25th IRG–OECD meeting in Uppsala Sweden June 13–14 2005

International Research Group on Wear of Engineering Materials
Hosted by Professor Sture Hogmark and the Tribomaterials Group
at The Ångström Laboratory, Uppsala University

# EFFECT OF CHEMICAL COMPOSITION AND SURFACE ROUGHNESS ON FRICTION IN THE µN LOAD REGIME

Syed Imad-Uddin Ahmed[1*], G. Bregliozzi[1], Nicolas Conté[2] and Pierre-Albert Steinmann[1]
*[1] Laboratoire des Matériaux et de Technologie des Surfaces, Haute Ecole ARC Ingénierie,*
*Eplature-Grise 17, CH-2300 La Chaux-de-Fonds, Switzerland.*
*[2] CSM Instruments SA, Rue de la Gare 4, CH-2034 Peseux, Switzerland*

## 1. INTRODUCTION

The tribological problems associated with microelectromechanical systems (MEMS) with moving parts in contact with one another are well known [1]. The preferred use of silicon (Si), an established MEMS material with inherently poor tribological characteristics, is the main cause of this situation. Among the various surface forces, which play an important role due to the large surface to volume ratios of microsystems, capillary forces are known to be a problem with respect to adhesion and friction [2]. Such forces can be overcome or reduced considerably by changing the chemical composition of the surface by using hydrophobic coatings such as self-assembled monolayers (SAMs) or Langmuir-Blodgett films for low load and low temperature applications [3,4]. However, these films tend to deteriorate under high loads [5] and also under certain operating conditions [4,6,7]. In these cases, coatings such as diamond-like carbon (DLC) [8,9], silicon carbide (SiC) [8], silicon nitride ($Si_3N_4$) [8] as well as titanium carbide (TiC) [10] can be effective. For the case of titanium carbide, Radhakrishnan et al. [11] have recently shown that it is possible to integrate TiC thin films into the MEMS fabrication process.

Besides the chemical composition of the surface, another key issue is the surface topography. Most tribological studies involving coatings have been performed on polished surfaces, even though most technical surfaces, including those of micromachined MEMS, possess a finite roughness. Therefore, it is important to understand the effects of roughness associated with technical surfaces. In addition, it is important, whenever possible, to perform studies especially for MEMS using the same material pairs. This work reports on the microfrictional properties of SAMs applied on Si(100) samples as well as Si balls (counterbodies), to understand effects associated with the chemical composition, and TiC tribo-pairs for gaining insight on roughness effects.

## 2. MEASUREMENT SYSTEM: MICROTRIBOMETER

A reciprocating precision microtribometer was used in this work. The system is described in detail elsewhere [2,12,13]. Briefly, the microtribometer (Figure 1) is a system

---

[*] Corresponding Author. Present Address: Institut für Physik und Institut für Mikro- und Nanotechnologien, Technische Universität Ilmenau, PF 100565, Ilmenau 98684, Germany. Email: imad.ahmed@tu-ilmenau.de

consisting of a combination of precision drives, a force transducer made from photostructured glass and fiber-optic length detection system to measure the deflections of the force transducer. This system makes it possible to measure friction forces from the μN to several hundred-mN with normal loads within the same range. The advantages over scanning probe techniques (AFM) are that various types of material combinations can be readily tested and the friction force and the applied normal load can be precisely determined [13].

In this work, microfriction tests were performed by measuring friction force as a function of the applied normal load. The friction coefficient was then determined by taking the slope of this curve. All samples and counterbodies used in this study were used after sequential cleaning in acetone, isopropanol and finally in ethanol, each for 10 min, using ultrasonic assistance. Furthermore, all experiments were repeated at least three times to ensure reproducibility of results.

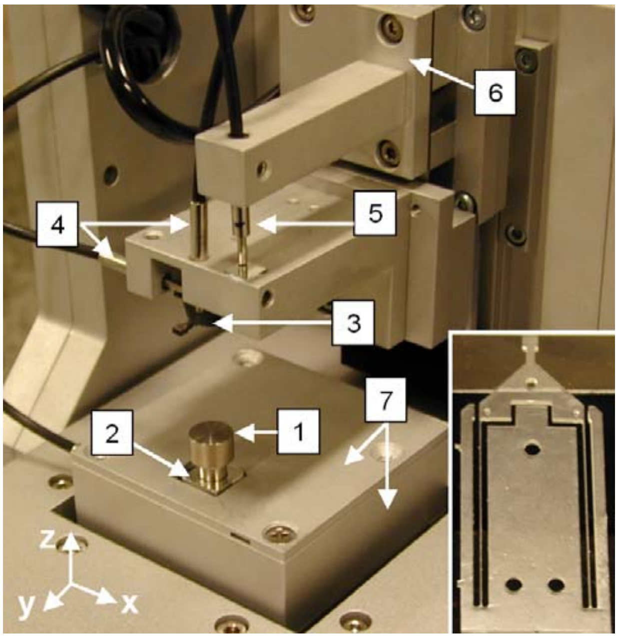

**Figure 1:** *Image of the microfriction tester. The main components are: 1) Sample, 2) reciprocating unit, 3) force transducer, 4) fiber-optic sensors to detect normal and lateral deflections of the force transducer, 5) fibre-optic sensor for normal force adjustment detection, 6) precision drive to adjust normal force and 7) the xy micropositioning stages powered by stepper motors with an integrated sample tilt adjustment. The inset shows a photostructured glass spring (force transducer).*

## 3. MICROFRICTIONAL PROPERTIES OF SAMS SLIDING AGAINST SAMS AND SILICON

The SAMs in this study were coated on smooth Si using 1H,1H,1H,2H,2H-perfluorodechyltrichlorosilane ($CF_3(CF_2)_7(CH_2)_2SiCl_3$ (referred henceforth as FDTS) and octadecyltrichlorosilane ($CH_3(CH_2)_{17}SiCl_3$ (referred henceforth as OTS). Prior to SAM coating, the Si surfaces were immersed in piranha solution, subjected to an oxygen plasma etch, then immersed in the desired SAM solution (FDTS or OTS in a solvent) and subsequently rinsed with hexadecane and isopropanol. All immersions were performed in a nitrogen glove box to avoid polymerization.

After microfrictional testing, the coefficient of friction (CoF) was obtained by a linear fit of the measured friction versus load curves for each tested tribopair and then determining the slope (Figure 2). The lowest CoF was obtained when an OTS-coated ball slides against a flat FDTS- or OTS-coated surface. A higher CoF was measured when the OTS-coated ball slides on the native oxide-covered Si surface; in that case, the coefficient of friction was even higher comparable to the case when a bare Si ball slides against a flat OTS-coated surface. This is attributed to the different contact situations: in case of the OTS-coated ball sliding on a flat Si surface the monolayer chains are in constant contact during sliding, while when a bare Si ball slides on a monolayer coated surface, the ball is in contact only with a given group of chains for a finite time period, depending on the velocity. Similar arguments apply for the corresponding results using a Si ball coated with FDTS. Also, in this case, low friction only occurs when sliding against a flat OTS-coated sample, although the friction values were higher than in the case of an OTS-coated ball sliding against a FDTS-coated surface. Interestingly, in the case of a FDTS-coated ball sliding against a FDTS-coated surface, a high coefficient of friction was measured compared to self-mated surfaces involving OTS.

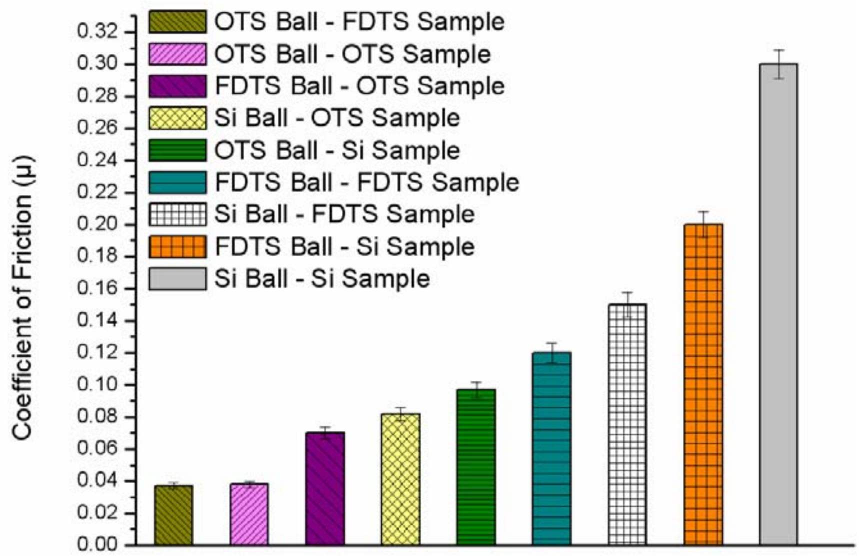

**Figure 2:** *Coefficient of Friction of various SAM coated material pairs, SAMs sliding against Si and Si sliding against Si.*

## 4. MICROFRICTIONAL PROPERTIES OF SELF-MATED TITANIUM CARBIDE SURFACES

The TiC surfaces investigated in this paper were all surfaces of TiC thin films deposited on AISI 440C steel by chemical vapor deposition (CVD) [11,12]. The deposited single phase films were about 6 µm thick, with a grain size of 0.1 µm, and a hardness of HV = 3500. The as-deposited surfaces are very rough, but can be polished down to a very smooth finish. It has been shown that air-exposed TiC surfaces have an oxide layer that is up to 5 nm thick [13].

The topography of the films was characterized using a commercially available atomic force microscope (AFM). All measurements were performed using the same microfabricated V-shaped $Si_3N_4$ cantilever with a manufacture-quoted radius of curvature of 30 nm.

Table 1 lists the roughness measurements of the various samples from rough (Sample A) to very smooth (Sample D) as well as the TiC ball used in the study.

| Sample | Ra [nm] | Rms [nm] | Rmax [nm] |
|---|---|---|---|
| **A** | 129.00 ± 1.0 | 161.00 ± 1.0 | 1120.0 ± 5.0 |
| **B** | 1.70 ± 0.1 | 2.50 ± 0.1 | 153.4 ± 5.0 |
| **C** | 0.70 ± 0.1 | 1.15 ± 0.1 | 62.5 ± 4.0 |
| **D** | 0.50 ± 0.1 | 0.65 ± 0.1 | 22.5 ± 2.0 |
| TiC Ball | 1.7 ± 0.1 | 2.8 ± 0.1 | 125.0 ± 5.0 |

**Table 1:** *AFM roughness measurements of TiC samples and TiC ball*

The effect of surface roughness was examined by analyzing the friction-load curves of the smooth TiC ball (Fig. 3) sliding against TiC surfaces with different roughnesses. Starting from the smoothest surface (Sample D), by increasing the surface roughness it was observed that a decrease of the friction force occurred for a fixed normal load. A minimum was found for the sample C, characterized by a mean roughness value of 0.70 nm.

Then, by gradually increasing the sample roughness, an increase of the friction force was measured. The samples D and B, characterized by a mean roughness value of 0.5 nm and 1.70 nm, respectively, exhibit the same frictional behavior. In the case of the as-deposited TiC thin films, characterized by a mean roughness value of 129 nm, a further increase of the friction force was measured.

Different effects could influence the decrease of the CoF, due to increased surface roughness. The most important is that the rougher the surface is, the smaller is the contact area and, thus, the area that can be wetted and that leads to reduced capillarity. Also, this decreased contact area provides less interaction between the ball and the samples leading to a lower coefficient of friction.

The high coefficient of friction measured for the very rough sample is attributed to the initiation of deformation of surface asperities [16] and removal of the surface oxide present on all air-exposed TiC surfaces. While deformations have been shown to be elastic [16], plastic deformations due to higher contact pressures were also found to occur [17].

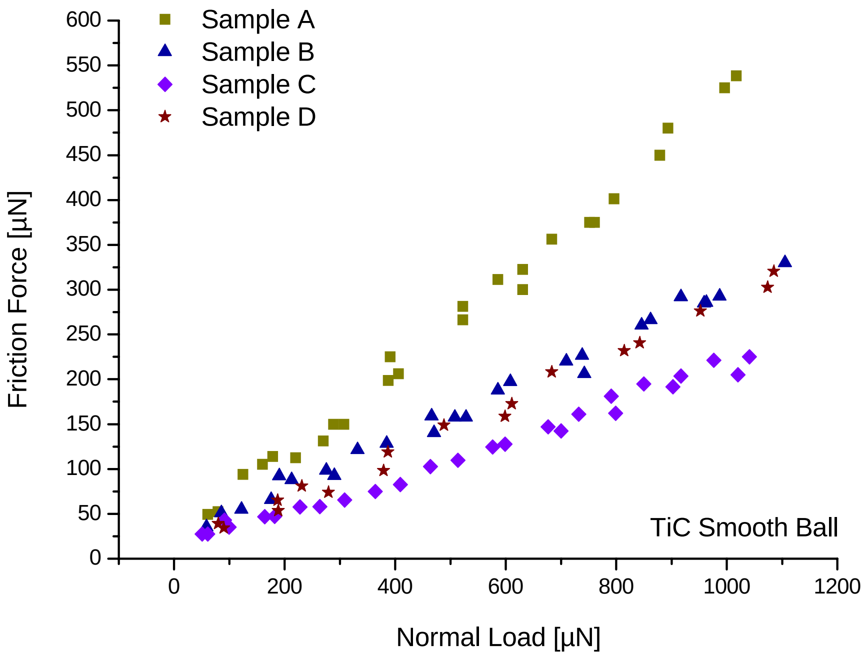

**Figure 3***: Friction versus normal load curves for the smooth TiC ball sliding against TiC surfaces with different roughnesses. All measurements were carried out at 20 °C, sliding velocity of 50 µm/sec and a sliding distance of 100 µm.*

## 5. CONCLUSIONS

In the µN load regime, microfriction occurring between flat hydrophilic surfaces can be reduced by the application of hydrophobic coatings such as self-assembled monolayers on both surfaces of a tribopair. In cases, where the application of hydrophobic self-assembled monolayers is not feasible and hard coatings such as titanium carbide are required, the ideal tribological contact situation is obtained with contacting surfaces that are slightly rough. Very smooth as well as rough surfaces result in higher friction.

## REFERENCES

[1] M.P. de Boer and T.M. Mayer, *MRS Bull.* 26, 302 (2001).
[2] M. Scherge, S. N. Gorb, in: *Biological Micro- and Nanotribology*, Springer-Verlag, Berlin, (2001).
[3] R. Maboudian, *Surf. Sci. Rep.* 30, 207 (1998).
[4] H. Liu, S.I.-U. Ahmed and M. Scherge, *Thin Solid Films* 381, 135 (2001).
[5] S. Ren, S. Yang, Y. Zhao, J. Zhou, T. Xu and W. Liu, *Tribol. Lett.* 13, 233 (2002)
[6] W. Hild, G. Hungenbach, S.I.-U. Ahmed, M. Scherge and J.A. Schaefer (in Press - Tribotest).
[7] R.R. Rye, G.C. Nelson and M.T. Dugger, *Langmuir* 13, 2965 (1997).
[8] N. Rajan, C.A. Zorman, M. Mehregany, R. DeAnna and R.J. Harvey, *Surf. Coat. Technol.* 108–109, 391 (1998).
[9] S.I.-U Ahmed, G. Bregliozzi and H. Haefke, *Wear* 254, 1076 (2003).

[10] H.J. Boving and H.E. Hintermann, *Thin Solid Films* 153, 253 (1987); H.J. Boving, H.E. Hintermann and G. Stehle, *Lubr. Engin.* 39, 209 (1983).
[11] G. Radhakrishnan, P.M. Adams, R. Robertson and R. Cole, *Tribol. Lett.* 8, 133 (2000).
[12] O. Mollenhauer, I. Ahmed and H. Haefke, Extended abstracts of the 23rd IRG-OECD Meeting in Coimbra, Portugal, 6 - 7 May, 2002.
[13] M. Scherge, S.I. Ahmed, O. Mollenhauer and F. Spiller: Technisches Messen, 67 324 (2000).
[14] H.J. Boving and H.E. Hintermann, *Thin Solid Films* 153, 253 (1987).
[15] H.J. Boving, H.E. Hintermann, and G. Stehle, *Lubr. Engin.* 39, 209 (1983).
[16] K. Meine, T. Schneider, D. Spaltmann, and E. Santner, *Wear* 253, 725 (2002).
[17] L. Kogut and I. Etsion, *Tribol. Trans.* 46, 383 (2003).