Micro Materials

Excellence in Nanomechanics

Advanced Scratch Test Module for Platform 5 NanoTest Vantage

Introduction

The scratch and wear functionality in the earlier Vantage systems has been significantly enhanced in the new module for Platform 5. The improvements enable NanoTest users to make the most of the technical advantages of the NanoTest high lateral rigidity and thermal stability. The user has full control over load/unload/hold segments enabling scratch and wear tests with complex loading histories to be designed. New experiment types include abrasion simulation and reciprocating wear tests. To simulate abrasive wear processes more directly multiple parallel scratches can be run in a single experiment, with software-generated random offsetting of scratch starting position. To study wear/fracture processes in greater detail the user can choose regions for analysis and for every scan in the test the system automatically calculates the mean and standard deviation of all measured parameters (including on-load and residual depths, friction forces and friction coefficients, 6 different surface roughness measurements etc).

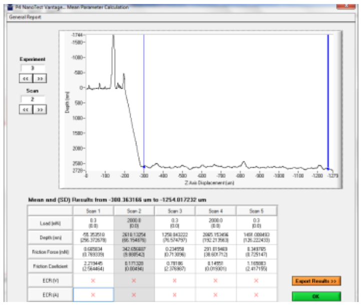
What's new in Platform 5?

Data acquisition

- User-defined multi-load loading history option for single scratches and multi-pass tests
- Abrasion simulation
- Reciprocating scratch and wear tests
- User-defined data densities

Data Analysis and Reporting

- Multi-axis composite plots
- Enhanced levelling options
- Integrated microscopy / positioning
- Automatic analysis of surface roughness statistics, depth, friction, μ and ECR for user-defined regions
- Step height calculation
- ISO/CEN 17627 test reports



Mean and SD of depth and friction data for every scan in a multi-pass scratch test from user-defined region

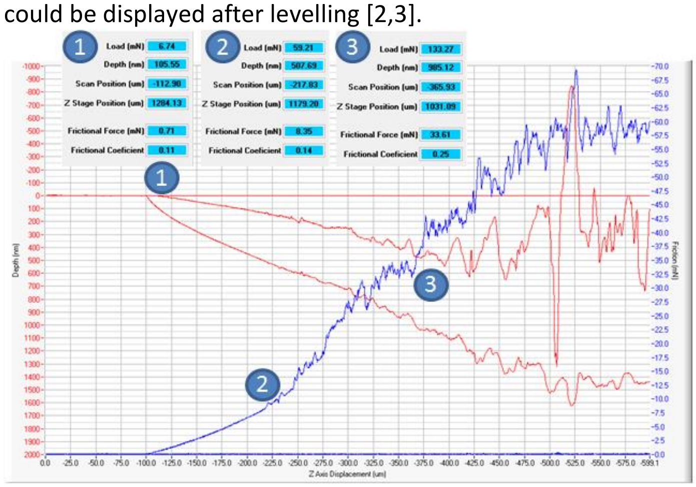
Case study examples

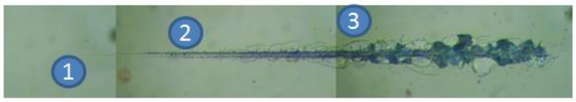
1. Nano-scratch

Using the NanoTest 500 mN loading head ramped load scratch tests were performed as 3-pass topography-scratch-topography tests on (i) 80 nm ta-C PVD coating on glass and (ii) 1.1 μm TiN/Si_3N_4 PVD nanocomposite coating on Si(100) with a R = 3.8 μm radius spherical diamond indenter. Abrasion simulation tests were also performed on the nc-TiN/Si_3N_4 coating.

3-scan tests on 80 nm PVD ta-C coating on glass

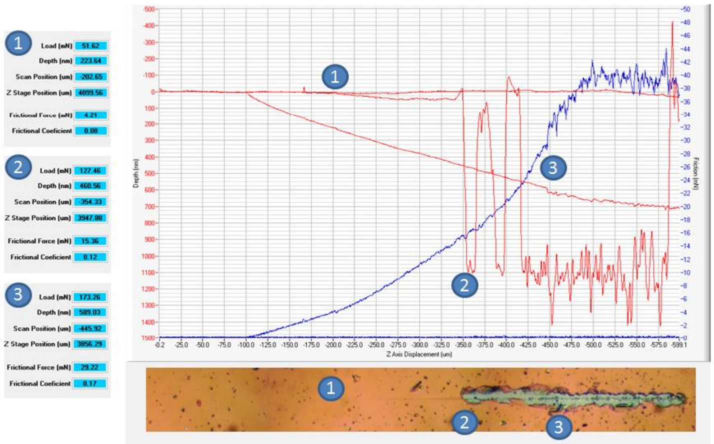
The 3-scan procedure (topography scan – ramped load scratch - topography scan) described in CEN 17627 [1] enables identification of failure mechanisms, such as the role of stress, in more detail. First reported by IBM in 1989, multi-pass scratch and wear tests have been part of the NanoTest scratch module since 1991. Micro Materials later showed that by removal of the instrument compliance contribution to the measured deformation the true nano-scratch and nano-wear depth data could be displayed after levelling [2,3].





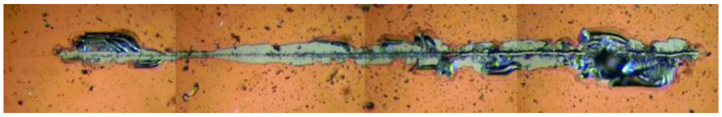
The figure above shows an example on the 80 nm ta-C on glass where the load is ramped linearly to 200 mN and the depth data has been corrected for slope and compliance following the method developed in [1-3]. The ta-C coating is thin, hard and stiff (H = 25 GPa, E = 175 GPa, ISO14577-4 measurements performed with a sharp cube corner indenter to ensure full plasticity in the coating before substrate yield). In the scratch tests with the 3.8 μm probe the ta-C yields at low load (point 1 in figure) despite having high H^3/E^2 due to plastic yield occurring in the softer glass substrate. Coating failure occurred at higher load due to high coating bending.

3-scan tests on 1.1 μm PVD nc-TiN/Si_3N_4 coating on Si



1 = onset of plasticity, 2 = delamination 3 = compressive failure

In the above example on the thicker nc-TiN/Si_3N_4 coating on Si(100) with H = 28.9 GPa and E = 425 GPa, the load for yield is much larger. Analysis shows yielding within the coating. The high H/E and H^3/E^2 of the coating resulted in delamination around the scratch track.

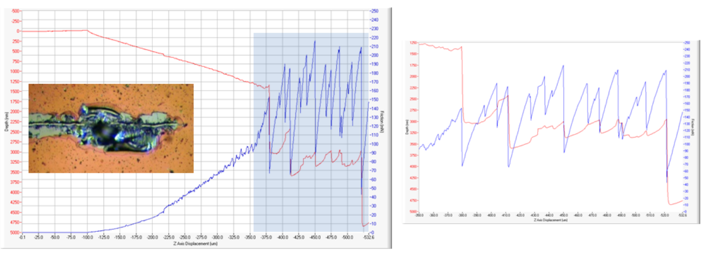


Extensive substrate fracture at high load

Nano-scratch tests to a higher maximum load of 500 mN revealed a transition substrate fracture-dominated wear at >415 mN.

Friction-topography investigations can be very useful to show location of failure relative to sliding probe and hence whether tensile or compressive stresses induce failure, and to study how wear proceeds in multi-pass tests.

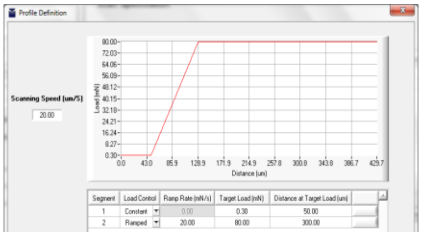
In the scratch test to 500 mN fracture in the Si(100) substrate at high load causes abrupt changes in contact area that alter the measured friction forces, as shown below.

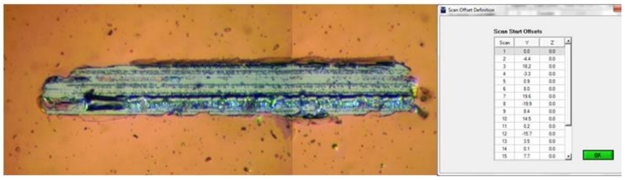


Abrasion simulation by parallel scratching

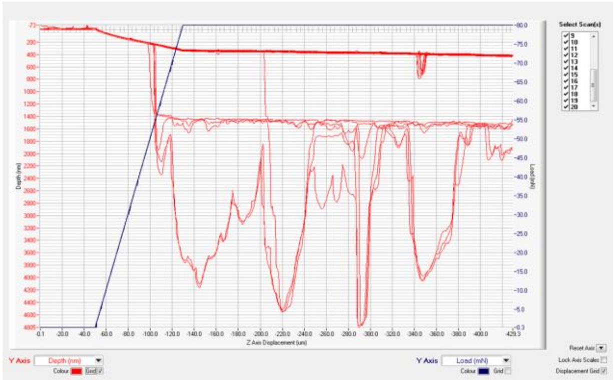
Parallel scratches can be set up as a single experiment with a random start offset to simulate abrasive wear processes where wear (and delamination on coated components) processes involve complex interactions with surface damage, pile-up and wear debris generated by previous contacts.

An example on a 20 cycle abrasion test at 80 mN on the nc-TiN/Si3N4 coating is shown below. The wear load was chosen to be between the critical loads for yield and cracking in a single scratch.

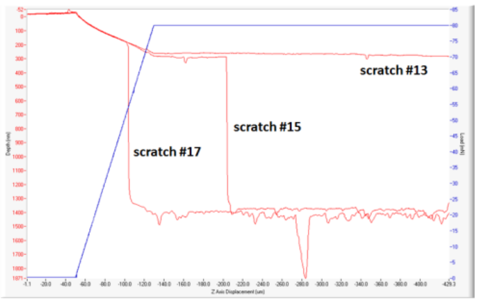




The box-width is chosen by the user and then random scratch positions are generated in the NanoTest software, as illustrated schematically above for 3 of the 20 scratches. Composite-plot analysis reveals a multi-stage failure process involving (i) a gradual fatigue process (ii) partial coating failure over track (iii) coating failure over entire track (iv) extensive substrate failure and dramatic increase in surface roughness. The substrate failure (large scale lateral cracking of the silicon seen near the bottom of the worn region) required >400 mN in a single scratch.



Composite plot analysis of abrasion experiment

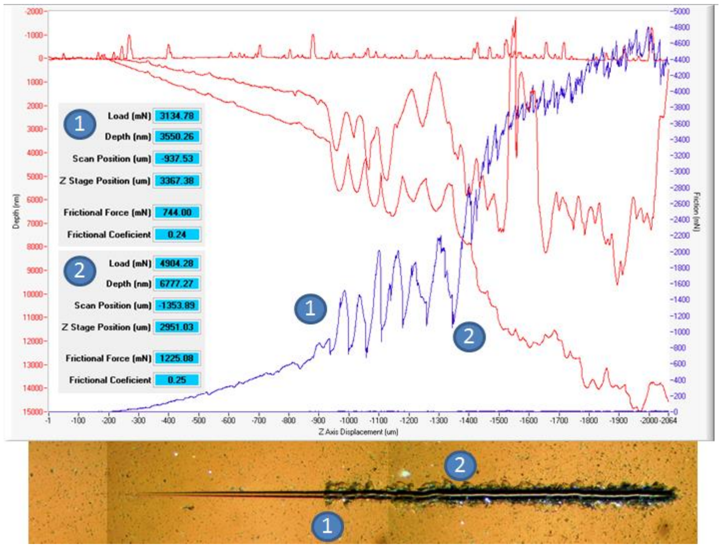


Selected scans reveal damage progression mechanism

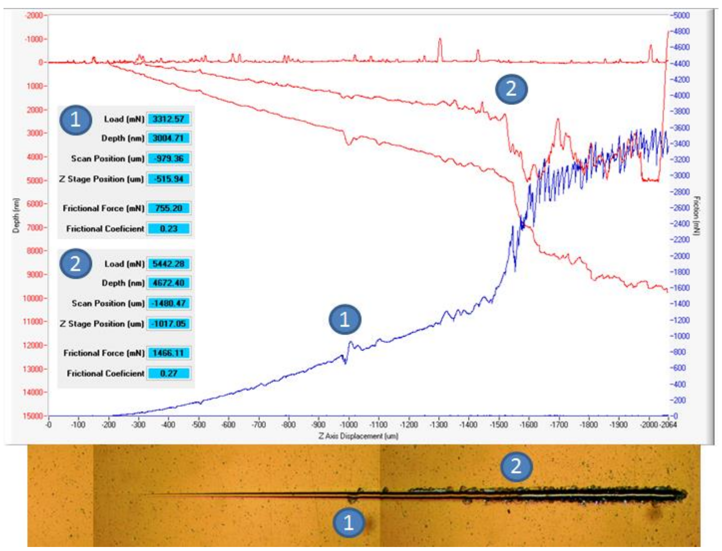
These type of abrasion simulation experiments are equally applicable to bulk samples, e.g. as part of novel alloy development.

2. Micro-scratch tests of TiN coatings on steel

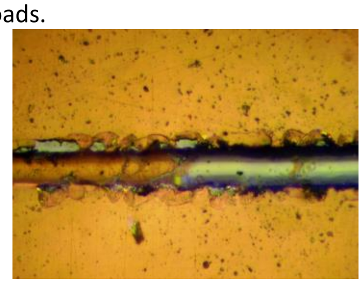
With the 30 N head it is possible to test thicker coatings and also investigate the influence of hardened sub-layers on scratch test critical loads and failure mechanisms. Ramped load scratch tests performed as 3-pass topography-scratch-topography tests with a R = 25 μm radius spherical diamond indenter on 2 μm TiN PVD coatings with high surface roughness on (i) D2 tool steel and (ii) ion-nitrided D2 tool steel.



Micro-scratch test on TiN on D2 tool steel (above) and ion nitrided D2 tool steel (below). 1 = critical load for chipping 2 = total coating failure

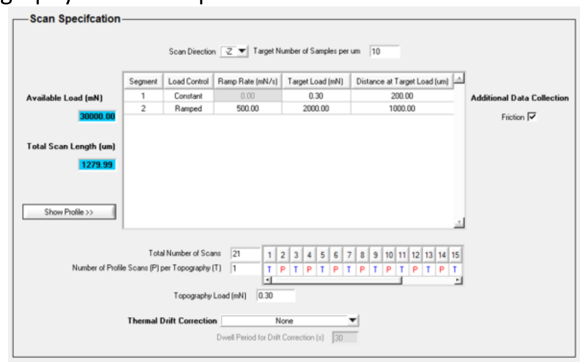


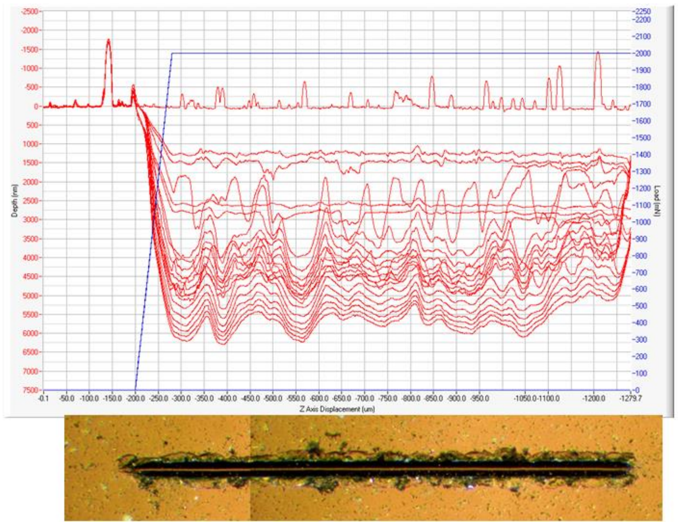
The figures above show typical data from ramped tests to 7.5 N after correction for slope and compliance. The increased load support shown in the duplex coating results in less chipping and higher critical loads.

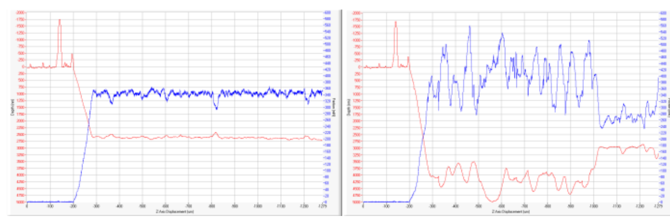


3. Repetitive micro-scratch tests of TiN on tool steel

Multi-pass tests involving 10 scratches at 2 N were performed on the TiN deposited on un-modified tool steel. Repetitive sub-critical load tests were performed on the non-nitrided TiN. These involved 21 scans with 10 scratches and 11 intermediate topography scans set up as:-

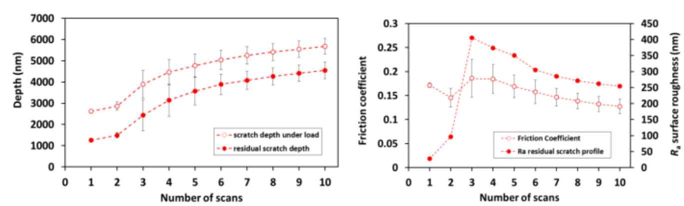






Correlation between friction (blue) and topography (red) in scans before (left) and after failure (right)

Multiple signals are available to pin-point the onset of wear processes and transition between plasticity-dominated to fracture-dominated deformation. The user can select any region of the wear track for analysis and export.



Isolated failure during second scan at 2 N and dramatic failure over entire track on scan 3.

Specifications

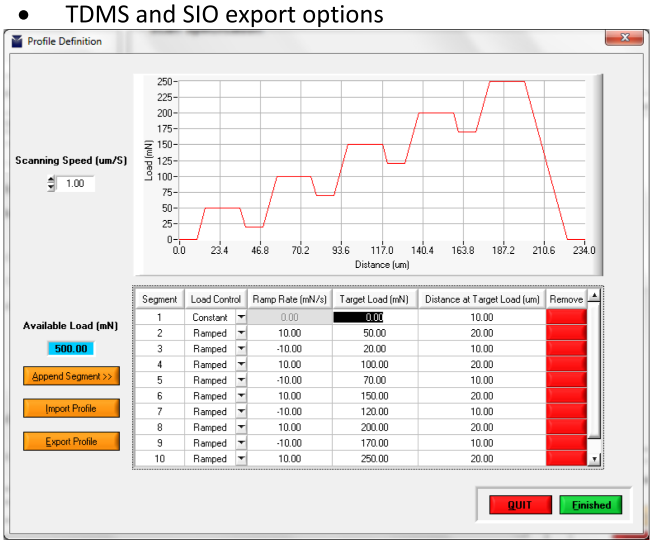
- Nano- (to 500 mN) and micro- (to 30 N) scratch test capability designed to fully meet and exceed requirements of CEN 17627
- Available as an option for either (i) as part of a Platform 5 upgrade (ii) in a new NanoTest Vantage system.

Advanced software for data acquisition

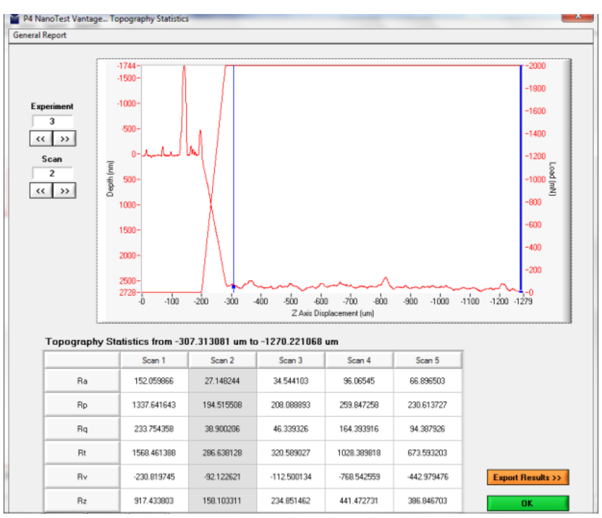
- User-defined multi-load loading history option for single scratches and multi-pass tests
- Abrasion simulation
- Reciprocating scratch and wear tests
- User-defined data densities

Advanced software for analysis

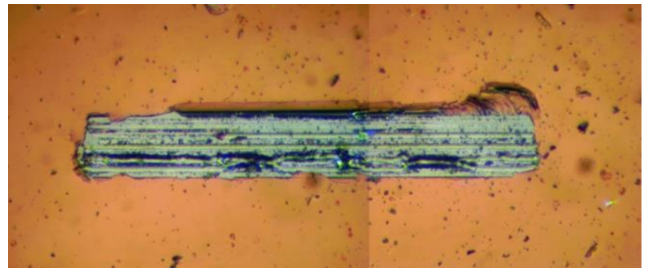
- Multi-axis composite plots
- Enhanced levelling options
- Integrated microscopy / positioning
- Automatic analysis - surface roughness statistics, depth, friction, friction coefficient in user-defined regions
- Step height calculation
- ISO/CEN 17627 test reports
- Plot-linked event logger
- TDMS and SIO export options



Complex load history



Surface roughness statistics



Abrasion simulation by 10 scratches at 100 mN

References

1. CEN/TS, 17629:2021 *Nanotechnologies — Nano- and micro-scale scratch testing* (2021).
2. BD Beake, VM Vishnyakov, R Valizadeh, JS Colligon, *Influence of mechanical properties on the nanoscratch behaviour of hard nanocomposite TiN/Si_3N_4 coatings on Si,* J Phys D: Appl Phys 39 (2006) 1392-1397.
3. BD Beake, AJ Harris, TW Liskiewicz, *Review of recent progress in nanoscratch testing,* Tribology 7 (2013) 87.

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