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Rheological Characterization of Feedstocks for Metal Injection Molding

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Metal injection molding feedstocks are typically highly filled thermoplastic systems. In this study, the effects of feedstock composition on flow properties are examined using the capillary rheometer and the extrusion plastometer. The two techniques are also compared with respect to sensitivity to feedstock composition and their utility in predicting variations in processing performance.

Keywords

metal injection molding, powder injection molding, rheological characterization

1. Introduction

THE quality of metal injection molded (MIM) parts is determined to a great extent by the feedstock, that is, the mixture of metal powder and plastic binder. Rheological characterization of feedstocks provides a means of evaluating homogeneity, lot-to-lot variability, and stability. Capillary rheometry and extrusion plastometry are among the most common industrial techniques for measuring the flow properties of thermoplastic materials (Ref 1-4). Although the two techniques are similar in many respects, there are important differences.

Both methods are routinely used to characterize MIM feedstocks. The melt plastometer, which provides a single datum per test, is usually employed as a quality control tool. The capillary rheometer is capable of characterizing flow behavior over a wide range of shear rates in a single test; thus, it can be used for determining differences between production samples, for evaluating new formulations, and for providing data at processing shear rates. This paper examines the precision of the two techniques and the correlation between the data they provide when applied to developmental and production feedstocks.

2. Rheological Considerations

In capillary rheometry, material is forcibly extruded through a cylindrical orifice of known dimensions. Usually, the rate of extrusion is the independent variable and the pressure drop across the orifice is the dependent variable. The former variable provides the apparent shear rate at the wall, while the latter provides the apparent shear stress at the wall. The ratio of apparent shear stress at the wall to the apparent shear rate at the wall is the apparent viscosity at that shear rate. The apparent viscosity is equal to the true viscosity for Newtonian fluids; for non-Newtonian materials, corrections must be applied to both shear rate and shear stress in order to obtain the true viscosity.

In essence, an extrusion plastometer, widely referred to as a melt indexer, is a capillary rheometer. However, unlike most capillary rheometers, which operate in a fixed shear-rate mode, the extrusion plastometer employs a dead weight to drive the extrusion; thus, it operates in a fixed shear-stress mode. Rather than measure directly the rate of extrusion, the dependent variable in extrusion plastometer testing is usually the mass of material extruded in a given time, a quantity termed the melt index or melt flow rate (MFR). In principle, the apparent shear rate can be calculated from the MFR if the density of the material at the test temperature is known. Knowledge of the apparent shear rate and apparent shear stress (from the dead weight employed and the orifice dimensions), allows calculation of an apparent viscosity. The conversion from MFR to apparent viscosity is not without pitfalls, and by and large the results are reported as MFR rather than as apparent viscosity.

The strength of extrusion plastometer testing lies in the rigid standardization of its use (Ref 5). Additionally, melt plastometers offer simpler operation, and easier data interpretation, and lower cost than capillary rheometers. The primary shortcoming of the instrument is the use of an orifice with a low length to diameter ratio (L/D), which means that entrance effects may be significant. There is also evidence that the extrusion rate may vary during the test (Ref 6). Thus, MFR, while a useful indicator of variations in a single parameter (usually molecular weight) among resins of the same type, is not a material property, as is true viscosity. The ASTM standard method carries a warning to this effect (Ref 5). However, it is valid to ask whether an extrusion plastometer can provide a useful estimate of the viscosity of a material.

There are two approaches one may take in attempting to measure viscosity using an extrusion plastometer: (1) infer the shear rate from the mass of material extruded in a given time, and (2) measure the time required for the plunger to travel a given distance. The former method requires knowledge of the density of the material at the test temperature, and both methods require steady flow throughout the test.

The apparent viscosity (η_{ap}) is the ratio of the apparent shear stress (σ_{ap}) and the apparent shear rate ($\dot{\gamma}_{ap}$), i. e.:

$$\eta_{ap} = \frac{\sigma_{ap}}{\dot{\gamma}_{ap}} \qquad \text{(Eq 1)}$$

For extrusion through a cylindrical orifice:

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$$\dot{\gamma}_{ap} = \frac{4Q}{\pi R^3} \quad \text{(Eq 2)}$$

$$\sigma_{ap} = \frac{RP}{2L} \quad \text{(Eq 3)}$$

where Q is the volumetric flow rate (vol/s), P is the pressure drop (Pa) over the length of the orifice, and R and L are the radius and length of the orifice.

For an extrusion plastometer with an orifice diameter of 2.1 mm (0.0825 in.) diameter, and an L/D of 3.8, the following expressions provide accurate estimates:

$$\dot{\gamma}\,(s^{-1}) = \frac{2000}{t} \quad \text{(Eq 4)}$$

$$\sigma_{ap}\,(Pa) = 9100m \quad \text{(Eq 5)}$$

$$\eta_{ap}\,(Pa \cdot s) = 4.55mt \quad \text{(Eq 6)}$$

where t is the time in seconds required for the plunger to travel 25 mm (1 in.) during steady flow, and m is the total mass (kg) driving the extrusion.

Corrections are applied to apparent viscosity values to obtain true viscosity, which is independent of the measuring device. These include the Rabinowitsch (R) and Bagley corrections (B). The Rabinowitsch correction corrects the apparent shear rate at the wall of the capillary, and the Bagley correction corrects for entrance effects on pressure drop and thus shear stress.

The corrections are applied as follows:

$$\dot{\gamma} = \dot{\gamma}_{ap} R \quad \text{(Eq 7)}$$

where $R = (3n + 1)/4n$, and n is the slope of the line on a log-log plot of shear stress versus shear rate (Ref 7). R can be derived from one graph. To determine B, a series of tests with capillaries of varying length are performed (Ref 7). B takes the form:

$$\sigma = \sigma_{ap} B \quad \text{(Eq 8)}$$

where $B = (L/D)/(L/D + e)$ and e is a correction factor that will vary with shear rate. From the form of B, it is apparent that an entrance pressure effectively increases the L/D of the orifice by an amount equal to e. Since $R \geq 1$, and $B \leq 1$, the apparent viscosity is typically greater than the true viscosity.

In determining viscosity, it is assumed that the material at the orifice wall is stationary. When this assumption is not valid, the measurements will give values that are orifice dependent, unless a slip correction is applied, which is tedious to determine.

3. Experimental Method

The capillary rheometer tests were performed with an Instron (Instron Corp., Canton, MA) 3210 rheometer mounted on a Model 1123 frame. Software from Laboratory Microsystems, Inc., Marina Del Rey, CA, was used for both data acquisition and reduction. An orifice of L/D 16.2 was used throughout the testing.

Approximately 35 g of sample were placed in the rheometer barrel and allowed to equilibrate thermally for 5 min under a modest load before initiating testing. Testing—with both the rheometer and the plastometer—was conducted at temperatures of 175 and 190 °C. Data at shear rates from 10 to 10,000 s^{-1} were obtained.

This rheometer allows control of the extrusion rate (shear rate) and measures the resulting force (shear stress) with a load cell. By using a strip chart recorder to monitor the output of the load cell during testing, the operator can ensure that steady flow has been achieved before taking a reading. Monitoring the load cell output provides other information, as well. High-frequency oscillation of the force is indicative of flow instabilities (e.g., slip-stick); low-frequency or random fluctuations may indicate inhomogeneity; and slow, steady drift may indicate thermal alteration of the material. As a check for alteration over the duration of the test, readings are taken twice at one of the shear rates—once at the beginning and once at the end of the test.

Capillary rheometer data are plotted on log-log plots because of their wide range and because the resulting plot is often nearly linear. Although the data are usually presented as plots of viscosity versus shear rate, plots of viscosity versus shear stress or of shear stress versus shear rate can be useful.

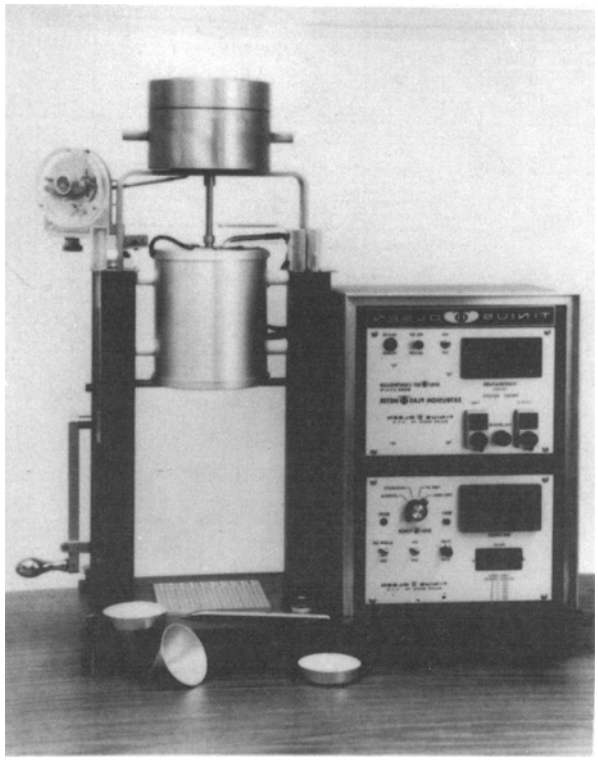

Fig. 1 Extrusion plastometer. Source: Ref 8

Figure 1 illustrates an extrusion plastometer. In this study, a Tinius Olsen Testing Machine Co., Willow Grove, PA, Model U E-4-78 was used. Twenty grams of sample were charged to the heated barrel and allowed to equilibrate for 6 min before extrusion, which was driven by a 6.7 kg load.

After the initiation of extrusion, the time required for the plunger to travel a distance of 25 mm (1 in.) was measured. This time, along with a knowledge of the barrel dimensions, allows calculation of the apparent shear rate (Eq 3). Apparent shear rates from 20 to 50 s^{-1} were observed.

4. Results

4.1 *Capillary Rheometry*

Figures 2 and 3 show the capillary rheometry testing results for seven feedstock batches at 175 and 190 °C, respectively. The differences among the feedstocks were the metal powder content and the date of mixing. Batches 2026 and 2072 illustrate the range of volume percentage of metal, from 58 to 60%.

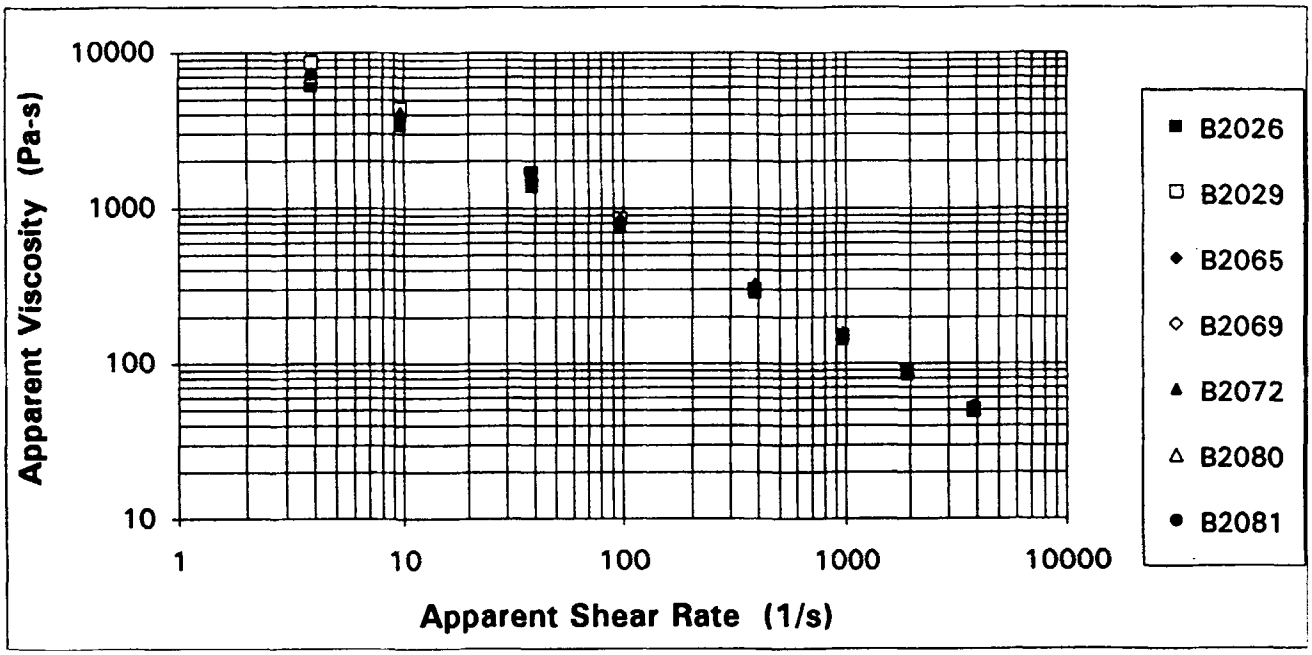

Fig. 2 Capillary rheometry testing results at 175 °C

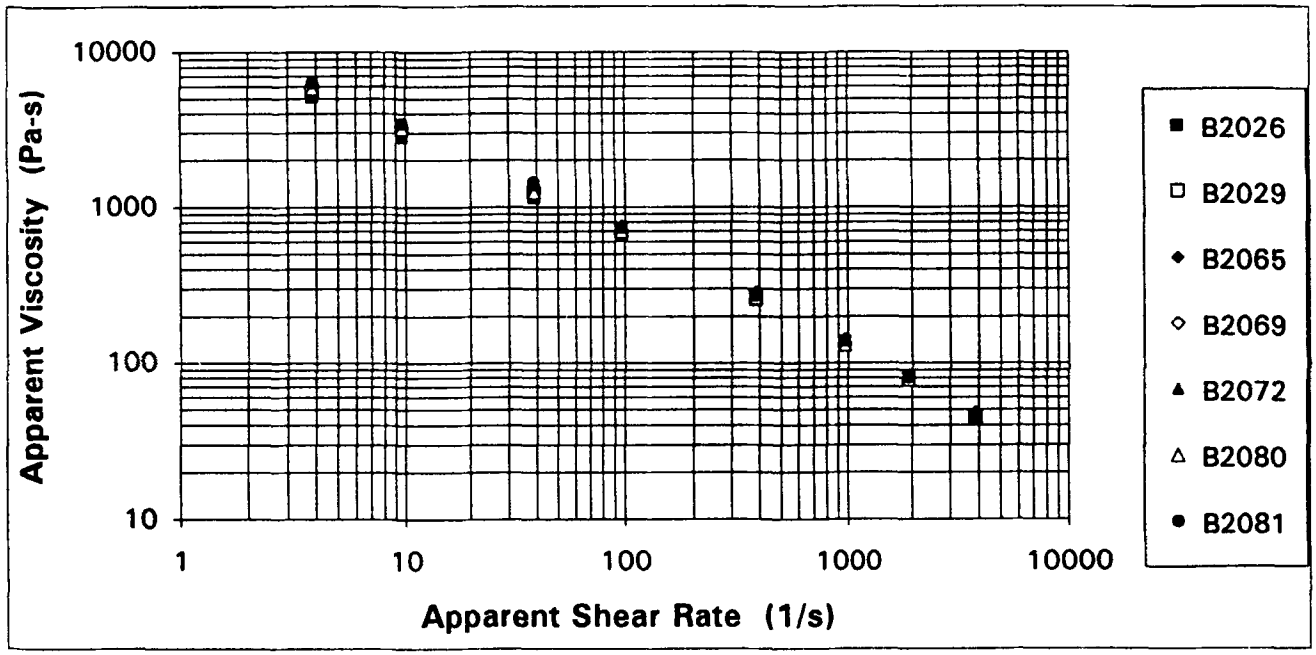

Fig. 3 Capillary rheometry testing results at 190 °C

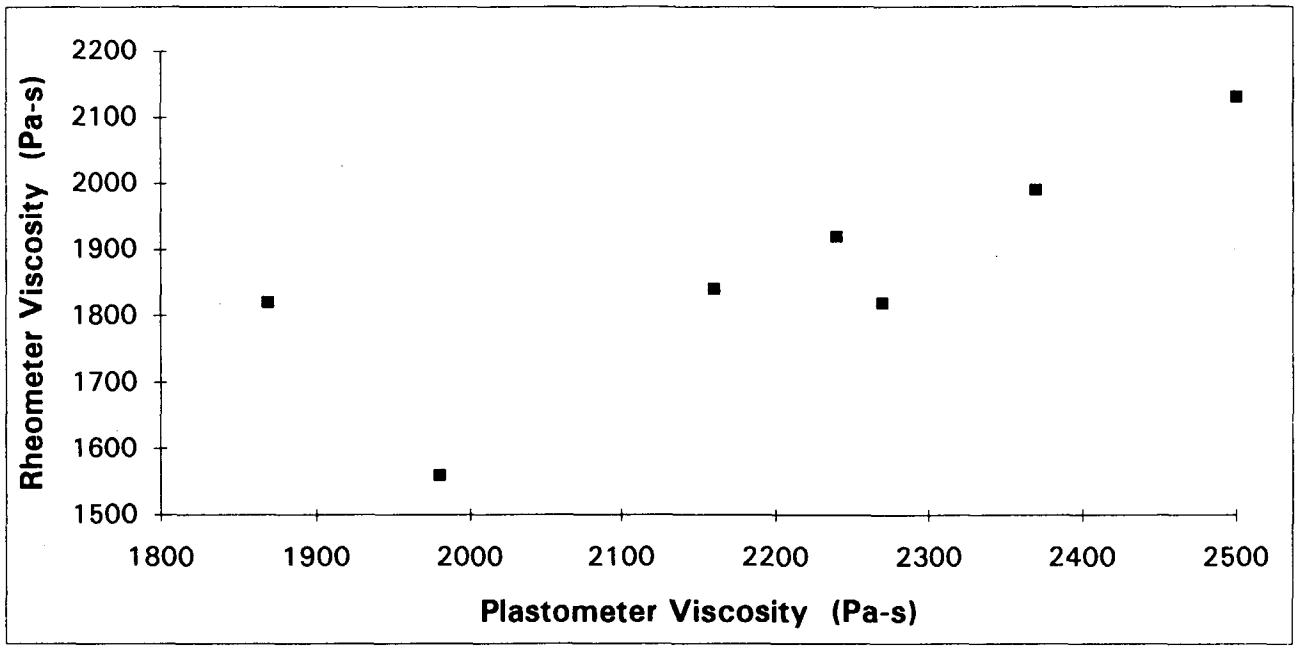

Fig. 4 Rheometer versus plastometer apparent viscosity at 175 °C

Table 1 Comparison of apparent viscosities obtained with a rheometer and a plastometer at 175 °C

	Apparent viscosity, Pa · s		
Batch	Rheometer	Plastometer	Difference, %
2026	1560	1980	27
2029	1820	1870	3
2065	1820	2270	25
2069	1920	2240	17
2072	1840	2160	17
2080	2130	2500	17
2081	1990	2370	19

Table 2 Comparison of apparent viscosities obtained with a rheometer and a plastometer at 190 °C

	Apparent viscosity, Pa · s		
Batch	Rheometer	Plastometer	Difference, %
2026	920	1180	28
2029	1040	1210	16
2065	1270	1430	13
2069	1140	1460	28
2072	1090	1380	27
2080	1230	1570	28
2081	1250	1460	17

4.2 *Comparison of Capillary Rheometry and Extrusion Plastometry*

After the results of the plastometer tests have been converted to viscosity (Eq 4 to 6), the problem arises of how best to compare the plastometer viscosities with those obtained using the capillary rheometer. Does one select for comparison the capillary viscosity at the apparent shear rate of the plastometer test, or does one select the capillary viscosity at the apparent shear stress of the plastometer test (61,000 Pa)? The two methods will give identical results only if measurements by the two instruments yield identical viscosities.

The authors obtained the best agreement between the two instruments by using the first method—that is, by using the capillary viscosity at the plastometer shear rate. An analysis of the two methods confirmed that this should be so.

Table 1 lists viscosities at 175 °C for seven feedstock batches measured using the capillary rheometer and the extrusion plastometer, along with the percent difference between the two measurements. Table 2 shows results at 190 °C. The same results are shown graphically in Fig. 4 and 5. If best-fit lines were to be drawn for Fig. 4 and 5, the correlation coefficients would be 63% and 75%, respectively.

4.3 *Correction of Capillary Rheometry Data*

The Rabinowitsch correction was applied to the shear rates, but the Bagley correction for entrance effects was not applied. However, the value of e required to make the rheometer and plastometer shear stresses equal was calculated, and it typically fell between 1.0 and 1.5. Regarding R, the true shear rate was found to be 1.1 times the apparent shear rate, on average, for the seven feedstocks, at both 175 and 190 °C. If there is a significant entrance effect, one would expect the apparent viscosities measured by the plastometer to be greater than those measured by the capillary rheometer. This was the case.

Slip at the capillary wall was indicated in a test of feedstock batch number 2026, in which an orifice with nearly the same L/D (16.0 versus 16.2), but with a larger D (1.59 versus 1.17 mm, or 0.0625 versus 0.046 in.), was used in determining viscosity. When slip is present, orifices of the same L/D but with different Ds will give different viscosities, and the difference

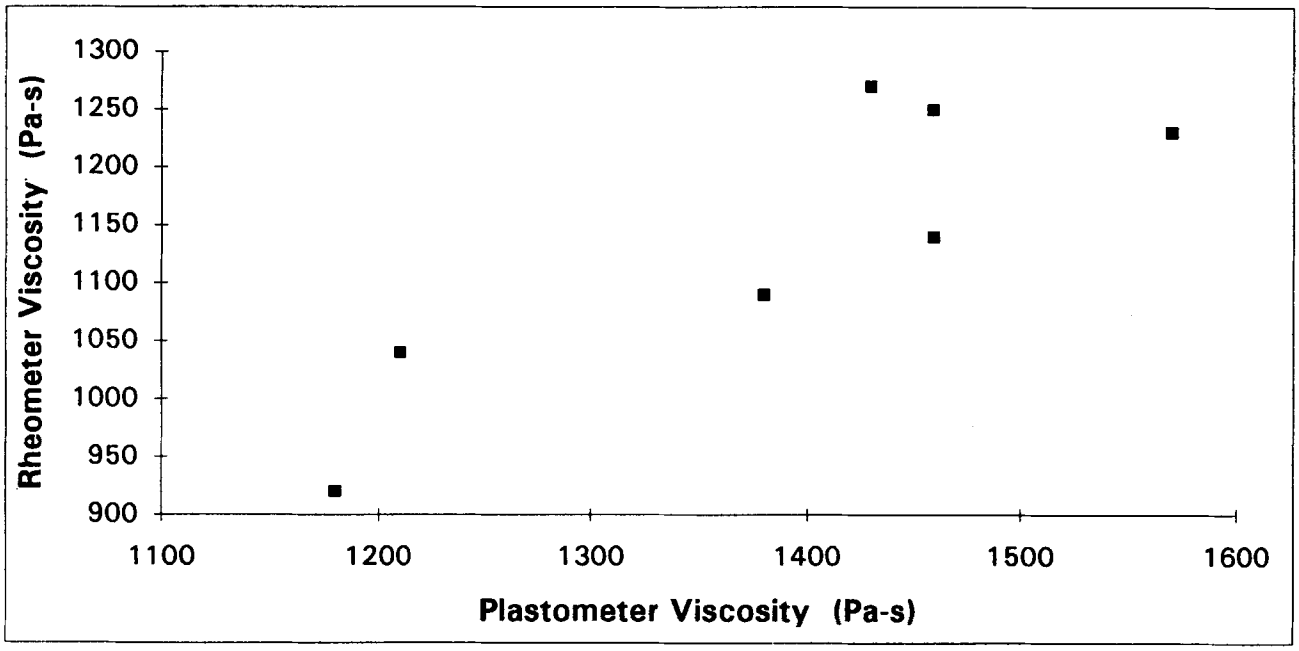

Fig. 5 Rheometer versus plastometer apparent viscosity at 190 °C

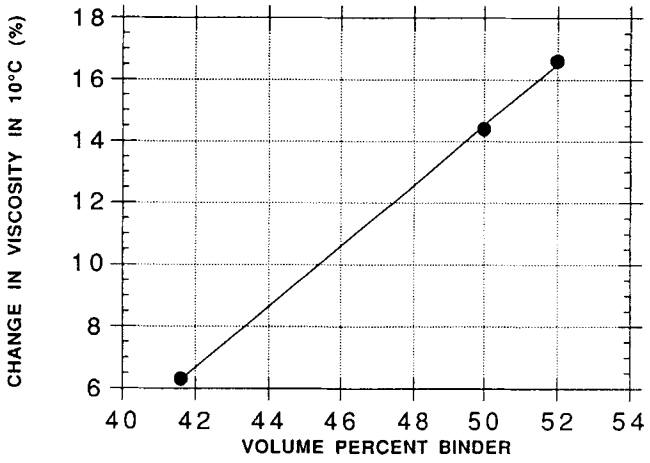

Fig. 6 Change in viscosity in 10 °C versus volume percentage of binder. Source: Ref 9

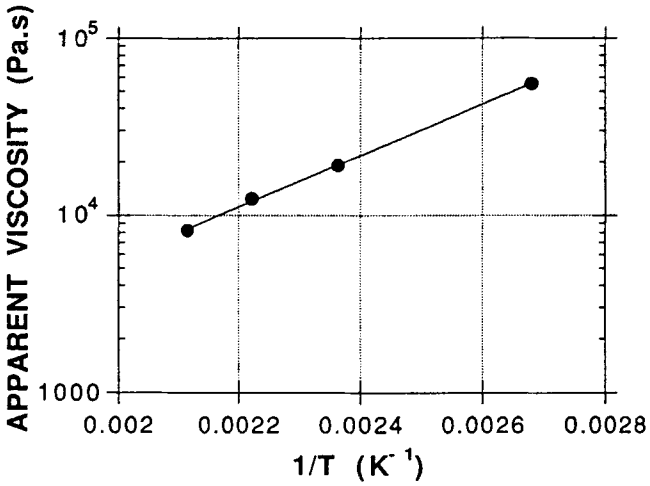

Fig. 7 Apparent viscosity versus reciprocal temperature. Source: Ref 9

will be in the direction of lower apparent viscosities obtained with the smaller *D*s (Table 3).

4.4 *Applications of Capillary Rheometry*

Individually, the capillary rheometer is used to study high shear rate, (i.e., molding and flow properties), and the extrusion plastometer is used to study day-to-day variations in feedstock. Figure 6 shows the change in viscosity in 10 °C versus volume percentage of binder at a shear rate of approximately 1000 s^{-1} (Ref 9). This type of information may be used in determining and adjusting molding parameters. The temperature sensitivity of viscosity increases with increasing binder content.

Figure 7 is an Arrhenius plot of the activation energy for feedstock flow at a shear rate of approximately 40 s^{-1} (Ref 9). Because the capillary rheometer data are more accurate with respect to obtaining true viscosity, and can be corrected to obtain true viscosity, this technique was used. Again, as with the data in Figure 6, this information can be used to establish successful molding temperatures for feedstocks.

4.5 *Applications of Extrusion Plastometry*

Figure 8 is a graph of the variation in apparent viscosity as measured with the extrusion plastometer versus the variation in work input during mixing (Ref 10). Sources of work input include mixing temperature, time, and material shear rate. In this

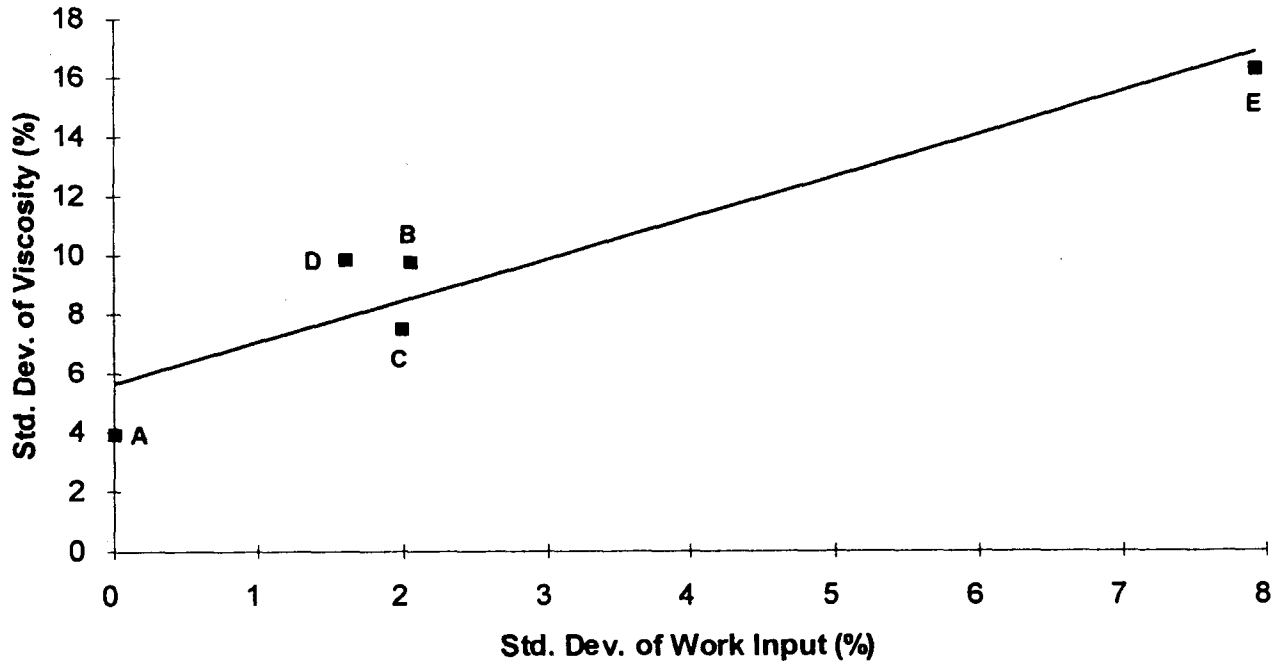

Fig. 8 Standard deviation of apparent viscosity versus standard deviation of work input. Source: Ref 10

Table 3 Slip study of batch 2026 at 175 and 190 °C

Shear rate, s^{-1}	Viscosity, Pa · s (D = 1.59 mm, or 0.0625 in.; 175/190 °C)	Shear rate, s^{-1}	Viscosity, Pa · s (D = 1.17 mm, or 0.046 in.; 175/190 °C
3.85	7250/6600	3.86	6140/5090
9.70	3650/3380	9.66	3420/2810
38.5	1510/1350	38.6	1400/1190
97.3	778/708	96.6	751/653
385	295/271	386	283/254
967	148/131	966	143/128

study of five groups of feedstock (A through E), a correlation between deviations in the mixing work input and the apparent viscosity was found, with a correlation coefficient of 88%. Viscosity variations were minimized when work input remained constant.

Figure 9 shows how apparent viscosity can be manipulated simply by changing the ratio of plasticizer to resin in otherwise invariant mixes. These results, established with the extrusion plastometer, were used as the basis for formulating a lower-viscosity feedstock.

5. Discussion

5.1 *Capillary Rheometry*

Although there are differences in metal loading among the batches—samples 2026 and 2029 being 58% metal by volume; 2080 and 2081, 59%; 2065, 2069, and 2072, 60%—the feedstocks are essentially indistinguishable at shear rates above 100 s^{-1}. There is some separation of the feedstocks at low shear rates, especially at 175 °C. (It is generally true in rheometry that sensitivity to material differences decreases as shear rate increases.) Based on these and other data accumulated for a variety of feedstocks, the low shear-rate range is thought to represent the degree of metal dispersion, whereas the high shear-rate regime is characteristic of the overall feedstock composition.

5.2 *Comparison of Capillary Rheometry and Extrusion Plastometry*

Regarding the correlation between the viscosities obtained using the capillary rheometer and the extrusion plastometer, there is a higher correlation coefficient at 190 °C (75%), versus 175 °C (63%), although neither data set exhibits a strong correlation. The primary source of discrepancies between the two types of measurements is the small L/D, 3.8, for the extrusion plastometer, versus the 16.2 L/D for the capillary rheometer. The smaller the capillary L/D, the greater the entrance effects, which contribute to inaccurately large pressure drops and thus shear stresses. Also, the presence of slip tends to make the viscosities greater for smaller orifices. The orifice of the capillary rheometer has a diameter of 1.17 mm (0.046 in.), versus 2.1 mm (0.0825 in.) for the extrusion plastometer, thereby further contributing to the difference between the results established with each technique. Also, the viscosity range for these feedstocks is only about 30%. Capillary rheometry precision is approximately ±5%, and that can lead to significant scatter in this viscosity range. If the range were increased to a few hundred percent, it is expected that the correlation would improve.

Considering these effects on the measurements, agreement of the viscosities to within 20 to 30% is better than expected.

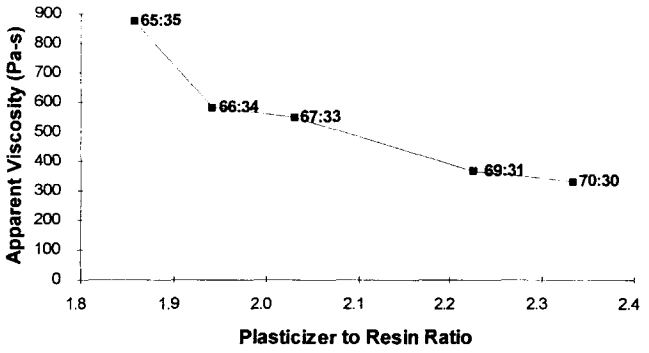

Fig. 9 Apparent viscosity versus plasticizer-to-resin ratio

The 3% difference for batch 2029 at 175 °C is not readily explained. Moreover, if the data for this batch are eliminated, the correlation coefficient from the regression analysis increases to 93% at 175 °C.

5.3 *Correction of Capillary Rheometry Data*

From the results, we know that R is not large and that slip has a significant effect on viscosity. For example, at approximately 39 s^{-1} shear rate and 175 °C, there is a 7% reduction in viscosity as measured with the smaller capillary. At 190 °C and the same shear rate, there is a 12% reduction. The difference between the results with the two capillaries is greater at 190 °C for shear rates of approximately 100 s^{-1} or less. This may account, in part, for the greater discrepancy between the capillary rheometer and the extrusion plastometer data at 190 °C (Table 2) versus 175 °C (Table 1).

5.4 *Applications of Capillary Rheometry*

The strength of capillary rheometry is its ability to provide data over a broad range of shear rates (of crucial importance in light of the fact that few materials of industrial interest are Newtonian) and the flexibility it offers in testing conditions, which allows one to make the corrections necessary to render the measurements instrument independent.

5.5 *Applications of Extrusion Plastometry*

The extrusion plastometer has its place in the day-to-day control of feedstock production. Figures 8 and 9 show the sensitivity of the instrument to variations in mixing and feedstock composition. Relatively minor feedstock variations can be resolved in the low shear-rate regime with the extrusion plastometer.

6. Conclusions

- At shear rates above 100 s^{-1}, minor changes in the degree of mixing or metal dispersion are indistinguishable. Both capillary rheometry and extrusion plastometry can be used to test in the shear-rate range below 100 s^{-1}, in which even minor feedstock variations are indicated.
- A strong correlation between viscosities measured by capillary rheometry and extrusion plastometry was not established; however, at 175 °C, a correlation coefficient of 93% was established when the data for batch 2029 were eliminated.
- Factors contributing to the discrepancies between the results of the two measurement techniques include entrance effects due to the smaller L/D for the extrusion plastometer and slip at the walls of the capillaries.
- Capillary rheometry can be used to establish true viscosity, independent of the measurement technique, for engineering calculations. Also, guidelines for molding parameters can be established.
- Extrusion plastometry indicates mixing and compositional variations, and can be used as an in-house quality control tool.

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