APPLICATIONS BULLETIN

Modified Pin-on-Disc Tribometer for controlled lubricant studies

Introduction

In this issue we feature a modification made to a standard CSM pin-on-disk Tribometer which allows the friction coefficients of different lubricants to be accurately assessed and compared. Users in the field of automotive engine oils are not necessarily interested only in the lifetime of a particular lubricant but also in a quantitative method of determining the service frictional properties. This means that a service temperature of 100-140°C must be maintained during the test and that a load arm capable of compensating for thermal drift must be used in order to reduce discrepancies in the frictional signal.

The standard pin-on-disk Tribometer uses a simple load arm with a tangential force sensor mounted close to the contact point so as to reduce errors due to arm compliance. However, for elevated temperature use, a different configuration is needed to avoid local heating and dilation within the sensor. For this reason a load arm from a CSM High Temperature Tribometer was adapted and fitted to a standard Tribometer. This arm uses two LVDT (Linear Voltage Differential Transformer) sensors which measure the tangential force independently after which an average value is computed and plotted as either a straight or an integrated signal.

The sample is clamped in an oil bath (see Fig. 1) and an external heating element is located around the sample so as to give homogeneous heating. A thermocouple is placed in the bath in order to accurately monitor the sample temperature. A separate regulator controls both heater and thermocouple. The oil bath shown below can be supplied on request together with the necessary fixture for securely clamping the sample.

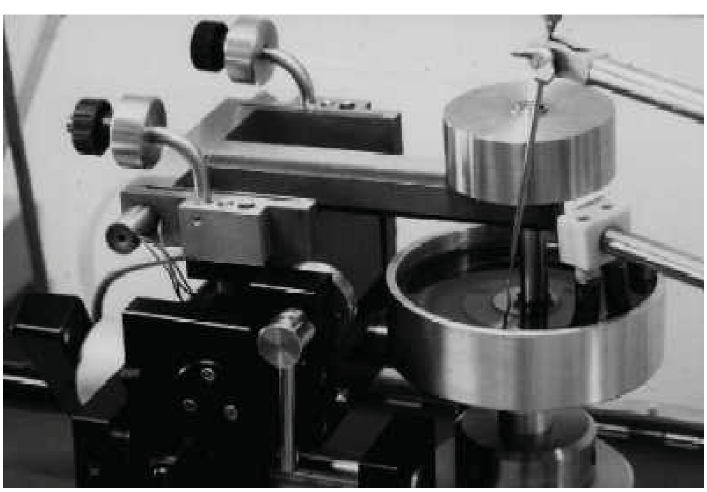


Figure 1 : Detail of the modified Tribometer with external heating element and thermocouple. The sample is completely submerged in the oil bath.

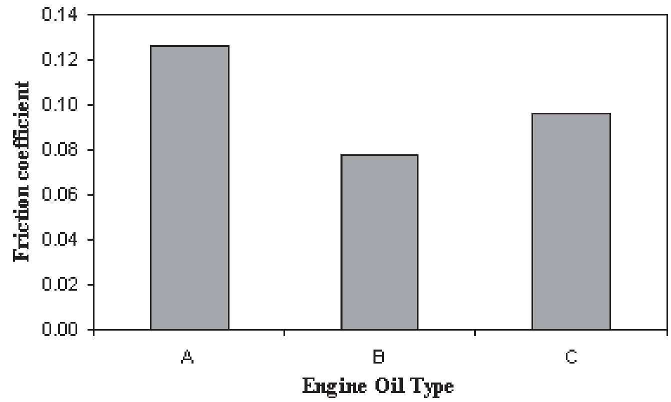


Figure 2: Friction coefficients for a selection of three commercially available automotive engine oils, after ball-on-disk testing at 120°C. Both ball and disc were of 100Cr6 steel and test duration was 60 minutes (speed = 30 rpm).

Results

To demonstrate the modified Tribometer, three similar automotive engine oils having slightly different composition were selected for ball-on-disk testing. The results are summarised in Fig. 2, in which it can be seen that oil type B has the lowest friction coefficient for the specified testing conditions.

The stability of the high temp. load arm, in comparison to that of a standard Tribometer, is demonstrated in Fig. 3 for a constant temperature of 120°C. Clearly the variation of friction coefficient is very stable for the high temp. arm. However, a normal load arm used at such temperature has quite a significant drift.

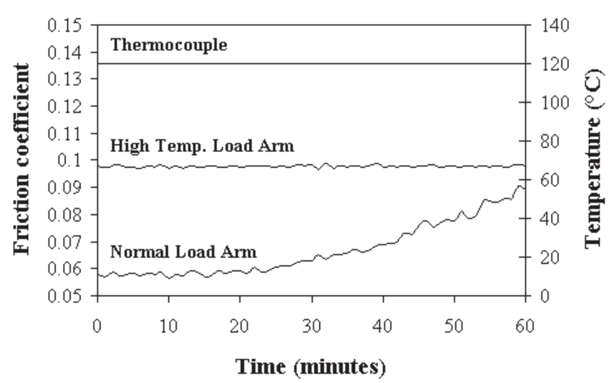


Figure 3: Comparison of friction coefficient variation between the high temperature load arm and the normal load arm over a period of 60 min. at 120°C.

Diamond knife for MST characterisation of coated optical fibres

Introduction

In the rapidly growing field of optical fibres for telecommunication and data transmission, such fibres are often coated for aesthetic, protective and structural reasons, as well as to prevent light intensity being lost to the surroundings. These coatings usually have thicknesses of the order <500 nm, the fibres having a diameter of ~ 200 m m.

Measuring the mechanical properties, in particular the adhesion between coating and fibre, is very difficult owing to the curved geometry of the surface and the problems of positioning a pointed diamond tip so that it moves along the longitudinal axis of the fibre during a scratch test.

This problem has been solved by using a specially manufactured diamond knife with a tip angle of 80°, although other angles have also been tested. The knife (see Fig. 1) is made using a high-purity natural diamond with its [110] orientation along the axis of the blade, ground to the desired geometry and angle, and mounted on a standard stub for fitting to the Micro Scratch Tester (MST). The company producing the knives is Diatome Ltd., Bienne, Switzerland.

Application

A selection of optical fibres having different metallic coatings have been tested with the modified MST (as outlined in Application Bulletin No. 3). It was found sufficient to hold the samples securely by taping them down to the flat sample table, and leaving a small section exposed for testing. The large blade width (several mm) made positioning very easy without any risk of the blade sliding off the side of the coated fibre. However, it was found important to mount the blade exactly perpendicular to the fibre in order to ensure reproducible results.

A typical load range was from 0 to 100 mN over a length of 5 mm, giving an adequate load ramp and well-defined critical failure points. Scanning Electron Microscopy (SEM) of the knife blade before and after use confirmed that no visible damage had occurred as a result of scratching under load.

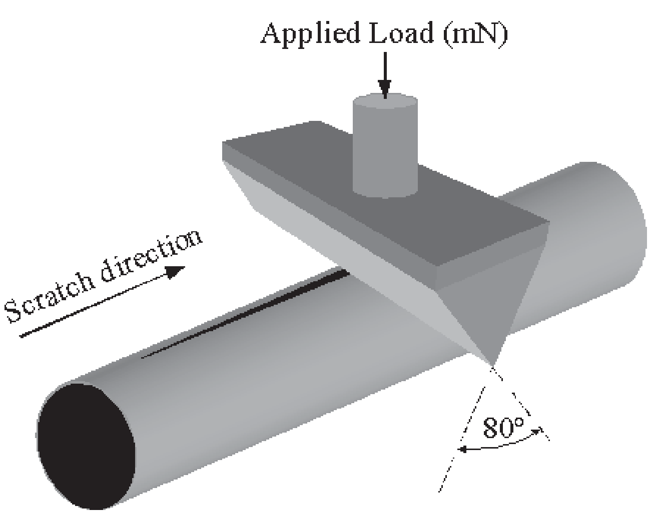
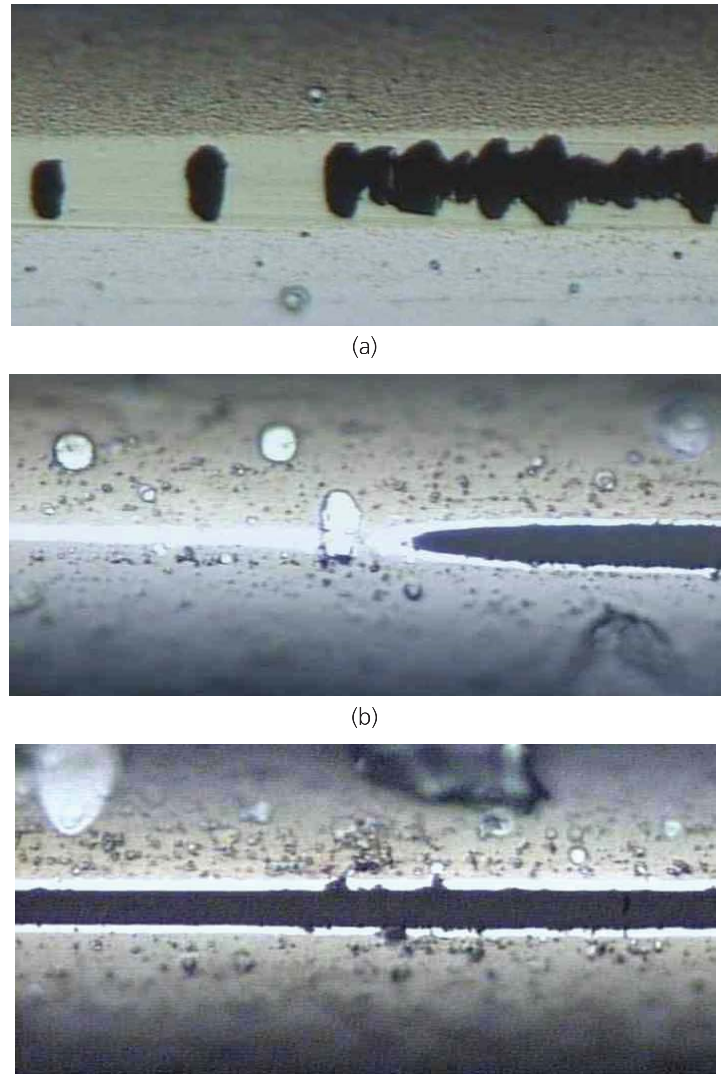


Figure 1 : Principle of scratch testing on coated optical fibres using a sharp diamond knife of angle 80°.



(a)

(b)

(c)

Figure 2 : Results for a 200 nm coating (a) and for a 500 nm coating, (b) and (c). Note the brittle flaking of material for the former, whereas the latter has a distinct point at which the substrate is reached, after which cracking and delamination occur. Scratch direction is from left to right (mag. 500x).

Results

The optical micrographs in Fig. 2 show the different types of coating failure encountered using this method. The 200 nm coating had two critical failure points; the first being the onset of chipping, the second being the point at which continuous chipping begins and the substrate is attained. Both modes are shown in Fig. 2 (a). For the 500 nm coating, no initial chipping occurred, the only critical failure point being where the substrate was reached. However, it is interesting to note that at loads greater than this, cracking and delamination was evident along the sides of the scratch path, as depicted in Fig. 2 (c).

The successful use of a diamond knife have been shown for an industrial application where such a method is the only one capable of adequately controlling the adhesion between such coatings and optical fibres of small diameters. Future work is envisioned with different knife angles to investigate their potential for other sample geometries which cannot be measured by the conventional MST technique.

Controlling magnetic hard disk adhesion to ceramic substrates with the MST

Introduction

In this application the Micro Scratch Tester (MST) is demonstrated for the routine quality control of the adhesion between magnetic hard disk multilayered coatings and their ceramic substrates. This a good example of where the acoustic emission, frictional force (Ft) and subsequent optical microscopy together yield a multitude of information relating to the modes of failure.

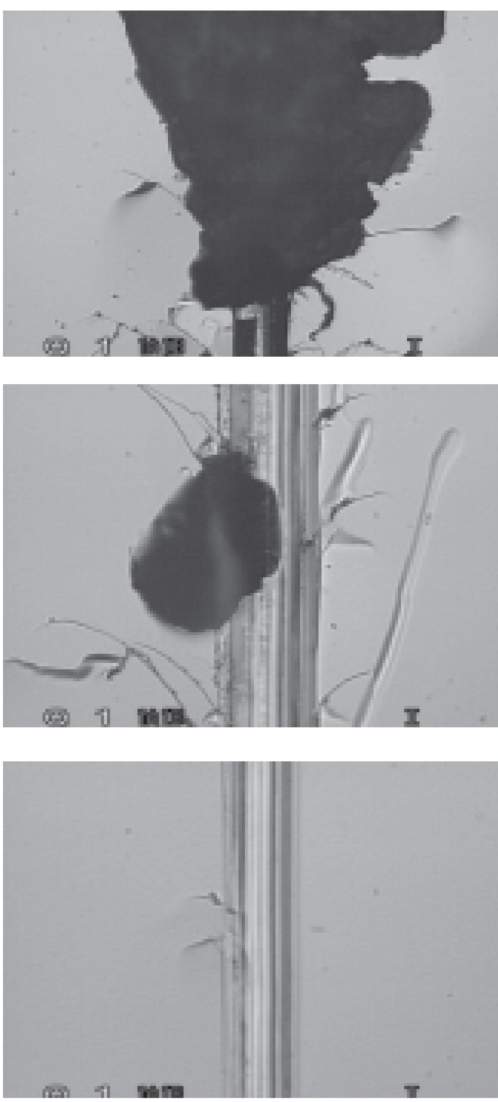
In the scratch test method, scratches are generated on the sample using a diamond stylus which is drawn across the sample under either constant or progressively increasing load, resulting in elastic and/or plastic deformation. At a certain load, damage occurs in the scratched region: the adhesive strength of the coating-substrate system being characterised by the critical load, Lc. For the progressive load scratch test, it can be defined as the smallest load at which some recognisable failure event occurs and the load bearing capacity is lost. For the constant load test, the regular occurrence of failure along the scratch path is required. Not all failures depend on the adhesion of the coating-substrate system; the properties of the coating and substrate will also play an independent role, e.g., the surface roughness and friction coefficient. However, it should always be remembered that the scratch test is basically a comparison test.

Results

Fig. 1 shows a complete set of results obtained with the MST for a multilayered hard disk coating on a ceramic substrate. The graph shows the acoustic emission (AE) signal and the frictional force (Ft), both plotted as a function of applied load. For this coating-substrate system there are three distinct critical loads at which different failure modes can be recognised by optical microscopy. These critical loads are also seen to correspond directly with sudden increases both in the acoustic signal and the frictional force.

The first critical failure point, Lc1, is characterised by a sharp increase in AE and Ft and corresponds to cracking of the coating at the sides of the scratch path. The second critical failure point, Lc2, shows a smaller jump in AE and represents the first sign of delamination from the substrate, seen as removal of a chip or flake from the surface. Interestingly enough, the optical micrograph also shows the greater propagation of scratches away from the scratch path and some buckling to the right of the image. As this is a multilayered coating it is probable that the buckling effect is caused by mismatch between the uppermost layer(s) and those beneath, resulting in delamination at a certain distance from the track. Final failure, Lc3, is characterised by total delamination from the substrate, an event which could only be observed accurately from the optical micrograph.

These results clearly show the importance of combining both the data plotted by the instrument with that from subsequent optical microscopy in order to gain a more overall picture of the true failure mechanisms.



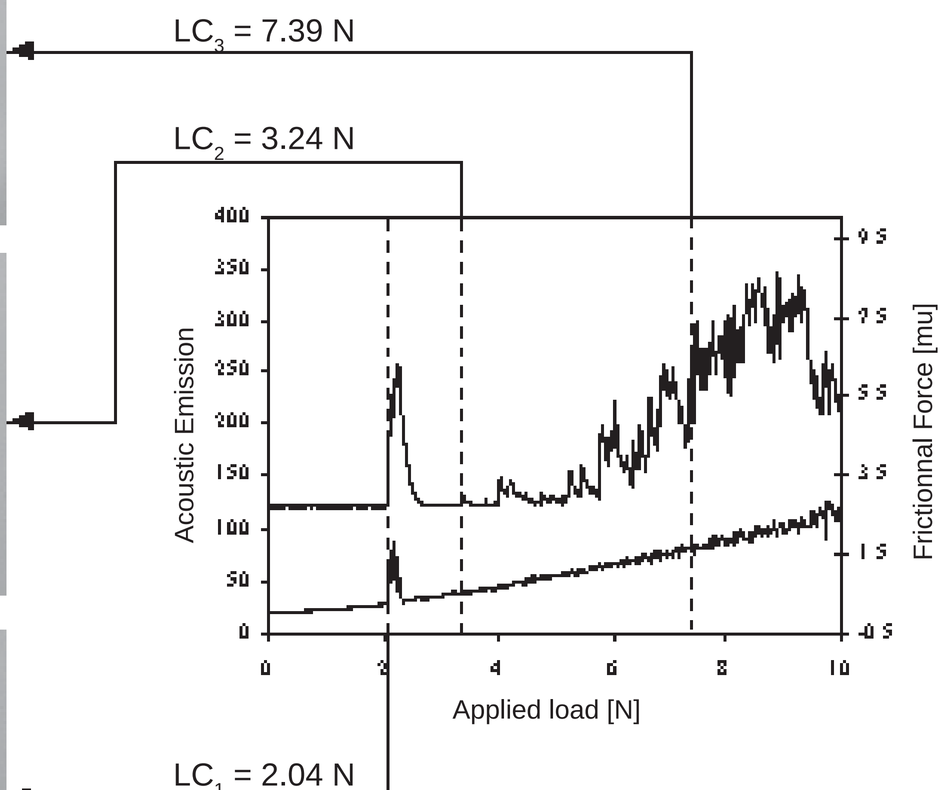


Figure 1: Typical results obtained with the MST for a scratch performed on a hard disk multilayer with a 20m m diamond tip, a loading rate of 10 N/min. and table speed 10 mm/min.

Scanning force microscopy of low load indentations into aluminium thin films

Introduction

Following previous application notes describing the integrated scanning force microscope (SFM) mounted on the Nano Hardness Tester (NHT), this note highlights a typical example of results which can be obtained with such a system. The Winter '96 edition featured the use of the NHT for quality control of IC bonding pads which are typically composed of a thin aluminium film sputtered onto a Si substrate.

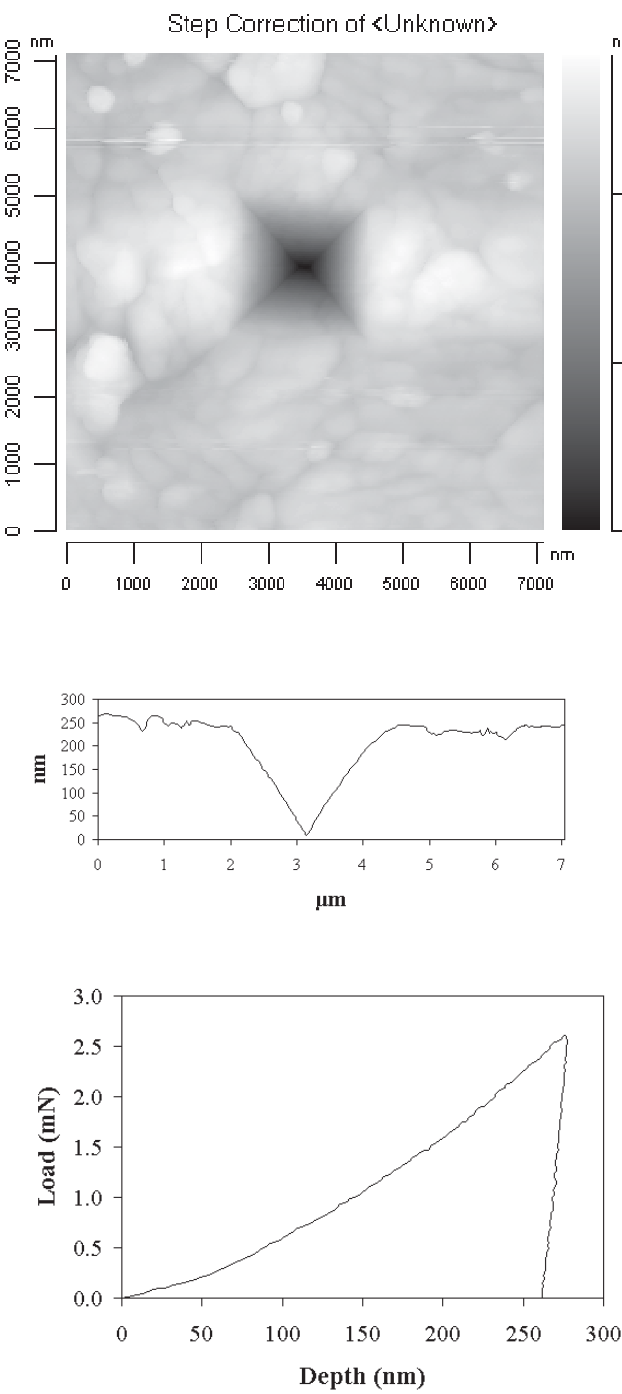


Figure 1: Typical data for a 3 mN indentation into an aluminium thin film (thickness = 2 m m); (a) SFM image of residual imprint; (b) cross-sectional profile through imprint; (c) load-depth curve giving H = 1.157 GPa and E = 112 GPa.

Apart from simply characterising the hardness and modulus of the material, the use of the SFM to measure the surface profile of the residual imprint can provide useful additional information concerning the response of the material to indentation (e.g., pile-up, sink-in effects), surface roughness around the imprint, and an idea of the surface structure. This can all be achieved at the touch of a button with the easy-to-operate SFM acquisition software.

Results

A typical set of results is shown in Fig. 1 for a low load (3 mN) indentation into a bonding pad Al film. The SFM image clearly shows the residual imprint and the extent of piled-up material around the impression. The grain structure of the film can also be seen as well as radial relaxation at the edges of the Vickers imprint. The cross-sectional profile (Fig. 1 (b)) confirms the residual depth as measured with the NHT (258 nm) and allows any pile-up to be precisely quantified. The load-depth curve (Fig. 1 (c)) enabled the Vickers hardness and elastic modulus to be calculated, respectively 1.157 GPa and 112 GPa.

Previous work [1] has already shown the advantages of SFM profiling for the characterisation of bonding pads and future work is envisaged to investigate the effects of pile-up on the true contact area used in the hardness calculation. This is because the projected contact area is calculated from the load-depth curve and thus does not take into account the increased area as a result of pile-up.

N. X. Randall, E. Holländer and C. Julia-Schmutz, Characterisation of IC aluminium bonding pads by nanoindentation and scanning force microscopy (to be published in Surface & Coatings Technology 199

CSM Instruments

This Applications Bulletin is published quarterly and features interesting studies, new developments and other applications for our full range of mechanical surface testing instruments.

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