High temperature nanoindentation

Tony Maxwell, Nigel Jennett, Xiaodong Hou and Laura Mera Alvarez

6th June 2014
Objectives

Purpose: mapping of creep, modulus and hardness at temperature using nanoindentation.

- Developed calibration procedures.
- Evaluated uncertainty budgets.
- Developed procedures to map mechanical properties as a function of temperature.
Why?

- Increased use of micro-components in industry.
- Need to identify inhomogeneities in micro-components to reduce wastage by detecting defects, impurities and poor blending.
High temperature indentation with hot-stage

Nanoindentation instrument

Load displacement curves obtained from technique
Project output: NanoTest Traceably calibrated as f(T) + Validation + Ref. materials’ specification

- Indentation cycle
- Control (F, h, T)
- Thermal drift + stability

Traceable Calibration

- Force & Displacement
- Cf
- A(h)
- T

- RT→ Elevated T
- Sensors; Thermal camera

- RT→ Elevated T
- Direct measurement
- Role of Ref. materials

- RT→ Elevated T
- Role of Ref. materials

Uncertainty budgets

Good practice guide

Reference materials for high temperature indentation

Determine properties of reference materials
- Hot-Impact Resonance
Displacement and load calibrations as function of temperature

<table>
<thead>
<tr>
<th></th>
<th>Calibration values</th>
<th>Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>LOAD</td>
<td><img src="image1" alt="Force calibration graph" /></td>
<td><img src="image2" alt="LOAD method" /></td>
</tr>
<tr>
<td>DISPLACEMENT</td>
<td><img src="image3" alt="Displacement calibration graph" /></td>
<td><img src="image4" alt="DISPLACEMENT method" /></td>
</tr>
</tbody>
</table>

For LOAD:
- Formula: \( y = -0.0057x + 57.781 \)
- \( R^2 = 0.8612 \)

For DISPLACEMENT:
- Formula: \( y = -0.143x + 2017.9 \)
- \( R^2 = 0.4963 \)
Displacement calibration

New high precision jamin interferometer using for calibration

Voltage output of nanoindenter calibration to displacement from interferometer
Calibration method for Area function and Frame compliance

• Two reference materials with known properties used to obtain
  • Soft material used to determine area function
  • Hard material used to determines frame compliance

• Modulus of reference materials at elevated temperatures determined using impact excitation
Determination of indenter area function with soft reference material at 100°C

\[ y = 5.286x + 118.98 \]
\[ R^2 = 0.9979 \]

Curve fit gives depth to contact area function
Determination of frame compliance with hard reference material at 100°C

Measured compliance at zero depth gives frame compliance

\[ y = 3104.5x \]

\[ R^2 = 0.9894 \]
# Area function and Frame compliance as function of Temperature

<table>
<thead>
<tr>
<th></th>
<th>Calibration values</th>
<th>Method</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>COMPLIANCE</strong></td>
<td><img src="image1.png" alt="Graph showing frame compliance as function of temperature" /></td>
<td><img src="image2.png" alt="Image of compliant frame" /></td>
</tr>
<tr>
<td></td>
<td>[ y = 0.0002x + 0.4581 ] [ R^2 = 0.1634 ]</td>
<td></td>
</tr>
<tr>
<td><strong>AREA FUNCTION COEFFICIENT</strong></td>
<td><img src="image3.png" alt="Graph showing area function coefficient as function of temperature" /></td>
<td><img src="image4.png" alt="Image of area function coefficient" /></td>
</tr>
<tr>
<td></td>
<td>[ y = 0.0013x + 4.9579 ] [ R^2 = 0.2618 ]</td>
<td></td>
</tr>
</tbody>
</table>
Temperature determination

- Indenter and specimen are heated with thermocouple control
- Specimen attachment with **highly conductive cement** (i.e. 1.4-1.7 W/m.K)
- Two additional thermocouples attached in the middle and edge of a sample
Actual Temperatures

- Graph shows readings from k-type calibrated thermocouple attached to the sample surface with hot indenter on surface.
- Actual surface temperature differs from ‘Nominal’ temperature due to sample size, thickness and thermal conductivity of specimen.
Acoustic sensitivity

The acoustic experiments have been done with a Brüel and Kjær Pulse Labshop in conjunction with a Genelec 7050B speaker and two microphones.
- Acoustic sweeps in the range of 22 to 104Hz (low frequency range)
- The damping capacity of the nanoindenter cabinet is being evaluated

Region of NanoTest resonance @71Hz
Nanoindenter acoustic sensitivity

- Results correlated to the nanoindenter response
- Signal display monitoring (i.e. the electronic signal responsible for the displacement data)

Pronounced signal display disturbance corresponding to 71Hz

SENSITIVITY COEFFICIENT; \( \approx 22.6 \text{ nm} \)
Good Practice Guide

High temperature nanoindentation

Materials Division
National Physical Laboratory

Abstract:
This Measurement Good Practice Guide covers technical issues surrounding nanoindentation measurements at high temperature. Recommendations are made for reaching isothermal equilibrium, but also a comprehensive study of other alternative methods is also present. An uncertainty budget for NPL nanoindenter has been estimated. The thermal properties of the sample under study appear to be one of the major contributions to the temperature uncertainties, obstructing isothermal equilibrium detection.

Contents

1. High Temperature Nanoindentation ........................................3
   1.1. Description of Technique ...........................................3
   1.2. NPL High Temperature Nanoindenter ...........................4
   1.3. Measurement Issues ...............................................6
       1.3.1. Instrument and Calibration ..................................6
       1.3.2. Temperature ..................................................17
       1.3.3. Specimen ....................................................29
       1.3.4. Indenter .......................................................30
   1.3.5. Procedure for Hardness and Young’s Modulus Determination .............30
   1.3.6. Analysis of Results .............................................31
   1.4. Nanoindentation standards .........................................31
       1.4.1. Room temperature standard ................................31
       1.4.2. High temperature standard .................................32
   2. Determination of Uncertainty .........................................32
       2.1. Fundamentals of Measurement Uncertainty ....................32
       2.2. Uncertainty of Nanoindentation Measurements ............33
       2.3. Uncertainty Budget for NPL High Temperature Nanoindenter ................38
   3. Bibliography ..................................................................40
       2.1. Instrumented Indentation Standards ............................40
       2.2. Nanoindentation at High Temperature .........................40
       2.3. Uncertainty .......................................................41
   Appendix 1: Procedure for high temperature nanoindentation 43
Comparing indentation to tensile creep in POM
Mapping of properties

Region of moulding examined in tests

Mapping the modulus cross-section of injection moulded POM at 60°C
Summary

- Calibration procedures for elevated temperature nanoindentation developed.
- Uncertainty budget developed for nanoindentation at elevated temperatures.
- Mapping of mechanical properties across specimens on the microscale at elevated temperatures achieved.
- Good Practice Guide on measurement uncertainties produced.