CSM Instruments
Advanced Mechanical Surface Testing

APPLICATIONS BULLETIN

Nanoindentation with spherical indenters for characterisation of stress-strain properties

Introduction

This application note describes the additional material properties which can be obtained from nanoindentation measurements performed with a spherical indenter tip. Although most low load nanoindentation measurements are made using a Berkovich indenter geometry (to minimise indenter tip chisel effects), such a geometry still suffers from a finite tip bluntness which is very difficult to define accurately.

Spherical indentation overcomes many of the limitations associated with pyramidal indenters and allows hardness to be evaluated by following the transition from elastic to plastic behaviour. This also enables the yield stress to be calculated [1]. Indentation behaviour depends on the ratio between the actual strain and the yield strain of the material; low ratios produce elastic behaviour whereas high ratios produce plastic behaviour. The actual strain can be represented by tanb where b is the angle between the indenter and sample surfaces. Clearly, a spherical indenter will behave in a fundamentally different way from one with a pyramidal geometry: the strain will increase as the indentation depth increases. Therefore, a series of spherical indentations with progressively increasing load will produce stress-strain curves which follow the transition from purely elastic to plastic behaviour.

The example in Fig. 1 shows a plot of stress versus strain for five different material types, produced from multicycle indentations over the range 10 - 260 mN. The stress (y-axis) corresponds to the measured hardness of each cycle in GPa, whereas the strain is calculated by dividing the radius of the residual impression by the indenter radius.

[1] T. J. Bell, J. S. Field and M. V. Swain, *Mat. Res. Soc. Symp. Proc.*, Vol. 239 (1992) 331 - 336

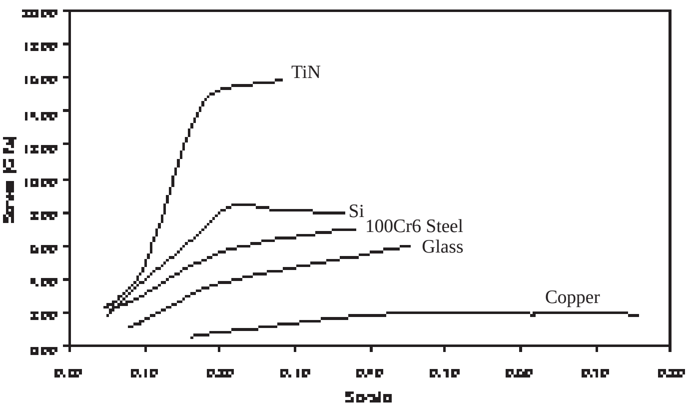

Figure 1 : Stress-Strain data for TiN (coating thickness = 3 µm), pure Si, 100Cr6 steel, glass and pure copper calculated from multicycle indentations performed over the range 10 - 260 mN with a spherical indenter of radius 1 µm. Each data set corresponds to the average of five sets of measurements.

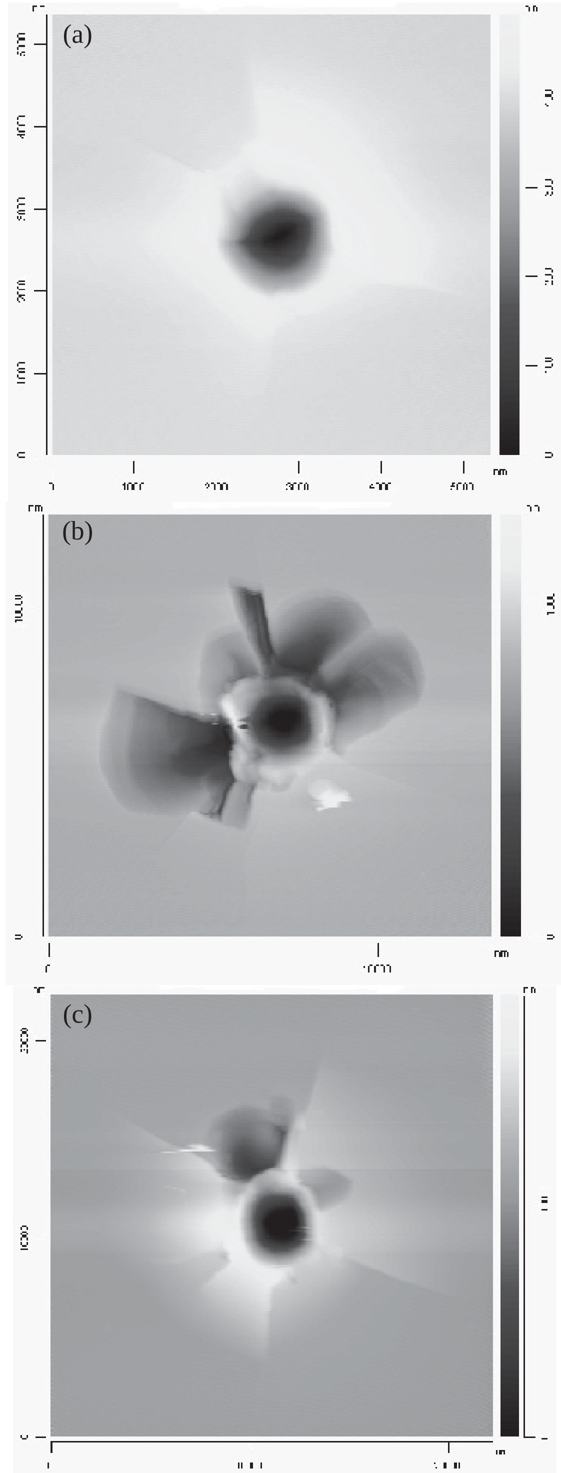

Figure 2 : SFM images of residual monocycle indentations performed in Si with maximum applied loads of (a) 30 mN, (b) 100 mN and (c) 200 mN. The indenter was a spherical diamond of radius 1 µm. Cracking becomes apparent even at low loads with severe chipping occurring at higher loads.

Results

The results presented in Fig. 1 clearly show the type of elastic-plastic transition which is observed in hard materials (TiN, Si) and softer materials (Cu). Harder materials tend to exhibit a more pronounced gradient change as the strain is increased.

Although a spherical indenter theoretically produces a uniform stress field around its circumference, severe cracking is nevertheless observed in materials such as silicon, especially when several load-unload cycles are performed on the same area. Fig. 2 shows the cracking and chipping which results after indentation at loads from 30 up to 200 mN using an indenter of radius 1 µm. Scanning Force Microscopy (SFM) is particularly useful for imaging such nanoindentation effects.

The SFM images shown in Fig. 4 represent residual indents in pure copper and the corresponding cross-sectional profiles are plotted together in Fig. 3. Previous measurements have shown that multicycle indentations produce less pile-up in soft metals such as copper than a monocycle indentation to the same maximum load. This phenomenon can be attributed to the effects of gradual work hardening under the indenter as the load is increased on every subsequent cycle. A careful choice of indenter radius, coupled with a suitable load range, allows a very wide range of materials to be investigated with this technique.

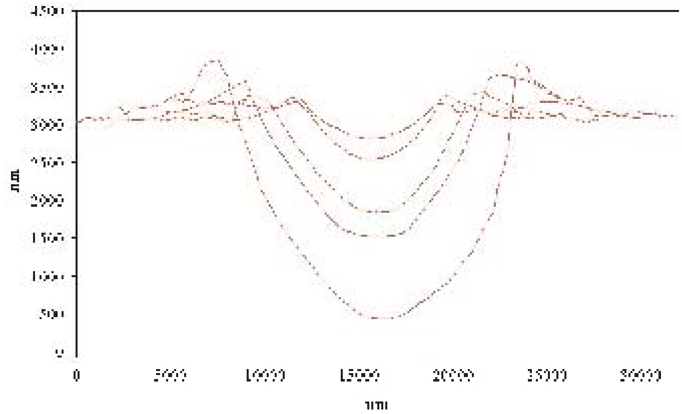

Figure 3 : Cross-sectional profiles through the residual imprints shown in Fig. 4. Note the evolution of pile-up as a function of maximum depth and the variations of the profiles due to indenter non-symmetry.

Future work will involve indentation of ceramics, for which Hertzian contact of a spherical indenter can initiate cone fracture (*brittle* mode) or sub-surface deformation (*quasi-plastic* mode) if the elastic limit is exceeded.

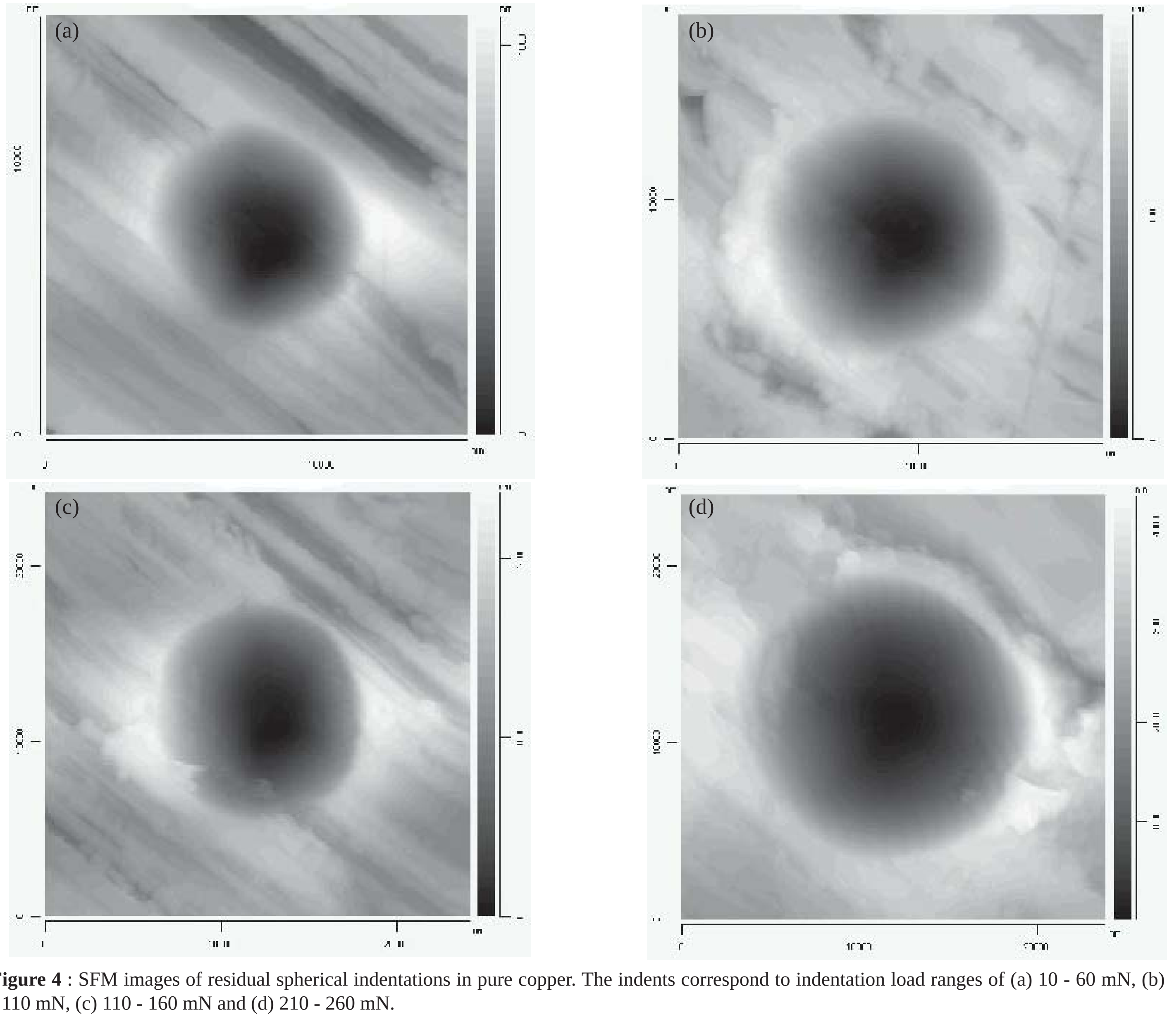

Figure 4 : SFM images of residual spherical indentations in pure copper. The indents correspond to indentation load ranges of (a) 10 - 60 mN, (b) 60 - 110 mN, (c) 110 - 160 mN and (d) 210 - 260 mN.

Characterisation of IC bonded gold wires with the Nano Hardness Tester (NHT)

Introduction

Following on from the previous application note concerning the quality control of Integrated Circuit (IC) bonding pads (featured in the No. 2, Winter 1996 issue), this example shows the Nano Hardness Tester (NHT) being used for characterisation of gold bonding wires *in-situ* on the pad surface.

For this type of nanoindentation measurement the bonded circuit is mounted in an epoxy resin and mechanically polished so as to reveal a section through the centre of a gold bonding wire. A series of equally spaced nanoindentations are then made in a line across the interface between the gold ball, pad coating and pad substrate. This allows the hardness and elastic modulus to be evaluated along the line, thus providing important information about certain parameters such as hardenability resulting from the bonding process.

Application

The optical micrographs shown in Fig. 1 show a typical sectioned sample on which nanoindentations have been performed from well into the substrate to the top of the gold wire ball. Note that the positioning accuracy of the NHT allows an indent to be made precisely on the plated silver coating (thickness = 1 µm) which acts as an interface between the gold wire ball and the copper substrate.

The values of hardness and modulus are plotted for each measured section in Fig. 2. These results clearly show the variation in mechanical properties as a function of distance from the Ag interface. The basic trend is that both the hardness and modulus decrease quite abruptly through the ball and then tail off to values approaching that of bulk gold in the neck and wire sections. However, the values are still slightly higher than the bulk value owing to work-hardening induced during the bonding process. The purpose of measuring the properties of the gold at different sections is to try and understand how they vary along the ball, heat affected zone (neck) above the ball and the drawn section further down the wire. This powerful technique has been shown to provide significant information which can help to establish better quality throughout the gold wire bonding process.

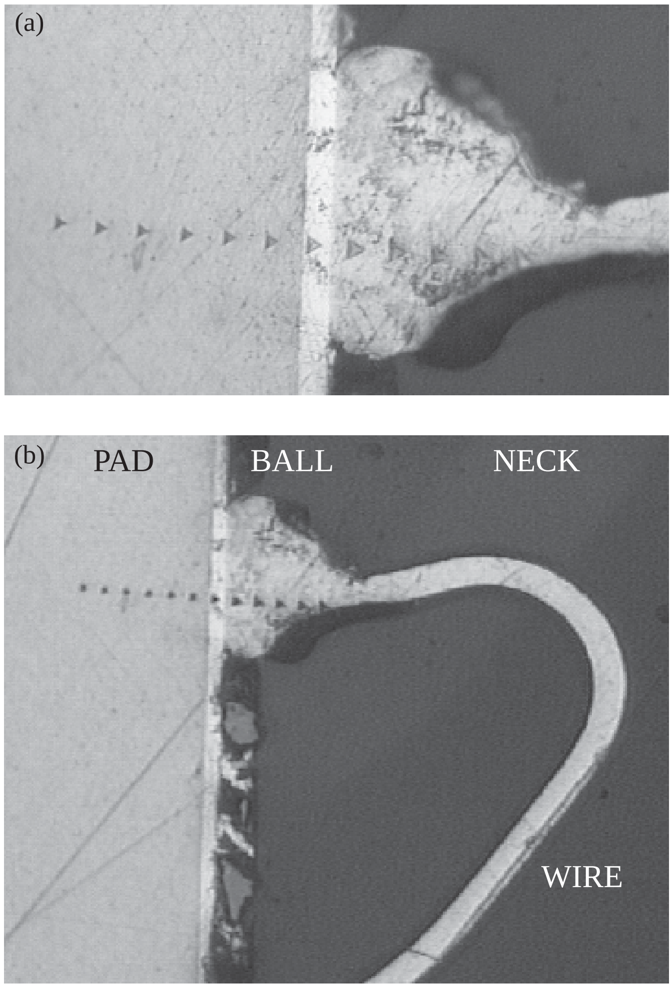

Figure 1 : Optical micrographs of a typical sectioned sample on which a line of 12 nanoindentations have been made. The overview (b) shows the different areas of the sample which can be compared, whereas the close-up (a) shows the positioning accuracy of the NHT in locating an indent on the silver coating which acts as an interface between the gold wire ball and the copper substrate.

Mr Johnny Yeung of ASM Technology Singapore is acknowledged for providing the wire-bonded samples presented in this application note.

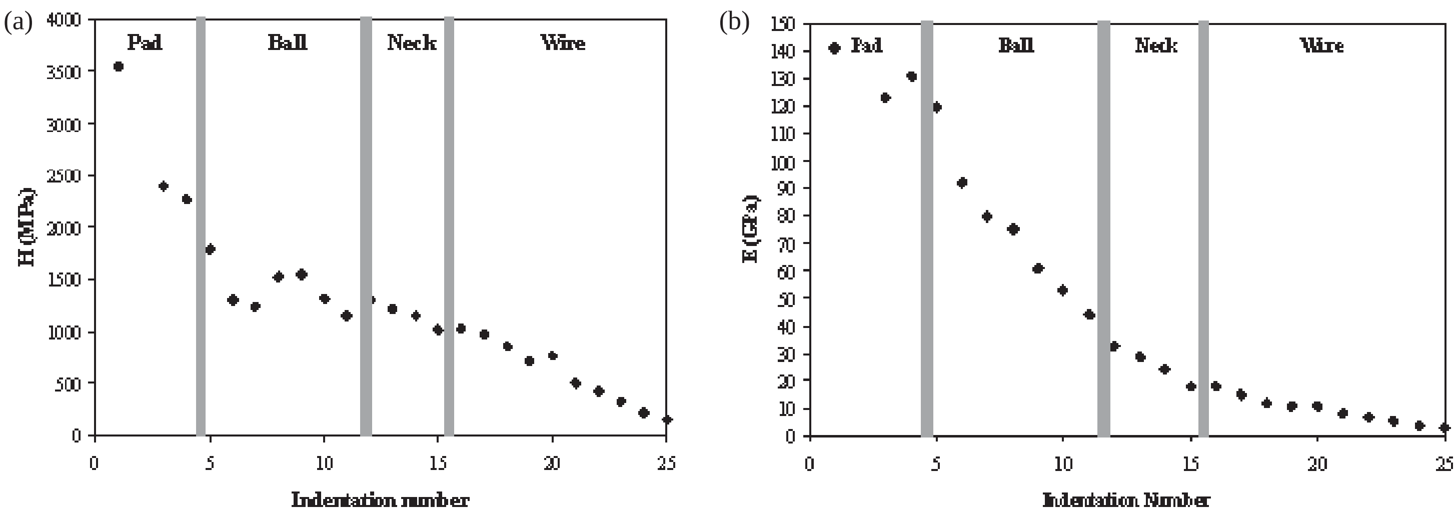

Figure 2 : Hardness (a) and Elastic Modulus (b) values plotted as a function of distance over the different tested areas of the gold wire bonded sample. Test conditions were 10 mN maximum load, 20 mN/min. loading rate and the indenter was of Berkovich geometry.

New Nano-Scratch Tester (NST) allows better magnetic hard disk characterisation

Very thin hard films of amorphous carbon (or diamond-like carbon (DLC)) are now widely used as protective overcoats for magnetic hard disks. This has stimulated a lot of work on adhesion assessment, as well as friction and wear properties of such materials. This application note features the results of nanoscratches performed on a typical hard disk surface [1], made up of different multilayers: these films consist of a 13 nm carbon overcoat on the top, with some magnetic, metallic and other films having a total thickness of about 120 nm.

Taking into account such thicknesses, a spherical diamond was chosen with a small tip radius (2 µm) in order to generate a maximum stress field near the sample surface. In order to study the friction fatigue of the sample, a multipass constant load cycle was used. By successively increasing the number of cycles, *N*, under a low load (2 mN), fatigue failure assessment was possible. Scratches were made with *N* varying from 1 to 70. Damage was seen to initiate when $N = 50$ and rapidly increased until catastrophic damage occurred by blistering. To verify the adhesive character of the coating damage, the blistered damage area can be removed by making a scratch in a transversal direction to that of the original scratch. This can also provide valuable information about multilayer plastic deformation below the DLC overcoat.

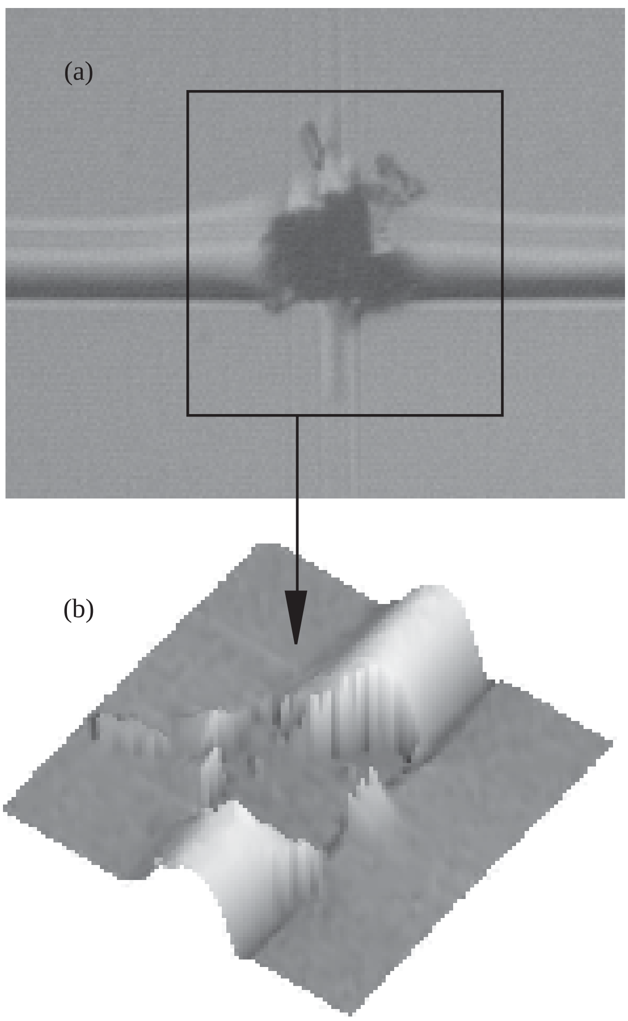

Figure 1 : Result of transversal scratches on a typical damage area having undergone 70 cycles with an applied load of 2 mN and spherical indenter of radius 2 µm. The optical micrograph (a) clearly shows the resulting decohesion and the SFM image (b) confirms the depth at which failure has occurred.

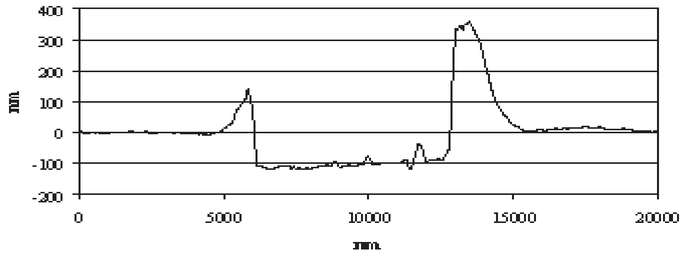

Figure 2 : SFM cross-sectional profile through the image shown in Fig.1 (b). The depth at which coating failure occurs can be accurately measured as being 100 nm below the surface. This corresponds to the interface between the substrate and first coating.

Transversal scratches were performed with the Y-axis translation table of the NST, the same tip and an applied load of 1 mN. The result is shown in Fig. 1, where coating decohesion and chip evacuation can be observed. SFM analysis with transversal profiles allows an accurate study of the damage process; the SFM cross-sectional profile of the delaminated area (Fig. 2) confirms that the adhesion failure is situated at the interface between substrate and first coating, because of the known multilayer thickness.

For a more accurate study of adhesion strength of the DLC film, a stylus with a smaller tip radius (0.5 µm or less) must be used. Experimental studies with different tip radii are currently being investigated. The NST is particularly suited to ultrathin coating characterisation of this kind, owing to its wide range of contact conditions and available loads. Under well-defined conditions, plastic deformation can be limited to the coating (s) with only minor effects, if any, on the substrate. This is especially important for the case of ductile or brittle coating/substrate composites.

[1] R. Consiglio, N. X. Randall, B. Bellaton and J. von Stebut, *Thin Solid Films* 332 (1998) 151 - 156

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

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:

6210.05.99V1 CSM Instruments / Printed in Switzerland