High temperature nanoindentation

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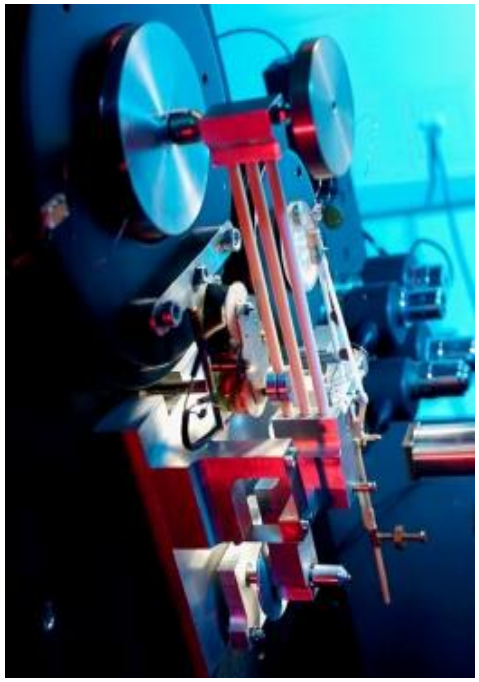
Objectives

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.



Nanoindentation instrumentation

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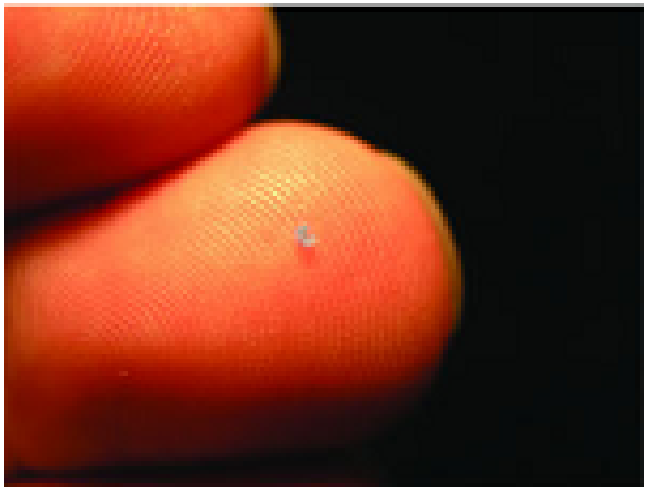
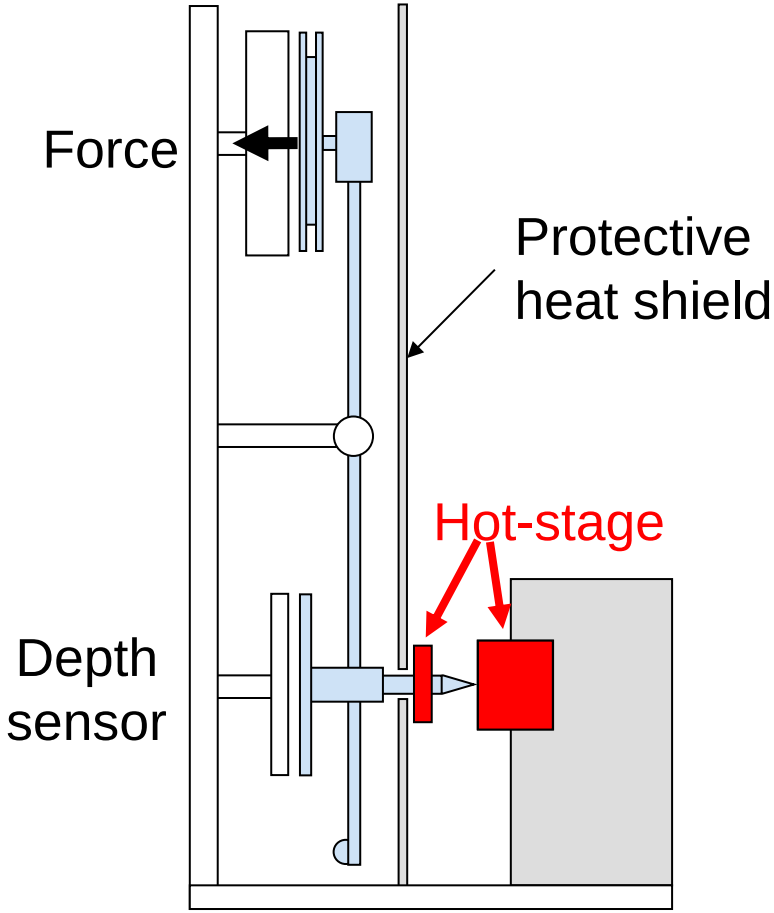
Why?

- Increased use of micro-components in industry.
- Need to identify inhomogeneities in micro-components to reduce wastage by detecting defects, impurities and poor blending.

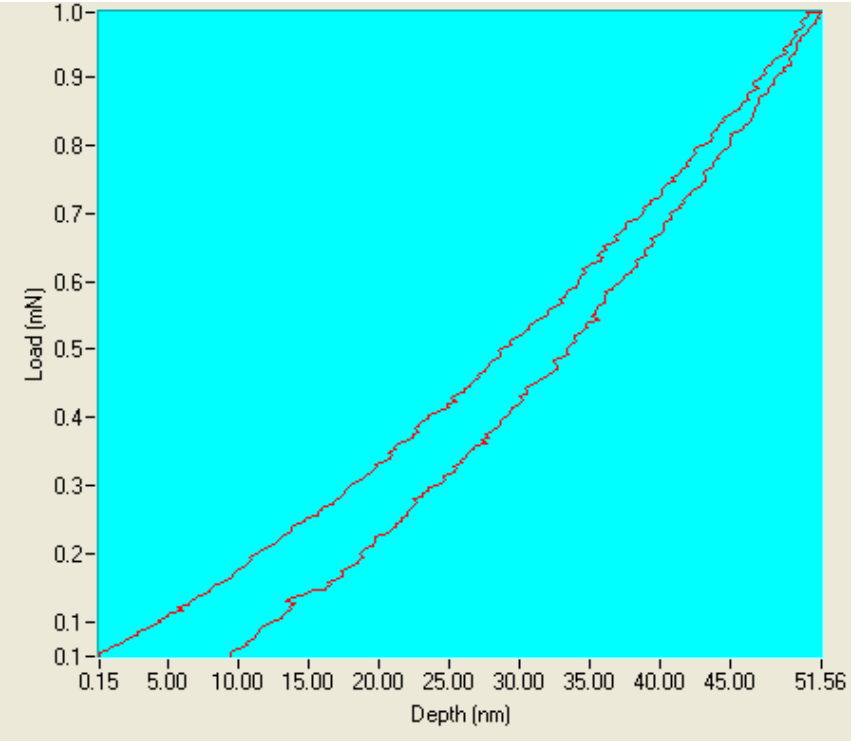


Micro-mouldings

High temperature indentation with hot-stage



Nanoindentation instrument



Load displacement curves obtained from technique

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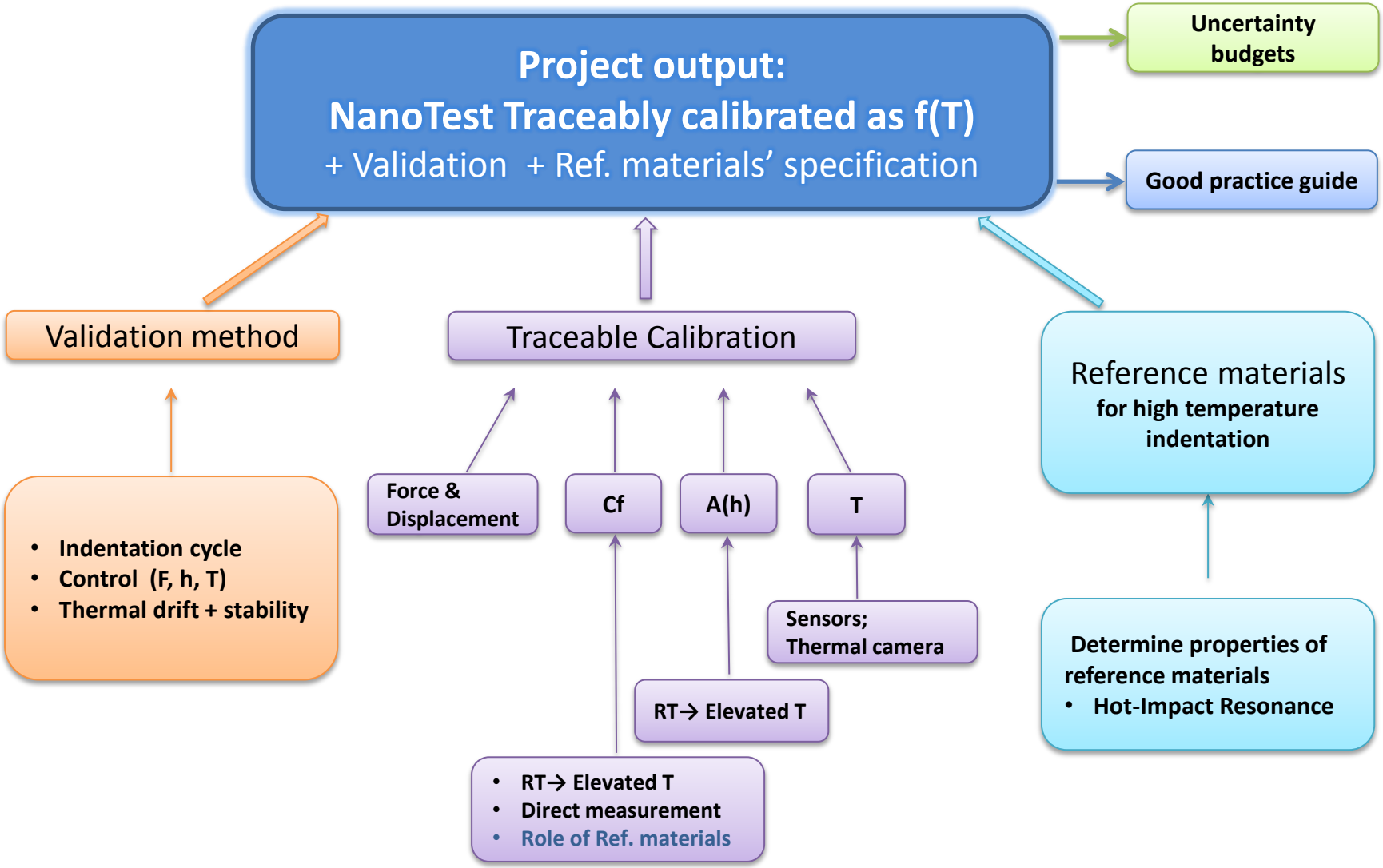

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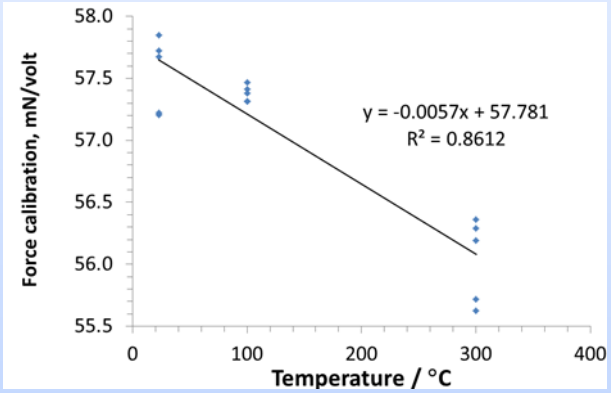
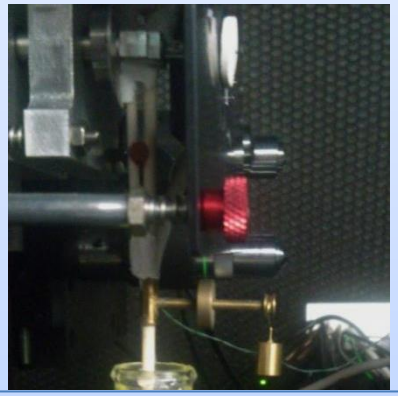
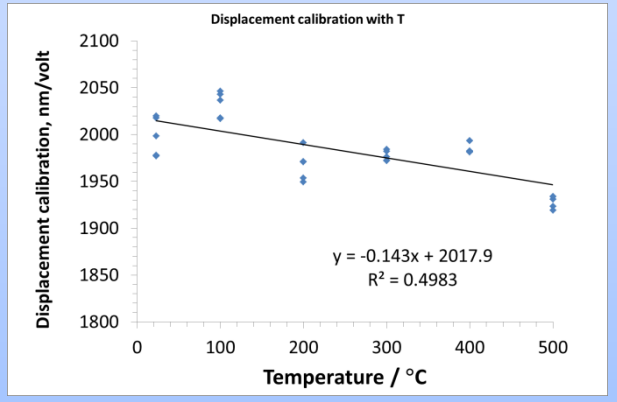
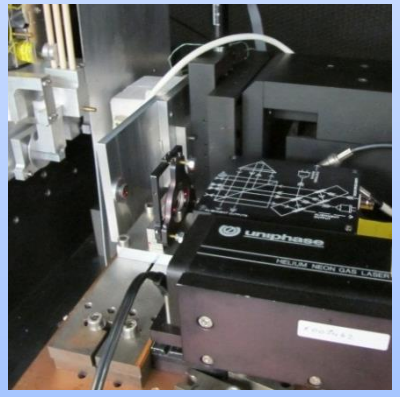
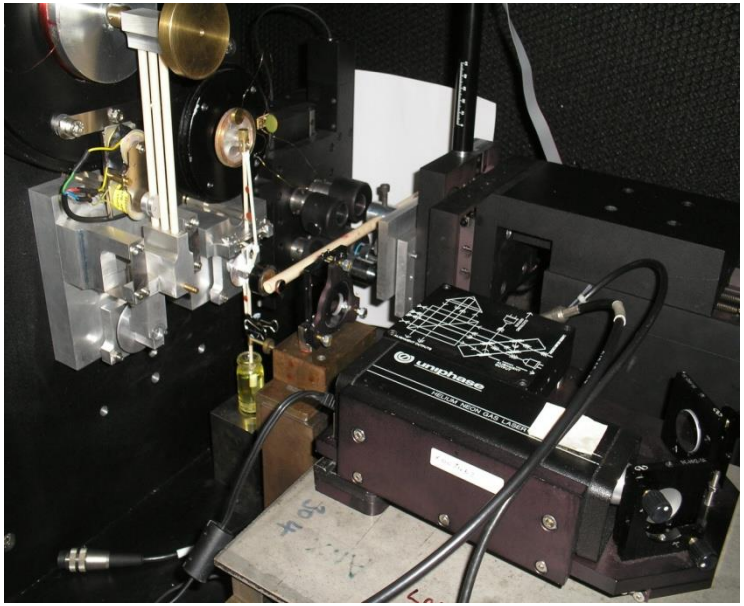
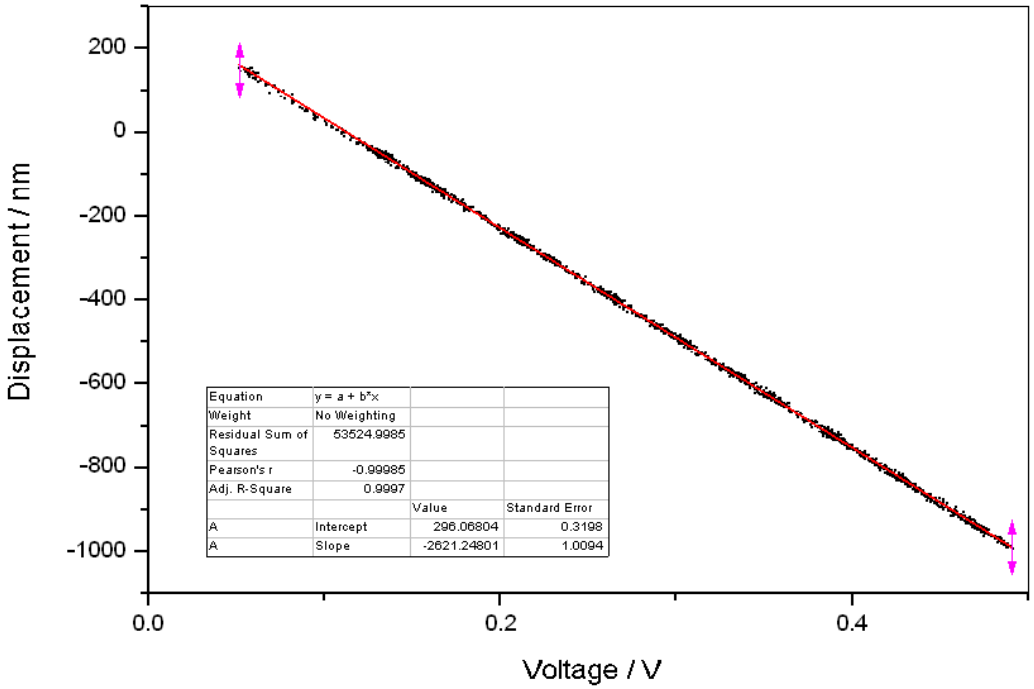
Displacement and load calibrations as function of temperature

	Calibration values	Method
LOAD		
DISPLACEMENT		

Displacement calibration



New high precision jamin interferometer using for calibration



Voltage output of nanoindenter calibration to displacement from interferometer

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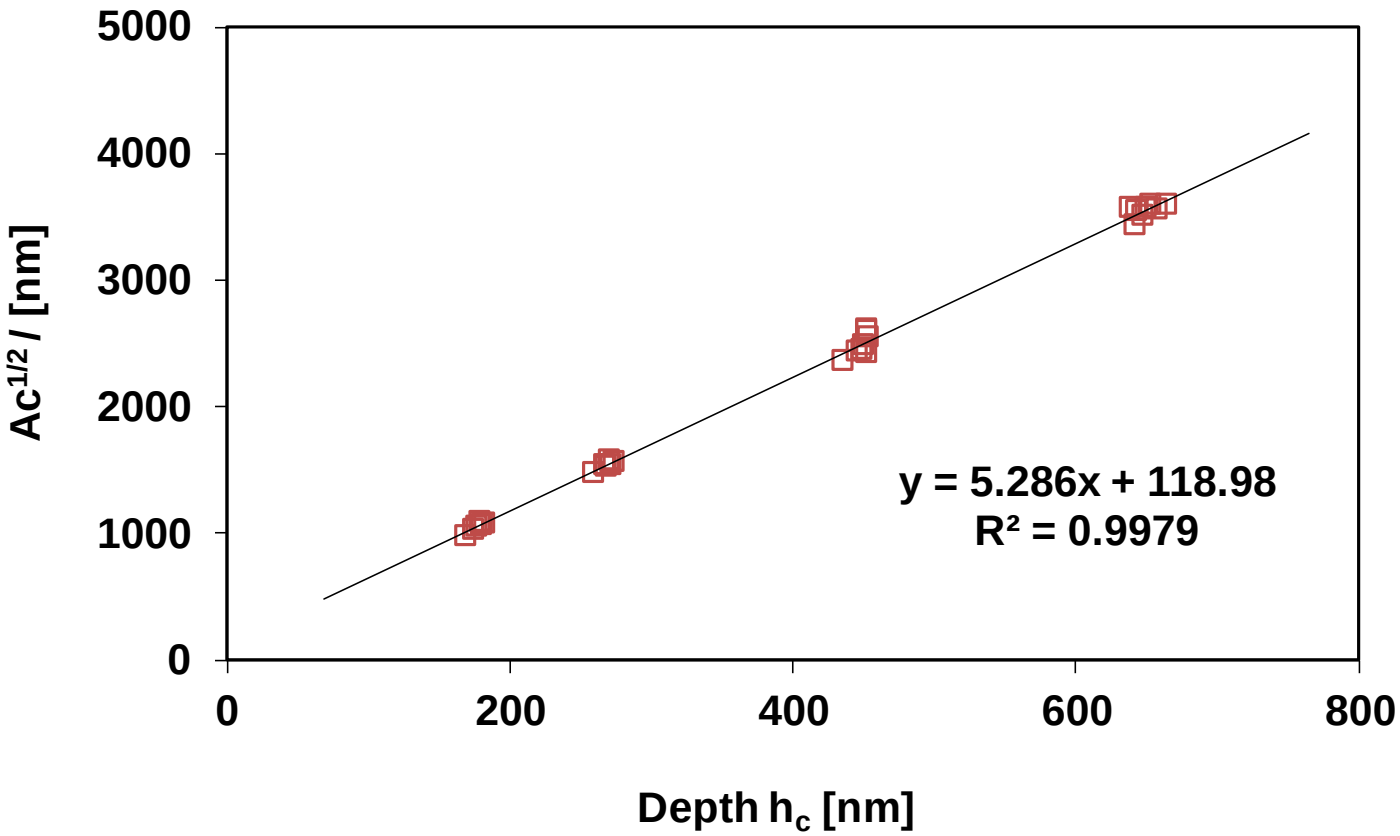
Calibration method for Area function and Frame compliance

- 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



Curve fit gives depth to contact area function

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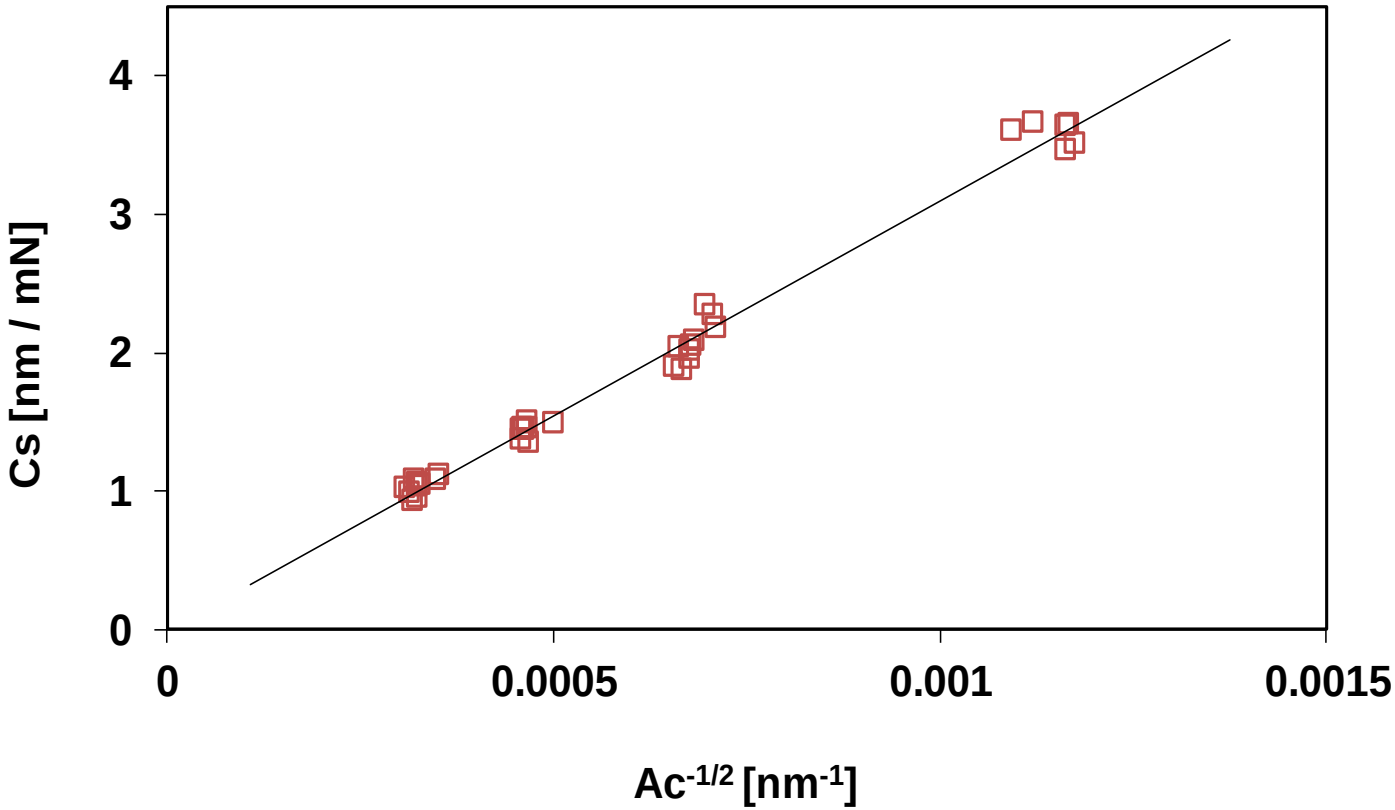
Determination of frame compliance with hard reference material at 100°C



Measured compliance at zero depth gives frame compliance

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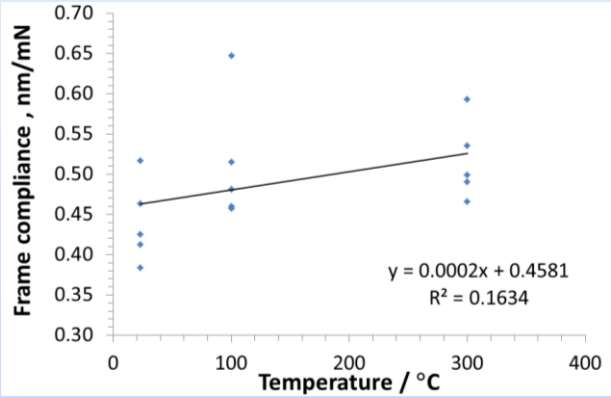
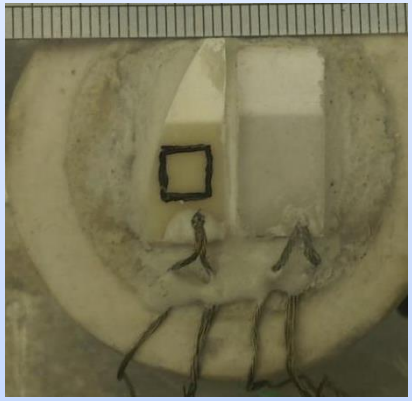
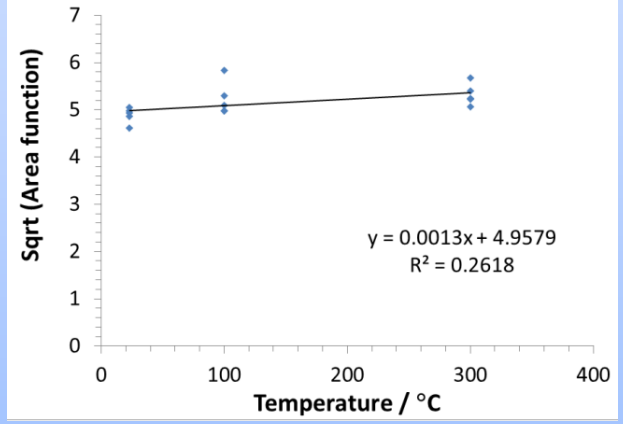
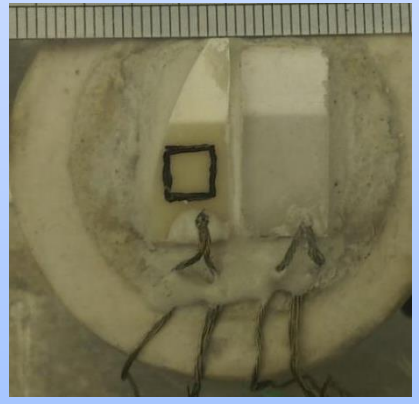
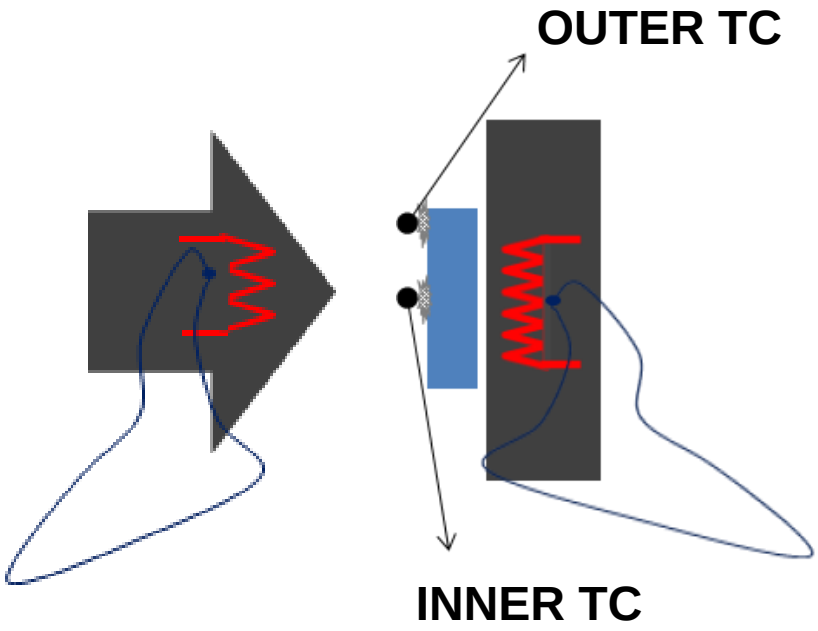
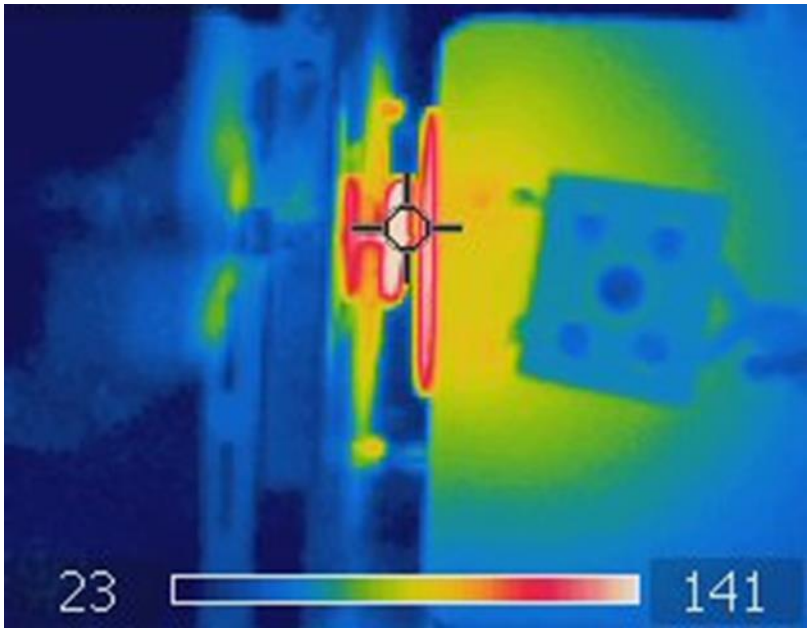
Area function and Frame compliance as function of Temperature

	Calibration values	Method
COMPLIANCE		
AREA FUNCTION COEFFICIENT		

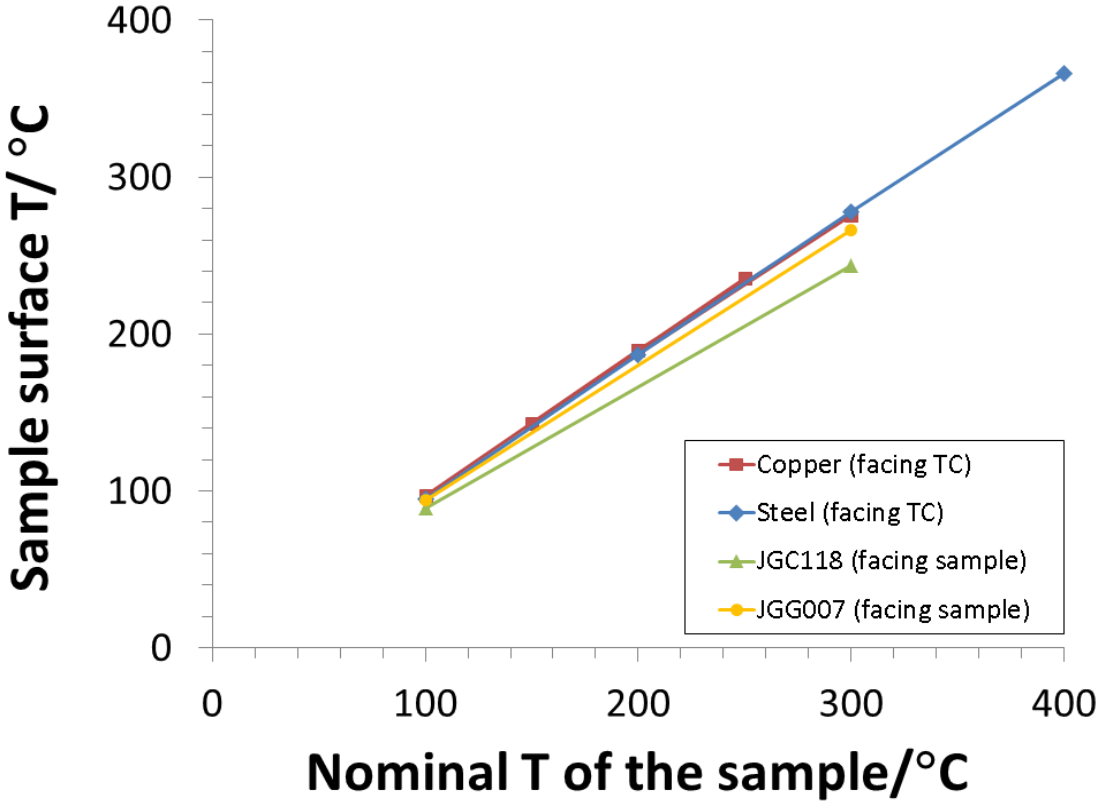
Temperature determination

- 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



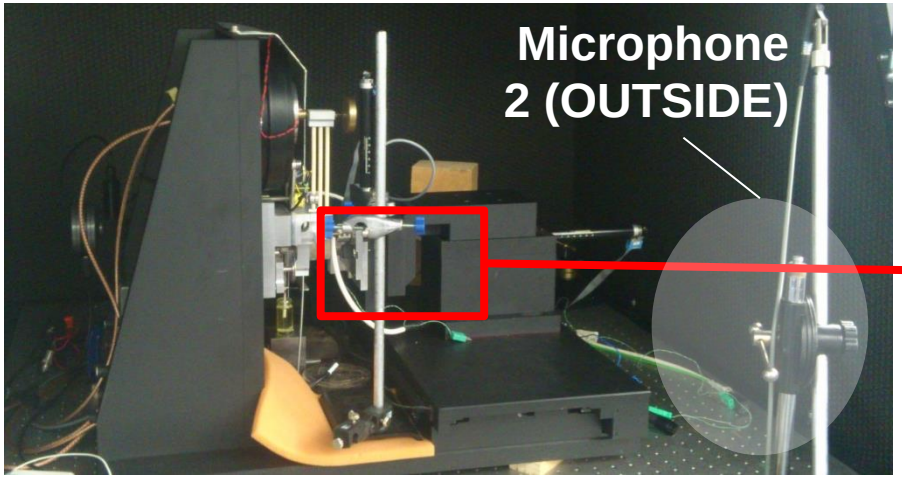


Actual Temperatures



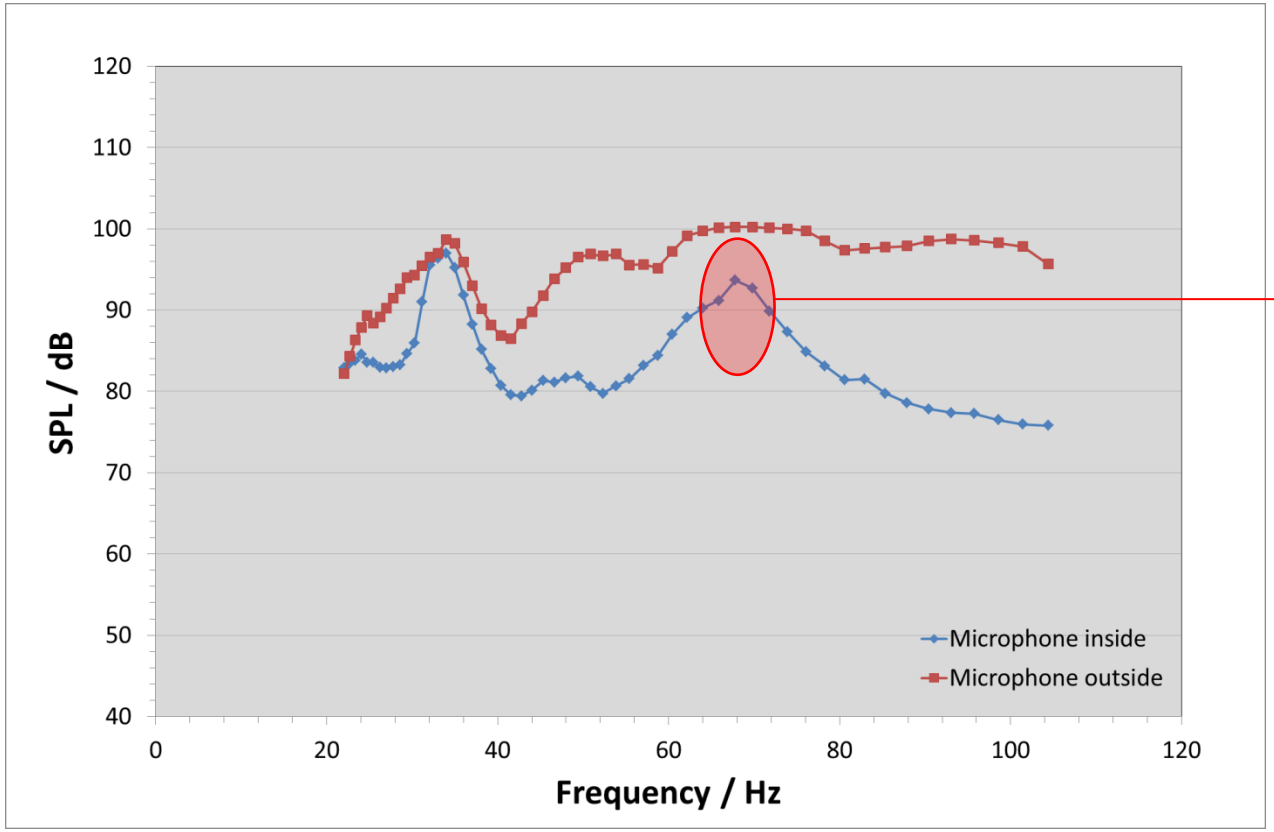
- Graph shows readings from k-type calibrated thermocouple attached to the sample surface with hot indenter on surface
- Actual surface temperature differs from 'Nominal' temperature due to sample size, thickness and thermal conductivity of specimen

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.

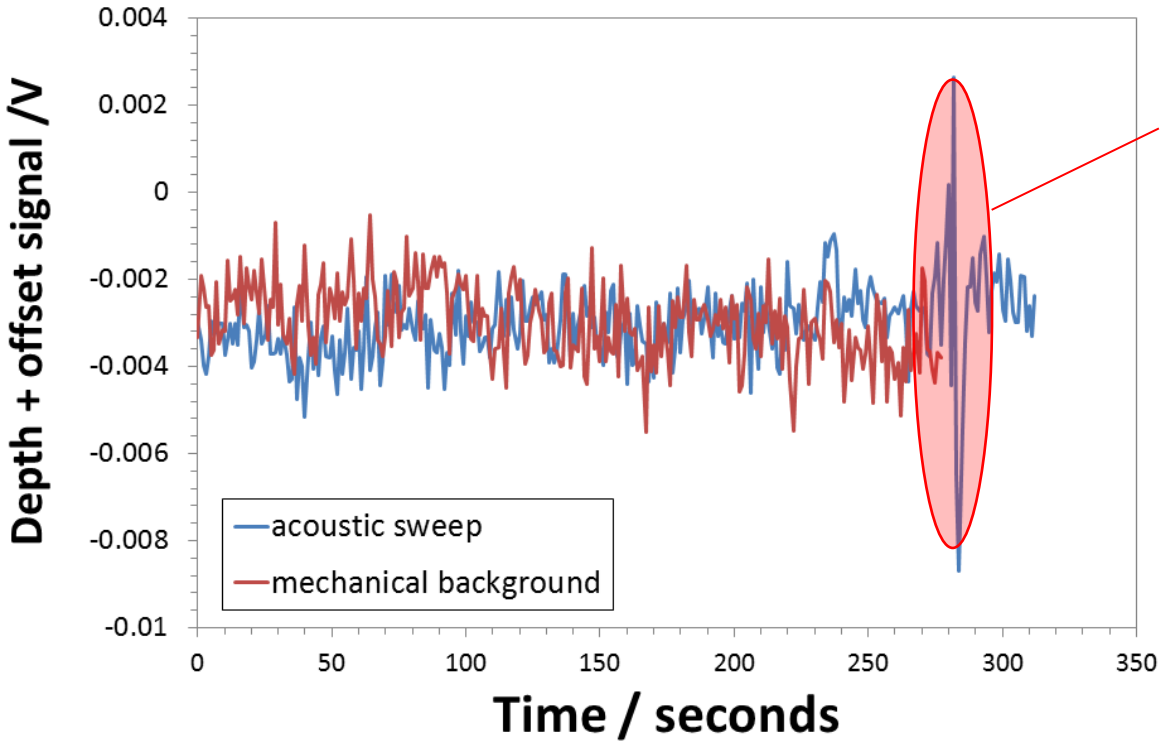
Resonant frequency



Region of NanoTest resonance @71Hz

- Acoustic sweeps in the range of 22 to 104Hz (low frequency range)
- The damping capacity of the nanoindenter cabinet is being evaluated

Nanoindenter acoustic sensitivity

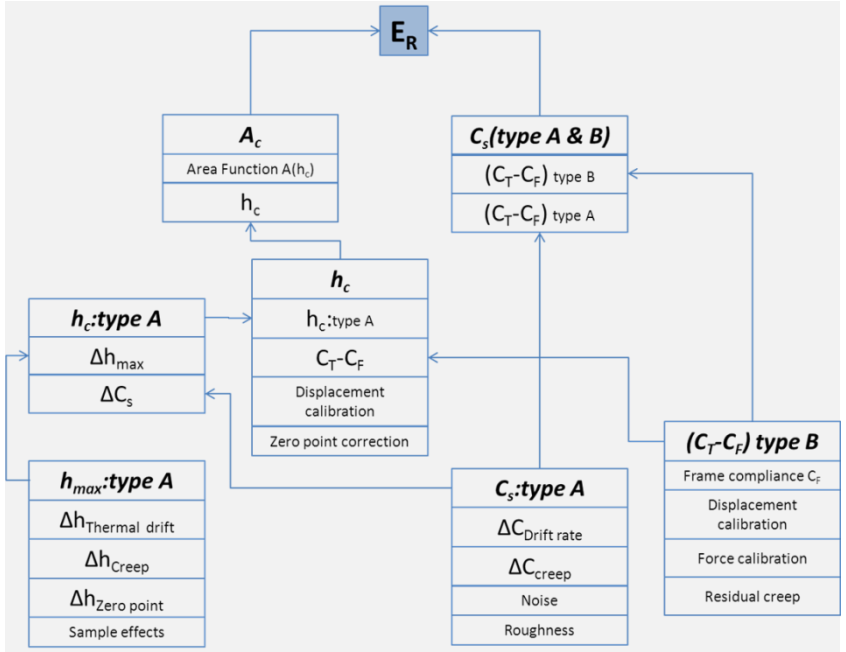


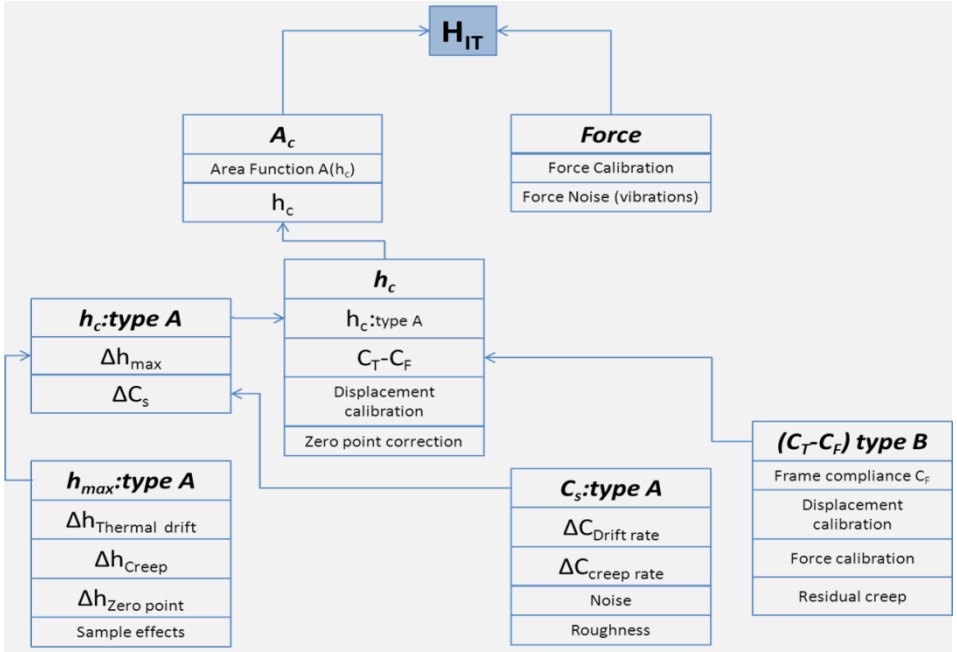
Pronounced signal display disturbance corresponding to 71Hz

SENSITIVITY COEFFICIENT; ~22.6 nm

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

Uncertainty budget





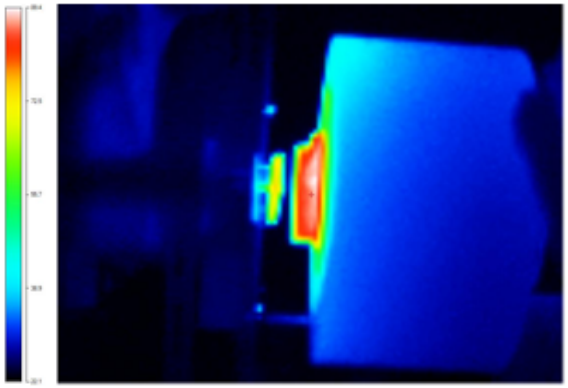
Good Practice Guide

NPL National Physical Laboratory

Measurement Good Practice Guide No.

High temperature nanoindentation

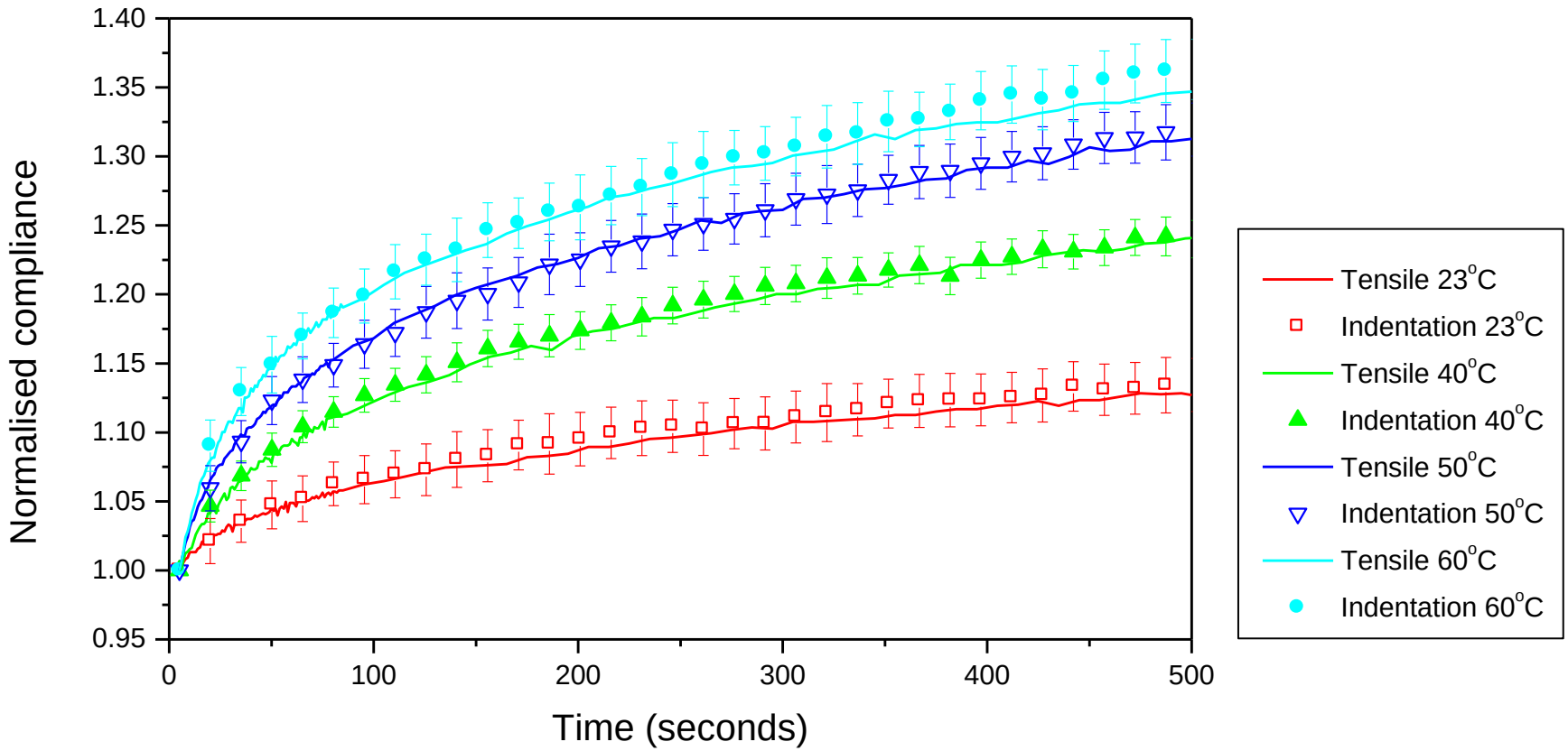
Materials Division
National Physical Laboratory



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

Comparing indentation to tensile creep in POM



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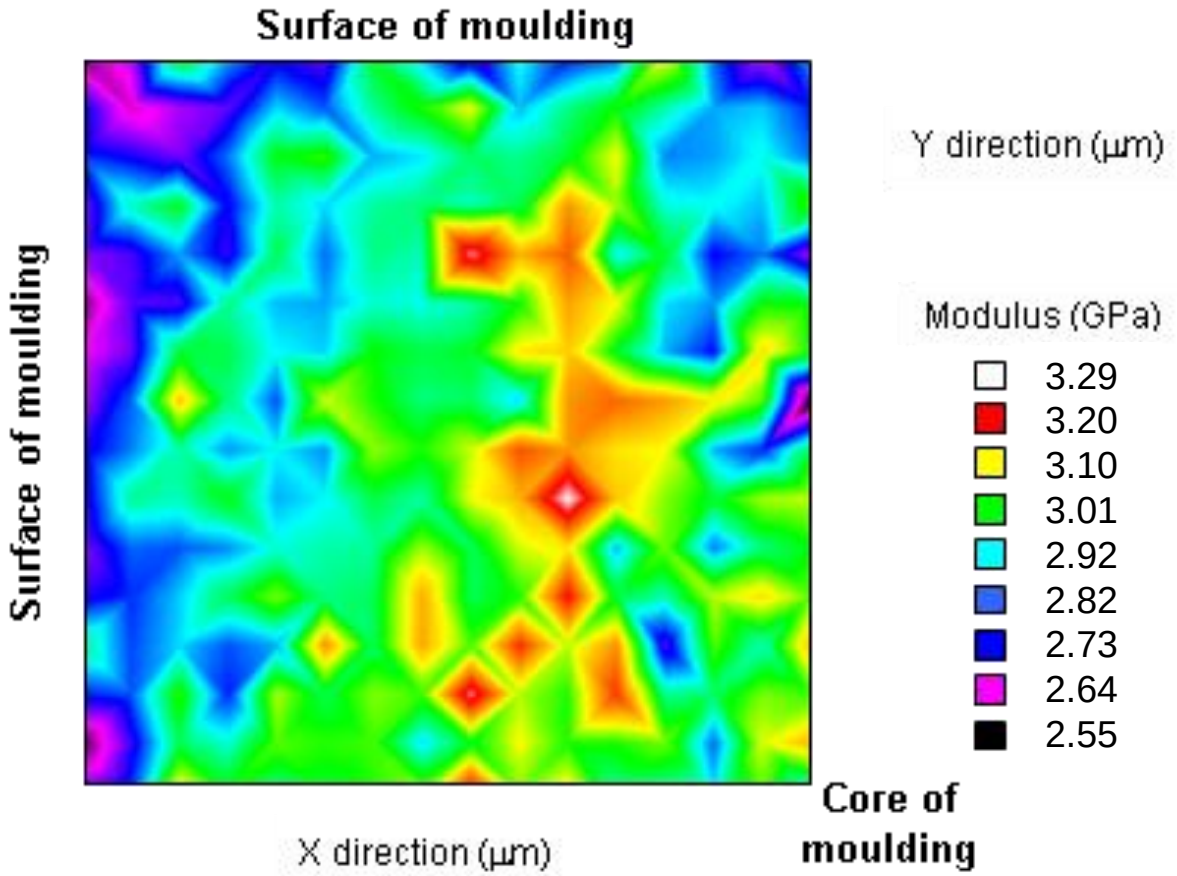
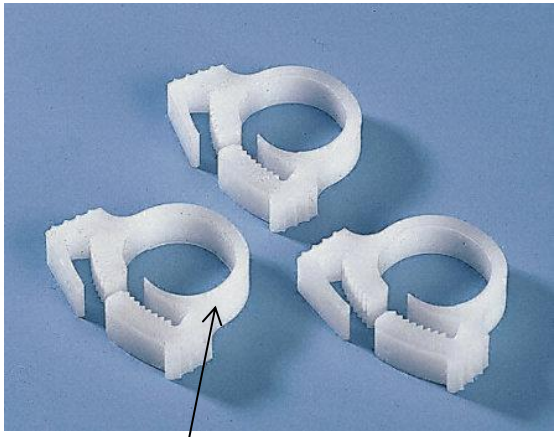
Mapping of properties





Region of moulding examined in tests

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

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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.

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