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DLC nano-dot surfaces for tribological applications in MEMS devices

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ABSTRACT

With the invention of miniaturized devices like micro-electro-mechanical systems (MEMS), tribological studies at micro/nano-scale have gained importance. These studies are directed towards understanding the interactions between surfaces at micro/nano-scales, under relative motion. In MEMS devices, the critical forces, namely adhesion and friction restrict the smooth operation of the elements that are in relative motion. These miniaturized devices are traditionally made from silicon (Si), whose tribological properties are not good. In this paper, we present a short investigation of nano- and micro-tribological properties of diamond-like carbon (DLC) nano-dot surfaces. The investigation was undertaken to evaluate the potential of these surfaces for their possible application to the miniaturized devices. The tribological evaluation of the DLC nano-dot surfaces was done in comparison with bare Si (1 0 0) surfaces and DLC coated silicon surfaces. A commercial atomic force microscope (AFM) was used to measure adhesion and friction properties of the test materials at the nano-scale, whereas a custom-built micro-tribotester was used to measure their micro-friction property. Results showed that the DLC nano-dot surfaces exhibited superior tribological properties with the lowest values of adhesion force, and friction force both at the nano- and micro-scales, when compared to the bare Si (1 0 0) surfaces and DLC coated silicon surfaces. In addition, the DLC nano-dot surfaces showed no observable wear at the micro-scale, unlike the other two test materials. The superior tribological performance of the DLC nano-dot surfaces is attributed to their hydrophobic nature and the reduced area of contact projected by them.

1. Introduction

Over the last decade, the field of MEMS has expanded considerably, catering to a wide variety of applications ranging from industrial, consumer, defense, aerospace, to biomedical applications [1]. MEMS devices can be categorized into two main classes, namely: (i) sensors and (ii) actuators. Sensors-based MEMS have only sensing elements, whereas the actuators-based MEMS have elements that undergo relative mechanical motion. Sensors-based MEMS devices are commercially available. However, the actuators-based MEMS have not been commercialized yet, owing to the fact that there exist no robust tribological solutions at micro/nano-scale which can make the devices operate smoothly and reliably. For the smooth functioning of the elements in the actuators-based MEMS, the contact between them must be properly lubricated. But lubrication becomes difficult when the dimensions of machine elements are in micro/nano-scale. At the micro/nano-scale, the surface-area-to-volume ratio is very large. Due to this, the surface forces such as adhesion and friction restrict the smooth operation and reliability of the actuator-based MEMS. In order to reduce these surface forces, solid lubricants (such as graphite or MoS_2) cannot be used as they are about the same size as those of the MEMS elements [2]. In addition, conventional liquid lubricants can also be hardly used, as they generate liquid-mediated adhesion [3]. Thus, minimizing the surface forces at small-scales is a real challenge.

Miniaturized devices are traditionally made from silicon (Si) due to the well established designing and fabrication processes related to the IC technology. However, Si does not have good tribological properties [4,5]. In order to improve the tribological properties of Si, researchers world-wide have resorted to a variety of surface modification techniques such as chemical and topographical modifications [5]. Chemical modification includes thin films/coatings such as self-assembled monolayers (SAMs) and DLC coatings [5–8]. Topographical modification includes roughening of surfaces [9] and surface texturing/patterning [10–14].

In this paper, we report a short investigation on the nano- and micro-tribological properties of DLC nano-dot surfaces. The aim of the investigation was to examine the suitability of the surfaces for tribological applications in MEMS devices.

2. Test specimens and experimental details

The DLC nano-dot surfaces were created by depositing DLC films on nano-sized nickel (Ni) dots fabricated on Si (1 0 0) wafers by

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Table 1
Water contact angle values of the test specimens.

Test material	Water contact angle (degrees)
Bare Si (1 0 0) flat surfaces	22
DLC coated silicon flat surfaces	78
DLC nano-dot surfaces	95

annealing Ni thin films. As the first step, Ni thin films (thickness ~9 nm) were deposited on Si (1 0 0) wafers by DC magnetron sputtering at room temperature. Next, the Ni thin films were converted into nano-sized Ni dots by annealing in a rapid thermal process furnace at 800 °C for 15 min in pure hydrogen atmosphere. As the final step, DLC films (thickness ~100 nm) were deposited by radio-frequency plasma assisted chemical vapor deposition method (bias voltage: −150 V, deposition pressure: 1.33 Pa) using methane gas. More details on the fabrication process and material characterization can be found in Refs. [15,16]. Representative scanning electron microscope (SEM) images of DLC nano-dot surfaces are shown in Fig. 1(a) top view and (b) side view. Fig. 1(c) shows an atomic force microscope (AFM) image of the DLC nano-dot surfaces (scan size ~2 μm × 2 μm, data scale ~100 nm), taken using a commercial AFM.

Tribological investigation at nano- and micro-scales were conducted on test materials that included bare Si (1 0 0) flat surfaces, DLC coated silicon flat surfaces and the DLC nano-dot surfaces. At the nano-scale, adhesion force and friction force were measured using an atomic force microscope (Multimode SPM, Nanoscope IIIa, Digital Instruments). A borosilicate ball (diameter ~1.25 μm) mounted on cantilever (Contact Mode type, nominal spring constant 0.58 N/m) was used as the tip. Adhesion measurements were conducted in the force-displacement mode. Tests were performed for 20 times and the average values were plotted. Friction force was measured in LFM (Lateral Force Microscope) mode (applied normal load ~0–120 nN, sliding speed ~2 μm/s, scan size ~5 μm × 5 μm). Each test was repeated for about 20 times and average values were plotted. At the micro-scale, friction was measured using a soda lime ball (radius ~0.5 mm, Duke Scientific Corporation) under reciprocating motion (applied normal load ~3000 μN, sliding speed ~1 mm/s, scan length ~3 mm) using a custom-built ball-on-flat type micro-tribotester [4,5,7,12]. Each test was conducted for about 15 min and the steady state values were recorded. Tests were repeated more than five times and the average values were plotted. All experiments were conducted at controlled temperature of 24 ± 1 °C and relative humidity of 45 ± 5%. Static water contact angles were measured for all the test materials using the sessile drop method, and the values are given in Table 1.

3. Results and discussion

Fig. 2 shows the adhesion force values at the nano-scale of the test materials, namely the bare Si (1 0 0) flat surfaces, DLC coated silicon flat surfaces and the DLC nano-dot surfaces. The error bars indicate the minimum and maximum values of adhesion force observed from the test results. It could be observed from the figure that the DLC nano-dot surfaces exhibit adhesion force values that are significantly lower than those of the bare Si (1 0 0) flat surfaces and DLC coated silicon flat surfaces. Under tribological contact, adhesion force is caused due to various forces such as capillary, electro-static and van der Waals forces [3]. Amongst these forces, the capillary force, which occurs as a result of water condensation from the environment that leads to the formation of meniscus bridge, is the strongest [3]. Eq. (1) gives the expression for the adhesive force generated due to the formation of meniscus bridge [4].

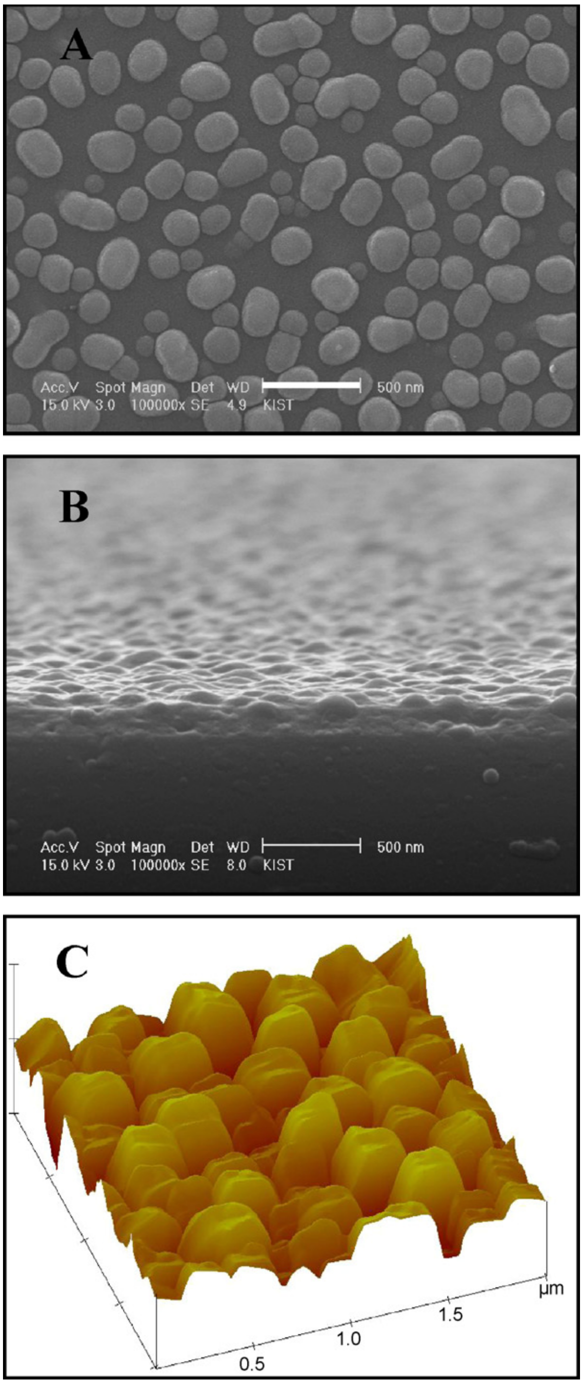

Fig. 1. Representative scanning electron microscope (SEM) images of DLC nano-dot surfaces: (a) top view (scale bar ~500 nm) and (b) side view (scale bar ~500 nm). (c) A representative atomic force microscope (AFM) image of the DLC nano-dot surfaces (scan size ~2 μm × 2 μm, data scale ~100 nm), taken using a commercial AFM.

$$F_m = 4\pi R\gamma \cos\theta \quad (1)$$

where R is the tip radius, γ the surface tension of water and θ the contact angle of water.

In the present case, the bare Si (1 0 0) flat surfaces show the highest values of adhesion force, owing to their hydrophilic nature (lowest water contact angle value, Table 1), which supports strong capillary force (Eq. (1)) [4]. The DLC coated silicon flat surfaces exhibit lower values of adhesion force than the bare Si (1 0 0) flat surfaces due to the increased water contact angle value (Table 1),

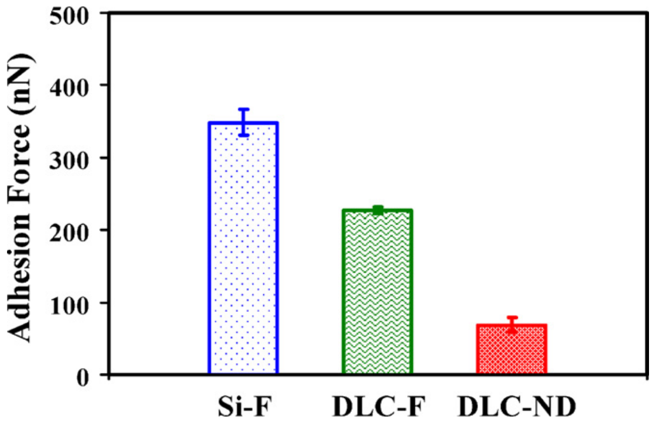

Fig. 2. Adhesion force of the test materials at the nano-scale. The DLC nano-dot surfaces (DLC-ND) exhibit adhesion force values that are significantly lower than those of the bare Si (1 0 0) flat surfaces (Si-F) and DLC coated silicon flat surfaces (DLC-F).

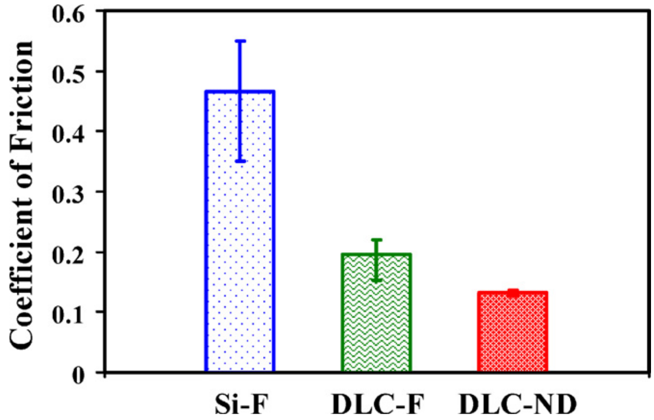

Fig. 4. Coefficient of friction of the test materials at the micro-scale, estimated as the ratio of the measured friction force to the applied normal load. The coefficient of friction values of the DLC nano-dot surfaces (DLC-ND) are the lowest when compared to the values of the bare Si (1 0 0) flat surfaces (Si-F) and DLC coated silicon flat surfaces (DLC-F).

which suppresses the capillary force. Further, the DLC nano-dot surfaces show the lowest values of adhesion force as a result of their hydrophobic nature (water contact angle value > 90°, Table 1), which suppresses the capillary force to a large extent. Under such circumstances, where the capillary force is unlikely to dominate the adhesion force, the weaker van der Waals forces would contribute to the adhesion force [3,4,17]. In the case of DLC nano-dot surfaces, the adhesion force decreases due to their hydrophobic property and also due to the reduced real area of contact. Reduction in adhesion force due to the increase in water contact angle value and due to the decrease in the real area of contact at the nano-scale has been observed earlier in the case of patterned surfaces [11,12].

Fig. 3 shows the friction force values of the test materials as a function of applied normal load at the nano-scale. It could be seen from this figure that for all the test materials, friction force exists even at the zero applied normal load. This is attributed to the influence of the inherent adhesive force on the friction force [4,5]. Friction at the nano-scale is in a regime where the contribution from inherent adhesion can outweigh those from the asperity deformation [3]. As mentioned earlier, the adhesive force is largely dominated by the capillary force that is the strongest force amongst the various forces that contribute. The bare Si (1 0 0) flat surfaces being hydrophilic in nature results in high inherent adhesion, which eventually causes high friction force. In contrast to the bare Si (1 0 0) flat surfaces, the DLC coated silicon flat surfaces and

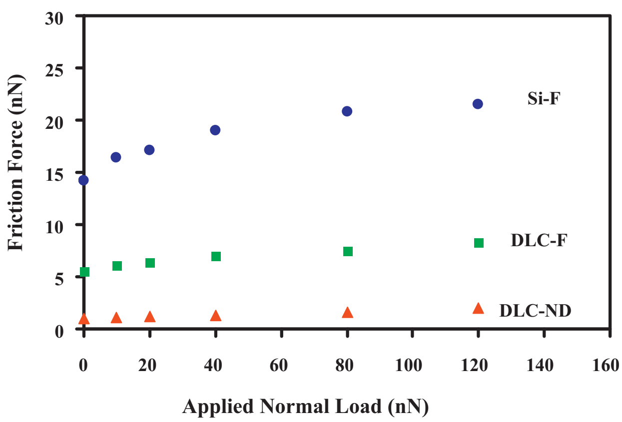

Fig. 3. Friction force of the test materials as a function of applied normal load at the nano-scale. The friction force values of the DLC nano-dot surfaces (DLC-ND) are significantly lower than those of the bare Si (1 0 0) flat surfaces (Si-F) and DLC coated silicon flat surfaces (DLC-F).

the DLC nano-dot surfaces have higher water contact angle values and hence, capillary force gets suppressed to a large extent in these materials, which lowers their inherent adhesion values. This eventually, reduces their friction force values. This phenomenon is more pronounced in the case of the DLC nano-dot surfaces as they are hydrophobic in nature (water contact angle value > 90°).

From Fig. 3, it could also be observed that the friction force increases with the applied normal load for the test materials. According to the fundamental law of friction given by Bowden and Tabor [18], for a single asperity contact, the friction force is given by the shear strength times the real area of contact between the surfaces. Eq. (2) gives the expression for the friction force [18].

$$F_f = \tau A_r \tag{2}$$

where, τ is the shear strength, an interfacial property and A_r the real area of contact.

The contact area can be estimated using contact mechanics models such as Hertzian or Johnson–Kendall–Roberts (JKR) models [9,19]. According to the JKR model (Eq. (3)) [19], the contact area is dependent on various parameters such as applied normal load, the size of tip and the interfacial energy of the materials.

$$A_r = \pi \left[\frac{R}{K} (F_n + 6\pi\gamma R + [12\pi\gamma R F_n + (6\pi\gamma R)^2]^{1/2}) \right]^{2/3} \tag{3}$$

where, R is the size of the tip, K the effective elastic modulus, F_n the applied normal load and γ, the interfacial energy of the material. The contact area according to the Hertzian model is given by the first term of Eq. (3) [9].

From Eq. (2), it could be seen that the friction force (F_f) depends directly on the contact area (A_r). Further, from Eq. (3), it could be seen that the contact area (A_r) in turn depends directly on the applied normal load (F_n). Therefore, any increase in the applied normal load (F_n), would increase the contact area (A_r), which in turn would increase the friction force (F_f). This explains for the increase in the friction force (F_f) with the increase in the applied normal load (F_n) for the test materials, as seen from Fig. 3. Amongst the test materials, the bare Si (1 0 0) flat surfaces show the highest values of friction force. This could be understood by considering the JKR model that includes the influence of the interfacial energy on the contact area (Eq. (3)). The bare Si (1 0 0) flat surfaces have high interfacial energy indicated by their lower water contact angle value (~22°) (cosine of water contact angle is a measure of surface energy [20]) and therefore their friction force values are high due to the high values of real area of contact [4,5]. Thus, the hydrophilic nature of the bare Si (1 0 0) flat surfaces supports high values of inherent

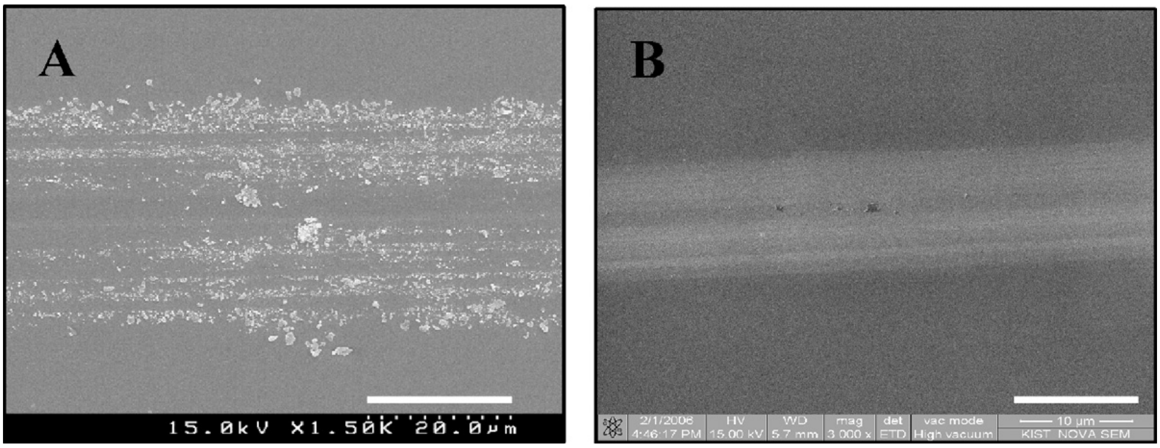

Fig. 5. Representative SEM images of the worn surfaces of (A) bare Si (1 0 0) flat specimen (scale bar ~20 µm) [4,5,14] and (B) DLC coated silicon flat specimen (scale bar ~10 µm) [5,8,14].

adhesion and also high values of real area of contact, thereby giving rise to high values of friction force.

The DLC coated silicon flat surfaces have lower interfacial energies, as indicated by their higher values of water contact angle (Table 1), due to which they exhibit lower friction forces owing to lower inherent adhesion and lower contact areas [5]. The DLC nano-dot surfaces show the lowest values of friction force due to their hydrophobic nature and also due to the reduced real area of contact projected by them. Reduction in friction force due to the increase in water contact angle value and due to the decrease in the real area of contact at the nano-scale has been observed earlier in the case of patterned surfaces [5,11,12]. Thus, in the present case, amongst the test materials, the DLC nano-dot surfaces exhibited superior tribological properties at the nano-scale with the lowest values of adhesion and friction forces.

Fig. 4 shows the results of the friction tests at the micro-scale. It gives the values of the coefficient of friction of the test materials at the micro-scale, estimated as the ratio of friction force to the applied normal load. It could be seen from the figure that the coefficient of friction values of the DLC nano-dot surfaces are the lowest when compared to the values of the bare Si (1 0 0) flat surfaces and DLC coated silicon flat surfaces. It is well known that the friction force is greatly influenced by the contact area even at the micro-scale [5,12,14]. Supported by the higher interfacial energy, large contact areas cause higher values of coefficient of friction in the case of the bare Si (1 0 0) flat surfaces [4,5,14].

The DLC coated silicon flat surfaces show lower values of coefficient of friction in comparison with the bare Si (1 0 0) flat surfaces owing to lower contact areas that reduce the values of friction coefficient [4,5,14]. The coefficient of friction values of the DLC nano-dot surfaces are the lowest when compared to those of the other test materials, which is attributed to the reduced real area of contact projected by the surfaces brought forth through physical reduction. Further to note, unlike at the nano-scale that is almost a 'wear-less' situation [4,5], wear was observed in the test materials at the micro-scale (except in the case of DLC nano-dot surfaces), which influenced their friction behavior. Formation of wear particles (debris) at the interface during sliding increases the overall value of the friction by the contribution of the plowing component [14,21–23] (friction force comprises of two components, namely adhesive and plowing [24]). Considering the case of the bare Si (1 0 0) flat surfaces, the influence of the wear debris on their friction property is another reason for the values of friction coefficient to be high at the micro-scale [14,22]. Fig. 5(a) shows the worn surface of a bare Si (1 0 0) flat specimen, where large amounts of wear debris are seen on the wear track [4,5,14]. Compared to the wear of the bare Si (1 0 0) flat surfaces, the DLC coated flat surfaces show fewer wear particles (Fig. 5(b)) [5,8,14], due to which the contribution of the plowing effect by the debris is reduced to a large extent, thereby lowering the overall value of the coefficient of friction. Amongst the test materials, the best performance is exhibited by the DLC nano-dot surfaces, which not only show the lowest values of coefficient of friction, but also exhibit no observable wear at the micro-scale.

In the context of the application of DLC surfaces to MEMS devices, thin DLC coatings have been directly tested for their performance in the MEMS device of electrostatic lateral output motor [25]. It was found that the DLC coated MEMS outperformed uncoated MEMS by about sixteen times in air and showed about a three hundred times increase in performance over the uncoated device in vacuum [25]. In the present investigation, it has been observed that the DLC nano-dot surfaces greatly improve the tribological performance when compared to the bare Si (1 0 0) flat surfaces and also when compared to the DLC coated silicon flat surfaces, both at the nano- and micro-scales. Looking at the superior tribological properties of the DLC nano-dot surfaces both at the nano- and micro-scales, it could thus be anticipated that in using the DLC nano-dot surfaces instead of DLC coatings, the tribological performance of the silicon based MEMS devices could be enhanced significantly.

4. Conclusions

In this short investigation, we have evaluated the nano- and micro-tribological properties of DLC nano-dots surfaces, in comparison with bare Si (1 0 0) flat surfaces and DLC coated silicon flat surfaces. The DLC nano-dot surfaces exhibited superior tribological properties than the rest of the test materials. They showed lowest values of adhesion, and friction at nano- and micro-scales. Further, they exhibited no observable wear at the micro-scale, unlike the other test materials. It was identified that their hydrophobic nature and the reduced real area of contact projected by them were the main reasons for their superior performance. We envision that the DLC nano-dot surfaces such as the ones investigated in the current work would have potential applications in miniaturized devices as tribological candidates.

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