Micro Materials
Excellence in Nanomechanics

NanoTest integrated modelling modules

Advanced simulation – deeper understanding for improved coatings design

Introduction

Analytical modelling utilising the effective indenter concept for stress calculations is a powerful tool for getting the most out of nanomechanical and nano-tribological tests [1]. Key strengths of the NanoTest design (such as high lateral rigidity for straight scratch tracks, ultra-low thermal drift at elevated temperature etc) enable production of high-quality artefact-free data to be used as direct input to the physical-based models.

The detailed simulated stress distributions enable data to be interpreted more effectively. They can provide mechanistic information which can be the key to unlocking exactly where and why coatings systems fail in scratch (see also figs. 1-3, 6-8) [2-4] and fretting [5] tests, and then to designing coatings with improved performance.
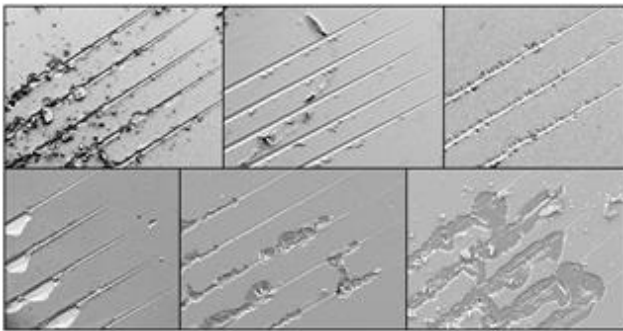

Figure 1 Different deformation mechanisms in nano-scratch tests on (Ti,Fe)N$_x$ coatings [2]

The effective indenter concept has also been applied to determine the coating elastic modulus free of substrate effects in a more reliable method than the standard unloading approach. At high temperatures indentation creep can make elastic modulus measurements less reliable, as shown at room temperature in the ISO standard, ISO14577-4, and to improve the accuracy of the high temperature data analysis a time-dependent effective indenter approach can be used.

Micro Materials have worked with SIO, an innovative modelling company who have developed these models and the accompanying intuitive software interface, to fully integrate this advanced modelling capability to the NanoTest system.

In this Technical Note we illustrate some of the possibilities from combining the advanced modelling with the NanoTest data on hard PVD coatings and bulk metallic materials.
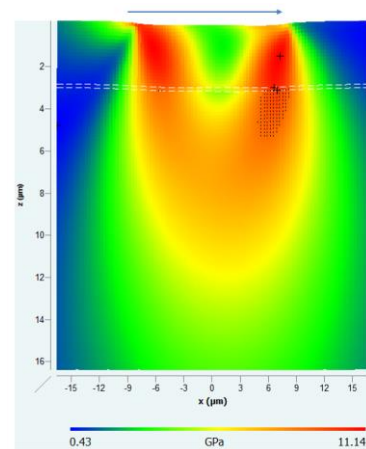

Figure 2 Simulated von Mises stress distribution at the L$_{c2}$ critical load in scratch testing TlAlCrSiYN/TiAlCrN bi-layer coating on WC-Co. Region where von Mises stress developing in the sliding contact exceeds the yield stress is marked [see ref 4]. Probe radius = 25 μm.
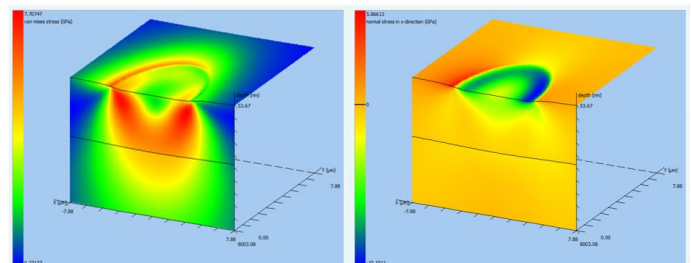

Figure 3 Simulated von Mises and normal stress distributions at the L$_{c1}$ critical load for Si-doped DLC on hardened steel. Probe radius = 5 μm.

Case studies

1. Nanoindentation on very thin coatings on low yield stress substrates

Nanoindentation on very thin coatings (100 nm or less) is more challenging when they are deposited on softer substrates. When coatings are very thin plastic flow can occur in the substrate before the true coating hardness is reached. Sensitivity to film properties is improved by using a sharper indenter so that coating-only properties could be measured. In tests of 40 nm and 80 nm ta-C films deposited on glass a sharp cube corner diamond indenter was used to ensure the measured hardness is due to the coating hardness and not a lower value due to plastic flow in the substrate.

Determining coating-only elastic properties is also challenging. When the relative indentation depth is 10% of the coating thickness there is some contribution from the less stiff glass substrate and the measured elastic modulus is lower than the real coating value. Two methods were used to avoid this:- (i) an ISO 14577--4 type approach - multi-cycle load-partial unload tests and extrapolate the results to zero depth (ii) analytically account for the substrate contribution to the measured result. It can be seen below that both methods provide improved accuracy, particularly for the 80 nm film, with the modelling being experimentally more convenient.
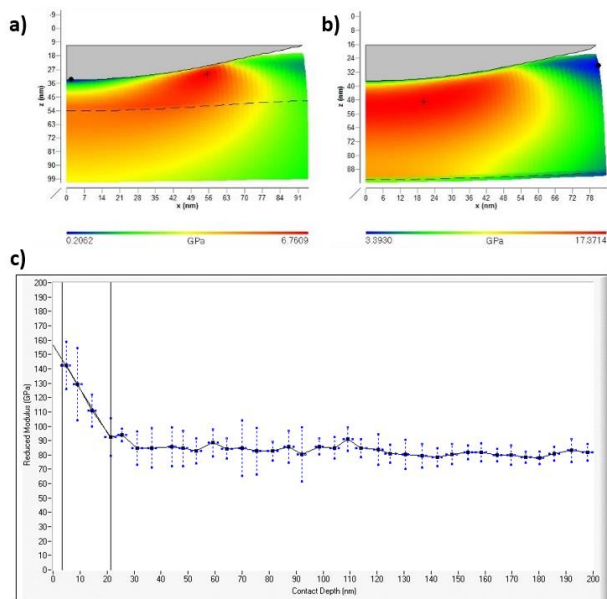

Figure 4 Illustrative stress distributions on the point of unloading for (a) 40 nm (b) 80 nm film. (c) NanoTest load-partial unload data and ISO extrapolation method.

ta-C	E (10%)/GPa	E (ISO)/GPa	E (SIO)/GPa
40 nm	105	115	111
80 nm	138	175	179

2. Nanoindentation – accounting for time-dependent behaviour at high temperature

To accurately determine the nanomechanical properties of weld and parent material for P91, a high Cr content creep resistant steel used in steam pipes in power plants, at its operating temperature requires (i) experimental capability to perform measurements with minimal thermal drift (ii) improved analysis to take into account the influence of creep.

Tests to 100 mN on the weld material with a cubic boron nitride indenter in argon purging atmosphere are shown below [6].
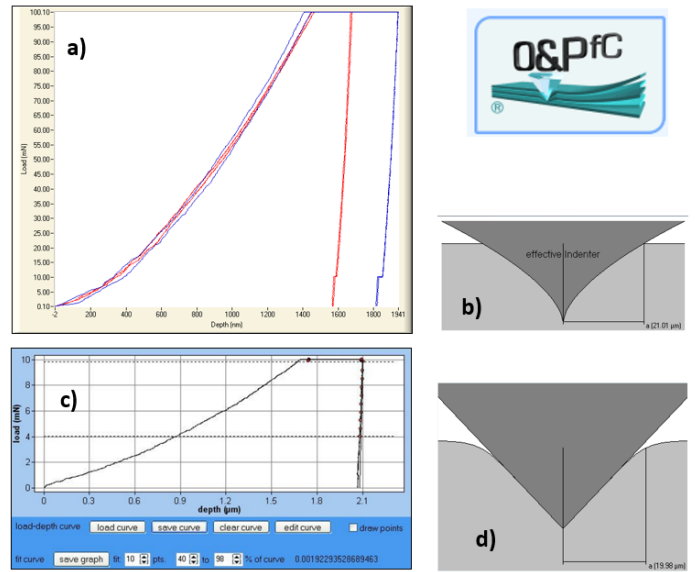

Figure 5 (a) 650 ℃ tests on P91 weld material with 40s (red) or 300s (blue) hold at peak load (b) effective indenter shape not accounting for time-dependent c) O&PfC module fitting creep and unloading (d) time-dependent effective indenter

The material appears stiffer due to the influence of creep on the slope of the unloading data, especially for the shorter hold time at peak load.

Conventional elastic unloading curve analysis does not account for the time dependent depth changes occuring during unloading resulting in the modulus being over-estimated.

Hold time (s)	Creep rate at end of hold (nm/s)	E/GPa from standard analysis	E/GPa from TDEI analysis
40	3.1	179	117
300	0.5	128	

Using the technique from SIO developed to modify the effective indenter geometry to compensate for time dependent depth change (TDEI – time-dependent effective indenter) resulted in improved results and agreement with literature data on bulk samples (E ≈110 GPa [7]).

3. Nano- and micro-scratch

The von Mises, tensile and shear stresses during the micro-scratch test were evaluated using the Scratch Stress Analyzer, which uses a physical- based analytical methodology to determine simulated stress distributions.

The input parameters to the physical-based analytical model are NanoTest data - the mechanical properties of the coating and substrate i.e., H, E, H/Y, their Poisson ratios, together with the applied load, scratch depth data, friction coefficient and probe radius used in the nano- and micro-scratch tests.
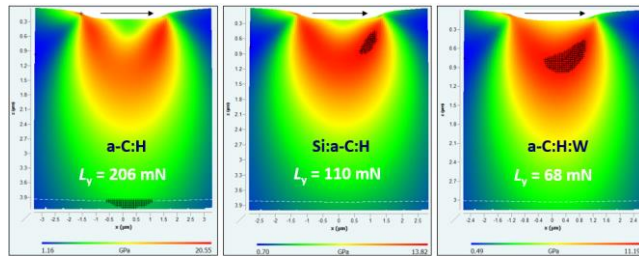

Figure 6 Von Mises stress distribution at the critical load for plastic yield for DLC coatings on hardened tool steel. Over-stressed regions are hashed.

Figure 6 shows that the different mechanical properties of the DLC coatings alter the deformation position when using a 5 µm probe.

For the harder a-C:H the interface is initially weakened by substrate deformation but for the softer Si- and W-doped DLC films the coating yields first.
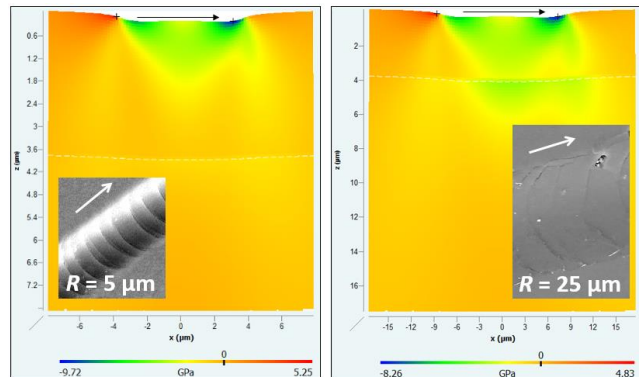

Figure 7 normal stress distributions at L_{c1} for Si-doped DLC tested with R = 5 and 25 µm probes

Figure 7 shows the tensile stresses developed at the L_{c1} critical load.

R (µm)	L_{c1} (mN)	Max VM in coating (GPa)	Max VM in surface (GPa)	Max normal stress at surface (Ga)
5	445	7.7	5.0	5.3
25	1830	6.7	5.1	4.8

4. High temperature micro-scratch

Experimentally it has been shown that on AlCrN and AlTiN coatings deposited on WC-Co the critical load for coating failure can be higher at elevated temperature than at 25 °C [3]. The modelling shows that this is not a result of improved adhesion at 500 °C. For AlTiN in particular the stress distributions were strongly temperature dependent. At 25 °C the coating failure occurred by interface weakening from substrate yield in combination with high tensile stress at the surface. At 500 °C the yield occurerd in the coating suggesting the deformation proceeds by a different mechanism. Analytically determined maximum shear stresses at L_{c2} were lower at 500 °C suggesting the coating-substrate bonding strength is actually slightly reduced.

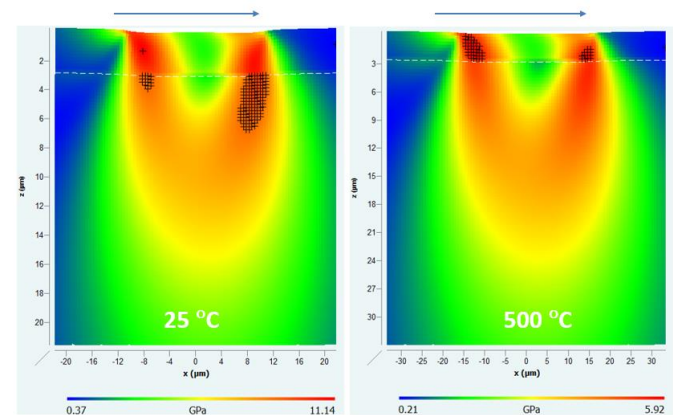*Figure 8 temperature-dependence of the von Mises stress distributions at L_{c2} when scratching AlTiN coating with 25 µm diamond. Over-stressed regions are hashed.*

5. Nano-fretting

In nano-fretting tests on DLC coatings the modelling approach provided closer simulation to the experimental data than determined from the Archard wear equation [5].
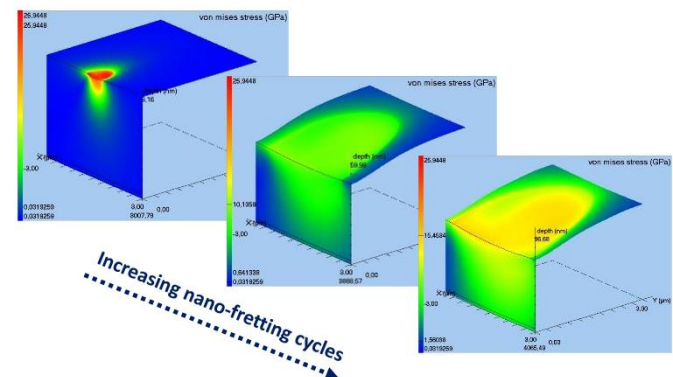

Figure 9 Evolution of the simulated stress distribution with number of nano-fretting cycles for a-C:H on hardened steel. Within a few cycles the contact pressure decreases due to the increasing size of the fretting wear scar. R = 5 µm diamond probe

6. Highly spatially resolved determination of residual stress by nanoindentation

Nanoindentation with modelling in Film Doctor was performed to determine residual stresses on a metallic steel sheet. Despite the highly rough surface the modelling was able to show good correspondence with scatter of residual stress determined hole-drilling methods [8].
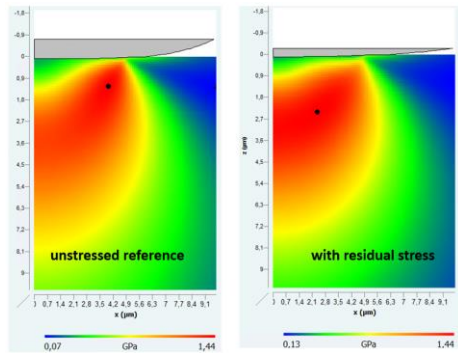

Figure 10 Von Mises stress distributions. The onset of yield (marked with the black dot) changes with residual stress

NanoTest Specifications

The Saxonian Institute of Mechanics (SIO) provide a range of different analytical modelling modules including:- Film Doctor, Oliver and Pharr for Coatings, Scratch Stress Analyzer, Test Optimizer, SIO Toolbox etc.

The NanoTest software includes data export routines for SIO formats so that the SIO modelling capability is fully integrated with the NanoTest indentation, scratch and fretting modules in low load (0-500 mN) and 0-30 N high load range, and across to the temperature range (to 850 °C in the NanoTest Vantage).


Micro Materials Ltd

At the forefront of nanomechanics since 1988:-

- First commercial high-temperature nanoindentation stage
- First commercial nano-impact stage
- First commercial liquid cell
- First commercial instrument for high-vacuum, high-temperature nanomechanics

Benefits of Modelling

- Improved accuracy of high temperature elastic modulus data
- Improved determination of coating-only properties of ultra-thin films
- Maximum interfacial shear strength measurement for true coating-substrate bonding information
- Simulated stress distributions to explain failure behaviour in scratch tests
- Understand temperature-dependent scratch test data
- Predict wear performance more reliably than Archard
- Highly spatially resolved residual stress determination
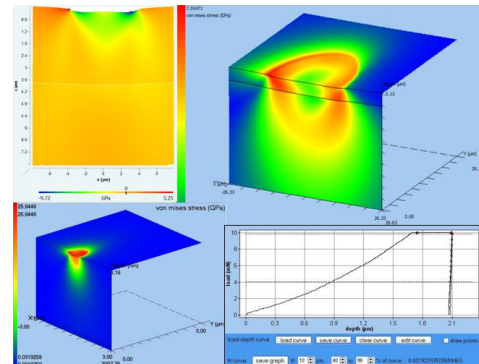
References and acknowledgements

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