Nanomechanical Testing in Materials Research and Development V

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Thermo-mechanical characterization of polymer samples using nanoindentation - From bulk characterization to thin film properties

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Thermo-mechanical characterization of polymer samples using nanoindentation
- From bulk properties to thin film applications -

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SURFACE nanometrology
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SURFACE nanometrology

Outline:

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- Motivation for studying polymer films
- Experimental techniques
- NanoLH Laserheater
- Comparison between DMA and nanoindentation spectroscopy
- Results on polymer thin film tests
- Technical aspects of polymer investigations
- Nanoindentor shape: flat top / spherical indentor
- Outlook

Motivation:

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- Polymer thin films used for coating and nanostructuring
- Modulus, hardness, creep, glass transition, scratch resistance
- Film preparation may change properties compared to bulk
- Properties change due to thermal history, oxidation or irradiation (UV)
- Measurements on the real length scale / device
- Nanoindentation is a versatile tool
- Thermo-mechanical properties are accessible
- ...

Conventional mechanical spectroscopy:

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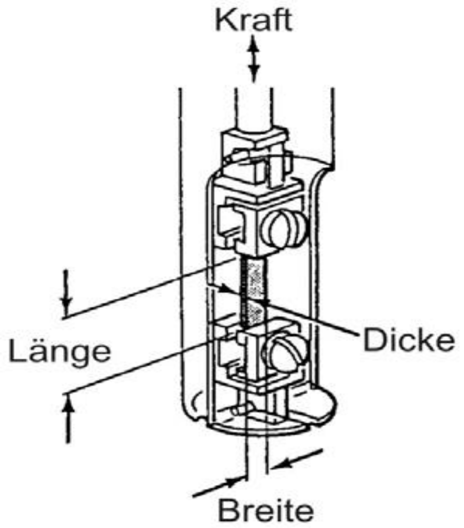
Dynamic mechanical analyzer, DMA

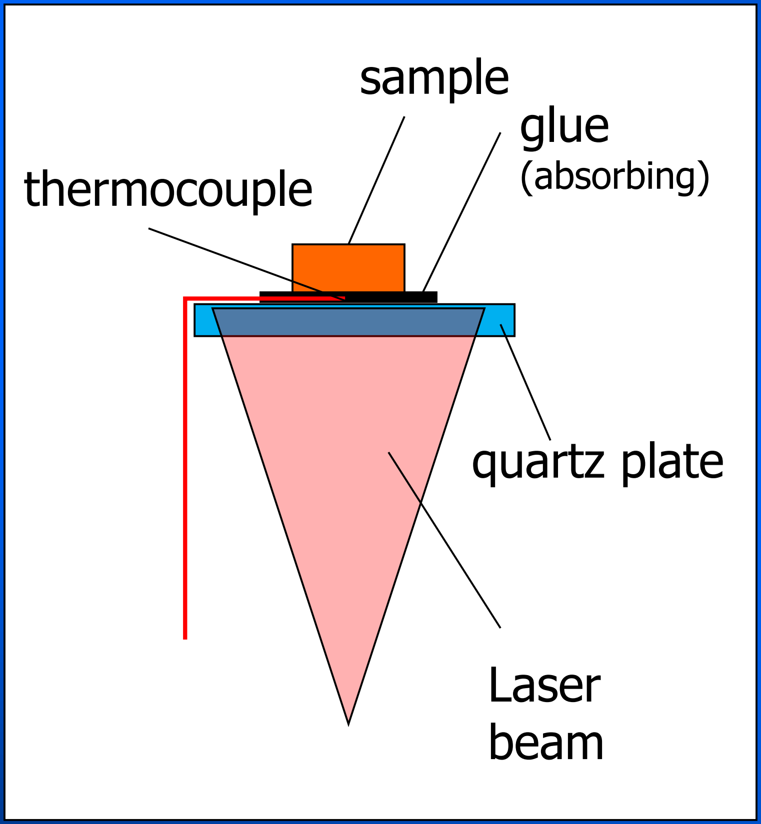


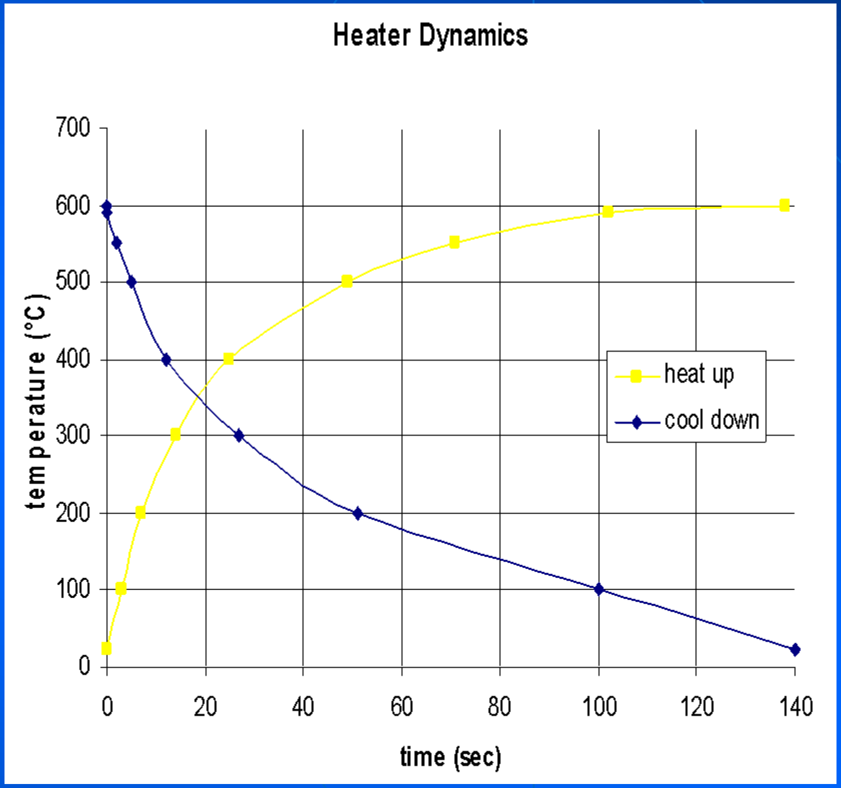
Perkin Elmer, manual 1994

Equations:

$\sigma = \sigma_0 \sin(\omega t)$	Stress
$\epsilon = \varepsilon_0 \sin(\omega t - \delta)$	Strain
$E = E' + iE''$	Complex modulus
$Q^{-1} = \tan(\delta) = E''/E'$	Loss factor

Sample heating for Nanoindentation:





Laser beam heats the sample and not the sample tray.

Very high heating / cooling rates possible

Indenter heating for Nanoindentation:

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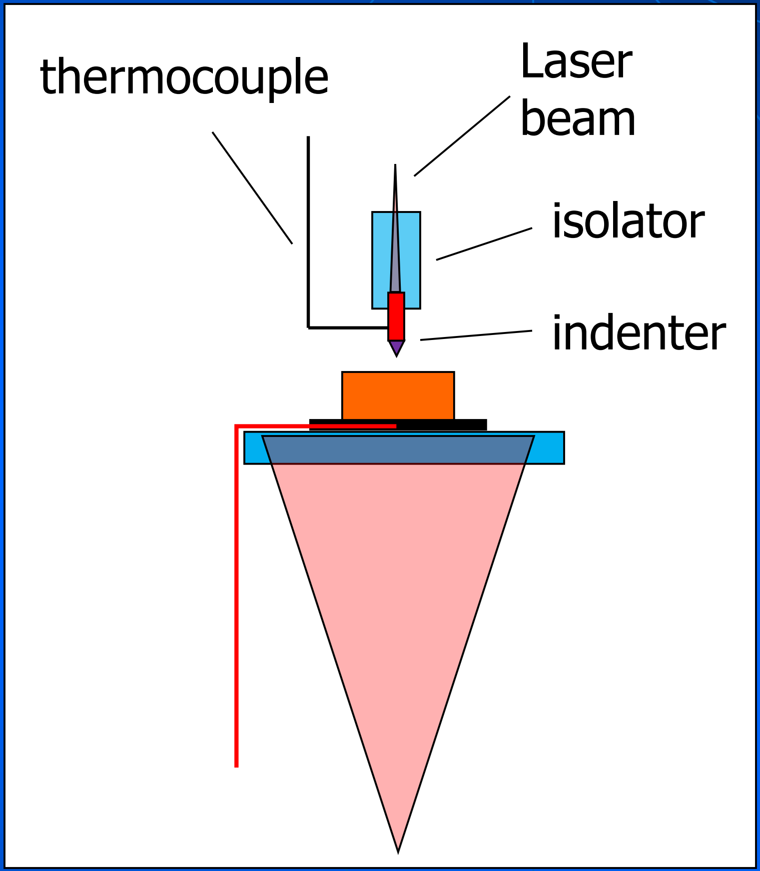
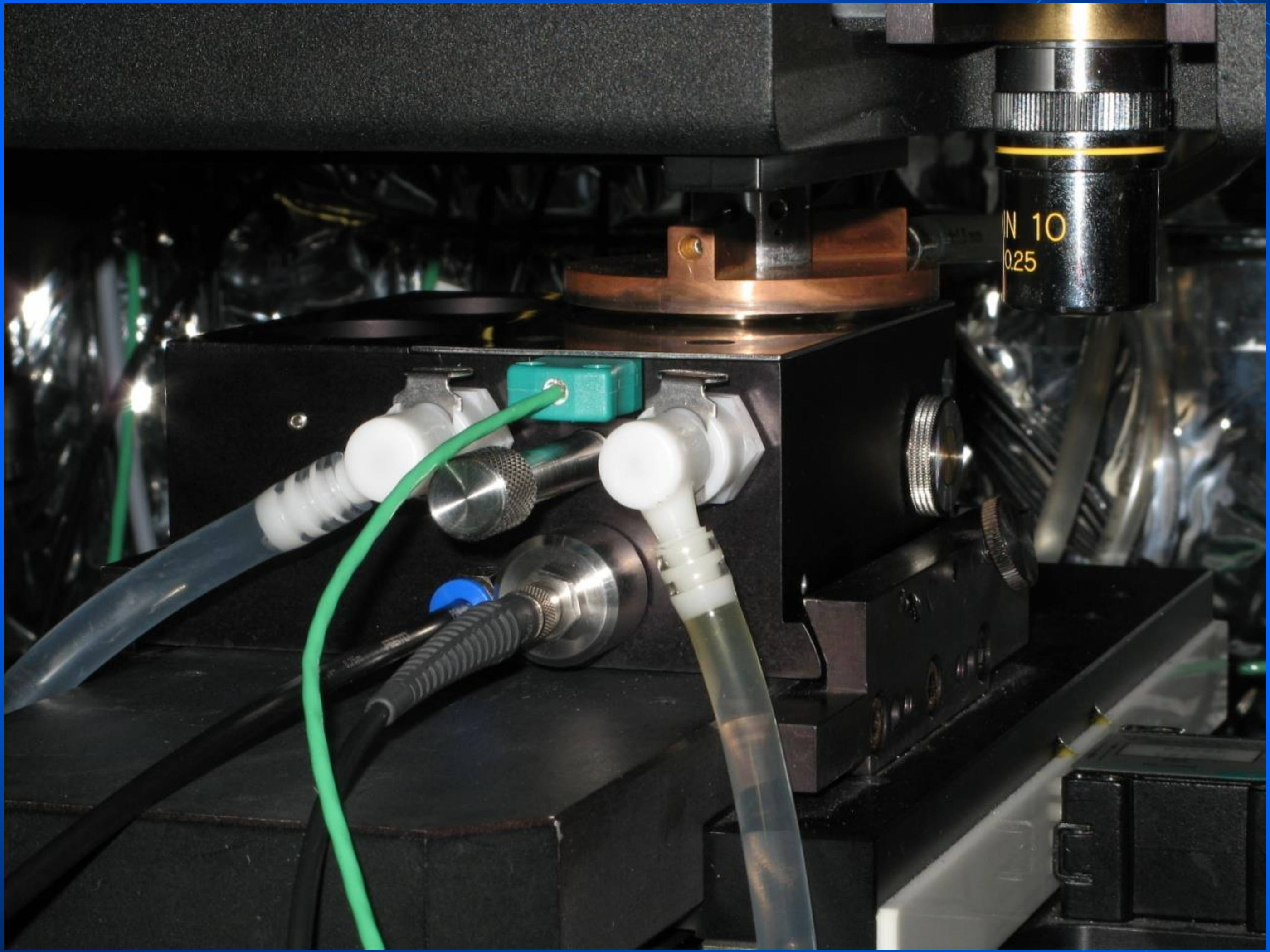
Heatable indenter tip controlled by an **independent PID loop**.

- No heat flow from sample to the tip
  → well defined sample temperature!

- No rapid change in tip temperature during indentation
  → Temperature of shaft remains constant (low drift)



SURFACE nanoLH in a G200 Nanoindenter:



Nanoindentation tests vs. DMA measurements:

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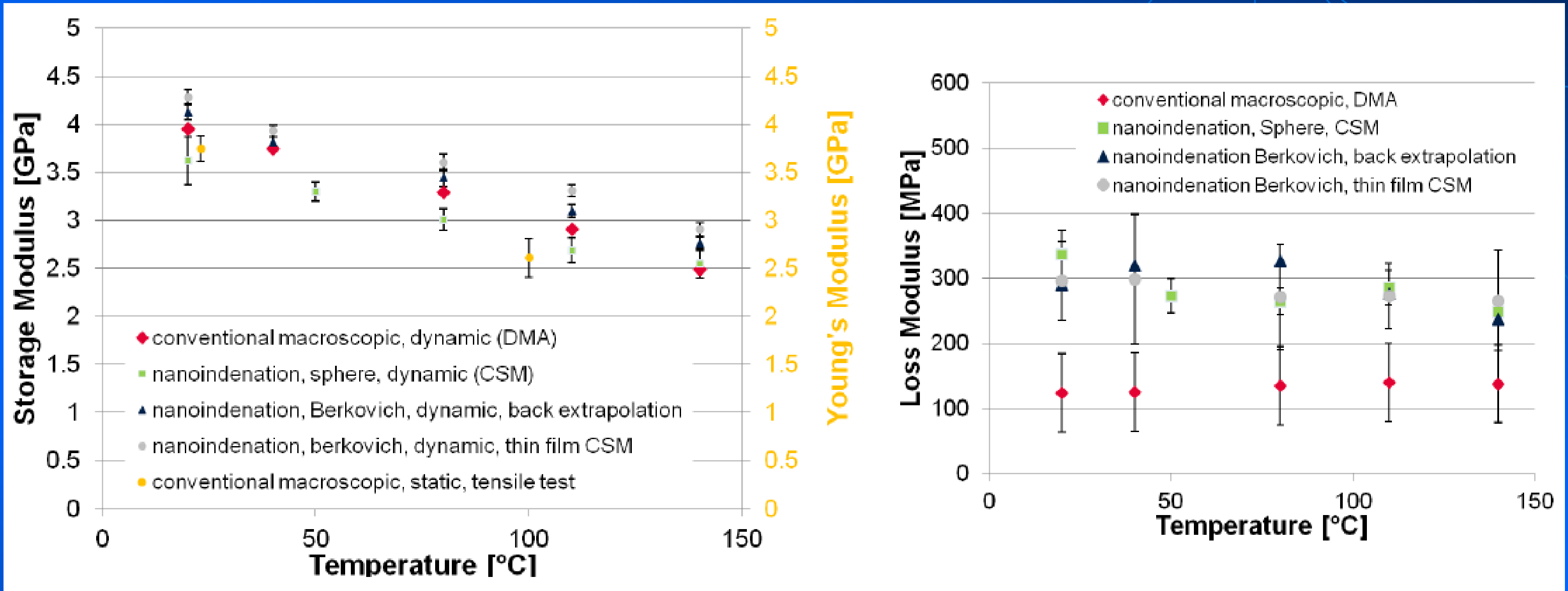

[K. Unterhofer, H. Preu, J. Walter, G. Lorenz, W. Mack, M. Petzold, EMPC conference, (2013), ISBN: 978-2-95-274671-7]

Very good agreement on storage date confirmed, loss modulus shows an offset, almost no temperature dependence.

Nanoindentation tests on polymer thin film samples:

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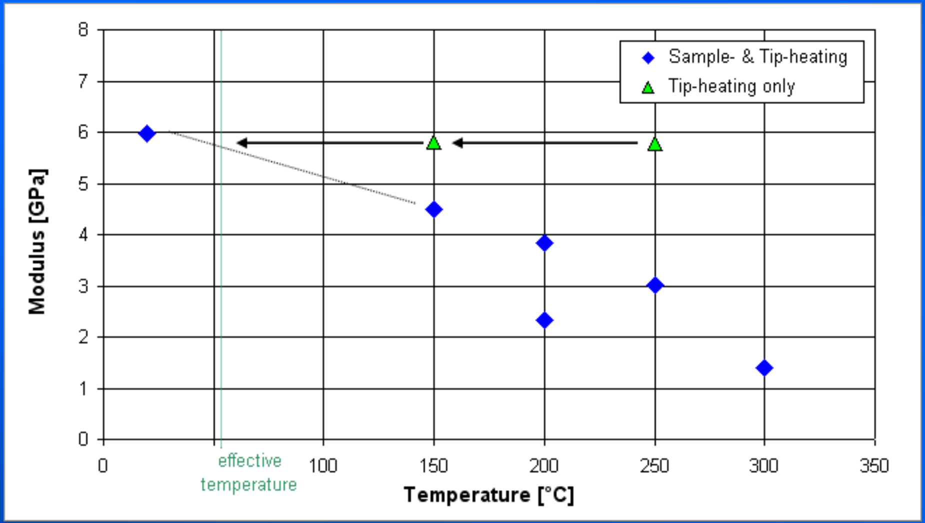
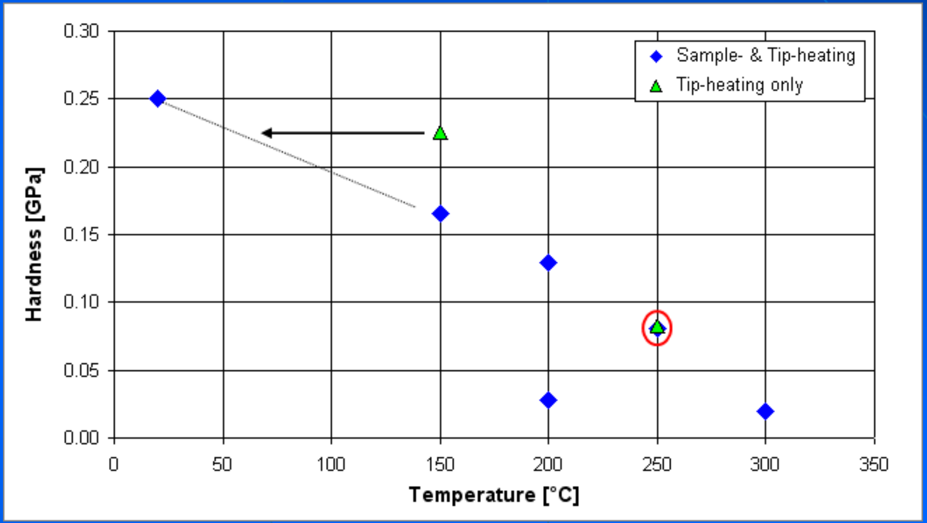
Comparison between sample and tip heating
to tip heating only (local heating)





Local heating (green) not effective for modulus estimation, requires sample and tip heating.

Local heating leads to correct hardness at higher temperatures (local flow around the indenter tip)

Nanoindentation tests on polymer thin film samples:

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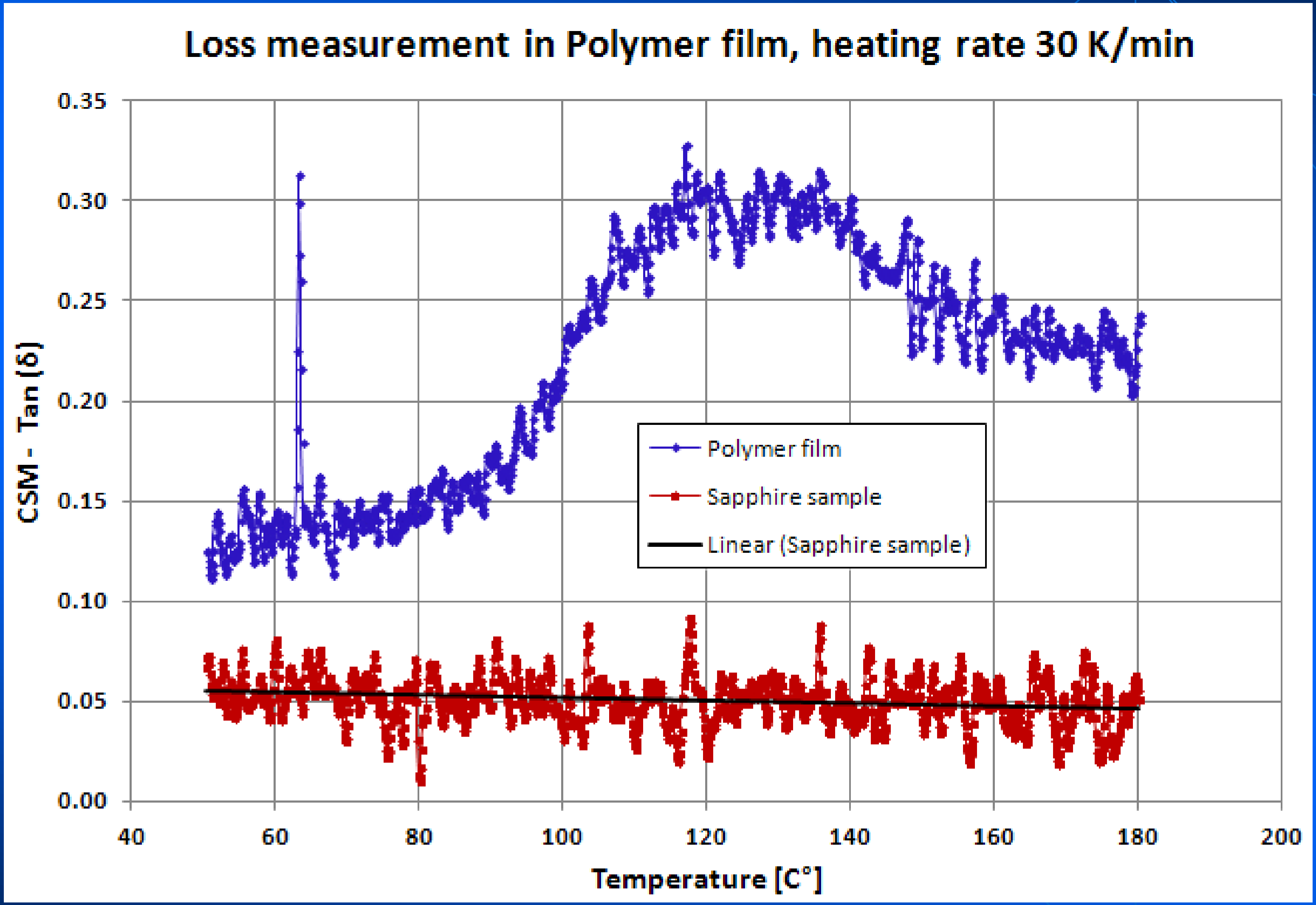

Glass transition visible in loss spectrum

In situ annealing tests of a polymer thin film:

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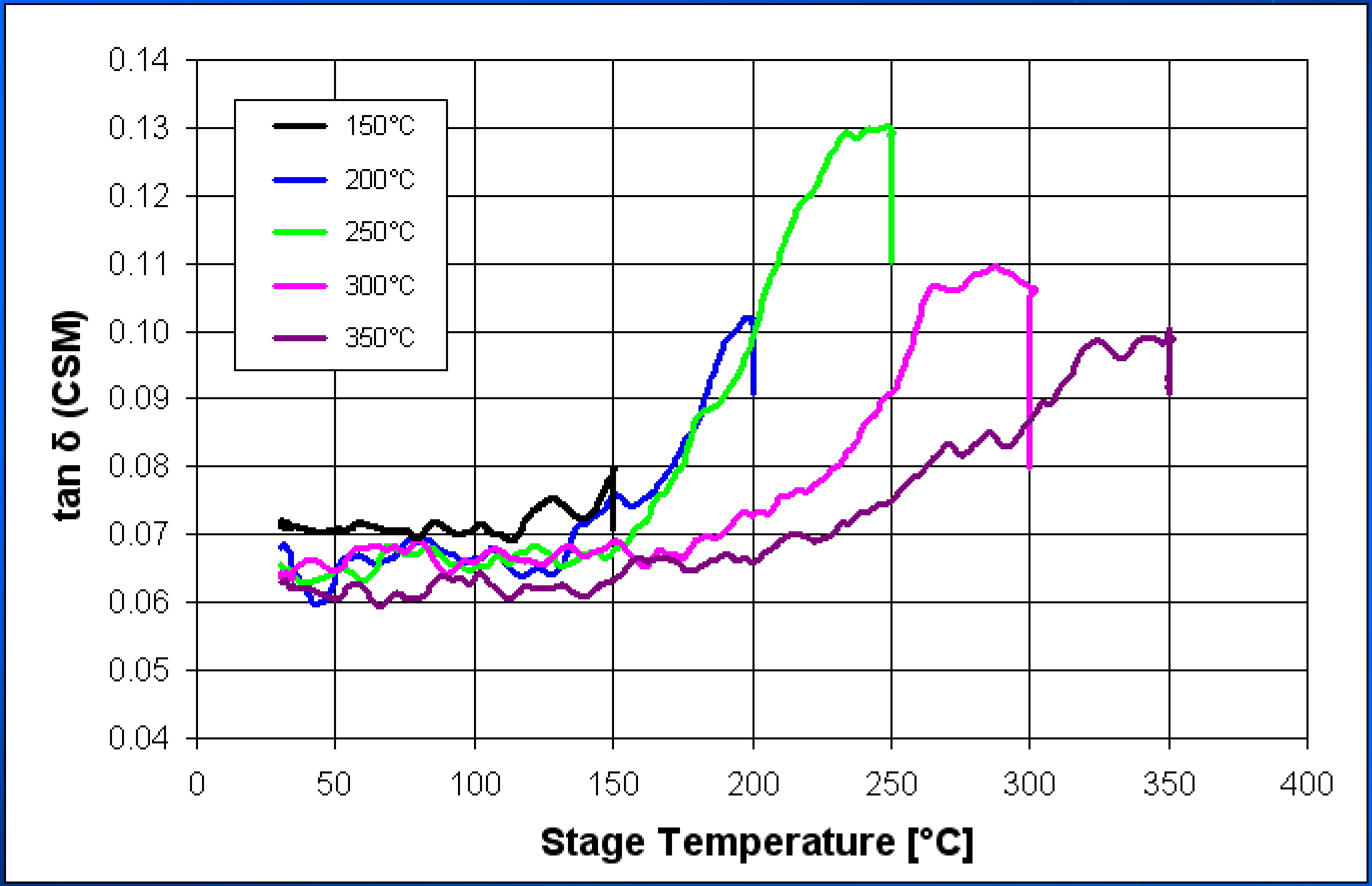
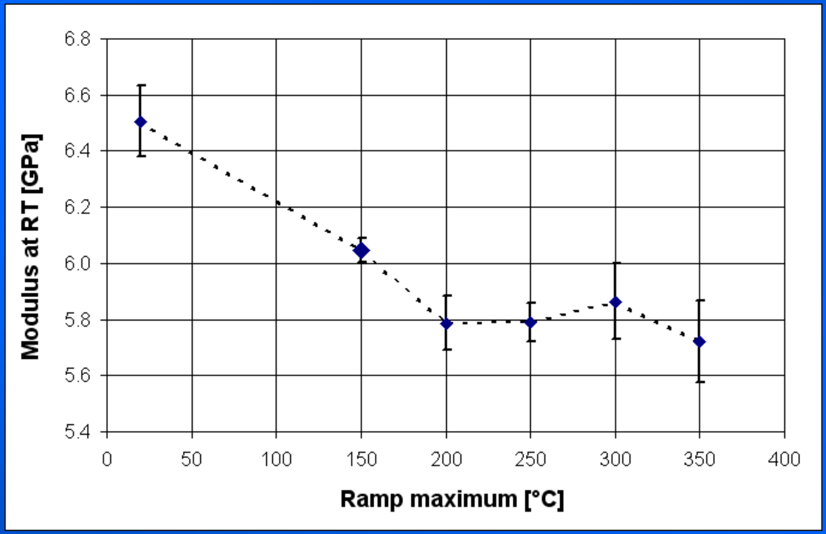
Ramp at 10 K / min to ramp maximum,
increase of maximum temperature with each run

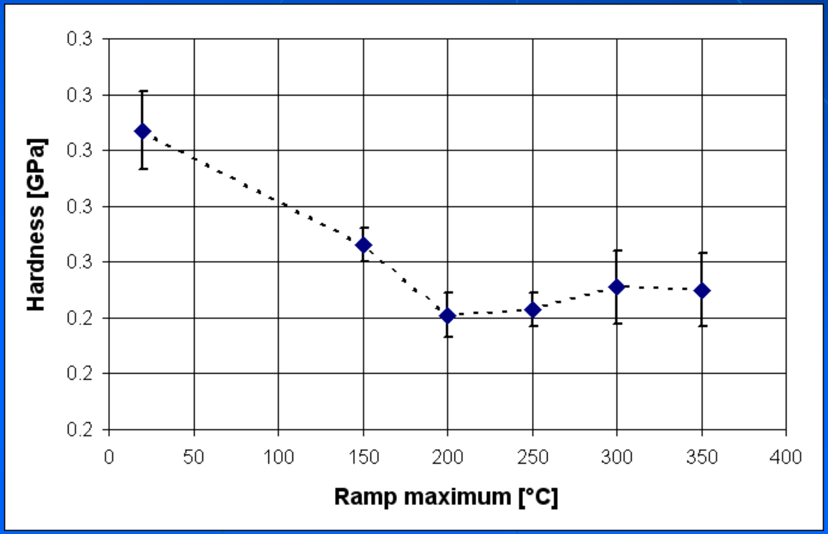


Relaxation of the film shifts the damping to higher temperatures.

In situ annealing tests of polymer thin film:

Modulus and hardness at RT can be studied in situ between the annealing steps





Relaxation changes the film up to 200°C, higher treatments have no significant effect.

Heating rates / frequency :

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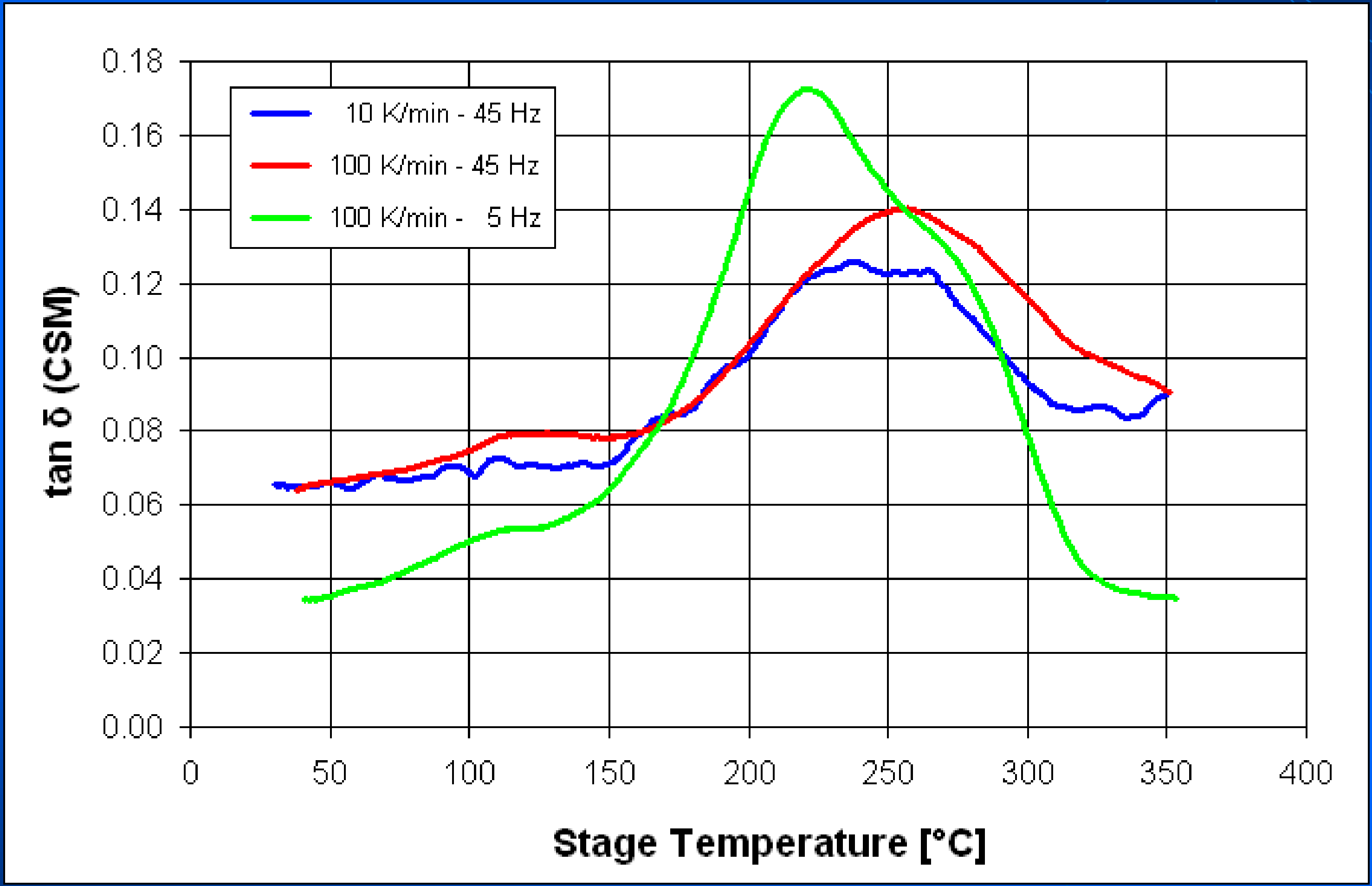

Heating rates and frequency can be varied to study the relaxation processes in polymers.

Technical aspects of polymer investigation:

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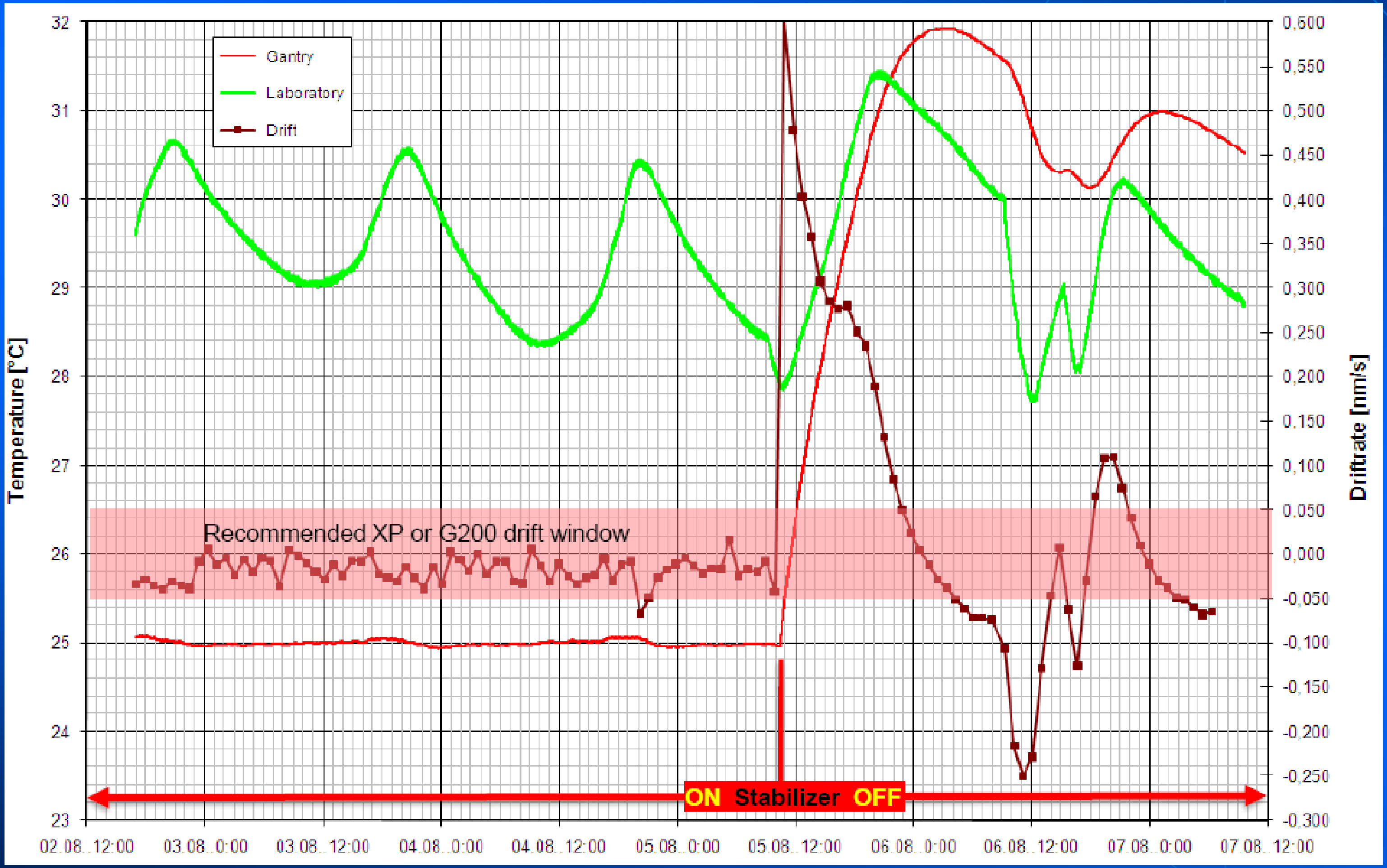
1. Stable temperature conditions



Active control of gantry temperature – low drift rates at varying conditions.

Technical aspects of polymer investigation:

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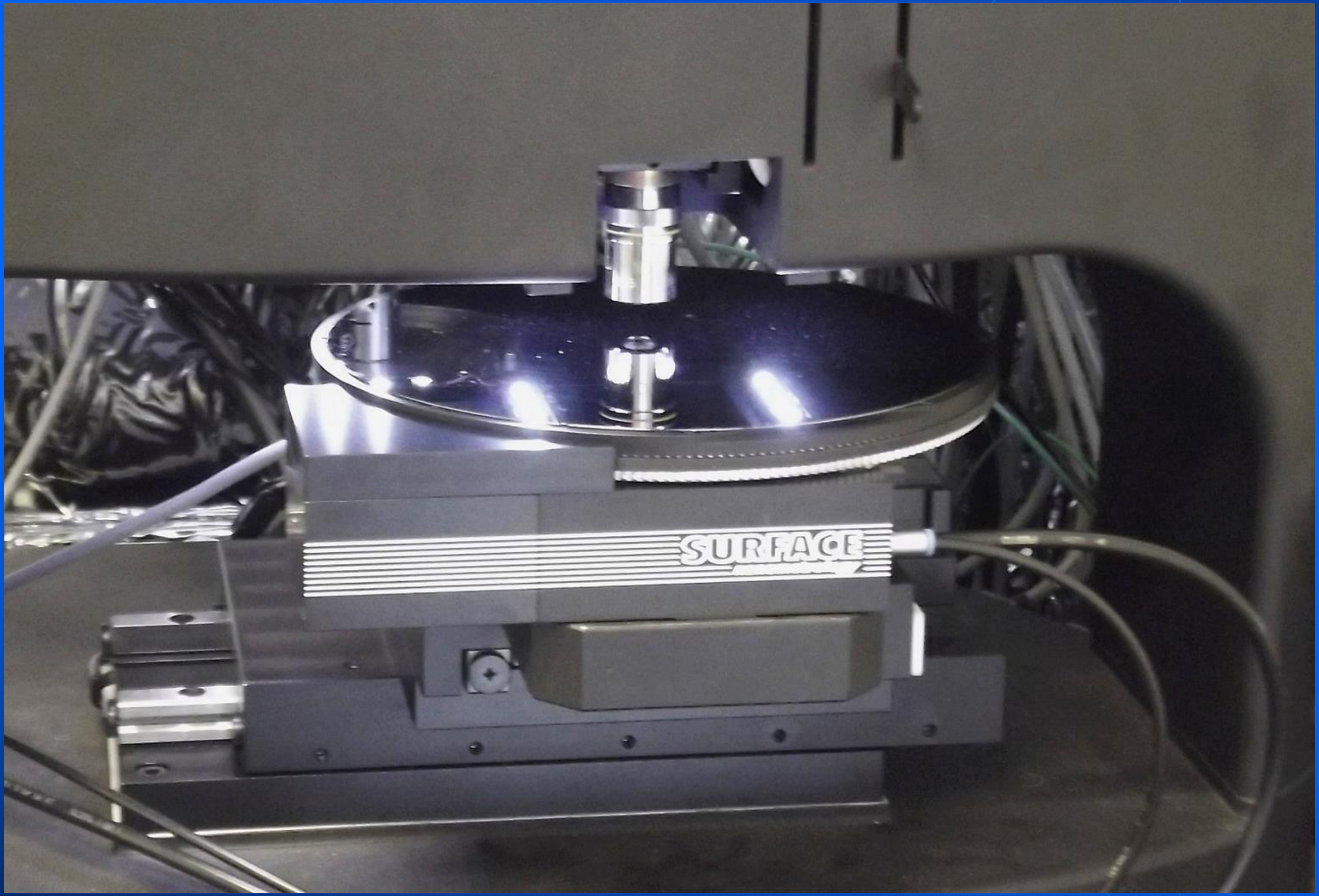
2. Handling of full wafer



Motorized vacuum stage for a full 200 mm wafer.

Technical aspects of polymer investigation:

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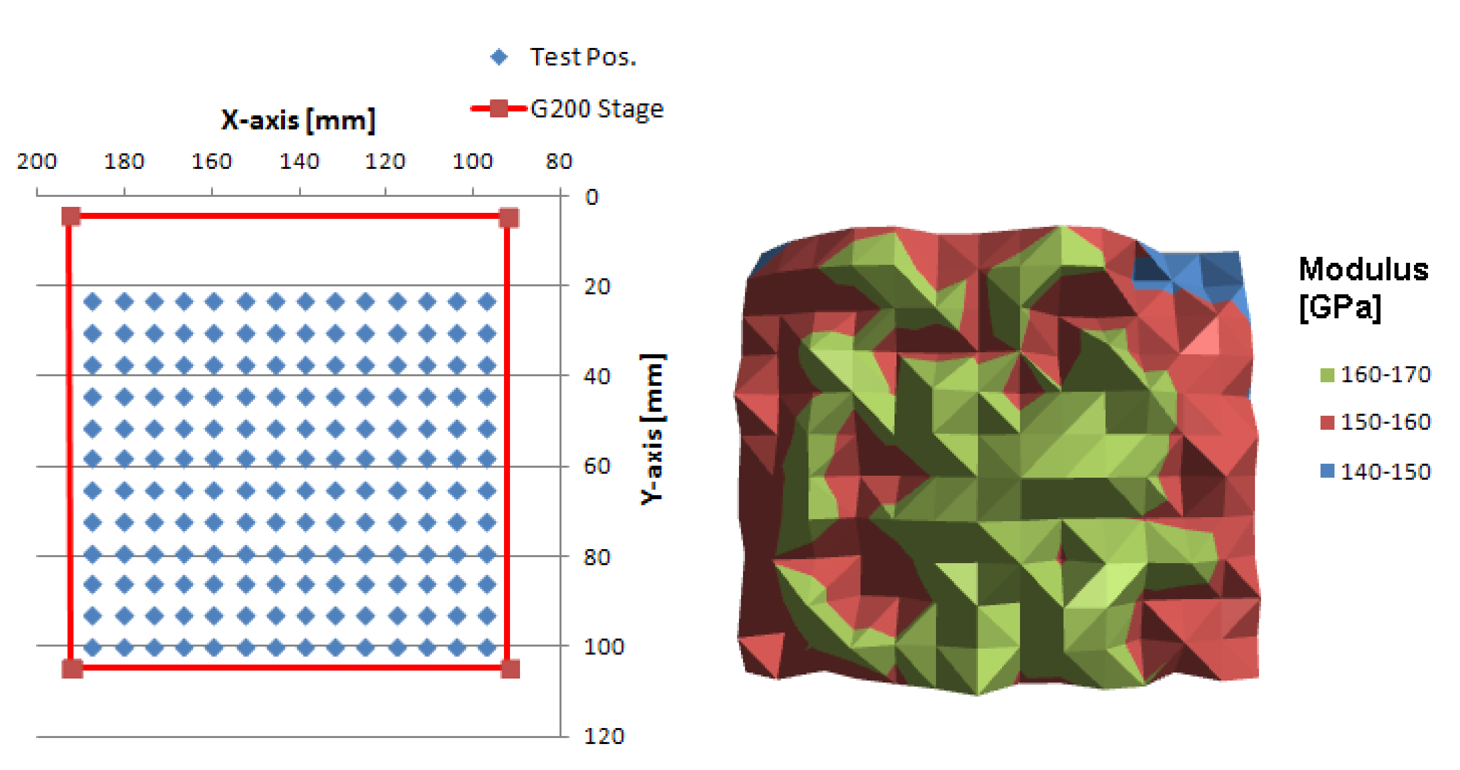
2. Handling of full wafer



Motorized vacuum stage for a full 200 mm wafer.

Technical aspects of polymer investigation:

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3. Flat punch indentation: demands very accurate substrate allignment

The nanoLab solutions:

the goniometer puck for 10x10 mm substrates

– precise manual surface alignment in the body of the XP/G200 puck
– not effecting the frame stiffness

the goniometer stage for wafer up to 2“

– manually or fully automated, software controlled alignment
– compatible to the XP/G200 sample tray

Technical aspects of polymer investigation:

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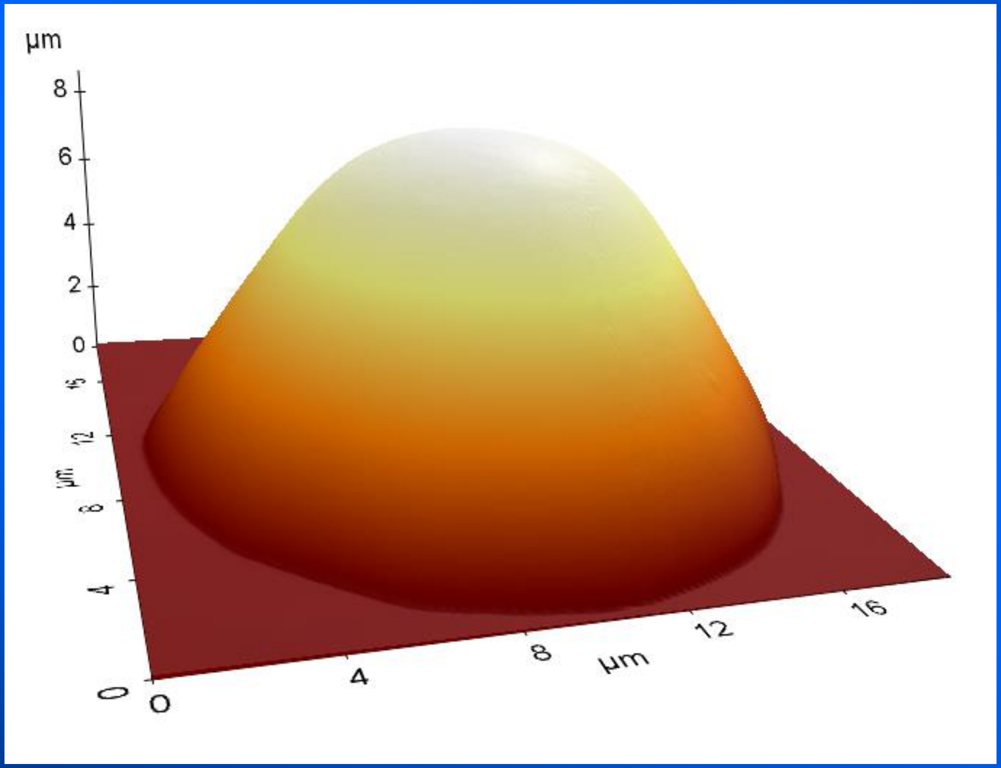
4. Proper calibration of spherical indenter



AFM topography of a cono-spherical indenter tip.

Cooperative Project:

Evaluation of spherical indenter tips made from sapphire.

Partner:

Fraunhofer Institut ENAS
Chemnitz

Infineon Technologies AG
Neubiberg

SURFACE, Hückelhoven

SYNTON-MDP AG
Nidau / CH

Summary and Outlook

- Thin polymer samples can be studied with nanoindentation

- Nanoindentation tests using CSM can reproduce bulk characterization tests

- Thermo-mechnical data can be obtained for µm-scaled samples

- CSM in combination with Laser heating enables mechanical loss spectroscopy

- **Ask for more tools!**

Thank you for your attention!