

# MICROSCALE TRIBOLOGY (FRICTION) MEASUREMENT AND INFLUENCE OF CRYSTAL ORIENTATION AND FABRICATION PROCESS

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## ABSTRACT

A microscale tribology test system has been developed at UCLA to measure friction and wear in MEMS components. Test results indicate that microscale friction may not follow Amontons' law that states the friction force is only related to the normal force with a coefficient of friction. In this study, test data indicate that the friction coefficient is not constant and it's influenced by crystal orientation, apparent contact area, MEMS material, and fabrication process, as well as the normal force applied. Explanation for the discrepancy may be related to adhesion, which is a critical issue at microscale.

## INTRODUCTION

Research on understanding microscale tribology is increasing in recent years. Microscale tribology (friction, wear and lubrication) is an important aspect for MEMS components including issues such as device service life, energy transfer efficiency, and friction drive mechanisms (SDA, wobble motor, e.g.). Previous research efforts to understand tribology in the small scale can be divided into two categories of experimental and analytical.

Most experimental approaches typically use atomic force microscopes (AFM) to evaluate tribology issues at the nanometer scale (Bhushan, etc. 1995 and references there in) where the AFM tip (in nanometer size and sharp) is used to contact the surface. This approach is similar to the pin on a plate method adopted in macroscale tribology measurements. Friction force and surface morphology can be obtained from these measurements. Usually the AFM tip is fabricated with a similar material ( $\text{Si}_3\text{N}_4$ , e.g.) to better understand the friction and wear between specific pairs of materials. Nevertheless, the sharp tip does not represent the contacting surface used in most MEMS applications and the stiffness of the tip's cantilever may influence the results, as bending is an issue. Therefore, while providing useful information the tests do not represent actual friction and wear situation.

While some data exists for MEMS wear, for example, gravity force has been used as normal force to measure MEMS components' wear (Beerschwinger, etc. 1994), very few friction measurements are available. Molecular dynamics (MD) have been used to simulate tribology responses at the atomic scale (Sorensen, etc. 1996; Shimizu, etc., 1998). In that modal a prearranged atomic wedding or pair of planes are used to simulate the sliding friction and wear. For a copper system, results show periodical stick-slip phenomena occur on a Cu (111) plane and not on Cu (100) plane owing to the close-packed (111) planes are the preferred slip planes (Sorensen, etc. 1996). A larger number of atoms contacted (contacting area) will generate larger friction force. Results such as this need more experimental data to evaluate their accuracy.

## MICROSCALE TRIBOLOGY (FRICTION) MEASUREMENT

### Setup of measurement system

A micro tribology measurement system has been developed at UCLA. The system is similar to the pin on a plate method used in standard wear test (ASTM standards). A picture of the test setup is provided in Figure 1. A MTS Tytron Microforce Test System applies a monotonic/cyclic load at the sub Newton level (0.01N) with a displacement resolution of 0.1 $\mu\text{m}$ . The sample holders including one center holder and two external holders are aligned parallel each other. They hold two pairs of samples face to face during the test (Fig. 1). Samples are placed vertically to eliminate gravity effects. One of the two samples is patterned using micromachining process (Fig. 2) while the other is a flat Si chip (8mmX8mm). The patterned chip has three equal cylinders fabricated on it (3 $\mu\text{m}$  high, similar to the ASTM's pin on plate method). The diameter of the cylinders is varied from 2 $\mu\text{m}$  to 1024 $\mu\text{m}$  (Fig. 2). A load cell is used to measure the normal force acting on samples during tests. A spring is used to apply the normal force and to keep samples contact. Bending is eliminated with a universal joint and symmetric

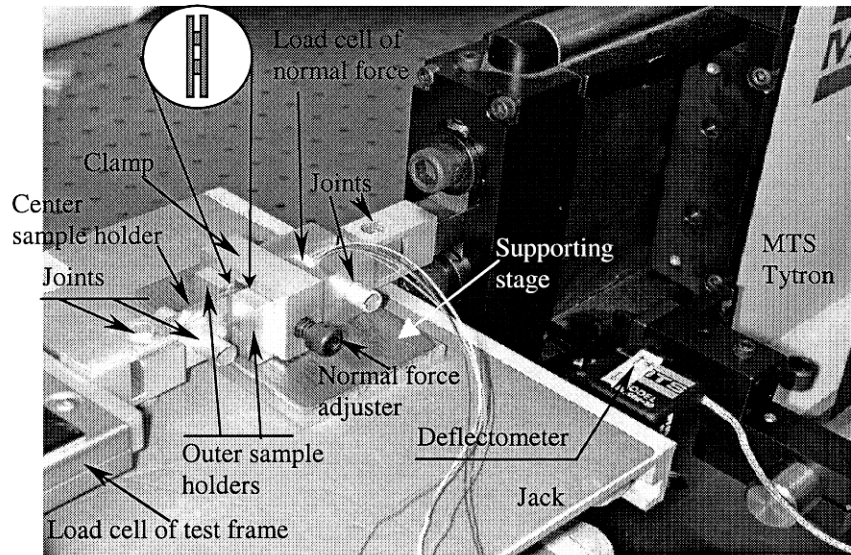


Figure 1 Test setup with MTS Tytron microforce test system

placement of the samples. The setup sits on an adjustable scissors jack to eliminate off-axis loading. Measurements indicate the friction force between the sample holders and the supporting stage is less than  $4 \times 10^{-3}$  N. Data is recorded with a MTS data acquisition system and a minimum of two samples were tested for each case. If the two tests displayed discrepancies, additional tests were performed.

### Sample fabrication

Sample fabrication consists of first producing the desired surface on the wafer and then fabricating the cylinder pattern. There were eight different surface conditions studied (e.g. crystal orientation, fabrication process, and deposition materials). Two different crystal orientation friction tests on silicon (111) and (100) wafer were conducted. Fabrication processes studied in this paper include KOH to etch (111) and (100) silicon wafers and deep RIE to etch (100) silicon wafer to produce different surface conditions. The deposition materials studied were high temperature silicon oxide, LPCVD silicon nitride and LPCVD polycrystal silicon. Each surface preparation group consists of at least two wafers. One of them was used as the flat piece and the other one was used to fabricate the cylinder pattern. The preconditioned wafer was first spin coated with photo resist to produce the circular shape by lithography method. Deep RIE etching was used to obtain  $3 \mu\text{m}$  high cylinders (3 per sample, see Fig. 2) and photo resist was used as an etching mask. Exceptions to this process are for the silicon nitride and silicon oxide. In the later cases fluorine RIE was used to etch down the deposition layer ( $\text{SiO}_2$  and  $\text{Si}_3\text{N}_4$ ) first and then deep RIE was used to etch silicon to produce the cylinder pattern. After

etching, the samples were diced to  $8\text{mm} \times 8\text{mm}$  chips.

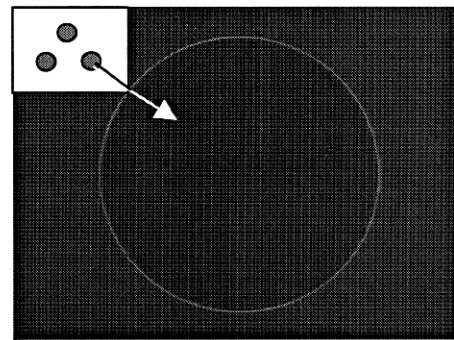


Fig.2 One of the three cylinder pattern after deep RIE etching. The diameter of the cylinder varies from  $2 \mu\text{m}$  to  $1024 \mu\text{m}$ .

Follow this, the photoresists were striped off and chips were cleaned. The surfaces of all chips were cleaned and inspected prior to testing.

## RESULTS AND DISCUSSION

### Crystal orientation

Friction force measurements on pure Si (111) and Si (100) wafers are presented in Figure 3. It's interesting that the periodical stick-slip phenomena observed in the Si (111) sample was not found in the Si (100) sample. Sorenson (1996) predicted this behavior using MD but it has not been experimentally observed to our knowledge. Our results suggest that crystal orientation strongly influence the microscale tribology. This maybe due to the fact that (111) are the preferred slip planes as

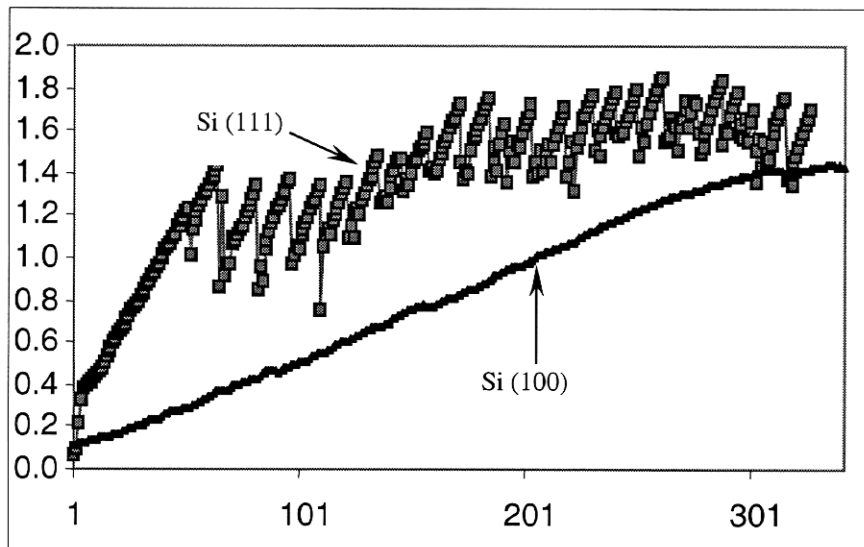


Figure 3 Friction force (N) versus test time. Periodical stick-slip occurs on Si(111), not on Si(100).

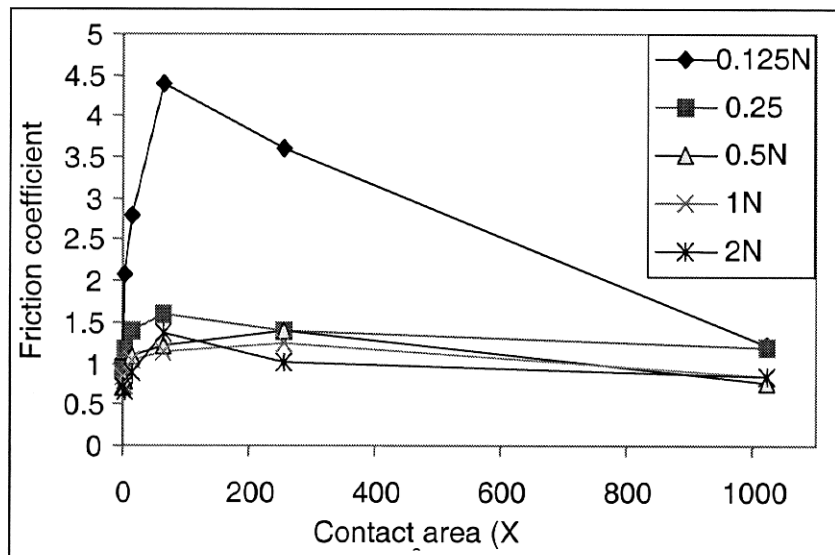


Figure 4 Friction coefficient of silicon (111) test wafer verses contact area and normal force

suggested by Sorenson. However, we must point out that the molecular dynamics simulations are order of magnitude smaller than the representing length of the samples in our experiments. Our results indicate that silicon (111) plane produces larger (10% to 60%) friction force than silicon (100) plane.

#### Contact area and normal force

Amontons' law ( $F = \mu N$ ) says that friction force can be calculated from the normal force and coefficient of friction for the material. In this equation,  $F$  is the friction force,  $N$  is the normal force applied and  $\mu$  is the

friction coefficient which is related only to the material and surface condition. Amontons speculated that friction was caused by the interaction of surface roughness peaks and only few of these peaks are actually in contact when one surface slide over another. Experimental results at the macroscale confirmed Amontons law.

It has been suggested that Amontons law maybe invalid at the atomic scale and nanoscale based on analytical (MD, e.g.) results (Sorensen, etc. 1996; Shimizu, etc., 1998, Landman, 1998). Research results (Shpenkov, 1995, references there in) on metals indicated that adhesion is an issue when the surface became very flat

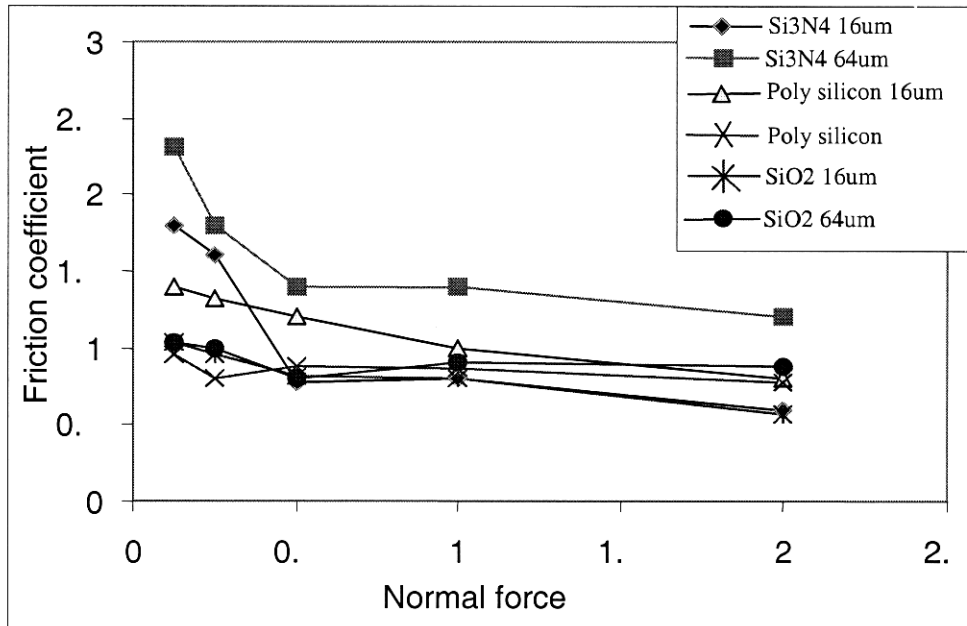


Figure 5 Friction coefficient of Si3N4, polysilicon and silicon oxide verses normal force. The diameters of the cylinder are 16um and 64um respectively.

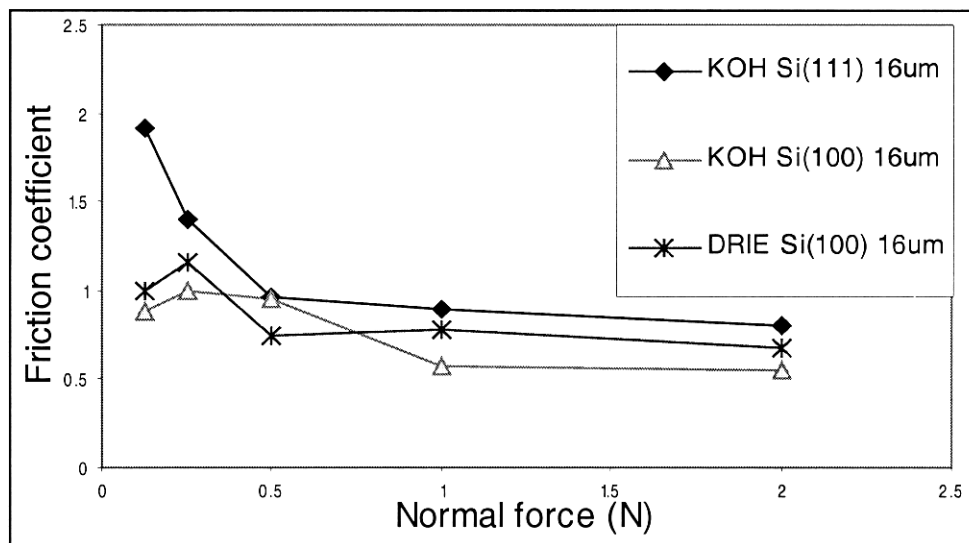


Figure 6 Friction coefficient of fabrication process, KOH etched Si (111), KOH etched Si (100) and deep RIE etched Si(100). The diameter of the cylinder is 16μm.

and not contaminated. Since the MEMS community uses micromachining processes that produce relatively flat surface on a small region, the question rises again is Amontons' law valid at the microscale?

The test results on Si (111) test wafers shown in Figure 4 indicate that the friction coefficient varies with contact area. The friction coefficient initially increases (by as much as two) up to a contact area of 64 μm in diameter

then decreases and becomes a constant. This suggests that Amontons' law maybe invalid at area less then 256μm in diameter. Fig. 4 also indicates that the normal force is influencing the friction coefficient, especially when the normal force is small. We believe the dependence of friction coefficient on contact area and normal force maybe due to adhesion becoming prominent at the small scale.

## Deposition materials and normal force

Thin film deposition is commonly used for surface micromachining. Some of the deposited materials are silicon oxide, silicon nitride and polycrystal silicon. It is important to understand the friction behavior of these thin films. Test results for friction coefficients of these thin films measured are shown in Fig. 5. The results indicate that the silicon nitride film (1500 Å thick) has the largest friction coefficient. The 64 μm diameter cylinder produce larger friction coefficient than the 16 μm one. Another trend is the friction coefficient decreases as the normal force increases and reaches a constant value at 0.5N. On the other hand, polycrystal silicon (1.5 μm thick) has a lower friction coefficient than silicon nitride. It also decreases as the normal force increases. The silicon oxide (0.5 μm thick) has the lowest friction coefficient when compared to the other two surface materials. A similar trend of friction coefficient versus the normal force was observed in silicon oxide too. The discrepancy between the three films is the influence extent of the normal force on the friction coefficient. Friction coefficient of silicon nitride decreases quickly versus the normal force and becomes constant at 0.5N. While the friction coefficient of polycrystal silicon and silicon oxide decreases much slower than silicon nitride does versus the normal force applied. In other word, friction of silicon nitride may not follow Amontons law, while silicon oxide and polycrystal silicon are more closer to that the Amontons law predicted (constant friction coefficient). We believe that this is due to the variation of surface conditions. The deposition thickness of silicon nitride is very thin (1500 Å), and the surface condition follows well with the surface profile of the silicon wafer. While surface condition of polycrystal silicon (1.5 μm thick) and silicon oxide (0.5 μm thick) may change a lot (more rough) during the deposition process because it's very hard to control the growth evenly at the microscale.

## Microfabrication process

MEMS devices are fabricated using various microfabrication processes, which are roughly divided as wet and dry etching. Fundamentally etching processes are chemical reactions that could change surface characteristics either chemically (absorbing) or physically (roughness). The test results presented in this paper focused on the most frequent commonly used methods of KOH wet etching and deep RIE dry etching. Figure 6 indicates that KOH wet etching of silicon (111) produces the largest friction coefficient among the three conditions. Friction coefficient of silicon (100) etched by deep RIE is larger than that of silicon (100) etched by KOH solution. Results also indicate in Figure 6 that

friction coefficients decrease as the normal force increases. A difference exist of this test is that for deep RIE etching silicon (100) and KOH wet etching silicon (100) where such trend is not as much as KOH etching Si (111) especially at small normal force (0.125N). This discrepancy maybe due to the fact that rough surfaces fabricated by deep RIE etching and KOH wet etching of Silicon (100) produced the smaller effect of adhesion on friction force at the microscale.

## CONCLUSION

A microscale tribology test system has been developed at UCLA to measure MEMS component's friction as well as wear. Test results indicate that the friction at microscale may not follow Amontons' law, when the contact area is less than 256 μm in diameter. The friction coefficient varies with MEMS materials and fabrication processes, as well as the normal force applied. Test results also indicates that silicon (111) surface produces larger friction than silicon (100). Thin silicon nitride film produces larger friction coefficient than thick film of polycrystal silicon and silicon oxide. KOH etched silicon (111) plane and deep RIE etched silicon (100) generate larger friction coefficient than that of KOH etched silicon (100) plane.

## ACKNOWLEDGMENT

Authors would like to thank Susie Maule for helping in fabricating the test setup. The authors would like also to acknowledge support by the Army Research Office on a Multidisciplinary University research Initiative (Contract Number: DAAH04-95-1-0095, Contract Monitor: John Prater).

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