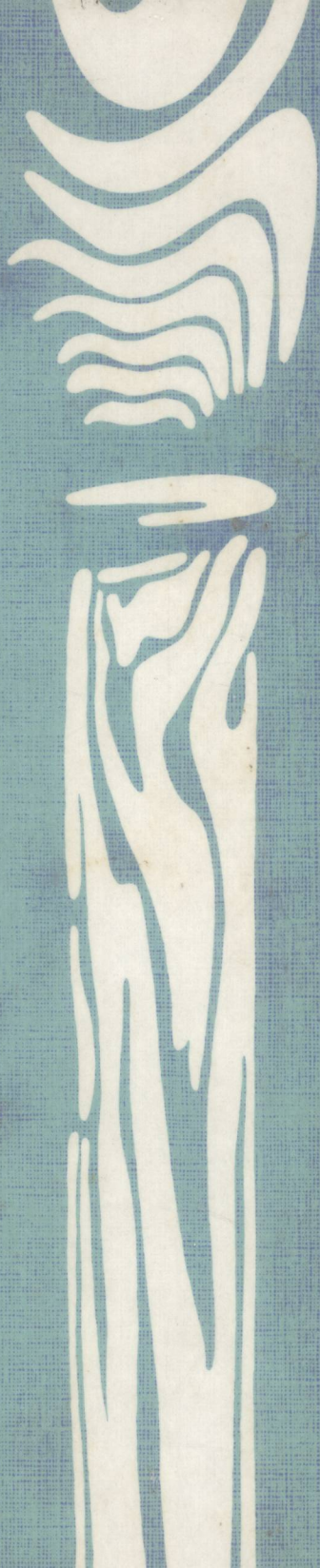


Rheological Measurement

Edited by

A.A. Collyer and
D.W. Clegg

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RHEOLOGICAL MEASUREMENT



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RHEOLOGICAL MEASUREMENT

Preface

In many cases rheological measurements are carried out in the simplest of geometries, but the interpretation involved in obtaining the rheological parameters of the test fluids from these measurements is surprisingly complex. The purpose of this book is to emphasise the points on which most workers in the field agree, and to let the authors deal with the contentious points according to their own beliefs and experience. This work represents a summary of the current thought on rheological measurement by experts in the various techniques.

When making measurements and obtaining from them parameters that describe the flow behaviour of the test fluids, it is essential that the experimentalist understands the underlying theory and shortcomings of the measurement technique, that he is aware of the likely microstructure of the fluid, and that from this he can appreciate how the fluid and the measuring system will interact with each other. It is this interaction that gives both the required rheological parameters of the fluids and the artefacts that confuse the issue.

This book covers the main rheological measurement techniques from capillary, slit and stretching flows to rotational and oscillatory rheometry in various geometries including sliding plate measurements. These topics are backed up by chapters on more practical aspects, such as commercial instruments, and on computer control and data acquisition. The chapters deal with the basic methods, how the measurements are taken, and what assumptions and interpretations are made to obtain valid data on the test fluids.

Another group of chapters, on die swell, shear heating and hole pressure measurement, build on the more commonplace techniques and enable the experimentalist to correct his data or to use further methods to obtain the rheological parameters of the test fluids. The chapter on flow visualisation techniques illustrates the need to use other techniques to assist the rheological measurement.

The final two chapters concentrate on the test fluids themselves rather than on techniques. One deals with the macroscopic flow behaviour of blends and complex fluids, and the other chapter examines the ways in which mathematical models of two-phase fluids can be derived from assumptions made on the fluid microstructure.

Rheology is a vast and exciting subject and can scarcely be covered in one book, albeit a large one. In this book it has been the aim to concentrate on the basic techniques and the physical principles underlying them, with some discussion of multiphase fluids, which are a large class of fluids of industrial interest and importance.

The work is of considerable importance and relevance to all establishments in which rheological work is carried out, be the materials polymeric, biological, slurries, food or other complex fluids. Materials scientists, engineers or technologists in industry, research laboratories or in academic institutions should find the book valuable in providing an up-to-date review of current thought from experts in the field of rheology from different parts of the world.

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Chapter 1

Capillary Rheometry

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1.1. INTRODUCTION

This chapter outlines the important physical aspects of the flow of fluids, especially polymeric fluids, within capillaries. The problem appears deceptively simple in that, if a steady viscometric flow is assumed within the

constant circular section tubing, it is generally very easy to derive equations from elementary constitutive equations which yield the velocity profile and pressure drop within the pipe. We shall discover that for polymer melts the flows are experimentally observed to be far from being viscometric within the capillary section; however, in spite of this, the 'classic' viscometric pressure drop equations are usually adequate for most engineering calculations, although of dubious merit for detailed characterisation of rheologically complex fluids.

Capillary rheometry is used as a method of characterising a test fluid's rheological properties. It also represents a particular case of the slightly more general problem of understanding the way in which fluids flow within a duct of variable cross section. This latter problem of course represents the heart of all extrusion processing which incorporates continuous extrusion such as the manufacture of plastic pipes, batch extrusion such as the flow of toothpaste out of a tube, blow moulding for the production of polymer film, and injection moulding of complex shapes. The current trend is towards increasingly diverse and sophisticated rheological fluids being processed faster within more complex geometrical dies, and consequently there is now a real need to understand fully both general duct flow and, in particular, capillary flow.

If it were possible to have an indefinitely long capillary section many of the problems associated with capillary rheometry would be removed. In reality the capillary section will have a beginning and an end. In terms of rheometry the entry region is very important, whilst the exit region has less influence on pressure drop measurements. The exit region does of course have profound significance for polymer processing because die swell and instabilities emanating from the exit are usually the two factors that control the maximum output of an extrusion process. In this chapter we choose not to examine exit effects too deeply but concentrate our attention on the capillary and entry region.

1.2. PHYSICAL ASPECTS

In this section we outline the main variables with which we will be concerned. It is important when dealing with unusual or novel fluids to check that the base assumptions applied to the analysis are valid for the particular fluid under test.

1.2.1. Geometrical Constraints

The primary geometric parameters shown in Fig. 1.1 are the L_0/D_0 ratio of the capillary section, the entrance contraction ratio D_1/D_0 , and the entrance angle ϕ of the capillary. Typically we might expect D_0 to vary from say $100\ \mu\text{m}$ to $5\ \text{mm}$ and L_0/D_0 ratios from 0 to 500. Entrance contractions D_1/D_0 vary from about 2 to about 200 and the entrance angle is usually 90° but can vary between say 15° and 90° .

When dealing with structured fluids and composite fluids the ratio D_0/l_f may be important, where l_f is the largest length scale associated with the structured fluid. In general the precision of circular holes is good and this together with surface finish and/or wall material composition will probably not influence measurements greatly.

1.2.2. External Fluid Boundary Condition

Desirably the choice is between a constant volumetric flow within the capillary or a constant pressure drop. Most capillary rheometers work on the basis of a constant velocity piston driving fluid from the reservoir to the capillary. Fluid pressure is measured either using a force transducer on the piston or a side entry pressure transducer upstream of the capillary entrance. The disadvantage of the piston force transducer is that piston friction and reservoir wall viscosity must be taken into account. The force divided by the piston cross-sectional area gives a measure of the mean upstream hydrostatic pressure in the reservoir. Side transducers usually present problems in ensuring a flush mounting on the inside convex face

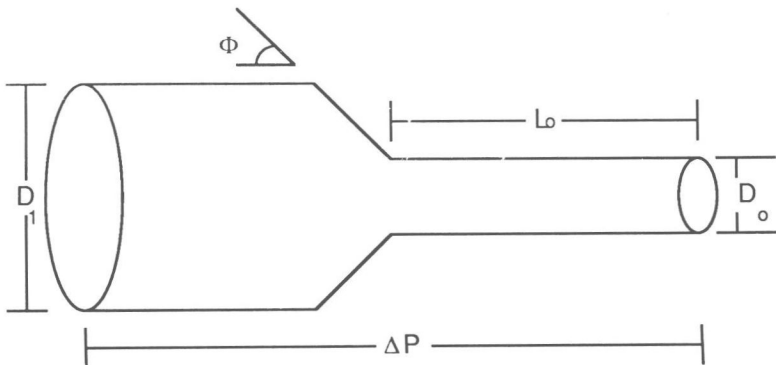


Fig. 1.1. Schematic diagram of duct geometrical parameters.

of the reservoir wall. The side transducer measures the normal force at the reservoir wall.

It is possible in principle to run a capillary rheometer at a constant pressure drop, where measurement of the volumetric flow can be obtained from continuous weight measurements of the extruding fluid; however, when working in this mode it is often difficult to run the fluid in a stable manner over a wide range of pressures.

1.2.3. Fluid/Solid Boundary Conditions

The fluid/solid boundary conditions are every bit as important as the constitutive equation of the fluid. It is usual to assume that the velocity of the fluid is zero at the wall, and this is generally the case for Newtonian fluids. The zero slip condition is also true for most but not all situations for flowing molten polymers. However, when one comes to examine e.g. structured fluids, composite fluids and some foodstuffs, the possibility of wall slip becomes a reality with very serious consequences in the rheological interpretation of data.

1.2.4. Inertia

The Reynolds number (Re) of capillary flow is usually but not always sufficiently low for inertia effects to be ignored, which means that creeping flow solutions are generally appropriate.

1.2.5. Temperature

Viscosity is a thermally activated process¹ and consequently the rheology of a fluid is very dependent on temperature. Accurate temperature control of fluid conditions is essential and there is the possibility that shear heating at the wall may influence results at high extrusion rates for high viscosity fluids, as discussed in Chapter 5.

1.2.6. Pressure

Absolute pressure also influences fluid viscosity¹ and Cogswell² for example considers that pressure can have a significant effect on polymer melt rheology.

1.2.7. Bulk Fluid Properties

Apart from the fluid/solid boundary conditions the bulk fluid properties represent the most difficult and intriguing aspect of fluid rheology. Firstly, the question of incompressibility should be considered. Generally, fluids that are dominantly viscous are incompressible, but viscoelastic fluids can

show significant compressibility effects.² The question of composition uniformity may also be important as polymer fluids can in principle segregate with different molecular weight components in different regions of the flow. When polymer solutions are processed there is the possibility of different polymer concentrations in different regions of the flow, and in particular there may be a depletion or enhancement³ of polymer concentration at or near the solid wall.

The constitutive equation of the fluid is of course the aspect of central importance in terms of the correct description of flow behaviour. Popular equations used in an engineering context are:

(1) Newtonian fluid

$$\tau(t) = \eta \dot{\gamma}(t) \quad (1.1)$$

where τ is the shear stress, $\dot{\gamma}$ the shear rate and η the viscosity.

(2) Bingham plastic fluid

$$\begin{aligned} \tau(t) &= \tau_y + \eta \dot{\gamma}(t) & (\tau > \tau_y) \\ \dot{\gamma} &= 0 & (\tau \leq \tau_y) \end{aligned} \quad (1.2)$$

where τ_y is the yield stress of the fluid.

(3) Power law fluid

$$\tau(y) = K \dot{\gamma}(t)^n \quad (1.3)$$

where K and n are constants.

(4) Cox equation

$$\tau(t) = \left(\eta_\infty + \frac{\eta_0 - \eta_\infty}{1 + \alpha \dot{\gamma}(t)^n} \right) \dot{\gamma}(t) \quad (1.4)$$

where η_0 and η_∞ are limiting viscosities at low and high shear rates respectively and α and n are constants.

In all these equations the shear stress τ at a particular position or (equivalent) time depends only on the strain rate at that particular position or time. The fluid does not have a memory of the past. Polymer fluids in particular do remember where they have come from and consequently their past deformation history does matter; thus the correct way to describe a polymer fluid should have a constitutive equation that takes past deformation histories into account. Constitutive equations of the