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Temperature-Independent Viscosity Characteristics of Polymer Systems

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Synopsis

The possibility of construction of temperature-independent viscosity characteristics of polymer systems by means of a "reduced variables" method similar to that of Ferry is discussed. It is stated that the viscosity as a function of temperature and shear rate can generally be regarded as the product of two functions, one of which depends only on the temperature and the other only on the shear rate. It is shown by dimension analysis and by assuming the flow of polymer systems to be viscoelastic in nature, that the ratio of apparent viscosity to initial Newtonian viscosity is a temperature-independent function of the product of the shear rate by the initial Newtonian viscosity. Hence, the latter (observed at shear rates tending to zero) appears to be a most important physical parameter governing the flow properties of polymer systems. At sufficiently low shear rates the apparent viscosity was found to be an exponential function of shear stress, so that the initial Newtonian viscosity can be determined by extrapolation. The above theoretical considerations were checked for the most reliable published data on the flow properties of condensed polymer sytems over a wide range of shear rates and temperatures. Experimental results obtained for polymer systems of different nature can be represented satisfactorily by corresponding temperature-independent viscosity curves.

Introduction

The steady viscosity of non-Newtonian polymer systems varies with the shear rate (or with the similarly dependent shear stress) and with the temperature. The relation between viscosity and temperature is most complicated and offers but little opportunity for theoretical analysis. This is why the problem of presenting viscometric data independent of temperatures deserves special attention.

The possibilities of temperature-independent presentation of the dynamical characteristics of elastomers, i.e., of the real and imaginary parts of a complex modulus of rigidity, compliance, etc., have already been investigated extensively. Ferry and his collaborators[1] found first, empirically, and later on the basis of the molecular theory of the high resilience of polymers, that experimental curves obtained at various temperatures can be matched by simple parallel shifting. A similar study was performed by Tobolsky et al.[2]

Arveson,[3] followed by Brunstrum et al.,[4] investigating the rheological properties of lubricants, proved by exclusively empirical methods, that in the materials investigated by them, the relative viscosity versus shear rate curves match well if shifted parallel along the shear rate axis. Relative viscosity is to be understood here as the ratio of the lubricant viscosity in the experiment, to the viscosity of the dispersion media at the same temperature.

A number of papers[5] give results of investigations on the possibility of presenting concentration-independent viscosity characteristics of polymer solutions, as well as empirically determined reduced coordinates, in which the relation of the solution viscosities versus shear rate can be given divorced from the polymer concentration in the system.

Philippoff and Gaskins,[6] investigating the flow of polymer melts, concluded that experimental data cannot be confined to a single curve if a wide range of shear rates (up to 6 or 7 decimal orders) is concerned. In a narrow variation range of this factor, however (within 2 or 2.5 decimal orders), Ito[7] who investigated melted polyamides, as well as Schott and Kaghan[8] in their research on polyethylene flow, succeeded in matching their viscosity–shear rate curves obtained at different temperatures. Ito used an exclusively empirical method. Schott and Kaghan, attempting to set up a theoretical basis for their method, assumed that the effective viscosity is inversely proportional to the square shear rate (at adequately high shear rates), which, however, seems quite doubtful.

Thus, the now available data on temperature-independent characteristics of the viscosity of polymer systems appear to be rather incidental, and in some cases baseless.

A theory is suggested in this paper for establishing temperature-independent viscosity characteristics of polymer systems. The possibility of such a representation is confirmed by an analytical review of various earlier published data on the flow of high polymers. The method developed in this paper is similar to some extent to that employed by Ferry.[1]

Theory

The steady viscosity of polymer systems depends on the applied stresses (or on the shear rates unambiguously related to them at a given temperature) as well as on the relaxation spectrum, characteristic of the viscoelastic materials concerned. It can be assumed that

$$\eta = \eta_{\max} f(\theta, D) \tag{1}$$

where η is the viscosity of the polymer system, D is the steady shear rate, and θ is the apparent relaxation time expressed in units of time and depending on the entire set of relaxation times of the investigated polymer system, i.e.,

$$\theta = \varphi(\theta_1, \theta_2, \ldots, \theta_n) \tag{2}$$

In the simplest event, a single characteristic relaxation time can be substituted for the entire spectrum. In eq. (1), $\eta_{\max}$ denotes the initial Newtonian viscosity of the system at the given temperature, or

$$\eta_{\max} = \lim_{D \to 0} \eta$$

and

$$\lim_{D \to 0} f(\theta,D) = 1$$

The following should be pointed out regarding the functions established above.

The function $f(\theta,D)$ is dimensionless, since it is simply the ratio of two viscosities $\eta/\eta_{\max}$, and its arguments have mutually inverse dimensions. This can occur if and only if $f(\theta,D)$ is a function of a product of its arguments, i.e.,

$$\eta/\eta_{\max} = f(\theta,D) = f(\theta,D) \tag{3}$$

The function $\varphi(\theta_i)$ is a homogeneous function of the first order. This is likewise the result of dimensional effects, since the dimensions of all arguments θ_i are the same as that of the function θ itself. Thus, multiplying all arguments θ_i by some coefficient means but a change in the θ_i scale and should give no other result than a change of the scale in which function θ is expressed. Hence,

$$\varphi(A\theta_i) = A\varphi(\theta_i) \tag{4}$$

Let us now consider the molecular theories concerning the temperature relations of those properties of polymer systems which interest us.

As far back as 1948 and 1949, Kargin and Slonimskiĭ[9] suggested a model for the mechanical behavior of polymers which most satisfactorily illustrated the main characteristic features of relaxation in the deformation of amorphous, linear polymers. Later, Rouse[10] studied the relationships between the internal structure of polymers, the temperature, and the characteristic relaxation spectrum. It can be concluded from these molecular theories, in particular, that the ith relaxation time can be represented by the formula

$$\theta_i = a_i \frac{\eta_{\max}(T)}{\rho(T)\,T} \tag{5}$$

where ρ is density.

The coefficient a_i, used in the theories of both studies,[9,10] is expressed in different ways. This is not the subject of the present discussion, but one fact is important, namely, that in neither of these theories does the value of a_i depend on the temperature. The relaxation spectrum appears in the said theories[9,10] as independent of the shear rate D; hence, a_i does not depend on it either. Actually, however, this is not always the case. If θ_i is a function of D, this function can be taken into account for a general consideration, assuming that the influence of the shear rate is contained in the coefficient a_i. The exact form of this factor is of no interest for the theory presented herein, only its independence of the temperature being essential.

It can be seen from eq. (5) that the relation between the *i*th relaxation time at a given temperature and at an arbitrarily selected reference temperature (all values pertaining to this temperature being henceforth marked with primes), can be expressed as follows:

$$\frac{\theta_i}{\theta_i'} = \left[\frac{\eta_{\max}(T)}{\eta'_{\max}(T)}\right]\left[\frac{\rho' T'}{\rho T}\right] \tag{6}$$

Let us now substitute the values of θ_i obtained from eq. (6) into eq. (4) resulting from the homogeneity of the function $\varphi(\theta_i)$. We obtain then a formula for reducing the relaxation spectrum at any temperature T to the reference temperature T':

$$\varphi(\theta_i) = \varphi'(\theta_i)\left[\frac{\eta_{\max}(T)}{\eta'_{\max}(T)}\right]\left[\frac{\rho' T'}{\rho T}\right] \tag{7}$$

or

$$\theta = \theta'\left[\frac{\eta_{\max}(T)}{\eta'_{\max}(T)}\right]\left[\frac{\rho'(T) T'}{\rho(T) T}\right]$$

Introducing the expression (7) into the general viscosity formula (2) we get the following relation:

$$\frac{\eta}{\eta_{\max}} = f\left\{D\eta_{\max}(T)\left[\frac{\rho' T'}{\rho T}\right]\left[\frac{\theta'}{\eta'_{\max}(T)}\right]\right\} \tag{8}$$

For a selected reference temperature the value of $\theta'/\eta'_{\max}(T)$ in eq. (8) will be constant; hence the ratio $\eta/\eta_{\max}$ may be regarded as a function of the type

$$\eta/\eta_{\max} = f(D\eta_{\max}\rho' T'/\rho T) \tag{9}$$

Thus, the complicated argument in the initial relation (1) can be replaced by another argument consisting only of variables subject to direct experimental measurement. If we denote the ratio $\eta/\eta_{\max}$ by η_r (reduced viscosity, dimensionless), and the argument of function (9) by D_r (reduced shear rate, but in dimensions of stress), the result will be $\eta_r = f(D_r)$. This is a single-valued function, which should not vary with the temperature.

Thus, the theory set forth herein is based on the main assumption that the *i*th relaxation time can be represented as a product of two functions, one of which is independent of the temperature and the other expressed as $\eta_{\max}/\rho T$, as follows from some earlier publications.[9,10] Hence, the above basic assumption is close to that used by Ferry[1] in considering the temperature relation of the dynamical characteristics of the viscoelastic properties of elastomers, Ferry having supposed that any relaxation time varies with the temperature in a similar manner.

Experimental investigations of the relation between viscosity and shear rate are often conducted over a very wide range of shear rates. Logarithmic graphs are therefore usually employed to present the experimental results. It becomes evident than that the relation between log η_r and log D_r may be treated as a temperature-independent characteristic. If the selected reference temperature T' is between the melting or softening temperature on the one hand and perceptible incipient polymer decomposition on the other, the value of $\rho'T'/\rho T$ will differ but slightly from 1 (by not more than 6–8% for most commercial polymers), i.e., the value of log $(\rho'T'/\rho T)$ will be within ±0.03. When the viscosity measurements are made over a shear-rate range covering several decimal orders (usually from 3 to 7 or 8), the above correction is negligible. And, where the value of $\rho'T'/\rho T$ can be neglected, the reduced shear rate can be calculated by the simplified formula:

$$D_r = D\eta_{\max}(T)$$

or

$$\log D_r = \log D + \log \eta_{\max}$$

This means that the choice of reference temperature is immaterial, and no temperature–density correction need be made when setting up temperature-independent viscosity characteristics.

The plotting method used for temperature-independent viscosity characteristics of condensed polymer systems may be applied to polymer solutions and gels as well. If the relation between the viscosity (η_0) of a solvent (or a dispersion media) and the temperature satisfies the condition

$$\frac{\partial(\eta_0/\eta_{\max})}{\partial T} = 0$$

the temperature independence of the function

$$\eta/\eta_0 = f(Dk_T)$$

will be ensured at once. The symbol k_T denotes here the experimentally determined temperature factor[3,4] of parallel shift of the flow curves in logarithmic coordinates. The condition

$$\frac{\partial(\eta_0/\eta_{\max})}{\partial T} = 0$$

means also that $k_T = \eta_0$. As observed experimentally by Brunstrum et al.,[4] the shift factor k_T equals η_0 only approximately. Consequently, the value of $\eta_0/\eta_{\max}$ varies somewhat with the temperature. For this reason, the temperature-independent viscosity characteristics of polymer solutions or gels may be represented by a graph in logarithmic coordinates obtained by shifting the relative viscosity versus shear rate curves for various temperatures along the shear-rate axis by

$$k_T = \eta_0(\eta_{\max}/\eta_0) = \eta_{\max}$$

and along the relative viscosity axis by $\eta_0/\eta_{\max}$. This was not taken into consideration in previous studies.[3,4]

Experimental Data on the Temperature Relation of the Viscosity of Condensed Polymers

In order to check the above theory and method of temperature-independent presentation of viscosity characteristics of polymer systems, special calculations were performed based on the most reliable experimental reports published on the flow curves of various condensed polymers.

Philippoff and Gaskins[6] published data on the viscosity of two polyethylenes and plasticized polyvinyl butyral. These figures deserve special attention, as the experiments were here carried out in a very wide shear-rate range and with a high degree of reliability. Flow curves were obtained within wide temperature limits, and initial Newtonian viscosities were directly measured. It should, nevertheless, be noted that the measurement results were widely scattered around the flow curves. Figure 1 illustrates the temperature-independent viscosity curve of polyethylene (sample II of the original reference[6]); experiments with the said material were conducted within a maximum temperature range of about 140°C.). Similar data on plasticized polyvinyl butyral are shown in Figure 2.

The temperature-independent viscosity curve of polyethylene, obtained from Dexter's experimental results[11] can be seen in Figure 3. A particular feature of Dexter's experiments is that the initial Newtonian viscosity region was not reached at any of the temperatures. The problem of determining $\eta_{\max}$ will be discussed later.

The possibility of constructing the temperature-independent viscosity characteristic of a typical elastomer is a point of particular interest. To check this, the experimental data of Treloar[12] on the flow properties of plasticized natural rubber have been plotted in log D_r versus log η_r coordinates in Figure 4. No initial Newtonian viscosity values were given in this paper either.

The method of presenting temperature-independent viscosity characteristics may be of use in the following cases.

If experimental determinations of apparent viscosity are conducted over a wide range of shear rates and temperatures, plotting the temperature-independent viscosity curve makes it possible to check the compatability of the experimental results.

Where experiments cover a wide temperature range but remain within a limited range of shear rates (due to limitations of the apparatus used, for instance), application of the temperature-independent presentation method enables evaluation of viscosity values at shear rates, which were not reached in the experiments.

Finally, there is no longer any need for direct viscosity measurements at all temperatures if data are required on the viscosity changes with the

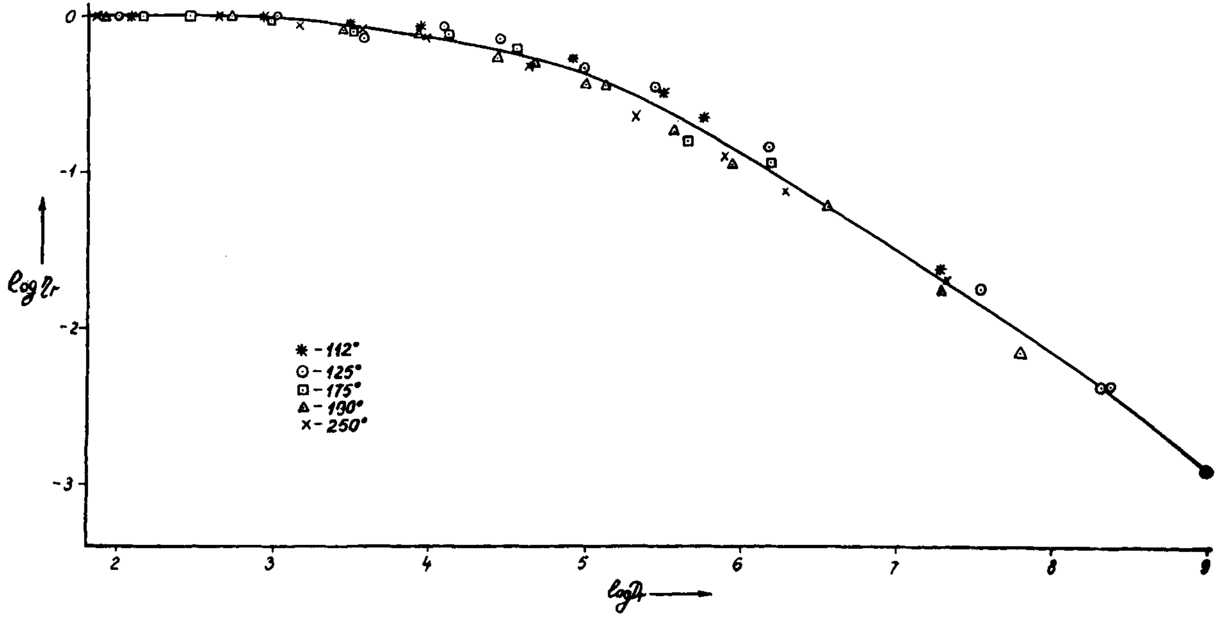

Fig. 1. Temperature-independent viscosity characteristic of polyethylene (based on data of Phillippoff and Gaskins[6]).

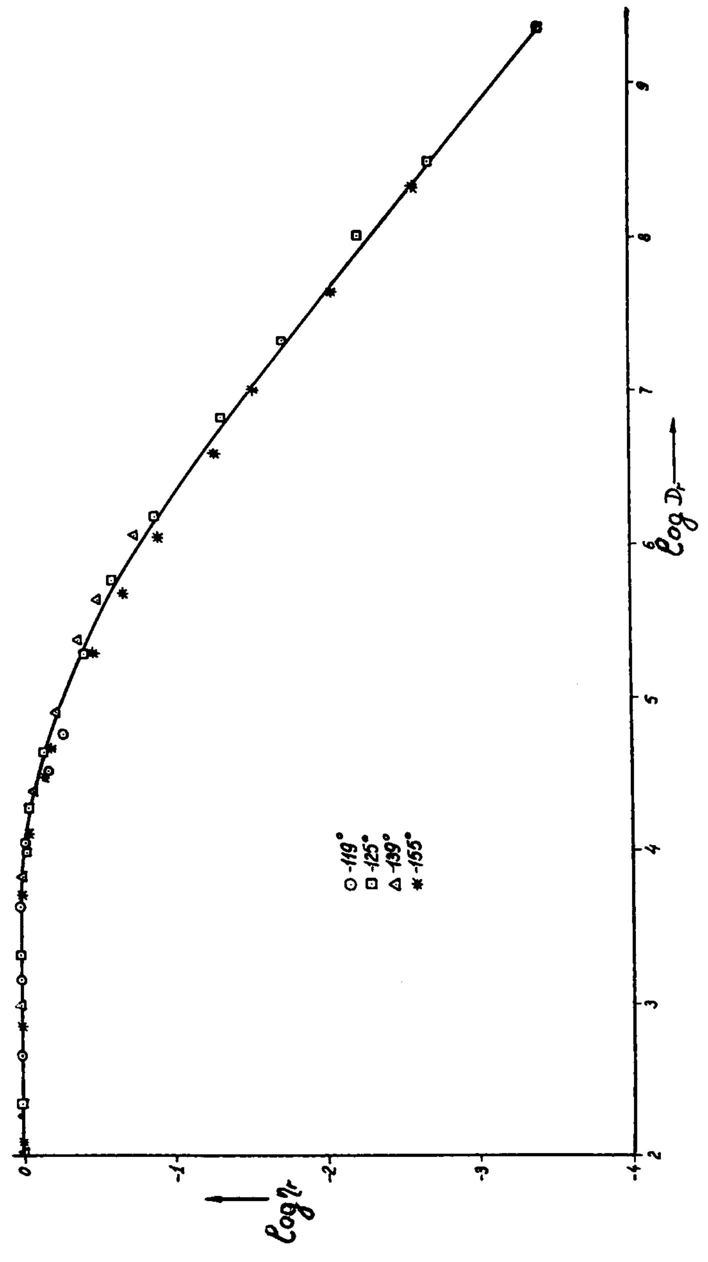

Fig. 2. Temperature-independent viscosity characteristic of plasticized polyvinyl butyral (based on data of Phillippoff and Gaskins[8]).

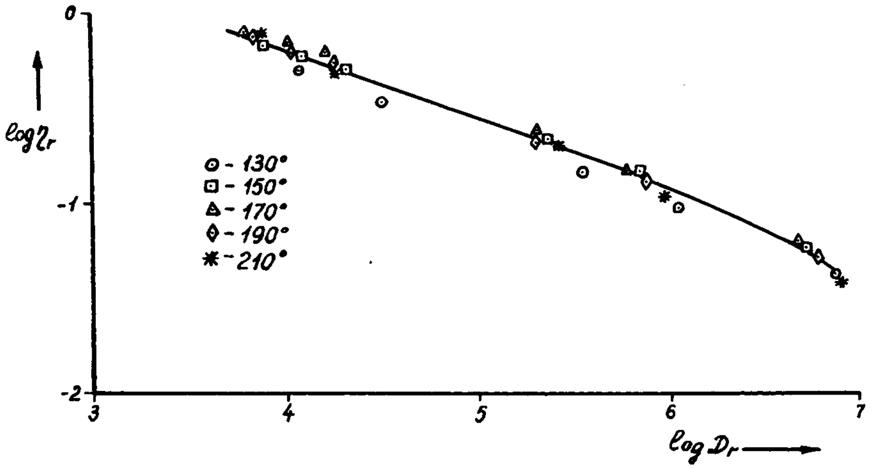

Fig. 3. Temperature-independent viscosity characteristic of polyethylene (based on data of Dexter[11]).

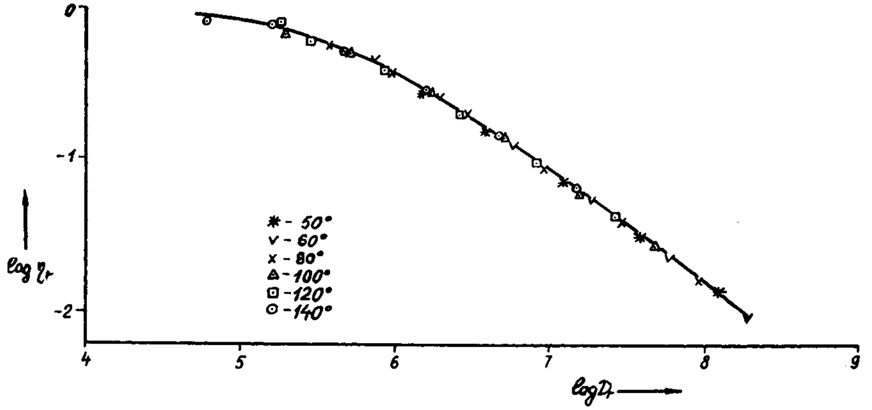

Fig. 4. Temperature-independent viscosity characteristic of plasticized natural rubber (based on data of Treloar[12]).

shear rate at various temperatures. For this purpose it is enough to obtain a flow curve, covering as wide a shear rate range as possible at any single temperature, preferably sufficiently remote from the melting point (for crystalline polymers), and to determine the initial Newtonian viscosities after which their variation with the temperature may be obtained with an adequate degree of reliability. The actual viscosities at various temperatures and shear rates can then be evaluated with the help of the temperature-independent viscosity characteristic.

Determining Initial Newtonian Viscosities

Initial Newtonian viscosities are of particular importance in the temperature-independent viscosity presentation method discussed. It is well

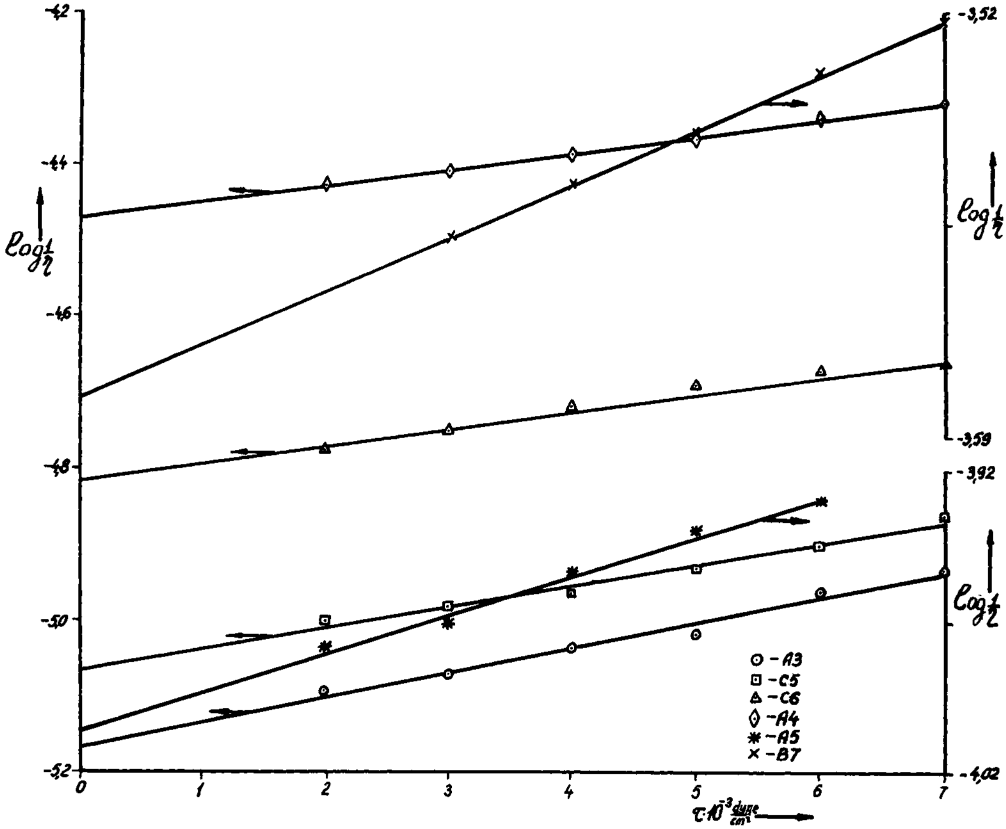

Fig. 5. Determination of initial Newtonian viscosity of polyethylenes (based on data of Tung[14]).

known that this parameter appears generally as a most essential physical characteristic of the material concerned, closely related to the molecular weight and the features of internal structure of high-polymer molecules. In experimental work, however, it is not always possible to reach the shear rate regions of Newtonian flow of condensed polymers. For this reason a dependable method of extrapolating experimental data on viscosity versus shear rate relations to zero shear rates (or, which is the same, to shear stress values tending to zero $\tau \rightarrow 0$) should be regarded as a matter of particular importance. Spencer and Dillon[13] suggested plotting log $1/\eta$ against τ and extrapolating to $\tau = 0$. The value of $\eta_{\max}$ can then easily be computed from the length of the intercept on the ordinate axis. Such a method was applied by the authors mentioned for the case of polystyrene; it was then observed that the relation between log $1/\eta$ and τ is linear at sufficiently low shear stresses, a feature which confirmed the feasibility of the above extrapolation.

We checked this method on the basis of the most trustworthy reports published so far. The extrapolated $\eta_{\max}$ values were compared with

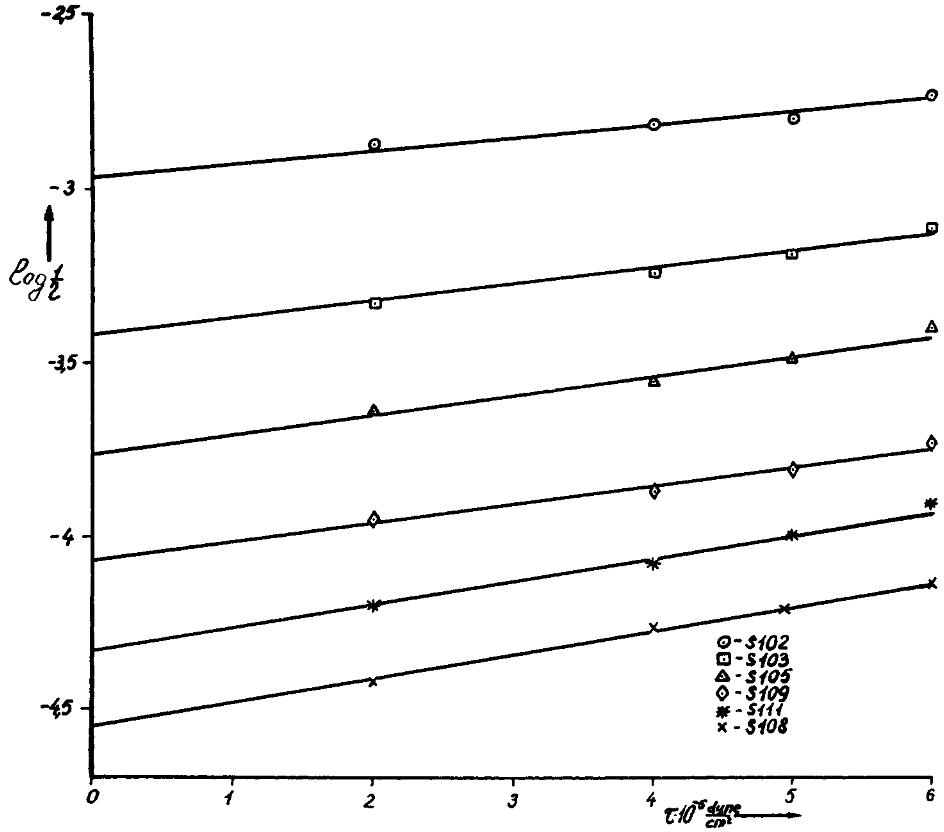

Fig. 6. Determination of initial Newtonian viscosity of polystyrenes (based on data of Rudd[15]).

directly observed experimental data. For this purpose a log $1/\eta$ versus τ coordinate system was used to represent Tung's[14] and Rudd's[15] results (see Figs. 5 and 6, respectively), both having conducted most exact measurements at low shear rates, and having reached the initial Newtonian viscosity regions. Tung made his investigations with narrow fractions

TABLE I
Initial Newtonian Viscosity Values of Polyethylenes

	log $\eta_{\max}$	
Polyethylene sample no.	Data of Tung[14]	From Fig. 5
A3	5.10	5.17
C5	5.01	5.06
C6	4.78	4.82
A4	4.44	4.46
A5	3.98	4.01
B7	3.58	3.58

TABLE II
Initial Newtonian Viscosity Values of Polystyrenes

Polystyrene sample no.	log η_{max} Data of Rudd[15]	log η_{max} From Fig. 6
S102	2.97	2.97
S103	3.40	3.42
S105	3.74	3.76
S109	4.08	4.07
S111	4.28	4.33
S108	4.52	4.55

of various polyethylenes, and Rudd with polystyrene. The sample symbols used in Figures 5 and 6, as well as in Tables I and II, are the same as in the original references.[14, 15]

The extrapolated log η_{max} values are compared with those found by experiment, in Tables I and II. The same method was used to determine

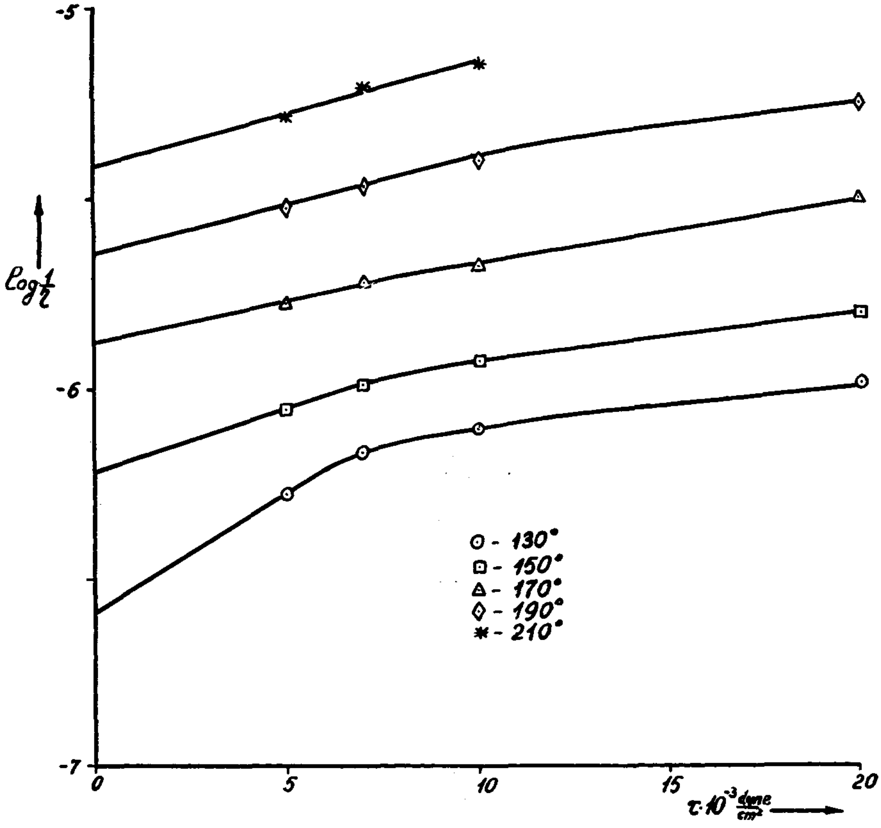

Fig. 7. Determination of initial Newtonian viscosity of polyethylene (based on data of Dexter[11]).

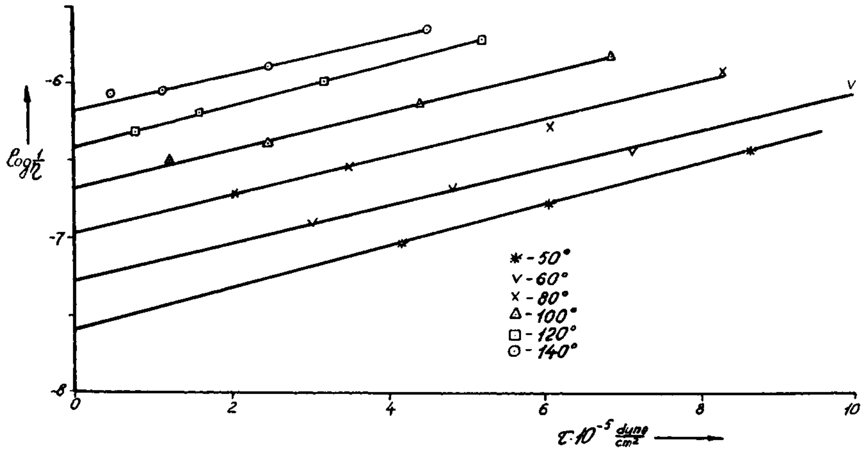

Fig. 8. Determination of initial Newtonian viscosity of plasticized natural rubber (based on data of Treloar[12]).

the initial Newtonian viscosities ($\eta_{\max}$) required for constructing the temperature-independent viscosity characteristics of polyethylene from Dexter's experimental data (see Fig. 7) and of plasticized natural rubber from Saunders and Treloar's report[12] in Figure 8.

Discussion

A detailed treatment of experimental results obtained by various investigators proved that temperature-independent viscosity characteristics can be obtained successfully even for polymer systems of widely differing chemical nature and structure. Consequently, the steady apparent viscosity can actually be represented as the product of two functions, one of which ($\eta_{\max}$) depends on the temperature but not on the shear rate, and the other, $f(D_r)$, depends on the shear rate but not on the temperature. For polystyrene, in particular, this fact was proved by experiment.[13]

The temperature relation of $\eta_{\max}$ appears similar to the temperature coefficient of shift in Ferry's investigations,[1] usually denoted by a_T. The widely used formula for computation of $a_T(T)$, known as the WLF formula, however, was found unsuitable for expressing the relation between the initial Newtonian viscosity ($\eta_{\max}$) and the temperature over a sufficiently wide temperature range.

The problem of the temperature relation of $\eta_{\max}$ is one of the most essential problems concerning the flow properties of polymers. At present, viscosity–temperature relations are usually expressed by Fraenkel-Eyring's exponential law. According to this, the relation between log $\eta_{\max}$ and $1/T$ should graphically be represented by a straight line whose slope is claimed to determine the activation energy of viscous flow. Figure 9 illustrates the relation between log $\eta_{\max}$ and $1/T$ for a number of polymer systems. It can be seen that only the curves plotted from Philippoff-

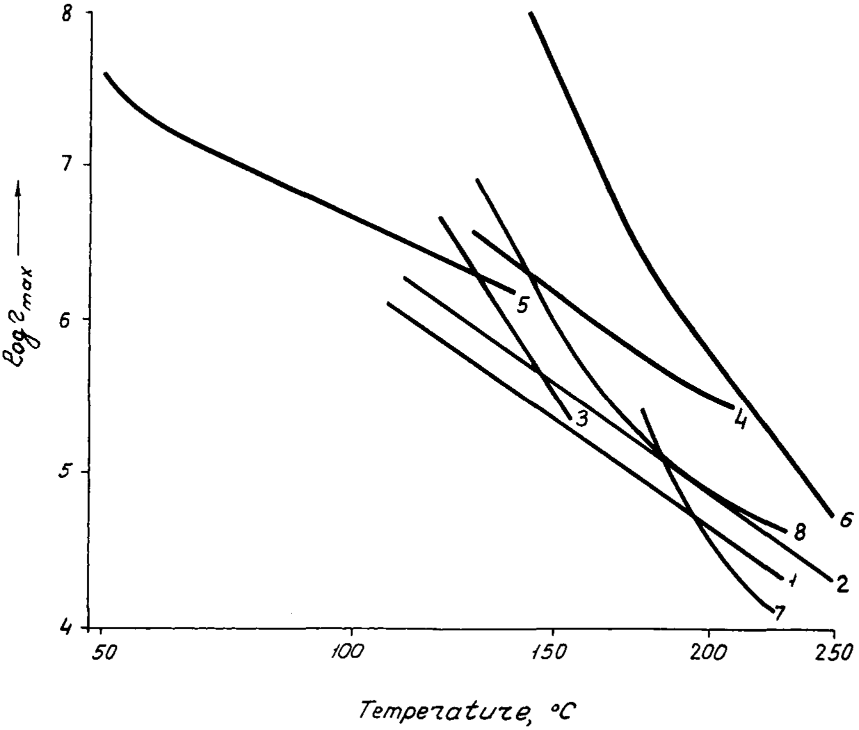

Fig. 9. Temperature relation of initial Newtonian viscosity for various polymer systems: (*1*) polyethylene I;[6] (*2*) polyethylene II;[6] (*3*) polyvinyl butyral;[6] (*4*) polyethylene;[11] (*5*) plasticized natural rubber;[12] (*6*) polystyrene, molecular weight 360,000;[13] (*7*) polystyrene, molecular weight 162,000;[13] (*8*) alkathene 2.

Gaskins' data[6] are actually straight lines. In all the other examples a noticeable curvature can be observed. Kobeko,[16] who examined viscosity–temperature relations for numerous products including most diverse ones, pointed out that whenever measurements covered an adequately wide temperature range, the log $\eta = F(1/T)$ graphs had a noticeable curvature. Hence, expressing the relation between $\eta_{\max}$ and T in exponent form and determining the viscous flow activation energy by the slope of the log $\eta_{\max} = F(1/T)$ curve would not be exact. Shishkin[17] supposed that the activation energy itself varies exponentially with the temperature. This means that the probability of "jumps" of molecular-kinetical units increases at rising temperatures not only as a result of their own elevated energy, but also due to a lowering of potential barriers to be overcome in the elementary kinetic act. This assumption permits a satisfactory interpretation of the experimentally observed viscosity–temperature relations of various materials. The same is true for the relation between $\eta_{\max}$ and T.

The $\eta_{\max}$ extrapolation method suggested by Spenser and Dillon[15] and checked above is substantially an application of a formula proposed earlier by Gurevich[17] representing the relation between apparent viscosity and shear stress:

$$\eta = \eta_{\max} e^{-a\tau} \tag{10}$$

where $\eta_{\max}$ and a are constants.

Since the relation between log η and τ can actually be represented in rectilinear form, it may be assumed that eq. (10) will be true for sufficiently low shear stresses. Detailed calculations, which confirmed the correctness of eq. (10) at low τ values, lead to the following important practical conclusion: experimental investigations of the relation between apparent viscosity and shear stress may be justifiably extended over low stress regions only until the test results plotted in log $1/\eta$–τ coordinates begin to fall positively onto a straight line. Further decreasing of τ would be impractical.

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Résumé

On a étudié la possibilité de construire une courbe caractéristique de la viscosité de systèmes polymères indépendante de la température avec utilisation du procédé des "paramètres réduits" analogue à celui de Ferry. On a établi que la relation entre la viscosité en la température ainsi que la vitesse de déplacement peut être représentée dans le cas général sous la forme d'un produit de deux fonctions dont l'une ne dépend que de la température et l'autre ne dépend que de la vitesse de déplacement. En partant des procédés de l'analyse dimensionnelle et des notions du caractère viscoélastique de la déformation des systèmes polymères on a montré que le rapport de la viscosité apparente et de la viscosité newtonienne maximum (initiale) est une fonction indépendante de la température du produit de la vitesse de déplacement par la viscosité newtonienne maximum (initiale). Il s'ensuit que la viscosité newtonienne maximum (déterminée à la vitesse de déplacement tendant vers zéro) joue le role d'un paramètre physico-chimique essentiel, caractérisant les propriétés visqueses des systèmes polymères. Dans le domaine des vitesses de déplacement suffisamment basses, la viscosité apparente est une fonction exponentielle des contraintes de cisaillement ce qui permet de trouver par extrapolation la viscosité newtonienne maximum (initiale). Cette théorie a été vérifiée sur les données publiées les plus sûres relatives à la viscosité des systèmes polymères condensés, dans de larges plages de vitesses de déplacement et des températures. Les données expérimentales sur des systèmes polymères de nature différente coincident d'une facon satisfaisante avec les courbes caractéristiques connexes, indépendantes de la température.

Zusammenfassung

Die untersuchte Möglichkeit, die temperatur-invariante Charakteristik der Zähigkeit von Polymersystemen unter Verwendung des Verfahrens der "reduzierten Parameter" zu konstruieren, ist analog der Methode von Ferry. Es wurde festgestellt, dass die Abhängigkeit der Viskosität von der Temperatur und der Verformingsgeschwindigkeit im allgemeinen als das Produkt von zwei Funktionen dargestellt werden kann. Hierbei hängt eine Funktion nur von der Temperatur, die andere nur von der Verformungsgeschwindigkeit ab. Anhand der Dimensionsanalysen-Methode und den Vorstellungen über den elastisch-zähen Charakter der Polymersystem-Verformungen wurde gezeigt, dass das Verhältnis der scheinbaren (effektiven) Viskosität zur maximalen Newton'schen Viskosität gleich der temperatur-invarianten Funktion des Produktes der Verformungsgeschwindigkeit und der maximalen Newton'schen Viskosität ist. Deshalb ist letztere (die bei einer sich Null nähernden Verformungsgeschwindigkeit bestimmt wurde) ein wichtiger physikalisch-chemischer Parameter, der die Viskositätseigenschaften von Polymersystemen charakterisiert. Die scheinbare Viskosität hängt im Bereich genügend kleiner Verformungsgeschwindigkeiten von der Schubspannung ab. Dadurch ist es möglich, durch Extrapolation die maximale Newton'sche Viskosität zu finden. Die Theorie wurde anhand der zuverlässigsten in der Literatur angeführten Daten in bezug auf die Viskositätseigenschaften kondensierter Polymersysteme in weiten Verformungsgeschwindigkeits- und Temperaturbereichen überprüft. Die Versuchsergebnisse ihrer Eigenart nach verschiedener Polymersystem können mit genügender Genauigkeit in Form von temperatur-invarianten Kurven dargestellt werden.

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