Specific knowledge about the properties of plastics under different thermal conditions is essential.

Application Note # 90

Determination of the Heat Influence on Plastics and Coatings Using the Dynamic Measurement Mode

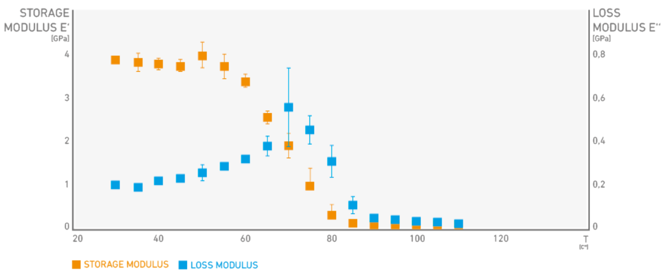
Properties of plastics and coatings vary significantly under different temperature conditions. For example, plastics are brittle and less elastic in a cold environment, whereas when heated, they become more ductile and flexible. The glass transition temperature is one of the most critical parameters for determining the properties of polymer materials. In particular, it provides information on their dimensional stability under the influence of heat.

In general, the glass transition temperature T_g is defined as the temperature at which amorphous or semi-crystalline plastics change from the highly viscous, viscous or rubbery state to the hard-elastic or glassy state. Concerning the required material behavior, materials are used either above or below their glass transition temperature.

Precise knowledge of T_g in the characterization of plastics is essential – even for survival. For example, as the cause of the accident of the Challenger space shuttle in 1986, a defective O-ring seal was identified. Cool temperatures at night made it insufficiently elastic and brittle. The elastomeric seal was operated below its T_g, the sealing function was no longer given, and fuel leaked out.

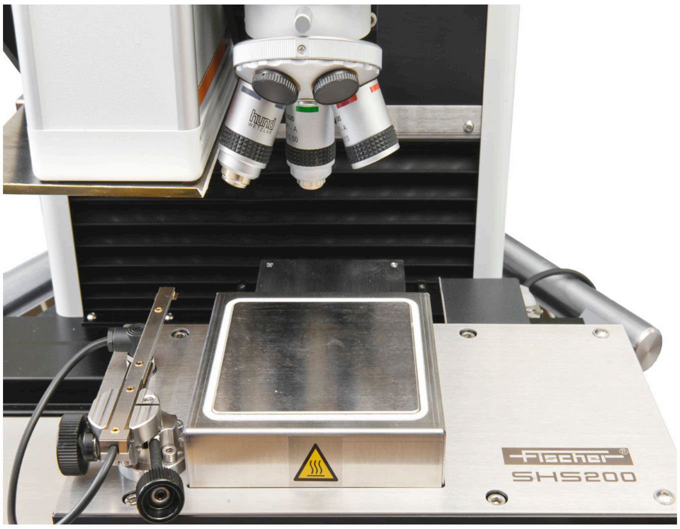
Dynamic mechanical thermal analysis is considered to be the essential characterization method in the field of thermal analysis. By applying an oscillating force to the sample, the viscoelastic properties and the elastic modulus values are determined as a function of frequency and temperature. The phase shift between the excitation and response signal provides information on whether a material behaves more viscously or elastically.

The graphic shows how elasticity parameters can be used to determine the glass transition in a paint sample. The elasticity modulus is composed of the storage modulus E' as the real part and the loss modulus E'' as the imaginary part. The storage modulus describes the mechanical energy that the system can store, while the loss modulus defines the measure of the energy dissipated by the material. In the example, at temperatures up to approx. 45 °C, the loss modulus is very low. The paint sample reacts like a solid, and the work of deformation



The elasticity modulus is composed of storage and loss modulus. The glass transition is reached at the maximum of the loss modulus or the storage modulus's inflection point.

is almost completely stored during the loading process. Above 50 °C, the loss modulus increases more strongly. The paint sample loses its elastic properties, and the mechanical energy introduced is primarily converted into thermal energy. At the same time, the storage modulus decreases. The glass transition is reached at the maximum of the loss modulus or inflection point of the storage modulus.



Heating table SHS200 for FISCHERSCOPE® HM2000 and PICODENTOR® HM500 to analyze mechanical material properties up to 200 °C.

With the SHS200 heating stage for the FISCHERSCOPE® HM2000 and PICODENTOR® HM500 nanoindentation systems, Fischer offers the right accessory for determining the hardness and elastic properties of coatings and plastics at temperatures of up to 200 °C. Using Dynamic Mode as an additional feature of the powerful WIN-HCU software, materials can be characterized even more precisely, and specific glass transition temperatures can be tested or determined.

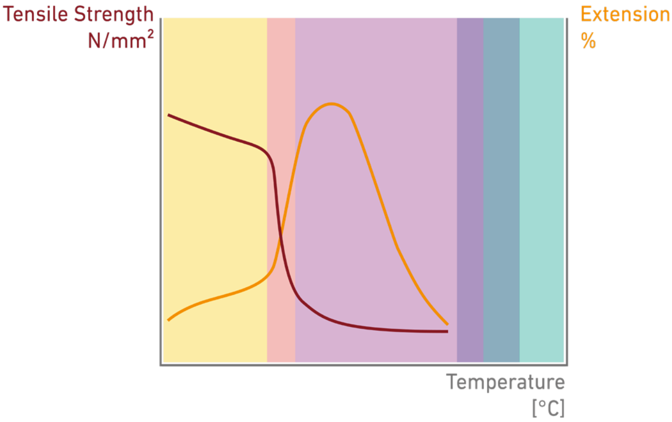
First, the specimen is loaded with a specific force of up to 2 N, analogous to an indentation measurement. Further, a minimal sinusoidal oscillation (amplitude of less than one millinewton) is applied to this force. The deformation of the material remains in the elastic range. The sample material's response is measured by looking at the amplitude (usually a few nanometers) and a phase shift between the excitation vibration and the response signal.
The measurement starts automatically when the heating stage reaches the predefined temperature. In addition to a heat shield to protect the measuring device, the indenter's base body is made of ceramic to minimize its thermal expansion. It ensures reliable values even when the measuring time is longer.

The glass transition is specific to each plastic and is based on the measurement method used. That's why it must be specified in the characterization. The point where T_g is located depends on how densely a plastic is crosslinked. For example, regarding a duromer, the point is much higher compared to an elastomer.

For amorphous thermoplastics such as polymethyl methacrylate (PMMA), the optimum application ranges are below T_g. Whereas, regarding the example of the Challenger accident, elastomers are used above T_g.

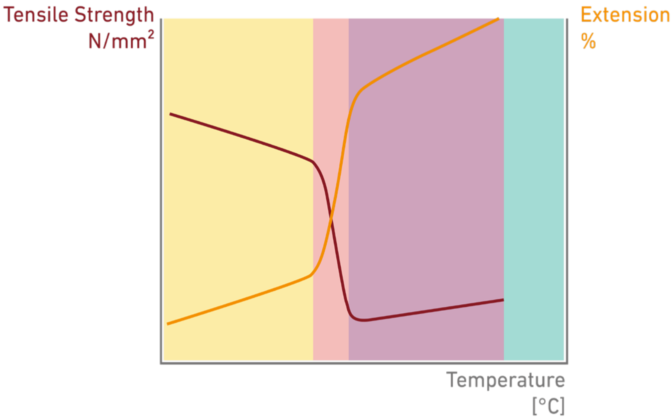
Conclusion:
Measurements with the nanoindentation systems FISCHERSCOPE® HM2000 and PICODENTOR® HM500 in conjunction with the SHS200 heating stage allow comprehensive characterization of plastics and coatings. In one series of measurements, both various viscoelastic properties (elasticity modulus, storage modulus and loss modulus) can be measured, and the glass transition temperature can be determined. An additional measurement of T_g with other methods such as differential scanning calorimetry is not necessary.

Amorphous Thermoplastic



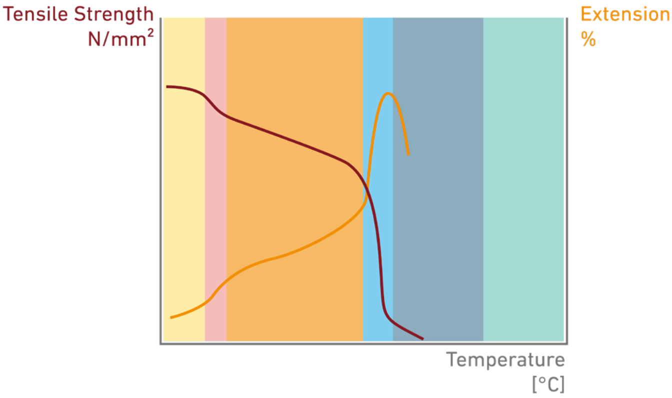
- GLASSY STATE
- T_G GLASS TRANSITION ZONE
- FLEXIBLE
- T_F FLOW TEMPERATURE
- PLASTIC
- T_Z DECOMPOSITION ZONE

Elastomer



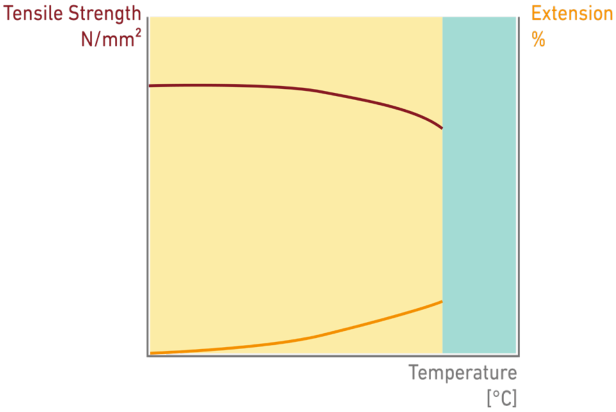
- GLASSY STATE, ENERGY-ELASTIC
- T_G GLASS TRANSITION ZONE
- FLEXIBLE
- T_Z DECOMPOSITION ZONE

Partially Crystalline Thermoplastic



- GLASSY STATE, BRITTLE
- T_G GLASS TRANSITION ZONE
- VISCOPLASTIC, STIFF
- T_M CRYSTALLITE MELTING TEMPERATURE
- PLASTIC
- T_Z DECOMPOSITION ZONE

Duromer



- GLASSY STATE, STIFF, BRITTLE
- T_Z DECOMPOSITION ZONE

Phase diagrams of amorphous and partially crystalline thermoplastics.

Phase diagrams of elastomers and duromers.