Impact

MICRO
MATERIALS
MEASURING NANOTECHNOLOGY

The NanoTest™

Bringing nanomechanical measurements into the real world

NANO-IMPACT AND NANO-FATIGUE TESTING

The NanoTest advantage for nanoscale impact and fatigue testing

- Reliable accelerated wear testing
- When toughness is more important than hardness
- When nanoindentation alone is insufficient
- Excellent correlation with cutting tool milling tests
- Clear failure identification
- Piezo sample oscillation impact for high cycle fatigue testing
- Impulse impact for low cycle fatigue testing
- Single impacts for work hardening, dynamic hardness and yield stress
- Rapid, automated determination of S-N curves

NANO-IMPACT AND NANO-FATIGUE TESTING

INTRODUCTION

Since the introduction of its nano-impact module almost 10 years ago the Micro Materials NanoTest has become the clear world leader in nano-impact testing [1-11].

A limitation of conventional nanoindentation testing is that the loading rate is too low (quasi-static indentation is another popular name for nanoindentation) to simulate the actual deformation rates occurring in a material's operation or production. For many applications what product designers really need is reliable high strain rate data rather than quasi-static data.

Realising this, Micro Materials have developed a fast indentation technique, nano-impact, to probe properties at high strain rates and investigate surface fatigue and fracture due to repetitive contact (analogously to a woodpecker pecking at a tree). This enables the simulation of the small repetitive stresses many modern materials suffer in real-life over time and allows more accurate prediction of their behaviour and the design of better materials.

Micro Materials have been granted a European Patent (No. 1095254) for the nano-impact technique.

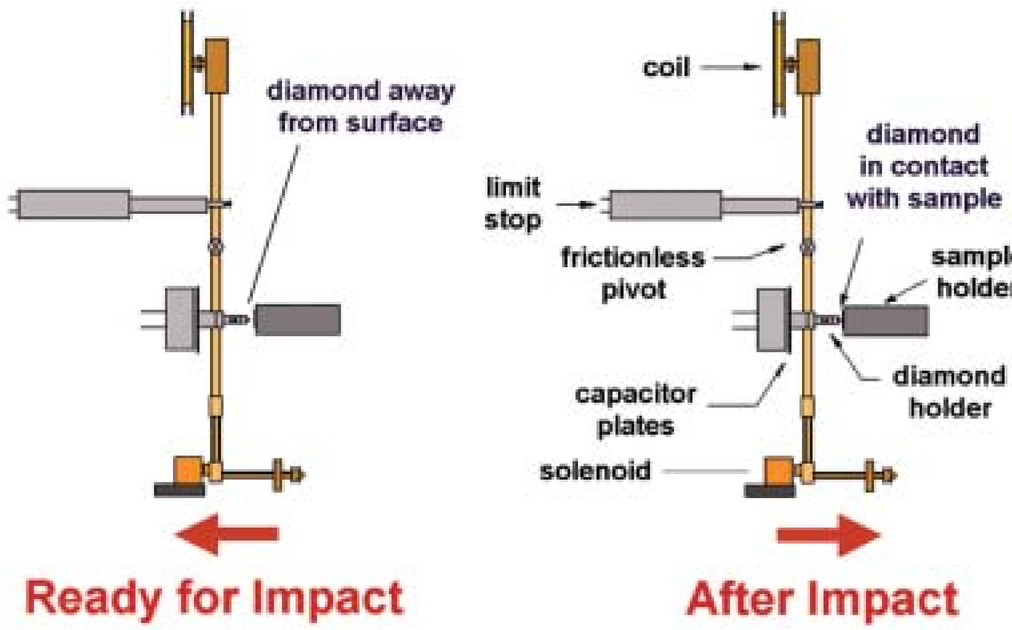
Materials often show differences in mechanical behaviour at high and low strain rates. The NanoTest is unique amongst indentation systems in having the (patent protected) ability to produce ultra-fast, high strain rate indentations.

It can be used to study material behaviour at strain rates (up to 300 s^{-1} or more) far in excess of those on any other commercial nanoindentation instrument.

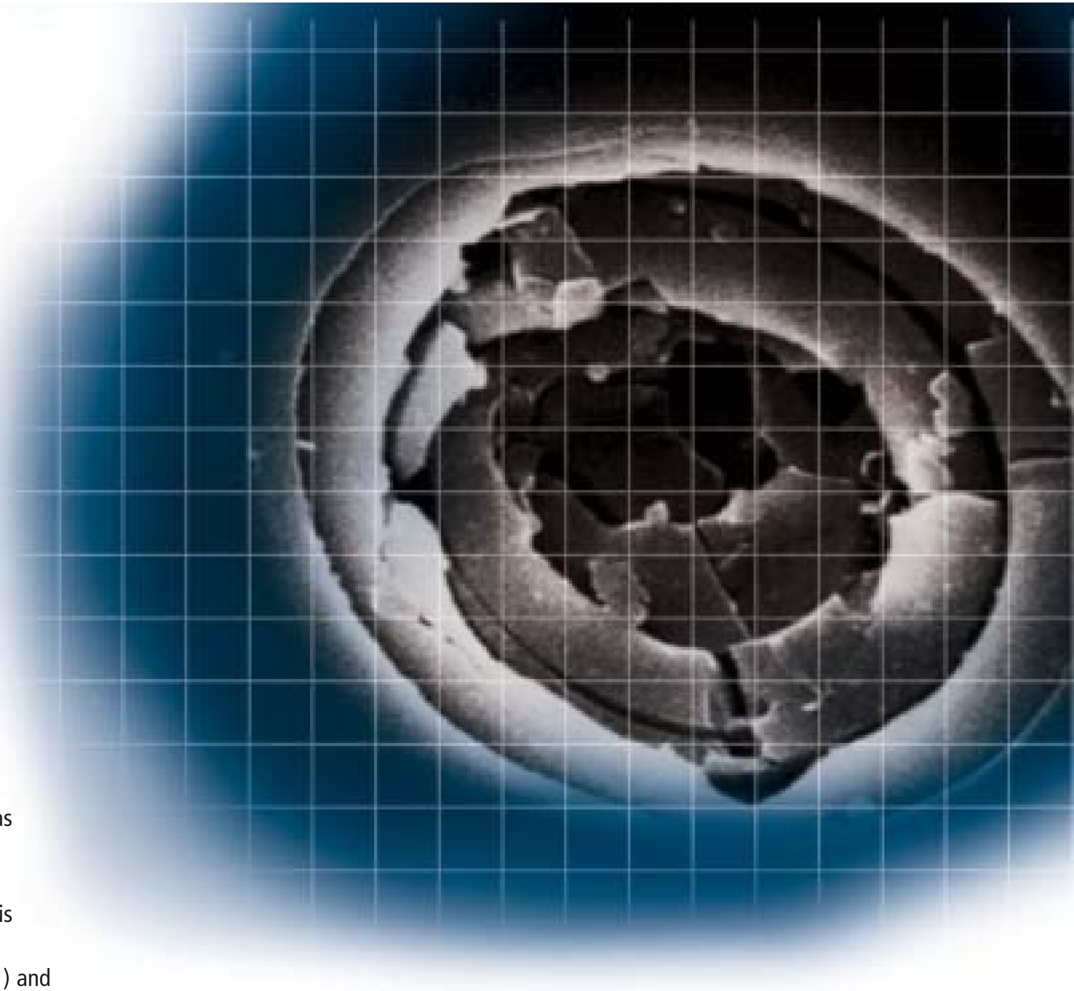
THE NANOTEST ADVANTAGE FOR NANOSCALE IMPACT AND FATIGUE TESTING

- Improve lifetime of coated cutting tools
- DLC-coated engine components
- Durability of biomedical devices
- Dynamic hardness
- MEMS
- Nanocomposites
- Simulation of erosive wear
- Fracture properties
- Strain rate sensitive behaviour of solder joints

Figure 1



Impulse impact mode for single and repetitive impacts.



Ring cracking in alumina caused by repetitive nano-impact with 25 µm spherical probe

HOW DOES IT WORK?

To provide both high cycle and low cycle fatigue testing capability two distinct impact/ fatigue modes – (1) Piezo Sample Oscillation Impact and (2) Impulse Impact - are included as standard in the impact module.

The NanoTest pendulum design is critical as this provides the space and geometry necessary to produce large amplitude oscillations in mode 1) and to use powerful solenoid actuation in configuration 2). Micro Materials have been granted a European Patent (No. 1095254) for the nano-impact technique. Solenoid actuation enables the probe to be accelerated over a precisely known distance and hence the energy delivered to be determined. The NanoTest (with the swinging pendulum analogous to conventional macro-scale impact tests) can accelerate the indenter fast enough to produce true high strain rate impacts (300 s^{-1} is possible with the NT in impact).

High cycle fatigue tests are performed by a piezo sample oscillation impact approach with oscillation frequencies up to 500 Hz and amplitudes up to 5 µm. Impulse impact is a popular nano-impact testing approach for investigating low cycling fatigue, work hardening, yield stress and dynamic hardness. In impulse impact a powerful solenoid is used to pull the test probe off the surface then accelerate it from a distance of typically 10 µm from the surface to produce each impact. Automation enables repetitive impacts at the same position on the sample surface with a set frequency.

The nano-impact test records the evolution of impact-induced damage with time on repetitive impact of a test probe that can be made of any material and any geometry. Small radius spheroconical probes and high contact strain cube corner indenters are used to induce fracture rapidly and accelerate the test. A typical test duration is 5 min.

Diagnostic methods for determining the time-to-fracture include (1) rapid changes in probe depth accompanying material removal (2) differences in energy absorption.

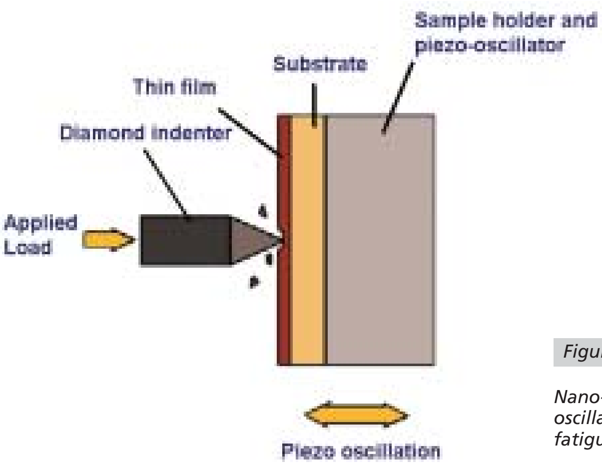


Figure 2

Nano-impact piezo sample oscillation mode for high cycle fatigue

NANO-IMPACT AND NANO-FATIGUE TESTING

THE CUTTING EDGE

PVD coatings such as TiAlN and AlCrN are often used as cutting tools, with their good oxidation resistance and high hot hardness. In collaboration with scientists at McMaster University in Canada, Polytech Tours in France and Balzers AG in Leichtenstein, the NanoTest nano-impact module has been used to measure the nanomechanical properties of these and other coatings at high strain rates and also elevated temperatures [1-2].

Conventional nanoindentation is quasi-static and it is not too surprising that it has proved difficult to optimise cutting tool coatings based on nanoindentation results. In complete contrast results with the nano-impact technique show excellent correlation with tool life-time in highly loaded interrupted cutting applications such as the end and face milling of steels and Ti alloys – but with a typical test time of only 5 minutes are much quicker!

THE NANOTEST ADVANTAGE FOR NANOSCALE IMPACT AND FATIGUE TESTING

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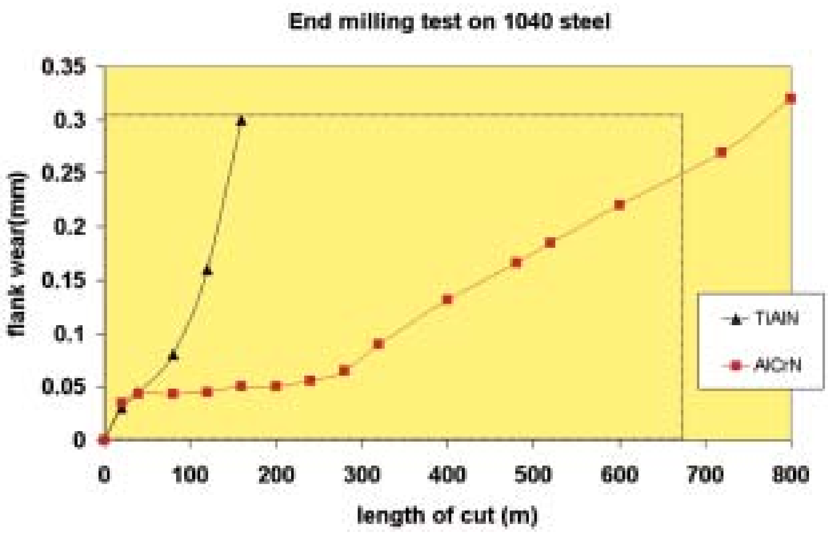


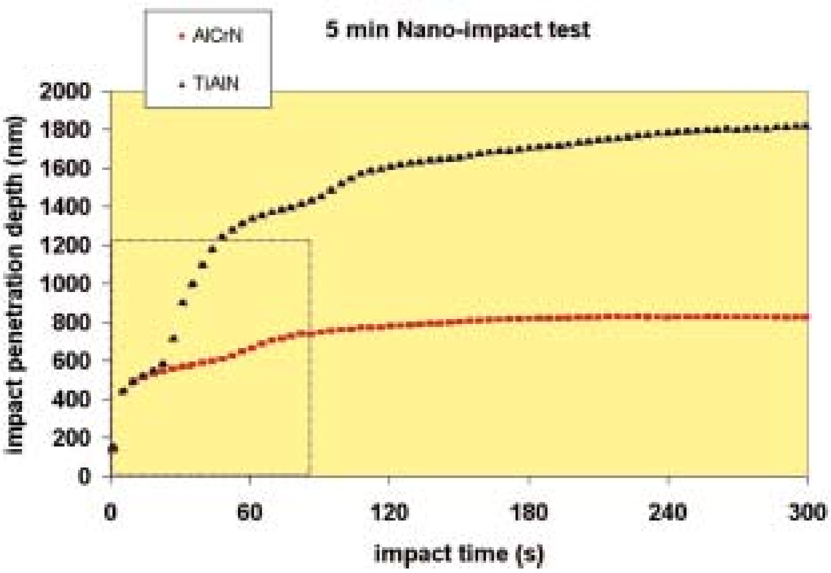
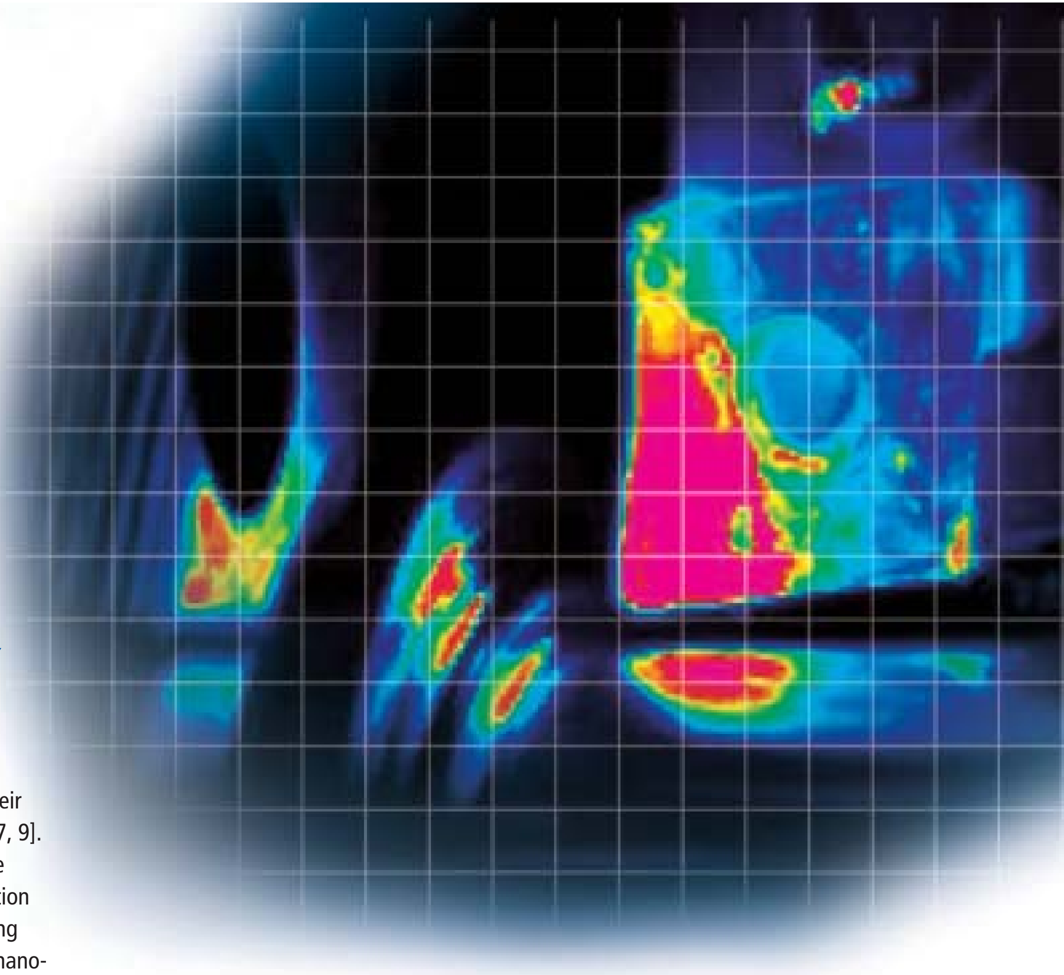


Figure 3

An excellent correlation between the rapid nano-impact test and the end milling trials on structural steel is observed. The non-constant tool wear rate is well reproduced in the accelerated test (compare highlighted regions).



IMPROVING THE DURABILITY OF DIAMOND LIKE CARBON COATINGS

DLC films can be stressed and brittle limiting their suitability for some demanding applications [5-7, 9]. Nevertheless DLC has a wide range of attractive properties including high hardness and low friction so possible applications for this coating are being continually investigated. Clear results with the nano-impact technique are driving the development of advanced DLC coatings.

Nano-impact is both an R&D and QC tool for both manufacturers and end-users of DLC coatings ensuring coating integrity and minimising costly failures. Debonding and dramatic film failure can occur at high force after only a few impacts on some DLCs (see figure 4). Low resistance to fatigue-induced fracture occurs when the coating is too highly stressed and/or there is poor adhesion.

In industry the use of rapid nano-impact tests is preferred to a complete engine test when evaluating new candidate coatings. For example, a major automotive parts manufacturer has been able to optimise their graded DLC coatings to reduce scuffing wear in fuel injector plungers.

High temperature nano-impact tests simulate cutting tool operation. Test results can be used to optimise coatings for demanding applications.

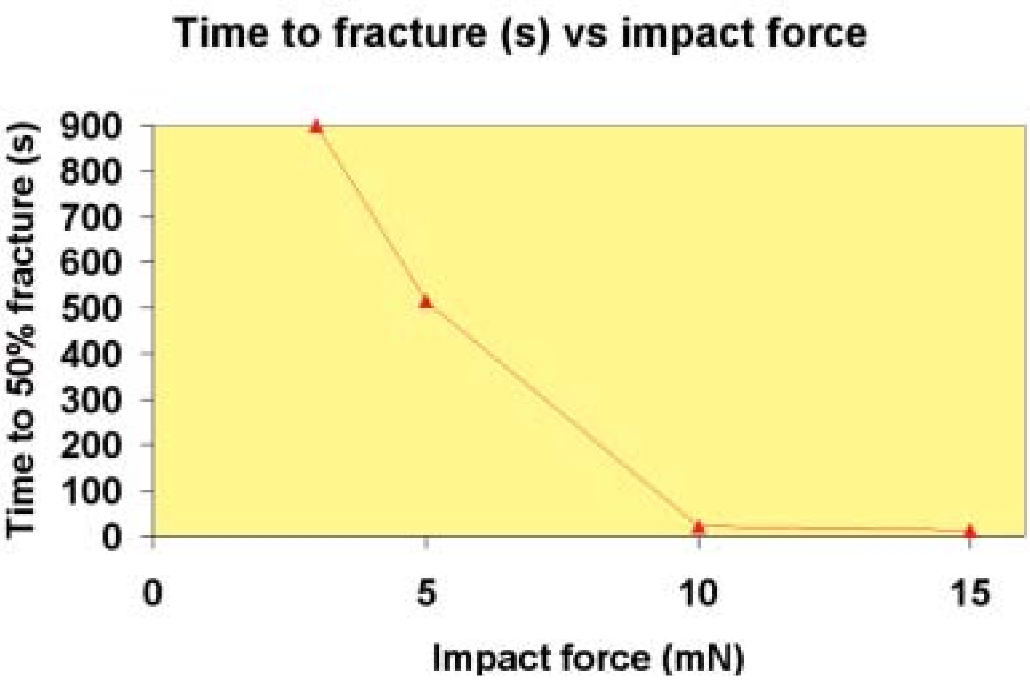


Figure 4

Nano-impact test on DLC-coated engine component. At or below 5 mN cohesive fracture occurs but at higher load there is a more damaging failure mechanism. Fracture over 5 mN is accompanied by coating delamination exposing the softer substrate.

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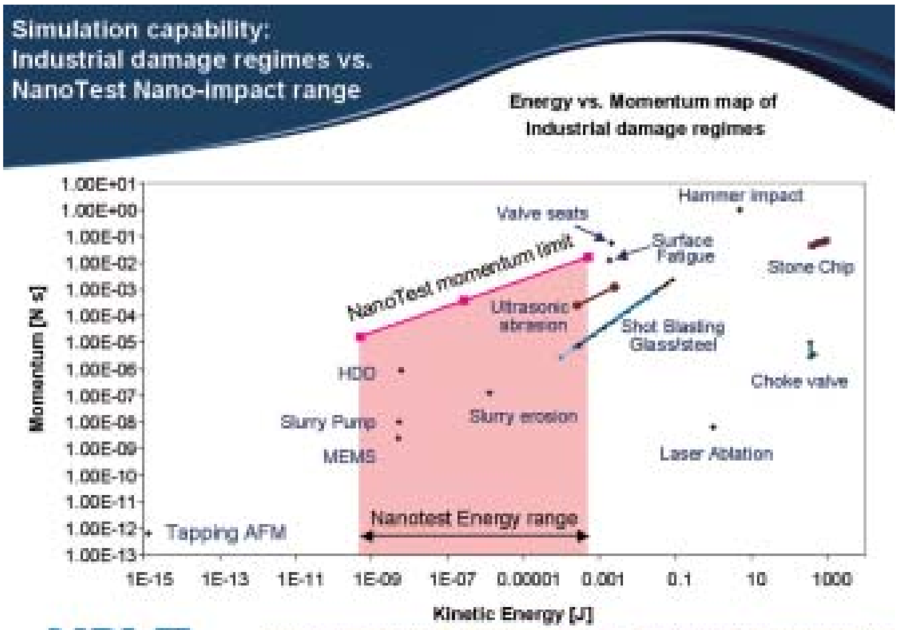
Figure 5

SIMULATION CAPABILITY

The ability of the nano-impact module to simulate wear conditions extends beyond cutting tools and coatings as the graphic below illustrates.



NPL

Original graphic by Dr Nigel Jennett, NPL. Reproduced with the permission of the Controller of HMSO and the Queen's Printer for Scotland.

The National Physical Laboratory use the nano-impact technique to probe the mechanical behaviour of materials at high strain rate and investigate a wide range of phenomena such as fracture toughness, yield stress, dynamic hardness, work-hardening and fatigue of tribological coatings.

TESTING INDENTATION BEHAVIOUR AT HIGH STRAIN RATE: DYNAMIC HARDNESS

Materials show differences in mechanical behaviour at high and low strain rates. The NanoTest is unique amongst indentation systems in having the (patent protected) ability to produce ultra-fast, high strain rate indentations and can be used to study material behaviour at strain rates far in excess of those on any other instrument.

This is possible due to the pendulum geometry that enables the probe to be accelerated to produce controlled high-energy impacts in a fraction of a second at the same location. With the aid of a fast DAQ system (a data acquisition rate of up to 500000 Hz is possible with the new NanoTest NTX Controller) all the probe displacement-time data is captured and can be analysed to produce dynamic hardness and viscoelastic property information.

Dynamic hardness is defined (after Tabor) as energy per unit volume and has units of pressure just as conventional hardness. Similarly, with the high data acquisition rate the parameter V(out)/V(in) (Leeb hardness = 1000 x V(out)/V(in) can be converted to HV and GPa) can also be obtained. The National Physical Laboratory (Teddington, UK) have found that this is a sensitive parameter which pin-points fracture events and time-to-failure on brittle materials.

Unlike the brittle materials that fracture during the nano-impact test, impact tests on polymers and biomaterials can reveal sensitive viscoelastic property information. For example the nano-impact (high-strain indentation) behaviour of commercial low-density polyethylene [LDPE], polycarbonate [PC] and polytetrafluoroethylene [PTFE] polymers is shown in the figure opposite. The probe (a diamond indenter in this case) bounces on the surface of all three polymers before the energy is dissipated but there are clear differences in how this occurs. PC shows essentially elastic behaviour, LDPE shows rubber-like behaviour and PTFE damps out the impact energy very effectively.

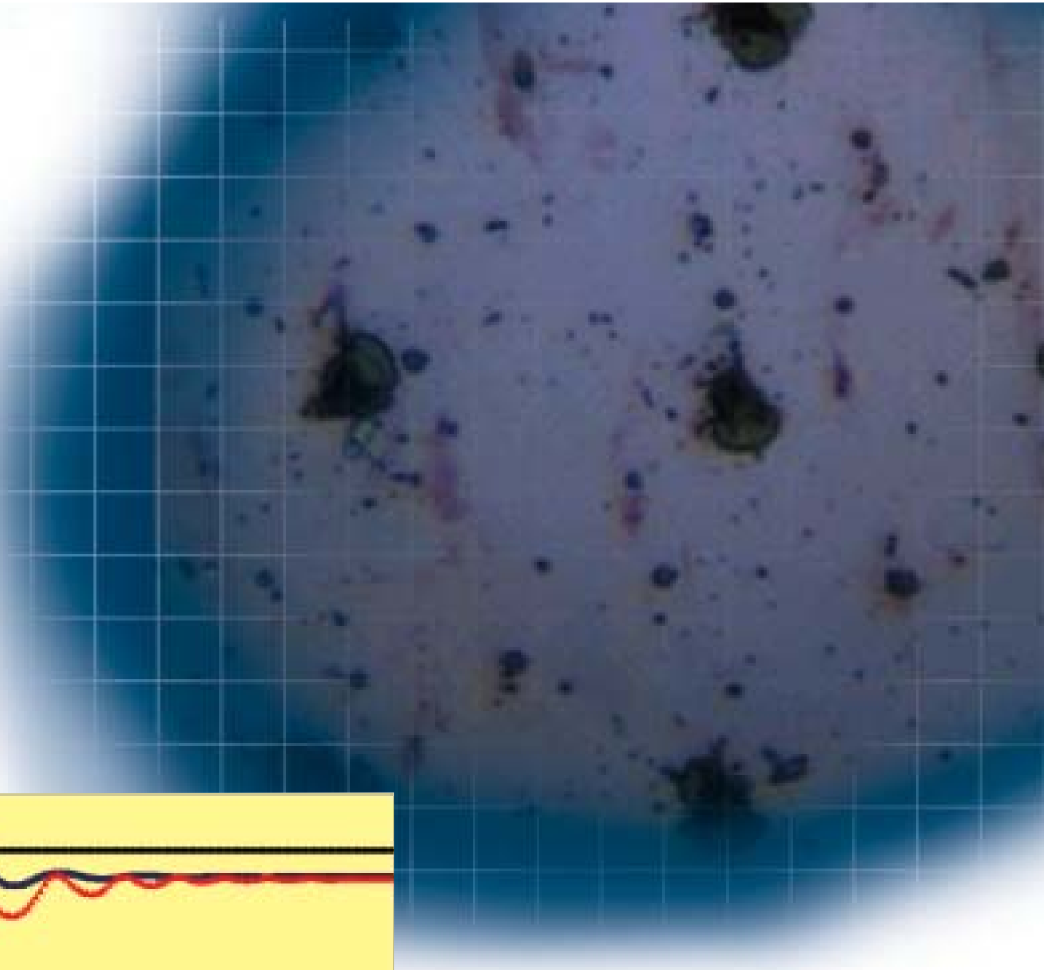
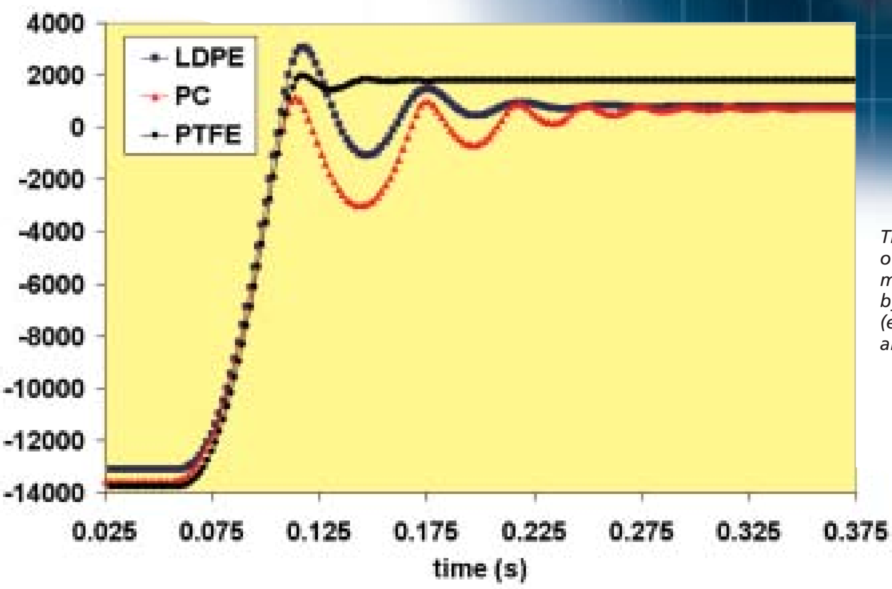


Figure 6



The damping ability of the PTFE material is shown by the lack of recoil (energy absorption).

The full scheduling capability of the NanoTest is maintained - automated arrays of impact tests can be scheduled

PATENTS AWARDED

Micro Materials have been granted a European Patent (No. 1095254) for the nano-impact technique.

SPECIFICATIONS

The nano-impact module includes two distinct impact testing modes as standard:-

Nano-impact piezo sample oscillation mode for high cycle fatigue:

- Piezoelectric oscillation system, signal generator, amplifier and software for control and data analysis.
- Enables both impact and contact fatigue tests to be performed depending on the magnitude of the static load.
- Frequency range 1-500 Hz.
- Amplitude 5 µm.

Pendulum impulse impact mode for low cycle fatigue:

- Pendulum impulse using A/C solenoid actuation to produce very high strain rate indentations (nano-impacts).
- Single and repetitive impacts can be performed.
- Dynamic hardness is determined from analysis of single impacts and fatigue behaviour from multiple impacts.
- Applied load, impact angle, acceleration distance, indenter geometry are user-definable.
- Both modes can be used on the MicroTest high load head.
- Any indenter geometry or material can be used.
- A cube corner diamond indenter is included with the nano-impact module.
- A 10 µm spheroconical probe is preferred for impact with the MicroTest high load head.

NANO-IMPACT AND NANO-FATIGUE TESTING

REFERENCES

1. *Investigating the correlation between nano-impact fracture resistance and hardness/modulus ratio from nanoindentation at 25-500°C and the fracture resistance and lifetime of cutting tools with $Ti_{1-x}Al_xN$(x=0.5 and 0.67) PVD coatings in milling operations* BD Beake, GS Fox-Rabinovich et al, Surf Coat Technol 201 (2007) in 4585.

2. *Impact of mechanical properties measured at room and elevated temperatures on wear resistance of cutting tools with TiAlN and AlCrN coatings* GS Fox-Rabinovich et al, Surf Coat Technol 200 (2006) 5738.

3. *Nanomechanical quantification of polymer energy absorption* CA Tweedie, JF Smith and KJ Van Vliet, Mat Res Soc Symp Proc 841 (2005) R5.6

4. *An investigation of the nanoindentation and nano/micro-tribological behaviour of monolayer, bilayer and trilayer coatings on cemented carbide* BD Beake and N Ranganathan, Mater Sci Eng A 423 (2006) 46.

5. *Evaluation of the fracture resistance of DLC coatings on tool steel under dynamic loading* BD Beake Surf Coat Technol 198 (2005) 90.

6. *Nano-impact testing – an effective tool for assessing the resistance of advanced wear-resistant coatings to fatigue failure and delamination* BD Beake and JF Smith Surf Coat Technol 188-189C (2004) 594.

7. *Evaluating the fracture properties and fatigue wear of tetrahedral amorphous carbon films on silicon by nano-impact testing* SP Lau et al, Surf Coat Technol 177-178 (2004) 611.

8. *Nanoscale repetitive impact testing of polymer films* F Gao et al, J Mater Res 19 (2004) 237.

9. *Investigating the fracture resistance and adhesion of DLC films with micro-impact testing* CA Rego et al, Diam Relat Mater 11 (2002) 1606.

10. *Simulation of erosive wear of boron carbide coatings by static and repetitive nanoindentation* K Bose and RJK Wood, Proc 2nd World Tribology Congress, Vienna, 2001, pp575-578.

11. *Micro-impact testing: a new technique for evaluating fracture toughness, adhesion, erosive wear resistance and dynamic hardness,* SR Goodes et al, Surface Engineering 17 (2001) 187.

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