Effect of Graphite Concentration on the Tribological and Mechanical Properties of Filled SU-8 Polymer

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Different concentrations of graphite powder (particle size < 20 µm) were added for the enhancement in mechanical and tribological properties of SU-8 polymer. The materials were studied as thick coatings with thickness in the range of ~ 35-40 µm on glass substrate. SU-8 and SU-8/graphite composite properties were examined using atomic force microscope (for nanoindentation test) and pin-on-disc tribometer (for friction and wear). The surface characteristics were studied using Optical Microscope, Goniometer and 3D Profilometer. At 10 and 20 wt% graphite concentration, it was found that several properties were enhanced such as elastic modulus ~ 2.2 times, marginal increment in hardness and approximately same water contact angle and same surface free energy (SFE) as compared with those of pure SU-8. More importantly, 10 wt% graphite concentration has given two-times lower steady-state coefficient of friction and ~ 10 times more wear life compared to those of pure SU-8. The 20 wt% composite gave higher coefficient of friction but lower wear rate than the 10 wt% composite.

Keywords: SU-8, graphite, polymer composite

1. Introduction

SU-8, an eight epoxy-based polymer with near ultra-violet (UV) sensitivity property, is a potential structural material for Micro Electro Mechanical System (MEMS) and other micro-machines. It has several advantages such as thermal stability (330°C) against decomposition, micro-fabrication capability and bio-compatibility etc. However, poor mechanical (elastic modulus and hardness) and tribological (friction and wear) properties are the major drawbacks [1]. Hence, improvement in both tribological and mechanical properties of SU-8 is necessary for the effective utilization of polymer MEMS based on SU-8.

Several researchers [2-7] have added various fillers to SU-8 in different weight percentages for the enhancement of tribological and surface mechanical properties. A reduction in the coefficient of friction by ~3-5 times and increase in wear life by ~2 times have been reported for these solid particle fillers. In many cases, the internal stress and the coefficient of thermal expansion were also reduced. PFPE liquid lubricant was added to SU-8 as filler which greatly improved the tribological properties without any noticeable gain or loss in mechanical properties of SU-8 [8]. Graphite powder is an important filler material for many matrices for the improvement in tribological properties. Graphite in different concentrations was added in pure metal, epoxy and alloy composites and a reduction in the friction and wear properties was observed with increasing graphite concentration [9-13].

No comprehensive work has been reported on the improvement in mechanical and tribological properties of SU-8 with optimum concentration of graphite powder as the filler. The main objective of this research work, therefore, is to investigate the effect of graphite concentration on the tribological and mechanical properties of SU-8 and optimize it for further study.

2. Experiment

Glass substrate of dimensions 30 mm × 30 mm was used to deposit SU-8 and SU-8/graphite composite films of 35-40 µm thickness by spin coating (S-2000 series MILLMAN, Pune India). The glass substrate was cleaned using oxygen plasma cleaner PDC 32G (Harrick plasma, NY, USA) prior to spin-coating. These
spin-coated samples were pre-baked at temperature of 60°C for 5 minutes followed by at 100°C for 15 minutes. The pre-baked samples were exposed to UV (ultra violet) rays with the wavelength of 365 nm and power 210 mJ/cm² for a duration of 50-60 seconds using UV lamp followed by post exposure bake at a temperature of 70°C for 5 minutes. Finally, baking was carried out at 110°C for 15 minutes. All the procedure and parameters of spin-coating, pre-baking, UV curing and post-baking were approximately same for both pure SU-8 and SU-8/graphite composites.

The thickness of the coatings was measured by partly removing the coating from the substrate using a glass piece. Since the substrate was also of the same glass material, the scratching action removed only the coating without scratching the substrate and created a step. The step height, which actually represented the thickness of the coating, was then measured using 3D optical profiler (Bruker, USA).

Friction and wear tests on the prepared samples of SU-8 and SU-8/graphite composites were performed on a lab- fabricated ball-on-disk tribometer using Si₃N₄ ball of 4 mm diameter with surface roughness of Ra ~ 5 nm. The ball-on-disk tribometer is shown in Fig 1. A phosphor- bronze ring was used as the load cell and the load cell calibration factor was experimentally found to be 0.90 N/mV.

The friction and wear tests were conducted in atmospheric environment at a normal load of 2 N and at a linear sliding speed of 0.36 m/s in rotational cycles. The wear life is reported as the number of cycles at which the ball contacted the substrate. This was judged by recording the coefficient of friction (COF) and observing a sudden change in the COF. All tests were repeated at least 3 times for every set of experimental conditions and average data are reported here. Standard deviations of the data set for each test were calculated and are provided as error bars.

The water contact angles and surface free energy of SU-8 and SU-8/graphite composites were measured by

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Table 1  Surface characterization and Tribology Data

<table>
<thead>
<tr>
<th>Materials</th>
<th>Elastic Modulus (GPa)</th>
<th>Hardness (GPa)</th>
<th>Calculated Hertzian Contact Pressure (MPa)</th>
<th>Roughness (µm)</th>
<th>WCA (Degree)</th>
<th>Wear Rate (× 0⁻³mm³/N·m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure SU-8</td>
<td>2.73 ± 0.02</td>
<td>0.29 ± 0.003</td>
<td>38.66</td>
<td>0.043 ± 0.02</td>
<td>82.03</td>
<td>4.95</td>
</tr>
<tr>
<td>SU-8/Graphite (2 wt%)</td>
<td>5.08 ± 0.13</td>
<td>0.3 ± 0.01</td>
<td>58.24</td>
<td>0.160 ± 0.03</td>
<td>77.78</td>
<td>1.71</td>
</tr>
<tr>
<td>SU-8/Graphite (5 wt%)</td>
<td>6.21 ± 0.06</td>
<td>0.40 ± 0.007</td>
<td>66.46</td>
<td>0.609 ± 0.20</td>
<td>78.7</td>
<td>2.02</td>
</tr>
<tr>
<td>SU-8/Graphite (10 wt%)</td>
<td>6.56 ± 0.18</td>
<td>0.41 ± 0.01</td>
<td>68.86</td>
<td>1.26 ± 0.41</td>
<td>81.91</td>
<td>0.406</td>
</tr>
<tr>
<td>SU-8/Graphite (20 wt%)</td>
<td>6.43 ± 0.33</td>
<td>0.45 ± 0.05</td>
<td>67.98</td>
<td>6.42 ± 1.16</td>
<td>71.0</td>
<td>0.281</td>
</tr>
<tr>
<td>SU-8/Graphite (30 wt%)</td>
<td>0.86 ± 0.07</td>
<td>0.03 ± 0.002</td>
<td>18.02</td>
<td>6.50 ± 2.0</td>
<td>59.25</td>
<td>0.43</td>
</tr>
</tbody>
</table>

Goniometer (Data Physics, Model: OCA35, 12 VDC, Germany). The 0.5 µl quantity of water was used for water contact angle test.

An optical microscope (Nikon) was used to study the cross-section and surface morphologies of wear track of pure SU-8 and SU-8/graphite composite films. The thickness of coating, roughness and wear volume of SU-8 and SU-8/graphite composites were calculated using topographical scans by 3D Optical Profilometer (Bruker, USA). The wear rate is calculated as the specific wear rate ($W_{sp}$), which is given as follows [14].

$$ W_{sp} = \frac{V}{L D} \text{mm}^3/\text{N m} \quad (1) $$

where $V$, $L$ and $D$ are the total wear volume, normal load and the total sliding distance, respectively.

The elastic modulus and hardness of SU-8 and SU-8/graphite composites were measured by using XE7 integrated AFM (Park system, South Korea) system. The test was performed using silicon cantilever with pyramidal shape diamond tip.

3. Results and discussion

SU-8 and SU-8/graphite composite coating of thickness ~ 35-40 µm were formed on glass substrate and tested under the conditions stated in the experimental section. The following results were obtained and are discussed in the sections below.

3.1. Surface characterization:

Table 1 shows the surface characterization data (elastic modulus, hardness, water contact angle (WCA), roughness, surface free energy and wear rate). From the
It is observed that increasing the concentration of graphite, surface roughness increases but after 20 wt% concentration, it remains constant. The elastic moduli of composites increase up to 10 wt% concentration but decreases for higher wt%. The WCA data show that the surface is hydrophilic in nature and hence it offers greater adhesive force. The reduction in the modulus and hardness beyond 10 wt% of graphite coupled with a reduction in the WCA indicate that the cross-linking of SU-8 molecules is affected for higher graphite concentration. We have found that poorly cured SU-8 shows more hydrophilic nature in comparison with properly cured SU-8. The composites with 20 and 30 wt% graphite have shown drastic decrease in the WCA. Hence, we believe that for higher wt% of graphite, UV light cannot penetrate into the SU-8 leaving uncured material at the base of the coating. This is the main reason for drastic reduction in the mechanical properties as well.

Using the elastic moduli of the composites from the table above and the elastic modulus of the ball material, which is silicon nitride, and their respective Poisson’s ratio, the Hertzian contact pressure is calculated. The Hertzian contact pressure data are shown in Table 1. The variation in Hertzian contact pressure is found to be from ~70 MPa for SU-8/graphite (30 wt%) to ~ 135 MPa for SU-8/graphite (10 wt%) for the same 2 N applied normal load. The values of Hertzian contact pressure are very close to the range available for MEMS (27 to 140 MPa) in literature for Si MEMS.

3.2. Tribology test:

Figure 2 presents typical raw data for the coefficient of friction (COF) versus the sliding cycles, the initial and steady-state coefficients of friction with the respective standard deviations. The raw data presented in Figure 2(a) show the general behavior of the coatings. There is sudden increase in the COF when the coatings fail with much fluctuating values. The number of cycles at the point of coating failure was used for estimating the wear life of the coatings. The initial coefficient of friction is the average value of initial ~ 600 numbers of cycles and the steady state friction is the average value of last ~ 1000 numbers of cycles before coating failure. Coating failure was defined as the point after which sudden increase or fluctuations in the coefficient of friction occurred, or, the coating was visibly seen to have failed which happened suddenly.

The SU-8/graphite (10%) composite shows low initial and steady-state (~ 0.36 and 0.38 respectively) coefficients of friction when compared with pure SU-8 and other SU-8 composites.

Figure 3 presents the ball images after the tribology test at the point of coating failure. The ball images show wear behavior of SU-8 and SU-8/graphite composites, either abrasive, fatigue or delamination. In the case of pure SU-8, the initial wear occurs, followed by delamination of the coating by fatigue due to the brittle nature of the coating and McCook et al. [18] have reported that delamination occurs for very thin coating because of the high interfacial pressure. In the cases of SU-8/graphite composites with 2 wt%, 5 wt% and 10...
wt% graphite contents, abrasive wear particles are seen on the ball surfaces. These particles are identified as the glass wear particles which are formed because of the complete failure of the coating and further sliding of the silicon nitride ball against the substrate. In the cases of 20 wt% and 30 wt% graphite composites, however, blackish wear material (mostly graphite) accumulate around the interface area on the ball surface. There is a reduction in the coefficient of friction for 30 wt% because of the presence of graphite debris at the interface and poor curing of SU-8. Some debris materials were stuck to the ball surfaces which occurred because of the delamination of the coating in uncured regions. In the steady-state, sliding occurred between the coating and the transferred debris material. This reduction in COF was not observed for the 20 wt% specimen because of the high roughness of the coating surface and there was very little debris transfer to the ball surface.

After tribology test, the surfaces are characterized by optical microscopy to find out track width and the images are shown in Figure 4. SU-8/20 wt% graphite surface has shown lesser wear track width (~1.09 mm) compared to those for pure SU-8 and other composites. Due the delamination of coating, the track width of SU-8 (~2.77 mm) is more in comparison to those for the composites. Few images show the whitish particles inside the wear track which are abrasive debris particles of sample surface.

Fig. 4  Optical Microscope images of SU-8 and SU-8/graphite composites after tribology test (2 N load at 0.36 m/s constant speed and room temperature). The scale bars (1 mm) are provided on each image.

The track widths, which are measured from Figure 4 and wear profile obtained by 3D Optical Profilometer, are shown in Figure 5 and Figure 6, respectively. The data of track width which is shown in Figure 5 are the averages of ~26 measurements taken at different places on the wear track with standard deviations.

The wear rate is calculated using equation (4) (depth and wear track from wear profile obtained from Figure 5 and Figure 6, respectively) and are presented in Table 1. From Table 1, SU-8/20 wt% graphite composite gives lesser wear rate in comparison to those for pure SU-8 and other graphite composites. Wear life (estimated from the long-term friction measurements) were also conducted for pure SU-8 and SU-8/graphite composites.
The wear life is evaluated in terms of number of cycle at which there was sudden change in the coefficient friction with much fluctuation. This point of change in the sliding test indicated complete failure of the coating and partial or full exposure of the glass substrate. Figure 7 presents the data for wear-lives for each sample in terms of sliding cycle with error bars. Increasing the wt% of graphite, the wear life is also increased but in the case of SU-8/graphite (10 wt%), 20 wt% and 30 wt% composites, there is a drastic increase in the wear life. The 20 wt% composite performs similar to the wear life of 10 wt% composite considering the error bars. For 30 wt% composite, we observed much coating material transfer to the ball because of uncured layers. There was deep wear track, though the COF was still in the lower range as seen in Figure 2(b) and (c).

4. Conclusions

In the present work, different concentrations of graphite powder were used to make SU-8 composites. Among them, SU-8/graphite (10 wt%) has given optimum results in terms of elastic modulus, hardness, initial and steady state coefficients of friction and the wear life. Based on the COF evaluation, 10 wt%, 20 wt% and 30 wt% composites showed high wear life; however the wear track width was large for 30 wt% composite than those of 10 wt% and 20 wt% samples. It is expected that 10 wt% graphite may provide optimum composition for mechanical and tribological property enhancement of SU-8.

References


