Apparatus and method for testing materials

This invention relates to apparatus and method for testing materials. In particular, it relates to testing the surface and thin-film mechanical properties of materials on a small scale.

The small-scale properties of materials are of importance to engineers because such properties can be of significant importance to the performance of an engineered component on a macroscopic scale. For example, where components move over one another in a machine, the rate and manner in which those components wear, and their resistance to fatigue and fracture may be largely determined by the microscopic surface properties of the components.

One type of testing to which the surface of components is subject involves causing a force to repeatedly be applied by a test probe to a surface of a sample of a material. An example of such apparatus is disclosed in EP-A-1 095 254 of the present applicant. Apparatus for performing such a test conventionally comprises a pendulum rod upon which a probe is mounted and a sample holder for holding a sample to be tested in a position in which the probe can be brought into contact with the sample. During testing, the probe is held still, and the sample is moved against the probe under the action of a drive system that generates a cyclic load.

A disadvantage with such known test apparatus is that the speed of the probe with respect to the sample is in the order of several mm/s. In many applications, for example, where the material is intended to provide protection against a ballistic impact, the impact speed might be several orders of magnitude greater than this.

An aim of this invention is to provide apparatus that can perform impact analysis of materials in which high impact speeds do not lead to a substantial increase in the total energy transferred to the test probe through the contact point. The aim is to supply increased impact energy to a localised volume, without excessively damaging the underlying substrate material.

To this end, from a first aspect, this invention provides apparatus for testing the surface properties of a sample of material comprising:

- an impact target supported to have a default location and being resiliently moveable from the default location in a displacement direction;

- a sample holder that can carry a sample to be tested;

- a drive system that can drive the sample holder towards the impact target such that a sample carried on the sample holder impacts with the impact target at its default location at a known speed, thereby causing the impact target be be displaced from the default location in the displacement direction; and

- a position measurement system for measuring movement of the impact target in the displacement direction.

By driving the sample into the target, rather than oscillating one against the other, a significant impact velocity can be achieved between the sample and the target. This allows dynamic properties of the material under test to be understood. The position measurement system allows the effect of the impact of the sample upon the target to be measured.

The drive system may include energy storage means for storing energy that can subsequently be delivered to the sample holder as kinetic energy. Such an arrangement can be calibrated to deliver a precisely known amount of energy to the sample holder. The energy storage means may comprise a spring, such as a compression spring. The spring typically acts between an abutment and the sample holder. The sample holder can be latched against movement towards the impact target while energy is stored in the energy storage means. Upon unlatching of the sample holder, energy is released from the energy storage means, so driving the sample holder towards the impact target. In preferred embodiments, the abutment is positioned by an electrically-controlled actuator such as a piezo-electric stack. Such an arrangement allows a precise amount of force to be applied to the spring through the abutment under the control of an electrical signal, which means that a precisely-controlled and readily-adjustable amount of energy can be stored in the spring.

The apparatus may further comprise a latch assembly that can be used to latch or unlatch the sample holder. The latch assembly may include a latching detent that can be moved between a latching position and an unlatching position under the control of an electrically-operated actuator such as a piezo-electric stack. Such an arrangement allows close control over the instant at which the sample holder is unlatched.

Apparatus embodying the invention typically further comprises a data logging system operative to store multiple data samples obtained from the position measurement system. Most preferably, the data logging system starts to operate a predetermined time after operation of the drive system. This ensures that the data logging system does not attempt to capture data from the position measuring system before a finite time prior to occurrence of the impact. Some data may be captured immediately prior to impact to record the initial probe position.

From a second aspect, the invention provides a method of testing a material in apparatus according to the first aspect of the invention, the method comprising, in an impact cycle, driving the sample holder towards the impact target to cause a sample to impact with the impact target and using the position monitoring system to determine the displacement of the impact target from its default location.

Testing methods embodying the invention may obtain useful information by analysing the displacement arising from individual impact cycles. Alternatively or additionally, analysis may be performed on the results of multiple impact cycles on one sample. For example, methods may compare the maximum displacement of the impact target that occurs during multiple impact cycles. In such methods, impact cycles are typically performed at a frequency that is sufficiently low to allow transient effects of one impact cycle to cease before commencement of a subsequent impact cycle. For example, impact cycles may take place at a frequency ranging from 0.1 to 2 Hz.

An embodiment of the invention will now be described in detail, by way of example, and with reference to the accompanying drawings, in which:

Figures 1 and 2 are plan and side views of test apparatus being an embodiment of the invention;

Figure 3 is a simple schematic of a electronic control system of the embodiment of Figure 1;

Figure 4 is a graph of probe displacement with respect to time during one impact cycle of operation of the embodiment; and

Figure 5 is a graph of maximum probe displacement during multiple impact cycles of a test procedure using apparatus embodying the invention.

Introduction

The purpose of this embodiment of the invention is to measure a range of surface and thin film mechanical properties. The basic properties that can be measured include:

- Young's modulus in the range MPa – GPa;
- hardness in the same range;
- scratch and wear resistance of typical wear-resistant coatings and surface treatments;
- fatigue resistance;
- fracture toughness; and
- test probe-to-surface frictional forces.

This embodiment has particular advantages in its measurement of the fourth and fifth of these properties, and of *dynamic* hardness.

The deformation response of a material depends to a greater or lesser extent on its strain rate as well as on its static strain. That is, some materials are more strain-rate-sensitive than others. Hardness tests, which are used as simple and rapid methods of estimating material strength, are usually carried out at strain rates considerably lower than those experienced in common industrial operations, such as machining. Hardness values measured at high strain rates are often higher than those obtained in standard hardness tests, and extrapolation of low strain rate results to material performance in high strain rate applications may not be valid.

One aim of the test apparatus is to measure the energy absorbed during an impact and the volume of an indentation caused by an impact. Energy *per* unit volume has the dimensions of hardness, but at high indentation speed (as opposed to slower, quasi-static testing) this quantity is known as *dynamic hardness*.

To determine the energy absorbed, the contact velocity is first measured in a calibration procedure. In this procedure, A 'hard' contact is made between the pendulum and the sample holder in order that the pendulum will follow the sample holder motion exactly without any bouncing taking place. This is achieved by first mechanically attaching a metal rod to the sample holder and then moving an outer end into the pendulum probe holder where it is clamped by means of a screw. Displacement *v*. time (and hence velocity) data are then recorded for various spring parameters.

The velocity at which the test probe loses contact with the sample is then calculated from displacement *v*. time data recorded during an impact test. Since the effective mass of the test probe and the associated components that move with it are known, this allows the difference in kinetic energy, and thus the energy absorbed, to be calculated. The indentation depth (and volume) is determined by placing the indenter back inside the indentation and recording the displacement relative to the initial surface position. The indenter load at this point is very low so the displacement measured is simply the plastic (or permanent) displacement. That is, there is no significant contribution from elastic deformation. Alternatively, the sample can be moved to another measuring device, such as an atomic force microscope for accurate measurement of the indentation

In addition to quantitative measurement of dynamic hardness, an extremely important application of embodiments of the invention is that of impact fatigue testing. This investigates the propagation of fatigue damage, which eventually causes an abrupt failure of a surface or coating. To achieve this, repetitive impacts are produced at one place upon the sample. In some cases a gradual increase in penetration depth occurs, but in other, more interesting cases, the depth remains almost constant until coalescence of defects leads to a sudden depth increase. This is associated with surface fracture and material removal (impact wear), coating fracture, or coating adhesion failure.

Embodiments of the invention can be used to test a wide range of materials, including, but not limited to:

- wear-resistant thin film coatings, such as those often applied to cutting tools;
- cutting tool materials *per. se.*;
- materials subjected to erosive wear, such as vehicle paint, aircraft windows, powder spray nozzles, *etc.*;
- polymers, including plastic bumpers or components for vehicles, polymeric windscreens for commercial aircraft, plastic bottles, electronic components, *etc.* Impact data allows optimisation of polymer composition, e.g. through the addition of components with good impact properties; and
- soft tissue and bone for the assessment of human injury risk.

Description of the Apparatus

The apparatus is shown in Figure 2 as it will be disposed for use. The terms “upper”, “lower” and related terms should be interpreted as referring to the apparatus in this position. What will be referred to as the “front” of the apparatus is towards the left of Figure 2, and the “rear” is towards the right. The terms “forward” and “rearward” should be interpreted accordingly.

Test apparatus embodying the invention comprises two main assemblies: the sample-holding assembly and the probe assembly. Only the sample-holding assembly is shown in Fig 1.

With reference to Figures 1 and 2, the sample-holding assembly comprises a sample holder for holding a sample 10 that is to be subject to testing and other components that act as a drive system for the sample holder. The sample holder is carried on a vertical end surface of a horizontal, forward-extending, elongate push rod 12. The push rod 12 extends from a front surface of a carrier block 14.

The carrier block 14 is supported on a stage 16 by a pivot 18 that has a pivot axis that is normal to a long axis of the push rod 12. The pivot is of a low-friction type, such as a spring pivot, to minimise frictional losses at that component. Thus, the carrier block 14, the push rod 12, and the sample holder on it can move around the pivot 18. Because the amount of rotation is small, this results in movement of the sample holder that is substantially along the axis of the push rod 12. The carrier block 14 has a rear surface normal to the long axis of the push rod, and an upper surface that is normal to it. A step 50 is formed in the upper surface, extending transversely of the long axis of the push rod.

Also carried on the stage 16 is a slider block 20. The slider block 20 can slide on the stage 16 in the direction of the long axis of the push rod 12. It has parallel front and rear vertical faces. A compression spring 24 is located between the rear surface 22 of the carrier block 14 and the slider block, such that it acts on the rear surface of the carrier block 14 and the front surface of the slider block 20.

An abutment block 28 is carried on the stage 16, and has a forward-facing, vertical abutment face that is directed towards and parallel to the rear face of the slider block 20. The abutment block 28 is fixed against movement with respect to the stage 16. A piezo-electric force stack 30 is located between the slider block 20 and the abutment block 28. The force stack 30 acts between the rear face of the slider block 20 and the abutment face.

The apparatus further includes a latch assembly 40 for the carrier block 14. The latch assembly 40 is supported above the carrier block 14 on a frame 42 that is secured to the stage 16. The frame 42 ensures that minimal movement can take place between the latch assembly 40 and the stage 16. The latch assembly 40 comprises a latch arm 44 that extends transversely above the carrier block. The latch arm 44 is pivoted close to one of its ends about an axis that is parallel to the long axis of the push rod 12. A latch detent 46 projects downwardly from the latch arm 44 generally towards the step 50 in the upper surface of the carrier block 14. The latch detent 46 has a rear-facing, vertical latching surface from which a lower surface extends, sloping upwardly towards the front of the detent 46.

A piezo-electric release stack 48 is positioned between the latch arm 44 and the frame 42 and is secured to both of them. Application of an electrical signal to the release stack 48 causes its length to change, which consequently causes the distance between the latch arm 44 and the frame 42 to change, this change being accommodated by pivotal movement of the latch arm 44. Thus, the vertical position of the latch detent 46 can be moved by variation of the electrical signal that is applied to the release stack 48.

The probe assembly will now be described. Although not shown in the figures, the probe assembly is constructed on a rigid base that is essentially immovable with respect to the stage 16. Components that are described as “fixed” should be taken as being immovable with respect to the rigid base.

The probe assembly comprises a vertical pendulum rod 80 formed from high-density alumina. The rod 80 is carried on a fixed pivot 82 that allows the rod 80 to pivot freely about a horizontal axis that is transverse to the long axis of the sample push rod 12. A coil 84 is carried on the pendulum rod 80 above the pivot 82, the coil 84 being positioned next to a fixed magnet 86. Variation of current in the coil 84 can cause it to be attracted towards or repelled from the magnet 86, with a variable force, so urging the rod to move about the pivot 82. An adjustable limit stop 88 is used to ensure that the rod 80 adopts a vertical disposition to ensure that the axis of the probe 92 (see below) is normal to (or is at a specific desired angle to) the specimen 10. Also carried on the rod 80 is an adjustable balance weight 98 that can be moved along a screw thread to vary its position with respect to the rod 80. This is adjusted to ensure that the rod 80 is balanced in a near-vertical position immediately before testing commences (that is, once the probe assembly is complete). Adjustment of current in the coil 84, and consequent variation in the force between it and the magnet 86, can be used to reduce or eliminate any remaining deviation of the rod 80 from vertical.

A probe holder 90 is carried on the rod 80 below the pivot 82. The probe holder 90 has a horizontally projecting rod that projects towards and is substantially coaxial with the sample push rod 12. A probe 92, which serves as the test target, is carried on an end surface of the horizontally projecting rod and is directed towards the sample 10. The probe holder 90 carries a conductive plate 94 that faces directly away from the probe 92 on the opposite side of the rod 90. The conductive plate 94 is closely spaced from and parallel to a fixed plate 96. The two plates 94, 96 together form a capacitor, the capacitance of which varies as the spacing between the plates 94, 96 changes as a result of pivotal movement of the rod 80. As is well recognised, it is possible to measure the capacitance of a variable capacitor and, with suitable calibration, establish a relationship between the capacitance and the separation of the plates. After suitable calibration, this allows the instantaneous position of the probe holder 90, and therefore, the probe 92, to be measured.

A damper 78 comprises a pair of optically flat damping plates with variable spacing. One plate is fixed and the other is attached to the pendulum 80. Normally, during testing, the spacing is set to be large and air damping that occurs between the plates is insignificant. The spacing can be reduced to reduce the impact velocity during testing, if required. The damping plates are normally used to alleviate the effects of vibration in high sensitivity indentation testing.

An adjustable stop 120 is provided to limit the extent to which the pendulum can swing and so limits the extent to which the sample 10 can move towards the probe 92. This stop allows high strain rates to be present during testing on the surface of the sample, but prevents the sample 10 from overshooting and damaging the substrate material or the pendulum rod 80 or its pivot 82.

With reference now to Figure 3, a control system of the apparatus of Figures 1 and 2 will be described in overview.

Operation of the control system is controlled by a microcomputer 100 connected to a system control and power bus 102.

The coil 84 is supplied with current from the output of a first digital-to-analogue converter D/A1. The output of D/A1 can be set by the microcomputer 100 in the range –Vref to +Vref. A second digital-to-analogue converter D/A2 has an output that is controlled by the microcomputer 100 and that is connected to a control input of D/A1 to set Vref for D/A1.

Therefore, the force applied to the arm through the reaction of the magnet 86 to the coil 84 can be controlled by the microcomputer 100 through D/A2.

The force stack 30 is driven by an amplifier AMP1 that has an output controlled by a digital-to-analogue converter D/A3 controlled by the microcomputer 100. The output of D/A3 is calibrated such that it can cause the force stack 30 to exert a known force on the slider block 20.

The release stack 48 is driven by an amplifier AMP2 that has an output controlled by a digital-to-analogue converter D/A4 controlled by the microcomputer 100. Output of D/A4 is also fed through a delay 104 to a control input of a trigger circuit 106.

The capacitance of the plates 94, 96 is measured by a bridge circuit 108. Output from the bridge circuit is fed, through the trigger circuit 106, to an analogue-to-digital converter A/D. Output from A/D is stored in a fast memory 110 that can be accessed by the microcomputer 100. Data is stored only when the trigger circuit is activated – this being at a time after a signal is sent to the release stack 48, the time being controlled by the delay 104.

In this example, the A/D has a maximum sampling rate of 625 kSamples/s and the memory 110 has a capacity of 100 kSamples, making a total sample time of 160ms. The memory capacity and/or sample rate could be increased if required for a particular measurement to be made.

Operation and use of the apparatus

Operation and use of the apparatus will now be described.

To use the apparatus, a sample is mounted on the sample holder and a suitable probe is selected. Test probes may be similar to those used in conventional test apparatus, these being multi-faceted (e.g., pyramidal) or spherical diamonds. However, for impacting softer materials the probe material could, in the alternative, be a ceramic or a hard metal. Pyramidal-shaped probes may be 3-sided, 4-sided or conical, with a tip radius of the order of 100 – 500nm. A preferred pyramid shape is the cube corner. Spherical probes can range in diameter from 2 μm to several mm. A current is then applied to the coil 84 of just sufficient magnitude to cause the rod 80 to rotate about the pivot 82 until the rod is vertical, to set the default position of the test target. This compensates for any variation in the weight of the probe 92 and normalises the output from the capacitor 94, 96.

A sample to be tested is mounted on the sample holder 10. Note that the sample does not necessarily need to be perpendicular to the impact direction. In some cases, it is advantageous to tilt the sample, such that impact from the probe occurs upon a surface angled to the impact direction, for example, at 45°. This can increase the interfacial shear stress and more closely simulate practical coating applications.

The complete assembly of Figure 2 is mounted on a motorised micrometer stage for coarse position adjustment. The position of the abutment block 28 is manually adjusted and then clamped (rather than fixed). This allows initial positioning of the force application components prior to energisation of the piezo stack.

The controller causes a signal to be applied to the force stack 30 such that its length is minimised, the slider block 20 is drawn rearwards, pulling the carrier block 14 rearwards also. The step 50 of the carrier block 14 moves towards the rear of the latch detent 46, force acting on the slope of its lower surface urging the latch detent 46 upwards until the step 50 has passed to its rear. The latch detent 46 can then move downwards, such that subsequent contact between the step 50 and the rear surface of the latch detent 46 prevents forward movement of the carrier block 14. The carrier block 14 is then said to be “latched”.

Small pre-loads are applied to both the pendulum rod 80 (using the coil 84) to place it on the limit stop 88 and to the carrier block 14. Optionally, an arbitrary additional load can be applied, for example, to reduce the tendency of the probe to rebound from the sample. The carrier block is latched and the complete assembly of Figure 2 is then moved towards the probe assembly by means of the motorised micrometer stage until the sample 10 just makes contact with the probe 92. It is then moved in the opposite direction to produce a pre-determined gap (typically several microns) between the sample 10 and the probe 92. The apparatus is then ready to start a test. If a signal is then applied to the force stack 30 to increase its length, this causes the slider block 20 to move forwards (the abutment block 28 preventing any rearward movement). Since the carrier block 14 cannot move forward, the spring 24 is compressed.

Subsequent application of a signal by D/A4 to the release stack 48 to cause its length to increase causes the detent to move upwards to unlatches the carrier block 14. Under the action of the spring 24, it accelerates rapidly forwards, thereby urging the sample holder 10 and a sample mounted on it towards the probe 92 to impact with it. A short time after unlatching of the carrier block, the trigger circuit 106 is activated, and then data is sampled from the bridge circuit 108 which effectively logs movement of the probe 92 caused by impact from the sample. A short time after the impact, a signal is applied to the force stack 30 to withdraw the sample so that it is not impacted by the probe 92 during a subsequent rebound, so completing an impact cycle.

It will be realised that, since it has mass, the sample 10 takes some time to reach its maximum speed after the carrier block 14 is unlatched. The probe 92 can be positioned at such a distance from the sample 10 that the maximum speed is achieved at the instant of impact.

While useful information can be gained from a single impact, to obtain further information, each test may comprise multiple impact cycles, with the result from each cycle as well as the pattern of change in results from one cycle to another both providing additional useful information about the properties of the sample. Sufficient time is allowed to elapse between the end of one impact cycle and the start of the next to allow any transient effects, such as "ringing" (vibrations within the sample or the probe) or localised heating, to disperse. Typical repeat frequencies are in the range 0.1 -2 Hz.

Interpretation of the Results

With reference to Figure 4, a typical result from one impact cycle is shown qualitatively, with displacement of the probe 92 (as captured in the memory) being shown on the y-axis against time on the x-axis. The direct measurement is of probe displacement against time. This allows the probe velocity to be calculated at each point along its trajectory.

In Figure 4, the carrier block 14 is latched at time $t=0$. Then, at time $t=t_1$, the carrier block is unlatched, and the carrier block 14 starts to move causing the sample to impact with the probe 92. The impact causes the probe 92 to be driven forwards, until the carrier block 14 is arrested, and further movement of the sample stops, at time $t=t_2$. The momentum of the probe

92, and all other components of the pendulum 80 means that the probe 92 does not stop immediately, but continues to move forward; however, this movement is against gravity and, optionally, against a mechanically-applied pre-load, so is eventually halted, at $t{=}t_3$, and the pendulum then returns to the vertical position.

Since the physical properties of the test apparatus are known, it is possible to calculate from the displacement curve such as that shown in Figure 4, the amount of kinetic energy that is absorbed by the probe as a result of the impact. Since the speed of the test probe is known, and the effective mass of the pendulum (including the test probe) can be determined, the kinetic energy transferred from the sample to the probe can be calculated. Since the kinetic energy of the pendulum for a fixed contact which does not experience energy absorption can also be determined simply by replacing the probe-sample with a probe-mechanical clamp, the difference between these is the amount of energy absorbed by the sample during the impact, from which the dynamic hardness of the material can be calculated

The effective mass of the pendulum is not the same as the actual mass since account must be taken of the position of the probe relative to the pendulum pivot and the moment of inertia of the pendulum. The effective mass is determined by connecting a spring of known stiffness between the probe holder and the sample holder, then manually displacing and releasing the pendulum, and finally measuring the resulting oscillation by means of the displacement measurement electronics. The resonant frequency thus obtained allows the effective mass to be calculated.

In order to assess the effect of wear that occurs over a period of time, typically in a periodic or cyclic fashion, the above-described impact test is performed multiple times, and a result of each test is plotted against time – time being, effectively, equivalent to the number of impact cycles. In the example shown in Figure 5, the result that is plotted is the maximum depth reached by the probe during consecutive impacts.

Figure 5 is typical of a test sequence in which a material coating or hardening case fails through fatigue after repeated impacts. In the initial phase, from $0 < t < t_1$, the material is resisting penetration of the probe, and the maximum depth remains constant. However, around $t = t_1$, there is a sudden increase in depth. For a further period $t_1 < t < t_2$ the depth of penetration is again constant (although greater than it was initially) before a further sudden increase occurs at around $t = t_2$.

A coalescence of defects in the sample can lead to a sudden depth increase, such as those shown in Figure 5. This is fatigue damage associated with surface fracture and material removal (impact wear), coating fracture, or coating adhesion failure.

Alternative embodiments of the invention can be contemplated. For example, hydraulic or pneumatic energy storage means or another mechanical arrangement may be provided as an alternative to the spring 24. Devices other than a piezo-electric stack can be used to supply energy to the energy storage means. For example, a motor, linear actuator, or other device may be used.