Materials processing in magnetic levitation furnaces

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Abstract

The magnetic levitation for diamagnetic materials provides a quasi-microgravity condition and enables containerless melting for materials synthesis. Laser furnaces have been developed and containerless melting experiments under the magnetic levitation condition were performed so far. In order to perform a homogeneous heating of a levitating sample, which was difficult in laser furnaces, a new magnetic levitation furnace has been developed using an electric furnace. Spherical samples of a cycloolefin polymer were obtained by using this furnace under the magnetic levitation condition.

Keywords: Magnetic levitation; Microgravity; Containerless melting; Electric furnace

1. Introduction

Diamagnetic materials receive a repulsive force in a magnetic field gradient. When this magnetic force balances with the gravitational force, the materials can be levitated [1–5]. The magnetic levitation is considered to be a quasi-microgravity condition because the counterbalance between the magnetic force and the gravity holds for each molecule constituting the material. Thus, the magnetic levitation enables containerless melting or containerless crystal growth for materials synthesis. Such a containerless technique provides a clean environment free from contamination due to a container. Suppressing uncontrollable heterogeneous nucleation leads to easily produce a supercooled or supersaturated state. In the case of the magnetic levitation, a magnetic orientation effect is expected additionally for materials with anisotropic susceptibility. Hence, materials processing under the magnetic levitation conditions is considered to be a new technique for materials synthesis.

In order to perform containerless melting, it is necessary to develop a special furnace available under the magnetic levitation condition. We call such an apparatus a magnetic levitation furnace. The design of the magnetic levitation furnace requires several specific features. The first is to heat samples in high magnetic fields. Because the furnace is placed in a hybrid magnet generating fields up to 30 T, it is necessary to operate satisfactorily in such an environment. The second is to install the furnace in a confined narrow space in a room temperature bore of a hybrid magnet, which is usually 52 or 32 mm in diameter. The third is to observe the behavior of a levitating sample in the furnace. It is indispensable to check whether the sample actually levitates or not in heating and cooling processes. If the magnetic susceptibility of the sample changes at melting or solidification, the sample falls down or flies away. We have developed various magnetic levitation furnaces for the containerless melting experiment under the magnetic levitation condition and have performed the experiments by using them so far.

2. Magnetic levitation furnace

2.1. Laser furnace

Laser furnaces have advantages when they are used for containerless melting under the magnetic levitation condition. A laser beam is not affected by magnetic fields, and the local irradiation just on a sample allows the use of observation systems including a CCD camera near the sample. We have developed two kinds of laser furnaces so far. One is the CO2 laser furnace, which uses an infrared laser beam from the CO2 laser to heat a levitating sample. A cubic glass has become a complete sphere through melting and cooling under
the magnetic levitation condition with the CO$_2$ laser furnace [6]. The other is the YAG laser furnace. In the YAG laser furnace, the laser beam is shaped to a ring by a cone mirror at the top part of the furnace. The beam is then reflected by a concave ring mirror and focused on the sample at the radial center of the furnace. Thereby, the sample is irradiated homogeneously when the sample is smaller than the beam width. In the case of the containerless melting of paraffin with the YAG laser furnace, the magnetic orientation of paraffin molecules was observed after solidification [7]. The homogeneous heating reduces the Marangoni convection caused by the surface tension gradient of the sample. As a result, the magnetic orientation occurred during the solidification process.

Laser furnaces, however, have also disadvantages in heating a levitating sample. When a sample does not absorb the laser light, the sample cannot be heated. If a sample is heated inhomogeneously and thermal conductivity of the sample is very small, the sample is partially heated just on the area irradiated by the laser light. In such a case, the temperature of the irradiated part of the sample rises rapidly and only thermal decomposition occurs before the whole sample melts. Such a behavior was observed when a piece of PMMA (polymethyl methacrylate), one of clear plastics, was heated by the CO$_2$ laser furnace.

2.2. Electric furnace

Electric furnaces for a cryogen-free superconducting magnet or a hybrid magnet [8] have been used for high field heat-treatment for high-$T_c$ superconducting materials and various magnetic materials [9–11] or DTA analysis in magnetic fields [12]. An electric furnace with an outer diameter of 50 mm produces high temperature up to 1200 °C in magnetic fields. A heater is made of platinum wires of 1 mm diameter. The outer furnace wall is cooled by water so as to eliminate thermal inputs to the magnet. The temperature of a sample is monitored and controlled using a thermocouple of Pt–Rh. Total height of the furnace is about 1250 mm.

For the use of an electric furnace as a magnetic levitation furnace, however, there was a problem how to observe the levitating sample in heating process. We intended to solve this problem by using a heatproof bore scope (Schöllly Fiberoptic GmbH). The bore scope with optical relay lenses cooled by a water jacket can be used up to 800 °C. Fig. 1 shows the schematic illustration of the electric furnace with the heatproof bore scope combined with a hybrid magnet. It is possible to observe a containerless melting process in the electric furnace by inserting the bore scope into the furnace from a Wilson seal port on the top end.

3. Application of a magnetic levitation electric-furnace to polymer processing

We performed containerless melting experiments with a magnetic levitation electric-furnace. A sample was a clear plastic, a cycloolefin polymer; ZEONEX® (ZEON Corp.). The sample was an elliptic cylinder with the major axis, the minor axis and the height of 3, 2 and 3 mm, respectively. The magnetic susceptibility, $\chi_m$, was $-10.63 \times 10^{-9}$ m$^3$/kg estimated by a SQUID magnetometer, and this value requires the magnetic force field, $B(\partial B/\partial z)$, of $-1159$ T$^2$/m for the levitation. The melting point of the sample is not clear but
the glass transition temperature is 138 °C. In order to achieve the magnetic levitation of ZEONEX, we used a cryogen-free hybrid magnet (27.5 T-CHM) with a room temperature bore of 52 mm [13].

A piece of ZEONEX sample almost levitated at 91 mm above the center of the magnet, when the center field became 17.0 T and calculated magnetic force field value was -1161 T^2/m at that position. The sample was heated in a N2 gas atmosphere. The heating process was shown in Fig. 2. The temperature was raised to 226 °C at a rate of 15 °C/min at a central field of 17.0 T. After the sample began to melt around 150 °C, the magnetic field was adjusted to 18.0 T (B(∂B/∂t) = -1338 T^2/m). Fig. 3 shows the top view of the heating process. The sample melted in the situation of slightly contacting with a sample holder. The sample solidified in spherical shape as is shown in Fig. 4(a). Sphericity was determined by measuring the diameter of the sample in two directions. The diameter of the horizontal direction in Fig. 4(a) was 3.212 ± 0.007 mm and the diameter in the direction tilted by 45° from the horizontal direction was 3.237 ± 0.017 mm. The coincidence of diameter between two directions means that the melt of the sample was under the microgravity condition. Fig. 4(b) shows a sample melted and solidified in

![Fig. 3. Melting process of a piece of ZEONEX. The temperature and the magnetic field are: (a) 18 °C, 17.0 T, (b) 147 °C, 17.0 T, (c) 213 °C, 18.0 T and (d) 226 °C, 18.0 T, respectively.](image1)

![Fig. 4. Samples melted and solidified (a) under the magnetic levitation condition and (b) in zero field.](image2)
zero magnetic field under the same heat-treatment condition and its shape was not spherical but ellipsoidal.

Small bubbles were observed in the solidified samples. In order to get an appropriate quality sample for the optical application, heating and cooling processes must be optimized.

4. Conclusions

We developed a new magnetic levitation furnace consisting of an electric furnace and a heatproof bore scope with a hybrid magnet. Homogeneous heating of a levitating sample in this furnace is available for containerless melting of polymers. We have succeeded in preparing a spherical sample of cycloolefin polymer. Developing the new furnace has made it possible to perform a containerless melting experiment for various kinds of materials.

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