Test Equipment

Development of a pressure-driven micro-rheometer

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Abstract

A pressure-driven micro-rheometer was developed. It uses 80 mg of material to measure the viscosity of polymer melts at shear rates ranging from 10 to several thousands s^{-1}. The maximum shear rate can be extended to several $10^4 \, s^{-1}$ with 200 mg of sample. The main part of the rheometer consists of two sample reservoirs connected through a slit channel ($H = 0.1$ mm, $W = 1$ mm, $L = 5$ mm) and two pistons. The double piston arrangement enables one to use the same material repeatedly by the reciprocating flow of the polymer melt from one reservoir to the other. In addition, by using a very thin slit channel, the viscosity of polymer melts can be measured over a wide range of shear rates whilst using only a small quantity of material. Measured viscosity was in good agreement with that by a capillary rheometer, and it was found that slip was negligible in the slit die used in this study.
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1. Introduction

The capillary rheometer and the slit rheometer based on a pressure-driven flow are the most commonly employed apparatuses for measurement of the viscosity of polymer melts, since they provide simple measurement and high shear rate range [1]. The rheometers based on drag flow provide a shear rate not much beyond $10 \, s^{-1}$, whereas higher levels are common in polymer processing equipments, such as injection molding, extrusion molding, transfer molding, etc.

In a typical test by a capillary rheometer, polymer samples of several tens of grams are needed. Often, a rheometer that consumes a small amount of material is needed when only a limited quantity is available. This is true for very expensive materials such as high-performance engineering plastic, polymer composite containing carbon nanotubes, etc. Additionally, many new materials are available in limited quantities since they are initially synthesized in small amounts. Here, we define a micro-rheometer as a rheometer that uses polymer samples of several tens of micro-grams.

Several attempts have been made to use a drag-flow-type rheometer as a micro-rheometer. Clasen and Mckinley built a flexure-based micro-gap rheometer consisting of miniaturized sliding plates [2]. They could measure the viscosity of polymer melt and polymer solution with samples of volume $<10 \, \mu L$ using optical plates 25 mm^2 in area with gaps ranging from several tens of μm to 200 μm. Soga et al. [3] used a similar principle in the

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Mckinley device. In both the cases, the alignment accuracy for the two plates is crucial as the gap distance decreases to micrometer scale. Since both the researches employ optical interferometry to monitor and control the spacing between the two plates, the equipment becomes very sophisticated, and the materials for the plates need to be transparent. It is well known that the physics of the confined system is much affected by the boundary between the polymer and the solid wall. For example, the slippage of polymer melts on the wall becomes considerable in a confined system, and is affected by the wall materials [4,5]. Therefore, the rheological information obtained from the optical plates may not be suitable to describe what really happens in polymer processing where most boundaries consist of polymer melts and metal solid. In addition, the maximum shear rate attained is limited by the inertial effect ($\sim 10\,\text{s}^{-1}$ in bulk systems, and $\sim 200\,\text{s}^{-1}$ in most in confined systems) in drag-type rheometry.

Most recently, Bur and Migler [6] introduced a multi-sample micro-rheometer based on pressure-driven channel flow. The driving force for the rheometer is pressurized nitrogen gas that forces polymer melt into a thin slit of $100\,\mu\text{m}$ height, maintaining the reservoir at constant pressure. The required sample size is between 50 and 100 mg. In order to obtain the flow rate, they measured the velocity of the flow front flowing through the slit die by deploying a video camera for obtaining a motion picture. Therefore, one side of the slit die must be a transparent material (sapphire). It is like an isothermal and isobaric injection molding process. Basically, the rheometer is operated in the unsteady state, though it can be regarded as a steady state due to the high Reynolds number of the system at the low shear rate. As the length of polymer melt in the slit die increases, the flow rate decreases because the pressure in the reservoir is maintained at a constant value. Therefore, the shear stress as a function of shear rate can be obtained with one run. However, the image analysis with a motion picture is very tedious and time consuming. They could measure the viscosity of polymer melts in the shear rate range $1\text{–}10\,\text{s}^{-1}$, which is somewhat low for a pressure-driven rheometer. To increase the maximum shear rate, a higher pressure in the sample chambers is needed. However, it may not guarantee a steady-state condition in the system, due to the increased Reynolds number.

Recently, we have built a micro-rheometer based on pressure-driven flow, and present its features in this paper.

2. Experimental

2.1. Materials

Polymers used were polystyrene (PS) from Cheil Industries Inc. (trade name: HF2660, $M_n = 120,000\,\text{g/mol}$), and linear low density polyethylene (LLDPE) from the Dow Chemical Company (trade name: EG8100, melt flow index $= 1.0\,\text{g/10 min}$).

2.2. Design and operation of the micro-rheometer

We designed a micro-rheometer which has the following features: (1) the required sample size is about $80\,\mu\text{L}$, equivalent to 80 mg when $\rho = 1.0\,\text{g cm}^{-3}$. (2) The shear rate attained ranges from ten to several thousands s^{-1}, to attain the target (2), a rheometer based on pressure-driven flow is the only choice. To accomplish target (1) the scale of the flow channel must be reduced to 1/10th of that of a commercial instrument. It is practically impossible to make a capillary die of 0.1 mm in diameter, hence we chose a slit die of 0.1 mm in height, 1 mm in width and 5 mm in length.

In a commercial instrument, the flow rate is obtained from the speed of the piston (U_p), corresponding to flow rate, $Q_p = U_p \pi R_p^2$, where R_p is the radius of the piston. In a micro-rheometer, the leakage-flow rate (Q_L) through the clearance between the barrel and the piston may be comparable to the flow rate through a die as the size of the channel decreases to micrometer scale. It is not assured that the piston can push all of the polymer melt in the reservoir through the die. In this case, the flow rate should be obtained from $Q_p - Q_L$, or the flow rate must be directly measured. To solve this problem, we directly measured the flow rate of polymer melt flowing through the slit die by the method explained below.

A schematic design of the micro-rheometer is shown in Fig. 1. It consists of a heated cylindrical metal block (#4 in Fig. 1) having two cylindrical cavities (hereafter denoted as a reservoir, #7 and#8 in Fig. 1) connected through a slit channel (#6), two cylindrical pistons (#1 and #2), a bottom plate (#5) and a dial gage (#3) which indicates the position of the ascending piston with a resolution of $0.5\,\mu\text{m}$. The bottom plate has a rectangular groove.