CSM Instruments
Advanced Mechanical Surface Testing

APPLICATIONS BULLETIN

New integrated SFM objective for better characterisation of NHT indentations

Introduction

The Nano Hardness Tester (NHT) has already established itself for the characterisation of thin films and coatings with force and displacement resolutions of 10mN and <1nm respectively, using a differential capacitive measuring technique. Although quantitative hardness and modulus data are of great importance in judging the applicability of a given material to a specific function, the actual response of the material to an indentation at such low loads can give substantial additional information about the indentation process.

This application note describes a new option for the NHT where a scanning force microscope (SFM) can be integrated into the optical microscope of the instrument, in place of a standard objective lens. This efficient and compact solution is shown in Fig. 1, and has a very user-friendly PC interface where all functions can be controlled directly from a powerful software package. The SFM uses an interferometric beam deflection system for detecting cantilever displacement and a piezoelectric scanner which is feedback controlled in the x, y and z directions. This latter feature greatly reduces the effects of piezo distortion and non-linearity and allows a standard lateral scan range of 20 □m, although larger scanners can also be supplied on special request.

Applications

The net advantages of combining surface topographic and indentation data in one instrument, with an accurate electromechanical positioning system, are that a particular sample site can be located and measured before repositioning either under an optical objective lens or the SFM.

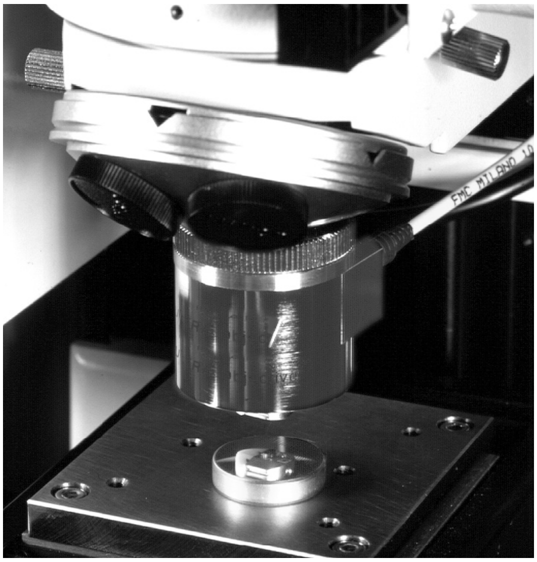


Figure 1 : The new SFM mounted as a standard objective on the optical microscope of th

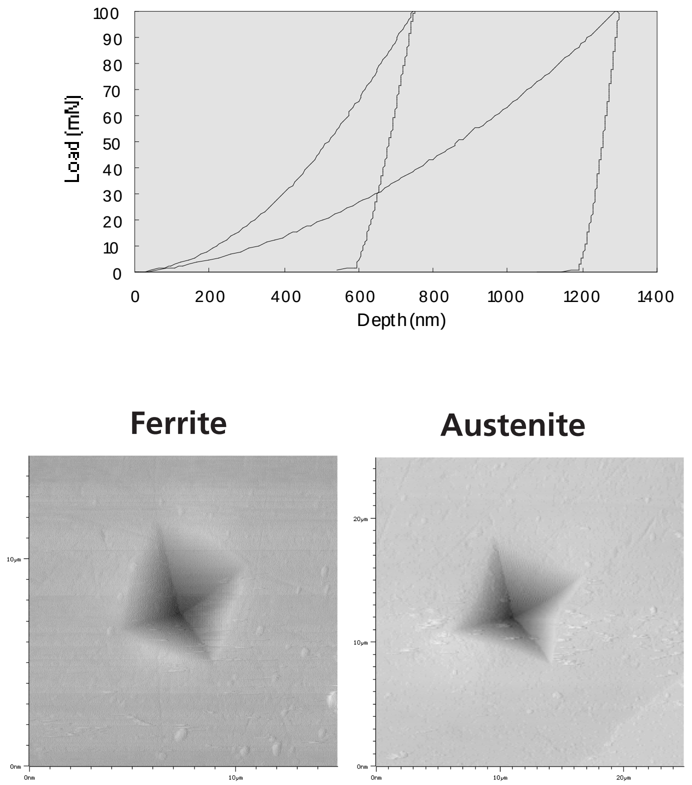


Figure 2 :Indentation curves (max. load = 100mN) for ferrite and austenite phases, together with corresponding SFM images of the residual imprints.

An interesting example of the use of this instrument is the characterisation of multiphase materials where the local mechanical properties of separate phases need to be measured. Duplex stainless steels, having a microstructure consisting of ferrite and austenite, have been investigated [1] in order to establish their susceptibility to long term ageing at intermediate temperatures (300-400°C).

Fig. 2 shows load-displacement curves for each phase with a maximum applied load of 100mN, together with SFM images of the residual imprints. The difference in imprint shape between both phases is particularly evident. For the austenite, the edges are concave which confirms the high work-hardenability and elastoplasticity of this phase, due to radial relaxation of material around the indentation during unloading of the indenter. In contrast, the ferrite imprint exhibits convex edges together with bulging and pile-up of material around it which suggests weak work-hardenability and a rigid plastic response to indentation. The respective Vickers hardness of the austenite and ferrite phases was measured to be 280 and 740 after 8000 hours ageing at 350°C.

[1] N. X. Randall, C. Julia-Schmutz, J. M. Soro, J. Von Stebut and G. Zacharie, Novel nanoindentation method for characterising multiphase materials, Proc. ICMCTF97, San Diego, (to be published in Thin Solid Films 1997)

No. 3 May 1997

Modified Micro Scratch Tester (MST) for improved force resolution at low loads

Introduction

In response to a growing demand amongst manufacturers and researchers of thin films and coatings (thickness < 1µm) for an accurate low loading regime with which to measure interfacial adhesion with the CSM Micro Scratch Tester (MST), certain improvements have been carried out to this end.

The conventional MST, whose load range of 1-30N is provided by a force cell measuring a 5V potential at full scale, has been modified simply by reducing the gain of the FN reference signal. This allows the applied force to be divided by a factor of 10 over the same 8 bit acquisition to give far higher resolution at low loads. In practical terms this means that very thin films that could not previously be measured with good reproducibility can now be tested with a tenfold increase in force resolution.

Obviously, with penetration depths in the nanometer range, the choice of scratching tip becomes of the utmost importance and the standard spherical diamond of radius 200µm is no longer suitable. Other options include hard metallic tips (usually of tungsten carbide) with radii of 10µm and various tip angles, e.g., 90°, 120°, etc.

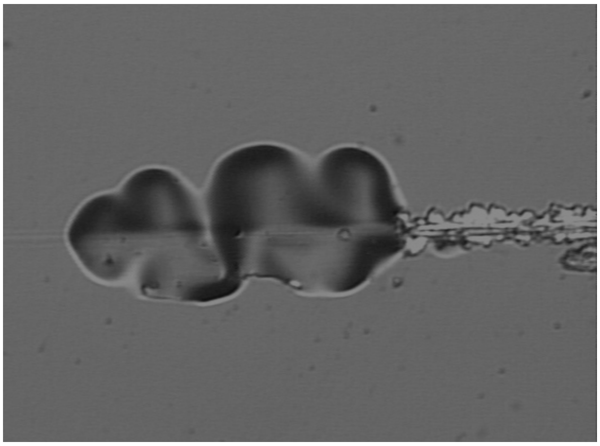


Figure 1 : Progressive load scratch performed on a carbon film deposited on a Si substrate. Total coating failure occurred at a normal force of 448mN (coating thickness = 100nm).

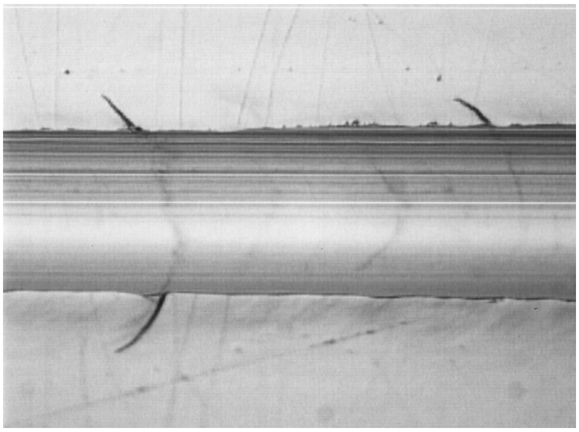
It should be remembered however that this modification is only a prototype system, used to expand the useful range of the instrument in its present state.

Results

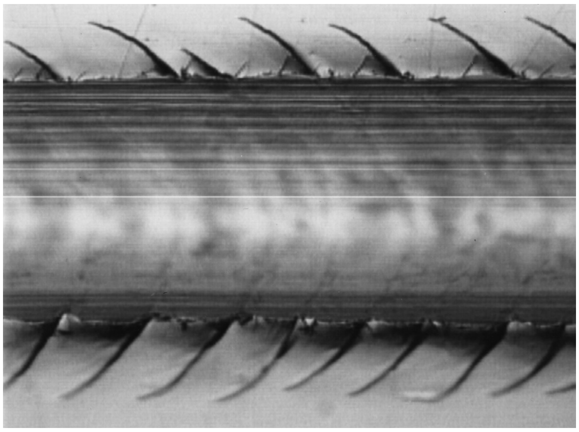
In order to demonstrate the effectiveness of increased force resolution, several thin films of thickness 100-300nm have been measured using a tungsten carbide tip of angle 90°. An example is shown in Fig. 1 where the adhesive strength of a carbon film has been evaluated in terms of two distinct failure modes. The scratch direction is from left to right and failure begins with deformation of the coating from the substrate causing buckling well away from the scratch path. At a certain critical force (in this case 448mN) the applied load can no longer be supported and brittle fracture of the coating occurs.

Owing to the increased force sensitivity of the instrument, the acoustic emission sensor does not provide sufficient signal at such low intensities and so critical failure points must usually be determined by optical inspection of the scratched area. This can sometimes be beyond the limits of the conventional optical microscope so other means are required. One solution is to mount the SFM objective on the microscope as described for the NHT on the previous page. Some such results will be presented in a future bulletin issue. However, this method can prove problematic owing to the contact between scanning cantilever tip and scratched sample. If the failure has been of a brittle nature in which debris has been produced then this may impair the imaging by movement of loose material at the tip-sample interface. Scanning Electron Microscopy (SEM) may prove a better solution in such cases.

Figure 2 shows a pair of optical micrographs of a scratch performed on an experimental hard coating for decorative applications where durability and scratch resistance are of paramount importance. Two distinct critical failure points are visible which are very characteristic of this particular material. The first is characterised by the initiation of small cracks on the sides of the scratch path and radial cracks propogating through the coating (Fig. 2 (a)).



(a)



(b)

Figure 2 :Progressive load scratch on an industrial hard coating. Note the first failure mode (a) which is characterised by small cracks on the sides and radial cracks propogated through the coating. Final failure (b) occurs when enough cracks coalesce to cause delamination and chipping.

The second is the evolution of these cracks until delamination and chipping occurs at a second critical force (Fig. 2 (b)). Note the frequency of the radial cracks along the length of the scratch. The relief of internal stresses can be decerned for each of the larger cracks by a small jump in the friction signal which can be directly correlated to the optical micrograph.

Hard coating characterisation with the CSM Tribometer

Introduction

Due to the increased interest amongst hard coating manufacturers for an adequate method of controlling friction and wear in dynamic machine components, this application note focuses on the use of the CSM Tribometer for routine testing of material pairs in dry and lubricated conditions.

The CSM instrument is a conventional pin-on-disk system although other geometries of the static partner are also in current use, such as the ball or truncated ball. The pin is mounted on a stiff lever and loaded onto the sample (in the form of a disk) with a precisely known weight. As the disk is rotated, the resulting frictional force acting between the pin and disk is measured from the small lateral deflections of the lever. The wear coefficients for the material pair are calculated from the volume of material lost after a specific number of revolutions. The resultant wear track left on the sample (see Fig. 1) can be analysed using a surface profilometer in order to accurately determine its depth.

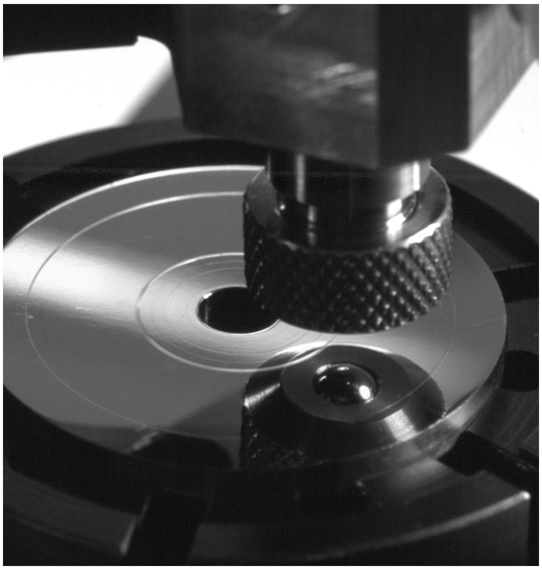


Figure 1 :Close-up view of a TiN sample mounted for a ball-on-disc wear experiment.

Results

A typical friction trace is shown in Fig. 2 for an MoS2 coating deposited onto a 440C steel disk, the static friction partner being a 100Cr6 ball of 6mm diameter. This continuous friction force recording not only provides numerical values for the friction coefficient, μ, but also allows changes in sliding behaviour to be monitored. Such changes can often be linked to variations in surface nature and topography, or in the wear mechanism.

The trace in Fig. 2 reaches stability after approximately 200 revolutions, the value of μ increasing abruptly from 0.02 to 0.15. This is quite characteristic of the wear test and is referred to as the running-in period, during which the instrument must stabilise and any surface contaminant films (e.g. oxide layers) must be broken down. After a finite number of revolutions at a fairly steady friction coefficient, the coating begins to break down after approximately 3500 revolutions, the value of μ rising rapidly up to the cut-off point (0.3). A relationship is often found between the sliding life (expressed in number of revolutions) and the coating thickness. For MoS2 this is usually linear.

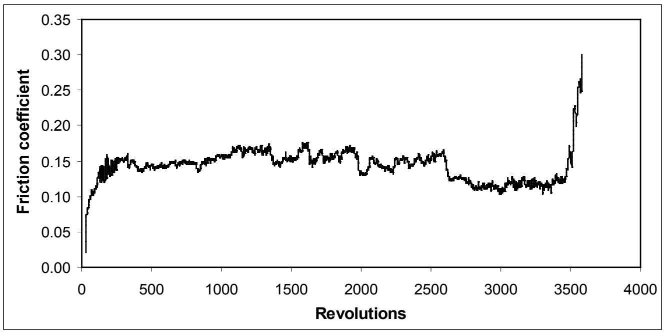


Figure 2 :Typical friction trace obtained with a steel ball on an MoS2-coated sample. Note the significant increase in friction after 3500 revs due to breakdown of the coating.

Apart from the major variables of normal load, contact area, sliding speed and testing time, several other factors must also be considered and monitored during a wear test. The influence of testing temperature on the mechanical properties of the material pair is very important and may induce thermally activated chemical processes, although these may often be dominated by frictionally-generated temperature rises. In lubricated systems it will also be important through its effect on lubricant viscosity. Atmospheric composition has a strong influence: reactive components such as water vapour and oxygen will seriously affect wear rates and mechanisms in all classes of material

Lubrication provides a powerful means of reducing wear in most sliding systems but the influence of different regimes should not be neglected. Full-film hydrodynamic lubrication leads to the lowest rates of sliding wear with a value of wear coefficient, K, typically less than 10-13. However, such wear rates are hardly significant because hydrodynamic conditions cannot be maintained eternally, and under the conditions of boundary lubrication the value of K may rise as high as 10-6, depending on the properties of the lubricant used. When the boundary film is penetrated and sliding occurs between essentially unlubricated surfaces, the wear coefficient may rise to 10-3, a value unacceptable in most practical applications.

Fig. 3 represents the measured friction coefficients for a selection of industrial hard coatings, for which the static partner was a truncated 100Cr6 ball loaded to a projected contact pressure of 10 MPa. Results are shown for tests performed with and without the presence of Motorex lubricant and a drastic reduction in friction coefficient is evident with all coating types. The CrN seemed to display the greatest reduction in friction as a result of lubrication, probably due to its plasma arc deposition process which caused a certain porosity and can be beneficial in retaining lubricant. In addition, the ability of any high-pressure additives in the lubricant to bond to the sample surface and thus prevent penetration of the boundary film could have contributed to the observed decrease.

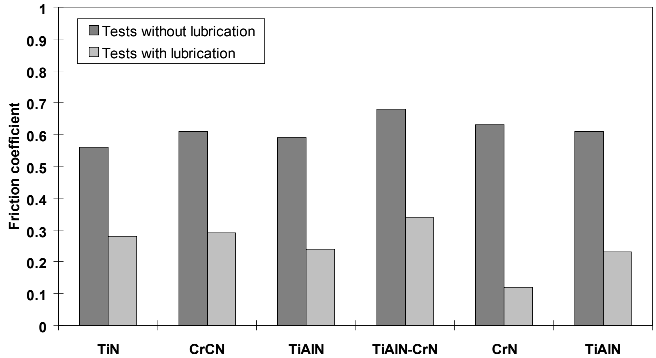


Figure 3 Friction coefficients for a selection of hard coatings tested with and without Motorex lubricant at 50%RH.

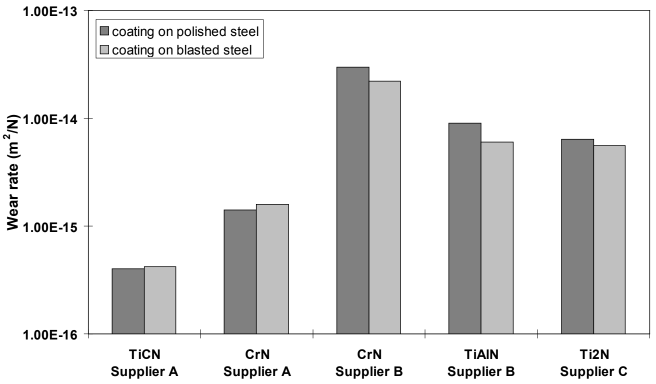


Figure 4 :Wear rates of typical hard coatings from three different manufacturers.

The wear rates for a selection of coatings from different manufacturers are displayed in Fig. 4, two different substrate pretreatments being used, namely polishing and sandblasting. In general it was found that the sandblasted samples gave better adhesion between the substrate and coating and a lower wear rate which could be attributed to a different contact area distribution between ball and disc. The wear rate in each case was determined from the residual wear track by measuring the surface profile with a stylus profilometer and calculating the worn area. An example is shown in Fig. 5. The wear rate is thus calculated by dividing the worn volume by the applied normal force. The wear rate can also be calculated by independantly weighing the pin and disc before and after the test and deducing the amount of material lost as a result of the wear process. Analysis of wear debris in and around the wear track can also yield important information about the wear process.

It has been verified experimentally that for many systems the loss of material by wear is proportional to the sliding distance. However, a strict relationship between wear rate and normal load is not often found in practice, an abrupt transition from a low to high wear rate being observed with increasing load.

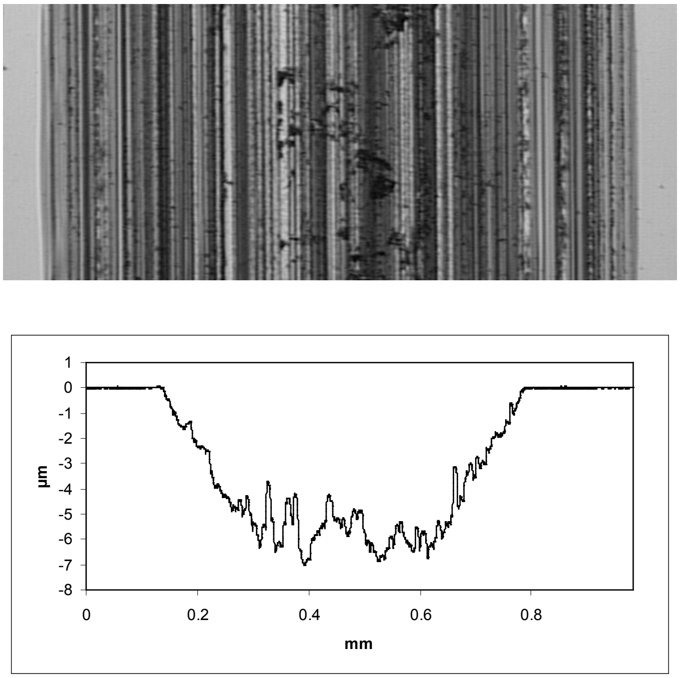


Figure 5 :Optical micrograph of a typical wear track together with the corresponding surface profile measured with a stylus profilometer after a conventional ball-on-disk test (area of profile = 2.82 x 10-3 mm2).

Conclusions

The CSM Tribometer provides a major contribution to better laboratory wear testing where controlled experiments can be used to evaluate new processes and establish the practical limitations of a material pair in near-service conditions. Wear testing becomes fully relevant when used for investigating the active wear mechanisms, for understanding the wear process dependence on testing parameters, and for developing relationships between the production of materials, their structural characteristics and their functional behaviour.

The CSM Tribometer has already established itself as a reliable and versatile tool and is used worldwide in over 100 industrial and research institutes. By constantly upgrading the basic design with better software control and more options, this instrument will remain at the forefront of wear research. Future application notes will focus on specific areas such as the study of lubricants, the high-temperature option and the measurement of electric current across the pin/disc interface.

Relevant Publications

(1) I. M. Hutchings, Tribology – Friction and wear of engineering materials, Edward Arnold, London, 1992

(2) C. Müller, C. Menoud, M. Maillat and H. Hintermann, Surf. Coat. Tech., 36 (1988) 351-359

(3) J. P. Celis, Surf. Coat. Tech., 74-75 (1995) 15-22

(4) H. Czichos, Tribology, a Systems Approach to the Science and Technology of Friction, Lubrication and Wear, Tribology Series 1, Elsevier, Amsterdam, 1978

(5) S. Lim and M. Ashby, Acta Metall., 35 (1987) 1-24

(6) F. F. Ling and C. H. T. Pan, Approaches to Modelling of Friction and Wear, Springer-Verlag, 1988

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.

Editor Dr. Nick Randall

Should you require further information, then please contact:

CSM Instruments
Rue de la Gare 4
CH-2034 Peseux
Switzerland

Tel: + 41 32 557 5600
Fax: +41 32 557 5610
info@csm-instruments.com
www.csm-instruments.com

DISTRIBUTOR:

6210.05.97 CSM Instruments / Printed in Switzerland