

# NanoTest Xtreme

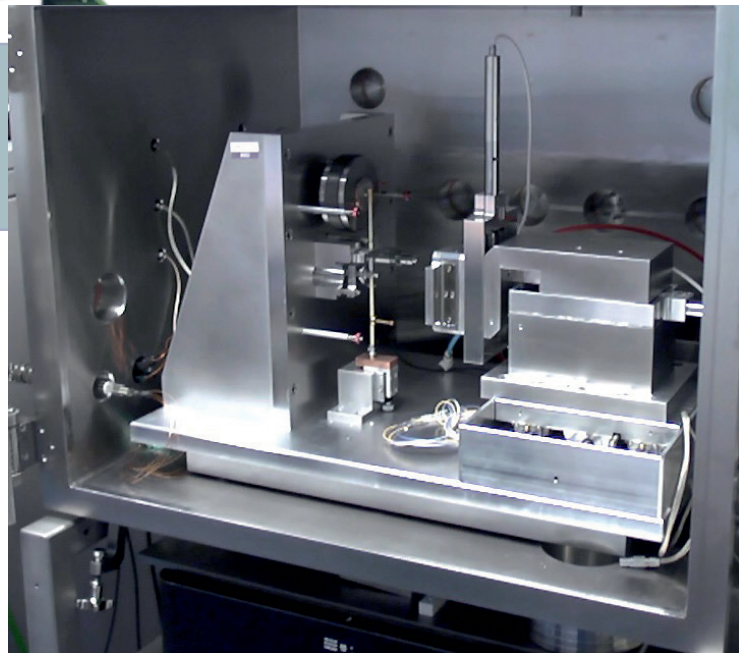
*Xtreme*



## Advantages

- Extended high temperature capabilities to 1000 °C – beyond the 850 °C provided by the NanoTest Vantage
- Enhanced low temperature capabilities to -40 °C without frosting of samples
- Ultra-low thermal drift due to same instrument design principles as those proven in the NanoTest Vantage
- Complete range of nanomechanical tests (e.g., indentation, scratch, wear, friction, impact)
- Ability to backfill with gas to match material operating environments

*Xtreme*



# NanoTest Xtreme



## The Nanomechanical testing centre that's optimised for investigating the effects of extreme environments

### Extreme Environments

Extrapolating results from ambient or near-ambient temperatures to predict high and low temperature properties has long been shown to be unrealistic and prone to error. In order to provide the most reliable and accurate prediction of properties, test conditions must closely mimic real-world environments.

Whilst Micro Materials already offers researchers the most comprehensive range of nanomechanical test options via the NanoTest Vantage, our NanoTest Xtreme now enables you to investigate more extreme environments than ever before, including:

- High temperatures for aerospace engine components
- Tool coatings for high speed machining
- High temperatures for power station steam pipes
- Irradiation effects in nuclear reactor cladding
- The effect of cold on weld repairs in oil/gas pipelines

### Testing under Vacuum

Until recently, nanomechanical testing instruments have been limited by oxidation at high temperatures and condensation/frosting at sub-zero temperatures. Testing under vacuum (see Figure 1) negates these problems, thus widening the temperature range over which testing is possible.

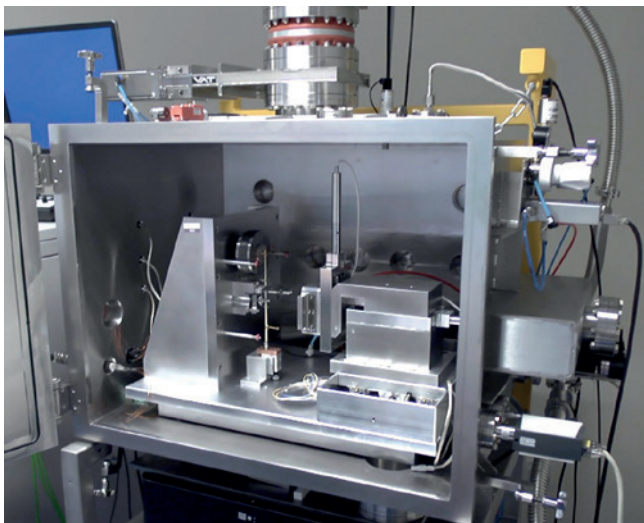


Figure 1: The NanoTest Xtreme situated inside the vacuum chamber.

### Testing Superalloys to 1000 °C

Nanoindentation is ideally suited to further the development of high temperature materials, such as the (Ni,Co) CrAlY bond coats protecting nickel-based superalloys in turbine blades. Until very recently, the operating temperatures of these materials were out of reach for nanoindentation systems. However, the unique design of the NanoTest Xtreme allowed scientists at RWTH Aachen University in Germany to push testing temperatures up to 1000 °C and gather valuable information on the hardness and creep behaviour of an Amdry-386 bond coat. See Figure 2.

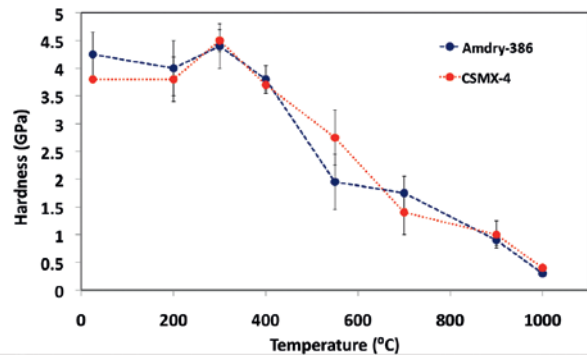


Figure 2. Scientists at RWTH Aachen have recently been the first group to perform nanoindentation tests at 1000 °C. Temperature dependence of hardness over the range 25-1000 °C for Amdry-386 bond coat and superalloy substrate.

The NanoTest Xtreme allows vacuum environment testing from -40 to 1000 °C ... without oxidation or frosting of samples

# NanoTest Xtreme



## Testing Tungsten to 950 °C

Elevated-temperature nanomechanical testing provides a convenient route to characterising the mechanical properties of materials used in high temperature applications at, or close to, their operating temperatures. This testing affords more relevant characterisation than measurements at room temperature. With ongoing advances in test instrumentation, elevated-temperature nanomechanical testing is becoming more commonplace in materials development for safety-critical sectors such as the nuclear industry.

Tungsten and its alloys are being considered as the main plasma-facing material in a fusion reactor. In collaboration with scientists at the University of Oxford, the NanoTest Xtreme has been used to test the mechanical properties of polycrystalline tungsten in high vacuum to 950 °C. Testing under high vacuum was essential as tungsten oxidises rapidly at >500 °C in air.

More significant time-dependent deformation was observed from 850 °C. The strain rate sensitivity determined by analysis of indentation creep data increased with temperature (see Figure 3).

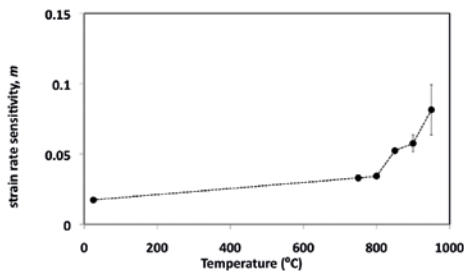


Figure 3. Strain rate sensitivity vs. T for polycrystalline tungsten.

With thermal drift at 750 to 950 °C typically as low as 0.05 nm/s, the NanoTest Xtreme has the stability to run longer duration indentation creep tests throughout the temperature range (see Figure 4).

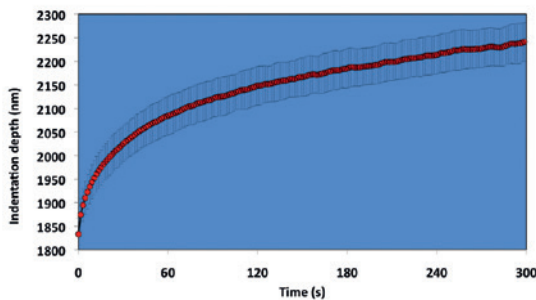


Figure 4. Development of indentation creep during 300 s at 945 °C. Mean and standard deviation from 3 repeat tests at 200 mN.

## Ultimate Nanopositioning at High Temperature

The NanoTest Xtreme's localised heating design allows the rest of the instrument to remain only a few degrees above room temperature. A key benefit of this design is that the SPM-nanopositioning stage (next to the hot stage) can be used throughout the temperature range. Images acquired at high temperature enable precise indentation positioning at temperature or targeting of specific features such as pillars for micro-compression tests or cantilevers for microscale bending experiments.

Researchers from the Department of Materials at the University of Oxford have used a NanoTest Xtreme to perform bending tests on microscale cantilevers at temperatures up to 770 °C with a cubic boron nitride indenter (see Figure 5).

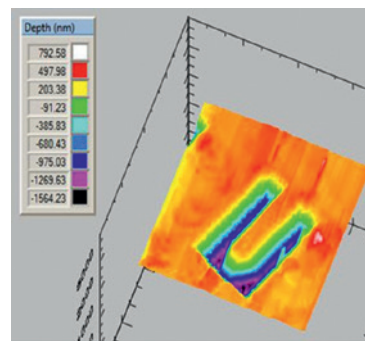
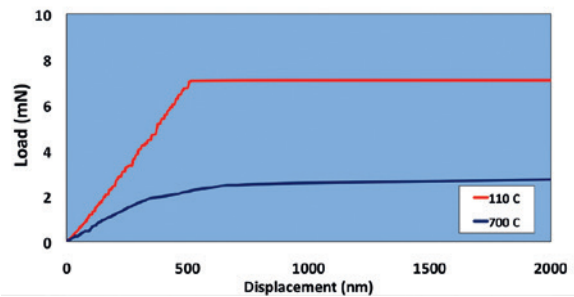


Figure 5. Image of a micro-cantilever FIB-milled on Si. The image was acquired with the integrated SPM-nanopositioning stage at 700 °C. Example micro-cantilever tests performed at temperatures above and below the brittle-ductile transition are shown.

Independent indenter and sample heating was used to ensure an isothermal contact and thus eliminate thermal drift. The high temperature images with the SPM-nanopositioning stage were used to position the indenter and perform micro-cantilever bend tests. These tests enabled temperature-dependent modulus, yield stress, and fracture behaviour to be determined and differences in ductility with increasing temperature to be investigated.

# NanoTest Xtreme

Xtreme

Specifications	
Load frame	Highly polished Aluminium for rapid degassing
Load application	electromagnetic
Maximum load with standard head	500 mN
Maximum load with optional high load head	30 N
Displacement sensor	capacitive
Load resolution	3 nN
Displacement resolution	0.002 nm
Repositioning accuracy	< 0.4 $\mu$ m
Sample manipulation	manual control, grid indentation, specific site selection, multiple simultaneously mounted samples
Thermal drift	<0.005 nm/s
Compliance with standards	compliant with ISO 14577 and ASTM 2546
High temperature stage	
Maximum temperature	1000 °C
Indenter tip heating	yes
Testable sample area	16 mm x 16 mm
Temperature control	feedback and constant power
Temperature accuracy	< 0.1 °C
Cold stage	
Minimum temperature	-40 °C
SPM nanopositioning stage	
Scan range	100 $\mu$ m x 100 $\mu$ m
X Y positioning accuracy	2 nm
Vacuum	
Operating modes	vacuum or gas purge
Vacuum level	ultimate $10^{-7}$ (Typical $10^{-6}$ ) mbar
Options	
	nano-scratch, nano-wear, nano-impact, dynamic hardness

## Key features

- Maximum testing temperature under vacuum for 500 mN loading head: 1000 °C
- Maximum testing temperature under vacuum for 30 N loading head: 800 °C
- Minimum testing temperature under vacuum: -40 °C
- Ultimate vacuum:  $10^{-7}$  mbar
- Compatible with all standard NanoTest techniques under vacuum (nanoindentation, nano-scratch, nano-wear, nano-impact, nano-fretting)
- Option of a second loading head increases maximum load from 500 mN to 30 N
- Backfill function to test in non-air gas environments
- High resolution optical microscope
- Option to add SPM imaging/nanopositioning stage which can be used over the full temperature range

## References

On extracting mechanical properties from nanoindentation at temperatures up to 1000 °C, J.S.K.-L. Gibson, S. Schröders, Ch. Zehnder, S. Korte-Kerzel, Extreme Mechanics Letters 17 (2017) 43–49.

Development of high temperature nanoindentation methodology and its application in the nanoindentation of polycrystalline tungsten in vacuum to 950 °C, A.J. Harris, B.D. Beake, M.J. Davies, D.E.J. Armstrong, Exp. Mech. 57 (2017) 1115–1126.

Bend testing of silicon cantilevers from 21 °C to 770 °C, D.E.J. Armstrong and E. Tarleton, Journal of Materials 67 (2015) 2914–2920.

## Micro Materials Ltd has been at the forefront of nanomechanics innovation since 1988 with:

- The first commercial high-temperature nanoindentation stage
- The first commercial nano-impact tester
- The first commercial liquid cell
- The first commercial instrument for high-vacuum, high-temperature nanomechanics