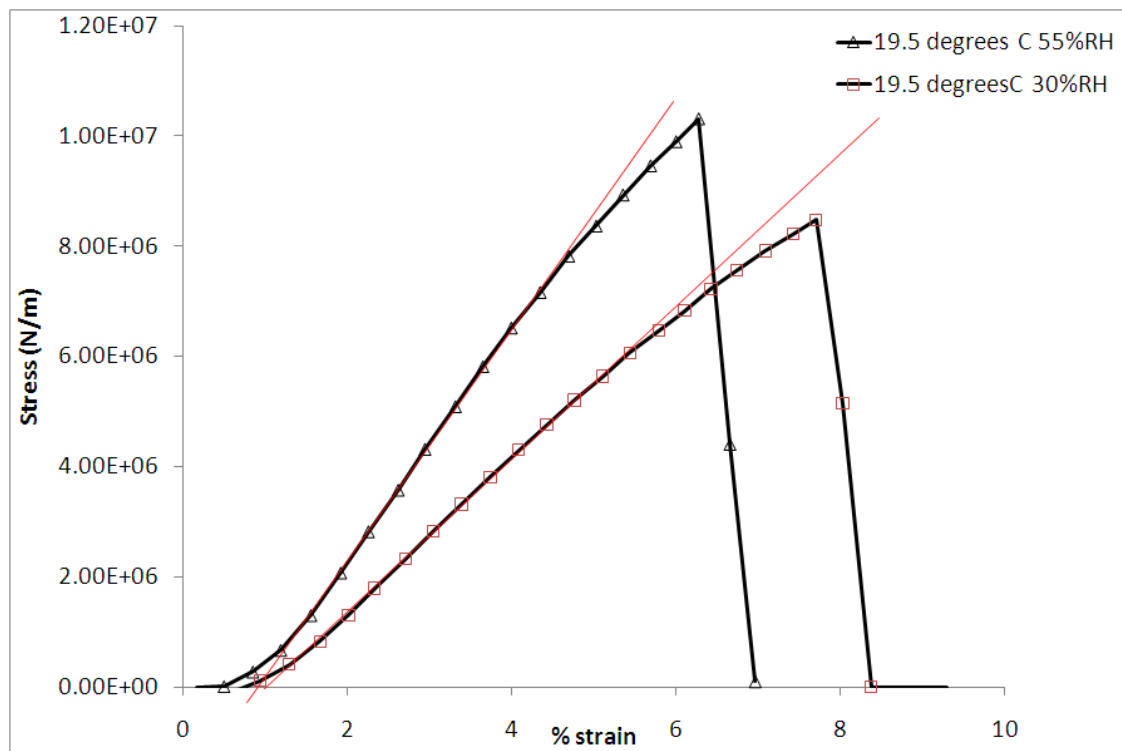


Figure 6 Tensile Stress vs. strain curve for epoxy (EA1253) at different RH.

Experimentally it was not possible to maintain the same RH for each test temperatures. Although the effect is not as pronounced as for PVAC, moisture is known to change the mechanical properties of epoxy (Foster 2004). Figure 6 shows the stress vs. strain curves in the glassy region at 20°C/30% RH and 20°C /55% RH. At 30% RH there is slight reduction in plastic deformation after the linear elastic region. The epoxy is, as expected, more brittle at the lower RH. Figure 7 summarises the strain to failure for all EA1253 tests. It can be seen that there is a consistent reduction in strain to failure (average value 7.3%) to (average value 4.1%) from 55% RH to 30% RH, respectively. This is a less significant effect when compared to the change in properties by a 10°C temperature change between 50°C and 60°C.

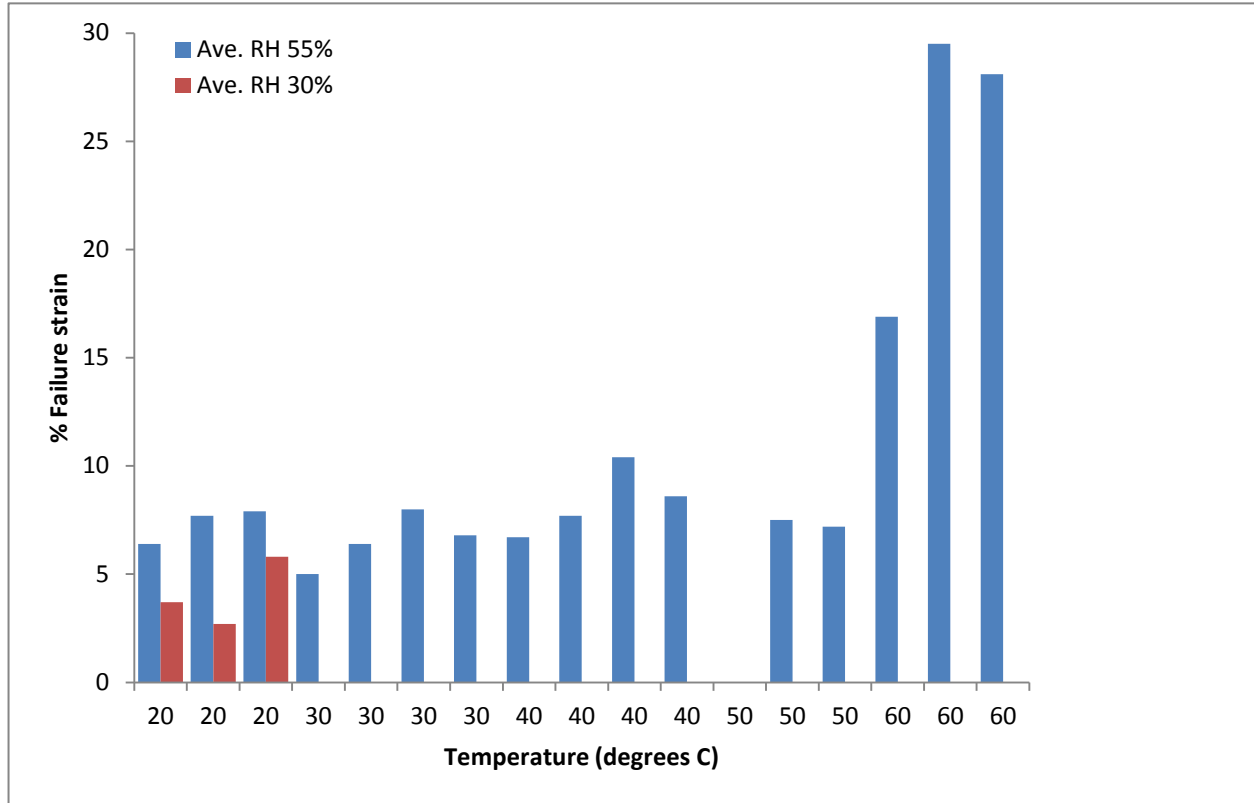


Figure 7 Summary of % Failure strain for epoxy (EA1253) at different test temperatures, and at two RH at 20°C (RH given is the average value).

## Using DMA for predicting bulk properties

### *Additional filler in EA1253*

The shape of the modulus curve for EA1253 (see Figure 1a) across the transition region is typical of a crystalline polymer (Chartoff et al. 2009), with a relatively small transition in modulus between 47°C and 77°C. The shoulder on the left side of the Tanδ curve (see Figure 1b) around 45-55°C is also typical of a filled crystalline polymer. Addition of microballoons alters the shape of all three curves (see Figure 8). Calculating the average values for each set of curves one sees a very small shift in properties: the Tanδ peak shifts from 57.8°C to 59.8°C and decreases in value from 0.46 to 0.41, respectively. Noticeable features are the greater variation

between samples (probably because of variations in the amount of additional microballoons manually mixed in), a reduction in the change of the modulus across the transition, and broadening of the loss modulus and  $\text{Tan}\delta$  curves. These features are all consistent with the addition of a filler to a crystalline polymer (Chartoff et al. 2009). The broadening of the  $\text{Tan}\delta$  curve is also important as it implies a high modulus of resilience (Chartoff 1977) e.g. better fracture toughness and thus a better ability to prevent crack propagation and brittle fracture. The broadening of the  $\text{Tan}\delta$  curve can be quantified by calculating the relative change in its width to its height, known as the full width half maximum (FWHM), see Appendix 2. Comparison of the FWHM of the  $\text{Tan}\delta$  curve at 1Hz shows that there is broadening from  $26.0^\circ\text{C} \pm 1.6^\circ\text{C}$  to  $29.2^\circ\text{C} \pm 3.6^\circ\text{C}$ . A similar trend is found when comparing the results obtained from the DMA in single cantilever mode. The  $\text{Tan}\delta$  peak decreases from 0.56 for EA1253 to 0.46 for EA1253Mi at  $64.5^\circ\text{C}$  and  $65.4^\circ\text{C}$ , respectively. The FWHM also increases from  $24.1^\circ\text{C}$  to  $27.4^\circ\text{C}$ . However, the storage modulus decreases from  $7.2 \times 10^8$  Pa to  $4.0 \times 10^8$  Pa (measured at  $30^\circ\text{C}$  in the glassy region). In this case the EA1253 is already filled with silica, the addition of the microballoons to a point where the commercial filler was at the limit of a workable paste has probably reduced its cohesive properties. Thus by comparing FWHM and the storage modulus one can measure how much filler can be added to an adhesive to increase fracture toughness while still retaining its cohesive properties.

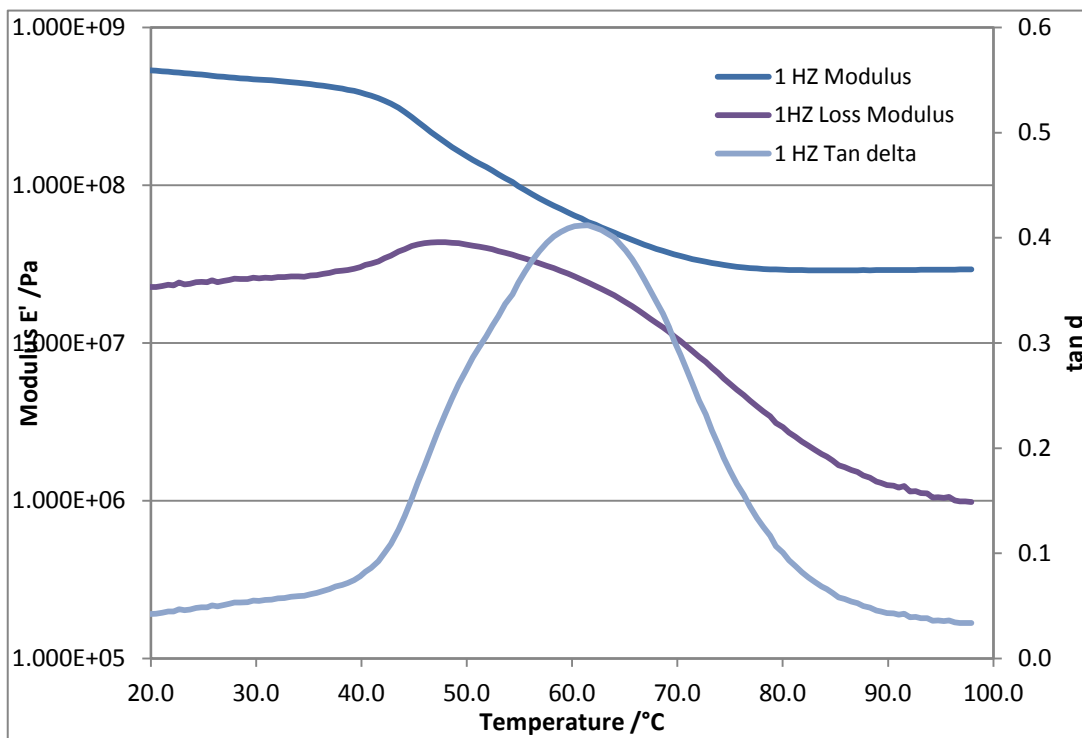


Figure 8 DMA curves for EA1253Mi at 1Hz in tension mode.

#### Addition of filler to PVAC (Resin W)

The thermomechanical properties for the unfilled and filled PVAC were measured using DMA. Although DMA in tension mode reduces the error in the determination of the modulus, it was found experimentally that the modulus for this PVAC dropped below the measurable range of the instrument in tension mode, and therefore, single cantilever mode was used for both the unfilled and filled samples. Typically for an amorphous polymer, unfilled PVAC exhibited a large drop in modulus (greater than two orders of magnitude) through the transition. The  $\text{Tan}\delta$  peak was at  $31.6^{\circ}\text{C}$  and has a value of 2.43. The transition in storage modulus started at  $10^{\circ}\text{C}$  and finished at  $30^{\circ}\text{C}$ . Addition of the filler reduced the modulus from 530 MPa to 180 MPa at  $10^{\circ}\text{C}$ , and reduced the range of the transition to one order of magnitude, with a corresponding slight increase in the temperature of the  $\text{Tan}\delta$  peak, a reduction in its height from 2.43 to 0.5, and an increase in FWHM from 38.8 to 40.5.

## Discussion and Conclusions

DMA dynamic modes measure the material in its elastic region, across the transition from glassy to leathery behaviour. The transition from brittle to ductile failure occurs when there is some plastic deformation. Nevertheless the ductile/brittle transition relates to a molecular configuration of the polymer and correlates with thermomechanical data.

Duncan has shown (Duncan 2008) that determination of the  $T_g$ , from the different methods of analysing DMA curves, leads to consistent trends with DMA  $\text{Tan}\delta$  giving the highest value and onset in reduction of the storage modulus the lowest value. For all the samples tested in this research the DSC onset was commensurate with the onset in the storage modulus reduction.

EA1253 was shown to be stable over time with respect to both natural and thermal ageing (Young et al. 2011). RwCoMi exhibits an increase in uniaxial tensile strength (UTS) and stiffness with time when tested under four point bending (Young et al. 2011). Thus the difference in the time between undertaking thermomechanical and tensile tests complicates the comparison. However, the features discussed in this paper have been found for a range of other adhesives and paints (Young 2011, Young 2008).

Thus, DSC provides a much easier and reliable method of predicting how an adhesive or polymer might behave in an object. It also requires much smaller sample sizes and very much shorter experimental time. For the materials tested the DMA onset in the change in the storage modulus at 1Hz correlates with the ductile/brittle transition.  $T_g$  derived using  $\text{Tan}\delta$  is at least  $15^{\circ}\text{C}$  higher than the onset storage modulus value.

There is a difference in the position and value of the  $\text{Tan}\delta$  peak for each DMA mode. Using the  $\text{Tan}\delta$  peak does provide a repeatable point of reference for measuring  $T_g$  because it does not include the dimensions of the samples. However, its value is always higher than that derived from the transition onset measured by the storage modulus which relates directly to the transition in stiffness of the material. Practically the prediction of adhesive properties needs to take this in account .i.e. an adhesive will become softer or start to creep at a lower temperature than expected.

Although T<sub>g</sub>'s are weaker events and less discernable from DSC data than DMA, the reproducibility and accuracy of the raw data is better. The transition has been easily discernible for a variety of PVAC, epoxy and acrylic, paints and adhesives tested. Calculation of the onset of the glass transition from the DSC curve gives a glass transition value closest to that of the onset measured from the DMA storage modulus curve. For consistency it is suggested that DSC should be used (as in many industrial applications) for comparison across materials.

DMA is useful for predicting fracture toughness and stiffness changes in a filled adhesive; information that cannot be obtained from DSC. DMA provides a powerful method for investigating the properties of viscoelastic polymers encountered in conservation.

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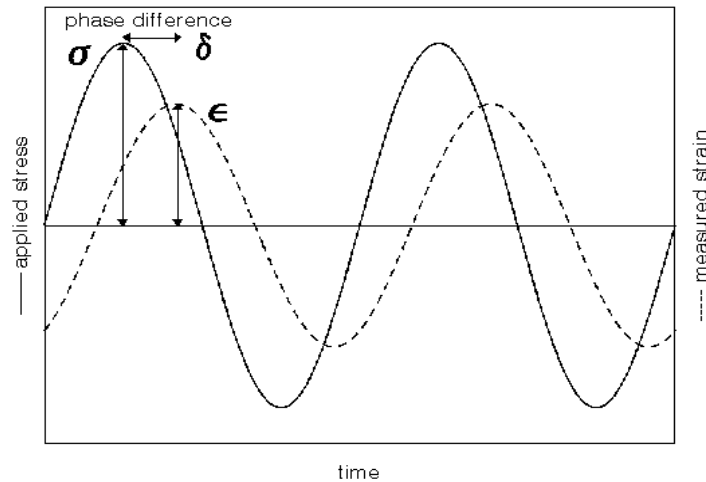
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# Appendix 1

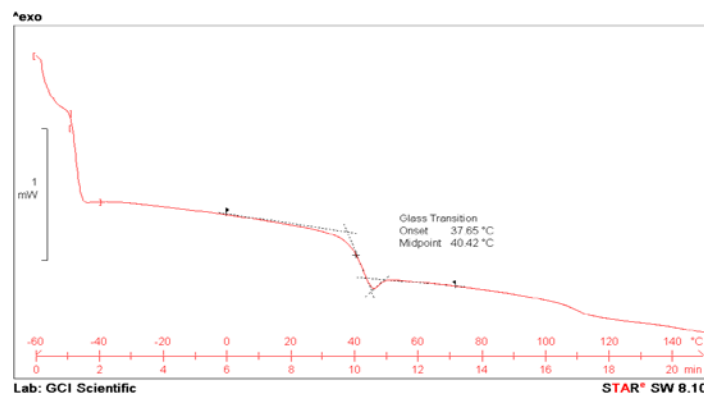
## Principles of DSC and DMA

A DMA instrument applies an oscillating force (stress  $\sigma$ ) to a sample and measures the resultant dimension change (strain  $\epsilon$ ). It is measured under controlled temperature (scanning or isothermal). For a perfectly elastic material the stress and strain are perfectly in phase. DMA measures the amplitudes of the stress and strain, as well as the phase angle ( $\delta$ ) between them, this value is used to resolve the modulus into the storage modulus  $E'$  and the loss modulus  $E''$ . For a perfectly elastic material,  $\tan \delta$  is zero whereas a perfectly viscous material  $\tan \delta$  is infinite (Duncan 2008).



## DSC

A DSC instrument measures the difference between the heat flow from the sample and a well characterised reference material as the temperature is changed. Changes in heat capacity during a glass transition are determined from the difference in heat flow (Gabbott 2008).



*DSC Method used for the tests (Schilling 1989)*

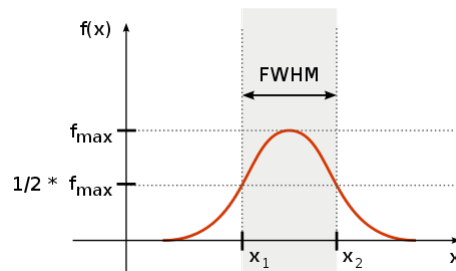
1. 25°C 5mins
2. 25-60°C at 10/min
3. 60°C 4mins

4. 60-100°C at 10/min
  5. 100°C 4mins
  6. 100-60°C at 10/min
  7. 60°C 4min
  8. 60-150 at 10.min
- All with N<sub>2</sub> 80ml/min

## Appendix 2

### Full width half maximum FWHM

The full width half maximum (FWHM) is defined as the width of the curve at a level which is half the maximum value of the peak ( $f_{\max}$ ).



## Materials and Suppliers

Araldite Epoxy 1253: [Conservation Resources \(U.K.\), Ltd](#), Unit 2, Ashville Way, Off Watlington Road, Cowley, Oxford OX4 6TU, UK.

Resin W Interior Wood Adhesive: Axminster Tools, Trafalgar Way, Axminster, Devon EX13 5PB, UK.

Microballoons: West System: Thames Ditton Marina, Portsmouth Rd., Surbiton Surrey KT6 5QD.UK  
<http://www.westsystem.com>

Coconut Shell Flour: Kindly supplied by Ray Marchant, Ebury St., London.

## Author Biographies and Contact Information

**Christina Young** has a BSc in Physics and an MSc in Applied Optics. After completing a PhD in the "Measurement of the biaxial tensile properties of paintings on canvas" at Imperial College, London (United Kingdom) in 1996, she joined Tate as a Research Fellow. She is currently a senior lecturer in easel painting conservation, as well as a conservation scientist and structural conservator, at the Courtauld Institute of Art in London. Christina supervises and undertakes canvas and panel structural conservation treatments, which helps ensure that her experimental research is relevant to conservation treatments. Her active areas of research are conservation mechanics, non-invasive monitoring techniques, methods/materials for structural conservation, and the conservation of contemporary art.

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## Biographies et coordonnées des auteurs

**Christina Young** détient un baccalauréat en physique et une maîtrise en optique appliquée. Après avoir terminé, en 1996, une thèse de doctorat intitulée « Measurement of the biaxial tensile properties of paintings on canvas » (mesure des propriétés des toiles en traction biaxiale) au Imperial College de Londres (Royaume-Uni), elle entre au Tate à titre de chercheuse universitaire. Elle est actuellement maître de conférence en conservation-restauration de peinture de chevalet, scientifique en conservation-restauration et restauratrice des structures au Courtauld Institute of Art, à Londres. M<sup>me</sup> Young supervise et réalise des traitements de restauration structurale des châssis, toiles et panneaux de bois, ce qui lui permet de vérifier la pertinence de son travail de recherche expérimentale du point de vue des traitements de restauration. Ses travaux de recherche en cours portent sur les techniques matérielles de la restauration, les techniques de surveillance non invasives, les méthodes et matériaux de restauration structurale et la conservation-restauration des œuvres d'art contemporain.

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Table 2 Results for all samples tested.

Sample	Film Cast	DSC Tg onset 2nd heating	DMA Tg onset 1Hz		Tg Peak 1Hz	Tg (area all data) 1H	Tanδ Peak Value	Tanδ FWHM	Modulus 1Hz (30C)	
<b>DMA Tension</b>										
EA1253_Ten_02	04/04/2010				57	12.5	0.499	27.4	8.36E+08	
EA1253_Ten_03	04/04/2010				58.2	14.9	0.494	26.8	6.69E+08	
EA1253_Ten_04	04/04/2010		42		58.2	13.9	0.493	23.7	6.41E+08	
					<b>Ave</b>	<b>57.8</b>	<b>13.8</b>	<b>0.495</b>	<b>25.967</b>	<b>7.15E+08</b>
					SDEV	0.57	1.0	0.003	1.621	8.6E+07
EA1253Mi_Ten_01_t4	04/04/2010		37		61.7	15.5	0.389	34.3	3.45E+08	
EA1253Mi_Ten_02	04/04/2010				61.5	13.7	0.412	27.3	4.68E+08	
EA1253Mi_Ten_03	04/04/2010				56.3	13.5	0.422	26.2	3.92E+08	
					<b>Ave</b>	<b>59.8</b>	<b>14.2</b>	<b>0.408</b>	<b>29.267</b>	<b>4.02E+08</b>
					SDEV	2.50	0.90	0.014	3.587	5.1E+07
<b>DMA Cantilever</b>										
EA1253_SC_01	04/04/2010				64.5		0.555	24.1	5.30E+08	
EA1253_SC_02_U2 *	04/04/2010				66.6		0.532			
EA1253Mi_SC_01	04/04/2010				65.4		0.46	27.4	1.80E+08	
RW_SC_02	22/03/2010				31.6		2.43	38.8	5.30E+08	
RWCoMi_SC_01	10/03/2010				33.7		0.653	40.5	1.80E+08	
<b>DSC</b>										
E1253_03			43.8							
EA1253Mi_02			46.3							
RW_02	22/03/2010		22.5							
RWCoMi_01	10/03/2010		23.2							
<b>Tensile</b>										
		<b>Ave. Test Temp</b>	<b>Measured Temp</b>	<b>Test RH</b>	<b>Bulk Elastic Modulus</b>	<b>UTS (N)</b>	<b>UTS (stress)</b>	<b>Strain to failure</b>	<b>Failure Strain %</b>	
RWCoMi_S10	10/03/10 50% 2	10		53	2.14E+08	10.6	2.75E+06	0.022	2.2	
RWCoMi_S14	10/03/10 50% 2	10		54	1.77E+08	15.06	4.73E+06	0.04	4	
RWCoMi_S15	10/03/10 50% 2	10		52	1.22E+08	10.09	3.26E+06	0.02	2	
RwCoMi_S16	10/03/10 50% 2	10		53	1.67E+08	18.78	5.76E+06	0.033	3.3	
RWCoMi_S02	10/03/10 50% 2	20		52	6.80E+07	15.7	3.90E+06	0.197	19.7	
RWCoMi_S03	10/03/10 50% 2	20		55	9.60E+07	17	5.20E+06	0.232	23.2	
RWCoMi_S04	10/03/10 50% 2	20		55	1.13E+08	18.2	6.20E+06	0.221	22.1	
RWCoMi_S05	10/03/10 50% 2	10		68	1.42E+08	22.9	6.70E+06	0.076	7.6	
RWCoMi_S06	10/03/10 50% 2	10		68	1.55E+08	30.3	7.80E+06	0.075	7.5	
RWCoMi_S07	10/03/10 50% 2	10		68	2.19E+08	30.1	8.70E+06	0.064	6.4	
RwCoMi_S13	10/03/10 50% 2	10		61	2.43E+08	26.1	8.97E+06	0.06	6	
EA1253_S22	04/04/10 52% 2	20	19.8	30.1		20.6	6.21E+06	0.037	3.7	
EA1253_S23	04/04/10 52% 2	20	19.5	30		10.5	2.95E+06	0.027	2.7	
EA1253_S24	04/04/10 52% 2	20	19.5	30		30.5	1.03E+07	0.058	5.8	
EA1253_S17	04/04/10 52% 2	20	19.5	54.9		28.6	8.74E+06	0.064	6.4	
EA1253_S18	04/04/10 52% 2	20	19.4	55.6		28.3	8.48E+06	0.077	7.7	
EA1253_S19	04/04/10 52% 2	20	19.5	54.4		31.5	8.63E+06	0.079	7.9	
EA1253_S01_U3	04/04/10 52% 2	30	30.8	44		28.8	7.67E+06	0.05	5	
EA1253_S03	04/04/10 52% 2	30	30.7	44		39.3	1.16E+07	0.064	6.4	
EA1253_S07	04/04/10 52% 2	30	30.1	53.8		37.1	9.48E+06	0.08	8	
EA1253_S08	04/04/10 52% 2	30	30.2	51.2		26.3	8.91E+06	0.068	6.8	
EA1253_S10	04/04/10 52% 2	40	39.3	25.3		24.5	5.73E+06	0.067	6.7	
EA1253_S11	04/04/10 52% 2	40	39.6	24.3		35.8	8.38E+06	0.077	7.7	
EA1253_S12	04/04/10 52% 2	40	39.7	25.9		23.1	8.14E+06	0.104	10.4	
EA1253_S14	04/04/10 52% 2	40	39.6	27		22.3	6.55E+06	0.086	8.6	
EA1253_S04	04/04/10 52% 2	50	49.3	21.5		15.7	5.69E+05	nk	#VALUE!	
EA1253_S05	04/04/10 52% 2	50	50.1	20.5		13.9	6.39E+06	0.075	7.5	
EA1253_S15	04/04/10 52% 2	50	50.1	17.5		13.5	4.92E+06	0.072	7.2	
EA1253_S16	04/04/10 52% 2	60	59.3	10.3		13	3.15E+06	0.169	16.9	
EA1253_S20	04/04/10 52% 2	60	60	10.2		8	3.01E+06	0.295	29.5	
EA1253_S21	04/04/10 52% 2	60	60	9.6		9.6	2.48E+06	0.281	28.1	