

Sliding wear, viscoelasticity, and brittleness of polymers

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(Received 11 March 2006; accepted 23 June 2006)

We have connected viscoelastic recovery (healing) in sliding wear to free volume in polymers by using pressure-volume-temperature (P-V-T) results and the Hartmann equation of state. A linear relationship was found for all polymers studied with a wide variety of chemical structures, except for polystyrene (PS). Examination of the effect of the indenter force level applied in sliding wear on the healing shows that recovery is practically independent of that level. Strain hardening in sliding wear was observed for all materials except PS, the exception attributed to brittleness. Therefore, we have formulated a quantitative definition of brittleness in terms of elongation at break and storage modulus. Further, we provide a formula relating the brittleness to sliding wear recovery; the formula is obeyed with high accuracy by all materials including PS. High recovery values correspond to low brittleness, and vice versa. Our definition of brittleness can be used as a design criterion for choosing polymers for specific applications.

I. INTRODUCTION

In his already classic book on friction and wear of materials, Rabinowicz¹ talks about the 1966 Jost report to the British government, which evaluated losses due to ignorance of tribology at M£ 515/per year. He says: “At the time the Jost Report appeared it was widely felt that the Report greatly exaggerated the savings that might result from improved tribological expertise. It has now become clear that, on the contrary, the Jost Report greatly underestimated the financial importance of tribology. The report paid little attention to wear, which happens to be (from an economical point of view) the most significant tribological phenomenon.”

For many reasons there is an ongoing process of replacement of metal parts by polymeric ones. More mileage per gallon in cars and airplanes due to lighter weight, for example, is one of the desired effects. In the case of metal parts and components, a simple—and fairly successful—procedure to mitigate wear consists in using external lubricants.¹ However, because polymers often swell in contact with liquid lubricants, the approach used for metals cannot be simply transferred. We have shown

before² that carbon black results in lower friction in polymer blends, irradiated as well as un-irradiated ones. However, in various applications lack of transparency and black color are excluded.

The gradual replacement of metals by polymers or materials with polymer matrices (PBMs, polymer-based materials) has not been accompanied by a sufficient growth in understanding of their tribology. PBMs include heterogeneous composites (such as with glass macrofibers) and nanohybrids (with carbon or ceramic nanofibers). Although new techniques for creation of a wide range of PBMs are being developed,³ literature on the tribology of such materials is limited. Rabinowicz discusses polymers in his book at only two locations.¹ In a collective book on tribology the treatment of PBMs consists essentially in tabulation of some friction values.⁴ A 500-page book by Mathieu, Bergmann, and Gras (MBG)⁵ discusses various kinds of polymers available for coatings and several coating deposition procedures. The procedures seem to deserve further improvement; MBG say soberly: with dexterity, bubble formation can be avoided. Nanohybrids—such as those containing carbon nanofibers^{6–8}—seem much more promising in this respect.

The situation is similar in books that focus on PBMs rather than on tribology. Mechanics of polymers has been covered well in the book by Goldman⁹ and also discussed

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DOI: 10.1557/JMR.2006.0300

in a collective book edited by Mark¹⁰; the latter also covers nicely thermophysical properties. Lucas, Soares, and Monteiro (LSM)¹¹ provide a thorough book on characterization of polymers. There exist in the literature sophisticated treatments on optimization of fiber-reinforced PBMs^{12–14} as well as on using sensors to damp vibrations in composites.¹⁵ However, words “friction” and “wear” do not appear in the subject index of the Goldman, Mark, or LSM books, while the excellent papers on mechanics hardly venture into tribology. The small number worldwide of groups active in polymer tribology with good results^{6–8} (see also a review of the field¹⁶) does not change the overall situation in a decisive way.

There are reasons why the tribology of PBMs is not more advanced. It is a difficult area. For metals and ceramics time is not a variable at all, but PBMs are viscoelastic: time dependence of properties plays a primordial role. Moreover, polymers have often complicated multiphase structures. Goldman⁹ says: “the situation is made considerably more complex by the existence of multiple domains and subdomains in which the mechanical behavior of the polymer varies.”

Clearly tribological properties are affected in a similar way as mechanical ones.

Given the need for more understanding of PBM tribology, and the existence of strong PBM mechanics, we have decided to connect the two areas. Since macroscopic properties of any kind depend on structures and interactions at the molecular level, connections between different properties should exist. As an example, in our earlier paper² we have shown that the concentration of the carbon black additive at which the static friction drops rapidly is the same at which the electric resistivity drops rapidly. We describe below some results obtained by connecting macroscopic properties of different kinds.

II. EXPERIMENTAL PROCEDURES

A. Materials

Materials were selected to represent several classes of polymers with different chemical structures and different mechanical properties. The polymers examined were: polystyrene (PS, Aldrich Chemicals Company), polycarbonate (PC, Dow Chemical Company), acrylonitrile/butadiene/styrene copolymer (ABS, Dow Chemical Company), polytetrafluoroethylene (PTFE or Teflon®, Dow Chemical Company), isotactic polypropylene (PP, Huntsman), low-density polyethylene (LDPE, Huntsman), polyethersulfone (PES, Solvay Engineered Plastics), Santoprene™ (a copolymer of PP and ethylene-propylene-diene-monomer, Advanced Elastomer Systems), styrene/acrylonitrile copolymer (SAN or Luran®, BASF, Ludwigshafen/Rhein), Surlyn® 8140 [a copolymer of ethylene and methacrylic acid (MAA), E.I. du Pont de Nemours, Wilmington, DE].

B. Sliding wear

Sliding wear tests were performed on a micro scratch tester (MST) (CSEM Instruments, Neufchatel, Switzerland) in multiple scratch mode. The instrument and procedure have been described previously.^{17–19} The test consists of 15 scratches by a diamond tip along the same groove. Each material was tested under constant loads of 5.0, 10.0, and 15.0 N at room temperature (25 °C). Scratch speed was 5.0 mm/min, and the total scratch length was 5.0 mm. Values for penetration depth R_p and residual depth R_h were determined from the midpoint (2.5 mm along the scratch length). The percentage of viscoelastic recovery f was calculated from R_p and R_h for the 15th scratch using the definition formulated in¹⁹:

$$f = [(R_p - R_h) \cdot 100\%]/R_p \quad (1)$$

Note that Eq. (1) applies to single scratch resistance tests as well as to sliding wear.

C. Storage and loss modulae

Dynamic mechanical analysis (DMA) to determine storage E' and loss E'' modulae was performed^{20,21} using the DMA7e apparatus (Perkin Elmer Co.). Specimens of rectangular form (20.0 × 6.0 × 3.0 mm nominal dimensions) were analyzed by three-point bending in temperature scan mode at the frequency of 1.0 Hz. Results recorded at 25 °C are used.

D. Elongation at break

Values for elongation at break ϵ_B were obtained from the MatWeb Online Materials Database (www.matweb.com). Averages were taken in cases of ranges of values.

III. SLIDING WEAR AND RECOVERY

Tribology deals with contacts between moving interfaces; energy dissipation accompanies the contacts. We find convenient for our purposes to consider contacts between approximately flat interfaces (static and dynamic friction) separately from those contacts in which one interface is used to probe the resistance to deformation and wear of the other one. The latter category involves the use of indenters or abrasers.

In the past 5 years or so we have repeatedly found useful micro scratch resistance determined with a diamond indenter. One measures first the instantaneous penetration depth R_p . While viscoelasticity of PBMs is a complication, it is also a blessing. Healing takes place, the bottom of the scratch groove goes up, and the residual or final depth R_h is typically much shallower than R_p . This was the basis of the definition¹⁹ provided as Eq. (1) in Sec. II. B.

Scratch resistance and sliding wear determination can be performed under a force linearly increasing with time

t, increasing stepwise, or maintaining a constant load. The last mode requires more experimentation but provides a more comprehensive set of results.

Do higher forces applied in single scratching or in sliding wear tests cause more permanent damage to the material and thus lower recovery? We have investigated a set of polymers belonging to different classes from the point of view of their chemical structures, including engineering thermoplastics and elastomers (see Sec. II. A.). The resulting values of *f* as a function of the force *F* are shown in Fig. 1.

Figure 1 tells us that the dependence of the recovery on the force applied can be neglected. Necessarily, higher forces result in higher values of *R_p* and *R_h*. However, we see here something akin to the Le Chatelier-Brown principle in action: the more a physical system is attacked by an external force, the more it resists the change. For most purposes, our viscoelastic scratch recovery turns out to be a material property independent of the regime imposed by the experimentalist.

IV. RECOVERY IN WEAR AND FREE VOLUME

Since mechanical properties of PBMs change with time, for instance as a result of creep,^{9,10,14,22,23} reliable methods of prediction of long-term service performance on the basis of short-term tests have been developed.^{9,10,22-27} These methods rely largely on the concept of free volume *v^f*. At higher temperatures (higher *v^f*) one observes at much shorter times creep and other property changes which at lower temperatures (lower *v^f*) would take years and decades (the time-temperature correspondence). The free volume is defined as

$$v^f = v - v^* \quad (2)$$

where all volumes are typically in cm³·g⁻¹, *v* is the total specific volume, and *v** is the incompressible (hard core)

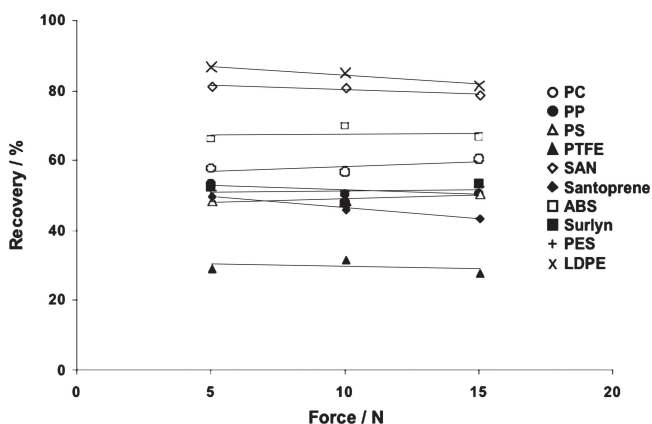


FIG. 1. Viscoelastic recovery *f* in sliding wear (after 15 scratch tests) calculated from Eq. (1) as a function of the applied force *F* (in Newtons) for representatives of different classes of polymers.

volume.²⁸ The use of Eq. (2) relies on an equation of state of the form *v* = *v*(*T*, *P*), where *T* is the thermodynamic temperature and *P* is pressure. We have found that for all kinds of PBMs, including polymer liquid crystals (PLCs), which form a variety of phases, reliable predictions over decades of years are obtained^{22,23,25-27} when one uses the Hartmann equation of state²⁹

$$\tilde{P}\tilde{v}^5 = \tilde{T}^{3/2} - \ln \tilde{v} \quad (3)$$

The use of reduced parameters such as featured in Eq. (3) goes all the way back to Johannes D. van der Waals. These parameters are defined as

$$\tilde{v} = v/v^*; \quad \tilde{T} = T/T^*; \quad \tilde{P} = P/P^* \quad (4)$$

The hard-core or reducing parameters *v**, *T**, and *P** enhance the comparability of different materials.^{28,29}

Fairly extensive *v*(*T*, *P*) results have been determined and reported by Zoller and Walsh.³⁰ We have applied Eq. (3) to these data and obtained *v**, *T**, and *P** for our polymers. Then we have used Eq. (2) to obtain free volume values for our polymers.

It is reasonable to expect that more free volume should result in more scratch or sliding wear recovery defined by Eq. (1). In Fig. 2 we present percentage recovery (an average of several forces for sliding wear determined by scratching each groove 15 times) as a function of free volume. Again results for important representatives of several classes of polymers are shown (for those for which equation of state data were available).³⁰

Figure 2 confirms our expectations: more free volume has as a result more viscoelastic recovery. The results form a single curve (except for polystyrene which is an outlier). If one excludes polystyrene, a linear regression fits the data very well. We shall address below the issue of the singular behavior of PS.

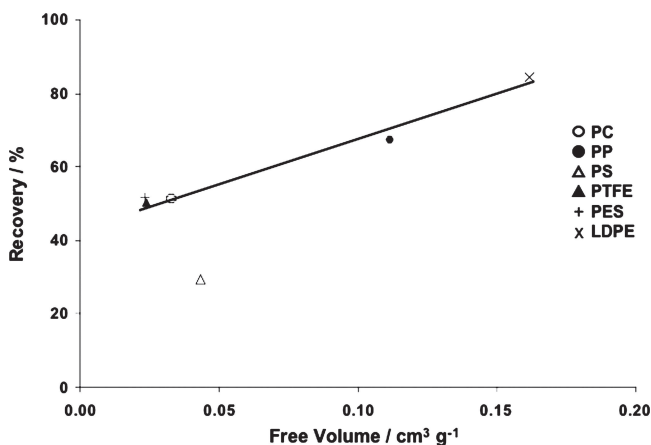


FIG. 2. Percentage recovery determined in sliding wear tests for representatives of different classes of polymers. The average viscoelastic recovery *f* is plotted as a function of the free volume *v^f* of the corresponding material. The correlation line is for a linear least squares regression of all points excluding that for PS (*R*² value = 0.9779).

V. STRAIN HARDENING AND BRITTLINESS

In 2004 we discovered strain hardening in sliding wear determined by repetitive scratching.¹⁷ We studied then three polymers: polypropylene, Teflon, and a polyester. After, say, 10 scratch tests (this number depends on the force level) the residual depth remains the same. That is, further passages of the indenter do not have any effects. The result is particularly interesting for Teflon, a polymer notorious for its poor scratch and wear resistance.

Since the original work,¹⁷ we have investigated more materials, also at varying sliding speeds.^{31,32} All polymers studied so far exhibit strain hardening in sliding wear—except for polystyrene that is an outlier also here, as seen in Fig. 3.

How do we explain the results for polystyrene seen in Figs. 2 and 3?

We recall now a detailed study by Karger-Kocsis and coworkers³³ of polyamide 12 containing blends with copolymers containing styrene units. As argued by them, PS has the advantage of easy melt processability. However, PS does not have good mechanical properties; malleation of the styrene-containing copolymers is needed to achieve them.

A qualitative explanation of our and other results is easy: PS is notorious for its brittleness. This brings about the obvious next question: How can we quantify brittleness?

Since the main feature of PBMs is their viscoelasticity, we characterize the viscoelastic behavior of our materials by dynamic mechanical analysis (DMA).^{20,21}

One imposes a sinusoidal stress σ at frequency ν as a function of time t :

$$\sigma(t) = \sigma_0 \sin(2\pi\nu t) \quad (5)$$

This causes the following behavior of the strain ϵ :

$$\epsilon(t) = \epsilon_0 \sin(2\pi\nu t - \delta) \quad (6)$$

where δ is a measure of the lag between solid-like (elastic) and liquid-like (viscous flow) response of the material. The results are typically presented in the form of the complex modulus:

$$E^* = E' + iE'' \quad (7)$$

Here $i = (-1)^{1/2}$; E' is the storage modulus, which represents the solid-like response while the loss modulus E'' corresponds to the liquid-like response.

With these results at our disposal, consider how they can be used in our quest for a measure of brittleness. Clearly the extent of liquid-like behavior represented by E'' is not related to brittleness. By contrast, E' seems a useful candidate; a material with high storage modulus will not be brittle.

One can consider brittleness as contrary to ductility. This does “not” help us; there is more than one measure of ductility in use. One can alternatively consider brittleness as contrary to toughness. A widely used measure of toughness is based on integration of stress versus strain curves obtained in quasi-static tensile testing. The resulting value represents the energy absorbed at break (failure). There is a lesson for us here; not only the resistance to deformation (which can be represented by E') but also the strain at break ϵ_b is important. A material with high strain at break will not be brittle, so an inverse relationship between the brittleness and ϵ_b seems appropriate.

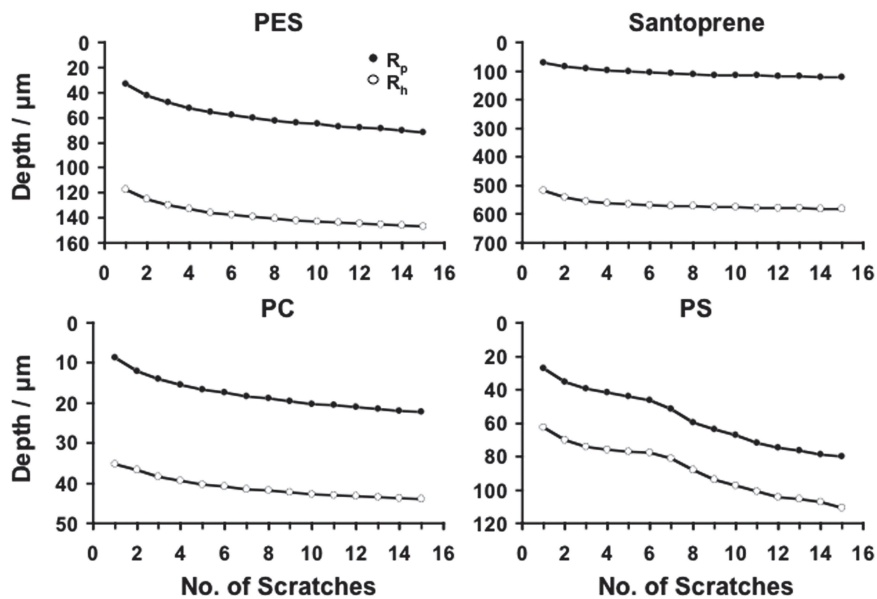


FIG. 3. Penetration depth R_p and residual depth R_h values as a function of the number of scratching tests performed in sliding wear determination. Strain hardening observed for all materials, also for different force level, except for polystyrene. Plots are for PES at 22.5 N, Santoprene at 15 N, PC at 5 N, and PS at 15 N.

Reasoning along these lines leads us to the following definition of brittleness:

$$B = 1/(\epsilon_b \cdot E') \quad (8)$$

In accordance with our approach of taking advantage of results obtained by different techniques, the first term in the denominator comes from quasi-static tensile testing and the second from dynamic mechanical testing.

We now verify whether the definition (8) does the job it should. Connecting to Fig. 2, we show in Fig. 4 brittleness B as a function of the recovery f [again Eq. (1)] for the same representative set of different classes of polymers. The line in the figure corresponds to the following equation:

$$B = b_1 e^{-f/b_2} \quad (9)$$

On the l.h.s. we actually use $10^{10}B/(\% \cdot \text{Pa})$. A least squares fit gives $b_1 = 422.5$ and $b_2 = 7.644$. The correlation coefficient $R^2 = 0.9941$ shows that Eq. (9) is obeyed within the limits of very high accuracy for *all* materials, with b_1 and b_2 as “universal” constants. b_1 determines the magnitude of B and thus depends on the unit used for the storage modulus. b_2 represents the strength of the coupling between the brittleness and the recovery; a larger b_2 value would result in a smaller effect of f on B . Above all, Eq. (9) tells us that high values of the recovery in sliding wear correspond to low brittleness, and vice versa. This agrees with the intuitive concept of brittleness.

We see in Fig. 4 a dramatic fall of the brittleness between polystyrene and all other materials. One can assume a threshold brittleness B value—below which all polymers show “normal” or non-brittle behavior. The normal behavior is such as seen in Fig. 2 for the recovery as a function of free volume. Also the fact that PS alone

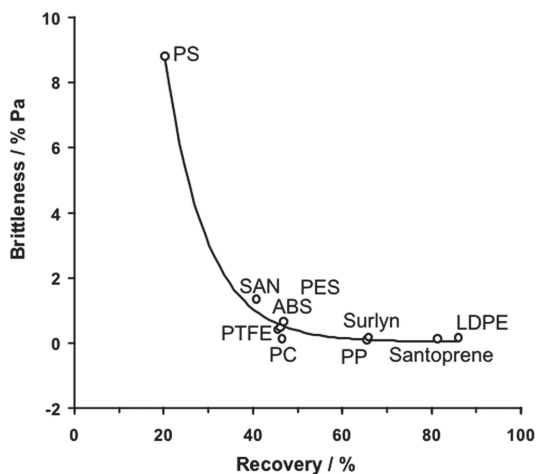


FIG. 4. Brittleness B , defined by Eq. (8), as a function of the viscoelastic recovery f for representatives of several classes of polymers. Circles represent experimental points. The units are $10^{10}B/(\% \cdot \text{Pa})$. The continuous line has been calculated by using Eq. (9).

does not exhibit strain hardening in sliding wear (Fig. 3) now can be explained quantitatively in terms of its strikingly high B value. We can see how this arises from the data presented in Table I.

VI. DISCUSSION

After Eq. (8) was formulated, we were asked how polymers such as poly(phenylene sulfide) (PPS) and poly(ether ether ketone) (PEEK) fit into our definition. We have not found the necessary data for PPS. For PEEK, however, we have found in Chapter 23 of¹⁰ $\epsilon_b = 50\%$; in a tabulation by Sepe³⁴ we find E' (1 Hz, interpolated value for 298 K) = $3.634 \cdot 10^9$ Pa. In the units we use, Eq. (8) gives us $10^{10}B/(\% \cdot \text{Pa}) = 0.055$, a value quite close to that for PP (see again Table I). PPS and PEEK are usually highly crystalline and sometimes are called brittle. Eq. (8) shows that they are not brittle. Our definition is supported by the fact that both PPS and PEEK serve as matrices in high performance thermoplastic composites.

Given the similarity of PP and PEEK found using the definition (8), we have also calculated free volume for PEEK using Eqs. (2) to (4) and the tabulation of P-V-T data by Zoller and Walsh.³⁰ Actually, since $1 \text{ atm} = 0.101 \text{ J} \cdot \text{cm}^{-3} = 0.101 \text{ MPa}$, the l.h.s. term in Eq. (3) is negligible and we simply have:

$$\ln \tilde{v} = \tilde{T}^{3/2} \quad (10)$$

The result is $v^f(298 \text{ K}, 1 \text{ atm}) = 0.115 \text{ cm}^3 \cdot \text{g}^{-1}$. This value for PEEK is close to the v^f value for PP displayed in Fig. 2. PP has the recovery in the sliding wear $f = 67.3\%$. We believe this similarity of v^f values is one more confirmation of our model. Therefore, we predict for PEEK $f \approx 70\%$.

The embrittlement of polymers with “aging” is well known from experience and is reported in the literature.^{35,36} What is not well characterized is what happens

TABLE I. Values of brittleness B expressed as $10^{10}B/(\% \cdot \text{Pa})$ for representatives of several classes of polymers compared with the viscoelastic recovery f .

Polymer	B (% Pa)	$f/\%$	$\epsilon_b/\%$	E'/Pa
PC	0.106	51.4	97.9	9.66E+08
PP	0.056	67.3	120.0	1.48E+09
PS	8.783	29.6	6.9	1.65E+08
PTFE	0.375	50.6	400.0	6.67E+07
SAN	1.316	46.6	4.0	1.90E+09
Santoprene	0.087	80.5	525.0	2.18E+08
ABS	0.443	51.1	27.3	8.26E+08
Surlyn	0.138	67.6	325.0	2.23E+08
PES	0.625	51.7	30.2	5.30E+08
LDPE	0.132	84.5	190.0	4.00E+08

Components of B are elongation (i.e., strain) at break ϵ_b and loss modulus E' according to Eq. (8), $B = 1/(\epsilon_b E')$.

to E' and ϵ_b with aging: if both decrease, then brittleness increases according to Eq. (8); or, if E' increases, then ϵ_b must decrease faster to maintain an increase in brittleness as defined in Eq. (8). Data on E' and ϵ_b for neat polymers are not abundant while more often only one of these parameters is reported for a specific blend or composite. We have found for a chlorosulfonated polyethylene polymer known as Hypalon® and for trilayered Hypalon with Neoprene® that E' increases and ϵ_b decreases with aging.³⁷ After aging 88 days, E' for Hypalon increased an estimated 16% while the average decrease in ϵ_b for samples aged between 1 and 88 days was 20%. For the trilayered Hypalon, the changes for the trilayered Hypalon were a 24% increase in E' accompanied by a 45% average reduction in ϵ_b . In both cases, therefore, elongation at break decreased faster than storage modulus increased, validating our measure of brittleness Eq. (8) in accord with the observation in³⁶ that aging produced embrittlement.

Schwarz and coworkers report values for storage modulus and strain at yield for neat PP after aging.³⁸ If one makes the reasonable assumption that elongation (i.e., strain) at break behaves similarly to strain at yield, then we see from their work that both E' and ϵ_b for PP decrease with aging. Thus, two reports from the literature indicate that our definition of brittleness can be used even to explain the brittleness that occurs with aging. This does not take into account the possibility of crystallization that might occur with aging. Under such conditions, the aged material is not the same as the starting material, and we have not addressed this complication.

As already mentioned, there exists a survey of polymer tribology.¹⁶ It describes the extant knowledge in this field and also points out areas where work is needed. As noted in the beginning, fiber-reinforced plastics constitute an important class of PBMs. The use of fibers is expected to enhance mechanical as well as tribological properties of the base materials. However, Fallon and Eiss Jr. report that neat polyetherimide shows “less” wear than the same polymer reinforced with short carbon fibers.³⁹ This is one more example why a better understanding of tribology of PBMs is needed. That understanding should unite information coming from several sources. Polymeric chains containing polar groups can be oriented in external fields. We have shown how a polymer liquid crystal oriented in a magnetic field shows different values of recovery f in function of the angle between the scratching and the field orientation direction.¹⁸ Other orientable polymers might show similar effects. Another source of information on tribological behavior is molecular-dynamics computer simulations. While scratch resistance testing provides us only with two averages for a given sample, R_p and R_h , simulations provide us with continuous dependence of the scratch depth on time t for every chain segment at the surface.⁴⁰

Werwa⁴¹ describes interviews conducted as a front-end study for the Materials MicroWorld project created by the Materials Research Society (MRS) and funded by the US National Science Foundation (NSF). Both adults and children were interviewed at the Maryland Science Center to discern their knowledge regarding materials science. The term “brittleness” has long been used by laymen even though no definition existed, and in answering questions and describing their impressions about certain materials, the interviewees talked in terms of hardness, toughness, and brittleness.⁴¹ Somewhat similarly, undergraduate students taking their Introduction to Materials Science and Engineering course at North Texas used such terms even before mechanical properties were covered and available definitions discussed.⁴² These students attended the MRS and NSF exhibit Strange Matter (the physical exhibit that resulted from the Materials MicroWorld project) shown in Dallas, and used terms from mechanics relying on their everyday meaning.⁴² Our definition of brittleness seems to agree with the everyday meaning of the word as it has been used before.

VII. CONCLUSION

As noted by Mayer,⁴³ “the designer needs to be aware of what is and is not possible with today’s materials and production techniques.” Mayer’s book is on fiber-containing PBMs, but his statement has a general validity. The close values of B for PP and PEEK, and also of ν^f for PP and PEEK, are interesting from this point of view also. We expect that the definition of brittleness now formulated can be used among others as a design criterion.

ACKNOWLEDGMENTS

One of us (W.B.) acknowledges discussions with the late Prof. Paul J. Flory at Stanford on the importance of free volume. Constructive comments of a referee are appreciated. Terry Glass and his colleagues at the Dow Chemical Co., Freeport, TX, have also provided comments on the manuscript. Partial financial support for this work was given by the Robert A. Welch Foundation, Houston (Grant No. B-1203) and also by a National Defense Science and Engineering Graduate (NDSEG) Fellowship, Washington, DC (to H.E.H.L.).

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