CSM Instruments
Advanced Mechanical Surface Testing

APPLICATIONS BULLETIN

Influence of tip size on the critical load during scratch testing with the Micro Scratch Tester

Introduction

With the Micro Scratch Tester (MST), the critical load can be determined by acoustic emission, optical microscopy, variation in penetration depth or variation in the tangential frictional force between tip and sample. However, it is difficult to express the adherence of a coating-substrate system in a quantitative way because the critical load depends on several parameters related to the testing conditions.

Some previous work [1] has already considered both intrinsic parameters (scratching speed, loading rate, diamond tip radius and diamond wear) and extrinsic parameters (substrate hardness, coating thickness, substrate and coating roughness, friction coefficient and friction force) which need to be taken into account in order to improve the interpretation of critical load results.

This application note responds to a growing demand from both MST and Revetest users for more information concerning the effect of different tip radii on the measured critical loads. The samples used for this study consisted of TiN, W, DLC, Al and Au in order to compare results between coating materials with different mechanical properties. Full details of all samples are summarised in Table 1.

Spherical diamond indenters were used with radii of 20, 50, 100 and 500 µm. Five scratches were performed with each indenter on each sample and average values of critical load were calculated. The scratch length in each case was 5 mm and the loading rate was kept constant for all measurements at 30 N/min. Either two or three critical loads were determined for each sample type by optical microscopy inspection of the damaged area after scratching. In some cases, the acoustic emission signal was also used to confirm the measured values.

[1] P. A. Steinmann, Y. Tardy and H. E. Hintermann, Thin Solid Films 154 (1987) 333 - 349

Coating	Deposition Method	Thickness (µm)	Substrate
TiN	CVD	1.50	440C steel
W	CVD	1.20	440C steel
DLC	CVD	0.56	Silicon wafer
Al	Sputtering	0.50	Silicon wafer
Au	Sputtering	0.50	Silicon wafer

Table 1: Summary of tested coating-substrate combinations. Note that CVD describes the Chemical Vapour Deposition method. Pure Si was chosen as the substrate for the thinner coatings in order to have a very low surface roughness (negligible influence on the critical loads).

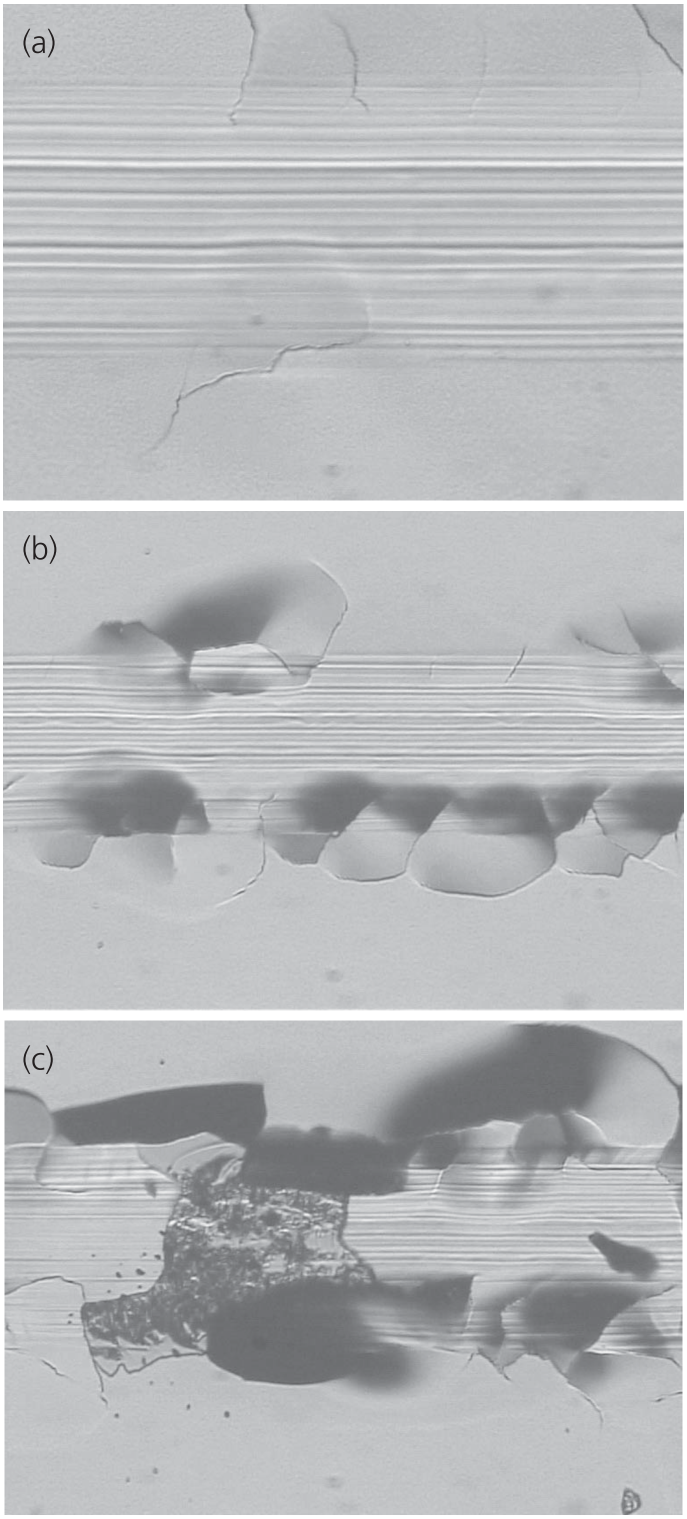

Figure 1: Optical micrographs of critical failure points along a progressive load scratch performed on a Tungsten (W) sample. The first failure, Lc1 (a), corresponds to initial cracking, the second failure, Lc2 (b), to extensive cracking and final failure, Lc3 (c) to partial delamination of the coating from the steel substrate.

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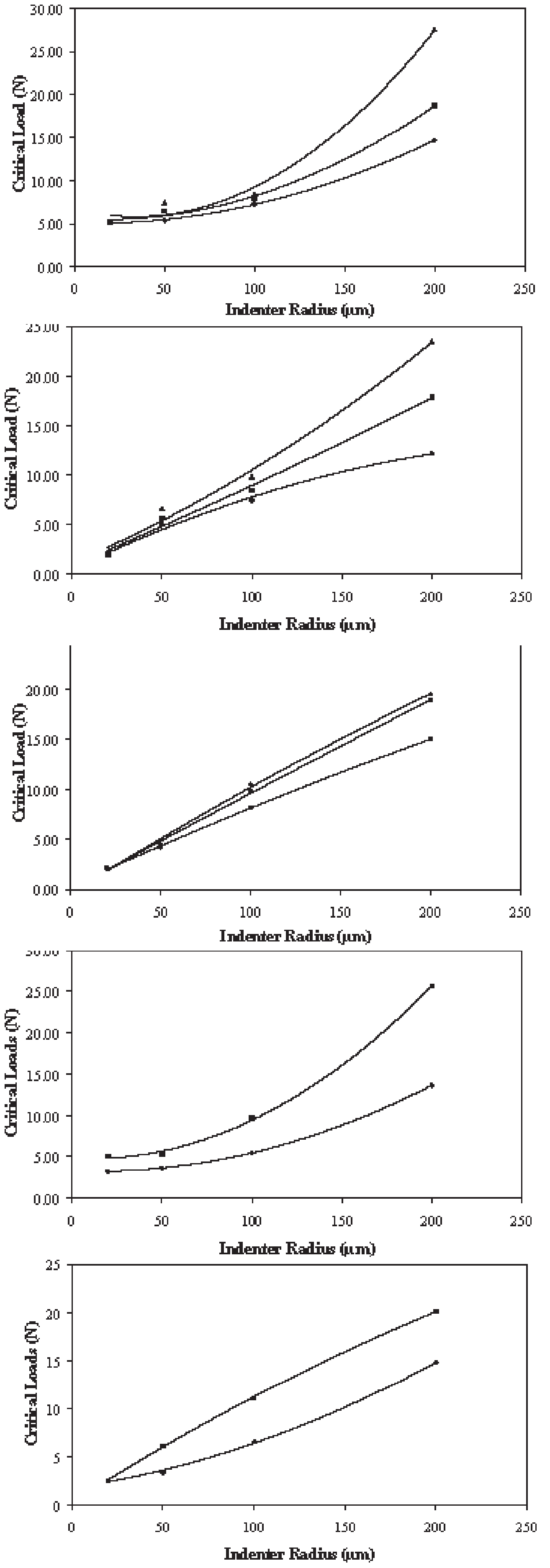

Figure 2: Variation in the critical load as a function of different indenter radii for each critical failure point measured on (a) TiN, (b) W, (c) DLC, (d) Al and (e) Au.

Results

The results presented in Fig. 1 show the three typical critical failure points observed on the W sample. The variations in critical load are summarised for all samples as a function of different indenter radii in Fig. 2. The optical micrographs in Fig. 3 show the characteristic chipping which occurs when scratch testing TiN.

The basic trend of the results was the same regardless of the sample type: the critical load values increase as a function of indenter radius. This can be explained simply by considering that the smaller the tip radius, the higher the contact pressure, and thus the lower the load required to cause failure in the coating. However, it should not be forgotten that the critical load depends not only on the indenter radius, but also on the loading rate and the scratching speed. Furthermore, when choosing a suitable indenter radius for a particular coating-substrate system, it is important to consider the extent of the stress field which will be produced under the indenter tip. For characterising the adhesive properties of a coating it is recommended to create a stress maximum at the interface between coating and substrate.

Future work is envisaged to relate the loading rate and scratching speed to the measured critical load, and therefore to be able to define standard testing conditions for a wide range of coated systems which take account of both the intrinsic and extrinsic parameters of the scratch test.

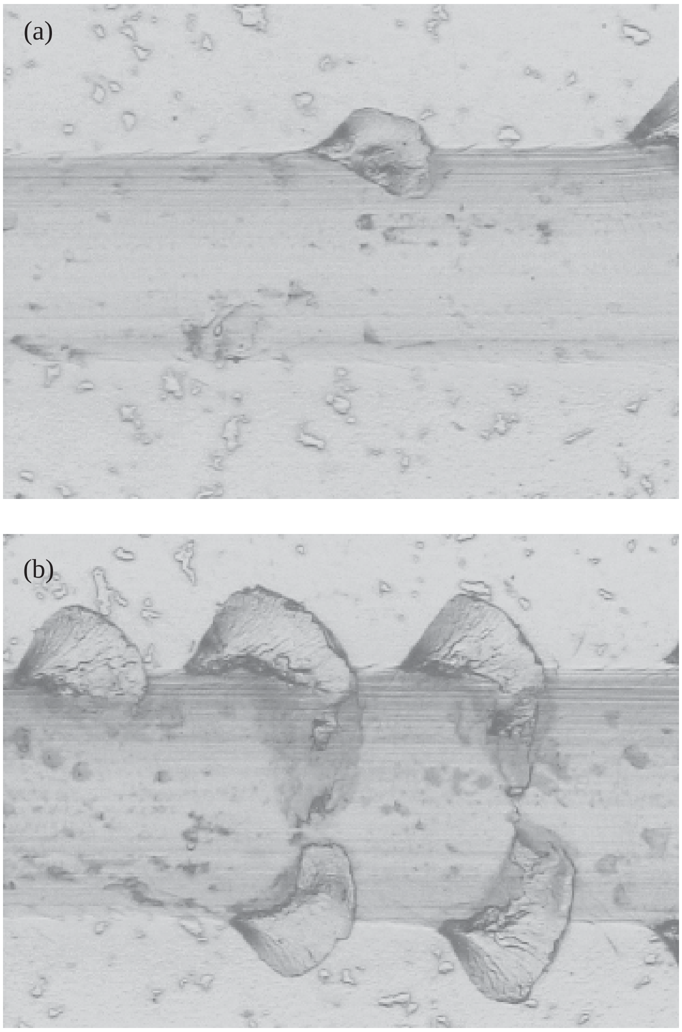

Figure 3: Optical micrographs of critical failure points along a progressive load scratch performed on a TiN sample. The first failure, Lc1 (a), corresponds to first chipping and the second failure, Lc2 (b) to extensive chipping along the sides of the scratch path.

Failure of TiN-coated polymer measured with the Pin-on-Disk Tribometer

Introduction

This application note features the Pin-on-Disk Tribometer as a perfectly suited tool for characterising the rupture point of a TiN-coated polymer used in printing applications. Of interest is the fact that this type of sample consists of a very hard coating deposited onto a very soft substrate. Scratch testing would not be recommended for this kind of sample as the soft substrate would fail much earlier than the coating, causing inaccurate results.

The testing procedure consists of a lightly loaded static partner (standard 100Cr6 ball of radius 6 mm) in sliding contact with the sample which is glued to a hard substrate. The entire contact zone is submerged in a distilled water lubricant to prevent premature failure. By using a slow sliding speed (1.0 cm/s), the friction coefficient can be accurately monitored until a sharp decrease signals that the coating has failed and that the substrate has been reached. The actual failure mechanism can be confirmed by optical microscopical observation of the wear track at various stages during the test.

Results

Some typical results are presented in Fig. 1 for a typical TiN-coated polymer sample. The Tribometer trace shows the abrupt change in friction coefficient (after a sliding distance of 0.7 m) when the TiN coating fails and the static partner (in this case a 100Cr6 ball) breaks through to the polymer substrate. The optical micrograph in Fig. 1 (b) shows the initial failure of the coating which is characterised by partial delamination along the direction of the wear track. Complete rupture of the coating (Fig. 1 (c)) corresponds to the sharp decrease in the friction trace. The static partner has completely ruptured and removed the coating along the wear track. Note also the small cracks which are visible along the sides of the track.

This application is a typical example of the kind of coating/substrate system which is becoming ever more common in several industries. The use of hard metallic coatings on polymeric components has been around for several years, particularly in the automobile industry where many exterior trim parts are produced by chrome-plating onto an etched polymer substrate. Similar components are now being fabricated for decorative applications, e.g., shower fittings, door knobs, kitchen accessories, etc. With such new types of coating/substrate system has come the need for satisfactory methods of adhesion quality control, of which the Pin-on-Disk Tribometer has shown considerable promise.

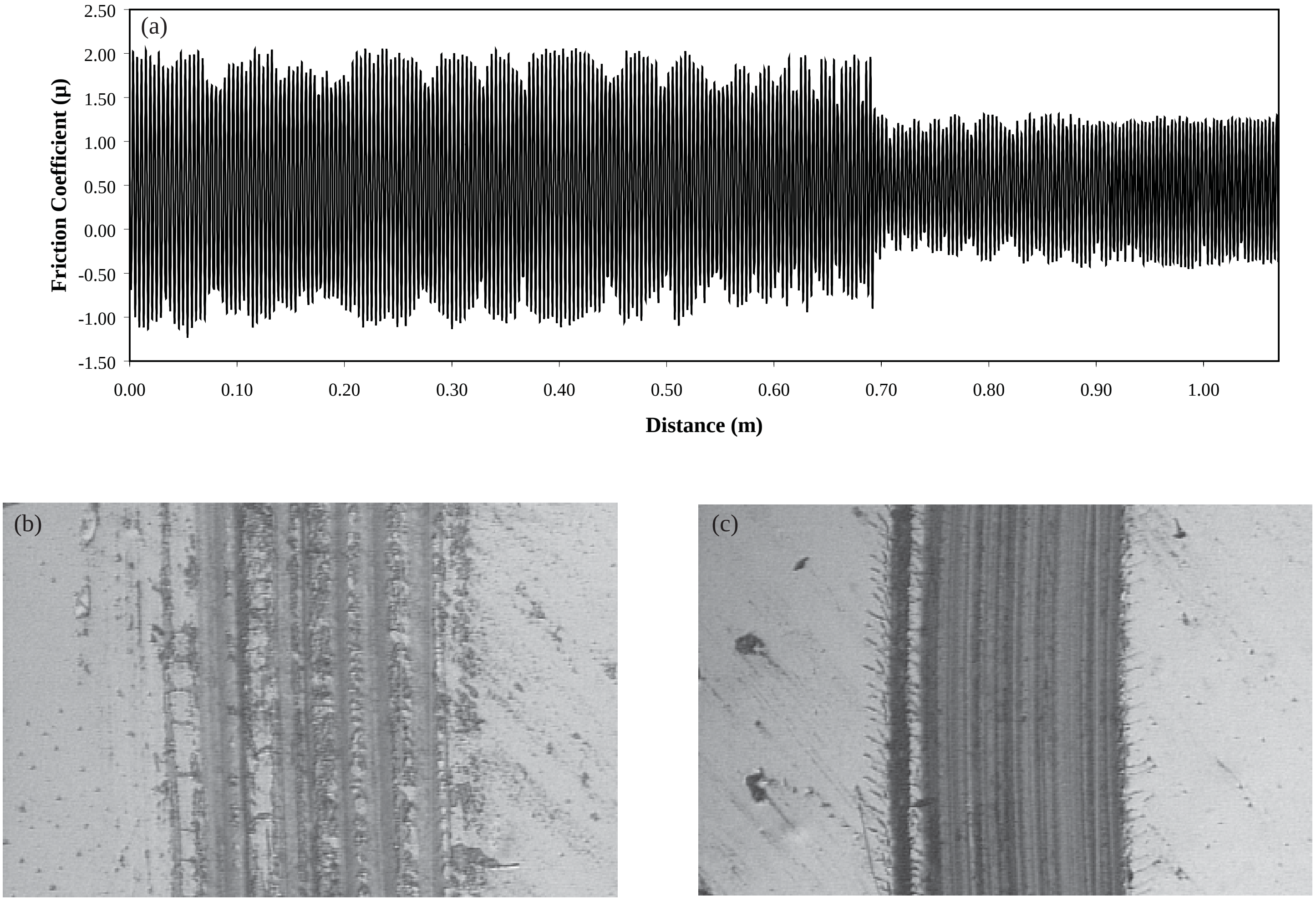

Figure 1 : Pin-on-disk Tribometer trace (a) showing the abrupt change in friction coefficient (after a sliding distance of 0.7 m) when the TiN coating fails and the static partner (100Cr6 ball) breaks through to the polymer substrate. The optical micrographs show initial failure of the TiN coating (b) and complete rupture (c). The test was performed with speed 1.0 cm/s, applied load 1.0 N and sliding radius 6 mm.

Nano-Scratch Tester (NST) for measurement of dielectric thin films

Since the introduction of advanced deposition techniques, the formation of thin films and coatings exhibiting dielectric properties has become commonplace for applications where specific electrostatic properties are required. Dielectric thin films have the unique feature of being able to store electrostatic charge and are finding increased use in areas such as printed circuit boards (PCBs), solar cells and touch-pad screens. In the form of hard coatings, materials in use today include Titanium dioxide, Borosilicates and Titanates (Ba, Sr, Ca, Mg and Pb), these being found in specific applications such as DRAM chips and sensors. Photodefinable dielectric coatings serve many functions such as passivation layers, stress buffers, planarising layers and protective masks for subsequent assembly operations. Their mechanical integrity, as well as adhesion, are important considerations.

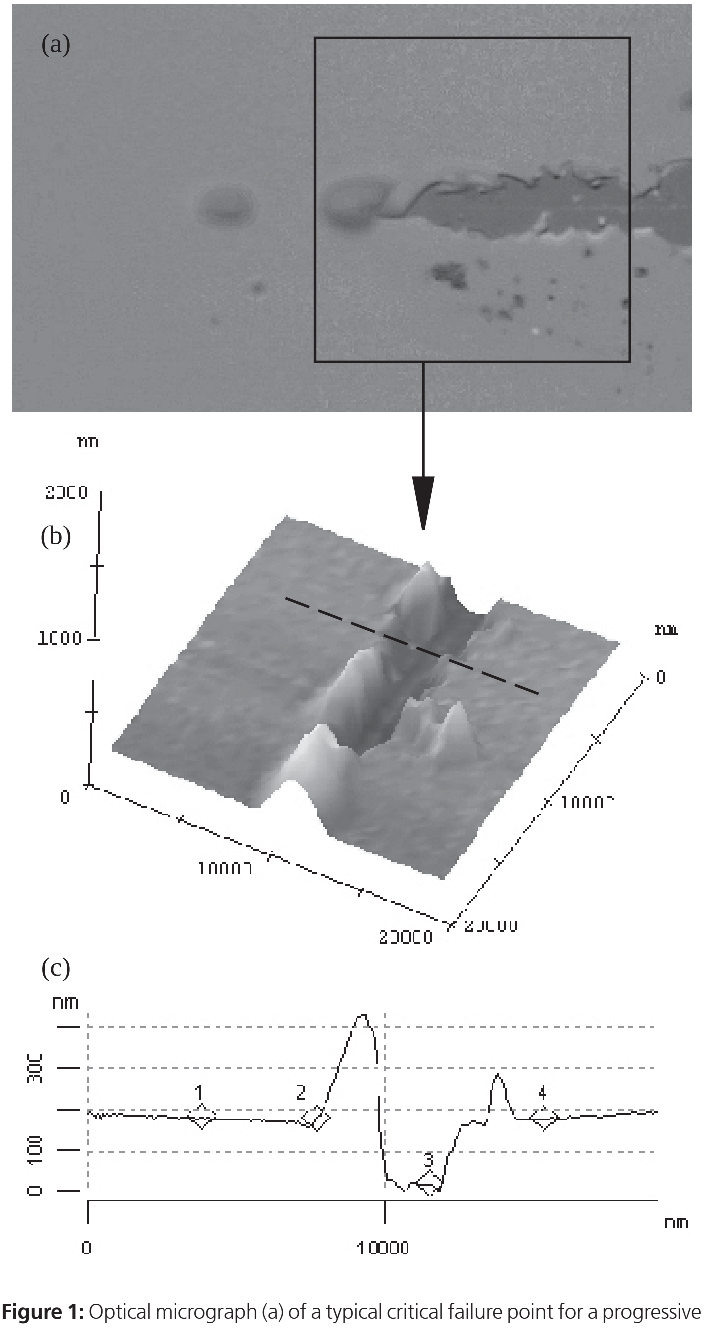

Figure 1: Optical micrograph (a) of a typical critical failure point for a progressive load scratch on a dielectric thin film. The SFM image (b) confirms the extent of delamination, from which a profile (c) could be extracted in order to verify pile-up phenomena (dotted line in (b)).

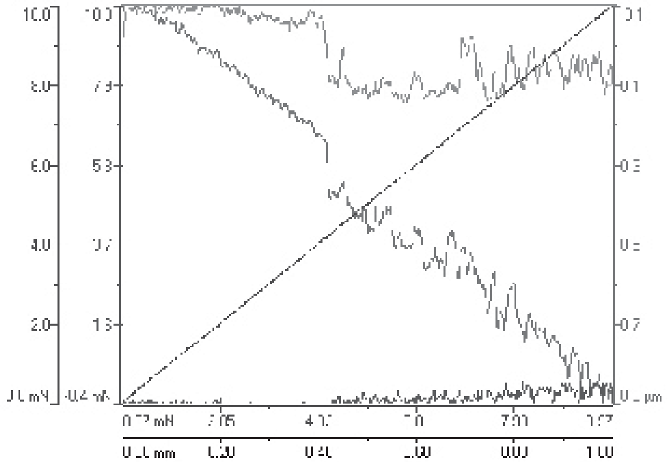

Figure 2 : NST results for the sample shown in Fig. 1 including residual depth (R_D), penetration depth (P_D), tangential force (F_T) and normal force (F_N) curves. The progressive load scratch test was performed over the range 0 - 10 mN with a spherical diamond tip of radius 2 µm. The critical load at which failure of the coating occurred was 4.2 mN.

This application note features a typical example of a complete test carried out with the Nano Scratch Tester (NST) on a hard dielectric coating of thickness 100 nm. Fig. 1 shows the result of a progressive load scratch which caused coating failure at an applied load of 4.2 mN. The area around this critical point has been imaged at high resolution with the Scanning Force Microscope (SFM) in Fig. 1 (b) and a profile extracted (Fig. 1 (c)) which confirms the presence of pile-up along the sides of the scratch path. Fig. 2 shows the corresponding measurement results: of particular interest is the observed relaxation of the Tungsten substrate (seen as the difference between the penetration depth, P_D, and the residual depth, R_D, as measured using the post-scan facility).

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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

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