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Note

A HIGH TEMPERATURE THERMOGRAVIMETRIC ANALYZER

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Thermogravimetric analyzers (TGA) have now reached a high state of sophistication and numerous commercial units are available. Wendlandt [1] summarized various types of thermogravimetric analyzers and their respective capabilities. Most of the units could be used below the temperature limit of 1600°C in various atmospheres. Gardner and Swiss [2] described a unit that could be used in oxidizing or reducing atmospheres up to 1700°C. A high alumina muffle tube was used to separate the sample from a graphite resistance furnace. Two types of metals such as platinum (oxidizing atmosphere) and molybdenum (reducing atmosphere) were used to make sample boats. In continuing the effort to extend the temperature limit of the TGA, a unit was assembled with a design similar to that of Gardner and Swiss' [2]. The new TGA unit allows the operating temperature as high as 2500°C in inert or reducing atmospheres and 1800°C in oxidizing atmospheres. The TGA has been successfully used for high temperature kinetic studies of solid-decomposition, gas–solid and solid–solid reactions.

APPARATUS

The TGA apparatus consists of a high temperature furnace, an electrobalance, sample boat assembly, gas manifold, and data readout instruments (Fig. 1).

The furnace is an Astro Model 1000 with graphite resistance heating elements. In inert or reducing atmospheres, the instrument can be operated continuously up to 2500°C. Heating rates for these conditions can be as high as 100°C min^{-1}. A high purity (99.9%) recrystallized alumina muffle tube is installed to protect the graphite elements when oxidizing atmospheres are utilized. When the muffle tube is in place, heating rates are recommended not to exceed 250°C h^{-1}, especially in the range 1000–1400°C. The maximum working temperature then is 1800°C. (The maximum working temperature for oxidizing atmospheres can be extended to 2200°C if a high purity stabilized zirconia muffle tube is used instead.) The sample thermocouple tip is in close proximity of the sample to insure accurate sample temperature

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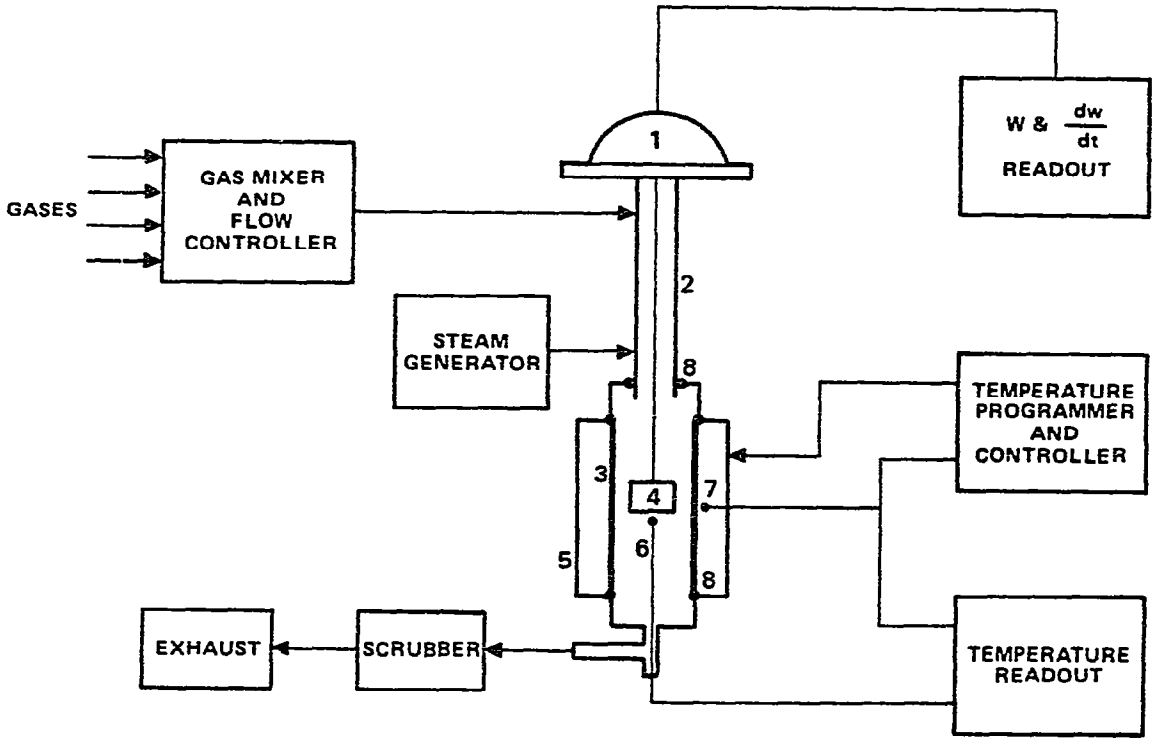

Fig. 1 Schematic diagram of the TGA assembly. 1, Electrobalance; 2, hangdown tube; 3, muffle tube; 4, sample boat assembly; 5, furnace; 6, sample thermocouple, 7, control thermocouple; 8, O-ring seal.

measurement. Type S thermocouple is used to measure the sample temperature up to 1600°C and a W–5% Re vs. W–26% Re thermocouple is used above 1600°C. These thermocouples are enclosed inside a high purity alumina sheath. A curve-following temperature programmer controls the furnace temperature up to 2000°C using a boron/graphite thermocouple and up to 2500°C using a power control module.

Weight measurements are made with the Cahn 1000 electrobalance system. The electrobalance is positioned above the furnace and the two are connected with a 60 cm long glass hangdown tube. A 50 cm stainless steel rod hangs from the balance stirrup down the center of the glass tube and passes through a graphite-felt radiation shield. The sample boat assembly hangs from the bottom of this rod. Note that the electrobalance is protected from the furnace thermal radiation by both distance and shielding.

The sample boat assembly is detailed in Fig. 2. It consists of three parts: the rod, eyelet and sample boat. The rod is connected to the eyelet and steel rod via threaded coupling when using graphite. When the ceramic material is used, a simple retaining pin holds the rod and adjacent parts together. The eyelet is a rectangular frame upon which a cylindrical sample boat is mounted. The boat is held in place with a pin. Graphite of high purity is

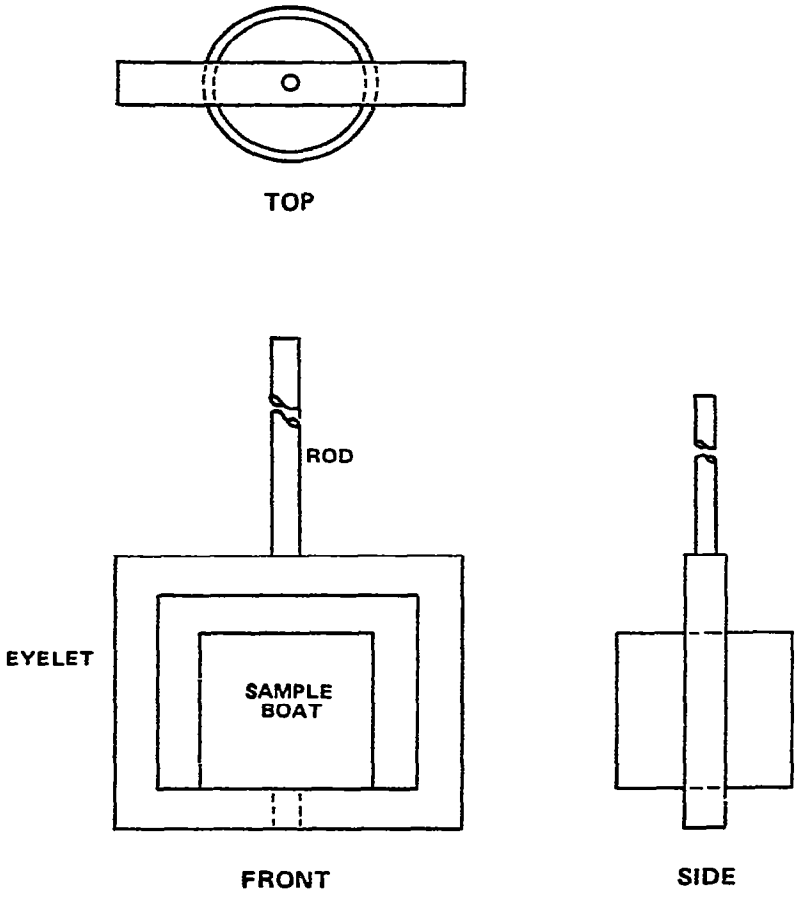

Fig. 2. TGA sample boat assembly.

used to make the boat assembly if reducing or inert conditions are required. High purity alumina is used to make the boat assembly if oxidizing conditions are required. The high alumina boat assembly is stable with respect to attack from silicon, boron, oxygen and lead at high temperatures, whereas platinum or molybdenum would be susceptible to attack under the same conditions.

Gas flow rate and composition are controlled with a Matheson multiple flow controller. A wide range of flow rates and a variety of gas compositions can be introduced into the TGA. Water vapor can be independently introduced into the TGA from a steam generator. The tube connecting the generator and the glass hangdown tube is heated and insulated to prevent steam condensation.

This TGA has accelerated the work on the kinetic studies of various high temperature reactions and the data have provided useful information for optimizing the pilot plant work. This unit will be computerized in the near future.

REFERENCES

1 W.W. Wendlandt, Thermochim. Acta, 7 (1972) 505.
2 R.A. Gardner and W.R. Swiss, Rev. Sci. Instrum., 44 (1973) 1428.