INTRODUCTION

The heat distortion temperature (HDT), deflection temperature under load (DTUL) or softening temperature usually denote the highest temperature to which a thermoplastic polymer may be used as a rigid material (1). Up to this maximum temperature, a material is able to support a load for some appreciable time. For amorphous polymers, the DTUL is close to the glass transition temperature while for semi-crystalline polymers the DTUL is in the vicinity of the melting temperature.

ASTM International Standard D 648 defines the DTUL temperature at which a polymer reaches a defined modulus value. According to D 648, a rectangular bar cross-section is testing in thre-point bending by applying a load at its center that fixes a maximum stress of 0.455 MPa (66 psi) or 1.82 MPa (624 psi). The specimen is 127 mm (5 in) in length, 12 mm (0.5 in) in depth/thickness and by any width from 3 mm (0.12 in) to 13 mm (0.5 in). The temperature is raised at 2 ± 0.2 °C/min. The temperature at which the test bar deflects by 0.25 (0.10 in) is recorded as the DTUL.

DTUL on the DMA Q800

The largest three-point bending clamps available on the Q800 hold a sample of maximum length, width and thickness of 50 mm (2.0 in) 15 mm (0.59 in) and 7 mm (0.27 in), respectively. These dimensions are very small compared to the ASTM dimension requirements. The DMA Q800 is capable of exerting a certain force on the sample such that there is a stress of 0.455 or 1.80 MPa on the sample. But, for the test to be valid under the ASTM conditions, this smaller DMA sample must deform to the same strain induced in the sample at a load of 0.455 or 1.82 MPa as that in the ASTM sample. So, given, the sample dimension for testing on the DMA Q800, the following calculation must be performed.

1. Calculate the force (F) required to achieve the desired stress.
2. Calculate the strain (ε) in the ASTM sample at the deflection of 0.25 mm, and
3. Calculate the required deflection (d) in the DMA sample so that there is an induced strain equivalent to ε.
FOR EXAMPLE

To use the largest three-point bending clamps available for evaluation of DTUL, for an example polymer of length of 55 mm (2.26 in), width of 11.95 mm and thickness of 4.45 mm. The sample length is deliberately kept longer than the 55 mm span of the clamps. Assuming that the stress on the sample is 455 kPa:

Step 1: Calculation of Force

According to the three-point bending equation (reference)

\[
F = \frac{2}{3} \left( \sigma \frac{T_{DMA}^2 W_{DMA}}{L_{DMA}} \right) \\
= \frac{2}{3} \left( 0.455 \text{ N/mm}^2 \left[ (4.45 \text{ m})^2 \times 11.95 \text{ mm} / 50 \text{ mm} \right] \right) \\
= 1.44 \text{ N}
\]

Note: The total sample length is 55 mm, but for the calculations, the 50 mm total span between the two outer points on the clamp should be used.

Step 2: Calculation of the ASTM Sample Strain

\[
\varepsilon = 6 \frac{d_{ASTM} T_{ASTM}}{L_{ASTM}^2} \\
= 6 \times (0.25 \text{ mm}) \times (13 \text{ mm}) / (127 \text{ mm})^2 \\
= 0.00121 \\
= 0.121 \%
\]

Step 3: Calculation of DMA Sample Deflection

\[
d_{DMA} = \frac{\varepsilon L_{DMA}^2}{(6 T_{DMA})} \\
= 0.00121 \times (50 \text{ mm})^2 / (6 \times 4.45 \text{ mm}) \\
= 0.113 \text{ mm} \\
= 113 \mu\text{m}
\]

Where:

- \( \sigma \) = Stress on the ASTM and DMA sample *
- \( \varepsilon \) = Strain on the ASTM and DMA sample *
- \( d_{ASTM} \) = Deflection in the ASTM sample = 0.25 mm
- \( d_{DMA} \) = Deflection at \( \varepsilon \) strain
- \( F \) = Force
- \( L_{ASTM} \) = Length of the ASTM sample = 127 mm
- \( L_{DMA} \) = Length of the DMA sample = 50 mm
- \( T_{ASTM} \) = Thickness of the ASTM sample = 13 mm
- \( T_{DMA} \) = Thickness of the DMA sample = 4.45 mm
- \( W_{ASTM} \) = Width of the ASTM sample
- \( W_{DMA} \) = Width of the DMA sample = 11.95 mm

* ASTM and DMA equivalence of these parameters is required.
Similar calculations may be performed for samples of any width and thickness.

The next step is to program a test to heat the sample under the conditions of the calculated applied load. The instrument mode must be DMA Controlled Force.

A typical DTUL curve shows a negative dimension change, which indicates upward motion of the fulcrum due to the thermal expansion of the sample. As the temperature rises, there is a downward movement that overcomes the thermal expansion and the sample starts to deform under the load. When the dimension change reads a positive 113 µm dimension change, that is the temperature we are looking for when reporting DTUL. For the polycarbonate used in this example, the DTUL is about 140 °C.

REFERENCES


KEYWORDS

dynamic mechanical analysis, thermoplastic polymers
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