1 | INTRODUCTION

Microelectromechanical system (MEMS) technology is sophisticatedly integrating miniaturized components such as sensors, actuators, and signal processing over a small area using microfabrication techniques, and offers new functionalities and performance advantages over conventional devices. Furthermore, due to the availability of mature fabrication and design techniques of IC technology, most of the MEMS devices are using silicon as a substrate material [1, 2]. But, the main problem with silicon is that silicon has poor tribological properties like non-lubricious nature, high friction, and poor wear resistance particularly at micro-scale [3]. In addition, as the overall size of the MEMS device decreases, all forces associated with the volume and inertia of the device also decrease. Thus, the microscopic forces such as capillary forces, van der Waals forces, chemical bonds, and electrostatic forces contribute to adhesion, which in turn considerably affect the friction. As a result, all these factors are also severely restraining the development of MEMS technology [4]. Until now, several methods have been proposed for the lubrication of silicon-based MEMS devices. For example, carbon-containing ultrathin films [5], self-assembled monolayers [6], diamond-like carbon films [7] and hydrogen terminations [8] helped in the reduction of friction and wear problems in MEMS, but all these were proved impracticable during prolonged operations because of their hard and non-lubricious nature and were damaged or scratched, eventually resulting in a high wear rate during high sliding operations. Asay et al. [9] had used the alcohol for the lubrication and showed that the lifespan of silicon microdevices was increased by four orders of magnitude. In vapour phase lubrication with 1-pentanol vapour results in a replenishable lubricating film and provides an extended operational lifetime compared to monolayer alone [10, 11]. Vapour phase lubrication proved feasible in the reduction of friction but requires very good sealing conditions and stable gas supply, so vapour phase lubrication is not widely used [12]. Deng et al. [13] tested liquid lubricants for the lubrication of micromotors and stated that liquid lubricants are producing over damping and drag in large amounts and concluded that liquids are inappropriate for lubrication of MEMS devices [14]. However, this statement was based on a very small amount of published literature and their test lubricants were of high viscosities (20–60 cS), so it is possible that over damping can be alleviated by using low viscous lubricants. Later, Ku et al. [15] used liquids of low viscosities for lubrication of MEMS thrust pad bearing and achieved low coefficients of friction at high speeds. Additionally, under the same operating conditions liquid lubrication exhibited smaller friction than vapour phase lubrication for the same friction-pair. This is not always similar for all conditions because friction in liquid lubricated surfaces varies significantly with operating speeds [16]. However, liquid lubrication was also proved ineffective, due to leakage, loss of lubricants during high sliding operations, and unavailability of suitable lubricant baths to properly hold the lubricants during the sliding process. Hence, it is the need of the time to investigate the possibility of using smart lubricants such as ferrofluids for the lubrication of MEMS devices.

Ferrofluids are considered as a stable colloidal suspension, formed by carrier liquid, surfactant, and single-domain magnetic nanoparticles with a diameter of about 10 nm [17]. In recent years, ferrofluids have gained considerable attention of researchers because of their unique properties like smartness under the influence of magnetic field and potential to offer promising solutions for challenging problems like sealing, lubrication, detection, heat transfer, separation, grinding, and damping [18–21]. Moreover, as a lubricant, ferrofluids have higher potential than traditional lubricants, because, with the employment of an external magnetic field on ferrofluids lubrication zone, ferrofluids can significantly be augmented, retained, located, shaped, and can act as a supporting lubricant to reduce the dosage and the leakage of lubricant [20]. Additionally, the friction behaviour of ferrofluids can also be controlled by the distribution of the magnetic field and the particle concentration of nanoparticles in ferrofluids [19, 22]. Uhmann et al. [23] showed that by employing an external magnetic field, the viscosity of ferrofluids can be increased by increasing the intensity of the magnetic field. This will improve load-bearing capacity, reduce wear, and improve surface morphology of the sliding contacts. All these distinctive properties extend the applications of ferrofluids in the field of tribology.

Therefore, the main objective of this research is to investigate the possibility of using ferrofluids for the lubrication of sliding silicon MEMS under different magnetic texture conditions. Such a method can provide, low-friction lubrication method for
MEMS devices. For the investigation of proposed work, a novel friction-pair was designed which accurately mimics the MEMS devices sliding conditions and has the potential to employ a magnetic field on the ferrofluids lubrication zone. Different tribological tests and surface morphology analyses were carried out with a reciprocating tribometer and an optical microscope to understand the possible mechanisms of ferrofluid lubrication under the influence of the magnetic textures.

2 | EXPERIMENTAL DETAILS

2.1 | Preparation of the test lubricant

In this paper, the Fe₃O₄ (magnetite) nanoparticles-based ferrofluids were prepared using the usual chemical co-precipitation method, which is the most efficient method for magnetite-based ferrofluids preparation. The prepared magnetite nanoparticles were dispersed into a low viscosity carrier liquid by a surfactant of oleic acid, and then the ferrofluids were produced. The mass fraction of magnetite nanoparticles dispersed in ferrofluids was 0.9 percent, and diester was used as the carrier liquid of ferrofluids. The density of the ferrofluids is 0.986 g mL⁻¹ the viscosity of the ferrofluids at 20 °C is 12 mPa·s, and the saturation magnetization (Ms) of the ferrofluids is 3.4 kA m⁻¹.

2.2 | The working principle of the test rig

For the tribological testing, a pin-on-disc reciprocating micro tribometer was used in this work. The schematic of the micro tribometer is shown in Figure 1. A micro scaled tribo-contact is produced between the flat surfaces of two MEMS test specimens. The lower specimen (rectangular silicon disk) is mounted on a platform driven by a DC servo motor and a crank-linker for reciprocating motion, the displacement of the lower specimen is controlled by the crank-linker. The upper specimen (cylindrical silicon pin) is attached to a two-dimensional force sensor for friction measurement, the height of the upper specimen was adjusted via a computer-controlled vertical stage. The friction coefficient between the surfaces is obtained by the normal load and generated tangential force measured by the two-dimensional force sensor.

Figure 2 illustrates the schematic of the pin-on-disc friction-pair assembly. The ferrofluids-based magnetic texture was designed by placing low viscous ferrofluids on the lower specimen which was assembled on the top of a magnets matrix, and the adjustment of the magnetic field was achieved by changing the parameters of the magnets matrix.

2.3 | Development of the friction-pair

Silicon-based friction-pair was used to mimic the MEMS device working conditions. The upper specimen is a cylindrical silicon pin of 2 mm as shown in Figure 3, a safety margin of 0.5 mm wide is kept to prevent the structure of the surface pattern from being damaged during laser cutting. As a result, the actual diameter of the upper specimen is 3 mm. The surface pattern of the pin is 100 µm wide, the spacing of each
2.4 Analysis of the ferrofluids-based micro-texture designed by the magnetic field

To investigate the relationship between ferrofluids and the magnetic matrix, ANSYS finite element analysis software was used to simulate the magnetic field of the lower specimen. In the simulation, Nd-Fe-B-N35 was used as the permanent magnet material, Al was used as the matrix material, and the airfield was set outside the simulation model. Then, ferrofluids texture was tested under the action of the magnets matrix based on above analysis. Before the test, a drop of 0.1 mL of ferrofluid was placed in the centre of the lower specimen for 5 min to ensure that the ferrofluid texture was formed under the influence of the magnetic matrix. An optical microscope (Leica DM2700M) was used to find the ferrofluids-based magnetic texture with the best quality by taking the photos of ferrofluid texture.

2.5 Tribological testing

2.5.1 Tribological testing under different magnetic field texture configurations

For the performance evaluation of ferrofluids under different magnetic fields, three types of magnetic texture configurations namely using no magnets, a bulk magnet and an optimized matrix magnets were designed and tribological tests were carried out using the reciprocating pin-on-disk tribometer. The reciprocating speeds of microtribometer come at 3.822, 5.742, 7.547, 9.600, 11.538, 13.187, 15.000, 17.143, 19.048, 21.053, 23.077, 25.000 mm s\(^{-1}\), the applied normal load was 10 N. The reciprocating displacement of the pin was 6 mm, and the duration of the test under each speed condition was 130 s. For each tribological test, the volume of ferrofluids was controlled at 0.1 mL on the lower specimen.

2.5.2 Wear analysis of the rubbed specimens

After the tribological testing, specimens were taken down from the microtribometer, placed in the ultrasonic cleaner for 10 min to remove the wear impurities from their surfaces. Later, deionized water and 99.9% absolute alchohol were used to clean the specimen. Then after being dried by hot air, the surface morphology of the specimens was analysed with an optical microscope, and images at different magnifications (50, 200, and 500 times in order) were taken.

3 RESULTS AND DISCUSSION

3.1 Analysis of the formation of ferrofluids texture

The magnetic field distribution of nine kinds of magnets matrix and bulk magnet have been simulated. The typical magnetic field pattern is 100 µm, and the depth is 50 µm, which was fabricated by deep reactive ion etching (DRIE).

As shown in Figure 4, the lower specimen is a rectangular silicon disk (15 × 10 mm) was assembled on the top of a magnets matrix. The magnets matrix as shown in Figure 5 is composed of regularly arranged permanent magnets in the lower specimen holder. The distribution of the magnetic field generated by matrix magnets can be adjusted by the height and spacing of the permanent magnets. In this paper, the diameter of the permanent magnet is 1 mm, the height, and spacing of each permanent is designed with nine kinds of matrix magnets of 2 × 1.5, 2 × 2, 2 × 3, 3 × 1.5, 3 × 2, 3 × 3, 5 × 1.5, 5 × 2, and 5 × 3 mm respectively. Moreover, to investigate the influence of the ferrofluids texture formed by magnets matrix, a bulk magnet and a bulk aluminum (non-magnetic) are also set up with a size of 13 × 7 × 2 mm.
distribution is shown in Figure 6. The magnetic field intensity of the bulk magnet is larger but the gradient is lower at the centre zone. The magnetic field gradient of the magnets matrix is very high and the spikes-shaped texture of the magnetic field had been successfully formed on the top of each permanent magnet. The texture is also affected by the height and spacing of the permanent magnets. The height of the magnet mainly affects the intensity of the spikes, and the spacing of the magnets affects the distribution of the spikes. Considering the effect of the magnetic field gradient on texture formation, the magnets matrix with a magnet size of 3 × 3 mm is the best.

Figure 7 is the image of ferrofluid texture formed by magnets of 3 mm height with different spacings. The ferrofluids are distributed on the surface of the specimen according to the distribution of the magnetic field as the ferrofluids flow from the low magnetic field area to the high magnetic field area. No distinct texture had been produced by the bulk magnet however the spikes-shaped micro texture has been formed by magnets matrix, which is consistent with the distribution of the magnetic field. Among all the micro textures, the ferrofluid texture with a magnet spacing of 3 mm is the best one. So, the magnets matrix of 3 mm was used for the tribological experiments.
3.2 The processing of original friction signal

For the signal processing of the original friction signal, a model tribological test at the normal load of 10 N and a 3 mm high bulk magnet was carried out. The signal diagram of friction data is shown in Figure 8. During the reciprocating motion, the friction coefficient changes periodically due to variation of speed and direction, but there is no obvious difference in friction coefficient between each period. Therefore, to avoid deviation caused by the effect of slight interference the average of 1560 'raw' data acquired over the 130 s was taken as the friction coefficient of each speed.

3.3 Friction coefficient curves under different magnetic field texture configurations

Figure 9 shows the friction coefficient curves under different magnetic field configurations. It is observed clearly that, under the no magnetic field conditions, the friction coefficient is much higher than both the bulk magnet and matrix magnets conditions. For no magnetic field conditions at the lower speeds, the lubricant was unable to form an effective lubricating film between the sliding contacts. The sliding specimens were in boundary lubrication stage as surfaces contact directly with each other, therefore, the friction coefficient is high. With the increase of the speed, the lubricating film is gradually formed between the surfaces of the test specimens, the hydrodynamic lubrication stage is started and friction coefficient at higher speeds is reduced due to the viscosity of the ferrofluids. When the load is small, the dynamic pressure parameters are large. As a result, it is possible to enter the stage of mixed lubrication or hydrodynamic lubrication stages earlier. Therefore, the friction coefficient curves in the without magnetic field conditions are dependent on the applied load and reciprocating speeds. While at bulk magnetic field conditions ferrofluids had shown much better results than without magnetic field conditions because the magnetic field helped the ferrofluids to properly position on the friction-pair, which had contributed to increased load-bearing capacity and lowered the friction coefficient.

It is believed that at bulk magnetic field conditions, the friction coefficient at low speeds was lower, but increased sharply at higher speeds due to the viscous resistance caused by the viscosity of the ferrofluids. However, at matrix magnets conditions the coefficient of friction was the lowest in all three stages of lubrication at all reciprocating speeds. It is deduced that matrix magnetic field had facilitated the proper positioning, retention, and allocation of ferrofluids on the friction-pair. And the spike-shaped texture had increased the load-bearing capacity in all three lubrication stages, and hence the lowest friction coefficient values were achieved as compared to other two conditions. Therefore, it is concluded that the friction coefficient has been greatly improved at low-speed after the external magnetic field has intervened, while the friction coefficient has increased at high-speed. So, the proper height and spacing of the magnetic field texture are very important for the achievement of better lubrication performance.

3.4 Surface morphology analysis of rubbed specimens

The images of surface wear morphology of upper silicon specimen under different magnetic field conditions are shown in Figure 10. Figure 10(a) shows the surface morphology of worn specimen under no magnetic field condition. It can be seen that specimen had deeper wear marks, scratches, and abrasions. Clear signs of material cracking and abrasive wear marks on both edges of the specimen texture can be seen in the
highlighted box. This can be attributed to micro-protrusions on the silicon surface, which were deformed due to excessive load, results in the peel-off material from the surface, and produced deep abrasive marks. Figure 10(b) shows the surface examination images of the rubbed specimen under the bulk magnet. It is observed that specimens have wear and abrasive marks but the depth of wear scars and abrasive marks is reduced. Besides, cracking and peeling on both sides of the specimen surface was also reduced a lot. The surface characterization of a worn specimen under the matrix magnets is shown in Figure 10(c). It can be seen that wear is significantly lower than that of the bulk magnetic field condition and no magnetic field conditions. The surface quality of the specimen is remarkably improved. Wear mainly occurs in small linear stripes at one end. Hence, it is concluded that the ferrofluids-based magnetic matrix texture greatly improves wear conditions between silicon surfaces. The texture increases the load-carrying capacity and self-repairs the wear marks on the specimen, and thus the significant reduction in wear was achieved.

4 CONCLUSION

In this paper, low viscous ferrofluids containing magnetic nanoparticles were used for the lubrication of silicon MEMS device under the influence of different magnetic field texture configurations. A novel silicon pin-on-disk friction-pair was fabricated by DRIE to mimic the working conditions of the MEMS devices. Tribological tests were carried out in both the absence and presence of different magnetic field textures using reciprocating pin-on-disk tribometer and the surface morphology of the rubbed specimens was characterized by an optical microscope. The main results are as follow:

(1) The application of magnetic field can improve the lubrication performance of ferrofluids for silicon surfaces, but it will lead to a very high friction coefficient at high speed by only using a simple bulk magnet due to the viscous resistance of the ferrofluids.

(2) The spikes-shaped micro ferrofluids texture formed by matrix magnetic field had shown a remarkable reduction in friction and wear from low speed to high speed, because the texture increases the load-carrying capacity and self-repairs the wear marks and scratches on the surface of the specimen.

(3) The pattern parameters of the magnetic field distribution are important for the optimal design of magnetic textures during ferrofluids lubrication.

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