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Nanotechnologies — Guidance on methods for nano- and microtribology measurements

Nanotechnologies — Directives relatives aux méthodes de mesure en nanotribologie

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Foreword

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In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

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Introduction

Evaluation of wear and friction in systems where interactions occur in the nanoscale is becoming increasingly important. There are two main areas of application. The first is in MEMS and NEMS devices, where tribological issues can determine the overall performance of the device. It is also true that, in many cases, the tribological performance of macroscale contacts depends on the combination of what occurs at the micro- and nanoscale asperity contacts which actually take place when two surfaces come into contact.

The development of nanotribology testing provides a way of generating information and understanding these small-scale contacts. This understanding can then be used to model the performance of microscale devices and provide the basis for future models of sliding wear.

Nanotechnologies — Guidance on methods for nano- and microtribology measurements

IMPORTANT — The electronic file of this document contains colours which are considered to be useful for the correct understanding of the document. Users should therefore consider printing this document using a colour printer.

1 Scope

This Technical Report establishes techniques for the evaluation of tribological performance of sliding contacts with a lateral size of between a few nanometres (nm) and 10 µm, and where the applied load is between 50 µN and 100 mN. It describes procedures for undertaking these measurements, and provides guidance on the effect of parameters on test results. It does not cover existing SPM techniques, such as frictional force microscopy and atomic force microscopy (AFM).

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1
wear
damage to a solid surface, generally involving progressive loss of material, due to relative motion between that surface and a contacting substance or substances

[ASTM G40]

2.2
frictional force
resisting force tangential to the interface between two bodies where, under the action of an external force, one body moves or tends to move relative to the other

[ASTM G40]

2.3
coefficient of friction
μ
f
dimensionless ratio of the frictional force, F, between two bodies to the normal force, N, pressing these bodies together

[ASTM G40)]

NOTE 1 $\mu = F/N$.

NOTE 2 $\mu \geq 0$.

3 Significance and use

This Technical Report provides guidance on how to carry out micro- and nanotribology tests, paying particular attention to the likely effect of test conditions and test parameters on the results to be obtained. This Technical Report does not specify a particular set of test conditions which should be used in a test. Appropriate test conditions should be chosen after considering the eventual application for the materials being evaluated.

4 Principle

Tribology tests are conducted in test systems, which are designed to press one sample against another with a controlled relative force, while also imposing controlled relative motion. Conventionally, sliding/rolling tests are carried out with samples where the nominal contact areas have dimensions of several millimetres or more, and with test loads of the order of 1 N or greater. The focus of this Technical Report is on tribological tests where the contact areas have dimensions of between a few nanometres (nm) and 100 mN, and the loads are between 50 µN and 100 mN.

Both friction and wear can be measured using these tests. A major aim of the tests is to provide information on the tribological performance of materials at the micro- and nanoscale. This information can be used to develop an understanding of the nanoscale mechanisms, which determine the wear and friction performance of the materials and the dependence of these mechanisms on the structure of the material.

Application areas for these measurements are

— micro- and nanoscale devices where there are sliding/rolling contacts, and

— the simulation of micro- and nanoscale contacts, which underlie all macroscale tribological contacts.

5 Apparatus and materials

5.1 Test systems

5.1.1 Typical probe and sample geometries

Typically, a probe with a well-defined geometry is used to contact a flat sample (see 5.2.11). It is often important to simulate real contacts in these tests, where features such as the shape of the contact and geometrical parameters, such as the radius of curvature of the tip that is in contact in the real application are reproduced. The assumed contact geometry, such as a pointed cone, cannot always be assumed to be correct at the contact scales experienced in the tests described in this Technical Report. The real contact geometry almost always has a rounded form at the very end of the contact probe. If a series of tests is to be carried out, it is also important to consider the repeatability of the probe geometry so that contact conditions can be repeated from one test to the next. Other details of the samples are given in this Technical Report.

Although the words “probe” and “sample” are used in this subclause and in many places throughout this Technical Report, it should be emphasized that wear and damage to both probe and sample can take place.

5.1.2 Holding samples

The sample and probe need to be held firmly and in a well-defined way so that only intended motion of the samples can take place. Mechanical clamping of samples is often preferable, but in some cases, an adhesive may be used to hold samples in place, e.g. where balls are used as the probe and need to be attached to a probe holder. If adhesives are used, it is important that the thickness of the adhesive be minimized to reduce the effect of any time-dependent flow in the adhesive and also to reduce the effect of the reduced stiffness introduced by the adhesive. Furthermore, if adhesives are used, sufficient time should be allowed for some adhesives to fully cure, develop maximum bond strength, as well as allow for dissipation of any exothermal effects prior to the start of test.

5.1.3 Motion generation

The relative motion generated between the probe and the sample can be achieved by either moving the sample or moving the probe. In either case, the motion that is generated should be well defined and reproducible so that repeated pass tests can be achieved. The small vertical displacements and applied loads which are applicable in tests mean that particular care is needed so that irregularities in the motion itself do not cause artefacts in the load that is applied.

Additional care should be taken in order to minimize motion fluctuations and other effects due to ground motion, ambient thermal variations and air flow current (caused by ventilation systems, operator and laboratory equipment, to mention a few possible sources).

Motion can be generated in several ways. Piezoelectric actuators can be used, but these have limited range (normally about 100 μm). Servo electric actuators, voice coils or stepper motors can also be used with gearing to give the requisite precision of motion. In all cases, it is important to have an independent measure of displacement.

It is also important to design the sample stage and drive systems so that artefacts in either the z-motion and the x-y motion, such as hysteresis or backlash, are minimized.

Three axes of motion are required to give the necessary x-y motion and also coarse z-motion to enable the probe to be brought close to the sample. The z-axis motion should be orthogonal to the motion in the x-y plane.

Different types of motion can be used in tests. The most common is reciprocating motion in a back and forth manner in a single linear direction. A variant of this type of motion is where unidirectional motion is required, such that movement takes place in a single direction with lift-off before return motion, followed by repeated contact to give multiple contact in the same direction. Circular motion is also quite common where the flat sample is simply spun by a motor drive.

5.1.4 Application of normal force

The applied normal force can be generated by several different mechanisms.

The simplest method is to use dead-weight loading. This is a passive technique, but care needs to be taken that load artefacts, such as parasitic friction, are not generated in the loading mechanism. Parasitic friction is friction generated in the elements of the loading mechanism such that the actual applied force is different from the required force.

Another common method for generating the applied normal force is to use the compression of a compliant element to generate a force, with the normal force determined by measurement of the dimensional compression of the compliant element. The dimensional compression of the compliant element can be measured by displacement transducers such as fibre optic sensors, light deflection sensing or capacitance devices. It is important that the range and precision of the displacement transducers be matched to the deflection of the compliant element in the loading system so that the resolution and load range that are required can be achieved. Systems can be designed so that interchangeable compliant elements can be used to give different load resolutions and ranges.

In both open and closed-loop control, the force magnitude is directly controlled, and the controlled data needs to be filtered appropriately for noise and spikes in values.

Loading systems that use a compliant element to generate the applied load can be used without active load control, but if a non-level sample surface is used or the sample is rough, or when wear of the probe or sample occurs, unwanted changes are generated in the applied load. For this reason, active load control is often used such that the load that is achieved is compared with the required load and the position of the loading mechanism adjusted through a feedback mechanism, often with a piezo-actuator, so that the actual load matches the required load.

To do this, either closed-loop or open-loop control is used. In closed-loop control, a direct comparison is made between the actual and required load with a difference signal generated, which is used to drive the piezo-actuator so that the required load is achieved. This has the advantage of being very fast, but it can be difficult to adjust the parameters of the closed-loop control such that feedback and control artefacts (like hunting) are not observed.

Open-loop control is where an external computer makes the comparison and sends commands to the loading system so that the correct load is achieved. The disadvantage of this approach is that the response time of the motor can be slow.

A useful technique that can facilitate tests on samples which have a complex surface form is to make a pre-scan of the sample surface under a light load, recording the vertical position of the sample probe as this pre-scan is made. This measured form can then be used to define the measurement path reducing the magnitude of feedback motion control that is needed so that better load control can be obtained.

5.1.5 Friction measurement

Friction measurement is normally carried out by measuring the deflection of a compliant element using displacement transducers or by applying strain gauges directly to the compliant element. If displacement measurement is carried out, either fibre optic sensors, light deflection sensing or capacitance devices can be used for this purpose. In view of the low friction values that are seen for some materials, it is even more important than for the control of applied load, that the range and precision of the displacement transducers be matched to the deflection of the compliant element in the loading system so that the resolution and load range that are required can be achieved.

In this respect, it is important that the axes of the two measured forces (N and F) be ensured to remain orthogonal to one another through the measurement process.

5.1.6 Real-time wear measurement

In principle, real-time wear measurement can be achieved by measuring the relative displacement of the sample and the probe. This can be achieved by the same sensors as for friction measurement, fibre optic sensors, light deflection sensing or capacitance devices. However, the very small displacements likely to be generated in these tests mean that artefacts generated, e.g. by thermal expansion of mechanical path between the two samples or irregularities in the sample stage mean that accurate real-time measurements are difficult.

Relative displacement measurements (displacement of one sample relative to the other due to wear) can be attempted, but are difficult to achieve when one of the samples is moving relative to the other.

5.1.7 Temperature control

Some test systems have the capability for controlled temperature testing. Care needs to be taken in the design of test systems for controlled temperature testing. It is important to heat or cool both the sample and the probe so that test results are not affected by unexpected temperature gradients. It is also important to ensure that measurement instrumentation is not affected by the sample heating or cooling; here, extensometry can be required, which is difficult to achieve for low loads and small contact sizes. Temperature measurement should be arranged so that reasonable confidence can be gained that the real temperature of the samples is measured, usually by careful placement of thermocouples adjacent to the test samples. This is a particular issue for the moving sample where the stiffness of the thermocouple leads can affect the measurement of friction.

5.2 Test parameters

5.2.1 General

The behaviour of materials at tribological contacts is very dependent on a wide range of different test parameters. As the scale of the contact is reduced from the macroscale, the dominance of different factors changes so that factors, such as capillary forces due to the presence of liquids at the tribological contact, become critical. Typical test parameters that need to be considered include:

— contact geometry;

— applied load;

— motion type;

— relative speed;

— test system stiffness;

— interface materials;

— body material (probe);

— counterbody material (sample);

— surface cleanliness;

— surface topography and roughness;

— environment.

These different test parameters are discussed in the rest of this subclause.

5.2.2 Contact geometry

A critical factor is the geometry of the contact. Often the contact is between a shaped probe and a flat sample. This is normally preferred to using two flat samples as it is very difficult to get good alignment between flat samples. The contact stresses generated in the samples are critically dependent on their shape, so it is very important that the shape of the tip of the probe be well characterized before tests are started. In some cases both samples may be shaped such as the geometry used traditionally in surface force apparatus where two cylindrically curved samples are contacted.

Tip shape characterization is normally carried out by different microscopy techniques such as optical microscopy, scanning electron microscopy (SEM) and AFM.

Optical microscopy is used for the evaluation of overall tip shape and quality. Some optical microscopes based on confocal or focus variation techniques also have height measurement capability with a resolution of 10 nm or better, but the lateral resolution is limited to about 150 nm.

Scanning electron microscopy can be used very effectively but care is needed to ensure that the orientation of the probe is optimized for measurements of shape. It is also important to view the probe from several different angles as the image that is obtained from the SEM is 2D.

For very fine probes transmission electron microscopy (TEM) can also be used.

Perhaps the best method for evaluating the shape of probes is to use AFM to carry out a measurement of the tip form. This has the appropriate instrumental resolution in both height and in the lateral direction, and the information that is gained can easily be processed to give an analysis of how the tip shape deviates from that required. However, care is needed with the calibration and control of the AFM itself to eliminate or reduce non-linearities and scan hysteresis from the AFM scanning motion, and the calibration of the AFM tip shape can be in itself an issue.

Tip shape can also be evaluated by indentation into reference materials which have known indentation response. The shape of the indentation can be measured, and/or the shape of the tip can be calculated from the known indentation behaviour of the reference material.

Typical tip shapes are spherical, conical, and pyramidal shapes such as the Berkovitch indenter. The Vickers indenter is not recommended due to potential for wedge tip due to the four sided shape. Balls can be used for rolling contact tests, but care is then needed to ensure free motion with the small balls typically used in these tests.

Often diamond probes are used due to their ready availability and the perception that they do not degrade in contact with test materials. The use of diamond probes should be considered carefully as it is can be better to use materials more appropriate to the end application. Considerable degradation of diamond probes can also take place (see 5.2.8).

5.2.3 Applied load

The load that is applied between the probe and the test surface has a major effect on the magnitude of friction and wear which are observed. There is often an increase in wear as the applied load is increased, but if a change in mechanism occurs, this does not always apply. Particularly at low applied loads, the normal relationship that frictional force is proportional to the applied load (Amonton’s law) does not necessarily apply due to the effect of phenomena such as surface tension of any adsorbed water, which can dominate behaviour at small scales.

Normally, constant applied loading is used in these tests, but in systems where the load can be varied during the test other loading profiles such as ramping or incremental loads can be used.

5.2.4 Motion type

The motion type that is used also has a major effect on the wear and friction obtained. Common types of motion are reciprocating motion, where the probe moves backwards and forwards in the same path over the sample, uniaxial circular motion, where the flat sample is rotated in contact with the probe, and a raster test where the probe is moved in a raster over the surface of the sample.

One of the differences between these different types of motion is that they affect the ability of any debris that is generated in wear processes to escape from the contact zone. Thus in uniaxial sliding, the debris is often swept out of the contact zone. If the debris remains in place in the contact zone, it can form a third body that acts to separate the probe from the sample affecting friction and any further wear that may take place.

Another major effect is due to the magnitude of the motion itself, where if only a small magnitude of motion occurs such that an appreciable portion of the area in contact remains in contact through the motion, debris is trapped in the contact giving different mechanisms compared to the case where the surfaces move out of contact.

The magnitude of the motion is also important to consider. This should always be defined by consideration of the magnitude of motion that is experienced in the application concerned. As the magnitude of the motion is reduced it is more and more important to minimize unwanted backlash and slack in the motion. For very small magnitudes of motion with a scale of a similar size to the contact size, the contact mode changes from a sliding wear contact to a fretting mode contact and the mechanisms of damage and degradation change.

5.2.5 Relative speed of contact

The relative speed between the probe and the sample can also affect the magnitude of friction and wear that occur.

With a reciprocating motion different speed profiles can be used such as sine wave, triangular wave and square wave profile.

As the speed increases, the heat generated at the contact is also likely to increase since the power that is dissipated at the contact is given by $P = \mu N v$ where μ is the coefficient of kinetic friction, N is the normal applied load and v is the relative speed.

If the deformation that occurs in wear is rate dependent (e.g. visco-elastic material behaviour), changing the speed can affect the wear processes which occur.

5.2.6 Test system stiffness and inertial effects

The stiffness and inertia of the elements of the test system can have a major effect on the magnitude of friction and wear that occurs in a test.

Stiffness can affect results in two ways. If the force that is applied to the sample is generated through a compliant element in the system, any inaccuracy in the plane of motion such that the probe does not travel parallel to the surface leads to a tendency for the applied load to vary due to the altered force generated in the compliant element in the loading mechanism.

Where the sample surface is not flat, spring elements in the loading mechanism of the system can generate varying forces which can affect wear. In the worst case, surfaces which are rough can promote resonances in the mechanical elements of the loading system, creating high peak loads which can create great damage to the surface. Inertial elements, such as masses in the system can cause similar effects. Measurement of the resonance of the test system with and without samples in place can be carried out to measure the vibrational response of the test system. Vibration of the test system during a test is induced by the motion of the probe over the sample. The vibration often increases as the relative speed of the test is increased. It is important that the frequency of the induced vibration be kept away from the resonance frequency of the test system to minimize artefacts caused by the generated vibration.

5.2.7 Interfacial material

The presence of any interfacial material at the contact between the probe and the sample affects wear and friction. This effect becomes more and more important as the scale of the contact decreases, as the effect of water or other interfacial contaminants becomes more dominant and as the scale of the contact becomes smaller.

5.2.8 Materials

The materials of the two surfaces is important, not only because of reactions that can occur between the materials of the probe and the sample, but also because of potential reactions between the sample materials and the surrounding atmosphere and any interfacial material that can be present.

The materials are also important through their physical properties, such as thermal conductivity, which have a major effect on aspects such as the dissipation of frictional energy generated at the interface. The metallurgical state of the materials should also be considered, e.g. heat treatment, crystallographic orientation or work hardening.

It should be emphasized that both the material of the probe and the sample itself should be considered. A series of tests is often carried out with the same probe, but it is nevertheless essential that the probe material be considered as part of the tribological system.

5.2.9 Temperature

The temperature of the test samples needs to be kept constant or at least monitored during the test. For tests under ambient conditions, it is usually adequate to monitor the temperature of the environment of the test, as the heat generated in the tests described in this clause is normally very small and does not have a great effect on the overall temperature of the sample. However, the local temperature at the contact itself may be large, particularly for nanoscale contacts. There are few techniques which can be used to monitor the temperatures generated at these contacts.

For test systems with heating or cooling systems, the overall temperature of both of the samples needs to be monitored so that the sample temperatures are controlled to the desired values.

5.2.10 Surface condition

It is particularly important for small-scale contacts that the surface condition is well controlled to ensure that the likelihood of the presence of surface contaminants such as water and grease is reduced. Good sample handling procedures, such as the use of clean gloves and washing the sample with pure reagent grade solvents to remove contamination, should be followed.

It should be noted, however, that many materials such as polymers are sensitive to or can dissolve in solvents. In this case, another appropriate cleaning procedure should be used.

5.2.11 Surface roughness and topography

The roughness of the surface affects the results obtained through stiffness and inertial effects as outlined above. It is also difficult, if not impossible, to achieve meaningful results if the roughness of the surface is greater than the scale of the damage produced by the test. It is, therefore, important that the surface of the sample be prepared so that it is smooth.

5.2.12 Environment

The test environment needs to be controlled or at least measured so that test conditions can be reproduced. Tests may be carried out in air, in other gases or in vacuum, or in liquids. The purity of any gases or liquids that are used should be checked, as contaminants can have a major effect even at low concentrations.

Moisture has a major effect on the results of tests whose effect increases as the scale of the test is decreased. This arises from the likelihood of water adsorption or even condensation at high humidity levels at the contact. It is therefore important to measure the humidity of the air or gas in the environment surrounding the test, and it can be beneficial to have control of humidity with the ability to vary the humidity to examine its effect on results.

6 Test procedure

6.1 Different types of test

6.1.1 General

There are two main types of tests that can be carried out. These are friction measurements where the main goal is the accurate measurement of friction between the probe and the sample. The second type of test is focused on the measurement of the removal of material from the sample surface by wear processes.

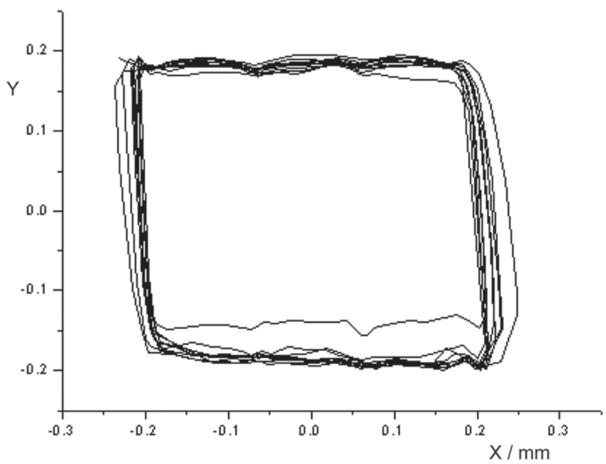
Of course, combined tests can be carried out where both friction and loss of material are measured.

6.1.2 Friction measurement tests

In these tests, the primary aim is the measurement of friction. Test systems that have the capability for friction measurement normally record the friction measurements digitally. In these tests, it is essential that the friction force itself be recorded, as Amonton's law is not necessarily obeyed, particularly as the scale of the contact is reduced.

If fluctuations in the applied load occur during the test, it is important also to record the applied load. If the friction coefficient is required, it can be beneficial to calculate the instantaneous friction coefficient by dividing the instantaneous values of the friction force and the applied load. However, if this is done, care needs to be taken to ensure that the data for both signals really were acquired at the same time; otherwise, large errors can occur in the calculation of friction coefficient.

The friction results can be presented in several ways. For reciprocating tests, plotting the friction against displacement throughout the contact path can be very useful giving a friction loop plot [see Figure 1 a)]. If the variation of the friction loop with time is also required, then a waterfall plot is a good way of presenting this information [see Figure 1 b)]. A trend plot of variation of average friction with time or number of cycles is also useful, care needs to be taken with the calculation of average friction. Several different methods can be used for this. One common method is to define a region of the friction loop, either a specific set of displacement positions or e.g. the central 50 % of the cycle and calculate an average value of friction for this region for every loop. Another method is to derive an average friction value for the loop from an integration of the area of the loop [see Figure 1 c)].

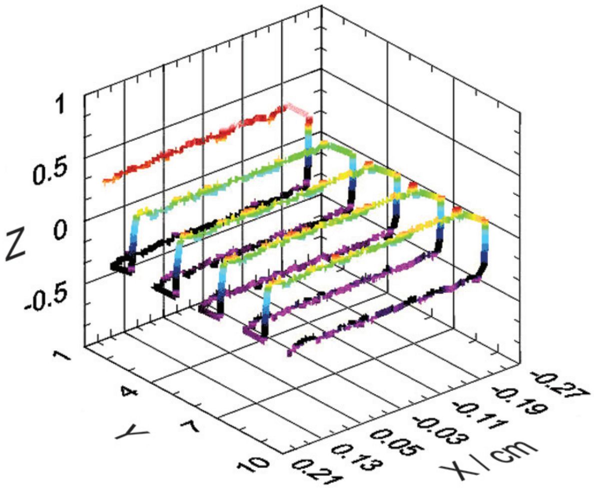


a) Friction loop

Key

X displacement

Y coefficient of friction



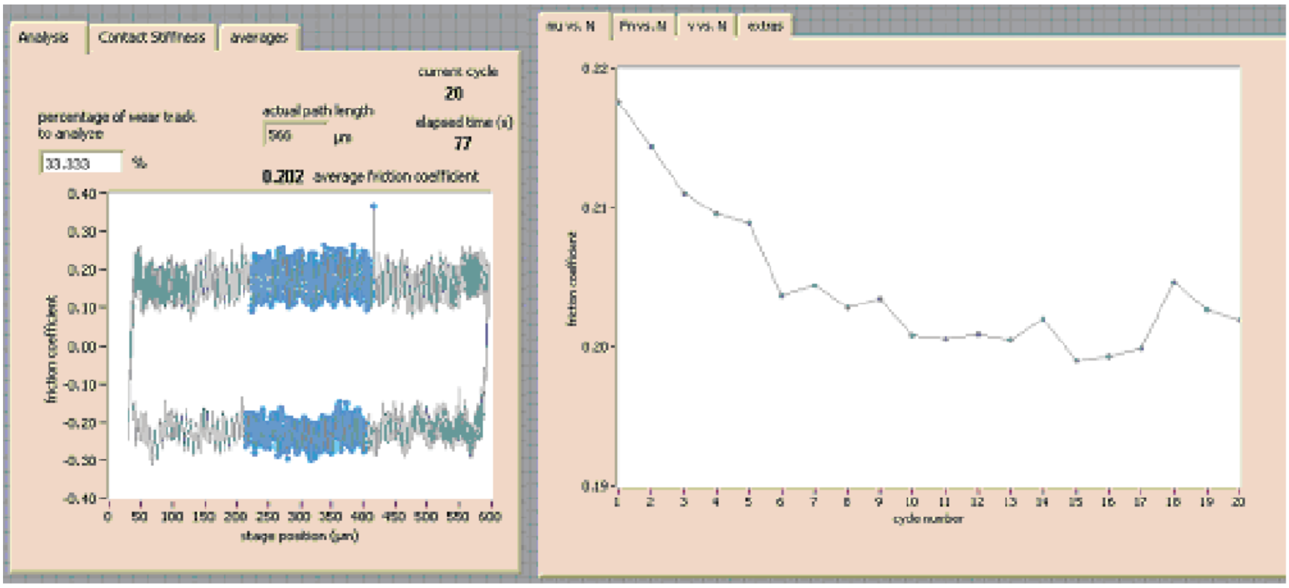
b) Waterfall plot

Key

X displacement

Y passes

Z coefficient of friction



c) Calculation of average friction value from central 50 % of friction loop and resultant trend graph

Figure 1 — Friction results from microtribology tests on patterned silicon wafer

6.1.3 Wear tests

In wear tests, the loss of material from the surface as repeated movement of the probe over that surface is measured. This can either be done through real-time tests where the movement of a fixed point on the probe towards the sample is monitored throughout the test, or can be carried out by measurement of damage after the test has finished. Note that the probe may also move away from the sample during a wear test, in the case of build-up of interfacial layers.

Real-time monitoring of the relative probe position with respect to the surface can be achieved and provides useful information, but it should be realized that this only gives a measure of the damage to both the materials in contact, that is the probe and the sample. In some cases, it is possible to use the probe to measure the original surface profile of the sample by carrying out an initial scan of the surface under a low load that is chosen to cause little or no damage to the surface before the test is started. This initial profile can then be used as an initial reference which can be subtracted from the subsequent measurements of probe position.

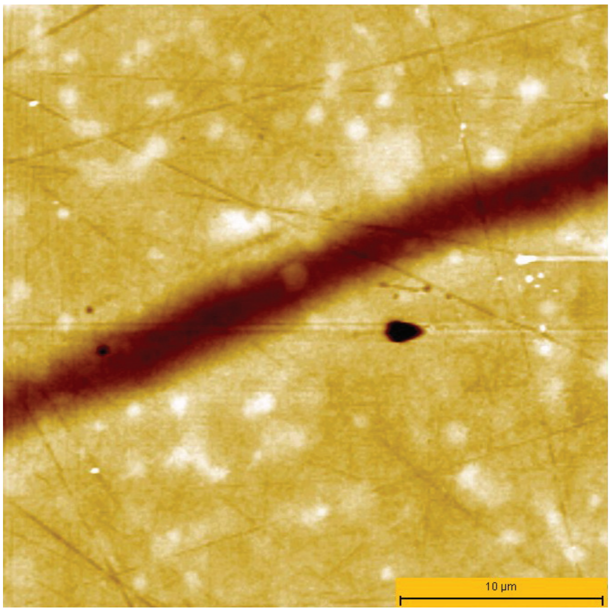
Measurements of damage to the sample can be carried out in a number of ways. Profilometry using AFM provides a good method for measurement of damage. Often this is carried out by measuring the profile of the wear track perpendicular to the direction of travel. This can either be done by measuring at a number of points (at least 3 points are recommended) along the track. Parameters such as the depth of damage, the width of the wear track, and the cross-sectional area of any damage are all important. If available, it is better to use an area scan and then calculate an average cross-sectional area from the area data (see Figure 2). This is particularly useful for scratches where there is variation in scratch morphology along the length of the scratch.

Confocal optical microscopy can also be used. This has good depth resolution (10 nm or better), but care should be taken to ensure that the technique has adequate lateral resolution to evaluate the wear damage effectively. The lateral resolution is limited by the wavelength of light.

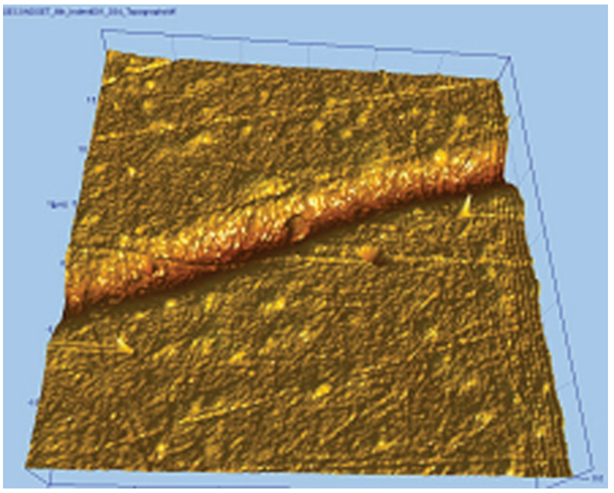
Another method to measure the damage is to use stereo SEM. In this technique, two or more images are acquired in an SEM at different tilt angles, and software is used to calculate the 3D form of the damage. It should be noted that there is often uplift at the edges of wear tracks so that some care is needed in defining these edges.

Since wear depths and wear volumes are measured relative to a reference plane, it is important to ensure that the data sets that are acquired are large enough, extending well beyond the damaged zone, so that the reference plane can be determined precisely.

If the material tested is a polymer or a material with significant time-dependent behaviour, the time between the test and the measurement of wear should be carefully chosen and documented. Some low Tg polymers flow and heal over long periods after damage is produced.



a) Conventional 2D image



b) 3D stereo image

NOTE The depth of the scratch is about 40 nm.

Figure 2 — Scanning electron micrograph of microtribology scratch made at load of 50 mN on 3 µm thick TiAlN coating on steel substrate with 10 µm radius diamond indenter

6.2 Surface examination techniques

An important part of any nanotribology test is the analysis of the changes to the surface which have taken place during the test. A very wide range of instruments is available.

It is important to examine both contacting surfaces, i.e. both the probe and the sample as changes can have taken place to both. It is often useful to examine surfaces before and after a test to identify whether or not any changes have taken place or if transfer films or contaminants are present.

Examination with optical microscopes is useful for looking at the overall form of the damage; however, their application is limited by the optical resolution limit.

Scanning electron microscopy (SEM) is frequently used, and with a resolution down to 1 nm or better for field emission instruments, has sufficient resolution for most purposes concerned with nanotribology tests (see Figure 3). The SEM images can also be coupled with energy dispersive X-ray spectrometry (EDS), which can give elemental chemical analysis with a resolution down to about 0,2 μm, and electron back-scattered diffraction (EBSD), which can give diffraction analysis (information on crystal structure and deformation).

Transmission electron microscopy (TEM) requires the preparation of electron transparent samples; however, if this is achieved, the TEM gives unprecedented information on structure with near atomic resolution. It is particularly useful to examine changes to the structure of samples below the surface of the contact area.

AFM is also often used to examine the topography of damage brought about by nanotribology tests and has resolution down to atomic levels.

There are also many other surface analysis techniques which can be used, such as secondary ion mass spectrometry, (SIMS), Auger and X-ray photoelectron spectroscopy (XPS), which give information on chemical structure with a depth resolution of a few nanometres.

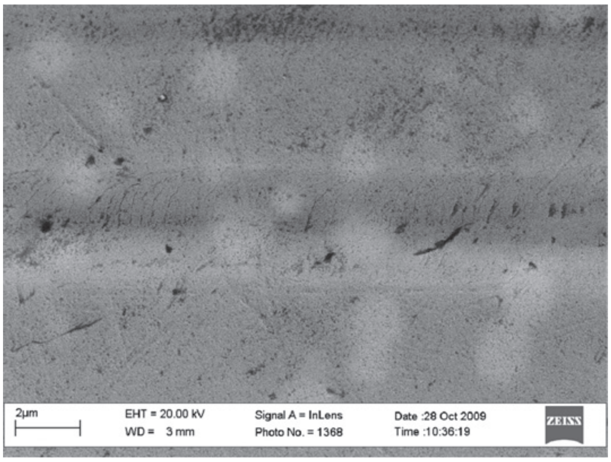


Figure 3 — Scratch made by microtribology test on 3 µm coating on steel substrate sample with 50 repeat passes of a 1 µm radius diamond indenter under a load of 50 mN

7 Test reproducibility, repeatability and limits

There are few data available on the reproducibility and repeatability of the types of testing described in this Technical Report. Interlaboratory exercises are needed to fill this gap.

Sources of uncertainty in these measurements come from machine effects, operator effects and intrinsic variability in results due to the stochastic nature of wear processes. The uncertainty due to machine effects can be reduced by good system design; the uncertainty from different operators can be reduced by following good, well-designed test procedures.

Many of these aspects of uncertainty can be checked with the use of well-defined reference materials. However, there are no commercially available reference materials for this type of testing, although in-house reference materials can be developed for in-house testing.

8 Test report

A coherent and comprehensive test report should be written to describe any work that is carried out. This should cover all the main elements, such as

— details of the test equipment and materials,

— the test procedure,

— the results,

— the discussion, and

— conclusions.

The report should be written in such a way that a competent person can reproduce the tests described. It is recommended that all of the parameters listed in 5.2.1 be reported.

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