LECTURE-28

THERMAL ANALYSIS

- Thermal analysis represents the group of techniques in which specific physical properties of a material are measured as a function of temperature.
- These techniques are mainly based on the measurement of temperatures at which phase changes or any other changes may occur, the measurement of the energy absorbed (endothermic transition) or energy evolved (exothermic transition) during a phase transition or a chemical reaction, and the assessment of physical changes resulting from changes in temperature.
- Various environments such as vacuum, inert, or controlled gas composition and heating rates from 0.1 to 500°C min^{-1} are used for temperatures in the range of −190- 1400 °C.

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- The analysis of gases released by the specimen as a function of temperature is possible when thermal analysis equipment is coupled with a mass spectrometer.
- On the basis of applications of type of thermal analysis techniques are varied.
- For environmental measurements, these parameters can be measured: vapor pressure, softening temperatures, thermal stability, flammability,, and boiling points.
- Compositional analysis offers phase diagrams, solvent retention, mineral characterization, free versus bound water, and polymer system analysis.
- In the important area of product reliability, thermal methods provide heat-capacity data, liquid-crystal transitions, purity, polymer quality control, glass transitions, Curie point, and fiber properties.

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- Information on stability can be obtained from modulus changes, creep studies, expansion coefficients, and antioxidant evaluation.
- Dynamic properties of materials are found from cure characteristics, impact resistance, elastic modulus, loss modulus, viscoelastic measurements, and shear modulus.
- Eventually, the chemical reactions can be followed through catalyst evaluation, reaction kinetics, metal–gas reactions, heats of transition, and crystallization phenomena.

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There are usually three types of thermal analysis techniques are preferred such as

I. Differential scanning calorimetry (DSC)

II. Differential thermal analysis (DTA)

III. Thermo gravimetric analysis (TGA)

Differential scanning calorimetry (DSC) :

- It consists the measurement of power required to maintain ΔT equals to zero with reference to temperature or time.
- **Instrumentation:** Use two separate furnaces below 650 °C and control both specimen and reference temperature independently.

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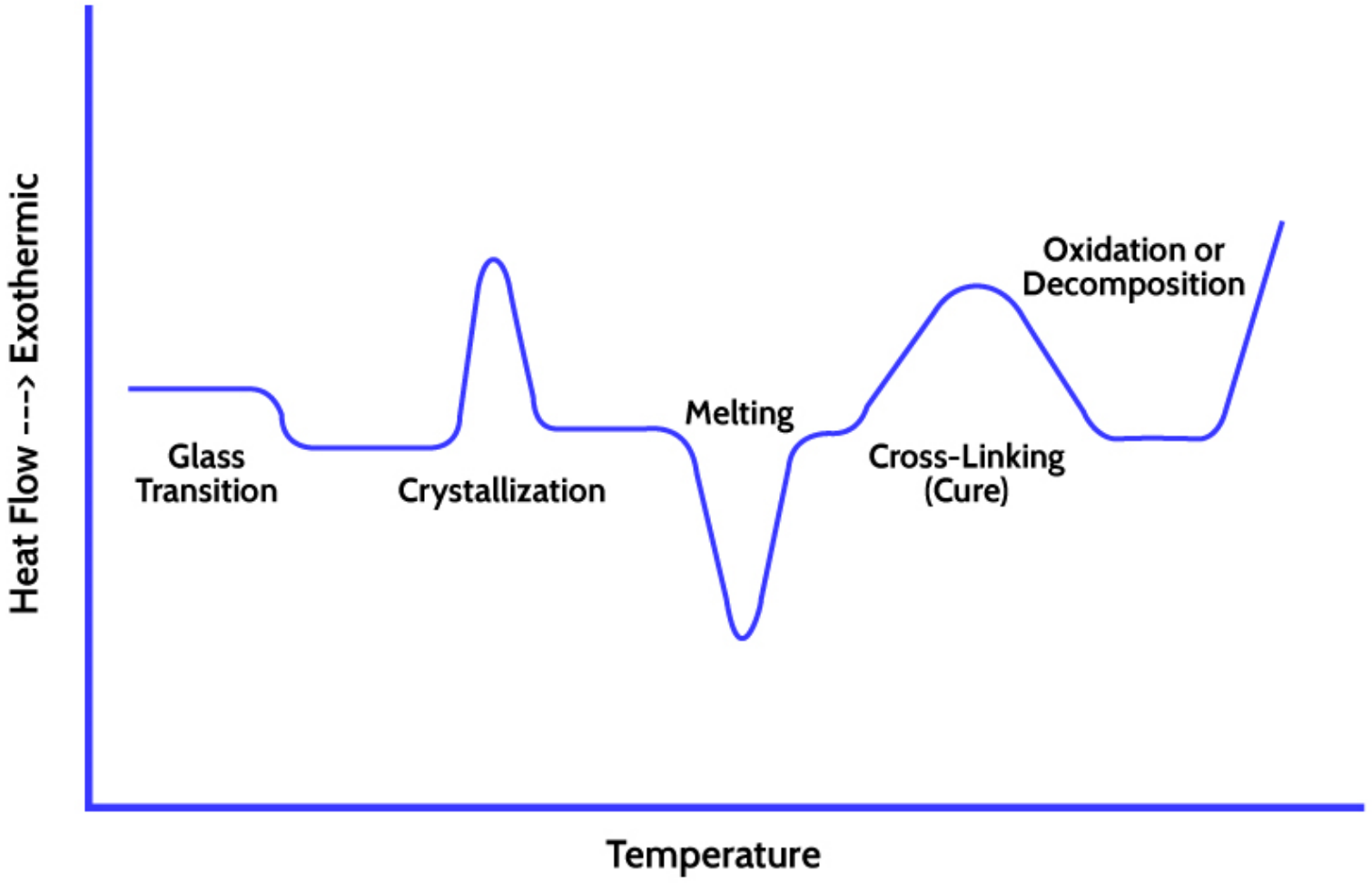

Schematic of various transition under DSC analysis

Ref: https://www.particletesting.com/thermal-analysis/

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- DSC is an accurate technique.
- Common applications are measurement of glass transition temperature, fusion temperature, predict the chemical reaction and oxidative stability, determination of curie point and crystallization temperature

Differential thermal analysis (DTA):

- It consists the measurement of heat change as a function of temperature or time.
- **Instrumentation:** Use one furnace around 500 °C in which both the specimen and reference temperature are simultaneously controlled.

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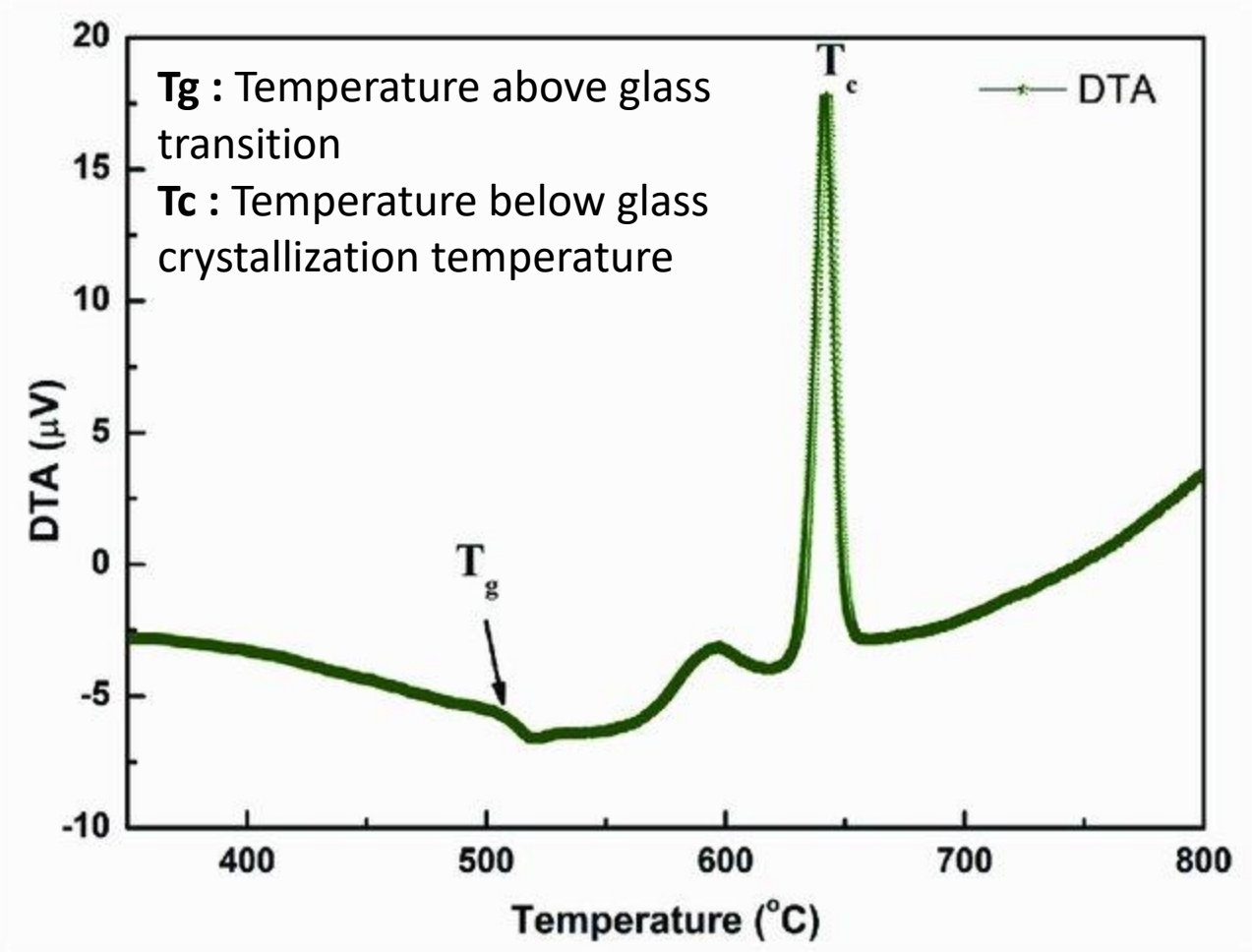

Schematic DTA analysis for glass sample

Ref: Munishwar et al., 2018, *Optical Materials*, *86*, 424-432.

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- DTA is less accurate technique as compare to DSC and TGA.
- Common applications of DTA technique are almost same as in case of DSC technique such as predicting the chemical reaction and oxidative stability, measurement of glass transition temperature, fusion temperature, curie point and crystallization temperature.

Thermo gravimetric analysis (TGA)

- It consists the measurement of mass change as a function of temperature or time by means of kinetic value determination and/or determination of of moisture, diluent etc. loss rate using weight loss method.

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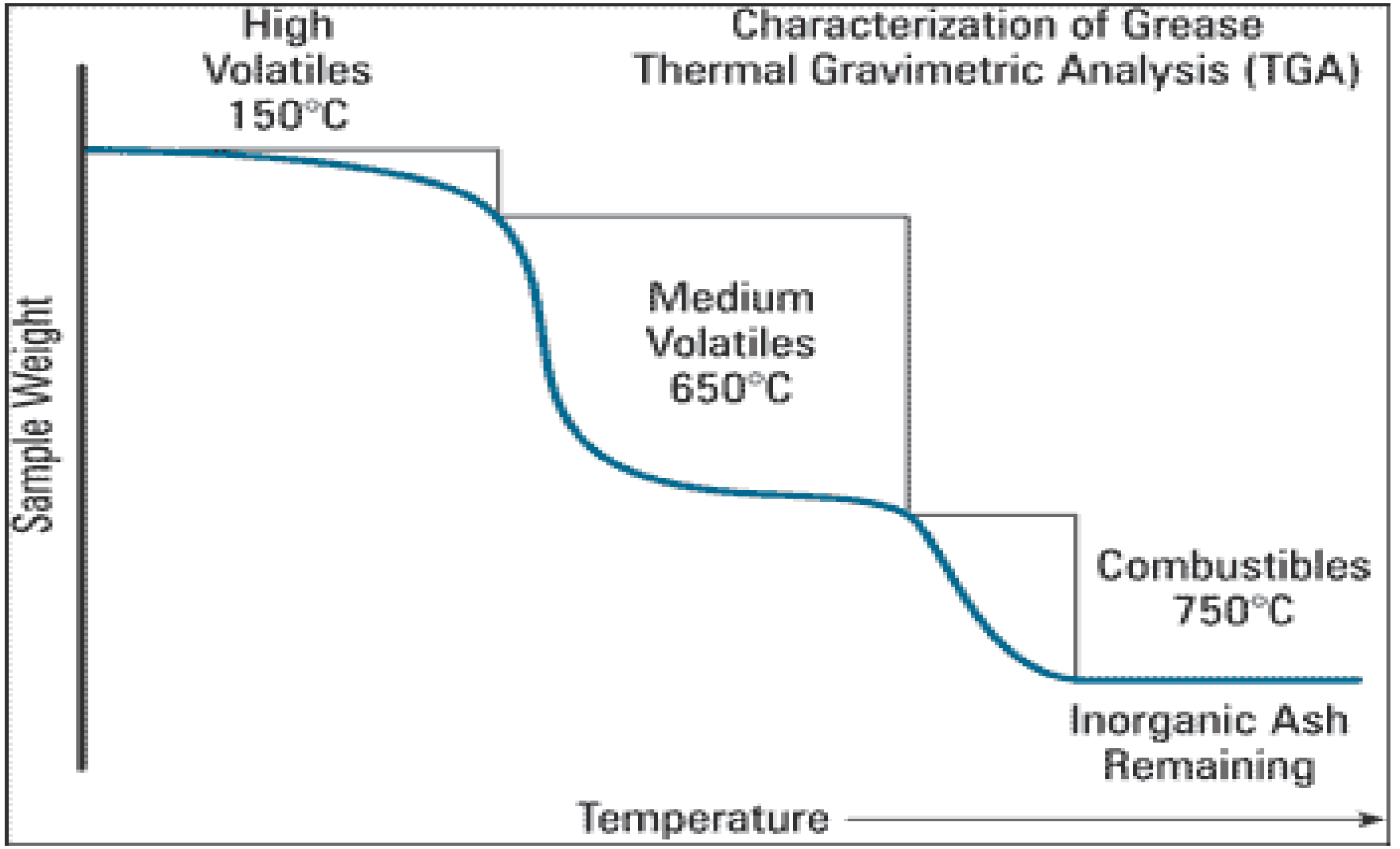

Schematic of TGA Grease (Greases, like oils, contain a variety of additives) Profile

Ref: https://www.machinerylubrication.com/Read/296/grease-analysis

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- **Instrumentation:** Use one furnace around 1000 °C with linear heating rate of (5-10) °C/min. Most of the TGA techniques are followed isothermal monitoring by controlling the both specimen and reference temperature change to zero.
- More accurate technique and overcome the difficulties of overlapping of both endothermic and exothermic values as by weight loss method.
- Common applications of TGA technique are determination of moisture loss, volatile materials loss, and oxidation temperature of specimen etc. Further TGA technique is used for decomposition and compositional analysis of multi-component system.