

Measurement of Reduction Process of Metal Oxides by TG Thermogravimetric Analysis

Recently, researchers have undertaken the task of developing many new materials. However, the complicated circumstances under which such new materials are used necessitate a solid grasp of various high-temperature gaseous reactions in order to evaluate durability and heat stability of the materials.

Thermogravimetric (TG) is a technique used to measure change in weight of a sample at high sensitivity as a temperature function, and is also effective in providing a reaction amount or the reactivity attained through the solid (liquid)-to-gaseous phase reaction between the sample and the atmosphere.

TGA, which employs a pending balance mechanism, allows for (1) independent adjustments of the sample and the balance mechanism atmosphere, and (2) easy replacement of gases using a vacuum pump. It also enables relatively simple measurements in various atmospheric conditions, including corrosive activated gases such as SO_2 , NH_3 and H_2 .

Reported herein are measurements of the reduction process of metal oxides in an H_2 atmosphere.

Figure 1 shows a gas channel of TGA. There are four gas inlet and outlet ports. Ports (2) and (4) are closed in a normal measurement; a gas is then introduced from Port (1), passes through a sample and is discharged from Port (3).

Since the complete replacement of atmosphere in the analyzer with H_2 was necessary to perform such measurements, Ports (2) and (3) were closed after the sample was set, and a vacuum pump was connected to Port (4). While introducing the Hydrogen from Port (1), the vacuum acted as an exhaust fan, pumping the other gas out. When the gas was fully replaced, Port (3) was opened, and measurement began.

The balance mechanism has to be protected during the measurement in corrosive gases. Thus, Port (4) was closed, and an inert gas such as N_2 or Ar was introduced from Port (1).

Simultaneously, a corrosive gas was introduced from Port (2), passed through the sample and discharged from Port (3). Here the atmosphere of sample was a mixture of the inert gas from Port (1) and the corrosive gas from Port (2). As flow of the gas from Port (1) increased, concentration of the corrosive gas around the sample reduced.

By arranging the gas channel of TGA-50, measurements in various atmospheres are possible.

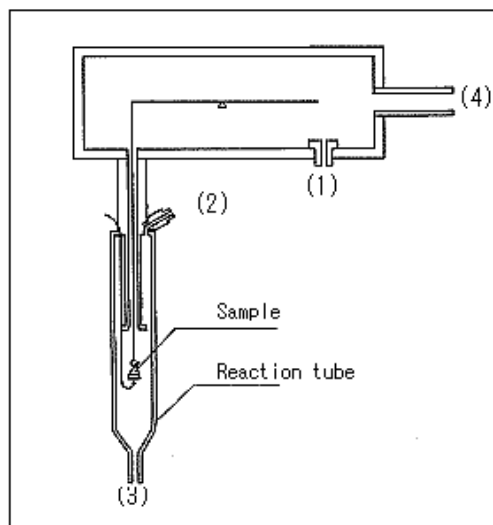


Fig. 1 Gas inlet/outlet ports of TGA-50

Measurement of metal oxides in H_2 Figures 3-8 show TGs and their differential curves in situations where several kinds of metal oxides were heated in the H_2 atmosphere. Based on such data, detailed analyses of temperatures and reduction processes should be feasible. Weight loss in the data applies to separate O_2 amounts (%).

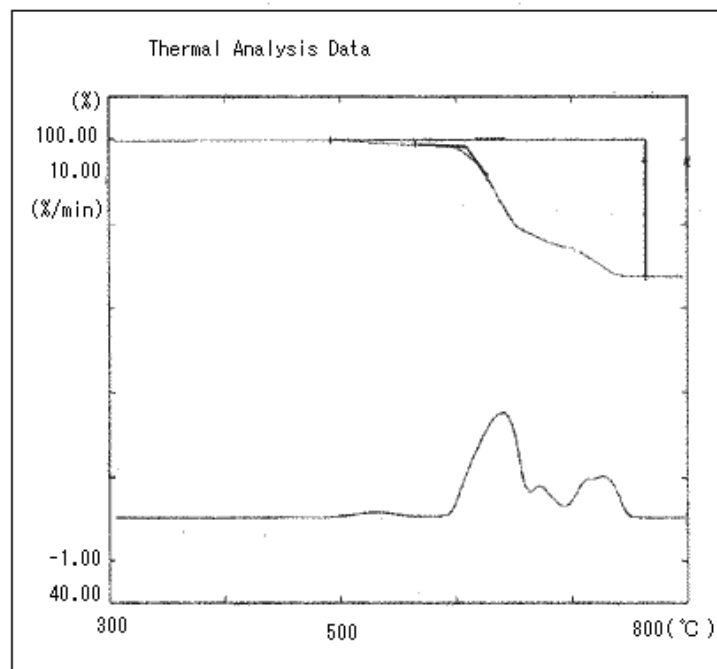


Fig.2 tungsten oxide (WO_3)

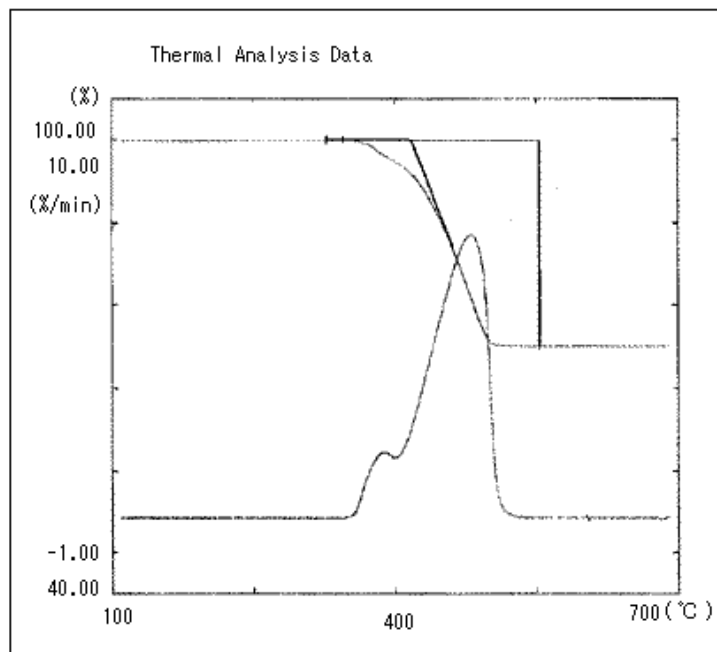


Fig.3 iron oxide (Fe_2O_3)

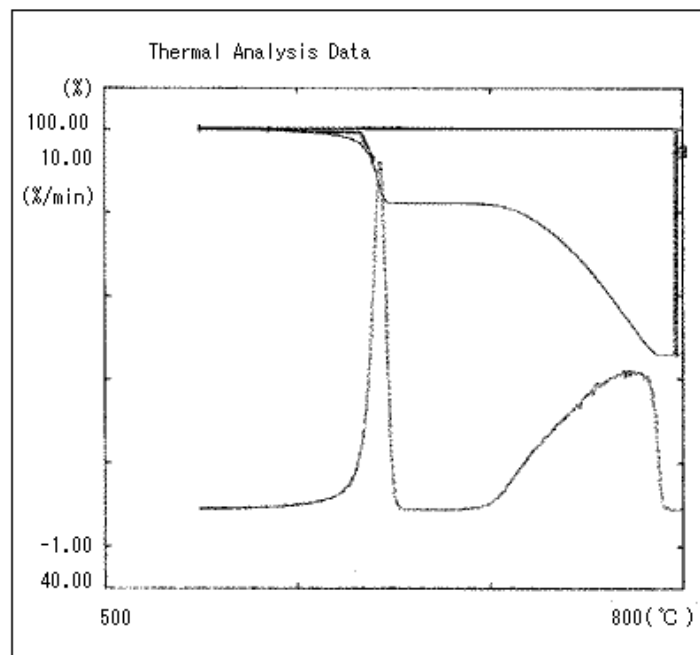


Fig.4 molybdenum oxide (MoO_3)

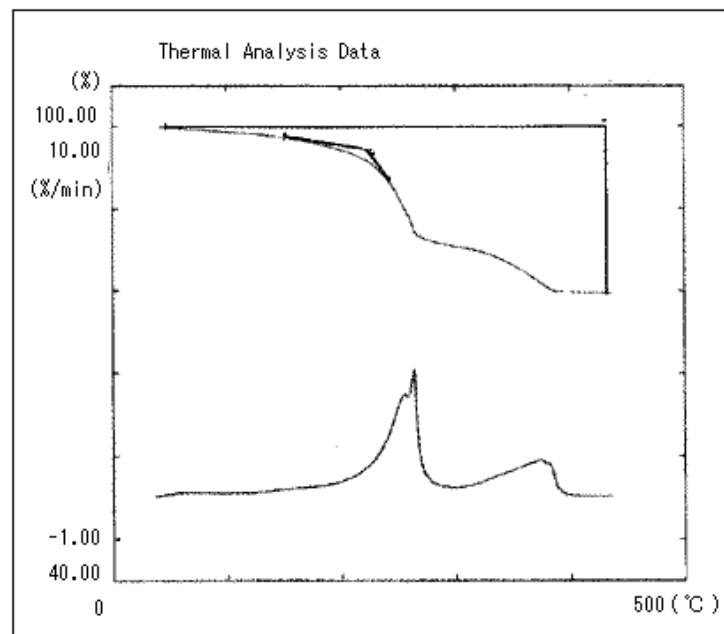


Fig.5 manganese oxide (MnO₂)

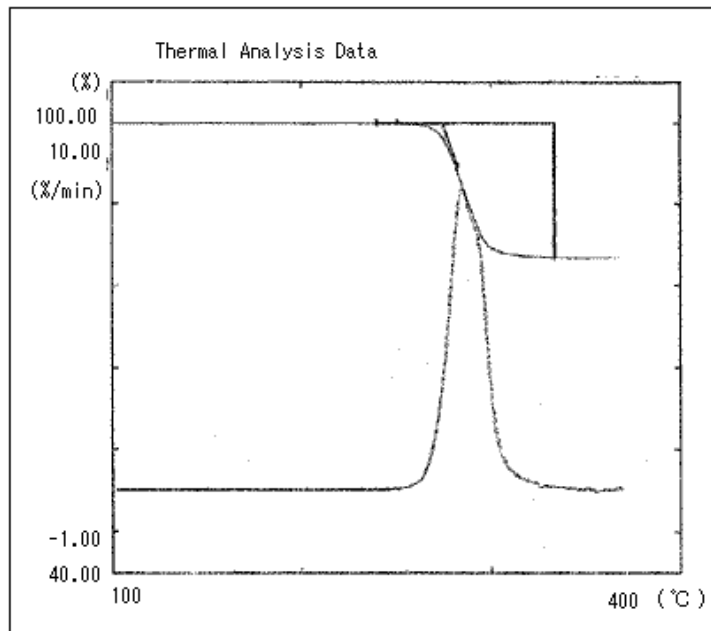
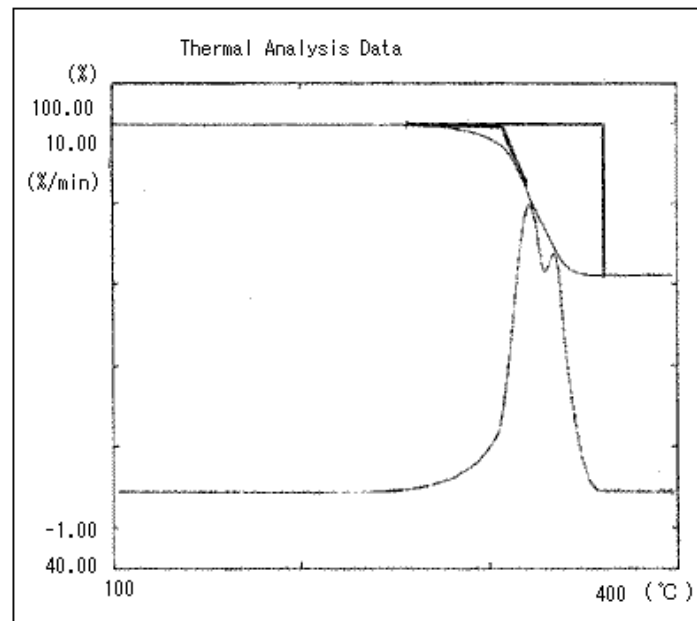


Fig.6 copper oxide (CuO)

Fig.7 cobalt oxide (Co₃O₄)

* Please be advised that data obtained before the implementation of the current Weights and Measures Law may be presented in terms of gravimetric unit.



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