Fluid

MICRO MATERIALS
MEASURING NANOTECHNOLOGY

The NANOTEST™

Bringing nanomechanical measurements into the real world

NANOTEST FLUID CELL

Test nanomechanical properties wet

- Measure nanomechanical properties of polymers and biomaterials in fluid
- Probe nanotribological behaviour and viscoelastic response
- Heat NanoTest fluid cell to body temperature and above
- Easy calibration, setup and indenter exchange

EXCELLENCE IN NANOSCALE MATERIALS TESTING

NANOTEST FLUID CELL

INTRODUCTION

Mechanical properties of biological and polymeric samples often vary considerably when in a fluid environment compared to the usual dry testing conditions. If we wish to understand their properties and behaviour in fluid media it is highly desirable to test under these conditions rather than to attempt to infer from measurements on dry (or 50% relative humidity) samples.

To meet this need, the testing capability of the NanoTest has been extended by the development of a fluid cell allowing nanoindentation, nano-scratch and nanowear testing of samples fully immersed in fluids. This development builds on the earlier success of the NanoTest humidity control chamber which allows testing under 25-90% RH. The fluid cell works with the existing pendulum design and the horizontal loading has several key advantages for testing in fluid (see box).

> "This liquid cell feature has enabled us to explore the mechanical properties of hydrated materials, which is particularly important to our studies of biologically relevant and biodegradable surfaces and which is not accessible to us through other instrumentation with comparable ranges of calibrated force and depth."
>
> *Krystyn J. Van Vliet, Lord Foundation Assistant Professor, MIT Department of Materials Science and Engineering.*

KEY POINTS OF HORIZONTAL LOADING DESIGN FOR TESTING IN FLUID

- **Indenter adapter allows indenter to be immersed in cell**
- **All electronics are well away from the cell**
- **Cell can be heated to body temperature and above (no issues with steaming the capacitive sensor)**
- **Fluid exchange during experiment is possible (flow cell option)**
- **Full scheduling capability is maintained**

APPLICATIONS OF FLUID CELL TESTING

In this Technical Note we focus on three specific applications of the fluid cell:

(1) mechanical behaviour of electrophoretic and hydrogels

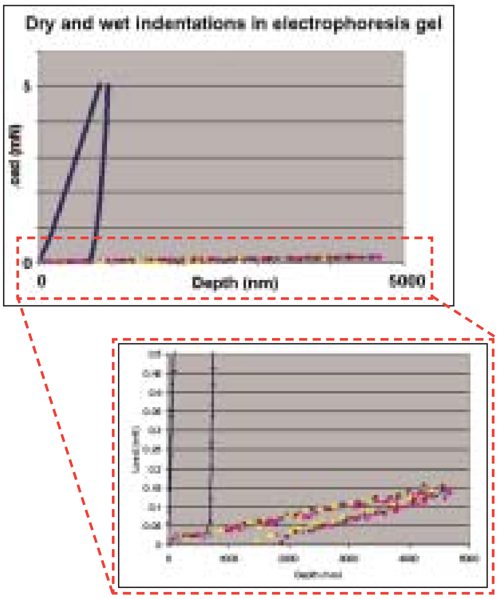
(2) plasticization and swelling of nylon by water

(3) testing of biological samples.

ELECTROPHORETIC GEL POLYMER

Hydrogels such as HEMA, the polymeric material used in contact lenses, change their surface morphology and mechanical properties on exposure to fluid. Recently scientists at MIT's NanoLab have used the NanoTest fluid cell to investigate how the indentation behaviour of an electrophoretic gel is influenced by its enironment. The difference in response wet vs. dry is striking (see figure 1) as expected due to the gel swelling in fluid. Several repeat tests were performed under the wet conditions to confirm the reproducibility of this result (see expanded area). Analysis showed a drop in modulus of over an order of magnitude when testing the gel wet.

Figure 1



Indentation into an electrophoretic gel polymer wet and dry. Indentations were performed so that the load ramp was terminated when either a set load (5 mN) or set depth (4.7 μm) was reached

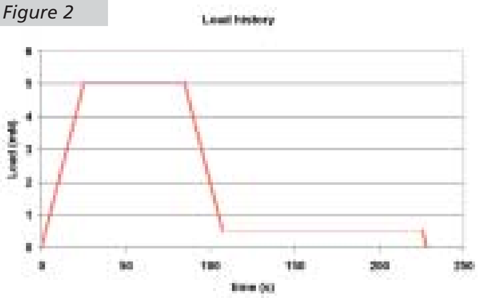
NYLON PLASTICIZATION BY WATER

Some polymers (such as polypropylene) do not absorb water significantly and so are negligibly influenced by test media. In contrast, nylon (PA6) can swell by 7-9% at saturation. The NanoTest fluid cell

has been used to investigate the role of the molecular weight and processing history of the polyamide (PA6) on its nanomechanical properties (primarily elastic modulus and creep compliance) dry and saturated.

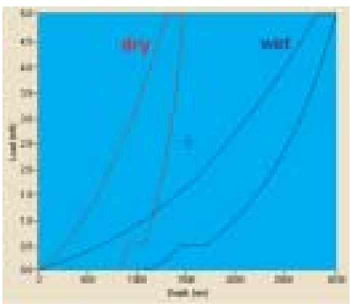
Typical indentation curves for a low MW PA6 sample are shown for dry (~50% relative humidity) and after immersion in deionised water for several hours are shown in figure 3. There is a decrease in elastic modulus of about 67% after 24 hours immersion.

Figure 2



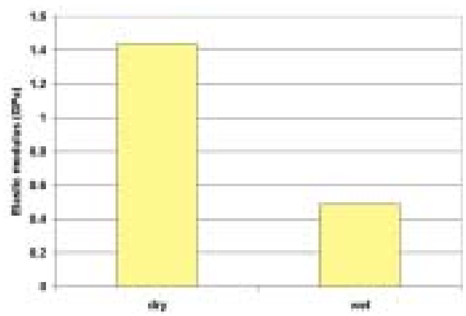
Loading history for indentation to 5 mN on nylon wet and dry

Figure 3



Typical dry and wet indentation curves for a low MW PA6 sample. Berkovich indenter loading at 0.2 mN/s to a peak load of 5 mN. Appreciable creep and creep recovery effects are observed during the hold periods both wet and dry.

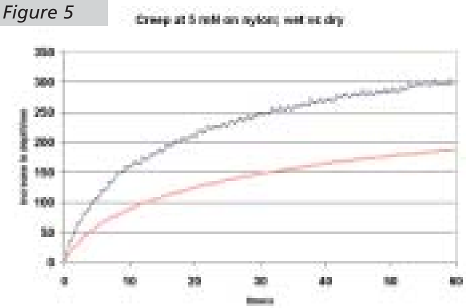
Figure 4



Influence of test environment on elastic modulus of PA6 after 24 hr immersion

The use of holding periods in the load history (see figure 2) allows investigation of creep and creep recovery processes. Analysis of this time-dependent response enables viscoelastic properties to be determined. In fluid there is an increase in indentation creep (figure 5). However, the proportion of time-dependent deformation compared to the total deformation decreases, consistent with a decrease in the magnitude of tan delta at the testing temperature [1].

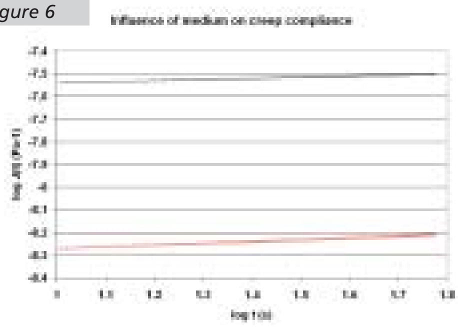
Figure 5



Nanoindentation creep during 60 s hold at 5 mN; wet = squares; dry = circles

The NanoTest software produces values of creep compliance vs. time, as has been done previously in ambient and elevated temperature experiments [2]. Typical experimental data are shown in figure 6. The increase in creep compliance is due to appreciable plasticization of the nylon by water molecules.

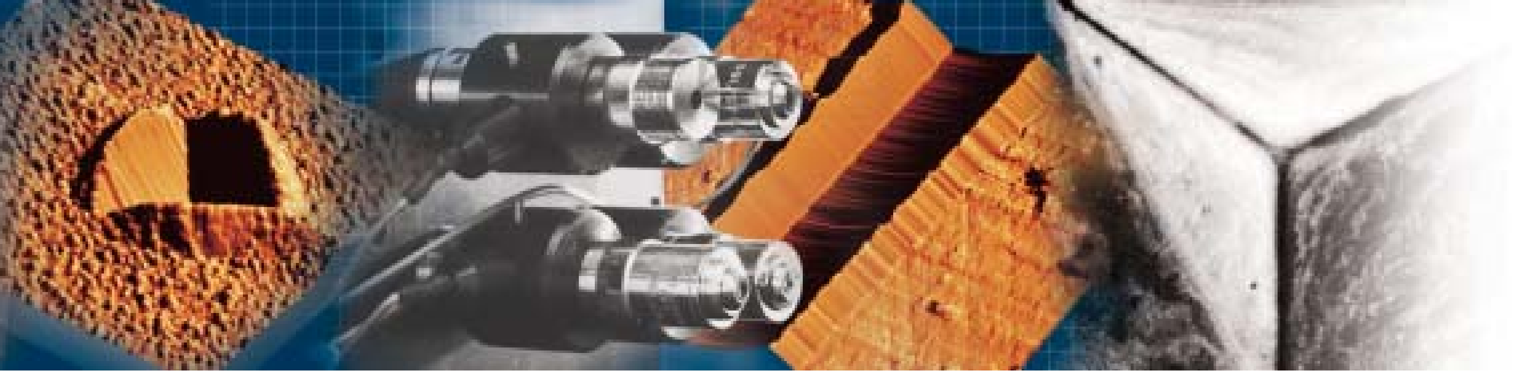
Figure 6



Creep compliance during 60 s hold at 5 mN; wet = squares; dry = circles

KEY POINTS OF HORIZONTAL LOADING DESIGN FOR TESTING IN FLUID

- **Ease of calibration, setup and indenter exchange**
- **Compatible with any NanoTest system**
- **No significant buoyancy problems**
- **No large change in meniscus position during indentation**



NANOTEST FLUID CELL

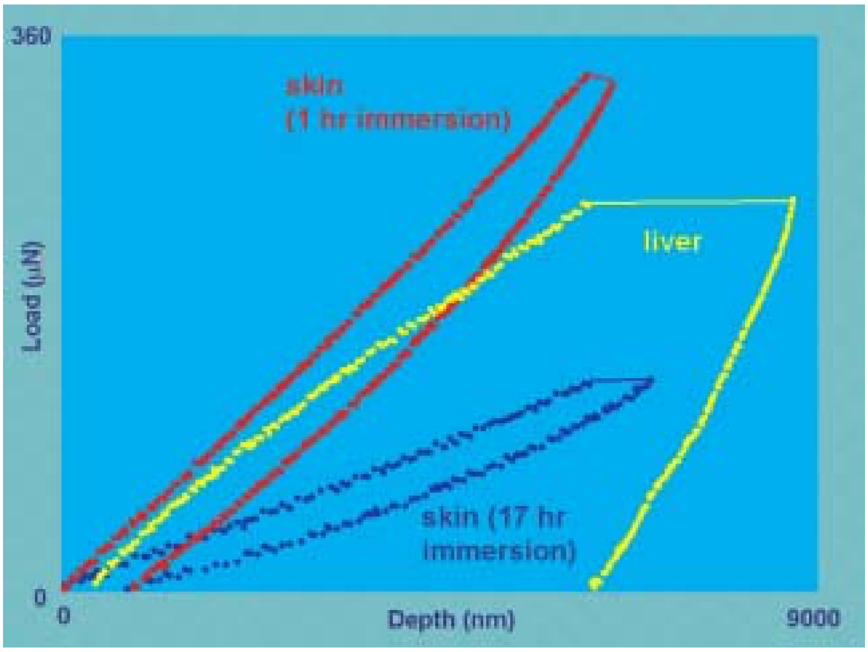


Figure 7

Nanoindentation to maximum set depth on skin and liver samples. Additional immersion time alters the elasticity of the skin sample

BIOLOGICAL SAMPLES

Probing the mechanical properties of biological samples in fluid media should prove a closer mimic of in vivo conditions than conventional dry nanoindentation testing. The NanoTest fluid cell has been used to investigate the indentation behaviour of skin and liver samples in deionised water. A large radius (500 μm) spherical indenter was used to reduce the contact pressure and produce a predominantly (visco)elastic response.

Comparison of the skin indentation response in water between 1 and 17 hr immersion shows that saturation can require considerable immersion time. Notably the liver sample exhibited significant creep; the viscoelastic properties of this sample were more pronounced than the skin sample.

MICRO MATERIALS

MEASURING NANOTECHNOLOGY

For further information contact us at

Micro Materials Limited
Willow House
Yale Business Park
Wrexham Technology Park
Wrexham LL13 7YP UK
Tel: +44 1978 261615
Fax: +44 1978 356966
info@micromaterials.co.uk
www.micromaterials.co.uk

Micro Materials is a member of the Millbrook Scientific Instruments PLC group.

SPECIFICATIONS

Fluid cell package includes an indenter adapter, liquid cell software and the liquid cell itself.

Load range 0-500 mN

For hardness, elastic modulus and creep compliance determination in fluid media.

Retrofittable to all existing NanoTest systems.
In addition to the examples of wet indentation reported here, the fluid cell can be used in conjunction with:-

1) Micro Materials nanofretting™ module
2) nanoscratch and nanowear experiments
3) humidity control chamber

A flow cell option is required for controlled fluid exchange during experiments.
Optional upgrades to include hot and cold stages.
In comparison to other methods (such as Dynamic Mechanical Analysis) the NanoTest fluid cell enables more highly localised measurements of mechanical properties and testing of thinner and more heterogeneous samples.

The fluid cell opens up the possibility to use the range of nanomechanical test methods to optimise biomedical device performance in conditions that closely mimic those in vivo.

ACKNOWLEDGEMENTS

We would like to acknowledge Prof Krystyn VanVliet and co-workers at the NanoLab (MIT, Boston) and also Prof Dariusz Bielinski (Institute of Polymers, Lodz) for kindly providing some of the experimental data for this Technical Note.

REFERENCES

1. The influence of fluid environment on the nanoindentation creep behaviour of nylon 6, BD Beake, G Bell and D Bielinski, in preparation.

2. Multiscale creep compliance of epoxy networks at elevated temperatures, TF Juliano, MR VanLandingham, CA Tweedie and KJ Van Vliet, Experimental Mechanics (2006).