Sliding Plate Rheometer and Its Applications

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1. Introduction

Rheology †, a study of flow and deformation of matter, described the interrelation between force, deformation, and time. It is a wide discipline including classical fluid mechanics and elasticity of Newtonian fluid such as water and small deformations of hard solids such as wood and steel. However, the word "rheology" normally refers to the flow and deformation of "non-classical" materials such as rubber, molten plastics, polymer solutions, slurries and pastes, electrorheological fluids, blood, muscle, composites, soils, and paints. These materials exhibit varies and striking rheological properties that classical fluid mechanics and elasticity cannot describe.

There are two fundamental catagories of rheology [1]. One is the development of correlation between deformation and force for a material of interest from experimental measurements. For example, we may observe that force requiring to compress a rubber ball for a certain distance is proportional to the distance. Thus one can establish a general relation from this observation. This kind of relation is known as "constitutive equation." In simple materials such as a linear elastic material or a Newtonian fluid, a constitutive equation is generally established. However, for more complex materials such as molten plastics, the developments of a constitute equation is more difficult and requires many types of experiment.

The second catagory is to relate the material properties such as material structure, composition, temperature, and pressure to the constitutive equation. That is, we can relate the viscosity and the relaxation modulus to molecular structure, composition, temperature, and pressure. This has only little success for the complex materials.

Because constructing the constitute equation need a simple measurement for the complex materials to correlate the material behavior to the equation. A sliding plate rheometer incorporated a local shear stress transducer [2] has been developed to suit a wide range of viscoelastic materials such as molten plastics, concentrated polymer solution, and

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† Though rheology is an old discipline, the word "rheology" was coined in 1929 by Professor Marcus Reiner and Professor Eugene Bingham. It means "everything flows depending on time interval."

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raw elastomers. It can generate steady shear rates up to 500 s^{-1} and not only can be used to measure linear viscoelasticity, but also can be used to measure non-linear one, a large deformation with very short time. For instance, the sliding plate rheometer can generate total strains of 10 in about 10 milliseconds, which is normally used in the industrial processes.

From those two study aspects, many researchers are trying to find a developed constitutive equation model that predicts many kinds of experimental data in complex flows. Certainly sliding plate rheometer is one of the key to study these correlations. For example, Jeyaseelan [3] successfully uses data from modified sliding plate rheometer to study biaxial shear behavior of a polybutylene and a low density polyethylene by using kinetic network theory. Later, Giacomin and his coworkers [4]-[6] using the sliding plate rheometer as a main apparatus to study the network theory to predict many kinds of flows such as large amplitude oscillatory shear, exponential shear, and step strain.

Since molten polymer behaviors in the working conditions of processing processes such as high strain rate of the molten polymers in extrusion or injection are far beyond current rheometrical techniques, because the material properties in the working ranges are highly nonlinear, to closely predict those processing processes, it is necessary to generate a large uniform, transient deformation involving high strain rate for a broad spectrum of nonlinear viscoelastic properties. A sliding plate rheometer incorporated with a local shear stress transducer is a possible solution for those nonlinear problems. This paper intends to review the sliding plate rheometer and its applications in the past.

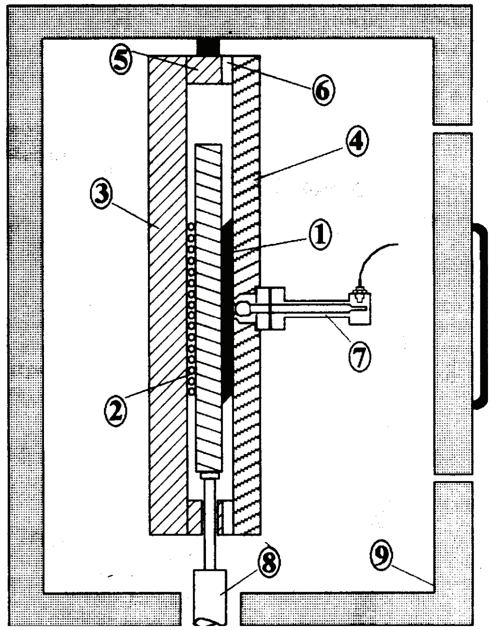

Figure 1 Cross section showing the essential elements of a sliding plate rheometer incorporating an elastic type shear stress transducer [17]. (1) sample; (2) moving plate; (3) back support; (4) stationary plate; (5) end frame; (6) gap spacer; (7) shear stress transducer incorporating a rigid beam supported by a steal diaphragm; (8) linear actuator; (9) oven.

2. Sliding Plate Rheometry

Figure 1 illustrates the operating principle of a sliding plate rheometer incorporating a shear stress transducer (7). By using the simple Couette flow fundamental, a molten plastics sample (1) is squeezed between parallel plates: one fixed (4) and the other moving at a controlled speed (8). When the moving plate is operated by the linear actuator, the sample senses the move and then propagates the deformation to the local shear stress transducer (7) that is flush-mounted in the stationary plate. Finally, the capacitance proximeter [Capacitec, Ayer, MA] at the transducer tail detects deflection proportional to the shear stress on the active face.

Giacomin et al. [7], [8] first built this kind of rheometer incorporated with a US patented local shear stress transducer [9] at McGill University in 1987. Next, the rheometer has been commercialized by Interlaken Technology Corporation [10]. Later, a high-pressure version has been developed to study the influence of pressure on the viscoelastic properties and on shear induced crystallization of molten polymers and elastomers. The high-pressure one can be operated at pressures up to 70 MPa and temperatures up to 225°C [11], [12]. Another high pressure version is also used in an on-line rheometer to be mounted directly on plastics manufacturing equipment [13]-[15]. Recently, McKinley et al. [16] develops the rheometer for microparticles for up to 200μm flexure-based microgap.

Because the sliding plate rheometer uses rectilinear shearing action, so it is useful for constructing material functions in both steady and unsteady shear flow, which is normally studied between two rectangular plates.

For this reason the sliding plate rheometer can be used for various conventional experiments such as steady shear flow, small amplitude oscillatory shear, stress growth, stress relaxation, and creep [18]. Moreover, it also can be used in complicated experiments such as interrupted shear [19], large amplitude oscillatory shear, exponential shear [20], The examples of rheological properties of linear viscoelastic fluids, usually described in terms of material function, that can be measured by the sliding plate rheometer [21], are the viscosity, $\eta(\gamma)$, the storage, $G'(\omega)$, and loss, $G''(\omega)$, modulli or the relaxation spectrum, g_i, λ_i [22].

In unsteady shear flow, for example, the material functions are the viscosity, $\eta(\gamma)$, the first, $\Psi_1(\omega)$, and second, $\Psi_2(\omega)$, normal stress coefficients‡. In addition, transient shear flow has the strain dependent relaxation modulus, $G(t, \gamma)$, the shear stress growth coefficient, $\eta^+(t,\gamma)$, and the tensile stress growth coefficient, $\eta^+_E(t,\gamma)$. Because of the rheological complexity in nonlinear viscoelastic fluids, no complete pictures from a material function to describe the flow behavior. In practice, an interested material function depends on the working conditions. Thus, one has to design a test that can simulate the working process. For this reason, the most versatile rheometer such as the sliding plate rheometer is needed.

3. Shear Stress Transducer in the Rheometer

To measure the viscoelastic properties of viscous liquids, a shear stress transducer is incorporated in a shearing fixture. The transducer, flush mounted and centered on the fixed plate, serves to circumvent uncontrollable flows near the sample's edges.

In the rheometer, a local shear stress transducer is used in materials characterization. Specifically, these are incorporated in shear fixtures whenever a total force (or torque) measurement fails. Consider a slab of polymer between sliding plates, for example. Though the middle of the sample undergoes simple shear, the ends and edges do not. These free boundary errors corrupt the total force measurement. This is

‡ The first and second normal stress coefficients are very difficult to measure and cannot be obtained from the sliding plate rheometer.

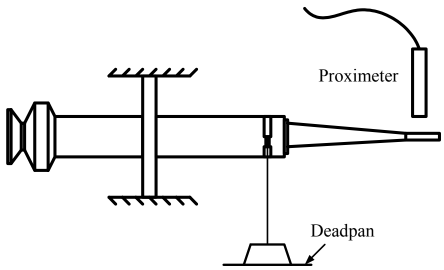

Figure 2 Static calibration for the shear stress transducer in a sliding plate rheometer.

why many flush-mount a shear stress transducer in the fixed plate [3], [21], [23]-[28]. This type of transducer was patented in 1986 by Dealy [9]. More details on its configuration and limitation can be found elsewhere [7].

This is one way that plastics engineers characterize molten plastics [29], [30]. Those interested in the large shear strain behavior are especially reliant on shear stress transduction, as the free boundary error corruption worsens with strain amplitude.

4. Calibration

4.1 Static Calibration

Static calibration is performed at the working temperature to ensure linear response between the measured wall shear stress and the transducer's output voltage. To achieve this, a deadpan is hung from the transducer as Figure 2 shows. The mechanical design of the transducer relates this calibration weight to the equivalent shear stress on the active face. Thus, one can adjust the output voltage related to the calibration weight. The static calibration is described in the rheometer user manual [31].

4.2 Dynamic Calibration

To study material viscoelasticity (or damping),

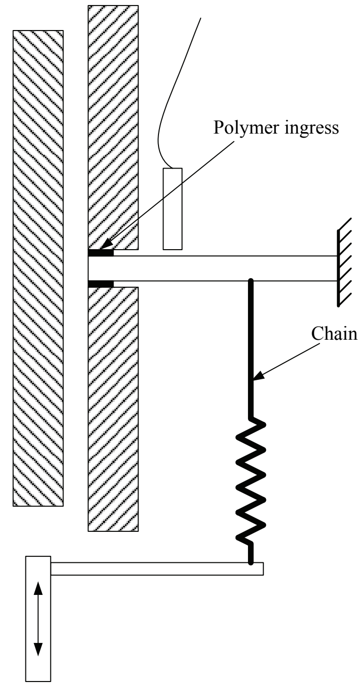

Figure 3 The *ex situ* dynamic calibration with applied sinusoidal force to the beam.

static calibration is not enough. Here the shear stress must be tracked accurately in time. Thus dynamic calibration is performed to see how much phase delay the transducer introduces. Figures 3 and 4 show dynamic calibration fixtures. When the actuator displaces sinusoidally with time,

$$d_a = d_{ao}\sin(\omega t) \qquad (1)$$

where d_{ao} is the actuator displacement amplitude, and ω is a frequency.

When the spring exerts a sinusoidal force on the cantilever, the cantilever beam moves toward the given signal with a phase shift, δ. We then compare the actuator displacement with the measured cantilever displacement,

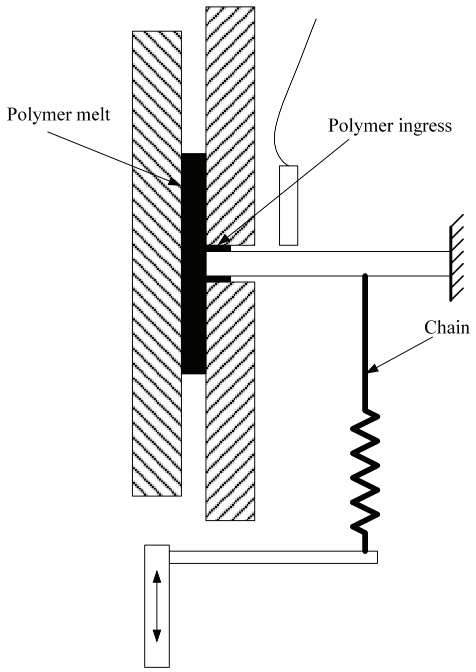

Figure 4 The *in situ* dynamic calibration with applied sinusoidal force to the beam. In the figure, the polymer sample is left inside the rheometer.

$$d_c = d_{co}\sin(\omega t+\delta) \qquad (2)$$

where d_{co} is the cantilever displacement amplitude.

A shear stress transducer can be calibrated with a sample in the rheometer (*in situ*) [32] or just after scraping it out (*ex situ*) [23]. In either case, for the dynamic calibration to be meaningful, there must be polymer ingress.

When scraping a molten polymer sample off the transducer (*ex situ calibration*), one can inadvertently remove some fluid ingress. *In situ* calibration circumvents this, but its analysis is more complicated. By leaving the sample in, cantilever displacement shears the sample between the transducer's active face and the fixed opposing plate.

Through the sliding plate rheometer is convincing to use for characterizing both linear and non-linear material functions of polymer melts, the ingress in the transducer housing may affect measurement data. Recently, Kolitawong and Giacomin [33]-[35] study the ingress effects in the sliding plate rheometer. Though the ingress is small, it can make measurement phase error in the material properties and the magnitude of the error depends on the material viscosity. Thus, they suggest to fill the transducer gap by an elastic material.

5. Conclusion

The sliding plate rheometer for trainsient, large deformation measurement is a solution for both researchers and plastics engineers to simulate the processing processes. Using the sliding plate rheometer as a main apparatus to find a general constitute equation to predict the polymer behaviors in nonlinear viscoelasticity, trial and error processes of the polymer processing can be diminished.

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