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

Lead Scientist: Nigel Jennett

Project Manager: Lesley Henderson

Characterisation Programme

(2006-2009)

Presentation outline

- Introductions to:
  - SE02 science team
  - The project motivations
  - Instrumented indentation
- SE02 Project outline
- Industrial involvement

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SE02 Science Team

Surfaces, Coatings and Nano-Mechanics Group

- Nigel Jennett (Lead Scientist)
- Miguel Monclus
- John Nunn
- Tony Maxwell (now in Polymers group)

Modelling / Finite Element Analysis

- Louise Crocker

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Motivation

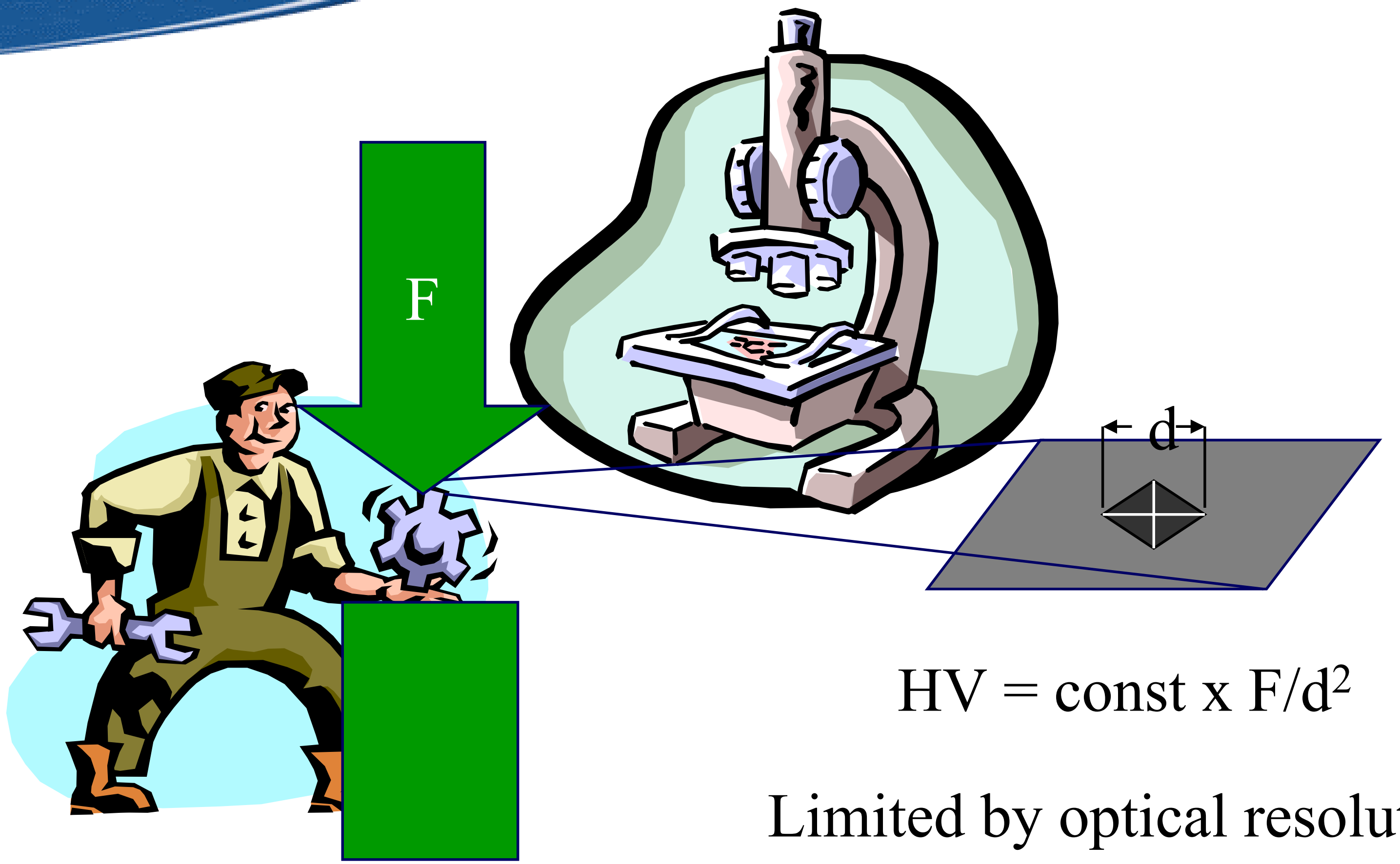
- Requirement for local polymer properties in part design:-
  - bearings, gears, cams, press-fit parts, composites (matrix and interfaces)
- Requirement for properties of small volumes
  - e.g. mico-mouldings, packaging, coatings.
- Production control and QA via sensitivity of surface to production parameters.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

Scientific Objectives

- Validate indentatin 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.

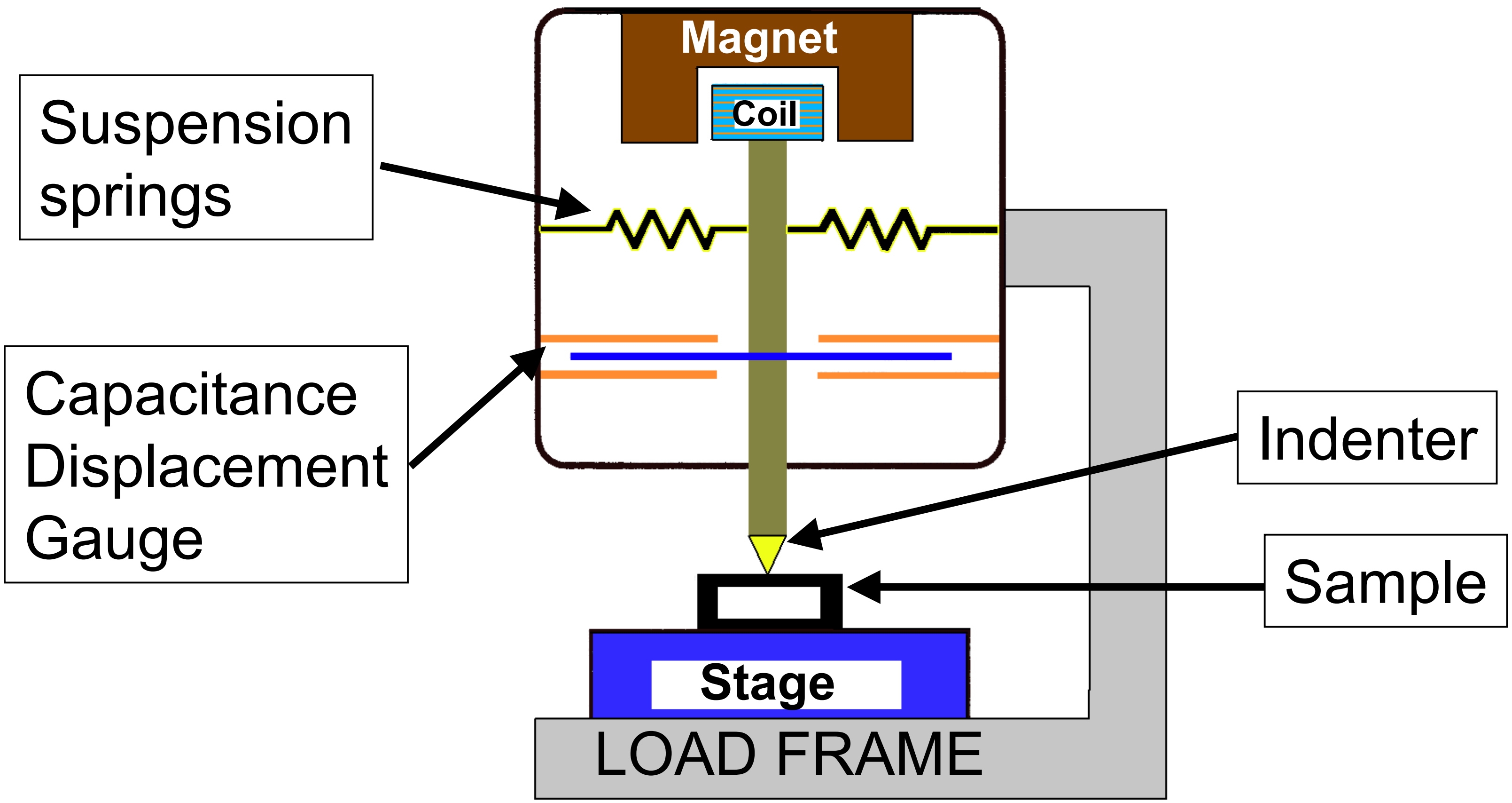
Traditional Hardness – a quick and easy QA tool



$HV = const \times F/d^2$

Limited by optical resolution

Instrumented Nanoindenter Schematic



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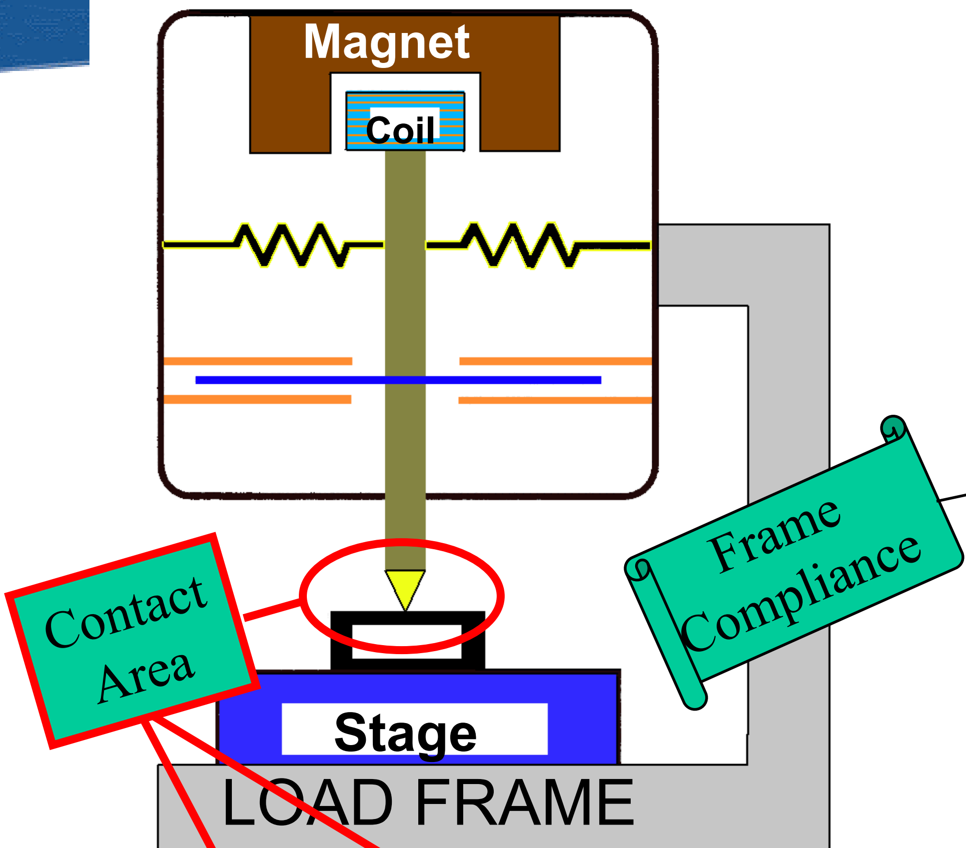
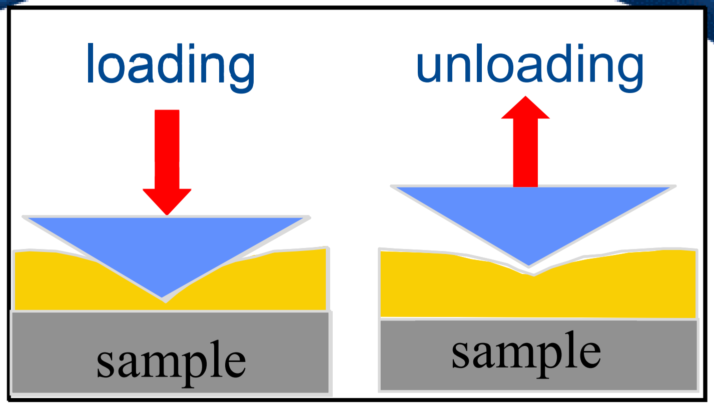
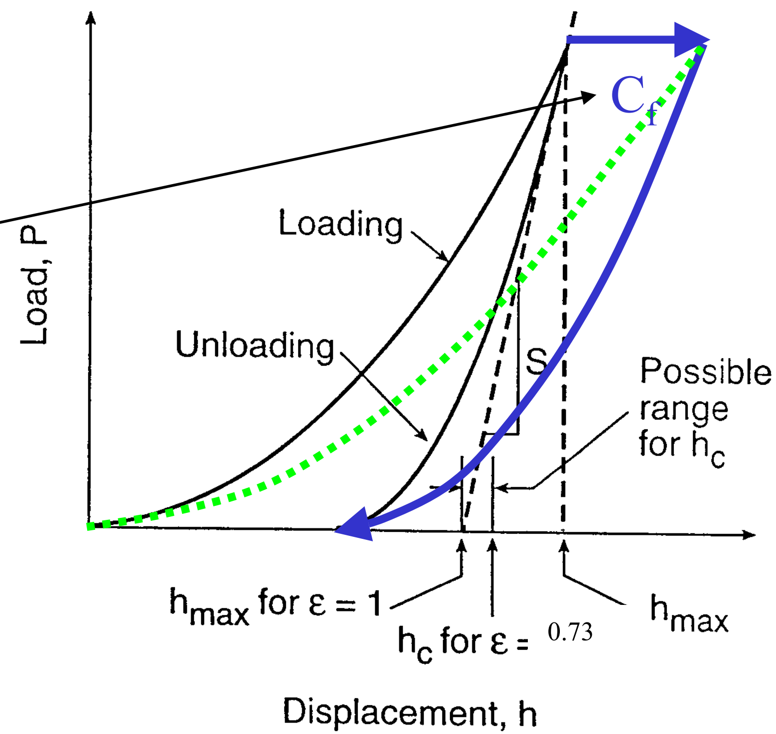
Instrumented (Nano)indentation







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

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

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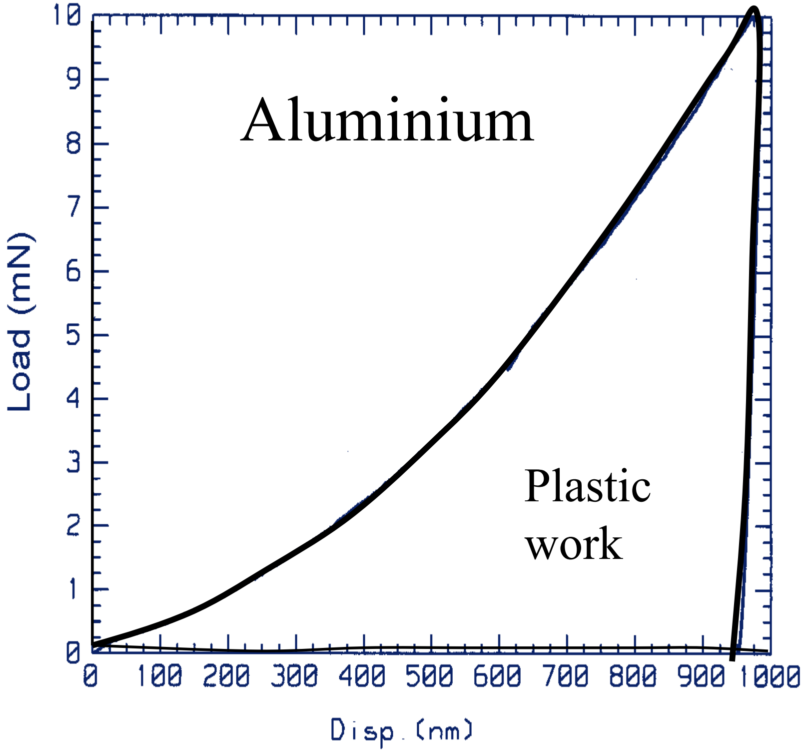
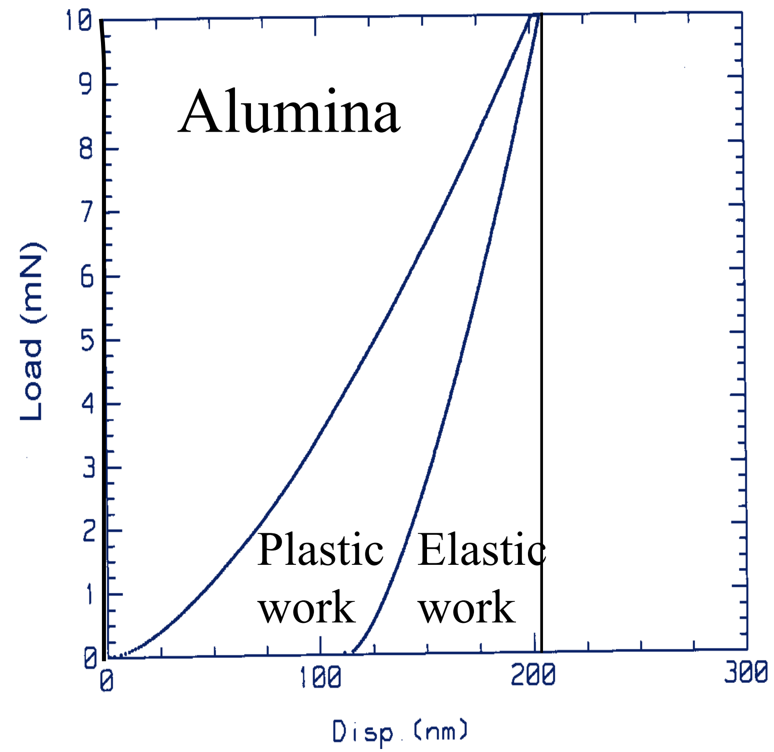
Typical material responses





Data courtesy of VAMAS TWA22

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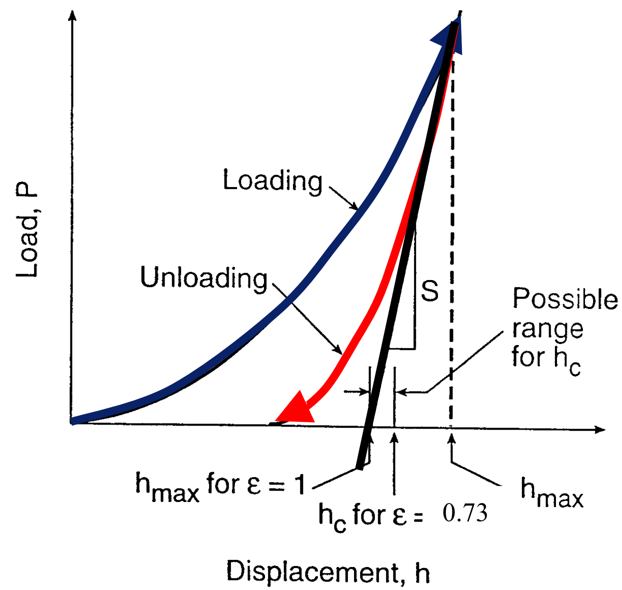
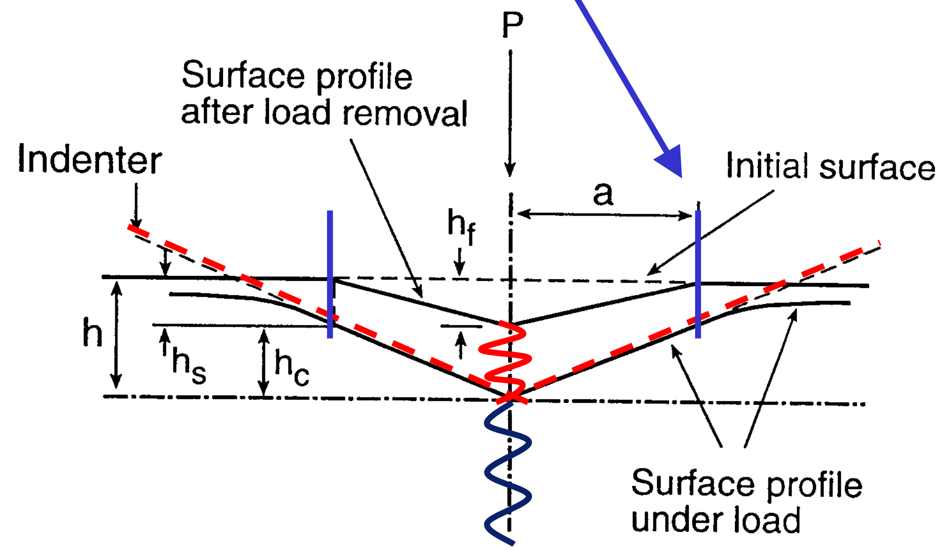
Indentation Contact Mechanics

For near perfectly plastic materials e.g. metals
$A_c \approx$ residual area
$\Rightarrow H_{IT} \; \alpha \; HV$

- Contact mechanics after Hertz and Sned





  – As with traditional Hardness, pile-up / sink-in is not accounted for
  – †*See I.N.Sneddon, Int. J. Engng. Sci.,* ***3*** *(1965) pp. 47-57*

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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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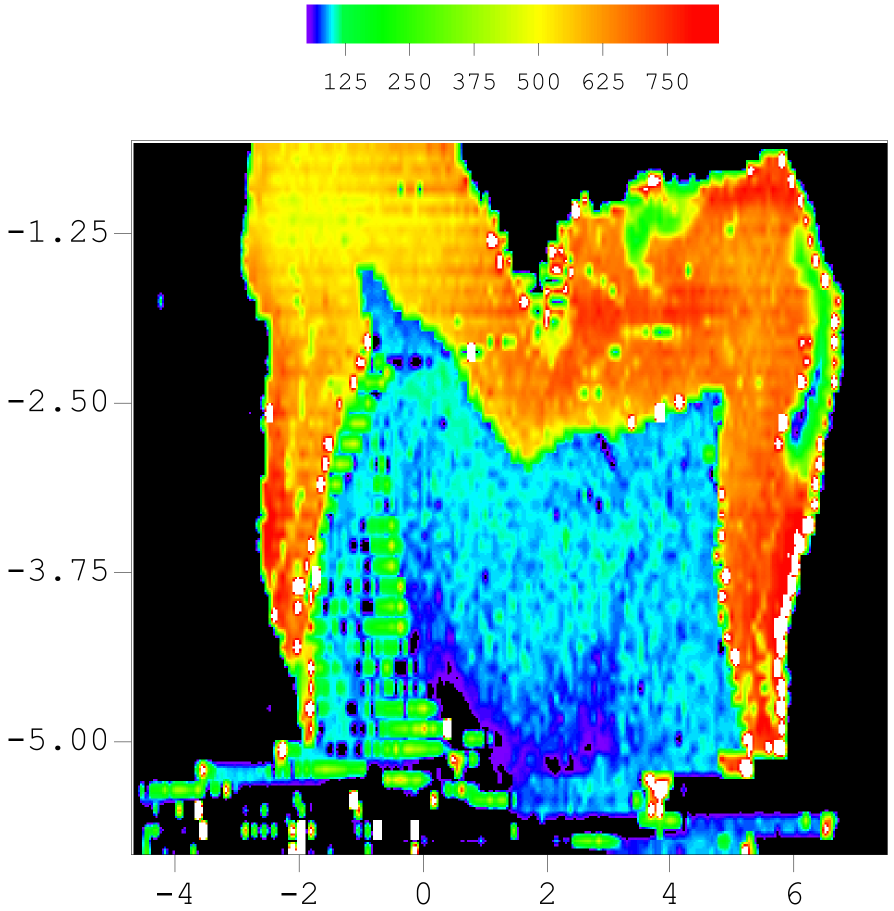
Scanning Indentation Mechanical Microprobe SIMM: Human Tooth



Typical scan, 200 by 100, takes ~30 hours (20,000 indents!)

Maximum load 5 N, load resolution 2 mN, depth resolution 20 nm

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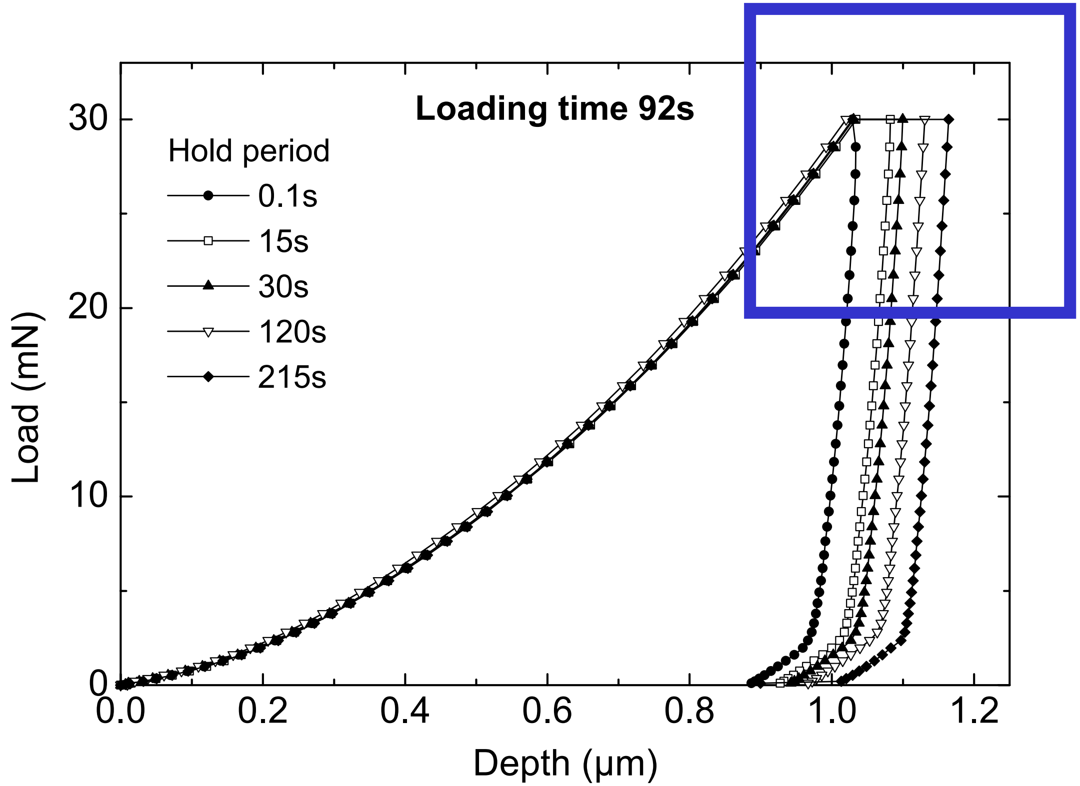
Creep affects H and E



[Chudoba and Richter Surf. Coat. & Tech. 148/2-3 (2001) 191-198]

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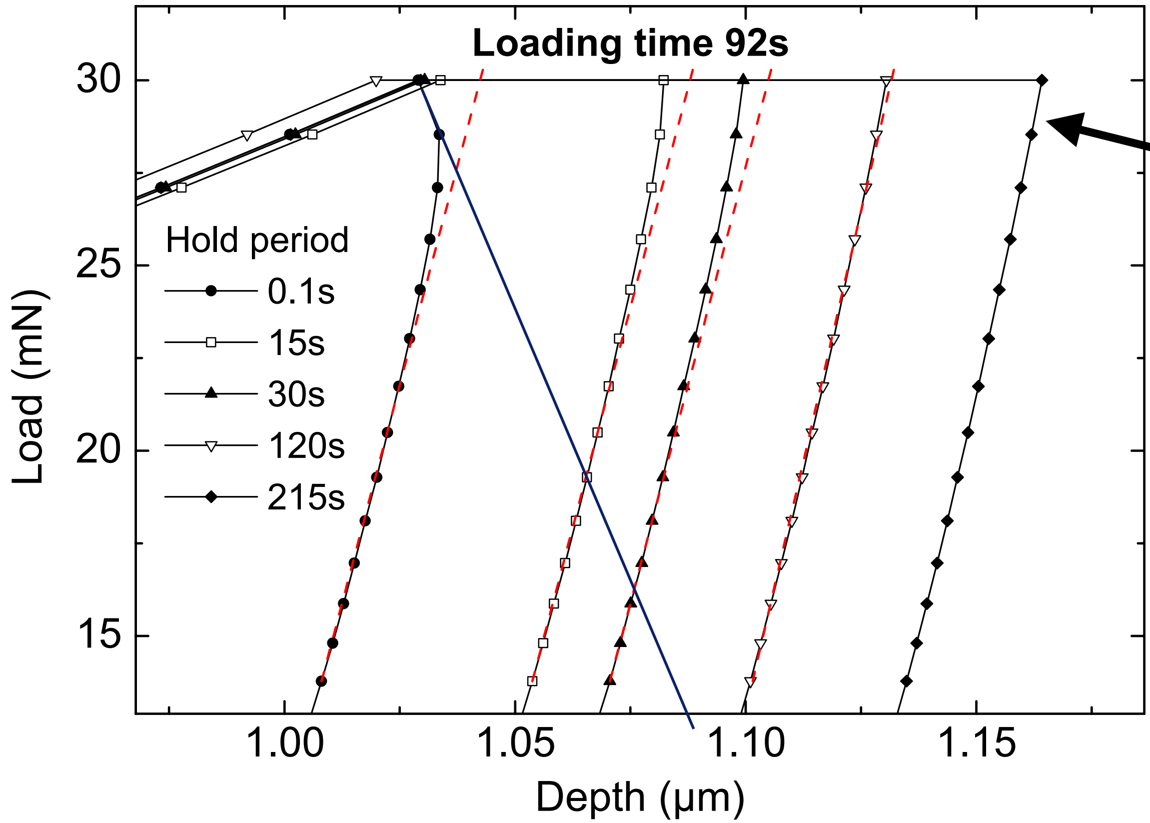
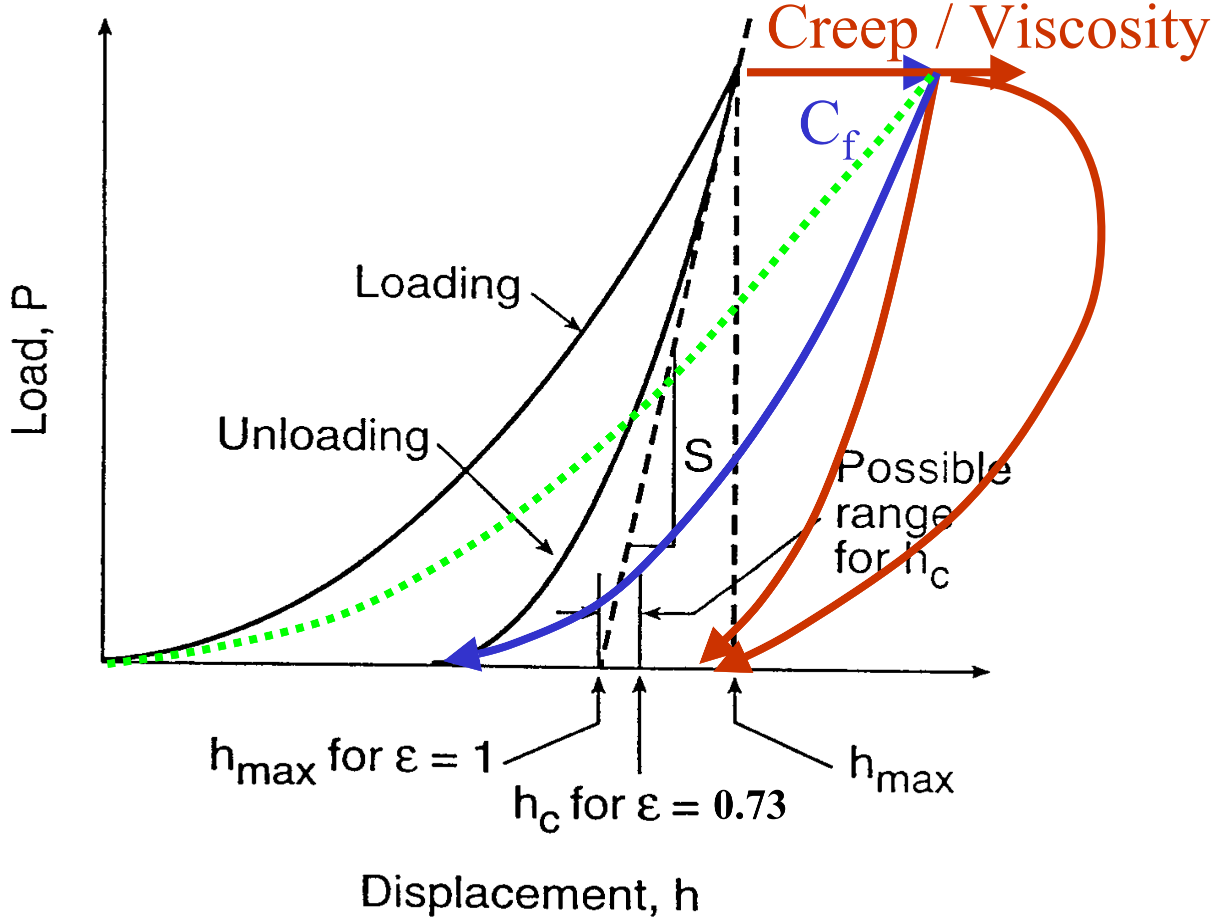
Wait until Creep rate drops (or unload quickly)

Diagram from: *Chudoba and Richter. Surf. Coat & Tech.* *148/2-3 (2001) 191-198*

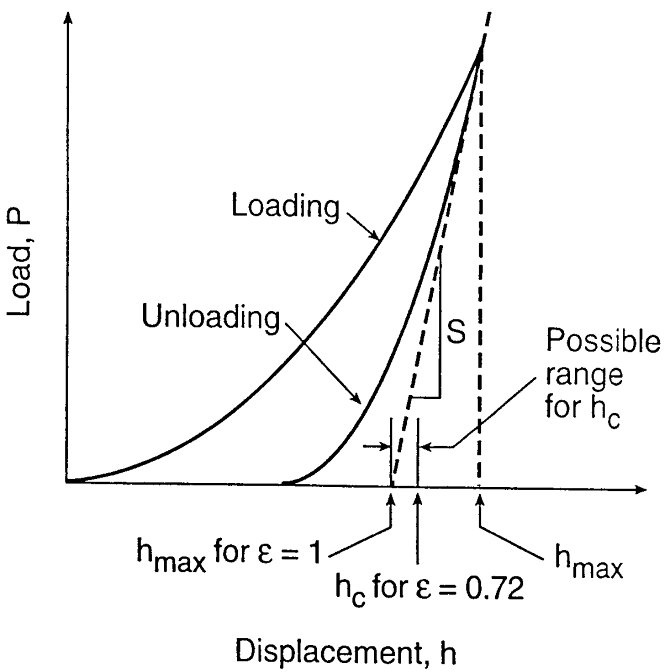


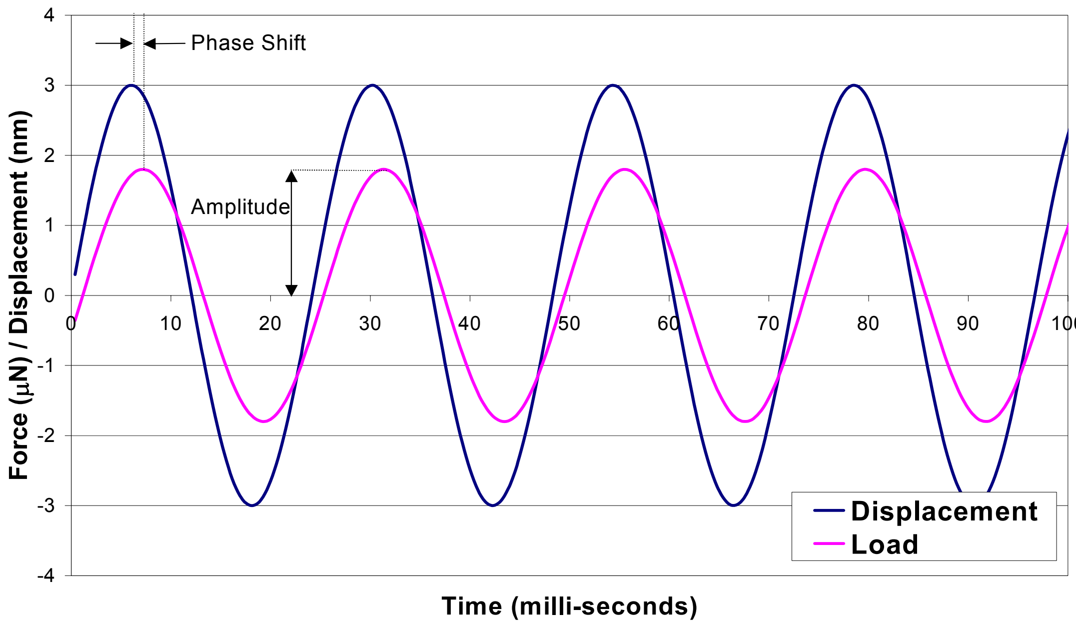
Creep rate Insignificant after 215 s (soft metal)

Schematic Indentation cycle



Polymers: Dynamic Indentation "Continuous Stiffness"





- Hardness and modulus of visco-elastic materials
- Superimpose a.c. signal on d.c. force ramp
- Phase shift and amplitude related to contact stiffness

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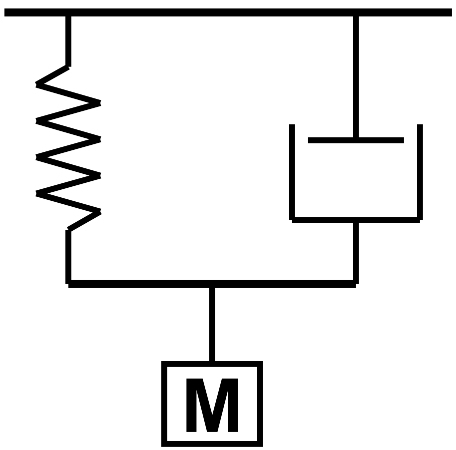
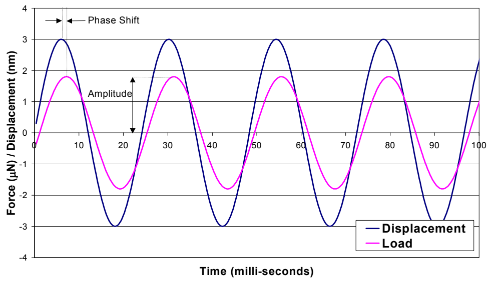
Spring and dashpot model



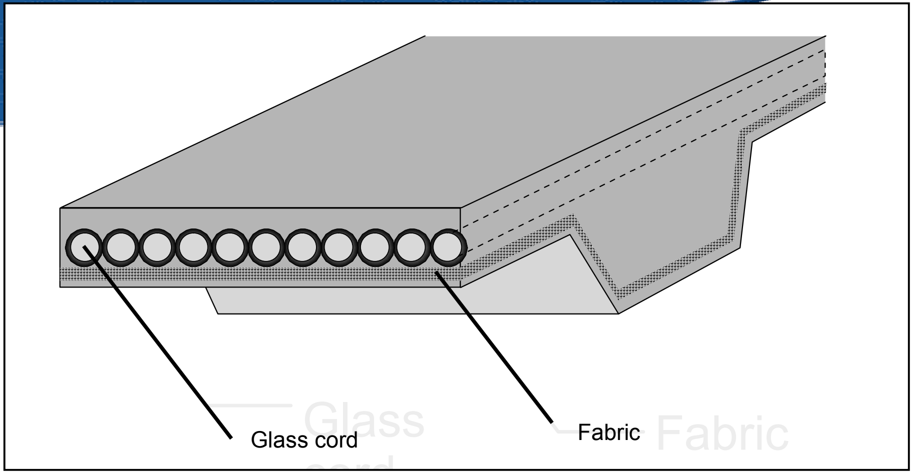
$$\sigma = \left(E' + iE''\right)\varepsilon$$

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

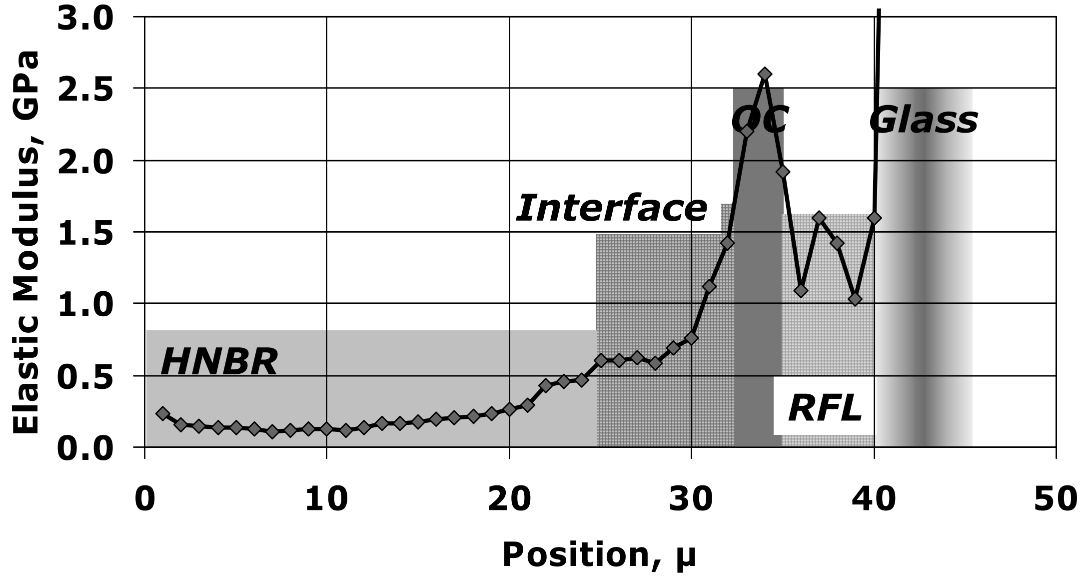


E′= Storage modulus
E″= Loss modulus
δ = Phase shift
Tan δ = Damping coefficient

Adhesive cure control – Car Timing Belts



Nanoindentation modulus measurement of Interface properties used to improve cure cycle and reduce adhesion failure.



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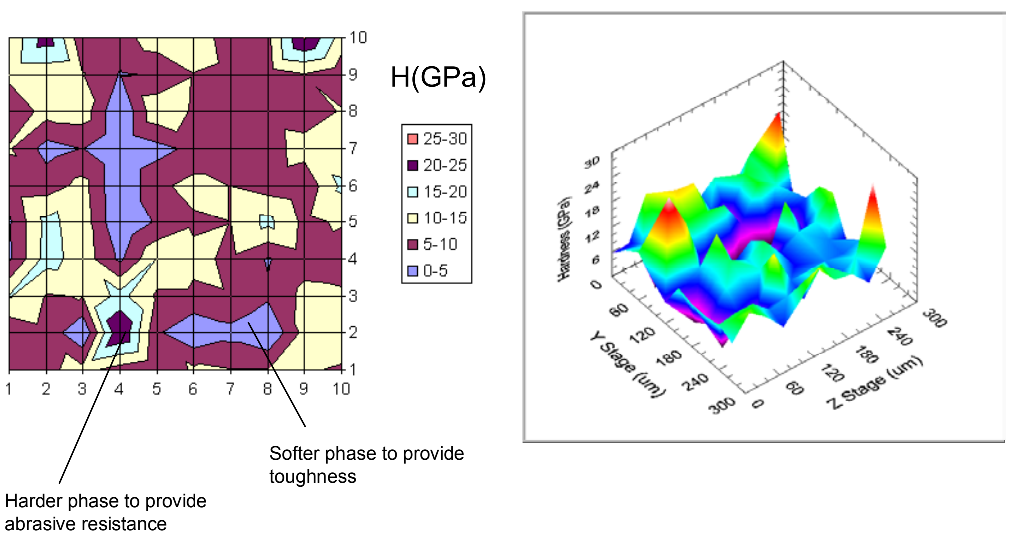
High-temperature Testing:
0.1 mN to 20 N

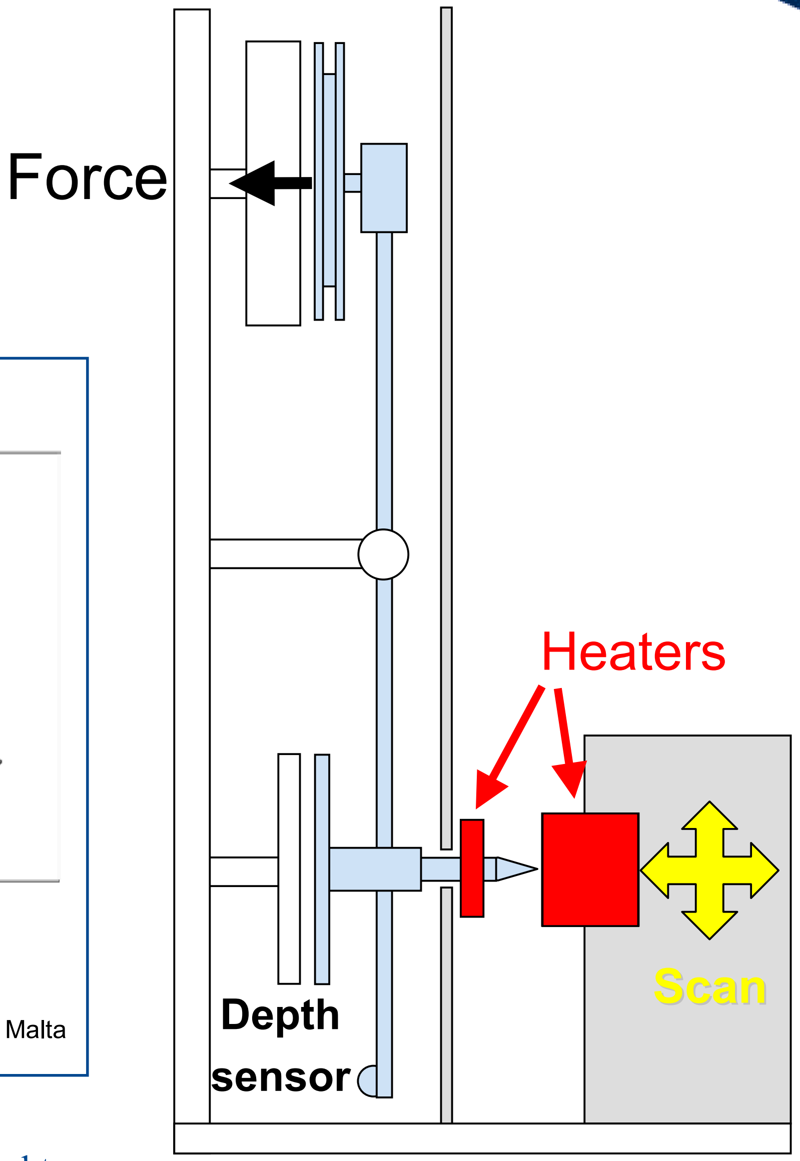
MicroMaterials NanoTest

Hot stage – sample temp ≤500°C

Mapping the hardness of tribological coatings



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



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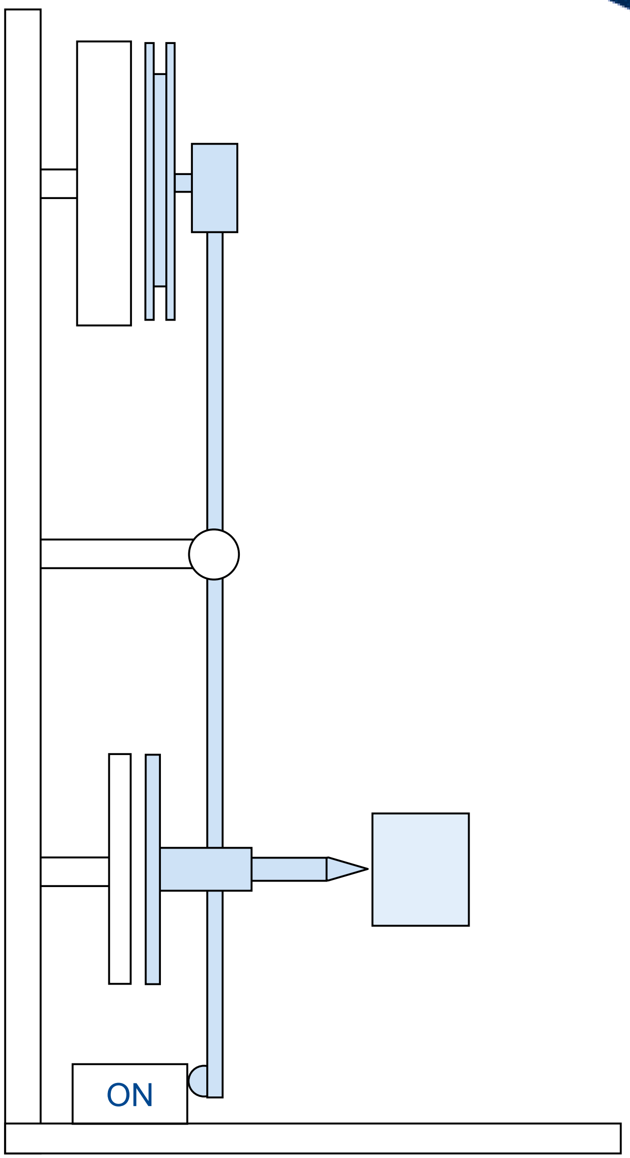
Dynamic indentation

NanoTest (Micro Materials Ltd, Wrexham)

- Dynamic nanoindentation
- Repetitive impacts at low force (mN)
- Displacement monitored with time
- Observe fracture & delamination

Test Variables

- Impact energy
- Impact Frequency
- Test probe geometry



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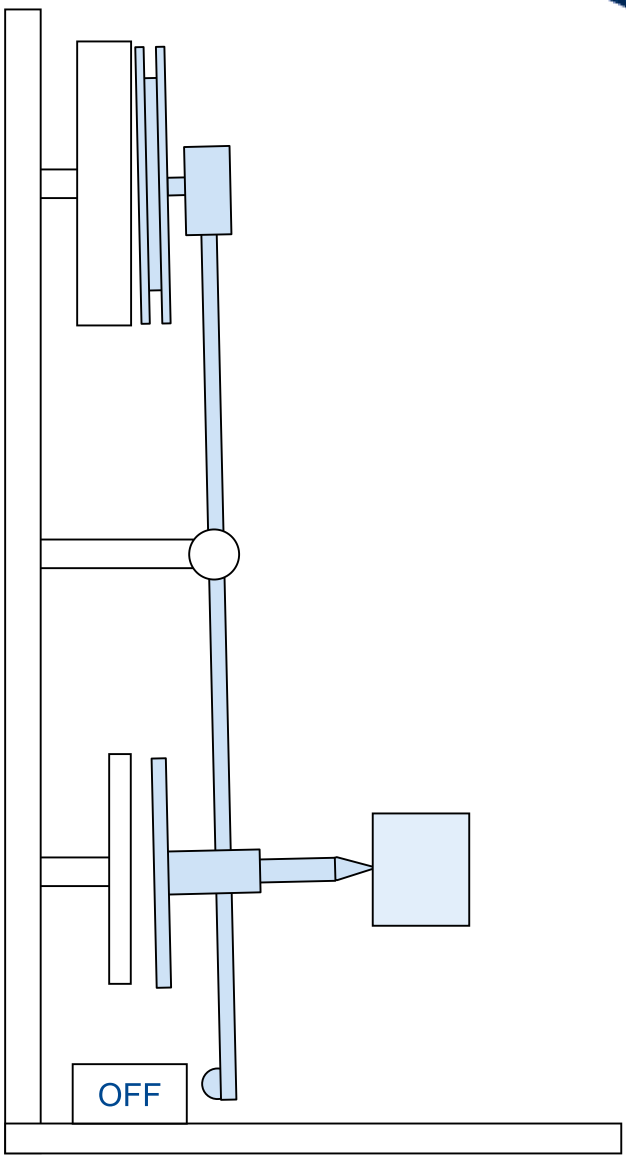

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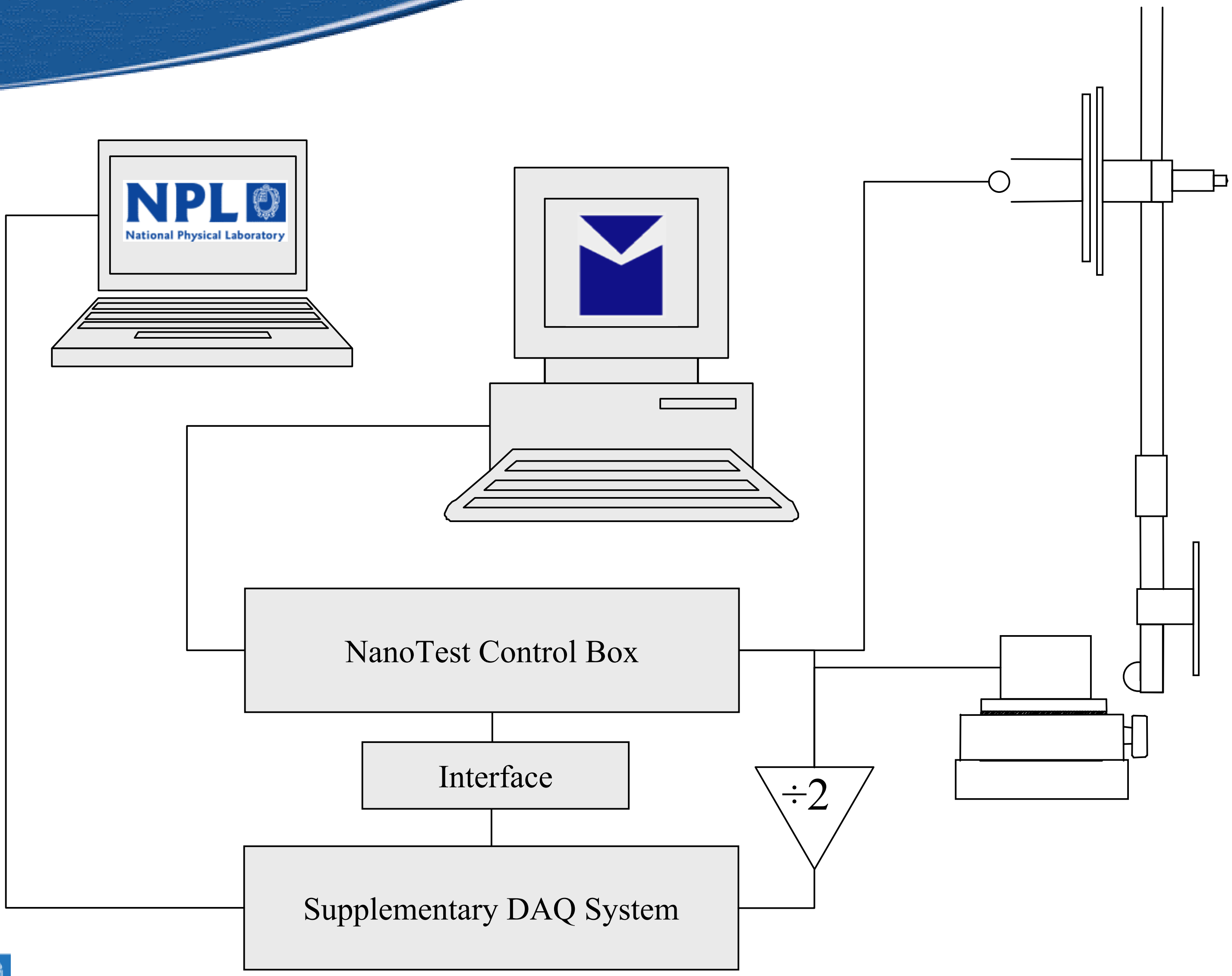
Supplementary Data Acquisition



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Practical Impact Parameters

Standard Set-up

Force	0.1 mN to 16 mN
Distance	5 µm to 20 µm
(I.e. Energy	0.5 nJ to 320 nJ)

Modified Set-up

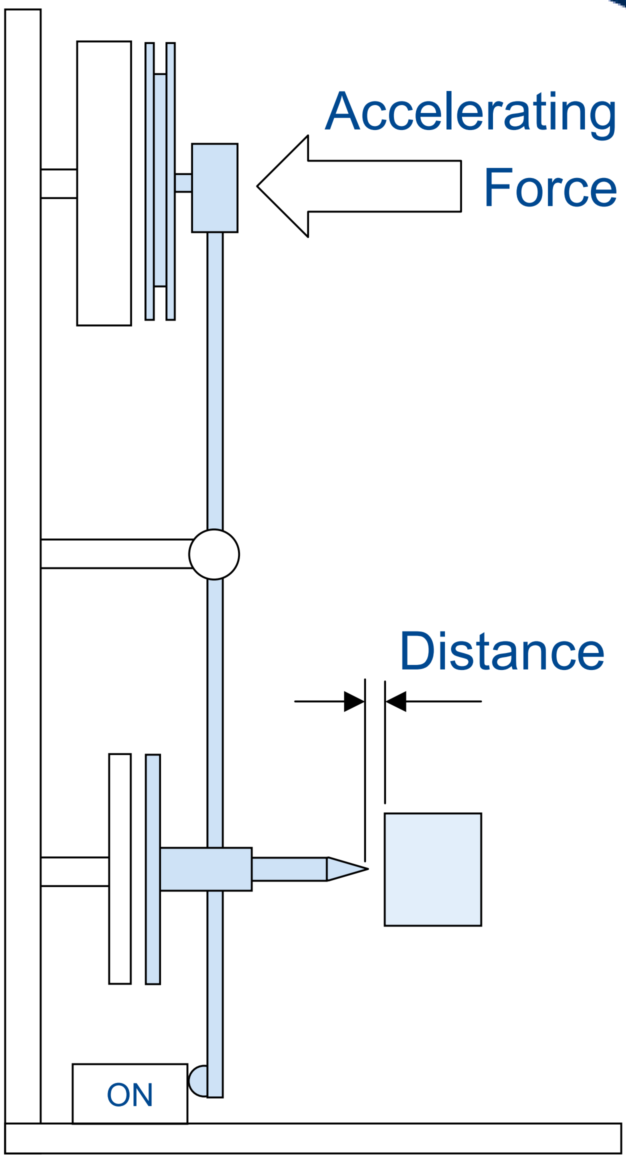
If larger solenoid used

Force	>500 mN

If Capacitor plate separation adjusted

Distance	>1mm

(I.e. Energy > 0.5mJ)



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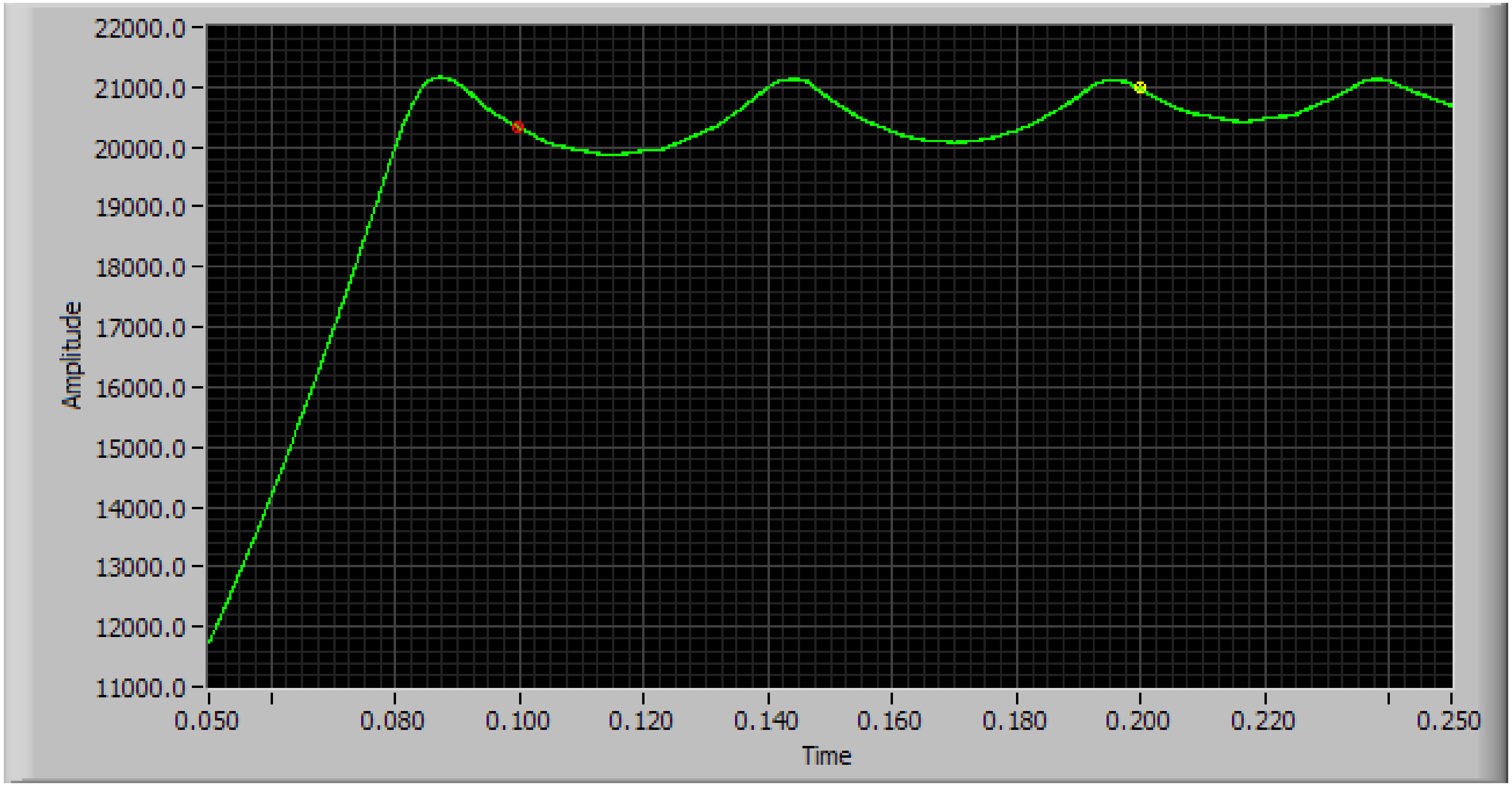
Impact test

5µm conical indenter
Single crystal aluminium

Individual data points…

Position 20k samples.s-1 (up to 500K/s possible)



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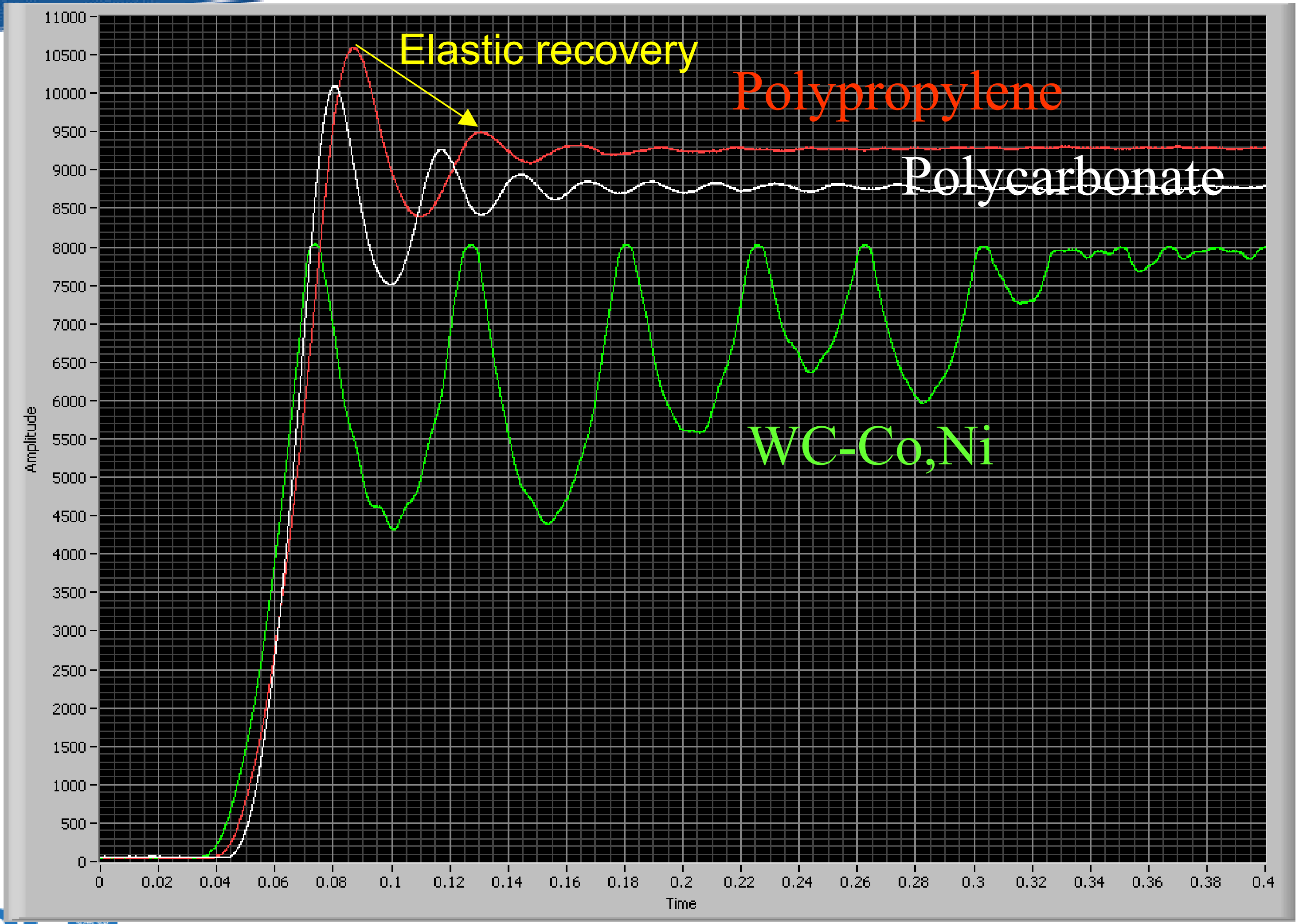
35 nJ impact energy



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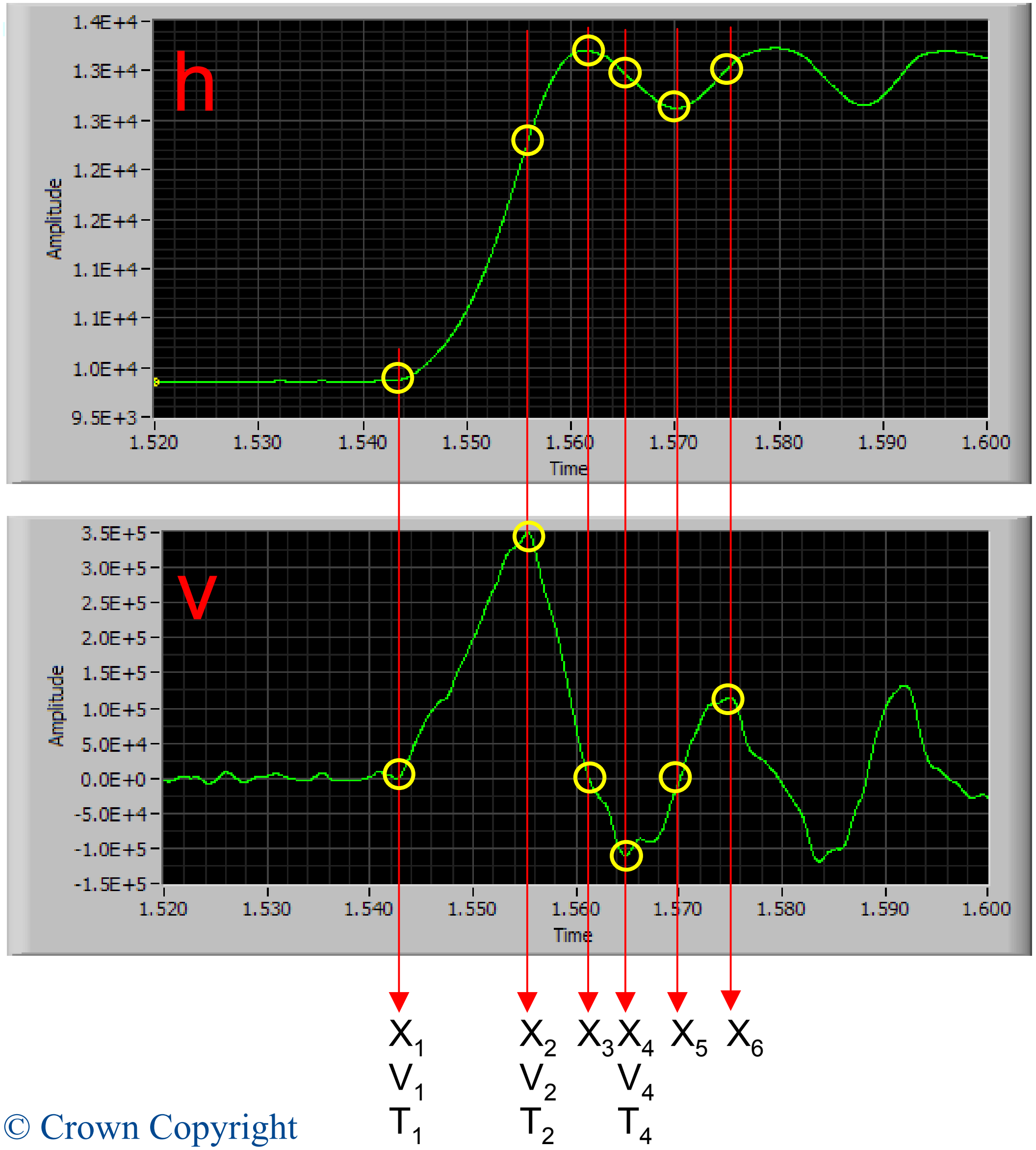
Automated determination of contact points

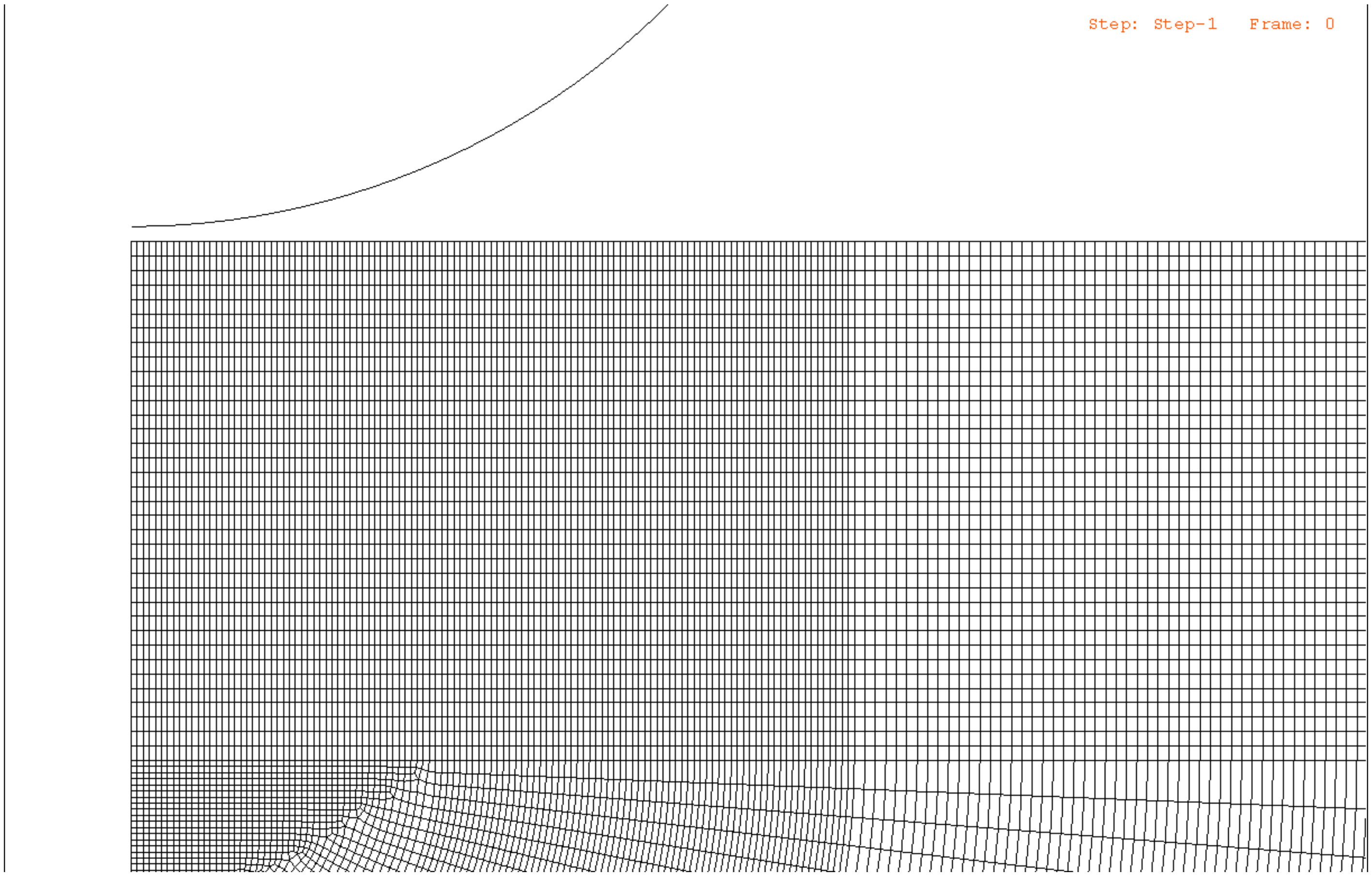
- Initial acceleration
- Velocity In
- Initial contact
- Max penetration
- Velocity Out
- Loss of contact
- Max rebound height
- Secondary contact



FEA of nano-impact testing (ABAQUS Explicit model)

Indentation of Aluminium single crystal

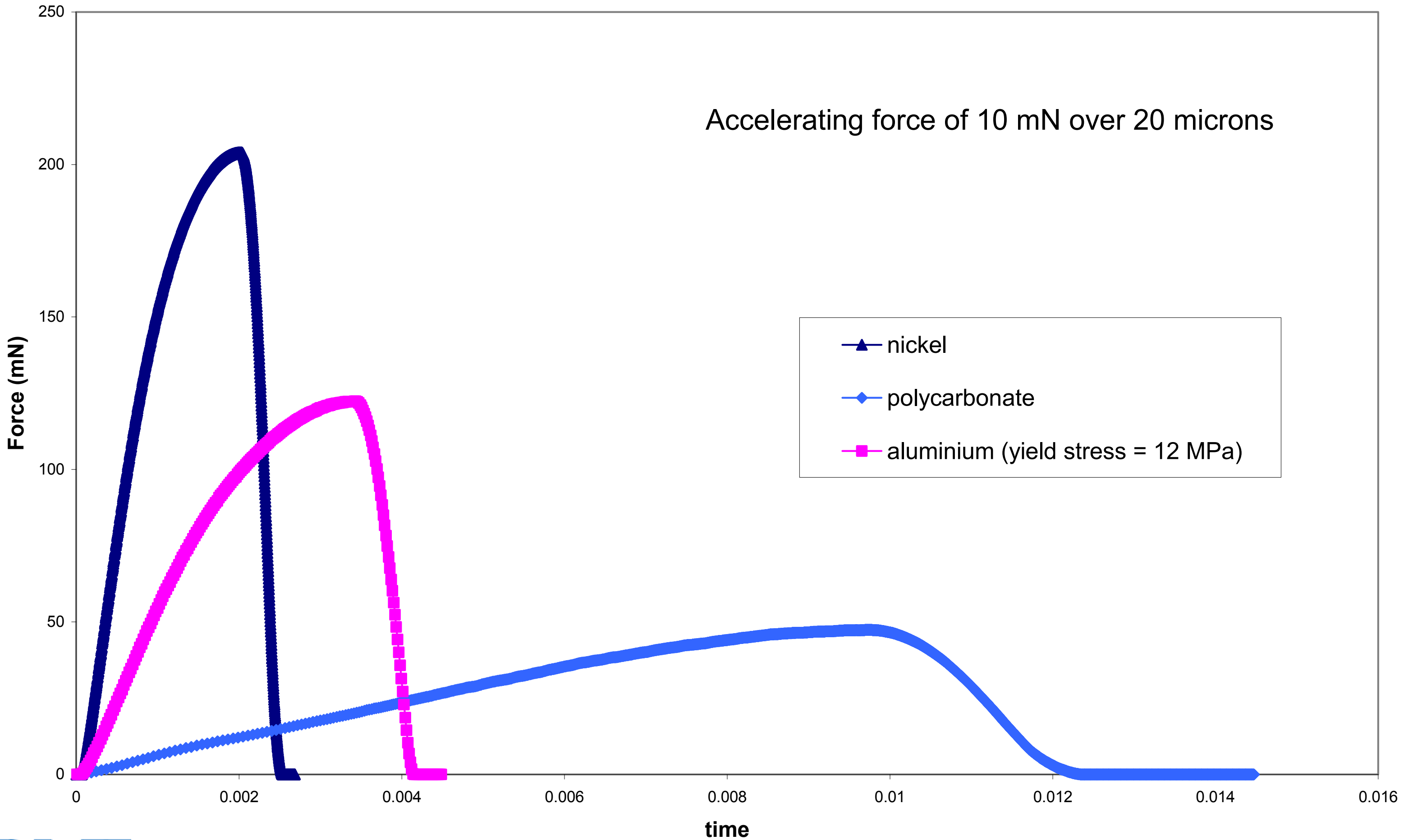
Indenter given mass and initial velocity



FEA of nano-impact testing

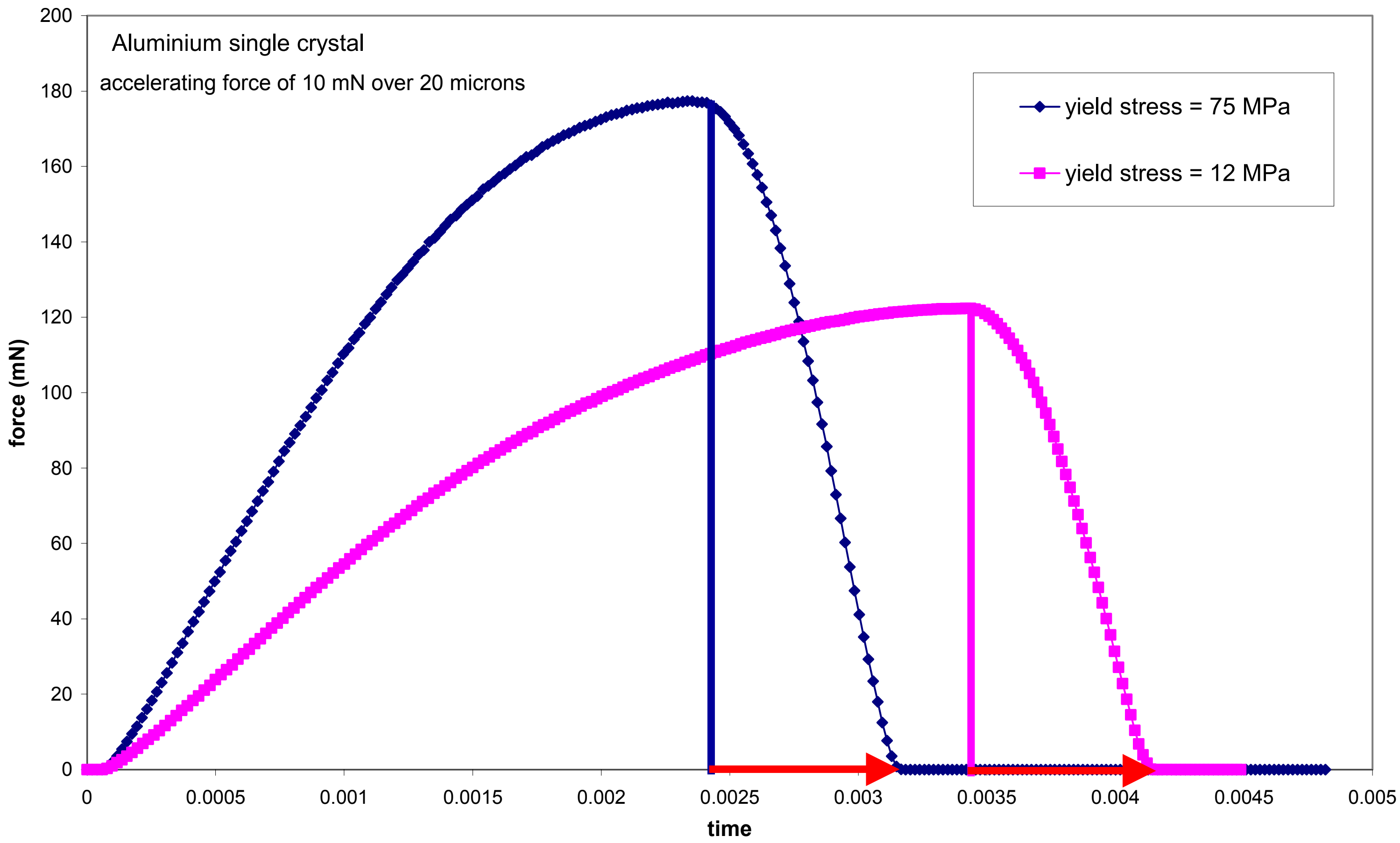
Indentation of different materials

- aluminium, nickel and polycarbonate



FEA of nano-impact testing

Indentation of Aluminium single crystal



Force predictions depend on yield stress used

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Stage Plans

- **Stage 1 (1/4/06 – 31/3/07)**
  - 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

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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).

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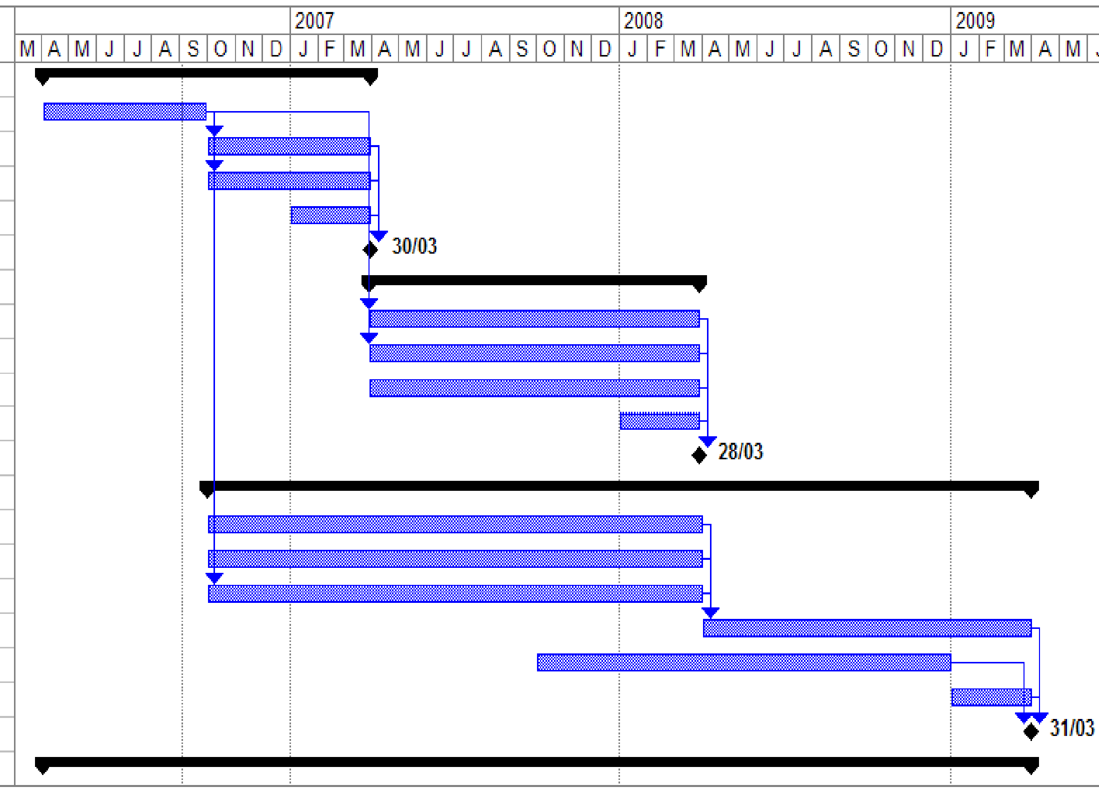
SE02 GANNT chart

ID	Task Name
1	**Dynamic Calibration**
2	Materials Procurement
3	Calibration Sensitivity Study
4	Algorith Sensitivity Study
5	Preparation and Submission of Report
6	Development of Dynamic Calibration Method
7	**Test Method Extended to Time/Frequency**
8	Ultra-Rapid Indentation Method
9	Frequency Sweep and Chirp
10	Viscoeleastic Models
11	Preparation and Submission of a scientific Paper
12	Development of High Rate Indentation Methods
13	**Test Methods Extended to Temperature**
14	Design Temperature Stage
15	Test Temperature Stage
16	Validation Data
17	Evaluate Use of Temperature Stage
18	Viscoelastic models
19	Preparation and Submission of a Scientific paper
20	High Rate Indentation Method at Elevated Temperatures
21	**Knowledge Transfer**



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