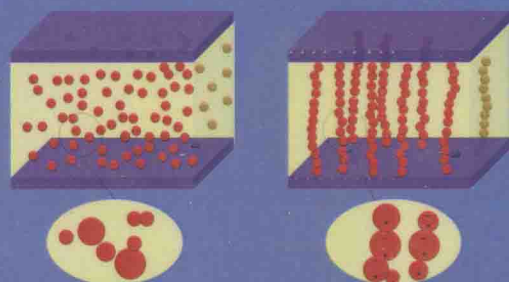


# Applied Polymer Rheology

POLYMERIC FLUIDS WITH  
INDUSTRIAL APPLICATIONS



Edited by  
MARIANNA KONTOPOULOU

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# APPLIED POLYMER RHEOLOGY

Polymeric Fluids with Industrial  
Applications

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Edited by

**Marianna Kontopoulou**

Queen's University  
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# APPLIED POLYMER RHEOLOGY

## PREFACE

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The topic of rheology of polymeric fluids has been the subject of intense research and has attracted some of the brightest minds in fluid mechanics, polymer physics, and chemistry. There are two main venues through which this research has been disseminated: The first is through numerous state-of-the-art textbooks, written by the top scientists in the field. These cover extensively the science and fundamental principles of rheology; constitutive equations; principles of rheometry, measurement methods, and techniques. The other dissemination method is through articles published in peer reviewed journals. These cover a vast array of topics, from purely theoretical concepts to the applied rheology of specific polymeric fluids and melts. Many review papers on various topics of applied rheology are also available. Although very comprehensive, these publications are often not readily accessible to professionals, consultants and industrial practitioners, who because of time constraints are not necessarily in a position to follow the advanced rheological concepts presented in many of the scientific papers published in top peer-reviewed journals.

The purpose of this book is to present the state-of-the-art for specific polymeric systems of current industrial interest, as studied by well-recognized researchers who are experts in their fields. It targets professionals, industrial practitioners, as well as researchers and graduate students in the fields of polymer engineering, materials science, and chemical engineering who need to know the most recent developments pertaining to the rheology of various polymeric systems. This target audience would benefit from the existence of a book that explains the distinct rheological characteristics of various industrial systems and their impact on their processing characteristics and ultimate engineering properties. This textbook provides them with a comprehensive overview of the rheological characteristics of the polymeric fluids of interest to their application, so they can interpret their findings or plan their experiments. It is also intended as a useful tool for processors and equipment designers, who need to plan carefully the processing conditions of their materials based on their rheological characteristics.

When teaching graduate courses I have frequently encountered the challenge of finding suitable review papers in many industrially relevant areas that I could present to my students. I envision this book as a useful starting point in the

research of graduate students in the fields of polymer science and engineering, materials science, and chemical engineering, who once exposed to a basic course on rheology will be ready to venture on the study of the polymeric fluids of interest in their graduate work. This book provides them with an overview of what has been done to date and, I hope, some insight on what needs to be done in the future.

Even though some basic concepts of rheology are introduced in Chapter 1, this textbook assumes a basic knowledge of rheometry and familiarity with the fundamental concepts of rheology. For in-depth coverage of these topics, the reader is referred to the many excellent books that cover the theoretical principles of rheology, fundamental concepts, constitutive equations and principles of rheometry.

This book comprises 11 chapters, organized loosely in subtopics. The first chapter establishes basic relations between the rheological properties and processability of polymer melts. Knowledge of the rheology of polymer melts can serve as a tool in predicting their behavior during processing. This chapter provides the readers with an appreciation of the importance of rheology to processing. It also includes some fundamental theory of rheology and constitutive equations. Processing aids, which are used extensively to improve the processability of polyolefins and other polymers are presented in Chapter 2. Chapter 3 covers the rheology of branched polyolefins, which have attracted great interest in recent years, because of their applicability in important industrial processes, such as thermoforming, extrusion coating, and foaming. The presence of branching affects profoundly the rheology and thus the processing characteristics of these polyolefins. This chapter is divided in two subsections, describing the rheology of branched polyethylene (PE) and polypropylene (PP). Filled polymers and composites are the topic of the next three chapters. These include the rheology of fiber suspensions, polymer nanocomposites, and wood fiber composites. Addition of solid particulates significantly affects the rheological properties and thus the processability of the melts. These effects are discussed extensively in Chapters 4–6. Chapter 4 introduces the theoretical framework on which the analysis of these materials is based. Chapters 7–10 include systems with microstructure, such as block copolymers, liquid crystalline polymers, electrorheological fluids, and reactive blends. Chapter 11 deals with a novel topic, that of paste extrusion of polytetrafluoroethylene paste.

I would like to thank all the contributors who provided their expertise and enthusiasm to this project and Wiley for making this work possible. I would like to dedicate this book to the memory of my father, Antonios Kontopoulos, who was a committed academic and educator.

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# CHAPTER 1

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## BASIC CONCEPTS IN POLYMER MELT RHEOLOGY AND THEIR IMPORTANCE IN PROCESSING

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## 1.1 INTRODUCTION

The vast majority of the nearly 250 million tons of polymers produced annually are thermoplastics, which are melted by heating, shaped by flowing through dies or filling of molds, and subsequently solidified into final useful products. The melt processing of thermoplastics is, to a large extent, determined by flow behavior, which in turn depends on structure and the processing conditions of temperature and pressure. The relations of structure, processing, and properties of polymers are of paramount importance in all their applications as film, sheet, pipe, tubing, profiles, wire and cable coatings, containers, appliance housings, automotive, and aerospace parts and as numerous other products.

Molten polymers exhibit rather unusual flow behavior and some rather counterintuitive phenomena, as it is explained in several books [1–14] and numerous publications. In fact, the majority of publications in rheology, the science of deformation and flow of matter, deal with polymer melts and solutions. First, rheological measurements are necessary for process equipment design. Knowing the viscosity as a function of shear rate permits the calculation of pressures and production rates. Process troubleshooting and process optimization can be carried out by detailed rheological characterization, as it will become apparent in the subsequent sections and chapters of this book. Some rheological measurements are sensitive indicators of polymer structure, such as molecular weight, molecular weight distribution, and chain branching. In fact, the sensitivity and frequently simplicity of some rheological characterization methods are the main reasons for the success on rheology in polymer processing. Textbooks on polymer processing [15–25] are mainly devoted to describing how rheological measurements can be combined with fluid mechanical and heat-transfer principles for the prediction of how molten polymers flow through extruders, channels, dies, molds, and other types of equipment.

## 1.2 VISCOSITY OF POLYMER MELTS

Viscosity represents the resistance to shearing, i.e., flow of imaginary fluid slices like the motion of a deck of cards. Referring to Figure 1.1, we can define

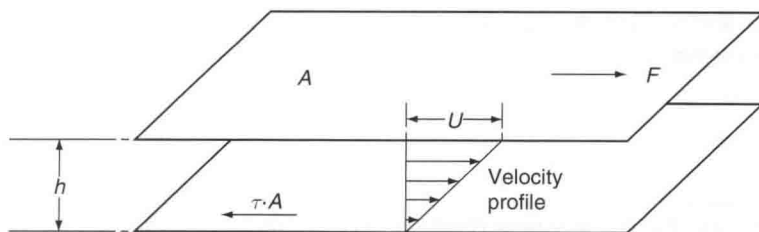


FIGURE 1.1 Simple shear flow.

viscosity as the ratio of the imposed *shear stress* (force  $F$ , applied tangentially, divided by the area  $A$ ), and the shear rate (velocity  $U$ , divided by the gap  $h$ )

$$\eta = \frac{\text{Shear Stress}}{\text{Shear Rate}} = \frac{F/A}{U/h} = \frac{\tau}{\dot{\gamma}} \quad (1.1)$$

This relation is known as Newton's Law of Viscosity. When the viscosity is independent of the shear rate, a fluid is called Newtonian. Molten polymers have viscosities dependent on shear rate, exhibit several other unusual flow properties, and are referred to as non-Newtonian.

One remarkable property of polymeric liquids is their *shear-thinning* behavior (also known as pseudo-plastic behavior). As the shear rate increases, the viscosity decreases, as shown in Figure 1.2. This reduction of viscosity is due to molecular alignments and disentanglements of the long polymer chains. The higher the shear rate, the easier it is for polymers to flow through dies and process equipment.

The most frequently used model to express the shear-thinning behavior of polymers is the power law:

$$\eta = m \dot{\gamma}^{n-1} \quad (1.2)$$

This expression is a straight line when plotted on double logarithmic coordinates. The value of the consistency index  $m$  can be obtained from the intercept at  $\dot{\gamma} = 1$ . The exponent  $n - 1$  is the slope, because

$$\log \eta = \log m + (n - 1) \log \dot{\gamma} \quad (1.3)$$

This model is not suitable for fitting low shear rate data because the viscosity predicted approaches infinity as the shear rate goes to zero, while polymeric liquids exhibit a characteristic Newtonian plateau at very low shear rates.

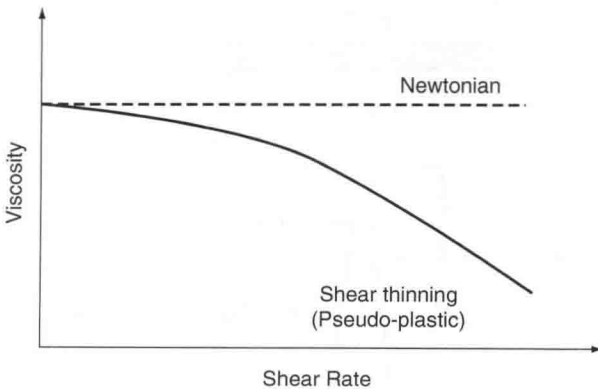


FIGURE 1.2 Newtonian and shear-thinning viscosity behavior.

For  $n = 1$ , the power law model reduces to Newton's law (constant viscosity). As  $n$  decreases, the polymer becomes more shear thinning. The power law exponent of commercial polymers varies between 0.8 (for some polycarbonate, PC, grades) and 0.2 (for some rubber compounds). For various polyethylene (PE) grades, the range is  $0.3 < n < 0.6$ , and depends on molecular weight and chain branching and does not change much with temperature. The consistency  $m$  varies a lot with temperature and under usual processing conditions the consistency index for the most common molten polymers varies between  $1000 \text{ Pa} \cdot \text{s}^n$  (for some polyethylene terephthalate, PET, resins) to  $100,000 \text{ Pa} \cdot \text{s}^n$  for highly viscous rigid polyvinyl chloride (PVC). The value depends on chain mobility and molecular weight.

During single-screw extrusion, shear rates may reach  $200 \text{ s}^{-1}$  in the screw channel near the barrel wall, and much higher between the flight tips and the barrel. At the die lip exit the shear rate can reach or exceed  $1000 \text{ s}^{-1}$ . During cavity filling in injection molding shear rates can reach  $10,000 \text{ s}^{-1}$  and in some wire-coating applications shear rates may exceed  $100,000 \text{ s}^{-1}$ . However, in the process of rotational molding shear rates are much less than  $1 \text{ s}^{-1}$ .

Melt index (MI), melt flow index (MFI), or melt flow rate (MFR) (for polypropylene) refers to the grams per 10 min pushed out of a die of prescribed dimensions according to an ASTM Standard [4] under the action of a specified load, as shown in Figure 1.3. For PE (ASTM D-1238) the load is 2.16 kg and the die dimensions are  $D = 2.095 \text{ mm}$  and  $L = 8 \text{ mm}$ . The experiment is carried out

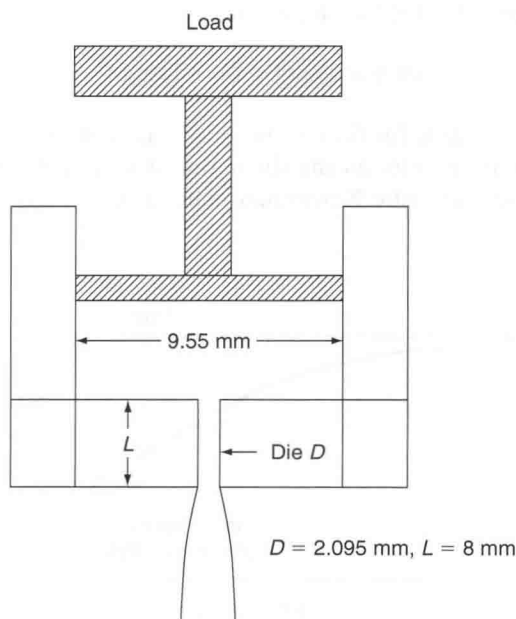


FIGURE 1.3 Schematic of a melt indexer.

at 190°C. For PP, the same load and die dimensions are used, but the experiment is carried out at 230°C.

When measuring the melt index with a 2.16-kg load, the wall shear stress can be calculated to be  $\tau_w = 1.94 \times 10^4$  Pa, and the wall shear rate approximately  $\dot{\gamma} = (1838/\rho) \times MI$ , where  $\rho$  is the melt density in  $\text{kg/m}^3$ . Assuming  $\rho = 766 \text{ kg/m}^3$  for a typical PE melt, we get  $\dot{\gamma} = 2.4 \times MI$ . A low melt index means a high molecular weight, highly viscous polymer. A high melt index means a low molecular weight, low viscosity polymer. When the melt index is  $<1$ , the material is said to have a fractional melt index. Such materials are used for film extrusion. For some film grades MI can be  $<0.1$ . Most extrusion PE grades seldom exceed  $MI = 12$ , however, for injection molding, MI is usually in the range of 5–50.

The MI (inversely) corresponds to just one point on a viscosity curve (usually at low shear rates). Frequently, the so-called high load melt index (HLMI) is measured, usually with either a 2.16-kg or a 10-kg load on the melt indexer. From two points it is possible to obtain a power law viscosity fit, but such approximations should always be used with extreme caution due to inherent inaccuracies in the measurement method.

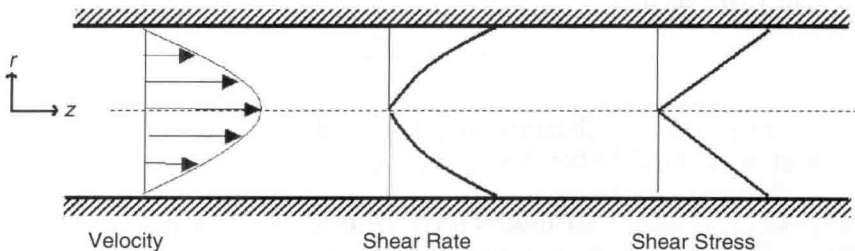
For fully developed pressure driven axial flow ( $z$  direction) of polymeric fluid in a tube of radius  $R$ , the velocity profile is quasi-parabolic, the shear stress varies linearly with the radius and the shear rate nonlinearly as shown in Figure 1.4.

For Newtonian fluids, the wall shear rate is given by

$$\dot{\gamma}_w = \frac{4Q}{\pi R^3} \quad (1.4)$$

This relation is referred to as apparent shear rate when used in non-Newtonian flows. A correction is necessary (Rabinowitsch correction) for shear thinning fluids. For the power law model, the true (Rabinowitsch corrected) shear rate becomes

$$\dot{\gamma}_w = \frac{3n+1}{4n} \frac{4Q}{\pi R^3} \quad (1.5)$$



**FIGURE 1.4** Velocity, shear rate, and shear stress profiles for pressure-driven flow through a tube of radius  $R$ .

This means that for a material with a power law exponent  $n = 0.4$  (very common for several commercial polymer grades), the relation between apparent and true shear rates is given by

$$\dot{\gamma}_{\text{true}} = 1.375 \times \dot{\gamma}_{\text{apparent}} \quad (1.6)$$

The above equations are used in capillary viscometry for measurement of viscosity from pressure drop  $\Delta P$  versus flow rate  $Q$  data.

The pressure drop  $\Delta P$  is measured in the reservoir of the viscometer, and at the entrance to the capillary there is an excess pressure drop  $\Delta P_e$ . The easiest way to determine the excess pressure drop  $\Delta P_e$  is to carry out the experiment using a twin-bore viscometer having two capillaries. One of them has very short length ( $L = 0$ ), and the pressure drop recorded is essentially the excess pressure due to the entrance (orifice). This correction is necessary when capillaries are relatively short ( $L/R < 50$ ) and is known as the Bagley correction. The Bagley correction is usually expressed as

$$n_B = \frac{\Delta P_e}{2\tau_w} \quad (1.7)$$

The Bagley correction ( $n_B$ ) may reach perhaps 20 when polymeric materials are extruded near the critical stress for sharkskin. For a Newtonian fluid the value for  $n_B$  is 0.587. For information on the Bagley correction and entrance and exit pressure drops the reader is referred to the monographs by Han [7].

Without the Rabinowitsch and Bagley corrections there can be significant errors in viscosity data obtained from capillary instruments.

In addition to the power law model, two other expressions are frequently used for better fitting of data over the entire range and to include the Newtonian plateau at low shear rates.

1. The Carreau–Yasuda model:

$$\eta = \eta_o \left( 1 + (\lambda \dot{\gamma})^a \right)^{\frac{n-1}{a}} \quad (1.8)$$

where  $\eta_o$  is the viscosity at zero shear and  $\lambda$ ,  $a$ , and  $n$  are fitted parameters.

2. The Cross model:

$$\eta = \frac{\eta_o}{1 + (\lambda \dot{\gamma})^{1-n}} \quad (1.9)$$

where  $\eta_o$  is the zero shear viscosity and  $\lambda$  and  $n$  are fitted parameters. Note that in this model when  $\lambda = 1/\dot{\gamma}$ ,  $\eta = \eta_o/2$

Capillary viscometers are usually used for the shear rate range from  $1 \text{ s}^{-1}$  to  $3000 \text{ s}^{-1}$ . The lower limit is determined by the ability of pressure gauges to measure low pressures, with accuracy, and the high limit is determined by instabilities, wall slip, and viscous heating phenomena. Rotational viscometers

are usually used for the range  $10^{-2}$  to  $5\text{ s}^{-1}$ . At higher rotational speeds, secondary flows and instabilities may occur that invalidate the simple shear assumption. For more information about viscosity measurements, the reader is referred to Macosko [2].

The viscosity of polymer melts varies with temperature and for most polyolefins it obeys the Arrhenius relation in the form

$$\eta = \eta_{ref} \exp \left[ \frac{E}{R} \left( \frac{1}{T} - \frac{1}{T_{ref}} \right) \right] \quad (1.10)$$

where  $E$  is the activation energy,  $R$  the gas constant and  $T_{ref}$  is the reference temperature.

In polymer processing this relation is frequently simplified into a simple exponential, which applies over a shorter temperature range but it is good enough for most extrusion calculations and simulations

$$\eta = \eta_{ref} \cdot \exp(-b \cdot \Delta T) \quad (1.11)$$

The temperature sensitivity coefficient  $b$  is usually between  $0.01$  and  $0.1\text{ }^{\circ}\text{C}^{-1}$ . For a linear polymer, high density polyethylene (HDPE) the value of  $b$  is roughly  $0.01$ , while for a branched low density polyethylene (LDPE) it may reach  $0.03$ .

The viscosity increases with pressure in the form

$$\eta(p) = \eta(0) \cdot \exp(\alpha \cdot p) \quad (1.12)$$

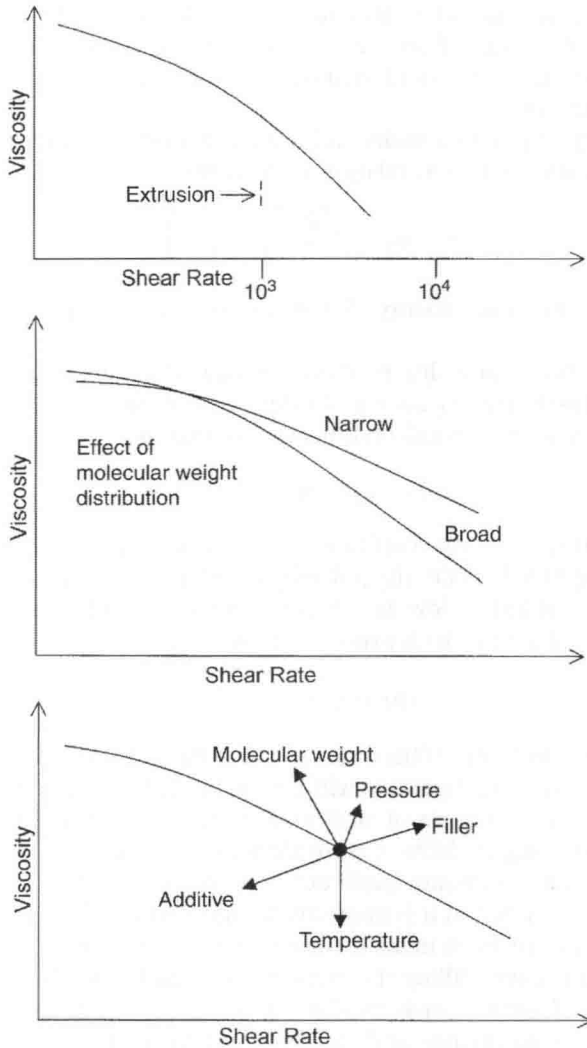
The coefficient is probably of the order  $2 \times 10^{-8}\text{ Pa}^{-1}$ . This means for a pressure increase of  $10\text{ MPa}$  the viscosity will go up by  $22\%$ . Cogswell [3] expresses pressure dependence in terms of an equivalent temperature change. His results suggest that applying  $10\text{ MPa}$  is equivalent to decreasing the temperature by about  $5\text{ }^{\circ}\text{C}$ . Usually, pressure dependence of viscosity is not taken into consideration in extrusion, but it is necessary in injection molding calculations and simulations. Pressure drops in die extrusion may reach at most  $50\text{ MPa}$ , while in injection molding cavity filling the pressure may reach  $200\text{ MPa}$ .

The effects of factors such as shear rate, molecular weight distribution, pressure, filler, temperature and additives on viscosity are summarized in Figure 1.5, following Cogswell [3]. Linear narrow molecular weight distribution polymers, such as metallocene catalyzed polyethylenes, are more viscous than their broad distribution counterparts. Fillers may increase viscosity (greatly). Pressure increases viscosity (negligible under usual extrusion conditions, but important in injection molding). Various additives, such as processing aids and lubricants, are available and are designed to decrease viscosity. The zero shear viscosity increases dramatically with the weight average molecular weight:

$$\eta_o = \text{const } M_w^{3.4} \quad (1.13)$$

For some metallocene catalyzed PEs with long chain branching, the exponent might be much higher (perhaps  $6.0$ ).





**FIGURE 1.5** The influence of various parameters on polymer viscosity.

In the above discussion of viscosity measurements, the assumption is made that the no-slip condition on the die wall is valid. This is, however, not always the case. In fact, at shear stress levels of about 0.1 MPa for PE, slip occurs. Wall slip is related to the sharkskin phenomenon [26, 27]. Wall slip is measured by the Mooney method in which the apparent shear rate ( $4Q/\pi R^3$ ) is plotted against  $1/R$  for several capillaries having different radii. In the absence of slip, the plot is horizontal. The slope of the line is equal to  $4 \times (\text{slip velocity})$ , as explained by Dealy and Wissbrun [5].