CSM Instruments
Advanced Mechanical Surface Testing

APPLICATIONS BULLETIN

New LOC module for the SFM provides high resolution frictional contrast

Introduction

This application note describes the recently developed Longitudinal Oscillation Contrast (LOC) module which is available as an optional extra for use with the standard contact mode scanning force microscope (SFM) system.

The SFM head houses a standard piezo tube which is feedback controlled in both horizontal axes by capacitive strain gauges. The deflection of the integrated cantilever beam is transformed into a measurement signal by a glass-fibre interferometer which serves as a position detector. This signal is sent to the control electronics where the tip position is continuously adjusted during scanning to compensate for height variations. This feedback control loop ensures that cantilever deflection remains constant during a measurement and is recorded as a direct representation of the surface topography of the sample.

The LOC module allows high resolution frictional contrast to be recorded simultaneously with the topography signal. The basic principle is shown in Fig. 1. During scanning in the conventional contact mode the scan head is oscillated parallel to the cantilever axis at a typical frequency of approximately 600 Hz. The type of friction encountered between the tip and the sample causes the cantilever to bend in one of two modes.

In the case of static friction (see Fig. 1 (a)), the cantilever bends towards the optical fibre (which is mounted above it) causing an increase in the intensity of the signal. In the case of sliding friction (see Fig. 1 (b)), the cantilever bends away from the fibre which results in a decrease in signal intensity. By mounting the optical fibre along the middle section of the cantilever allows the intensity difference to be optimised for either the ‘stick’ or the ‘slip’ condition.

At each measuring position, i.e., corresponding to each pixel of the image, the cantilever is oscillated in the LOC mode several times. By using a phase sensitive method (lock-in detection) allows the LOC signal to be separated from the topography signal. This means that, in practice, both a topography image and an LOC image can be acquired simultaneously.

By adjusting the amplitude of the LOC oscillation, the transition between static and sliding friction can be investigated in different materials as demonstrated in the results of Fig. 2. In this example, the friction signal has been plotted as a function of oscillation amplitude for chromium, gold and glass.

Starting at an amplitude of zero, the LOC signal is seen to increase linearly up to a certain maximum (depending on the characteristics of the measured sample material) before dropping sharply. The transition between static and sliding friction is characterised by this peak. The higher the peak, the greater the static friction between the tip and the sample. At higher amplitudes, the LOC signal remains alomost constant at a certain level, this value being an indication of the sliding friction encountered between the tip and the sample measured.

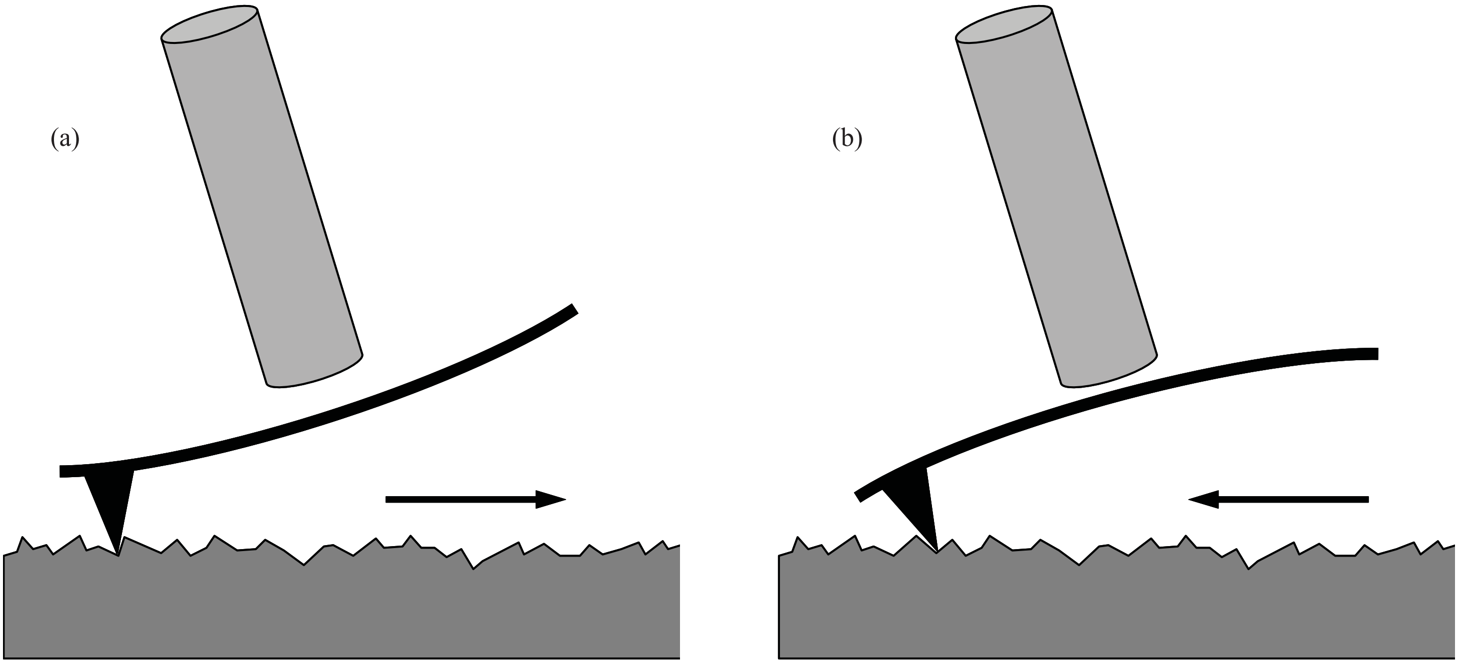

Figure 1 : Schematic representation of the Longitudinal Oscillation Contrast (LOC) principle. The cantilever is oscillated along its axis and bends depending on whether static friction is encountered in the backward (a) or forward (b) direction. In the case of sliding friction, the cantilever bends to a much lesser extent. By positioning the optical fibre interferometer along the middle section of the cantilever, the resultant changes in intensity can be monitored depending on the type of interaction between tip and sample.

Results

Some typical results are presented in Figs 3 and 4 for a magnetic floppy disk surface and a glass slide respectively. In both cases, a large difference between the topography image and the LOC (friction) image is observed, with greatly enhanced contrast in the latter. This improved contrast is due to the variations in frictional characteristics between different phases of the surface structure. It can be particularly useful in cases where the surface topography is very flat and structureless, but where phases are present which have measurable variations in either static or sliding friction. By adjusting the oscillation amplitude, the regime best suited to the measured sample can be used.

The LOC technique is particularly well suited to frictional characterisation. Conventional Frictional Force Microscopy (FFM) relies on torsion of the cantilever beam to measure variations in friction. Such a method is very difficult to calibrate owing to the non-linear torsional deformation mode of the cantilever and so can only be considered as a qualitative technique. On the other hand, the LOC method can be calibrated quite accurately, provided that a sample is used with a known coefficient of friction between its surface and the material of the tip (usually Si).

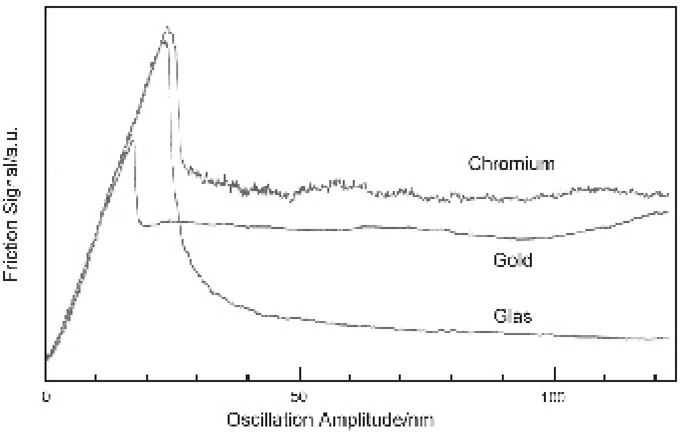

Figure 2 : Friction (LOC) signal plotted as a function of oscillation amplitude for chromium, gold and glass samples scanned with a Si cantilever tip in contact mode. The low amplitude peak corresponds to the transition between static and sliding friction, whereas the constant signal at higher amplitudes gives a measure of the sliding friction encountered between tip and sample.

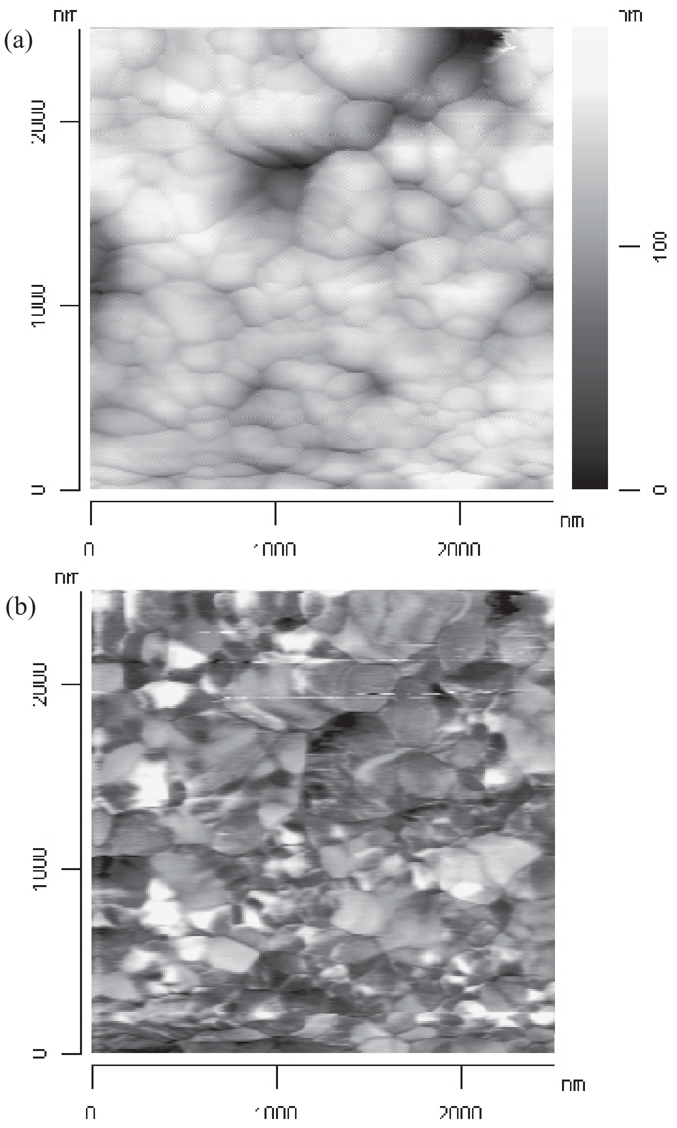

Figure 3 : Topography (a) and LOC/friction (b) data obtained on a magnetic floppy disk surface. The topography image shows the general morphology of the surface, but the LOC image gives a large contrast enhancement between different phases.

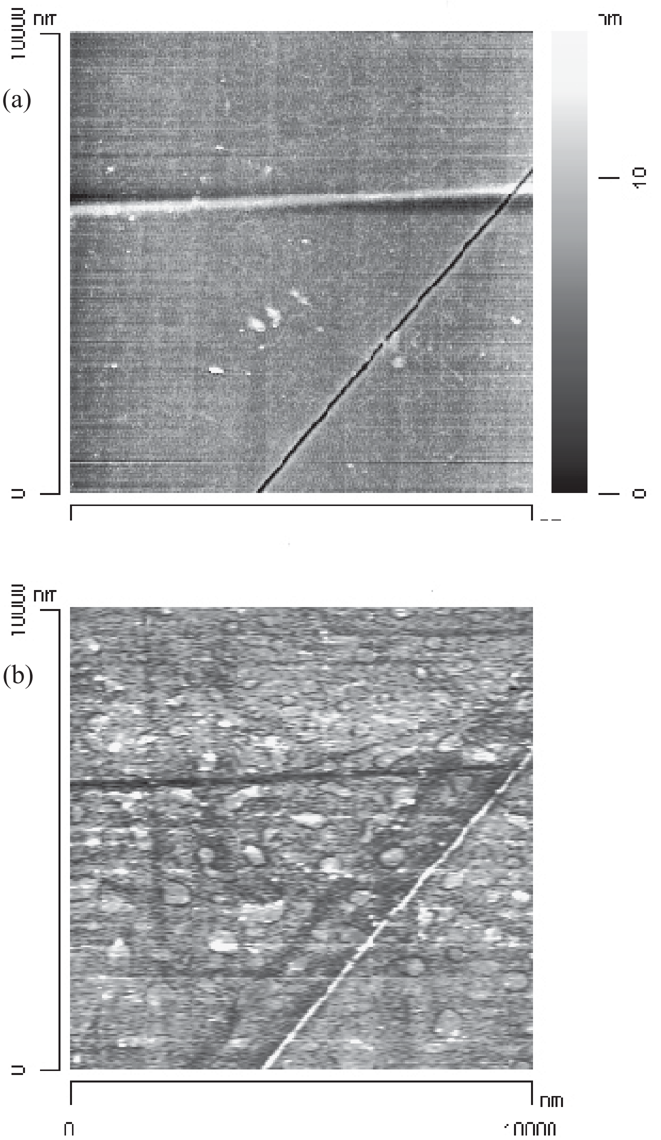

Figure 4 : Topography (a) and LOC/friction (b) data obtained on a standard glass substrate. Apart from a surface scratch, the topography image shows hardly any structure. In contrast, the LOC image shows the grain structure of the material.

Characterisation of tribological transfer films using combined NHT/SFM

Introduction

In conventional pin-on-disk testing of the tribological characteristics of two different materials in sliding contact, the main parameters of interest are notably the friction and wear properties of the material pair. However, when two bodies consisting of hard and soft materials respectively are subjected to such testing, the apparition of a transfer film, or *third body*, which can be a composite mixture of the two, is often observed. This layer is formed through mutual transfer and/or by mechanical mixing of the two materials. Until now the characterisation of transfer films in terms of their mechanical properties has been hampered by their non-homogeneous distribution across a tested surface, their small size, low thickness and the difficulty in accurately positioning a test probe such that the film properties can be measured independently from those of the substrate.

The recent development of nanoindentation techniques, in particular the CSEM Nano Hardness Tester (NHT), has allowed highly localised hardness and modulus measurements to be performed on very small material volumes. This method is ideally suited to tribological transfer films owing to their aforementioned dimensional limitations.

When combined with a surface imaging technique, such as the scanning force microscope (SFM) attachment on the NHT, surface deformation phenomena can also be investigated in and around the indentation area. Some previous work [1] has already shown the net advantages of nanoindentation as a unique tool for the characterisation of tribological transfer films created with a steel/polymer material pair in both ambient air and cryogenic environments. Distinct variations in hardness between the transfer films and their contacting bodies were observed and correlated to the wear behaviour and testing environment.

In this study, the mechanical properties of transfer films created between material pairs of significant industrial importance were investigated, namely hard coatings (TiN, TiCN, TiAlN, CrN) for tool applications and bulk ceramics (Al_2O_3, WC). In the case of high performance cutting tools, the film material must possess high strength at elevated temperatures, good oxidation resistance, a low coefficient of thermal expansion and high thermal conductivity.

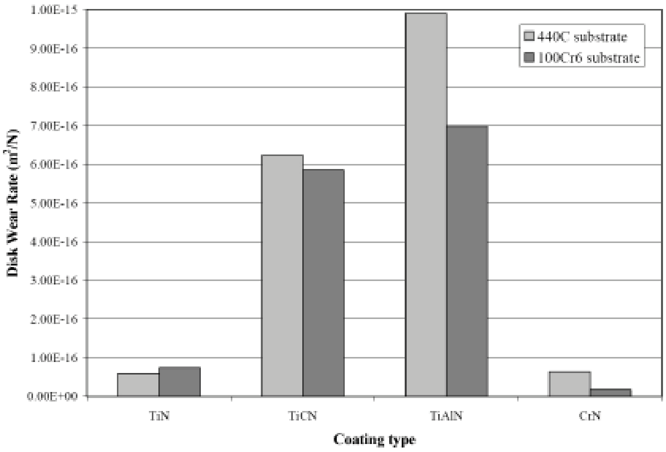

Figure 1 : Disk wear rate plotted as a function of coating type and substrate for TiN, TiCN, TiAlN and CrN combinations, as measured using a pin-on-disk tribometer with a WC ball as static partner, 5 N normal load, 10 cm/s speed and 1.2 µl/4 cm^2 of HBO 2222 lubricant.

Methodology

The tribological apparatus used consisted of a conventional CSEM pin-on-disk tribometer for room temperature experiments and a high temperature tribometer for experiments up to a maximum of 800°C. Separate samples of TiCN, TiAlN and CrN were produced by Physical Vapour Deposition (PVD) onto AISI 440C and 100Cr6 steel substrates. Coating thicknesses were measured using the CSEM Calotest method and were all in the range 2-4 µm. Room temperature sliding wear experiments were then carried out with a tungsten carbide (WC) ball of diameter 6 mm acting as the static partner. The wear rate results are shown in Fig. 1 for these samples (TiN data has been added as a reference for comparison purposes).

High temperature tribometer measurements were performed on TiN, TiAlCrYN and TiAlN/CrN coatings which had been deposited by PVD (coating thicknesses were measured as 1.9-3.4 µm). The substrate was high speed steel (HSS), the static partner was an Al_2O_3 ball of diameter 6 mm and the test conditions were 5 N normal load, 6 cm/s speed and the friction coefficient was averaged over a distance of 2000 m. The average roughness (R_a) was measured for the disk wear surface before testing using a three-dimensional stylus profilometer.

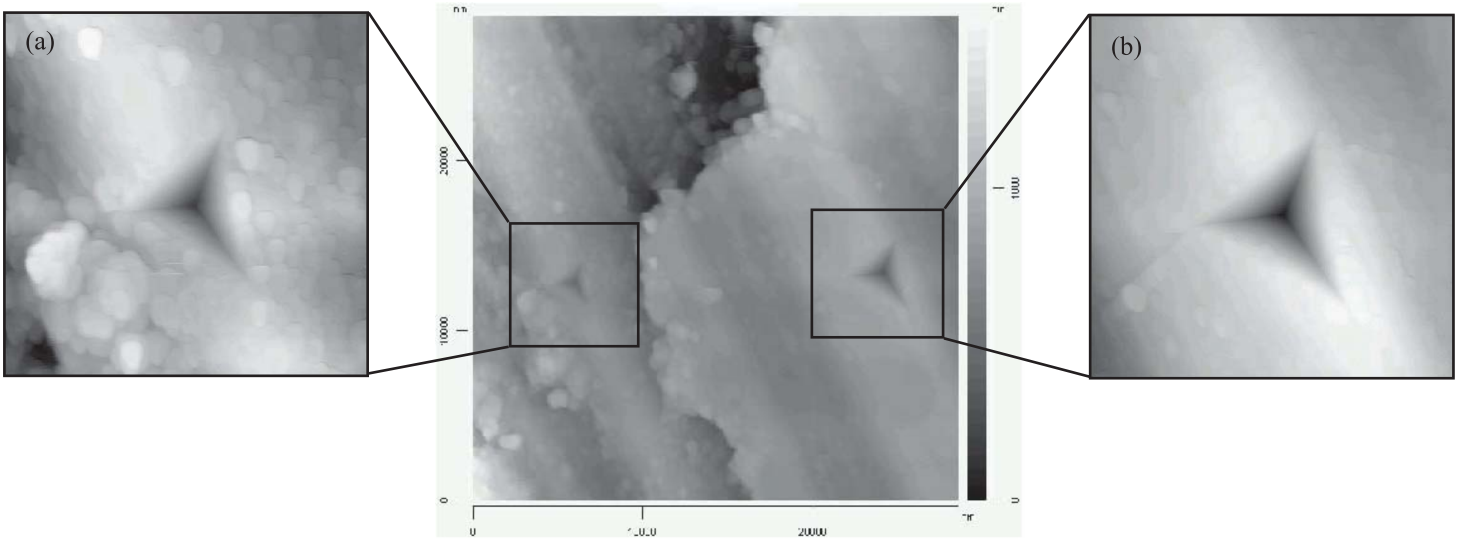

Figure 2 : SFM images of a TiCN wear track showing 20 mN residual Berkovich nanoindentations made on the coating (a) and the transfer film (b). Note the positioning accuracy of the NHT and the differences in surface morphology between the two areas.

The residual transfer films which had accumulated on both the ball and disk were analysed using nanoindentation, and their hardness (*H*) and elastic modulus (*E*) compared to that of the coating, substrate and ball materials. The maximum force and the loading rate were varied 8-50 mN and 16-100 mN/min. respectively. The smallest force value was used when indenting on the transfer films in order to keep substrate effects to a minimum. The optical micrographs of the residual wear tracks (Fig. 3) show the differences in wear behaviour between the various material pairs and test conditions. In the case of the TiCN, small amounts of transfer film are visible along the wear track, these being inhomogeneously distributed and of varying size and thickness. The wear tracks of the TiAlN/CrN and TiAlCrYN were both found to be highly oxidised as a result of high temperature testing.

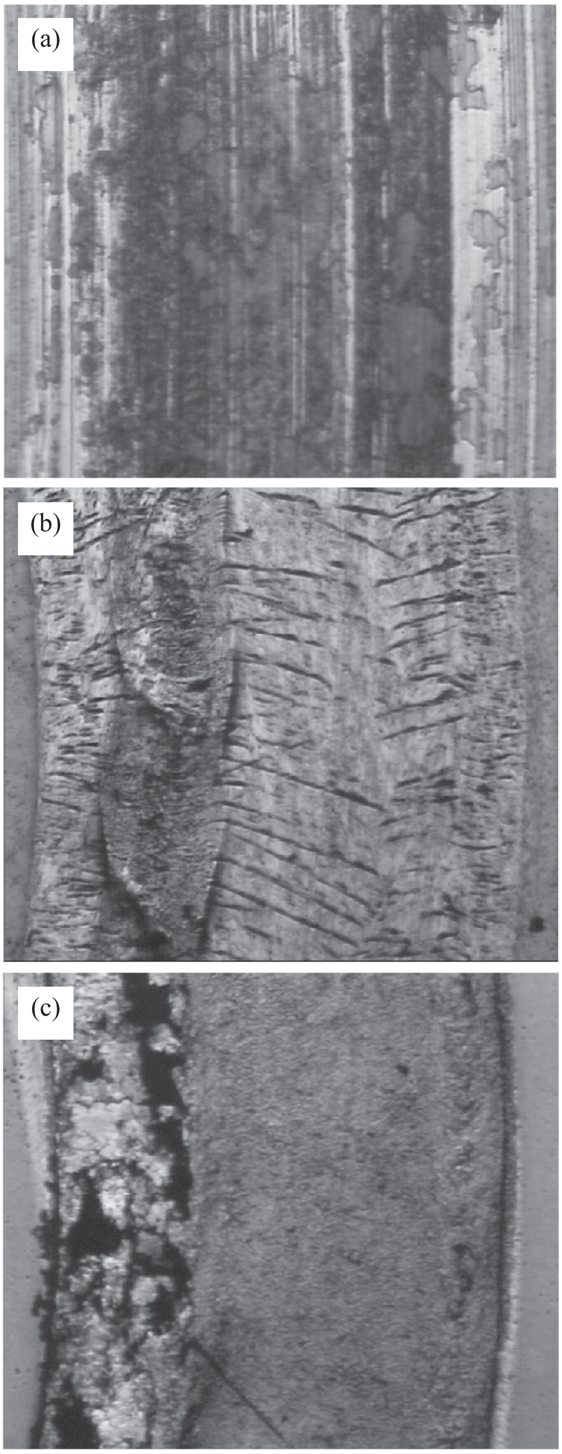

Figure 3 : Optical micrographs of the residual wear tracks on the following measured samples; (a) TiCN (200 x), (b) TiAlN/CrN (50 x) and (c) TiAlCrYN (50 x).

A thick mixture of transfer film and oxide was spread quite homogeneously over the surface of the track. Lateral cracking was visible throughout the transfer layer, probably due to the thermal stresses induced in the sample during the test.

Surface topographical characterisation of the wear track residual imprints by SFM gave significant additional information about the material response to the indentation as well as to surface morphological changes resulting from the sliding wear process. Fig. 2 shows an image of two 20 mN residual Berkovich nanoindentations made on the TiCN coating and transfer film. Both surfaces are quite obviously made up of sheared particles which can clearly be seen in both images, as well as shallow pits onm the surface where fragments have been ripped out during the abrasion process.

Conclusions

It has been shown that, for material pairs consisting of hard coatings in dry sliding with two types of bulk ceramic, an increase in the hardness of the transfer film which is inevitably produced can strongly influence the apparent wear rate. The wear rate itself is dependent on the testing conditions and on the resultant temperature at the ball/coating interface, as well as on the roughness of the mating surfaces. Sliding wear tests at high temperatures have been shown to result in significant amounts of transfer material and the formation of oxides which can strongly influence the measured mechanical properties.

Nanoindentation has been introduced as a valid method for measuring the hardness of ultra-thin transfer films and, by combining it with high resolution SFM, for investigating differences in material response and surface topography, and correlating them to the wear process. Elemental analysis will help to establish whether compositional changes resulting from the wear process have a major contribution to the formation and effect of transfer layers.

[1] N. X. Randall and J. L. Bozet, *Wear*, 212 (1) (1997) 18-24

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

Editor Dr. Nicholas Randall

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