스크류 레오미터를 이용한 고분자 용융점도 측정

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A novel method to measure the melt viscosity: the screw rheometer

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Introduction

The concept of screw rheometer using single screw extruder is not new. The first model, called "Helical Screw Rheometer (HSR)" as shown in Fig. 1, was introduced by Kraynik et al. in 1984 [1]. But it was only used the measurement of viscosity about polymer solution at room temperature. For the purpose of measurement of polymer melt viscosity, the "Helical Barrel Rheometer" (HBR) in Fig. 2 was commercialized by David B. Todd et al. in 1997 [2]. The defeats of HBR are that it is difficult to clean split barrel and helical grooving at barrel has fixed geometry. Screw Rheometer is able to measure melt viscosity like HBR despite of using single screw extruder which is a actual polymer processing instrument and can change geometry by exchanging kinds of screw.

Many common laboratory viscometers are rotational devices that employ cone-and-plate, concentric-cylinder or parallel-plate geometries. All of these instruments require the measurement of torque and rotational rate to obtain the shear-rate dependence of the viscosity [1,3]. Capillary and slit rheometers require the measurement of pressure drop and volumetric flow rate. But Screw Rheometer requires the measurement of pressure difference and rotational rate. So the viscosity measurement by screw rheometer does not need to measure either the torque or the flow rate.

For homopolymer such as LDPE, viscosities measured by screw rheometer and commercial capillary rheometer show good agreement with various temperature. The screw rheometer is capable of measuring the accurate melt viscosity of the rubber materials, which do not show the temperature dependency in the capillary rheometer type.

Experimental

The modified extruder for screw rheometer is Göttfert single screw extruder using blind plug for closed discharge. A square-pitched full flight single screw was used (D = 45 mm, L/D = 25, Compression ratio = 3.0) as shown in Fig. 3.
The pressure transducers that are capable of measuring small amount of pressure difference and easily being exchangeable are located in the 20D and 23D position.

For the purpose of a curve fitting and comparison between the results obtained on the screw rheometer and those on conventional viscosity measuring equipment, Göttfert capillary rheometer (RHEO-TESTER 1501) was used.

As shown in Table 1, the resins for experiment such as LDPE, GPPS, ABS, SBS and EPDM/PP are almost supplied by LG Chem Ltd except for Nylon 6 by KOHAP Ltd

Result and Discussion

Fig. 4 shows the original diagram by screw rheometer including raw data such as pressures as a function of screw speed (P20D, P23D: Pressure at the 20D & 23D barrel positions, n: RPM [1/min], TM23D: Melt temperature at the 23D position). With this raw data including the change of pressure as a function of screw speed, the viscosity and shear rate are easily calculated from equation.

Fig 5, 6 show the shear viscosities of homopolymers (Low Density PolyEthylene (LDPE) & General Purpose PolyStyrene (GPPS)) by the screw rheometer in comparison with those by the Göttfert capillary rheometer. In case of homopolymers in Fig 5 &6, the shear viscosities by screw rheometer are identical to those by capillary rheometer.

Fig. 7, 8 show the shear viscosities of ABS dry powder (LG DP215) obtained by capillary rheometer and by screw rheometer at three different temperatures (240℃, 260℃ and 280℃). Although the capillary rheometer does not show the difference of shear viscosity with various temperatures in Fig. 7, Screw rheometer indicates the clear temperature dependency of shear viscosity in Fig. 8.

Screw rheometer shows more accuracy at low shear rate than capillary rheometer in Fig 9 that the shear viscosities of Styrene-Butadiene-Styrene (SBS, LG LUPRENE 475P) by screw rheometer and capillary rheometer are compared with various temperatures.

Fig. 10 shows the shear viscosities with changing temperature of EPDM/PP (60/40 wt%, LG Keyflex TO) in comparison with screw rheometer and capillary rheometer. As far as viscosity at low shear rate region is concerned, the screw rheometer can give the temperature dependency that capillary rheometer cannot.

Fig. 11 shows the shear viscosities of Nylon 6 (KOHAP EN 300) by screw rheometer and capillary rheometer with various temperatures. Even screw rheometer has the sensitivity measuring low shear viscosities that capillary rheometer does not measure correctly.

Conclusion

The screw rheometer gives reasonable results regardless of the shape of material such as pellet, bead, powder, popcorn, paste, etc.In case of homopolymer the shear viscosities using the screw rheometer are well matched with those using capillary rheometer.

In case of high rubber content material such as ABS, SBS and EPDM/PP the screw rheometer shows the temperature dependency clearly.

For Nylon 6, with which the capillary rheometer has a difficulty at low shear rate region, the screw rheometer can measure shear viscosity accurately.In addition, screw rheometer can trace chemorheological behavior: polymer degradation during the processing and the reactive extrusion

Reference

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TABLE. 1 Materials used in the experiments

Name of resin	LDPE	GPPS	ABS	SBS	EPDM/PP	Nylon 6
Shape of material	pellet	pellet	powder	popcorn	pellet	pellet
Name of grade	LGLUTENE BB0808	LG GPPS 25SPE	LG DP215	LG LUPRENE 475P	LG KEFLEX TO	KOHAP EN300
Properties	MI = 0.8	MI = 2.9	BD = 55%	BD/SM=60/40 %	60/40 wt %	Mw=162,289

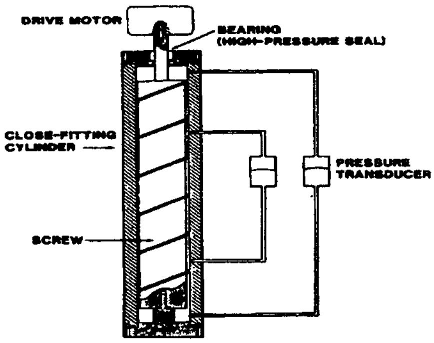

Figure 1. The Helical Screw Rheometer (HSR) investigated by Kraynik et al in 1984 [1].

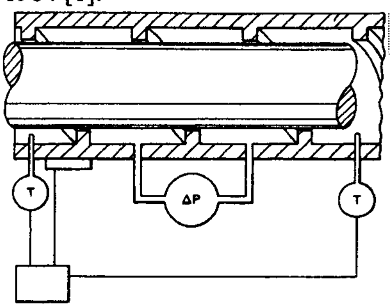

Figure 2. The Helical Barrel Rheometer (HBR) commercialized by David B. Todd et al in 1997 [2].

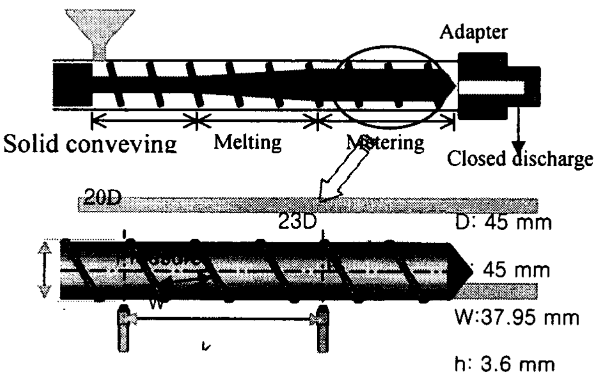

Figure 3. Schematics of screw rheometer.

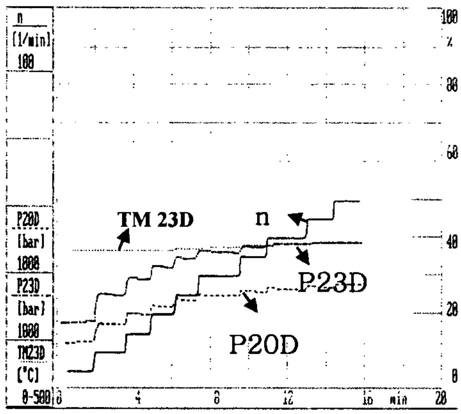

Figure 4. The original diagram by screw rheometer showing raw data such as pressures as a function of screw speed (P20D, P23D: Pressure at the 20D & 23D barrel positions, n: RPM [1/min], TM23D: Melt temperature at the 23D position).

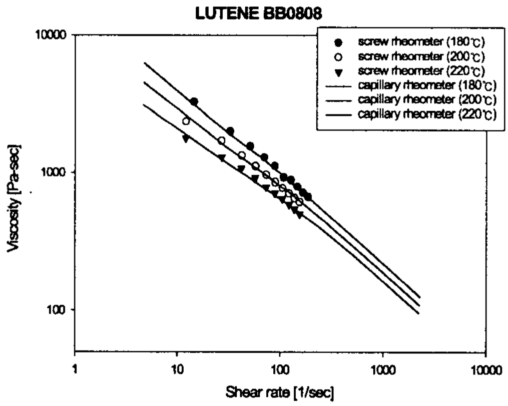

Figure 5. Shear Viscosity of LDPE (LG LUTENE BB0808): Screw Rheometer vs Capillary Rheometer

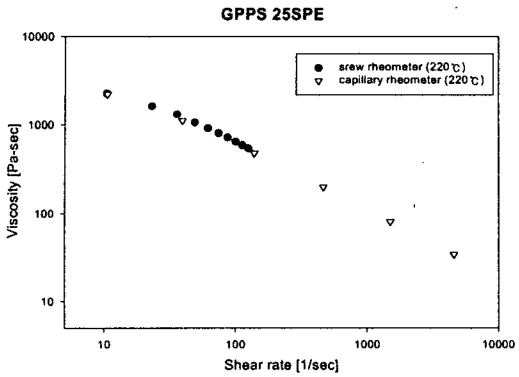

Figure 6. Shear Viscosity of GPPS (LG GPPS 25SPE): Screw Rheometer vs Capillary Rheometer.

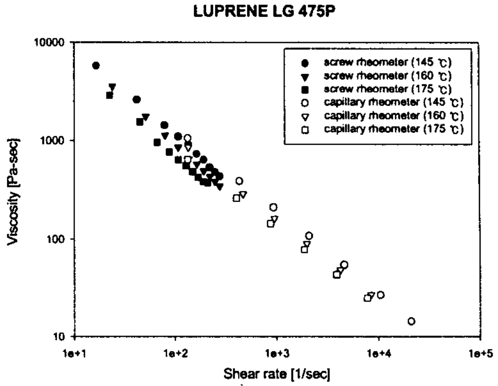

Figure 9. Shear Viscosity of SBS (LG LUPRENE LG 475P): Screw Rheometer vs Capillary Rheometer.

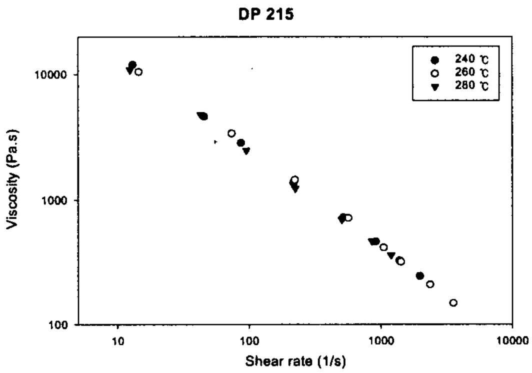

Figure 7. Shear Viscosity of ABS (dry power, LG DP215) obtained by capillary rheometer.

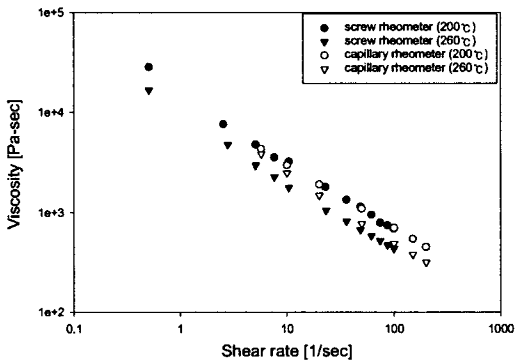

Figure 10. Shear Viscosity of EPDM/PP (LG Keyflex TO): Screw Rheometer vs Capillary Rheometer.

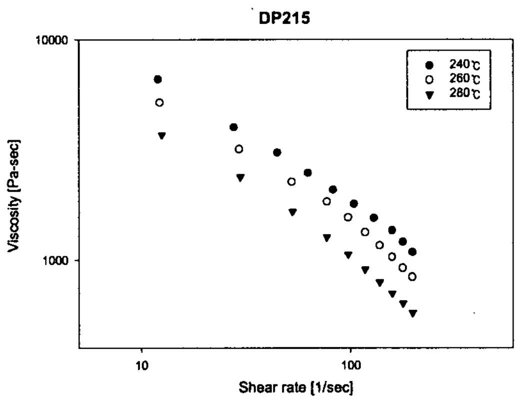

Figure 8. Shear Viscosity of ABS (dry power, LG DP215) obtained by screw rheometer.

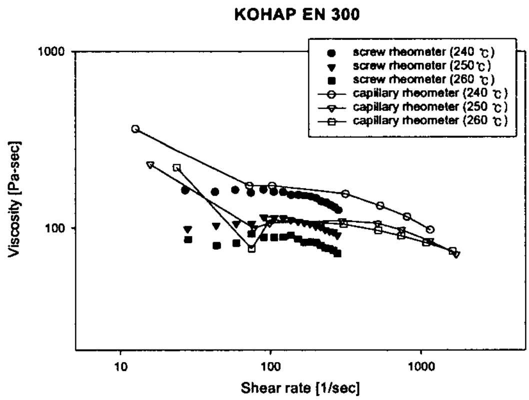

Figure 11. Shear Viscosity of Nylon 6 (KOHAP EN 300): Screw Rheometer vs Capillary Rheometer.