Experimental Equipment for Interfacial Force and Friction Measurements of Micro Scale Samples

N. K. MYSHKIN
Metal-Polymer Research Institute of National Academy of Sciences, Gomel, Belarus

N. D. SPENCER[1], A. Ya. GRIGORIEV[2], A.M. DUBRAVIN[2], O. Yu. KOMKOV[2], S. TOSATTI[1]
[1]Laboratory for Surface Science and Technology, Department of Materials, ETH-Zurich, Switzerland
[2] Metal-Polymer Research Institute NASB, Gomel, Belarus

SUMMARY

Wide spread of miniature mechanical systems such as micro scale actuators and sensors attracts attention of many researchers to the field of tribology and surface properties of these mechanical systems. Experiments have shown that there is a scale factor that doesn't allow one to apply directly results obtained for macroscopic tribosystems to nano/micro systems. So, the studies in microtribology call for use adequate experimental equipment. The paper describes the design and test data when using laboratory equipment for measuring adhesion forces and friction properties of micro systems. The set of developed devices consisted of adhesion meter, rotary and reciprocating microtribometers. The adhesion meter allows us to measure force-distance dependence between test samples in the range of the 10 to 10000 μN on the distance 1 to 10000 nm. The tribometers operate in the range of normal load from 1mN to 1N, velocity – from 0.1 to 1mm/s and allow to measure friction force and acoustic emission. The data on measurements of adhesion and friction forces of $DDPO_4$, $ODPO_4$, and OTS SAMs, as well as DLC coatings on silicon substrates are discussed.

1 INTRODUCTION

An analysis of modern tendencies for measurement techniques reveals that the dimensional range in technologies and research has shifted to micro- and nanometer scale [1]. High precision rubbing parts and other mated members for precise mechanics (e.g., magnetic recording devices, high-precision tools, microrobotic systems, etc.) are produced with very smooth surfaces and operate at low loads and speeds.

Experiments have shown that there is a scale factor that doesn't allow one to apply directly results obtained for macroscopic tribosystems to micro/nano systems. It is commonly accepted that main reason of observed phenomena is explained in terms of changing role and grade of influence of different factors of contact interaction on the process of friction and wear in micro/nano scale (Fig.1).

With decreasing scale of tribosystem the adhesion and interfacial interactions became a dominant factor, which define their behavior. For example, it is known that some difficulties related to failure of MEMS (micro-electro-mechanical systems) resulted mainly from adhesion leading to sticktion of parts [2].

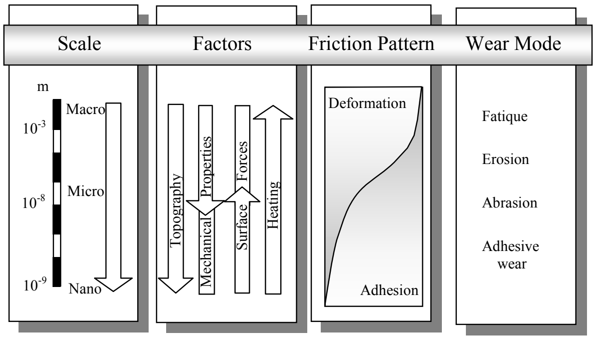

Figure 1: Combination of factors effecting friction andwear

In the present time most of the experimental support of investigations in the field of micro- and nanotribology is provided by such devices as atomic force microscope (AFM), lateral force microscope (LFM), and surface force apparatus (SFA) [3]. But these devices are intended for investigation of surfaces in nanoscale force/distance resolution and they did not well suited for measurements related to operation of precision mechanisms and MEMS. So, the studies in microtribology call for use the adequate experimental equipment.

The paper describes the design and test data when using laboratory equipment developed for measuring adhesion forces and friction properties of micro systems. The set of developed devices consisted of contact adhesion meter, rotary and reciprocating microtribometers. All the devices have similar contact geometry and range of measurement forces.

2 EXPERIMENTAL EQUIPMENT

2.1 Contact Adhesion Meter

Available evaluations of molecular forces between macroscopic bodies correspond to the sensitivity of an accurate analytical balance. At first sight this leads to a conclusion that the balance can be used to perform such measurements. Yet one of the main problems arising when measuring the molecular forces is that the latter increase rapidly with decreasing the distance between the specimens under testing. Hence, the measurements should be carried out at a very small speed that can not be implemented technically using the design of the common balance. Deryagin et al. proposed to solve the problem [4] by applying the principle of a feedback balance. This design with modification was used later in a number of experiments intended to measure molecular attraction forces.

When designing the apparatus we have taken into account the experience of development and application of already known designs. We have chosen the design of a vertical torsion balance with negative feedback. This design eliminates the problems with balancing and errors caused by friction in the balance support.

The measuring unit of the adhesion meter is vertically disposed frame 1 (Figure 2) suspended on string 2. One arm of the frame carries holder 3 of a probing specimen (ball). Another arm of the frame carries movable coil 4 of a measuring electromagnet connected with a highly stable current source. Mirror 5 is fastened to the frame. It reflects the beam of laser 6, which then passes expander of optical base 7 and impinges photodetector 8. When force starts to act between the specimens frame 1 with mirror 5 turns thus changing the light flux impinging photodetector 8. The signal of the photodetector forms a feedback signal, which varies current in coil 9 until the frame has turned to its initial position. So, any variation in the forces acting between the specimens is compensated by a corresponding current variation that remains the frame stationary in measuring process. Current in coil 9 is calibrated. So, the forces acting between specimen 10 and the ball can be measured. Specimen under testing 10 is placed on table 11 equipped with a system of rough positioning driven by stepping motor 12 and with a system of fine positioning driven by piezodrive 13.

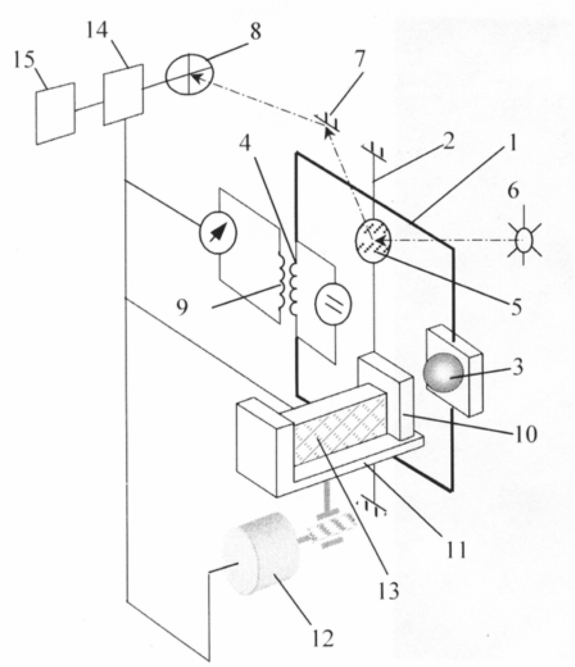

Figure 2 Principal scheme of adhesion meter

Initially the specimen and the ball are removed to a distance at which they do not influence each other. Piezodrive 13 is stretched. In this position current passing measuring electromagnet 9 is assumed to correspond to zero force between the specimens. Then specimen 10 is approached to the probing specimen by stepping motor 12 until a preset initial contact load is attained. Depending on the aim of the experiment the measuring cycle can start either on reaching the required contact load or after a certain period of time.

Adhesion is measured when moving off specimen 10 with piezodrive 13 by plotting the dependence of the force acting between the specimens on the distance. In the process of moving off the force varies and this variation is compensated by current in coil 9 that provides a stationary position of the frame. When a preset distance is reached the measurement is performed in reverse order, namely when approaching the specimen to the probe. As a result, two dependencies are obtained characterizing the force of interaction when moving off and approaching the specimen to the ball.

To protect the apparatus from vibrations and to retain a constant temperature it was mounted on a table with a magnetic damper. The table is suspended on strings into a wooden case with a sound-proof lagging made of cellular polystyrene and metallized polyethylene film. Control of the measuring process is performed by a programmable analog-digital signal processor 14. Experimental results are indicated and operation modes are set by software operating in Windows and connected with the signal processor through a parallel port of PC 15. Basic characteristics of the apparatus are listed in the Table 1.

Measured forces, μN	10 – 10000
Sample displacements, nm	10 -- 10000
Sample size, mm	20×20×5
Probe size (ball type), mm	0.2 -- 5

Table 1: Characteristics of contact adhesion meter

2.2 Reciprocating Microtribometer

The investigation of friction characteristics of thin films and coatings used in MEMS and similar technology makes necessary some special testing conditions such as light normal loads, low sliding velocity and high sensitivity of friction force measuring systems [5]. It is important that mechanical noise of the motor system must be limited. The loading part of test apparatus must be non-sensitive to the topography and small tilt angles of the specimen to prevent load fluctuation during testing. Presented design of microtribometer being based on sensitive measuring system [6] and frictionless linear electromagnetic motor can solve some of these problems.

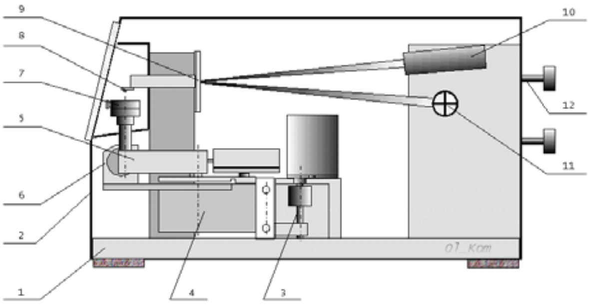

Figure 3 General scheme of reciprocal tribometer:

The microtribometer consists of the folloving parts (Figure 3): base plate 1, case 2; vertical positioning system 3; electormagnetic drive for vertical movement 4; voice coil motor 5; induction transducer 6; specimen holder 7; ball holder 8; spring suspension system 9; laser 10; photodetector11 and screws 12 for adjasment optical system of mesuring friction force.

The base plate 1 are used for support of the tester's parts and electric boards with power supply unit. The string suspension 9 supports the loading system 4. The electromagnetic motor 5 is used for smooth moving the sample holder 7 during friction tests.The laser system 10 is used for measuring angle position of the loading system which is depends on value of friction force.

The electric circuit based on digital signal processor is used for system control and connected to the PC by COM port. Figure 4 shows the scheme of friction force measurement. When the normal load is applied to the ball specimen the friction force causes rotation of the loading system suspended by vertical string. The laser beam is reflected from the mirror fixed on string suspension. The beam spot position is detected by photo detector. The position sensitive detector and electromagnetic system assembled with string suspension are connected with feedback circuit to compensate the friction force. The feedback system supports the angle position of the loading system to be constant during test. The value of current applied to the winding of electromagnetic system proportional to the value of friction force is recorded and visualized by PC software. The sample is moved by the frictionless voice coil motor without vibrations and mechanical noise in a wide range of speed. The voice coil motor winding and Hall effect position sensor are connected with feedback circuit to control the speed and stroke of motion.

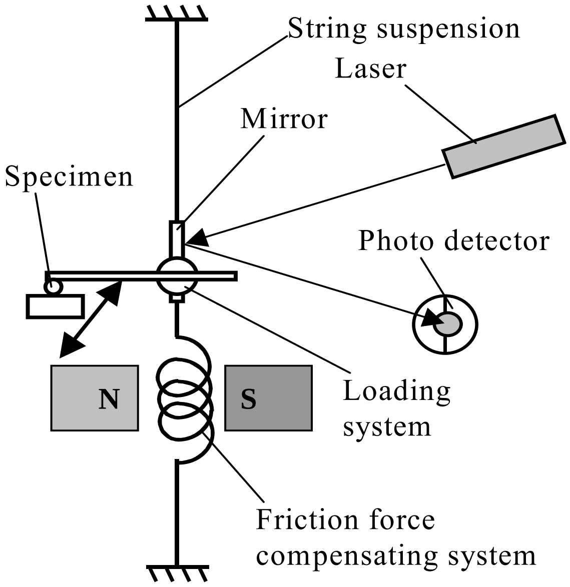

Figure 4 Scheme of friction force measurement

Moving parts of the sample holder are suspended by "knife" type support. It can be also substituted by flexible hinge system. The loading system consists of permanent magnet, steel ring, winding, holder of ball specimen and balancing weight. The loading force is are caused by the interaction between electrical current passed through the winding and magnetic field of the permanent magnet. The value of produced force is independent from the angular position of the holder. It means that the value of load of the loading system is independent from the topography effect and small tilt angles of the specimen. Technical specification of the reciprocal tribometer is listed in the Table 2.

Normal load, μN	100 – 10000
Friction force range, μN	10 – 2000
Sliding speed, μm/s	0.13 -- 4000
Stroke length (max), mm	5

Ball size, mm	0.2 -- 4

Table 2: Characteristics of reciprocating tribometer

2.2 Rotating Microtribometer

Rotating tribometer (Figure 5) has the same design as reciprocal one except that sample stage is rotated. The stage 6 is rotated by low speed revolution motor 4. and the device is equipped by system of horizontal positioning 2 for changing radius of wear track.

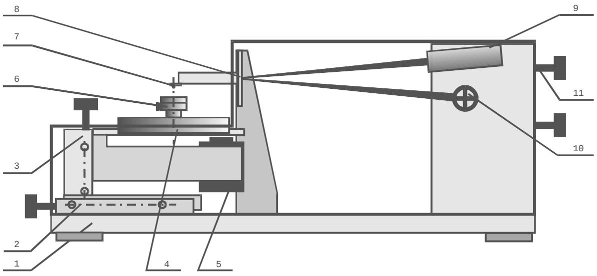

Figure 5: General scheme of reciprocating tribometer: 1 – base plate; 2 – horizontal positioning plate; system of vertical positioning; 4 – electromagnetic motor; 5 – electromagnet; 6 – sample stage; 7 – ball holder; 8 – spring suspension; 9 – laser; 10 – four-quadrant photo detector; 11 – tuning screws.

The specification of the device is listed in the Table 3

Normal load, μN	10 – 10000
Sliding speed, rev/min	1 – 20
Radius of wear track, mm	0 –10
Ball size, mm	0.2 -- 4

Table 3: Characteristics of rotating tribometer

3 EXPERIMENTAL DETAILS

The purpose of the investigation was measurements and comparative analysis of adhesion and friction forces of such self-assembled monolayers as DDPO$_4$ (dodecylphosphoric acid ester), ODPO$_4$ (octadecylphosphoric acid ester), and OTS (octadecyltrichlorosilane) SAMs, as well as DLC (diamond-like carbon) coatings on silicon substrates. For DDPO$_4$ and ODPO$_4$ SAMs the initial silicon substrates were covered by Ti or TiOx sublayers. Basic characteristics of the specimens being tested are listed in Table 4

The technique of preparing SAM-covered silicon samples is described in [7]. The tests were performed on the experimental equipment described above.

Two following technique of specimens preparation were involved. After covering of silicon substrates by the SAMs they were blown off through a microfilter (pore diameter 0.2 μm) by compressed air and were glued to the steel holder with cyanoacrilate adhesive. The conductive specimens were electrically connected with the apparatus case with electroconductive paste applied to specimen edges.

Sample	Description
Ti	Ti coating (100 nm) on Si substrate
TiOx	TiOx coating (20nm) on Si substrate
DLC	Diamond like carbon on Si substrate
Ti-ODPO4	Octadecylphosphoric acid ester on Ti covered Si substrate
TiOx-ODPO4	Octadecylphosphoric acid ester on TiOx covered Si substrate
TiOx-DDPO4	dodecylphosphoric acid ester on TiOx covered Si substrate
OTS	Octadecyltrichlorsilane on Si substrate

Table 4 Description of investigated specimens

All tests were carried out at a temperature 18±2°C, atmospheric pressure 740±15 mm Hg and relative humidity 60-70%.

4 RESULTS AND DISSCUSIONS

Typical dependence of adhesion force during approaching and retracting of silicon ball to the investigated samples test obtained by developed contact adhesion meter (CAM) is shown on the Figure 6.

All the samples are shown similar dependence of attraction forces vs. distance from sample to silicon ball. On the Figure 7 presented the forces normalized by radius of indenter in comparing of similar data obtained with AFM. The data well concise except with AFM pull-off force measurements on Ti and TiOx samples. For our opinion the difference can be explained by influence of capillary forces and low hydrophobic properties of the samples. For the much bigger size of indenter of CAM comparing AFM tip, capillary forces should play dominant role in the interaction of samples during retraction.

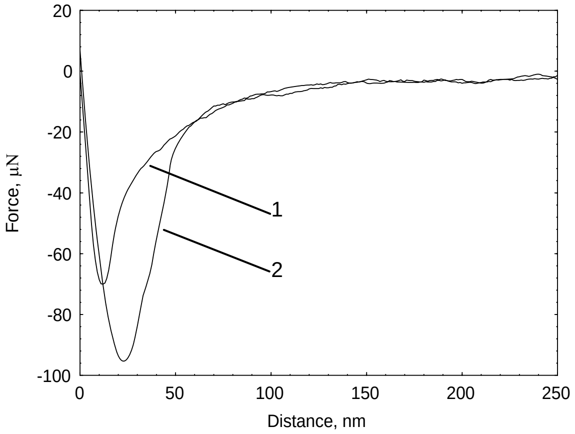

Figure 6. Force-distance curve for OTS samples and silicon ball. 1 – approaching; 2 – retraction.

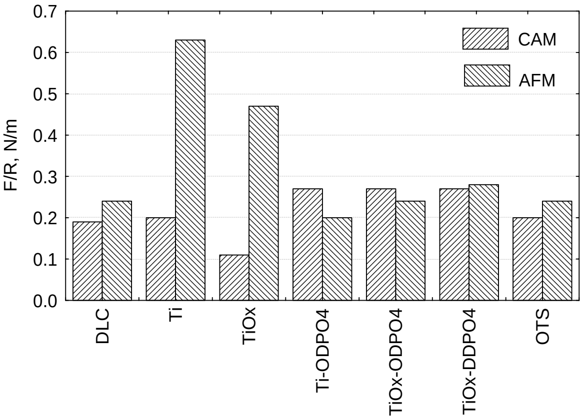

Figure 7. Adhesion (CAM) and pull-of forces (AFM) of the specimens.

Figure 8 presents data on friction coefficient of the investigated samples obtained by the reciprocating tribometer with load 10 mN and sliding speed 0.5 mm/s.

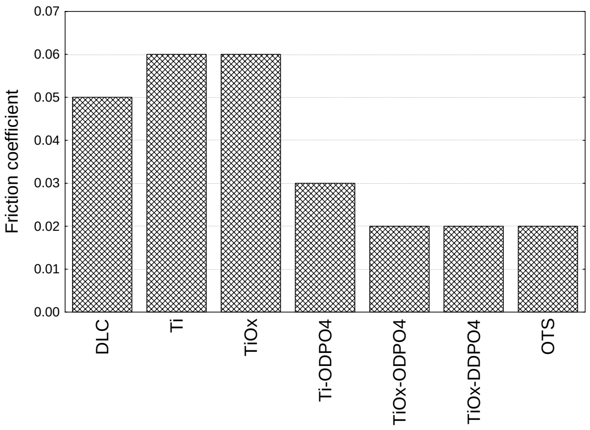

Figure 8. Friction coefficients of investigated samples.

5 CONCLUSIONS

Design and test data when using laboratory equipment for measuring adhesion forces and friction properties of micro systems are presented. The set of developed devices consists of adhesion meter, rotary and reciprocating microtribometers. The adhesion meter allows us to measure force-distance dependence between test samples in the range of the 10 to 10000 µN on the distance 1 to 10000 nm. The tribometers operate in the range of normal load from 1mN to 1N, velocity – from 0.1 to 1mm/s and allow to measure as friction force as acoustic emission.

The data on measurements of adhesion and friction forces of $DDPO_4$, $ODPO_4$, and OTS SAMs, as well as DLC coatings on silicon substrates are discussed.

Results showed that the tribological characteristics of $DDPO_4$ and $ODPO_4$ SAMs were the best among our test samples.

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