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Wear of silicon surfaces in MEMS

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ABSTRACT

High levels of friction and wear are problems which currently limit the development of sliding micro-electro-mechanical systems (MEMS) – devices which would otherwise offer significant technological advancement. The current paper focuses on the wear of MEMS silicon surfaces, and specifically looks at the effect of environment and surface preparation on wear behaviour. Included in the study is the assessment of two self-replenishing lubrication mechanisms; namely liquid and vapour phase lubrication. All tests were carried out using a tribometer which operated and measured friction and wear under conditions representative of MEMS.

It is shown that friction and wear behaviour depend strongly on subtle changes of the silicon surfaces prior to testing. Greatest wear was measured when the surfaces were tested immediately after plasma-cleaning, while subsequent exposure to ambient air for 15 h reduced wear to negligible levels. Exposure of plasma-cleaned surfaces to water-saturated argon prior to testing prevented wear to a limited extent. Based on this, and TOF-SIMS analysis, it is suggested that the observed wear reduction after exposure to air is caused by tiny amounts of lubricious long chain hydrocarbon contaminants present in ambient air.

Tests carried out with the specimens submerged in a liquid bath show that the presence of liquid water reduces friction and wear, but only if specimens have been plasma-cleaned beforehand. This behaviour is tentatively attributed to the hydrophilic nature of plasma treated silicon, reducing the corrosive action of water. When hexadecane or 1-pentanol was used as a liquid lubricant, friction was minimal, and wear was undetectable under all sliding conditions. This was the case even though the contact operated in the mixed lubrication regime, suggesting a boundary film is formed on the silicon surfaces by both of these organic liquids.

Results of tests carried out with the lubricant being supplied in the form of pentanol vapour also showed no appreciable wear. A considerable difference in friction was found between liquid and vapour lubricated contacts; under the conditions tested, the coefficient of friction for vapour was 0.28, while for liquid it was 0.05.

1. Introduction

Micro-electro-mechanical systems (MEMS) are a growing technology, with great potential to integrate sensors, actuators, and signal processing into miniature devices [1]. Based on developments in micro-electronics, production of such devices can offer large volumes, under tight tolerances, and at low cost. A well known obstacle to realising these benefits is the dominance of surfaces forces that occurs when length scales are miniaturized. Furthermore, silicon – the most commonly used material to fabricate MEMS due to its cost and good mechanical properties – is not naturally lubricious and suffers from low toughness. As a result, friction, stiction and wear are significant problems in MEMS. While the level of friction determines the operating efficiency and whether moving parts will function, wear will determine the reliability and stability of the device.

Many micromachine surfaces are impossible or impractical to protect from their operating environment [1]. As a result, not only water vapour, but also other airborne contaminants inevitably interact with component surfaces to form adhered films, which control tribological behaviour. It is therefore clearly of practical benefit to understand the link between ambient conditions, both during operation and prior storage, and the resulting tribological behaviour of the silicon MEMS surfaces. Despite this, there appears to have been very limited research into the effect of environment on the tribology of MEMS surfaces [1].

This paper describes a series of tests carried out to quantify the wear behaviour of sliding silicon surfaces within a MEMS contact. Particular attention is given to the relationship between the behaviour and the conditions of the surfaces both prior to, and dur-

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ing testing. Initially, a brief review of previous MEMS wear studies is given.

There are two main approaches to studying wear in MEMS, either an in situ measurement is made on an actual MEMS device (described in Section 1.1), or a test rig is used to simulate a MEMS contact (described in Section 1.2).

1.1. In-situ studies

The majority of in situ studies consisted of running a MEMS device and counting the number of cycles it completes before failure is detected [2,3]. An example of this is work by Henck et al., who assessed various storages of micro-mirror surfaces by measuring the time until stiction caused rotating part to cease functioning [2].

Due to the difficulty in measuring wear within small MEMS devices, there are only a few studies where in situ wear on the worn surfaces has been quantified [14,5,6]. One example is the study of a MEMS gear pair where wear was measured from the resulting variation in bearing clearances [4,5].

The second was by Tanner et al. [6], who studied the effect of humidity on the reliability of a microengine with connected gears. It was shown that perfluorodecyltrichlorosilane (FTS) coated motors had a longer wear life than those that were supercritically dried. Wear was quantified for a series of tests on gears coated with FTS at a range of humidities. It was found that higher humidity resulted in lower wear rate. At high humidity, hydroxides formed on the surfaces and acted as a lubricant.

1.2. MEMS tribometer tests

The most common method of simulating wear is to use a commercially available low load tribometer, typically a micro pin on disc tester [7–9]. The majority of such studies have been conducted in order to assess the merits of various potential surface coatings for MEMS, such as ceramic coatings, diamond-like carbon coatings (DLC) and hard metal coatings

Bhushan used a pin on disc tester to simulate the read/write head of a hard drive [7]. Pins of different materials were rubbed against a spinning disc, covered with either an amorphous DLC or a Teflon lubricating film. Friction and wear were shown to be dependent on either the transfer of carbon atoms from the disc to the pin, or on the fracture of the pin. The presence of the Teflon lubricating film delayed tribochemical reactions and transfer of carbon atoms at the interface, and hence the degradation of the interface. Bandorf used a pin on disc setup to investigate the friction and wear of various DLC coatings deposited on substrates of varying hardness [8]. It was found that the friction coefficient was load dependent at the microscopic scale, and friction was greatly reduced when a substrate of high elastic modulus was used. It was also shown that wear resistance was increased by the use of a hard coating on top of a soft substrate material. Haseeb used a commercially available pin on disc tribometer with a steel ball on flat (test surface) setup to investigate the effect of the tungsten content in Ni-W alloys on friction and wear [9]. Here it was found that pure nickel films sustained more damage than nickel–tungsten alloy films. Adhesive and oxidation wear mechanisms were found to be operative under these conditions, accompanied by appreciable material transfer from the ball to the substrate.

In addition to those available commercially, a number of tribometers have been custom built specifically for simulating MEMS conditions. Alsem et al. [10] used a set up known as the "MEMS sidewall friction device". This device involves two comb drive actuators, one to produce a reciprocating motion and the other to apply a normal load. It was shown that debris particles were produced by adhesion, which subsequently ploughed into to sliding surfaces. The extent of wear was not quantified. Asay et al. also conducted linear wear tests with a MEMS sidewall friction device to assess the effectiveness of vapour phase lubrication with pentanol [11,12]. It was found that, by maintaining a certain vapour pressure of the lubricant, surface film replenishment was sufficient to reduce friction and to limit wear. Under such conditions, wear was prevented by the formation on the surfaces of a high molecular oligomer species produced by tribochemical reactions in the contact regions. Patton et al. employed a set up known as the "MEMS electrostatic lateral output motor" [13,14]. This involves the slider (ball) reciprocating against a flat surface. It was found that the wear mechanism in dry air was different to that in vacuum, due to the former condition allowing for the regeneration of the native oxide film present on the silicon surfaces. Smallwood et al. [15] used a similar ball-on-flat set up to study the wear performance of MEMS coated with diamond-like carbon (DLC) coatings. Here it was found that the coated MEMS devices always performed better than those that were uncoated, in terms of friction, wear and lifetime. The coated devices also performed better at higher relative humidities. Most severe wear was observed for uncoated MEMS surfaces sliding in a vacuum, due to high adhesion forces and cold welding.

Suzuki et al. used a test rig similar to a typical pin-on-disc set up, but, by replacing the pin with a cylindrical "rider", a line, rather than point contact was produced [16]. This was done in order to simulate the contact within a micro-motor between shaft and rotor. In this case, DLC coatings and silicone liquid lubricants were studied. A further example where a MEMS tribometer has been used to study coatings is in research carried out by Beerschwinger et al. [17]. Here, it was found that DLC sliding on DLC showed the lowest wear rate, while the wear exhibited by silicon nitride was higher by three orders of magnitude. Under these conditions, two possible wear mechanisms were identified: asperity fracture or asperity deformation.

Gatzen and Beck [18] studied the effect of surface roughness on wear for a silicon-on-silicon micro-contact. This was achieved by ion milling raised studs in a Si crystal, which was then loaded against a rotating silicon disc. By measuring the reduction in height of the studs, it was shown that greatest wear was produced by surfaces with high initial roughness.

Generally, the pin-on-disc studies outlined above have been useful in proving the benefit of various dry surface coatings. However, less attention has been paid to the study of uncoated silicon surfaces and the effect of atmospheric conditions on their wear behaviour. Additionally, a minor shortcoming of the tribometers used in some of the above tests is that the contact size is usually larger than those present in actual devices. Therefore in order to achieve the correct contact pressure, the load must be scaled up. It is possible that this will affect the measured wear.

1.3. Self replenishing lubrication techniques

The coatings that are commonly proposed for MEM surfaces provide a solid protective layer to resist wear during sliding. However, once worn away, such a coating cannot replenish itself, so that any finite rate of wear will ultimately lead to loss of the coating. This is unacceptable for MEMS components which experience large sliding distances and this limitation incentivises the investigation of self replenishing lubrication mechanisms to reduce wear or even prevent in MEMS devices. Two self replenishing lubrication mechanisms have been investigated to date, vapour phase technique [11,12] described above, and fluid film lubrication. The latter is very effective at the macro-scale but has largely been ignored for MEMS devices. Generally this is due to the assumption that MEMS device operation will be limited by the presence of excessive viscous drag forces. For this reason, there have been very few studies where liquid lubrication has been applied to MEMS devices [16]. However, the recent development of a new MEMS tribometer has shown that a hydrodynamic film can reduce friction to acceptably low levels, provided a lubricant of sufficiently low viscosity is employed [19]. Furthermore, friction modifier additives can be blended with liquid lubricants to reduce friction under conditions where the entrainment speed is insufficient to generate a hydrodynamic film [20]. Regarding the liquid lubrication of MEMS, it should be noted that, in addition to the friction within the contact, churning losses could arise if moving components are submerged within fluid lubricant. However, analysis shows these losses are significantly reduced when low viscosity fluids are used.

In this current work, the wear behaviour of uncoated silicon surfaces is studied. The low load and small contact area afforded by the newly developed tribometer allows friction and wear to be measured under conditions that are representative of MEMS. The aim is to gain understanding of the relationship between surface conditions and the mechanisms and extent of wear, and the comparative merits of both self-replenishing MEMS lubrication mechanisms.

2. Experimental details

These tests were carried out using a tribometer outlined in [19]. A contact is produced by loading and rotating a flat silicon disc against a stationary, patterned silicon disc. The stationary lower specimen is mounted on a flexible platform allowing very small normal and rotational displacements, both of which are monitored. The rotational displacement of the platform, measured using an optical lever technique, is used to determine the frictional torque experienced by the contact. The normal displacement of the platform, measured using a laser device is used to determine the applied load. Normal and torsional stiffnesses required to evaluate these forces are found from calibrations, details of which are given in Ref. [21].

The motor speed control and data acquisition processes were automated with the aid of LabVIEW software. Friction data was acquired at a rate of one measurement every 6 s, with each measurement consisting of an average of ‘raw’ data acquired over a 5 s period at a rate 50 data points per second.

The wear tests described in this paper consisted of loading the two disc specimens together at 0.05 N load, and rotating the upper specimen at 500 rpm, with frictional torque being continuously monitored. These conditions give an average sliding speed, V, of $0.052\,\mathrm{ms}^{-1}$ and a nominal contact pressure, p, of 8.47 kPa. Therefore, the contact can be considered as a thrust bearing, with a pV value of $4.43 \times 10^{-4}\,\mathrm{MPa\,m\,s}^{-1}$. The duration of each test was 90 min which corresponds to 45,000 cycles, and a sliding distance of 283 m. After the test, the upper specimen was removed and the surface measured using a Wyko optical surface profilometer. Since the lower specimen (shown in Fig. 1), consists of four raised pads around a recessed centre, contact with the upper specimen only occurs over part of the radius, with an unworn section always being present on the upper specimen. The unworn central region of the upper specimen could therefore be used to as a datum, against which the depth of wear could be measured.

2.1. Specimen details

The silicon disc specimens used in this study were 2 mm diameter, and were fabricated using a Deep Reactive Ion Etching (DRIE) technique. The structure of the stationary disc was equivalent to a miniature thrust pad bearing, as shown in Fig. 1.

2.2. Storage of specimens

All disc specimens were initially plasma cleaned before study. This involves oxygen plasma exposure for 15 min to remove all organic substances and water on the surfaces. During this cleaning, high energy plasma particles react with organic contaminants to form carbon dioxide or methane [22]. Temperatures above 200 °C are also present, which will remove most water or other contaminant molecules from the specimen surfaces. The plasma cleaner used was manufactured by Harrick Plasma, Ithaca, USA.

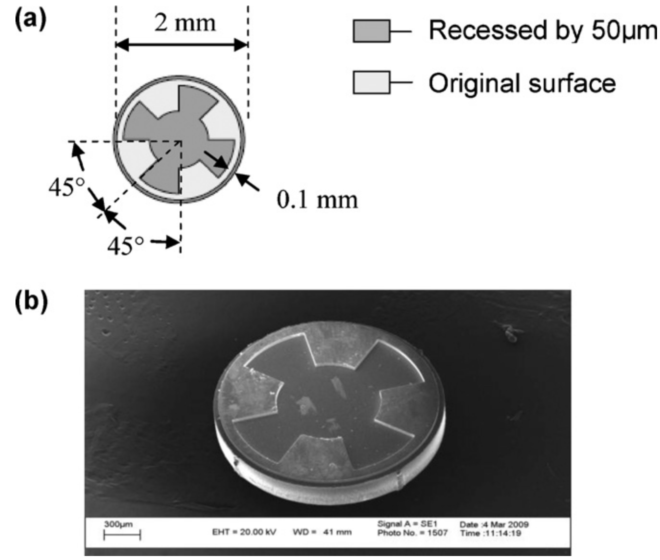

Fig. 1. Details of the lower pad specimen: (a) dimensions; (b) SEM image prior to testing.

After plasma cleaning, discs were treated in three ways prior to testing.

(i) “*Not stored*”: Some discs were tested immediately after plasma cleaning without further storage.
(ii) “*Water vapour stored*”: Once removed from the plasma cleaner, some specimens were left in a sealed desiccator for 15 h at room temperature (23 ± 2 °C). The desiccator contained a beaker of distilled water. Immediately the specimen had been placed inside, the chamber was pumped with argon. This ensured that the silicon specimen surfaces were exposed to water vapour and argon, but no other air-borne particles or vapours.
(iii) “*Air stored*”: Once removed from the plasma cleaner, some specimens were exposed to ambient laboratory air for 15 h. This allowed the possibility of contaminants from the air being deposited on the silicon surfaces.

2.3. Lubricants used

Tests were carried out under three different sets of conditions:

(i) “*Dry*”: These wear tests were carried out in ambient air atmosphere.
(ii) “*Submerged*”: A number of tests were run with the contact submerged in a liquid lubricant. This was achieved using small lubricant bath located around the specimens on the platform. The lubricants used were hexadecane ($C_{16}H_{34}$), deionised water, and 1-pentanol ($C_5H_{12}O$).
(iii) “*Vapour phase*”: For one set of tests, lubrication was constantly supplied in the vapour phase – following a technique similar to that of [11]. To do this, the contact was housed within a chamber and 1-pentanol saturated argon was pumped though. A diagram of the vapour phase set up is shown in Fig. 2.

A summary of the tests conducted in this study is outlined in Table 1. In all tests both the upper and lower discs had the same prior storage.

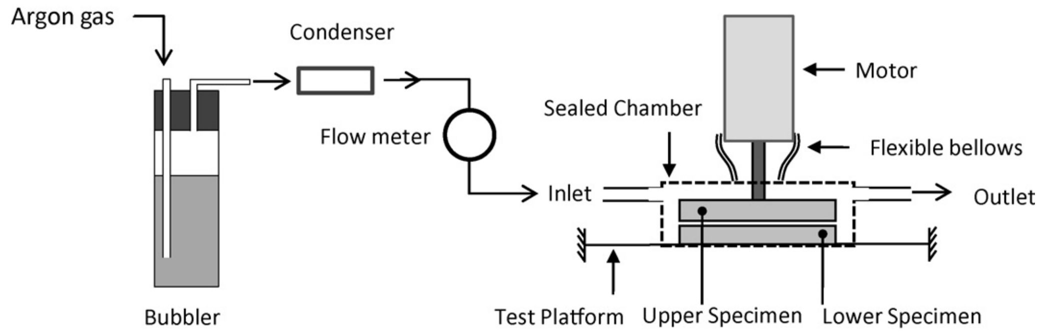

Fig. 2. Schematic diagram of vapour phase set up.

Table 1
Summary of tests conducted (l = liquid, v = vapour).

Description	Storage (after plasma cleaning)	Lubricant used
Not stored/dry	Not stored	Dry
Not stored/H_2O (l)	Not stored	Water liquid
Not stored/$C_{16}H_{34}$ (l)	Not stored	Hexadecane liquid
Not stored/$C_5H_{12}O$ (l)	Not stored	1-Pentanol liquid
Not stored/$C_5H_{12}O$ (v)	Not stored	1-Pentanol vapour
H_2O (v) stored/dry	Water vapour stored (desiccator)	Dry
Air stored/dry	Air stored	Dry
Air stored/H_2O (l)	Air stored	Water liquid

3. Results

Three tests were carried out at each condition listed in Table 1. Friction was monitored throughout each 90 min test, and wear was quantified once the test had finished.

3.1. Friction

Fig. 3 shows individual friction measurements from three repeat tests with the contact submerged with hexadecane. There is reasonable agreement between the three traces. Although conditions are kept constant, a variation in friction over time is evident within a test. If wear is present, this variation may be attributed to the change in surface topography and the evolution of debris in the contact (although no measurable wear occurred for the hexadecane test shown). For the test lubricated with hexadecane, the observed variation in friction with time is also possibly due to the formation a surface film on the silicon specimens, which varies throughout the test.

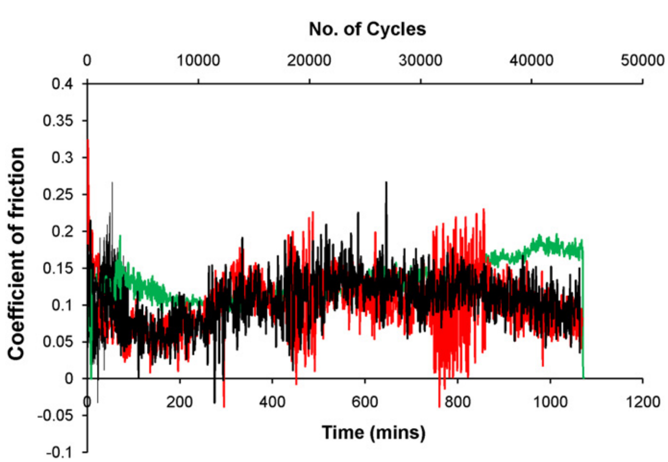

Fig. 3. Friction vs. rubbing time for three repeat tests with contact submerged in hexadecane not stored/$C_{16}H_{34}$ (l). Load = 0.05 N, rotational speed = 500 rpm.

For all subsequent results, friction will be given as the average values over the whole friction traces, showing the mean average value for the three tests at each test condition and also the spread of the averaged values at each test condition.

Fig. 4 shows the average friction coefficient for each test, with labels on *y*-axis corresponding to test conditions given in Table 1. Test results for each condition will be discussed separately later in this paper. For two of the test conditions, friction coefficient exceeded the maximum measurable value of 4.

3.2. Wear

Fig. 5 shows example wear profiles of upper specimen surfaces after rubbing. All profiles show a flat section at the central region of the pad (on the right in the figure) where no contact occurs with the lower specimen and thus no wear takes place.

In order to estimate the volume of material removed from the lower specimen during rubbing, the volume of revolution of each profile was calculated (integrated about the axis of rotation), and subtracted from that for the unworn case. Each wear volume found in this way was then used to calculate a wear coefficient K_b. This was done according to Archard's law, $K_b = V/xW$, where V is the volume of material removed, x is the sliding distance (283 m), and W is the load (0.05 N). The results are shown in Fig. 6.

4. Discussion

4.1. Dry tests

4.1.1. Dry tests with no storage after plasma cleaning – not stored/dry

In the tests under conditions identified as "not stored/dry", the samples were tested immediately after plasma storage with no liquid lubricant present. Friction results under these conditions are not displayed, since the values present were greater than the maximum measurable friction (above a coefficient of 4). It should be noted that the maximum measurable friction coefficient could be increased, if required, by fabricating a stiffer test platform, but this was not done in the current study.

Fig. 6 shows that under these conditions, extensive wear also occurred, with an average coefficient, K_b, of 0.0183 mm^3 N^{-1} m^{-1}. It is believed that the high wear rate results in damaging wear debris. This is a common problem experienced in MEMS [6,17], since debris is of a similar length scale to the size of the devices. The presence of debris is confirmed by the SEM images given in Fig. 7.

In previous unlubricated silicon-on-silicon tests carried out by the authors [19], a relatively low friction coefficient was observed. There are two reasons for the discrepancy between that study and

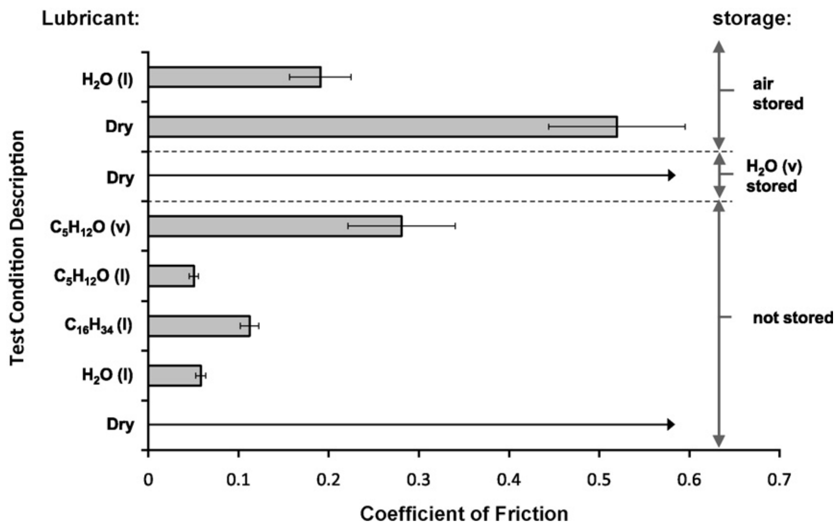

Fig. 4. Averaged friction coefficient for each test condition. In all tests load = 0.05 N, rotational speed = 500 rpm.

the current work. Firstly the preparation of samples for the previous study consisted only of washing in solvents, while the plasma cleaning preparation in the current work is likely to remove foreign material from the surface much more stringently. Also, the current tests were run for a considerably longer period of time (90 min rather than 11 min), so that wear debris might accumulate to impede the motion of the shaft, and result in considerably higher friction values.

Fig. 7 shows SEM images of the upper and lower specimen post test. The image showing the whole of the upper specimen (Fig. 7d), clearly indicates the worn and unworn regions which are compared to find the volume of material removed. The wear consists of concentric grooves, which suggest that wear is caused by abrasion or ploughing by wear debris particles (evident from Fig. 7e and f).

These particles are visible in Fig. 7a, where, due to the rotation of the upper specimen, they are visible at the furthest clockwise edge of the bearing land. The variation in size and shape of the debris particles can be seen from Fig. 7b and c.

From Fig. 7c (taken at the highest resolution of the SEM equipment), the debris can be seen in clusters, suggesting they agglomerate during sliding. The sizes of these clusters are approximately 3–20 μm in length. It is possible that the observed wear was initiated as a result of asperity contact between the two sliding surfaces. As suggested by Tanner et al., plastic flow occurs at these points and leads to cold welding and, eventually, to the asperities being broken off to form debris [6]. The debris is likely to be composed mainly of silicon oxide as indicated by EDS on similarly formed particles in other work [6]. Further evidence of this can be found in [10,13,14]. It is believed that debris is initially formed from the native oxide on the silicon surfaces. Subsequently, the "flash temperatures" caused by repeated sliding are sufficient to cause the underlying silicon to oxidise, which in turn is removed as debris.

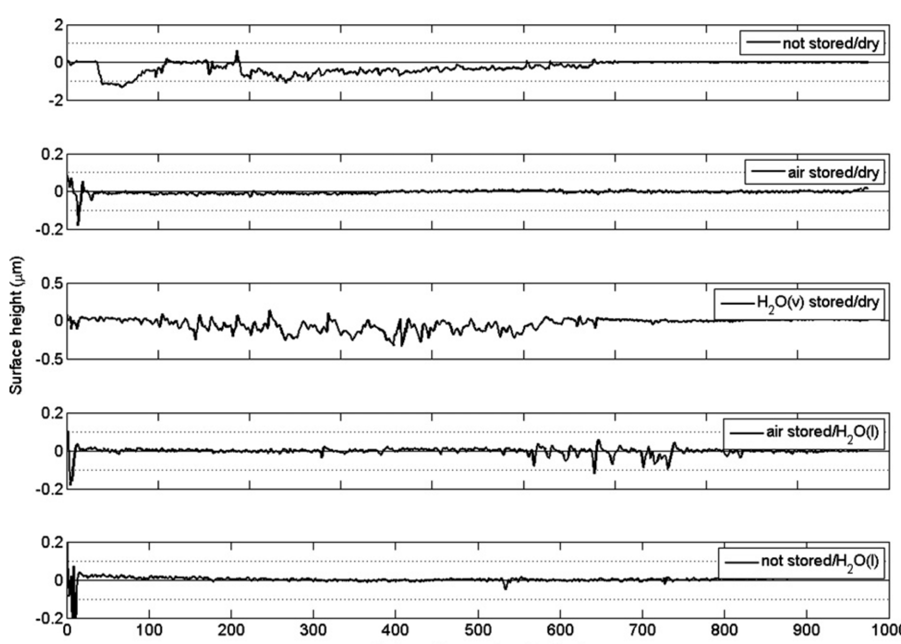

Fig. 5. Worn surface profiles for various test conditions. Note: scales on *y*-axis vary. In all tests load = 0.05 N, rotational speed = 500 rpm.

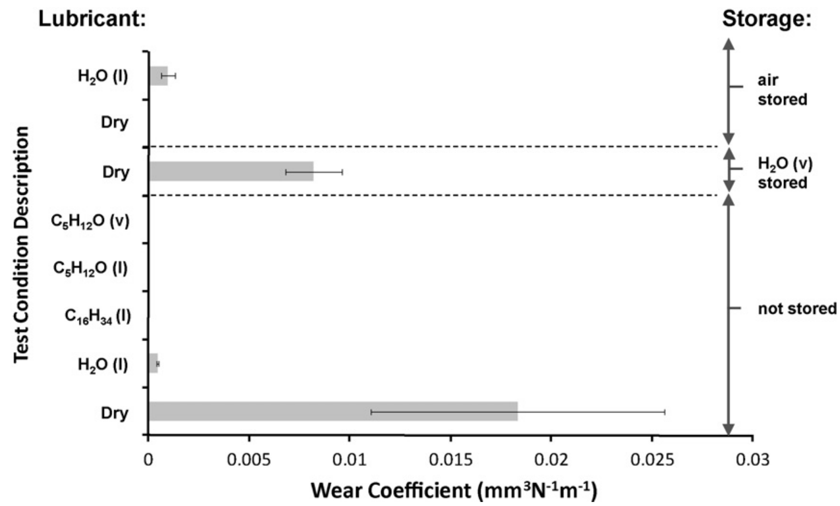

Fig. 6. Wear coefficient (K_b) for upper specimen at each test condition. Specimens with wear depth shown as zero (not stored, dry air and hexadecane (l)) had wear depths less than the minimum measurable value of 0.01 μm.

4.1.2. Dry tests after storage in air – air stored/dry

For this set of tests, the specimens were cleaned using the plasma cleaner but then exposed to ambient air for 15 h prior to testing. During these 15 h, it is likely that organic contaminant as well as water molecules from the air become adsorbed on the silicon surfaces.

Fig. 4 shows that the measured friction of the exposed samples (air stored/dry), are markedly lower than those samples which were tested immediately following plasma cleaning (not stored/dry). Fig. 6 shows that the corresponding wear volumes for the exposed samples are also much lower. In fact wear is too low to be measured using the optical profilometer, which can measure surface variation down to 0.01 μm.

The cause of the reduction in friction and wear after exposure to air is not immediately obvious, beyond the assumption that water vapour and/or other air borne contaminants have attached to the silicon surfaces to provide some form of lubricating film.

4.1.3. Dry tests after storage in water vapour – H_2O (v) stored/dry

In order to ascertain whether water vapour or some other air-borne species is responsible for the observed reduction in friction and wear, specimens were stored, after plasma cleaning, in a desiccator containing only water and argon gas. In this way, the specimens would be exposed to water vapour, while being free from all other air-borne contaminants.

As shown in Fig. 4, the friction coefficient recorded for these tests was extremely high, being above a maximum measurable value of 4. The only other condition, under which such high friction was observed were the tests run immediately after plasma cleaning (not stored/dry). The corresponding wear volumes, given in Fig. 6 show that significant wear is present, however, the average volume removed is only about half that when specimens were tested immediately after plasma cleaning.

Comparing results from the water vapour-stored specimens with both air-exposed specimens and immediately tested specimens reveals the following. Greatest wear protection results from exposure to air, showing that some species must be attaching to the silicon surfaces from air. Such lubricious contaminants cannot be solely water molecules; otherwise the friction and wear of desiccator prepared specimens would also be low. Some wear reduction is found in the desiccator stored specimens which suggests that adsorption water vapour affords some wear protection, but much less than that provided by other contaminants, or water molecules in combination with such contaminants.

Atmospheric water vapour is known to adhere to silicon surfaces by forming hydrogen bonds and results in films several monolayers in thickness [23]. However, the above results show that some other, important form of contamination must occur during the 15 h of exposure to room air. In an attempt to identify these contaminants, time-of-flight secondary ion mass spectroscopy (TOF-SIMS) was carried out on two sets of samples: (a) those that had been plasma treated immediately (<30 s) beforehand, and (b) those that had been exposed to room air for 15 h. TOF-SIMS analysis is sensitive to very low concentrations of surface species, and so can produce large quantities of detailed data. Therefore in order to differentiate between the two surfaces of interest, principle component analysis (PCA) was carried out on the TOF-SIMS results. PCA results showed two clear findings. Firstly, plasma cleaned surfaces produce higher concentrations of silicon ions. This observation is in keeping with the possibility that some surface film is covering the air-exposed sample. Secondly, the air-exposed specimens have considerably larger quantities of long chain organic molecules (comprising 4–14 carbon atoms). The TOF-SIMS concentration maps for $C_4H_{12}NO$ shown in Fig. 8a and b, give an example of this (though it should be noted that many other hydrocarbons were also found). The presence of these contaminants goes some way to explaining the greatly reduced friction and wear of the air-exposed silicon specimens. During cleaning, the oxygen plasma is likely to react with and remove such contaminants to form carbon dioxide. It appears that ambient air is providing a form of vapour phase lubrication, albeit somewhat uncontrolled. The origin of these contaminants is not obvious. Tests were carried out in a tribology laboratory in a major city (London, UK) and may originate from normal city atmospheric air or primarily from cleaning solvents or oil specimen samples present in the laboratory environment from other research.

4.2. Fluid lubricated tests

4.2.1. Tests with water lubrication – not stored/H_2O (l) and air stored/H_2O (l)

The tests carried out with the contact submerged in water are identified as "not stored/H_2O (l)" (surfaces were cleaned with oxygen plasma but not subsequently treated before being tested in liquid water), and "air stored/H_2O (l)" (surfaces treated with oxygen plasma then air exposed for 15 h prior to testing in water). As shown in Fig. 4, the average friction coefficient increases noticeably as a result of the plasma cleaned surfaces being exposed to air for 15 h. This is the converse of the behaviour seen in dry tests.

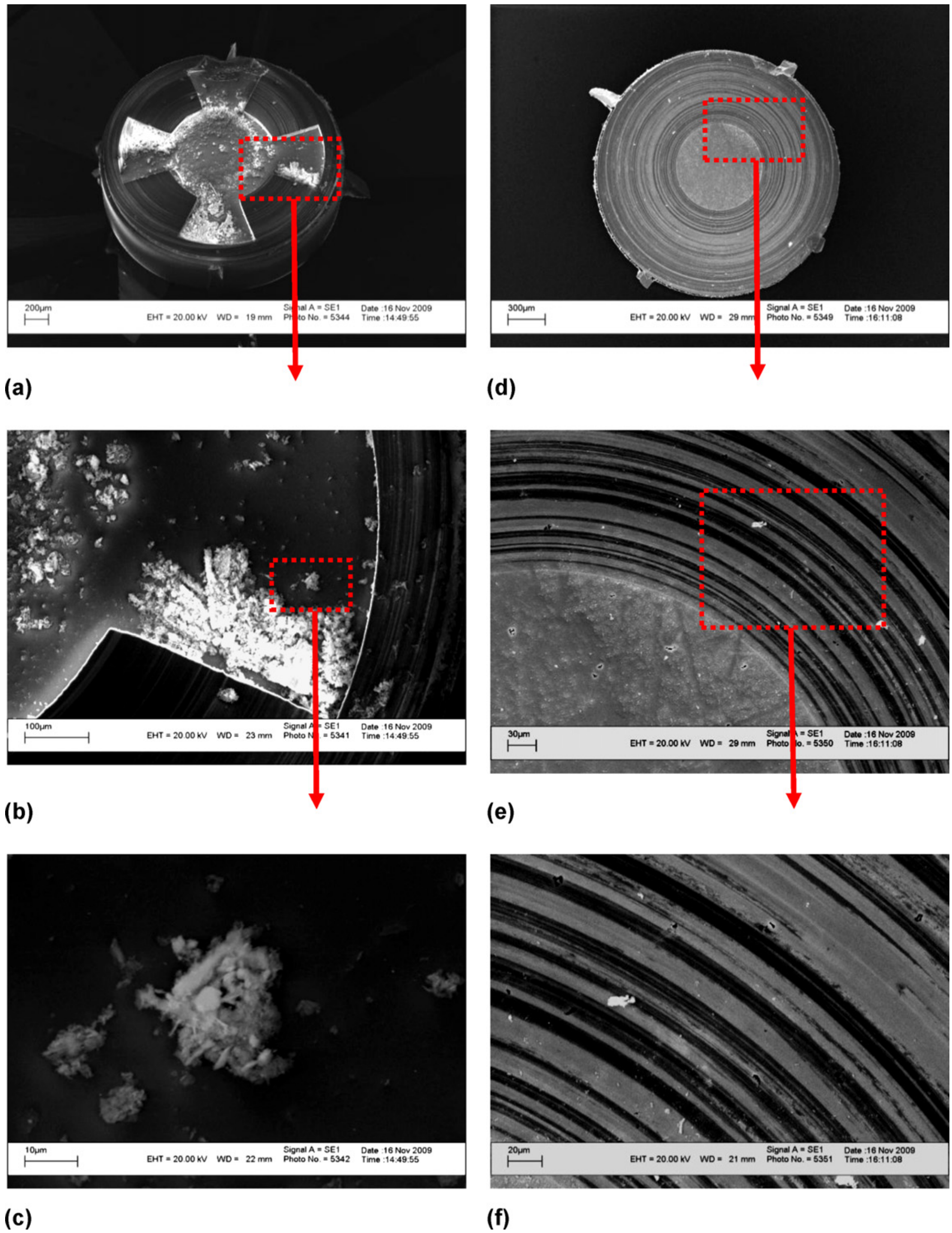

Fig. 7. SEM images of worn specimens (after 90 min sliding at 500 rpm and a load of 0.05 N).

During these submerged tests, a fully formed hydrodynamic film cannot be present, since the entrainment speed of 500 rpm is sufficient only to partially separate the sliding surfaces. This can be seen from the Stribeck curves (obtained under the same conditions) in Ref. [21].

It interesting to note that, under dry conditions, exposure of specimens to ambient air results in reduced friction and wear, while under conditions of submersion in water, exposure to air has the opposite effect. The reasons for this are not immediately obvious. It has been shown that although water can form provide a lubricating film and separate sliding surfaces, it also acts to attack silicon surfaces [20]. The corrosion process occurs as water acts to breaks the Si–Si bonds after first being dissociatively chemisorbed on its surface [24]. For specimen surfaces that have been exposed to air, the latter effect seems to dominate, while the action of plasma cleaning must reduce this effect. A possible explanation is that the plasma cleaning leaves the silicon surfaces highly hydrophilic, so that, when submerged a monolayer of water molecules attaches on the slightly oxidised surface, resulting in wear protection. If exposed to air following plasma cleaning, the surfaces will become partially hydrophobic and thus less likely to be protected by a very thin water layer.

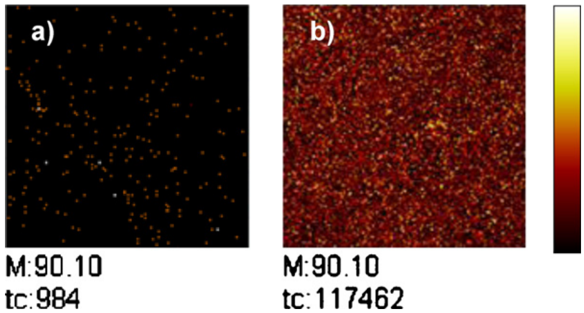

Fig. 8. TOF-SIMS map showing intensity of $C_4H_{12}NO$ peak present on (a) a sample immediately after plasma cleaning, and (b) 15 h after plasma cleaning.

4.2.2. Tests with hexadecane and 1-pentanol – not stored/$C_{16}H_{34}$ and not stored/$C_5H_{12}O$ (l)

For tests submerged in hexadecane, Fig. 4 shows the measured friction coefficient to be about 0.1, which is acceptably low. Additionally, Fig. 6 shows that no measurable wear occurred. Previous work [21] shows that at an entrainment speed of 500 rpm (as tested here), the hexadecane-submerged contact operates in the mixed/hydrodynamic regime. Within this regime, some degree of contact occurs. Despite this surfaces must be sufficiently well protected to prevent wear.

Tests were also carried out with the contact submerged in 1-pentanol (not stored/$C_5H_{12}O$ (l)). The results were similar to tests with hexadecane, with low measured friction and undetectable wear. Dugger and co-workers [12] used vapour phase lubrication of 1-pentanol to significantly improve lifetimes of sliding MEMS components. Using surface analysis, they showed that 1-pentanol vapour forms high molecular weight oligomers on the rubbing silicon surfaces, to effectively protect the surface. It is likely that a similar lubrication mechanism is operating in the current, liquid phase tests.

4.2.3. Vapour phase lubrication – not stored/$C_5H_{12}O$ (v)

One set of tests was carried out using plasma cleaned surfaces with no further storage with 1-pentanol lubricant supplied in the vapour phase (not stored/$C_5H_{12}O$ (v)). Fig. 6 shows that no measurable wear occurred. This is in agreement with work by Asay et al. [12], demonstrating the effective wear reduction using this form of lubrication.

No significant difference in wear could be observed between liquid and vapour phase 1-pentanol lubrication systems employed in the current study, since neither resulted in measurable wear. However, measured friction did vary quite markedly between these two methods of lubricant delivery. The average friction measured during the pentanol *vapour* lubricated tests was found to be 0.28, while that for pentanol *liquid* was found to be only 0.05. This difference may be attributed the partial hydrodynamic separation afforded by submersion in liquid pentanol which acts to reduce solid contact. The friction associated with a liquid lubricated contact varies with entrainment speed following a Stribeck curve, while a vapour lubricated contact is not affected by entrainment speed to such an extent. All tests in the current study were carried out at a speed of 500 rpm; conditions under which the liquid pentanol lubricated contact operates in the boundary/mixed regime.

As mentioned above, the average friction measured during our vapour phase tests was 0.28, rather than *ca.* 0.2 measured by Asay et al. [12], a discrepancy, which can be attributed to several factors including differences in surface roughness. A further difference is that our tests were run on plasma treated specimens, on to which pentanol vapour was deposited prior to (and during) testing, while those carried out previously were first coated with a self-assemble mono-layer (SAM) prior to rubbing in a vapour environment.

5. Conclusions

- Wear of high sliding silicon surfaces is a critical factor in determining the reliability of MEMS devices. To date however, few in situ quantitative micro-scale wear investigations have been carried out.
- Using a custom built MEMS tribometer, tests have been carried to measure friction and wear on 2 mm diameter silicon disc surfaces, under a range of conditions.
- Friction and wear behaviour are shown to depend strongly on the condition of surfaces prior to testing. When used in unlubricated conditions, greatest wear was observed on specimens which had been cleaned with oxygen plasma prior to testing. Specimens exposed to atmospheric air for 15 h prior to testing exhibited negligible wear and low friction. However, exposure to water-saturated argon prior to testing did not prevent wear to such an extent. This observation demonstrates that some species present in air, other than water, attaches to the silicon surface during air-exposure, *i.e.* a lubricious contaminant. This is corroborated by TOF-SIMS analysis, which showed a significantly increased presence of long chain hydrocarbon molecules on air-exposed surfaces.
- Friction and wear tests were also carried out on silicon specimens submerged in a liquid bath. Again, results depend on the prior storage of the test surfaces. If surfaces have been plasma treated prior to testing, liquid water reduces friction and wear compared to dry conditions. Conversely, the presence of water accelerates the wearing of specimens that have been exposed to ambient air prior to testing. When hexadecane or 1-pentanol was used as a liquid lubricant, friction was a minimum, and wear was undetectable for all conditions. This was the case, despite the contact being subject to mixed lubrication. This suggests that a boundary film is formed on the silicon surfaces by each of these organic lubricants.
- Tests carried out with the lubricant being supplied in the form of 1-pentanol vapour also showed no appreciable wear. A difference in friction was found between contacts lubricated with liquid and vapour; for the conditions tested, the latter coefficient of friction was 0.28, while for liquid it was 0.05. Further testing is needed to be carried out under more severe conditions to compare the relative wear reduction associated with liquid and vapour phase lubrication.

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References

[1] S.T. Patton, K.C. Eapen, J.S. Zabinski, Effects of adsorbed water and sample aging in air on the μN level adhesion force between Si(1 0 0) and silicon nitride, Tribology International 34 (2001) 481–491.
[2] S.A. Henck, Lubrication of digital micromirror devices, Tribology Letters 3 (1997) 239–247.
[3] C.M. Waits, B. Geil, R. Ghodssi, Encapsulated ball bearings for rotary micro machines, Journal of Micromechanics and Microengineering 17 (2007) 224–229.
[4] K.J. Gabriel, F. Behi, R. Mahadevan, M. Mehregany, In situ friction and wear measurements in integrated polysilicon mechanisms, Sensors and Actuators (1990) 184–188.
[5] M. Mehregany, S.D. Senturia, Measurement of wear in poly silicon micromotors, IEEE Transactions on Electron Devices 39 (1992) 1136–1143.
[6] D.M. Tanner, J.A. Walraven, L.W. Irvin, M.T. Dugger, N.F. Smith, W.P. Eaton, W.M. Miller, S.L. Miller, The Effect of humidity on the reliability of a surface micromachined microengine, EEE 37th Annual International Reliability Physics Symposium Proceedings (1999) 189–197.

[7] B. Bhushan, S. Venkatesan, Friction and wear studies of silicon in sliding contact with thin-film magnetic rigid discs, Journal of Materials Research 8 (1993) 1611–1628.
[8] R. Bandorf, H. Lüthje, T. Staedler, Influencing factors on microtribology of DLC films for MEMS and microactuators, Diamond and Related Materials 13 (2004) 1491–1493.
[9] A.S.M.A. Haseeb, U. Albers, K. Bade, Friction and wear characteristics of electrodeposited nanocrystalline nickel–tungsten alloy films, Wear 264 (2008) 106–112.
[10] D.H. Alsem, E.A. Stach, M.T. Dugger, M. Enachescu, R.O. Ritchie, An electron microscopy study of wear in polysilicon microelectromechanical systems in ambient air, Thin Solid Films 515 (2007) 3259–3266.
[11] D.B. Asay, M.T. Dugger, S.H. Kim, In-situ vapor-phase lubrication of MEMS, Tribology Letters 29 (2008) 67–74.
[12] D.B. Asay, M.T. Dugger, J.A. Ohlhausen, S.H. Kim, Macro- to nanoscale wear prevention via molecular adsorption, Langmuir 24 (2008) 155–159.
[13] S.T. Patton, W.D. Cowan, K.C. Eapen, J.S. Zabinski, Effect of surface chemistry on the tribological performance of a MEMS electrostatic lateral output motor, Tribology Letters 9 (2000) 199–209.
[14] S.T. Patton, J.S. Zabinski, Failure mechanisms of a MEMS actuator in very high vacuum, Tribology International 35 (2002) 373–379.
[15] S.A. Smallwood, K.C. Eapen, S.T. Patton, J.S. Zabinski, Performance results of MEMS coated with a conformal DLC, Wear 260 (2006) 1179–1189.
[16] S. Suzuki, T. Matsuura, M. Uchizawa, S. Yura, H. Shibata, Friction and wear studies on lubricants and materials applicable to MEMS, in: Proceedings of IEEE 4th Workshop on MEMS, Nara, Japan, 1991, pp. 143–147.
[17] U. Beerschwinger, D. Mathieson, R.L. Reuben, S.J. Yang, A Study of wear on MEMS contact morphologies, Journal of Micromechanics and Microengineering 4 (1994) 95–105.
[18] H.H. Gatzen, M. Beck, Wear of single crystal silicon as a function of surface roughness, Wear 254 (2002) 907–910.
[19] I.S.Y. Ku, T. Reddyhoff, J.H. Choo, A.S. Holmes, H.A. Spikes, A novel tribometer for the measurement of friction in MEMS, Tribology International 43 (2010) 5–6.
[20] T. Reddyhoff, I.S.Y. Ku, A.S. Holmes, H.A. Spikes, Friction modifier behaviour in lubricated MEMS devices, Tribology Letters 43 (2011) 239–246.
[21] I.S.Y. Ku, T. Reddyhoff, J.H. Choo, R. Wayte, A.S. Holmes, H.A. Spikes, Lubrication of MEMS devices using liquids of different viscosities, ASME Journal of Tribology, in press.
[22] Harrick Plasma, Plasma Cleaner User's Manual for the Expanded Plasma Cleaner (and Optional Plasmaflo).
[23] T. Takahagi, H. Sakaue, S. Shingubara, Adsorbed water on a silicon wafer surface exposed to atmosphere, Japan Journal of Applied Physics 40 (2001) 6198–6201.
[24] D. Graf, M. Grundner, R. Schulz, Reaction of water with hydrofluoric acid treated silicon(1 1 1) and (1 0 0) surfaces", Journal of Vacuum Science & Technology A7 (3) (1988) 808–813.