DYNAMIC MECHANICAL ANALYSIS AND ITS ADVANTAGES OVER DEFLECTION TEMPERATURE UNDER LOAD

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ABSTRACT

The ASTM D-648 is a standard method for evaluating the softening temperature of materials, commonly referred to as the Deflection Temperature Under Load (DTUL) or Heat Distortion Temperature (HDT). It can be shown that Dynamic Mechanical Analysis (DMA) provides information that pertains to the structural characteristics of the material, whereas DTUL does not.

INTRODUCTION

Historically, structural polymers and composites were tested to find a softening point in terms of temperature, above which, the material cannot support a load for any appreciably long times because of the loss in rigidity [5]. That temperature is the heat distortion temperature. The American Society for Testing and Materials (ASTM) has a few standard methods for evaluating this parameter, designated D-648 (flexure), D-1637 (tension), D-1043 and D-1053 (shear), and D1525 (penetration) [5].

In ASTM D-648 a bar of rectangular cross-section is subjected to a constant flexural load (455 Pa or 1820 Pa) and the temperature is ramped up at 2°C/min [1]. The temperature at which a certain deflection (0.25mm) is observed is the DTUL. The TA Instruments DMA 2980 can be used to evaluate DTUL. Based on the dimensions of the ASTM sample, 0.25mm is equivalent to a certain strain (0.121%). If the DMA is used in the 3-point bending mode and the force equivalent to 455 Pa is applied on a sample, then the temperature at which the DMA sample deflects by 0.121% strain is the DTUL [2]. This conversion exercise is necessary because the samples that can be tested using the DMA are much smaller than the ASTM recommended one.

The DTUL is a single-point measurement. The effect of certain molecular characteristics such as crystallinity and crosslinking affect the DTUL [5]. It is more a bulk property of the material than one relating to its microscopic structure. For example,
in immiscible polymer blends that have two distinct glass transition temperatures, each characteristic of one component in the blend, the DTUL would be unable to detect the two components. DTUL cannot provide information about secondary transitions due to small-scale relaxations.

The Dynamic Mechanical Analysis, especially when used with temperature ramps, can provide all the information that the DTUL can and cannot give. The DMA can discern the elastic and viscous components of deformation [4], [5], [6]. It gives a very sensitive profile of the viscoelastic properties like Storage Modulus, Loss Modulus and tan δ changing with temperature. By measuring true material properties, the DMA is able to better assess the effects of crystallinity, cross-linking, fillers, additives, molecular weight, aging, etc. Thus, the ability to differentiate between samples on a more fundamental basis is realized.

SAMPLE

There were two samples that were tested, namely, A and B. They were both fiber-filled composites of material constituents that cannot be disclosed. As such, not much was known about them prior to testing.

EXPERIMENTAL

The 3-point bending clamps were calibrated prior to testing. For the DTUL, the force (calculated using method outlined in [2]) was held constant while the temperature was ramped at 2°C/min. The displacements that would indicate DTUL for each sample were monitored. For the DMA temperature ramps, the oscillation was performed at 1Hz at an amplitude of 20 micrometers and force track of 130%. The temperature was ramped at 3°C/min from –50°C to 220°C. There was no need to maintain the same ramp rate between the two types of experiments because the aim was to show the presence of additional information in any generic DMA temperature ramp than in DTUL.

RESULTS AND DISCUSSION

Shown in Figure 1-3, are the displacements occurring in the samples as a function of the changing temperature for the evaluation of DTUL. As can be seen, the displacements at which the required strain for DTUL is achieved are different from one sample to another, based on the differences in the dimensions of those samples. But the DTUL for samples A and B are 208.2±2°C and 212.5°C, respectively. This is the extent of the capability of DTUL because that temperature is the only true measurement that is made!
In Figure 4 and 5, the Storage and Loss Moduli changing as a function of temperature are shown for samples A and B, respectively. In each of these figures, a glass transition temperature is noted by the drop in storage modulus ($E'$) and a peak in the loss modulus.
(E”). Taking the peak in loss as the indicator, there seems to be a difference in the Tgs of the two materials (47°C for A and 53°C for B). Furthermore, there are some additional E” relaxation peaks in B that are not present in A. These may be due to low molecular weight additives in B or other short-chain motions in the constituents [4]. (See [3])

![Figure 3. DTUL of sample B.](image)

![Figure 4. DMA temperature ramp of sample A.](image)
At higher temperatures, there is a precipitous drop in $E'$, denoting melting of the matrix material. The melting points, according to the $E''$ peak, for A and B are 207°C and 208°C, respectively. These values are very close to the DTULs of the two samples. This would indicate that DTUL is only an indicator of the softening of the bulk material.

Figure 6 shows the comparison of the DMA temperature ramps of A and B. This indicates yet another difference – in their storage modulus, a parameter that DTUL cannot ascertain. Sample B has a higher modulus than A.

![Figure 5. DMA temperature ramp of sample B.](image1)

![Figure 6. Comparison of DMA temperature ramps of A and B.](image2)
**SUMMARY**

DMA temperature ramp is a more versatile technique in evaluating mechanical properties of materials. In the same amount of time that it would take to run a DTUL experiment, one could have the storage and loss moduli at a variety of temperatures, and thereby $T_g$, secondary transitions, and $T_m$ at one’s fingertips. The viscoelastic parameters are better suited for linking mechanical properties to structural and/or morphological characteristics of materials.

**REFERENCES**