Development of an In-Line Viscometer in an Extrusion Molding Process

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ABSTRACT: In this work, an in-line viscometer to measure the viscosity of polymer melts under extrusion molding processes was developed. The in-line viscometer contains a stress sensor and a shear rate sensor which were installed between the screw and the die of an extruder. In this way, the flow line after the screw cannot be changed, unlike the present in-line capillary rheometer which can change the diameter of the pipe of the flow line and hence influence the throughput. All data acquisition is done by a computer such that the melt viscosity can be calculated automatically. The shear-thinning behavior of a low-density polyethylene (LDPE) under three different temperatures is presented in all experiments. It is concluded that the melt viscosity can be effectively monitored. © 1997 John Wiley & Sons, Inc. J Appl Polym Sci **63:** 919–924, 1997

Key words: in-line viscometer; shear stress sensor; extrusion; viscosity; in-line

INTRODUCTION

In the polymer processing industry recently, more and more stringent controls on properties relevant to the precision and geometric stability of products are required. Much research has emphasized quality control. The molecular orientation of a polymeric extrudate is dependent on the viscosity of the melt under processing, and the mechanical properties of an extrudate are influenced by the molecular orientation. Melt rheology plays a very important role in quality-control engineering.[1–5]

Currently there are many commercial rheological instruments used to measure the melt viscosity for quality identification.[6–8] Because the ranges of the shear rate are relevant to each processing need, the rheometers are mostly developed based on a capillary or a slit in an extrusion molding process, both on line and off line. The most accurate and widely accepted test procedure among these is the Laboratory Capillary Rheometer (LCR).[6] It is a better off line method for melt viscosity measurement, but it takes more time and the results being tested deviate from the conditions of the molding shop.

The rheological behavior of most polymeric materials is quite complex.[9] The melt viscosity is both shear and thermal history dependent. The flow characterization model obtained using an on line rheometer is more relevant to the extrusion molding process than the LCR data.[10] The viscosity of a polymeric melt can be measured on line using a modified molding machine and has been proven to be an important way to quality improvement.[5,11] Göttfert[12] offers two continuous capillary rheometers, the Kontirheograph and the Side Stream Capillary Rheometer. Melt under pressure is taken as a side stream from the flow line. In both instruments, a metering pump forces the melt through a die, and the pressure at the inlet is monitored. After leaving the rheometer, the sample stream may simply flow out as a waste stream. However, there is still a significant signal delay resulting from the time required for the

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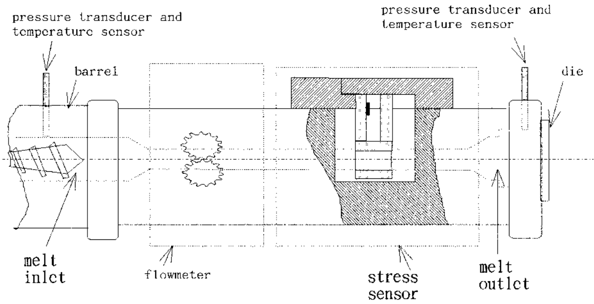


Figure 1 Schematic diagram of stress and flow rate sensors of the in-line monitoring system.

melt to flow through the transit lines and the gear pump.[13] In addition, the viscosity in the process line cannot be monitored in real time. Because of this, an in-line process instrument is needed.

The in-line viscometers most used commercially or academically are those which are made of a slit or a capillary.[14–17] The flow rate is measured by an extra flowmeter, and the stress on the wall is measured by the pressure drop of the two sides of the slit or capillary. Since the dimension of the die is small, there is a limitation to use in mass production.

This paper proposes a new in-line viscometer that can more directly test melt viscosity as a function of shear rate in an extrusion molding process. This mechanism is designed to include a stress sensor and a shear rate sensor which are mounted between the screw and the die of an extruder and do not change the dimension of the pipe of the main flow line. Moreover, with rapidly developing computer science, computer technology is applied in this study. All data (such as temperature, pressure, and flow rate, etc.) acquisition is done by a computer, and then the melt viscosity is measured automatically using the C programming language.

DESIGN OF THE IN-LINE VISCOMETER

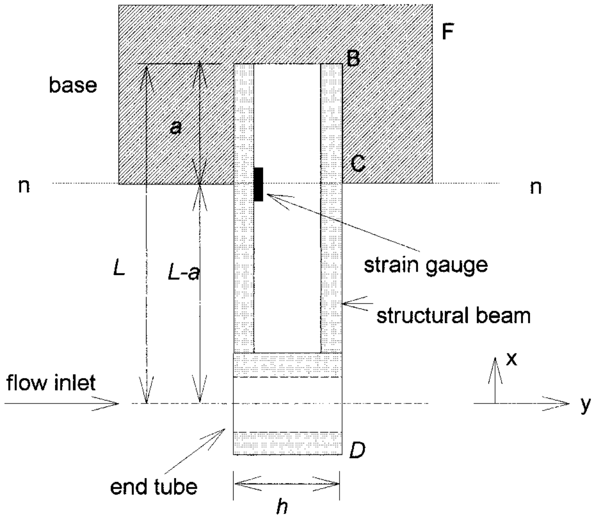
The definition of the viscosity is given by eq. (1). Measurement of the melt viscosity requires the use of a controllable flow in which both the stress and strain are known. The in-line viscometer in this study is then designed including a stress sensor and a shear rate sensor which are sequentially mounted between the screw and the die in the process line as shown in Figure 1.

$$\eta = \frac{\tau_w}{\dot{\gamma}_w} \tag{1}$$

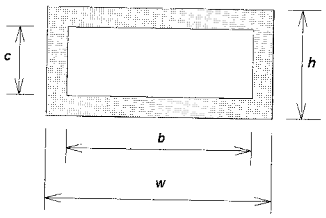
where $\dot{\gamma}_w$ means the shear rate and τ_w represents the shear stress.

DESIGN OF THE STRESS SENSOR

The stress sensor as shown in Figures 2(a) and 2(b) is based on the notion that the shear stress in the process line can generate a bending moment, and the bending moment will cause a strain. A strain gauge is pasted inside a hollow transverse structural beam to measure the strain for shear stress calculation. Once the melt is extruded out and passes the end tube of the stress sensor, the viscous flow will generate a shear force F_w, simultaneously generating a moment M where the strain gauge is pasted. The normal stress, considering section $n - n$, is as eq. (2).



(a)



(b)

Figure 2 (a) Schematic diagram of the stress sensor and (b) cross-section view of the stress sensor.

$$\sigma_x = \frac{My}{I} \tag{2}$$

where M is the bending moment in given cross section $n - n$, x is the longitudinal direction, and y represents the horizontal direction distance from the neutral axis. We simplify the moment of inertia of the rigid body in this case as the one of the structural tube beam, so that the moment of inertia I can be represented as eq. (3):

$$I = \frac{wh^3 - bc^3}{12} \tag{3}$$

where w, h, b, c are dimensions of the stress sensor shown in Figures 2(a) and 2(b).

There will be no permanent deformation, and Hooke's law for uniaxial stress applies. Assuming the material to be homogeneous, and denoting its modulus of elasticity by E, then

$$\sigma_x = E\varepsilon_x \tag{4}$$

where ε_x means longitudinal strain. Substituting eq. (3) and (4) into eq. (2), therefore

$$\frac{12F_w(L - a)y}{wh^3 - bc^3} = E\varepsilon_x \tag{5}$$

Finally, the equation can be written in the alternate form by substituting eq. (5) into $\tau_w = F_w/A$. If A represents the surface area of the pipe flow in the stress sensor, then

$$\tau_w = \frac{E\varepsilon_x(wh^3 - bc^3)}{12(L - a)A} \tag{6}$$

In this case, $A = 2\pi rh$, and r is the radius of the mainline of a pipe flow. The shear stress on the wall is consequently measured.

The stress sensor is connected to the data acquisition board in a personal computer. We use C programming language for automatic data gathering and processing.

INSTRUMENT FOR SHEAR RATE MEASUREMENT

The instrument developed uses a flowmeter installed right after the screw as shown in Figure 1. The melt from the screw in the flow line causes the gears to rotate. The volume generated by every revolution is designed to be 40 cc/rev. The flow rate of the melt is then calculated, applying the lubrication approximation to the measurement of shear rate on the wall and according to Rabinowitsch's correction for a non-Newtonian melt flow. The shear rate can then be written as

$$\dot{\gamma}_w = \frac{4Q}{\pi R^3}\left(\frac{3n + 1}{4n}\right) \tag{7}$$

where n is the power in the relationship of shear stress proportional to (shear rate)^n at a given shear rate, Q is the volume flow rate, and R is the radius of tube of flow.

Then, the melt viscosity can be represented as $\eta = \tau_w/\dot{\gamma}_w$. The melt viscosity is then measured.

EXPERIMENT

All of the experimental data presented in this paper are obtained from runs on a 25 : 1 L/D ratio single-screw extruder with full-way windows. The extruder is equipped with four temperature sensors and pressure transducers, with three located at the solid polymer transition section, the molten section and the melt transition section, respectively, and the rest located at the die. The in-line viscometer is installed between the screw and the die. A personal computer is used with A/D and D/A converters and an RS232 interface for monitoring all of the process information from the extruder, including temperature, pressure, screw speed, flow rate, and strain. Then C programming language is used for the automatic gathering and manipulation of data.

The material used in the experiment is low-density polyethylene (LDPE). The operating temperature is set at 150°C the first time, and the screw speed is set at 100 rpm. Data is recorded from sensors after a sampling time of 20 seconds, then the screw speed is changed to 200 rpm. The steps of recording and changing the screw speed are repeated until the screw speed is up to 700 rpm. The data on melt under processing at a certain temperature is then measured. The system flowchart is shown as Figure 3. All data acquisition is done under three temperature conditions: first at 150°C, next at 160°C, and finally at 170°C.

Tables I, II, and III show the results of the measurement of LDPE at 150°C, 160°C, and 170°C. The feature of decreasing viscosity with increasing temperature is illustrated in Figure 4.

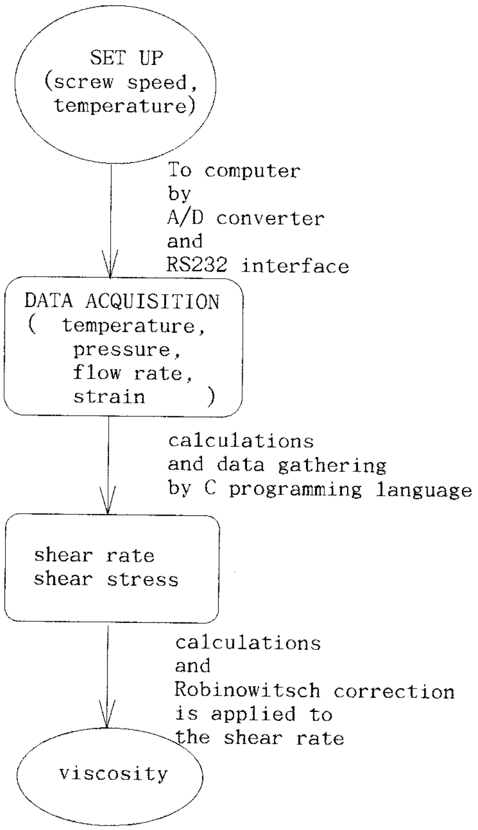


Figure 3 The flowchart of the in-line monitoring system.

All curves strictly follow the shear thinning behavior.

In order to verify the results of the in-line monitoring system, we had the sample tested in the ROSAND precision capillary rheometer, Model RH 7-2, developed by Rosand Precision Ltd., at 160°C for comparison with the one used in the in-line viscometer. The result is shown in Table IV. It is shown in Figure 5 that there are differences between the results of the in-line viscometer and those of the ROSAND capillary rheometer. The maximum difference between these two curves is 14.7% for the shear rate at the point of 6089 (1/s).

Table II **Experimental Data and Results of LDPE Processed at 160°C**

rpm (r/min)	Q (g/min)	τ_w (kPa)	n	$\dot{\gamma}_w$ (l/s)	η (Pas)
100	116	32	0.49	186.22	171.839
200	359	70	0.47	586.25	119.403
300	696	106	0.45	1157.54	91.574
400	1201	140	0.44	2016.73	69.419
500	1849	173	0.43	3115.99	55.166
620	2593	203	0.41	4491.53	45.196
700	3506	227	0.40	6141.08	36.964

CONCLUSION

An in-line viscometer in an extruder is demonstrated that can examine the melt viscosity as a function of shear rate under extrusion molding processing conditions. This study also simplifies the process of in-line monitoring in an extruder by computerizing the extrusion molding process, making the operation of the polymer processing easier.

The shear thinning behavior of LDPE which features decreasing viscosity with increasing temperature is detected. However, the viscosity being tested in the in-line monitoring system is higher than the result for the ROSAND precision capillary rheometer when the shear rate is below about 600 1/s and is lower when the shear rate is over 600 1/s, as shown in Figure 5. This is possibly due to instability of both the flowmeter and the stress sensor. Because some discrepancies still ex-

Table I **Experimental Data and Results of LDPE Processed at 150°C**

rpm (r/min)	Q (g/min)	τ_w (kPa)	n	$\dot{\gamma}_w$ (l/s)	η (Pas)
100	97	32	0.50	154.46	207.175
200	300	71	0.49	481.64	147.423
300	560	105	0.46	922.74	113.792
400	936	143	0.45	1556.69	91.862
500	1416	175	0.43	2401.60	72.868
600	1979	208	0.41	3427.97	60.677
700	2707	238	0.40	4741.56	50.194

Table III **Experimental Data and Results of LDPE Processed at 170°C**

rpm (r/min)	Q (g/min)	τ_w (kPa)	n	$\dot{\gamma}_w$ (l/s)	η (Pas)
100	168	33	0.48	271.97	121.335
200	486	69	0.47	793.64	86.941
300	952	103	0.44	1598.61	64.431
400	1602	135	0.42	2745.31	49.175
500	2505	160	0.41	4339.09	36.874
600	3662	164	0.40	6414.33	25.568
700	5058	168	0.39	8962.81	18.744

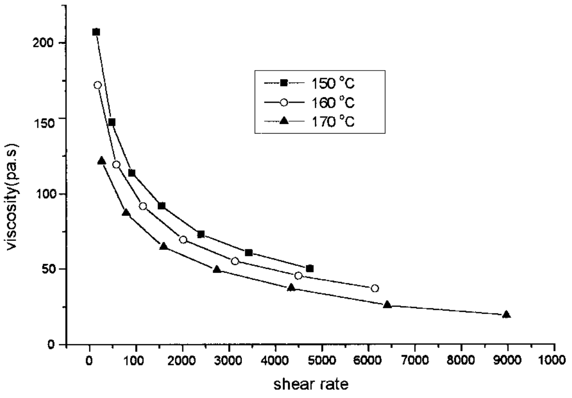


Figure 4 The shear thinning behavior of LDPE tested at three different temperatures.

ist in the results, more accuracy is needed for this system, the subject for further research.

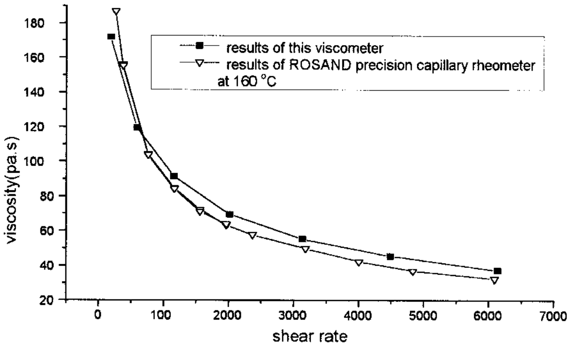


Figure 5 The results of the in-line monitoring system and the ROSAND precision capillary rheometer under 160°C.

NOMENCLATURES

A = the surface area of the flow tube in the stress sensor.
a, b, c, w, h = dimensions of the stress sensor.
B, C, D = points on the stress sensor.
E = modulus of elasticity of the stress sensor.
F = force apply to the suspended beam by the melt.
I = moment of inertia of the stress sensor.
M = bending moment in an given cross section $n - n$.
n = the power law model index.
Q = the volume flow rate.
R = radius of tube of the flow line.
x = longitudinal direction.
y = horizontal direction distance from the neutral axis.

GREEK SYMBOLS

ε_x = longitudinal strain.

Table IV Rosand Precision Capillary Rheometer Test Results of Polyethylene (Rabinowitsch-Corrected) Under 160°C

Shear Rate (l/s)	Time (s)	P1 (Mpa)	Ps (Mpa)	P0 (Mpa)	Shear Stress (kPa)	Shear Visc. (Pas)	Extern. Stress (kPa)	Elong. Visc. (Pas)	n
6089.4	8.5	15.88	3.520	3.324	196.14	32.21	1746.6	1.6379	0.40
4837.0	11.5	14.40	3.186	3.008	177.96	36.79	1588.6	1.8664	0.41
4007.9	14.5	13.61	2.972	2.803	168.85	42.13	1486.0	2.0987	0.41
3184.6	20.5	12.56	2.607	2.449	158.02	49.62	1304.7	2.3078	0.42
2368.2	23.6	10.78	2.178	2.042	136.51	57.64	1094.5	2.5874	0.43
1963.1	28.1	9.70	1.883	1.759	124.07	63.20	946.6	2.6892	0.43
1560.6	33.6	8.73	1.636	1.524	112.66	72.19	823.9	2.9302	0.44
1161.2	38.1	7.53	1.339	1.241	98.20	84.57	674.9	3.2066	0.45
765.8	42.1	6.07	1.051	0.971	79.69	104.06	532.9	3.8065	0.46
376.3	58.1	4.38	0.681	0.623	58.66	155.89	346.6	4.9671	0.48
261.2	75.2	3.57	0.501	0.453	48.74	186.64	253.7	5.2011	0.49
376.3	125.2	4.30	0.621	0.563	58.39	155.18	313.2	4.4878	0.48
765.8	137.3	6.00	1.001	0.922	79.34	103.61	505.8	3.6131	0.46
1161.2	143.7	7.46	1.310	1.212	97.66	84.11	659.4	3.1326	0.45
1560.6	149.3	8.57	1.584	1.473	110.95	71.09	796.3	2.8321	0.44
1963.1	153.3	9.69	1.809	1.684	125.17	63.76	906.1	2.5740	0.43

Test geometry: long die length 16 mm, diameter 1 mm; short die length 0.25 mm, diameter 1 mm.

$\dot{\gamma}_w$ = the Rabinowitsch corrected shear rate on the wall.
τ_w = shear stress on the wall of tube.
η = viscosity.
σ_x = longitudinal stress.

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