Scratch

MICRO
MATERIALS

MEASURING NANOTECHNOLOGY

The NANOTEST™

Bringing nanomechanical measurements into the real world

NANOTEST NANO-SCRATCH AND NANO-WEAR TESTING MODULE

NanoTest advantages

- Optimised design for robustness, reproducibility and sensitivity
- Ultra-low thermal drift – necessary for long duration nanowear tests
- Bespoke software

Use nano-scratch to

- Optimise coating deposition processes
- Understand failure mechanisms
- Obtain reliable quantified data on depth and frictional changes corresponding to each failure event

EXCELLENCE IN NANOSCALE MATERIALS TESTING

NANOTEST NANO-SCRATCH AND NANO-WEAR TESTING MODULE

INTRODUCTION

The NanoTest nano-scratch/nanowear module has been developing continuously over the past 10 years or so. Now the NanoTest is a state of the art, technology driven technique with advantages for the study of advanced coatings (see box) [1-9]. Conventional (macro-) scratch testing was designed to test the previous generation of hard and thick anti-wear coatings, so its experimental conditions (load, loading rate, probe radius etc) were optimised for testing these type of samples. They have obvious disadvantages for the testing of advanced coatings which are thinner and thin films in different applications. It was clear something was needed for studying thin coatings and the NanoTest nano-scratch module was developed to meet this need.

HOW DOES NANO-SCRATCHING COMPARE WITH TRADITIONAL SCRATCH TESTING?

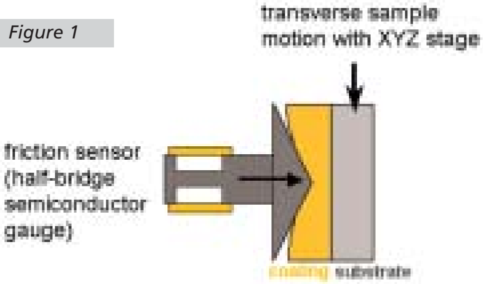
Disadvantages of macro-scratch testing

- Load is applied too fast
- Load is too high
- Probe radius is too large
- Results are sensitive to scan parameters
- Only single scratches are performed
- Results can be insensitive to adhesion

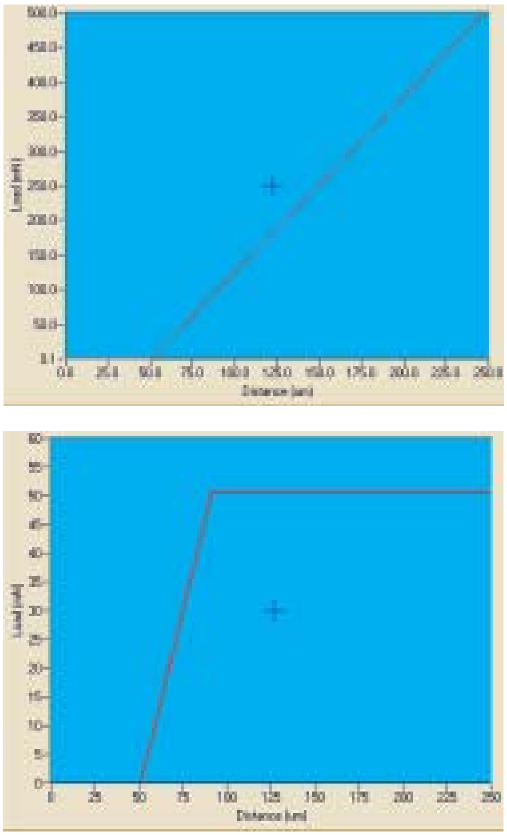
Advantages of NanoTest nanoscratch / nanowear

- Results are highly reproducible – robust yet sensitive loading head
- Results are not particularly sensitive to scan parameters
- Multi-pass wear scratches are very sensitive to subtle differences in adhesion
- Multi-pass wear for sliding/abrasive wear rates
- Three-scan procedure (with residual scan) allows identification of failure mechanisms – and the role of stress in particular - in greater detail
- Smaller probes, lower scan rates and loads, more sensitive to variations across surfaces
- Fully automated – multiple experiments on multiple samples in a single automated schedule

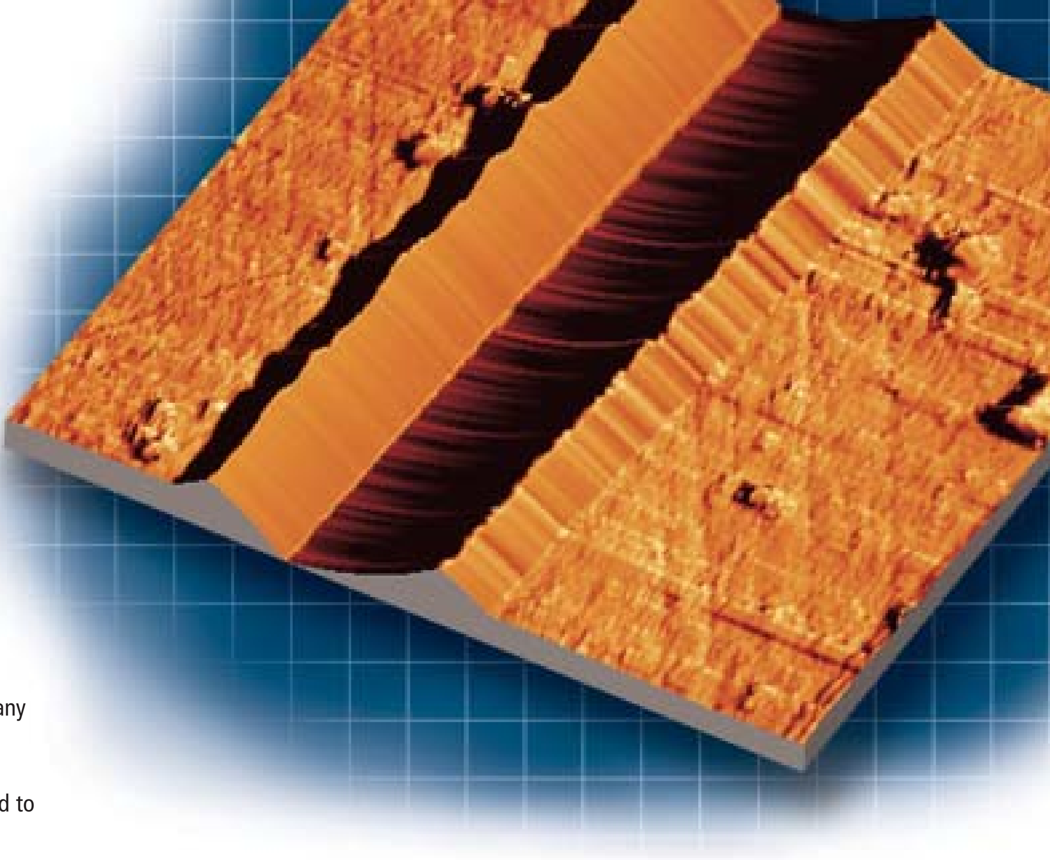
Figure 1



Principle of nano-scratch/nanowear



Illustrative load profiles for ramped and constant load scratches



NANOSCRATCH AND NANOWEAR APPLICATIONS

The nano-scratch/nanowear module has found many applications in sectors as diverse as optical, microelectronics, polymer/biomaterial, tribological coatings etc. A range of case studies are presented to illustrate some typical applications and show the potential for the technique to understand failure mechanisms and optimise coating deposition.

NANO-SCRATCHING 60 nm ta-C FILM FOR MEMS APPLICATIONS

The optimum deposition conditions necessary for improved scratch resistance [1] of tetrahedral amorphous carbon (ta-C) films produced by filtered cathodic vacuum arc (FCVA) plasma technology have been determined by nano-scratch testing. ta-C films have been developed for MEMS applications including capacitive sensors and protective coatings for micromachined components. They have a high sp^3 fraction of carbon atoms (up to 88 %) conferring high hardness, but the films can be highly stressed and their suitability for tribological applications needs to be assessed.

After the 3-scan test is complete the data is corrected for initial slope/topography and compliance in the NanoTest software. This procedure enables us to very clearly define several key transition points. These are L_{e-p} (critical load for the onset of plasticity), L_{c1} for the onset of edge cracking and L_{c2} for total film failure. These are confirmed by microscopy. Figure 2 shows an image taken with the NanoTest integrated optical microscope which confirms the excellent reproducibility of the NanoTest nano-scratch technique. The distance between L_{c1} and L_{c2} suggests this coating has adequate toughness under sliding wear conditions.

For the first time it has become possible to obtain quantitative data on changes in probe penetration occurring during different transitions. It is well known that the substrate (Silicon) undergoes contact-induced phase transformations during nanoindentation and the importance of these in nanoscratching is the subject of on-going research.

3-scan nano-scratch test of 60 nm ta-C film on Si wafer

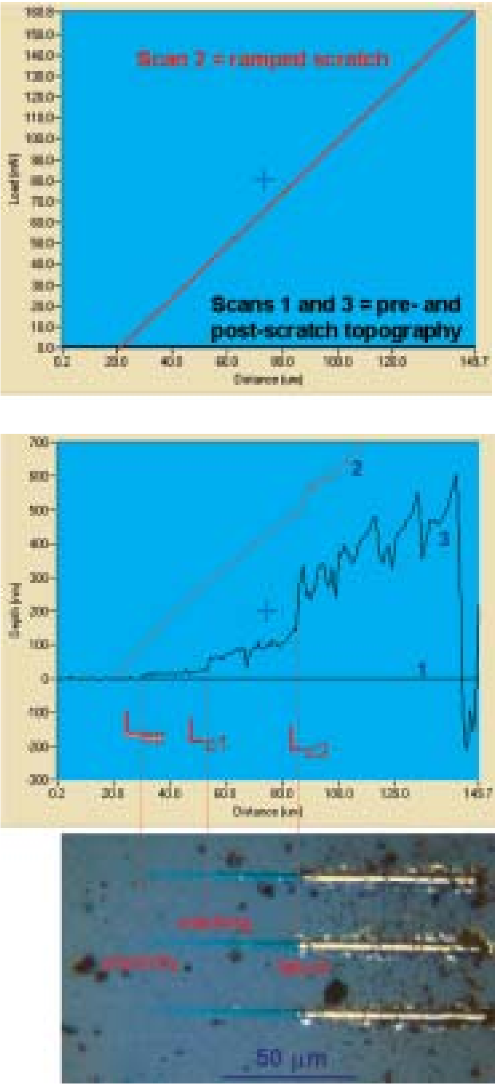


Figure 2

Experimental conditions: Three-scan scratch test with 3µm end radius probe scanning over a 150 µm track at a scan speed of 2 µm/s. In scan 2 after 20 µm the load is ramped at 2.5 mN/s. 3 repeat tests were performed to test the reproducibility of scratch behaviour. The replot shown was from the second of these.

NANOTEST NANO-SCRATCH AND NANO-WEAR TESTING MODULE

NANO-SCRATCHING OF Si-DOPED DLC FILMS

Silicon doping of DLC coatings can improve adhesion to some substrates and alter the surface wettability, although at the expense of some hardness. Scientists at Paisley University were keen to see if this altered their scratch resistance. They found differences in scratch resistance due to composition were relatively minor and the film thickness has an influence on the critical load in the nanoscratch test [2].

In principle, film thickness can have two opposing effects:- 1) thicker coatings that are harder than substrate provide more load support and delay the onset of the substrate deformation that is often the precursor of film failure (higher critical load) 2) thicker coatings can be more highly stressed and more easily through-thickness crack and delaminate when deformed (lower critical load).

In the case of the Si-doped films, higher critical loads are due to the greater load-carrying capability of the thicker films. Film failure is abrupt and easy to determine by any of (1) on-load scratch depth (2) residual depth (3) friction force (4) microscopy. In figure 3 the 366 nm film had a critical load (for total film failure) of 16.5 mN and the 614 nm a critical load of 34.3 mN.

As the DLC-coated glass shows clear film failures in nanoscratch tests, it is an ideal choice of coating system to use to investigate the possible influence of various experimental parameters on the measured critical load in the nanoscratch test. Encouragingly, no dependence on critical load was found on either (1) scratch speed, (2) loading rate, or (3) increase in load per unit scratch distance (dL/dx) when dL/dx is much less than 1 N/mm.

This suggests that tests with the NanoTest nanoscratch module - under widely different loading conditions - can be compared directly [2]. This shows the versatility and robustness of the NanoTest nano-scratch technique. This is in contrast to conventional macro-scale scratch testing where dL/dx is set to 10 N/mm and results are strongly dependent on both loading rate and scan speed.

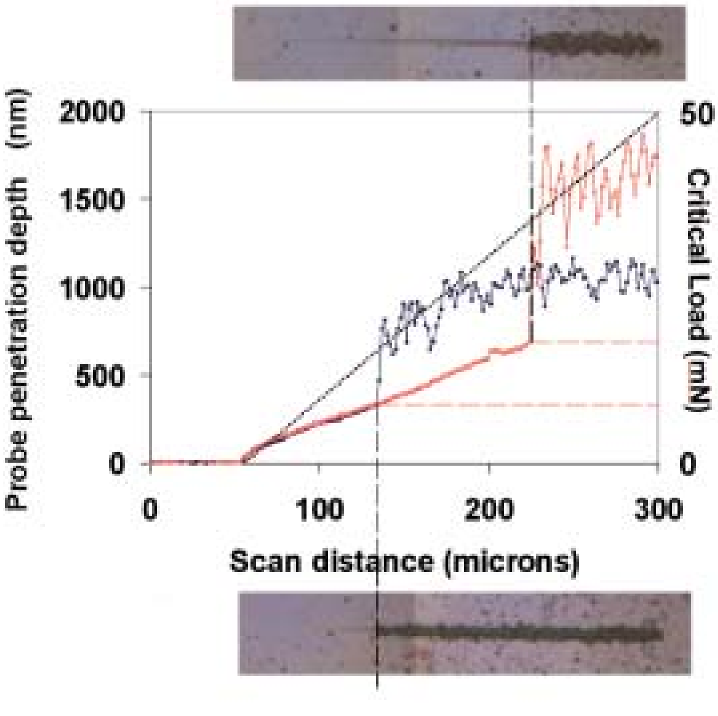
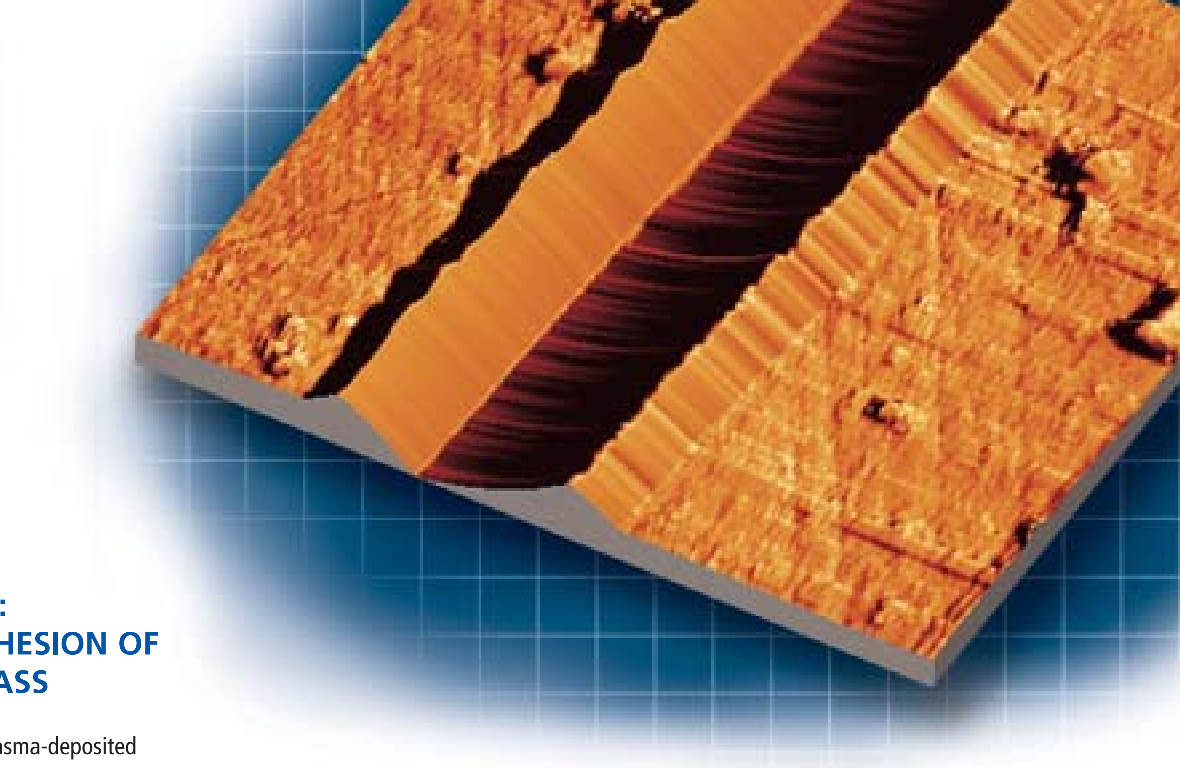


Figure 3

On-load scratch test traces together with accompanying optical micrographs from tests at 1 mN/s on Si-containing a-C:H films. Film failure is clearly seen as an increase in scratch depth and subsequent oscillations in the scratch trace as the probe encounters broken film. Microscopy confirms total film failure. Upper micrograph = open triangles = 20 sccm TMS film (614 nm); Lower micrograph = closed squares = 10 sccm TMS (366 nm).



NANOWEAR TESTING: EVALUATING THE ADHESION OF THIN Ti FILMS ON GLASS

The adhesion strength of pulsed-plasma-deposited titanium films is key to their durability and scratch resistance. In this study processing parameters were found to have a significant influence on their hardness and a dramatic influence on their adhesion, with films deposited under higher deposition pressure exhibiting weaker adhesion.

In the results below both Ti coatings were 1 µm thick, deposited on glass with minimal (~0.4%) oxygen inclusion and almost identical hardness (~6 GPa) and elastic modulus (~140 GPa). Critical loads in a ramped scratch test using a 3 µm radius probe were also rather similar (~175 mN).

Constant load multi-pass nano-wear testing was used to probe differences in adhesion. A constant load of 50 mN (30% of the critical load in a single ramped scratch) was chosen and 10 constant load wear cycles performed on both coatings.

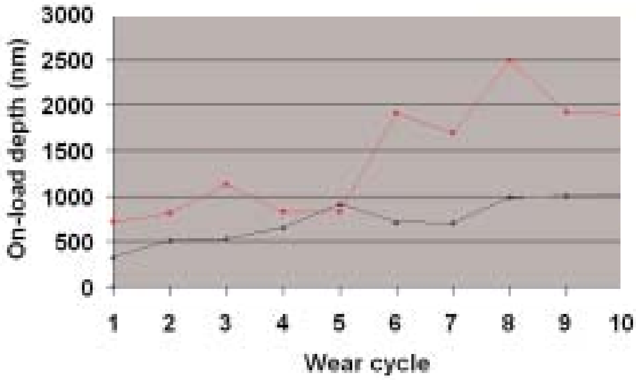
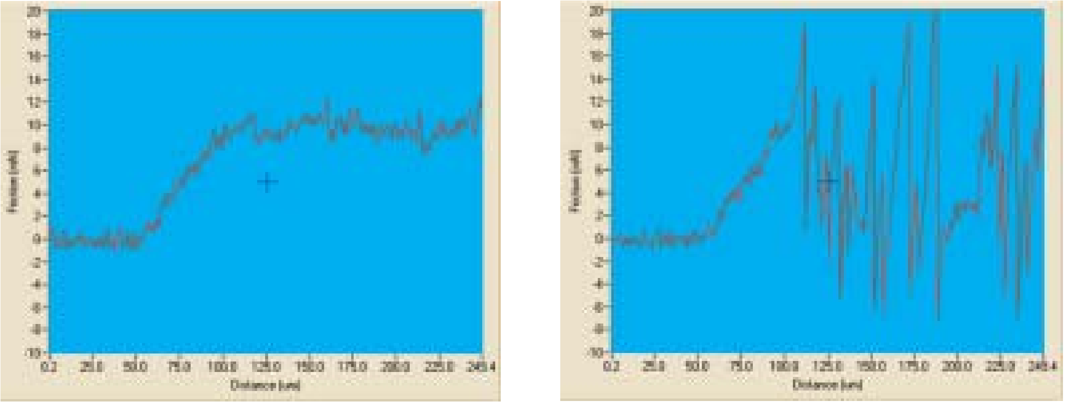


Figure 4

Constant load nano-wear testing procedure was found to more clearly show this difference in adhesion than single ramped scratches.



Friction traces on poorly adhering sample – wear scan 5 (left) and 6 (right)

Despite their virtually identical hardness and elastic modulus the nanowear test clearly reveals differences in adhesion between the two Ti coatings. The on-load depth and friction signals were used to determine the number of cycles to failure. The sample (red) exhibits poor adhesion failing after 6 cycles – as shown by the abrupt change in depth and the onset of oscillations in friction trace - whilst the other (blue) has better adhesion strength not failing within the duration of the test.

Researchers at Manchester Metropolitan University use the nano-scratch and nano-wear facility to optimise their advanced Ti coatings for improved adhesion and durability. In the example above the use of AC rather than DC pulsing during deposition was an important factor in improving the adhesion of the Ti film.

NANOTEST NANO-SCRATCH AND NANO-WEAR TESTING MODULE

INFLUENCE OF MECHANICAL PROPERTIES ON THE NANOSCRATCH BEHAVIOUR OF HARD NANOCOMPOSITE TiN/Si_3N_4 COATINGS ON Si

A link between mechanical properties and tribological performance is now well established, hence the popularity of combining nanoindentation and nano-scratch characterisations. In collaboration with scientists at Manchester Metropolitan University we have undertaken a systematic study of the interrelationships using nanocomposite TiN/Si_3N_4 coatings on silicon substrate as test samples. These coatings have attracted much interest recently due to the possibility to make them "super-hard".

In this work however, the aim was not to specifically produce super-hard coatings rather to produce coatings with an optimum combination of hardness and toughness in tribological situations.

Their mechanical properties were determined by nanoindentation and their tribological properties were measured by nanoscratch testing [3]. The nanoindentation showed that harder nanocomposites exhibited higher ratios of hardness to modulus (H/E) and that the H/E value clearly influences the nano-scratch behaviour as shown below.

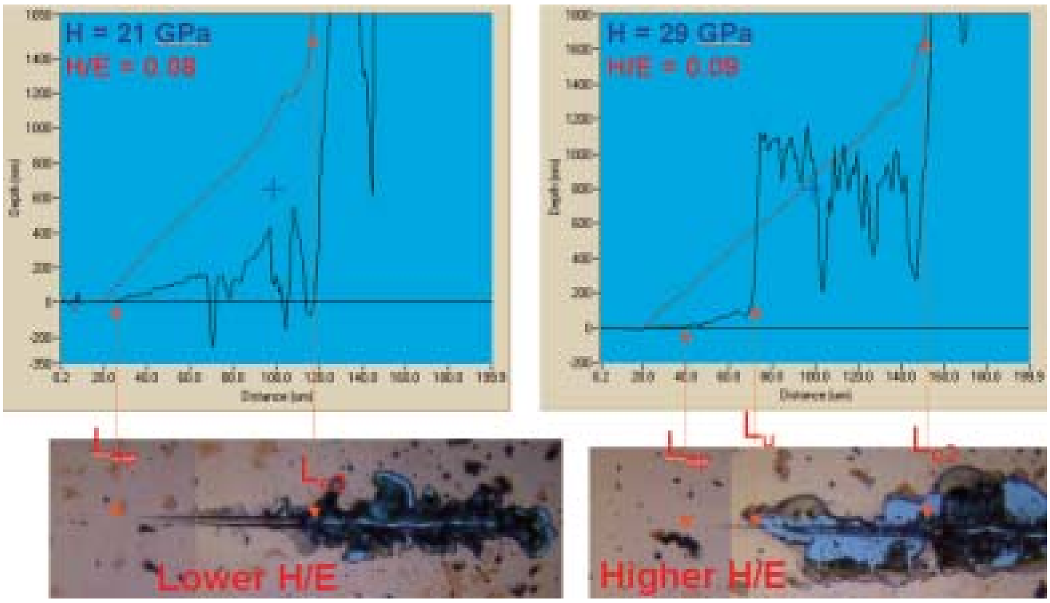


Figure 5 *nano-scratch tests on Ti-Si-N nanocomposite coatings and microscopic images of scratch tracks taken with the integrated NanoTest high resolution microscope.*

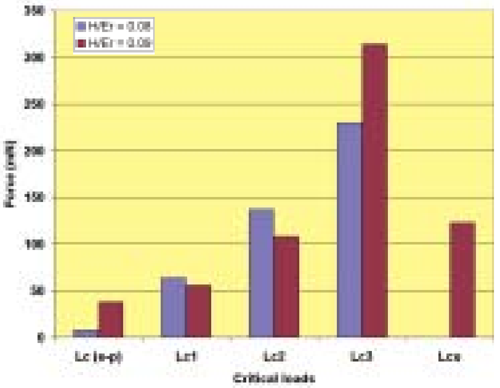


Figure 6

Coatings with higher H/E values showed higher critical loads for elastic-plastic transition and also the total coating failure occurring in front of the probe. However, coatings with higher H/E also exhibited an unloading failure, occurring behind the probe at much lower load than the loading failure. Optimising coating deposition conditions to avoid this stress-related unloading failure is critical for tribological applications.

ADVANTAGES OF NANOTEST NANOSCRATCH / NANOWEAR

- **Results are highly reproducible – robust yet sensitive loading head**
- **Results are not particularly sensitive to scan parameters**
- **Multi-pass wear scratches are very sensitive to subtle differences in adhesion**
- **Multi-pass wear for sliding/abrasive wear rates**
- **Three-scan procedure (with residual scan) allows identification of failure mechanisms in greater detail**
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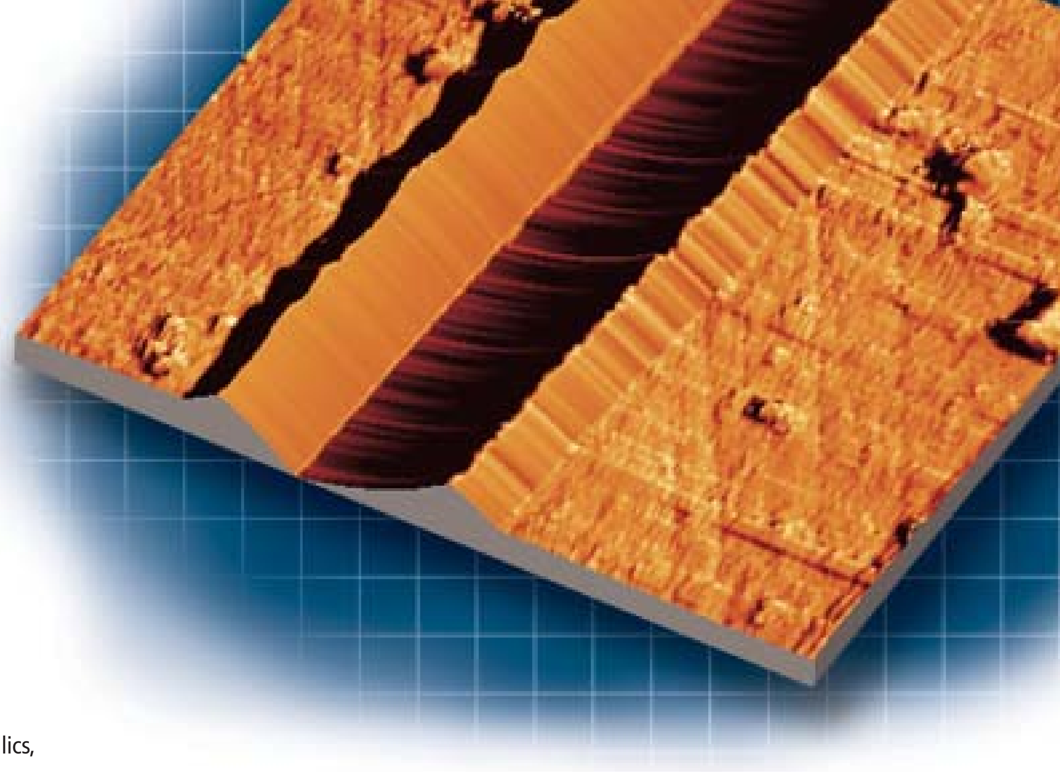


NANOSCRATCH TESTING OF THERMAL OXIDE ON FeAl ALLOY

Iron aluminides, like other lightweight intermetallics, are being investigated for use in the automotive industry. For applications involving tribo-contact – such as valves in IC engines – abrasion resistance could be a crucial factor in determining the life and performance of FeAl parts.

Researchers at Birmingham University have used both the constant load and ramped nanoscratch capabilities of the NanoTest to evaluate the effectiveness of surface treatments in improving their scratch and wear resistance [4]. The surface treatment involved thermal oxidation at 1000˚C for 30-150 hr. The critical load for brittle fracture (buckling and chipping) of the oxide layer could be increased by lengthening the oxidation time.

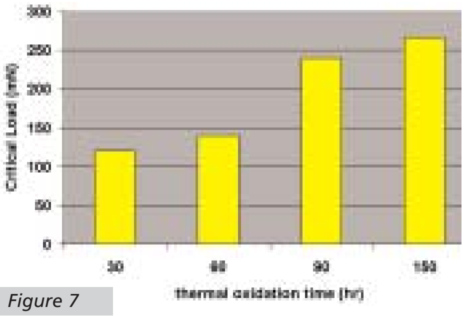
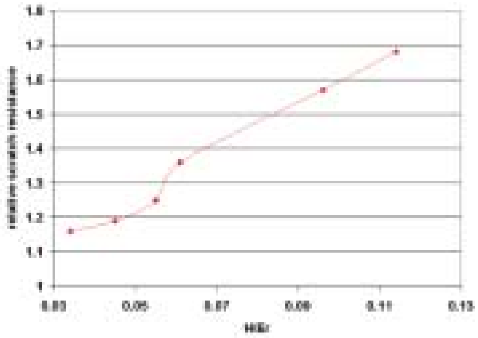


Figure 7

Variation in critical load with time of oxidation at 1000˚C

The Birmingham group also confirmed the expected interrelation between tribological and mechanical properties; finding that scratch rates (for removal of material) followed a linear relationship with (Load/H), and that relative scratch resistance scaled with the dimensionless (H/E_r) parameter.

Figure 8



Nanoindentation and nano-scratch testing showed that by increasing thermal oxidation time they could improve the abrasion resistance of the FeAl alloys. Surface treatment by the thermal oxidation route developed should extend the applicability of FeAl alloys to harsher tribo-contact conditions.

MICROSCRATCH TESTING OF TiB_2 COATINGS ON HIGH SPEED STEEL

Microscratch testing is performed with the NanoTest high load head (MicroTest) using a 25 µm probe. It is recommended for hard PVD and CVD coatings (typically1-5 µm thick). Micro-wear tests can also provide useful information [5]. The microscratch test can be used in conjunction with the NanoTest hot stage [6].

Researchers at Singapore's Nanyang Technological University have used the multi-pass microscratch technique – in conjunction with nanoindentation - to improve the properties of sub-micron nanostructured titanium diboride coatings on high speed steel, using the friction force to determine critical loads [7].

They found that coatings prepared without sputter-cleaning or substrate bias showed poor adhesion and failed by compressive spallation. However, with substrate bias the critical load increased and the failure mechanism changed to wedge spallation as the adhesion was strong enough to bear the stress (figure 7). By combining sputter pre-cleaning and substrate bias they were able to improve these properties yet further.

Figure 9

The scratch direction is from left to right in these images of microscratch traces. The use of substrate bias during deposition (lower scratch track) increased the critical load and altered the failure mode

NANOTEST NANO-SCRATCH AND NANO-WEAR TESTING MODULE

SPECIFICATIONS

The scratch and wear module can be used with the NanoTest low load head (nanoscratch/nanotribology) and/or NanoTest high load head (microscratch)

Easy and quick probe exchange (~ 1 min) – fully modular with nanoindentation and nano-impact modules.

Robust - no danger of damage to loading head springs during scratching.

No recalibration necessary on switching between nanoindentation and nanoscratch modules.

Choice of spheroconical diamond scratch probes with end radii 0.7-200 µm. Recommended radii are 5-10 µm for nano-scratching and 25 µm for micro-scratch.

The NanoTest low thermal drift allows multi-pass wear tests.

Bespoke software correction to instrument compliance and sample slope to pin-point transitions in depth traces.

Wide range of robust friction probes with different force constant available.

TYPES OF TEST POSSIBLE

Ramped scratch
3-pass multi-pass (2nd scan is ramp)
Multi-pass constant load wear test
Topography scan

MEASUREMENTS

On-load and off-load (residual) depth
Critical loads
No. of cycles to failure
Surface roughness (Ra, RMS, peak-to-valley, etc)
Friction (tangential force)
Acoustic Emission (optional extra)
Microscopy (optional extra)

ACKNOWLEDGEMENTS

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