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ORIGINAL CONTRIBUTION

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Extensional viscosity from entrance pressure drop measurements

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Dedicated to the memory of Tasos Papanastasiou

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Abstract Extensional rheological properties are important in characterization and processing of polymeric liquids. The use of entrance pressure drop to obtain extensional viscosity is particularly attractive because it can be applied to both low and high viscosity liquids using the Bagley correction obtained from a conventional capillary rheometer.

Low density polyethylene of three different melt index values, including IUPAC-X (a different batch of IUPAC-A), and a high density polyethylene were tested using a commercial capillary rheometer. The entrance pressure drop (ΔP_{en}) was obtained with a "zero-length" orifice die with an abrupt contraction. The contraction ratio was 12:1. Predictions from several approximate analyses to calculate the uniaxial extensional viscosity η_u (using an axisymmetric contraction) from ΔP_{en} were compared. These comparisons are summarized in the appendices.

Due to the transient nature of contraction flows, η_u is also a function of the strain (ε). This was examined by comparing η_u from ΔP_{en} (Cogswell's analysis was chosen for convenience) with transient extensional viscosity (η_u^+) at different magnitudes of ε from fiber-windup technique (Padmanabhan et al., 1996). η_u^+ at $\varepsilon \approx 3$ was found to be close to η_u from ΔP_{en} (using Cogswell's analysis) for two LDPE samples that had fiber-windup data available. The magnitude of the strain in the contraction did not vary with strain rate.

Key words Extensional viscosity – entrance pressure drop method – Cogswell's analysis – extensional strain

Introduction

The importance of extensional rheological properties of polymers has been recognized for nearly three decades now (Walters, 1992). However, due to the difficulty in generating a controlled extensional flow for rheometrical purposes, several ingenious techniques have been developed for polymer melts and solutions (Macosko, 1994). Among these techniques, the use of pressure drop through contractions has gained a great deal of attention (Binding, 1993). Contraction flows are also important from a processing standpoint. The entrance pressure drop method is particularly attractive because it allows a productive use of the Bagley correction obtained in conventional capillary rheometry using an existing instrument. In addition, the technique can be applied equally well to both high viscosity polymer melts (Laun and Schuch, 1989) and low viscosity polymer solutions (Binding and Walters, 1988)

The complexities in the contraction flow are well reviewed (Boger, 1987; White et al., 1987). To facilitate the calculation of extensional viscosity from entrance pressure drop measurements, several approximate analyses have been proposed. Metzner and Metzner (1971) assumed a shear-free sink flow to analyze the entry flow. Cogswell (1972) conducted a more elaborate analysis considering both shear and extensional components. Gibson (1988, 1989) modified Cogswell's analysis, but assumed sink flow kinematics. Binding (1988) followed the arguments used by Cogswell, and presented a more rigorous analysis by using variational principles to minimize the energy dissipation in the contraction. In all the aforementioned analyses, the effect of the so-called "shear elasticity," represented by the first normal stress difference (N_1) was assumed negligible. In a subsequent analysis, Binding (1991) took the effect of N_1 also into consideration and showed that as the level of shear elasticity of the fluid increases, there is a significant contribution of the first normal stress difference to the entrance pressure drop. The analyses of Binding were reviewed recently (Binding, 1993).

In Appendix A, a summary of the analyses of sink flow (Metzner and Metzner, 1971), Cogswell (1972), Gibson (1988, 1989), and Binding (1988, 1991) is presented. The results from Binding's and Gibson's analyses have been manipulated to explicitly obtain the extensional rheological quantites (Padmanabhan, 1993; Padmanabhan and Bhattacharya, 1994). The predicted extensional viscosity from all these approximate analyses are compared in Appendix B. In addition, the uniaxial extensional viscosity (η_u) data from axisymmetric contraction were compared with the planar extensional viscosity obtained using a slit orifice die.

In order to obtain an analytical solution, the approximate analyses assume that the shear and extensional flows in the contraction region are locally steady state. Thus, the analyses assume that the extensional viscosity is, at best, a power-law function of the extension rate. However, published studies have reported difficulty in generating steady-state extensional flow for LDPE, even up to a Hencky strain of 7 (Meissner et al., 1981; Laun and Schuch, 1989). The short residence time of the fluid in the contraction clearly insures that the flow is transient.

Since the extensional flow in the contraction is not steady state, a comparison of η_u^+ from a transient elongation technique with η_u from entrance pressure drop (ΔP_{en}) will be worthwhile in shedding light on the transient nature of the extensional flow in the contraction. η_u^+ data obtained using the fiber-windup technique (Padmanabhan et al., 1996) are compared with η_u from ΔP_{en} in this paper.

Experimental

Commercial LDPE samples with melt index values of 6 (DOW 752) and 12 (DOW 4012) donated by Dow Chemical Co. (Midland, MI) and IUPAC-X (Lupolen 1810 with melt index of 1.5 donated by Dr. H.M. Laun, BASF, Ludwigshafen) were used for the studies. The weight-average molecular weight $\bar{M}_w$ of DOW 752 is 194000 with a molecular weight distribution ($=\bar{M}_w/\bar{M}_n$, $\bar{M}_n$ being the number average molecular weight) of 14, while the corresponding values for IUPAC-X are 1.2×10^6 and 65, respectively. In addition, a high density polyethylene (HDPE) was also tested.

A Rheometrics Mechanical Spectrometer (RMS-800) fitted with a 25 mm diameter and 0.1 rad cone and plate geometry was used to obtain η and N_1 at 150 °C for the LDPE samples. Pellets of the sample were loaded on the plate and allowed to melt at 150 °C before lowering the cone. The maximum shear rate achievable was limited by the onset of edge failure. The onset of edge failure was determined by monitoring the real-time recording of the torque and normal force using a strip chart recorder. Failure to reach steady state and a continuous decrease in the magnitude of torque and normal force at a given shear rate was used as a sensitive indication of the onset of edge failure.

A commercial capillary rheometer (Goettfert Rheo-tester 1500) was used for the entrance pressure drop measurements. A pressure transducer with a range 0 to 100 bar (0 to 10 MPa) was used to record the pressures. Circular channel dies with a channel diameter of 1 mm and lengths of 10, 20 and 30 mm manufactured by Goettfert were used to obtain the end pressure drop following the Bagley precedure. Circular channel orifice dies of diameter 1.04 and 1.96 mm were made out of stainless steel with an exit angle of 120 degrees. A rectangular channel orifice die was also made with a width of 8.9 mm and height of 0.84 mm. The orifice dies all had small *L/D* (or *L/H*) ratios ($<<1$) with the inner edges machined sharp – in other words, they were all "zero-length" dies. All the dies used had an abrupt contraction.

The lowest shear rate achievable on the capillary rheometer was limited by the lowest measurable pressure (0.5 bar by calibration) by the pressure transducer. The limitation in achieving the highest shear rate was the onset of melt fracture.

Results and discussion

The steady shear viscosity and first normal stress difference data for the polyethylene samples from the rotational rheometer and the shear viscosity at higher rates from the capillary rheometer are shown in Fig. 1. Deviations between the cone and plate and the low shear rate capillary rheometer data are due to the low pressure readings obtained with the latter.

End pressure drop (ΔP_{end}) obtained using the Bagley procedure is compared with the orifice pressure drop (ΔP_0) in Fig. 2. Within experimental error, the two values are in agreement, in accord with the observation

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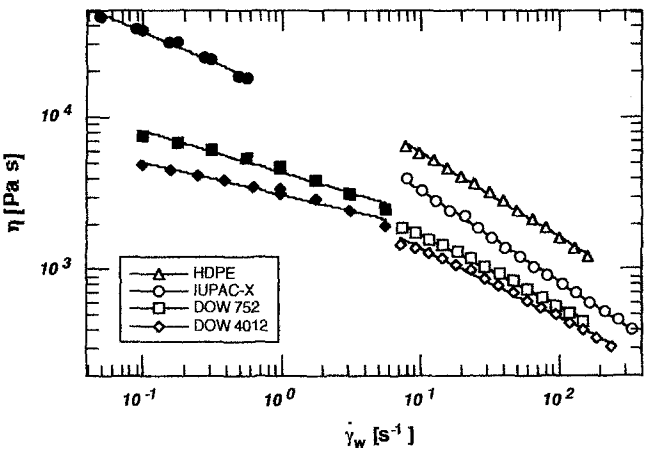

Fig. 1a Steady shear viscosity from cone and plate (filled symbols) and capillary (open symbols) rheometers. The lines shown are power-law fits, primarily for the purpose of a guide

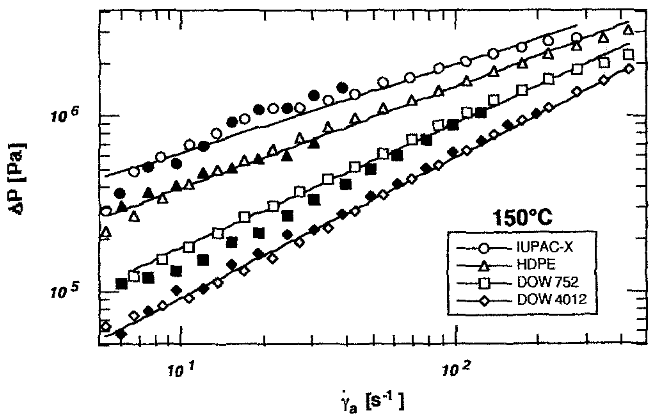

Fig. 2 Comparison of end pressure drop (filled symbols) with orifice pressure drop (open symbols) for axisymmetric contraction, shown as a function of the apparent shear rate. The lines shown are power-law fits to the orifice pressure drop. A greater difference is seen between the orifice and end pressure drop readings for DOW 752. However, this difference is less than the accuracy of the pressure transducer

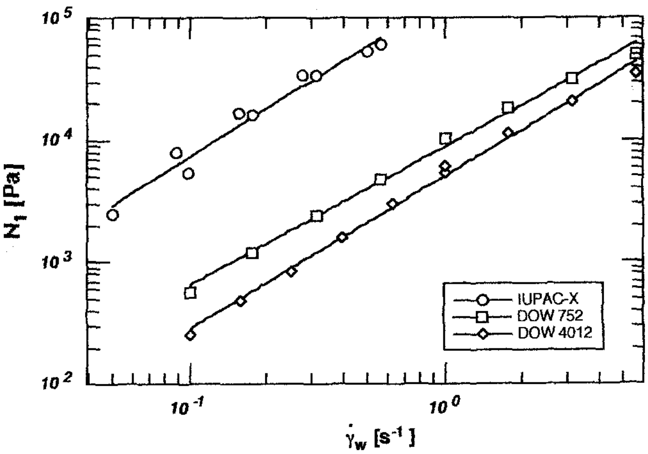

Fig. 1b First normal stress difference of the LDPE samples. The lines shown are power-law fits to the data

of Laun and Schuch (1989). However, there appears to be a greater scatter in ΔP_{end} (Fig. 2). This scatter is believed to be due to extrapolation error. Since the maximum shear rate that can be reached with a given pressure transducer decreases with increasing die length, ΔP_{end} was obtained over a smaller range of shear rates compared to ΔP_0. In addition, ΔP_0 can be obtained faster, being a direct measurement. Hence, ΔP_0 was used to calculate the extensional viscosity.

A comparison of the approximate entry flow analyses to predict the extensional viscosity using ΔP_0 is presented in Appendix B. For convenience, we have chosen the results from Cogswell's analysis to compare with η_u^+ from the fiber-windup technique below.

Recent reviews (James, 1990; James and Walters, 1993; Macosko, 1994) and studies (Ferguson and Hudson, 1993; Banfill, 1991) on extensional rheology have demonstrated the need to include the strain history and strain along with the strain rate when reporting extensional rheological data. Thus, transient extensional flow exists in the contraction region and the magnitude of the extensional viscosity will be a function of both extension rate and strain.

In the capillary rheometer, the strain history due to the amount of pre-shearing the fluid encounters before reaching the contraction may be considered negligible due to the low shear rates prevalent; for example, the experimental conditions employed in this study subjected the polymer melt to shear rates of magnitude $5\times10^{-4}\ s^{-1} \leq \dot{\gamma}_a \leq 5\times10^{-2}\ s^{-1}$ in the barrel.

In the contraction region, however, the extensional flow, particularly, imparts significant strains on the sample. The extensional strain and strain rate increase rapidly as the fluid approaches the die entrance plane (Feigl and Öttinger, 1994). Thus, the fluid does not have an opportunity to approach steady state in extensional flow (Feigl and Öttinger, 1994). However, it is interesting to note that for a given flow rate there is hardly any dependence of $\dot{\varepsilon}$ on the radial position of the streamline and the maximum value is similar at all streamlines (Feigl and Öttinger, 1994).

Due to the complex nature of the entry flow and the non-uniform strain and strain rates (in the axial direction) present, an analytical expression for calculating the extensional strain is not readily available. A simple approximate method to calculate an overall strain is given below. Consider a fluid element of unit length in the barrel with same cross-sectional area as the barrel (A_b). When this fluid element flows through the die, its cross-sectional area may be assumed to be the same as that of the die (A_d). Assuming the fluid to be incompressible, the resultant maximum Hencky strain is then given by

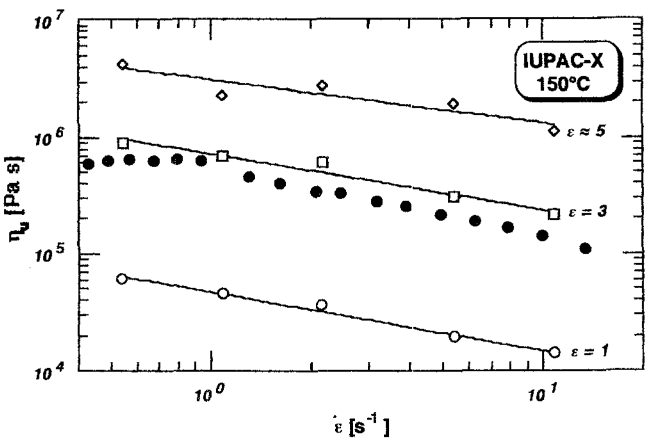

Fig. 3a Comparison of uniaxial extensional viscosities from the entrance pressure drop method, and the transient extensional viscosity (open symbols) from the fiber-windup method (Padmanabhan et al., 1996) at different values of the strain for IUPAC-X LDPE. The lines shown are power-law fits to the fiber-windup data

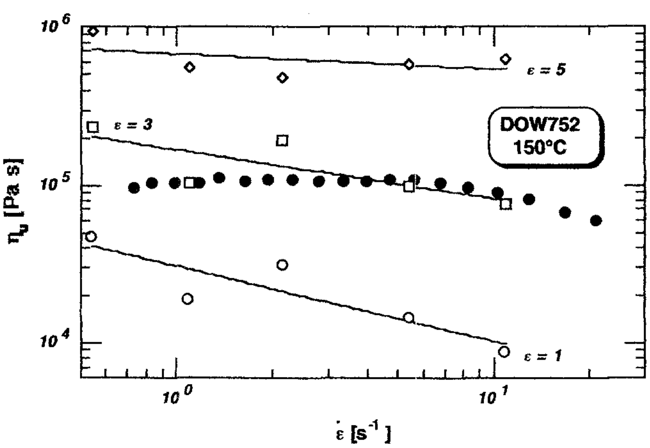

Fig. 3b Uniaxial extensional viscosity from entrance pressure drop method using Cogswell's analysis compared with transient extensional viscosity (open symbols) at specific values of the strain (ε) from the Fiber-windup method (Padmanabhan et al., 1996) for DOW 752 LDPE. The straight lines are power-law fits to the fiber-windup data

$$\varepsilon_{\max} = \ln\left(\frac{A_b}{A_d}\right)$$

which is same as the expression given by McKinley et al. (1991). For the contraction ratio of A_b:A_d:: 12:1 used in this study, $\varepsilon_{\max} \approx 5$. Feigl and Öttinger (1994) showed that the maximum strain obtained at various points along the streamline in an axisymmetric contraction flow is of similar magnitude to the value obtained using Eq. (3).

Comparison of η_u from ΔP_{en} with η_u^+ from the fiber-windup technique (Padmanabhan et al., 1996) is made in Fig. 3 for IUPAC-X and DOW 752 samples (fiber-windup data were available for these two samples only). From the fiber-windup technique, η_u^+ as a function of $\dot{\varepsilon}$ at various values of ε are shown for both materials. The IUPAC-X results of Fig. 3a using ΔP_{en} method were found to be in good agreement with the IUPAC-A results reported by Laun and Schuch (1989) using rod-pulling and ΔP_{en} methods.

For IUPAC-X, the strain rate dependence of η_u from ΔP_{en} is in agreement with the fiber-windup results. However, for DOW 752 (Fig. 3b), the ΔP_{en} results show a slight extension-thickening at the lower strain rates, in contrast to the fiber-windup results. More remarkable is the agreement in the magnitude of η_u from ΔP_{en} with η_u^+ from fiber-windup method at $\varepsilon \approx 3$ *for both materials*, within experimental error. Note that the same contraction geometry was used for both materials. This suggests that for η_u obtained from Cogswell's analysis using a contraction ratio of 12:1, the average strain in the contraction region is ~ 3. It is reasonable that the average strain is less than the maximum estimated value of 5. The magnitude of the average strain did not vary noticeably with the strain rate.

Conclusions

Several approximate analyses are available in the literature to calculate an extensional viscosity from entrance pressure drop measurements. Comparison of these analyses revealed considerable differences in their predictions.

Extensional viscosity from entrance pressure drop (using Cogswell's analysis) was in good agreement with the fiber-windup results at a strain of 3. Thus, for a large contraction ratio Cogswell's analysis predicts an average strain of 3 in the contraction.

Appendix A

Assuming the existence of only a shear-free flow, Metzner and Metzner (1970) used sink flow kinematics to evaluate the extension rate, $\dot{\varepsilon}$. The velocity field, using spherical coordinates, is given by

$$v_r = -\frac{Q}{A}\ , \quad v_\theta = v_\phi = 0$$

where Q is the volumetric flow rate, and A is the cross-sectional area in the converging region. Substituting for A and using an average convergence angle of 15° (Macosko, 1994), we have

$$\dot{\varepsilon} = \frac{Q}{2\,\pi R^3}$$

where R is the radius of the orifice. In terms of apparent shear rate at the orifice, $\dot{\gamma}_a$ we can rewrite the above equation as

$$\dot{\varepsilon} = \frac{\dot{\gamma}_a}{8} \tag{1}$$

where

$$\dot{\gamma}_a = \frac{4Q}{\pi R^3}$$

Since the shear contributions are assumed negligible, the extensional normal stress difference, $\tau_{11}-\tau_{22}$, may be written as (Balakrishnan and Gordon, 1976)

$$\tau_{11} - \tau_{22} = \Delta P_{en} \tag{2}$$

The extensional viscosity, η_u, is defined as

$$\eta_u = \frac{\tau_{11} - \tau_{22}}{\dot{\varepsilon}} \tag{3}$$

Cogswell (1972) assumed contributions from both shear and extensional flow in the contraction region. The shear viscosity was assumed to obey power-law, while the extensional viscosity was assumed to be constant. In the converging region, the flow was assumed to be locally fully developed. Thus, the axial velocity profile is the same as that for a power law fluid undergoing Poiseuille flow through a channel of cross-section defined by the converging walls. For an abrupt contraction, the fluid was assumed to define its own convergence profile corresponding to a minimum pressure drop. Based on this criterion, the extension rate was given by

$$\dot{\varepsilon} = \frac{4}{3(n+1)} \frac{\tau_w}{\Delta P_{en}} \dot{\gamma}_a \tag{4}$$

where τ_w is the wall shear stress, and n is the power-law index of the shear viscosity ($\eta = m\,\dot{\gamma}^{n-1}$). The extensional stress is given by

$$\tau_{11} - \tau_{22} = \frac{3}{8}(n+1)\,\Delta P_{en} \tag{5}$$

Cogswell (1978) has given a detailed discussion of the differences between the locally fully developed flow kinematics assumed in his analysis and the sink flow kinematics assumed by others.

Binding (1988) presented a more rigorous analysis, by essentially making the same assumptions as Cogswell. In addition, the extensional viscosity was assumed to be a power-law function of the extension rate, and variational principles were employed to minimize the energy dissipated in the contraction, from which explicit relations for the pressure drop were obtained. The results given by Binding can be manipulated to give explicitly (Padmanabhan, 1993)

$$\dot{\varepsilon} = \frac{(3n+1)(1+k)^2}{3k^2(1+n)^2} \frac{\tau_w}{\Delta P_{en}} \dot{\gamma}_a \tag{6}$$

and

$$\tau_{11} - \tau_{22} = \frac{3k\,2^{k-1}(1+n)^2}{(1+k)^2(3n+1)I_{nk}} \Delta P_{en} \tag{7}$$

where k is the index in the assumed power-law functional dependence of the extensional viscosity, in $\eta_u = s\,\dot{\varepsilon}^{k-1}$, evaluating using

$$k = \frac{d \ln \Delta P_{en}/d \ln \dot{\gamma}_w}{1 + n - (d \ln \Delta P_{en}/d \ln \dot{\gamma}_w)} \tag{8}$$

and

$$I_{nk} = \int_0^1 \left\{ abs\left[2 - \left(\frac{3n+1}{n}\right)\right] \xi^{(1+n)/n} \right\}^{k+1} \xi d\xi \tag{9}$$

Gibson (1988, 1989) assumed sink flow kinematics and the absence of vortices (i.e., a minimum pressure drop was not evaluated), even for an abrupt contraction, to arrive at (Padmanabhan, 1993)

$$\dot{\varepsilon} = \frac{\dot{\gamma}_a}{4} \tag{10}$$

and

$$\tau_{11} - \tau_{22} = \frac{\left\{\Delta P_{en} - \frac{2^{3n+2}\tau_w}{3n\pi^{3n+1}}\right\}}{\left\{\frac{2}{3k} + G_g\right\}} \tag{11}$$

where

$$G_g = \int_0^{\pi/2} (1+\cos\beta)^{g-1} (\sin\beta)^{g+1}\, d\beta \tag{12}$$

and

$$g = \frac{d \ln \Delta P_{ene}}{d \ln \dot{\gamma}_a} \tag{13}$$

where

$$\Delta P_{ene} = \Delta P_{en} - \left(\frac{2^{3n+2}\tau_w}{3n\pi^{3n+1}}\right) \tag{14}$$

In the earlier analyses attempting to extract the extensional characteristics of the fluid using contraction flow (Cogswell, 1972; Binding, 1988; Gibson, 1989), only the shear and extensional viscosities of the fluid were assumed to contribute significantly to the energy dissipation. In extensional flow, the elasticity of the fluid manifests itself through the large magnitude of the extensional viscosity typically observed. However, in shear flow, the elasticity is typically represented by the first and second normal stress differences, N_1 and N_2, respectively.

Assuming N_2 to be negligible, Binding (1991) reanalyzed the contraction flow problem by including the contribution of N_1. The results of this revised analysis can be rewritten in terms of the extension rate and extensional normal stress difference as follows (Padmanabhan, 1993). The extension rate and stress and related parameters are same as that given in Eqs. (6–9) for the older analysis of Binding (1988), with $\Delta P'_{en}$ replacing ΔP_{en} where

$$\Delta P'_{en} = \Delta P_{en} + \frac{p\,(3n-1)\,(3n+1)^{p+2}}{3n^{j+1}\,2^{j+1}\,(2n+j+1)\,(3n+j+2)}\,\dot{\gamma}_a^{j+1} \tag{15}$$

ΔP_{en} being the measured total entrance pressure drop, and p and j are the parameters obtained from fitting a power-law function to the steady-state values of the first normal stress difference, $N_1 = p\dot{\gamma}^{j+1}$. All the remaining parameters are the same as the older analysis of Binding (1988) given above.

Appendix B

Uniaxial extensional viscosities from the various entry flow analyses (Appendix A) are compared in Fig. B-1 for IUPAC-X and DOW 4012 samples. The results for only two of the four materials tested is shown in Fig. B-1 because these were found to contain all the observed features of the analyses.

Cogswell's and the older Binding's (1988) analyses showed the same trend of $\eta_u(\dot{\varepsilon})$ for all the materials. It is remarkable that even though Cogswell (1972) assumed η_u to be independent of $\dot{\varepsilon}$, the results show otherwise.

The magnitude of η_u predicted by Cogswell's analysis was higher than that of Binding's (1988) analysis. This is in agreement with observations reported by others for axisymmetric and planar contractions (Tremblay, 1989; Padmanabhan and Bhattacharya, 1993), but in contrast to the planar contraction results of Padmanabhan and Bhattacharya (1994). For Binding's analysis, an average extension rate cannot be obtained analytically. Hence, we have selected to represent the extension rate at the center of the entrance plane (Padmanabhan, 1992). On the other hand, an average extension rate is evaluated in Cogswell's analysis (Cogswell, 1972). Thus, the extension rate will be lower, while the extensional viscosity will be higher for Cogswell's analysis, compared to Binding's analysis, as seen in Fig. B-1.

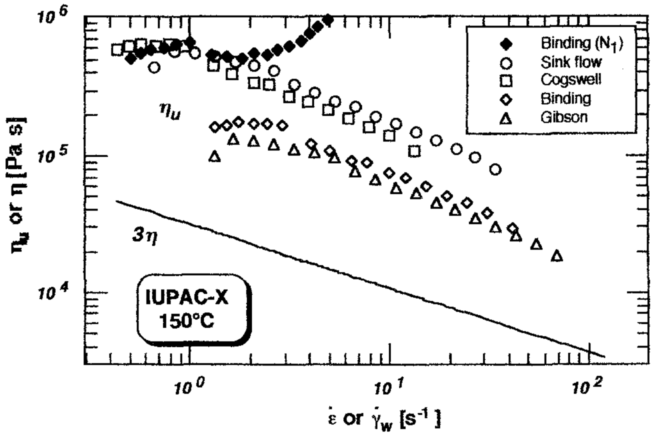

Fig. B-1a Uniaxial extensional viscosities of IUPAC-X obtained from the various analyses. Also shown is three times the shear viscosity (the straight line)

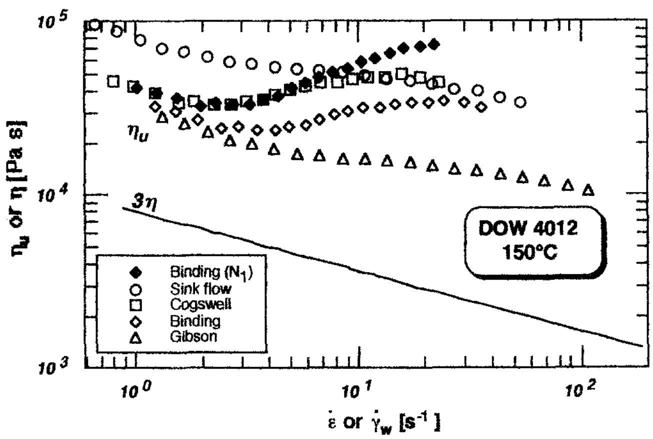

Fig. B-1b Uniaxial extensional viscosities of DOW 4012 obtained from the various analyses. Also shown is three times the shear viscosity (the straight line)

Both Cogswell's and the older Binding's (1988) analyses predict an extension-thinning behavior for IUPAC-X (Fig. B-1a) and extension-thickening behavior for DOW-4012. Independent confirmation of the actual existence of an extension-thinning trend was available only for the IUPAC-X sample (a different batch of IUPAC-A; results for IUPAC-A are given by Laun and Schuch, 1989 and references therein). Sink flow and Gibson's analyses showed extension-thinning behavior for all the samples tested. Binding's newer analysis predicted extension-thickening behavior for all samples.

Gibson (1988, 1989) assumed that even for the abrupt contraction geometry the converging flow profile of the fluid is defined by the walls of the contraction; in other words, recirculation zones are assumed to be absent for all flow conditions. This forced convergence assumption of sink flow and Gibson's analyses may be responsible for the failure of these analyses to capture

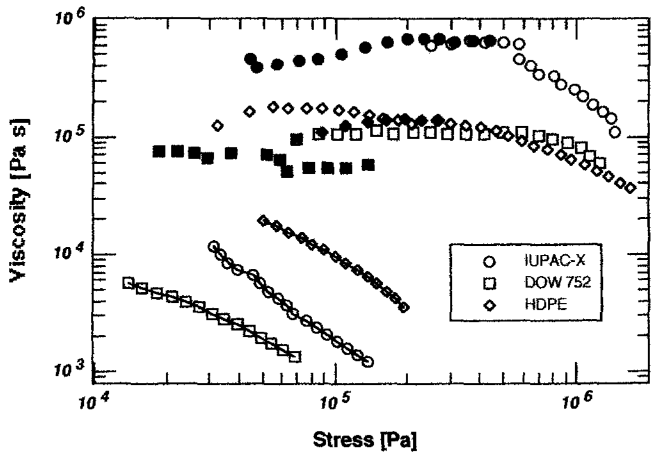

Fig. B-2 Three times the shear viscosity (open symbols with connecting lines), uniaxial extensional viscosity (open symbols) and planar extensional viscosity (filled symbols). Note that the viscosities are plotted against the corresponding stresses. The extensional viscosities were all obtained using Cogswell's analysis

the extension-thickening behavior. Consequently, extensional viscosities predicted using the forced convergence assumption are likely to be in greater error as demonstrated by Binding and Jones (1989).

Calculations of η_u obtained using the newer analysis of Binding (1991), that incorporates N_1 as a correction for shear-elasticity in the converging region, are also shown in Fig. 3 for both materials. Steady rotational shear N_1 data obtained using the cone and plate geometry (Fig. 1) were used to apply the newer analysis of Binding (1991). The exaggerated extension-thickening behavior observed with the newer analysis of Binding (1991) may be due to the use of extrapolated N_1 data in our calculations.

Planar extensional viscosity obtained using the slit orifice die are shown in Fig. B-2 and compared with η_u and η. The equations used were as follows (Cogswell, 1972):

$$\dot{\varepsilon} = \frac{2}{3(n+1)} \frac{\tau_w \dot{\gamma}_a}{\Delta P_{en}} \tag{1}$$

and

$$\tau_{11} - \tau_{22} = \frac{1}{2}(n+1)\,\Delta P_{en} \tag{2}$$

It is interesting to note that in most cases the planar extensional viscosity is close in magnitude to η_u (Laun and Schuch, 1989).

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