SU-8 Composites for Micro-systems Applications

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1. Abstract

An epoxy based SU-8 polymer is a suitable material for making microsystems such as MEMS because of its compatibility to the micro-fabrication methods. SU-8 has somewhat poor mechanical and tribological properties. In this research, we have used perfluoropolyether (PFPE) as in-situ lubricant filler to SU-8. This composite material has shown highly superior friction and wear performance over pristine SU-8 and any such alternative material for this application. We have shown that the mechanical property can also be enhanced by this method if we utilize nano-particles for further reinforcement. This self-lubricating SU-8+PFPE composite can be used for the fabrication of MEMS applications requiring no external lubrication, and also these composites can find applications in many tribological components of traditional machines.

2. Introduction

Microsystems such as MEMS (micro electro mechanical systems) are small scale devices (from few µm to several hundred µm), designed to perform variety of operations in various industries including automotive, bio-engineering, bio-medical and military etc. As the surface area to volume ratio gets larger, the surface forces become highly detrimental to operation of these devices. These forces, for small devices, are capable of stopping the operation of machine against its inertial forces [1]. Therefore, tribology of micro-systems becomes an important factor to be considered while designing any micro-electro mechanical systems (MEMS). Despite of the fact that Si has been a conventional MEMS structural materials for the past few decades, this trend may be about to change with SU-8 for certain applications. SU-8 is a negative thick-film photoresist [2]. SU-8 consists of three basic components: (a) an EPON™ SU-8 epoxy resin; (b) a solvent such as gamma-butyrolactone (GBL) and (c) a photoacid generator such as triaryl sulfonium salts. Each SU-8 molecule consists of an average of 8 epoxy groups (and hence the name SU-8).
SU-8 has some prominent advantages over Si, such as bio-compatibility, compatible with micro-fabrication process by its unique UV sensitive property and less hydrophobic nature [3]. However, it has two major drawbacks; its poor tribologoical and mechanical properties. Very limited studies have been conducted on improving the tribology of SU-8. Jiguet et al [4] have studied the effect of silica particles reinforcement with SU-8 and heat treatment of SU-8. The improvements in tribology from reinforcement are very marginal, but the heat treatment improved tribology considerably. Few other studies by Singh et al [5, 6] have developed a two-step surface modification process, which utilizes the oxygen-plasma treatment of SU-8 surface, followed by coating of a nanolubricant such as perfluoropolyether (PFPE). The tribology of SU-8 improved significantly by this method. Another study by the same researchers is on the chemical-modification of SU-8 surface by treating with ethanolamine-sodium phosphate buffer, followed by a coating of PFPE lubricant. The wear life is increased by three orders of magnitude, which attributed to end groups bonding between PFPE and ethanolamine.

In order to improve the tribology of SU-8 through self-lubrication, we have developed bulk SU-8 composites by using the liquid lubricants such as PFPE lubricant and nano-fillers such as SiO_2, CNTs and graphite as filler materials. These SU-8 composites have yielded much improved tribological properties when compared to the pristine SU-8.

3. Material and Experimental Procedure

The detailed sample preparation procedures for pristine SU-8 and SU-8 composite films can be found in our previous publication [7]. The fabrication procedures for the pristine SU-8 and SU-8 composite films are very similar except addition of liquid lubricant (i.e.PFPE) and nano-particles (i.e. SiO_2, CNTs or graphite) to SU-8 in the case of SU-8 composite.

Water contact angle measurements (WCAs) were carried out for pristine SU-8 and SU-8 composites using water volume of 0.5µl. The WCA measurements were also performed in inside the wear track.

X-ray photoelectron spectroscopy (XPS) characterizations were performed for pristine SU-8 and SU-8+PFPE composite on the fresh surface and inside the wear track. Two separate XPS instruments were used for fresh SU-8 surface and inside the wear track.

Tribological tests (friction and wear tests) were performed using UMT-2 (universal Micro Tribometer, CETR, USA) in a ball-on-disk setup. The typical number of sliding cycles versus coefficient of friction (COF) was recorded for the sliding tests. 4 mm diameter Si_3N_4 balls with a surface roughness of 5 nm were used as the counterface. From the sliding tests, an initial coefficient of friction (μ_i) was noted as an average of the first twenty sliding cycles. The steady-state coefficient of friction (μ_S) was measured as the average of all coefficients of friction values from the point where the steady-state behavior (after the observation period of initial coefficient of friction) was observed until the end of the test or until the failure point, whichever was earlier. The wear life for the tested conditions was taken as the number of sliding cycles after which the coefficient of friction exceeded 0.3 or a visible wear track was observed on the substrate with abnormally fluctuating friction values, whichever occurred earlier.

Nanoindentation characterization was carried out for pristine SU-8 and SU-8 composites using MTS Nano Identator XP with CSM (continuous stiffness measurement) methodology.

4. Results and Discussion

Table 1: *AFM images of freshly spin-coated Pristine SU-8 and SU-8+PFPE composite surfaces before and after washing* [8].

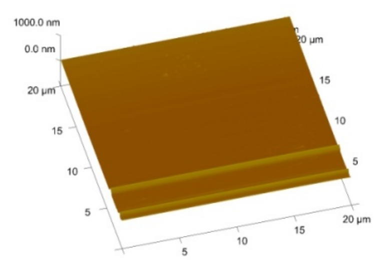
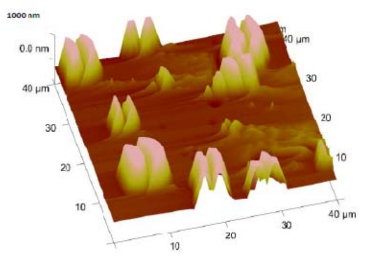
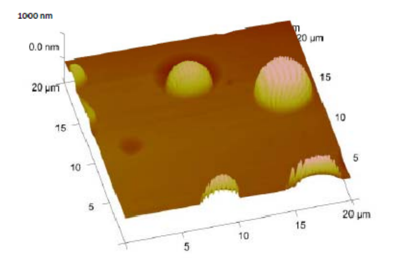
Composite	**AFM Images**
Pristine SU-8 (case 1)	
SU-8+PFPE – Freshly spin-coated (case 2)	
SU-8+PFPE – After washing (case 3)	

Table.1 shows the AFM images of freshly spin-coated surfaces of pristine SU-8 and SU-8+PFPE composite, before and after washing. The pristine SU-8 (case 1) shows plain surface without any feature.

Table 2: *Initial coefficient of friction (μ_i), Steady-state coefficient of friction (μ_S) and wear life (number of sliding cycles) of SU-8 and SU-8 composites obtained from sliding tests against 4 mm diameter Si_3N_4 ball at different normal loads and sliding rotational speeds.*

Composite Description	Initial coefficient of friction, COF (μ_i)	Steady-state coefficient of friction, COF (μ_S)	Wear Life (Number of Cycles)
Normal load :30g, Sliding Speed: 200 rpm (0.006 m/s), Spin Coated on Si			
Bare SU-8	0.82	-	0
SU-8+SiO2	0.77	-	0
SU-8+CNTs	0.39	-	0
SU-8+graphite	0.35	-	0
SU-8+PFPE	0.03	0.09	>100,000
Normal load :300g, Sliding Speed: 2000 rpm (0.06 m/s), Spin Coated on Si			
Bare SU-8	0.52	-	0
SU-8+PFPE	0.07	0.09	>1,000,000
SU-8+PFPE+SiO2	0.04	0.11	>1,000,000
SU-8+PFPE+CNTs	0.11	0.17	>1,000,000
SU-8+PFPE+graphite	0.09	0.14	>1,000,000

[Note: Experiment stopped due to long test duration for all PFPE contained films. No μ_s were reported for all non-PFPE samples as they fail very early.]

SU-8+PFPE surface before washing (case 2) shows uniformly dispersed droplets over the surface with negligible agglomeration, whereas the same surface after washing (case 3) with PFPE solvent H-Galden, shows uniformly dispersed droplets over the surface. These AFM images do indeed confirm that appreciable amount PFPE droplets have been retained even after intensive washing. The effect of observed features on tribological behavior of SU-8+PFPE composite will be discussed in the next paragraph.

Tribological results of pristine SU-8 and SU-8 composites are summarized in Table.2. Tests were conducted at various normal loads and sliding speeds. Pristine SU-8 and all non-PFPE composites were tested at 30g normal load and 200 rpm sliding speed. The pristine SU-8 exhibited very high μ_i ~0.82 and low wear life of n~0. SU-8+SiO2, SU-8+CNTs and SU-8+Graphite showed μ_i of 0.77, 0.38 and 0.35 respectively and low wear life of n~0. All the PFPE contained composites were tested at 300g normal load and 2000 rpm sliding speed and exhibited much superior tribological properties than non-PFPE composites. These self-lubricating SU-8+PFPE and SU-8+PFPE+nanoparticle composites have reduced the initial and steady-state coefficient of friction by *~6-9* and *~3-6* times, respectively, and increased wear life by more than five orders *(>10^5)* of magnitude.

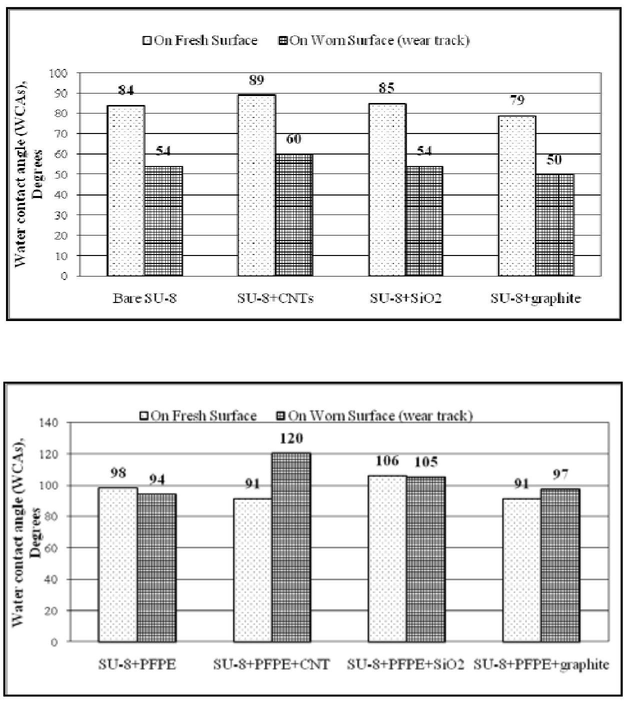


Figure 1: *WCA values for pristine SU-8 and SU-8 based composites over fresh (pristine surface of composites before experiment) and worn surfaces (wear track).* **(a)** *: Non-PFPE combinations at 10^4 cycles (composites did not contain PFPE).* **(b)***: PFPE combinations at 10^6 cycles.*

Water contact angle measurements were performed on freshly spin-coated surfaces and worn surfaces (after tribological tests) also shown in Figure.1. From tribological results, it is found that the nanoparticles (NP) contribution to tribological improvement is very minimal and PFPE is the major contributor for tribological improvement. Therefore, water contact angle (WCA) values were measured for (non-PFPE combinations) pristine SU-8, SU-8+NP combinations and SU-8+PFPE, SU-8+PFPE+NP combinations (PFPE combinations) on freshly spin-coated surfaces and worn surfaces (after tribological tests), as shown in Figure.1. Pristine SU-8 and SU-8+NP combinations without PFPE are showing contact angle values ~80° in fresh surface condition and ~55° in worn surface condition. The PFPE combinations are showing WCA value of >90° in fresh surface condition and increased significantly up to 120° in worn surface condition. There is 100% increase in WCA values from non-PFPE combinations to PFPE combinations in worn surface condition. It is clear that PFPE is responsible for this enhanced hydrophobic behavior.

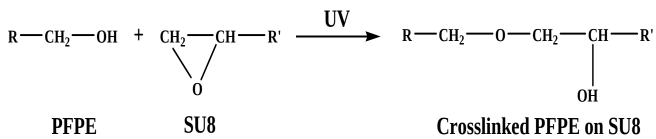


Figure 2: *A depiction of the formation of ether bonds between SU-8 and PFPE in SU-8+PFPE composite.*

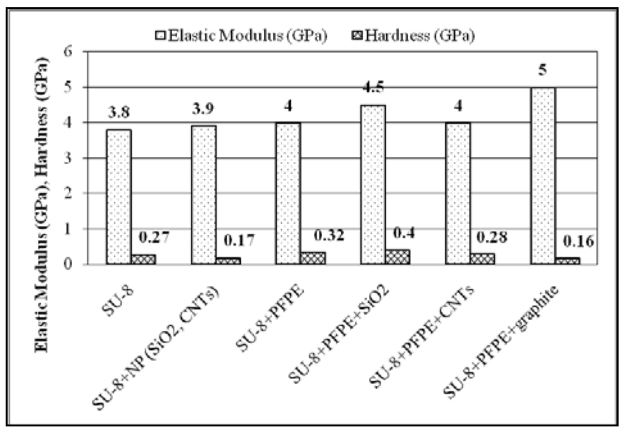


Figure 3: *Elastic Modulus (E) and Hardness (H) values of pristine SU-8 and SU-8 composites from nano-indentation characterization.*

The significant improvement in tribological properties of SU-8 can be attributed to the possible ether bond formation between the SU-8 and PFPE molecules [9, 10]. The possible etherification reaction is depicted in Figure 2. It is postulated that the -OH terminal groups in the PFPE polymer and the carbocations (C^+) in the SU-8 polymer (epoxide ring) undergo the reaction, which breaks the existing bond and forms new linkage which is called ether bond.

The Pristine SU-8 (Figure 3) has shown E and H values of ~3.8 and ~0.27 GPa respectively, while SU-8+PFPE has shown E and H of ~4 and ~0.32 GPa respectively. The improvement in mechanical properties of SU-8 by PFPE addition could be attributed to the chemical bonding between SU-8 and PFPE, which results in increased cross-linking. SU-8+PFPE+SiO_2 has shown H of ~0.4 GPa, which is 40% increase in H. SU-8+PFPE+Graphite has shown E of ~ 5 GPa, which is 22% increase in E. However, the selection of right composite entirely depends on the application i.e. high hardness/modulus requirements.

5. Conclusions

The developed self-lubricating SU-8 composites with PFPE are much more hydrophobic than SU-8 and SU-8+NP composites in fresh and worn surface conditions. The self-lubricating SU-8 composites reduced initial coefficient of friction of SU-8 by ~6-9 times and increased wear life by >10^5 times. The elastic modulus (E) and hardness (H) were also increased by ~1.4 times. The chemical bonding between SU-8 and PFPE molecules were partially verified by AFM images and tribological characterization indirectly.

Acknowledgements

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6. References

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