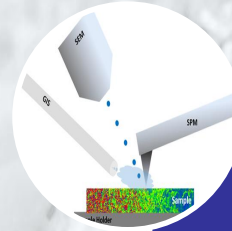


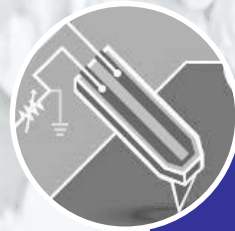
Thermo-physical properties of 500 nm thin Tungsten films investigated with Time Domain Thermoreflectance and Scanning Thermal Microscopy

Katrin Fladischer

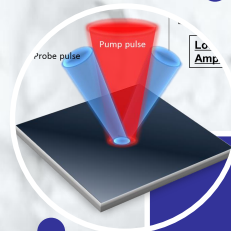




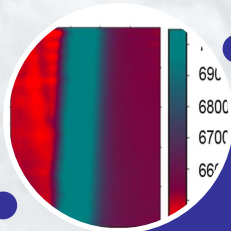
Outlook



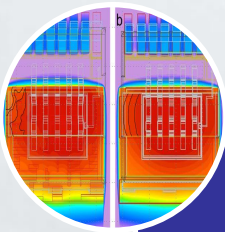
W thin film investigated by SThM



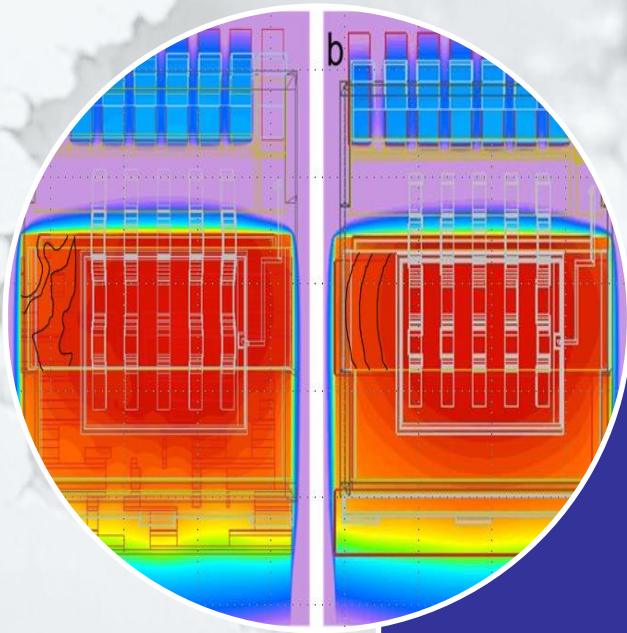
W thin film investigated by TDTR



SThM @ mcl

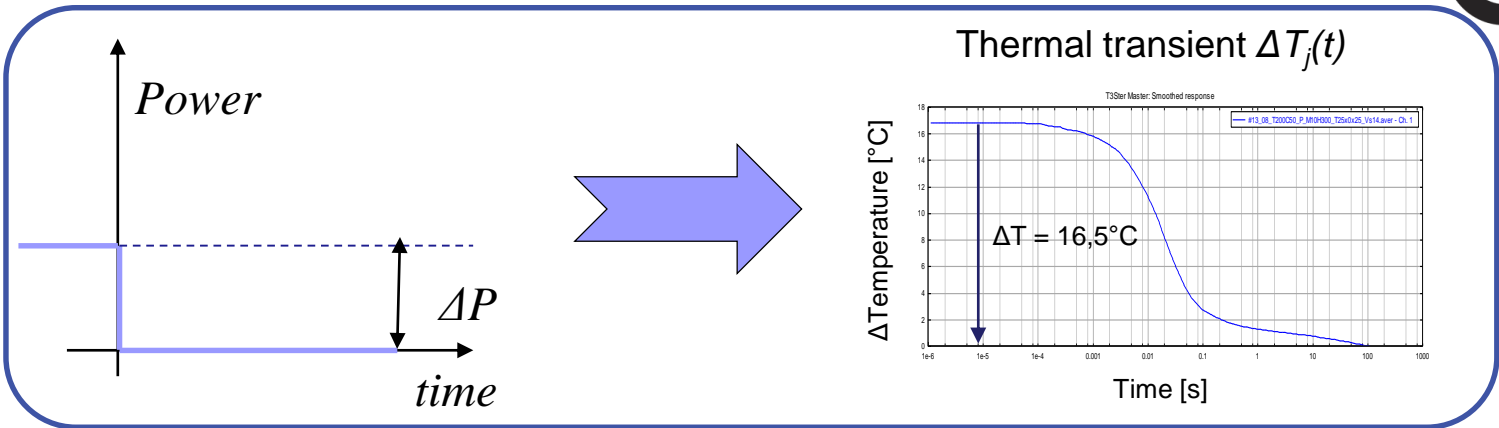


Motivation: Thermal Management @ mcl

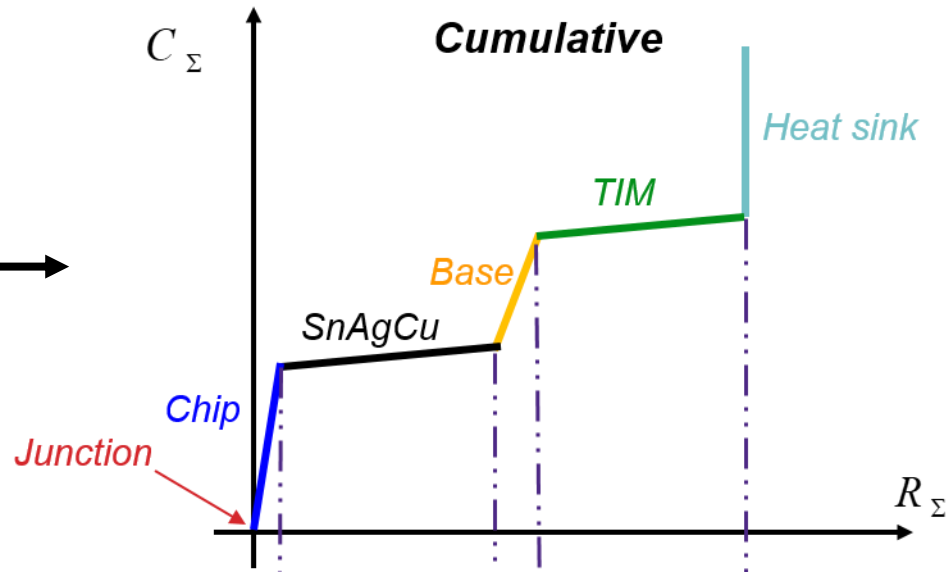
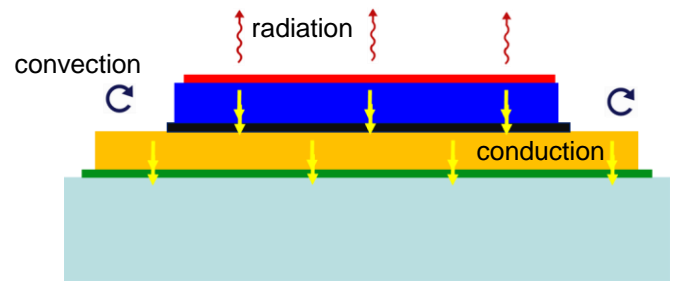
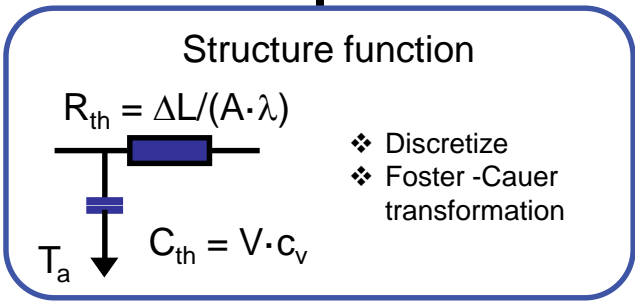


Motivation: Thermal Management @ mcl

Motivation: Thermal Transient Analysis

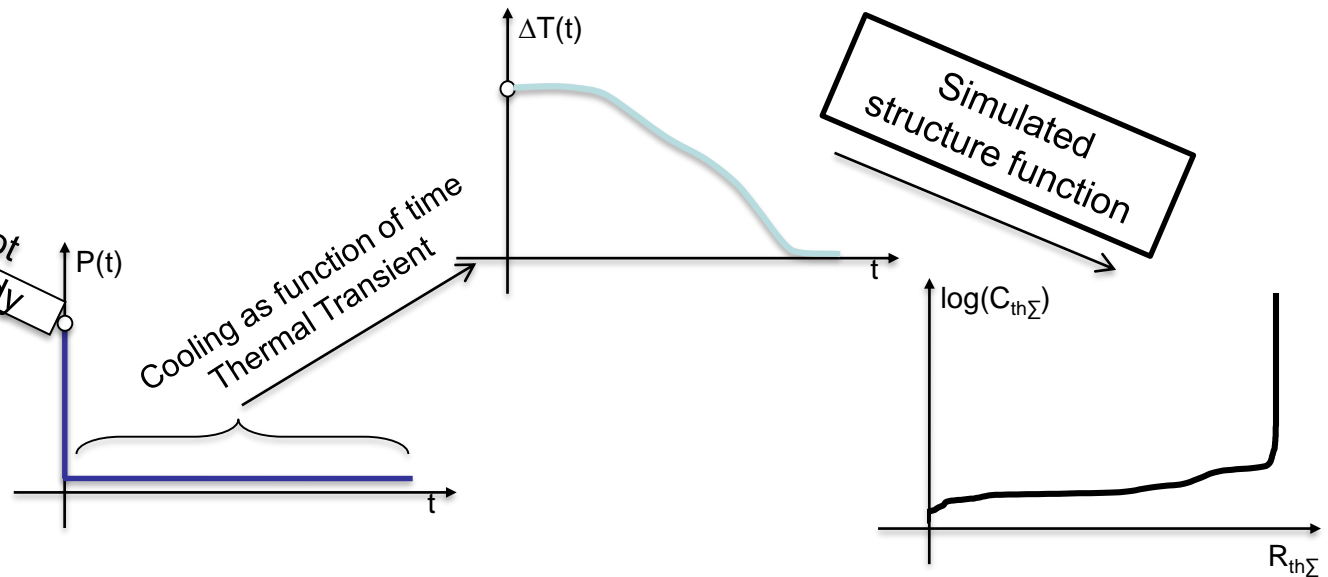
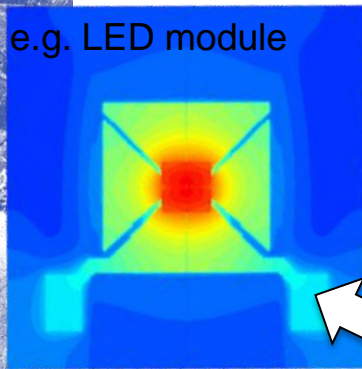


Time constant spectrum $R_\zeta(\zeta)$



→ can be measured

Simulation is used to create the thermal transient.



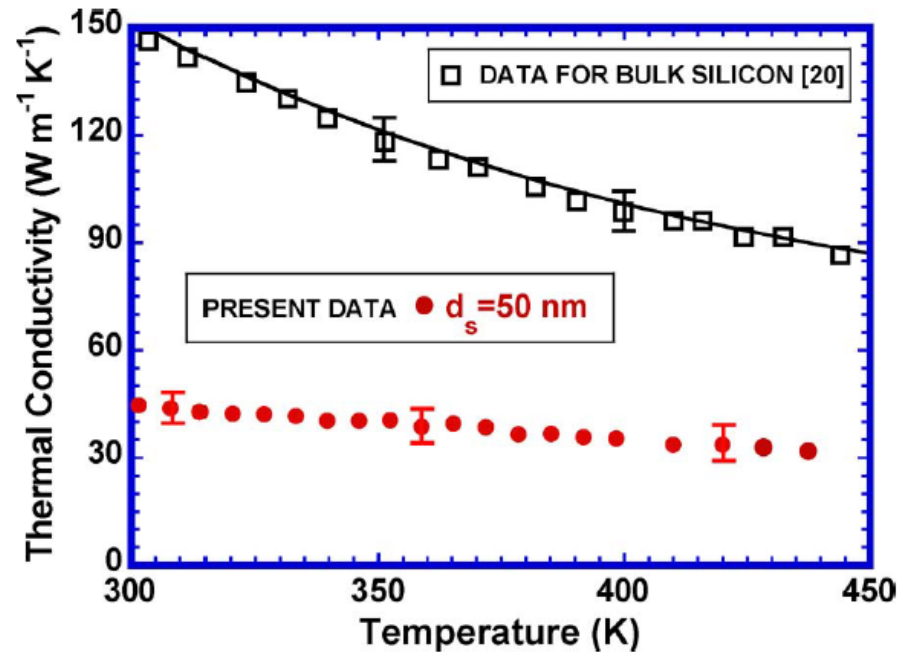
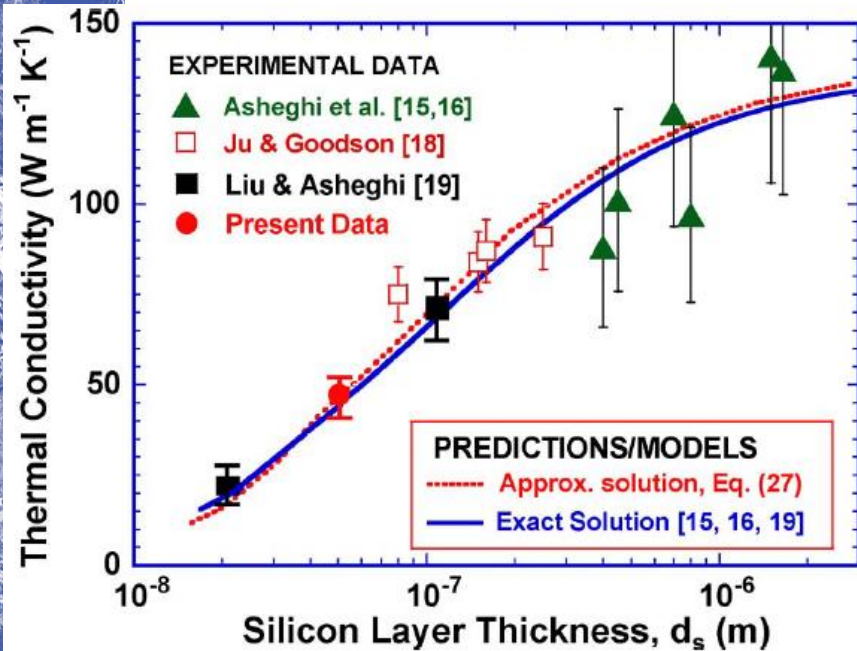
Comparison of simulation and measurements.

Simulated structure function = measured structure function

→ **Validated system**

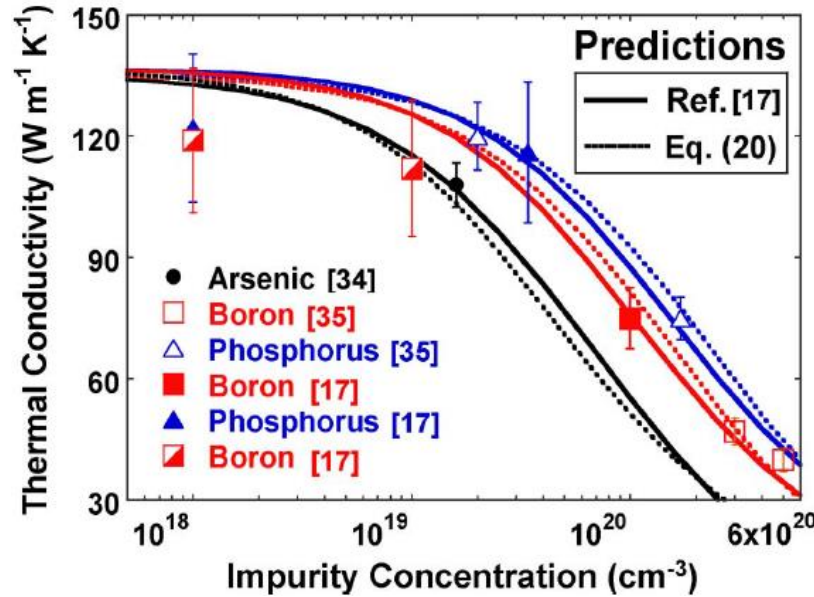
Adjustment of the module's material stack:

Variation of ρ, λ, c_p

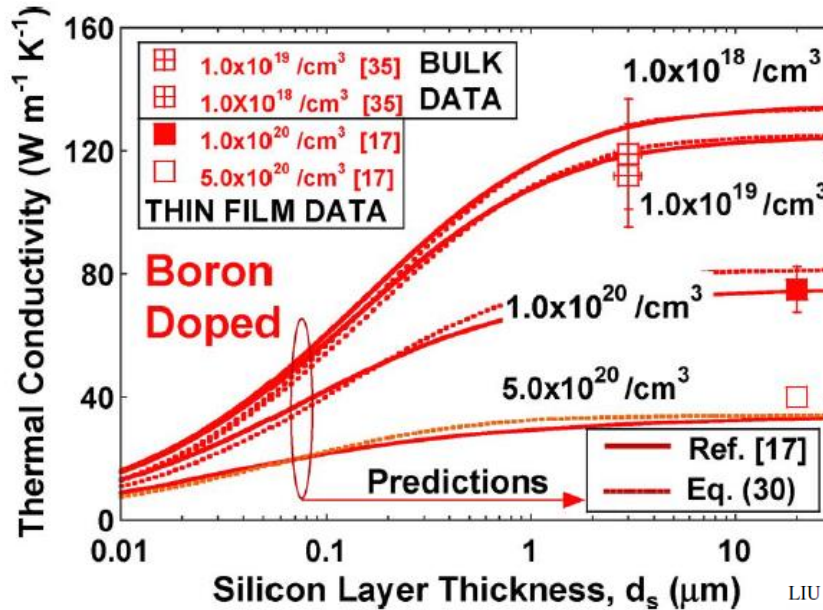


→ Thermal conductivity of 50 nm thin layer of silicon is significantly lower than that of the bulk: from 150 W/mK to 45 W/mK

→ Temperature dependency follows different law for bulk and thin layers.



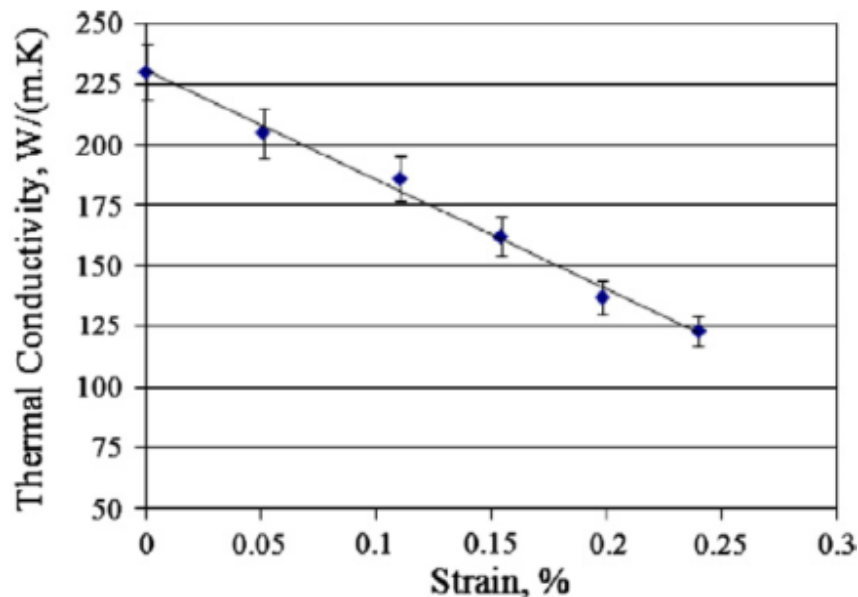
→ The higher the impurity concentration the lower the thermal conductivity.



impurity concentration ↑

125 nm Al-films with average grain size of 50 nm
(no thickness effect – 3 times the mean free path)

Strain–thermal conductivity coupling:

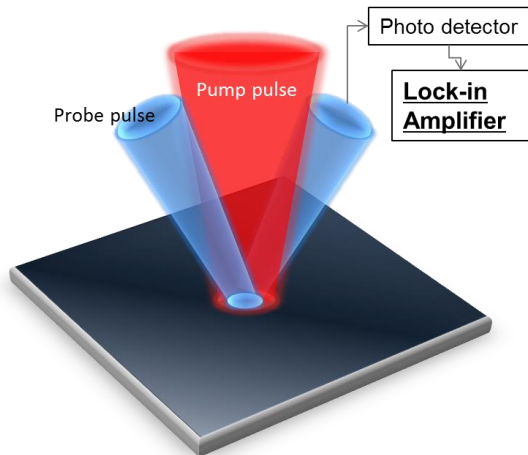


50% reduction in thermal conductivity at $\sim 0.25\%$ strain (~ 175 MPa of stress)

mechanical strain **decreases the electron mean free path**
→ enhanced scattering at the moving grain boundaries

Lee, H. F.; Kumar, S.; Haque, M. A. Role of Mechanical Strain on Thermal Conductivity of Nanoscale Aluminum Films. *Acta Mater.* 2010, 58, 6619–6627.

Time Domain Thermal Reflectance TDTR



Measurement principle

- Optical reflectivity R changes with the temperature.

What can be measured?

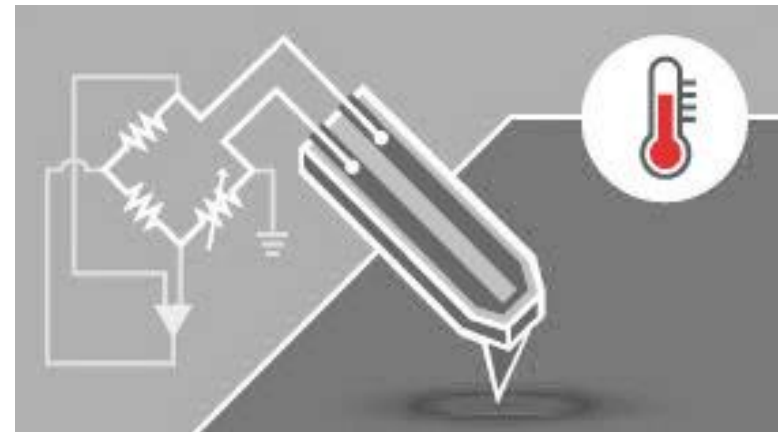
- Thermal effusivity, diffusivity, conductivity and interfacial thermal resistance of layers

On which scale?

- 10 nm to 20 μm thick layers averaged over probe pulse \varnothing 25 μm

Oven for sample investigation up to 500°C

Scanning Thermal Microscopy S_ThM



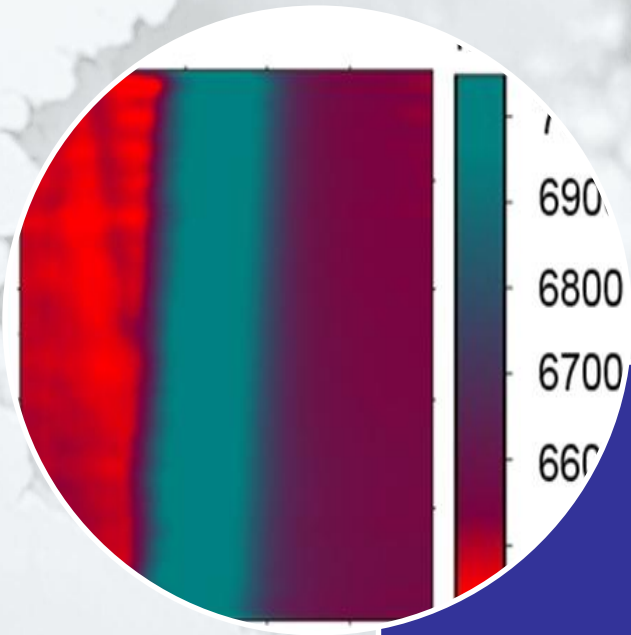
www.parkafm.com

What can be measured?

- Temperature measurement
- Thermal conductivity
- Topography

On which scale?

- Nanometer scale

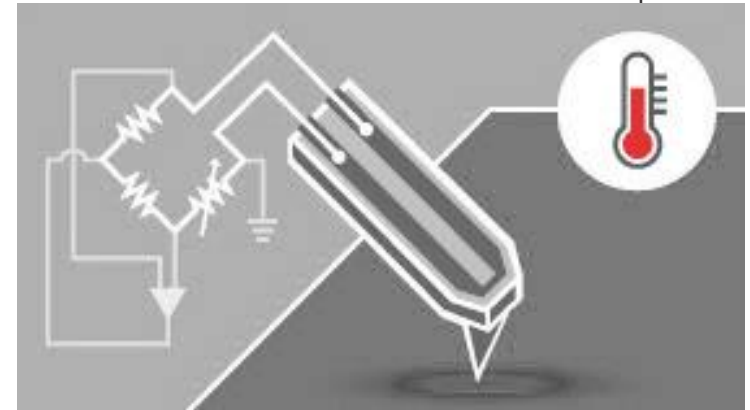


Scanning Thermal Microscopy - SThM

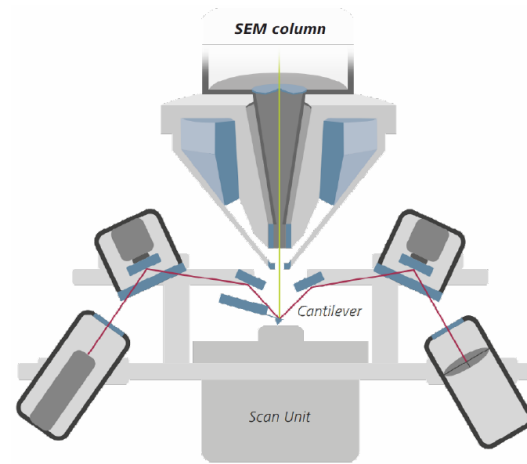
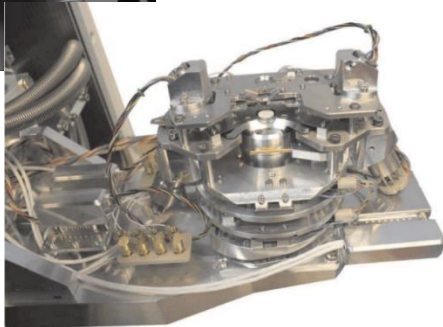
SThM @ MCL: August 2018

Temperatures and thermal conductivities in the sub-100 nm regime

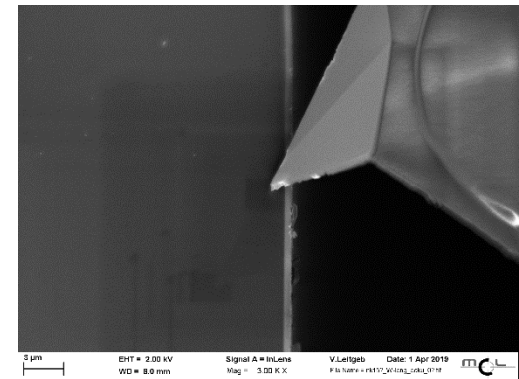
- lateral (in-plane) resolution < 30 nm

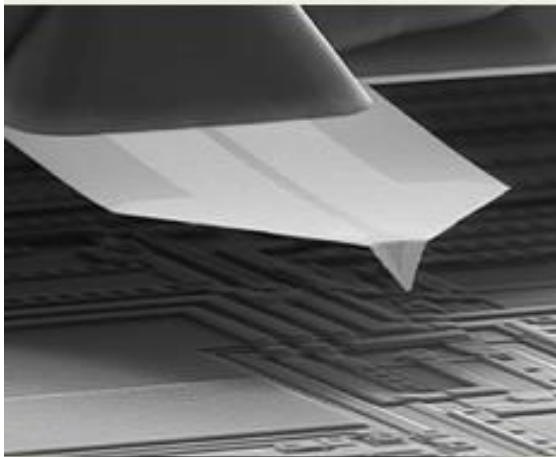


www.parkafm.com



SPM & SEM combination

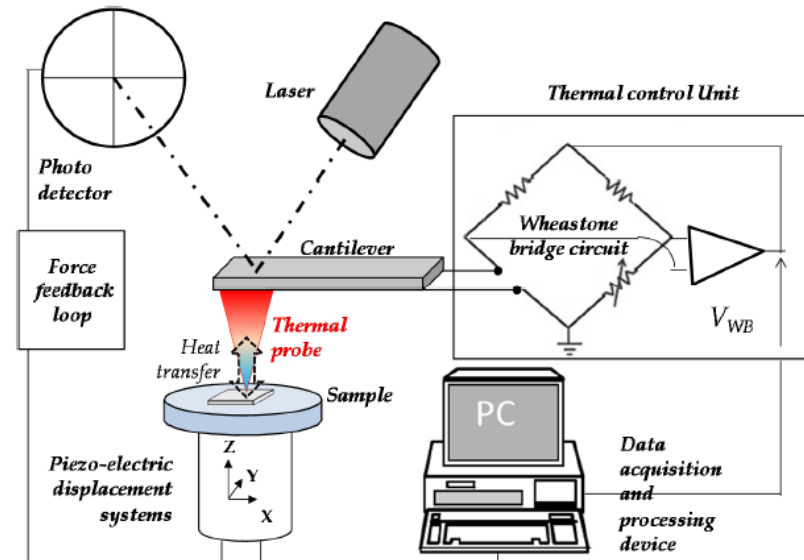




SEM images of nanolithographed SThM probe from Kelvin Nanotechnology

- Cantilever is made of SiN with a thin-film metal wire. Highest resistance of the wire is near the apex of the tip.
- Electrical resistance of thin-film resistor at probe tip correlates with temperature.

- **Resistive probe** incorporated in Wheatstone bridge
- Frequency-modulated measurements
→ 3ω -method (Fiege et al)
Quantitative results



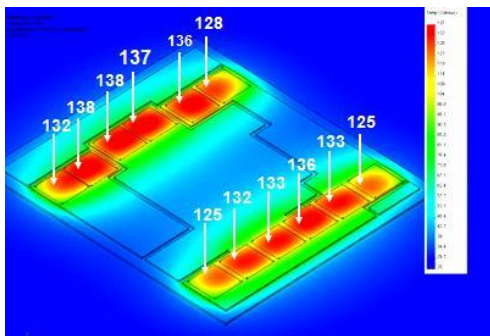
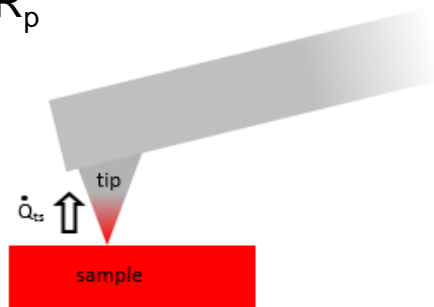
General layout of a SThM AFM-based system.

G. Fiege, A. Altes, B. Heiderhoff, L.J. Balk, Quantitative thermal conductivity measurements with nanometre resolution, J. Phys. D-Applied Phys. 32 (1999)

Gomès S., Assy A., and Chapuis P.-O., "Scanning Thermal Microscopy: a review", 2015, Physica Status Solidi (a) 212

Thermometry

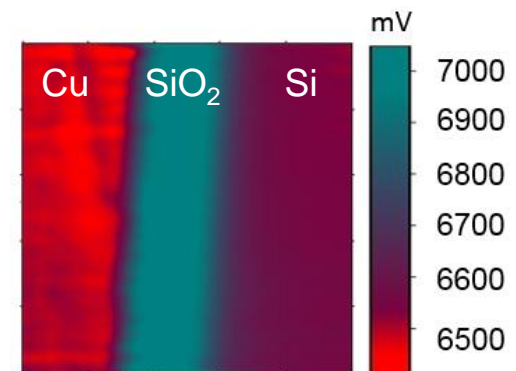
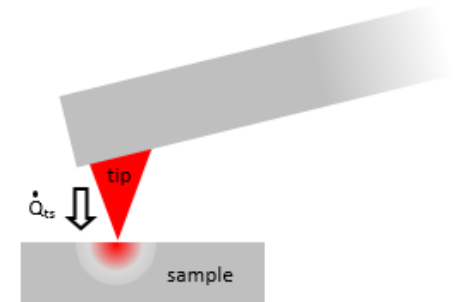
During a scan, heat flows from the **hot sample** to the probe and changes R_p



<http://www.powerguru.org/heat-transfer-in-power-semiconductor-devices/>

Thermal conductivity

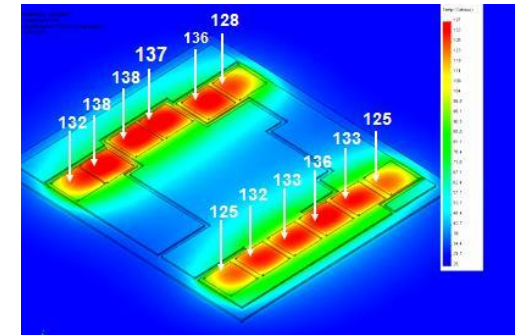
Hot tip apex acts as nanoscale heat source



SThM image, $5 \times 5 \mu\text{m}^2$, of Cu-SiO₂-Si stack system, scan in vacuum.

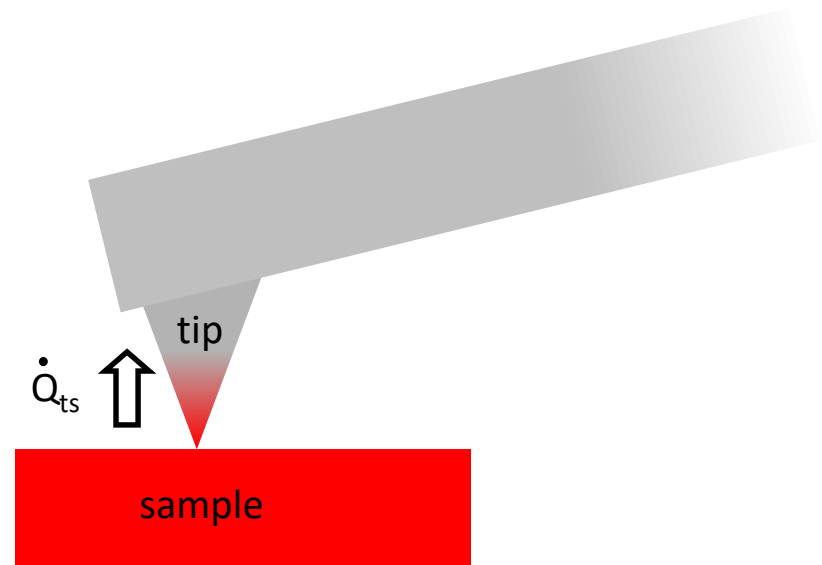
- Passive mode

- In this mode, a very small electrical current is passed through the probe
- Results in minimal Joule self-heating and enables measurement of electrical resistance R_p of the probe



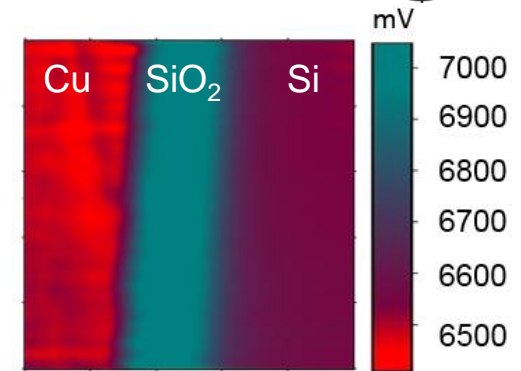
<http://www.powerguru.org/heat-transfer-in-power-semiconductor-devices/>

- During a scan, heat flows from the **hot sample** to the probe and changes R_p



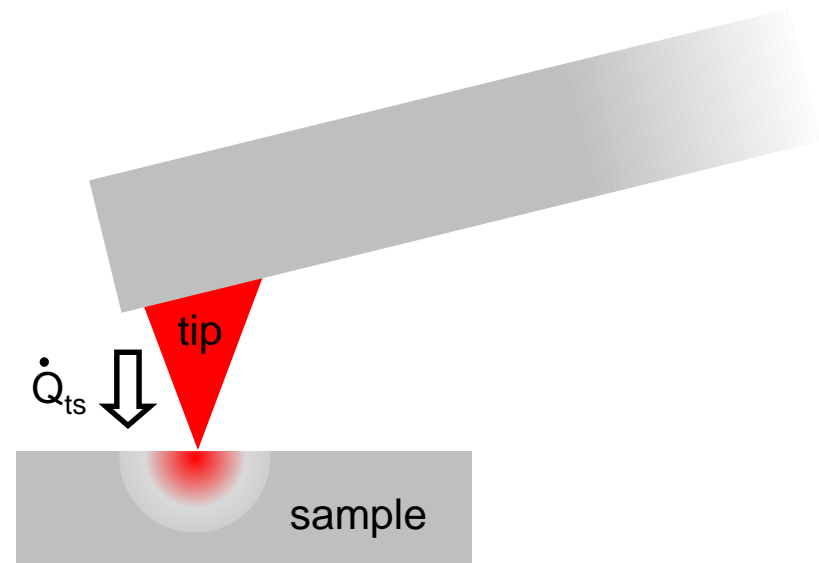
- Active mode

- A larger electrical current is passed through the probe, resulting in a significant Joule heating



SThM image, 5x5 μm^2 , of Cu-SiO₂-Si stack system, scan in vacuum.

- **Hot tip apex** acts as nanoscale heat source
- Thermal conductivity of sample affects SThM probe



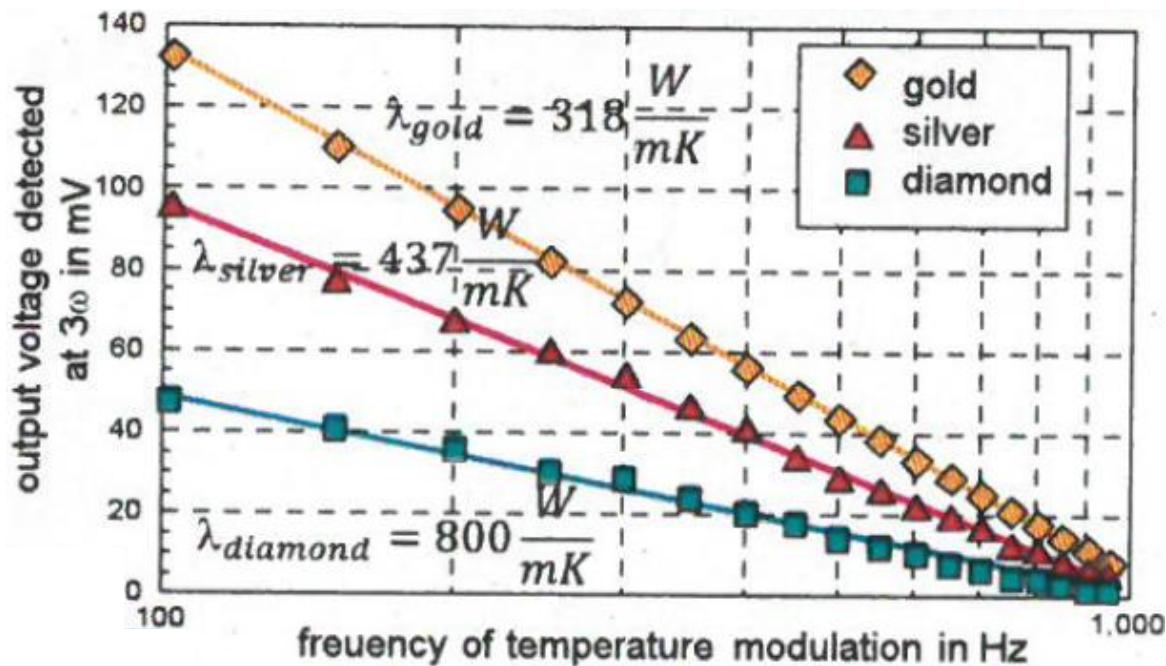
Quantitative measurements of thermal conductivity - 3ω method

Thermal conductivity λ as proportionality factor

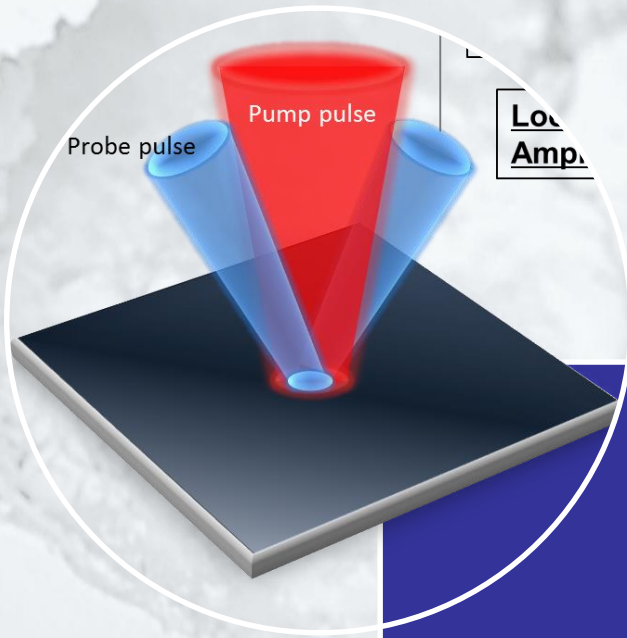
$$\frac{U_{3\omega_1} - U_{3\omega_2}}{\ln(\omega_1) - \ln(\omega_2)} = \frac{1}{4} I_0 \cdot \frac{dR}{dT} \cdot \frac{P}{\pi \cdot \lambda}$$

$\frac{dR}{dT}$... temperature coefficient of the probe

P ... power, from probe to sample



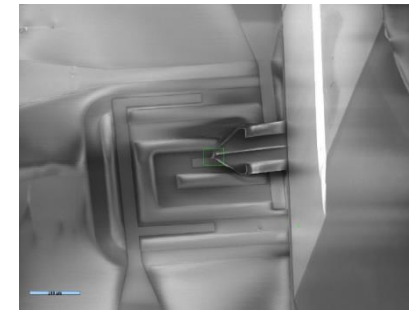
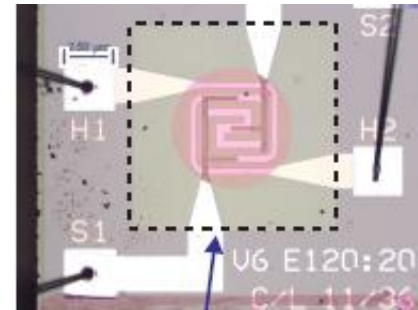
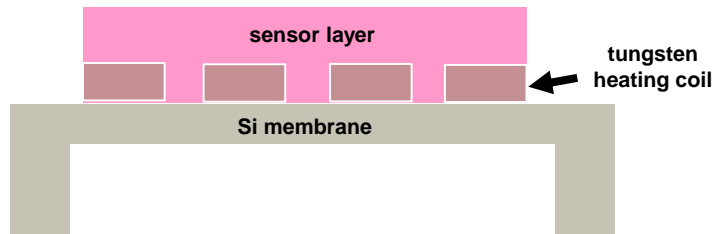
G. Fiege et al., J. Phys. D: Appl. Phys. 32 No 5 (7 March 1999) L13-L17.



W thin film
investigated by
TDTR

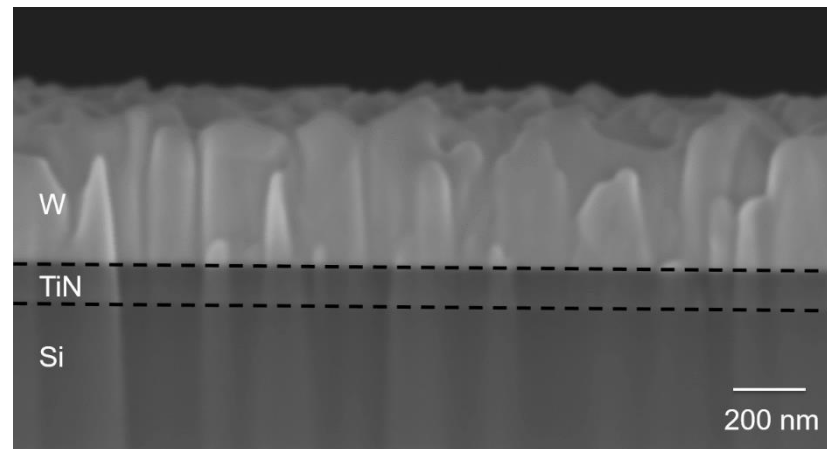
Thermal conductivities of thin films/ interfaces

- Heat management in metal oxide gas sensors



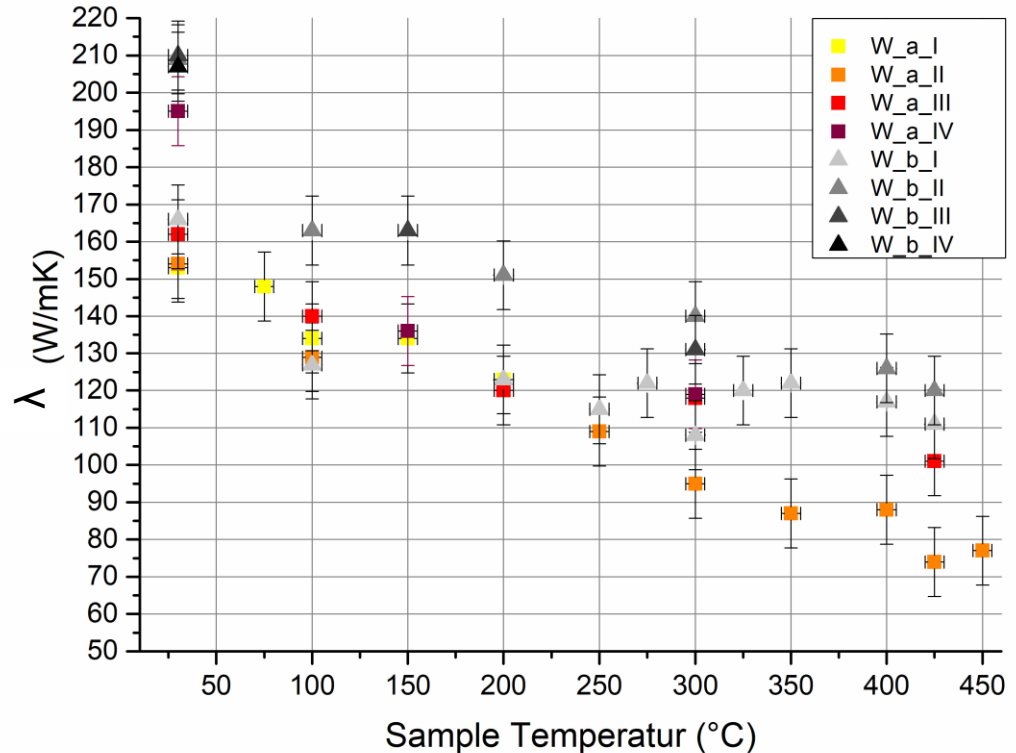
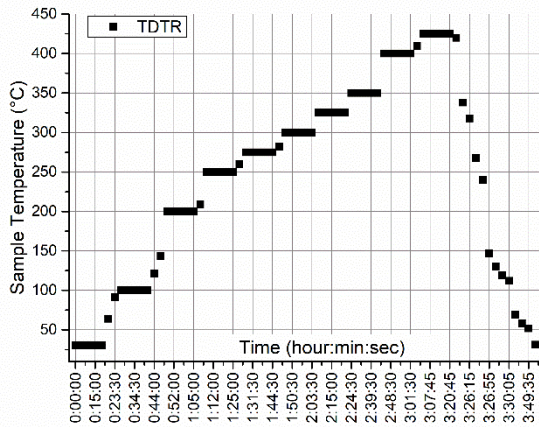
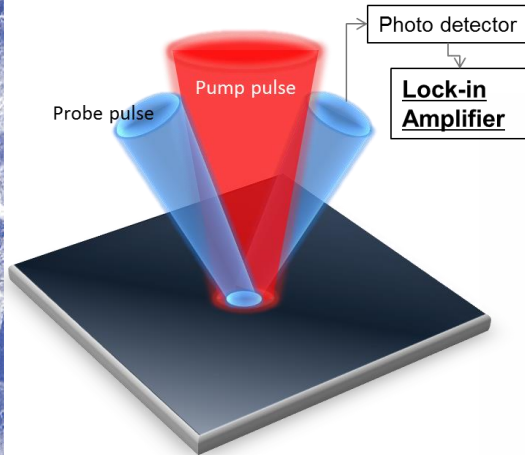
Miniature hotplate: optical image (left) and SEM image (right)

Sideview: Tungsten heating coil for next generation hotplate



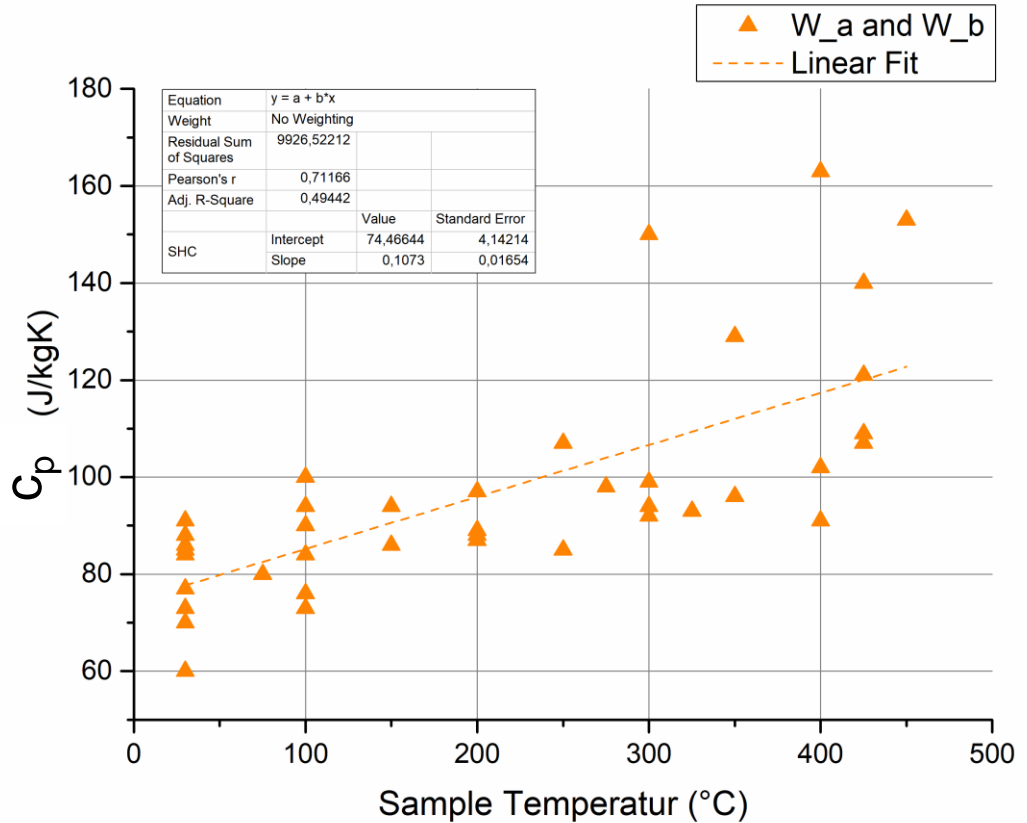
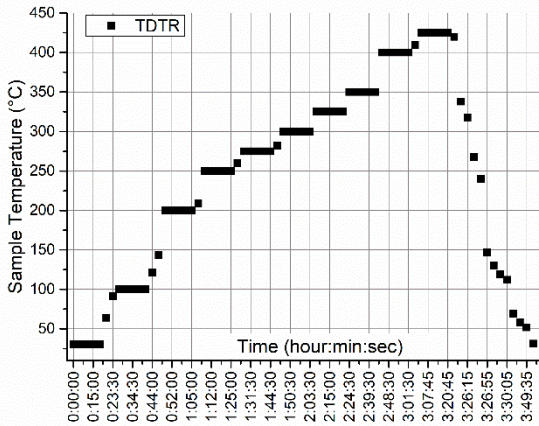
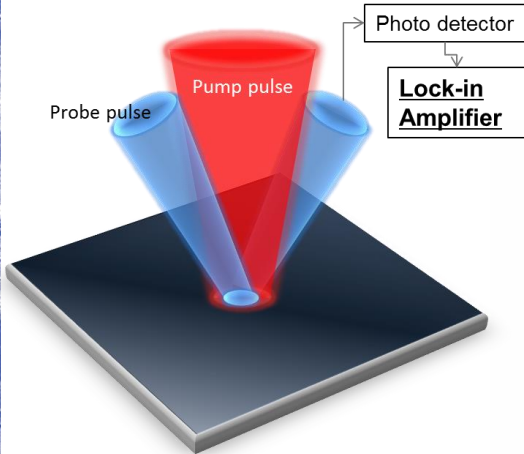
SEM image on cross section of CVD grown 500 nm thick W on a 100 nm TiN layer on (100) Si

Thermal conductivities (λ) of thin films



x 4 heating cycles

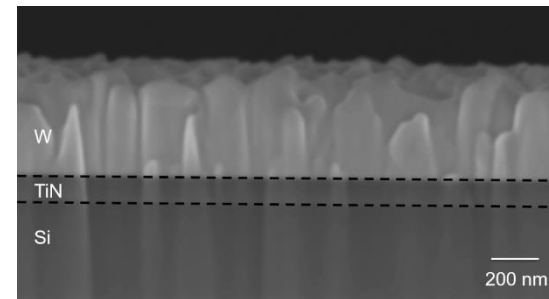
Specific heat capacity (c_p) of thin films



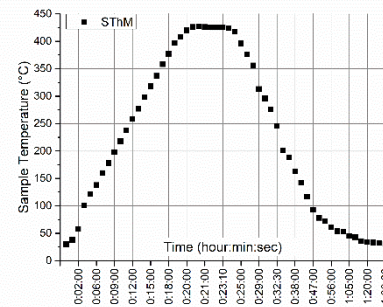
x 4 heating cycles

Thermal conductivities of thin films/ interfaces

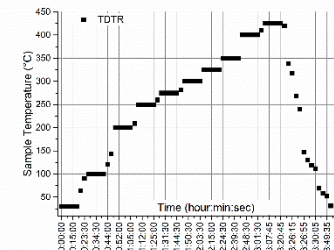
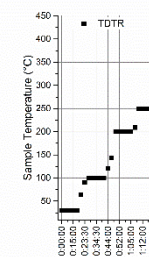
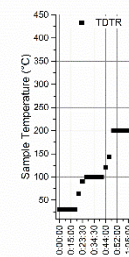
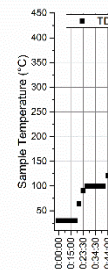
W_{native} = as deposited



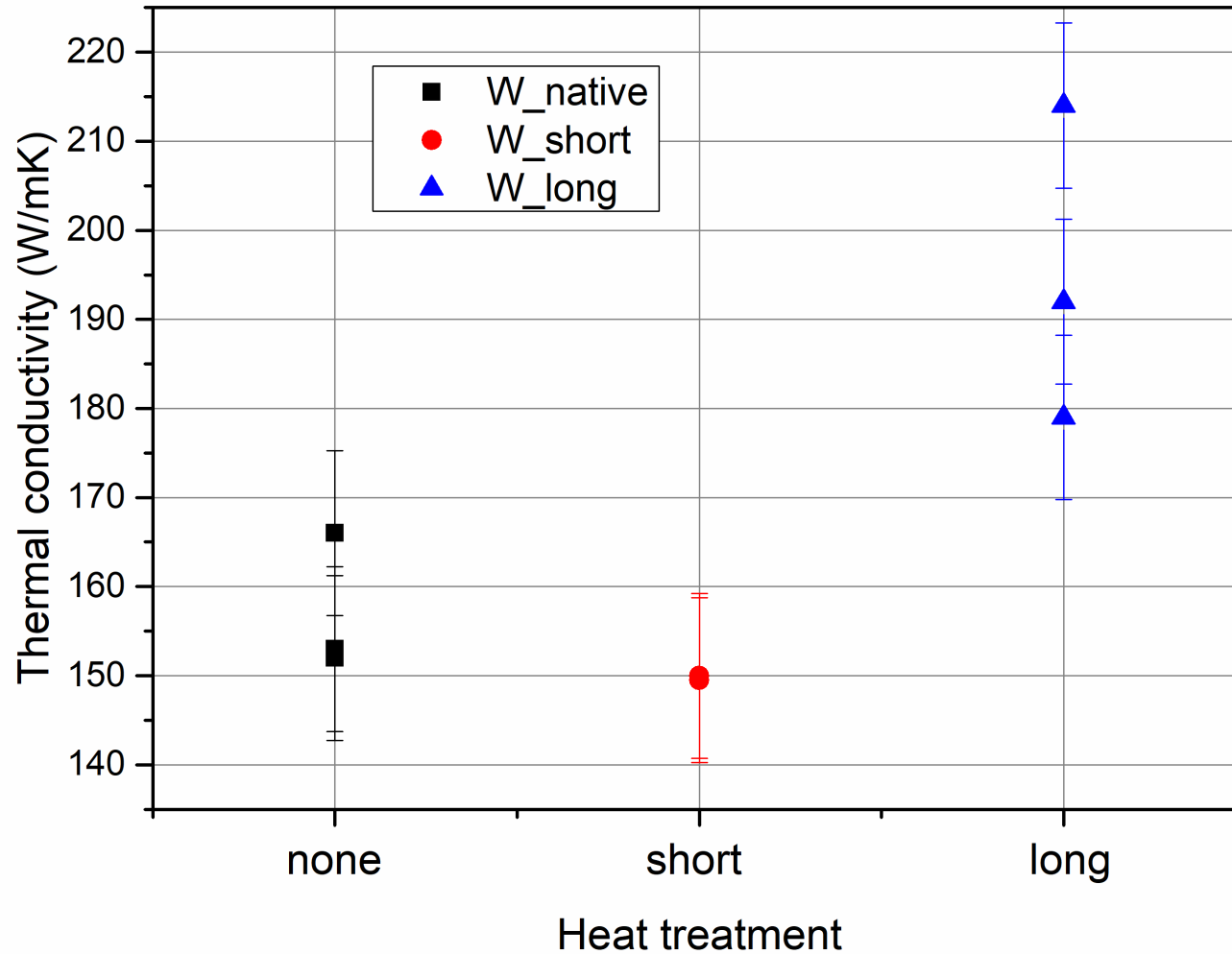
W_{short}: after a short heat cycle
2.5 min @ 425°C

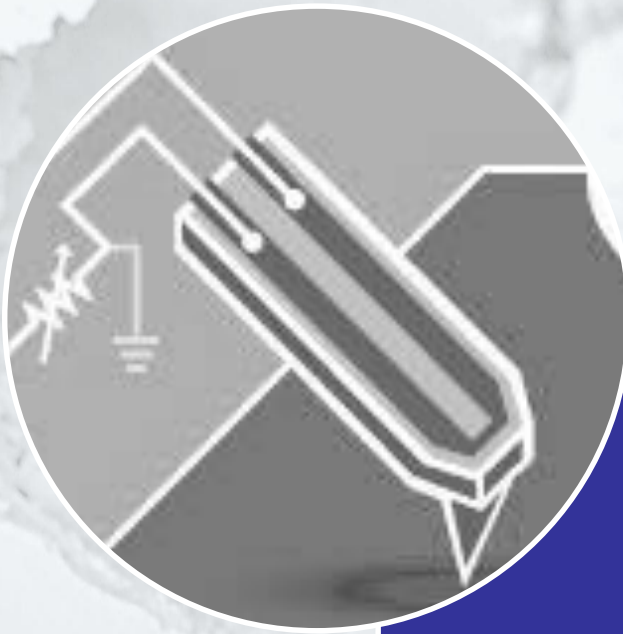


W_{long}: after a long heat cycle
x 4 heating cycles
up to 425°C



Thermal conductivities of thin films/ interfaces

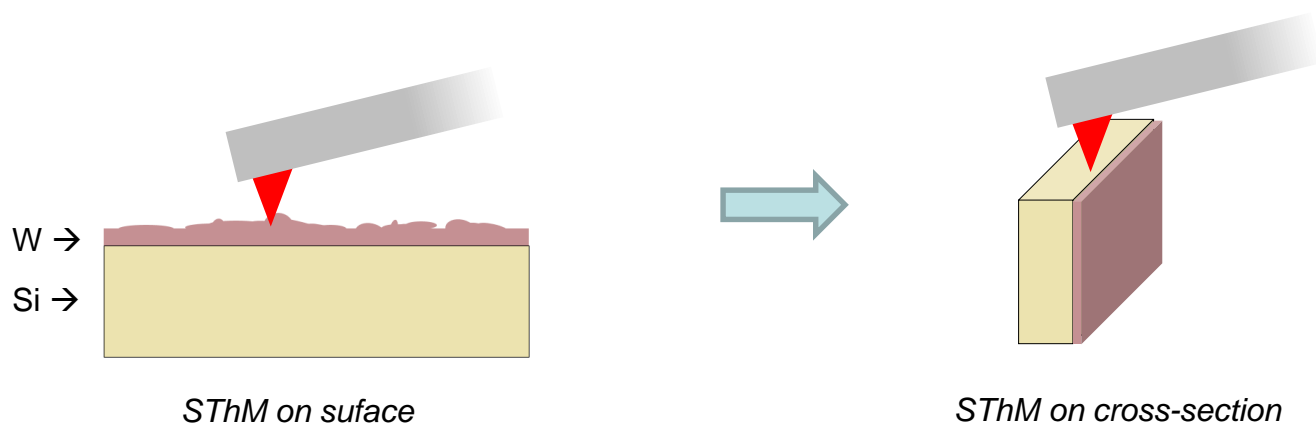




W thin film
investigated by
S_ThM

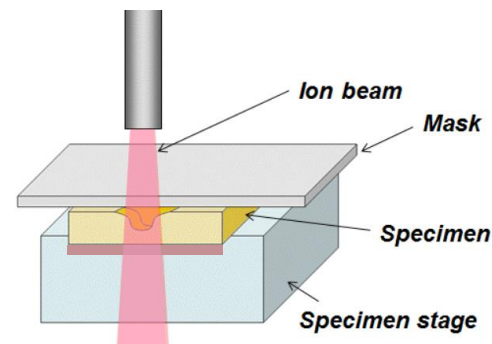
Thermal conductivity of tungsten thin layers

Sample preparation



SThM on cross-section to avoid

- Influence of topography
- Oxidation of surface
- Pollution of surface layer

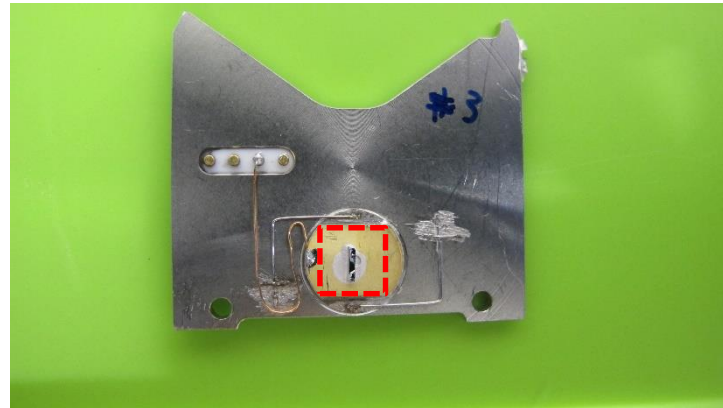


Ion Slicing to get smooth and even surface

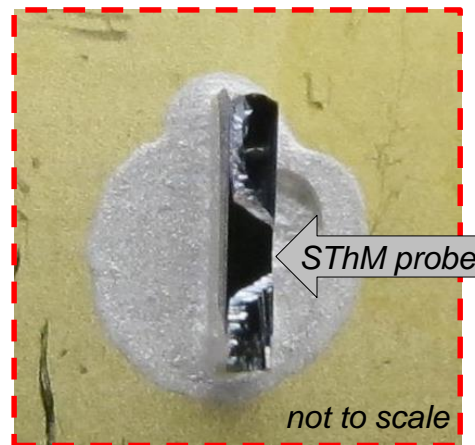
SThM: Tungsten (W) – thin film

Thermal conductivity of tungsten thin layers

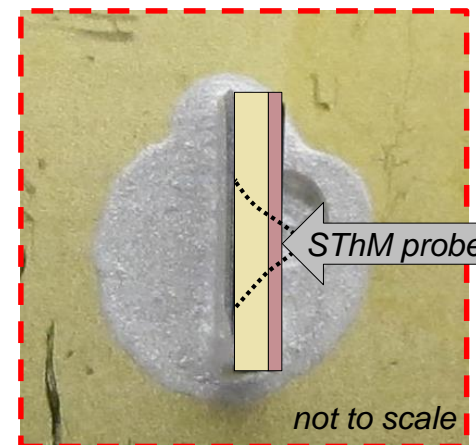
Sample preparation



Tungsten sample on sample holder



not to scale



not to scale

SThM on cross-section of tungsten sample

SThM: Tungsten (W) – thin film

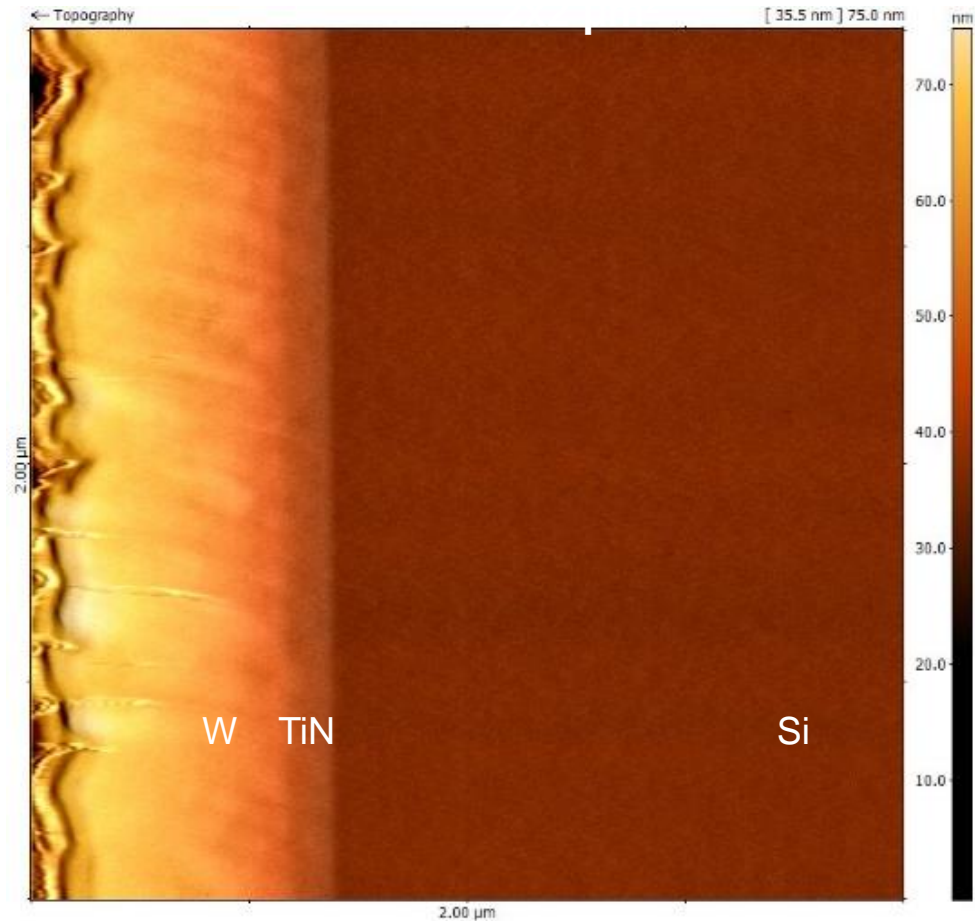
Thermal conductivity of tungsten thin layers

Topography on cross-section:

500 nm W

100 nm TiN

(100) Si substrate



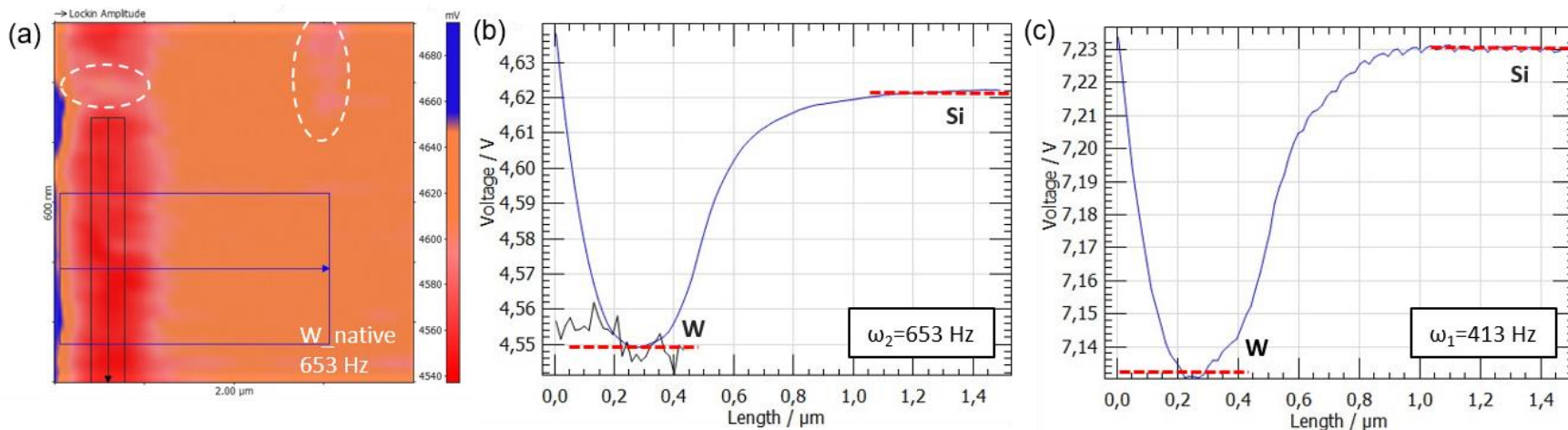
SPM-probe: Arrow-NCR

Thermal conductivity of tungsten thin layers

SThM on cross-section

Heating voltage 350 mV, in vacuum: $1.1 \cdot 10^{-6}$ mbar

W_{native}



(a) SThM image

(b) & (c) areas utilized for data evaluation of W and Si at different frequencies

Thermal conductivity, literature values for bulk:

Si ... 149 W/mK

W ... 173 W/mK

TiN ... 19 W/mK

But: TiN layer not visible

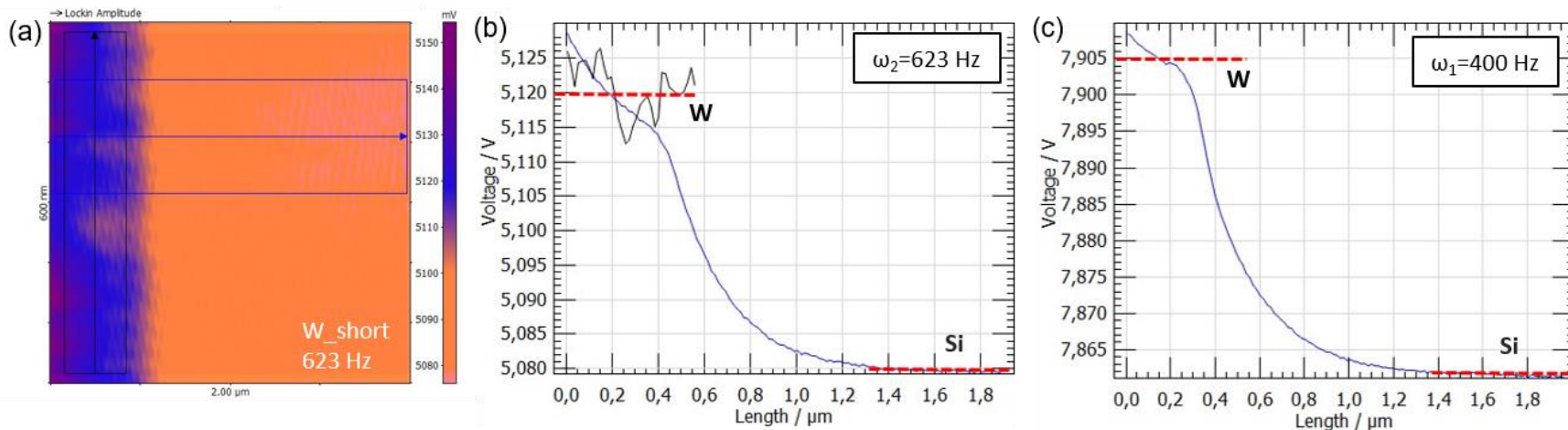
$$\lambda_{W_{\text{native}}} = 154.2 \pm 4 \text{ W/mK}$$

Thermal conductivity of tungsten thin layers

SThM on cross-section

Heating voltage 350 mV, in vacuum: $1.1 \cdot 10^{-6}$ mbar

W_short



(a) SThM image

(b) & (c) areas utilized for data evaluation of W and Si at different frequencies

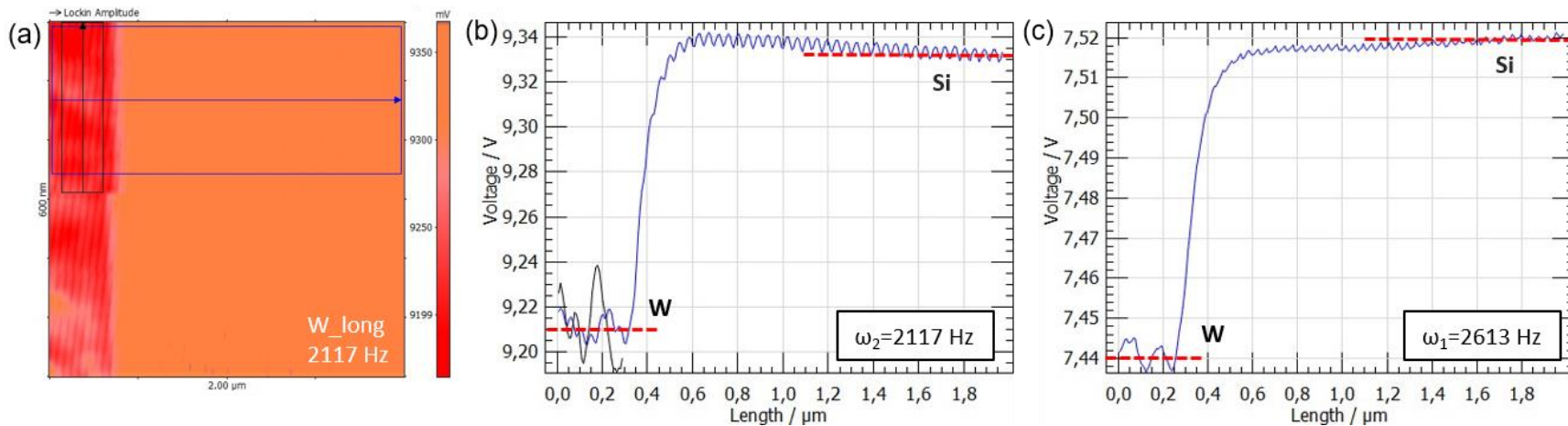
$$\lambda_{W_short} = 151.8 \pm 4 \text{ W/mK}$$

Thermal conductivity of tungsten thin layers

SThM on cross-section

Heating voltage 350 mV, in vacuum: $1.1 \cdot 10^{-6}$ mbar

W_long



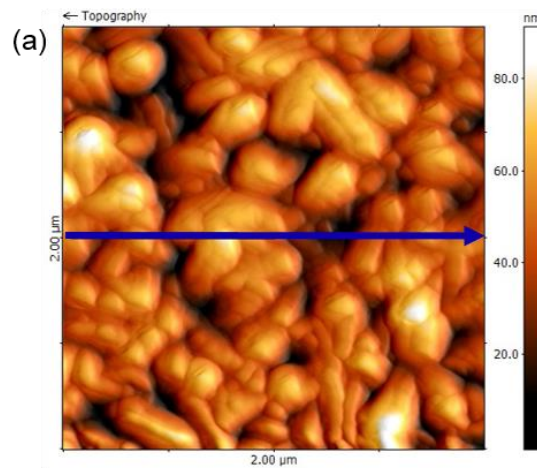
(a) SThM image

(b) & (c) areas utilized for data evaluation of W and Si at different frequencies

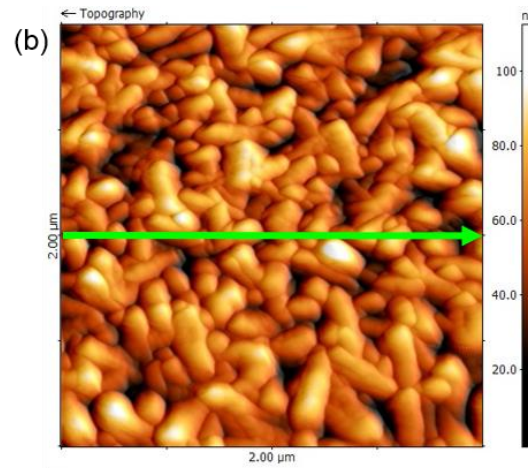
$$\lambda_{W_long} = 155.6 \pm 4 \text{ W/mK}$$

SPM topview – Grain sizes

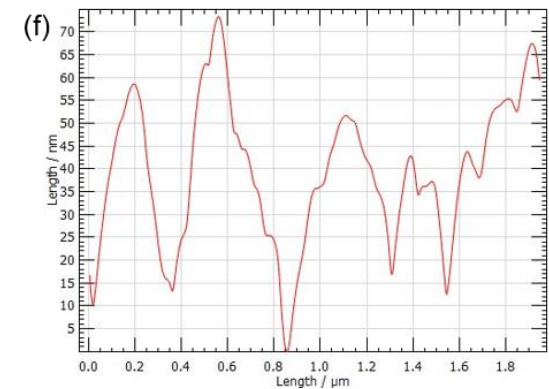
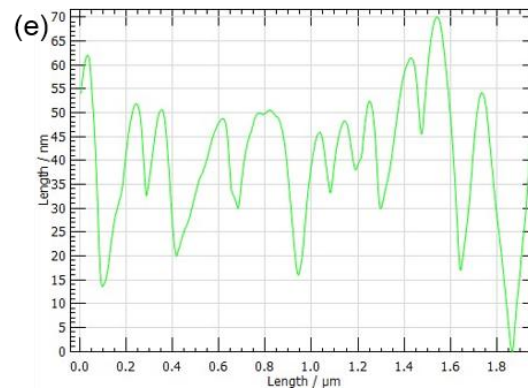
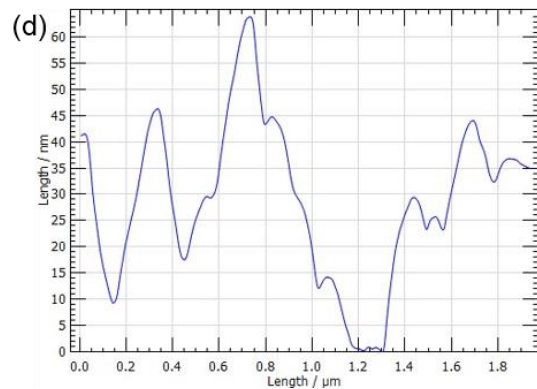
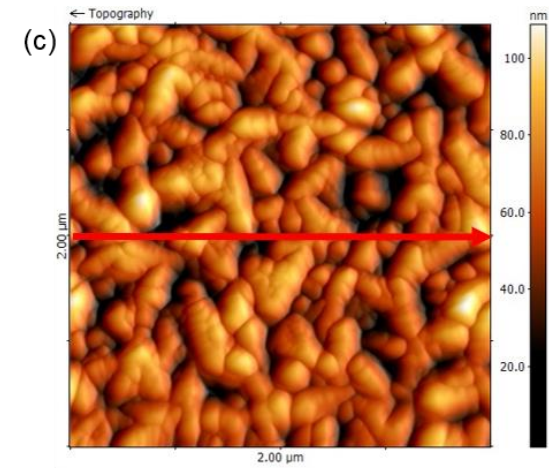
W_native



W_short

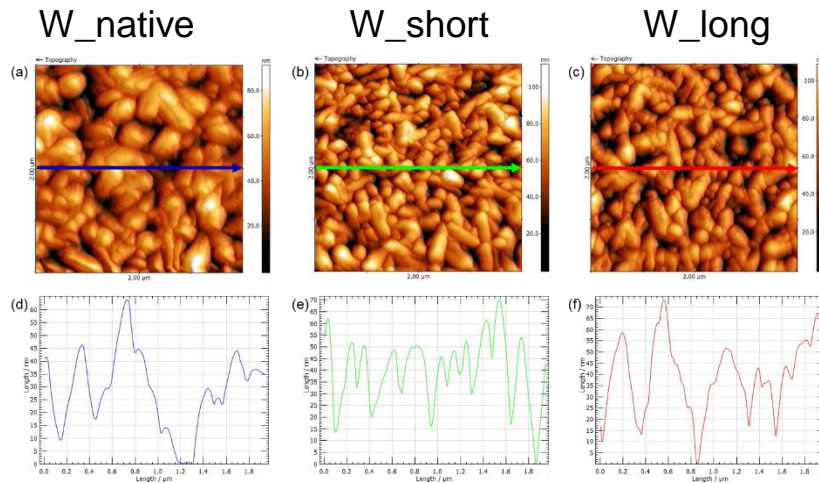


W_long



Tungsten (W) – thin film – at 30°C

SPM topview – Grain sizes



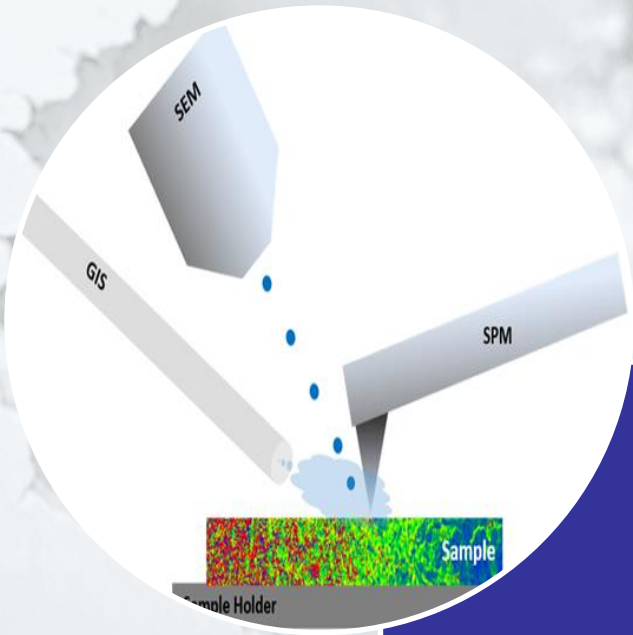
D. Choi, *The electron scattering at grain boundaries in tungsten films*, *Microelectron. Eng.* 122 (2014) 5–8. doi:10.1016/j.mee.2014.03.012:

Grain boundary scattering → increase of electrical resistivity due to

- surface scattering (Fuchs-Sonderheimer model $\Delta \sigma_{FS}$) and
- grain boundary scattering (Mayadas-Shatzkes model $\Delta \sigma_{MS}$)

→ Calculation of λ_{calc} out of grain size

Sample	Average grain size [nm]	λ_{calc} [W/mK]	λ_{TDTR} [W/mK]	λ_{STHM} [W/mK]
W _{native}	136 ± 51	152 ⁺⁵ ₋₁₁	157 ± 8	153.6 ± 4
W _{short}	105 ± 50	146 ⁺⁸ ₋₁₉	149.8 ± 0.5	151.8 ± 4
W _{long}	177 ± 84	156 ⁺⁵ ₋₁₃	195 ± 18	155.6 ± 4

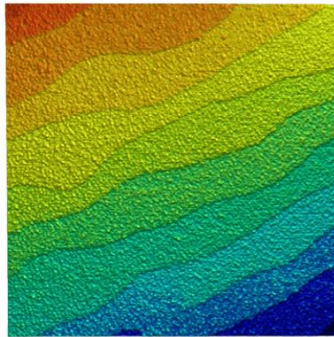


Outlook

Outlook – SThM at MCL

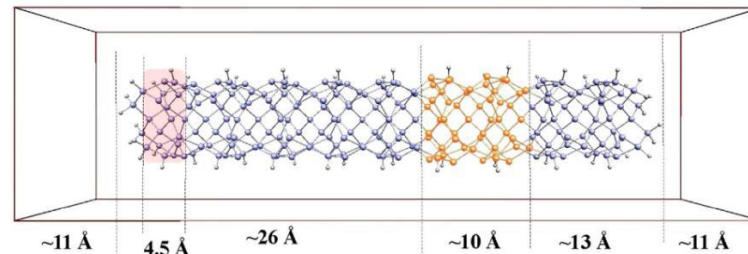
- **SThM & TDTR characterization of multilayer systems and interfaces**

Record topography and thermal properties simultaneously with nm-resolution
 → Create input to model thermal transport via ab-initio Molecular Dynamics



Topography showing steps of strontium titanate; image size 1.1µm

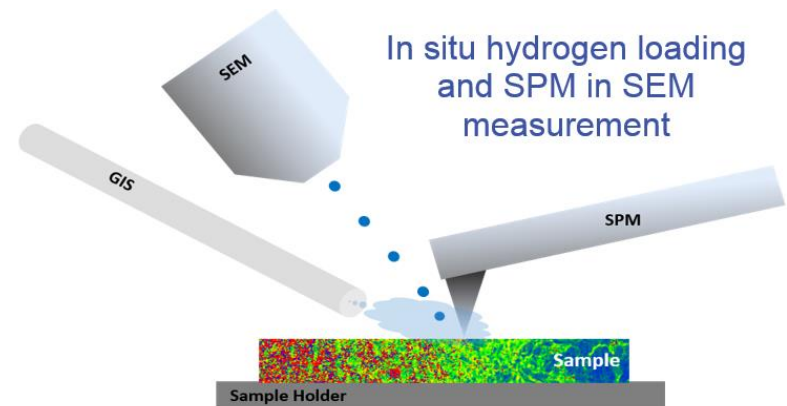
<https://www.nanosurf.com/en/application/547-topography-strontium-titanate-flexafm>



Simulation cell representing layered system. Gibbons, Bebek, Kang, Stanley, and Estreicher, J. of Applied Physics 118 (2015).

- **SThM - SEM characterization**

- Study thermal transport mechanism, phonons /electrons?
- In situ loading of hydrogen etc.



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Any questions?

Thank you for your attention!

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