MSE310 Lecture 2

Thermogravimetric Analysis (TGA)

The TGA technique measures the mass of a sample as it is heated, cooled or held at a constant temperature in a defined atmosphere. Thermogravimetric analysis (TGA) is ideal for characterizing the thermal properties of materials such as plastics, elastomers and thermosets, mineral compounds and ceramics as well as for chemical and pharmaceutical products.

A technique in which the mass of a substance is measured as a function of temperature while the substance is subjected to a controlled temperature programme.

Principle of Operation

A TGA analysis is performed by gradually raising the temperature of a sample in a furnace as its weight is measured on an analytical balance that remains outside of the furnace. In TGA, mass loss is observed if a thermal event involves loss of a volatile component.

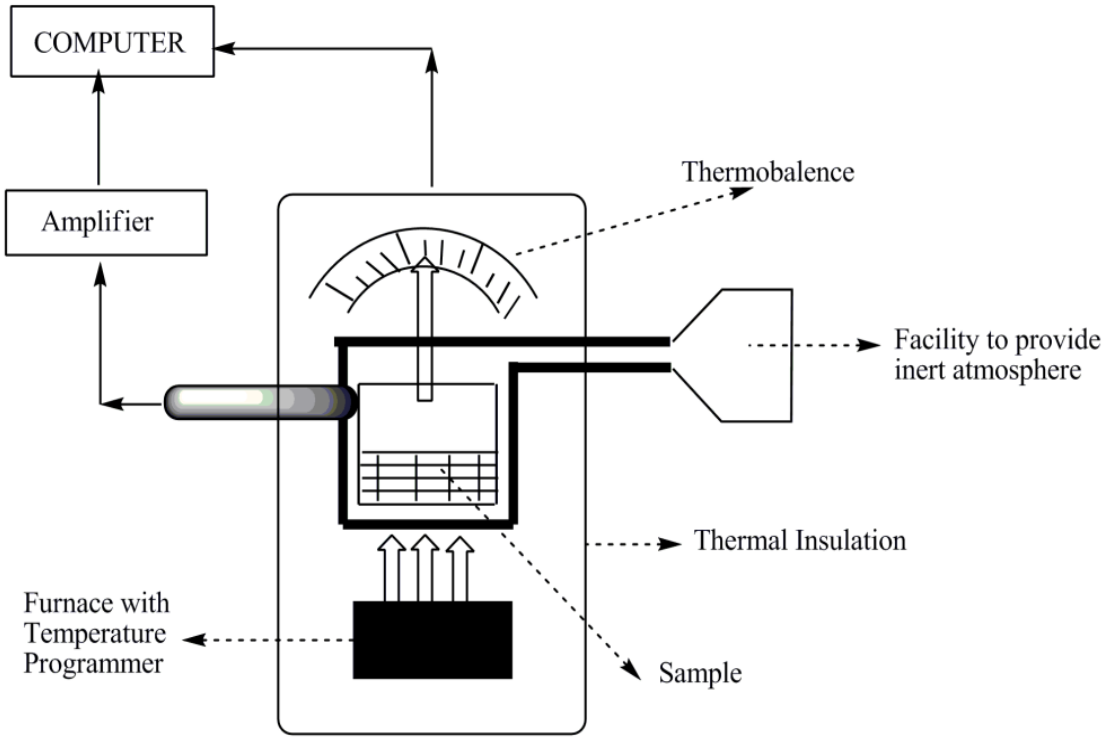
In TGA mass of the substance is continuously monitored as the temperature is linearly increased. TGA apparatus is used to measure TGA. It can scan over a wide range of temperature (25 – 1200 ºC).

The main components of TGA apparatus:

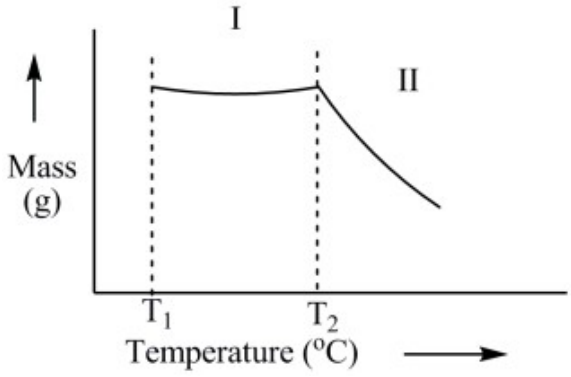
1. A high precision thermobalance. A thermobalance is used for weighing a sample continuously while it is being heated or cooled.
2. Furnace with temperature programming facility. The heating rate is the rate of temperature increase, which is customarily quoted in degrees per minute (on the Celsius or Kelvin scales). The heating or cooling rate is said to be constant when the temperature/time curve is linear.
3. Facility for providing inert atmosphere (like N_2 gas) Or Oxidizing environment.The preparation and manipulation of certain inorganic compounds requires the use of an **inert atmosphere**. **Inert** often implies that the **atmosphere** is free from water and oxygen. The reactions are carried out in a dry **nitrogen atmosphere**. **Inert gases** are **used** generally to avoid unwanted chemical reactions degrading a sample. These undesirable

chemical reactions are often oxidation and hydrolysis reactions with the oxygen and moisture in air.

4. A computer which can collect, store and process data – like plotting the graph.



The mass vs Temperature plot is called Thermogram.



Region I: The horizontal indicates the region where there is no mass change. i.e from T_1 to T_2 the material is thermally stable.
Region II: The graph declines indicating a weight loss (weight loss can be due to dehydration, decomposition, sublimation, desorption, Evaporation etc. [Weight gain during Metal Oxidation, adsorption etc]

The TGA Technique

- TGA measures the amount of weight change of a material, either as a function of increasing temperature, or isothermally as a function of time, in an atmosphere of nitrogen, helium, air, other gas, or in vacuum.
- Inorganic materials, metals, polymers and plastics, ceramics, glasses, and composite materials can be analyzed.
- Temperature range from 25°C to 900°C routinely. The maximum temperature is 1000°C.
- Sample weight can range from 1 mg to 150 mg. Sample weights of more than 25 mg are preferred, but excellent results are sometimes obtainable on 1 mg of material.
- Weight change sensitivity of 0.01 mg.
- Samples can be analyzed in the form of powder or small pieces so the interior sample temperature remains close to the measured gas temperature.

Working of TGA

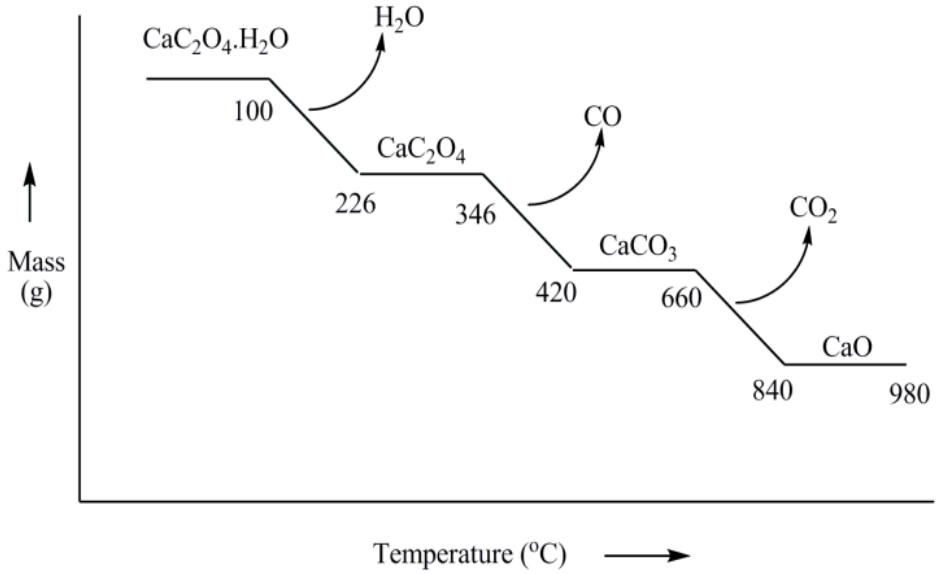
In **thermogravimetric analysis**, the change in weight in relation to a change in temperature in a controlled environment is measured. **Heat** is used in **TGA** to force reactions and physical changes in materials.A thermocouple is used to accurately control and measure the temperature within the oven.

The thermogravimetric analysis instrument usually consists of a high-precision balance and sample pan. The pan holds the sample material and is located in a furnace or oven that is heated or cooled during the experiment. A thermocouple is used to accurately control and measure the temperature within the oven. The mass of the sample is constantly monitored during the analysis. An inert or reactive gas may be used to purge and control the environment. The analysis is performed by gradually raising the temperature and plotting the substances weight against temperature. A computer is utilized to control the instrument and to process the output curves (temperature vs. weight).

Applications of Thermogravimetry

- Thermal stability of substance
- Decomposition mechanism of inorganic salts.

Consider the TGA-thermogram of calcium oxalate monohydrate $CaC_2O_4.H_2O$.



- From the graph it is seen that calcium oxalate monohydrate $CaC_2O_4.H_2O$ is stable upto 100ºC.
- Removal of water begins at 100ºC and gets completed at 226ºC.
- The horizontal portion 226-346ºC shows the thermal stability of CaC_2O_4.
- Decomposition of CaC_2O_4 to $CaCO_3$ with the elimination of CO occurs in the temperature range 346- 420ºC.
- The 420-660ºC horizontal portion gives the thermal stability of $CaCO_3$.
- Above 660ºC $CaCO_3$ decomposes to CaO and CO_2.
- From the mass difference the mechanism of decomposition can be deduced.

- Determines temperature and weight change of decomposition reactions, which often allows quantitative composition analysis. May be used to determine water content.
- Allows analysis of reactions with air, oxygen, or other reactive gases .
- Can be used to measure evaporation rates, such as to measure the volatile emissions of liquid mixtures.

- Allows determination of Curie temperatures of magnetic transitions by measuring the temperature at which the force exerted by a nearby magnet disappears on heating or reappears on cooling.
- Helps to identify plastics and organic materials by measuring the temperature of bond scissions in inert atmospheres or of oxidation in air or oxygen.
- Used to measure the weight of fiberglass and inorganic fill materials in plastics, laminates, paints, primers, and composite materials by burning off the polymer resin. The fill material can then be identified by XPS and/or microscopy. The fill material may be carbon black, TiO_2, $CaCO_3$, $MgCO_3$, Al_2O_3, $Al(OH)_3$, $Mg(OH)_2$, talc, Kaolin clay, or silica, for instance.
- Can measure the fill materials added to some foods, such as silica gels and titanium dioxide.
- Can determine the purity of a mineral, inorganic compound, or organic material.
- Distinguishes different mineral compositions from broad mineral types, such as borax, boric acid, and silica gels.

These notes have been prepared with the help of Materials Characterization books by Sam Zhang, Lin Li, Ashok Kumar.