RHEOLOGY IN MATERIALS ENGINEERING

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Contents

Summary

Rheology is the science that studies the deformation and flow of matter and it is a relatively young but highly multidisciplinary science that encompasses such different industrial areas of activity as plastics, ceramics, cosmetics, pharmaceutics, food and biotechnology, paints and inks, adhesives, lubricants and surfactants. In fact, it is quite straightforward to list situations where the deformation or the flow of matter (which depend on the rheological characteristics of the materials involved) determines the performance of a product, the effectiveness of a service, the rate of a manufacturing process. Therefore, the applications of Rheology in Materials Engineering are virtually endless and the subject would, in itself, be worth various books. Thus, it was the authors' decision not only to keep this chapter to a very introductory level, for the sake of generality, but also to focus on what are probably the two main areas of application, Polymers and Food.

1. Introduction

This chapter deals with the role of Rheology in Materials Engineering, which is a subject that would certainly be worth various books by itself. Thus, it was necessary for the authors to: a) Focus on specific techniques and materials and b) Keep the discussion at a very introductory level for the sake of generality.

In what regards the former, the chapter will focus on polymers and food and the respective processing techniques due to their economic, societal and scientific importance. In fact, polymers (plastics) are processed in more ways and give rise to more diverse products than any other materials and the relevance of food engineering is self-explanatory.

The chapter will begin with a discussion on material properties, applications and processability that will be followed by a very short review of some of the most common processing techniques. The emphasis will then shift to the role of Rheology in Polymer and Food Engineering, which continues with an introduction to the role that modeling has in modern-day engineering applications. Finally, we will conclude the chapter with a necessarily personal view of the upcoming challenges and developments in both areas.

2. Materials and Their Properties

This section is devoted to a brief explanation of the main chemical, physical and rheological properties of the two classes of materials we deal with in this chapter, polymers and food, and introduces the reader to the influence they may have on their processing characteristics as well on the final product properties.

2.1. Polymer Materials: Applications and Processability
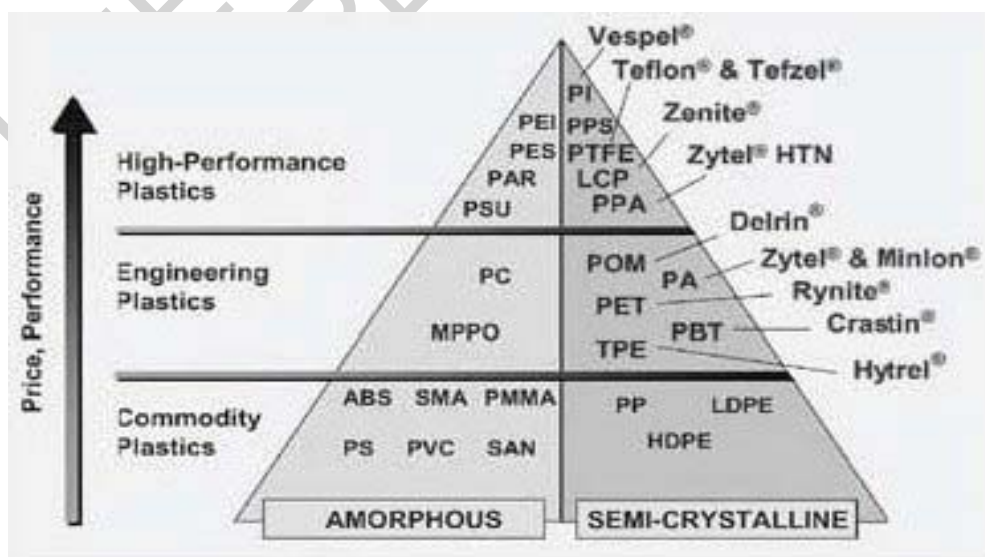
2.1.1. Applications

Figure 1. Polymer pyramid: a usual classification of polymers (source: Dupont Plastics)

Polymers consist of large molecules (*macromolecules*) containing from several thousands up to a few million of repeated chemical units (*monomers*) that are joined together. In turn, each monomer contains usually between 5 and 500 atoms. The terms plastics, elastomers, fibers, coatings, paints and adhesives refer to polymer materials exhibiting quite distinct behavior. Hundreds of different polymers exist, both natural and synthetic. Figure 1 reproduces a common classification of thermoplastic polymers: they can be either amorphous or (partially) crystalline and, depending on performance/price, they are known as commodity (typically for high consumption applications), engineering (in applications requiring at least one advanced characteristic) and high-performance (generally, in terms of mechanical properties and/or service temperature) [Cowie and Arrighi (2007); Powell and Housz (1998); Chanda (2006)].

As a class of materials, polymers have a number of characteristics that make them the best choice for a number of applications, namely:

- *Low density*, which leads to lighter products (CDs, PCs, ipods, mobile phones, automotive parts, packages). An illustrative example: the BMW series 1 car contains 219 kg of plastics parts. If these were replaced my metallic components, the car would weight approximately 1 ton more (given the differences in density of plastics to steel and aluminum), i.e., performance would decrease and energy consumption would increase;

- *Good thermal, electrical and acoustic isolation* (electrical cables and appliances, tool handles, panels for civil construction, window profiles, foams);

- *Low permeability to liquids and gases* (bottles, protective clothing, medical devices packaging);

- *Good mechanical properties* (helmets, bullet-proof clothing, automotive bumpers, surfboards, skis);

- *Easy conversion into products with complex and intricate geometries* (electric/electronic and mechanical parts – for example, small gears for watches and medical devices);

- *Excellent aesthetics* (Swatch® watches, plastics furniture, toys, household and office equipment, textile fibers, parts combining tough and soft touch zones – a simple illustration: the tooth brush).

Figure 2 shows the relative plastics consumption per industrial/application sector. Notice the importance of packaging, construction and household products, which account for 75% of all plastics utilization. As an example, Figure 3 identifies the most popular plastics that are used for packaging of food and other basic domestic products: Polyethylene, PE, Polypropylene, PP, Polyethylene Terephtalate, PET, Polystyrene, PS and Polyvinyl Chloride, PVC. Thus, most water and fizzy drinks bottles are made of PET, yoghurt cups and egg trays use PS, while the common plastic bag is generally made from PE.
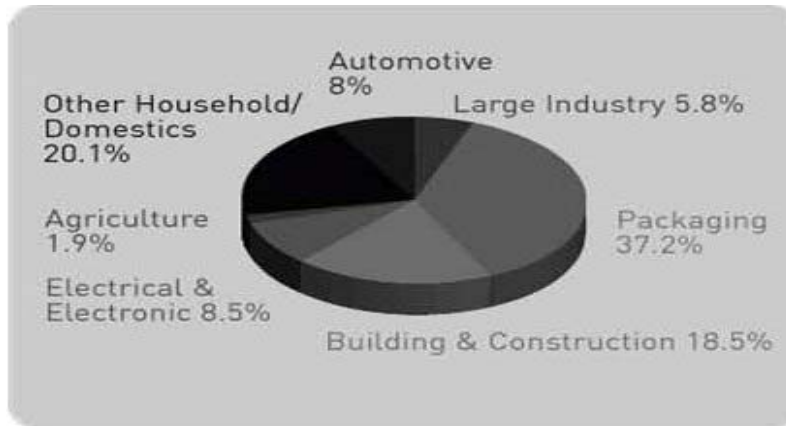
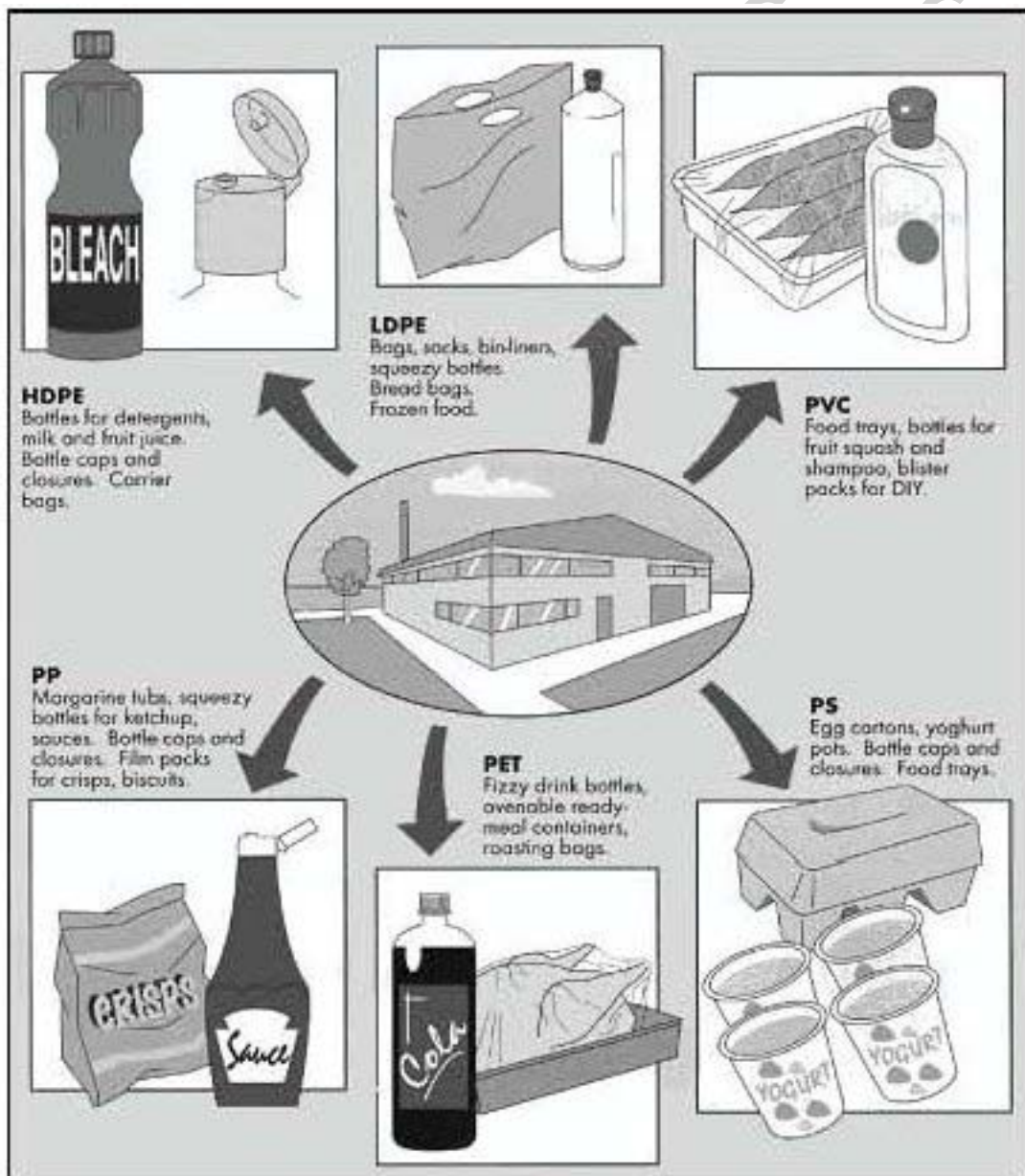Figure 2. Consumption of plastics per industrial sector (Source: APME 2003)

Figure 3. Packages and the plastics they are made from.

It is also important to consider if the properties that are advantageous in terms of applications are also interesting from a manufacturing point of view, i.e., whether some typical polymer characteristics facilitate or create practical difficulties to processing.

Figure 4 illustrates the typical sequence of operations involved in processing a thermoplastic polymer to yield a useful part. It starts with compounding, i.e., with the preparation of the raw materials. This is a very useful step, as it provides the possibility to either overcome the limitations of a given polymer to attain a specific performance for a given use, or to extend the properties to values beyond those initially expected for that particular polymer. For example, in the first case we might add UV-stabilizers to the polymer in order to decrease its rate of degradation when subjected to outdoor use, while in the second case we may add flame retardants, or carbon fibers, to increase significantly the fire and mechanical resistances, respectively. Substances that are incorporated into the polymer matrix are usually known as *additives* (these can be inorganic or organic, and in this case they can range from hydrocarbons to high molecular weight polymers). Another common example of additives is colorants (pigments or dyes), which are utilized to convey a plethora of aesthetical characteristics, such as color, gloss, or special optical effects (e.g., phosphorescent, metallic, or thermally triggered color changes). Compounding may involve more complex operations that are beyond the scope of this text, and that may require the use of specific equipment. The interested reader is advised to examine the literature specialized on this important topic [Todd (1998); Xanthos (1992); Utracki (2003)]. Examples include blending of (typically) two polymers, thus generating a new material that combines the properties of its components (but, as most polymer pairs with practical interest are immiscible, preparation of their blends may require physical or chemical interface compatibilization), polymer modification (for example, grafting chemical species to a polymer to promote its adhesion to other materials) and polymer degradation (a classical example would be the controlled peroxide-induced degradation of polypropylene, to generate grades with distinct average molecular weight). Raw materials should be available in pellet form, which is easier to transport and safer than powder. As pelletization is achieved through a special extrusion operation, this means that most polymers are subjected to two thermo-mechanical cycles, one during pelletization, the second during processing.

Once compounding is completed, the material (or, more adequately termed, the compound), usually in pellet form (i.e., granules of 2-4 millimeters in size), feeds a plasticating unit. This unit is present in most plastics processing technologies, even if with some modifications. It drags the material forward while heating and melting it, and generates pressure for the subsequent forming stage. Simultaneously, it must ensure proper melt homogenization, both in terms of size and concentration of any dispersed additives, as well as in terms of melt temperature. Once this is achieved, the plasticating unit pumps the melt through a die, or into the cavity/ies of a mold (extrusion and injection molding processes, respectively), thus creating the required product shape (we will see later that this statement is an oversimplification). Intense cooling will cause melt solidification, so that the processed part can be handled and subjected to eventual subsequent post-forming (the term denotes in-line procedures, such as blowing) and/or finishing (cutting, winding, printing, welding, etc) operations.

Given the above, polymer processing entails the following two basic ideas:

- Forming is carried out in the melt state, hence it must be preceded and followed by heating and cooling stages, respectively. Since polymers are good thermal insulators, heating and cooling are difficult

- Since it is interesting to guarantee high flow rates, the plasticating unit should be able to promote high shear rates and efficient pressure generation. Polymers having high viscosity levels, flow at high rates will require significant power from processing equipment, among other consequences.

The following paragraphs discuss, in greater detail, the interdependency between typical polymer characteristics and processability.
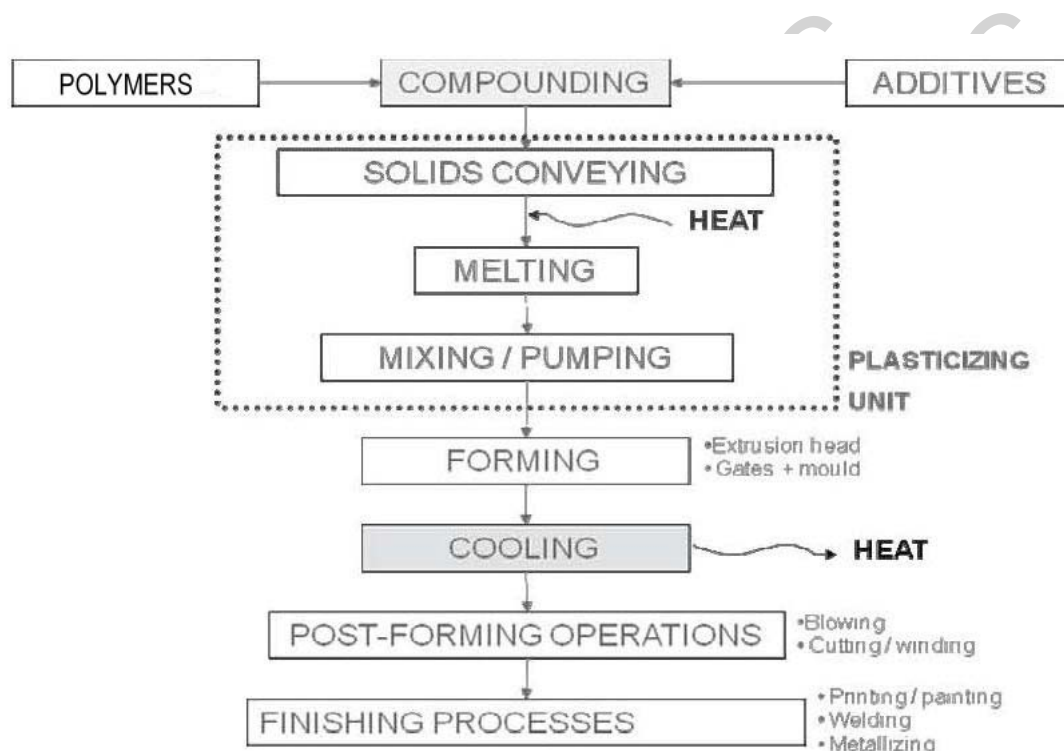
Figure 4. Usual steps in processing thermoplastics

2.1.2. Viscosity Levels

The melt viscosity of commercial polymers usually varies between 10^2 - 10^4 Pa.s (i.e, 10^5 - 10^7 times that of water). Table 1 compares the viscosity of polymer melts with that of other common fluids. Flow in channels at shear rates that can easily reach 10^3 to 10^5 s^{-1} can create significant pressure drops (typically from 5 to 50 MPa in an extrusion die). This feature brings about important practical consequences:

- Plastics processing equipment must be particularly robust and use high power drives to be able to induce flow at rates of industrial interest. For example, an extruder with a 60 mm diameter screw should have a 10 kW heater power and 60 kW drive power

to process polyamides. Similarly, all associated tools (such as extrusion heads, screws, and moulds) must be able to withstand the high stresses they are subjected to. A die or a mould that are not sufficiently rigid will deform as a result of the pressures and stresses generated upon flow, and thus affect the shape and/or the dimensional tolerances of the plastic item being produced. The overall volume of an injection mould is many times bigger than that of the part that it produces.

- High viscosities promote viscous dissipation (i.e., the self-generation of heat due to molecular friction). For example, in extrusion the contribution to material heating of dissipated heat is much higher than that of heat transfer. As a practical rule, such an increase in temperature is proportional to the product of the viscosity by the shear rate squared. Flow in a relatively short and simple duct can readily generate local temperature rises of 5 - 20ºC, but dissipations of 50 – 80 ºC have been reported for some situations (e.g., mixing zones in compounding extruders). Within certain limitations, viscous dissipation contains a self-regulating mechanism, since an increase in temperature due to shear heating will cause a decrease in viscosity, and the latter a decrease in the viscous dissipation.

- If the melt temperature inside a plasticizing unit is higher than the set temperature regulated by the operator, there is an obvious difficulty in controlling the process and avoid eventual local material degradation. The machine operator might deduce that an inappropriate operating condition is developing when he/she notices that the temperature controllers are permanently "off", instead of switching periodically between the "on" and "off" states, as the system tries to keep the barrel temperature close to the prescribed value, thus overcoming the thermal inertia.

MATERIAL	VISCOSITY (Pa.s)
Glass	10^{40}
Molten glass (500ºC)	10^{12}
Pitch	10^{8}
Molten polymers (150 - 300ºC)	$10^{2} – 10^{5}$
Liquid caramel	10^{2}
Honey	10^{1}
Liquid yoghurt	10^{0}
Olive oil	10^{-1}
Lubricating oil	10^{-2}
Water (25ºC)	10^{-3}
Air	10^{-5}

Table 1. Typical Viscosity Levels for Different Types of Materials

- Even when the polymer melt and the flow channel are initially at the same

temperature, polymer flow is inherently non-isothermal, as a result of the development of a shear rate profile across the flow channel, and of the above referred interdependence between shear rate and viscous dissipation. Temperature differences will become increasingly more important along the flow direction. For example, in the case of a pressure flow along a circular or rectangular channel, shear rate is nil at the centre and maximum at the wall, thus temperature would tend to increase in the same way. However, although the material flowing close to the wall exchanges little heat with the cooler adjacent inner flow stream, it is cooled by the much more efficient heat conductive metallic channel wall. As a global result, the temperature across the flow channel will be minimal at the center, increasing radially and reaching a maximum value not far from the wall, then decreasing sharply to the wall temperature as it contacts it.

- A number of additives have been developed to minimize the effects of high viscosity levels. For example, (internal) lubricants and plasticizers facilitate processing by lowering the viscosity, because they are compatible with the polymer and have much lower molecular weight. Heat stabilizers minimize the consequences of excessive viscous dissipation, by retarding the polymer thermal degradation rate. External lubricants promote wall-slip, i.e., facilitate flow and decrease the cross-channel shear rate gradient [Pritchard (2005)].

- Viscosity levels decrease with increasing temperature and presence of additives such as lubricants and plasticizers, but increase with molecular weight, hydrostatic pressure and presence of other additives, such as fillers and reinforcements. Since higher molecular weights favor mechanical performance but decrease processability, a compromise between both is generally aimed at.

Dimensionless numbers can provide a good estimation of the effects of viscous dissipation. The relative importance of viscous dissipation and heat transfer is measured by the *Brinkman number*:

$$B_r = \frac{\eta V^2}{k\left(\bar{T} - T_w\right)}$$

(1)

where η is viscosity, V is a characteristic flow velocity, k is thermal conductivity, $\bar{T}$ is average polymer temperature and T_w is the wall temperature. If $B_r > 1$, viscous dissipation will govern polymer temperature, otherwise the machine thermal regulation plays the major role. The *Nahme number* balances the viscosity temperature sensitivity and the temperature rise due to viscous dissipation:

$$N_a = \frac{\eta b V^2}{k}$$

(2)

where b is the temperature dependent viscosity factor. When $N_a < 1$, the mechanical

and thermal effects may be decoupled.

Commercial polymers are non-Newtonian and, generally, shear-thinning. The shear stress dependence on shear rate can be simply described by a power-law:

$$\tau = k \cdot \dot{\gamma}^n \tag{3}$$

where k is consistency and n is known as the power law index, usually ranging between 0.4 and 0.6. From a processing point of view this is a positive characteristic that reduces the effects of the high viscosity levels discussed above (again, although viscous dissipation is proportional to shear rate, viscosity decreases when the latter increases, thus viscous dissipation becomes less important). As shown in Figure 5, each of the major processing technologies may subject a polymer to distinct shear rates ranges which, from a machine point of view, poses different drive power requirements (see variation of shear stress with shear rate in Figure 5). In practice, polymer manufacturers develop different *grades* of the same polymer, i.e., materials with a rheological behavior meeting the requirements of a given processing technology and application. For example, grades for injection molding are usually much less viscous than for extrusion, as one aims at producing intricate parts, very often with thin walls, at very high flow rates. While the viscosity level is mainly a function of MW, the non-Newtonian character can be influenced by the molecular weight distribution, MWD: generally, the broader the MWD the less viscous the polymer becomes at high shear rates, little differences being perceived at low deformation rates.
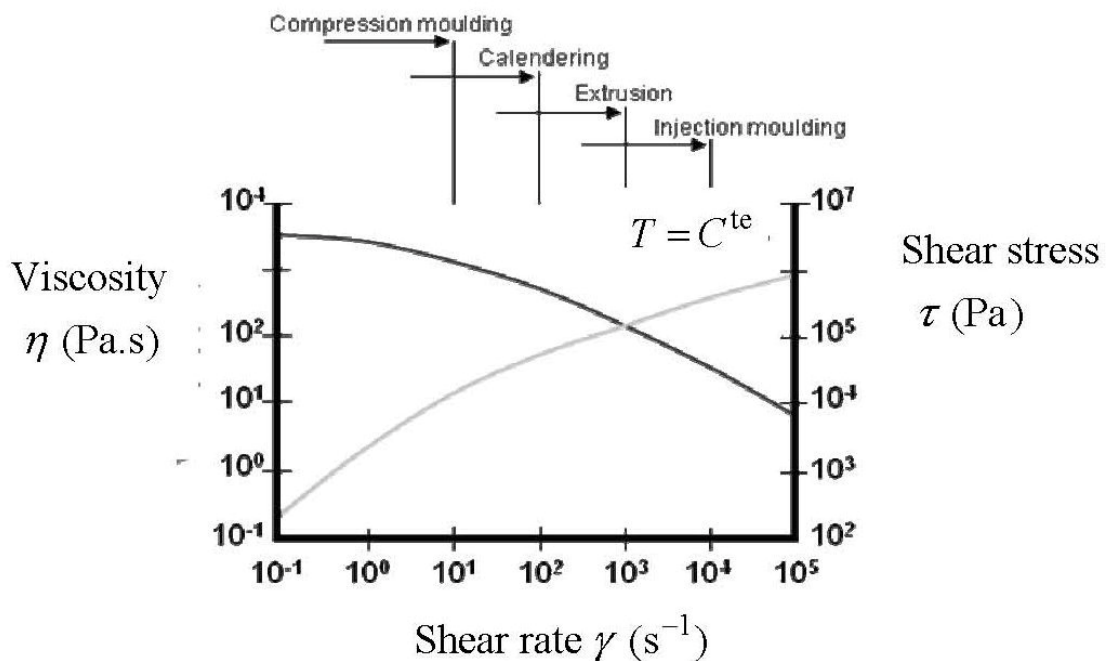

Figure 5. Shear flow curve with indication of the shear rate limits attained by major processing techniques

2.1.3. Viscoelasticity

From a processing point of view, the elasticity component of the rheological behavior of molten polymers is responsible for a number of relevant features:

- During extrusion, the *extrudate swell* of molten polymers at the die outlet is much higher (15-50%) than that observed for Newtonian fluids due to the rearrangement of the flow profiles upon exiting the channel (approximately 12%). In polymers, extrudate-swell is mainly caused by the relaxation of the (non-uniform across the channel) flow induced molecular orientation. It varies with temperature, average shear rate, channel length and distance from the die. In the case of axisymmetric channels, extrudate-swell is seen as an increase of the extrudate cross-section (with a simultaneous decrease of the average velocity, due to mass conservation). However, for any other channel shape, extrudate-swell causes also a distortion of the cross-section (see Figure 6 left, which shows how a square channel will produce a distorted extrudate - what would be the required shape of the die lips to produce a square shape?). The differences between die and product cross-sections are even more complicated in industrial practice, due to the simultaneous contribution of several phenomena: i) the extrudate-swell is not instantaneous, instead it develops until an equilibrium value is attained; ii) immediate cooling of the extrudate is applied, which prevents die-swell to develop fully; iii) stretching of the extrudate by an haul-off downstream, which partially balances extrudate-swell, ensures consistent evolution of the product along the line. Figure 6 right illustrates the typical evolution of a pipe cross-section as it leaves the die.

- The occurrence of rheological anomalies, such as *sharkskin melt fracture*, compromise the surface quality of the extrudate. The term sharkskin is utilized to identify a rough extrudate, whereas melt fracture denotes a more serious disruption of the melt stream. Both are usually associated with the attainment of limiting stresses, which of course have an equivalent in terms of shear rate, hence output. The two phenomena can be combated with a number of measures, from changing the operating conditions (reduce flow, increase melt temperature), using special additives (external lubricants, processing aids), or redesigning the die (decrease convergence angles, avoid sharp exits at the die outlet). A different approach consists in increasing the flow rates to values well beyond those where sharkskin had been observed (this regimen is sometimes known as *super-extrusion*). In principle, the corresponding high wall shear stresses will promote wall-slip, in turn this condition minimizing the tendency for the appearance of sharkskin.

- Polymer melts have significant *extensional viscosities* and, most importantly, some exhibit strain hardening behavior. This characteristic is fundamental in many extrusion processes (for example, in tubular blown film extrusion), and in blow molding and thermoforming, where the melt is subjected to substantial uni-axial or bi-axial stretching before or while being cooled down. If the polymer does not have the appropriate melt resistance, practical processing difficulties arise, or the process may be simply not viable. This was the case of several PP homopolymers before metallocene catalysis and co-polymerization provided the possibility to produce improved grades, e.g., for film blowing and thermoforming.
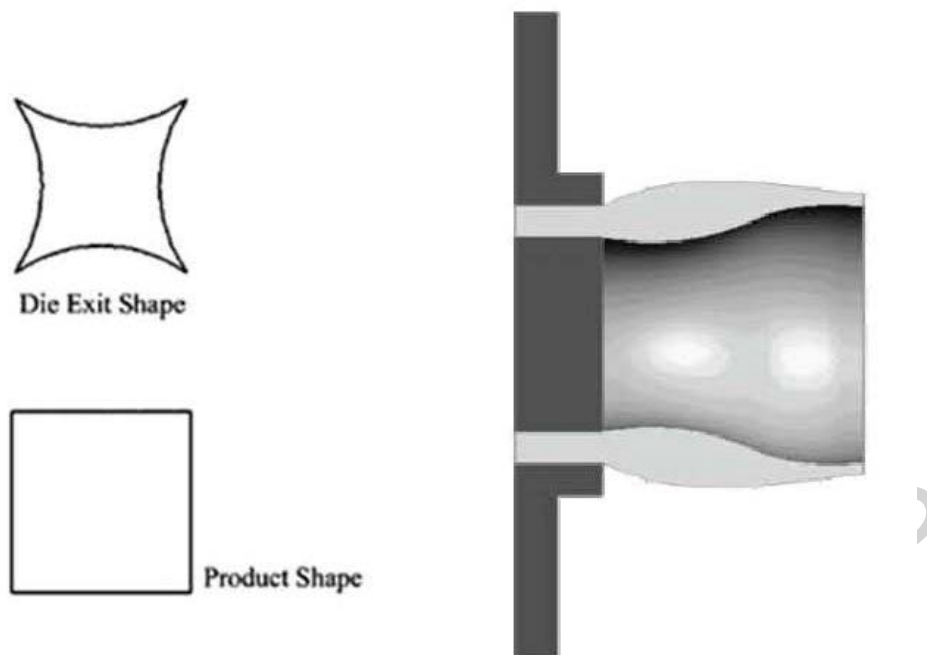
Figure 6. Extrudate swell. Left: Distortion of the flow cross-section; Right: Evolution of a pipe cross-section as it leaves the die.

A more detail discussion on the rheological behavior of polymer melts can be found in the open literature [Barnes et al. (1989); Piau and Agassant (1996); Hatzikiriakos and Migler (2004); Baird and Collias (1998)].

2.1.4. Thermal Conductivity

The thermal diffusivity of most polymers is circa 10^{-7} m^2/s, which is within the range of many insulating systems. As discussed above, low thermal conductivities are useful in terms of applications, but reduce the efficiency of any heat exchanges required during processing (see Figure 4). In fact, this characteristic has the following practical consequences:

o Discontinuous processes, such as injection molding, may necessitate rather long production cycle times, since the material cools down slowly inside the mold. Figure 7 shows the importance of the cooling stage in comparison with the other steps involving the manufacture of a plastic part by injection molding;

o Continuous processes like extrusion will require long lines, with lengthy cooling tanks, in order to guarantee sufficiently high throughputs (in other words, lengthier tanks ensure longer times for heat exchanges);

o Parts made of plastics are generally thin, as they are economically more interesting to produce than thicker ones (they cool faster);

o Plastics processing equipment is generally designed to keep the surface/volume
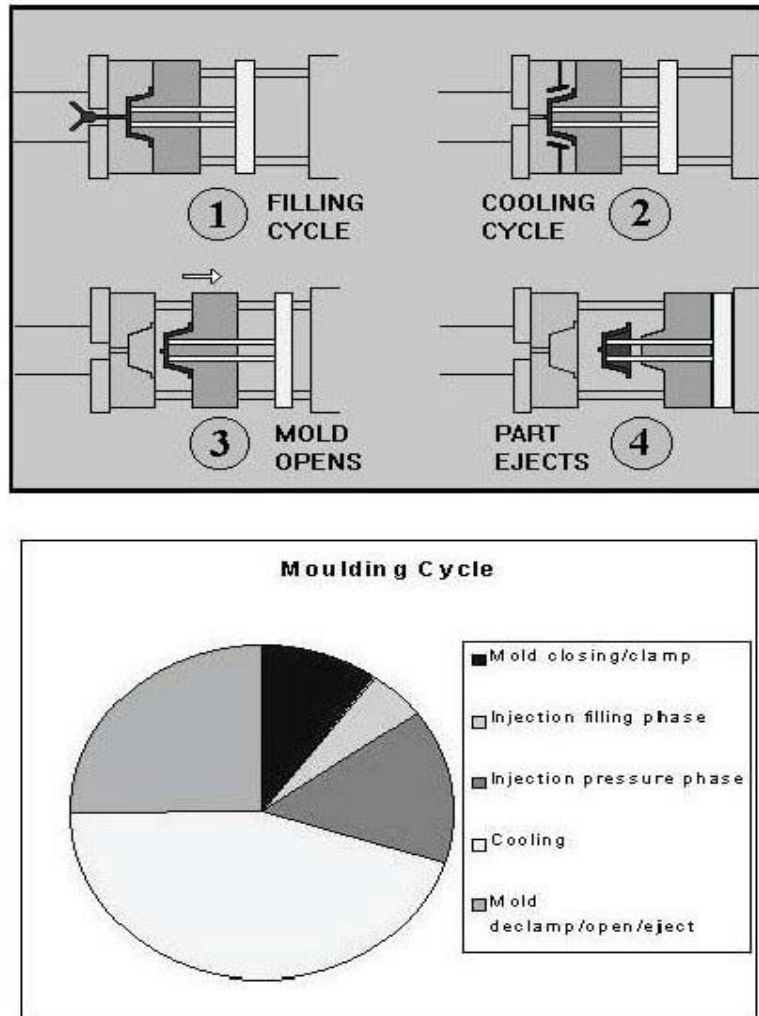
ratios of flow channels as high as possible.

Figure 7. The injection molding cycle. Top: Sequence of operations (source: www.devco-design.com). Bottom: The relevance of cooling (source: www.pitfallsinmolding.com)

As in the case of viscous dissipation, dimensionless numbers can provide an order of magnitude of the thermal phenomena. The *Cameron number* can be used to estimate the temperature rise in a confined flow, by weighing convection in the flow direction against diffusion in the transversal direction:

$$C_a = \frac{\alpha L}{V h^2} \tag{4}$$

Here, α is thermal diffusivity, L is flow length and h is the heat transfer coefficient. If C_a is lower than 10^{-2}, thermal conduction towards the channel wall is negligible (adiabatic condition). If $C_a > 1$, the thermal regime is fully developed and the

temperature will remain constant along the flow channel. The *Peclet number* compares the convection and diffusion terms in the flow direction:

$$P_e = \frac{VL}{\alpha}$$
(5)

Usually, it takes very high values.

2.1.5. Crystallinity

Polymers are either amorphous or partially crystalline, crystallinity taking various forms (spherulites, shish-kebab, lamella …) and percentages typically up to 80%. Several features of a polymer structure may affect crystallinity (chain length, chain branching and interchain bonding), this being an extensive and important topic of polymer physics [Gedde (1995); Bower (2002); Rubinstein et al. (2003)], which can only be treated here quite superficially.
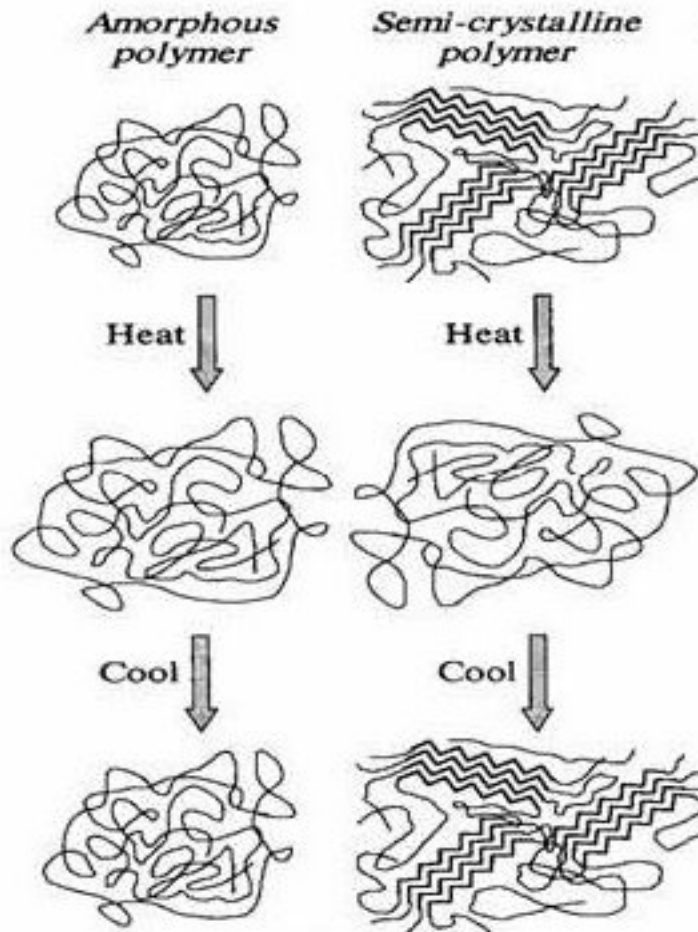

Figure 8. Processing of amorphous and semi-crystalline polymers (adapted from www.nbmold.net)

From a processing point of view, it is important to note that polymer melt flow (see Figure 4) involves the displacement of an essentially homogeneous amorphous material, as it is accomplished either above T_m (crystalline polymers), or well above T_g (amorphous polymers) – see illustration in Figure 8. In the case of semi-crystalline materials, both flow induced and thermally induced (due to cooling) crystallization develops. The rate of thermally induced crystallization (both nucleation and growth) depends on temperature, the fastest rate occurring midway between T_g and T_m. Hence, the longer a semi-crystalline polymer remains between the two temperatures the greater the crystallinity, i.e., in practice there are relationships between processing conditions, microstructure and properties (for example, slow cooling of a PP film will induce larger spherulites and a more opaque product). Conversely, annealing (heating and holding at a temperature below the crystalline melting point, followed by slow cooling) will produce a significant increase in crystallinity, as well as relieving stresses.

Crystalline and amorphous polymers also shrink differently. As a crystalline polymer solidifies, the improved packing due to the formation of crystals leads to shrinkage values far greater than those seen in amorphous polymers. This factor is of great importance when designing processing tools such as moulds and dies, as these tools must compensate for those phenomena.

2.1.6. Molecular Orientation

Given their structure and organization, polymers are inherently anisotropic. If a melt at rest can adopt a random-coil macromolecular conformation, forcing it through dies or cold moulds, or stretching it (i.e., subjecting it to shear or extensional stresses), induces molecular orientation in the flow direction (and, eventually, crystallization, as seen above), whose intensity is proportional to the stress levels and residence time. If solidification due to cooling occurs before the polymer molecules are able to fully relax to their state of equilibrium, molecular orientation is locked in.

The level of orientation dictates the properties that the product will exhibit in service (not only in terms of their values, but also of their differences with direction). For example, most plastic bags used in supermarkets are able to withstand relatively high weights, but they tear easily along their length when carrying volumes with sharp edges. This is because molecular orientation is predominant in the axial direction, hence tensile stresses are resisted by intramolecular forces, while tear is almost entirely opposed by the much weaker intermolecular bonds. Thus, in this example, either adequate operating conditions, or the use of specific processing technologies, should guarantee a balanced bi-axial orientation. Conversely, when producing a fiber, significant axial orientation will guarantee good mechanical behavior.
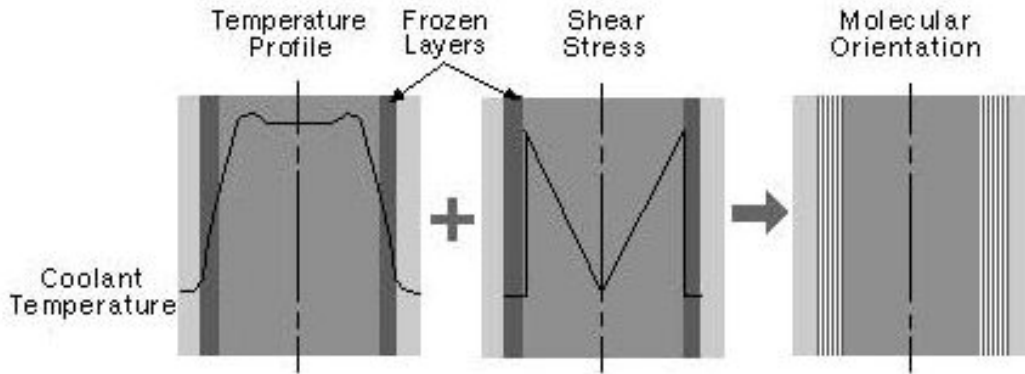
Figure 9. Frozen-in molecular orientation during the filling stage of injection molding

Together, molecular orientation, crystallinity and low thermal conductivities (low thermal diffusivities) can create various important phenomena during polymer processing. For example, Figure 9 illustrates how in the case of injection molding the combined effects of shear stresses and of high cooling rates near to the mould wall (as discussed above, heat transfer is almost negligible in the opposite direction) create highly oriented layers near to the part surface. Due to the thermal insulating effect of these outer layers, the polymer melt in the hot core is able to relax to a higher degree, leading to a low molecular orientation zone (if the polymer is semi-crystalline, the core will exhibit big largely undeformed spherulites, in contrast with the small elongated crystals in the oriented layer).
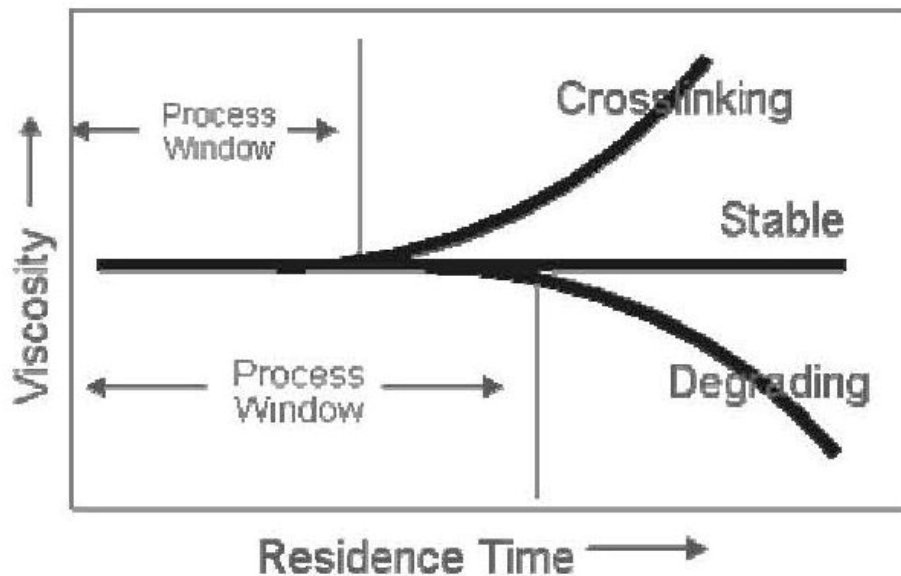
2.1.7. Thermal Stability

Figure 10. Using rheological measures to determine the processing time at a given temperature

In general, polymers have limited ability to withstand high temperatures, but the

susceptibility to thermal degradation varies from polymer to polymer. Rheology can be used to ascertain whether a given material has enough thermal stability to be subjected to a certain temperature during a certain time. By performing isothermal time sweeps, one may determine the amount of time a material can be subjected to thermo-mechanical stresses before cross-linking or chain scission start occurring (see Figure 10).

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Biographical Sketches

João Maia was born on January 2, 1970, in Barreiro, Portugal. He has a BEng in Technological Physics Engineering obtained from the Technical University of Lisbon, Portugal (1992), a PhD in Applied Mathematics – Rheology from the University of Wales Aberystwyth, United Kingdom (1996) and an Aggregation in Polymer Science and Engineering from the University of Minho, Portugal (2007).

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Professor Maia is currently a member of the Portuguese Society of Materials, SPM, the Portuguese Society of Rheology, SPR (of which he is the Vice-President), the British Society of Rheology, BSR, the European Society of Rheology, ESR, the Society of Rheology, SoR, the American Institute of Physics; AIP, the Polymer Processing Society, PPS and the University of Wales Institute of Non-Newtonian Fluid Mechanics, UWINNFM (Associate Member).

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Bruno de Cindio was born on June 30, 1946, in Napoli, Italy. He has a MSc in Chemical Engineering obtained from the University of Napoli "Federico II", Italy (1972).

He was Assistant Professor and Associate Professor at the Department of Chemical Engineering of the University of Napoli "Federico II", Italy, since 1977 to 1993; he was Associate Professor at University of Calabria since 1993 to 2001 and he is currently Full Professor at the Department of Engineering Modeling of the University of Calabria where he has been a Faculty member since January 1993. He published more than 100 papers on national and international journals, more than 200 contributions in proceedings of International and National refereed conferences and he is co-author of 1 patent concerning a novel apparatus for rheological properties determination. Main areas of his scientific activity are: Rheology of complex systems (foods, emulsion, suspensions, biological fluids), setup and optimization of novel rheological techniques, Thermodynamics of complex systems, Food process engineering.

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Domenico Gabriele was born on May 30, 1971, in Cosenza, Italy. He has an MSc in Chemical Engineering obtained from the University of Calabria, Italy (1997), a PhD in Chemical Engineering, concerning Food Rheology, from the University of Napoli "Federico II", Italy (2000).

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