MSE 310 Lecture 7

Dielectric Thermal Analysis (DETA)

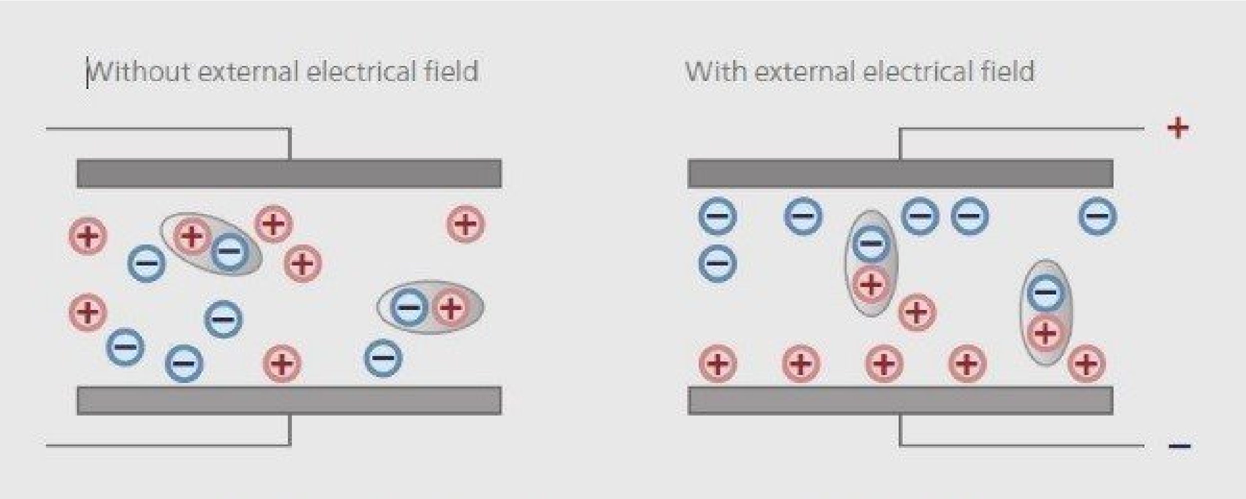
Dielectric thermal analysis (DETA) is a materials science technique in which an oscillating electric field is used to analyze changes in the physical properties of a number of polar materials. A **polar material** in the strict (crystallographic) sense is a **material** containing a nonvanishing dipole moment. This thermal analysis technique can be used with materials in a range of forms, from thin films and sheet materials to powders or liquids. Another similar technique is dynamic mechanical analysis (DMA), in which a mechanical force is used instead of an electrical field.

Fundamental Principle

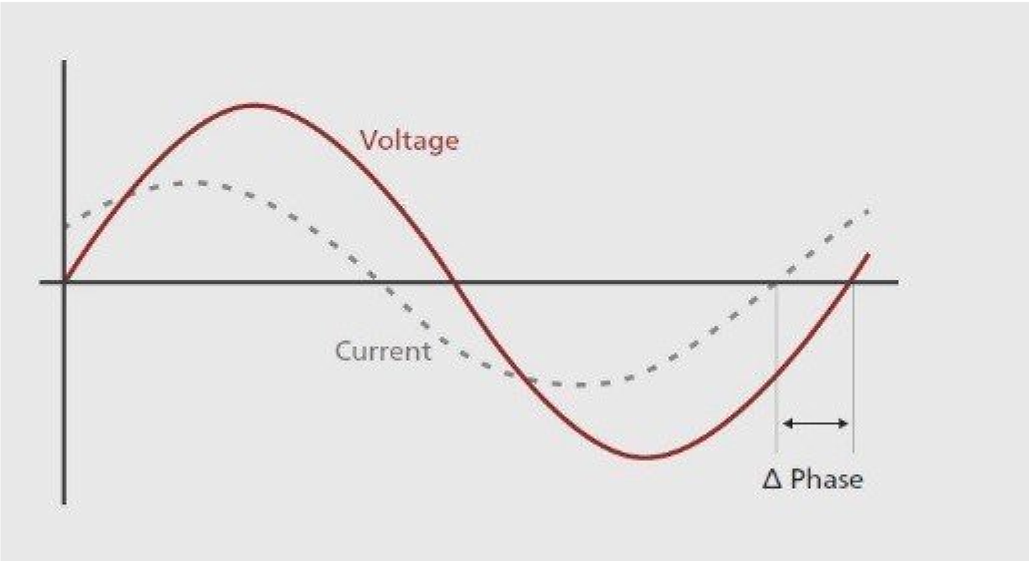
The functional principle is consistent with that of an impedance measurement. In a typical test, the sample is placed in contact with two electrodes (the dielectric sensor). When a sinusoidal voltage is applied, the charge carriers inside the sample are forced to move: positively charged particles migrate to the negative pole and vice versa. This movement results in a sinusoidal current with a phase shift.

In the frequency range (up to 1 MHz), the charge carriers are mainly ions (often present as catalysts or impurities) and additionally dipole alignment takes place within the electrical field.

The response signals – current and phase shift – are a function of the ion and dipole mobility. This relationship makes dielectric (thermal) analysis an ideal method for monitoring a curing process, where the sample's viscosity is increasing dramatically. As a consequence, the mobility of the charge carriers decreases, causing a corresponding attenuation of the amplitude and an increased phase shift in the resulting signal.



Behavior of ions and dipoles inside an external electrical field



DEA – Measurement principle

Results

An external electric field generated by an excitation voltage is applied to the sample and the response, occurring as a current through the material, is measured. Dipoles will be aligned and the ions will move towards the oppositely charged electrode, which can be seen in permittivity ε' and loss factor ε'', respectively. Based on the sample's characteristics, a time shift between the excitation and response signal is detected which, along with voltage and current, allows for calculation of the dielectric magnitudes.

In detail, this yields information about:

- Flow behavior
- Reactivity
- Cure progress

How does Dielectric Thermal Analysis Work?

The electrical **Capacitance** C (=Q/V) **is** defined as, the ratio of the electric charge Q on each conductor plate of a **Capacitor**, to the potential **difference** V **between** them. Also, the electrical **conductance** G (=i/V) **is** defined as, the ratio of the electric current i (=dq/dt) to the potential **difference** V.

Capacitance pertains to the ability of capacitors to store electric charges in them.The SI unit used for the quantity is the farad.

Conductance pertains to the ability of conductors to easily pass electric current through them. The SI unit of this quantity is the mho. This property is the opposite of resistance which has an SI unit of ohm.

In dielectric thermal analysis, an oscillating AC electrical field is applied to a sample of material, and measurements of the material's capacitance and conductance are taken as a function of time, temperature and frequency. Some of the electrical charge will be stored in the material sample (capacitance) and the rest of the electrical charge will be dissipated through the material sample (conductance). The relaxation properties of the material sample will determine the sample's rates of capacitance and conductance.

In practice, a material sample will be placed in contact with two parallel plate electrodes, and a sinusoidal voltage is applied to one of the electrodes. The response is then measured at the second electrode, with the sinusoidal current being weakened and shifted in accordance with ion mobility and dipole alignment. The dipoles will attempt to align with both the ions and the electrical field, and move toward the electrode of opposite polarity. The oscillating voltage signal is applied at frequencies from 0.001 to 100,000 Hz.

Data are presented in terms of the relative permittivity (ε') and dielectric loss factor (ε'') – these are related to capacitance and conductance by:

$$\varepsilon' = C/(\varepsilon_0.A/D)$$

&

$$\varepsilon'' = G/(w.\varepsilon_0.A/D)$$

where ε_0 is the permittivity of free space (8.86×10^{-12} F m^{-1}) and A/D (in m), is the ratio of electrode area (A) to plate separation or sample thickness, D for a parallel plate capacitor. ε' & ε'' are dimensionless quantities.

The ratio $\varepsilon''/\varepsilon'$ is the amount of energy dissipated per cycle divided by the amount of energy stored per cycle and known as the dielectric loss tangent or dissipation factor (tan δ).

Applications of Dielectric Thermal Analysis

Dielectric thermal analysis can be used in investigations into the curing behavior of thermosetting resin systems, adhesives, paints, and composite materials. Dielectric thermal analysis can also be used to characterize polar materials including:

- PVC – polyvinyl chloride
- PVDF – polyvinylidene fluoride
- PMMA – poly(methyl methacrylate)
- PVA – poly(vinyl acetate)

Polymer properties examined by DETA are permittivity (ε'), the measure of the degree of alignment of the molecular dipoles to the electrical field, and the loss factor (ε"), which represents the energy required for the reorientation of the dipoles and ions. The dissipation factor ($\tan \delta = \varepsilon''/\varepsilon'$) and the conductivity are also examined.

With DETA, the dielectric constant and polarizability of polymers are easily detected during phase transitions such as the glass transition (Tg), a unique characteristic of polymers in which a material will transition from a hard state (glassy) to a viscous state as the temperature of the polymer is increased. Other transitions include melting and crystallization and secondary transitions.

Other applications of dielectric thermal analysis are monitoring curing kinetics of epoxy and urethane systems.

Benefits of Dielectric Thermal Analysis

The applications for dielectric thermal analysis are very similar to that of dynamic mechanical analysis and differential scanning calorimetry (DSC). DSC is the most widely used thermal analysis technique and has been used to analyze food, pharmaceuticals, polymers, plastics, glasses, ceramics, and proteins. However, dielectric thermal analysis has a number of advantages over both DMA and DSC.