Self-lubricating polymer composites

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Appendices

Appendix 1: Matlab code script for wear calculations

To measure the topography of the wear track, a μSurf Nanofocus confocal microscope is used. Based on the 3D confocal images of the wear track and assuming the polished surface is flat, a Matlab script is used to calculate the wear volume of the composite. From every wear track, four images (north, east, south and west sides) were taken to calculate the average wear volume, with each of 1455×1410 μm size. The wear calculation is explained in Fig. A.1. After defining two shoulders (the red rectangles), the script reconstructs the original surface by interpolating a flat surface between these shoulders. Then, the wear volume is obtained via subtracting the wear track from the interpolated flat surface.

Fig. A.1 An illustration of the method to calculate wear volume via Matlab.

```matlab
% 1st step, input parameters
radius=input('please input the radius of the wear track:');
plan1minx=input('min x of plan area1:'); plan1minx =round(plan1minx *512/1455);
plan1maxx=input('max x of plan area1:'); plan1maxx =round(plan1maxx *512/1455);
plan1miny=input('min y of plan area1:'); plan1miny =round(plan1miny *512/1410);
plan1maxy=input('max y of plan area1:'); plan1maxy =round(plan1maxy *512/1410);
plan2minx=input('min x of plan area2 (two plans should be symmetric):'); plan2minx =round(plan2minx *512/1455);
plan2maxx=input('max x of plan area2:'); plan2maxx =round(plan2maxx *512/1455);
plan2miny=input('min y of plan area2:'); plan2miny =round(plan2miny *512/1410);
plan2maxy=input('max y of plan area2:'); plan2maxy =round(plan2maxy *512/1410);
trackmin=input('min of track:'); trackmin =round(trackmin *512/1430);
trackmax=input('max of track:'); trackmax =round(trackmax *512/1430);

% 2nd step, calculate average plan height
h1=0; c1=0; h2=0; c2=0; h=0; diff=0;
for i= plan1minx:1: plan1maxx; for j= plan1miny:1: plan1maxy;
    h1=h1+data(512*(j-1)+i,3); c1=c1+1;
end; end;
```

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for i= plan2minx:1: plan2maxx;  for j= plan2miny:1: plan2maxy;
    h2=h2+data(512*(j-1)+i,3); c2=c2+1;
end;end;

h=(h1/c1+h2/c2)/2; diff=h1/c1-h2/c2;
%3rd step, leveling

distplan=0; step=0;
distplan=abs(plan2minx-plan1minx); step=diff/distplan;
for i=1:512; inc=0; for j=1:512;
    inc=(i-1)*step; data(512*(j-1)+i,3)=data(512*(j-1)+i,3)+inc;
end;end;

for i= plan1minx:1: plan1maxx;  for j= plan1miny:1: (plan1miny+20);
    h1=h1+data(512*(j-1)+i,3); c1=c1+1;
end; end;

for i= plan1minx:1: plan1maxx; for j= (plan1maxy-20):1: plan1maxy;
    h2=h2+data(512*(j-1)+i,3); c2=c2+1;
end;end;

diff2=0; diff2=h1/c1-h2/c2; distplan=abs(plan1maxy-plan1miny-20); step=diff2/distplan;
for i=1:512; inc=0; for j=1:512;
    inc=(j-1)*step; data(512*(j-1)+i,3)=data(512*(j-1)+i,3)+inc;
end; end;

% 4th step, get the plan height

h1=0; c1=0; h2=0; c2=0;
for i= plan1minx:1: plan1maxx; for j= plan1miny:1: plan1maxy;
    h1=h1+data(512*(j-1)+i,3); c1=c1+1;
end; end;

for i= plan2minx:1: plan2maxx; for j= plan2miny:1: plan2maxy;
    h2=h2+data(512*(j-1)+i,3); c2=c2+1;
end;end;

h=(h1/c1+h2/c2)/2;
% 5th step, cutoff the very higher points (some debris landing on the edge)
for i=1:512; for j=1:512;
    if data(512*(j-1)+i,3)-h>1; data(512*(j-1)+i,3)=(data(512*(j-1)+i,3)-h)/5+h;
end;end;end;

% 6th step, calculate the wear volume

dV=0; c=0; dA=0; dZ=0;
for i=1:512;
    dV=dV+dA*2.754; % total volume change equation
dA=0; dZ=0; % set variable-- area change in one x-parallel line, height change per x point;
    for j= trackmin:1:trackmax; % x-min to x-max
        dZ=data(512*(i-1)+j,3)-h; % height change per x
        if dZ<1; % cutoff the debris in the wear track >1m
            dA=dA+dZ*2.842; % area change in one y line
        c=c+1;
    end; end;% end of j loop
end; % end of i loop

mV=dV*2*pi*1000*radius/1465;
Appendix 2: Supporting figures

**Fig. A.2.** The evolution of wear volume of the phenolic composite liner and the Epomet-PTFE-15 sliding against ø13 mm 100Cr6 steel ball, under 60 N normal load, 2 cm/s sliding speed, 22±2 °C and 35% ±2% relative humidity.

**Fig. A.3.** The evolution of wear volume of (a) the epoxy 25036 composite, under 60 N normal load, and (b) the epoxy 6078 composite, under 40 N load, sliding against ø13 mm 100Cr6 steel ball, 5 cm/s sliding speed, 22±2 °C and 35% ±2% relative humidity.
**Fig. A.4** The histogram of the measured transfer film thickness on (a) the wear scar of the TiC/a-C:H coated honing ball surface and (b) the wear scar of the steel honing ball surface. Both balls were sliding against the composite liners for 700 m, under 60 N normal load and 2 cm/s speed.
Fig. A.5 Measured size distribution of the diameter of (a) hollow glass spheres, (b) S, (c) M and (d) L solid glass spheres. The average diameter is indicated in the legend.
Fig. A.6 Measured length distribution of (a) carbon fibers, (b) short glass fibers (Short-GF), (c) glass fibers and (d) long glass fibers (Long-GF). The average length is indicated in the legend.
Fig. A.7 CoF curves of the Epomet-PTFE composites with 7.5 wt.% PTFE sliding against 100Cr6 steel, Al₂O₃ and Si₃N₄ counterpart balls under different conditions: (a) 2 cm/s velocity and 5 N normal load, (b) 20 cm/s velocity and 40 N normal load. Note the different scales of the ordinate of the figures.
Fig. A.8 Schematic illustrations of a steel ball sliding over (a) a SiO₂-epoxy composite without PTFE; (b) a PTFE-SiO₂-epoxy composite; (c) a PTFE-SiO₂-epoxy composite in water-lubricated conditions, with formation of metal-F bonding; (d) a PTFE-SiO₂-epoxy composite in water-lubricated conditions, without formation of metal-F bonding. The relative scale bars have the same length in all figures, showing the different gaps (between the ball and the composite surfaces) in different cases.
Appendix 3: On the dispersion and polar interfacial energies

The work of adhesion between two dissimilar materials fulfills a central role in this thesis and therefore it is relevant to understand the various approximations and physics behind Eq.(1) in Chapter 3 and Chapter 7, that reads

$$W_{adh} = \gamma_1 + \gamma_2 - \gamma_{12} = 2\sqrt{\gamma_1^d \gamma_2^d} + 2\sqrt{\gamma_1^p \gamma_2^p} \quad (A3.1)$$

where \(\gamma_1, \gamma_2\) and \(\gamma_{12}\) denote the surface free energy and interface energy of materials 1 and 2.

We can derive this expression as follows. Suppose an attractive constant \(A_j\) determines the work of adhesion \(W_{adh} = -W_{ij}\) (where \(W_{ij}\) is defined here as the negative of the energy required to separate two bodies from equilibrium to infinity):

$$W_{ij} \propto -A_{ij} \quad (A3.2)$$

We can consider \(A_{ij}\) as the attractive constant in a Lennard-Jones description, but that is not relevant as long as \(A_{ij}\) represents the attractive component of a force field. Let us assume that energy additivity holds on dipole-dipole (pp), dipole-induced dipole(pi) and dispersion forces (d) independently, i.e.

$$A_{ij} = A_{ij}^{pp} + A_{ij}^{pi} + A_{ij}^d \quad (A3.3)$$

Often pp and pi interactions are taken together and are defined as polar interactions:

$$A_{ij}^d = \frac{3}{2} \alpha_i \alpha_j \frac{I_i I_j}{I_i + I_j} (i, j = 1, 2) \quad (A3.4)$$

$$A_{ij}^p = \left(\alpha_i \mu_j^2 + \alpha_j \mu_i^2\right) + \frac{2\mu_i^2 \mu_j^2}{3kT} \quad (A3.5)$$

where \(\alpha_i\) represents the polarizability of \(i\).

**Dispersion forces:**

\(A_{ij}^d\) can be expressed in the attractive constants of the \(i\) and \(j\) by elimination from (Eqs.A3.4-A3.5) either \(\alpha_{i,j}\) or \(I_{i,j}\), i.e.
\[ A_{12}^d = 2 \sqrt{\frac{I_1 I_2}{I_1 + I_2}} \sqrt{A_{11}^d A_{22}^d} \quad (A3.6) \]

or

\[ A_{12}^d = 2 \frac{A_{11}^d A_{22}^d}{A_{11}^d \alpha_2 + A_{22}^d \alpha_1} \quad (A3.7) \]

If \( I_1 = I_2 \) then from Eq.(A3.6)

\[ A_{12}^d = \sqrt{A_{11}^d A_{22}^d} \quad (A3.8) \]

**Dipole-dipole and dipole–induced dipole interactions**

If pp interactions dominate over pi interactions:

\[ A_{12}^p = \sqrt{A_{11}^p A_{22}^p} \quad (A3.9) \]

whereas if pi interactions dominate:

\[ A_{12}^p = \frac{A_{11}^p + A_{22}^p}{2} \quad (A3.10) \]

Assuming in the interaction functions that

\[ r_{ij} = \sqrt{r_ir_j} \quad (A3.11) \]

In that case the work of adhesion can be simply connected to the work of cohesion \( E_{c,i,j}^{d,p} \)

\[ W_{12}^d = -\sqrt{E_{c,i,j}^{d,e} E_{c,j}^{d,e}} \quad (A3.12) \]

\[ W_{12}^p = -\sqrt{E_{c,i,j}^{p,e} E_{c,j}^{p,e}} \quad (A3.13) \]

Taking \( W_{ij} \) as the free energy functions, the Dupre relation yields:

\[ W^{ad} = -W_{12} = \gamma_1 + \gamma_2 - \gamma_{12} \quad (A3.14) \]

\[ 2\gamma_i = -E_{c,i} \quad (A3.15) \]

and Eqs. A3.8 and A3.9 yield:

\[ \gamma_{12} = \gamma_1 + \gamma_2 - 2\sqrt{\gamma_1^d \gamma_2^d} - 2\sqrt{\gamma_1^p \gamma_2^p} \quad (A3.16) \]
The *adhesion energy* is defined as the change of the surface energies of 1 upon contact over a certain contact area $S$ due to the interactions with 2, or simply:

$$U_{\text{adhesion}} = -W_{12}S = 2\left(\sqrt{\gamma_1^d\gamma_2^d} + \sqrt{\gamma_1^p\gamma_2^p}\right)S \quad (A3.17)$$

Eq. A3.17 is called the ‘geometrical mean equation'; it can only be derived under the specific assumptions that $I_1 \approx I_2$ and pp interactions dominate over pi interactions. Various other relations $\gamma_{12}$ may be obtained by selecting other assumptions.

The geometrical mean equation is most suitable for liquid-liquid interfaces (no ionic forces and no long-range force fields). The geometric mean equation will be less suitable for interfaces among ionic materials and between ionic materials like oxides and metals (induced dipolar fields [1]) but for interfaces among polymers and polymer-metals it will be not too bad (except for a complete rotational freedom in polymers, randomness will contribute to a geometrical mean description).

Using contact angle measurements, the geometric mean equation can be employed for the determination of the surface free energy. However, due to the many assumptions involved in the derivation, it is unlikely that the $\gamma_{12}$ data obtained using the concepts of dispersion and polar components will equal the thermodynamically derived surface free energy. Therefore it is concluded that all the data derived from Eq. A3.16 as presented in this thesis should be considered as semi-quantitative. Besides these theoretical considerations, there is another argument for precaution.

The experimental determination of the work of adhesion is not as easy as it looks. Delamination from a substrate is not only driven by applied stresses in the materials, but, sometimes rather, by residual stresses. Such stresses are inevitable in certain manufacturing techniques and are enhanced when the materials have vastly different thermo-physical properties. These stresses are still rather extrinsic due to deposition or to thermal expansion mismatch, but they can relax by annealing or by plastic deformation of some kind. In fact, the energy stored in the system, which becomes available when failure occurs depends on the thickness of the layers and on residual stresses in the layer or film.
If the solid phases 1 and 2 become strained to an amount \( d\varepsilon_{ij} \) along the interface, the area of the interface is increased by \( dS \) and the work, \( dW = \gamma_{12} dS \) is provided by an intrinsic stress, which is called the interface stress:\(^2\):

\[
\sigma'_{ij} = \gamma_{12} \delta_{ij} + \frac{\partial \gamma_{12}}{\partial \varepsilon_{ij}}
\] (A3.18)

The physical origin of the interface stress lies in the long-range interactions and the specific structure of the interface. Although the interface energy is a scalar, the interface stress in Eq. A3.18 is a tensor and different interfaces may have different interface stresses.

**Appendix 4: Stokes law**

Stokes law is a mathematical equation which expresses the velocities of small spherical particles in a fluid.

The frictional force of viscosity (Stokes drag) on a small sphere moving through a viscous fluid is given by:

\[
F_d = 6\pi \mu R V \quad (A4.1)
\]

where \( \mu \) is the dynamic viscosity, \( R \) is the radius of the spherical object, and \( V \) is the flow velocity relative to the object.

In an epoxy medium, the spherical glass particle also feels the gravity and the buoyancy force, the sum of which is:

\[
F = \frac{4}{3} \pi R^3 g (\rho_1 - \rho_2) \quad (A4.2)
\]

where \( g \) is the gravitational acceleration, \( \rho_1 \) (2.4 g/cm\(^3\)) and \( \rho_2 \) (1.2 g/cm\(^3\)) are the density of the spherical particle and the viscous medium.

Therefore, in an equilibrium status, \( F_d \) is equal to \( F \). Then, the velocity is

\[
V = \frac{2}{9} \frac{(\rho_1 - \rho_2)}{\mu} R^2 g \quad (A4.3)
\]

Let us take \( \Phi80 \) µm glass spheres and LY556/HY917/DY070(1\%) epoxy system for example. The difficult part of this calculation is that the real time viscosity of the epoxy system during curing is unknown. However, with this high performance composite epoxy system, three viscosity values is given by the manufacturer (as shown in the following table)
Table A.1 The development of viscosity during curing of the LY556/HY917/DY070(1%) epoxy system at 25°C.

<table>
<thead>
<tr>
<th>Curing time</th>
<th>0 h</th>
<th>1.5-2 h</th>
<th>6-7 h</th>
</tr>
</thead>
<tbody>
<tr>
<td>Viscosity (Pa·s)</td>
<td>0.75</td>
<td>1.5</td>
<td>3.0</td>
</tr>
</tbody>
</table>

However, adding the fillers in the epoxy system will increase the viscosity of the suspension. The wt.% of glass fillers is 37.5 wt.% in this study, which is corresponding to 22.5 vol.%. Einstein has theoretically addressed the viscosity behavior of suspension in the dilute limit in 1906:

\[ \eta_r = 1 + B\Phi \quad (A4.4) \]

where \( \eta_r \) is relative apparent viscosity, \( B \) is a constant which ranges between 1.5 and 5.5 (depend on particles concentration, particles shape and size, fluid rheological nature), \( \Phi \) is the volume fraction of particles. Mueller [3] and Barnes [4] have provided that the viscosity is about 2.4-2.7 times in a 22.5 vol.% filled suspension than that of the unfilled fluid. If we take 2.6 times, then the initial settling speed is:

\[ V' = 2.15 \times 10^{-6} \text{ m/s} \]

To reach this velocity from zero m/s, the time it cost is negligible in comparison with the whole curing process.

**Settling distance:**

Since the real time development of viscosity is unknown, the real time settling velocity and settling distance can be only be estimated. To simplify the calculation process, we take the average velocity between two known time points. The calculated settling distance is presented in Table A.2.

Table A.2 Calculation of settling distance of Φ80 μm glass spheres.

<table>
<thead>
<tr>
<th>Curing time</th>
<th>0 h</th>
<th>1.75 h</th>
<th>6.5 h</th>
</tr>
</thead>
<tbody>
<tr>
<td>Viscosity (Pa·s)</td>
<td>0.75</td>
<td>1.5</td>
<td>3.0</td>
</tr>
<tr>
<td>Velocity (×10⁻⁶ m/s)</td>
<td>2.15</td>
<td>1.12</td>
<td>0.56</td>
</tr>
<tr>
<td>Average velocity (×10⁻⁶ m/s)</td>
<td>1.64</td>
<td>0.84</td>
<td></td>
</tr>
<tr>
<td>Settling distance (mm)</td>
<td>10.3</td>
<td>14.3</td>
<td></td>
</tr>
</tbody>
</table>

It can be seen that the settling distance when reaching 3.0 Pa·s viscosity is already more than 24 mm, which is much larger than the thickness of the curing samples (usually around 8-9 mm). Particles settle to the bottom side of the sample. If using an epoxy which has 5 times higher initial viscosity (i.e. Epoxydharz C) and two times faster curing speed, the settling distance is still about 2.4 mm, when the viscosity of the system reaches 14 Pa·s.
Appendix 5: Strength-bond failure?

The question we like to address here is if bond breaking of C-F due to mechanical force fields is likely? Two factors are important for the stiffness of the bond, $k_B$, and density of the bonds per unit of area, $r_a$, (where $r_a$ is the ‘atomic size’ effect, order of 0.1 nm). The zero-order approximation is that the modulus $E$ can be related to the bond stiffness, simply formulated as:

$$E = \frac{k_B}{r_a} \quad (A5.1)$$

where $k_B$ can be taken from experiments (IR spectroscopy). The stretching frequency of C-F bond is 1100 cm$^{-1}$ (1 cm$^{-1} = 1.23 \times 10^{-4}$ eV, 1 eV = $1.6 \times 10^{-19}$ Jmole$^{-1}$) and in a quasi-harmonic force field approximation follows for the stiffness constant [5] per diatomic bond:

$$k_B = 596 \text{ Nm}^{-1} \quad (A5.2)$$

This seems to be quite reasonable since all spring constants $k_B$ of purely covalent bonds lie between 10-1500 Nm$^{-1}$, depending on the character of single, double or triple bond order. C-F has a polarized covalent bond and the binding in fact is quite strong because of its partial ionic character. Polymers contain both strong covalent and weaker van der Waals bonds (0.5-2 Nm$^{-1}$). Elastomers have a low stiffness because the glass temperature is below room temperature leaving only the weak ‘entropic’ force fields associated with long-chain, tangled molecules, etc.

Obviously electrostatic/dipole interactions and stereoelectronic interactions with neighboring C-F bonds play an important role in PTFE [6]. Suppose to a first approximation a localized-bond picture is the base for our prediction of bond breaking and we ignore electrostatic/dipole interactions and stereoelectronic interactions in the estimate of bond breaking. The force to break a single bond is roughly after stretching to 0.2% (no plasticity):

$$F_c = \frac{k_B r_B}{500} \quad (A5.3)$$

and the (shear) stress to break the bonds is then estimated by:
\[ \sigma_F = \frac{F_c}{r_B^2} \quad (A5.4) \]

leading to a prediction of the failure stress of (covalent bond length is 0.135 nm) of:

\[ \sigma_F = 8.8 \text{GPa} \quad (A5.5) \]

Obviously, this is far too big in comparison with our experiments. The reason is twofold:

a): polymers do not derive their stiffness and strength from localized bond stretching but from change in entropy of the tangled molecular chains when the material deform. It means that van der Waals bonds between the chain matter but not so much stretching of individual bonds.

The experimental values of the \( E \) modulus of epoxies, of PMMA and of PVC at a density of 1-2 g/cm\(^3\) are close to 5 GPa. With Van der Waals atomic radii of F and C which are ranging between 0.149 nm and 0.170 nm a stiffness constant \( k_B \) of 0.85 Nm\(^{-1}\) is calculated, i.e. at least 3 orders of magnitude smaller than predicted based on a localized-bond description.

b): stress concentrators in the material

In conclusion: external force fields in our experiments will lead to entropic restoring and relaxing forces, but not to localized C-F bond-breaking effects. The answer to the question whether the force fields in our experiments are sufficient for bond breaking of C-F is negative.

List of Publications

Journal papers


Conference proceedings

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