MEASURING RESIN TEMPERATURE DURING EXTRUSION USING A FLUORESCENCE TECHNIQUE

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Abstract

We have used a temperature sensitive fluorescent dye, doped into polycarbonate, to monitor the true resin temperature during extrusion processing. For this measurement, a fluorescent dye, perylene, was doped into the polycarbonate at very low concentration. We apply this measurement concept to extrusion processing by using an optical sensor that accesses the machine at standard instrumentation ports. The sensor has a confocal optics design that permits the measurement of temperature profiles. With the sensor looking over the screw of a single extruder, temperature profiles from the barrel wall to the core of the screw were obtained as a function of screw speed, screw design and melt flow index.

Introduction

Temperature measurements during processing by single or twin screw extrusion are usually made using thermocouples that are inserted in instrumentation ports along the extruder barrel. Sensors that reach through the barrel wall and into the interior of the extruder have erroneously been given the name "melt thermocouples" implying that they are measuring the resin melt in the extruder. But, it has been established by many workers that the thermocouple measurement is dominated by heat conduction from the barrel wall to the thermocouple junction so that the thermocouple reading is essentially that of the temperature controlled extruder wall.(1,2) Effects due to shear heating and temperature gradients in the resin flow stream are not recorded by the thermocouple.

The inadequacies of temperature measurements have major implications regarding rheological understanding of the process and the onset of resin degradation temperatures. To address these issues, we have engaged in a program to use fluorescence spectroscopy as a tool for monitoring resin temperature during processing. The hardware of the measurement apparatus involves an optical fiber sensor that is inserted into existing instrumentation ports and is used to transmit excitation light to the resin and to collect the resultant fluorescence and transmit it to the detector.(3-5) Fluorescence is produced from a fluorescent dye, perylene, that is mixed with the resin at dopant concentrations, less than 10^{-5} mass fraction of dye in the resin. The concept regarding fluorescent dyes is that they are molecular probes, i.e. they respond to the molecular environment in which they exist and report the conditions of that environment via their observed spectra. Thus, a temperature deduced from fluorescence spectra yields a true resin temperature.

The fluorescence temperature method has been described previously.(1,2) Briefly, the technique involves measuring fluorescence intensity at two wavelengths and calculating temperature from a calibration function involving the ratio of the two intensities and the applied pressure. For perylene doped into polycarbonate, the two wavelengths of interest are 464 nm and 475 nm and the calibration function for these measurements was

$$T = 807.3\frac{I_{464}}{I_{475}} - 373 + 0.57P \qquad (1)$$

where the T is temperature in ^{o}C, I is intensity and P is pressure in MPa. The last term is a pressure compensation term that adds 0.57 ^{o}C to the calculated temperature per MPa of applied pressure. Equation (1) was obtained using a temperature/pressure calibration cell that was constructed with a standard half-inch threaded port to accommodate the sensor.

Experimental Procedure[a]

Perylene dye was obtained from Aldrich and used as received. Two DOW Chemical polycarbonates (PC), melt flow rate (MFR) 23 g/10 m and melt flow rate 6 g/10 m respectively (ASTM D-1238), were prepared by doping with perylene. Powdered perylene was added to a starting core batch of PC pellets, extruded and repelletized. This batch was diluted by combining it with undoped PC pellets,

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[a] Identification of a commercial product is made only to facilitate experimental reproducibility and to describe adequately the experimental procedure. In no case does it imply that it is necessarily the best product for the experiment.

extruding and repelletizing again. These steps were repeated until the concentration of dye in the resin was 1.6 x 10^{-5} mass fraction of perylene in the PC resin.

The confocal design of the sensor has been described in reference 3 and is depicted in Figure 1. A sleeved half-inch sensor bolt was machined to receive an optical fiber that transmits light from the light source. The lens, which is an essential feature of the design, focuses the excitation light to a point in the resin. The resulting fluorescence transmits back through the lens to collection fibers that conduct the light to the detector. The collection fibers act as a pinhole and the point-to-pinhole confocal design is thus achieved.

The experimental setup is shown in Figure 2. Experiments were performed on a 6.35 cm (2.5 inch) diameter, 21:1 length-to-diameter single-screw extruder. This extruder has been modified with 12 instrumentation ports equally spaced along the barrel starting at a position 3.6 diameters from the feed hopper with a port every 1.5 diameters thereafter. The optical sensor was placed in position 11 (18.6 diameters from the feed hopper) and the remaining ports were equipped with pressure transducers. In this position, the optical sensor looked directly over the screw and was approximately 15.2 cm from the extruder exit. Pressure transducers in positions 10 and 12 were used to deduce the pressure at position 11 by a linear interpolation. Extrusion was carried out with the set point temperature at 275 ^{o}C for all positions along the barrel.

Three different screws were used in this investigation. Two of the screws were single-flighted, square-pitched screws and the other was a barrier-flighted, energy transfer screw. In this paper, results obtained from one of the single-flighted, square-pitch screws will be presented. This screw is characterized by six flights of 8.89 mm (0.350 inch) feed, eight flights of taper, and seven flights of 3.18 mm (0.125 inch) meter. Data obtained with the other two screws is presented in a companion paper to be presented at this meeting.(6)

The light source was a 30 mW, 407 nm diode laser from Power Technologies. Laser light was filtered with a 10 nm bandpass filter centered at 405 nm and focused onto a single 200 μm diameter optical fiber that transmitted the excitation light to the resin. A reinforced industrial grade optical fiber cable protected the optical fiber for its connection to the sensor head in the extruder and its connection to the detector. At the sensing head, the optical fiber is assembled in a sheath with a collar that holds a focusing lens. The sheath is inserted into the standard bolt sleeve and at the same time it is connected to a micrometer that can move the sheath/lens/fiber assembly in the axial direction of the sensor bolt. In this way the position of focus is moved in the radial direction of the barrel and screw while carrying out the profile temperature measurement. The length of travel for the micrometer is 3.175 mm and the spatial resolution of the profiles reported here is 0.5 mm.

Fluorescence light is collected by six 200 μm diameter fibers that transmit the light to a beamsplitter that separates the light into two channels. The two channels are filtered one at 464 nm and the other at 475 nm. The ratio of these intensities along with pressure is used in equation (1) to calculate the temperature. The standard uncertainty in the temperature measurements presented here is 1.5 ^{o}C, the relative standard uncertainty in the pressure measurements is 1 %, and the standard uncertainty of the depth profile dimension is 0.025 mm.

In order to maintain the uncertainty in the temperature measurements at 1.5 ^{o}C, total photon counts for each measurement needed to be greater than 10^5. We have reason to be concerned about the photon count because, in carrying out temperature profile measurements, we focus the excitation light to different depths within the resin. Fluorescence from the surface yields high count rate, but for the excitation beam focused at 3 mm below the surface the fluorescence production falls off because of light absorption, i.e. as excitation light traverses through the dye doped resin, it is absorbed during transit to the point of focus (Beers law). The phenomenon is demonstrated in Figure 3 where we have plotted fluorescence intensity at 476 nm versus distance from the window of the sensor and fitted the data with a Beers law exponential function. In order to counteract this effect, we increased the count integration time whenever necessary so that total photon counts remained above 10^5.

Results and Discussion

Figure 4 depicts the sensor in its instrumentation port facing a screw that is turning at 20 rpm (revolutions per minute). If the data acquisition time is set for 0.5 s, then there will be six data points per cycle of the screw. The six dashed lines in the axial direction of the screw depict the spatial distribution for a 0.5 s integration time for each data point. If the excitation light is focused at a position that is cut off by the rotating screw flight, then these interruptions will be reflected in a decrease in intensity counts once per cycle. As the point of focus is moved very close to the window, a small region between window and screw flight is not sliced rotating screw and, when accessed by the focused beam, is unaffected by screw rotation. This is the benefit of confocal optics whereby spatial effects can be separated.

Figure 5 shows data for the MFR 6 PC resin as a function of time for excitation light focused at 0.95 mm from the wall for processing at 20 rpm. The periodic spikes in both the count data and the temperature data coincide with the periodic translation of a screw flight in front of the sensor window. The decrease in counts and increase in temperature was seen at every sixth data point. (It is apparent that the data acquisition rate and the screw rotation were not exactly synchronized, but the discrepancy is small.) Each time a screw flight passed the window, the counts decreased as the flight cut off a part of the focused beam, but at the same time, the flight pushed a film of highly stressed resin between it and the wall. Spikes in the temperature data of approximately 15 °C above the ambient are associated with shear heating of the thin film of molten resin between the flight and barrel wall.

As noted above, when the position of the focus is moved very close to the window, interference from the screw flight becomes less apparent. With focus at d = 0.64 mm, steady state temperature of MFR 6 PC resin is plotted in Figure 6 for 20 rpm. The standard deviation for these data is 1.0 °C and there is no evidence of a periodic pattern in the data as was observed for d = 0.95 mm (Figure 5). The steady state conditions of resin flow near the wall are very stable for 20 rpm. Similar results were observed for 60 rpm.

To measure temperature profiles the micrometer was changed in steps of 0.32 mm or 0.64 mm, holding constant at each position while the fluorescence temperature was measured for approximately 10 min. A moderately long measuring time was used in order to investigate the stability of the steady state and to collect enough data to determine the uncertainty in the measurement. Figure 7 is a plot of temperature versus distance from the barrel wall for MFR 6 PC processed at 20 rpm. The highest temperatures were located at the wall and at the core of the screw, i.e., at the interfaces with the confining surfaces where the shear stress is highest. Generally, the profile temperatures were observed to be higher than the set point temperature of the barrel, 275 °C, due to viscous heating.

The dynamics of change from one process condition to another can be seen in Figures 8 and 9 where the temperature dynamics associated with change from 20 rpm to 60 rpm and from 100 rpm to 20 rpm are plotted for the MFR 6 PC resin. At 100 rpm and d = 1.27 mm, we observed the temperature varying over a wide range, from 250 °C to 295 °C. The very low temperatures observed here are evidence for solids conveying, i.e. pellets that have not reached the processing temperature at this location in the extruder. A screw turning at high rpm translates resin pellets through the extruder at such a rapid rate that some of the pellets do not fully blend with the melt stream at position 11 on this extruder. After the change to 20 rpm, a much smaller temperature spread was observed and the average temperature of the resin decayed slowly over time. (No data were collected as sspeed was decreased to 20 rpm resulting in the 30 s gap in the data.)

In summary, we have described the operation of a fluorescence temperature sensor and demonstrated its use for real-time monitoring of single screw extrusion. The measurement yields new information about the distribution of temperature between screw flights, about transient temperature spikes associated with shear heating of a thin film of resin melt between screw flight and barrel wall, about the stability of the steady state, and about the dynamics of temperature change associated with a change in processing conditions.

References

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Keywords: extrusion, resin temperature, processing, polycarbonate, fluorescence spectroscopy, temperature profiles

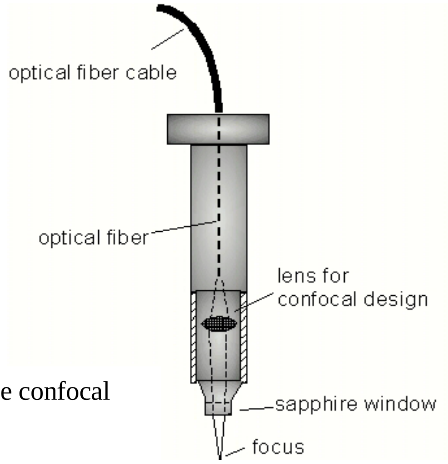

Figure 1(a) The confocal sensor design

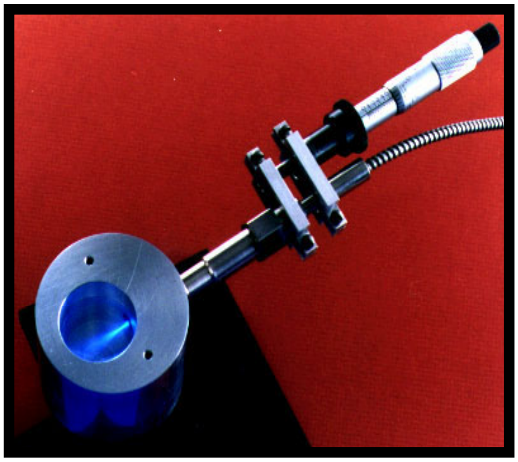

Figure 1(b). The confocal sensor bolt with micrometer

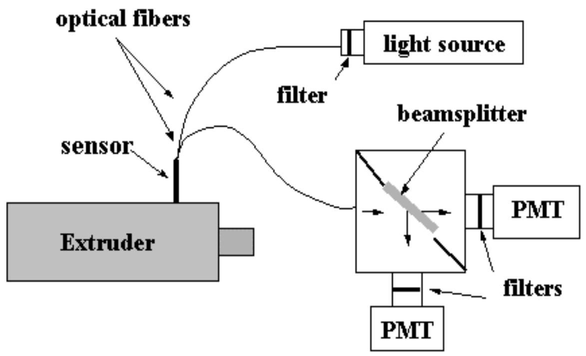

Figure 2. The experimental setup.

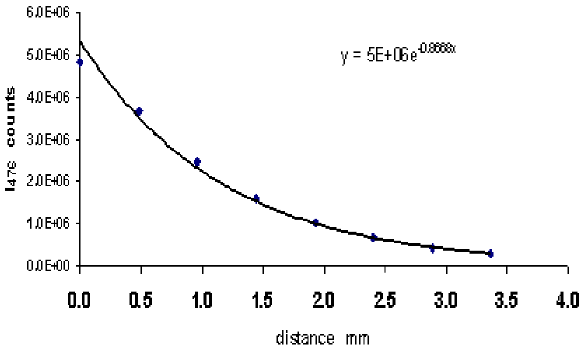

Figure 3. Fluorescence intensity at 476 nm is plotted versus depth of focus into resin where zero is the window of the sensor. Line is exponential fit.

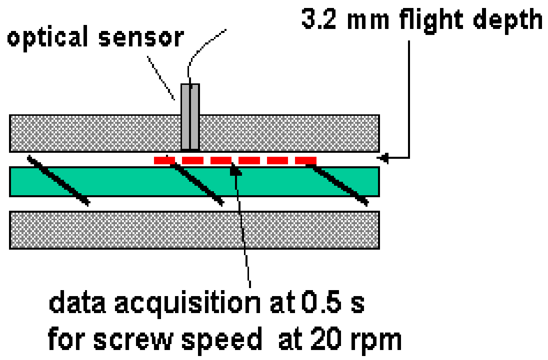

Figure 4. The sensor is shown in position overlooking the screw. The six dashes represent the spatial integration equivalent to 0.5 s optical integration time when the screw is turning at 20 rpm.

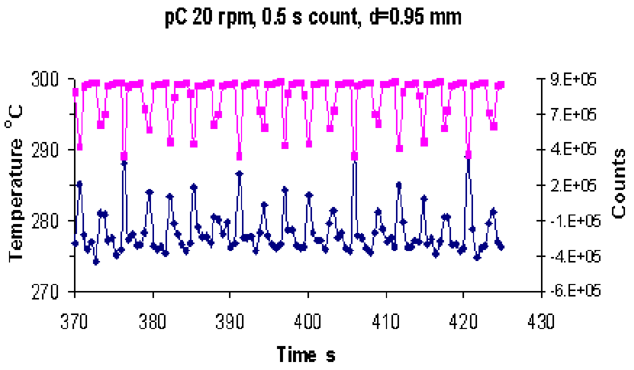

Figure 5. Temperature (lower trace) and counts versus time showing periodicity in the data corresponding to the screw rotation rate, 20 rpm. Data acquisition rate was 0.5 s or six data points per screw rotation.

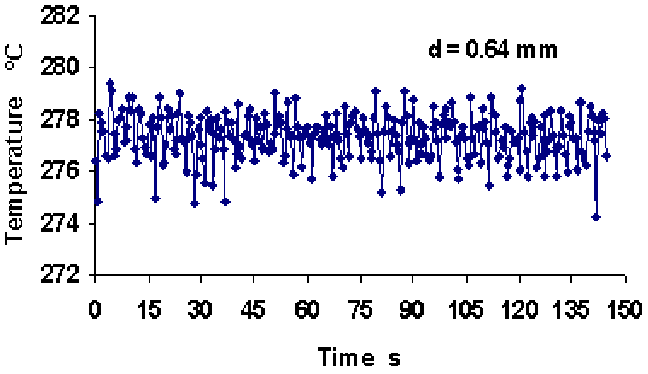

Figure 6. Steady state temperature for depth of focus near the sensor window, d = 0.64 mm.

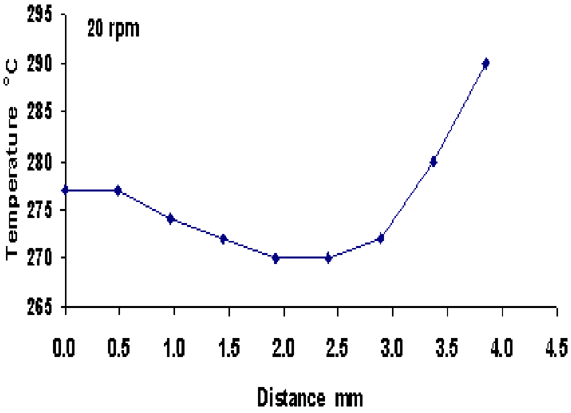

Figure 7. Temperature versus depth of sensor focus shows profile from sensor window to the core of the screw. Line is drawn to aid reader's eyes.

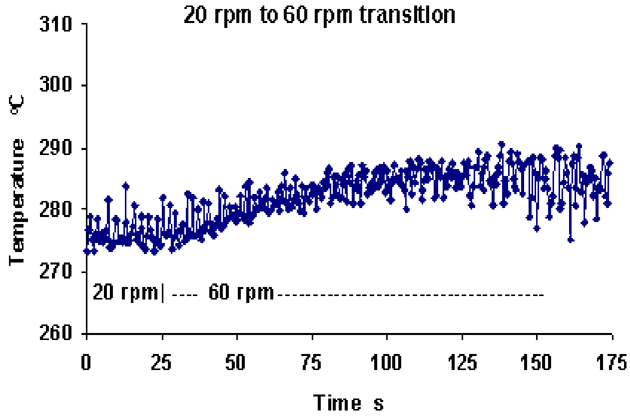

Figure 8. The transition from 20 rpm to 60 rpm screw speed is reflected in the temperature increase. Depth of focus was 0.96 mm.

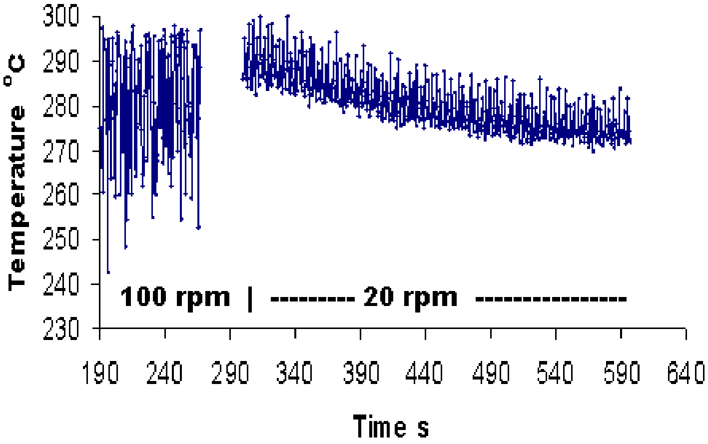

Figure 9. The transition from 100 rpm to 20 rpm screw speed is reflected in the change in temperature. Depth of focus was 0.96 mm.