FACTORS AFFECTING THE EXPERIMENTALLY RESOLVED SHAPES OF TG CURVES

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From a theoretical consideration concerning the factors affecting the experimentally resolved shapes of thermogravimetric curves, the possible way to increase the accuracy of the thermogravimetric measurements were discussed.

Keywords: kinetics, shapes of TG curves

Introduction

Although the present thermogravimetric (TG) apparatuses have achieved a very sophisticated state [1-7], the information obtained from the experimentally resolved shapes of TG curves is still at the level of qualitative characterization of the reaction temperature and rate [8]. On the other hand, as is the case of the use of TG curves as a source of kinetic data, the accuracy of the TG measurements have always been desired [9]. The accuracy of TG data is not only limited by the sensitivity of the balance, but by the magnitude of spurious effects due to the conditions of the experiment. Further sophistication would be enabled by solving the problems connected within unified concept of the fluid density gradients convenient to describe melts behaviour during single crystal growth [10], as adopted for discussion in the present paper.

Problems

Gradients in the atmospheric density and temperature

In the closed system shown in Fig. 1, gradients in both the atmospheric density, ρ, and temperature, T, should be arose. These gradients are also affected by
the temperature of the system. In the case of $\rho <<$, e.g., in vacuum, both the gradients and their temperature dependence can be ignored. On the other hand, the correct measurement of sample mass is disturbed by these factors and shift of TG base line would be observed, when the $\rho$ is large enough. Typical example is the case of measurements of crystal growth from fluid [11].

**Atmospheric flow caused by heating**

When the closed system were heated from outside, a kind of heat flux, $\dot{q}$ is arose within the system (Fig. 2(a)). In the gaseous atmosphere, the heat flow is governed by the conduction or convection. When $\rho$ is large enough, this is accompanied by the flow of atmosphere, $f$, as shown in Fig. 2(b).

In addition, the gradients in $T$ and $\rho$ would be also observed in the vertical direction (Fig. 3). These gradients introduce the mixing flow shown in Fig. 3. It

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**Fig. 1** Density, $\rho$, and temperature, $T$, gradients within the closed TG system

**Fig. 2** (a) The heat flow, $\dot{q}$ caused by the furnace heating, $Q$, and (b) consequent flow of atmosphere $f$