High T

MICRO
MATERIALS
MEASURING NANOTECHNOLOGY

The NanoTest™

Bringing nanomechanical measurements into the real world

ELEVATED TEMPERATURE NANOINDENTATION TESTING

The NanoTest advantage for high temperature testing

- Ultra-low thermal drift at elevated temperature
- Depth sensing nanoindentation up to 750°C
- Stability for nanoscale creep testing
- Active control of sample and probe temperature

ELEVATED TEMPERATURE NANOINDENTATION TESTING

INTRODUCTION

The Micro Materials NanoTest has become the clear world leader in depth sensing nano- and micro-indentation testing at elevated temperature [1-9]. The horizontal loading design of the NanoTest is critical for accurate and reliable testing at elevated temperatures.

The NanoTest hot stage controller utilises separate heating (and active temperature control) of both probe and sample to ensure no heat flow occurs during the indentation process.

The NanoTest is unique in this isothermal contact for elevated temperature testing. As no significant thermal drift occurs during elevated temperature measurements it becomes possible to perform long-duration tests – such as indentation creep tests – at elevated temperatures aswell as normal nanoindentation tests up to 750ºC.

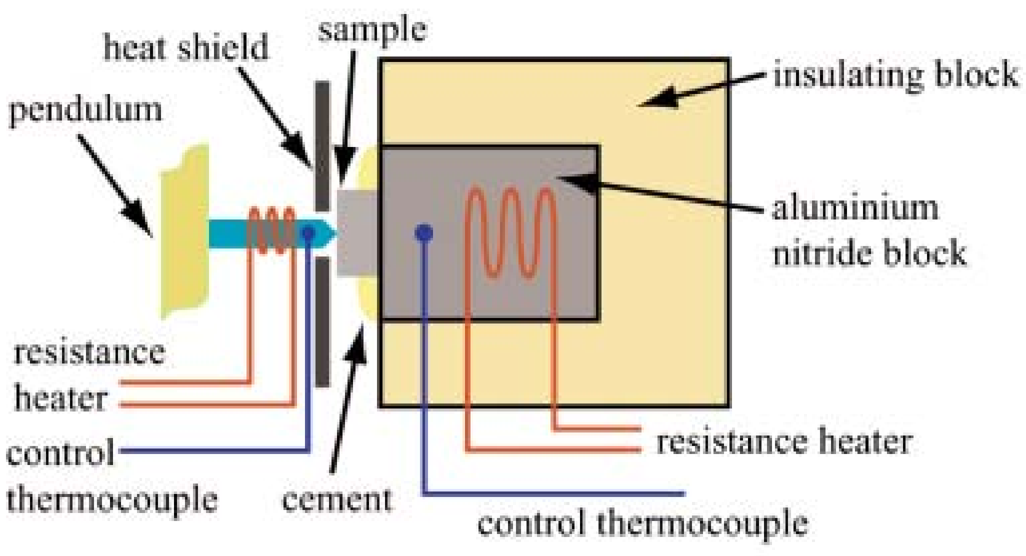
Leading-edge researchers at the University of Cambridge, MIT, General Motors, Loughborough University, SimTech, Cranfield University, PolyTech Tours, KAIST, Northwestern University, and many others worldwide, use the NanoTest hot stage and control system to probe material behaviour well beyond the capability of any other commercial nanomechanical test instruments.

As it becomes possible to accurately measure material properties and performance at high temperatures product developments will speed up accordingly. No longer will the actual testing be an afterthought.

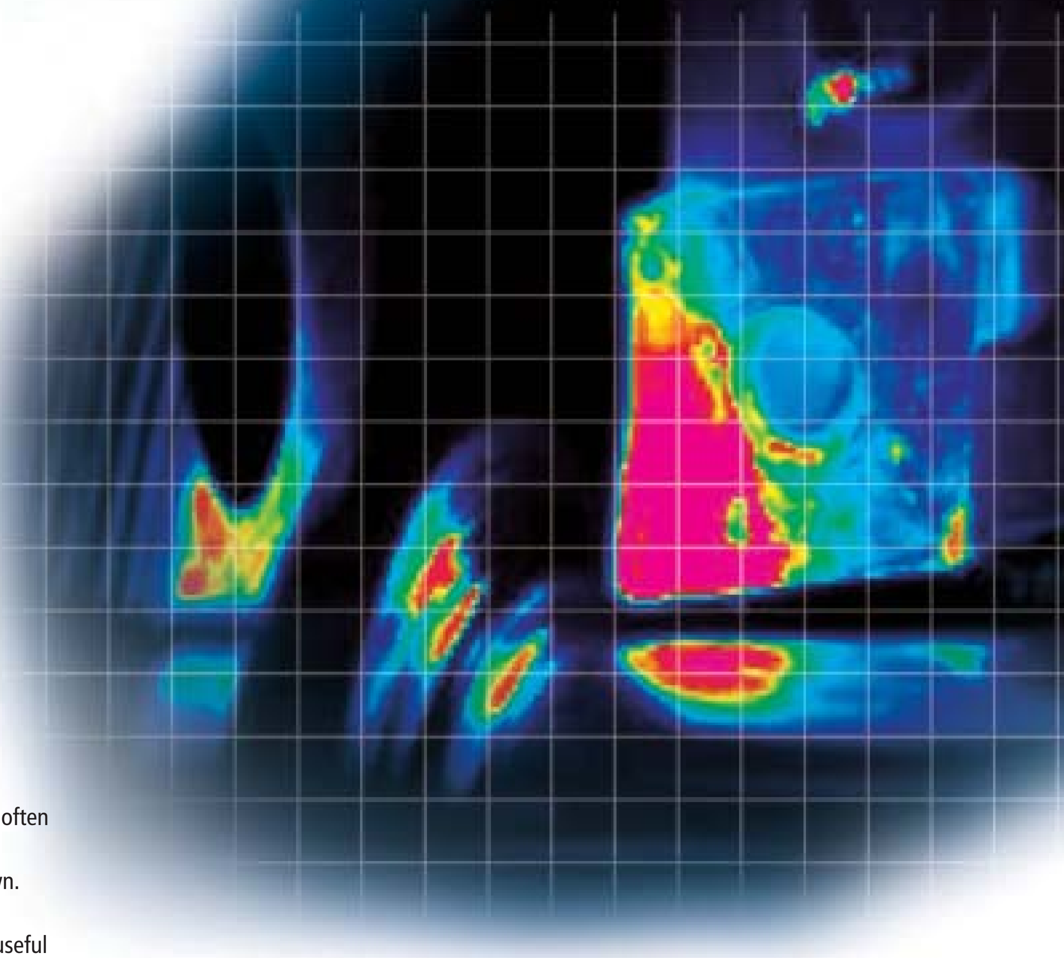
HIGH TEMPERATURE APPLICATIONS

- Phase changes in Shape Memory Alloys
- Lead-free solder
- Thermal barrier coatings
- Polymers - properties close to/in glass transition region
- Hot hardness for Cutting tools
- Coatings, Superalloys for power generation
- Fuel cells – mechanical integrity at operating temp – phase changes
- Elevated temperature nanoscale fatigue
- Nuclear materials
- Bulk metallic glasses
- Adhesion and friction testing

Figure 1



Schematic of NanoTest hot stage showing separate tip and sample heaters, figure courtesy of AJ Muir Wood, University of Cambridge



WHY TEST AT ELEVATED TEMPERATURE?

Before the advent of high temperature nanoindentation, thermal stability and high temperature performance of new materials was often inferred from measurements taken at 25°C on samples previously heated and cooled back down.

Although this approach can occasionally prove useful it is much better to actually test at temperature – where dislocation activity and creep is more significant and hardness can be greatly reduced – as actual performance depends on material properties (hardness, modulus, H/E, toughness etc) at operating temperature not at 25°C!

With the NanoTest it is possible to test at elevated temperature (750°C) samples that have previously been annealed at even higher temperature. Differences in hardness between annealed and as-deposited AlTiN coatings can be minimal at room temperature but dramatic at elevated temperature [1-2].

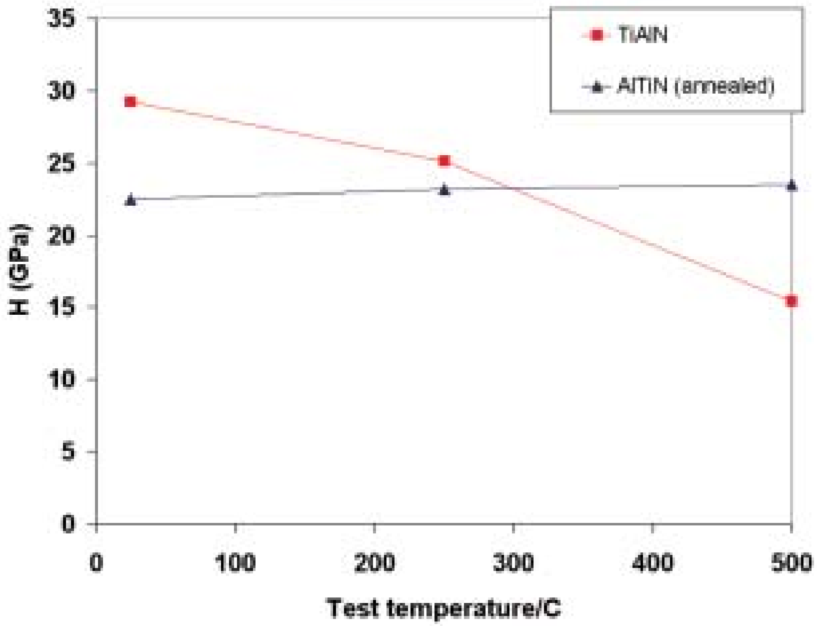


Figure 2

Variation in hardness of TiAlN and AlTiN coatings on WC-Co with test temperature. Annealing at 900°C prior to testing has a beneficial effect on the hot hardness of AlTiN coating [2].

WHY IS A HEATED PROBE ESSENTIAL?

Finite element modelling has conclusively shown that without a heated indenter it is impossible to make meaningful high temperature measurements due to heat flow from the hot sample to the cold indenter

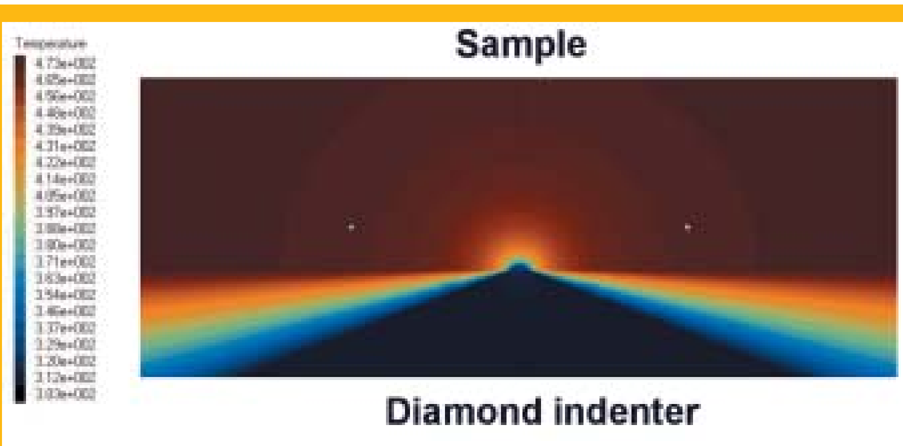


Figure 3

Heat flow for a sample of thermal conductivity 200 WmK^{-1} if the indenter is not heated. The indenter has to be heated to avoid this.

ELEVATED TEMPERATURE NANOINDENTATION TESTING

NANOTEST KEY ADVANTAGES FOR HIGH TEMPERATURE TESTING

- **Horizontal loading**
- **True depth sensing indentation**
- **Isothermal contact**
- **Minimal thermal drift**
- **Localised heating approach**
- **Separate tip and sample heaters**
- **Stability for low load long duration creep tests**
- **Maximum temperature 500°C (with option to 750°C or more)**
- **Sapphire indenters can be used above 500°C**

PHASE CHANGES: SHAPE MEMORY ALLOYS

Researchers at Cambridge [3] and General Motors [4] employ elevated temperature nanoindentation with the NanoTest to study phase changes occurring in NiTi shape memory alloy. A shape memory effect is observed in the nanoindentation test at elevated temperature, where up to 8% strain is accommodated by phase transformation when a spherical test probe was used. The stress-induced phase transition from the parent phase to metastable martensitic phases is reversible (superelastic effect).

The NanoTest high temperature nanoindentation capability has also been used to reveal that indentation-induced phase changes in silicon are also strongly dependent on the measurement temperature.

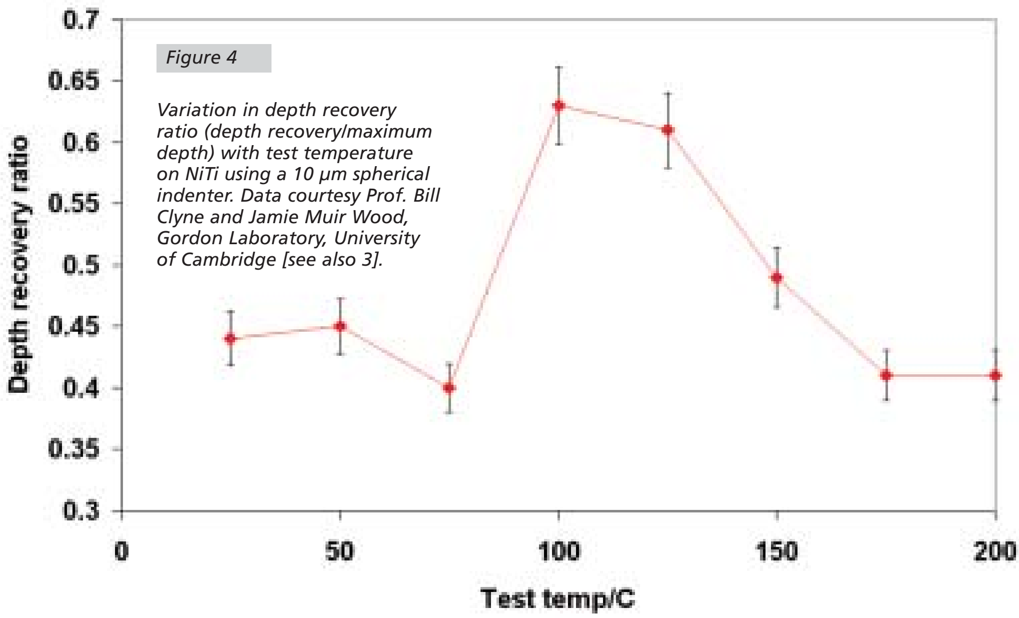


Figure 4

Variation in depth recovery ratio (depth recovery/maximum depth) with test temperature on NiTi using a 10 μm spherical indenter. Data courtesy Prof. Bill Clyne and Jamie Muir Wood, Gordon Laboratory, University of Cambridge [see also 3].

SILICON CARBIDE

The influence of grain refinement on the nanomechanical properties of experimental bulk SiC samples has been investigated by scientists at KAIST, Korea. There was no observable change in elastic modulus over the temperature range 25-500°C. Dislocation slip was activated at low temperatures in these materials. At low temperature grain refinement significantly alters hardness but at higher temperatures it has little effect.

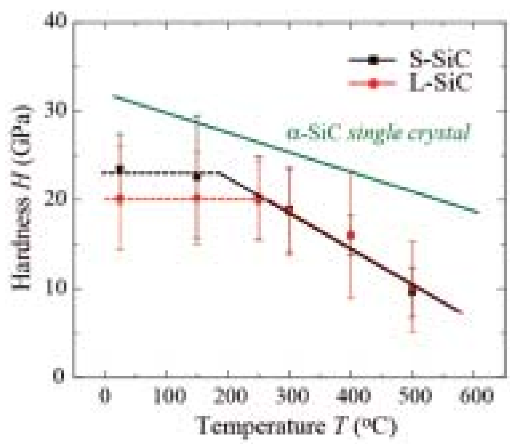
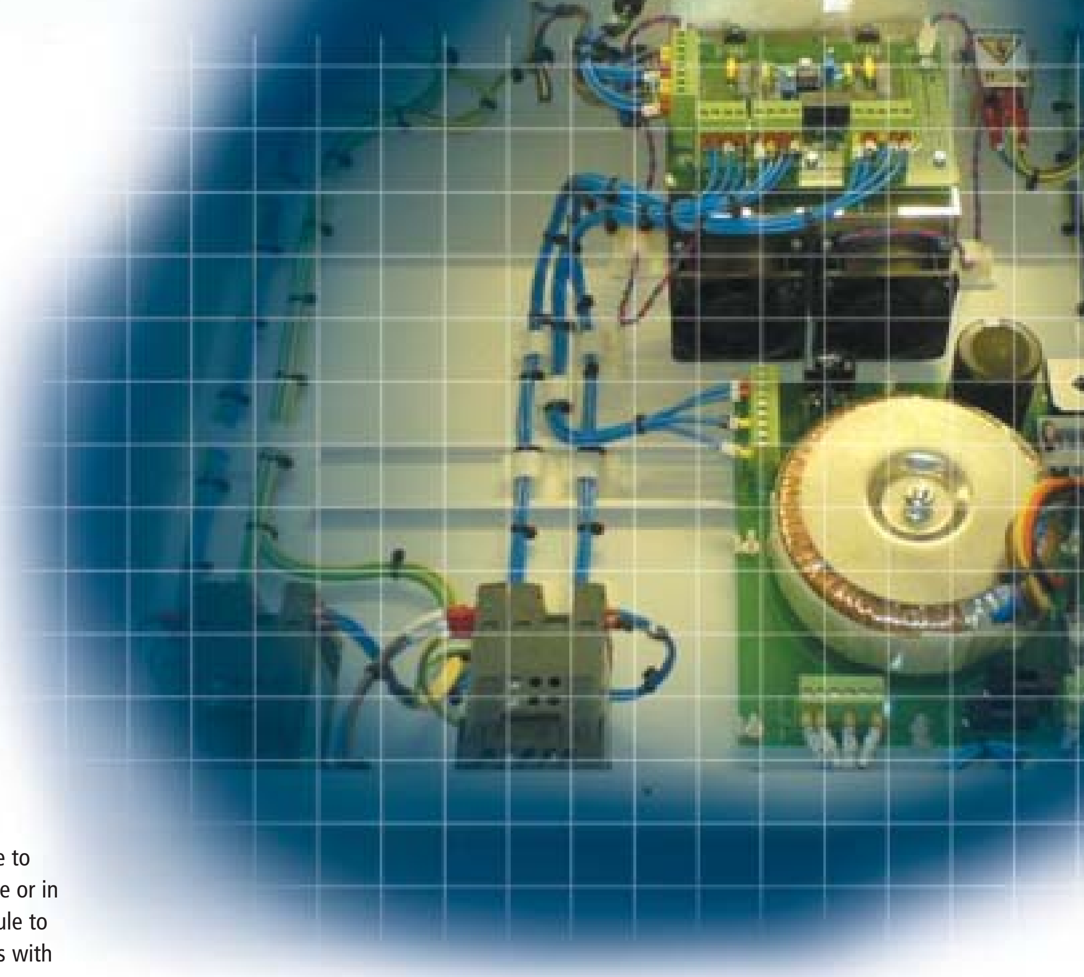


Figure 5

Hardness vs. temperature for SiC. Over the same temperature range there is no change in elastic modulus. Figure courtesy Dr Jong Ho Kim, KAIST, Korea.



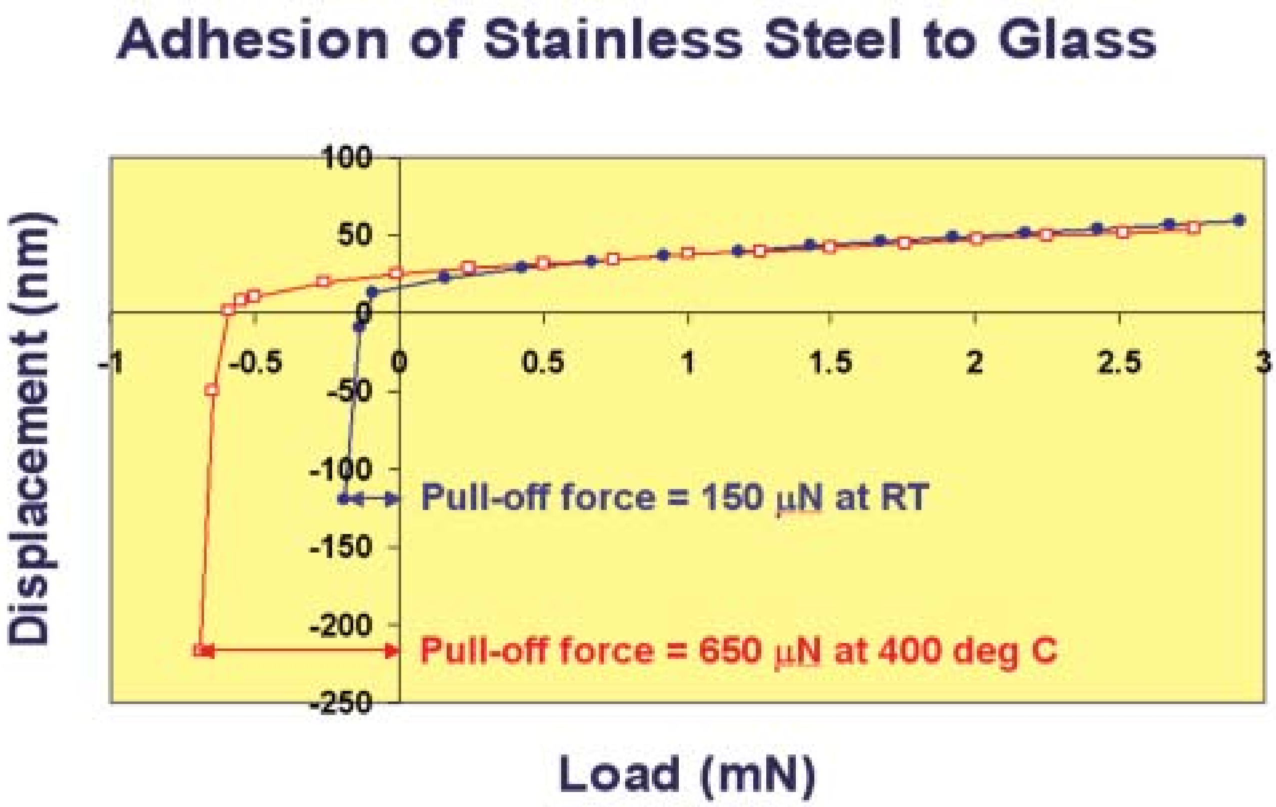
ADHESION – PULL-OFF FORCES VARY WITH TEMPERATURE

Adhesion and friction are a strong function of temperature. The hot stage can be used in conjunction with the NanoTest Scratch module to study stick-slip friction at elevated temperature or in conjunction with the NanoTest Adhesion module to study the variation in adhesive (pull-off) forces with temperature.

It is not necessary to use a diamond indenter. For example in figure 6 the pull-off force between a 500 µm steel sphere and a glass plate was measured as a function of temperature (data courtesy Northwestern University).

Bespoke electronics inside NanoTest hot stage controller

Figure 6



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NANOSCALE GLASS TRANSITION BEHAVIOUR

With the advent of reliable high temperature testing it becomes possible to observe how the nanomechanical and viscoelastic properties of polymeric thin film materials change as they go through the glass transition temperature. Elevated temperature nanoindentation has revealed that changing mechanical properties with temperature on PET [poly(ethylene terephthalate)] film is a dramatic function of processing history and crystallinity [5].

For example, Figure 7 shows high temperature nanoindentation into an undrawn (amorphous, non-heat set) PET film material. A sharp decrease in mechanical properties between 70 and 80°C occurs due the presence of a glass transition and indentation creep is dramatic at both 80°C and 90°C. At 110°C a noticeable improvement in modulus was observed due to the onset of cold recrystallization.

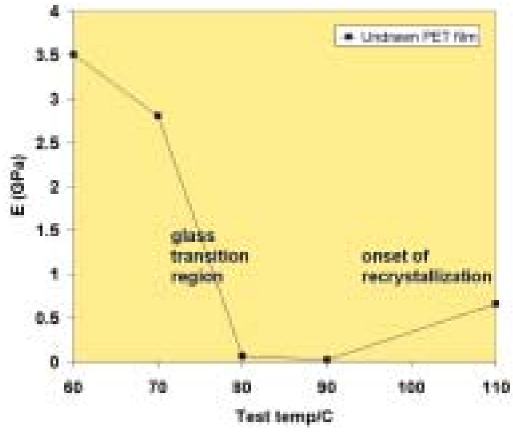
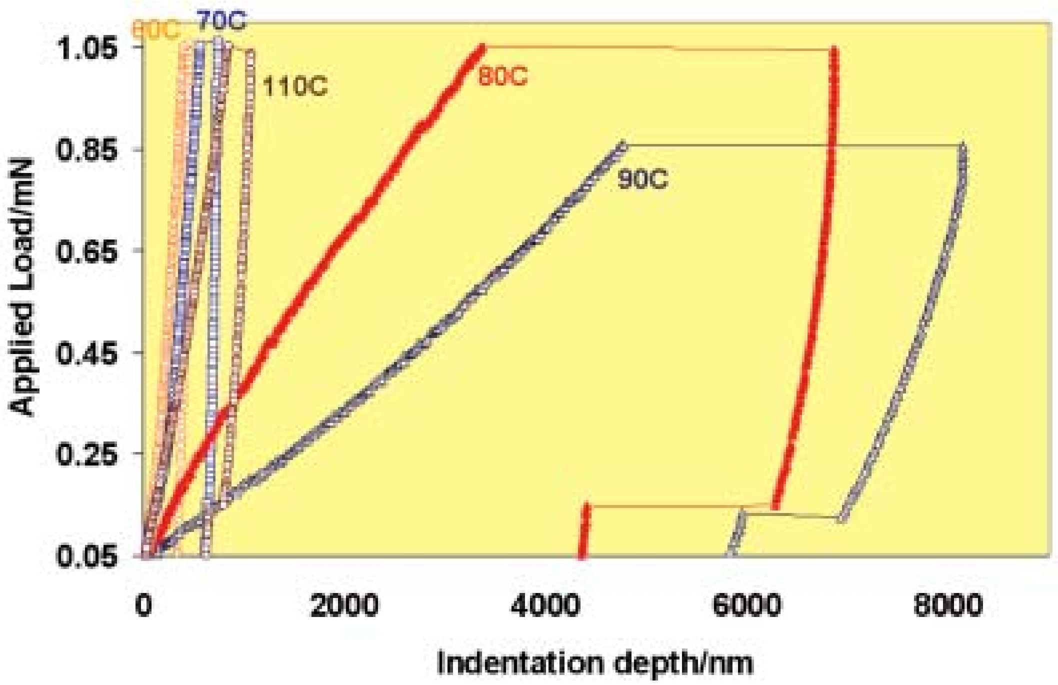
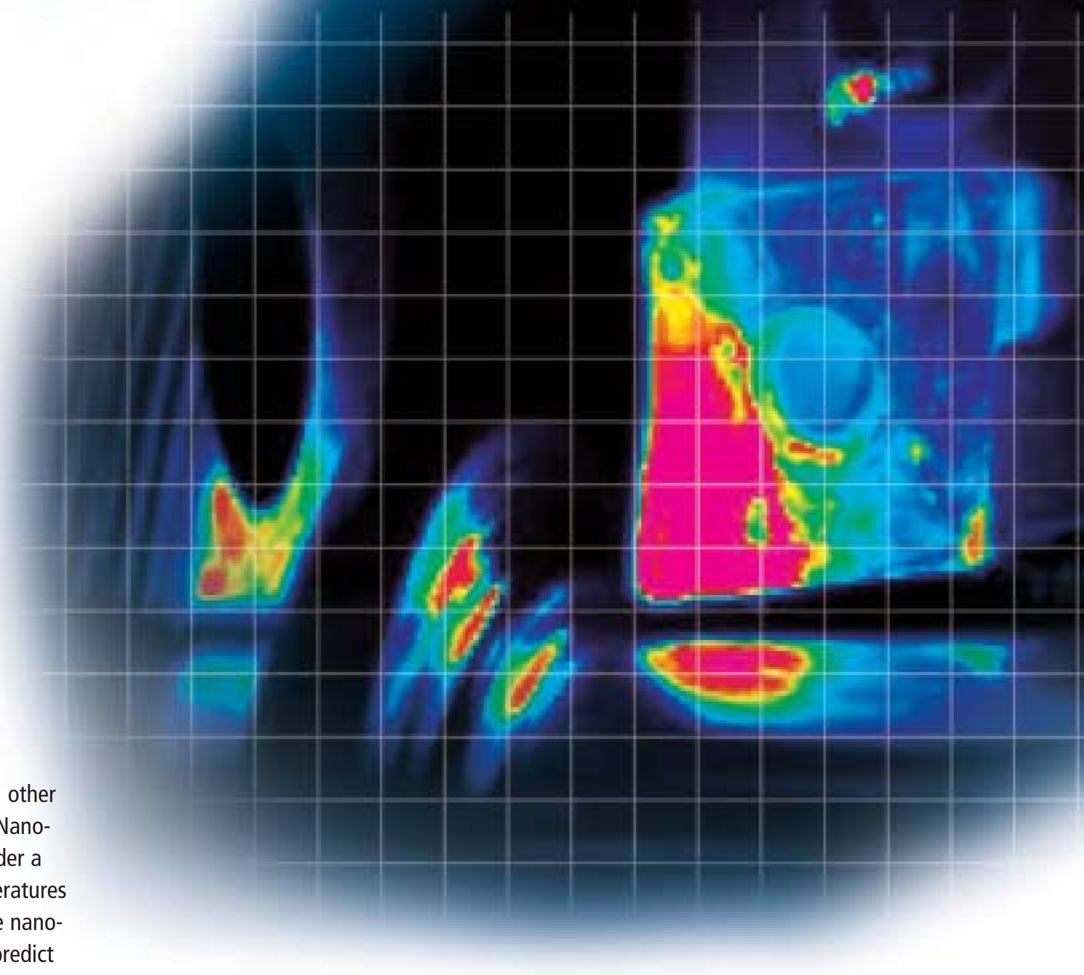


Figure 7

Variation in nanoindentation behaviour and elastic modulus with test temperature over the range 60-110°C for an undrawn (amorphous) PET thin film





REPETITIVE IMPACT AT HIGH TEMPERATURE

The hot stage can be used in conjunction with other NanoTest modules such as Nano-scratch and Nano-impact. This enables tests to be performed under a very wide range of strain rates and test temperatures as illustrated in figure 8. Elevated temperature nano-impact testing has been used successfully to predict the performance of coated cutting tools in demanding applications such as end and face milling of Ti alloys [2].

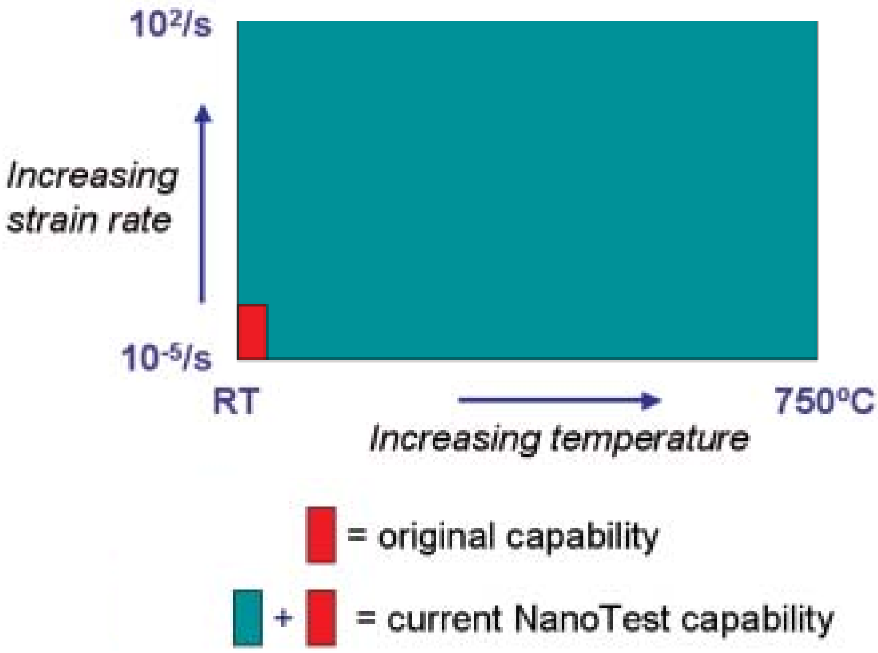


Figure 8

All materials properties are temperature dependent and materials can fail by fatigue aswell as overload; advances in NanoTest instrumentation mean we can now study these effects separately or together at the nano- and micro-scale.

SPECIFICATIONS

- The standard NanoTest hot stage, heated indenter and thermal control system operates to 500°C.
- The hot stage can be used in conjunction with the indentation, scratch, impact and adhesion modules.
- Separate PID controlled heating (and active temperature control) of both probe and sample ensuring no heat flow occurs during the indentation process (isothermal contact).
- Minimal instrumental thermal drift at elevated temperatures allows indentation creep tests at elevated temperatures.
- Option to 750°C.
- Option for controlled atmosphere testing.
- Option for elevated temperature testing with MicroTest high load head.

ELEVATED TEMPERATURE NANOINDENTATION TESTING

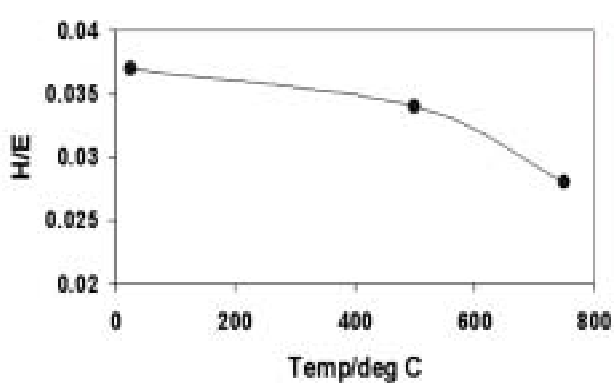


Figure 9

Variation in H/E from nanoindentation of an EB-PVD thermal barrier coating (Zirconia/8wt% yttria) at 750°C – diamond can oxidise above 500°C so a sapphire Berkovich indenter was used

THERMAL BARRIER COATINGS

At Cranfield University Prof. John Nicholls uses elevated temperature nanoindentation to investigate the mechanical properties of advanced TBCs for turbine engines. Electron beam deposited YSZ coatings are heterogeneous and columnar (see figure 3) with individual erosive events occurring during use on the scale of the individual columns and so it is necessary to probe their properties by nanoindentation testing rather than by bulk measurements.

Variation in the TBC hardness/modulus ratio (a useful dimensionless index often important in tribological situations) with temperature is shown in figure 9. Above 500°C the TBC loses stiffness and plastic deformation becomes more important, which helps avoid sintering across columns and top-coat debonding.

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