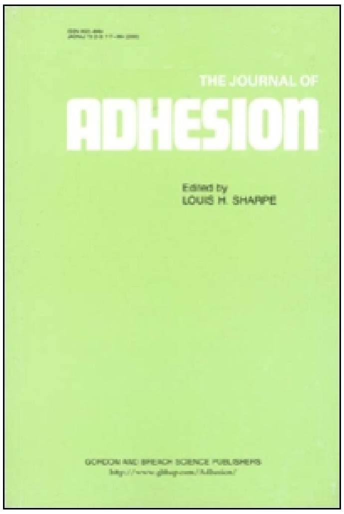

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# Shear Properties of Thin Polymeric Films

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# Shear Properties of Thin Polymeric Films†

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When a thin solid organic coating is interposed between two contacting and sliding surfaces we may define a strength property $\tau$ as the frictional force per unit area of solid–solid contact. This paper reviews recent work on the influence of contact pressure, temperature and sliding velocity on $\tau$ for a range of high molecular weight organic polymers and lower molecular weight organic solids. It is shown that the shear properties of these thin films resemble those of the corresponding bulk polymers if allowance is made for the high degree of molecular orientation produced in the film during sliding.

## INTRODUCTION

It is a great privilege to contribute a paper on the shear properties of thin polymeric films to this Symposium at which the Borden Foundation Award will be presented to Dr. Zisman. Those of us who work in this field recognize the important and seminal contributions that Dr. Zisman has made in this area.

Earlier work on the shear properties of thin films by Hardy and his collaborators,[1] Bowden and Tabor[2] and Bridgman and his associates[3] was mainly concerned with long chain fatty acids and similar amphipathic molecules, used in the investigation of boundary lubrication. It was Zisman and his colleagues who were amongst the first (*c.* 1953) to study the shear properties of other materials which might be applied as "thin dry films to reduce friction and wear between sliding solids".[4] Their first paper in 1968 dealt with paraffin wax, gold and molybdenum disulphide[5] and later papers dealt more specifically with various organic materials.[16]

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† Presented at the Borden Award Symposium honoring William A. Zisman during the National Meeting of the American Chemical Society in New Orleans, LA, March 21–25, 1977.

In this paper we review work largely carried out in our own laboratory during the last 5 years on the shear properties of thin organic films trapped between hard substrates. The materials range from stearic acid and calcium stearate to polythene, polystyrene, PMMA and other polymers. In particular we shall consider the influence of contact pressure, temperature and sliding speed on the quantity $\tau$ which we define as the frictional force per unit area of contact. The results show that this strength property resembles the shear strength of the material in bulk if allowance is made for the high degree of molecular orientation developed in the film during sliding and to the mode of shear involved.

## THE EFFECT OF CONTACT PRESSURE

If a smooth sphere of radius $R$ is loaded against a smooth plate upon which is deposited a thin film of organic material then to a good approximation the area of contact, $A$, is given by[8]

$$A = \pi(KWR)^{\frac{2}{3}} \qquad (1)$$

where $W$ is the normal load and $K$ is an elastic constant determined by the elastic properties of the sphere and the plate. In the experiments to be described the sphere and the plate are generally of glass which provides a good smooth and highly elastic surface. The mean pressure in the contact, $P$, is $W/A$. If a force, $F$, parallel to the plate, is required to maintain a sliding velocity, $V$, then the frictional force per unit contact area, $\tau$, is $F/A$.

Figure 1 shows $\tau$ against $P$, plotted logarithmically, for a range of low molecular materials including stearic acid, various stearates, and paraffin wax. We note that in spite of the rather different chemistry and experimental method the data fall broadly on the same line, and that $\tau$ increases markedly with pressure. Higher molecular weight polymers show a similar trend but the values of $\tau$ are generally displaced to high values for a given value of pressure. It is however of more interest to plot $\tau$ against $P$ on linear ordinates as is done in Figure 2 where we see that $\tau$ is a linear and increasing function of $P$[16-18];

$$\tau = \tau_0 + \alpha P \qquad (2)$$

where $\tau_0$ and $\alpha$ are constants. This expression appears to be quite general for thin solid films and few exceptions have been noted.[16] $\tau_0$ is normally a function of temperature to a first approximation, while $\alpha$ is not.† Table I lists values of $\tau_0$ and $\alpha$ for a range of organic materials.

It is seen that $\alpha$ lies between 0·05 and 0·8: these values are close to those obtained from the pressure dependence of the shear flow stress of the corresponding bulk material.[20] On the other hand the values of $\tau_0$ lie between

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† There are cases where $\alpha$ is a marked function of both temperature and sliding velocity. This occurs when viscoelastic retardation in compression is important. See later.

one tenth and one hundredth of the isotropic bulk shear strength of the film material at atmospheric pressure.[19, 20]

Because of this large discrepancy it is relevant to ask if the quantity $\tau$ is a true measure of the shear strength of the film material or a measure of some other property, for example, the shear strength of the *interface* between the solid and the film material. The evidence indicates that $\tau$ does not depend on the nature of the hard solid provided it is hard and smooth. Secondly, with thin films of metals the quantity $\tau$ agrees well with the bulk values. Thirdly, with polymers which are not readily oriented, the discrepancy between $\tau$ and the bulk shear strength is small. Fourthly, if bulk polymers are heavily drawn and coupons are cut out in various orientations for subsequent testing, the modulus and the shear strength may vary by a factor of 10 or more being smallest along the direction of chain orientation and largest at right angles to this direction.[21] Although these observations do not provide an unequivocal answer they suggest that, in most cases, $\tau$ is a true measure of the shear strength of the film material: the low value compared with the bulk material is apparently due to the high degree of orientation induced in the film by the sliding process itself.

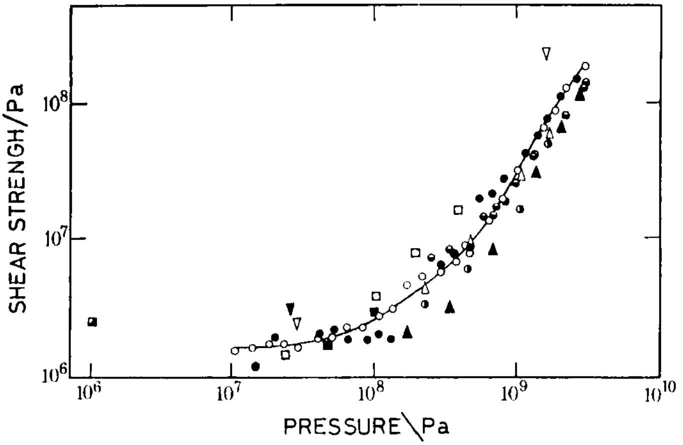

FIGURE 1 Pressure dependence of the shear strength of certain stearates and stearic acid at 20°C. Sliding speeds of the order of 2–200 $\mu$m $s^{-1}$. ○, calcium stearate in the form of Langmuir–Blodgett layers, see Table I, 1(i),† Table Legend (6); ●, copper stearate as a thick layer, Table I 1(ii) (6); ◪, calcium stearate–stearic acid mixed film sheared between mica, Table I, 3(9); ▽, stearic acid, deposited from solution, Table I, 1(ii) (15); ▼, bulk stearic acid sliding on platinum (14); △, stearic acid as a thin film between metal plates, Table I, 4 (12); △, ferric stearate and ◑ sodium stearate sheared between plates, Table I, 4 (13); ⊖, paraffin wax sheared between glass surfaces, Table I, 1 (ii) (5); ■, calcium stearate sheared between mica, Table I, 3 (10); and □, stearic acid sheared between mica (10) and (11). For more complete description see Ref. 19.

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† See Legend to Table I.

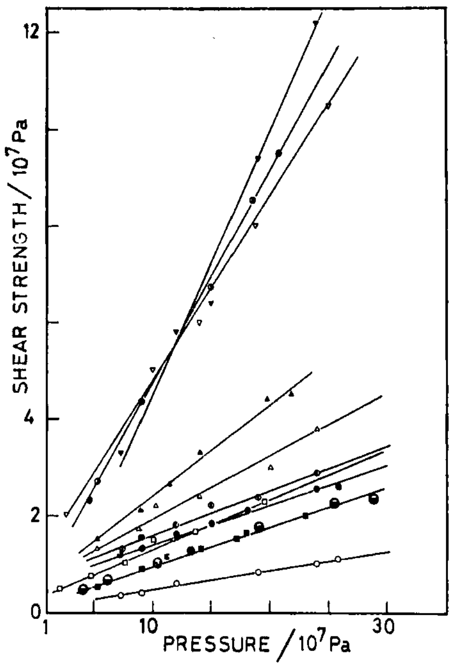

FIGURE 2 Shear strength, $\tau$, against mean calculated pressure $P$, at 20°C for ■, high density polythene (pure); ○, high density polythene (filled with 5% CuO, 30% $Pb_3O_4$ 2/2); □, PTFE; △, low density polythene; ▲, polypropylene; ▽, polymethylmethacrylate; ▼, polyvinylchloride; ◑, polystyrene; ○,●, oleamide, stearamide; and ◒, calcium stearate. Sliding speed approximately 0·05 mm $s^{-1}$. $\tau$ is a linear function of pressure. Data taken from Ref. (6) and (24). Substrates were glass and the sliding configuration that of a sphere on a flat.

We may now return to Eq. (1) and use it to predict the frictional force and coefficient of friction, $\mu$, of polymeric contacts as well as those contacts which consist of a thin film being sheared between hard substrates.[5, 24] For example the coefficient of friction, $\mu$, of such contacts is.[25]

$$\mu = \frac{A\tau}{W} = \frac{F}{W} = \frac{1}{P}(\tau_0 + \alpha P)$$

$$= \frac{\tau_0}{P} + \alpha \tag{3}$$

where for a thin film $P$ is defined either by an extension of Eq. (1) or by the flow stress, $P_0$, of the substrates. For a bulk polymer sliding on a smooth clean flat or on another piece of polymer $P_0$ is the appropriate flow stress for the polymer. The limiting value of $\mu$ at high loads is $\alpha$ when $P > \tau_0$. This is approached for most polymer layers when $P$ is greater than $10^9$ Pa;

# TABLE I

"Shear strength" $\tau$, pressure coefficient $\alpha$, and activation energy $Q$, for organic layers sheared between hard solids

| | | | Above Tg or Tm (b) | | | Below Tg or Tm (b) | | |
|---|---|---|---|---|---|---|---|---|
| Material | Method (a) | Ref. | $10^{-7}\tau_0$ /Pa | $\alpha$ | $-Q$ /kJ mol$^{-1}$ | $10^{-7}\tau_0$ /Pa | $\alpha$ | $-Q$ /kJ mol$^{-1}$ |
| St Ac | 1(i) | 6 | — | — | — | 0·15 | 0·07 | 16 |
| St Ac | 2 | 10, 11 | — | — | — | | | 16 or 30 |
| Ca St | 1(i) | 6 | — | — | — | 0·10 | 0·08 | 16 |
| Ca St | 2 | 11 | — | — | — | — | — | — |
| PTFE | 1(iii) | 6, 23 | 0·10 | 0·08 | 20 | — | — | — |
| PTFE | 4 | 18 | 0·23 | — | — | — | — | — |
| PTFE | 4 | 18 | −0·3 | 0·051 | — | — | — | — |
| HDPE | 1(ii) | 6, 23 | 0·25 | 0·10 | 16 | — | — | — |
| HDPE | 5 | 20, 22 | 1·40 | 0·034 | — | — | — | — |
| PP | 1(ii) | 19 | 0·50 | 0·17 | 17 | — | — | — |
| LDPE | 1(ii) | 19 | 0·60 | 0·14 | 9–30 | — | — | — |
| PS | 1(ii) | 19 | 1·40 | 0·17 | 26 | 0·40 | 0·45 | $\sim 0$ |
| PVC | 1(ii) | 19 | 0·45 | 0·18 | 18 | −0·90 | 0·57 | $\sim 0$ |
| PMMA | 1(ii) | 6, 23<br>19 | 0·80 | 0·10 | 14 | 1·0 | 0·24 | $\sim 0$ |
| PMMA | 5 | 20, 22 | — | — | — | 5·03 | 0·20 | — |
| PEMA | 1(ii) | 24 | — | — | 14 | 2·0 | 0·77 | $\sim 0$ |
| PBMA | 1(ii) | 24 | — | — | 10 | 1·0 | 0·95 | $\sim 0$ |
| PNMA | 1(ii) | 24 | 2·5 | 0·72 | 9·0 | — | — | — |
| PCSMA | 1(ii) | 24 | 1·2 | 0·39 | 9·0 | — | — | — |

(a) Experimental method:

1. Sliding experiment where a thin film is sheared between smooth glass substrate. Sliding speeds of the order of a few tenths of a millimeter per second.
   - (i) Film thickness: a few monolayers
   - (ii) Film thickness: *c.* 100 nm essentially unoriented
   - (iii) Film thickness: *c.* 10 nm highly oriented.
2. Sliding experiment where thin layers are sheared between glass and metal substrates. Sliding speed a few tenths of a millimeter per second.
3. Sliding experiment where a few monolayers are sheared between mica surface. Sliding speed *c.* 3 $\mu$m s$^{-1}$. For stearic acid two values of $Q$ are tentatively observed. With breakdown of the Langmuir–Blodgett structure, $Q = 16$ kJ mol$^{-1}$. If the structure is retained $Q \simeq 30$ kJ mol$^{-1}$.
4. Anvil experiment after Bridgman. The values of $\tau_0$ and $\alpha$ are generally for 20°C except where values "above Tg" are quoted for polymers whose Tg is above 20°C and here the values refer to a temperature about 40°C above Tg. The values of $Q$ are for a mean pressure of about $10^8$ Pa.
5. Bulk shear tests under hydrostatic pressure after Ward *et al.* (for more details see Ref. 22).

(b) Tg is the glass transition temperature. Tm is the melting temperature. PTFE shows a pressure induced phase change at 20°C at about $1{\cdot}8 \times 10^8$ Pa.

*Key*:

| | | | |
|---|---|---|---|
| St Ac | Stearic acid | Ca St | Calcium stearate |
| PTFE | Polytetrafluoroethylene | HDPE | High density polythene |
| PP | Polypropylene | LDPE | Low density polythene |
| PS | Polystyrene | PVC | Polyvinyl chloride |
| PMMA | Polymethylmethacrylate | PEMA | Polyethylmethacrylate |
| PBMA | Polybutylmethacrylate | PNMA | Polynonylmethacrylate |
| PCSMA | Polycetylstearylmethacrylate | | |

about the flow stress of gold and copper. Certainly we know that for any contact, $\mu$ cannot be less than $\alpha$. It is interesting to take Eq. (3) and the values of $\tau_0$ and $\alpha$ (obtained from experiments where a thin film of polymer is "sheared" between a sphere and flat) from Table I, and compare these with the values obtained directly when the same polymer is slid over itself or on a clean glass surface.[25] We take $P$ as being $P_0$ where $P_0 \simeq H_v$, and $H_v$ is the Vickers pyramidal hardness of the polymer.† This comparison is made in Table II, where it can be seen that the predicted values of $\mu$ are close to those obtained by direct measurement. This is good confirmation of the "adhesion" model of friction (2).

TABLE II

Comparison between coefficient of friction deduced from thin film experiments and those directly measured

| Polymer | $P_0$ (Pa) | $\tau_0$ (Pa) | $\alpha$ | $\tau_0/P_0+\alpha$ | $\mu_b$ | $\mu_g$ |
|---|---|---|---|---|---|---|
| PTFE | $3{\cdot}18\times10^7$ | $1{\cdot}0\times10^6$ | 0·08 | 0·11 | 0·16 | 0·13 |
| HDPE | $5{\cdot}46\times10^7$ | $2{\cdot}5\times10^6$ | 0·10 | 0·15 | 0·15 | 0·08 |
| LDPE | $2{\cdot}14\times10^7$ | $6{\cdot}0\times10^6$ | 0·14 | 0·42 | 0·52 | 0·42 |
| PP | $6{\cdot}65\times10^7$ | $5{\cdot}0\times10^6$ | 0·17 | 0·25 | 0·26 | 0·26 |
| PMMA | $3{\cdot}75\times10^8$ | $1{\cdot}0\times10^7$ | 0·36 | 0·39 | 0·36 | 0·41 |
| PS | $1{\cdot}95\times10^8$ | $4{\cdot}0\times10^6$ | 0·45 | 0·47 | 0·42 | 0·45 |

$\mu_g$: coefficient of friction obtained when the bulk polymer is slid over a flat glass substrate; $\mu_b$: coefficient of friction obtained when the bulk polymer is slid on itself.

For most polymers $\tau_0$ is a strong function of temperature and sliding velocity. $\alpha$ is generally a monotonic function of these variables. We deal with the effects of temperature in the next section.

## THE EFFECT OF TEMPERATURE

Organic films show two characteristic variations of $\tau$ with temperature; either $\tau$ is independent of temperature or it decreases, sometimes markedly, as the temperature is raised. When $\tau$ decreases with temperature it is generally found that to a good approximation

$$\tau = \tau_0' \exp\{-Q/RT\} + \alpha P \tag{4}$$

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† The contact pressure, $P_0$, under these conditions will be of the same order as the indentation hardness, $H_V$, of the polymer though there are reasons for supposing that it might be somewhat less. In Ref. (25) $P_0 \approx H_V/1{\cdot}4$. For simplicity we shall take $P_0$ as equal to $H_V$.

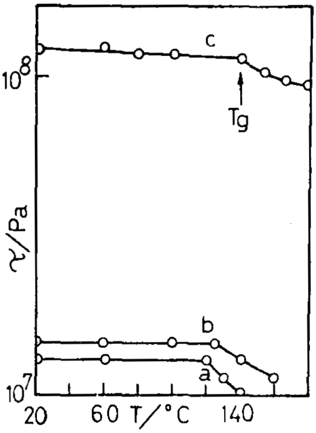

FIGURE 3 Shear strength, $\tau$, as a function of temperature for polymethylmethacrylate at contact pressures of $a = 6{\cdot}30 \times 10^7$ Pa, $b = 7{\cdot}2 \times 10^7$ Pa, and $c = 7{\cdot}2 \times 10^8$ Pa. Sliding speed 0·03 $\text{mms}^{-1}$. The glass transition temperature, Tg, is indicated. Below Tg $\tau$ is not a marked function of temperature. The value of Tg increases slowly with increasing contact pressure. Data taken from Ref. 26. Technique as for Figure 2.

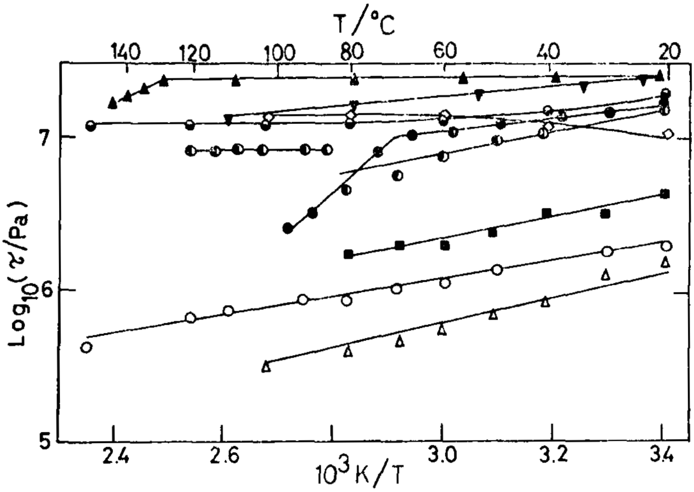

FIGURE 4 Temperature dependence of the shear strength plotted according to Eq. (2) as $\log_{10}(\tau/\text{Pa})$ against the reciprocal of temperature. Mean contact pressure $c$. $6{\cdot}0 \times 10^7$ Pa and sliding speed 0·2 $\text{mms}^{-1}$. Data, △, PTFE; ○, calcium stearate; □, stearic acid; ◐, high density polythene; ○, low density polythene; ⊖, anthracene; ◇, sebacic acid; ▲, polymethylmethacrylate; and ▼, polycetostearylmethacrylate. Sliding members were a glass flat and a glass sphere.

In this relation $\alpha$ is apparently not a function of temperature. Films of PMMA show both types of behaviour; Figure 3.[26] Below its Tg the shear strength is independent of temperature while above the Tg Eq. (4) holds and $Q$ is *c.* 14 k J mol$^{-1}$. Figure 4 shows data for other polymeric films and without exception Eq. (4) appears to provide an accurate description of the data. $Q$ is either zero ($\tau \neq f(T)$) or in the range 10–30 k J mol$^{-1}$. Table I lists some values of $Q$ for various polymers. It is found experimentally that $Q$ is not usually a function of pressure or sliding velocity. The form of Eq. (4) and the magnitude of $Q$ will be discussed later. We note also that the values of $Q$ correspond quite closely to those found for the temperature dependence of the shear yield stress of bulk polymers.[7]

## THE EFFECT OF SLIDING VELOCITY

It is found, under isothermal conditions, that the effect of changing the sliding velocity is of two kinds for a fixed contact geometry. First we have the effect on increasing the strain rate in the films and this leads to an increase in $\tau$. The rate of strain in the film is approximately $V/h$ where $h$ is the film thickness. Following the conventional approach for bulk polymer deformation we may write[27]

$$\tau = \tau_0'' \ln\left(\frac{V}{h} \cdot \frac{1}{\phi}\right) \quad \text{(at constant } P, T\text{)} \tag{5}$$

where $\phi$ is a "characteristic" frequency and $\tau_0''$ is a constant. Conceptually it seems sensible to assume that this factor only operates on $\tau_0'$ of Eq. (4) although there is evidence[27] which suggests that this may be an over-simplification.

The second effect of increasing $V$ is to reduce the mean contact time (approximately $V/d$, where $d$ is the diameter of the contact area) during which a given element of the films suffers compression. This is termed visco-elastic retardation in compression.[28, 29] It means simply that the film does not have adequate time to respond to the application of the normal stress; this leads to a reduction of $\alpha$ as $V$ is increased. Intuitively we suppose that this effect modifies $\alpha$ (Eq. 2) only and that[17]

$$\alpha = \alpha_0 \exp\left(-\frac{V}{d} \cdot \frac{1}{\theta}\right) \quad \text{(constant } T\text{)} \tag{6}$$

where $\theta$ is another "characteristic" frequency. $\phi$ and $\theta$ differ in that the former corresponds to a high strain, high strain rate process while the latter to a lower strain, lower strain rate process. Both frequencies are functions of temperature and may be thought of as weighted mean values over all the relaxation processes occurring in the polymer.

We may combine Eqs. (4), (5) and (6) and produce a general empirical expression

$$\tau = \tau_0''' \ln\left(\frac{V}{h}\cdot\frac{1}{\phi}\right)\exp\left(-\frac{Q}{RT}\right)+\alpha_0\exp\left(-\frac{V}{d}\frac{1}{\theta}\right)P \tag{7}$$

where $\tau_0''$ is a constant. Typical data for the influence of sliding velocity on the shear strength of films is shown in Figure 5. Most polymers show an increase in $\tau$ with increasing velocity but there is one exception: PMMA. Thus for most polymers the strain rate in shear appears to be the dominant effect, and where comparisons can be made the value of $\phi$ (Eq. (5)) is similar to that found in bulk shear yield experiments.[19, 22] The case of PMMA is of special interest and recent experiments[30] have demonstrated that viscoelastic retardation in compression is an important factor in controlling $\tau$ for this material at temperatures below its glass transition temperature.

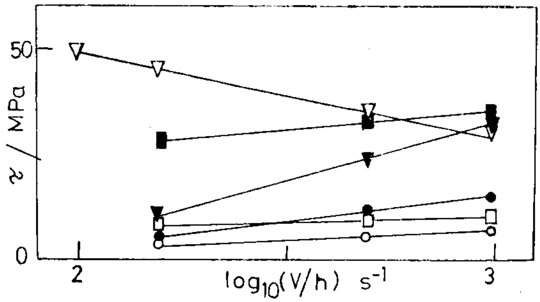

FIGURE 5 The shear strength of polymer films plotted as a function of the logarithm (to the base ten) of the estimated strain rates. The film thicknesses have been taken as 2000 Å. ○, PTFE (contact pressure, $P = 6{\cdot}7\times10^7$ Pa; temperature, $T = 25$;C); ●, PP ($P = 7{\cdot}2\times10$ Pa; $T = 25$°C); □, HDPE ($P = 7{\cdot}2\times10$ Pa; $T = 25$°C); ■, PS ($P = 7{\cdot}2\times10$ Pa; $T = 26$°C); ▽, PMMA ($P = 7{\cdot}2\times10$ Pa; $T = 30$°C); ▼, PMMA ($P = 7{\cdot}2\times10$ Pa; $T = 200$°C).

## GENERAL FEATURES OF THE SHEAR BEHAVIOR OF THIN ORGANIC FILMS

The response of thin films, as manifested by changes in $\tau$, to changes in contact pressure, temperature, sliding velocity (or contact time) is accurately described by Eq. (7) with the properly chosen material constants, $\tau_0'''$, $Q$, $\alpha_0$, $\phi$, and $\theta$. The geometry of the contact and the normal load defines the pressure and the contact diameter (and hence the contact time). The temperature in the contact is made up of a contribution from the bulk temperature $T'$ and a part due to adiabatic frictional heating in the film. In the experiments described previously simple calculations suggest that the latter contribution is small; that is $T' = T$.

The form of Eq. (7) and the magnitude of the material constants indicates that these thin films, many only a few hundreds of Ångstroms thick, behave in a way which closely resembles that of the corresponding bulk polymer during yield or fracture. The interfacial shear strengths are however generally a factor of between ten and one hundred less than the isotropic bulk strengths of the polymers. This reduction in strength is probably due to the extensive reorientation of the molecular structure in the interfacial layers as well as to the existence of a well defined interfacial shear plane during sliding.

The significance of the material constants $Q$, $\alpha$, $\phi$ and $\theta$ may be described in terms of a stress aided thermally activated rate process as for many rheological systems including bulk polymer flow.[24, 27] These constants may then be given some molecular significance. While this approach has definite short-comings it is possible to account for the changes in $\alpha$ for the homologous series of poly(n-alkyl)methacrylates in terms of the relative sizes of the pendent n-alkyl side groups.[24] $Q$ reflects the temperature dependence of the mobility of these segments.

## CONCLUSION

During the past ten years we have seen an increase in our understanding of the way in which energy is dissipated in thin organic layers when they are sheared in concentrated contacts. Little has so far been done to apply this approach to practical problems where such factors as frictional heating, surface topography and lubricant failure are additional and important considerations. Nevertheless we can now predict with some certainty $\tau$, the energy dissipated per unit area per unit sliding distance, for most well defined model contacts.

## Acknowledgements

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