Micro Materials
Excellence in Nanomechanics

NanoTest high temperature nanomechanical measurements portfolio

Introduction

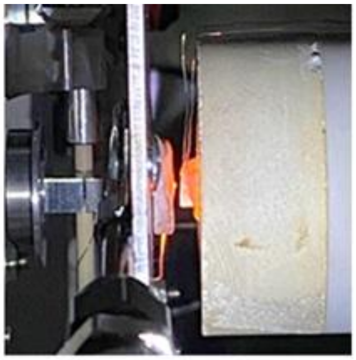
For 25 years Micro Materials NanoTest systems have been at the forefront of elevated temperature nanomechanical testing. From the earliest published work in 2000 [1] there has been continuous development enabling a wide range of nano- and micro-mechanical and tribological tests to be performed at increasingly higher temperatures and under different environments (air, argon, reducing gas and high vacuum) [2]. Maximum test temperatures are 850 °C for the NanoTest Vantage and 1000 °C or more for the NanoTest Xtreme.

Test Capabilities

- Nanoindentation ✓
- Nanoindentation Creep ✓
- Micro-Pillar Compression ✓
- Micro-Cantilever Bending ✓
- Nanoscratch Testing ✓
- Reciprocating Nanowear ✓
- Cyclic Impact ✓
- SPM Nanopositioning ✓
- Spatially-Distributed Multiple Impact ✓
- SPM Imaging ✓
- Spatially-Distributed Scratch Testing ✓

Properties Obtained

- Hardness ✓
- Elastic Modulus ✓
- Fracture Toughness ✓
- Position-Sensitive Local Mechanical Properties ✓
- Strain Rate Sensitivity ✓
- Impact Resistance ✓
- Plastic Deformation Mechanisms ✓
- Phase Transformation Temperatures ✓
- Nanowear Resistance ✓
- Cyclic Impact Behaviour (S-N) ✓
- Brittle-Ductile Transition Temperature ✓
- Abrasion Simulation ✓
- Solid Particle Erosion Simulation ✓
- Scratch Adhesion ✓

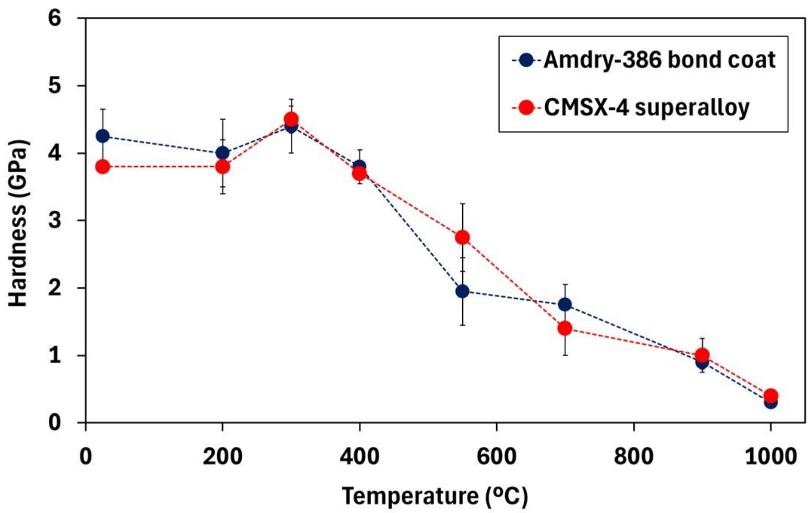

Nanoindentation to 1000 °C

Indenter materials and test environment in publications with maximum test $T \geq 750$ °C

Materials system	Indenter material	Test environment	Max. test temperature (°C)	Reference	Year
NiCoCr medium entropy alloy	cBN	Air	750	3	2022
PVD TiAlN and TiCN coatings	cBN	Argon	750	4	2014
Tungsten	cBN	Vacuum	750	5	2014
G18 glass-ceramic	cBN	Argon	750	6	2011
Silicon	cBN	Vacuum	770	7	2017
Inconel 617	cBN	Air	800	8	2017
Ti_2AlN MAX-phase	Sapphire	Vacuum	800	9	2022
SiC ceramic matrix composites	Sapphire	Argon	800	10	2021
MgCoNiCuZnO entropy stabilised oxide	cBN	Air	950	11	2022
Tungsten	cBN	Vacuum	950	12,13	2017, 2018
Cr_2AlC MAX-phase	Sapphire	Vacuum	980	14	2019
CMSX-4 Ni-base superalloy and MCrAlY bond coat	Sapphire	Vacuum	1000	15	2017

Measurements in air or argon with NanoTest Vantage; measurements in vacuum with NanoTest Xtreme instruments.

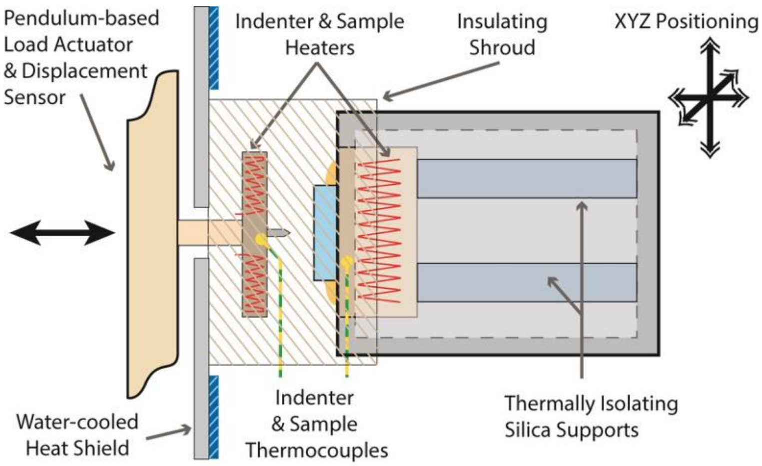
In 2017 Prof Sandra Korte-Kerzel, Dr James Gibson and colleagues at RWTH Aachen were the first group to perform nanoindentation tests at 1000 °C.



Temperature dependence of hardness over the range 25-1000 °C for Amdry-386 bond coat and superalloy substrate. NanoTest Xtreme data from measurements with sapphire indenter to 1 μm indentation depth under vacuum [15].

Independent active heating of sample and indenter

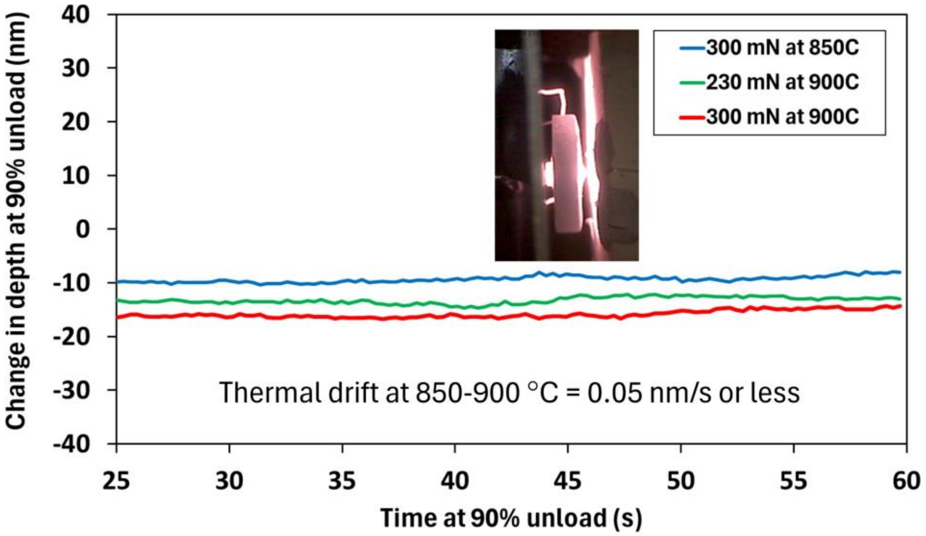
To achieve an isothermal contact and minimise thermal drift in an elevated temperature nanoindentation test the indenter and sample are both actively heated.



A schematic illustration of NanoTest High temperature set up [2]. Additional surface thermocouples can also be used to improve accuracy in assigning the sample surface temperature.

Ultra-low thermal drift even at the highest temperatures

Active tip heating is combined with additional design factors to achieve minimal thermal drift: (1) localised heating – heat shield and insulating shroud (2) patented control protocol to match indenter and stage temperatures to 0.1 °C and (3) for measurements in gaseous environments the horizontal loading configuration eliminates convection effects.

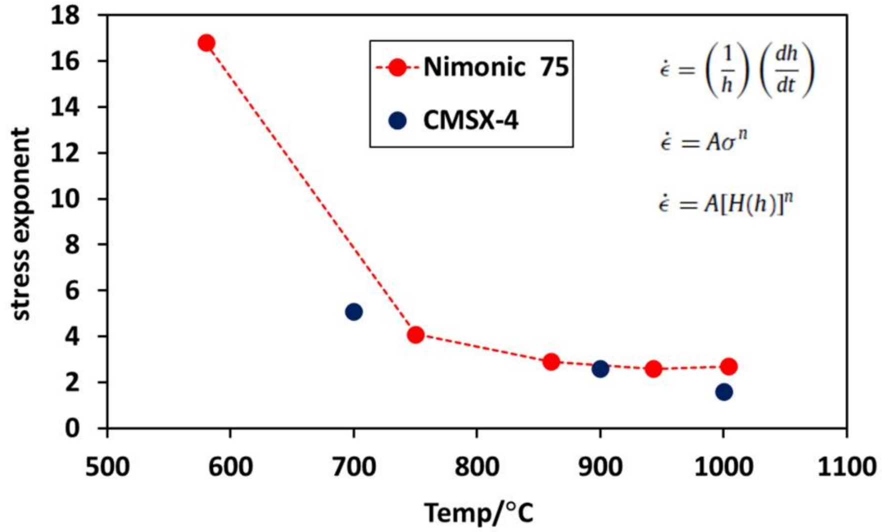


Assessment of thermal drift on a polycrystalline tungsten sample during a hold period at 90% unloading. NanoTest Xtreme with cBN indenter [12,13].

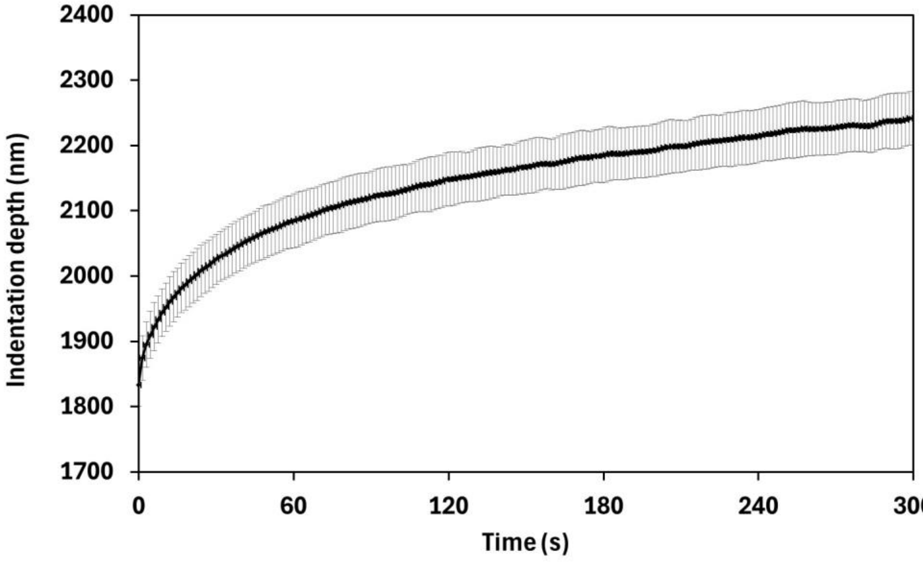
Indentation creep analysis

Developing advanced alloys with enhanced creep resistance at elevated temperatures

Creep is responsible for many component failures at high temperatures. Creep deformation becomes increasingly dominant at homologous temperatures of T/T_m = 0.5 and above. Because grain boundaries in materials usually facilitate diffusional processes in creep, eliminating grain boundaries is a primary approach in resisting high-temperature creep in metals, e.g. as in single-crystal Ni-base superalloys for turbine blades.



Creep stress exponents for CMSX-4 [15] and Nimonic 75 measured with sapphire indenters under vacuum.



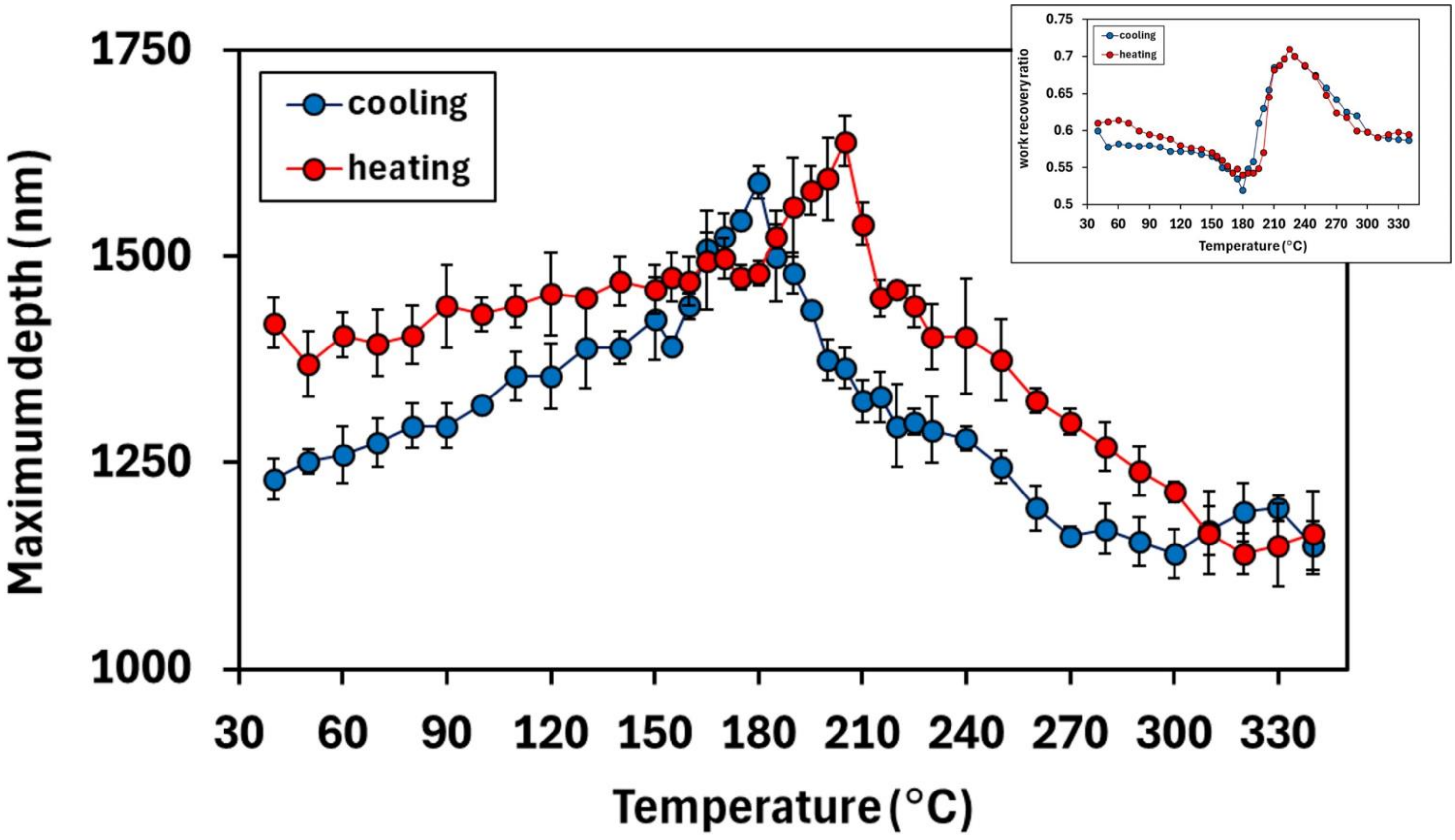
Development of indentation creep on polycrystalline tungsten over 300 s at 200 mN at 945 ℃. Mean and standard deviation from 3 repeat tests. cBN Berkovich indenter in vacuum (NanoTest Xtreme).

Precise thermal control for studying phase transformations

Nanoindentation enables rapid characterization of local shape memory properties. Li et al performed a systematic study of the behaviour of the high-temperature NiTiHf alloy from 30-340 °C by spherical indentation [16]. Tests were performed at closely spaced temperature intervals during heating to characterise the austenite start and finish temperatures and cooling to identify the martensitic phase transformation during cooling.

On heating the highest maximum depth was at 205 °C (see figure) marking the austenite finish temperature matching that from bulk DSC. On cooling the martensitic transition was at 180 °C, also very close to that from bulk DSC.

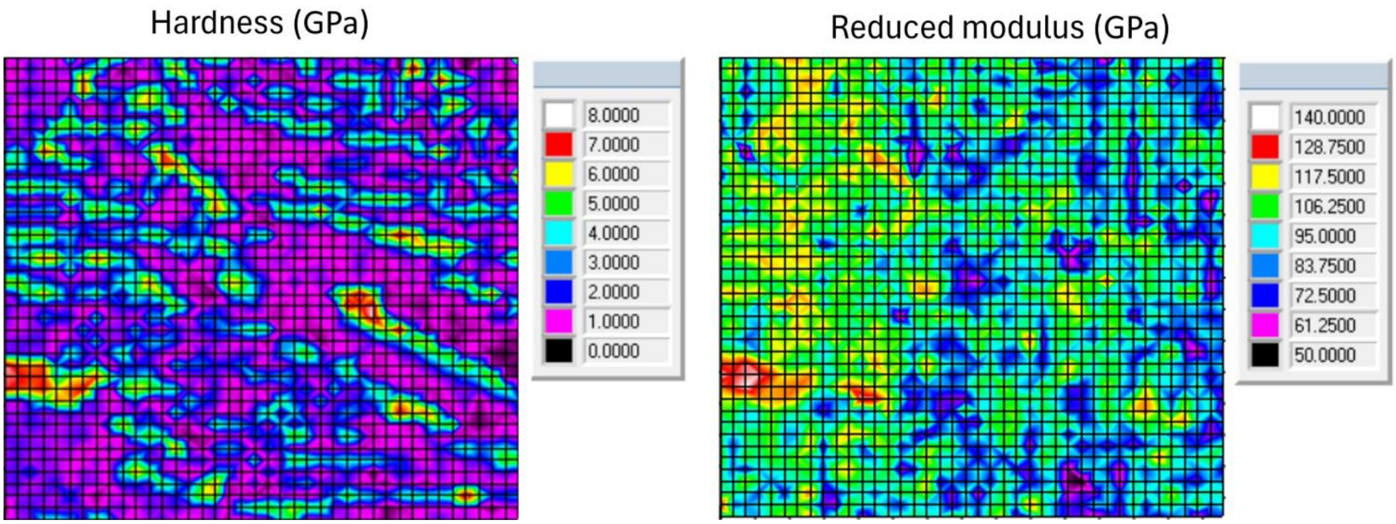
The depth recovery (work recovery ratio, see inset) was used to highlight the superelastic region, which was 205-225 °C.



500 mN tests with 25 μm diamond indenter. Mean values and standard deviations from 3 repeats at different locations [16].

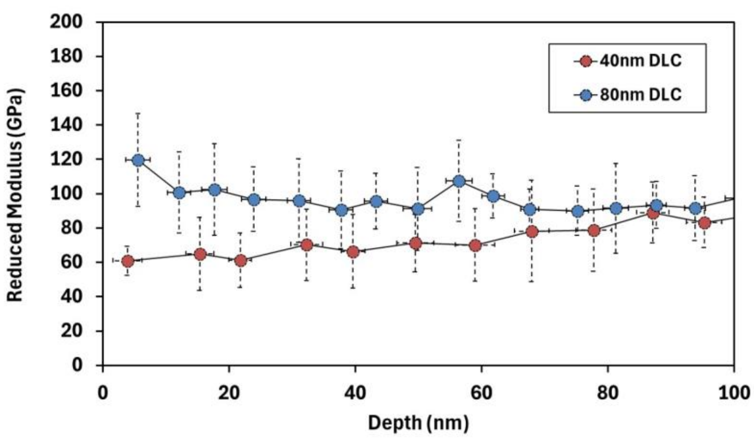
Nanoindentation – mapping and depth profiling mechanical properties

Localised mechanical property mapping is achieved by performing arrays of tests. The indentation size and spacing can be optimised to maximise the number of indentations possible whilst ensuring that they are not spaced too closely that the stress fields interact appreciably. A typical example is the 40 x 40 array on a novel Al alloy designed for high temperature performance and AM processing shown below.



1600 x 80 nm deep indentations at 200 ℃, spaced 1 µm apart. Data shown courtesy of Taha Waqar and Prof Michael Benoit, University of Waterloo.

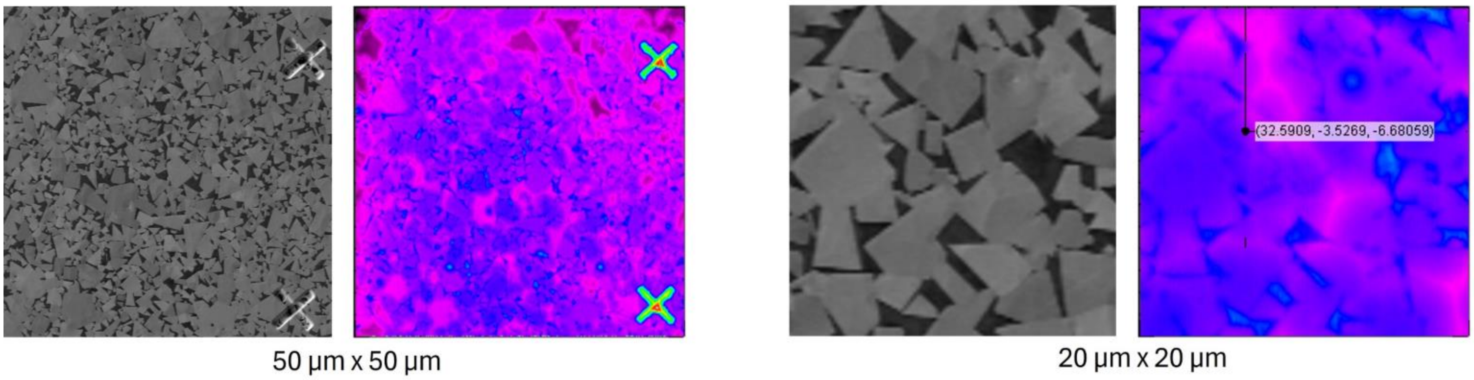
Mechanical properties can also be conveniently studied as a function of depth by load-partial unload or by continuous stiffness methods. This can be useful when studying the mechanical properties of coated or layered systems. The approach is illustrated below for measurements on ultra-thin carbon films deposited on glass.



Tests on 40 and 80 nm DLC films on glass performed in air with a diamond indenter at 200 ℃.

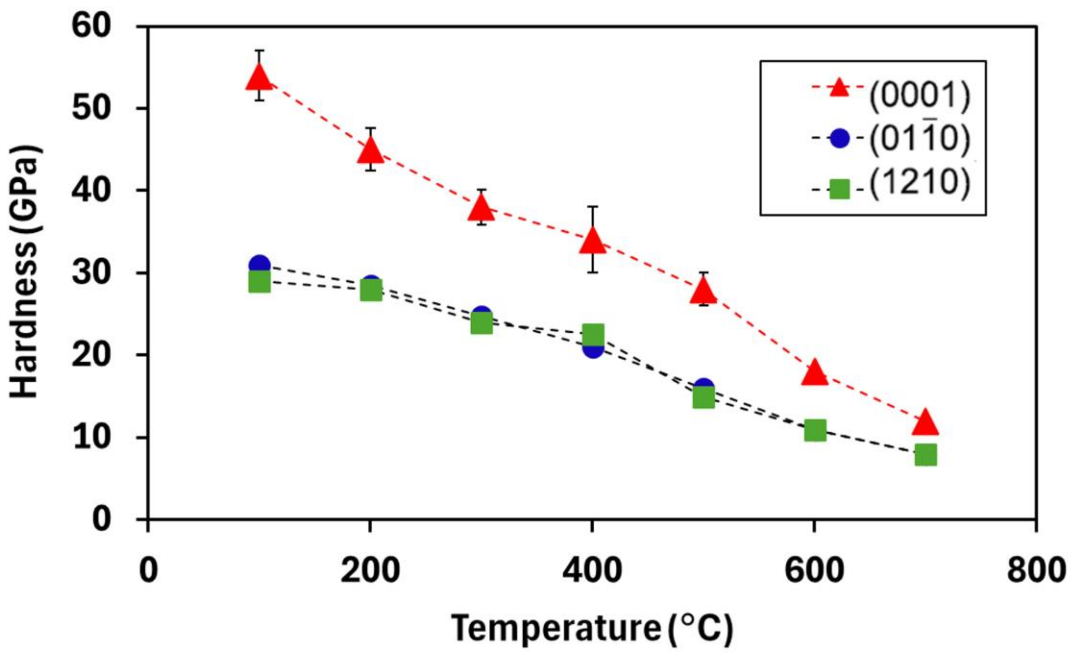
Precision targeting and indent placement

With the localised heating approach employed in the NanoTest Vantage and Xtreme instruments, the rest of the instrument remains only a few degrees above room temperature. A benefit of this 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 micro-scale bending experiments [7, 17-20].



Comparing images of WC-Co with integrated SPM-nanopositioner and SEM [17].

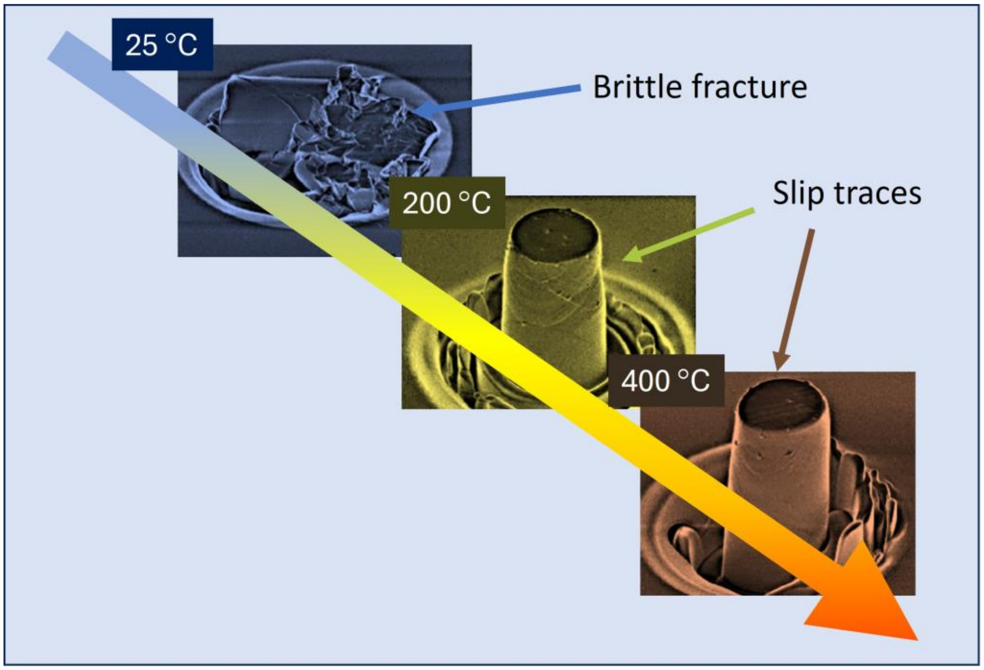
De Luca and colleagues at NPL [17] used a NanoTest Xtreme with an integrated SPM nanopositioning stage to study the temperature dependence in the hardness of WC crystals with different crystallographic orientations.



Tests in vacuum. cBN indenter used above 400 ℃. Error bars generally smaller than the symbols [17].

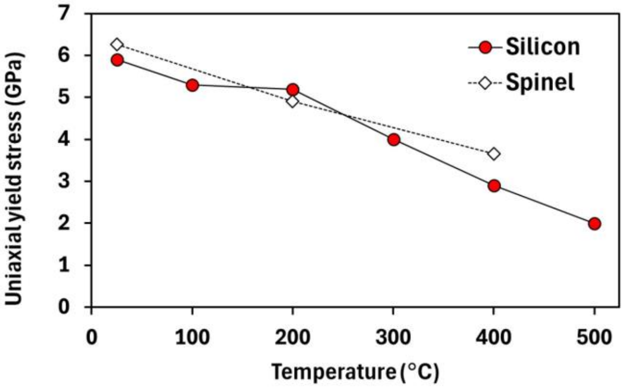
Micro-pillar compression

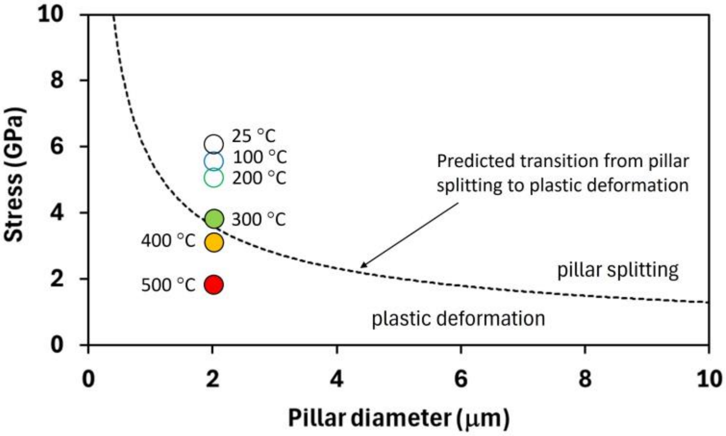
Sandra Korte-Kerzel and colleagues at the University of Cambridge performed compression tests on microscale pillars of intrinsically brittle materials [18,19]. Due to the brittle-to-ductile transition the deformation morphology changed with temperature.



Temperature dependence of compression of 2.5 µm diameter pillars of (001) $MgAl_2O_4$ spinel, compressed with a 10 µm diameter diamond flat punch. [18]

A transition from splitting to predominantly plastic deformation was predicted as the yield stress falls below the splitting stress.

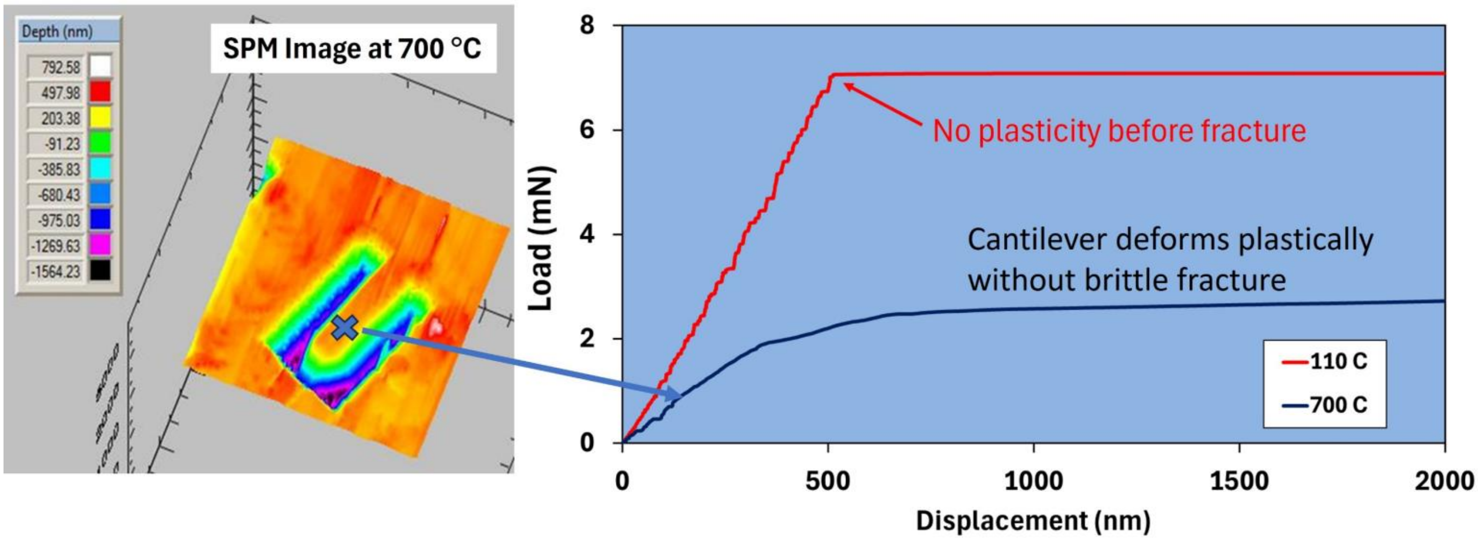




The uniaxial yield stress drops with increasing temperature on both materials. On silicon (right) the results were consistent with the predictions of an axial splitting model [19].

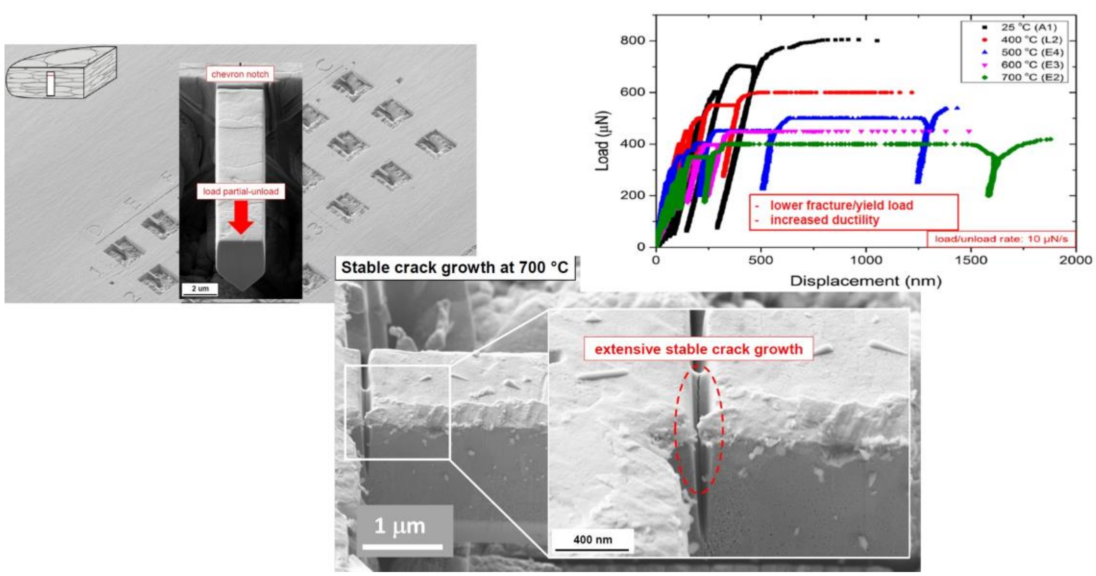
Micro-cantilever bending

High temperature images acquired with the SPM-nanopositioning stage can be used to position the indenter and perform micro-cantilever bend tests. Tests at the University of Oxford on silicon over the range 110-770 °C [7], enabled temperature dependent modulus, yield stress and fracture behaviour to be determined and differences in ductility with increasing temperature to be investigated.



cBN indenter, vacuum (NanoTest Xtreme) [7].

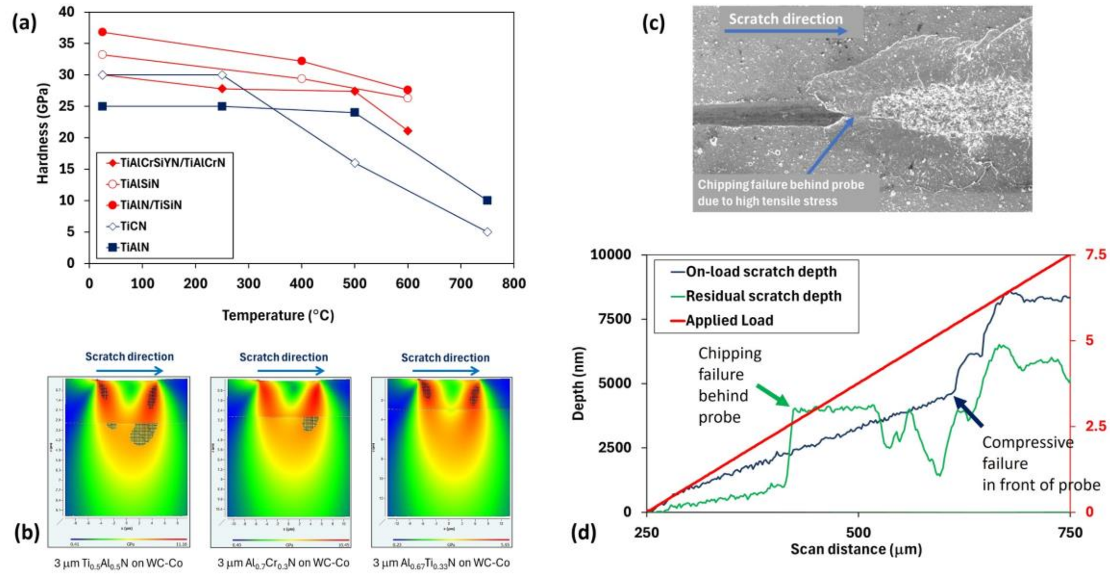
Due to the very high thermal stability it is possible to perform extremely long contact duration tests. Using Chevron-notched micro-cantilevers, Dr Bo-Shiuan Li and Prof David Armstrong at the University of Oxford used this approach [20] to study stable crack growth at elevated temperatures and determine micro-scale brittle-ductile transition temperatures.



Multi-cycle loading-unloading tests on tungsten-tantalum alloy with >30 min contact duration [20]. cBN indenter in vacuum.

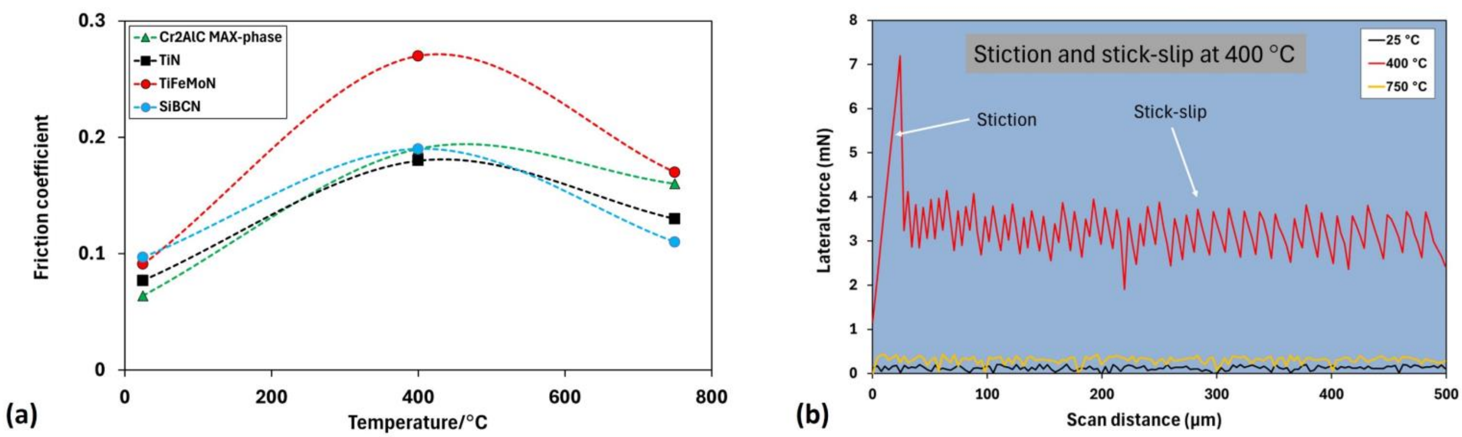
Nano- and micro- scratch testing

The mechanical properties of hard coatings used for wear protection can reduce at higher temperatures (e.g. see (a) below). High temperature mechanical properties influence the initial interface weakening - by initial substrate or coating yielding or both – affecting the deformation failure mechanism – with overloading from substrate side being particularly problematic for coating fracture. In collaboration with Cranfield University and McMaster University an integrated modelling package was used to show that high tensile stresses developed in highly loaded sliding contact (in a micro-scratch test) explaining the experimentally observed chipping failure behind the probe [21].



(a) Temperature dependence of some hard PVD coatings measured with a cBN indenter. Si-containing coatings showed higher hardness at temperature. (b) Simulated von Mises stress distributions [21]. (c,d) chipping behind sliding probe.

Experimental conditions can be chosen to measure interfacial friction by minimising ploughing friction. In tests on hard coatings [22] the highest friction was found at 400 °C. The reduction in friction at 750 °C is due to formation of lubricious surface-oxides and oxidation-associated roughening of the surface lowering the contact area.



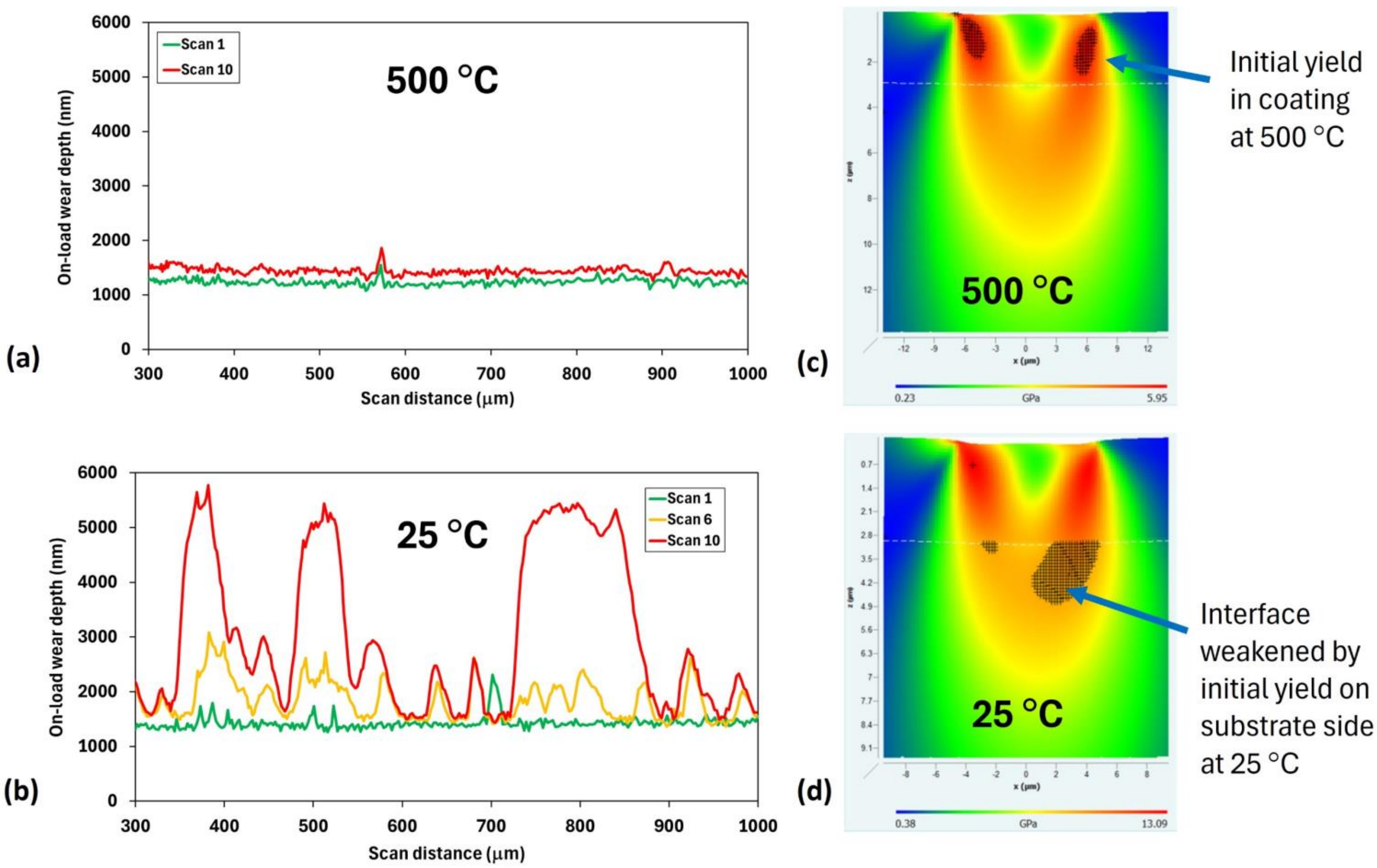
Nano-scratch test at constant 2 mN load with 350 μm radius WC indenter (nominal contact pressure <1 MPa) in air.
(a) Temperature dependence of friction coefficient (b) stiction and stick-slip was also observed [22]

Repetitive nano- and micro- scratch testing

Several types of high temperature wear test are possible including:

(i) repetitive (unidirectional) scratch test with multiple cycles over the same track
(ii) reciprocating wear test with multiple cycles over the same track
(iii) multiple parallel scratch test to simulate spatial distribution in abrasive wear.

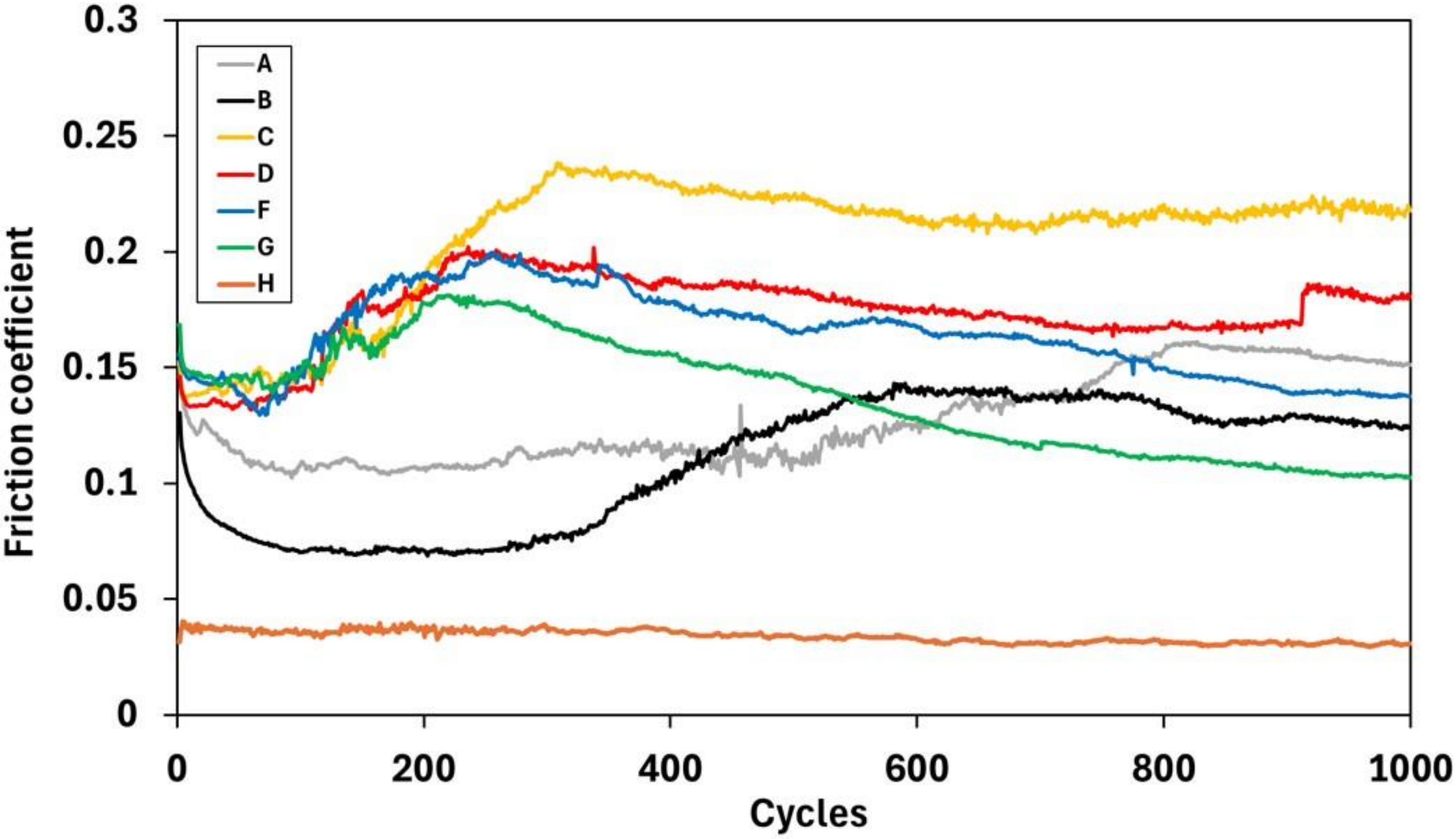
An example of a repetitive (unidirectional) micro-scratch test with multiple cycles over the same track on a PVD AlTiN coating [21] is shown below.



Repetitive scratch tests on 3 µm PVD AlTiN coating on WC-Co. Load = 1 N, spheroconical diamond indenter with 25 µm end radius. On-load depth vs. scan distance over constant load region at (a) 500 ℃ and (b) 25 ℃. Corresponding simulated von Mises stress distributions shown in c and d. The hashed areas show where the system is overloaded (local von Mises stress > yield stress).

Reciprocating nano-wear

Elevated temperature reciprocating nano-wear and small-scale fretting tests can reveal changes to deformation mechanism with temperature. As part of a collaborative research project with Teer Coatings developing advanced low friction coatings for space applications 1000-cycle tests were performed on different coatings at 150 °C. These revealed significantly less wear debris at high temperature than 25 °C. On the MoS_2-based coatings there was more plasticity-dominated than micro-wear dominated deformation at the higher temperature. The best performance was for the coating labelled H in the figure below which retained low friction throughout the test.



1000 cycle reciprocating tests on 1 µm Ti-doped MoS_2 and Ti/Ta co-doped MoS_2 films deposited on 304 stainless steel by CFUBMSIP (Teer Coatings).
30 mN load with a 5 µm end radius spheroconical diamond probe. Track length = 40 µm; Scan velocity = 40 µm/s

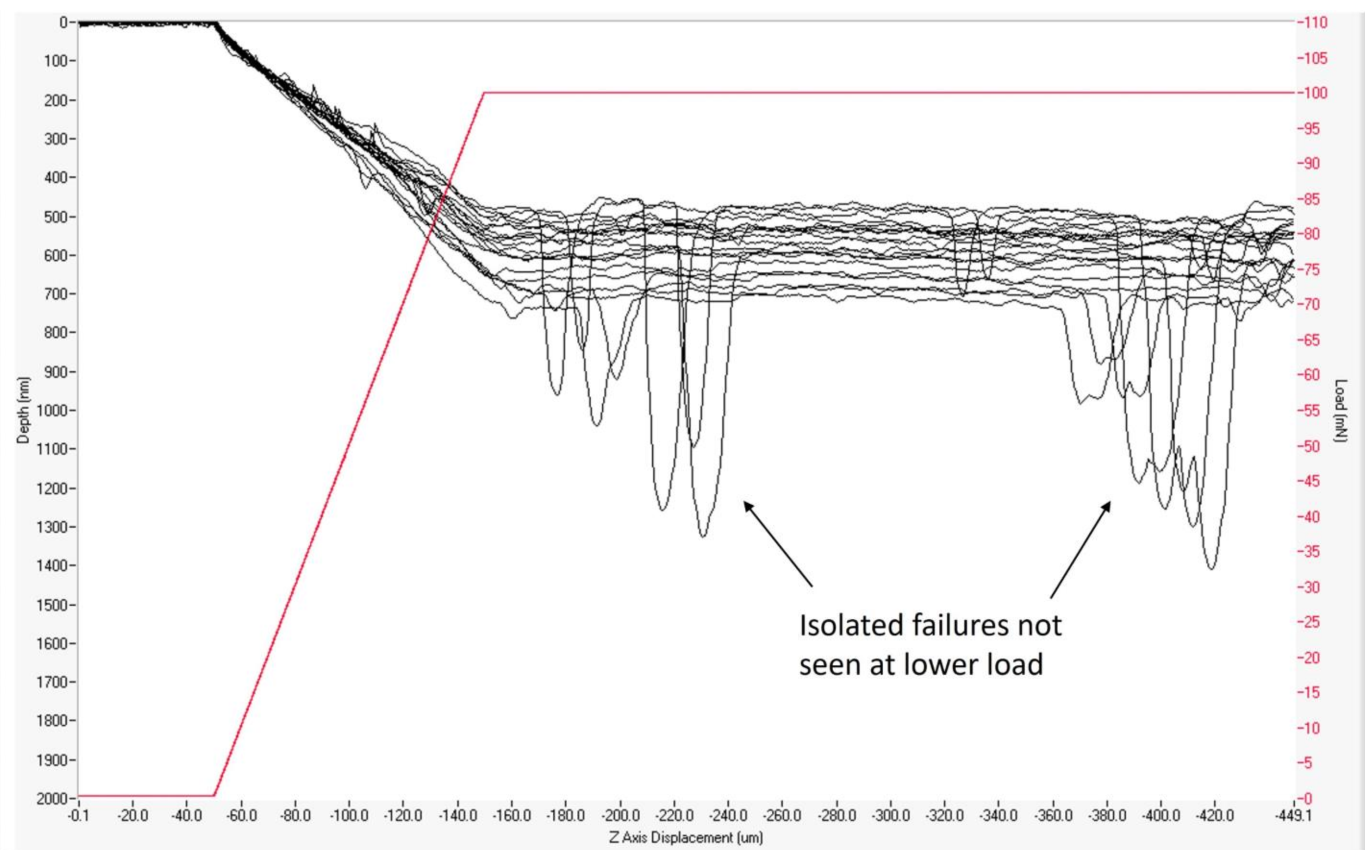
Simulating abrasion with more complex scratch tests

Although multiple scratch tests in the same track have been used to simulate abrasive wear they do not replicate the spatial distribution of contacts that occur in real abrasive wear. More complex high temperature scratch tests involving parallel scratches with controlled spatial distributions more closely the high temperature abrasive wear. An example definition screen is shown below.

Scan Offset Definition

Scan	Y	Z	Scan	Y	Z
1	0.0	0.0	11	-13.6	16.3
2	-2.2	10.6	12	5.3	6.2
3	15.5	-0.4	13	7.4	12.0
4	16.8	-15.8	14	-6.8	2.1
5	4.5	-2.9	15	-9.7	-6.9
6	-18.6	18.3	16	6.2	1.5
7	-2.5	-12.0	17	-2.7	9.2
8	18.9	7.4	18	19.7	-8.1
9	6.7	9.6	19	-19.8	14.4
10	-4.2	-4.3	20	16.5	-0.8

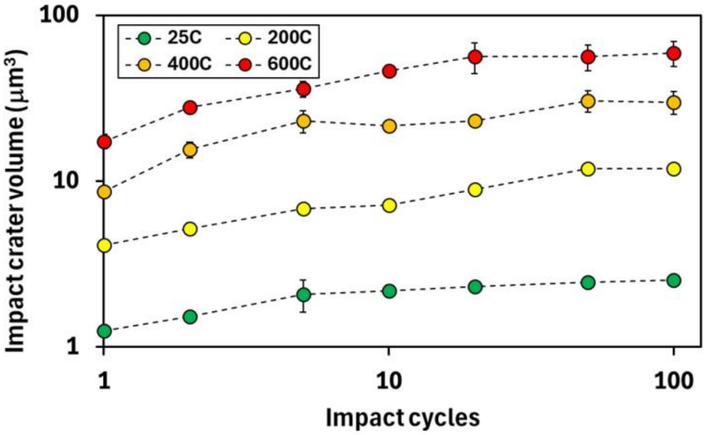
Definition screen from a 20-cycle spatially-distributed nano-scratch test. The positions of the scratches are defined by applying a chosen statistical distribution in the NanoTest Scratch module software.

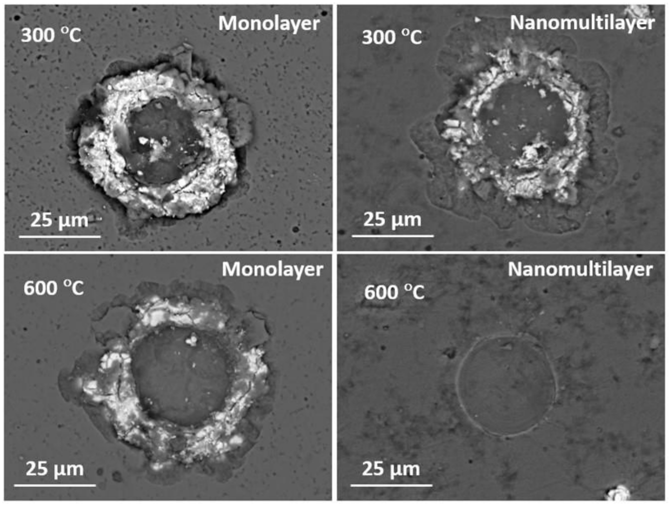


An example of a 100 mN test at 150 ℃ on a 1 µm thick Ti-doped MoS_2 coating using a 5 µm end radius spheroconical diamond probe. In similar tests at lower load there was minimal coating damage. At this relatively high load isolated failures occurred after 14 scratches but the coating remained intact over most of the track.

Cyclic impact

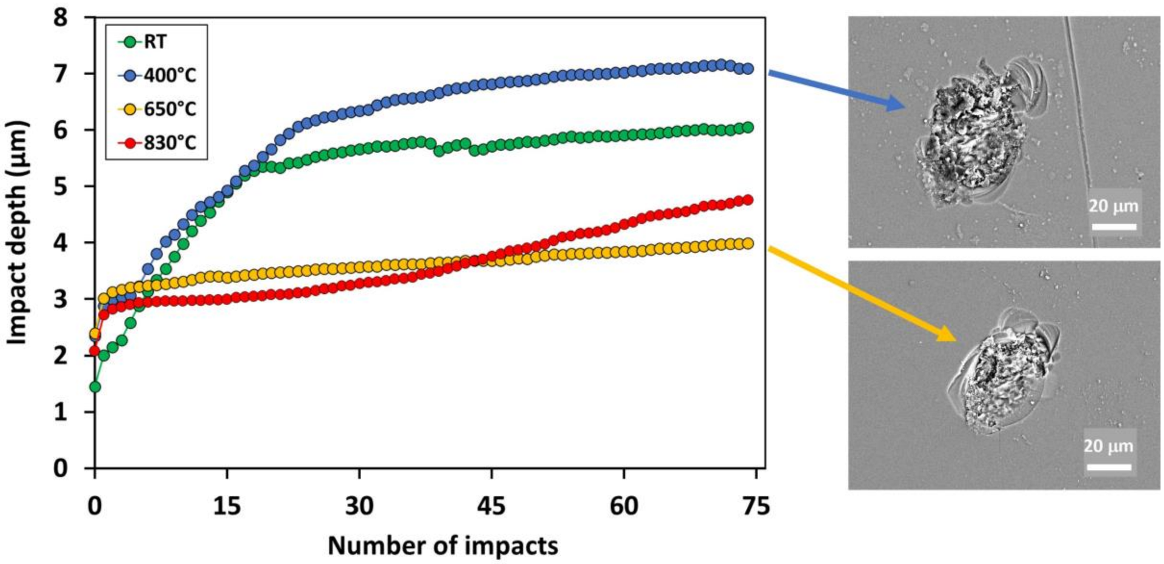
Mechanistic changes in deformation behaviour at high strain rate can be studied by single impact and cyclic (repetitive) impact tests. Typically there is a reduction in yield stress and greater plasticity at elevated temperature. This can result in more impact damage at elevated temperature – e.g. as in the tests on WC (below left [23]) or in some cases a reduction in fracture, (bottom right [24]).





Left – Temperature dependence of impact crater volume after cyclic nano-impact testing on WC [23]
Right – temperature dependence of crater morphology after cyclic micro-impact tests on monolayer TiAlSiN and nano-multilayered TiSiN/TiAlN coatings [24]

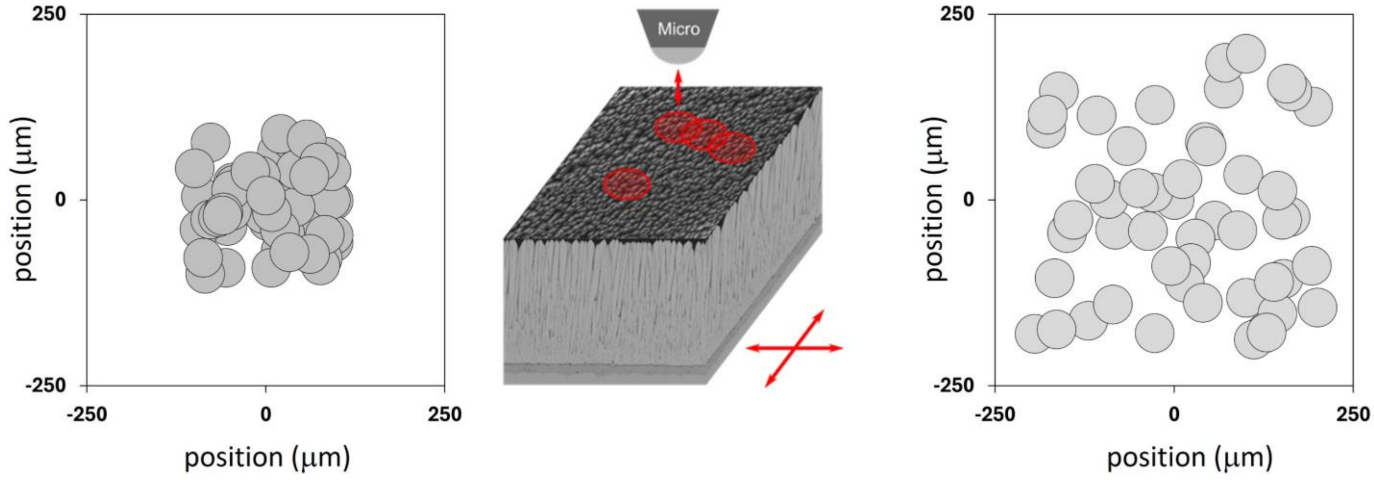
Monitoring the depth during the elevated temperature tests provides additional information about the deformation mechanism that may not be obvious from the final crater.



Cyclic micro-impact on fused silica. At 650 ℃ and above the glass structure weakens gradually with continued impact, in contrast to the more abrupt deformation observed at lower temperature.

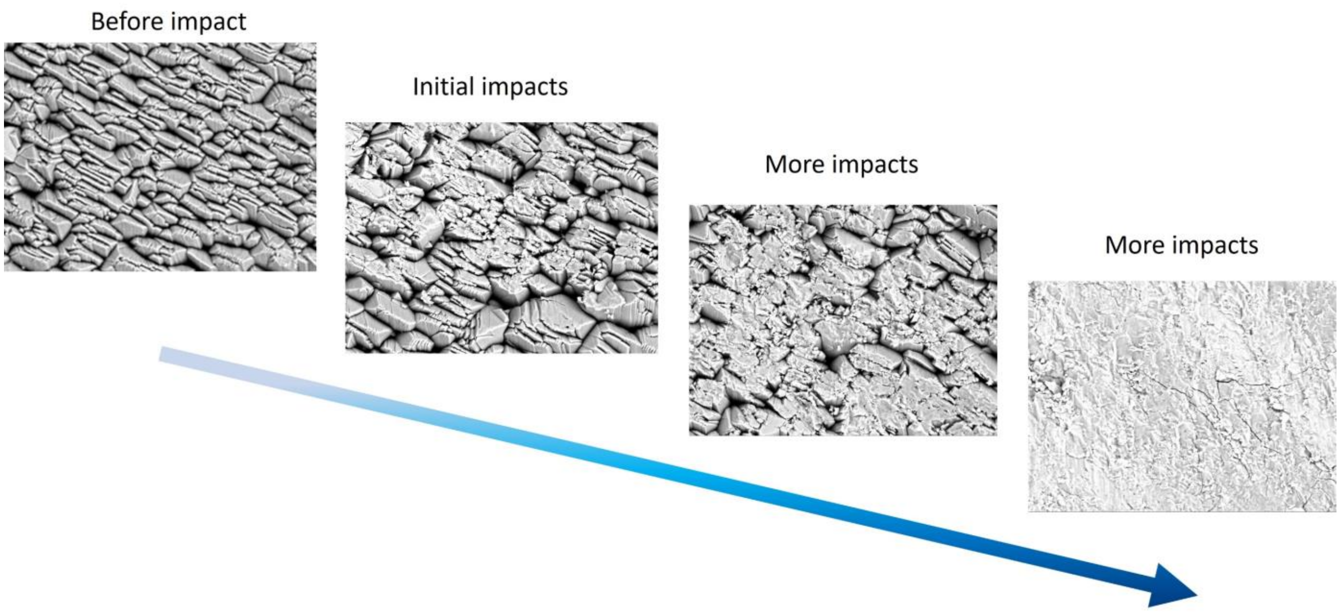
Simulating erosion with spatially distributed impact

In a standard cyclic impact test the repetitive contact occurs at the same point on the surface. A recently introduced novel test method for more closely replicating the spatial distribution of multiple impacts that occur in solid particle erosion is to perform cyclic impacts at different locations on the sample surface, where movement of the test instrument's sample stage between impacts enables each impact to be at a new position on the surface.



Examples of different spatial distributions of micro-scale impacts on a thermal barrier coating

To enable higher engine temperatures, thermal barrier coatings need to be developed with improved erosion resistance. To replicate high temperature erosion, spatially-distributed micro-impact tests have been performed on the thermal barrier coating 7YSZ at 850 °C as a baseline. Compaction dominated behaviour with some cracking was observed in these tests.



Influence of increasing number of impacts in spatially-distributed micro-impact tests at 850 °C

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Instrumentation specifications

Micro Materials manufacture two high temperature multifunctional nanomechanical test instruments:-

- NanoTest Vantage to 850 °C testing in air, argon or reducing atmosphere
- NanoTest Xtreme to >1000 °C testing under high vacuum

These instruments offer a wide combination of test modes, load ranges and multi-sensing capabilities.

With the additional of the integrated nanopositioning stage SPM topographic imaging and test targeting with nm precision becomes possible across the full temperature range.

Instruments can be configured as single- or dual- load head systems. In the dual-load head systems the low (0-500 mN) and high (0 – 30 N) load heads are always permanently mounted.

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