Glass Transition Temperature Measurements Using Dynamic Mechanical Analysis

In addition to first-order phase transitions such as melting and vaporization, there are second-order transitions that occur in certain materials. One of these second-order transitions is the glass transition exhibited by amorphous materials. When amorphous materials are cooled below their glass transition temperatures, they become hard and brittle, like glass; when the amorphous materials are heated above their glass transition temperatures, they are in the rubbery state, where they are soft and flexible. This glass transition phenomenon is mainly because the molecular chains become entangled and cannot slip pass each other at low temperatures. The temperature at which the molecular chains become 'locked' upon cooling or 'released' upon heating is referred to as the glass transition temperature (Tg).

However, the glass transition does not occur at a single temperature. Depending on the definition in use and method to measure, it can be defined as the temperature at which the material (a) suddenly loses mechanical strength, (b) exhibits a step-change in specific heat capacity, or (c) exhibits increased thermal expansion. There are even more definitions depending on which properties are important for the end use.

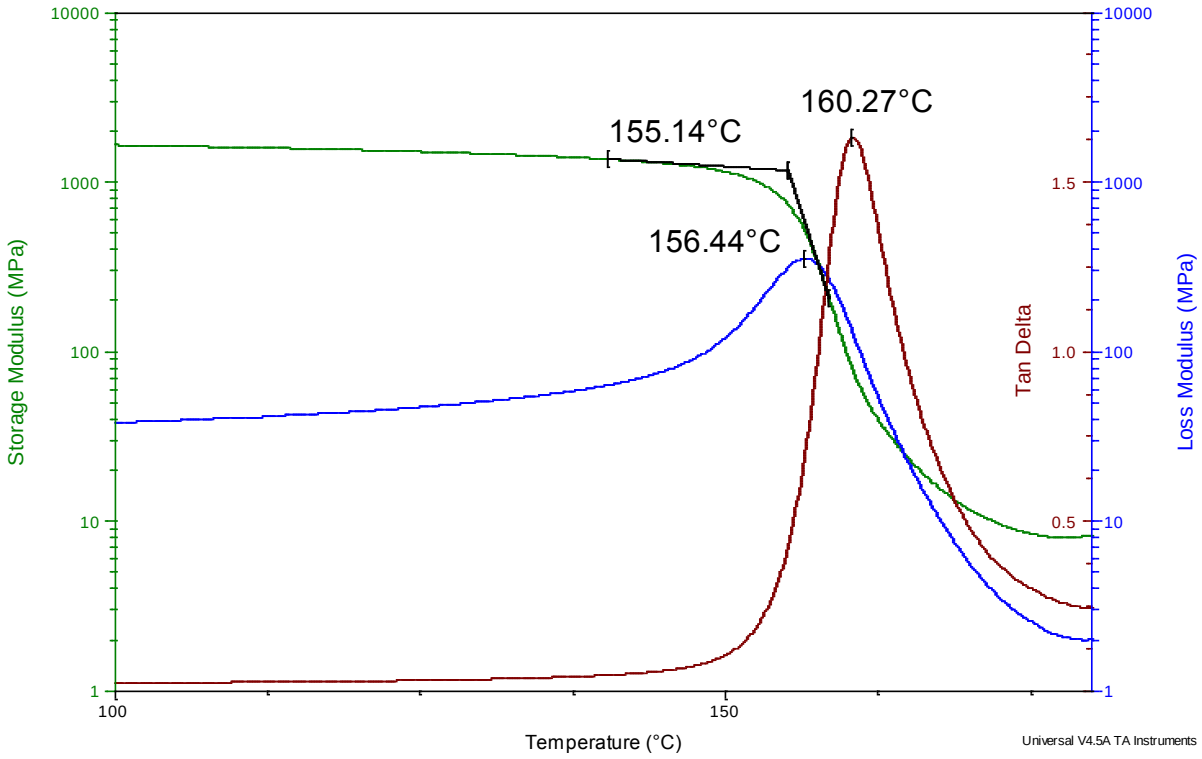

Figure 1. Dynamic mechanical analysis of the glass transition of polycarbonate.

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In this application study, the glass transition temperature of polycarbonate was determined using a Q800 Dynamic Mechanical Analyzer (DMA) manufactured by TA Instruments. This test system characterizes the viscoelastic behavior of materials by applying a sinusoidal force to a specimen and measuring the material response. For viscoelastic materials, the responses are time-dependent and vary depending upon the frequency and temperature at which the force is applied. By loading the specimen in tension, compression, bending, or shear, the DMA is able to determine storage and loss moduli and damping properties of a material for a specific temperature and frequency. This gives the system unparalleled sensitivity in characterizing the thermo and dynamic mechanical properties of materials.

With DMA, a material's transition from the glassy to the rubbery state is signaled by a dramatic decrease in storage modulus, a peak in loss modulus and a peak in damping coefficient (tan delta), all occurring immediately one after another. Each of these events occurs at a slightly different temperature but combine to describe the temperature range over which the glass transition occurs. The sudden decrease in storage modulus occurs at the lowest temperature and indicates physical softening. If the material is heated above this temperature in mechanical service, it will fail. The peak in loss modulus occurs at the middle temperature and indicates segmental motion within the material. The peak in tan delta occurs at the highest temperature and indicates the midpoint of the material's transition between the glassy and rubbery states. This is illustrated in Figure 1 using the results obtained on polycarbonate.

The polycarbonate was tested using a load frequency of 1 Hz. Due to polymer's time-dependent properties, a change in test frequency will alter the measured Tg. Ebatco's Application Note on the principle of time-temperature superposition discusses this phenomenon in more depth.