Rheological Characterization Laboratory (RKL)

Rheology is a science of flow and deformation of materials. In METU Central Laboratory, Rheometer, Capillary Rheometer and Dynamic Mechanical Analyser (DMA) are available for measuring rheological properties of materials.

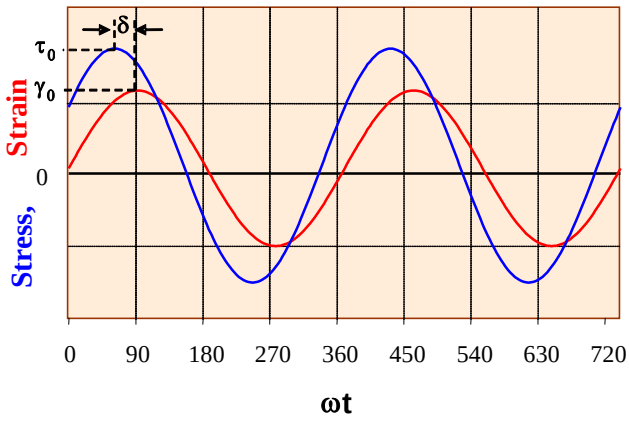
BASIC PRINCIPLES

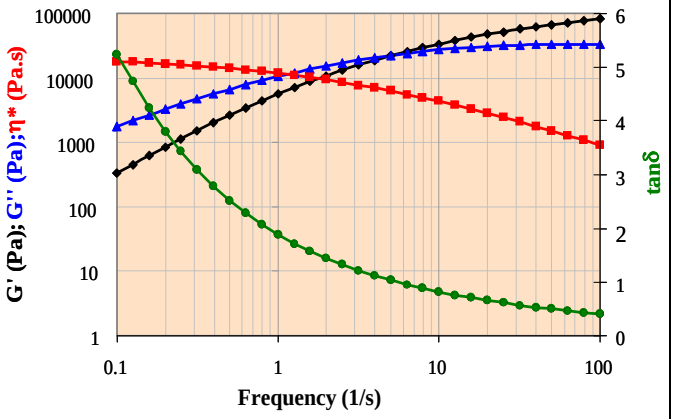
Capillary rheometer is a high-shear instrument consisting of a heated barrel and a piston that drives molten material through a calibrated die, applying pressure (force) at a constant piston speed (constant shear rate). As a result, flow curves (apparent viscosity against shear rate) for materials are obtained.

In a rheometer, it is possible to carry out steady (flow curves), dynamic, and transient measurements. A dynamic rheological measurement consists of subjecting a material to a continuously oscillating strain over a range of frequencies and measuring the peak value of the stress τ_0 and the phase difference between the stress and strain δ. If the material behaves as an ideal elastic material, the resulting stress is proportional to the strain amplitude and the stress and strain signals are in phase. The behaviour of an ideal elastic material is represented by Hooke's Law. For an ideal viscous material, the stress is proportional to the strain rate and the stress is 90° out of phase with strain. The behaviour of an ideal viscous material is represented by Newton 's Law. For viscoelastic materials, the phase lag (δ) between stress and strain occurs somewhere between the elastic and viscous extremes (i.e. between 0° and 90°) (Figure (1)). The stress signal of a viscoelastic can be separated into two components: an elastic stress that is in phase with strain, and a viscous stress that is 90° out of phase with strain. The storage modulus (G') (the ratio of the elastic stress to strain) represents the ability of a material to store energy elastically. The loss modulus (G'') (the ratio of the viscous stress to represents the ability of a material to dissipate energy. The complex modulus (G^*) represents the overall the resistance of a material to deformation.

The working principle of a DMA is the same as a rheometer. The most significant difference between a DMA and a rheometer is the forces applied to the samples during measurements. Extensional and shear forces are applied to DMA and rheometer, respectively. The following viscoelastic properties are determined using a DMA or a rheometer.

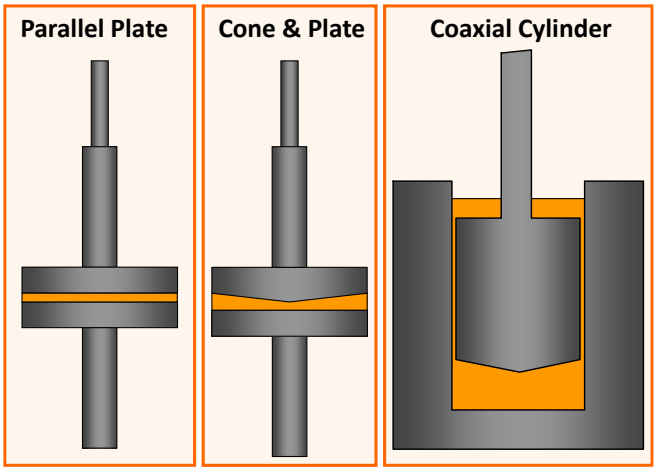
	Rheometer	DMA
Complex Modulus	$G^*=\tau_0/\gamma_0$	$E^*=\sigma_0/\varepsilon_0$
Storage Modulus	$G'=G^*\cos\delta$	$E'=E^*\cos\delta$
Loss Modulus	$G''=G^*\sin\delta$	$E''=E^*\sin\delta$
Complex Viscosity	$\eta^*=G^*/\omega$	$\eta_{ext}^*=E^*/\omega$

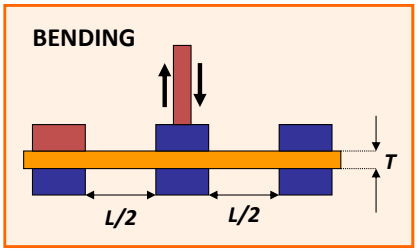


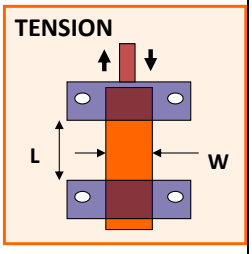


SAMPLES

In the rheometer, liquid (or semi-liquid) samples can be characterised using a parallel plate, cone & plate, or co-axial cylinder measuring systems. In the DMA, the rheological properties of solid samples are determined using tension or bending measuring systems. Schematic illustrations of the measuring systems are given in the following figures.







For capillary rheometer, 15 gram powder or granule sample is sufficient. For rheometer, 20 ml sample would be sufficient. When the bending mode is used in the DMA, the length, width, and thickness of the samples should be 50 mm, 5-10 mm, and 1-3 mm, respectively. For the tension mode, the length, width, and thickness of the samples should be 40-50 mm, 5-10 mm, and 0.1-3 mm, respectively.

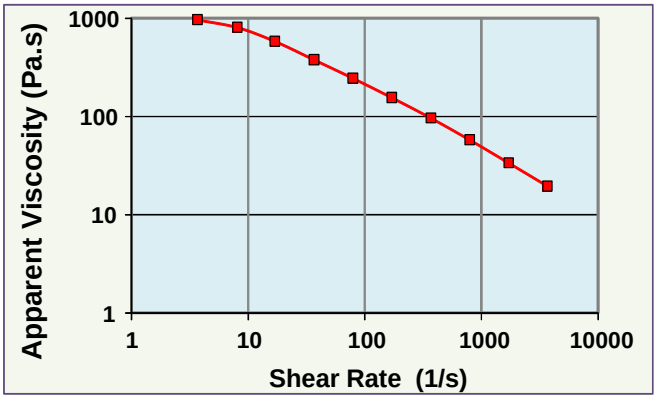
MATERIALS

By using the instruments available in the Rheological Characterization Laboratory, it is possible to determine the rheological properties of the following materials: polymers, polymer blends, elastomers, composites, foods, and aerospace materials.

EXAMPLES

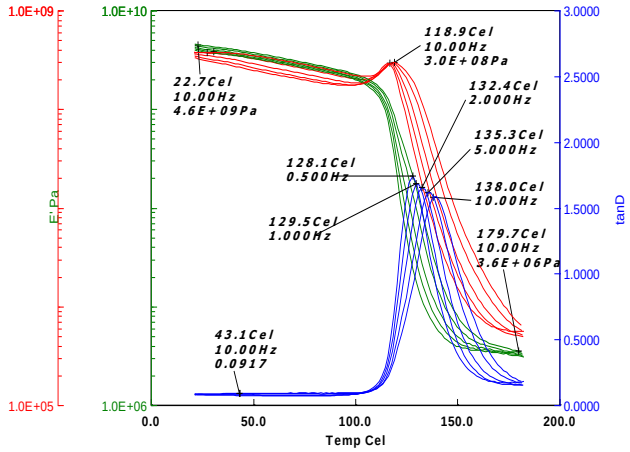
Determination of flow curves (apparent viscosity versus shear rate) for molten polymers:

The knowledge of the flow curves for materials is very important for determining the materials processing parameters. In the following figure, the flow curve for polypropylene obtained using the capillary rheometer is shown.

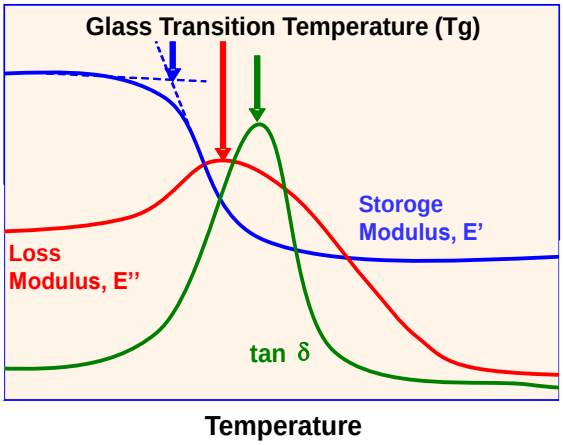


The viscoelastic properties (storage, loss, and complex moduli; complex viscosity) of polymers:

DMA and rheometer are used to determine the viscoelastic properties various types of materials including polymers. The following graph shows the viscoelastic properties of PMMA sample as a function of temperature and frequency.



Glass Transition Temperature (T_g): The T_g of polymers can be determined using various techniques such as differential scanning calorimetry, dilatometer, and DMA). Among those techniques, DMA is the most sensitive technique for determining the T_g of polymeric materials. When DMA is used, the T_g of the polymers is generally determined from $tan\delta$ curves.



STANDARD TEST METHODS

ISO 11443: Fludity of plastics (Capillary Rheometer)

ISO 6721-10: Complex shear viscosity (Rheometer /Parallel Plate)

ISO 6721-4: Plastics- Determination of dynamic mechanical properties - Tensile vibration (DMA)

ISO 6721-11:2012: Plastics -- Determination of dynamic mechanical properties: Glass transition temperature (DMA)

INTERNATIONAL PROFICIENCY TESTS

In 2013, RKL participated in two proficiency tests (ISO 11443 and ISO 6721-10) organized by Kunststoff-Institut Lüdenscheid, Germany. Satisfactory results were received for all the measured properties using both the rheometer and the capillary rheometer.

CONTACT DETAILS

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