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Recent publications highlight unique NanoTest capabilities

The wide range of capability offered by the MML NanoTest system means that the system is active in a wide range of application areas. The following are examples of recent work published by the MML user base. Full references for each paper discussed are given at the end of the article.

Micropillar compression of ceramics at elevated temperatures

Sandra Korte and W.J. Clegg, University of Cambridge, UK

This paper describes micropillar compression tests carried out using a flat punch indenter mounted on an MML NanoTest system. Tests were carried out on $MgAl_2O_4$ spinel, at a range of temperatures from room temperature to 400°C. The spinel deformed plastically at 200°C and above without a confining pressure. The yield stresses measured were consistent with those obtained elsewhere by compressing larger crystals under a high confining pressure, suggesting that micropillar compression has the potential to be a useful technique for studying plastic flow in brittle materials.

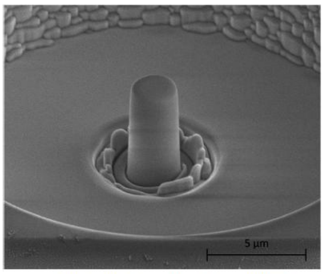

Figure 1: Pillar after focused ion beam milling prior to deformation, viewed at an angle of 52°.

Compression of such micropillars requires an extremely stable system. The NanoTest ensured this stability at high temperature by using heaters in both the sample stage and diamond tip. This minimises thermal drift, thereby allowing micro-compression tests to be carried out at reasonably low strain rates.

The authors state 'Both sample and tip heaters are required to minimize thermal drift … In both cases the rate was constant over the 20s period. The values obtained were 0.15 and 0.20 nm s^{-1} at 200 and 400°C respectively.'

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. PID loop control of both indenter and sample heating ensure excellent temperature stability, thus long duration creep tests.

Probing mechanical properties of fully hydrated gels and biological tissues

Georgios Constantinides[1], Z Ilke Kalcioglu[1], Meredith McFarland[1], James F Smith[2], Krystyn J. Van Vliet[1].
[1]MIT, Boston, US
[2]Micro Materials Ltd, Wrexham, UK

This innovative paper from NanoTest users at MIT details the work they have done in conjunction with Dr Jim Smith of MML to validate a liquid cell which enables nano- to micro-scale indentation of fully immersed, hydrated polymers and tissues. This is done while maintaining the high precision force and displacement signals normally acquired with the NanoTest.

The group state, '*We anticipate that the platform will enable quantitative studies of tissue mechanics as a function of source, disease state, and exposure to soluble toxins or adjuvants, particularly as a function of submicrometer position within the tissue*'

A range of hydrated gels and biological tissues were investigated using the cell, and the accuracy of measurement was validated using viscoelastoplastic analysis of relatively stiff, water-insensitive materials.

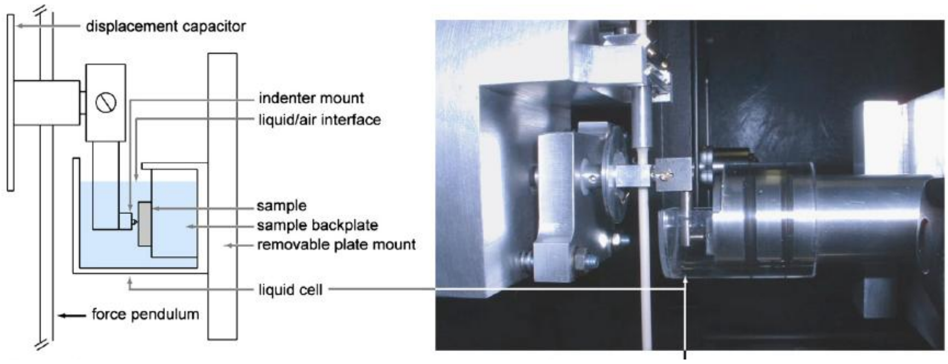

Figure 2: NanoTest configured for indentation in liquid, obviating vertical loading effects such as buoyancy and surface tension.

<u>NanoTest capabilities that made this work possible:</u>

The authors highlight the advantages of the NanoTest's horizontal-loading configuration (shown above) which, in the words of the authors, 'Confers significant advantages for indentation in liquids, obviating vertical-loading effects due to variation in buoyancy and surface tension during vertical indenter displacement through liquid. In absolute terms such forces are quite small, but can be significant for the nanoscale forces and/or displacements relevant to compliant hydrogels (Kaufman et al., 2008) or tissues.'

<u>The work demonstrates clearly that the NanoTest has the required sensitivity and repeatability to measure elastic properties of synthetic gels/biological tissues in a liquid environment.</u>

<u>An investigation into the effect of film thickness on nanowear with amorphous carbon-based coatings.</u>

G.M. Wilson and J.L. Sullivan, Aston University, UK

Aston University
Birmingham

NanoTest users at Aston University used the newly developed 'nanofretting' capability to perform nano-scale wear testing on a series of sputtered Cr doped amorphous C films.

The authors explain the background behind their work as follows 'It is expected that carbon-based coatings may provide solutions to tribology issues associated with micro-scale devices involving moving mechanical arrays (MMAs), such as MEMS and other microscale engineering systems, due to the high hardness, high elastic modulus and chemical inertness of the films. These types of applications will require a low friction/wear carbon-based film of just a few nm or tens of nm thickness, operating under conditions of low contact pressure, low contact area, with small displacements.' These low pressure, low contact area conditions are perfectly replicated using the MML nanofretting option.

The authors expand further to state 'It was shown that mean contact pressures, contact radii and displacements relevant to MEMS and micro-scale engineering devices could be achieved over hundreds of thousands of oscillation cycles using a spherical ruby counter-body. Contact pressures of 60–2200MPa; contact radii of 2–7μm and displacements of 2–14μm were employed in this initial investigation.'

The results section state 'There was a clear exponential reduction of specific wear rate with reducing film thickness over the range of 2000–10 nm.'
The authors state that 'such a clear relationship with quantified wear data as shown here has not previously been reported under conditions relevant to MEMS devices.' Furthermore 'a rapid reduction of specific wear rate (defined as volume of worn material per unit applied load per unit of slid distance) was observed during the first 3000 oscillation cycles. This was analogous to the 'running in' process observed with macroscopic tribology systems.'

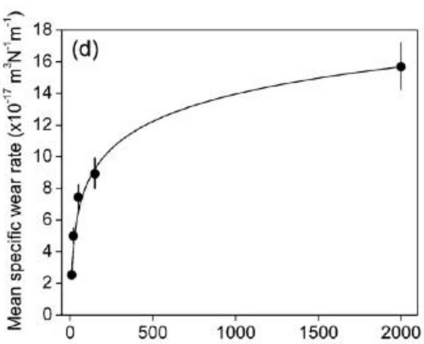

Figure 3: Exponential reduction of specific wear rate with reducing film thickness over the range of 2000–10 nm.

<u>NanoTest capabilities that made this work possible:</u>
MML offer a newly developed nanofretting option, which allows oscillation of sample whilst applying a known load. The user can vary both the frequency and amplitude of sample oscillation.
The extremely low thermal drift experienced by the NanoTest allows long duration wear tests such as these to be carried out with no need for drift corrections.
Various experiments may be scheduled to run automatically, ensuring best use of system time.

Coating optimisation for high speed machining with advanced nanomechanical test methods.

B.D. Beake[1], G.S. Fox-Rabinovich[2], S.C. Veldhuis[2], S.R. Goodes[1],
[1]Micro Materials Ltd, UK
[2]Mc Master University, Canada

This paper describes how advanced nanomechanical testing has been used to evaluate key factors influencing tool life. These tests include:
(1) a plasticity index at room and elevated temperature
(2) high temperature indentation
(3) fatigue fracture resistance

Results presented show that elevated temperature nanoindentation showed decreasing hardness and increasing plasticity index with temperature. In high-speed turning hot hardness is the dominant factor, whilst for interrupted cutting high hot hardness should be combined with improved plasticity for longer tool life. A novel test technique nano-impact, which is patented to MML, was used to simulate the interrupted contact.

Elevated temperature mechanical properties were determined from nanoindentation to 25–60 mN at 20–500 °C.

From a combination of nanoindentation measurements and cutting tool tests on ≥3 μm hard PVD coatings onWC–Co substrates the authors showed a correlation between the plasticity index and tool life for the coatings studied. Furthermore, the elevated temperature data acquired revealed a range of coating mechanical behaviour at temperature that shows correlation with tool life.

Nano-impact, a novel test technique was used to simulate the interrupted contact (and cyclic loading) conditions occurring in milling applications and evaluate the fatigue fracture resistance of coated tools. It was reported that tests (at both room and elevated temperature) show correlation with tool life in end and face milling.

The authors conclude 'the results from the various advanced nanomechanical tests … can provide compelling data that correlate with tool life. With care, the techniques described can be used to aid reliable prediction of which coatings have longer life in severe cutting conditions.'

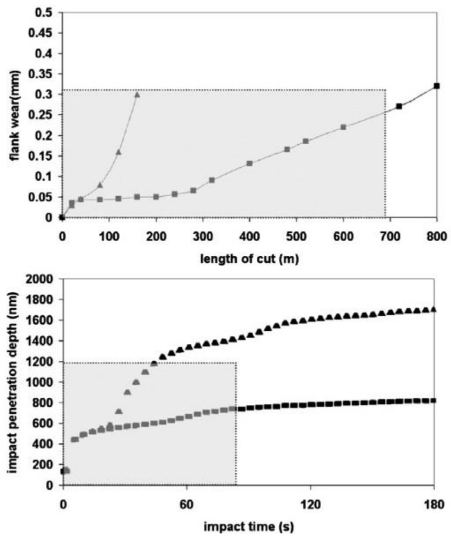

Figure 4: Clear correlation between end milling tests (top) with nano impact results (bottom) is shown.

NanoTest capabilities that made this work possible:
Elevated temperature nanoindentation can be carried out with excellent levels of system stability, due to the fact that both indenter and sample are heated to ensure isothermal contact. This work involved use of the unique NanoTest nano-impact technique, which is subject to an MML patent. These two unique capabilities can be combined to allow the user to carry out elevated temperature nano-impact.

References:

Micropillar compression of ceramics at elevated temperatures. Sandra Korte and W.J. Clegg **Scripta Materialia** 60 (2009) 807-810

Probing mechanical properties of fully hydrated gels and biological tissues. Georgios Constantinides, Z. Ilke Kalcioglu, Meredith McFarland, James F. Smith, Krystyn J. VanVliet, **Journal of Biomechanics** 41 (2008) 3285–3289

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