CSM Instruments
Advanced Mechanical Surface Testing

APPLICATIONS BULLETIN

MST Swinging Module for variable frequency scratch testing

Introduction

This application note describes the CSEM Swinging Module which is available as an optional extra with the standard Micro Scratch Tester (MST). In conventional scratch testing, the sample is displaced in one direction (x) under the constant or progressively-loaded indenter tip. In the *swinging* mode the sample can be additionally modulated in a perpendicular direction (y) as a function of time.

Fig. 2 (b) shows the basic principle of the technique. The sample is mounted on a specially adapted table which has an additional motor in order to operate transversely to the conventional scratch direction. Although this motor rotates in one direction only, an oscillating motion is achieved by a cam-drive, where the size of the cam dictates the amplitude of the motion. Currently, cams are available for amplitudes in the range 50 - 200 µm.

The frequency of oscillation is simply controlled by varying the current in the motor, this being adjusted by turning a potentiometer on the front panel of the swinging module control unit. The full frequency range available is 0 - 30 Hz. By controlling both the frequency (y direction) and the scratch speed (x direction), a wide range of sinusoidal scratch paths is possible. However, such a system can also be used for investigating the wear properties of surface coatings. This is achieved by maintaining the sample stationary in the x direction and oscillating only in the y direction.

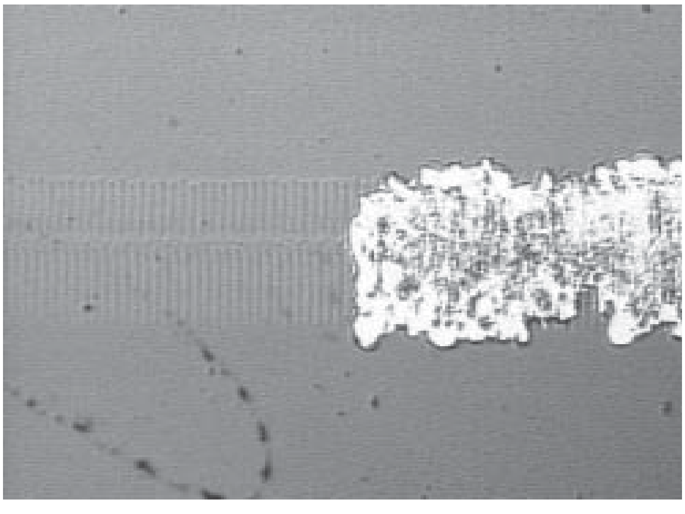

Figure 1 : Optical micrograph of a progressive load scratch using a swinging frequency of 15 Hz together with a loading rate of 1.6 N/min. over the normal load range 0 - 3 N. The sample is a DLC thin film of thickness 1 µm deposited by CVD onto a Si substrate. Coating failure occurs abruptly at a normal load of 1.88 N.

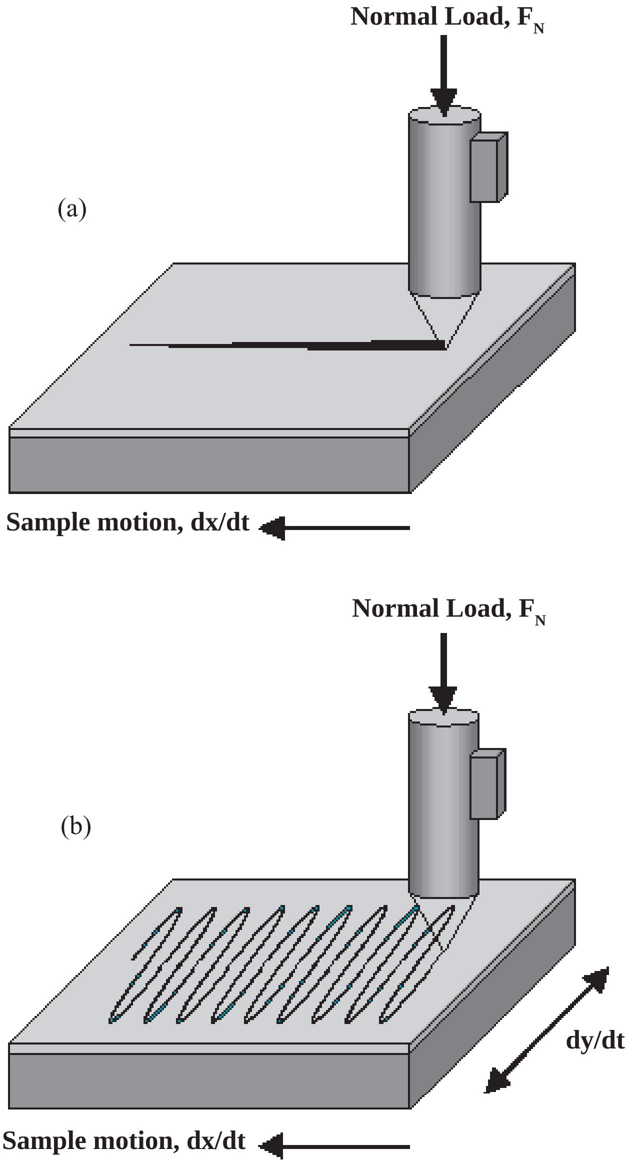

Figure 2 : The basic principle of the conventional scratch testing technique (a) and the use of the swinging module adaptation (b) where the sample motion is additionally modulated in the y direction (i.e., transversely to the scratch direction).

Wear properties, as well as coating integrity, are measured by varying the number of cycles of each stationary oscillation. For example, a run of 10 cycles is performed after which the sample is displaced in the x direction by a small distance. Another run of 20 cycles is then performed and compared to the first by optical microscope observation. This process can be continued until, at a certain number of cycles, the coating loses its structural integrity and the substrate is reached. Alternatively, the number of cycles remains constant but the applied load is varied.

Results

The advantages of the *swinging* mode can be demonstrated by looking at various examples where coating failure can be accurately determined along the length of a progressive load scratch.

Fig. 1 shows an optical micrograph of a scratch made on a DLC thin film of thickness 1 µm. The swinging oscillations are clearly visible on the left side of the image together with the point at which abrupt failure of the coating occurs (scratch direction is from left to right). One advantage of horizontally oscillating the sample is that the effective scratch length is much longer than if a conventional scratch had been made. In the case of progressive loading, this translates to a much more precise load gradient between the minimum and maximum preselected load values.

Some swinging mode data for a TiN film is presented in Fig. 3. The critical point at which failure occurs is very clearly defined, both on the Acoustic Emission (AE) and Frictional Force (Ft) signals. Sudden failure of thin films and coatings is a characteristic of swinging data and can provide more reproducibility in results for certain materials, in particular coatings of a hard nature which have been deposited on a relatively hard substrate. Additionally, it can be argued that for larger amplitudes of oscillation the scratch path has a far greater width than if a conventional scratch had been performed: this means that small imperfections in the coating (e.g., porosity, foreign particles, cracks, etc.) will have less influence on the final result.

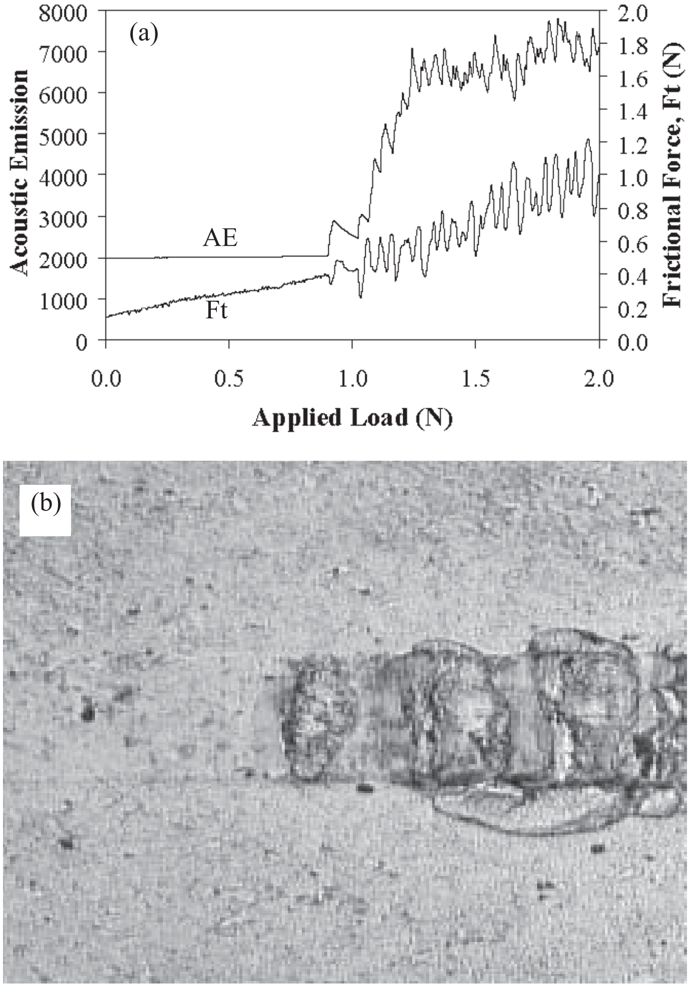

Figure 3 : Swinging module data for a TiN coating (thickness = 1 µm) on a 440C steel substrate. The sudden jump in both the AE and Ft signals in the MST data (a) can be directly correlated to the coating failure shown in the optical micrograph (b).

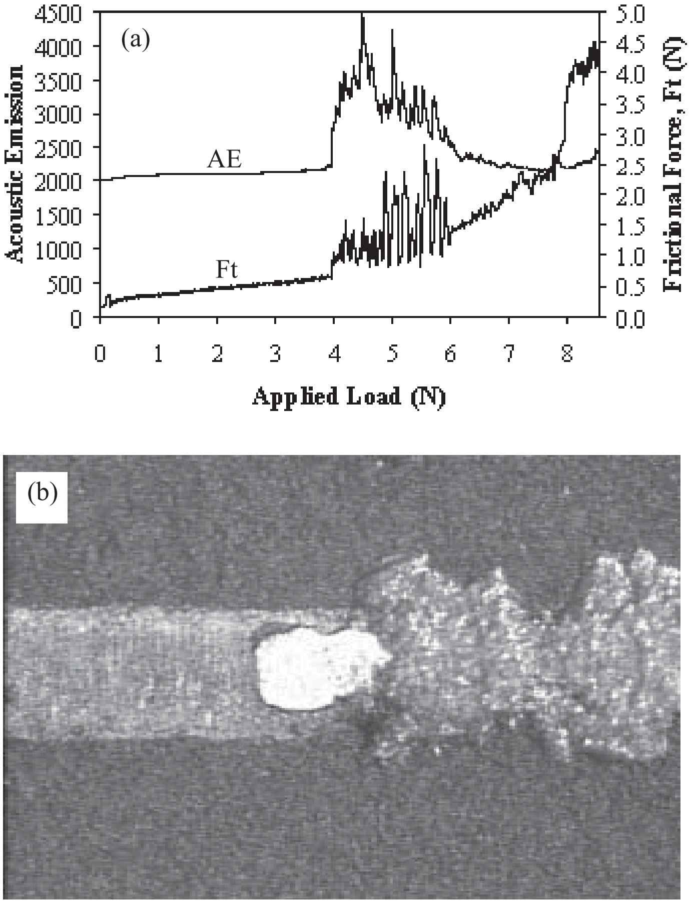

Figure 4 : Swinging module data for a TiN/TiC multilayer coating (each layer has a thickness of 1 µm) on a hard metal (WC) substrate. The MST data (a) clearly shows the two regions where each coating has successively failed and this is also visible in the optical micrograph (b).

For the case of multilayered coatings, the swinging mode can also prove advantageous for defining separate critical points. Fig. 4 shows some swinging mode data for a TiN/TiC multilayered coating deposited on a hard metal (WC) substrate (scratch direction is from left to right). Distinct differences are visible between the surface scratched TiN layer (left side of image), the delaminated TiC coating beneath (centre of image) and the subsequent total delamination of the multilayer (right of image). Both the AE and Ft signals give distinct increases at the points of failure and can be directly correlated with the optical micrograph. A conventional scratch, using the same 0 - 10 N load range (not shown), showed only the failure point at which total delamination of the multilayer occurred and not the point at which each component layer failed.

Conclusions

The CSEM Swinging Module has been demonstrated as a very useful addition to a standard Micro Scratch Tester (MST). It allows a variety of additional scratching modes to be incorporated into a measuring sequence, namely dynamic as well as stationary oscillation of the sample. This permits wear properties to be evaluated in addition to coating integrity and interfacial debonding. By using different spherical tip radii and different oscillation amplitudes and frequencies, the swinging mode can provide significant additional information concerning coating mechanical properties.

An additional application of this method is to use a material of interest in place of the standard diamond tip. By making stationary passes at different loads, the wear properties of the particular material pair can be accurately evaluated.

Combined NHT/SFM for investigating the mechanical properties of Cr_2O_3 thin films

Introduction

It is now well established that Cr_2O_3 is one of the hardest oxides both on the mineralogical scale (8.5 Mohs) and on the microhardness scale (up to 29.5 GPa). Much of the recent research work has focussed on Al_2O_3 due to its chemical and thermodynamic stability which makes it a viable option as a barrier layer in tribological and microelectronic applications. Comparatively little work has been dedicated to chromium oxide and its mechanical properties. The major drawback of oxide films compared to the generally softer transition metal nitrides, which are now standard in the wear protective coating industry, is their lower toughness. However, Cr_2O_3 has now found application as a protective coating on read-write heads in digital magnetic recording units.

This application note summarises some results obtained in a recent study of various Cr_2O_3 thin films prepared by reactive RF sputtering. The target material was metallic chromium (99.5 % purity) and all depositions were performed at a total pressure of 10^{-1} Pa in mixed Ar and O_2. The ratio between the inert gas (Ar) and reactive gas (O_2) was varied in the range 5 - 30 % in order to obtain different oxygen concentrations in the films produced. Nanoindentation studies combined with scanning force microscopy (SFM) were thus used to investigate mechanical properties.

Application

The results presented in Fig. 1 are for a Cr_2O_3 thin film deposited with a substrate temperature of 90°C and a deposition rate of 0.73 Å/s. The film in this case has an amorphous structure, in contrast to films produced at higher substrate temperatures which contain small crystallites (5 - 15 nm diameter).

At low loads (< 20 mN) the *coating only* properties are measured giving values of H = 21 GPa and E = 205 GPa. At higher applied loads, for example 50 mN (see Fig. 1 (a)), the values are already lower as the Si substrate begins to influence the measurement (Si has a hardness of ~ 9 GPa). The corresponding SFM image shows pile-up around the indentation site which is no doubt due to delamination of the coating during unloading of the indenter.

An indentation at a relatively high applied load of 200 mN (Fig. 1 (b)) displays a *flat* in the loading curve in the depth range 550 - 650 nm. Only subsequent SFM imaging allows this phenomenon to be fully investigated, confirming that subsurface median and lateral cracking has taken place around the indentation site. The measured mechanical properties are almost entirely those of the Si substrate. The considerable extra information provided by the SFM is again demonstrated for the case of nanoindentation on thin films and coatings.

Peter Hones from the Ecole Polytechnique Fédérale de Lausanne (EPFL) is acknowledged for providing the Cr_2O_3 samples.

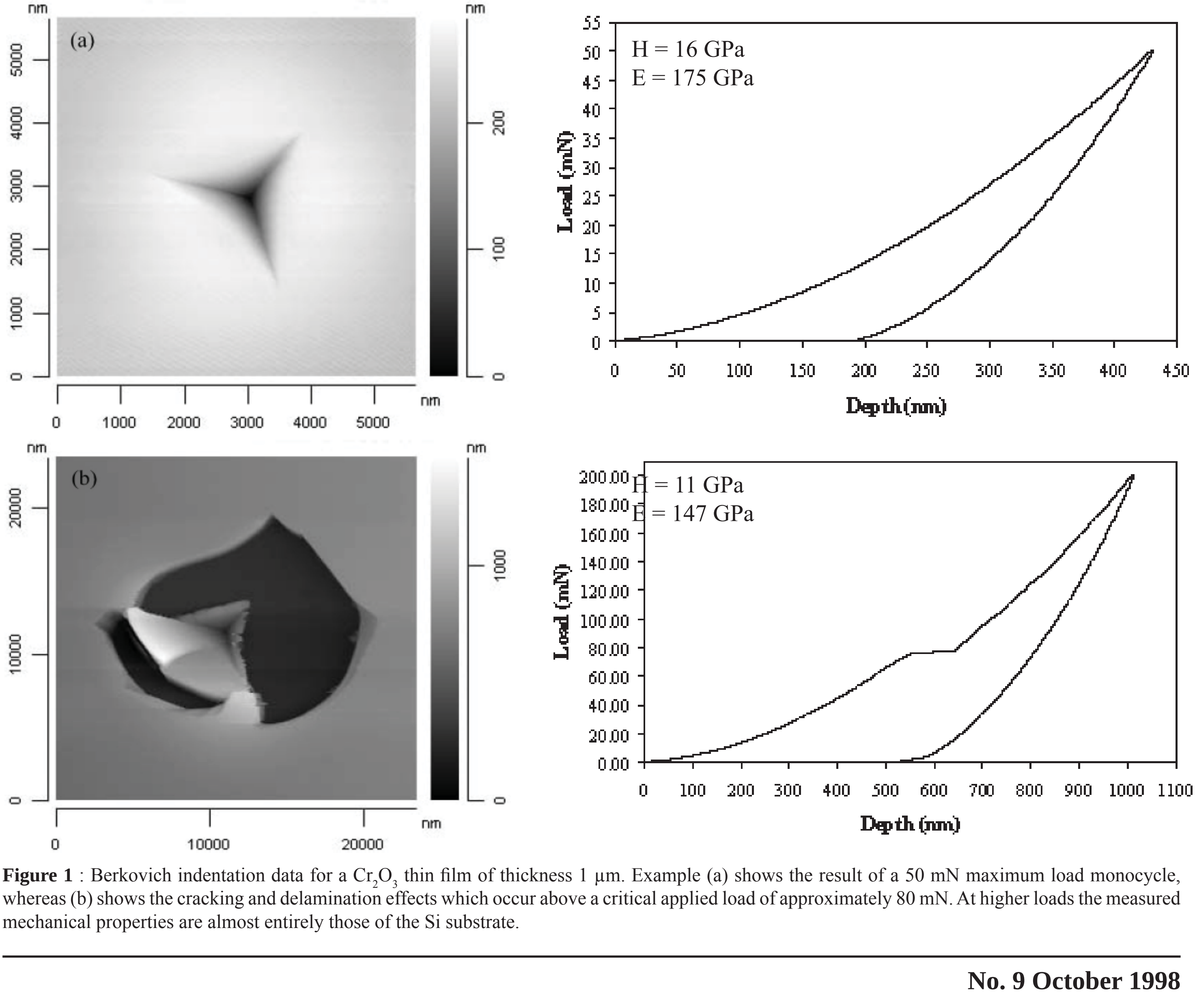

Figure 1 : Berkovich indentation data for a Cr_2O_3 thin film of thickness 1 µm. Example (a) shows the result of a 50 mN maximum load monocycle, whereas (b) shows the cracking and delamination effects which occur above a critical applied load of approximately 80 mN. At higher loads the measured mechanical properties are almost entirely those of the Si substrate.

New Industrial Calotest allows greater flexibility for measuring over/undersized samples

Introduction

Although the basic principle and advantages of the new CSEM Industrial Calotest were presented in a previous issue (No. 8, July 1998), there have been several requests concerning the use of such apparatus for the measurement of non-standard sample sizes and geometries.

The motorised shaft on which the ball rotates is held by an adjustable arm which allows great flexibility in how the ball is positioned on the sample. For particularly bulky samples which are too big to be mounted in the sample holder provided, the complete arm and motor assembly can be removed and directly clamped to the side of the sample using the magnetic foot provided. The range of rotation speeds of the shaft has been increased to 10 - 3000 rpm for ball diameters between 10 and 30 mm. By keeping the motor assembly away from the electronics has the added advantage of making cleaning much easier than when integrated with the electronics.

Application

The example in Fig. 1 shows the Calotest being used to measure the coating thickness of a large TiN coated component. The ball can be precisely positioned on a specific area of the sample by simply moving the adjustable arm and correctly seating the ball.

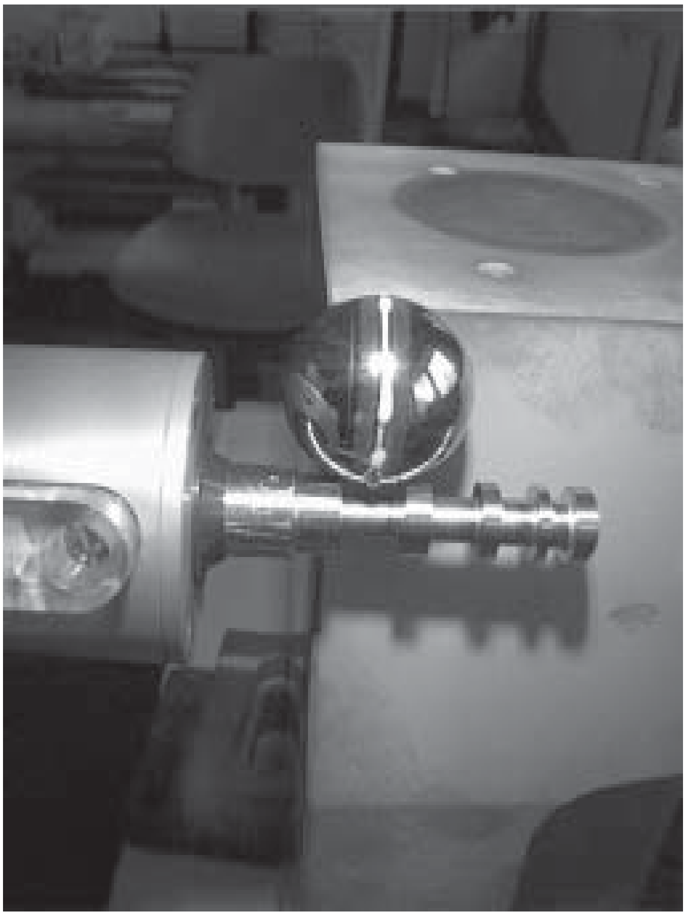

Figure 1 : Detail of the Calotest being used to measure a coated object of large dimensions. The adjustable arm allows great flexibility in positioning of the motorised shaft.

Figure 2 : Detail of the Calotest being used to measure an end milling bit of small diameter. The dimensions of the ball, as well as the angle of the motorised shaft are important considerations.

In the case of particularly small sample areas, Fig. 2 shows the Calotest being used to measure the coating thickness at the edge of a milling bit thread. After firmly clamping the tool between the sample jaws, the motorised shaft must be adequately positioned such that the ball is in contact only with the required area of the sample. The angle of the shaft can also be optimised to aid the measurement. For very small sample areas, reduced ball diameters are recommended (i.e., 10 mm).

Martin Hess from Eifeler Werkzeuge GmbH, Düsseldorf is acknowledged for providing the optical micrographs in Figs 1 and 2.

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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