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High temperature nanoindentation –Publications in 2011

2011 proved that high temperature nanoindentation continues to be a huge growth area in materials science. The operation and function of the NanoTest instrument from Micro Materials Ltd (MML) at temperatures up to 750°C is now well established and recognised, meaning that the challenge to the nanomechanical community is no longer in the acquisition of reliable data, but in the interpretation of the resulting data produced.

2011 saw the publication of some seminal papers in the area of high temperature nanoindentation. This article will offer a summary of selected work from users of the MML NanoTest system, which was the only instrument to produce publications featuring data above 200°C. This article will précis work carried out at temperatures above 600°C only.

High temperature nanoindentation – the importance of isothermal contact

N.M. Everitt, M.I. Davies & J.F. Smith

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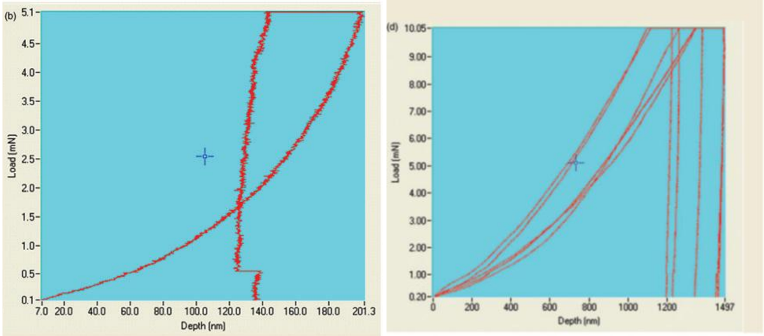
A key issue in high temperature nanoindentation is instrument stability, and the need to minimise drift during testing. This is important for accuracy of hardness and modulus data, and even more so for long-duration creep data.

A major focus area in the past few years has been the assessment of heat flow and stability during the indent itself, when the indenter material is brought into contact with the sample. It makes logical sense that the diamond should be heated as well as the sample in order to ensure isothermal contact and prevent unwanted system instability, and this paper demonstrates this.

Finite element analysis modelling was used to give a qualitative view of how the thermal picture develops under a diamond indenter without controlled heating of the diamond. In the case of a low-conductivity sample such as fused silica, the thermal gradient below the indenter tip can be relatively insignificant, whereas with a high-conductivity sample such as gold, only a small region of the sample reaches thermal equilibrium with the tip. As a result, a very steep thermal gradient is created in the sample.

Such a thermal gradient will result in heat flow between the indenter and sample as soon as the indenter moves into the sample, causing unwanted contraction/ expansion of both during indentation, and thus inaccuracy in measurement.

The results of the model were validated by comparing results obtained by heating the indenter either indirectly by contact with the sample or utilising a separate heater for the indenter (an isothermal contact method).



Figures 1a (left) and 1b (right) demonstrating the need for tip heating.

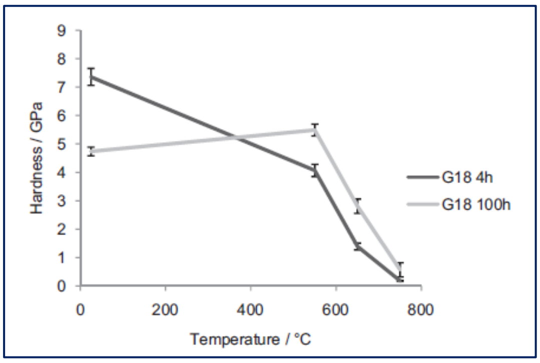
Figures1a (left) shows a nanoindentation curve acquired on a gold sample at 300°C, using a method where the heater is indirectly heated by prolonged contact with the sample prior to indentation. The curve appears to exhibit negative creep, with the unloading curve crossing the loading curve. This is as a result of instrument drift. Figure 1b shows how this can be avoided by heating the tip separately so that contact is isothermal.

Nanoindentation results were presented for experiments on fused silica at temperatures up to 600°C, and annealed gold at temperatures up to 300°C. The results showed that indentation without separate indenter heating tended to produce unacceptable thermal perturbation in the system, whereas the isothermal contact method maintained acceptable thermal drift and produced values of modulus and hardness that compared well with those in the literature.

Mechanical properties of solid oxide fuel cell glass-ceramic seal at high temperatures

J. Milhans, D. Li et al

This group at Georgia Tech recently published NanoTest data describing the mechanical properties of solid oxide fuel cell glass-ceramic seal material, G18. Hardness, modulus and creep properties were investigated via depth-sensing nanoindentation at room temperature, and then at temperatures of 550, 650 and 750°C.

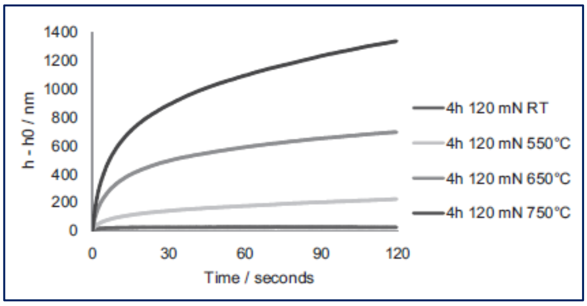


Results showed a decrease in reduced modulus with increasing temperature, with significant decrease above the glass transition temperature, while hardness generally decreased with increasing temperature (Fig 2).

Fig 2: Hardness measurements show that aging the G18 sample for longer improved stability.

Creep data acquired over 120s at a maximum load of 120mN showed that creep increased with increasing temperature, but then decreased with further aging (Fig 3).

Fig 3: High temperature creep data for G18 aged for 4 hours



The tip heating used by the NanoTest ensures excellent instrument stability even at these very high temperatures, allowing such creep data to be acquired.

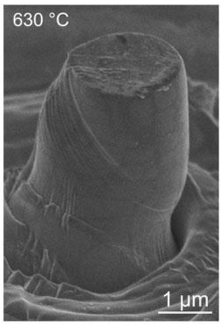
High temperature microcompression and nanoindentation in vacuum

S. Korte, R.J. Stearn, J. Wheeler, W.J. Clegg

Nanoindentation is now commonly used as a method of studying micropillar compression.

At elevated temperatures there is sometimes the need to test in an inert environment so as to minimise oxidation effects. Furthermore, impurities in inert gases can pose problems so that testing in vacuum is desirable. NanoTest users in Cambridge have modified their instrument to allow it to be used in a vacuum chamber, allowing high temperature nanoindentation in a vacuum environment.

By carefully controlling the temperatures of the indenter tip and the sample, the group were able to carry out flat punch indentations of gold, a good thermal conductor, over several minutes at 665 °C in vacuum.



This tip heating capability also permitted thermal stability to be quickly re-established in site-specific microcompression experiments. This allowed compression of nickel superalloy micropillars up to sample temperatures of 630°C with very low levels of oxidation after 48 h. Furthermore, the measured Young moduli, yield and flow stresses were consistent with literature data.

NanoTest capabilities that made this work possible:
The MML NanoTest uses a unique horizontal loading mechanism, meaning electronics and measurement hardware are free from the influence of heat convection. This, combined with the separate heating of both sample and indenter, ensure makes the NanoTest stand out as the only option for high temperature measurements. Patented PID loop control of the heating mechanisms ensures excellent temperature stability, thus long duration creep tests.

References:

N.M. Everitt, M.I. Davies & J.F. Smith (2011): High temperature nanoindentation – the importance of isothermal contact, **Philosophical Magazine, 91:7-9, 1221-1244**

J. Milhans, D. Li, M. Khaleel, X. Sun, M. Al-Haik, A. Harris, H. Garmestani: Mechanical properties of solid oxide fuel cell glass-ceramic seal at high temperatures **Journal of Power Sources, 196(13):5599-5603 volume 196 issue 13**

S. Korte, R. J. Stearn, J.M. Wheeler and W.J. Clegg: High temperature microcompression and nanoindentation in vacuum **Journal of Materials Research, Available on CJO 14 September 2011 doi:10.1557/jmr.2011.268**

Established in 1988, Micro Materials Ltd are manufacturers of the innovative NanoTest system, which offers unique nanomechanical test capability to materials researchers for the characterisation and optimisation of thin films, coatings and bulk materials. The current model, the NanoTest Vantage was launched on June 1st 2010.

Further information on the NanoTest Vantage can be found at
www.micromaterials.co.uk