Characterisation Programme 2006-2009
Polymer Multi-scale Properties Industrial Advisory Group
28th June 2007

SE02: Improved Design and Manufacture of Polymeric Coatings Through the Provision of Dynamic Nano-indentation Measurement Methods

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Project Manager: Rob Brooks

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Presentation outline

- Brief review of:
  - The project motivations
  - Instrumented indentation
  - SE02 Project outline
- Sensitivity study of parameters affecting dynamic indentation results
- Adaptation of NanoTest for variable rate, frequency and temperature dynamic indentation

Nano-indentation applications

Ideal for measuring elastic and plastic properties of small volumes of materials, e.g. thin films and micro/nano-structures.

- Coatings and Surface Engineering sectors / users, e.g. Electronics, optics, automotive, aerospace, biomedical, pharmaceutical sectors..
- Local/surface properties of biological or polymeric materials (very soft, low force indentations)
- Micro-mouldings, nano-composites and ultra hard coatings where indentation depths are very small.
- Nano-mechanical testing of nano-engineered structures and materials
- Designers needing input data for models

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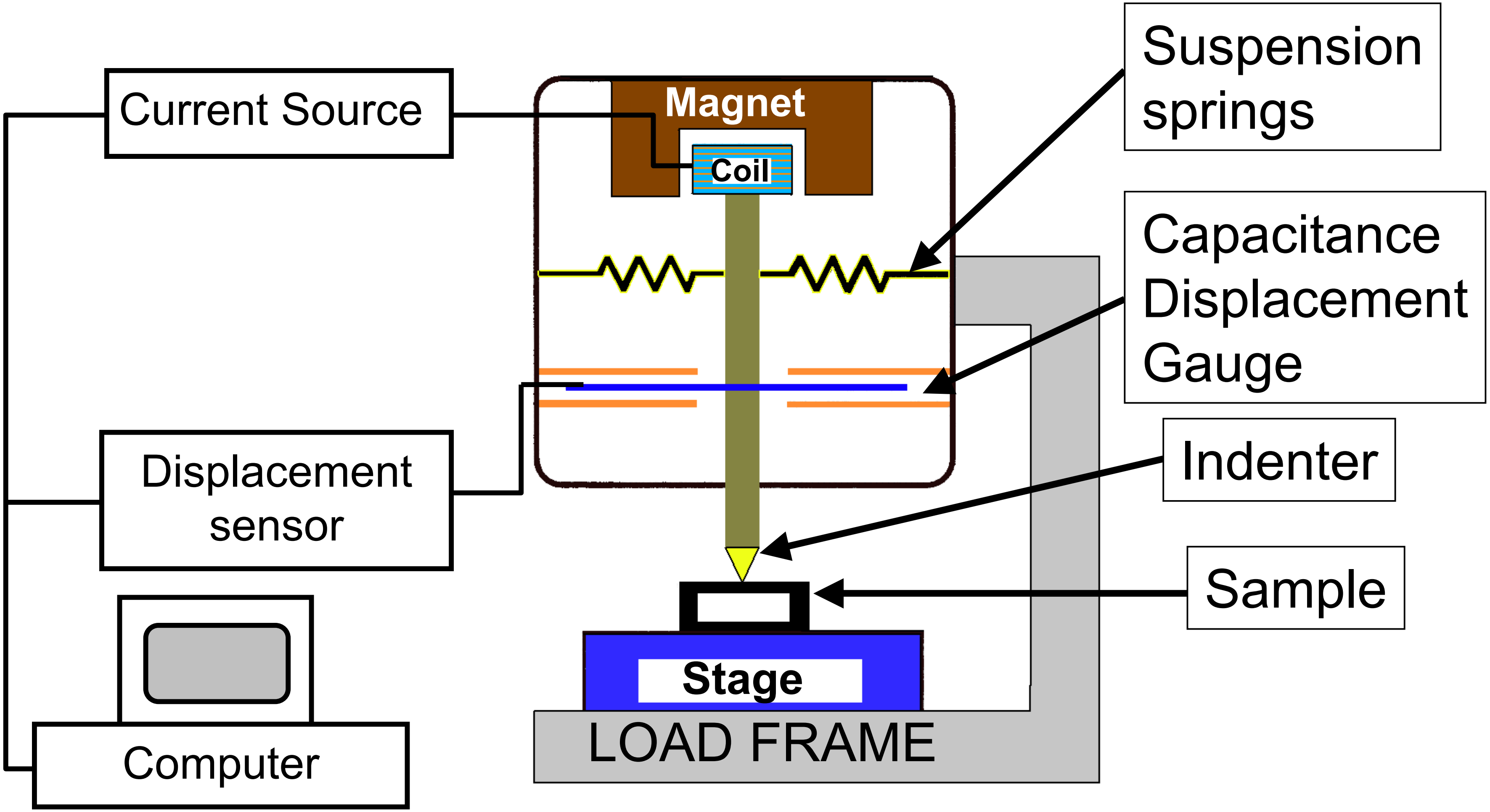
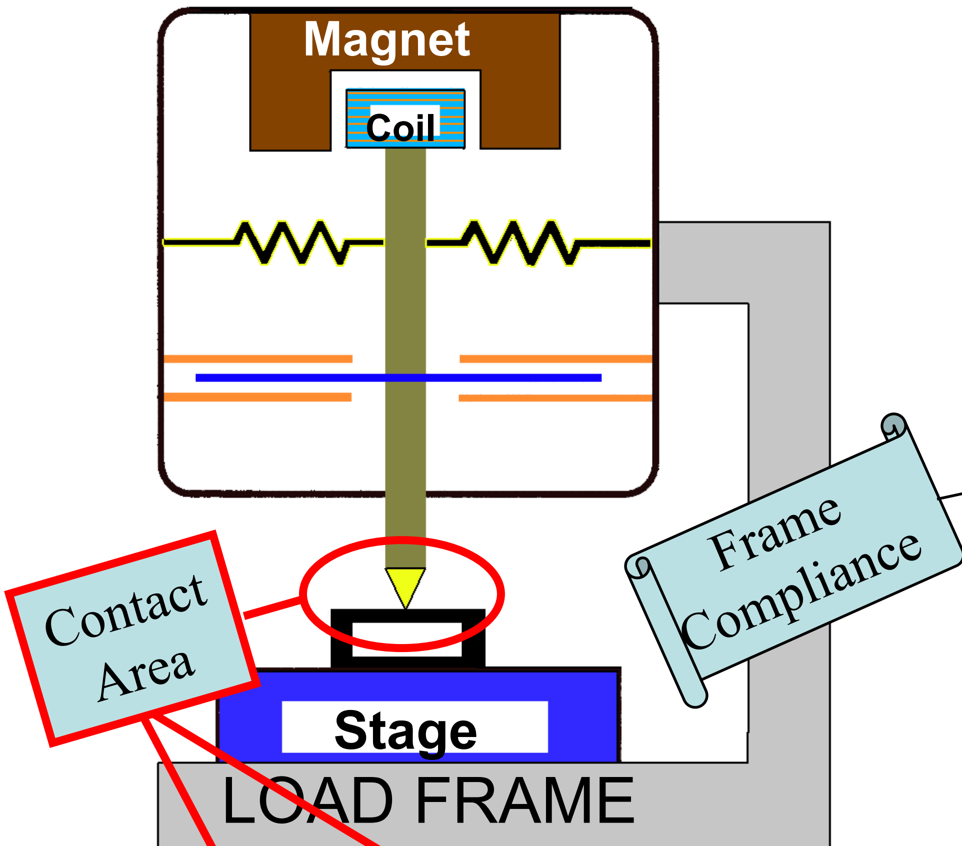
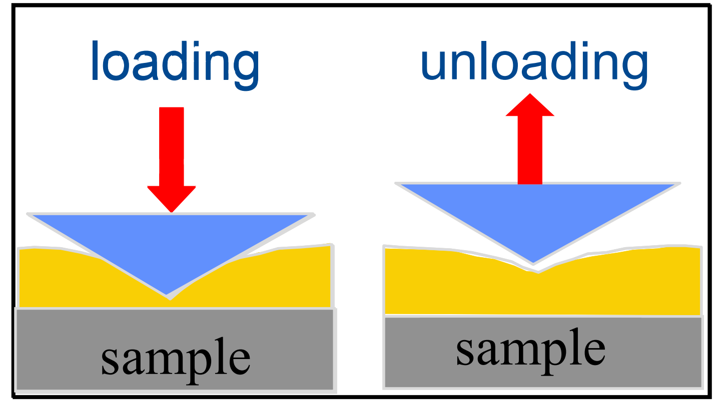
Instrumented Nanoindenter Schematic

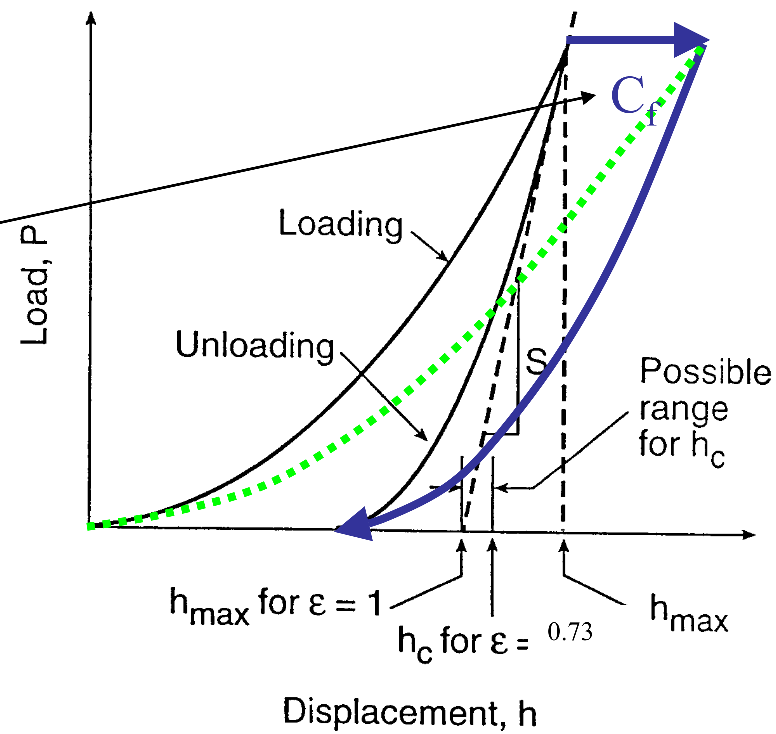


Quasi-static

Instrumented Indentation







$$H_{IT} = \frac{Force}{Area}$$

$$E^* \propto \frac{1}{\sqrt{A_C}} \times \frac{1}{C_S}$$

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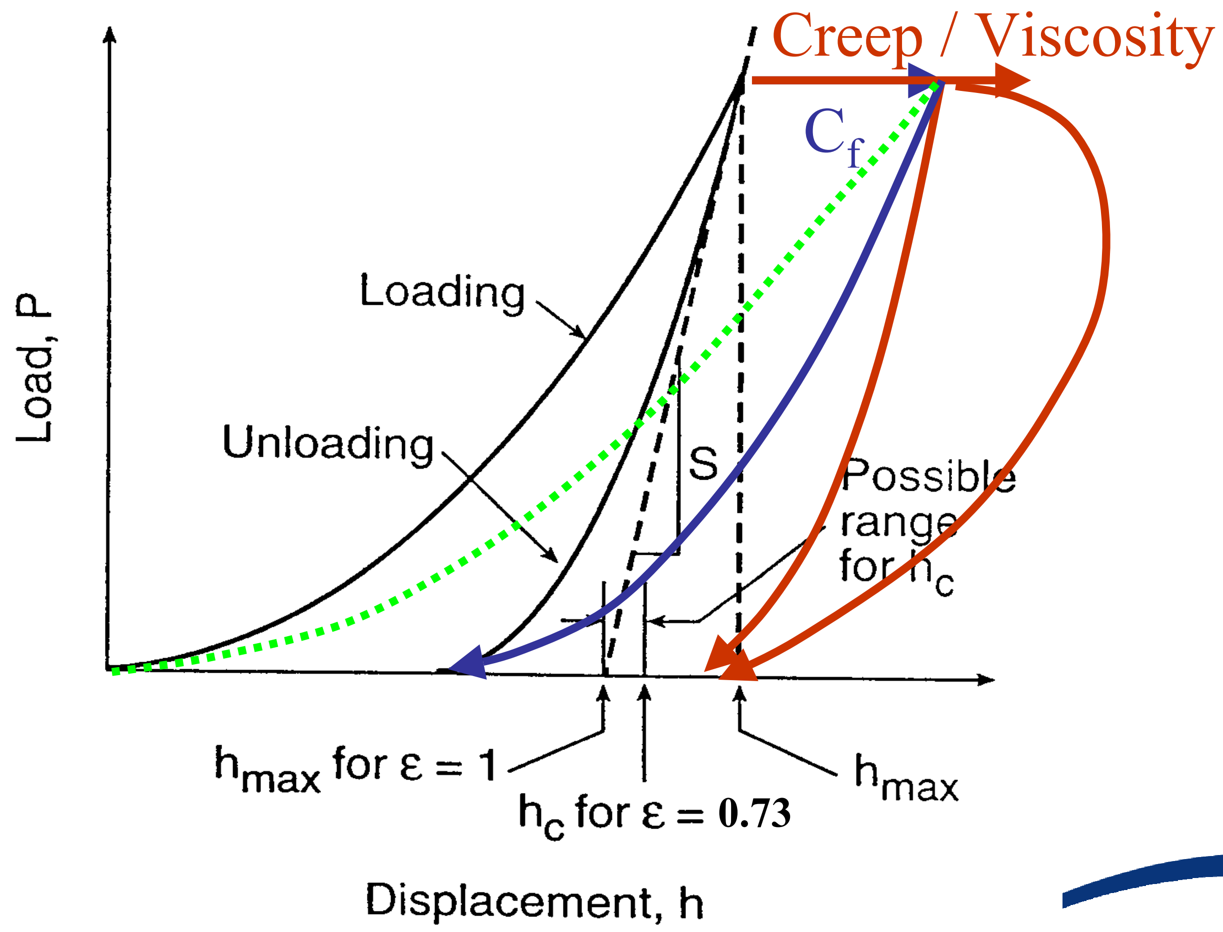
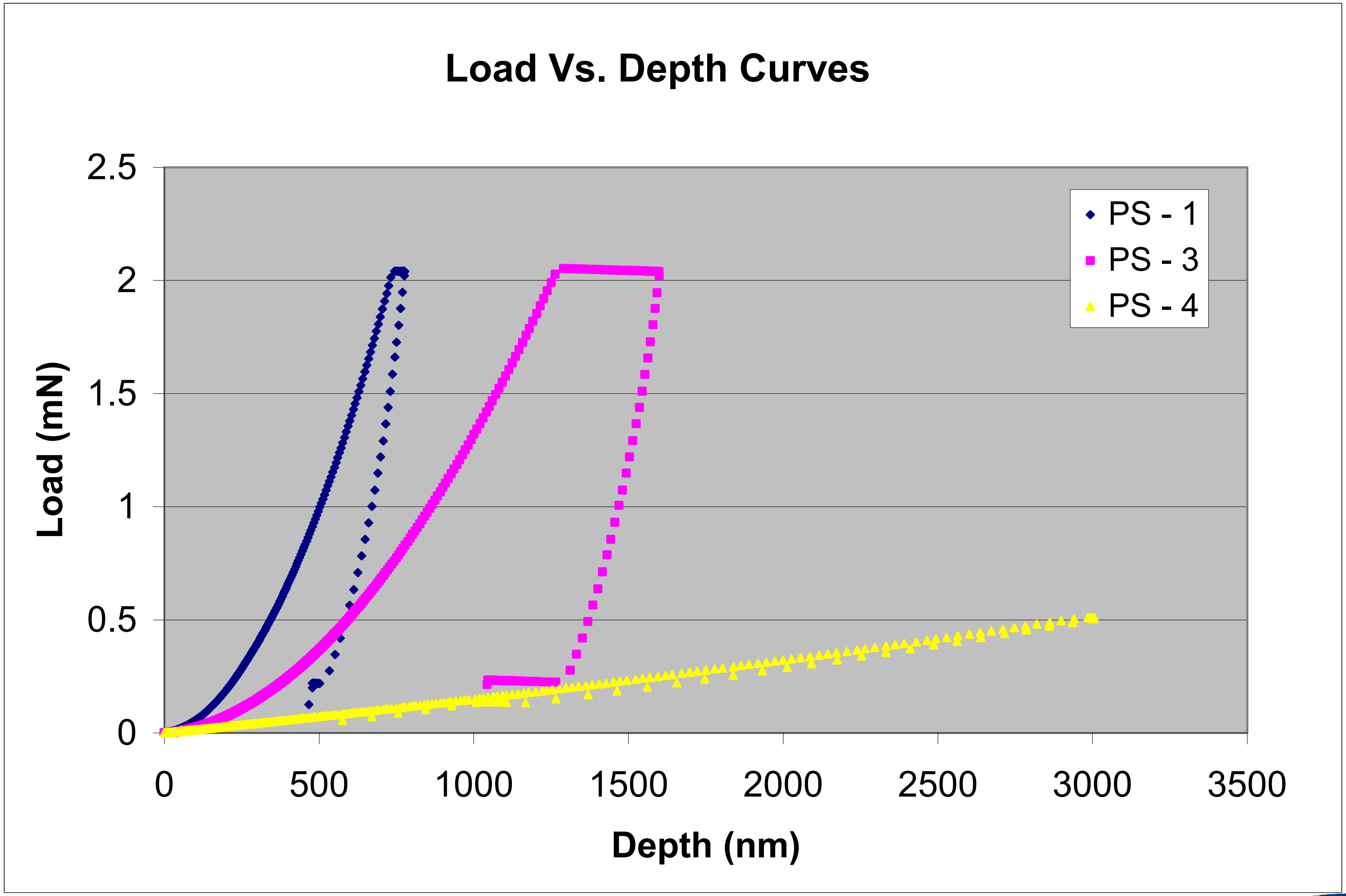
Schematic Indentation cycle



Standard Polymers: quasi-static response

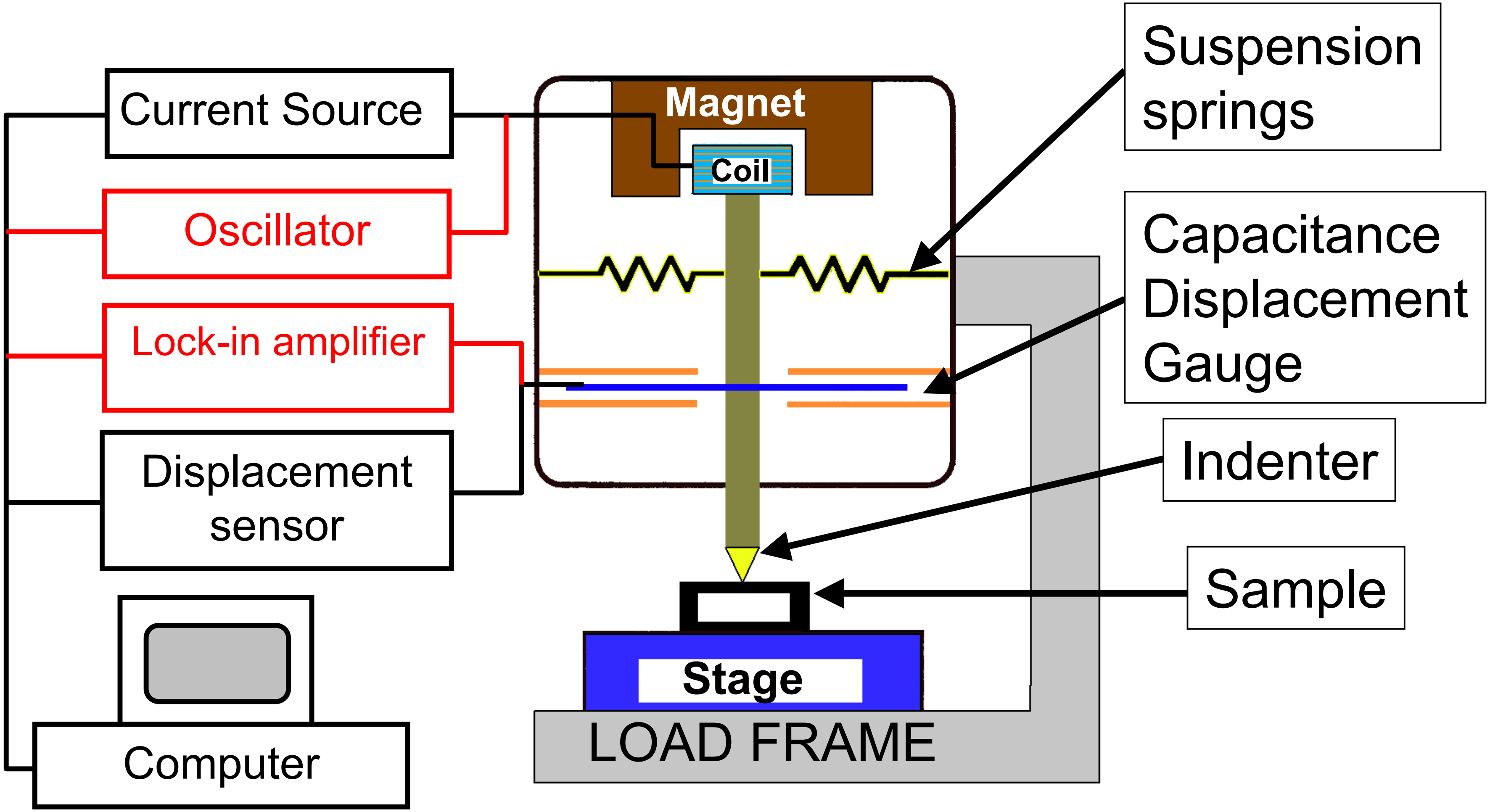


Motivation

- Requirement for local polymer properties in part design:-
  - bearings, gears, cams, press-fit parts, composites (matrix and interfaces)
- Requirement for properties of small volumes
  - e.g. mico-mouldings, packaging, coatings.
- Production control and QA via sensitivity of surface to production parameters.
  - Thermal history affects surface properties and can be detected by indentation.

Nanoindentation has the resolution but polymers have time/rate dependent properties.
$\Rightarrow$ Dynamic measurement methods are required!

Instrumented (Nano)indenter Schematic



Quasi-static / Dynamic

Scientific Objectives

- Validate indentation protocols for measuring loss and storage modulus and time constants of visco-elastic materials and feed into:
    - ISO standardisation (new work item)
    - Development of 1GPa certified reference material
- Compare methods to measure polymer properties as a function of frequency and temperature.
- Develop ultra-rapid indentation and creep-relaxation measurement methods for characterisation of visco-elastic materials.

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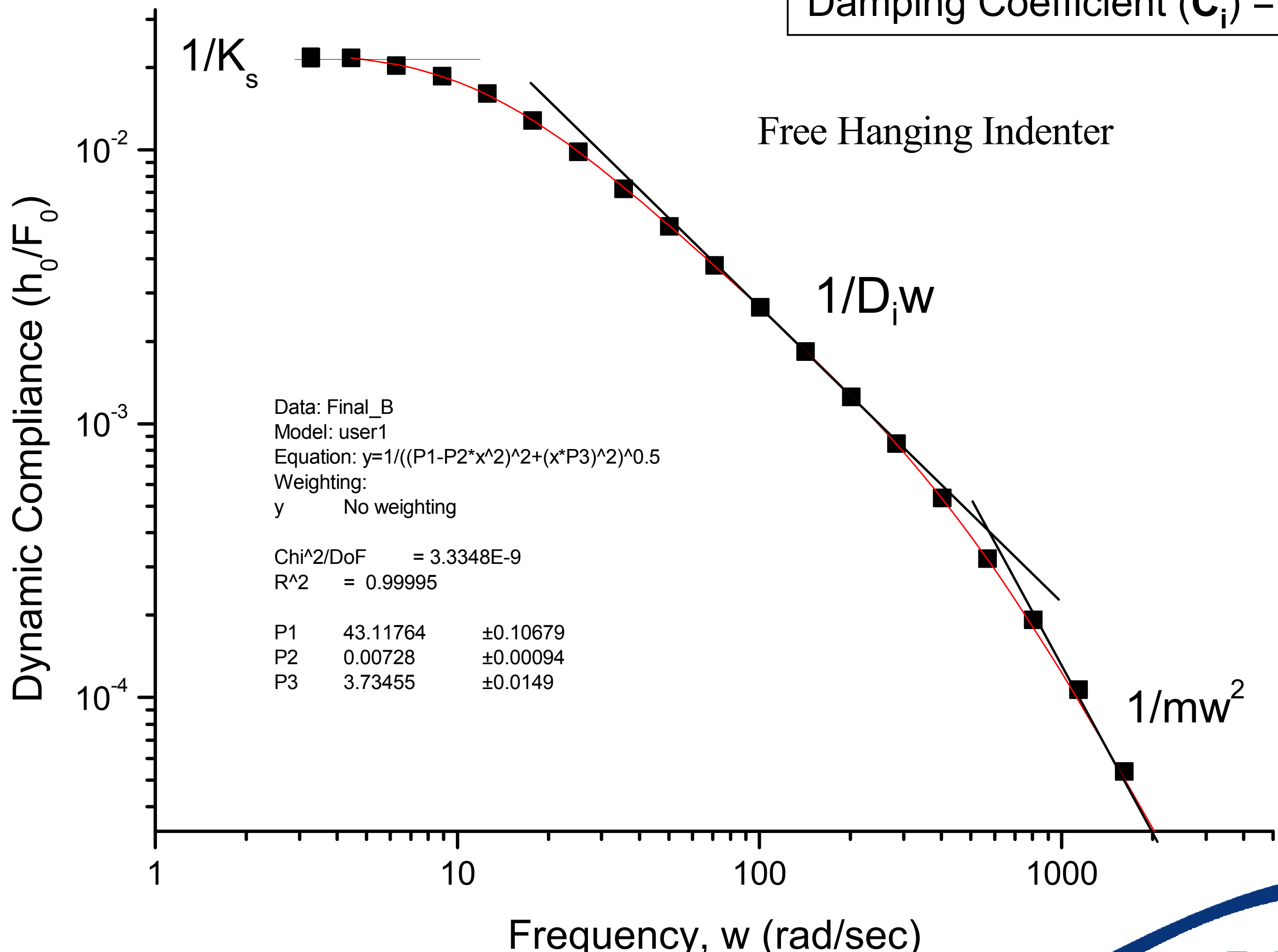
Dynamic Calibration #1

Support spring stiffness ($\mathbf{K_s}$) = 43.12 N/m
Indenter mass (**m**) = 0.0073 Kg
Damping Coefficient ($\mathbf{C_i}$) = 3.73 Ns/m



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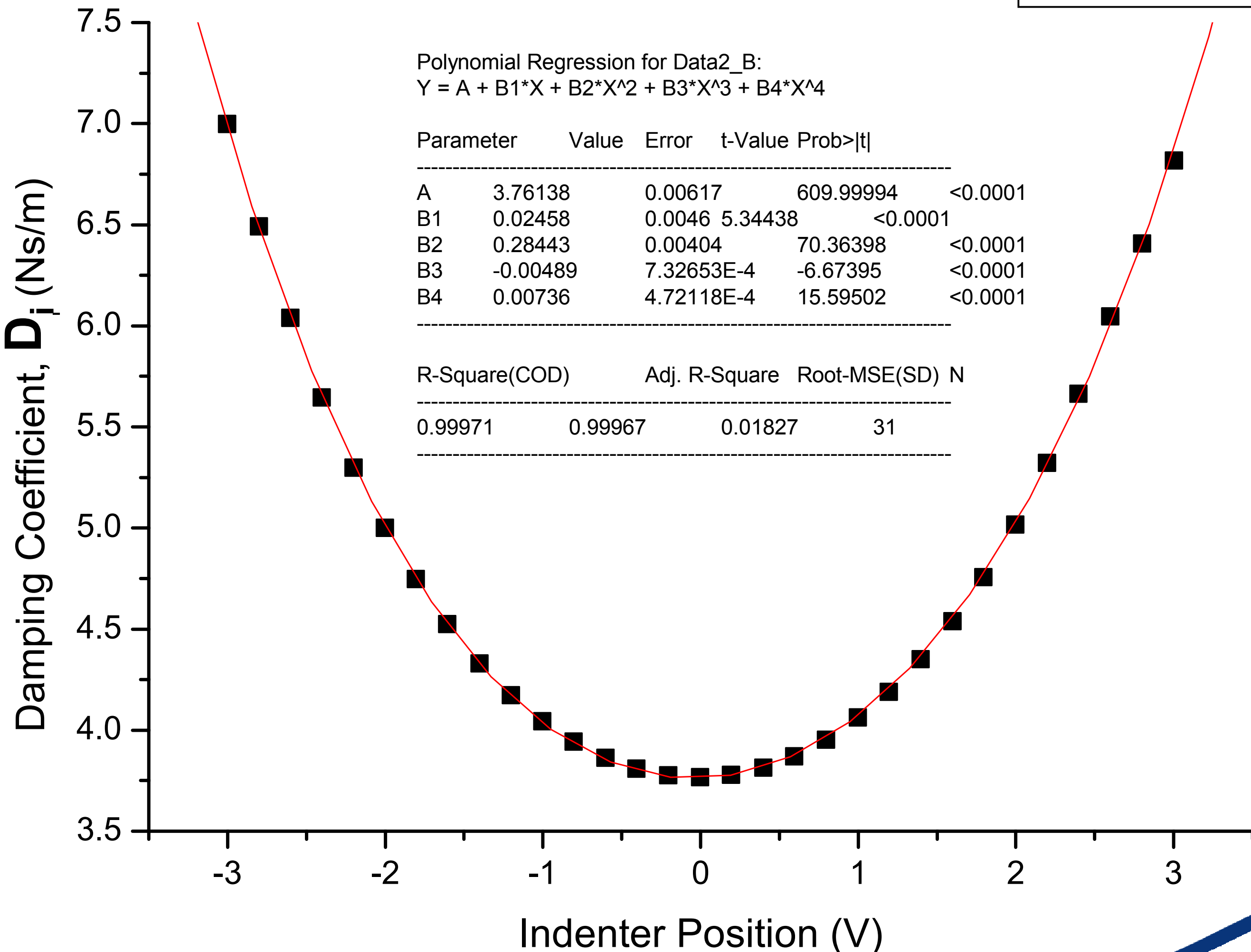
Dynamic Calibration #2

Damping Coefficient vs. Position

$$D_i = D_o + D_1x + D_2x^2 + D_3x^3 \text{ Ns/m}$$



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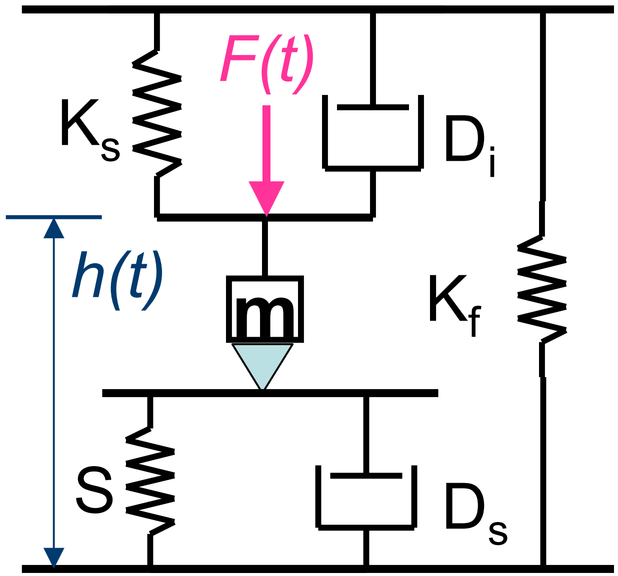
Spring and dashpot model



E′= Storage modulus

E″= Loss modulus

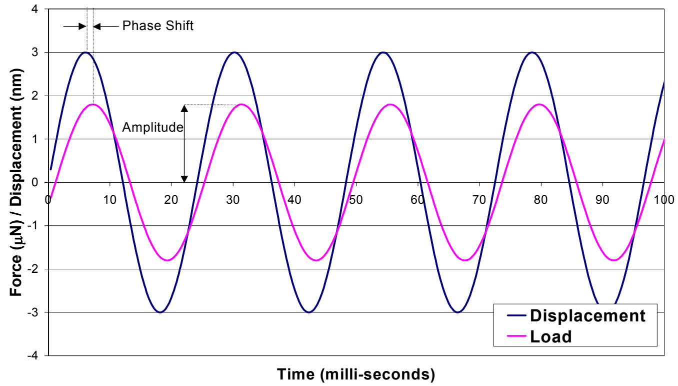
δ = Phase shift

Tan δ = Damping coefficient

$$\frac{F_0}{h_0} = \sqrt{\omega^2 D_i^{\ 2} + \left(K_s - m\omega^2\right)^2}$$

$$\sigma = \left(E' + iE''\right)\varepsilon$$

$$\tan\delta(\omega) = \frac{E''(\omega)}{E'(\omega)}$$



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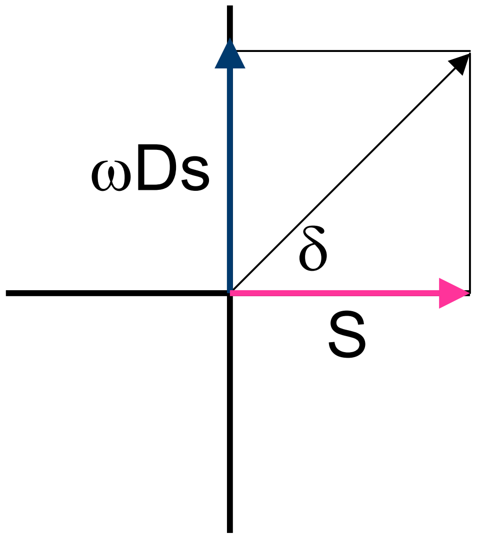
Dynamic contact mechanics



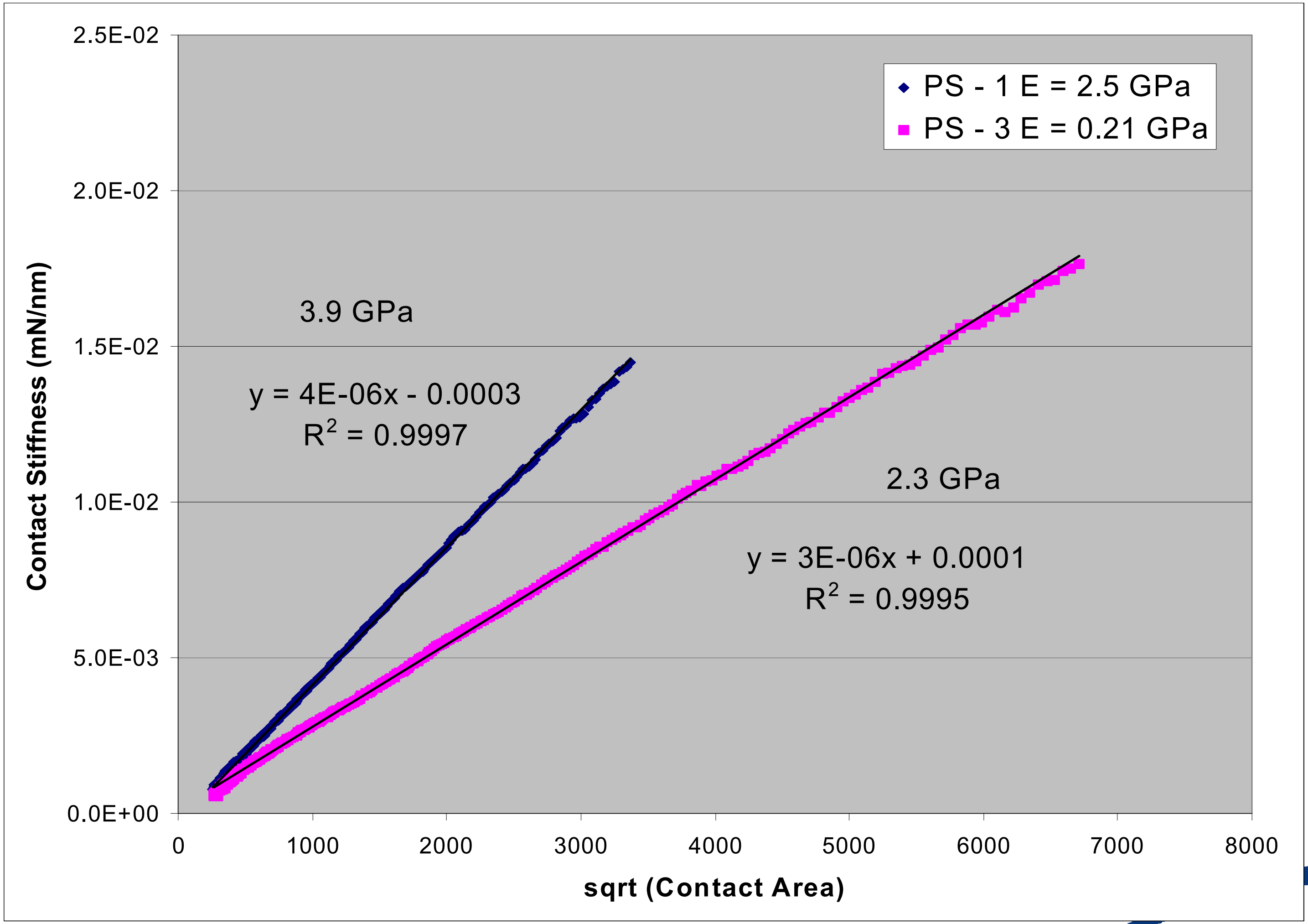
$$E' = \frac{S\sqrt{\pi}}{2\sqrt{A(h_c)}}$$ Storage Modulus

$$E'' = \frac{\omega D_s\sqrt{\pi}}{2\sqrt{A(h_c)}}$$ Loss Modulus

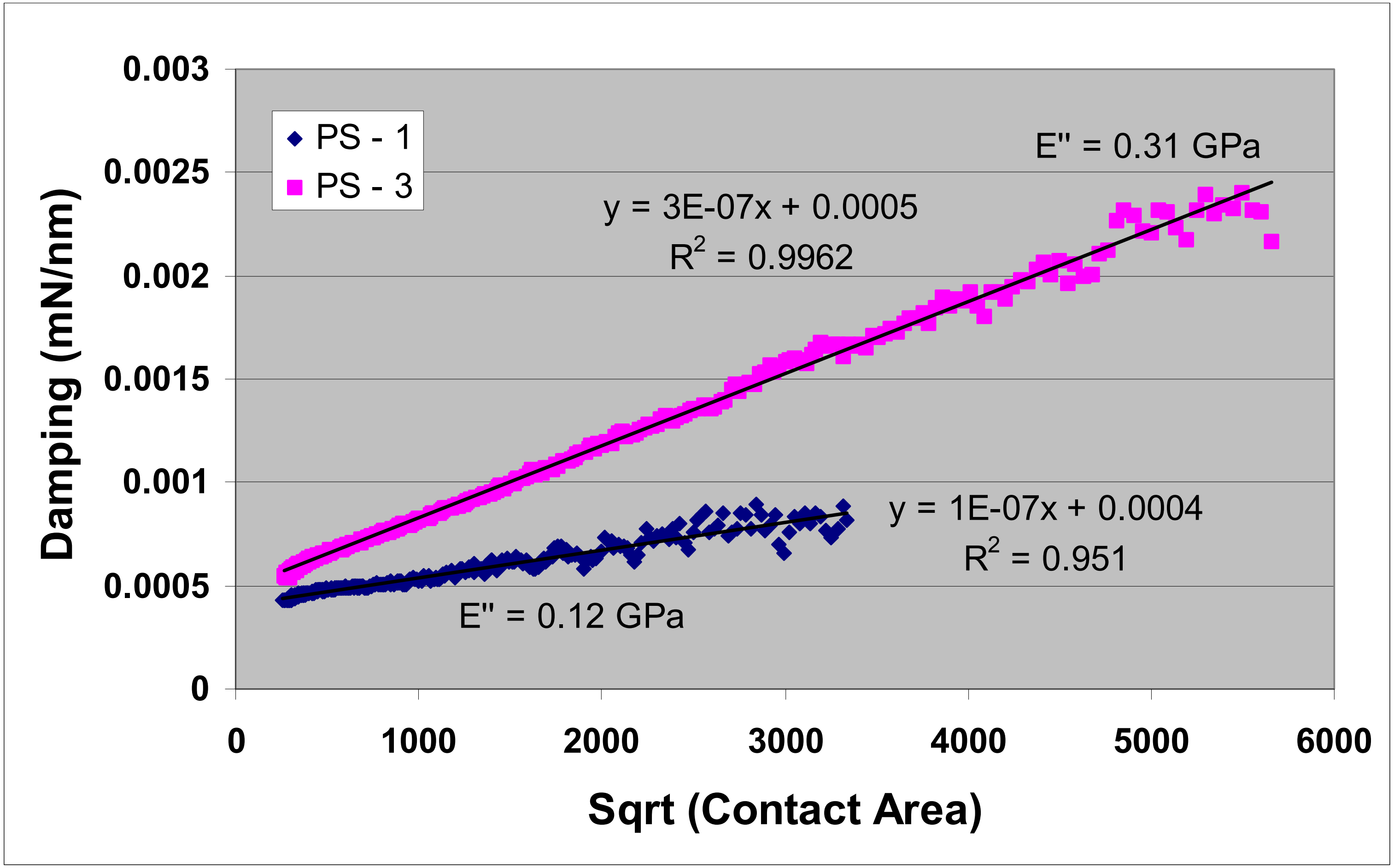
Results are available point wise

Or by plotting S or D vs. $\sqrt{A(hc)}$

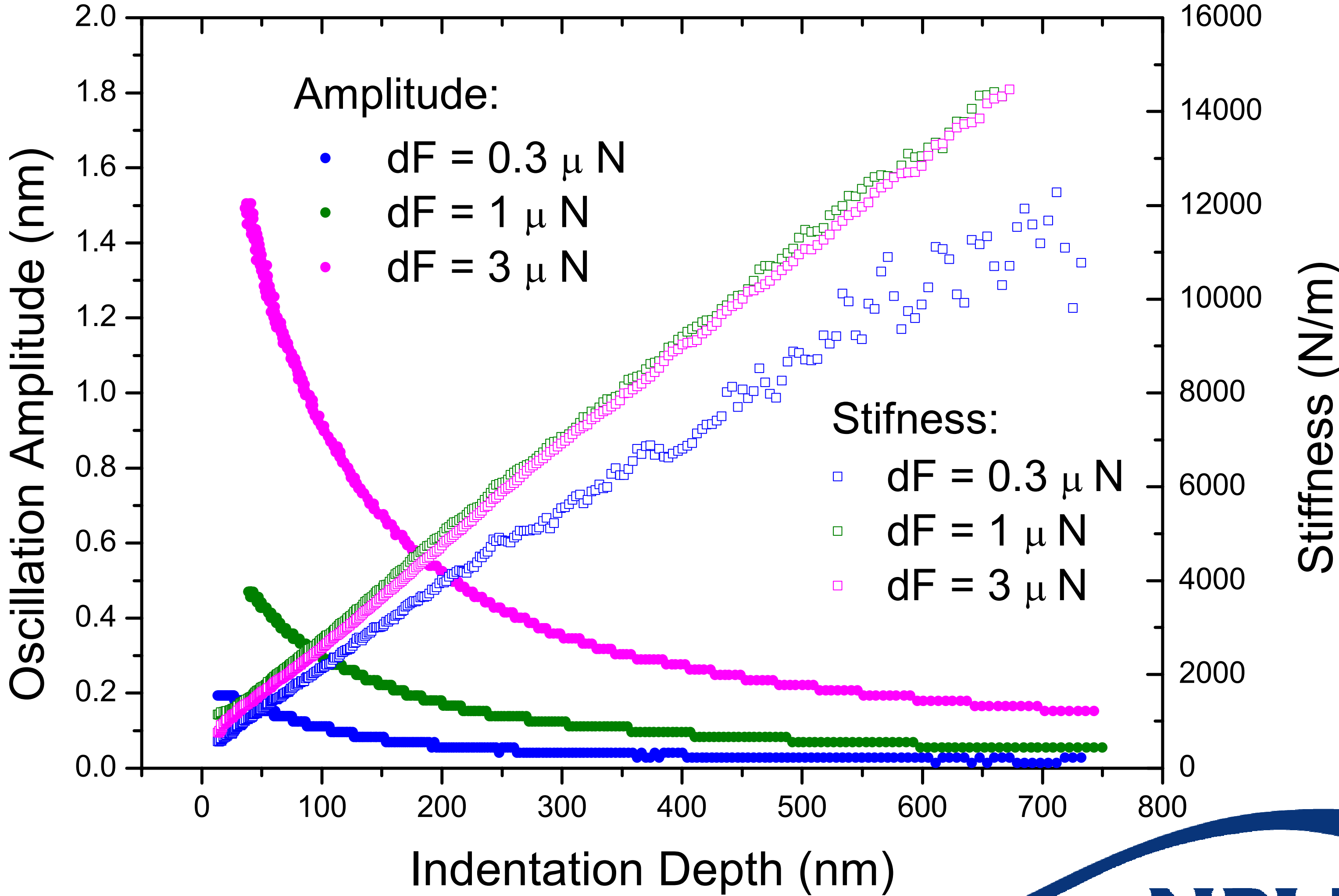
Storage modulus by graphical method



Loss Modulus by graphical method



PS1: Stiffness dispersion vs. drive amplitude



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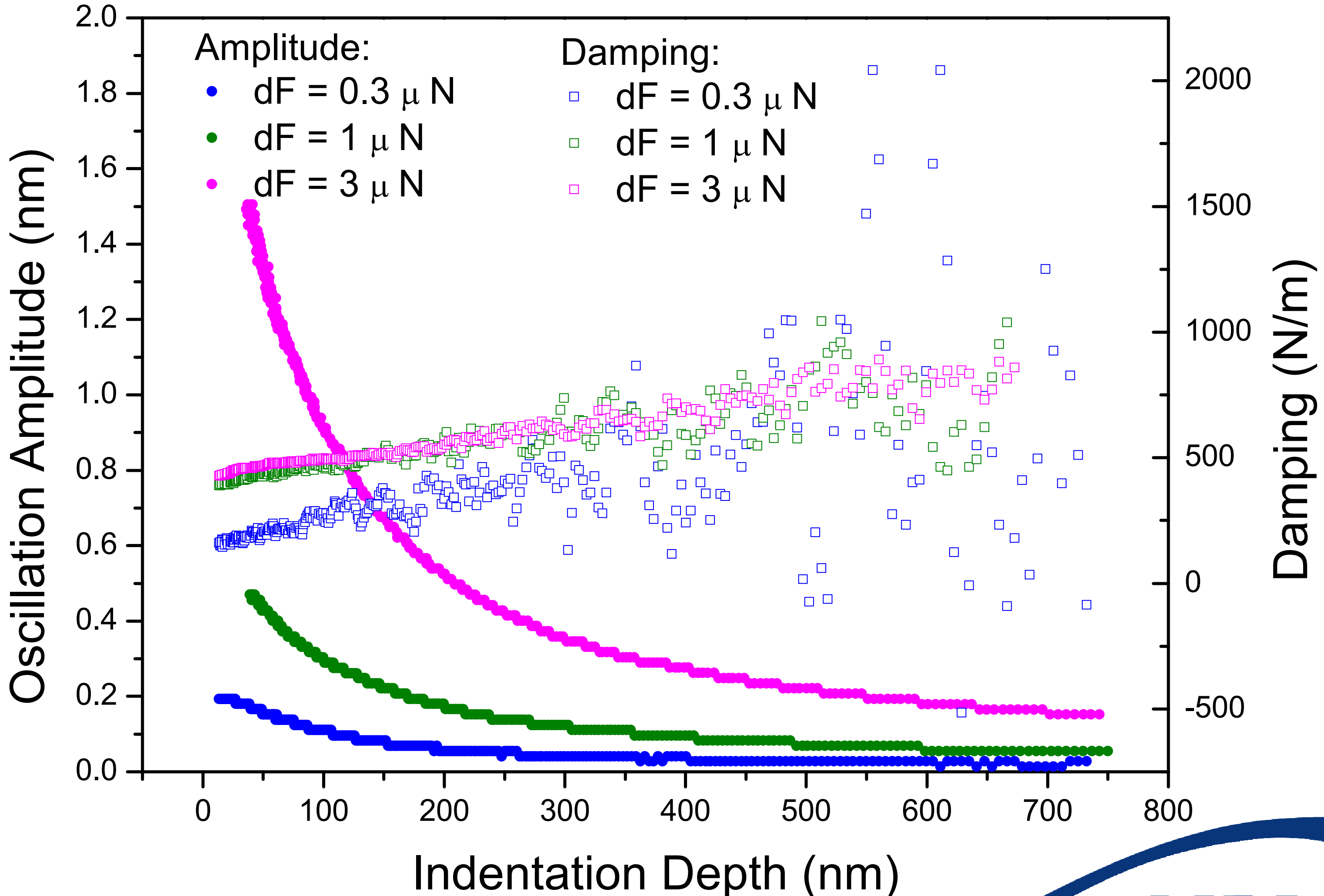
PS1 Damping dispersion vs. drive amplitude



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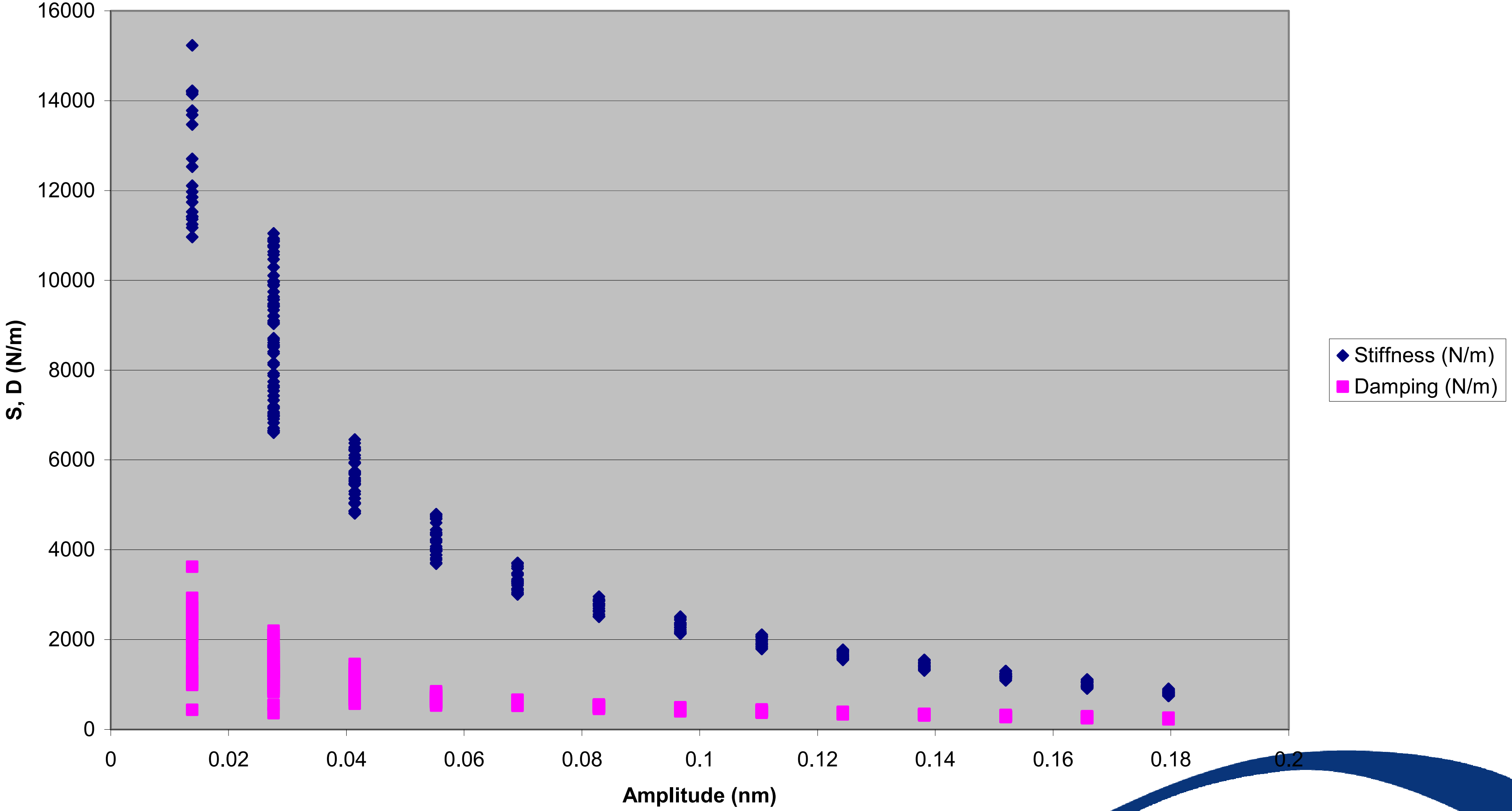
PS3 Data uncertainty vs. drive amplitude



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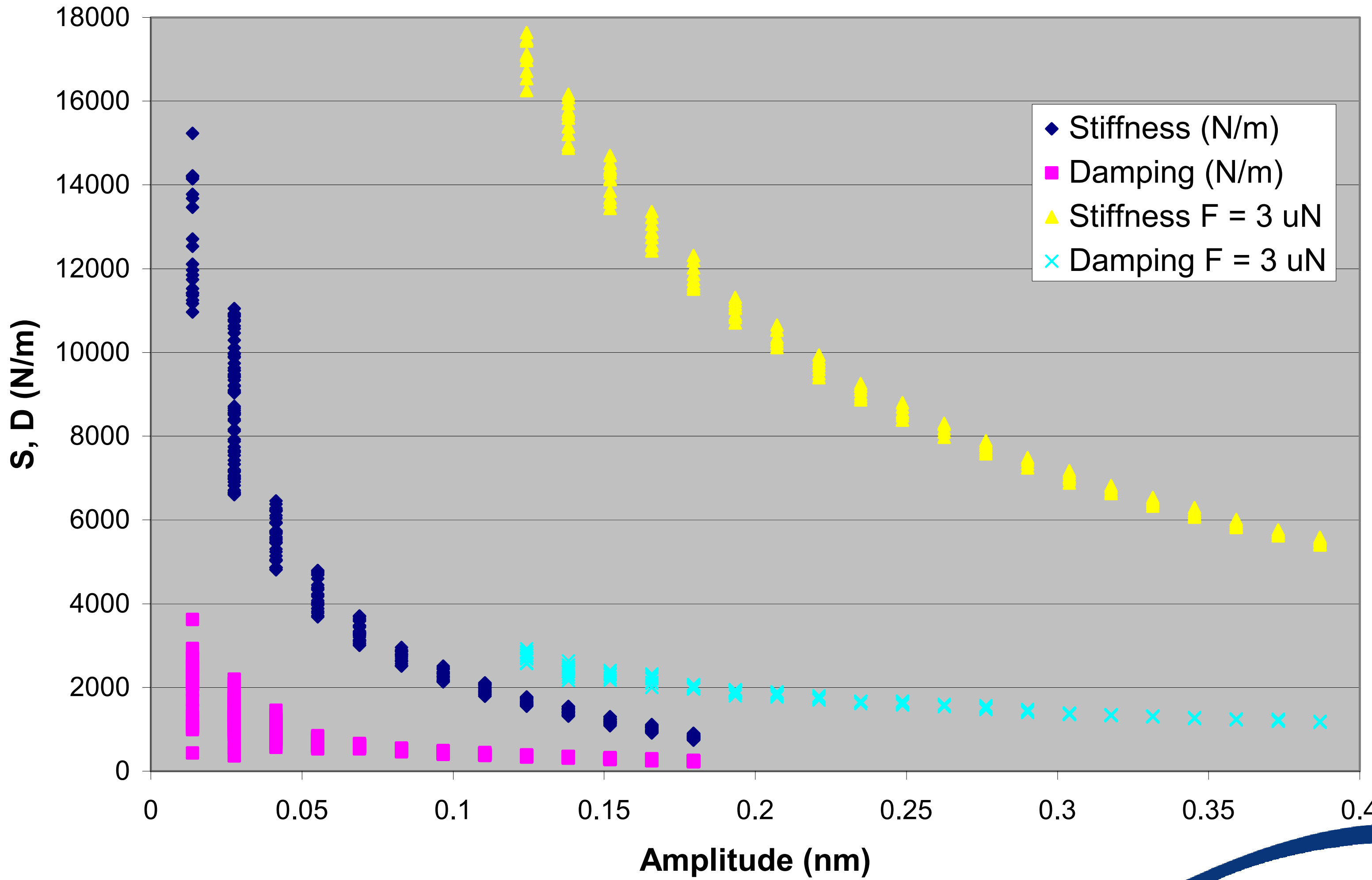
Effect of amplitude on dispersion PS3



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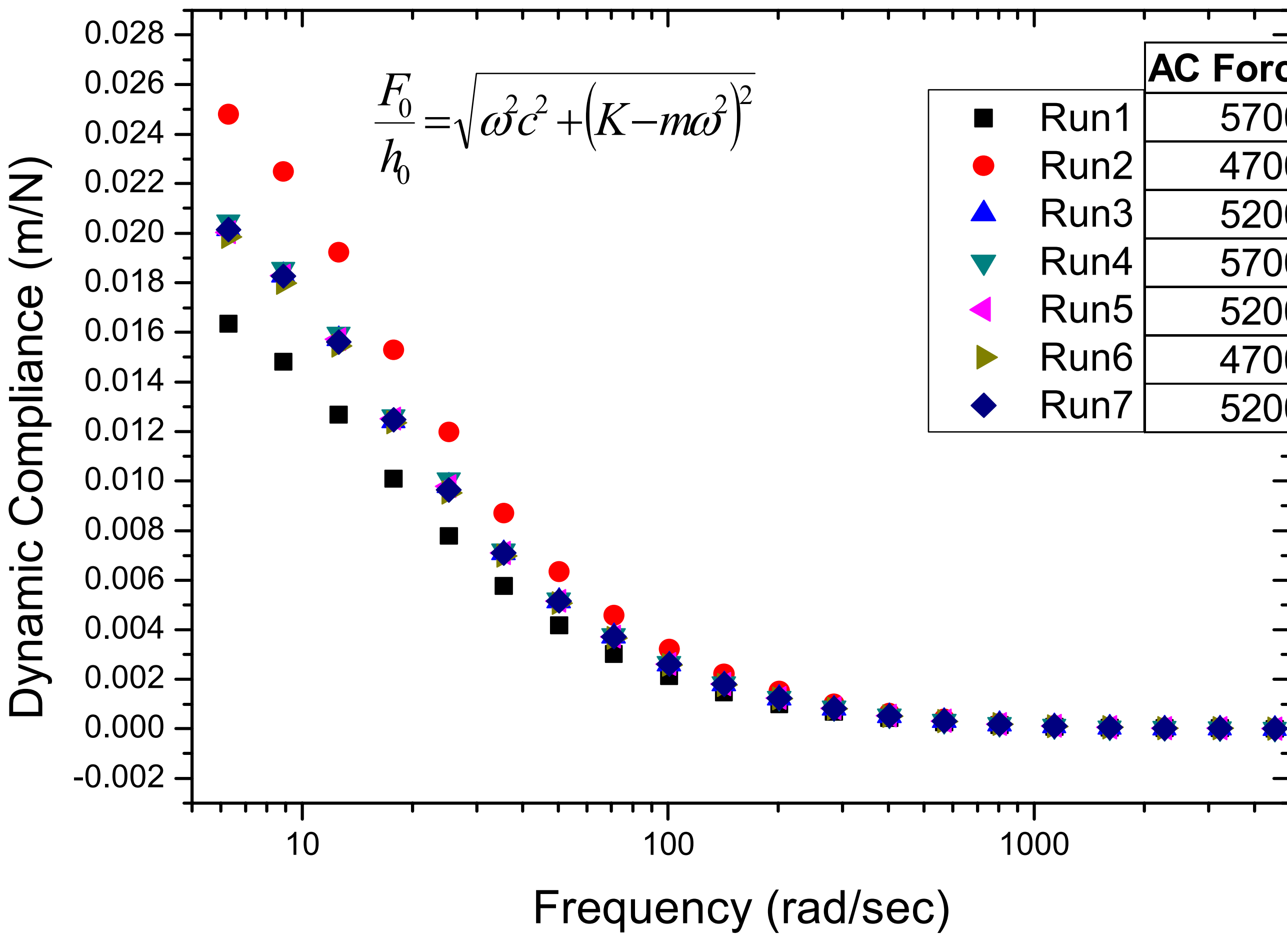
Sensitivity to static calibration



	AC Force Cal.	AC Displacement Cal
Run1	57000	12000
Run2	47000	15000
Run3	52000	13500
Run4	57000	15000
Run5	52000	13500
Run6	47000	12000
Run7	52000	13500

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Sensitivity of E', E" to dynamic calibration

Er' and Er" calculated from slope of Stiffnes and Dar		
Run	**Factor 1 Ko (N/m)**	**Factor 2 m (Kg)**
1	44.5	0.008
2	42	0.005
3	44.5	0.008
4	47	0.005
5	44.5	0.008
6	42	0.011
7	47	0.011
8	53.87	0.00857
9	35.55	0.0058
Original	43.12	0.00728

Software sensitivity design

Run	Factor 1 Ko (N/m)	Factor 2 m (Kg)
1	44.5	0.008
2	42	0.005
3	44.5	0.008
4	47	0.005
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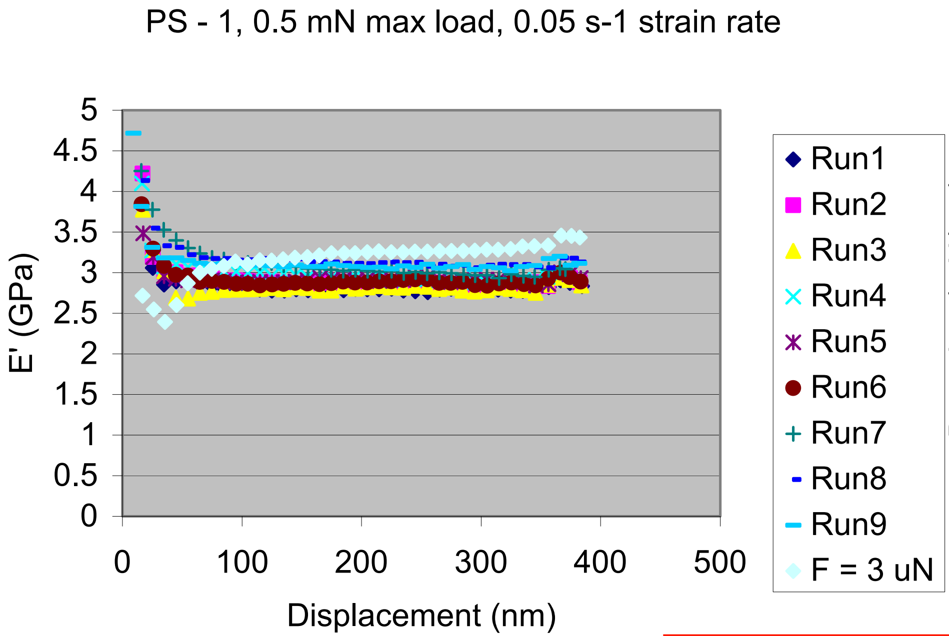
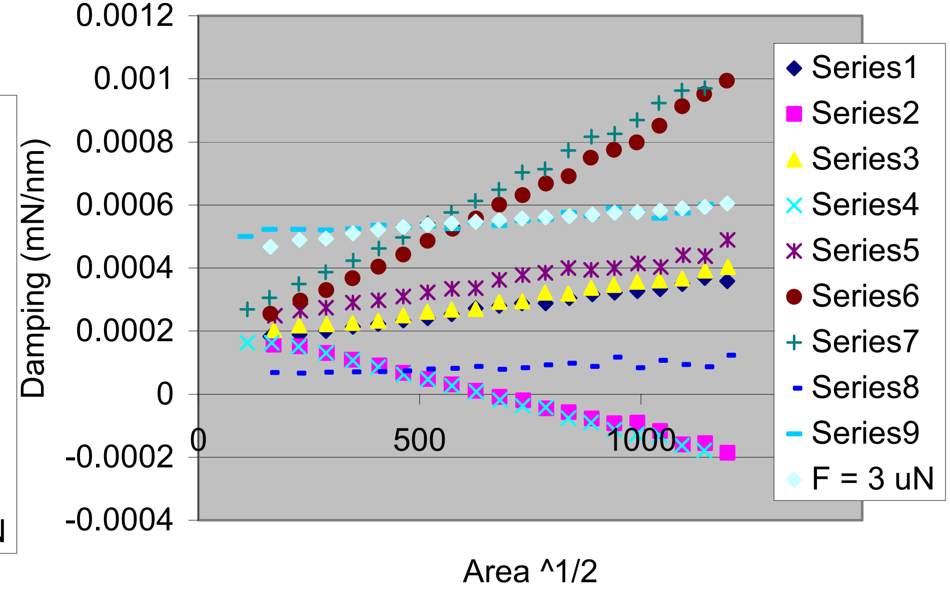
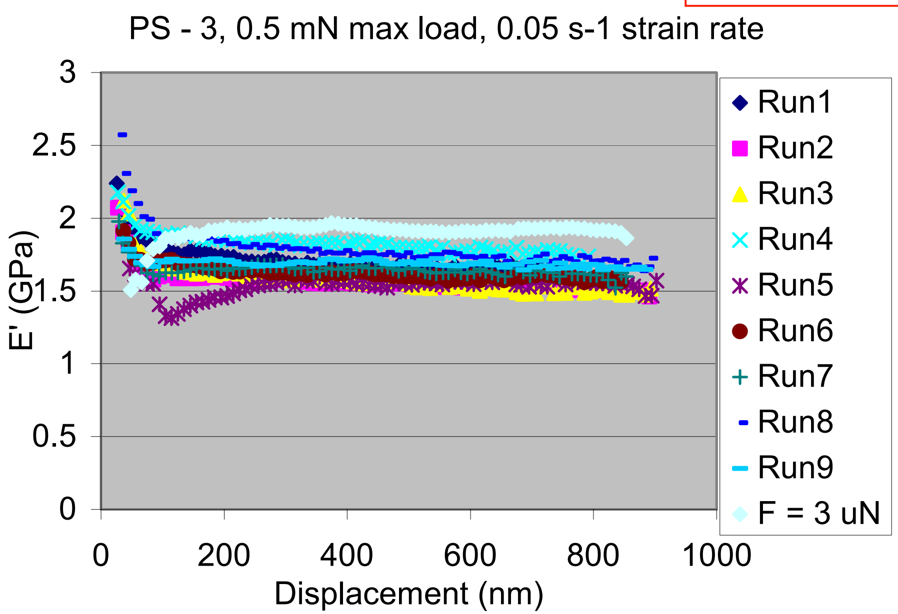
6	42	0.011
7	47	0.011
8	53.87	0.00857
9	35.55	0.0058

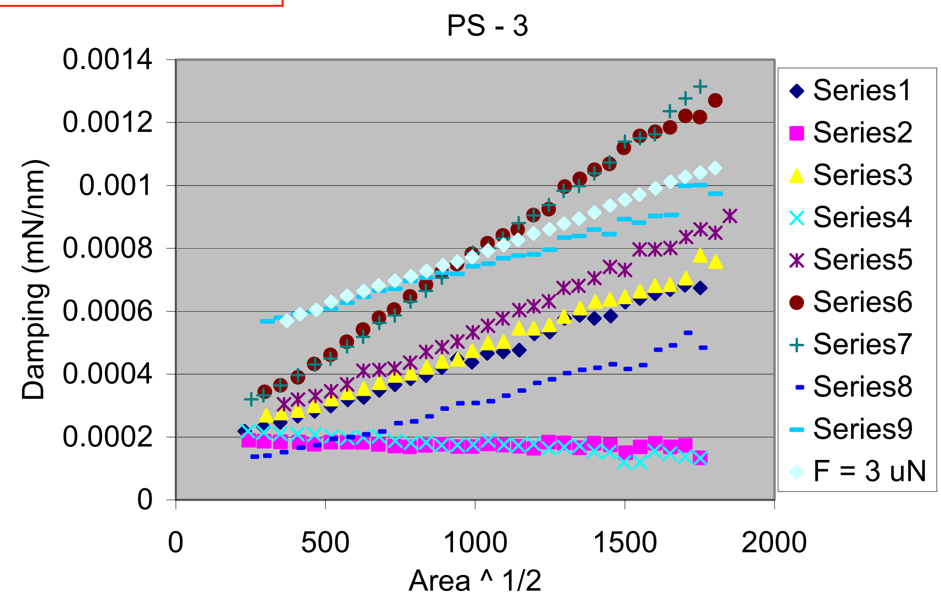
Experimental Sensitivity design

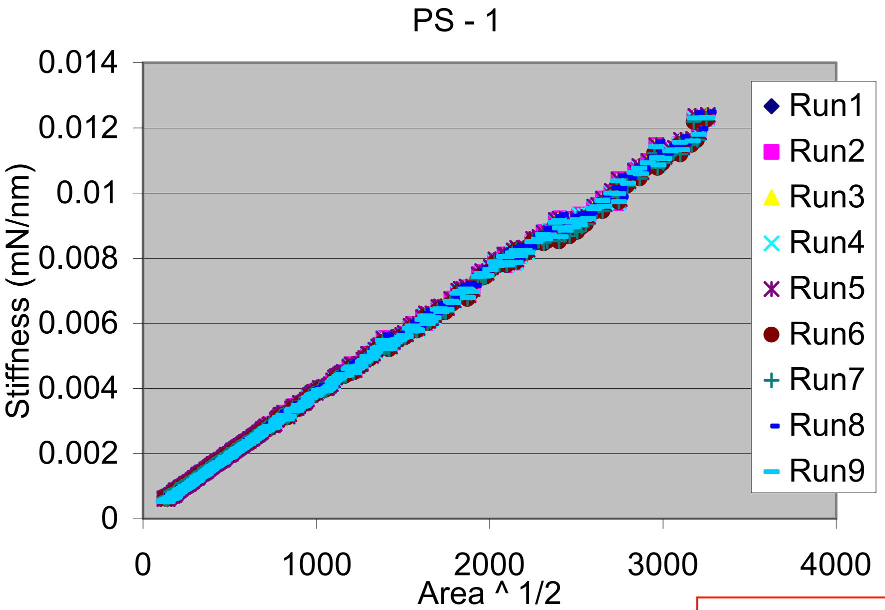


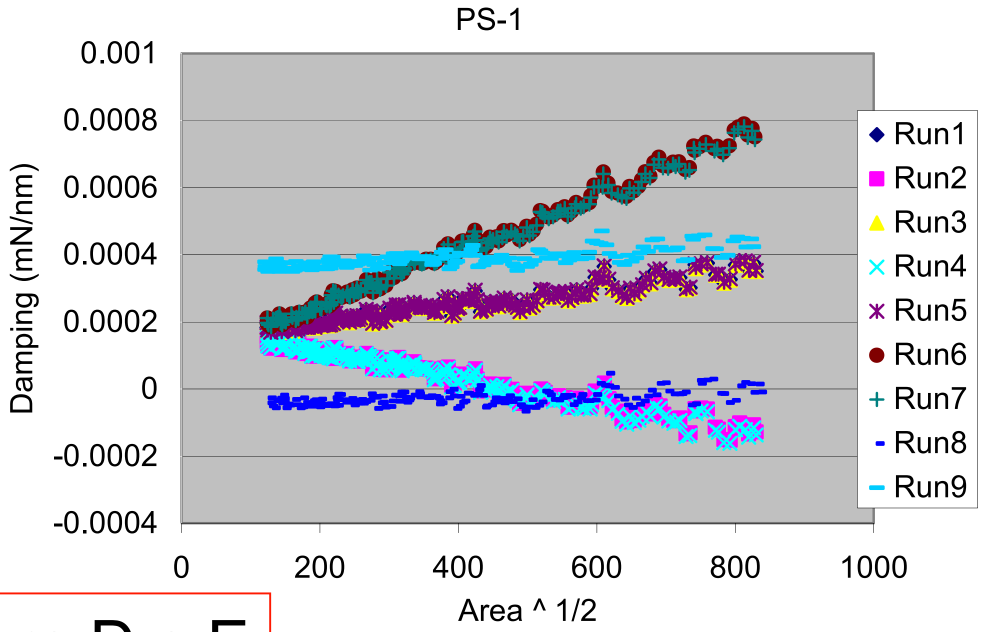


Experiment D.o.E

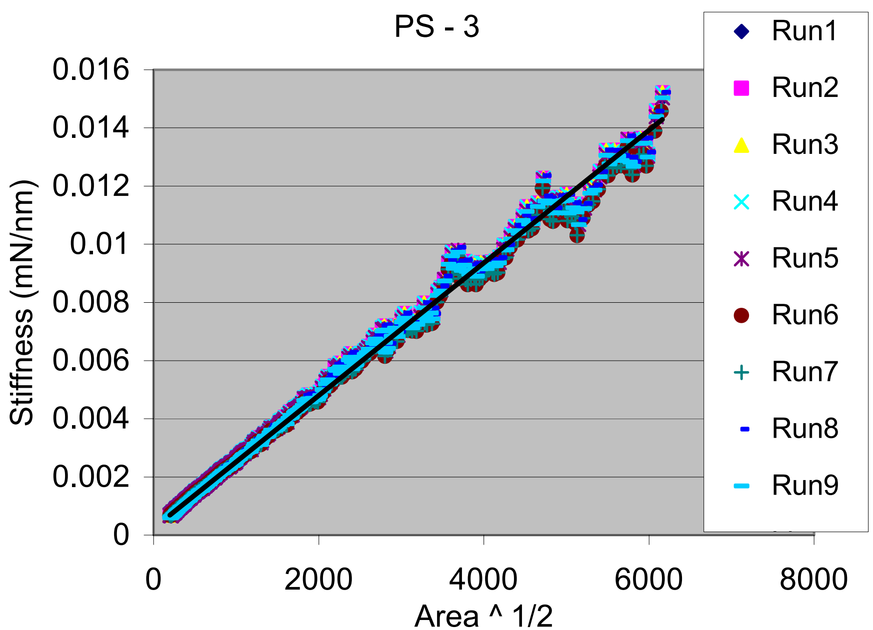


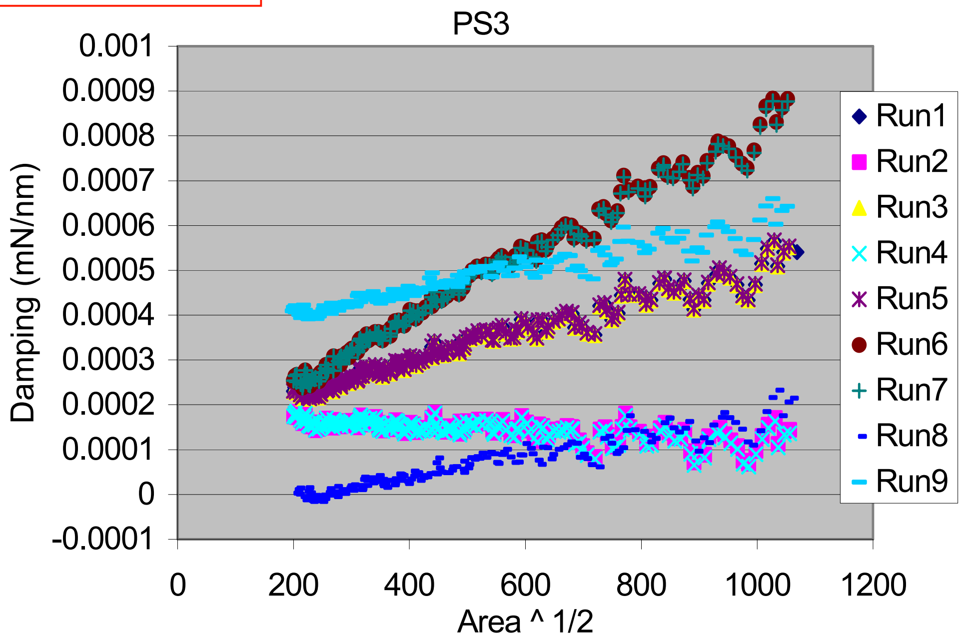




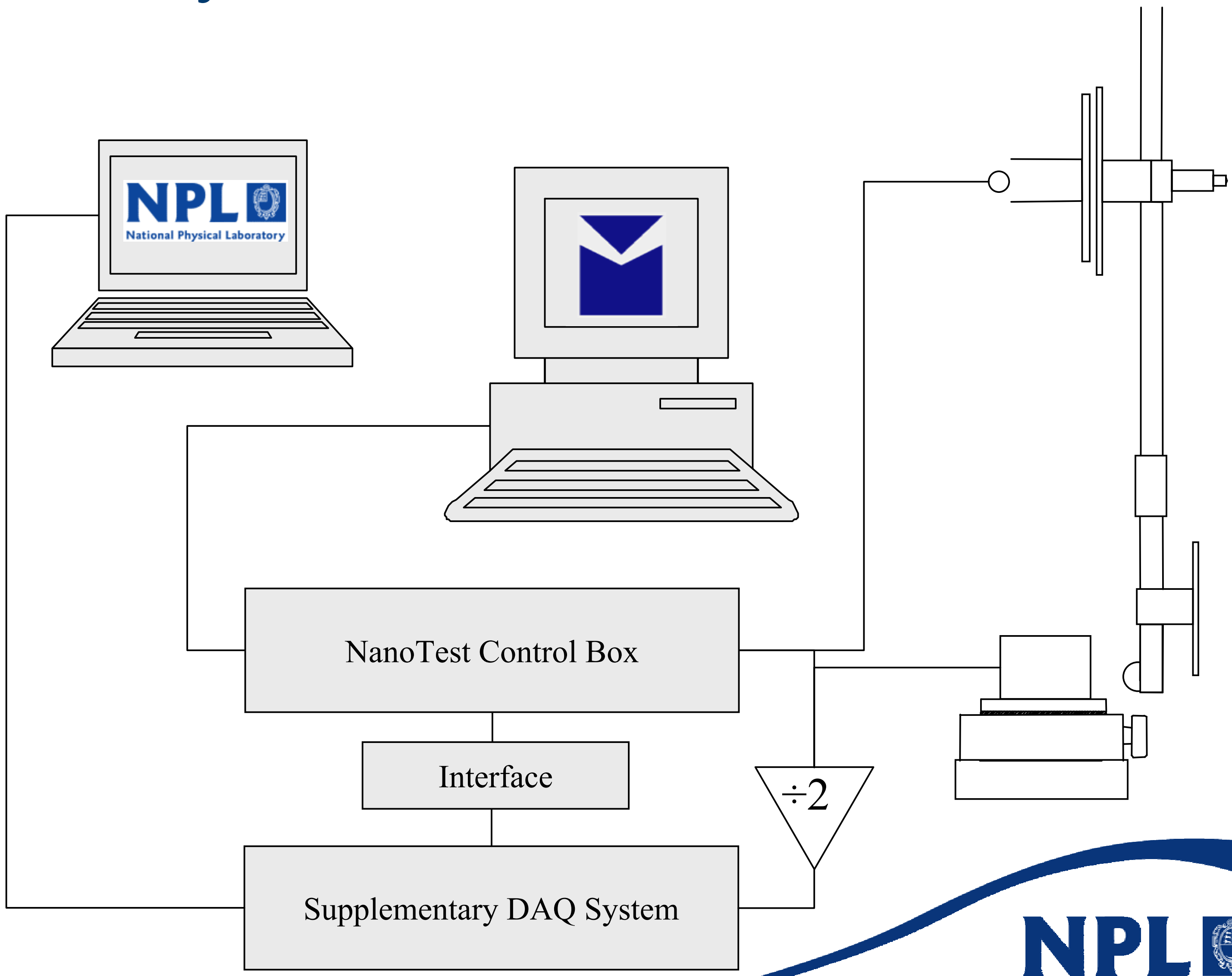


Software D.o.E





Ultra-fast dynamic indentation



Dynamic indentation

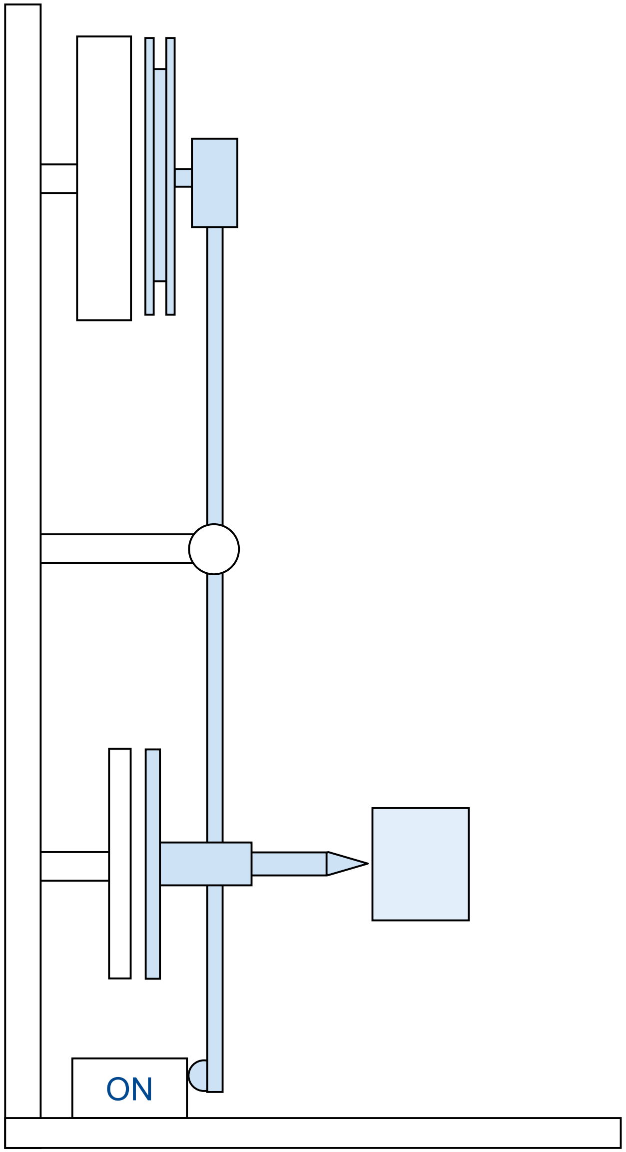
NanoTest (Micro Materials Ltd, Wrexham)

- Dynamic nanoindentation
- High and low rate indentations
- Displacement monitored with time

vs. Lock-in amplifier Amplitude & Phase

Test Variables

- Impact energy
- Oscillation Frequency
- Strain rate
- Test probe geometry
- Temperature



Dynamic indentation

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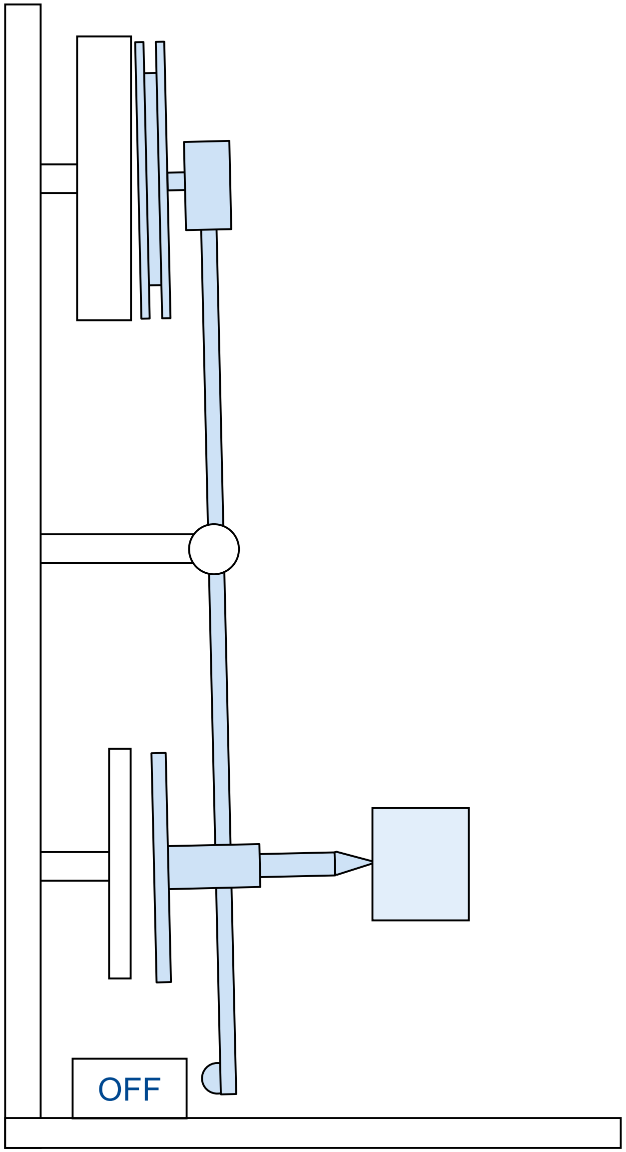
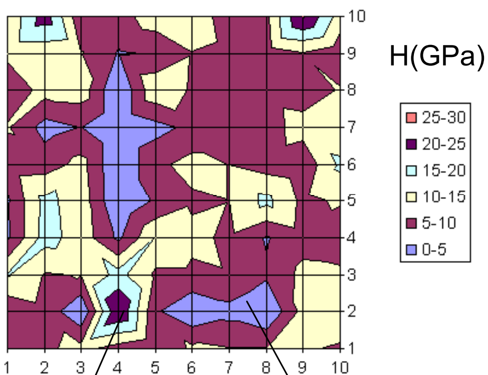


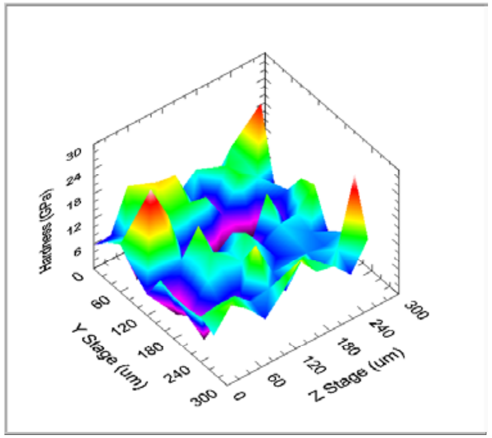
High-temperature Testing:
0.1 mN to 20 N

MicroMaterials NanoTest

Hot stage – sample temp ≤500°C

Mapping the hardness of tribological coatings

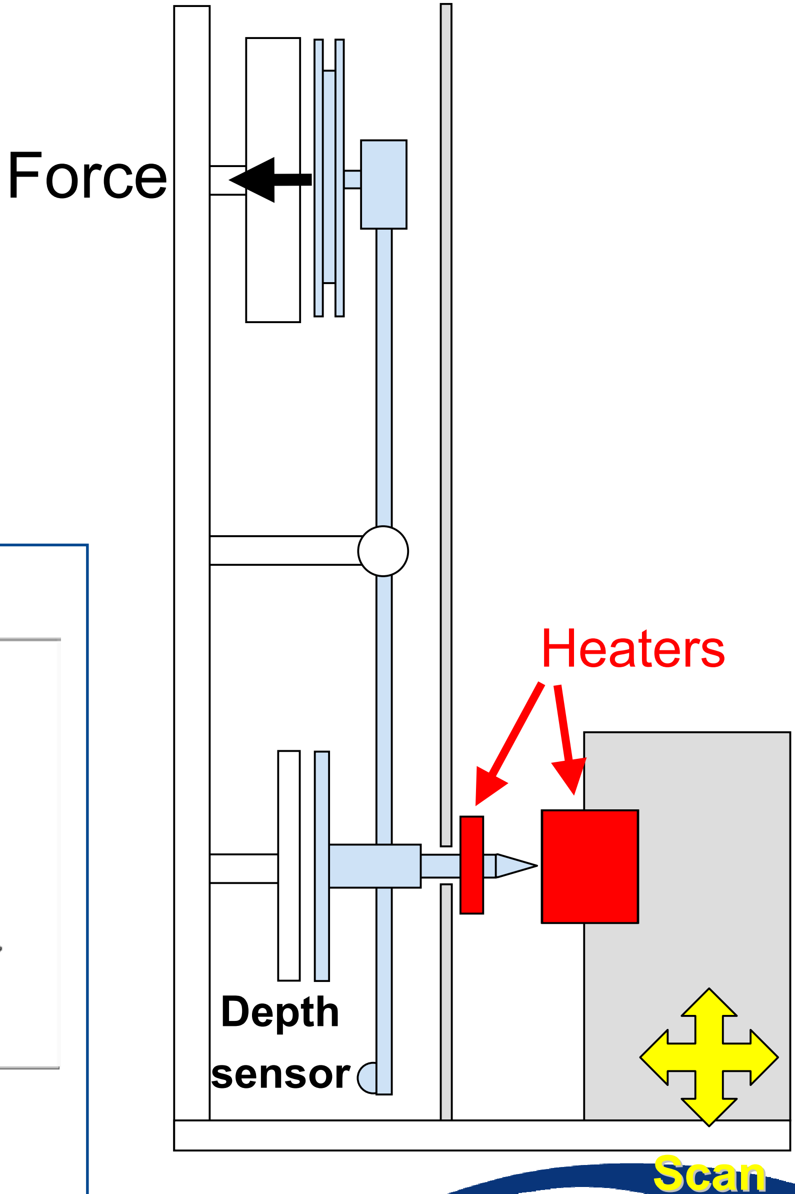




Harder phase to provide abrasive resistance

Softer phase to provide toughness

5.2 micron Ion-beam Assisted Deposition (IBAD) Alumina; Stephen Abela, Unversity of Malta



Stage Plans

- **Stage 1 (1/4/06 – 31/6/07)**
  - **Selection/procurement of materials**
  - **Calibration and algorithm sensitivity studies**
  - **Development of dynamic calibration method (NPL Report)**
- **Stage 2 (1/4/07 – 31/3/08)**
  - **Design and test temperature stage**
  - **Development of ultra-rapid indentation method**
  - **Frequency, sweep and chirp**
  - **High rate indentation method (scientific paper)**
- **Stage 3 (1/4/08 – 31/3/09)**
  - **Visco-elastic models**
  - **Evaluation of temperature stage**
  - **High Rate indentation method at elevated temperatures (scientific paper)**
  - **Validated protocols/Input to ISO standards**

Industrial involvement

- Materials supply
  - Highly reproducible “standard” polymers
  - Industrial components / materials
- Collaboration for characterisation comparisons
  - Property data from other test methods as function of frequency and temperature
- Case studies / feasibility of adoption of method

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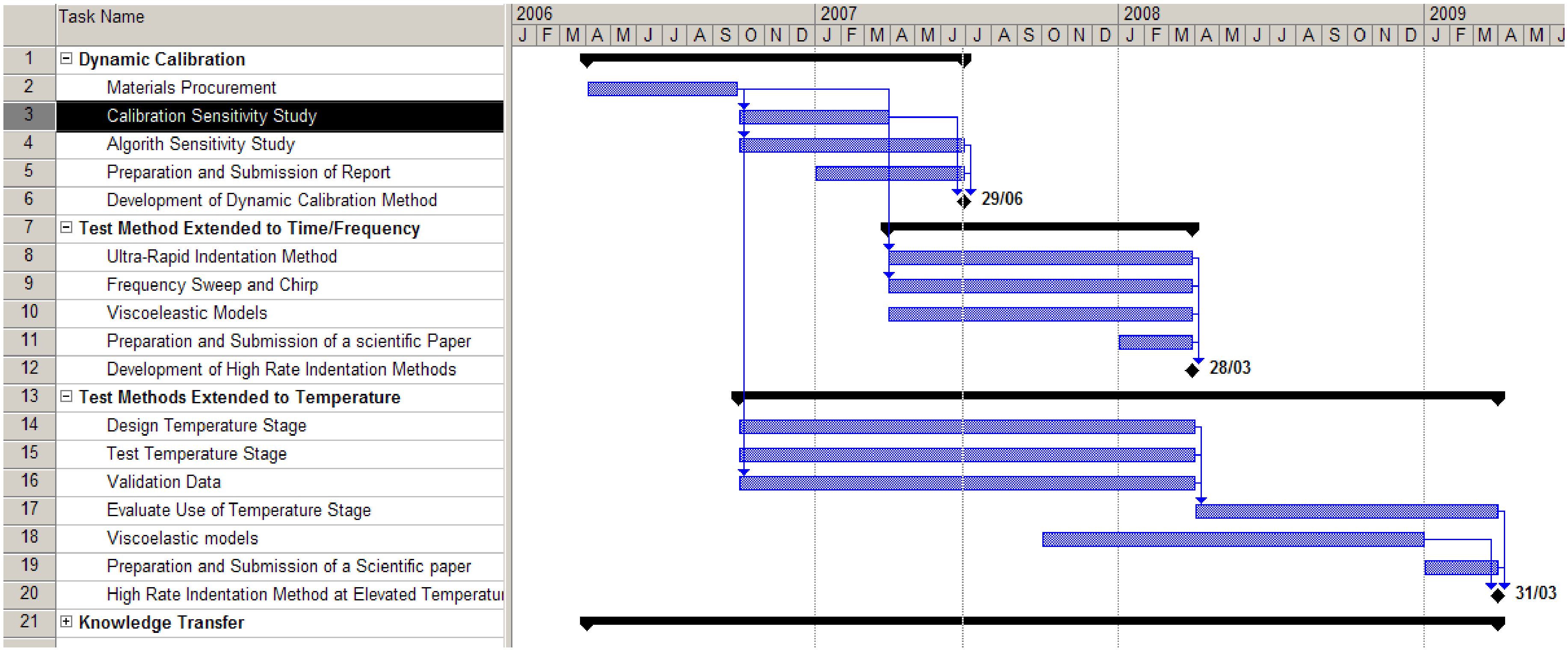
Deliverables

Project Description

- **D1: Validated protocols for room temperature measurement of mechanical properties of polymer surfaces or coatings (NPL Report).**
- **D2: Refinement of current methods into intelligent miniaturised tests (scientific paper).**
- **D3: Mechanical properties of polymer surfaces or coatings as a function of temperature (scientific paper).**

SE02 GANNT chart



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