Lecture (3)

Thermogravimetric analysis (TGA)

Thermogravimetric analysis or thermal gravimetric analysis (TGA) is a method of thermal analysis in which changes in physical and chemical properties of materials are measured as a function of increasing temperature (with constant heating rate), or as a function of time (with constant temperature and/or constant mass loss). (Note that mass is a measure of the amount of matter in a sample, whereas weight refers to the effect of the gravitational force on a mass), A thermobalance is a combination of a suitable electronic microbalance with a furnace, a temperature programmer and computer for control, that allows the sample to be simultaneously weighed and heated or cooled in a controlled manner, and the mass, time, temperature data to be captured. The balance should be in a suitably enclosed system so that the nature and pressure of the atmosphere surrounding the sample can be controlled (see Figure 1). Care is usually taken to ensure that the balance mechanism is maintained at, or close to, ambient temperature, in an inert atmosphere. The furnace should (i) be non-inductively wound.

(ii) be capable of reaching 100 to 200°C above the maximum desired working temperature.

(iii) have a uniform hot-zone of reasonable length.

(iv) reach the required starting temperature as quickly as possible, (i.e. have a low heat capacity).

(v) not affect the balance mechanism through radiation or convection.

The sensitivity of a thermobalance and the maximum load which it can accept, without damage, are related. Typical values are maximum loads of 1 g and sensitivities of the order of 1 μ m.

The information obtained from TGA

1)physical phenomena, such as second-order phase transitions, including vaporization, sublimation, absorption, adsorption, and desorption.

2)chemical phenomena including chemisorptions, decomposition, and solid-gas reactions (e.g., oxidation or reduction).



Fig. (1)schematic thermobalance of (TGA).

The Sample

Although solid samples may be nominally of the same chemical composition, there may be considerable differences in their behaviour on heating. These differences arise from structural differences in the solid, such as the defect content, the porosity and the surface properties, which are dependent on the way in which the sample is prepared and treated after preparation. For example, very different behavior will generally be observed for single crystals compared to finely ground powders of the same compound. In addition to the influence of defects on reactivity, the thermal properties of powders differ markedly from those of the bulk material.