Developing advanced alloys with enhanced creep resistance at elevated temperatures

Micro Materials
NanoTest
XTREME
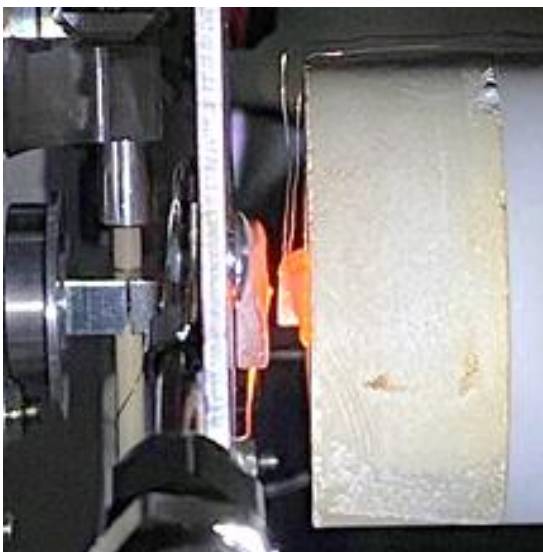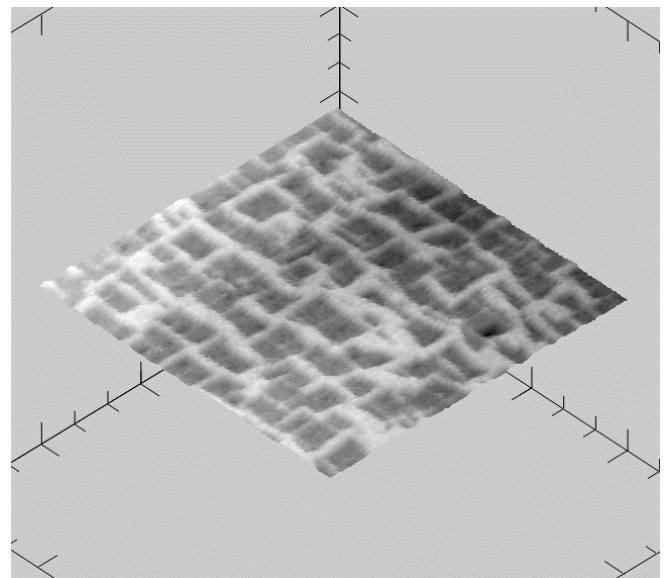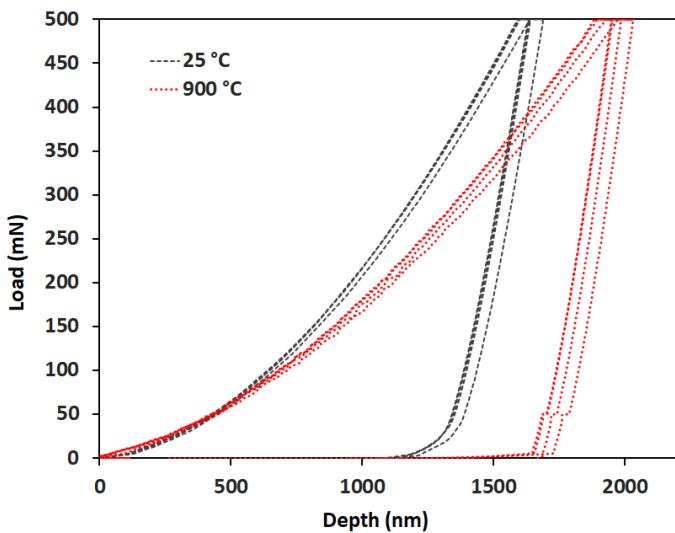
Introduction

To provide the most reliable, accurate measurement of mechanical properties and prediction of materials behaviour, researchers are increasingly demanding that test conditions closely mimic real-world environments.

The NanoTest Xtreme, with its active tip and sample heating, horizontal loading, highly localised heating and high vacuum environment enables users to reliably measure mechanical properties on the nano-scale as a function of temperature, free from the influence of thermal drift or oxidation.

Properties that can be measured up to 1000 °C include hardness, elastic modulus, creep, strain rate sensitivity, viscoelasticity, fracture toughness, yield stress, scratch resistance, coating adhesion, wear, fatigue resistance and impact resistance.

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.

The NanoTest Xtreme has been used to measure creep resistance in Ni-base superalloys to over 1000 °C. The figure below shows the creep stress exponents for CMSX-4 [1] and Nimonic 75 [2] obtained with sapphire indenters under high vacuum with the NanoTest Xtreme.
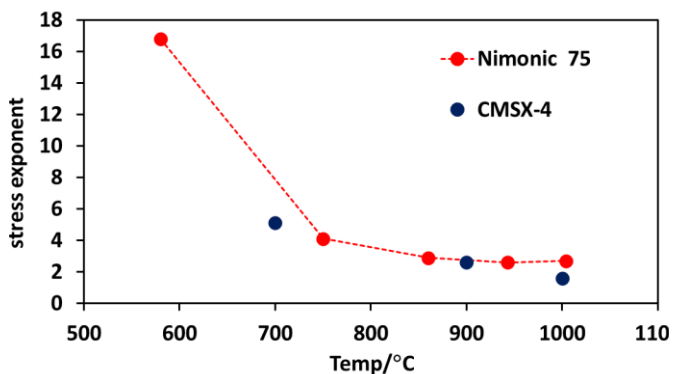

Figure 1 – creep stress exponents for CMSX-4 and Nimonic 75

Although these superalloys have high creep resistance at moderate temperatures there is a transition to much faster creep processes above 600 °C in Nimonic 75.

Other Ni-base alloys with potentially improved creep resistance are being investigated. A case study on one of these is shown below.

Indentation creep of 80Ni-3Ta-2Y-15Mo at 800 °C

Since the alloy sample [3] had high surface roughness with porosity the surface was imaged with the integrated SPM nanopositioner to select suitable porosity-free regions for indentation creep tests.
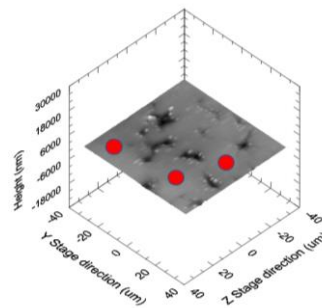

Figure 2 – quick SPM image to target suitable pore-free regions for testing

The procedure for the creep measurements at 800 °C was:
(i) pump down to high vacuum, heat to 800 °C, short stabilisation period
(ii) SPM nanopositioner 70 µm x 70 µm image size at 800 °C to select porosity-free regions for indentation
(iii) Creep testing (14 indents) = 10.5 hrs at 800 °C

The test conditions were:
- 500 mN peak load
- Sapphire Berkovich indenter
- 100 mN/s loading (5 s)
- 900 s dwell at 500 mN
- 50 mN/s unloading (10 s)
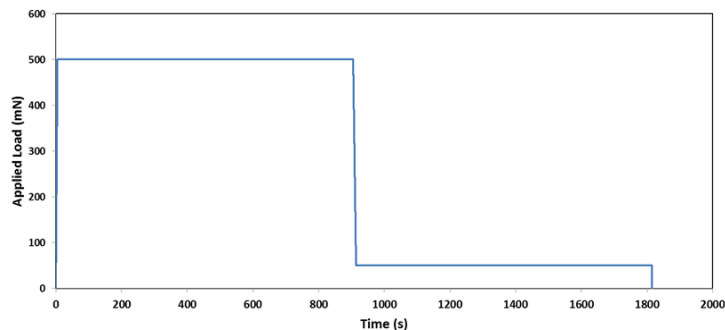- 900 s at 90% unload for thermal drift correction
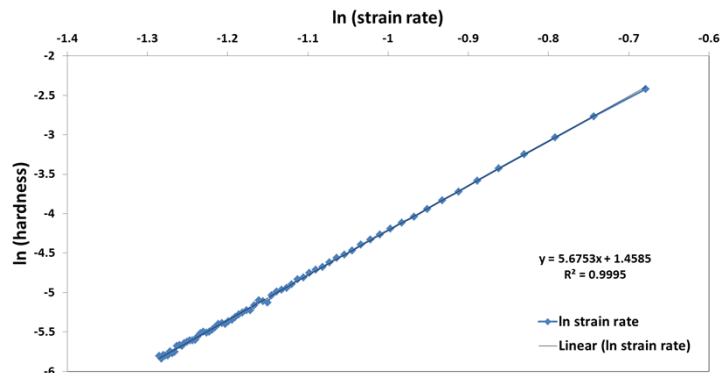

Figure 3 – load profile for creep test

Figure 4 – example creep stress exponent plot

Stress exponent	R² fit
7.07	0.9992
5.68	0.9995
7.07	0.9992
6.75	0.991
6.23	0.996
6.92	0.9994
7.09	0.999
6.85	0.998
6.96	0.997
5.78	0.9996
7.53	0.991
Mean = 6.71 ± 0.60	

The testing in vacuum with the NanoTest Xtreme did not result in any deterioration of the surface so SPM imaging and extended creep testing at 800 °C was possible. The low thermal drift at 800 °C allowed >30 min tests to be performed. Although the creep stress exponent of (6.7 ± 0.6) at 800 °C compares reasonably well with Ni-base superalloys there was a greater decrease in hardness for this alloy. Alternative approaches are therefore needed and two of these are described briefly below.

A refractory High Entropy Alloy with high creep resistance – MoNbTaVW

High-entropy alloys (HEAs) represent an innovative class of materials that have garnered significant attention recently. HEAs are characterized by their composition of multiple (usually at least 5) principal elements, each present in roughly equal proportions. This distinctive composition can create a unique microstructure leading to enhanced mechanical and thermal properties.

In the example below, high temperature nanoindentation was performed on a refractory high-entropy alloy (R-HEA) based on five principal elements (Mo, Nb, Ta, V, and W) to investigate the effect of temperature on hardness, modulus and creep resistance [4].
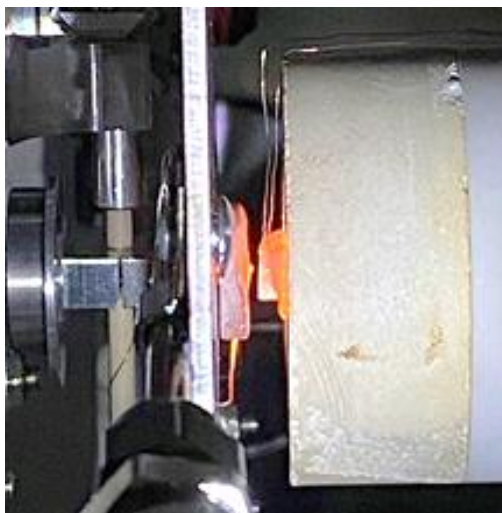

Figure 5 – sample and indenter at 900 °C on the NanoTest Xtreme

The R-HEA sample was secured to the high temperature sample mount using a thermally conductive cement. A heated cubic boron nitride Berkovich indenter was used since active tip heating is essential to minimise thermal drift and ensure measurement reliability.

500 mN indentations were performed at 25, 300, 500, 700 and 900 °C, each with 10 s loading time, 5 s unload and 10 – 20 s creep hold period. Reference indentations were performed on a fused silica sample before and after the R-HEA tests to determine the indenter area function and to confirm that the tip geometry did not change during the high temperature tests.
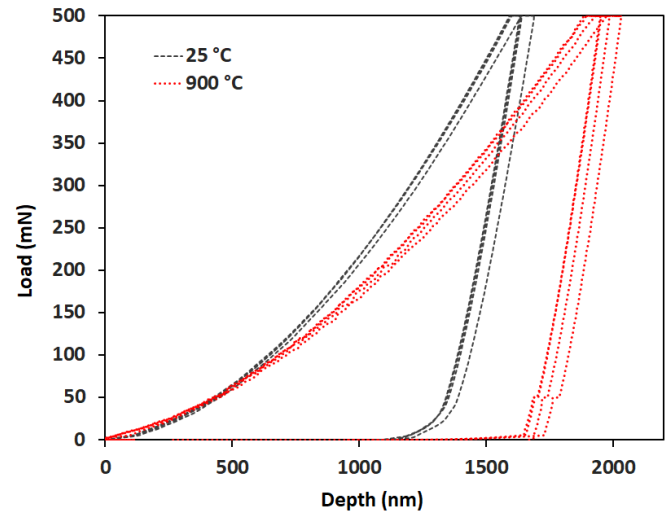

Figure 6 – 500 mN indentations at 25 °C and 900 °C
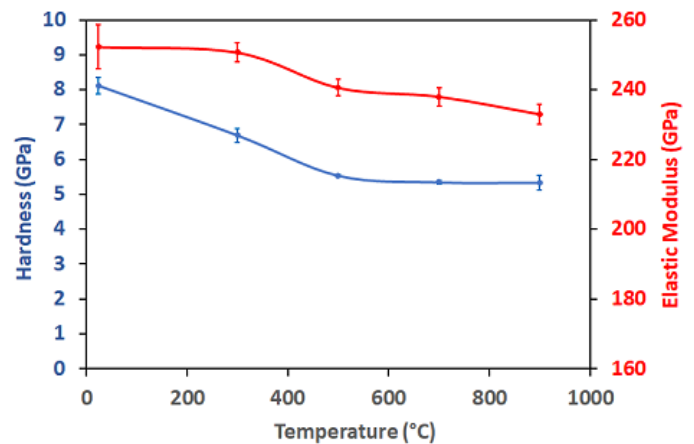
Figure 7 – hardness and modulus vs test temperature

The R-HEA showed excellent creep resistance with stress exponents around 80 at 900 °C. The dependence of hardness on temperature follows a similar general trend to that found elsewhere, e.g. for an oxide-dispersion strengthened steel [5] and the refractory metal tungsten [6].

The very low standard deviation in the data – even at the highest test temperatures – is indicative of inherent stability of the NanoTest Xtreme.

Improved high temperature creep resistance with stable grain boundary networks in plastically strained nano-grained NiCoCr

In a recent paper published in Science [7], Zhang and colleagues adopted a different strategy to inhibit high temperature creep by using stable networks of grain boundaries. These were obtained by plastic strain of a commercial single-phase NiCoCr (34.1Ni-33.9Co-20.9Cr-10.2Mo-0.9Ti). Introducing the stable grain boundary network by the plastic straining into a nanograined medium-entropy alloy improved its creep resistance.

The NanoTest was used to test the creep resistance of coarse-grained, nano-grained and grain-boundary network samples to 750 °C, with a 600 s hold at peak load for creep analysis. It was shown that although the nano-grained and coarse-grained samples showed some creep at 700 °C, the grain-boundary network sample showed extremely low steady-state creep rates (10^{-7} s^{-1}) with stress exponents of >100 at 750 °C (T/T_m >0.6). Grain size distribution analysis showed that there was an inherent stability against grain coarsening due to the grain-boundary network with numerous twin boundaries and stacking faults [4].

Contrary to general assumption that smaller grains lower creep resistance, it appears that the grain-boundary network was able to effectively suppress diffusional creep processes under gigapascal stresses, at least to above T/T_m >0.6.

Conclusions

The NanoTest Xtreme is proving a highly effective tool for measuring the creep resistance of advanced alloys at elevated temperatures. Key benefits of the Xtreme include (i) inherently high thermal stability – enabling long duration creep tests (ii) high vacuum environment to avoid sample oxidation (iii) wide load range so that tests can be performed either at low load in individual phases, or at higher load to obtain representative behaviour, as required.

NanoTest advantages for high temperature creep testing

The NanoTest Vantage and Xtreme have the necessary ultra-low thermal drift for creep testing due to design advantages including:

1. Active tip heating – the indenter and the sample are both actively and independently heated, resulting in an isothermal contact before the experiment begins.
2. Horizontal loading – the unique load head configuration of the NanoTest systems means that there is no heat flow onto the loading head or depth measurement sensor.
3. Highly localised heating – a heat shield and insulating shroud around the heated zone ensures instrument stability during high temperature experiments.
4. Patented control protocol – software routines are used to precisely match the indenter and stage temperatures to 0.1 °C.

References and Acknowledgements

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

[2] Micro-impact tests of novel thermal barrier coating systems and >1000 °C nanoindentation of Ni-base superalloy, B.D. Beake, S.R. Goodes, AJ. Harris, L. Isern, C. Chalk, J.R. Nicholls, H. Zhang, M.G. Gee, ICMCTF, San Diego (2023).

[3] Sample provision from Lehigh University is acknowledged.

[4] Sample provision from Indian Institute of Technology Kanpur is acknowledged.

[5] Z. Huang, A.J. Harris, S.A. Maloy, P Hosemann, Nanoindentation creep study on an ion beam irradiated ODS alloy, J. Nucl. Mater. 451 (2014) 162-167.

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

[7] B.B. Zhang, Y.G. Tang, Q.S. Mei, X.Y. Li, K. Lu, Inhibiting creep in nanograined alloys with stable grain boundary networks, Science 378 (2022) 659-663.

Contact Us

Micro Materials Ltd
Willow House, Yale Business Village,
Ellice Way, Wrexham, LL13 7YL, UK

Tel: +44(0) 1978 261615
E-mail: info@micromaterials.co.uk
www.micromaterials.co.uk