

DETERMINATION OF MOLAR MASS DISTRIBUTION
OF POLY(TETRAFLUOROETHYLENE)

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Abstract: High-temperature static and dynamic laser light scattering characterization of polytetrafluoroethylene in perfluorotetracosane has been accomplished after solving the following experimental difficulties: (1) constructing a high-temperature laser light scattering spectrometer which can be operated at a temperature as high as 340 °C, (2) developing a new all-glass apparatus for dissolving and filtering, (3) measuring the refractive index increment of polytetrafluoroethylene in perfluorotetracosane at 325 °C, and (4) establishing a scaling relation between the molecular weight (M) and the translational diffusion coefficient at both infinite dilution and the zero scattering angle from only two broadly distributed polytetrafluoroethylene samples so that we can convert the translational diffusion coefficient distribution to the molecular weight distribution. This paper presents a summary of our published results of the characterization of poly(tetrafluoroethylene), which emphasizes the experimental details of high-temperature laser light scattering and the principle of the transformation from a translational diffusion coefficient distribution to a molecular weight distribution.

INTRODUCTION

Laser light scattering (LLS) has been used successfully to characterize a range of relatively intractable polymers, such as polyethylene (denoted as PE) in 1,2,4-trichlorobenzene (Ref.1,2), poly(ethylene terephthalate) (denoted as PET) in hexafluoroisopropanol (Ref.1,3) and poly(1,4-phenylene terephthalamide) (denoted as PPTA) in concentrated sulfuric acid (Ref.4-8). An absolute determination of the molecular weight of polytetrafluoroethylene (denoted as PTFE), $-(CF_2CF_2)_x-$, commercially known as "Teflon" TFE fluorocarbon resin (a registered trademark of Du Pont) by means of laser light scattering represents another advance in the development of LLS as an analytical technique for polymer characterizations (Ref.9). In order to carry out the experiments at high temperatures (340°C), we have built a high-temperature light-scattering spectrometer (HTLLS) and developed a new all-glass dissolution/filtration apparatus, capable of dissolving, transferring and clarifying the polymer solution at elevated temperatures under an inert gas and which has no moving components except for the stopcock. In this report, we present details of our light scattering studies on PTFE solution properties around the melting point of PTFE

($\approx 330^{\circ}\text{C}$). Although Teflon was invented more than 40 years ago, an absolute determination of the molecular weight represents an interesting challenge by any analytical techniques. To date, no solvents which are suitable for Teflon have been found. Thus, in our approach to create a Teflon solution, we used very low molecular weight Teflon, e.g., $n\text{-C}_{24}\text{F}_{50}$, and oligomers of Teflon-like polymers, e.g., oligomers of poly(chlorotrifluoroethylene), as solvents with the following criteria: (a) a boiling point sufficiently high for the oligomer melt to form a liquid at temperatures greater than 330°C , (b) a refractive index sufficiently different from that of the Teflon polymer to give a reasonable refractive index increment, and (c) the oligomer molecular weights sufficiently low for the characteristic decay times to be distinctly different from those of Teflon. The same approach could be applicable to other polymers which are difficult to dissolve.

Oligomers of poly(chlorotrifluoroethylene) appear to be poor solvents for Teflon. We were able to study only a low molecular weight Teflon polymer in detail. Precise measurements of intensity-intensity time correlation function permit us to make a Laplace inversion using a variety of techniques including multiexponential singular value decomposition (MSVD), regularized inversion of Laplace integral equation (RILIE), and the CONTIN algorithm developed by Provencher. We were able to obtain an estimate of the normalized characteristic line-width distribution which could be reduced to a distribution of translation diffusion coefficient at infinite dilution, independent of the method used in the Laplace inversion.

As a delicate balance exists among the three criteria above, higher molecular weight Teflon polymers can be expected to dissolve in higher molecular weight fractions of polyperfluorocarbons. Nevertheless, the present study demonstrates our new approach to light scattering characterizations of such intractable polymers. It should also be noted that we are aware of the effects associated with the presence of multicomponent solvents (Ref.10). However, as the refractive index difference among oligomers within a narrow boiling-point range should be fairly small, corrections for preferential interactions between different solvent components and the polymer are relatively small. Furthermore, the oligomers are chemically similar, which could provide another reason to consider such a "solvent" as a pseudo one-component

fluid in light-scattering studies. In order to show the validity of this approximation, we used two different boiling-point fractions of the oligomer as solvents to characterize the same sample and the oligomers as solvent to characterize a known PTEFE sample. The results agree with each other within experimental error limits. In addition, we also characterized two Teflon samples in $n\text{-C}_{24}\text{F}_{50}$, which is a pure single component solvent. We found that the results obtained in the oligomers are very similar to those obtained in $n\text{-C}_{24}\text{F}_{50}$, which confirms our assumption about the oligomers. We have established a scaling relationship between D_0^0 and M , i.e., $D_0^0 = 3.99 \times 10^{-4} M^{-0.55}$. Using this scaling relationship, we obtained the molecular weight distributions (MWD) of both Teflon samples.

Our approach to Teflon solution characterization is also influenced by the high melting point (T_m) exhibited by Teflon. With $T_m \approx 330^\circ\text{C}$, our instrumentation must be capable of high-temperature ($\approx 350^\circ\text{C}$) operation, not only for the light-scattering spectrometer but also for all aspects of solution preparation, transfer, and clarification. The success of our study depends essentially on experimental developments in being able to achieve high-temperature operations at all levels. In this report, we shall be concerned with the refinements for high-temperature operations and show that our results are independent of the multicomponent nature of the oligomers used, as well as the methods of data analysis.

EXPERIMENTAL

Preparation of Solvent: The solvents used in this work were $n\text{-C}_{24}\text{F}_{50}$ and a mixture of oligomers of poly(chlorotrifluoroethylene). $n\text{-C}_{24}\text{F}_{50}$ (courtesy of W. Buck, Polymer Products Department, Du Pont Experimental Station, Wilmington, Del.) was used as received. The oligomers were made by Hooker Industrial & Specialty Chemicals Division of Occidental Chemical Corporation. The specific grade used was "Fluorolube" LG-160. Prior to use, the "Fluorolube" was distilled at a pressure of 0.01 mm Hg. The fraction boiling between 169 and 179°C (56% of the mixture) was used for the majority of the light-scattering work. This fraction had an atmospheric pressure boiling temperature of about 345°C . Vapor phase chromatography of this material (6 ft. x 1.4 in. column, 20% OV-210 on "Chromasorb" PAW/DMCS, 150° to 240° at $10^\circ/\text{min}$, then

isothermal at 240°C, thermal conductivity detector) showed 85 area % to be present as three triplet peaks with retention times centered at 6.3, 9.6, and 13.9 minutes. In order to assess thermal stability of the "Fluorolube" solvent, a sample of the distilled solvent was sealed under vacuum in a heavy-walled glass tube, then placed in a circulating air oven at 340°C for twenty-four hours. After this heat treatment, the solvent had changed from colourless to yellow. Vapor phase chromatography of this material showed a complex mixture with a large increase in the amount of low elution time components. Mass spectroscopic analysis of several of the peaks showed them to be chlorofluorocarbon residues from solvent degradation. Thus, the solvent degrades slowly at this temperature, possibly due to end group instability. The oligomers were prepared by W. Buck.

Preparation of Solutions: Two distilled fractions of Fluorolube with boiling points $\approx 350^\circ\text{C}$ and $>350^\circ\text{C}$ and indices of refraction 1.320 and 1.325 at 488 nm and 340°C, respectively, were used as solvents for the low molecular weight Teflon polymer. $n\text{-C}_{24}\text{F}_{50}$ was used as solvent for both the low and high molecular weight Teflon samples. Solutions at different concentrations were prepared individually by dissolving known amounts of Teflon in the fractionally distilled oligomer solvents at $340^\circ\pm 2^\circ\text{C}$. For $n\text{-C}_{24}\text{F}_{50}$ as solvent, the solution was prepared at $327^\circ\pm 2^\circ\text{C}$. Particular care should be taken to consider polymer dissolution at only a few degrees centigrade below the solvent boiling point. Over periods of hours at elevated temperatures, the oligomers may degrade or decompose to form lower boiling-point fluids resulting in a pressure increase which could cause a mild explosion if a sealed dissolution/filtration apparatus were used. Our experience with the unexpected explosions requires the use of a pressure-release mechanism. Therefore, we have designed a dissolution/filtration apparatus (Ref.9) as shown schematically in Fig.1.

Sleeve A (without a bottom) is joined to the shaded stopper, which is connected to a reflux condenser by means of a grease-free glass joint. Cup B, with magnetic stirrer E, sits on top of the lower (fine grade) sintered glass filter F_2 and is used for polymer solution clarification. The procedure is as follows. Add solvent and polymer to B of the argon-filled apparatus with the dust-free light-scattering cell D already attached. Insert the reflux condenser, which has been flushed with dust-free argon, making sure that the apparatus with PTFE and oligomers is under an inert atmosphere at room

temperature. Raise temperature to 340 ± 2 °C. With gentle stirring, complete dissolving should take a few hours. Apply argon pressure through the coarse-grade sintered glass filter F_1 to move the polymer solution from B to C, which has the fine-grade sintered glass (bottom) filter F_2 . The additional pressure difference plus gravity will filter the polymer solution directly to the dust-free light-scattering cell D. After the filtration process is completed, the additional argon pressure is released. The stopcock above the upper filter F_1 could be closed during light-scattering experiments. The high-temperature light scattering chamber in the light-scattering spectrometer accepts the entire light scattering cell, which could be controlled to ± 0.1 °C at 340°C over a region of ≈ 1 cm where the polymer solution was located. Temperature could be controlled to ± 0.1 °C over a 1 hour period. The important point is to be able to dissolve a polymer and to filter the polymer solution under an inert atmosphere at elevated temperatures, without having moving components as all glass stopcocks or joints tend to lock themselves at very high temperatures. We must also provide a pressure release mechanism but without permitting the low vapor pressure oligomers to escape. So, the miniature water-cooled condenser was introduced. The all glass dissolution/filtration apparatus can be cleaned by pyrolysis so that it can be used repeatedly without degradation of performance.

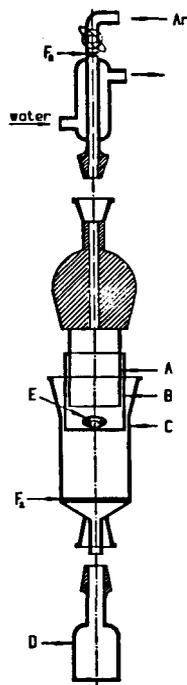


Fig.1 Dissolution/
filtration
apparatus

Methods of Measurements: A HTLLS spectrometer was constructed for the PTFE characterization. Fig.2. shows schematic top and side views of the essential features of the high-temperature, multiple detector light-scattering spectrometer. The main components are listed in the following: 1. silicone rubber insulation; 2. heating wires for the brass thermostats; 3. outer brass thermostat with fluid circulation facilities; 4. inner brass thermostat which has a separate temperature controller and thermometer and can accommodate scattering

cells up to 27 mm o.d.; 5. stainless steel shield to thermally isolate and decouple, at least partially, the temperature environment of the inner light-scattering chamber from the outside chamber; 6. Glan-Thompson polarizers; 7. fluid circulation paths; 8. lens; 9. field aperture; 10. optical fiber bundle; 11. rotating plate for multiple detectors; 12. RT-200 Klinger rotary table with 0.01° step size; 13. cooling plate to isolate the outer thermostat from the rotary table; 14. stainless steel standoffs for thermal isolation.

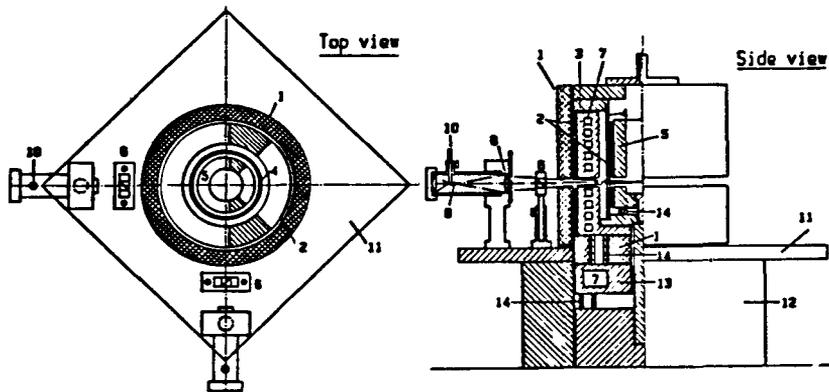


Fig. 2 HTLLS spectrometer (Laser and PMT are not shown)

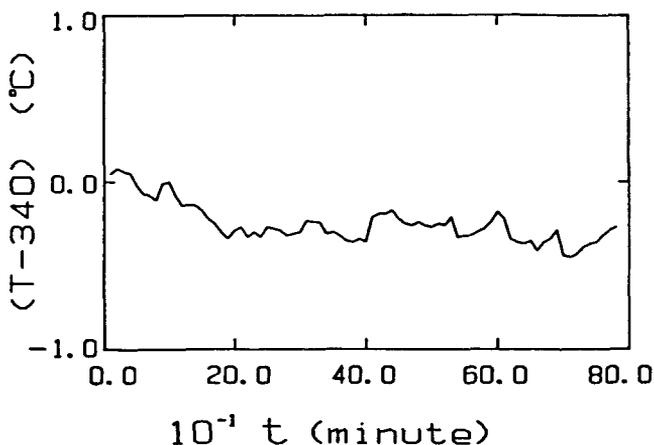


Fig. 3 Typical temperature fluctuation in the cell

At 340 °C, temperature gradient becomes a more critical factor. The platinum resistance thermometer is located very close to the polymer solution. Fig.3. shows a typical tracing of the temperatures monitored by the platinum resistance thermometer located near the light scattering cell in the inner thermostat. Intermediate term (1 hour) control of $\pm 0.1^\circ$ and long term (10 hours) control of $\pm 0.5^\circ$ could be achieved at 340 °C. Long term temperature control depends partially on room temperature fluctuations, even in the presence of an outer thermostat and stainless steel shielding between the two thermostats.

Intensity and line-width measurements were performed in a similar manner as described elsewhere (Ref.10).

RESULTS AND DISCUSSION

Refractive Index and Refractive Index Increment: We used a deformed cylindrical light-scattering cell so that the solution/glass/air interface was not perpendicular to the incident laser beam at the exit side of the light-scattering sample chamber. Refraction could be measured and calibrated using organic solvents of known refractive index. At 340°C and $\lambda_0 = 488$ nm, the two boiling fractions have $n_0 = 1.320$ and 1.325, respectively. We obtained $(\partial n/\partial C)_T = 0.053$ mL g⁻¹ at 340°C and $\lambda_0 = 488$ nm for the lower boiling fraction oligomers, $(\partial n/\partial C)_T = 0.047$ mL g⁻¹ for the high boiling fraction oligomers ($n_0 = 1.325$) and $(\partial n/\partial C)_T = 0.011$ for C₂₄F₅₀ ($n_0 = 1.361$) at 325°C.

Light Scattering Intensity Measurements: The excess absolute integrated scattered intensity of light scattered by a dilute polymer solution at concentration C (g/mL) and scattering angle (θ) has the approximate form:

$$\frac{HC}{R_{VV}(K)} \approx \frac{1}{M_w} (1 + K^2 R_g^2/3) + 2A_2 C \quad (1)$$

where H (in units of mole cm²/g²) is equal to $4\pi^2 n_0^2 (\partial n/\partial C)_T^2 / (N_A \lambda_0^4)$ with N_A , λ_0 and $(\partial n/\partial C)_T$ being, respectively, Avogadro's number, the wavelength of light in vacuo, and the refractive index increment. R is the excess Rayleigh ratio and the subscript vv denotes vertically polarized incident and

scattered light. A_2 (in units of mole mL / g²) is the second virial coefficient and $K = (4\pi n/\lambda_0) \sin(\theta/2)$. From a Zimm plot,

we can determine the weight-average molecular weight M_w , A_2 and the root mean square z-average radius of gyration R_g . Fig.4. shows a typical Zimm plot of Teflon in low boiling fraction of poly(chlorotrifluoroethylene) oligomers.

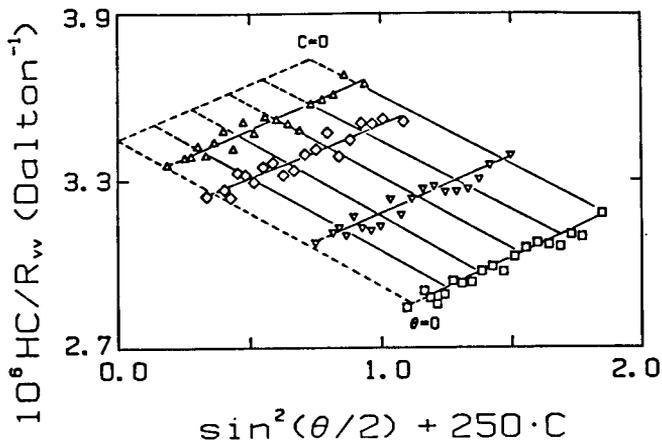


Fig.4 Typical Zimm plot of PTFE in the oligomers.

We recognize that oligomers within a narrow boiling point range still represent a multicomponent fluid. However, in light-scattering studies, some of the essential characteristics can be approximated as those of a single component. For example, the effect due to preferential absorption among oligomers should be weak. As refractive indices of oligomers are similar, the preferential absorption effect becomes less important in the molecular weight determination by light-scattering intensity measurements. In order to prove the assumption of the oligomers acted as a pseudo one component solvent, we further used $C_{24}F_{50}$ as a solvent. We repeated measurements for the same low molecular weight Teflon sample in $C_{24}F_{50}$. All static results are summarized in Table I.

Table I. Static Properties of PTFE in Various Solvents

PTFE type	solvent type	n	$\partial n / \partial C$ (mL/g)	M_w 10^5 g/mol	$A_2/10^{-5}$ mol·ml/g ²	R_g nm	T °C
DLX-6000	MO-I	1.320	0.053	2.84±10%	-6.7±20%	17.8±10%	340
	MO-II	1.325	0.047	2.78±10%	-6.2±20%	18.3±10%	"
	n- $C_{24}F_{50}$	1.361	0.011	2.6±15%	1.9±20%	20±25%	325
PTFE-5	"	"	"	21±15%	1.0±20%	59±14%	"

Light Scattering Line-width Measurements: The measured intensity-intensity time correlation function $G^{(2)}(K, \tau)$ in the self-beating mode has the form:

$$G^{(2)}(K, \tau) = A(1 + b|g^{(1)}(K, \tau)|^2) \quad (2)$$

where A is the background and b is a parameter depending on the detection optics. In our correlation function measurements, we do not use A as an adjustable parameter and insist on having A and $\lim_{\tau \rightarrow \infty} G^{(2)}(K, \tau)$ agree to within 0.1%. For polydisperse polymers, the measured first order electric field correlation function $b|g^{(1)}(K, \tau)|$ is related to the normalized characteristic line-width distribution $G(\Gamma)$:

$$g^{(1)}(K, \tau) = \int_0^\infty G(\Gamma) e^{-\Gamma(K)\tau} d\Gamma \quad (3)$$

We shall continue to use four different established methods of correlation function profile analysis:

(i) Cumulants expansion (Ref.14)

$$\ln|g^{(1)}(K, \tau)| = -\bar{\Gamma}(K)\tau + \frac{1}{2}\mu_2(K)\tau^2 + \dots \quad (4)$$

where $\bar{\Gamma} = \int_0^\infty \Gamma G(\Gamma) d\Gamma$ and $\mu_2 = \int_0^\infty (\Gamma - \bar{\Gamma})^2 G(\Gamma) d\Gamma$ with Variance (VAR) = $\mu_2/\bar{\Gamma}^2$. The cumulants method is applicable for $G(\Gamma)$ distributions which are not too broad ($\mu_2/\bar{\Gamma}^2 < 0.4$).

(ii) Multiexponential singular value decomposition (MSVD) (Ref.8,15,16)

$$G(\Gamma) \approx \sum_j P_j \delta(\Gamma - \Gamma_j) \quad (5)$$

such that $g^{(1)}(\tau) = \sum_j P_j \exp(-\Gamma_j \tau)$ with P_j being the intensity weighting factor of the j th representative fraction. The MSVD technique can be used to fit $G^{(2)}(K, \tau)$ exhibiting fairly broad unimodal characteristic line-width distributions. With $\mu_2/\bar{\Gamma}^2 \geq 0.5$, the cumulants method becomes difficult to use.

(iii) Regularized inversion of Laplace integral equation (RILIE) (Ref.16). A modified version of the method developed by Abbiss et al. and is described in detail elsewhere (Ref.16) RILIE, like MSVD, is applicable for unimodal $G(\Gamma)$ but has a slightly faster algorithm.

(iv) CONTIN (Ref.18,19): kindly furnished by S. W. Provencher.

We determined $\text{VAR} \sim 0.2$ by using a 3rd-order cumulants fit for a typical time correlation function of the low molecular weight Teflon sample in the oligomers measured at $\theta = 30^\circ$ and $C = 7.51 \times 10^{-4}$ g/mL as shown in Fig.5.

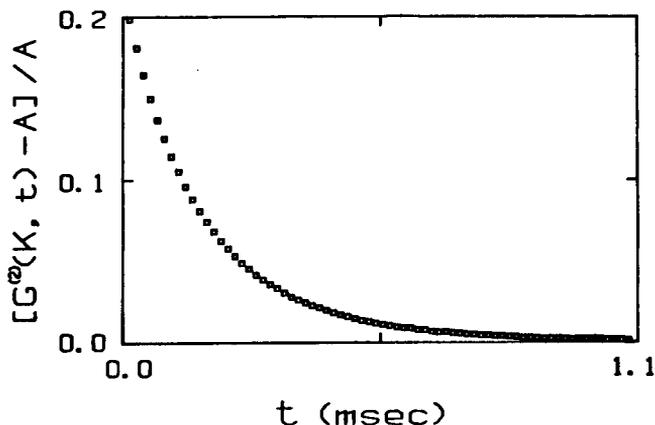


Fig.5 Typical time correlation function of PTFE in low boiling fraction of oligomers.

The same correlation function was analyzed by using methods (ii), (iii) and (iv) with $G(\Gamma)$ vs Γ shown in Fig.6.

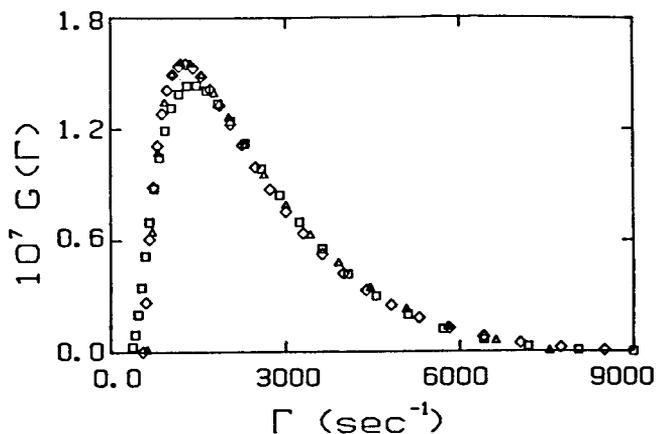


Fig.6 Plots of $G(\Gamma)$ versus Γ obtained by using three different methods of Laplace inversion: Squares, CONTIN; diamonds, RILIE; triangles, MSVD.

The inversion results clearly show that estimates of $G(\Gamma)$ using different methods of Laplace inversion agree for the Teflon solution. The same procedure has been tested by a variety of approaches, such as use of simulated data and polymers of known polydispersity. Thus, we are quite confident in our present estimate for $G(\Gamma)$, especially when $\mu_2/\bar{\Gamma} \approx 0.2$. By extrapolating $\bar{\Gamma}/K^2$ to zero K , we obtain the z-average translational diffusion coefficient $\bar{D}_0 (= \lim_{K \rightarrow 0} \bar{\Gamma}/K^2)$. By extrapolating \bar{D}_0 to $C = 0$, we are able to obtain \bar{D}_0^0 and the diffusion second virial coefficient k_d according to

$$\bar{D}_0 = \bar{D}_0^0 (1 + k_d C) \quad (6)$$

For the low molecular weight Teflon sample in the oligomers at 340°C , $\bar{D}_0^0 = 4.26 \times 10^{-7} \text{ cm}^2/\text{sec}$ and $k_d = -53.4 \text{ mL/g}$. For the low molecular weight and high molecular weight Teflon samples in $\text{C}_{24}\text{F}_{50}$ at 325°C , $\bar{D}_0^0 = 3.66 \times 10^{-7} \text{ cm}^2/\text{sec}$, $k_d = -35.2 \text{ mL/g}$, $\bar{D}_0^0 = 1.21 \times 10^{-7} \text{ cm}^2/\text{sec}$ and $k_d = -130$, respectively. By using the scaling relation $\bar{D}_0^0 = k_D M_w^{-\alpha_D}$ with $k_D = 3.07 \times 10^{-4}$ and $\alpha_D = 0.54$ obtained for Teflon in $\text{C}_{24}\text{F}_{50}$ at 325°C , we can transfer D to M and $G(D)$ to $f_w(M)$. Further, we obtained $(M_w)_{\text{calc}} = 2.2 \times 10^5$ and 1.8×10^6 for the low and high molecular weight Teflon samples, respectively. Both are less than the measured M_w . The errors come from two aspects. One is from the uncertainty of the value k_D and α_D which we obtained only from two different molecular weight Teflon samples. The other is due to the broad molecular weight distribution (MWD) of the two samples. For a set of broad MWD samples, the k_D and α_D in $\bar{D}_0^0 = k_D M_w^{-\alpha_D}$ are not the same as in $D_0^0 = k_D M^{-\alpha_D}$, i.e. we cannot simply use \bar{D}_0^0 and M_w instead of D_0^0 and M to obtain k_D and α_D . In order to obtain the correct k_D and α_D , we applied the following principles. By letting $\theta \rightarrow 0$ and $C \rightarrow 0$, we have

$$\int_0^\infty G(D_0^0) dD_0^0 = \int_0^\infty F_z(M) dM = 1 \quad (7)$$

where $F_z(M)$ is the z-average molecular weight distribution function. By assuming $D_0^0 = k_D M^{-\alpha_D}$ and using Eq.(7), we can obtain

$$\int_0^\infty -G(D_0^0) \alpha_D D_0^0 d[\ln(M)] = \int_0^\infty F_z(M) M d[\ln(M)] \quad (8)$$

Further, by comparing both sides of Eq.(8), we have

$$F_z(M) = \frac{G(D_0^0) \alpha_D D_0^0}{M} \quad (9)$$

According to the definition of M_w , we have

$$M_w = \frac{\int F_w(M)M \, dM}{\int F_w(M) \, dM} \quad (10)$$

where $F_w(M)$ is weight average molecular weight distribution function and $F_w(M)M = F_z(M)$. We can rewrite Eq.(10) as

$$M_w = \frac{\int F_z(M) \, dM}{\int [F_z(M)/M] \, dM} \quad (11)$$

By combining Eq.(11), Eq.(9) and $D_o^0 = k_D M^{-\alpha_D}$, we finally obtain

$$M_w = \frac{k_D^{1/\alpha_D} \int G(D_o^0) \, dD_o^0}{\int G(D_o^0) D_o^{0/1/\alpha_D} \, dD_o^0} \quad (12)$$

For two (Teflon) samples of different molecular weight and distribution, we have two $G(D_o^0)$, denoted as $G_1(D_o^0)$ and $G_2(D_o^0)$. From them, we could calculate two $(M_w)_{\text{calc}}$, denoted as $(M_w,1)_{\text{calc}}$ and $(M_w,2)_{\text{calc}}$. The ratio of $(M_w,1)_{\text{calc}}$ and $(M_w,2)_{\text{calc}}$ is

$$\frac{(M_w,1)_{\text{calc}}}{(M_w,2)_{\text{calc}}} = \frac{[\int G_1(D_o^0) \, dD_o^0][\int G_2(D_o^0) D_o^{0/1/\alpha_D} \, dD_o^0]}{[\int G_2(D_o^0) \, dD_o^0][\int G_1(D_o^0) D_o^{0/1/\alpha_D} \, dD_o^0]} \quad (13)$$

The two calculated $(M_w)_{\text{calc}}$ have to equal the two measured M_w . It means that we already know the value of the left side of the Eq.(13). So, we can vary the value of α_D and calculate the right side of Eq.(13) until the left side equals the right side. In this way, we could find the correct α_D value. After we obtained α_D , we could get the k_D value by using Eq.(12). By using the above principle, we obtained $\alpha_D = 0.55$ and $k_D = 3.99 \times 10^{-4}$ in $D_o^0 = k_D M^{-\alpha_D}$ for Teflon in $C_{24}F_{50}$ at 325°C . Then, we made the transform to get $F_w(M)$ for both the low and the high molecular weight Teflon samples. They are shown in Fig.7. With only one Teflon polymer characterized in oligomers of poly(chlorotri- fluoroethylene) at this time, we could not determine the scaling relation $\bar{D}_o^0 = k_D M_w^{-\alpha_D}$ for Teflon in the oligomers at 340°C .

Fig.8. shows a log-log plot of R_g versus M_w for different polymer solutions. As A_2 for PTFE is negative, the Teflon coil appears to be substantially contracted, in agreement with our

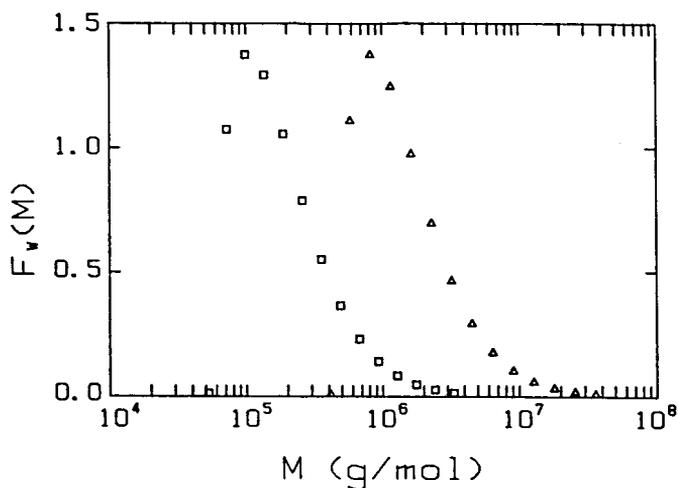


Fig.7 Molecular weight distributions of two Teflons obtained from $G(\Gamma)$. □: $M_w = 2.6 \times 10^5$, $M_z:M_w:M_n = 3.8:2.1:1.0$; Δ: $M_w = 2.1 \times 10^6$, $M_z:M_w:M_n = 4.4:2.3:1.0$.

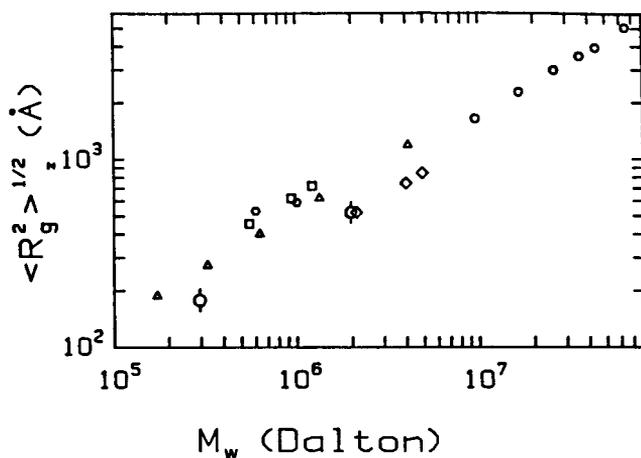


Fig.8 Log-log plot of R_g versus M_w for different polymer solutions. (Same as Fig. 1.2.9). Δ: poly(styrene) in benzene; ◇: poly(methylmethacrylate) in methyl methacrylate; ○: polyethylene in 1,2,4-trichlorobenzene; □: PETFE in diisobutyl adipate; and ⊙: Teflon in $n\text{-}(C_{24}F_{50})$.

present perception that PTFE coils are similar to those of polyethylene (PE) or PETFE. There could be subtle differences in the configuration of such polymer coils (PE, PETFE, PTFE) because of size differences between H and F. However, once in their respective solvents at whichever temperatures, the statistical configurations are essentially the same. Finally, we also made a time study on the stability of Teflon solution in the oligomers at 340°C. M_w remains constant for longer than 24 hours while \bar{r} ($\theta = 30^\circ$) appears to change after about 10 hours. Therefore, all our measurements were performed within an 8-hour period.

CONCLUSIONS

By characterizing poly(tetrafluoroethylene) known as Teflon, we have established a methodology of the high-temperature laser light scattering (HTLLS). As a non-invasive analytical technique, HTLLS is suitable or even irreplaceable sometimes for the characterizations of some intractable polymers, such as PETFE (or Tefzel, a registered trademark of Du Pont) and Teflon (a registered trademark of Du Pont). With information on M_w and MWD as well as R_g and second virial coefficients, further improvements on polymerization reactions and materials processing could be considered.

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