Thermo-mechanical and tribological properties of SU-8/h-BN composite with SN150/perfluoropolyether filler

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Abstract: In this study, SU-8 and its composites are fabricated by blending 10 wt.% hexagonal boron nitride (h-BN) fillers with/without lubricants, such as 10 wt.% base oil (SN150) and 20 wt.% perfluoropolyether (PFPE). The thickness of SU-8 and its composites coating is fabricated in the range ~100–105 μm . Further, SU-8 and its composites are characterized by a 3D optical profilometer, atomic force microscopy, scanning electron microscopy, a thermal gravimetric analyzer, a goniometer, a hardness tester, and an optical microscope. Under a tribology test performed at different normal loads of 2, 4, and 6 N and at a constant sliding speed of 0.28 m/s, the reduction in the initial and steady-state coefficient of friction is obtained to be ~0.08 and ~0.098, respectively, in comparison to SU-8 (~0.42 and ~0.75), and the wear resistance is enhanced by more than 103 times that of pure SU-8. Moreover, the thermal stability is improved by ~80–120 °C, and the hardness and elastic modulus by ~3 and ~2 times that of pure SU-8, respectively. The SU-8 composite reinforced with 10 wt.% h-BN and 20 wt.% PFPE demonstrated the best thermo-mechanical and tribological properties with a nano-textured surface of high hydrophobicity.

Keywords: SU-8; boron nitride; perfluoropolyether (PFPE); base oil (SN 150); *in-situ* lubrication

1 Introduction

Surface treatments and coatings have provided tremendous achievements in micro-electro-mechanical system (MEMS) industries from metallurgy to processing [1]. Composite deposits can be coated on steel, light metals, ceramics, plastics, and other materials to enhance the surface properties, such as mechanical, tribological, corrosive, and thermal [2]. For all polymeric materials, SU-8 is a promising material for next generation 3D fabrication. It can also be used as a coating material in different applications, such as bearing steel [3]. Currently, silicon wafers are used for the fabrication of MEMS devices; however, they have several disadvantages, such as a low thermal stability,

difficulty in 3D fabrication, and non-biocompatibility. These disadvantages can be diminished by introducing a polymer, such as SU-8. SU-8 is an epoxy-based negative photo resist that is ultraviolet (UV) curable and can mitigate the disadvantages of a silicon wafer; however, it has poor mechanical and tribological properties [4, 5]. If the mechanical and tribological properties are improved, SU-8 could be used to effectively create small 3D devices. Therefore, researchers have tried to improve these properties by adding solid fillers, liquid fillers, or a solid with liquid fillers. These solid fillers could be a silicon powder, silicon carbide, graphite, graphene, single and multiwall carbon nanotubes, etc., and the liquid fillers could be perfluoropolyether (PFPE), multiply alkylated cyclopentane

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(MAC), SN 150 base oil, mineral oils, etc.

For blending solid fillers in SU-8, Jiguet et al. [6–8] first mixed different weight percentages of silica nano particles. They performed friction and wear tests using a polyoxymethylene (POM) ball and carbon steel ball with a 6 mm diameter as the interface on the sliding friction apparatus. They observed that wear was reduced by a heat treatment process and an optimal concentration of (2.5 wt.%) silica nano particles. They also reported that the coefficient of friction was inversely proportional to the elastic property of SU-8, and the nanoparticles lowered the internal stress of the surface and reduced the coefficient of thermal expansion. Further, Saravanan et al. [9–11] mixed solid fillers, such as graphite, silicon dioxide, and single wall carbon nanotubes in SU-8. They performed a tribology test using a ball-on-disk setup with a 4 mm diameter silicon nitride ball as the interface. Minimal reduction in friction was observed. Therefore, they further added liquid fillers, such as PFPE, MAC, and SN 150, with solid fillers and found that the coefficient of friction was reduced by approximately 5–6 times, and the wear life was increased by more than 10^4 times. They also performed a nano-indentation test to measure the elastic modulus and hardness and found a marginal increase in the mechanical properties. To continue this study, Katiyar et al. [12–14] mixed carbon fillers, talc, graphite, and a combination of talc and graphite, in different weight percentages in SU-8 and fabricated SU-8 composites. Friction and wear tests were performed on the fabricated composites with a ball-on-disk setup using a silicon nitride ball with a 4 mm diameter, and the coefficient of friction was ~ 4 –5 times lower than that of pure SU-8. The mechanical properties of SU-8/talc (30 wt.%) were ~ 3 –4 times greater than that of pure SU-8.

From the above methods, the solid fillers alone in SU-8 did not show encouraging results in terms of the tribological properties. Therefore, researchers [10, 11] have proposed to mix liquid lubricant fillers with or without solid fillers in SU-8. Liquid lubricant fillers, such as PFPE, MAC, and SN 150 have been mixed with solid fillers, such as CNT and graphite, in an SU-8 matrix, and a tribology test was performed on a ball-on-disk machine. The wear resistance property and wear life were improved by $\sim 10^5$ times. Moreover,

the initial and steady-state coefficients of friction were reduced. Further, Batooli et al. [15] added ionic liquid in different weight percentages into SU-8 and fabricated an SU-8 composite. These composites were tested on a surface force apparatus (SFA) using a carbon steel ball with a 6 mm diameter. The friction and wear were directly proportional to the concentration of ionic liquid. Katiyar et al. [16] added PFPE lubricant fillers in different weight percentages with talc and noticed a significant reduction in the coefficient of friction.

Furthermore, from the literature, a nitride (Cr_xN) surface demonstrated high hardness, enhanced wear resistance, and residual compressive stress [17]. Boron-based compounds, such as boron carbide, boron nitride, and transition metal borides, exhibited numerous attractive properties, such as a high melting point and hardness, good wear and corrosion resistance, excellent electrical conductivity, and resistance to attack by molten metals [18]. Hexagonal boron nitride had a graphite-like structure and was suitable for applications where the interface temperature reached $\sim 400^\circ\text{C}$. A majority of the studies evaluated the effect of h-BN in clutches, bearing, etc. Kimura et al. [19] mixed h-BN with a lubricant as an additive and performed sliding experiments. They reported that h-BN added to a lubricant marginally increased the coefficient of friction and drastically reduced wear. Furthermore, the effect of h-BN on the tribological properties for a sliding bearing was studied by Pawlak et al. [20, 21]. They observed a low coefficient of friction (0.02–0.03) and concluded that the load and pressure-velocity parameters affected the coefficient of friction. They also reported that h-BN was biocompatible and performed *in vitro* and *in vivo* tests. Here, h-BN yielded a lower coefficient of friction than that of phospholipids. Chen et al. [22] reported a reduction in the coefficient of friction and an improvement in the wear resistance property after adding h-BN in a copper matrix, which was used for the sleeves of high-speed sewing machinery.

From the published literature, h-BN also acts as a good solid lubricant and provides *in-situ* lubrication in various applications. The studies have evaluated tribological issues or mechanical issues; however, the tribological and mechanical properties were not simultaneously investigated. Therefore, the main

objective of this study was to improve the mechanical and tribological properties. Based on this objective, hexagonal boron nitride (h-BN) with and without lubricant fillers SN 150 and PFPE was blended in an SU-8 matrix. To investigate the composites, a thick coating was fabricated, and mechanical and tribological tests were performed on a micro hardness tester and ball-on-disk setup, respectively. Here, h-BN with PFPE enhanced the mechanical and tribological properties.

2 Experimental procedure

2.1 Materials

The composite coatings of h-BN and h-BN with liquid fillers were fabricated on a 3 cm × 3 cm glass substrate. SU-8 (Grade-2025, Microchem Ltd USA) was used as the coating material. h-BN (Sigma Aldrich, India) was used in powder form with a particle size of ~1 µm (from supplier data). The SU-8 composites were fabricated in two forms: a solid filler, i.e., h-BN, and a solid filler, i.e., h-BN combined with liquid lubricant fillers (SN 150 and PFPE). These lubricant fillers were intended to fabricate a self-lubricating composite. SN-150 (Indian Oil Cooperation Ltd, India), Grade-I, with a viscosity of 4 cSt and Fomblin-Y (also known as functionalized PFPE) (Sigma Aldrich, India) with a viscosity of 60 cSt were used for this research study.

2.2 Sample preparation

A glass substrate of ~3 cm × 3 cm was cleaned using an ultrasonicator (Citizen Scales, India) with deionized water for ~30 min, followed by drying at room temperature (25 °C). The cleaned glass substrate surface was cured by an oxygen plasma cleaner, PDC 32G (Harrick plasma, NY, USA) for ~20 min. The oxygen plasma treatment removed the foreign contaminants and generated the functional group –OH on the top surface of the glass.

A microbalance (Citizen Scales, India) was used for preparation of the composite solution. Here, 10 wt.% h-BN was blended, and liquid fillers (SN150 and PFPE) were added 10 wt.% and 20 wt.%, respectively, to the SU-8 matrix. The volume of the liquid fillers was the same because the density of SN 150 is half the density of PFPE. The composite solution was initially mixed

by a stirring rod, followed by an ultrasonic homogenizer (Sonics VCX 130 manufactured by Sonics & Materials Inc. Newtown, USA) for 25–30 min. The ultrasonic homogenizer prevented agglomeration of the composite solution.

Finally, the prepared mixture of the composites was coated on the glass substrate by a spin coater (S-2000 series MILLMAN, Pune India). The spin coater created a homogeneous coating over the surface and maintained a coating thickness in the range of ~100–105 µm. The prepared coatings were pre-baked at a temperature of 90 °C for 5–8 min followed by heating at 100 °C for 10 min. The pre-baked samples were exposed to UV rays of a 365 nm wavelength and 210 mJ/cm² power for 80–110 s. The UV exposers generated cross linking between the SU-8 molecules. The UV cured samples were post baked at a temperature of 95 °C for 5–8 min followed by heating at 120 °C for 10 min. Finally, the prepared samples were placed in a desiccator to protect the prepared samples from foreign contaminations. A similar procedure and parameters were used for preparing the SU-8 composites. The abbreviations used in this study are listed in Table 1.

2.3 Tribology testing

Tribology tests were performed on the prepared samples at different normal loads (2 N, 4 N, and 6 N) with a constant sliding speed of 0.28 m/s at room temperature, 25 °C, until coating failure or 4 × 10⁵ cycles. The ball-on-disk setup (Ducom Instruments, India) was used to conduct the tribology test using a silicon nitride ball with a 4 mm diameter and 5 nm surface roughness (R_a) (per the supplier's data) at the interface. The silicon nitride ball was used because of its nonreactive nature with most materials and its high hardness. In each test, a new silicon nitride ball was used. For the measurement of friction and wear, a

Table 1 Abbreviations used for the different compositions.

Materials	Nomenclatures
Hexagonal boron nitride	h-BN
Pure SU-8	SU-8
SU-8/BN (10 wt.%)	BN
SU-8/BN (10 wt.%) / SN150 (10 wt.%)	BNSN
SU-8/BN (10 wt.%) / PFPE (20 wt.%)	BNFY

full bridge ring shape load cell was used with a calibration factor of 0.75 N/mV. The load cell was connected to the stain measurement system (SCAD500, Pyrodynamics Ltd., India), followed by the data acquisition system (DAQ, Technocomm Instruments Pvt. Ltd, India) and lab view software (provided by supplier). The friction data are presented in the form of the initial and steady-state coefficients of friction with the standard error. The initial and steady-state coefficients of friction were obtained from the average value of the first 100 sliding cycles and the average value of the last 5,000 sliding cycles, respectively. The average value of the wear life is also presented in this investigation. The wear life showed that the sample contained a visual wear track or the coating failed owing to a fluctuation of the friction. All tests were repeated at least 3 times for each coating sample, and the average value of the data was reported with the standard errors.

2.4 Mechanical and surface characterization

The composites were characterized using a 3D optical profilometer, an atomic force microscope, a scanning electron microscope, a thermal gravimetric analyzer, a goniometer, a hardness tester, and an optical microscope.

2.5 Wettability test

Wettability tests were performed on the prepared samples before the tribology test using a goniometer (data physics, model: OCA35, 12 VDC, Germany). A volume of 2 μ l of deionized water was used to measure the contact angle. All tests were performed at room temperature. The tests were repeated six times for each sample, and the average value of the contact angles with the standard error is presented in this study.

2.6 Thermogravimetric analysis

The thermal stability of the composite materials was analyzed using a simultaneous thermal analyzer (STA) 8000TGA (PerkinElmer, USA) and is presented by the weight loss with respect to time. The temperature range for the test was maintained between 30 to 550 $^{\circ}$ C, and the test was performed in the presence of a nitrogen gas environment. The heating rate for the test varied

at 10 $^{\circ}$ C $\cdot$ min $^{-1}$, and the cooling rate for test varied at 30 $^{\circ}$ C $\cdot$ min $^{-1}$.

2.7 Micro-hardness tester

For measurement of the mechanical properties (elastic modulus and hardness), a micro hardness tester (CSM, Switzerland) was used. Micro indentation was performed using a Vickers indenter with a 50 mN applied load. The indentation depth was in the range of $\sim$ 1–2 μ m, which was small compared to the coating thickness. For each sample, 4–5 indentations were performed, and the average value of the hardness and elastic modulus with the standard errors are presented.

2.8 3D optical profilometer

The Contour GTK (Bruker, USA) 3D optical profilometer was used to measure the surface roughness before the tribology test, and after the test, the wear depth and wear volume were observed. Furthermore, the specific wear rate in mm 3 /(N $\cdot$ m) for each coating was measured using the given equation [22].

$$W_{sp} = \frac{V}{LD} \quad (1)$$

Here, V is the wear volume (mm 3); L is the normal load (N), and D is the total sliding distance (m).

2.9 Optical/scanning electron microscopy

Finally, the surface morphology of the composite coatings after the tribology test, and the cross-sections of the coatings were observed using a scanning electron microscope (SEM, Σ IGMA, Zeiss, USA). Before obtaining the image from FE-SEM, a titanium coating with a thickness of $\sim$ 12 nm at 10 mA for 75 s was applied on each sample using a plasma sputter coater (SC7620-CF, Quorum Laughton Lewes). The surface morphology of the composite coatings before the tribology test was also observed using an atomic force microscope, XE7 integrated atomic force microscope (AFM) (Park system, South Korea). The test was performed in a non-contact mode using a silicon cantilever. After the tribology test, the ball interface surface was also evaluated using an optical microscope, Eclipse E100 (Nikon Instruments Inc., USA).

3 Results and discussion

3.1 Surface characterization

The surface of the composite material was characterized using a 3D optical profilometer and goniometer. The following results were obtained, as discussed in the following sections.

3.2 Coating surface

The coating surface was characterized at a micro- and

nano-scale using an optical microscope and atomic force microscopy, respectively. Figures 1(a)–1(d) shows the optical microscopic images of SU-8 and its composite surface. From Fig. 1, more liquid droplets were present in the BNFY composite coating at the same volume than that of BNSN, demonstrating changes in the surface after the addition of fillers. The sizes of these liquid droplets were in the range of 50 μm to 1 nm .

The coating surfaces were also observed using AFM, as shown in Figs. 2(a)–2(d). From Fig. 2, the pure SU-8 coating contained a flat surface; however,

Fig. 1 Optical microscope images of the composite surface: (a) SU-8, (b) BN, (c) BNSN, and (d) BNFY.

Fig. 2 Atomic force microscope images of the composite surface: (a) SU-8, (b) BN, (c) BNSN, and (d) BNFY.

after adding the fillers, nano-textures were observed. The surface roughness of the composite coatings was increased. However, after adding the liquid filler (SN 150 or PFPE) in BN, more homogeneous nano-textures were observed over the surface. These homogeneous nano-textures could entrap liquid droplets, which could reduce the coefficient of friction and change the surface from hydrophilic to hydrophobic.

To measure the thickness of the coating and the distribution of the liquid droplets throughout the thickness, scanning electron microscopy was used. The images are shown in Figs. 3(a)–3(d). From Fig. 3, the cross-section of the SU-8 coating surface was smoother than that of the BN composite coating. After adding the liquid fillers in the BN composite coating, the cross-section showed a homogeneous dispersion of the liquid droplets; however, for the BNFY composite, there were more liquid droplets, and the size of the liquid droplets was $\sim 50\text{ }\mu\text{m}$ to a few nm. From the figure, the thickness of prepared coating was in the range of $\sim 100\text{--}105\text{ }\mu\text{m}$.

3.3 Wettability test

From the wettability test (contact angle is shown in

Figs. 4(a)–4(d)), the composite surface was changed from hydrophilic to hydrophobic. From Fig. 4, the pure SU-8 and BN coatings were hydrophilic. However, after the addition of the liquid fillers (SN 150 and PFPE), the surfaces of the composites BNSN and BNFY were hydrophobic. The hydrophobic nature of BNFY could be because of the greater surface roughness, as shown in Fig. 5. This phenomenon was also suggested in a previous study; however, they considered different oils and surfaces [23].

3.4 Roughness test

Different wear modes were found to link the load to the roughness dependence between the bodies and counter body [24]. The data of the surface roughness with the standard errors are shown in Fig. 5. From Fig. 5, the roughness increased drastically after the addition of the fillers (BN or BN + SN 150/PFPE) in pure SU-8; however, a higher roughness was shown with the BNFY composite, i.e., this composite surface demonstrated a hydrophobic nature. The surface roughness of BNSN was also higher than that of the BN composite. The high surface roughness was owing to the nano-texture, as shown in Figs. 2(b)–2(d).

Fig. 3 Cross-sectional images of the composite surface: (a) SU-8, (b) BN, (c) BNSN, and (d) BNFY.

Fig. 4 Contact angle of the composite coating: (a) SU-8, (b) BN, (c) BNSN, and (d) BNFY.

Fig. 5 Surface roughness of the different coating materials.

3.5 Material characterization

The material was characterized using a thermogravimetric analyzer and micro hardness tester, as stated in the experimental section. A detailed discussion of these characterization methods is given below.

3.6 Thermogravimetric analysis (TGA)

The thermal stability of the different composites was measured by a TGA, and the consolidated data of the thermal decomposition are shown in Fig. 6. In all samples, approximately 18% weight loss was observed at a temperature of $\sim 100^\circ\text{C}$. The weight loss at this temperature was owing to the evaporation of the water molecules present in the samples. A significant weight loss in the range of $180\text{--}240^\circ\text{C}$ was observed.

Fig. 6 TGA spectra of SU-8 and its composites.

This weight loss was because of the thermal decomposition of the oxygen carrying functionalities. However, in the liquid carrying samples, the liquid fillers also evaporated at this temperature. Moreover, another major loss began at a temperature of $\sim 280^\circ\text{C}$, owing to the decomposition of SU-8 and its composite moieties. The spectra of coating material represented an exothermic weight loss over a wide range of temperatures, $180\text{--}500^\circ\text{C}$, owing to the slow decomposition. Therefore, the thermal stability of SU-8 was improved by $\sim 80\text{--}120^\circ\text{C}$ after adding the fillers because of the bonds formed between SU-8 and the fillers.

3.7 Micro hardness test

The micro hardness tester was used to measure the elastic modulus and hardness of the prepared coatings. Micro indentation was performed using a Vickers indenter at a 50 mN applied load. The indentation depth was in the range of $\sim 1\text{--}2\ \mu\text{m}$, which was smaller than the coating thickness. After indentation, the Oliver and Pharr method was used to calculate the elastic modulus and hardness of the sample surface, which was incorporated in software supplied by the manufacturer. The consolidated data with the standard error are shown in Figs. 7(a) and 7(b). From Fig. 7(a), the BN composite contained ~ 2 times greater hardness than that of pure SU-8. Similarly, after adding the liquid filler (SN 150), the hardness of the composite was reduced relative to the BN composite; however, for PFPE, the hardness was marginally increased relative to the BN composite. Therefore, after adding

Fig. 7 Mechanical properties of SU-8 and its composite materials: (a) hardness of the coating materials and (b) elastic modulus of the coating materials.

the fillers, the hardness was increased ~3 times. From Fig. 7(b), the BN and BNFY composites contained a higher elastic modulus (~2.5 times) than that of pure SU-8. The elastic modulus of the composite affects the Hertzian contact pressure, which is responsible for the friction and wear of the coating. This phenomenon was also discussed by Jiguet et al. [8] with a different material (SU-8/silicon nano-powder composite).

The Hertzian static contact stress is calculated using the following equation [25]

$$p_m = \frac{L}{A} = \frac{L}{\pi \left(\frac{3RL}{4E^*} \right)^{2/3}} \quad (2)$$

where L is the normal load; A is the Hertzian contact area; R is the radius of the spherical ball, and E^* is given as Eq. (3).

$$E^* = \left(\frac{1-\nu_1^2}{E_1} \right) + \left(\frac{1-\nu_2^2}{E_2} \right) \quad (3)$$

Here, E_1 and E_2 are the elastic modulus of the two interacting materials, and ν_1 and ν_2 are their Poisson's ratios, respectively. The values of E and the Poisson's ratio (ν) for silicon nitride were taken as 310 GPa and 0.27, respectively, from the supplier data. The Poisson's ratio for SU-8 and its composites was 0.22 [26]. The Hertzian contact stress is listed in Table 2.

3.8 Friction and wear test

The friction and wear tests were performed on a

Table 2 Hertzian contact stress (MPa) for the static contact between the 4 mm diameter silicon nitride ball and coated materials.

Materials	Hertzian contact stress (MPa)		
	2 N	4 N	6 N
SU-8	49.57	—	—
BN	84.13	—	—
BNSN	65.09	—	—
BNFY	84.55	106.54	121.96

ball-on-disk tribometer at a constant sliding speed of 0.28 m/s and different loading conditions (2, 4, and 6 N). The raw data of friction for the BNFY composite coating conducted under a 4 N normal load and 0.28 m/s sliding speed at room temperature are shown in Fig. 8. The initial and steady-state coefficients of friction were obtained from the average value of the first 100 sliding cycles and the average value of the last 5,000 sliding cycles, respectively, as shown in Fig. 8. Each tribology test was conducted until failure of the coating or a maximum of 4×10^5 cycles. The average data of the initial and steady-state coefficients of friction are shown in Figs. 9(a) and 9(b) with the standard errors (SE) as error bars based on at least three repetitions for each measurement.

First, the tribology test was conducted at a 2 N load and 0.28 m/s sliding speed for all fabricated composite coatings. At this load, greater initial and steady-state coefficients of friction were observed for pure SU-8. However, after adding the fillers into the SU-8 matrix, the initial coefficients of friction were drastically reduced because the surface of composite became textured, as shown in Fig. 2. These textures

Fig. 8 Raw friction data of the BNFY composite coating at a 4 N normal load and 0.28 m/s sliding speed

Fig. 9 (a) Effect of the load on the initial coefficient of friction and (b) the effect of the load on the steady-state coefficient of friction for SU-8 and its composites. The error bars in (a) and (b) show the standard errors of three repetitions for each data point.

reduced the contact area. For the BN composite coating, the initial coefficient of friction was reduced by ~ 3 times that of pure SU-8, while the steady-state coefficient of friction was reduced by ~ 1.5 times. Moreover, the increase of the steady-state coefficient of friction in the BN composite was owing to the increase in abrasive wear after $\sim 20,000$ cycles. The abrasive debris were observed at the ball-surface interface after the tribology test, as shown in Fig. 11. After adding the liquid fillers in the BN composite, the BNSN composite contained a low coefficient of friction; however, the coating was not sustained at a 2 N load. This could be because of the lower hardness and elastic modulus than those of the other composite coatings. Furthermore, the BNFY composite coating showed ~ 9 times lower the initial and steady-state coefficients of friction than those of pure SU-8 sample coating, which sustained up to 4×10^5 sliding cycles. Therefore, for further load variation, only the BNFY composite coating was tested because of its excellent properties in terms of the coefficient of friction and wear life. From Fig. 9, the BNFY composite provided lower initial and steady-state coefficients of friction even though the normal load increased from 2 to 4

and 6 N. The low coefficient of friction for the BNFY composite coating could be because of its hydrophobic nature, which reduced adhesion on the surface, and the texture on the surface, which reduced the contact area.

After the tribology test, the surfaces of SU-8 and its composite coatings were characterized by a scanning electron microscope. The images are shown in Figs. 10(a)–10(f). From Fig. 10(a), at 2 N load, the pure SU-8 sample coating was delaminated and contained a zig-zag shape. This could be because of high contact stress, which caused brittle fracture. This brittle-fracture phenomenon was also proposed in a previous investigation using different materials (PTFE/epoxy composite) [27]. After adding the fillers, the delamination of the coating was reduced, and a smooth wear track formed because of the self-lubrication properties of h-BN. Moreover, a homogeneous wear profile was developed, and wear debris were generated after a few cycles. These debris particles rubbed against each other and generated a higher friction than that of the other composites. The BN composite coating showed a three-body wear phenomenon. Therefore, adhesive and abrasive wear occurred in the BN composite coating.

Fig. 10 Optical microscope images of the samples after the tribology test: (a) SU-8 at 2 N, (b) BN at 2 N, (c) BNSN at 2 N, (d) BNFY at 2 N, (e) BNFY at 4 N, and (f) BNFY at 6 N. All tests were carried out at a constant sliding speed (0.28 m/s).

At a lower load (2 N), the BNSN composite coating contained lower initial and steady-state coefficients of friction; however, complete removal of the coating occurred. The lower coefficient of friction occurred because of the formation of a lubrication film at the interface. Furthermore, at the lower load, the BNFY composite coating showed negligible wear marks on the surface (Fig. 10(d)) after 4×10^5 cycles. This could be because of the high elastic modulus and *in-situ* lubrication from the PFPE lubricant inside the matrix. With a further increase in the load from 2 to 4 N, small wear marks were shown in the BNFY composite coating; however, the coating was sustained up to 4×10^5 cycles. The wear marks were less because of the lubrication film and texture formation on the coating surface, which reduced the contact area. The surface of BNFY was also sustained at a higher load because this composite coating showed a higher elastic modulus and lower coefficient of friction. This phenomenon was also suggested in a previous study [8] using a different material, such as SU-8 with graphite and a PFPE lubricant. When the load was increased from 4 to 6 N, wear on the coating was present because of the high contact stress (~ 1.5 times greater) at the interface; however, the wear track was smooth owing to the *in-situ* lubrication at the interface.

For confirmation of the wear phenomenon, ball interface images were obtained using a microscope, as shown in Figs. 11 (a)–1(f). Figure 11(a) shows that the coating was delaminated because of brittle fracture, and the delaminated coating parts are represented

by an arrow. Furthermore, the BN composite coating showed initial adhesive wear followed by abrasive wear (Fig. 11(b)). However, the BNSN composite demonstrated ploughing of the coating and caused ploughing wear (shown in Fig. 11(c)). Moreover, from Fig. 11(d), a large number of liquid molecules of PFPE in the form of droplets were present at the interface, providing *in-situ* lubrication at the interface. Therefore, wear of the material did not occur. When the load increased from 2 to 4 N, the quantity of liquid lubricant was reduced because of the high heat generated at the interface by increasing the load. This caused mild wear marks on the surface, as shown in Fig. 10(e).

Moreover, after increasing the load from 4 to 6 N, adhesive and abrasive wear developed owing to the high contact pressure; however, these abrasive particles consisted of PFPE lubricant, which reduced the coefficient of friction at a higher load.

The wear test (also known as a long-term friction test) was performed on SU-8 and its composites until failure of the coating or a maximum 4×10^5 sliding cycles. The wear-rate of the coating was calculated by Eq. (1), explained in experiment section, and the data with the SEs are shown in Fig. 12.

From Fig. 12, at a lower load, the BNFY composite showed lower wear resistance than that of pure SU-8 and other composite coating materials. The lower wear rate of the BNFY composite was owing to the *in-situ* lubrication film at the interface (Figs. 9 and 10(d)), higher elastic modulus, and hydrophobic nature of the coating surface. After increasing the load at the

Fig. 11 Portable microscope images of the ball after the tribology test: (a) SU-8 at 2 N, (b) BN at 2 N, (c) BNSN at 2 N, (d) BNFY at 2 N, (e) BNFY at 4 N, and (f) BNFY at 6 N. All tests were carried out at a constant sliding speed (0.28 m/s). The scale is 0.5 mm in each image.

Fig. 12 Wear rate of SU-8 and its composites until failure of the coating at different normal loads and a constant 0.28 m/s sliding speed.

interface, the wear rate slightly increased owing to an increase in the contact pressure at the interface, which increased the frictional heating at the interface. Furthermore, with an increase of the load from 4 to 6 N, the formation of the *in-situ* lubrication film for the BNFY composite at the interface was reduced, which caused abrasive wear and changed the wear phenomenon from two body to three body. This increased the wear rate by ~ 19 times that of the same coating materials tested at a 2 N load. Therefore, at a lower load (2 N), the BNFY composite showed a high

wear resistance property, greater than 10^3 times that of pure SU-8.

4 Conclusions

From this study, the following conclusions are drawn.

1. After adding the fillers, textures were generated on the surface, which changed the surface from hydrophilic to hydrophobic.

2. The thermal stability was improved by 80–120 °C owing to the bonds formed between SU-8 and the fillers. Moreover, the hardness was ~ 3 times higher and the elastic modulus by ~ 2.5 times higher than those of pure SU-8.

3. For the friction test, at a lower load (2 N), the composite fillers showed a lower coefficient of friction than that of pure SU-8. However, after adding h-BN, the initial and steady-state coefficients of friction were reduced by ~ 3 times and ~ 2 times, respectively. Moreover, after adding lubricant fillers with h-BN, the initial and steady-state coefficients of friction were reduced by ~ 8 times and ~ 9 times, respectively.

4. At a higher load, the BNFY composite yielded lower initial and steady-state coefficients of friction than those of pure SU-8.

5. From the wear test, the BNFY composite showed a high wear resistance property, 10^3 times greater than that of pure SU-8. However, after adding the fillers, delamination of the coating was reduced, and a smooth

wear track was formed. In addition, the BNFY composite showed negligible wear marks on the surface after 4×10^5 cycles at a low load (2 N).

The SU-8 composite with 10 wt.% BN and 20 wt.% PFPE lubricant provided a low coefficient of friction, high wear resistance property, and high wear life at low loads. Moreover, the composite coating improved the mechanical and surface properties. Therefore, the composite coating could be useful for bearing applications and MEMS fabrication.

Furthermore, this work could be extended to optimize the coating thickness for lower load and higher load applications.

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