COMBINED NANOINDENTATION AND AFAM FOR MECHANICAL CHARACTERIZATION OF ULTRA LOW-K THIN FILMS

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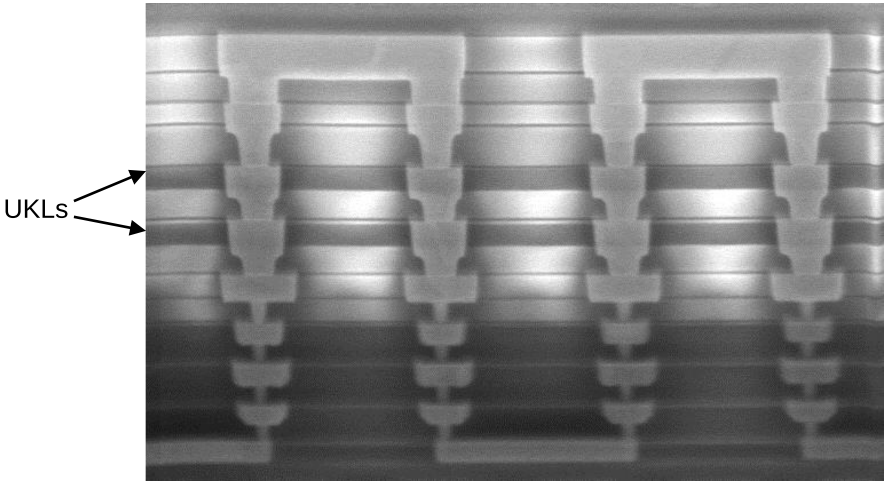
AGENDA

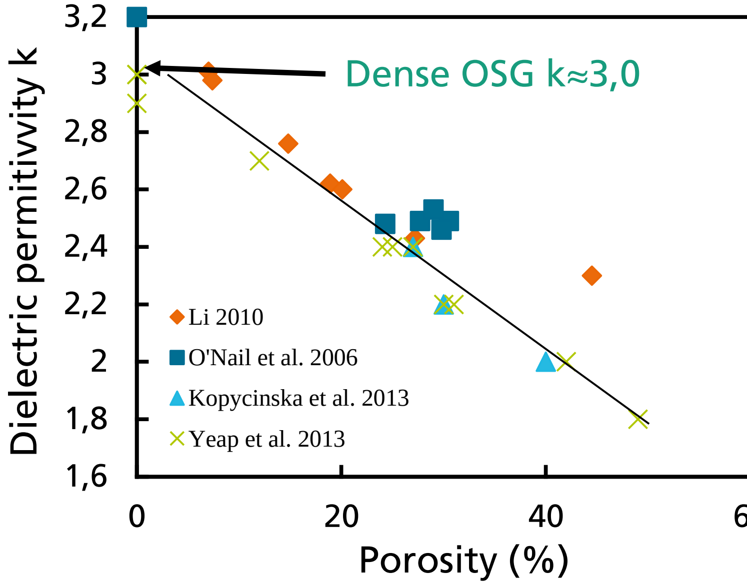
- Introduction, materials
- Nano-indentation of ULK film
- Combined Nano-indentation and AFAM
- Drawing conclusions on the pore topology using mechanical data
- Take home messages

Ultra Low-k nano-porous materials in nano-electronics



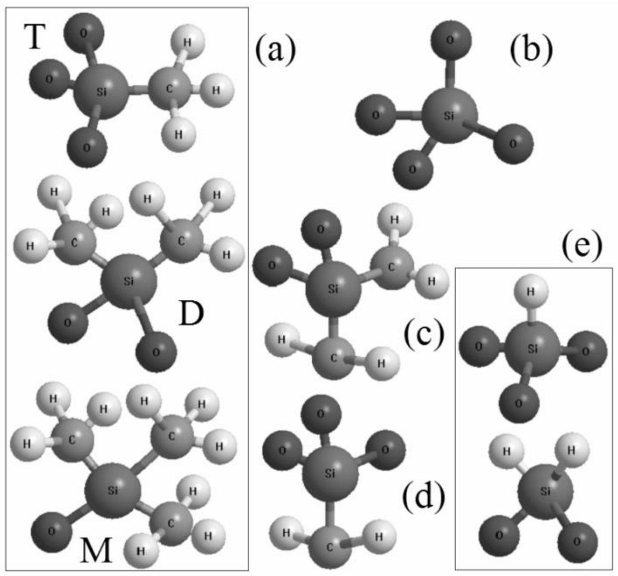
FIB cross-section of a 9 metal layer structure with Cu/low-K (OSG) interconnect system.

k-values of organosilicate glasses (OSGs)

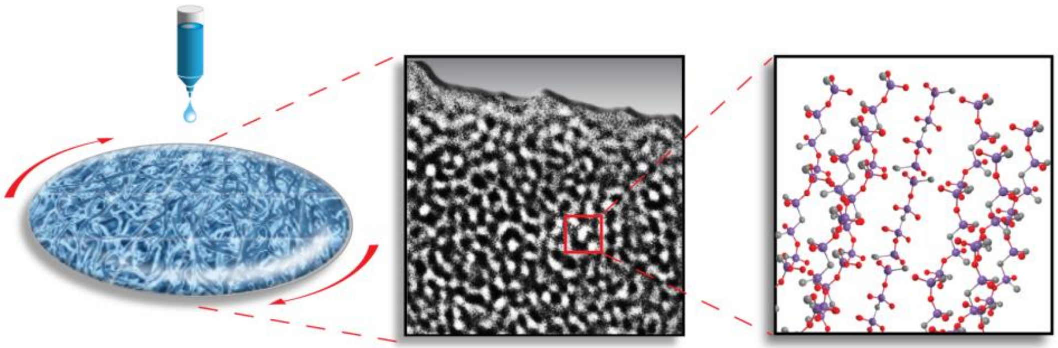


- Decreasing on-chip interconnect pitch (including inter-layer dielectrics dimensions) in nano-electronic products → higher signal delay, power loss, …
- Need of dielectric materials with ultra low k-values (ULKs)
- **Nano-porous organosilicate glasses (OSGs)** for k-values below 3,0

Fabrication of organosilicate glass UKL thin films



Samples of building block in the OSG network [1].



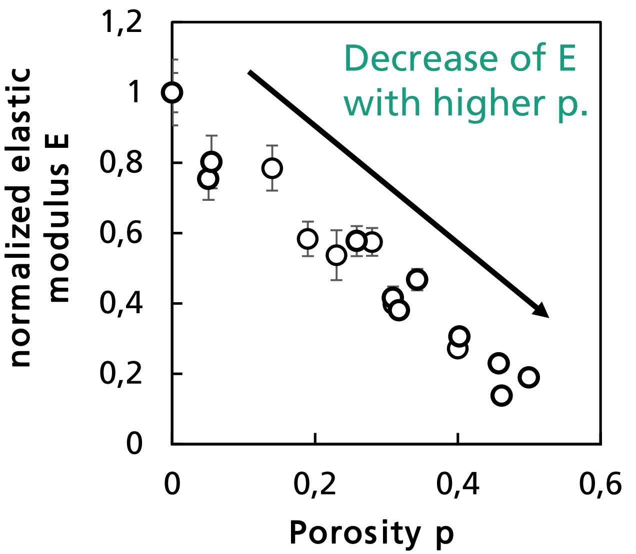
(Sol gel process)

Application: sol-gel processes using spin coating and final curing or CVD deposition.

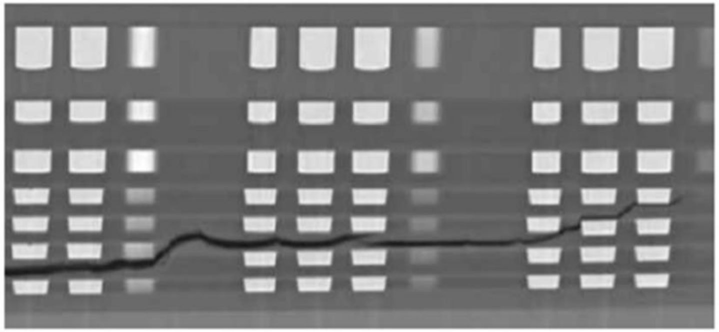
OSG chemistry can include porogens for insertion of controlled porosity.

[1]: Youbo Lin, PhD thesis, Cambridge, 2007.

Mechanical strength of nano-porous OSG ULKs



Criterion for crack propagation: $$\frac{\pi\sigma^2 a}{E} \geq 2\gamma$$ Low E leads to easy crack propagation.



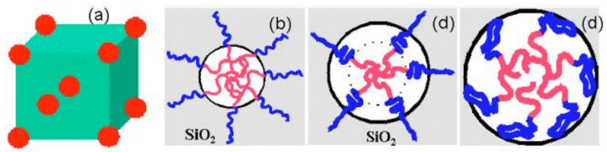
Crack propagation in a multilevel interconnect.

- Reliability issues caused by crack propagation (CPI, thermo-mech. stresses)
- Gradients in the ULK film can lead to electrical failure even if mean k-value is OK

➢ **Mechanical characterization of the ULK films is important (E, Gradients)**

Motivation: high elastic modulus at a given k-value

- Optimizing the chemical structure and/or the pore topology
- One example: producing an ordered pore structure:



Self assembly sol gel process (SBA materials): Triblock co-polymer, removed by thermal or UV curing.

TABLE 1. Sample description and experimental results for SA-OSG and CVD films.

Sample	Target *k*	Actual *k*	Porosity, *p* (%)	Thickness (nm)	Elastic Modulus, *E* (GPa)	Cure process
SA-OSG6	2.4	2.41	24	693	7.3 ± 0.3	Thermal
CVD-OSG1	2.4	N.A.	25	530	3.7 ± 0.3	UV & Thermal

Techniques needed for the measurement of the mechanical properties of nano-porous thin OSG films

→ Nanoindentation and AFAM

Samples: OSG thin film samples from SBA materials

First set of SBA SA-OSG ULKs

Sample	K	Porosity p	Film thickness (nm)
1	3	0,000	582
2	2,88	0,051	607
3	2,87	0,055	553
4	2,39	0,258	564
5	2,27	0,309	512
6	2,25	0,318	491
7	2,19	0,343	490
8	2,05	0,403	492
9	1,92	0,458	490
10	1,91	0,462	470
11	1,82	0,500	504

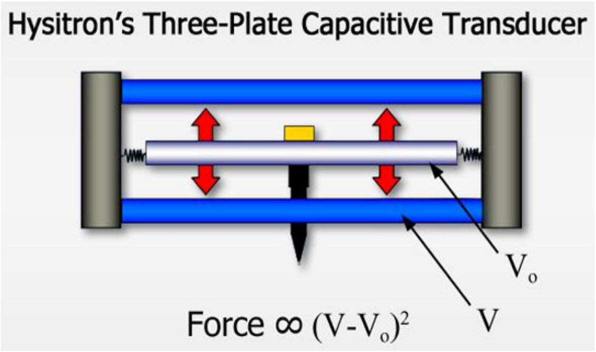
Second set of SBA SA-OSG ULKs

Sample	k	Porosity p	Film thickness (nm)
1	2	0,4	511,14
2	2,2	0,31	502,38
3	2,3	0,28	468,87
4	2,4	0,23	321,09
5	2,5	0,19	393,14
6	2,6	0,14	219,92
7	3	0	335,93

- SBA Spin-on OSG ULKs featuring a self-assembly process of the porogen

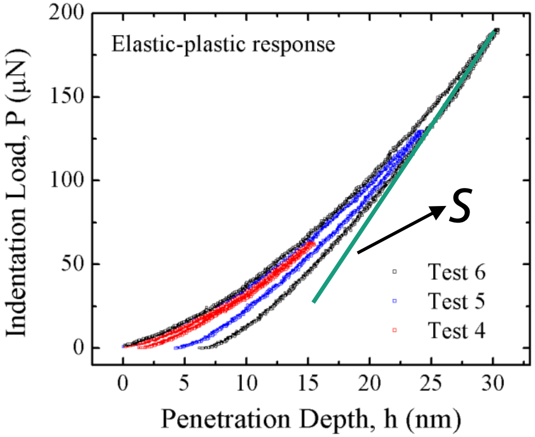
Nano-indentation

Hysitron TI-950



A schematic of the Hysitron nanoindentation system.

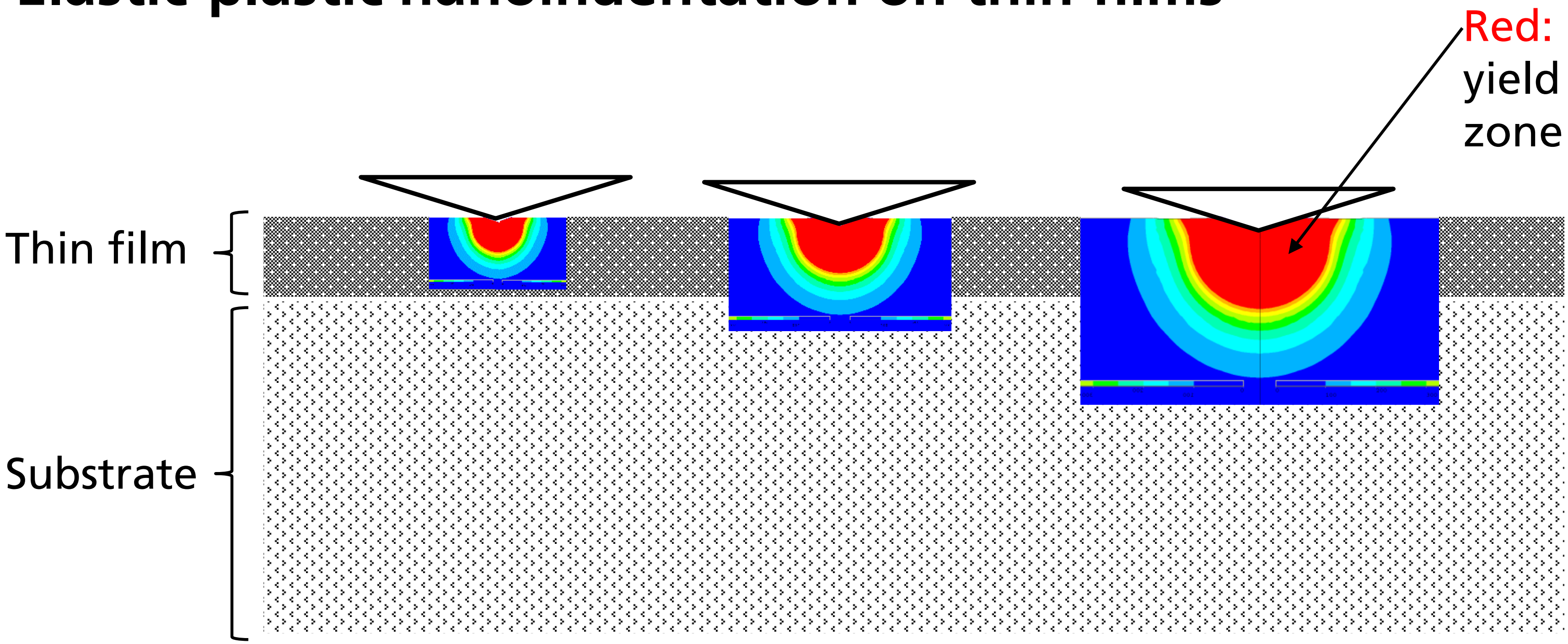
Elastic-plastic contact with Berkovich tips



$$S = \frac{dF}{dh} = \beta \frac{2}{\sqrt{\pi}} E^* \sqrt{A_c}$$

- **Hardness H**
- **Elastic modulus E**

Elastic-plastic nanoindentation on thin films



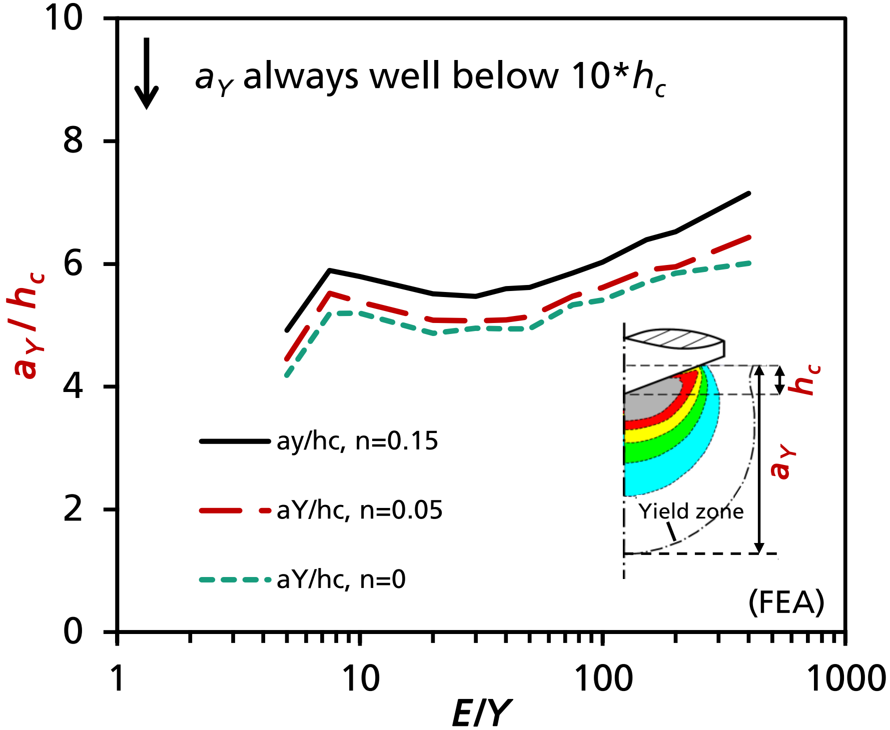
- **Hardness H**
  - Mainly determined by the yield zone
  - Local property
  - Low Substrate influence
- **Elastic modulus E**
  - Mainly determined by the elasic field outside the yield zone
  - More a global property
  - High subtrate influence

Indentation hardness and modulus of thin films

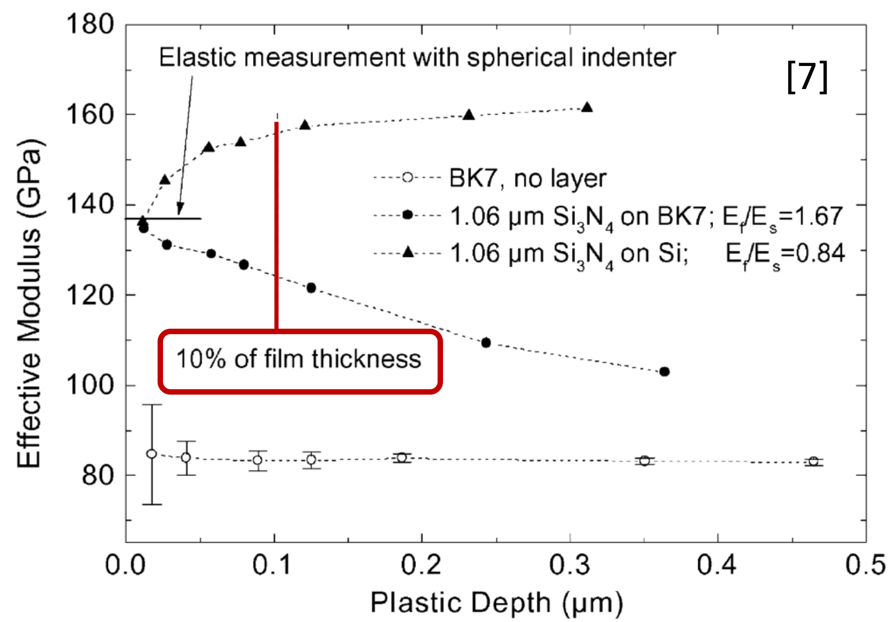
- Hardness is ruled by the yield zone
- Yield zone should not reach substrate
- ➢ Safe contact: $h_c < 1/10 * h_{film}$ (Bückle rule)

- For E, 10% rule is **not** appropriate
- Elastic fields outreach much further
- ➢ Indentation depths < 1% of h_{film} needed

Yield zone radius vs. contact depth



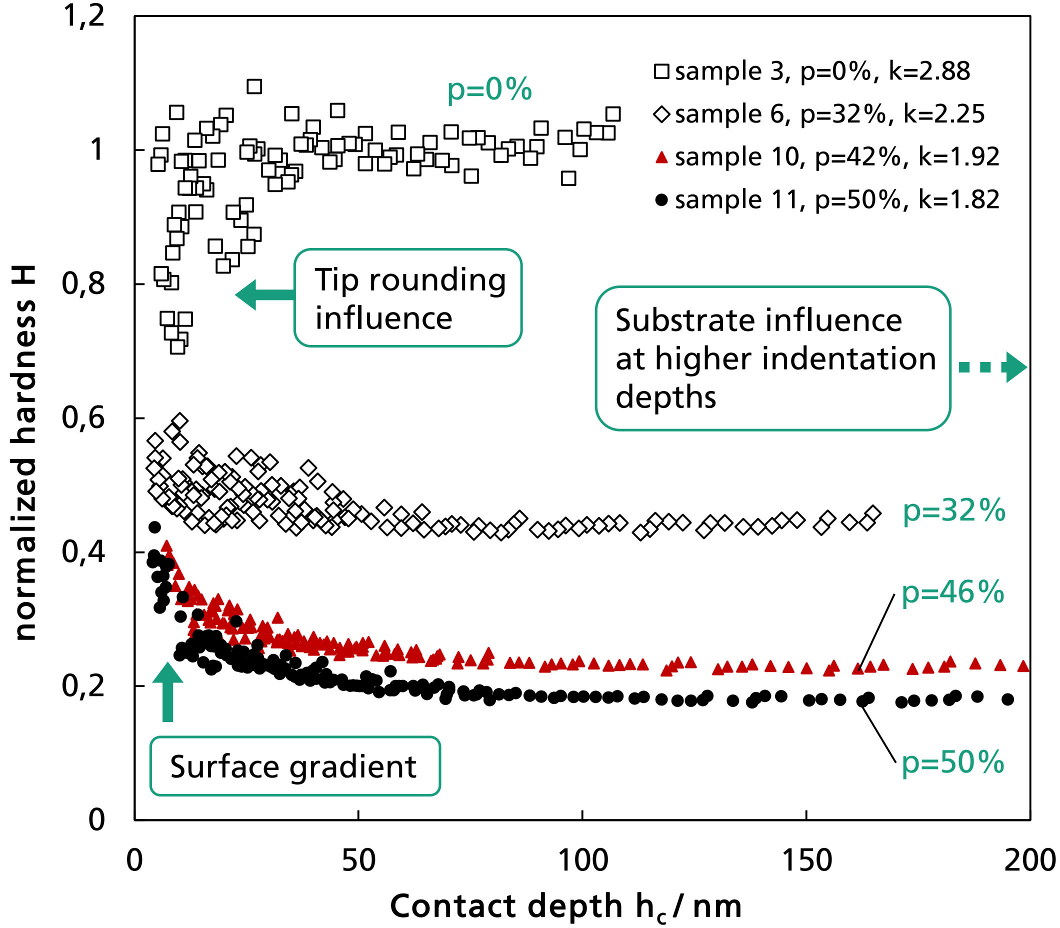
Film- vs. substrate modulus



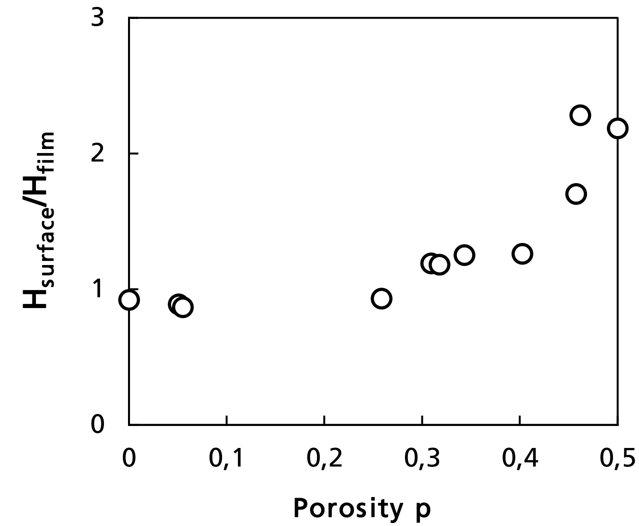
[7]: Surf. Coat. Tech. 154, 140–151 (2002).

Indentation hardness H for the first sample set

Hardness gradients vs. porosity p



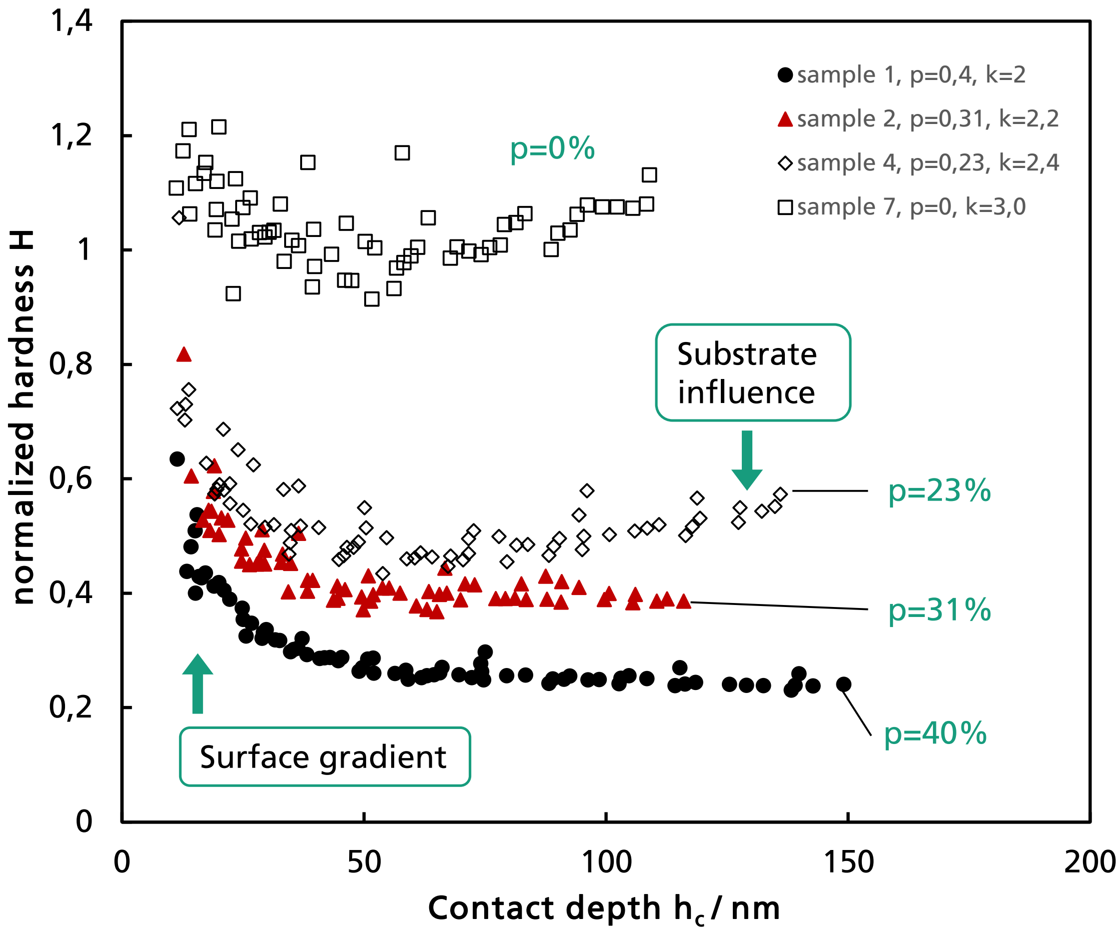
Hardness increase at the surface vs. p



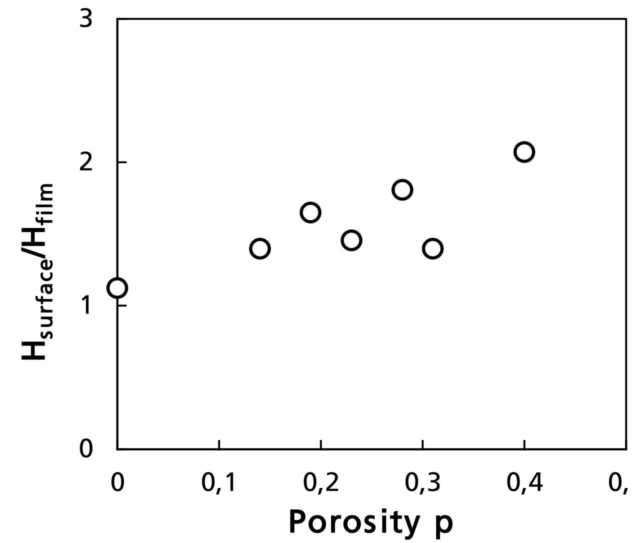
- Porosity dependent surface gradient
- Harder and maybe denser top layer

Indentation hardness H for the second sample set

Hardness gradients vs. porosity p

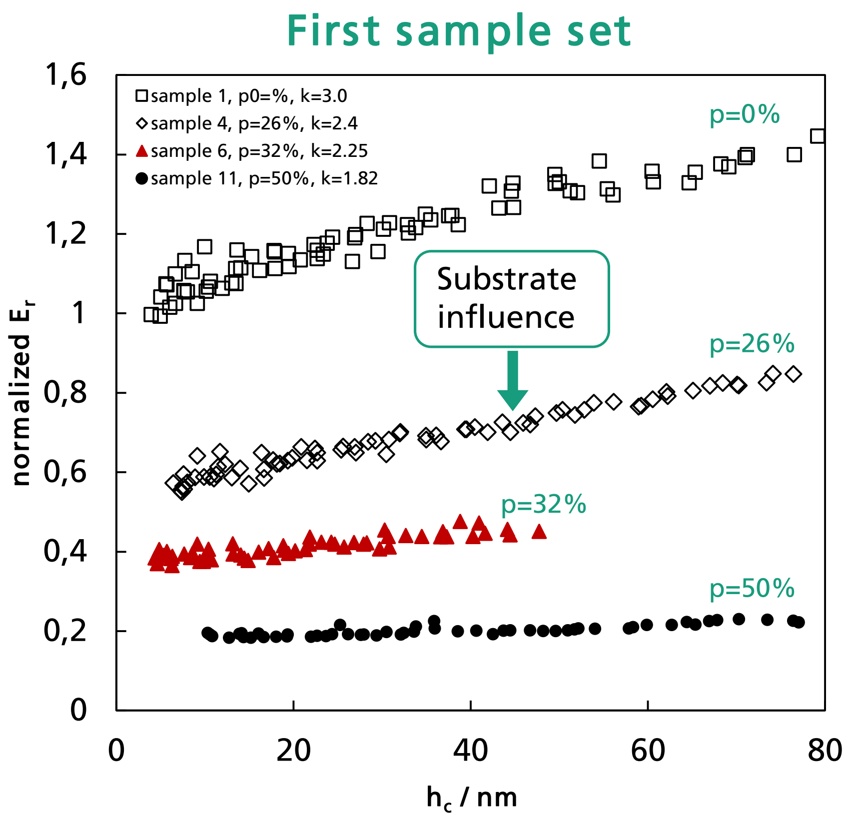


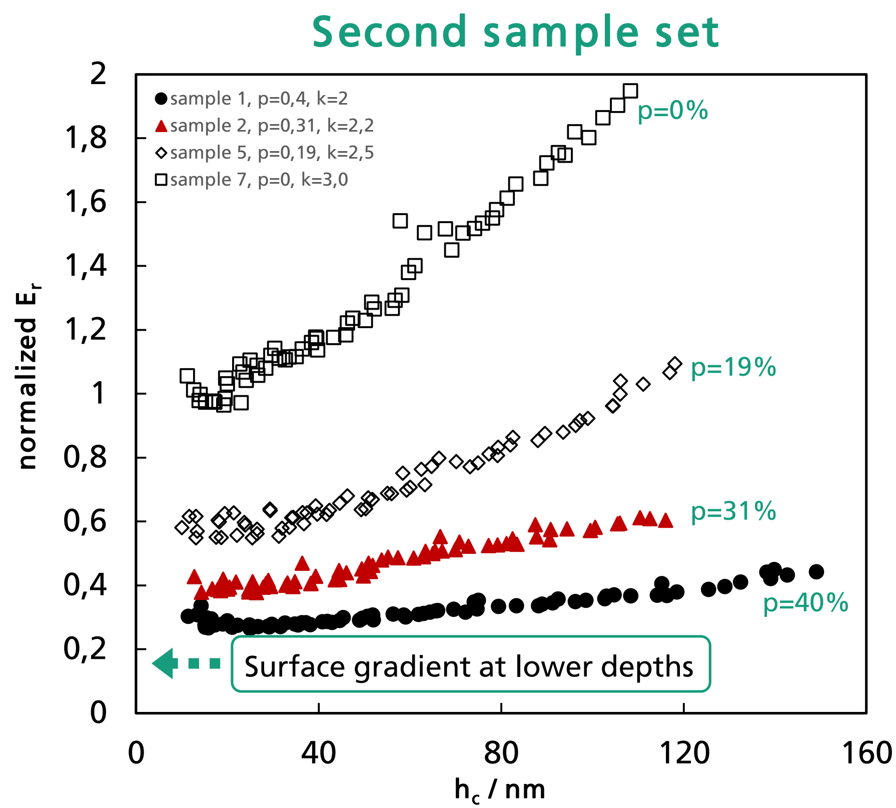
Hardness increase at the surface vs. p



- Porosity dependent surface gradient
- Harder and maybe denser top layer

Nano-indentation results for the Elastic modulus E





- Forces are too high to significantly surpass substrate influence
- Surface gradient not visible
- Need for a higher resolution E measurement → **AFAM**

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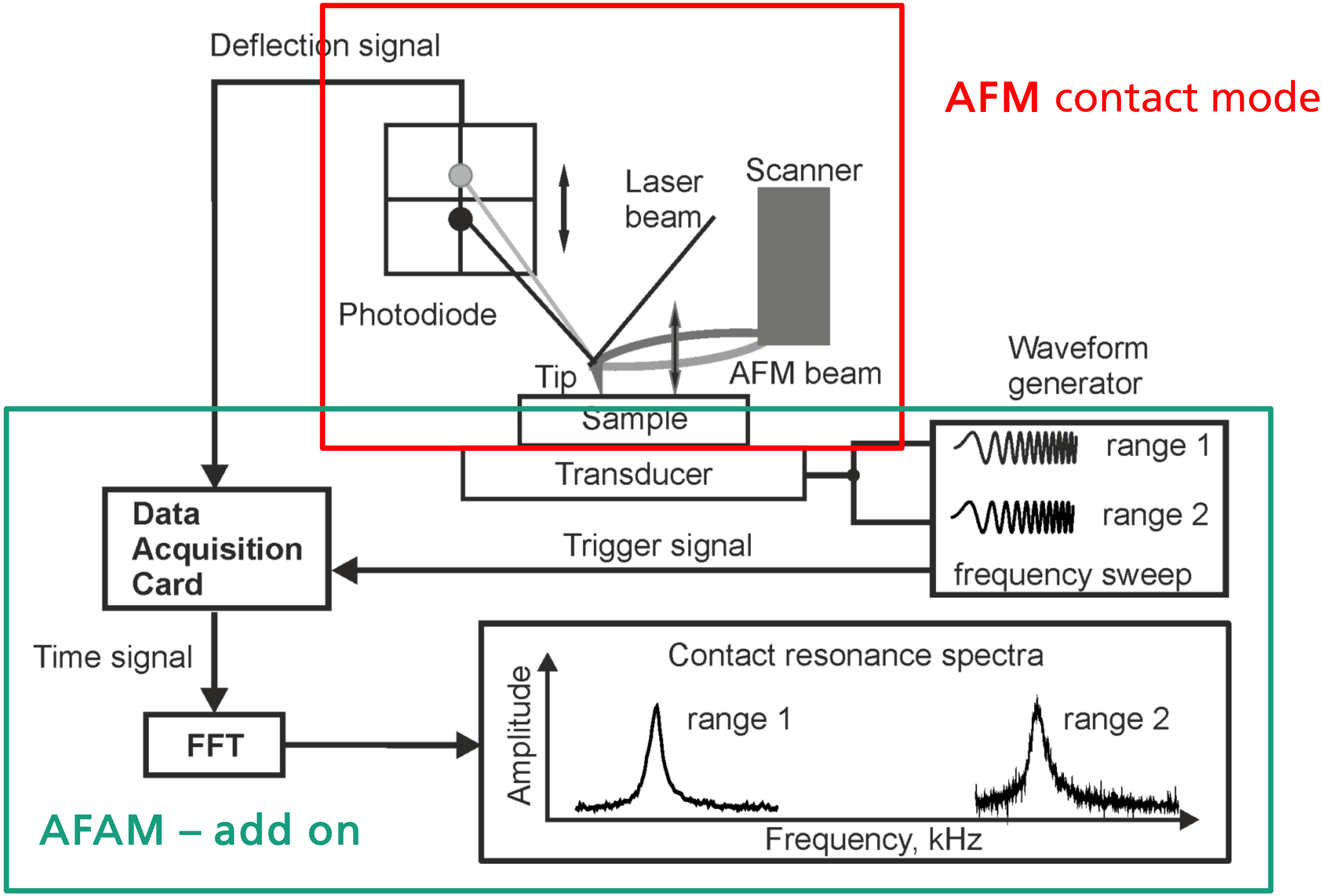
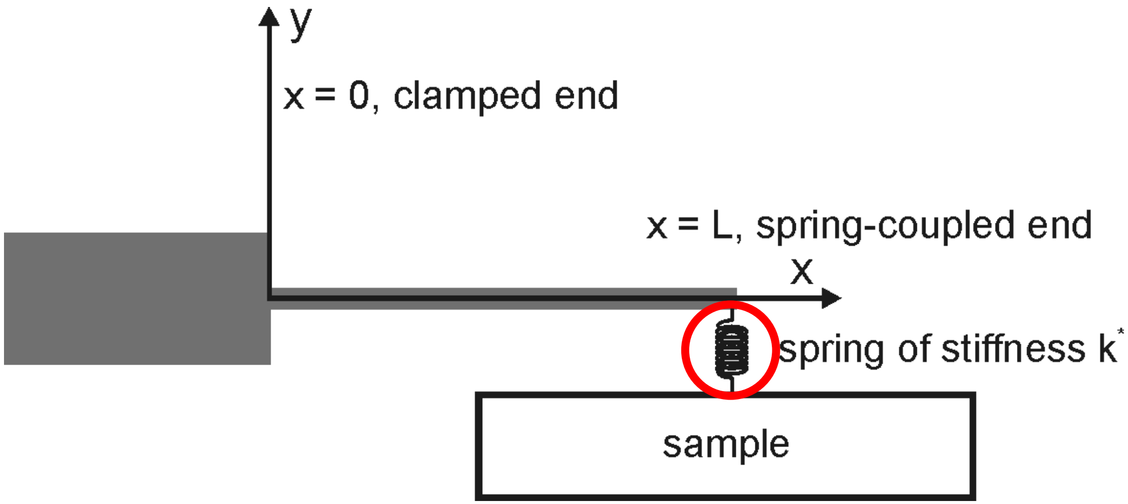
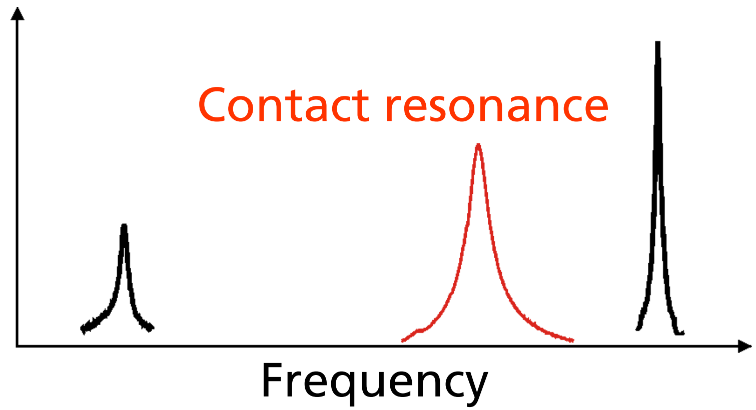
AFAM principle



Contact resonance frequencies of an AFM cantilever





dynamic behavior of the cantilever

tip-sample contact stiffness

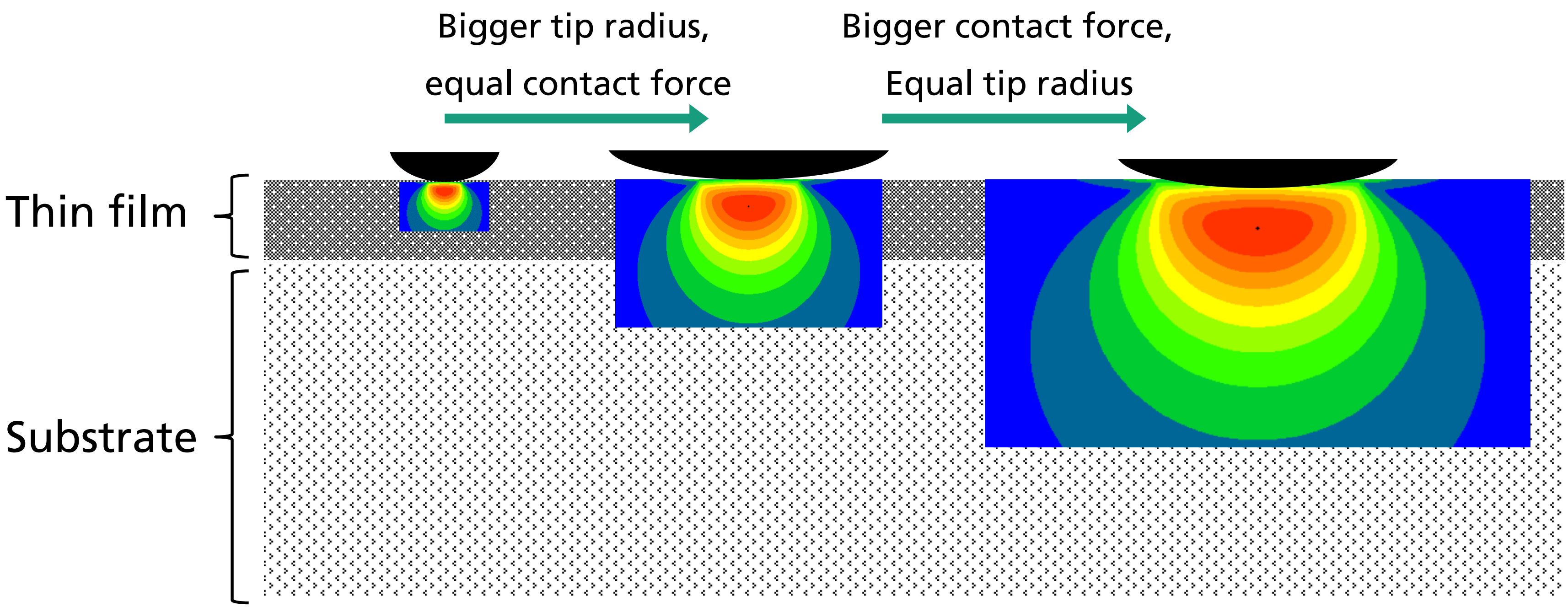
contact mechanics

$$f_n = \mathrm{F}_i(k^*, k_c, L_1, L)$$

k^*

$$k^* = 2aE^*$$

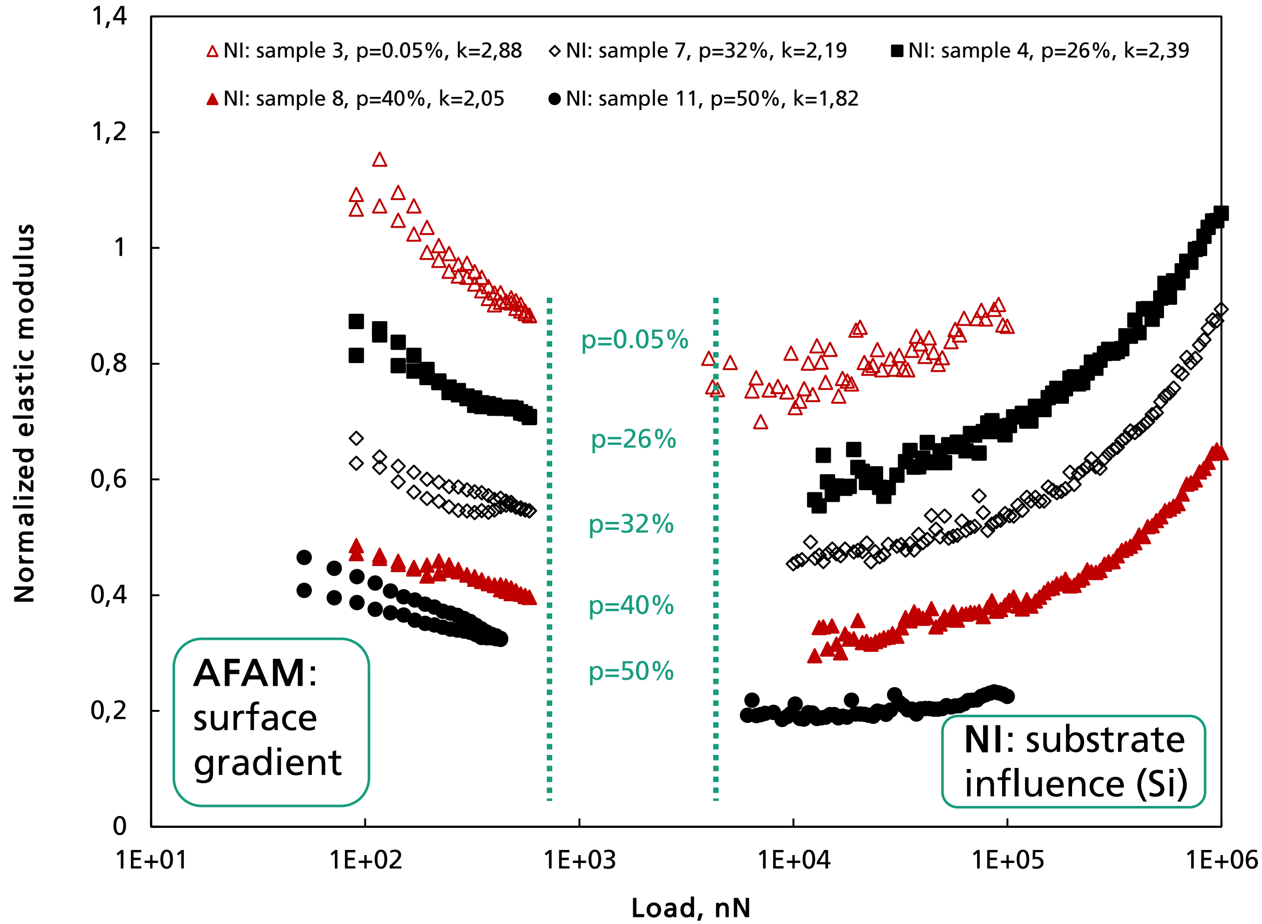
Elastic contact on thin films



- **Main aspects for the substrate influence on the E measurements**
  - Tip radius → the bigger the tip radius the deeper reaches the elastic field
  - Contact force → the bigger the contact force the deeper the elastic field

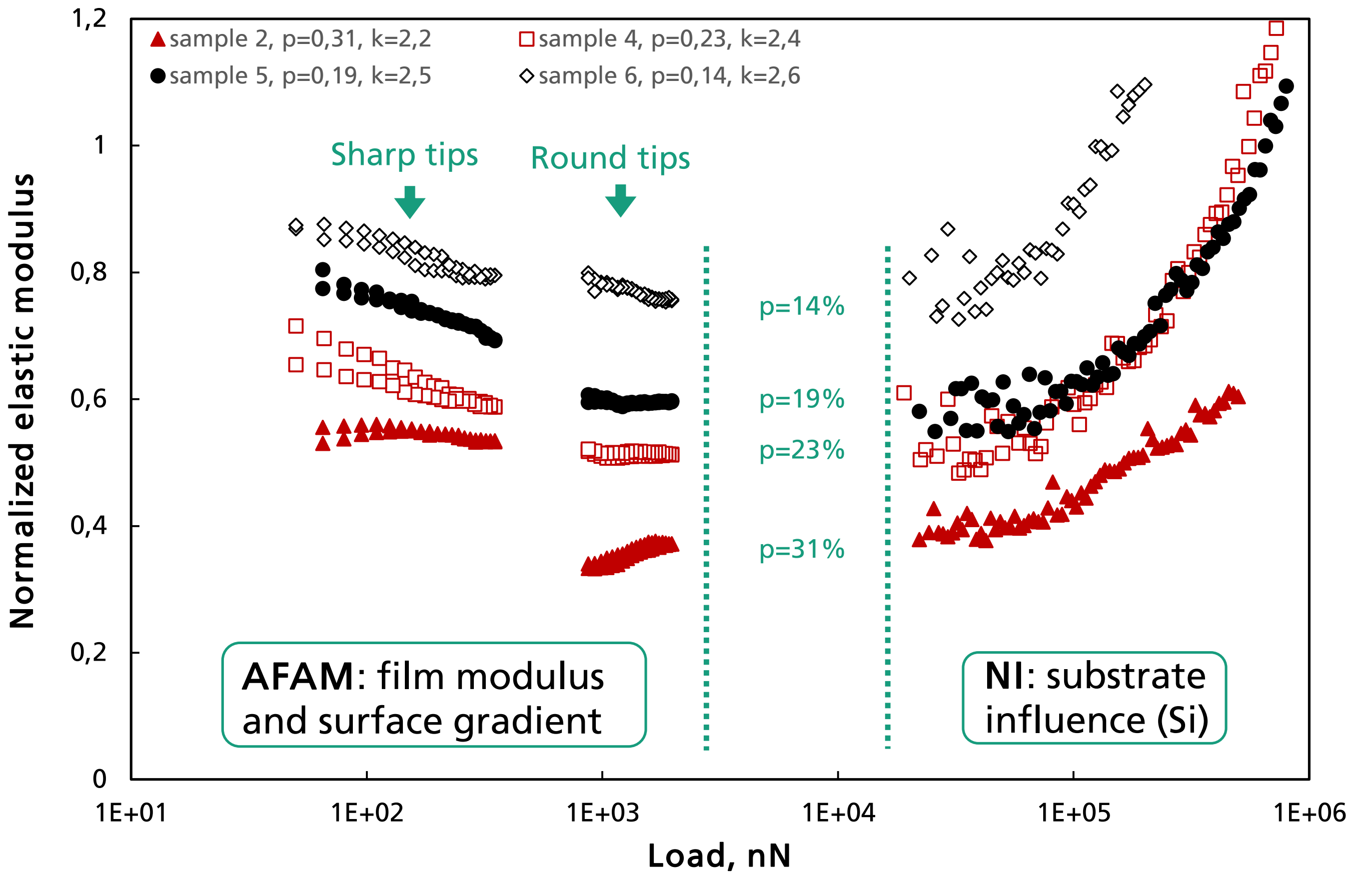
Low forces and sharp tips for the E-gradient measurements

Combined AFAM and nano-indentation: First sample set



- Surface gradients for the elastic modulus become visible via AFAM!
- AFAM studies intensified for the second sample set

Combined AFAM and nano-indentation: Second sample set



- Surface gradients for the elastic modulus become visible with sharp tips
- Film modulus becomes visible for round tips

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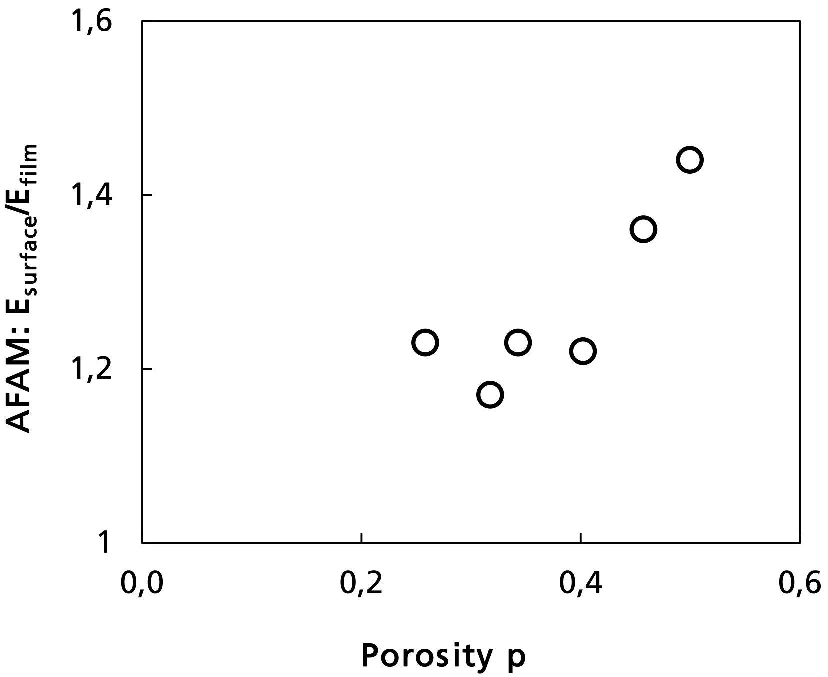
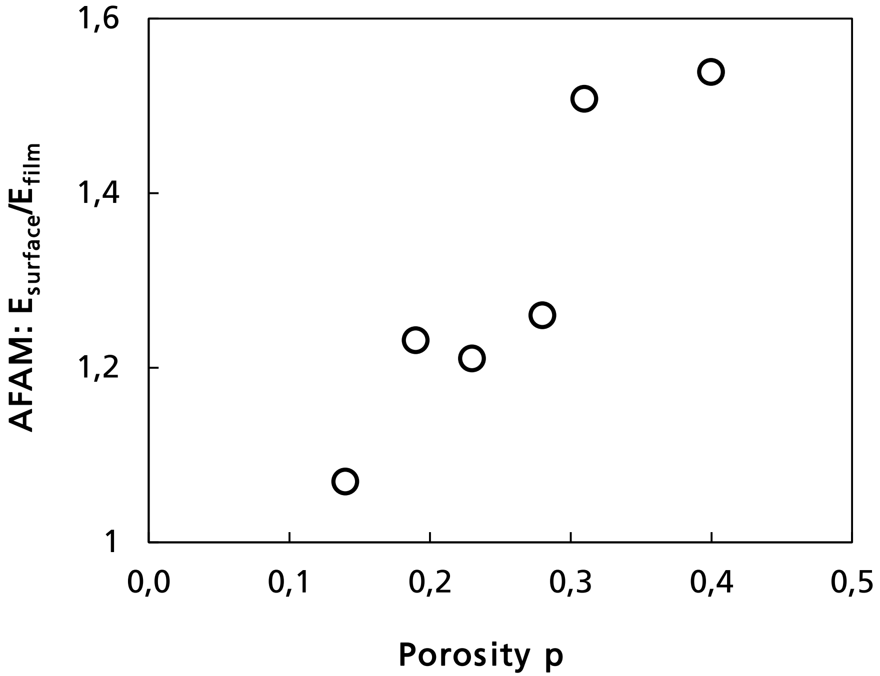
Surface gradient in the elastic modulus, AFAM results

First sample set



Second sample set



➢ AFAM also shows porosity dependent surface gradient

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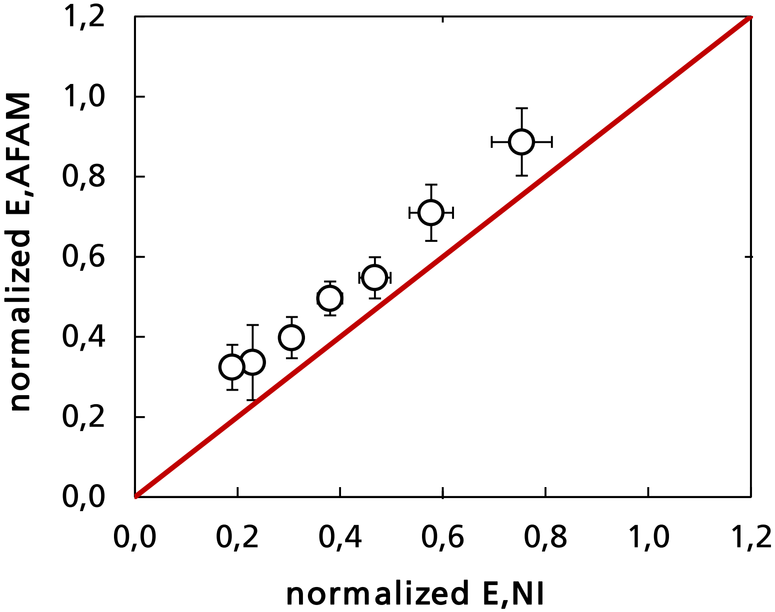
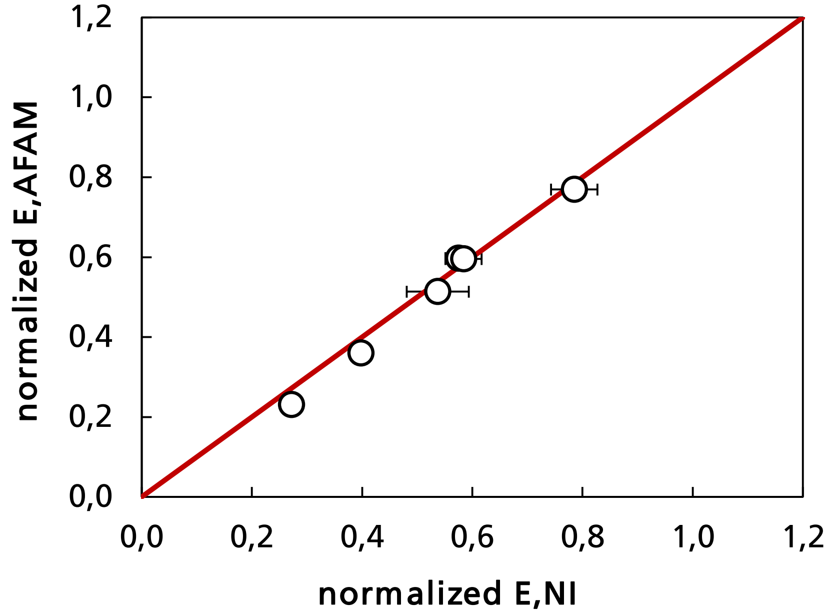
Comparison of AFAM and nano-indentation results

First sample set

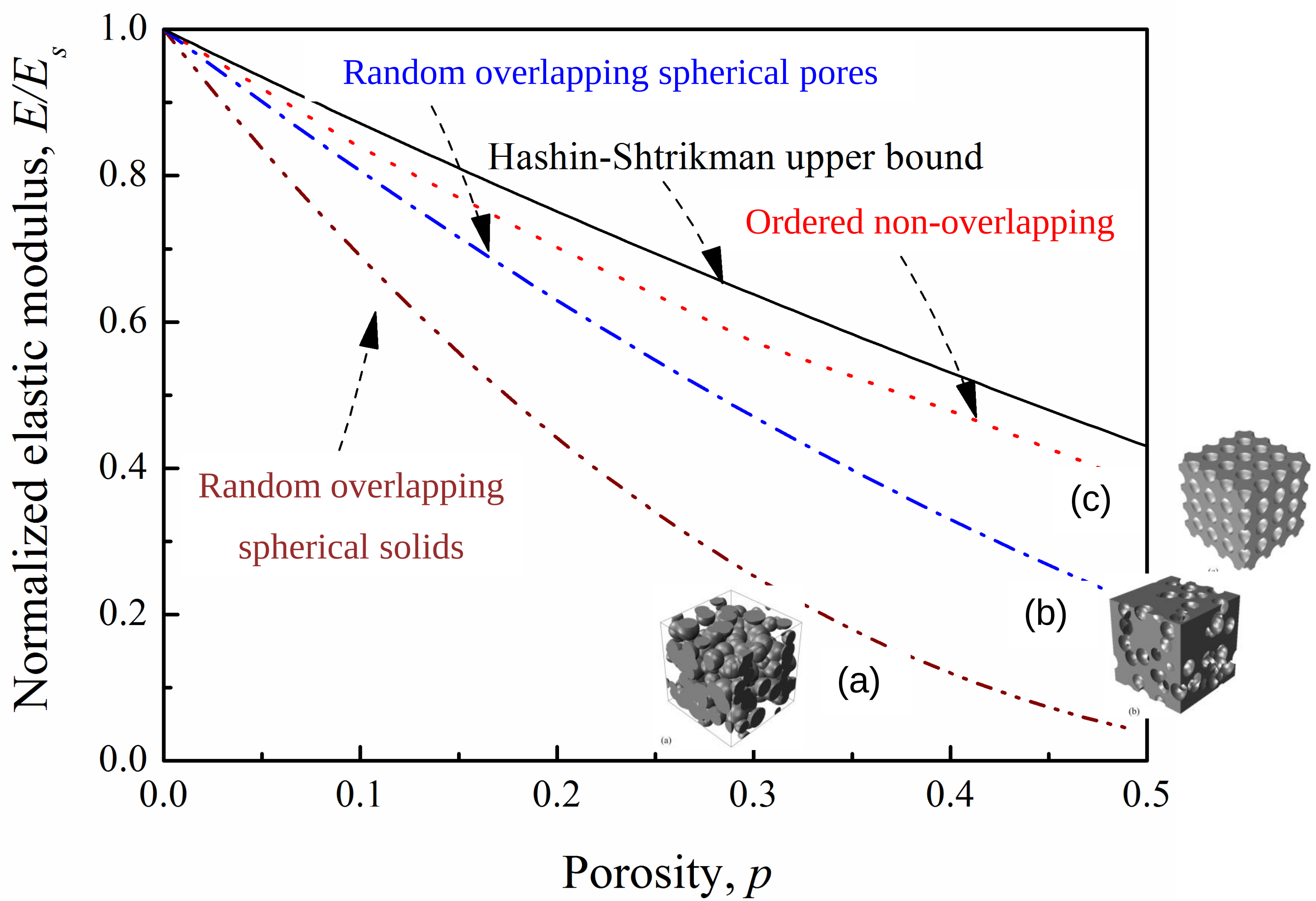


Second sample set



➢ AFAM shows very comparable results to nano-indentation

OSG pore topology and elastic modulus



K. B. Yeap et al., J. Mater. Res. (2013)

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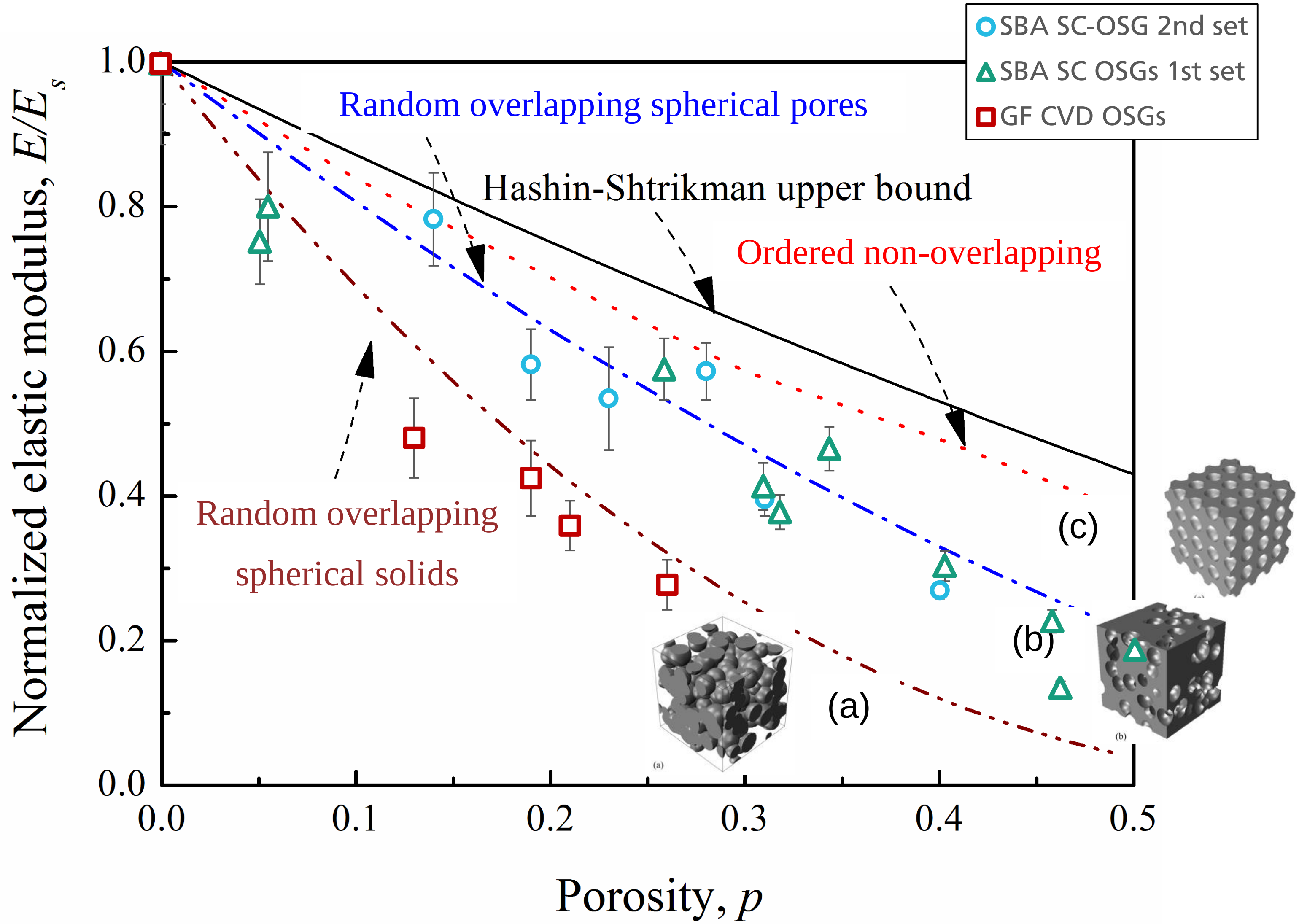
OSG pore topology and elastic modulus



K. B. Yeap et al., J. Mater. Res. (2013)

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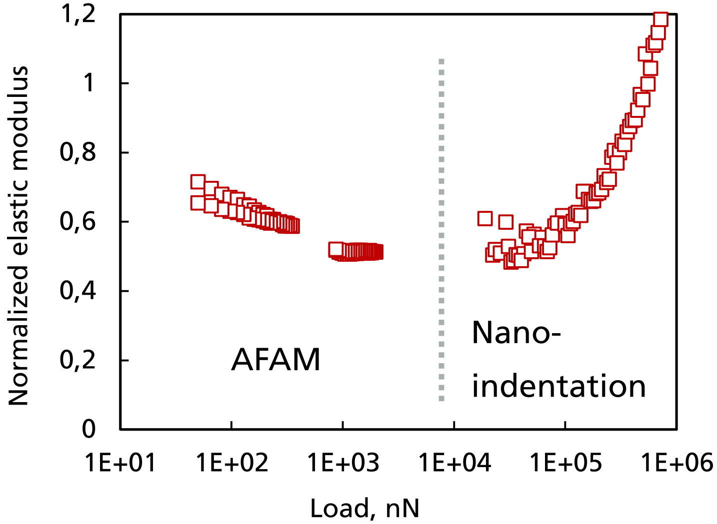
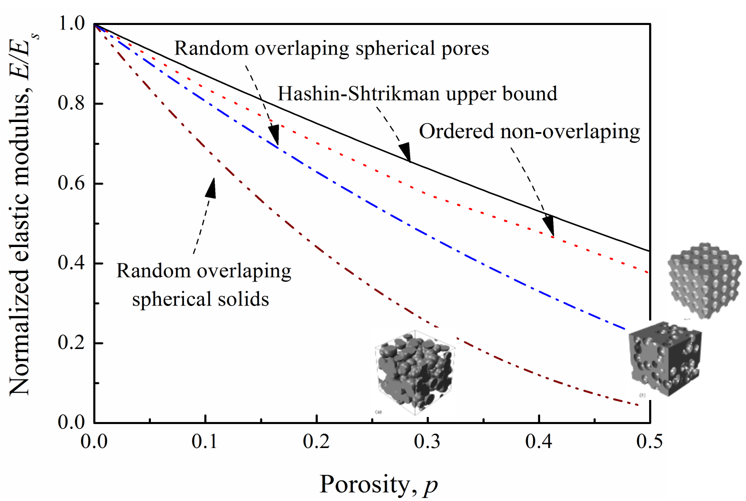
Take home messages



- AFAM and nano-indentation complement each other well for the mechanical characterization of porous thin films



- From mechanical data of porous thin films, conclusions about the pore-topology can be drawn

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