

Scanning Thermal Microscopy of functional layers used in halide perovskite devices

Ralf Heiderhoff

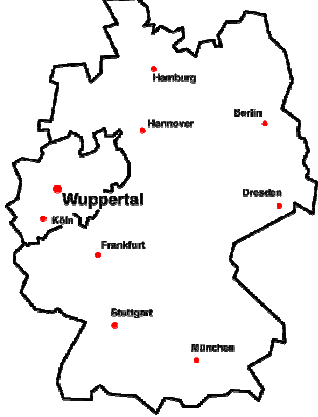
CM@S School of Electrical, Information and Media Engineering
Institute of Electronic Devices

Summer School Khiva | May 2023


BERGISCHE
UNIVERSITÄT
WUPPERTAL

Introduction

University of Wuppertal




Germany



suspension
railway

- since 1863 School of Engineering
- 1972 Foundation of BU Wuppertal
- 8 Faculties
- 24,000 Students



CM@S Scanning Thermal Microscopy
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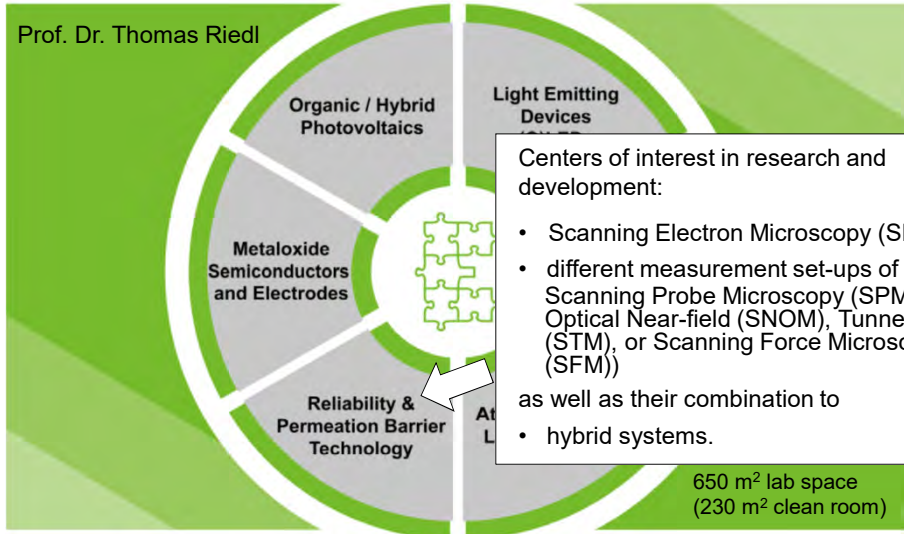
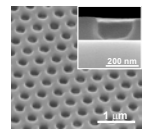
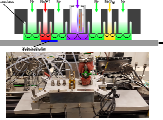
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WUPPERTAL

Introduction



Prof. Dr. Thomas Riedl



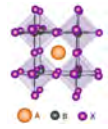
Centers of interest in research and development:

- Scanning Electron Microscopy (SEM),
- different measurement set-ups of Scanning Probe Microscopy (SPM) (like Optical Near-field (SNOM), Tunneling (STM), or Scanning Force Microscopy (SFM))

as well as their combination to

- hybrid systems.

650 m² lab space
(230 m² clean room)

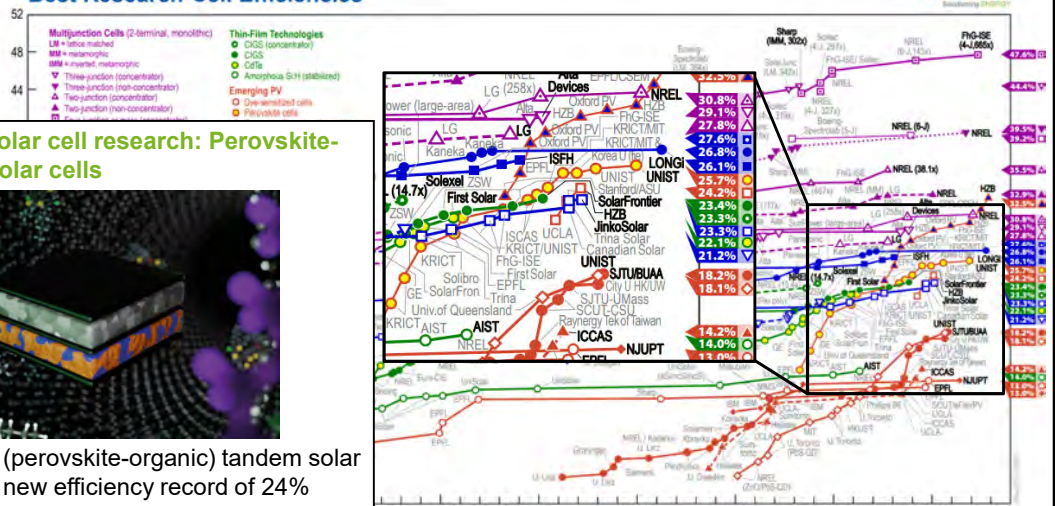


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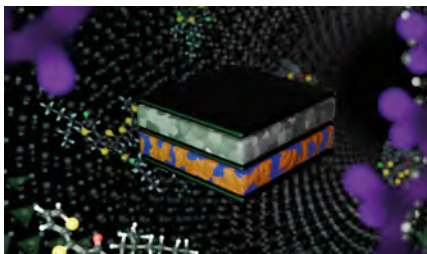


Motivation and Purpose

Best Research-Cell Efficiencies



World record in solar cell research: Perovskite-organic tandem solar cells



- next-generation (perovskite-organic) tandem solar cells reaching a new efficiency record of 24%
- published in Nature 604, 280 (2022)



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Motivation and Purpose

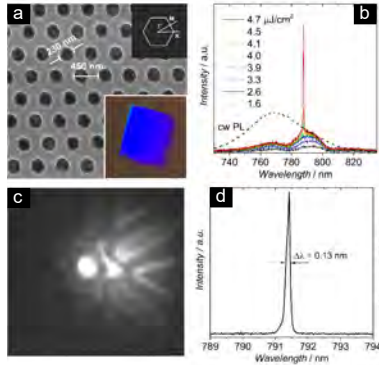
ADVANCED SCIENCE NEWS
www.advancescience.com

MAPbI₃



Photonic Nanostructures Patterned by Thermal Nanoimprint Directly into Organo-Metal Halide Perovskites

Neda Pourdavoud, Si Wang, André Mayer, Ting Hu, Yiwang Chen, André Marianovich, Wolfgang Kowalsky, Ralf Heiderhoff, Hella-Christin Scheer, and Thomas Riedl*



Pourdavoud et al. Adv. Mater. 29, 1605003 (2017)

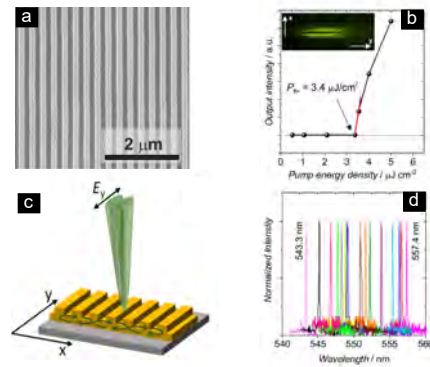
COMMUNICATION
Perovskite Lasers

MAPbBr₃

ADVANCED TECHNOLOGIES
www.advantech.de

Distributed Feedback Lasers Based on MAPbBr₃

Neda Pourdavoud, André Mayer, Maximilian Buchmüller, Kai Brinkmann, Tobias Häger, Ting Hu, Ralf Heiderhoff, Ivan Shutsko, Patrick Görm, Yiwang Chen, Hella-Christin Scheer, and Thomas Riedl*



Pourdavoud et al. Adv. Mater. Technol. 3, 1700253 (2018)



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Motivation and Purpose

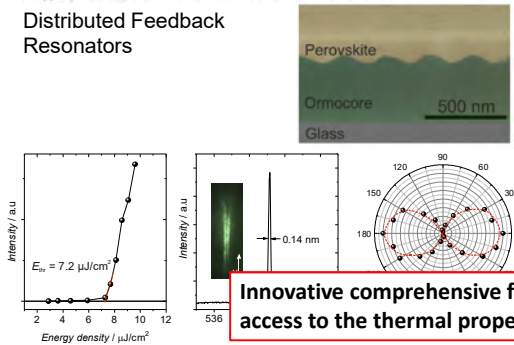
COMMUNICATION
Perovskite Lasers

ADVANCED MATERIALS
www.advmat.de

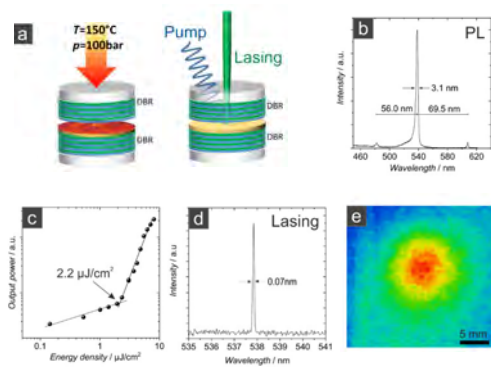
Room-Temperature Stimulated Emission and Lasing in Recrystallized Cesium Lead Bromide Perovskite Thin Films

Neda Pourdavoud, Tobias Haeger, Andre Mayer, Piotr Jacek Cegielski, Anna Lena Giesecke, Ralf Heiderhoff, Selina Olthof, Stefan Zoelffer, Ivan Shutsko, Andreas Henkel, David Becker-Koch, Markus Stein, Marko Cehovski, Ouacef Charfi, Hans-Hermann Johannes, Detlef Rogalla, Max Christian Lemme, Martin Koch, Yana Vaynzof, Klaus Meerholz, Wolfgang Kowalsky, Hella-Christin Scheer, Patrick Görm, and Thomas Riedl*

Distributed Feedback Resonators



Vertical Cavity Surface Emitting Laser



Innovative comprehensive failure and reliability investigations are mandatory to get access to the thermal properties of these devices and systems as a whole!

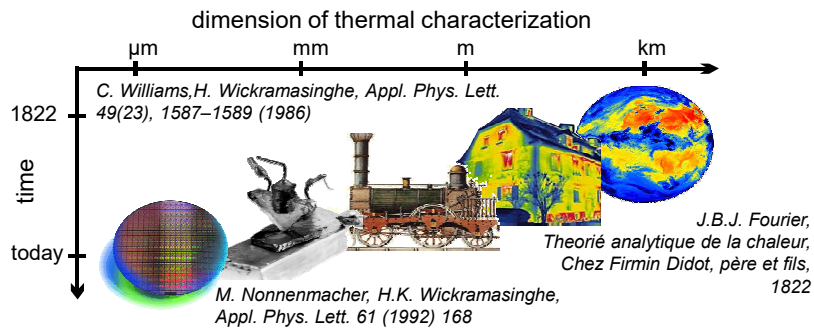


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Motivation



Fourier law

$$\vec{q} = -\vec{\lambda} \cdot \text{grad } T(\vec{r})$$

general equation of heat conduction

$$\rho c \frac{\partial T(\vec{r})}{\partial t} = \frac{\partial}{\partial x} \left(\lambda_x \frac{\partial T(\vec{r})}{\partial x} \right) + \frac{\partial}{\partial y} \left(\lambda_y \frac{\partial T(\vec{r})}{\partial y} \right) + \frac{\partial}{\partial z} \left(\lambda_z \frac{\partial T(\vec{r})}{\partial z} \right) + \dot{q}_E$$

ρ : specific density \dot{q}_E : amount of heat generated per unit time and unit volume

A. Freeman, *Theory of heat*, Cambridge University Press, London, 1878



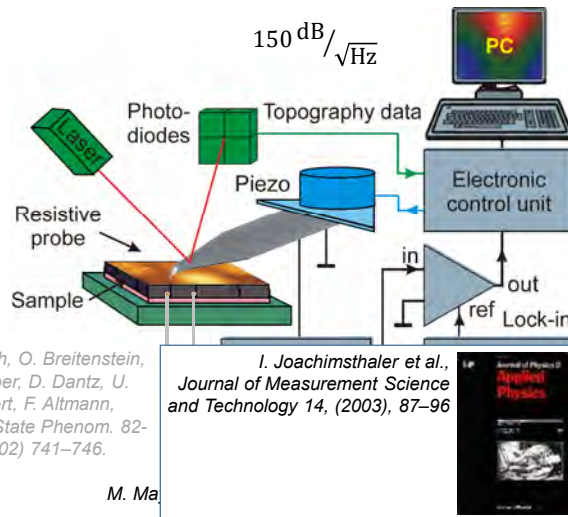
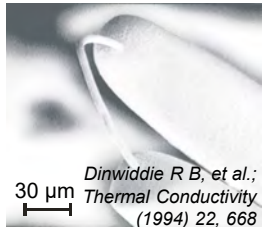
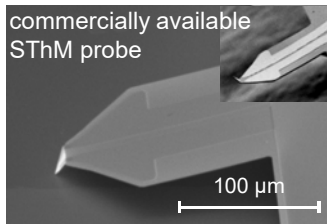
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Set-up of Scanning Thermal Microscope

P. Tovee et al.; *Journal of Applied Physics*, (2012) 112, 114317



S. Huth, O. Breitenstein, A. Huber, D. Dantz, U. Lambert, F. Altmann, *Solid State Phenom.* 82-84 (2002) 741-746.

I. Joachimsthaler et al., *Journal of Measurement Science and Technology* 14, (2003), 87-96

M. Ma



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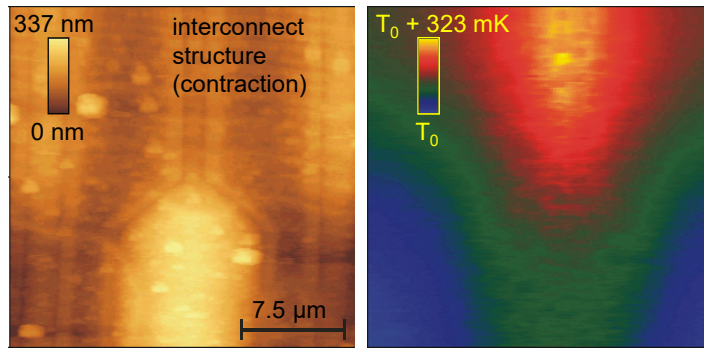
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Measurements of temperature distributions

Voltage source:
local heat
dissipation in
dependence on
the heating power
density

G.B.M. Fiege et al.
Microelectron. Reliab.
38 (1998) 957–961.



Topography

Temperature

$$5 \frac{mK}{\sqrt{Hz}}: \text{thermal noise}$$

$$\frac{\Delta x}{\Delta T}: 1/\text{temperature gradient}$$

30 nm: effect. contact area in air

achievable spatial resolution:

$$\Delta x = 5 \frac{mK}{\sqrt{\Delta f}} \cdot \sqrt{\Delta x} + 30 \text{ nm}$$

10 nm: in ultra high vacuum

W. Jeong et al., *Sci.Rep.4* (2014) 4975

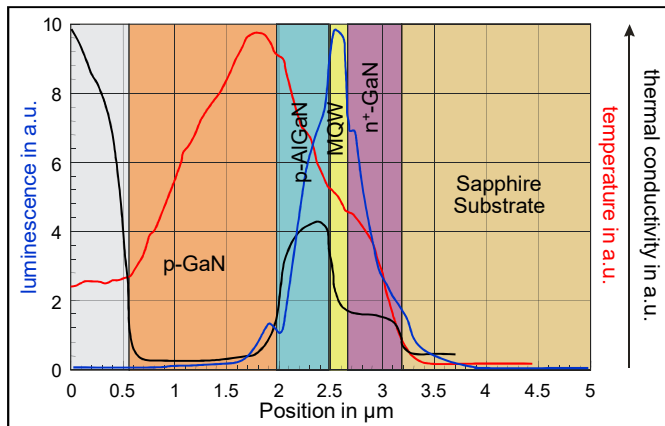


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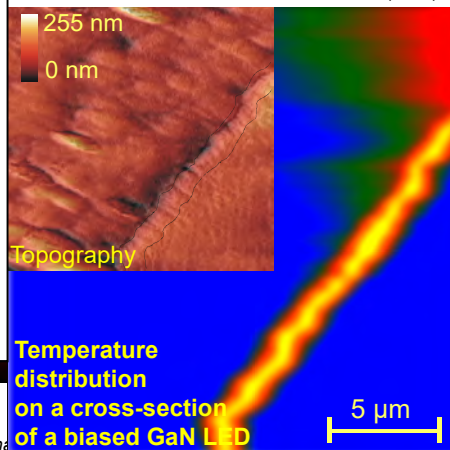
Reliability investigations by means of SThM



Topography

M. Palaniappan et al., *Proc. 25th Int. Symp. Test. Fail. Anal.*

R. Heiderhoff et al., *Microelectron. Reliab.* 40 (2000) 1383–1388



Temperature distribution on a cross-section of a biased GaN LED



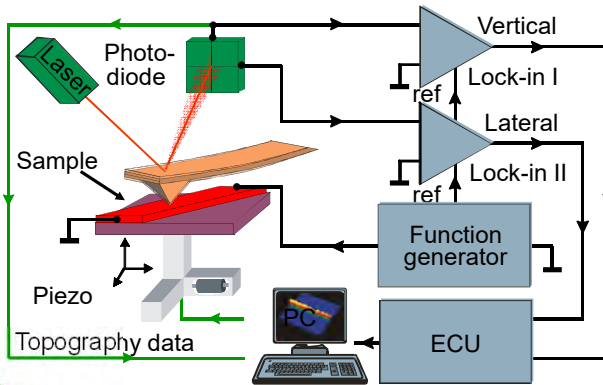
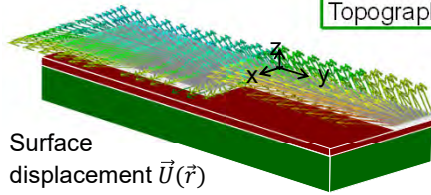
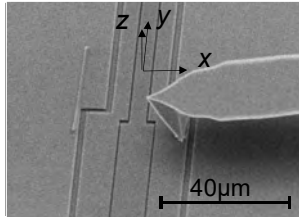
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Scanning Joule Expansion Microscopy

Vertical und lateral surface displacement detection on an interconnect structure



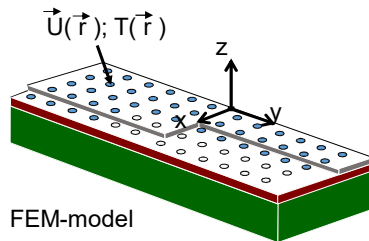
J. Varesi and A. Majumdar, *Appl. Phys. Lett.* **72** (1998), 37-39

Fakhri M. et al., *Microelectronics Reliability* **50** (2010), 1459-1463

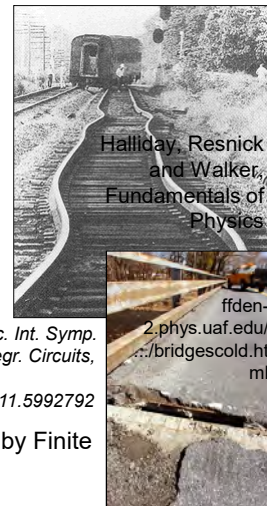


Thermally induced mechanical stress analysis

Electric current → Thermally induced mechanical stress



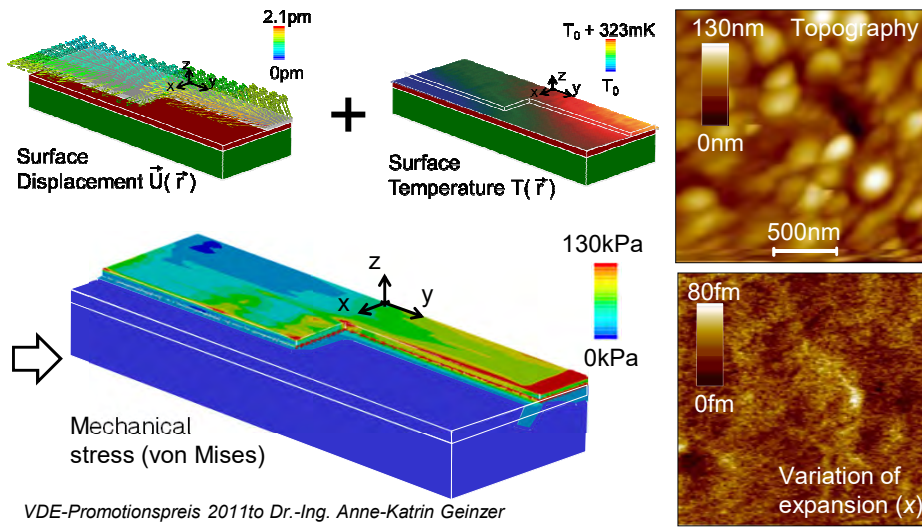
Solution of thermal and thermo-elastic differential equations by Finite Element Method and using experimental data as boundary conditions



M. Fakhri et al., *Proc. Int. Symp. Phys. Fail. Anal. Integr. Circuits, IPFA.* (2011).
doi:10.1109/IPFA.2011.5992792



Thermally induced mechanical stress analysis

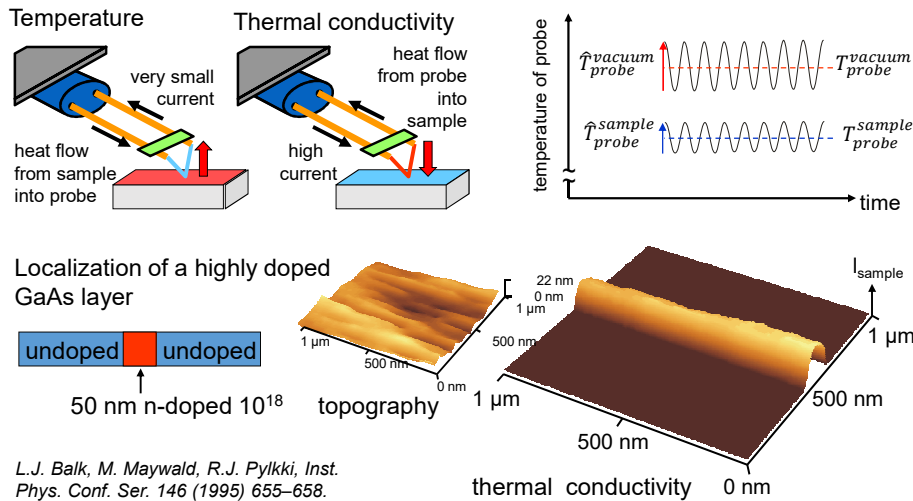


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Heat flows in Scanning Thermal Microscopy



L.J. Balk, M. Maywald, R.J. Pylkki, *Inst. Phys. Conf. Ser. 146 (1995) 655-658.*



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Motivation and Purpose



thin film batteries



flexible solar cells



bendable thermoelectrics

Performances/efficiencies are significantly influenced by thermal properties (reliability, life time, thermoelectric figure of merit, etc.)

➔ Investigations of cross-plane and in-plane thermal transports in dependence on thin film thicknesses



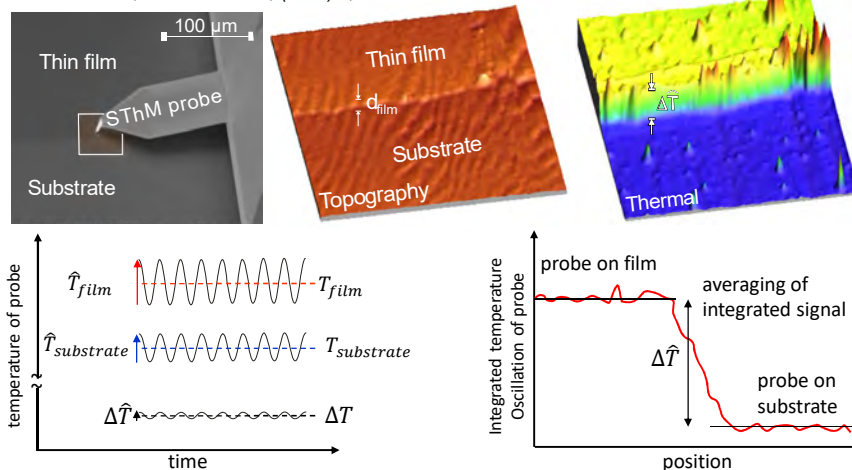
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Scanning Thermal Microscopy on thin films

A. Makris et al., RSC Advances, (2016) 6, 94193–94199.

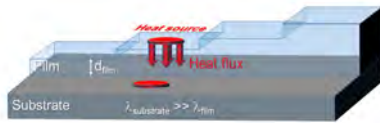


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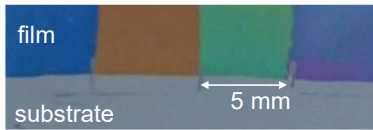
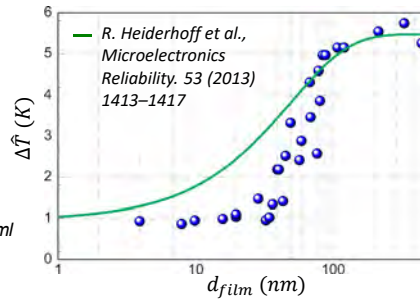


Out-of-plane heat transport



*Si substrate λ_{Si} : $150 \text{ Wm}^{-1}\text{K}^{-1}$
 Al_2O_3 films $\lambda_{\text{Al}_2\text{O}_3}$: $0.8 \text{ Wm}^{-1}\text{K}^{-1}$
 ** $\Lambda_{\text{Al}_2\text{O}_3}$: $0.47\text{--}3.5 \text{ nm}$

*DETHERM, (2017) <http://www.dechema.de/Detherm.html>
 **N. Oka et al., *Thin Solid Films*, (2010) 518, 3119–3121



optical image of fabricated step-like film matrix

$$*** \Delta\hat{T}(d_{film} \ll w) = \frac{\hat{p}}{w \cdot \lambda_{film}} \cdot d_{film}, \quad w = 2 \cdot r_{contact}$$

$$\Delta\hat{T}(d_{film}) = \frac{\hat{p}}{\lambda_{film}} \cdot (1 - e^{-\frac{d_{film}}{2 \cdot r_{contact}}}) + \hat{T}_{contact}$$

⇒ enhanced out-of-plane heat dissipation

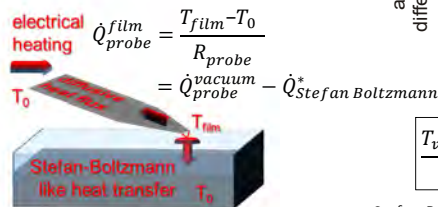
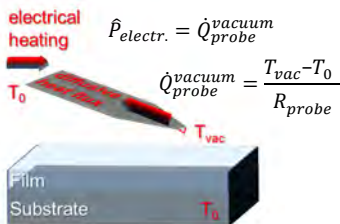
***S.-M. Lee and D. G. Cahill, *Journal of Applied Physics*, (1997) 81, 2590–2595



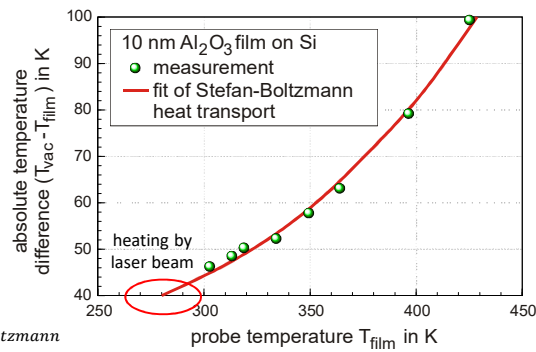
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Stefan-Boltzmann-like heat transport



*A. Majumdar, *Journal of Heat Transfer*, (1993) 115, 7–16



$$\frac{T_{vac} - T_{film}}{R_{probe}} = e^{-\frac{d_{film}}{\Lambda_{film}}} \sigma_{Stefan\ Boltzmann} A_{contact} (T_{film}^4 - T_0^4)$$

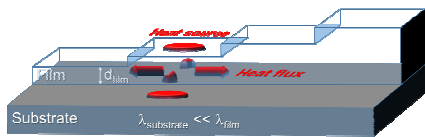
σ : Stefan-Boltzmann constant for phonons
 Λ_{film} : phonon mean-free path
 $A_{contact}^{Stefan\ Boltzmann}$: effective contact area for ballistic heat transport



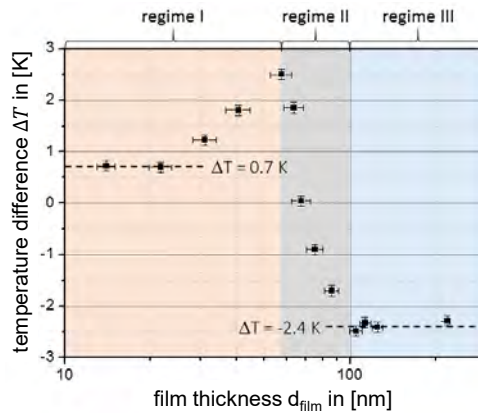
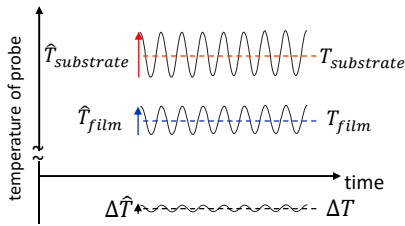
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In-plane heat transport



- *glass substrate $\lambda_{\text{glass}} : 0.8-1.4 \text{ Wm}^{-1}\text{K}^{-1}$
- **TiO₂ films $\lambda_{\text{TiO}_2} : 2-8 \text{ Wm}^{-1}\text{K}^{-1}$
- *** $\Lambda_{\text{TiO}_2} : 0.4 -1 \text{ nm}$

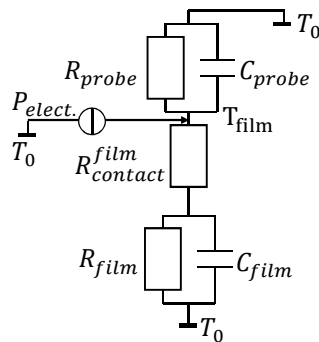


*DETHERM, (2017) <http://www.dechema.de/Detherm.html>.
 **D.J. Kim et al., *Int. J. of Thermophysics*, (2004) 25, 281–289.
 ***C. B. Carter, M. G. Norton, in *Ceramic Materials*, (2013), 641–657

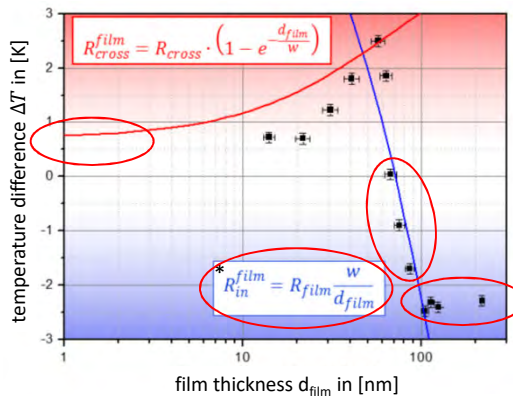


In-plane heat transport

Heiderhoff R. et al., *Microelectronics Reliability* (2017),
 doi: 10.1016/j.microrel.2017.06.064



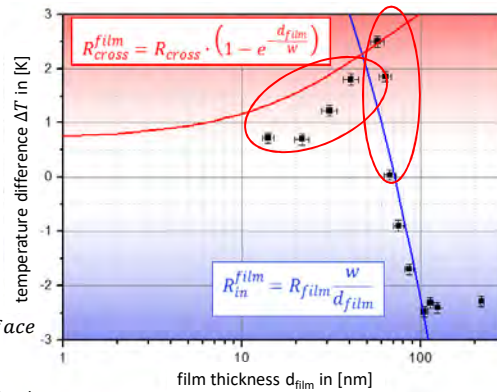
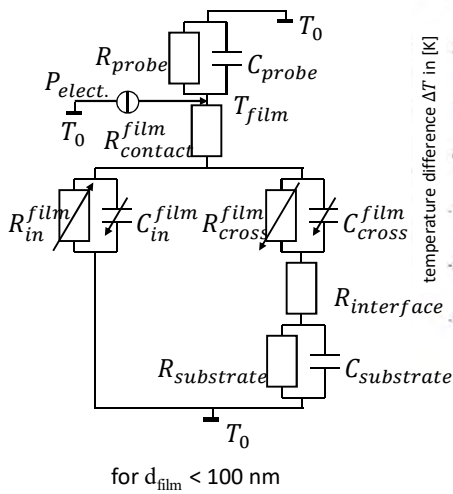
TiO₂ layer considered as bulk material in case of $d_{\text{film}} > 100 \text{ nm}$



*D. Chu et al. *Journal of Vacuum Science & Technology B: Microelectronics and Nanometer Structures*, (2001) 19, 2874



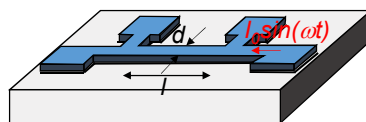
Disparate anisotropic heat transport



significant out-of-plane Stefan-Boltzmann-like heat loss due to phonon scattering
 phonon transport, Journal of Heat Transfer, (2013) 135, doi:10.1115/1.4023577



Frequency dependence of 3ω-method



D. G. Cahill, Review of Scientific Instruments, 1990, 61, 802–808

$$\text{wave number } k = \sqrt{\frac{\omega}{2a}}$$

$$\text{thermal diffusivity: } a = \lambda / \rho c$$

$$1/k \gg d : \omega_{\max} \ll 2a/d^2$$

$$1/k < l : \omega_{\min} > 2a/l^2$$

temperature distribution of line heat source with thermal penetration depth $\frac{1}{\alpha}$ (approximation):

$$\hat{T} = \frac{\hat{P}}{l \cdot \pi \cdot \lambda} \cdot J_0(\sqrt{j}ar) = \frac{\hat{P}}{l \cdot \pi \cdot \lambda} \cdot \left(\ln(2) - 0.5772 - \frac{j\pi}{4} + \frac{1}{2} \ln\left(\frac{a}{r^2}\right) - \frac{1}{2} \ln(\omega) \right)$$

quantities to be insert in SI without units!

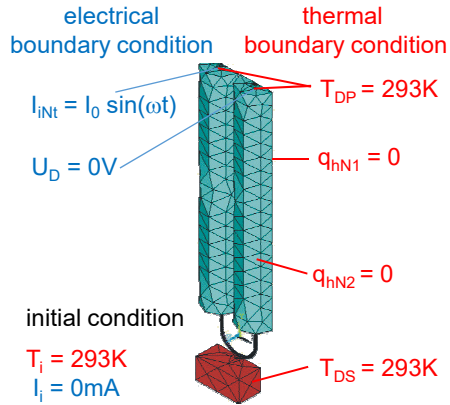
analysis in the frequency domain

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

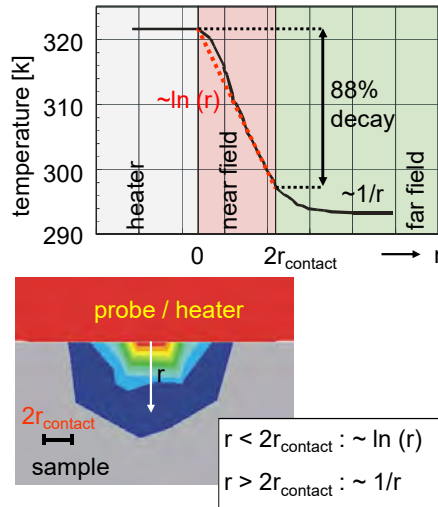


Validation of 3ω -method

FEM model of thermal probe:



Altes A. et al.: *Journal of Physics D: Applied Physics*, 37 (2004), issue 6, pp 952 - 963



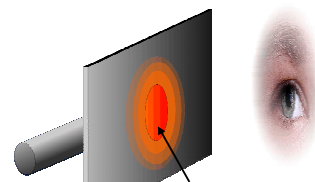
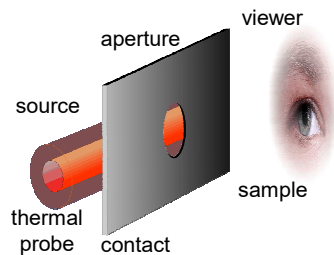
Validation of 3ω -method

Near field condition of thermal probe:

Altes A. et al.: *Journal of Physics D: Applied Physics*, 37 (2004), issue 6, pp 952 - 963

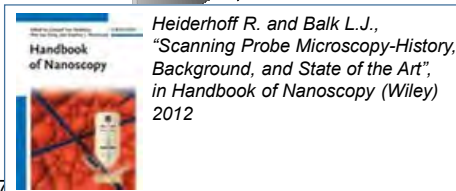
New approach of estimation

Conventional estimation



temperature distribution and frequencies depend on probe characteristic

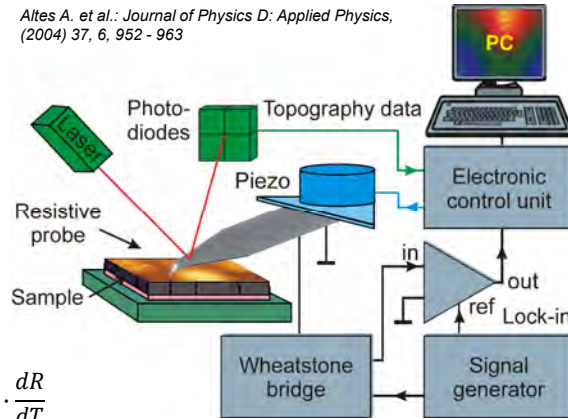
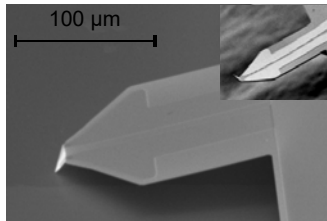
S. Gomès et al., *Phys. Status Solidi A* 212, No. 3, 477



Set-up of Scanning Thermal Microscope

P. Tovee et al.; *Journal of Applied Physics*, (2012) 112, 114317

Altes A. et al.; *Journal of Physics D: Applied Physics*, (2004) 37, 6, 952 - 963



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

D. G. Cahill, *Review of Scientific Instruments*, (1990) 61, 802–808

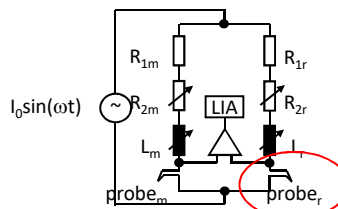
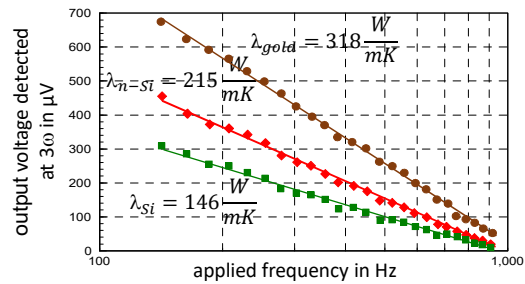
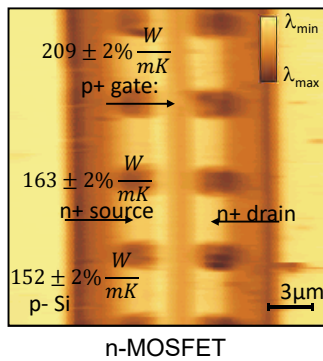
S. Lefèvre, S. Volz, *Rev. Sci. Instrum.* 76 (2005) 033701
S. Gomès et al., *Phys. Status Solidi A* (2015) 212, 3, 477–494.



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Quantitative thermal conductivity analysis on doped areas



T.H. Lee et al., *27th Int. Symp. Test. Fail. Anal.*, 2001: pp. 191–197

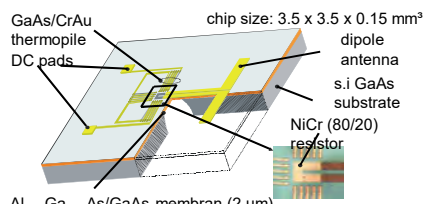


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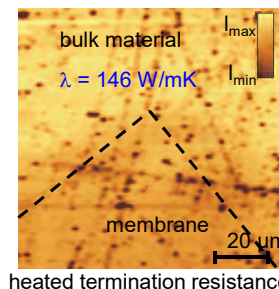
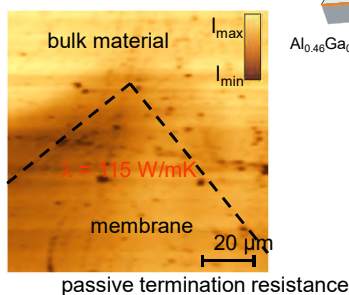


Thermal conductivity distribution on active devices

Local thermal conductivity $\lambda(T)$ analyses on micro-machined thin membrane using 3ω method



Interface membrane – bulk



A. Altes et al., *Superlattices Microstruct.* 35 (2004) 465–476



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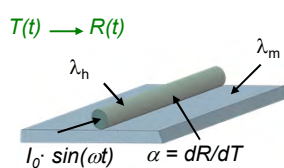
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Frequency domain of Scanning Thermal Microscopy

Electrical 3ω -signal generation:

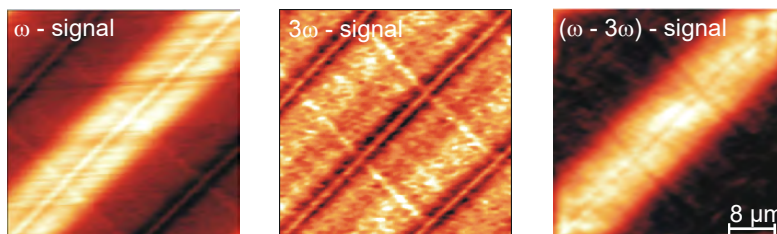
D. G. Cahill, *Review of Scientific Instruments*, 1990, 61, 802–808



$$R(t) = R_0 - dR/dT \cdot \hat{T}/2 \cdot \cos(2\omega t - \varphi)$$

$$U(t) = I_0 \cdot R_0 \cdot \sin(\omega t) + I_0 \cdot \hat{T}/4 \cdot dR/dT \cdot \sin(\omega t - \varphi) - I_0 \cdot \hat{T}/4 \cdot dR/dT \cdot \sin(3\omega t - \varphi)$$

Removal of conductivity information and impact of tip-sample thermal resistance from temperature measurements



see also: A. Reihani et al., *ACS Nano*, 2021, doi:10.1021/acsnano.1c08513



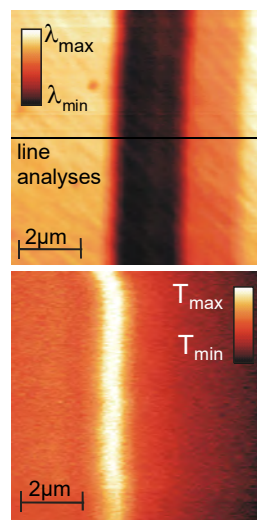
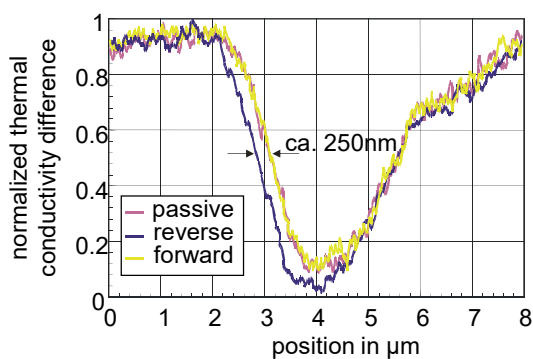
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Scanning Thermal Microscopy on active devices

Local thermal conductivity analyses on a InGaAlP-LED under different biasing conditions:



R. Heiderhoff et al., *J. Phys. Low Dimens. Struct.* 1/2 (2004) 63–70

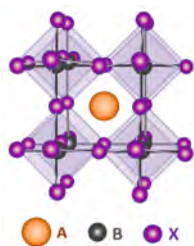


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Motivation and Purpose



Lev Perovski (1792-1856)



cation A =



Caesium



Methylammonium

Formamidinium

B = Pb, Cd, Ti, or Sn

anion X = I, Br, or Cl

Adv. Energy Mater., 2015, 5, 1500477
www.advenergymat.de

Intrinsic Thermal Instability of Methylammonium Lead Trihalide Perovskite

Bert Conings,¹ Jeroen Drijkoningen, Nicolas Gauquelin, Aslihan Babayigit, Jan D'Haen, Lien D'Olieslaeger, Anisha Eshirajan, Jo Verbeeck, Jean Manca, Edoardo Mosconi, Filippo De Angelis, and Hans-Gerd Boyen^{1*}

ADVANCED MATERIALS
Adv. Mater., 2016, 28, 6804–6834
www.advonlinelibrary.com

Perovskite Materials for Light-Emitting Diodes and Lasers

Sjoerd A. Veldhuis, Pablo P. Boix,¹ Natalia Yantara, Mingjie Li, Tze Chien Sum, Nirpan Mathews, and Subodhi G. Mukhopadhyay^{1*}

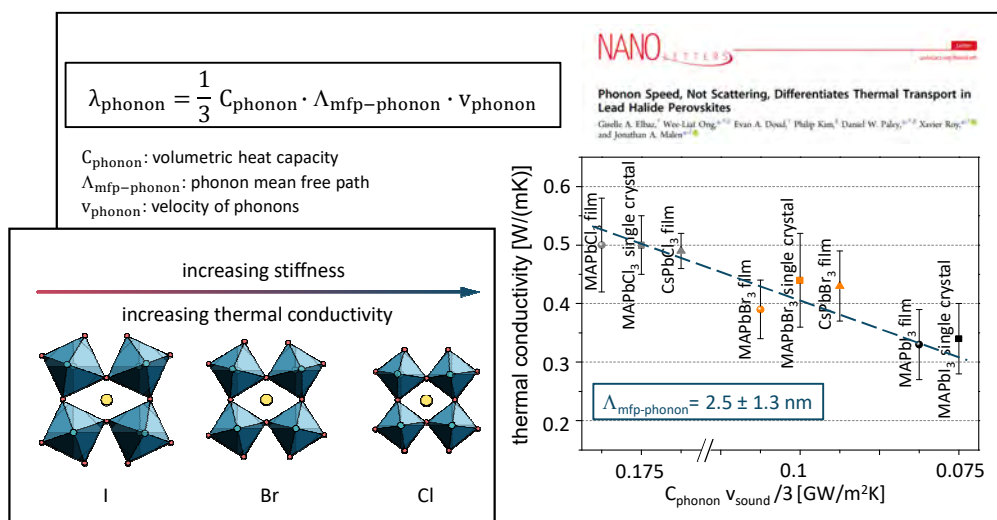


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Thermal conductivity of halide perovskites



Haeger T. et al., *J. Mater. Chem. C*, (2020), DOI: 10.1039/D0TC03754K

& improved air-stability



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Quantitative thermal conductivity mapping

Haeger T. et al, *J. Phys. Chem. Lett.* (2019), 10, 3019–3023, DOI: 10.1021/acs.jpclett.9b01053

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

applying mixed voltage signal:

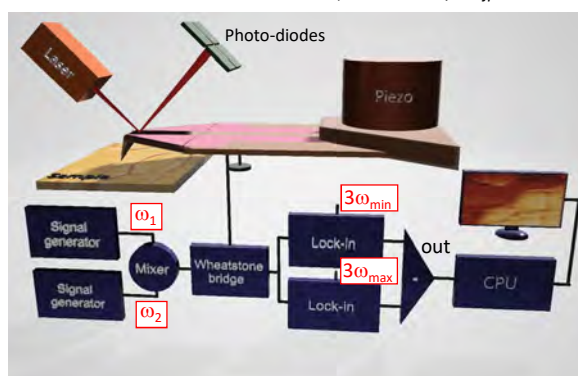
$$U(t)_{\text{bridge}} = \frac{1}{c} U_a \sin(\omega_a t + \varphi_a) \cdot U_b \sin(\omega_b t + \varphi_b)$$

c: coupling factor [V]

signal: phase neglected

$$U(t)_{\text{probe}} = (I_0 R_0 - I_0 \frac{dR}{dT} \cdot \frac{\hat{T}}{4}) (\cos(\omega_1 t) + \cos(\omega_2 t)) + I_0 \frac{dR}{dT} \cdot \frac{\hat{T}}{4} (\cos(3\omega_1 t) + \cos(3\omega_2 t))$$

$$-I_0 \frac{dR}{dT} \cdot \frac{\hat{T}}{4} (\cos((2\omega_1 - \omega_2)t) - \cos((2\omega_1 + \omega_2)t) - \cos(2\omega_2 - \omega_1)t + \cos(2\omega_2 + \omega_1)t)$$

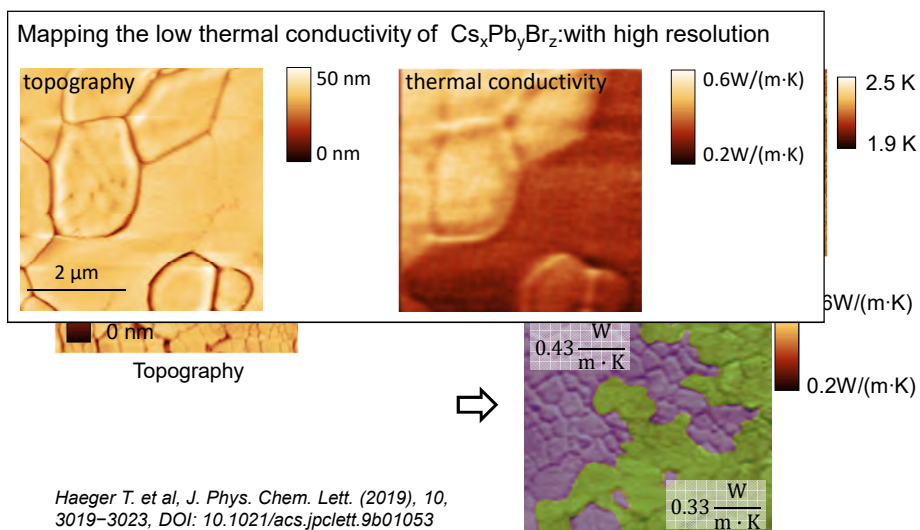


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Quantitative thermal conductivity mapping



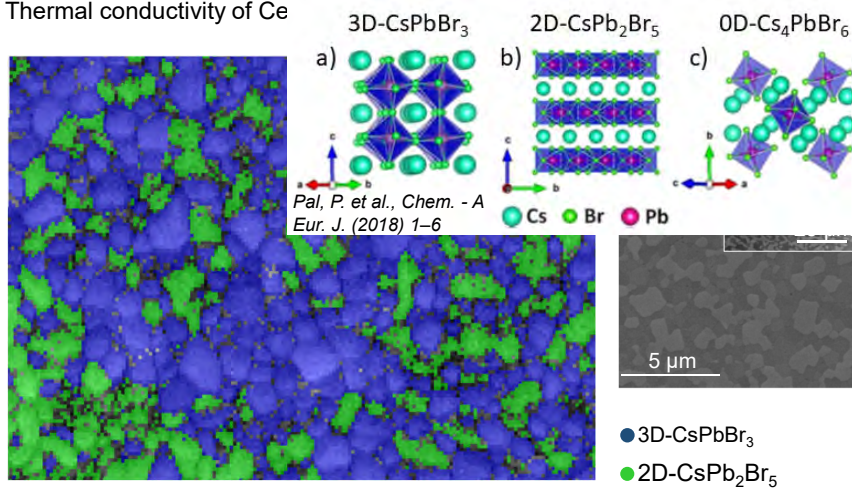
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Quantitative thermal conductivity mapping

Thermal conductivity of Ce

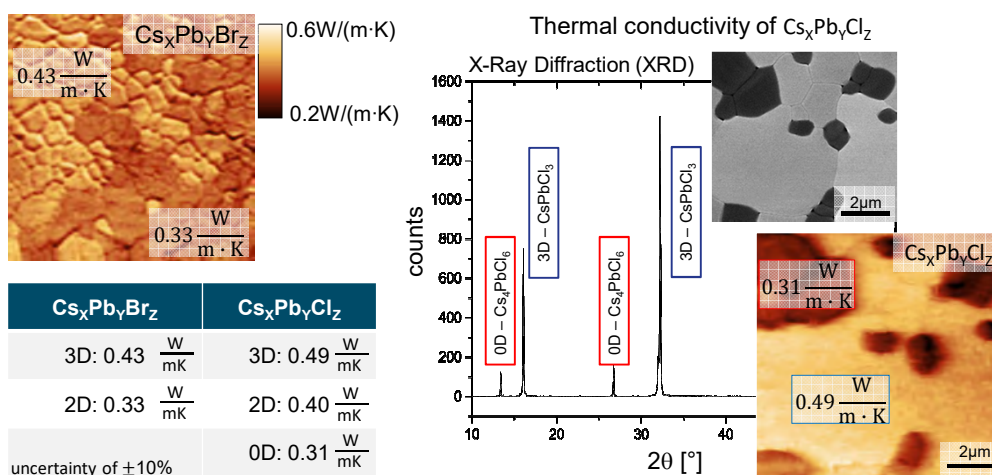


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Quantitative thermal conductivity mapping



3D- CsPbBr_3 : 0.4 $\text{W}/(\text{m}\cdot\text{K})$ Woochul Lee et al., PNAS 114 (2017), 33, 8693–8697

T. Haeger et al., in MRS Fall Meeting, Boston, 2019, p. EN09.15.02



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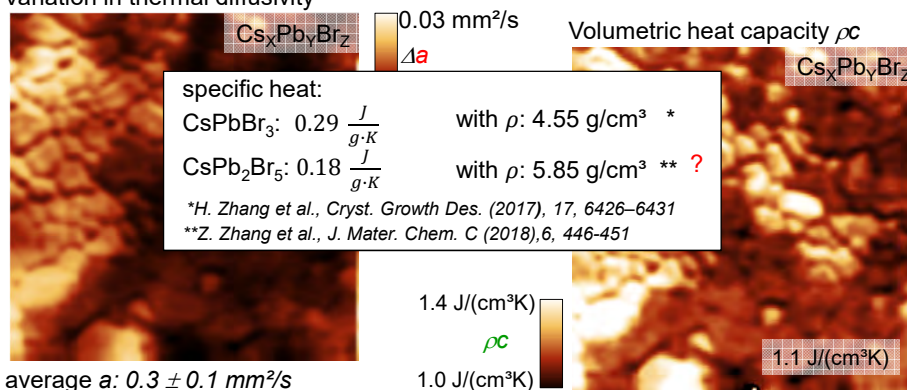
Simultaneous Mapping of Thermal Conductivity, Thermal Diffusivity, and Volumetric Heat Capacity

Haeger T. et al., J. Phys. Chem. Lett. (2019), 10, 3019–3023, DOI: 10.1021/acs.jpcclett.9b01053

$$\hat{T} = \frac{\dot{Q}_s}{\lambda} \cdot \left(\frac{1}{2} \ln\left(\frac{a}{r^2}\right) - \frac{1}{2} \ln(\omega) + \ln(2) - 0.5772 - \frac{j\pi}{4} \right) \quad \frac{\partial T(\vec{x}, t)}{\partial t} = a \cdot \Delta T(\vec{x}, t) \quad [\dot{Q}_s] = \frac{\text{W}}{\text{m}}$$

D. G. Cahill, Review of Scientific Instruments, 1990, 61, 802–808

Variation in thermal diffusivity



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Simultaneous Mapping of Thermal Conductivity, Thermal Diffusivity, and Volumetric Heat Capacity

Haeger T. et al, *J. Phys. Chem. Lett.* (2019), 10, 3019–3023, DOI: 10.1021/acs.jpcclett.9b01053

$$\hat{T} = \frac{\dot{Q}_s}{\lambda} \cdot \left(\frac{1}{2} \ln \left(\frac{a}{r^2} \right) - \frac{1}{2} \ln(\omega) + \ln(2) - 0.5772 - \frac{j\pi}{4} \right) \quad \frac{\partial T(\vec{x}, t)}{\partial t} = a \cdot \Delta T(\vec{x}, t) \quad \frac{W}{m}$$

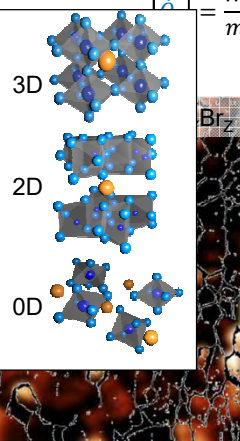
D. C
Var

Thermal properties of Cs based chloride perovskites

perovskite	λ [W/(mK)]	a [mm ² /s]	c_v [J/(cm ³ K)]
3D-CsPbCl ₃	0.53	0.5	1.1
2D-CsPb ₂ Cl ₅	0.40	0.6	0.7
0D-Cs ₄ PbCl ₆	0.30	0.5	0.5

T. Haeger et al., in *MRS Fall Meeting, Boston, 2019*, p. EN09.15.02

T. Haeger et al., *J. Phys. Mater.*, 2020, 3, 024004



average a : 0.3 ± 0.1 mm²/s

$\Delta\rho c$
0 J/(cm³K)

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Motivation and Purpose

Phase transitions of
halide perovskites

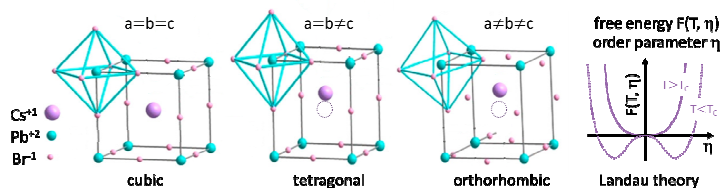
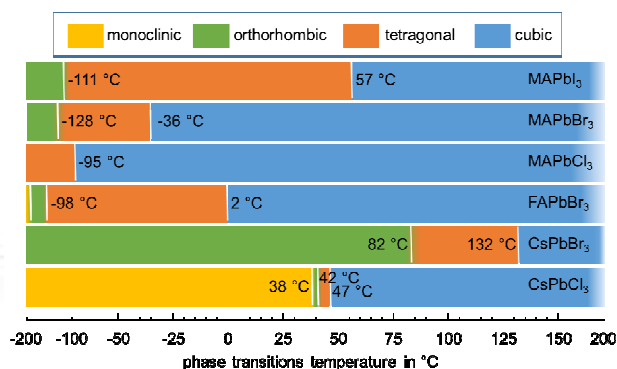
T. Haeger et al., *J. Mater. Chem. C*,
2020, 8, 14289–14311

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www.nature.com/amt

Adv. Mater., 2016, 28,
6804–6834

Perovskite Materials for Light-Emitting Diodes and Lasers

Spond A, Wüthrich, Pablo P. Boix, Natalia Yanina, Mingjie Li, Tsz Cheri Sum, Nirpan Mathew, and Subash C. Mohankumar



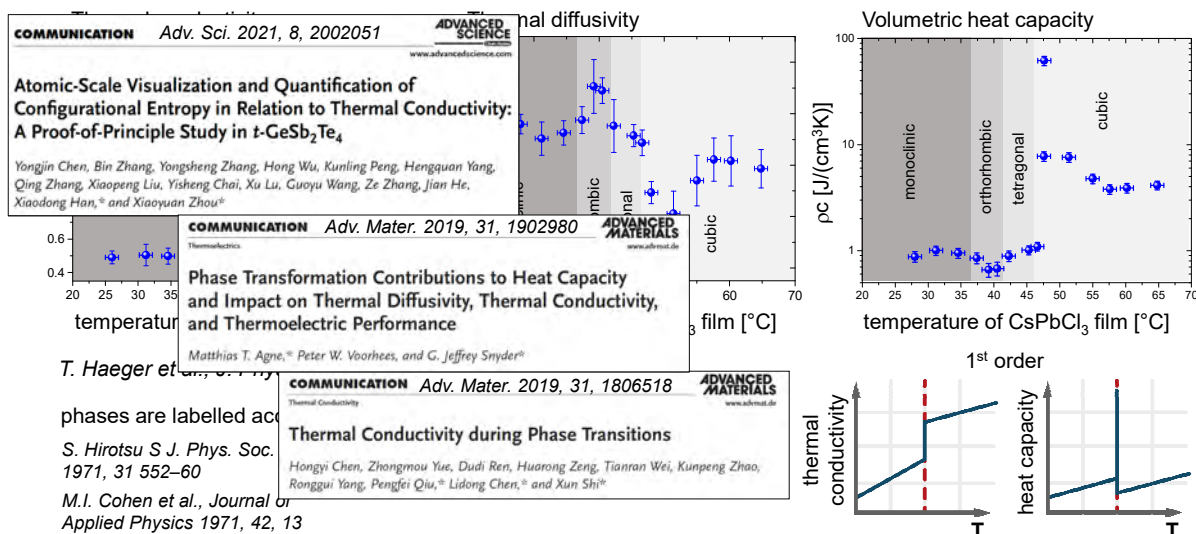
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Thermal properties of CsPbCl₃ at phase transitions



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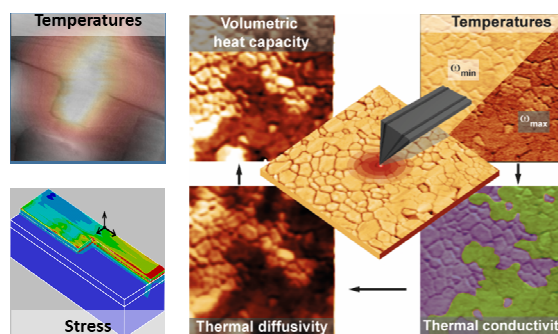


Closing remarks

Thermal and thermoelastic quantities determinable

- on ultra-high und ultra-low thermal conductors
- with spatial resolution in the nanometer range
- with femto-meter sensitivity
- on thin films
- at phase transitions

Scanning near-field thermal microscopy techniques assisted by complementary finite element simulation



- Reliability investigations on smart materials and devices
- Transition from diffusive heat flux to ballistic Stefan–Boltzmann like heat transport in thin films
- Thermal properties of hybrid and all-inorganic perovskites in dependence on choice of cations & halides, dimensionality, and crystal-phase
 - ultra low thermal conductivities
 - low thermal diffusivities
 - low volumetric heat capacities



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 J. Bahr
 M. Ketterer

Prof. Dr. rer. nat. Dr. h.c. (bsuir) Ludwig Josef Balk
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DFG Deutsche
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Thank you for your attention



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