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Nanotechnologies — Guidance on methods for nano- and microtribology measurements

Nanotechnologies — Directives relatives aux méthodes de mesure en nano- et microtribologie

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Foreword

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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.

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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.