Nanoindentation creep testing

Introduction

Nanoindentation provides a unique opportunity to probe the creep response of individual components or phases in complex microstructural materials that are not possible with conventional bulk testing techniques. Elevated temperature creep resistance is important in many applications of high temperature materials. Understanding the fundamental relationship between creep and microstructure is a route to developing improved materials with enhanced stability at elevated temperatures.

To measure indentation creep accurately it is essential that the test instrument displays ultra-low thermal drift across the entire measurement range. The NanoTest Vantage (-25 °C to 850 °C) and NanoTest Xtreme (-50 °C to 1000 °C) display industry-leading thermal stability in conjunction with the flexibility to measure the widest range of sample geometries.

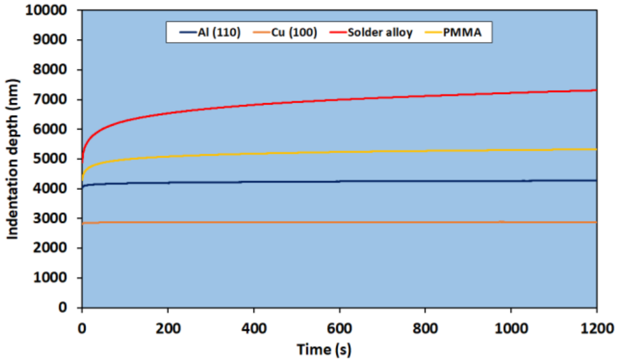

Figure 1. 1200 s tests at 100 mN with a Berkovich indenter.

NanoTest systems have the stability for reliable creep measurements over longer experiment durations, and under different environmental conditions (e.g. hot/cold/variable humidity etc) [1-7].

The NanoTest configuration for enabling isothermal contact is shown in the figure below.

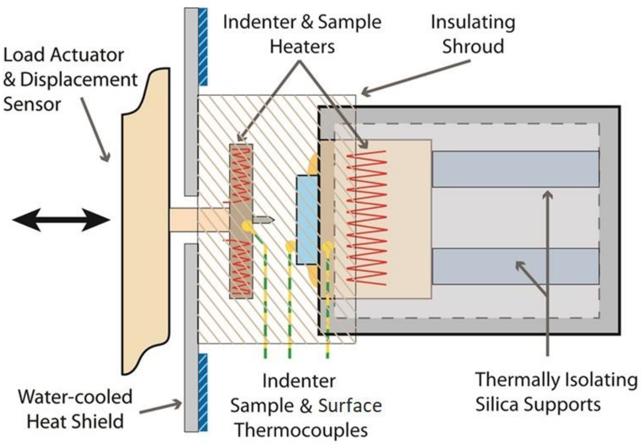

Figure 2. NanoTest high temperature testing configuration

In this Technical Note we show examples of where nanoindentation tests have been performed to 750 °C (solid oxide fuel cell materials), 950 °C (refractory metals), 1000 °C (Ni-based superalloys) together with testing of materials (polymeric materials, fcc metals, solder alloys) that are more strongly rate-sensitive at room temperature.

Applications include

- Development of novel high temperature creep resistant materials
- Creep behaviour of specific phases or microstructural elements
- Reliability of lead-free solders
- Time-dependent behaviour in viscoelastic materials
- Spatially resolved measurements of creep resistance across welded components

Elevated temperature creep testing

Solid Oxide Fuel Cell Materials

High temperature nanoindentation creep tests have been performed on a barium calcium aluminosilicate glass–ceramic (G18) used for seals that separate the fuel and air sides of a solid oxide fuel cell [2]. The glass transition temperature for this material was 620 °C. Creep of the seal at the operating temperature of 800 °C can lead to early failure of the cell. Using a cubic boron nitride indenter the influence of a thermal "pre-ageing" process of 4 or 100 h at 800 °C was studied.

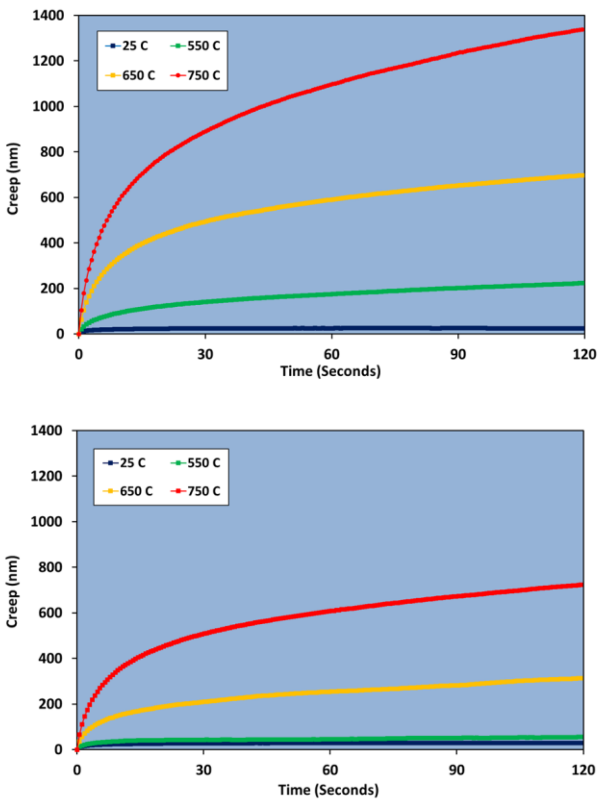

Figure 3. Indentation creep for G18 after 4h (top) and 100 h (bottom) thermal pre-ageing treatment

Marked differences in indentation creep resistance were observed from 550 °C (approaching its glass transition temperature). The creep resistance of the sample pre-aged for 100 h at 550 °C was only slightly worse than at room temperature. As the test temperature increased, both 4 h and 100 h samples showed more time-dependent deformation, although this was relatively lower after the longer duration of pre-ageing.

Weld and parent material for P91 steel

Nanoindentation can reveal the temperature dependence of the creep exponent for the process governing plastic flow providing evidence for changing dominant mechanisms of plasticity with temperature since their magnitudes vary strongly for different rate limiting processes. Measurements were made in the parent and weld regions of P91, a high Cr content creep resistant steel used in steam pipes in power plants at its operating temperature of 650 °C [3]. In the 300 s tests the maximum indentation loads were sufficiently high (≥ 100 mN) to encompass several grains of the steel and be representative of "bulk" behaviour for the region of interest. The measurements were with a cBN Berkovich indenter in an Argon gas purging environment. Stress exponents were determined from ln(strain rate) vs. ln(stress) graphs.

	Test performed at 650 °C	Stress exponent
Parent material	Nanoindentation (100 mN)	8.95 ± 0.47
	Nanoindentation (200 mN)	7.89 ± 0.41
	Uniaxial tensile test	8.46
Weld material	Nanoindentation (100 mN)	7.49 ± 0.45
	Nanoindentation (200 mN)	7.44 ± 0.35
	Uniaxial tensile test	7.22

Measurements of stress exponents agree with those determined from uniaxial tensile tests on bulk specimens.

Superalloys and bond coats

Creep exponents were determined [4] at operationally-relevant temperatures on a sample of the Ni-base superalloy CMSX-4 and a 200 μm thick NiCoCrAlY bond coat (Amdry-386). Nanoindentation measurements were performed in high vacuum to a depth of 1 μm at 1000 °C with an actively heated sapphire indenter.

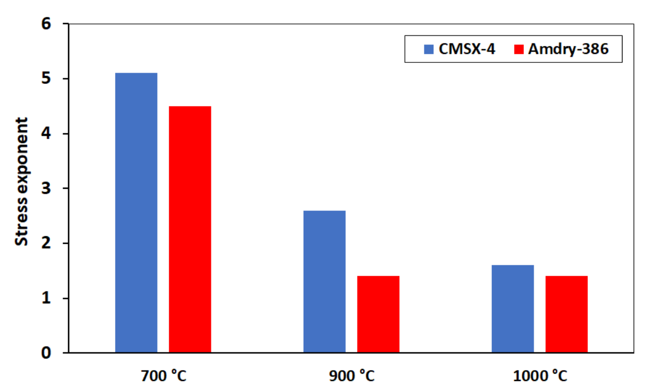

Figure 4. Superalloy and bond coat stress exponents

The temperature dependence of stress exponents matched that of the literature values between 700-1000 °C. For the bond coat there was good agreement between the measured values and those obtained by tensile testing [4].

Nanoindentation creep testing as a tool for probing chain constraint in polymer nanocomposites

Extensive research into the performance of polymer-clay nanocomposites has gradually recognized the strength and weakness of these materials. In terms of stiffness, they have advantages over traditional micro-sized fibre and filler technology at low filler contents but at higher filler loading the efficiency of reinforcement in the polymer-clay nanocomposite rapidly declines due to difficulties in exfoliating the multi-layered structure of clay and the onset of filler agglomeration.

Despite much research it is not yet clear to what extent the improvements in stiffness are simply due to the incorporation of a much stiffer clay filler, or if the filler improves the stiffness and creep resistance by interaction with the polymer matrix exerting a constraining effect on the polymer chains.

Therefore, Chen and co-workers [5] studied nylon 6-clay nanocomposites (NC) with different filler loading produced by melt compounding and compared their properties to model nylon 6 submicro composites (SMC) reinforced by submicro-thick silica flakes in which polymer chain constraint cannot occur due to the difference in filler geometry. Both processing routes produced composites that were stiffer than the base nylon polymer, with the nanocomposites showing higher elastic modulus for a given wt.% filler.

The extent of polymer chain constraint was assessed by the analysis of nanoindentation creep data from a 120 s hold at the 10 mN peak load. The NC samples showed improved creep resistance for all filler loadings.

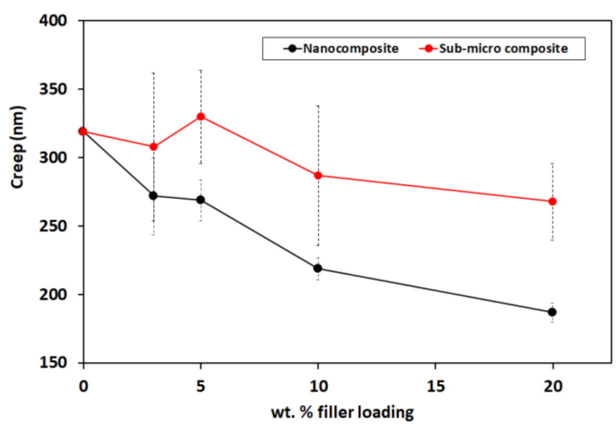

Figure 5. Total creep during 120 s at 10 mN.

Strain rate sensitivity analysis and creep compliance analysis of the creep data revealed clearly that the time-dependent deformation in the NC decreased with filler loading consistent with the clay platelets exerting a constraint effect on the polymer chains which increased with filler loading.

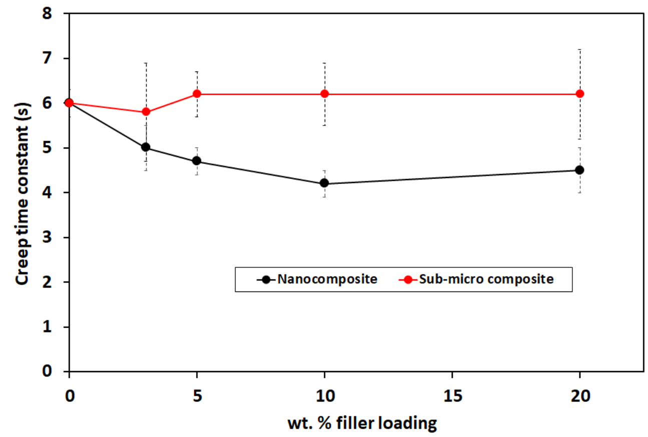

Figure 6. The creep time constant changes with filler loading for the NC.

In sharp contrast, on the SMC samples there was no evidence any reduced time-dependent creep or changing strain rate sensitivity, consistent with a lack of constraint expected from the much lower aspect ratio of the silica flakes. The methodology applied in this study provided strong evidence that constraint does occur in nylon 6-clay nanocomposites.

Due to the high stability of the NanoTest it is easily applicable to the optimisation of other systems such as CNT/polymer composites.

The indentation creep behaviour of polymers and polymer nanocomposites can be highly sensitive to the test environment conditions (hot/cold/moisture) [6].

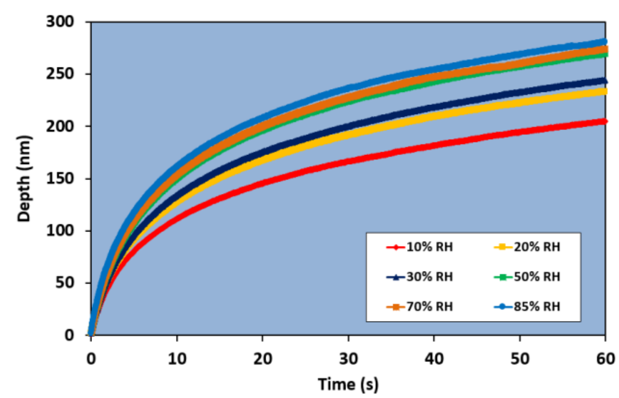

Figure 7. Changing creep resistance with humidity for nylon-clay nanocomposite material

Elevated temperature testing of refractory metals

Tungsten and its alloys are being considered as the main plasma-facing material in a fusion reactor. The NanoTest Xtreme has been used to test the mechanical properties of tungsten in high vacuum at 25-950 °C [7]. Testing under high vacuum was essential as tungsten oxidises rapidly at >500 °C in air.

With thermal drift at 750-950 °C typically as low as 0.05 nm/s the NanoTest has the stability to run longer duration indentation creep tests throughout the temperature range. Greater time-dependent deformation was observed starting from 850 °C.

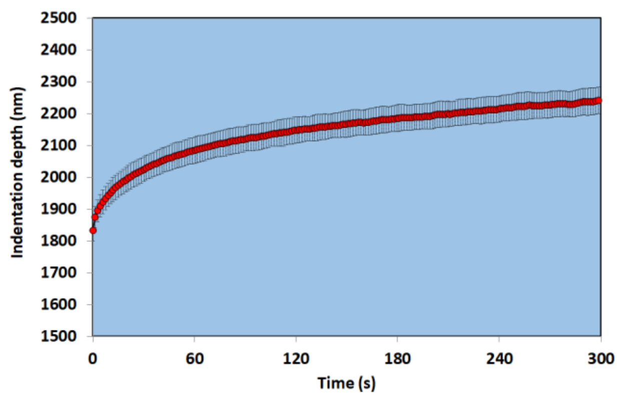

Figure 8. Nanoindentation creep on polycrystalline tungsten at 945 °C at 200 mN with a cBN Berkovich indenter under high vacuum.

NanoTest Vantage Specifications

High stiffness granite frame. Ultra-low thermal drift (~0.004 nm/s) provides the stability for long-duration creep tests.

NanoTest Xtreme Specifications

NanoTest Xtreme thermal drift rates as low as 0.01 nm/s in vacuum at 750 °C and 0.05 nm/s at 950 °C.

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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

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