Improving the properties of polymer blends by reactive compounding
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CHAPTER 7 MODELLING AND EXPERIMENTAL EVALUATION OF MIXING IN AN INTERMESHING COROTATING TWIN SCREW EXTRUDER

Abstract.

Using the temperature modelling described in the previous chapter, mixing of two polymers in the extruder has been modelled in this chapter. Coalescence is included in the modelling. This model and the model in chapter 6 (temperature and mixing) will be used in chapter 8 to model reactive compounding in the extruder. Finally this will be used to improve the process control of reactive blending, as described in chapter 9. The size and deformation of the dispersed phase of a blend mixed in the intermeshing corotating twin screw extruder have also been measured. By comparing the measured and calculated size of a blend for several extruder parameters the model developed will be validated. The influence of most of the processing parameters will be determined. The throughput, the temperature of the barrel, the geometry of the screw and the screw speed have been varied.

1 Introduction to mechanical properties of blends.

The model developed in this chapter is a one dimensional model for the average size of the dispersed phase. It is obvious that the final size distribution can only be predicted when all parameters of interest to mixing are known at every 3D-position of the extruder, which is not done here. Mixing, as will be described in this chapter, is of vital importance for improved mechanical properties of blends (which is the final goal of this thesis). Sometimes combination of the properties of two polymers can be achieved by merely mixing. However improving the mechanical properties of a blend is often needed since in many applications of blends the properties get worse instead of better. Especially the morphology of the blend has been investigated in the literature (1-10) as well as blending and extrusion (11-24). In the conventional way the mechanical properties of a blend are improved by improving the mixing of the different parts of the blend. However in doing so for PS usually the mechanical properties were very poor. Therefore we first need to understand what determines the mechanical properties of an amorphous polymer. In a second step we also need to understand what determines the mechanical properties of a blend. In the literature it has been stated that for optimal mechanical properties of PS blends the dispersed phase has an optimal size (14). This has been found for blends where there was no chemical adhesion between PS and the dispersed phase. A small size means a large stress concentration but also a larger interface.
For a pure polymer it is well known from the literature that the structure of the chain of an amorphous polymer has a large influence on its mechanical properties. Kramer et al (7) ascribed the transition between brittle and ductile behaviour to a low value for entanglements resulting in a higher surface tension (see also chapter 10). Wu (6) studied the effects of the size of the dispersed phase for blends with rubber as the component added. He found that the distance between the particles of the dispersed phase of a blend should be smaller than a certain critical inter-particle distance. This was shown to be one of the determining factors, whether a blend was tough or brittle. The minimum adhesion required for toughening was also discussed. It was found that the critical size of the dispersed phase of a blend depends on the rubber contents. PS is the matrix phase in most of the blends studied in this thesis. The toughness increases with decreasing particle size of the dispersed phase for a blend with a matrix of this type of polymer. The highest toughness was found for blends of PS and rubber at an optimum particle size of 2-5 µm.

With the results of the previous chapter where the temperature modelling has been developed (and verified with measurements) we now are able to develop a computer code to model dispersive mixing of two polymers in the extruder.

1.1 Mixing in the intermeshing corotating twin screw extruder.

In the intermeshing corotating twin screw extruder mixing mostly takes place in the kneading section due to shear and/or elongation. The dispersive mixing performance of the kneading elements in the intermeshing corotating twin screw extruder has been studied only by a few investigators. The minor component in many blends is the dispersed phase of a blend (drops or filaments) mixed in a continuous phase of the major component. Heating, deformation and break-up of the dispersed phase occur during mixing. An elementary step in the mixing process is the deformation of a dispersed drop in a flow field. An increase in the interfacial area between the two components is accompanied by a decrease in local dimensions perpendicular to the flow direction (the striation thickness). Drop deformation is mainly governed by the capillary number and the ratio of the viscosities of both phases. Any mixing model should incorporate the governing mechanisms in a principally transient way in order to be applicable to a practical situation.

A number of papers have been published dealing with morphology development during processing. Most working models developed measure morphology development in a single-screw extruder. Not many models for mixing in a corotating twin screw extruders with these break-up models are known so far. Elmendorp (8) has reported most of the expressions needed to model the deformation and break-up time of the dispersed phase of a blend during mixing of immiscible fluids. The size of the dispersed phase of a blend in a 2-zone mixing model with an estimated value for
the shear rate has been modelled by Janssen (17). The results of his calculations showed a
decrease in size of the dispersed phase of a blend to approximately 1 μm after passing 6
kneading elements. However, the values of the elongation and shear rate were not based
on real-case values for twin screw or single screw extruders. Sufficiently accurate values of
the shear and elongation rate can be calculated from 3D modelling (20, 21) which is used
in our calculations.

The time needed for deformation and break-up of the dispersed phase of a blend into
smaller spheres has been studied for a number of decades. In particular the time scales of
the rheological processes must be considered to determine whether the available process
time in the extruder suffices for a specific event to occur.

Wu (6) published a dimensionless master curve of the Capillary number, \( \gamma \eta d/s \), versus the
viscosity ratio, \( p \), for melt extrusion in a corotating twin screw extruder for several blends
of PET, nylon, and EP rubbers. From these curves it is clear that the size of the dispersed
phase of a blend can only decrease due to elongation for a viscosity ratio larger than 3.5.
From our earlier calculations shear rate was found to dominate elongation rate in the flow
of the twin screw extruder used in our experiments except for in the intermeshing regions.
Both, the shear and elongation rate, have been incorporated in our calculations.

2 Modelling of mixing in the corotating twin-screw extruder.

The 3-D flow and temperature profiles were calculated in chapter 2, 3, and 4 and partially
validated in chapter 6. Deformation, break-up, the break-up time of the dispersed phase in
the melt, the residence time, and the occurrence of coalescence have been calculated.

The effectiveness of mixing of two polymers is influenced by several processes:

- The heating or cooling of the polymers by the barrel wall and heating by viscous
dissipation determines the melting of the mixture of polymers and therefore the position
where they start to mix.
- The shear rate in the melt determines the deformation of the dispersed phase in the fully
filled sections.

The model to describe mixing in the extruder has been based on the following
assumptions:

A subdivision of the mixing process can be based on the local capillary number in the
extruder. The capillary number decreases continuously along the extruder due to the
decreasing size and is calculated at every axial position at the modelled temperature and
viscosity.
The extended lamellar pattern of striations becomes unstable due to periodic distortions and striations rupture into fibrils and later into globules. Depending on their size these drops may eventually be stretched and broken again unless the interfacial tension, $\sigma(T)$, protects them against further deformation. The parameters which are calculated at every temperature and position along the extruder are:

$$\frac{\eta_1}{\eta_2} = \rho; \text{the viscosity ratio between dispersed (1) and continuous (2) phase}$$

$$\frac{\gamma kd \eta_2}{\sigma} = \text{Ca; the capillary number : We/Re}$$

$$\frac{\rho vd}{\eta_2} = \text{Reynolds number}$$

$$d; \text{diameter of the sphere}$$

$$\frac{t_b}{t_r} = \text{dimensionless time}$$

Formation of thin layers occurs mostly in kneading sections in the corotating twin screw extruder. Due to the layer-forming mechanism the phase size decreases tremendously in the first minute.

Note that:

- Little mixing occurs in the powder transport section. Also little mixing is expected in the melt transporting section where shear rate will be very low in comparison to the kneading section. In the initial stage, under conditions where both polymer components are melting, the pellets produce fine lamellar structures. In the direction of the die of the extruder, the thickness of these striation layers is reduced due to stretching when $Ca >> Cacrit$.

- The kneading section in the extruder is completely filled.

- In the first set of kneading elements the dispersed phase has not been melted and therefore no deformation occurs on this location. This delays break-up of the dispersed phase and decreases the mixing efficiency.

- The transporting elements are not completely filled, which makes the heating of the mixture less efficient.

- Mixing takes place mostly in the fully filled kneading section of the extruder due to high shear- and elongation rates. The flow of material between the flights and the barrel has been neglected.

- It has been assumed that the average size distribution of the dispersed phase of a blend results from the average residence time and the average shear in the kneading elements.
These assumptions have been validated and found to be reasonably accurate in chapter 2, 3, and 4.

- If no break-up of the dispersed phase takes place, the average diameter of the dispersed phase of a blend will relax to its original size. This is the case when the residence time is shorter than the time needed for break-up.

- After break-up, the dispersed phase of a blend consists of deformed droplets that relax (within less than one second) to spherical shapes. The spherical shape results in a maximum volume/surface ratio of the dispersed phase of the blend.

- Coalescence of the dispersed phase has been taken into account even when only very small amounts of the dispersed phase of a blend are used.

- Although most polymers exhibit viscoelastic behaviour, our modelling has been based on an analysis for Newtonian liquids.

### 2.1.1 A simplified modelling of the average size of the dispersed phase of a blend.

In a simplified approach it can be assumed that the sphere breaks up into two spheres. The rate of deformation has been taken into account by dy/dz, figure 1a. If the shear rate is small the deformed sphere in figure 1a will have only a small angle with the vertical axis.

The deformation and break-up mechanism of the dispersed phase of a blend is independent of the size of the dispersed phase and can be found by calculating the volumetric deformation in a shear field, as shown in equation 4.

For this simplified approach it is assumed that the surface tension is zero and the total volume of the sphere stays the same while the dispersed phase deforms. The volume of the deformed sphere is constant. The deformation is the ratio between dz and dy in figure 1a. The volume of the dispersed phase is constant as long as no break up occurs for which \( d_1 \) is the diameter of dispersed phase before deformation. The simplified approach leads to equation 2 and 3 while a detailed approach provides equation 4:
if deformation is linear in shear:

\[ \frac{\partial d_1}{\partial t} \cong -C_{\gamma} \gamma_1 \cdot \frac{d_1}{d} \] (2)

factor which takes break up into two spheres into account:

\[ C_{\gamma} = 1 - \frac{1}{3/2} \] (3)

Elmendorp: \[ \frac{\partial d_1}{\partial t} \cong \gamma_1^{0.5} \cdot \frac{d_1}{d} \] (4)

where \( \gamma \) is the shear rate while the dispersed phase changes as dv/dz (in the flow field as a function of the axial parameter z). From equation 4 an approximation of the final size of the dispersed phase can be found. However this is only valid in a pure shear flow. Under the assumption made an estimation of the deformation of the sphere can be calculated. In a more extensive research the deformation as described in the thesis of Elmendorp has been found which is therefore used in our modelling.
2.1.2 Modelling of the average size of the dispersed phase of a blend.

The opposing force against deformation is the surface tension. Therefore the force due to surface tension is subtracted from the deformation force, resulting in the following semi-empirical equations for the size of the dispersed phase which can be used as an estimation:

\[
\frac{\partial d}{\partial t} = -(C_1 \cdot \frac{\sigma_d^{0.5}}{d} - C_2 \cdot \frac{\sigma}{\eta})
\]

\[
\tau_m = \tau_d \cdot \dot{\gamma}_d = \gamma_m \cdot \frac{\eta_m}{\eta_d}
\]

\[C_1, C_2: \text{constants}\]

At the start of the break-up process the surface tension usually is small compared to the first term on the right hand side in equation 5. In the computer calculations the shear and elongation rate at every rotation speed and throughput are calculated for the extruder (11, 18). From chapter 4 it could be concluded that the elongation is small in the channel. The equations used for the average shear- and average elongation rate in a kneading paddle were derived in chapter 2, 3, and 4. Equation 3 is a strongly simplified version of the reality of mixing. Elmendorp gives the equations for the deformation and mixing of the dispersed phase in a blend (equation 6).

\[d \approx \frac{\gamma}{1/2}\]

After deformation break up of the dispersed phase takes place after some time. The total strain (elongation and shear) applied to the dispersed phase in the extruder can be expressed as:

\[\gamma = (\dot{\gamma}_f \cdot t_f + \dot{\gamma}_c \cdot t_c) \cdot \frac{2 \cdot t_r \cdot N}{60}
\]

\[f: \text{flight, } \quad c: \text{channel}\]

\[N: \text{rpm}\]

\[t_r: \text{residence time at the actual throughput}\]

At every axial position in the kneading section of the extruder, the deformation and the time for break-up of the dispersed phase is calculated with expressions from the literature (8, 17). The time for break-up has been calculated with:

\[t_b = \frac{\eta_v(T)R_0}{\sigma(T)\Omega} \ln\left(\frac{10^{23} \sigma(T)R_0^2}{T}\right)
\]
Where $\Omega$ is a constant which is calculated with the expressions of Tomotikata (10), and $R_0$ is the thickness or minor axis of the long slender body formed during deformation. The time needed for break-up in a simple flow has been studied elsewhere (8). If the time needed for break up is too long, compared to the average residence time, the average size of the dispersed phase of a blend will not decrease over the axial position in the extruder. The algorithm in the modelling at every axial step along the extruder is therefore:

- Evaluate the average residence time in the (kneading and transporting) channel.
- Compare the residence time with the break-up time.
- Dispersive flow can be obtained if the residence time in a high shear flow is longer than the time for break-up and the viscosity ratio is smaller than 3.5, since the flow is mostly shear flow in the extruder and break-up and deformation is mostly due to shear rate.

The deformation of the dispersed phase of a blend leads to a large length over width ratio ($L/B$). The long slender bodies formed will break-up into spheres with a diameter comparable in size with $B$.

In many cases the equilibrium size (when $d$ is not anymore changing in time as shown in equation 6) is never reached because equilibrium between coalescence and break-up of the dispersed phase is reached at a larger size of the dispersed phase. This size was described by Favis (5, 22, 23) as:

$$d \approx \frac{24P_r \phi \sigma(T)}{\pi \eta(T) \dot{\gamma}} \left(1 + \frac{4P_r \phi E_{dk}}{\pi \eta(T) \dot{\gamma}}\right)$$

(9)

where $P_r$ is the probability that a collision between two spheres will result in coalescence, $j$ is the volume fraction of the dispersed phase, and $E_{dk}$ is the bulk breaking energy.

With increasing shear (and/or elongation) the average size of the dispersed phase of a blend decreases. This size of the dispersed phase decreases until equilibrium in equation 6a is reached.

Mixing has been investigated by many authors such as Chella and Ottino who presented an analysis of mixing in cavity flows and in single screw extruders (24).
3 Experimental set-up.

PS/HDPE mixtures are extruded with a Baker Perkins 50 mm, self wiping corotating twin-screw extruder (figure 1b). Two different screw configurations were used as also described in (2). Samples were taken at the die and studied with SEM (figure 1c, In the figures um means $\mu m = 10^{-6} m$).

![Screw geometry](figure1b)

$figure\ 1b\quad screw\ geometry.$

![Morphology of the blend measured with SEM](figure1c)

$figure\ 1c\quad Morphology\ of\ the\ blend\ measured\ with\ SEM$

The experimental procedure contained the following measurements:
-Pressures are measured at the die and 80 mm before the die and were in reasonable comparison with the calculated values. The temperature of the polymer in the melt at the kneading section is measured with an IR thermometer.

-Visually the degree of fill is estimated in the partially filled transporting section. Directly after a sample is taken it is cooled with liquid nitrogen. With S.E.M the morphology of the blend is made visible from which the average length and thickness of the dispersed phase is measured, figure 1c.

4 Results and discussion.

The average size of the dispersed phase of a blend, calculated with our computer model, has been validated. Only the calculated size of the dispersed phase in the kneading section will be shown since little mixing is found in the partially filled section. The influence of several processing conditions on mixing are studied such as the temperature of the melt, residence time, and shear rate. For the matrix Styron 7000 (PS, SHELL) was chosen. The barrel temperature was 200 °C and the number of kneading elements 7.

4.1 Modelling the average size of the dispersed phase in the kneading section.

The thickness of the dispersed phase of a blend is chosen as the best measure for its deformation since often the length of the dispersed phase can not be determined with SEM. In figure 2a the development of the thickness of the dispersed phase as calculated in the kneading section is shown. The thickness of the dispersed phase is constant in the partially filled section and decreases in the kneading section.

![Thickness versus Axial Position](image)

*figure 2a  thickness versus axial position in extruder.*
figure 2b  *Length of dispersed phase versus axial position in the extruder.*

The length of the dispersed phase increases to a value which is very large relative to the small volume of the channel in the kneading section, figure 2b. Since the thickness of the dispersed phase decreases the Capillary number also decreases along the axial position in the extruder, figure 3.

figure 3  *Capillary number versus axial position in the extruder.*
Usually it is assumed in the literature that small perturbations in the flow cause waves at the interface of the dispersed phase. These waves can grow and cause break-up of the dispersed phase. The time for break-up depends on this wave but also on the type of flow in the extruder. A sudden change of thickness of the dispersed phase causes a jump in the capillary number in figure 3. After the material leaves the kneading section at 0.47 m the (effective) shear rate drops to a smaller value resulting in a jump in the Ca number. The thickness of the dispersed phase (B), figure 2a, decreases resulting in figure 3, $Ca = \frac{\eta B}{\sigma}$.

Average values of the shear rate have been used. In order to properly describe mixing in the extruder a complicating factor is that all the processes take place in a very complicated 3-D flow. Folding of the dispersed phase, which has the shape of a long slender body, may cause an enhancement of the break-up especially in the intermeshing region of the twin screw extruders (4). However no proof was found in the literature that this phenomena needs to be included in our modelling. Therefore reorientation and details from the 3-D flow in the extruder were not taken into account in the calculations.

When the capillary number in figure 3 is decreasing it will become smaller than the critical capillary number. Only when the thickness of the dispersed phase is small enough, the time needed for break-up of this long slender body is shorter than the residence time available and break-up can take place.

![Figure 4a](image-url)

**Figure 4a**  The time for break-up versus axial position in the extruder.

The time for break-up decreases due to the decrease of the thickness of the deformed dispersed phase along the axial position in the extruder, figure 4a. An important parameter
in these calculations is the average temperature ($T_b = 200 \, ^\circ C$) as modelled earlier in chapter 6. The time needed for break-up decreases with increasing rotation speed because the diameter of the thread decreases with increasing rotation speed, figure 4b.

**figure 4b**  the time for break-up after 7 kneading elements, $Q = 3 \, \text{kg/h}$, varying $N$.

Increasing the rotation speed usually causes an increase of the deformation and therefore a decrease of the thickness of the deformed dispersed phase. However other parameters such as the temperature of the melt, are also influenced when the rotation speed increases. The distribution in size of the dispersed phase is measured with SEM, figure 5.

**figure 4c**  the time for break-up after 7 kneading elements, $N = 60 \, \text{rpm}$, varying $Q$. 
usually a broad distribution of the size of the dispersed phase is found, figure 5. The diameter of the dispersed phase as calculated suddenly drops by a factor 1000 in a very short length in figure 6. This occurs in the kneading section because the shear rate and
residence time is relatively large. The dispersed phase first has a large deformation before break up occurs.

4.2 The influence of the rotation speed of the screws.

One of the most important parameters for mixing is the rotation speed of the extruder. With increasing rotation speed the shear rate increases, the viscosity decreases and the temperature of the melt increases (18). The size, the thickness, and the length of the dispersed phase will be calculated at the position of the seventh paddle of the kneading section.

![Graph showing equivalent diameter versus rotation speed](image)

**figure 7a** Equivalent diameter $(6V/\pi)^{0.33}$ versus rotation speed;

$Q = 3 \text{ kg/h}, T_b = 200 \degree C, \text{ number of kneading elements : 7.}$

As can be expected the average size of the dispersed phase decreases with increasing rotation speed, figure 7a. If the rotation speed increases the length of the dispersed phase decreases if break-up occurs. When no break up occurs the length of the dispersed phase increases due to an increase in deformation. In figure 7b break up is delayed for $N$ smaller than 270 rpm. This is because the residence time is smaller than the break-up time at $N < 270 \text{ rpm}$ and therefore the dispersed phase did not break up.
Increasing the throughput causes a decrease of the residence time which causes a decrease of the length of the dispersed phase and a decrease of the temperature of the melt as shown in figure 8a. With decreasing length of the dispersed phase the thickness of the dispersed phase increases. This increase in thickness results in an increase of the break up time. Combined with a shorter residence time this leads to less break up and a larger size of the dispersed phase, figure 8b.

figure 7b  The length of the dispersed phase versus rotation speed , $Q = 3$ kg/h.

figure 8a  Length ($L$ [m]) and temperature ($T_{melt}$) versus the throughput of the extruder, $N = 60$ rpm.
The time for break up of the dispersed phase at the last paddle of the kneading section has been calculated. From the calculations it is found that the break up process is strongly influenced by the temperature (equation 8) and the thickness of the dispersed phase. We find that the size of the dispersed phase is determined by coalescence if the blend contains to much of the minor phase.
4.3.1 Comparison between modelling and measurements, PS/HDPE blends.

The calculated size of the dispersed phase will be compared with the average values of the size of the dispersed phase as measured. Calculation of the distribution of the size of the dispersed phase is possible with the results from chapter 4. However due to a lack of time no 3-D residence time distribution was calculated (11). For simplicity it will be assumed that the average size distribution of the dispersed phase in figure 5 results from the average residence time and average shear in the kneading elements. The extruder has vent ports at two axial positions before and after the kneading section. The vent ports are at the top of the extruder, above a partially filled transporting element.

![Figure 10](image)

**Figure 10** Thickness of the dispersed phase (calculated and measured) versus \( Q \), \( N = 314 \text{ rpm}, T_b = 200 \, ^\circ \text{C}, 7 \text{ kneading paddles} \).

The average thickness of the dispersed phase as calculated at a position directly after the kneading section is comparable with the measured values, figure 10. With increasing throughput the residence time and temperature decrease, both having an influence on the size of the dispersed phase. Usually an increase of the throughput has the opposite effect, as an increase in rotation speed on the deformation of the dispersed phase. The most important reason for this is the decrease of residence time, specific energy, and shear.
4.3.2 Comparison of our computer modelling with measurements of others.

Favis et al. (23) measured the size and size distribution of the minor phase in melt blended polypropylene/polycarbonate blends in a ZSK30 with a barrel temperature of 240 [°C] and a rotation speed between 100 and 300 [rpm].

![Figure 11](image1.png)

**Figure 11** Calculated results and measurements by Favis (5).

![Figure 12](image2.png)

**Figure 12** Calculated results and measurements versus viscosity ratio, $p$ by Favis (5).

Little influence of the rotation speed was found, figure 11. The line represents values from our computer calculations for which the rheological data and surface tensions were used as
given by Favis (22). From our calculations it became clear that, due to a very long mixing section used and the very large rotation speeds, the size of the dispersed phase is determined by the equilibrium between mixing and coalescence (5 % PP was mixed in).

![Figure 13](image)

**Figure 13**  Calculated results and measurements (by Favis (5)) versus volume of the dispersed phase.

The calculated size of the dispersed phase as a function of the viscosity ratio again shows a very good resemblance to the measured values of Favis, figure 12. Mixing efficiency decreases if the viscosity ratio increases. Mixing is obviously determined by shear, but also a little by elongation. If the flow was purely shear no mixing would have been found for \( P>4 \), according to Grace (16).

An increasing viscosity ratio causes an increase in the size of the dispersed phase in figure 12. Increasing the volume of the dispersed phase increases the minimum attainable size due to an increasing coalescence. The size of the dispersed phase is calculated correctly with the computer calculations, figure 13.

### 5 Conclusions.

For optimising polymer blend properties it is essential to control and to predict the size of the dispersed phase of a blend during mixing in the extruder. The shear rate in the extruder, due to the rotating movement of the screw, causes the shape of the dispersed phase of a blend to deform. Due to the break-up of the deformed
dispersed phase of a blend its average size decreases. From the comparison of the measured and the calculated values of the size of the dispersed phase a number of conclusions are found:

- The calculated values are found to be in reasonable agreement with the measured ones.
- The time needed for decreasing the size of the dispersed phase by break-up is very short for thin threads. Nevertheless a dispersed phase with the shape of a thin thread is found for many practical situations of extrusion where polymers are mixed.
- If the kneading section is long enough the size of the dispersed phase of the blend is in equilibrium with the surface tension. For a higher shear rate the equilibrium value for the size of the dispersed phase is smaller.
- To calculate the size of the dispersed phase coalescence is a very important process to take into account.

Nomenclature.

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<tr>
<td>A</td>
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<td>C_p</td>
<td>Heat capacity</td>
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<td>(axial) Length</td>
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Greek symbols

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Subscripts
b break up
r residence
c channel
f flight
k kneading element

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11 Chapter 4, this thesis.
14 S. Wu, Polymer, 26, 1855 (1985).
18 Chapter 6, this thesis.