#### **Residence Stress Distributions in a Twin Screw Extruder**

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## Residence Stress Distributions in a Twin Screw Extruder

*David Bigio, William Pappas, Harry Brown II, , Beserat Debebe and William Dunham, University of Maryland, College Park, MD* 

## Abstract

An experiment has been created to directly relate the stress distribution history (RSD) with the Residence distribution. Stress beads are used to determine the percentage of material that experiences a certain amount of stress at each location on the residence distribution. This experiment directly relates stress data as a function of the residence distribution and will be recorded for a range of specific throughputs. A comparison of a mixing section comprised of narrow versus wide kneading blocks is performed to demonstrate the power of the approach.

# Introduction

Extrusion is a viable technique for creating an array of different products across a broad spectrum of disciplines. Examples include pipes and tubes, chemical pressure-sensitive adhesives, and active ingredient patches for pharmaceutical industries [1]. One of the more intriguing applications for extrusion is the production of composite materials using fillers, such as carbon nanofibers. This process produces an extruded material with altered properties than that of the base polymer, such heightened thermo-conductive and mechanical strength properties. There are several factors that determine the change in properties of an extruded material. One such factor is the amount of mixing a material within an extruder experiences. To be able to determine the degree of mixing, it is important that one accurately model the stress that is induced for a given screw geometry and operating condition.

Mixing with a twin-screw extruder is categorized as either distributive or dispersive. Distributive mixing uniformly distributes particles throughout the melt and dispersive mixing breaks large particles and distributes them throughout the melt. Narrow kneading blocks are used for distributive mixing as the melt flows in and around the paddles, producing low shear. Dispersive, however, is a high shear process as it utilizes wide kneading blocks. Wide kneading blocks force the material around the blocks that is squeezed between the paddles and the barrel wall [2].

There have been numerous attempts in the past to find a correlation between stress in an extruder and a residencetime distribution. Efforts have been made to use the residence distribution to imply intensity of mixing by various methods, e.g.; Peclet number or a number of passes [3]. Typical residence distributions, however, only provide an axial history of the flow and give no information regarding the stress history. There have also been some attempts to quantify the stress in an extruder. Curry et al using the percent breakage of glass beads as a way to quantify the stress under different conditions and screw designs [4, 5]. The limitation of the approach is the effort to obtain a few values along the residence distribution. Gallant used stress beads to quantify the maximum stress in an extruder for an energetic application [6]. Cheng et al used stress beads to characterize different mixing section designs [7].

The purpose of this research is to establish the methodology to measure the Residence-stress Distribution (RSD) and compare it to the RTD. Another goal is to determine the stress history of different screw designs using narrow and wide kneading blocks and whether it can distinguish the difference between different screw geometries, as a function of operating conditions.

### **Materials**

The extrusion material used for this experiment was high-density polyethylene (HDPE) Alathon H6018 in pellet form from Equistar Chemicals. The HDPE had a density of 0.960 g/cc and a melt index of 18.0 g/10 min.

Calibrated microencapsulated sensor (CAMES) beads from Mach I, Inc. were utilized as the method to measure the stress in the extruder. The beads are designed to measure shear stress during mixing by breaking at specific critical stress levels which are dependent on the bead diameter. The selected range of diameters for the experiment was 53 -  $63\mu$ m. When the beads are subjected to stress levels beyond the critical shear stress, the beads break and release their contents (Red B Disazo dye) which stain the extruded material.

Using the same dye (Red B Disazo) that is encapsulated by the CAMES beads, "reference" dye shots were produced. These shots were prepared by dissolving polystyrene pellets in xylene, and adding concentrated amounts of dye. The solution was allowed to solidify over a 24 hour period. The batch was then cut and weighed to the appropriate amount to match that of the measured CAMES shots. The dye shots were used to produce reference RTD curves that represented 100% breakup of the CAMES beads.

#### Equipment

The experiments were performed using a 28-mm Coperion CoTSE extruder with an L/D = 32 using two screw configurations of interest. The melting section of both screw designs remained unchanged for the duration of the experiment and consisted of three right-handed kneading blocks (15, 30, and 45-mm), two neutral blocks (20-mm), and one left-handed kneading blocks (15-mm). The mixing section of the first screw design contained two right-handed kneading blocks (15- and 45-mm) and one left-handed convey element (15-mm) with a 5-mm pitch. The second screw configuration consisted of five righthanded kneading blocks (15-mm), one neutral block (20-mm), and one left-handed kneading blocks (15-mm) with a 5-mm pitch. The second screw configuration consisted of five righthanded kneading blocks (15-mm), one neutral block (20-mm), and one left-handed conveying element (15-mm) with a 5-mm pitch. The extruder set up consists of a feed port and one vent port just before the mixing section, as seen in Figure 1. The extruded polymer was cooled via a chill roller where it was then collected as strips.



Figure 1. 28-mm Coperion Co-Rotating TSE

A reflective optical probe was placed just before the die of the extruder. The probe consists of a split fiberoptic bundle, where light was transmitted from one fiber bundle. The light entered the melted polymer, reflected off the polymer and screw elements, and was collected by the other fiber bundle, where it was converted into a voltage signal.

# Experiment

The extruder and pellet feeder were then set to the desired operating condition. The five barrels were set to a temperature of 160oC and the die was set to  $155^{\circ}$ C. The data acquisition program began sampling for 15 seconds to establish a baseline. Due to the transparent nature of HDPE during extrusion, TiO<sub>2</sub> pellets were fed at a constant rate during the experiment to provide a white background on the polymer melt for the optical probe. The solid pieces of dye were fed into a channel via the vent port as an impulse. Since small amounts of the dye shots were fed into the

extruder, the concentration of the dye and polystyrene had a negligible effect on the viscosity of the HDPE melt. Once the stained polymer had been completely extruded, the data acquisition program was reset and the program was again run for 15 seconds to establish a baseline. CAMES beads were then fed into the vent port. In total, nine operating conditions were tested, with one dye and three CAMES shots fed per condition.

#### **Experimental Grid**

A central composite design (CCD) of experiment grid was used to layout the data to show relationships between percent breakup and different operating conditions, as seen in Figure 2. This approach was chosen because of the expected non-linear relationship between breakup and the chosen parameters. The specific locations on the grid represent the operating conditions for this study.



Figure 2. CCD grid of experimental conditions

The ranges of the conditions were determined by the capabilities of the extruder. The first number of a condition represents the throughput, Q, measured in mL/s. The second number represents the screw speed, N, in rpm. N vs. Q/N was chosen because RTDs scale with Q and N. It was found that Q/N was a key variable in determining the characteristics of RVDs and RRDs, which will be of interest in a later study [8].

In order to ensure that the stress beads would break in the designed operating region, viscosity data was generated using a Rheometrics RDA-III rheometer and the results plotted in Figure 3.



Figure 3. Shear viscosity for HDPE at 160°C

Furthermore, the slope of this graph was used to calculate the shear stress by using the equation:

$$\tau = \eta \dot{\gamma}^{(n+1)/n} \tag{1}$$

Where  $\tau$  is shear stress (Pa),  $\eta$  is viscosity of the polymer (Pa-s), ? is shear rate (1/s), and n is the slope. The shear rate is defined as:

$$\dot{\gamma} = \frac{\pi ND}{h} \tag{2}$$

where N is screw speed (rpm), D is the screw diameter (mm), and h is the channel height (mm).

Using the dimensions of the 28-mm conveying element that has a channel height of 4-mm, the stresses induced at each operating screw speed were evaluated and are displayed in Table 1.

Screw Speed, N (rpm)	Shear Stress, τ (Pa)
40	101.9
75	123.7
110	148.4
145	181.6
180	193.3

Table 1. Calculated shear stress

The approximate critical stress value at which the CAMES beads break at was determined to be 120 Pa, using data obtained from MACH I. Table 1 shows that, for the given range of screw speeds, the amount of bead breakup would occur at a low level for the lower screw speeds and increase at the highest speeds.

#### **Experimental Results**

#### **Percent Breakup**

The average percent breakup for all nine experimental conditions was calculated and the results inserted into the Central Composite Design grid for analysis as a way to compare the breakup history in the wide and narrow kneading block configurations. To determine the percent breakup of CAMES beads, the areas under the RTD and RSD curves were calculated and used in the followed equation:

Percent Breakup = 
$$\frac{A_c}{A_r} \times 100\%$$
 (3)

Where  $A_c$  is the area under the CAMES RSD curve and  $A_r$  is the area under the dye RTD curve.

Percent breakup generated from screw configuration 1 (i.e. wide kneading blocks) is presented in Figure 4.



Figure 4. Percent breakup using wide kneading blocks

Figure 4 presents the percent breakup for the wide kneading block configuration and indicates that there are trends within the data as a function of screw speed and specific throughput. As screw speed increases for a given Q/N, the percentage of broken beads also increases. This is expected as shear rate increases as screw speed also increases as given by equation (2).

Another noticeable trend within the data is percent breakup of beads increases as specific throughput increases. As the specific volume of material in the channel increases, the pressure on the melt while inside the kneading blocks also increases, inducing higher levels of stress on the beads.

Average percent breakup was calculated using the narrow kneading blocks screw design (screw configuration 2) and inserted in the CCD grid as shown in Figure 5.



Figure 5. Percent breakup using narrow kneading blocks

Figure 5 above shows similar trends within this data to that of Figure 4. Again, it can be seen that increasing N and Q/N yields a higher percent breakup.

Comparing the two average percent breakup grids (Figure 4 and Figure 5), it is apparent that screw configuration 1 (wide kneading blocks) broke up a larger percentage of CAMES beads than in the narrow kneading blocks configuration. This result is consistent with the common understanding that the wide kneading blocks are better for dispersive mixing due to the wider paddles and the higher extensional flows. The narrow kneading blocks are commonly described as better for distributive mixing, as confirmed by the lower stress levels.

To confirm that the range of obtained breakup was outside of experimental error, an error analysis was performed on both sets of data. It was determined that there is approximately a 2% error for both wide and narrow kneading blocks. This provides validation that the range of percent breakup is independent of experimental error and that there is in fact a significant increase in percent breakup as N and Q/N are increased.

#### **Residence Stress Distributions**

Residence time and stress distribution curves were generated from the data acquisition program and plotted together for each condition. Figure 6 displays the RTD and RSD curves for a given operating condition using the wide kneading blocks configuration.



Figure 6. Normalized wide kneading blocks RTD and RSD curves for .236/110 condition

Figure 6 shows the normalized RTD and RSD curves generated by the dye and CAMES shots, respectively. This figure shows the quality of the data collected by each individual run, which validates the claim that the feed method did not affect the output curves. The two RSD curves are essentially identical to each other demonstrating the experimental repeatability. The curves also show that the CAMES beads are passive tracers, following identical paths in the extruder as described by the RTD. Upon inspection, it is clear that the RTD curve has higher amplitude than the RSD curves, indicating that 100% breakup of the CAMES beads was not achieved.

Since the residence distribution is the time history through the screw's geometry, it can be viewed as the history of the flow path through the mixing zone. The dye that comes through first transverses the least tortuous path, staying in the center of the channels; whereas the tail of the distribution passes back through the paddles. The difference in the RTD and RSD curves confirms that description. Initially the percentage of the flow that experiences the critical shear stress is a small percentage of the total flow; whereas, the figure does indicate that at the tail end of the curves, the majority of CAMES beads did break during this segment as the RTD and RSD curves overlap.

The RTD and RSD curves generated using the narrow kneading blocks are plotted in Figure 7.



Figure 7. Normalized narrow kneading blocks RTD and RSD curves for .236/110 condition

The comparison of the RTD and RSD curves for the narrow kneading blocks configuration follows the same pattern as the wide kneading blocks where the initial part of the flow experiences little of the critical stress whereas all of the flow in the tail of the RSD experiences the critical stress. The one difference is there is a lag time between the start of the RTD and the RSD. This can be interpreted as the initial part of the RSD experienced little to no shear stress until about 20% of the RTD is complete.

#### Conclusions

A novel method for directly generating a Residence Stress Distribution using a stress sensitive bead (CAMES, MACH I, Inc.) has been demonstrated to be an effective tool to measure the stress history in a complex geometry as might be experienced in a twin screw extruder. A comparison of wide and narrow kneading block screw configurations was performed over a range of specific throughputs (Q/N) and screw speeds (N). The wide kneading block configuration showed a higher percentage breakup of the stress beads when compared with the narrow kneading blocks of equal length. The interpretation of that result was that a higher percentage of the CAMES beads experienced the critical stress at the same operating conditions, in all cases.

For a given screw configuration, there was an increase in percentage bead breakup as a function of screw speed and as a function of specific throughput. The higher values of each resulted in higher bead breakup.

Finally, plotting the Residence Time Distribution and the Residence Stress Distribution simultaneously reveals more in depth insights. A residence distribution is generally understood to be the time history through the extruder, but it can also be understood to represent the actual path through the machine. The average percentage bead breakup is not evenly experienced over the time distribution. The initial percentage is low, whereas, the percentage breakup near the tail of the distribution is nearly 100%. That can be interpreted as the initial flow through the mixing section experiences little breakup, perhaps it mostly remains in the middle of the channels; and the tail of the flow has followed a path that repeatedly goes through the paddles resulting in a 100% breakup of the beads.

These insights can be used to help screw configurations for breakup and distribution of many important additives like carbon nanotubes or pharmaceuticals, one needing a high degree of stress and the other needing a low degree of stress but good distribution.

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