Entwicklung und Charakterisierung von nanoskaligen Thermoelementen

Tristan Schlagregen

Vortrag zur Masterarbeit

Outline

- Motivation
- Thermocouple (ThC)
    - Theory
    - Structure
    - Measurement method
- Measurement Results
    - 3 Different Material Pairings
- Summary

Motivation

Conventional thermometers: Limitation of size or access

Development of thermometers with a spatial resolution > 1 µm

Nanothermometry

No universal thermal sensor

Different applications require different properties

My work

Development and characterization of nanoscale thermocouples (ThCs)

Measurement of nanoparticles in TEM

State of the Art

*estimation

Nanothermometer	Temperature Range	Temperature Resolution	Spatial Resolution	Main Advantage	Main Disadvantage
PNIPAM Nanoparticles	306 K- 314 K	~1 K*	< 1 µm*	Reversible	Aqueous environment
UCNPs	273 K-900 K	0.1 K	< 1 µm	Wider temperature range (than PNIPAM)	Calculation of the intensity relations
Ga-filled CNT	323 K-773 K	> 13 K*	10 µm × 75 nm*	(Simple) In situ procedure	Determination of Ga-level
Ga-filled MgO-NT	303 K-1073 K	> 10 K*	2 µm × 50 nm*	Wider temperature range (than CNT)	Spatial resolution
SThEM	~RT*	~1 K *	20 nm	Precise	Expansion of the subtrate
AFM Thermistor	~RT*	< 1 mK	100 nm	Topography and temperature	Fragile
AFM Thermocouple	~RT*	< 0.1 K	≥ 50 nm	Precise	Complex structure
Monometal-Thermocouple	~RT	~0.1 K*	> 150 nm*	Simple structue	Few possible application
Bimetal-Thermocouple	~RT*	300 µK	85 nm × 230 nm	Temperature Resolution	Complex structure

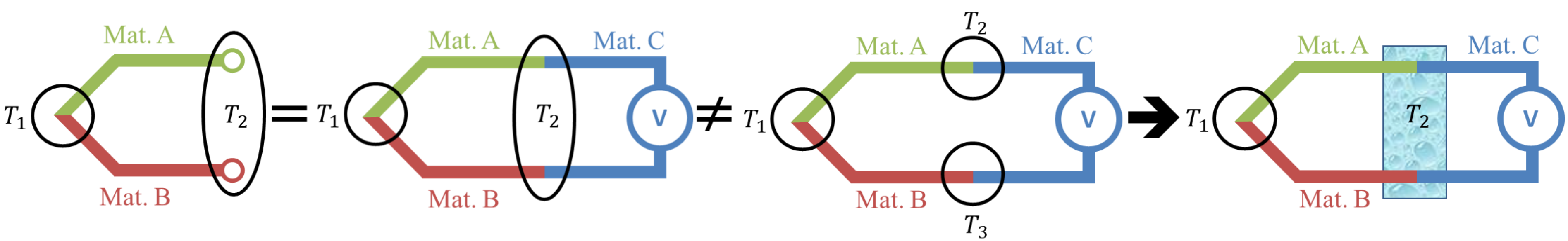
More details in my last presentation.

Contact vs. Thermal Voltage

- Compensation of all contact voltages in closed electric circuits
- Thermal voltage = temperature dependence of contact voltage (no compensation, if contact are on different temperatures)

  ➔ Thermocouple

  - Measurement of ΔT between thermal contact and measurement contact
  - Independent of number of materials (provided all contacts are at the same temperature) ➔ Ice water



Seebeck Coefficients

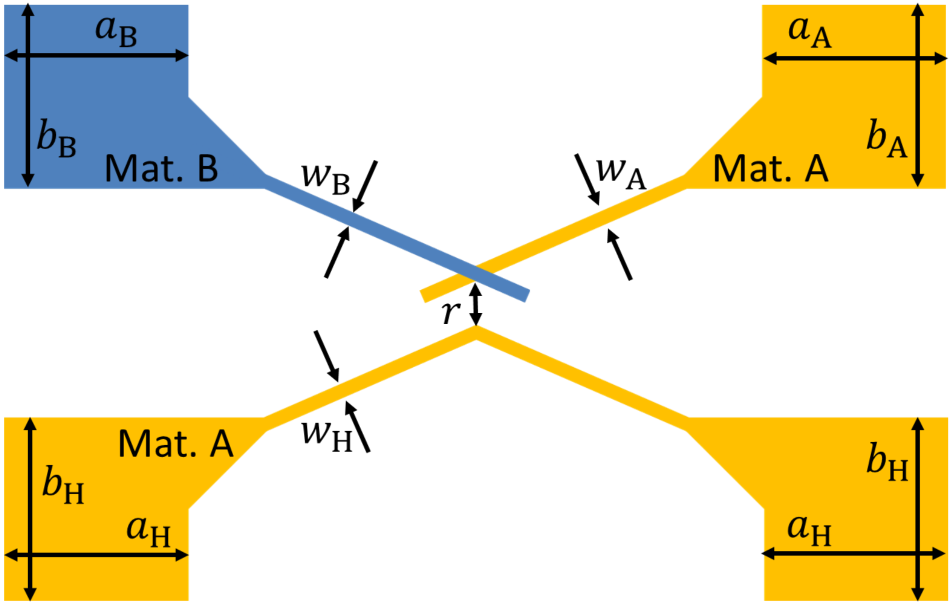
Material	$S_{A,Pt}(273\ K)\ \left[\frac{\mu V}{K}\right]$
Ge	300,0
NiCr	25,0
Fe	19,0
Cd	7,5
Cu	6,5
Au	6,5
Ag	6,5
Rh	6,0
Pb	4,0
Al	3,5
C	3,0
Hg	0,6
Pt	0,0
Na	-2,0
K	-9,0
Ni	-15,0
NiCu	-35,0

- Seebeck coefficients $S_{\mathrm{A,B}}$ as the temperature sensitivity of thermocouples
- $U_{\mathrm{A,B}} = S_{\mathrm{A,B}} * (T_1 - T_2)$
- Temperature and material dependent
- Estimation:

$$U_{\mathrm{Ge,Au}} = 293{,}5 * (22\ °\mathrm{C} - 20°\mathrm{C}) = 587\ \mu\mathrm{V}$$
$$U_{\mathrm{NiCr,Au}} = 18{,}5 * (22\ °\mathrm{C} - 20°\mathrm{C}) = 37\ \mu\mathrm{V}$$

https://www.efunda.com/designstandards/sensors/thermocouples/thmcple_theory.cfm? Orderby=Seebeck0C#Sensitivity, 12.12.2020 17:50.

2-Step-Lithography



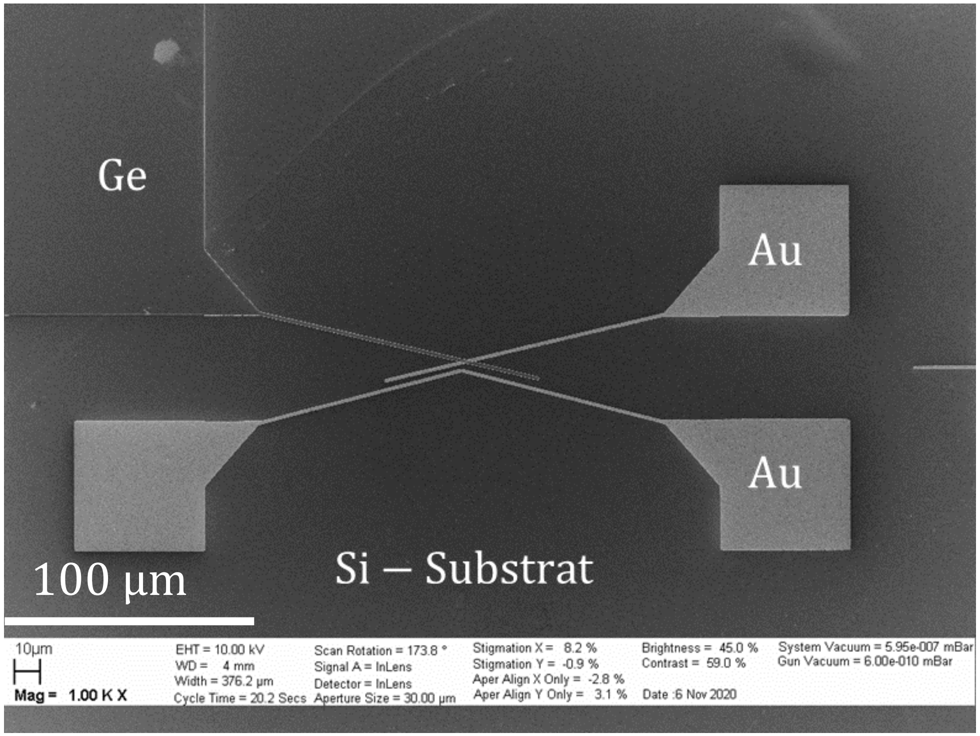
Sample preparation

1. Sample cleaning: acetone, water, isopropanol
2. PMMA Coating: Thickness: 200 nm
3. Electron beam lithography
4. Developer
5. Stopper
6. Evaporation
7. Lift-off
8. Repeat from step 2.

Materialpaarung	1	2	3
Material A	Au	Au	Chromel
Material B	Ge	NiCr	Ni
Haftvermittler (Ti)	5 nm für Au	5 nm für Au	5 nm für Ni

Au, Ge ➔ Weimann (BHE)
Chromel, NiCr, Ni ➔ Lorke

2 Step Lithography



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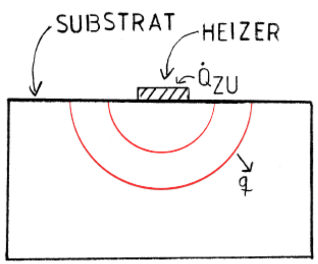
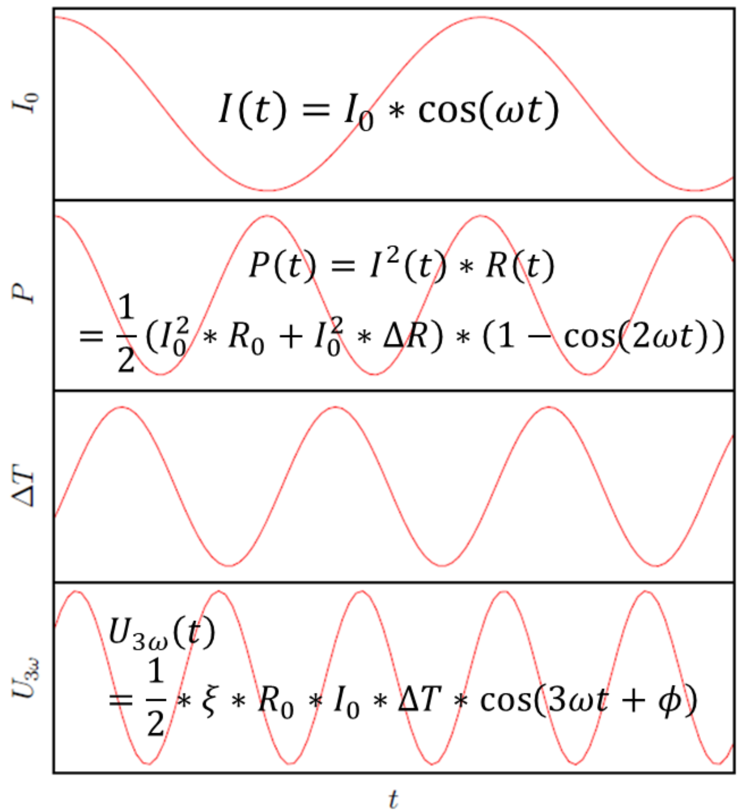
Measurement Method: 3ω-Model





$I(t)$: AC-Current
I_0: Current amplitude
ω: Angular frequency
t: Time
$P(t)$: Electrical power
$R(t)$: Resistance
ΔR: Change in resistance
$U_{3\omega}(t)$: 3ω Voltage
ξ: Temperature coefficient of resistance
ϕ: Phase shift
ΔT: Temperature difference

$$U_{3\omega} \sim \Delta T$$

M. Becker, Entwicklung eines Messverfahrens zur Bestimmung der Wärmeleitfähigkeit von dünnen Schichten, Dissertation, Fakultät für Physik der Universität Duisburg-Essen, 2019.

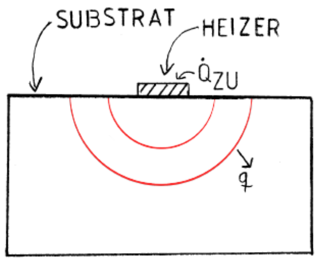
3ω-Model

Cahill & Pohl 1987:

$$\Delta T = \frac{P}{l\pi\kappa} K_0(qr)$$

If $qr \ll 1$:

$$\Delta T = Re\left[\frac{P}{l\pi\kappa}\left[\frac{1}{2}\ln\left(\frac{\alpha}{r^2}\right) + \ln(2) - 0{,}5772 - \frac{1}{2}\ln(\omega) - \frac{i\pi}{4}\right]\right]$$

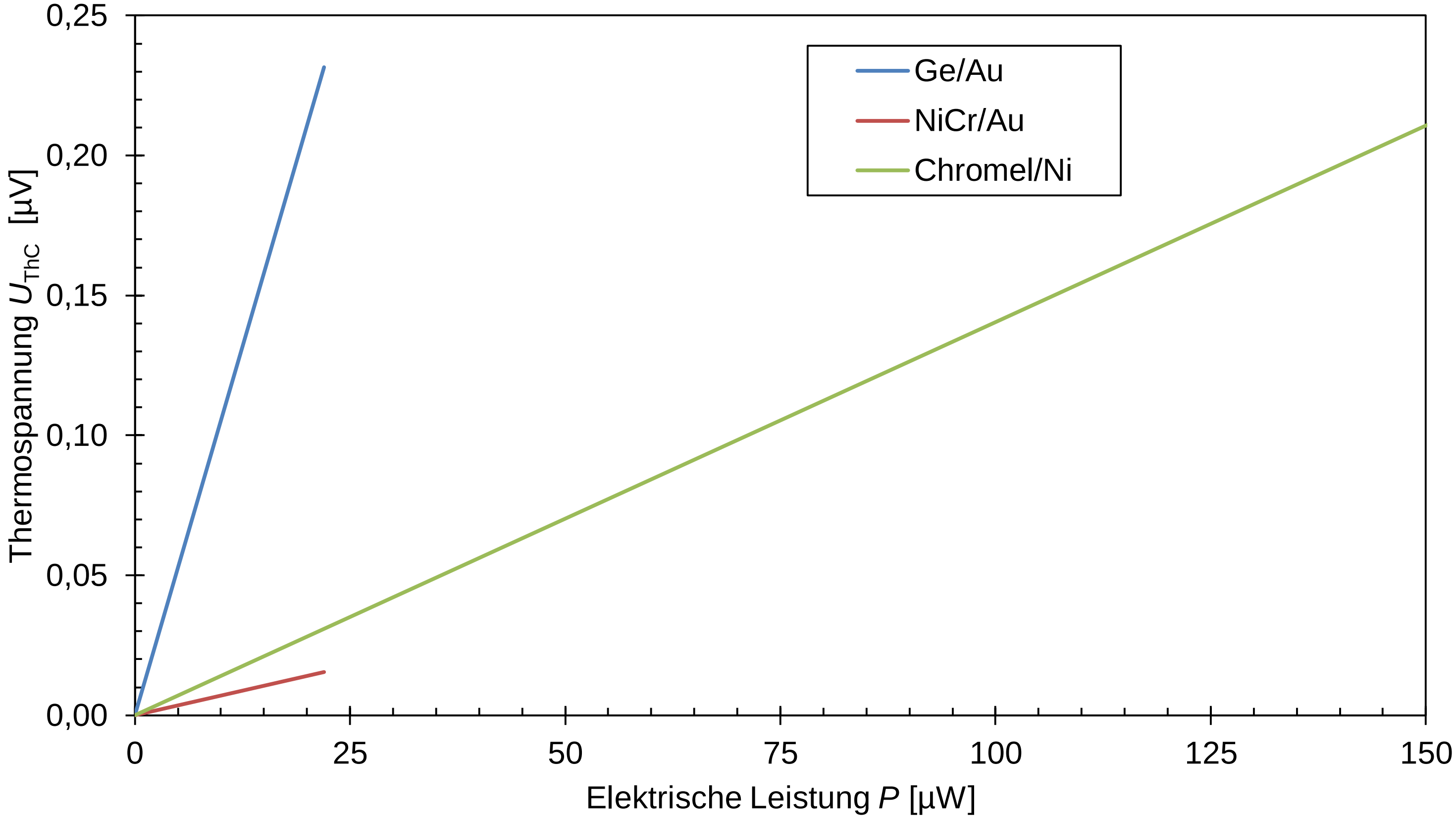


ΔT: Temperature difference
P: Electrical power
l: Wire length
κ: Thermal conductivity
K_0: function
q: Reciprocal value of the thermal wavelength
r: Distance
α: thermal Diffusivity
ω: Angular frequency
i: Imaginary unit

$$P \sim \Delta T$$

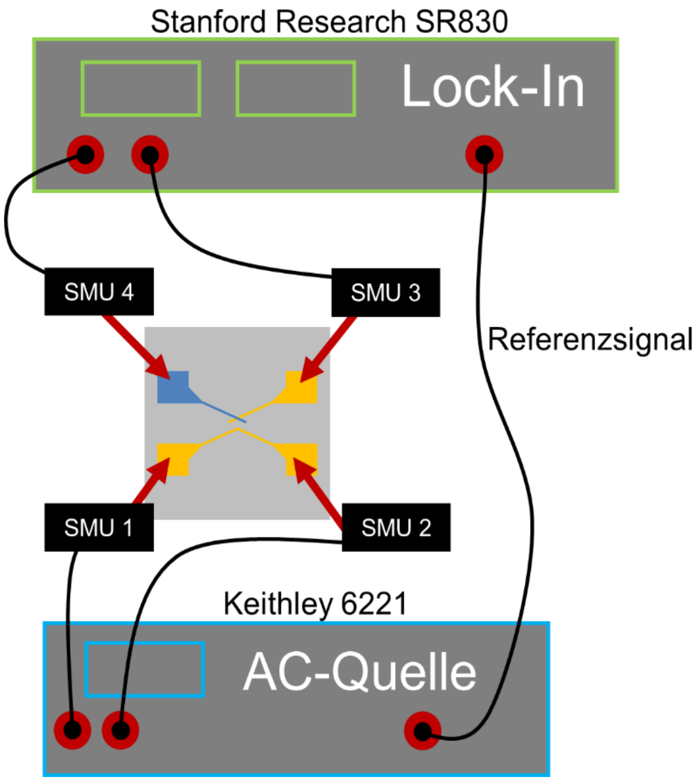
D. G. Cahill und R. O. Pohl, Thermal Conductivity of Amorphous Solids above the Plateau, Phys. Rev. B, vol. 35, no. 8, pp. 4067-4073, 1987.
M. Becker, Entwicklung eines Messverfahrens zur Bestimmung der Wärmeleitfähigkeit von dünnen Schichten, Dissertation, Fakultät für Physik der Universität Duisburg-Essen, 2019.

3ω-Simulation

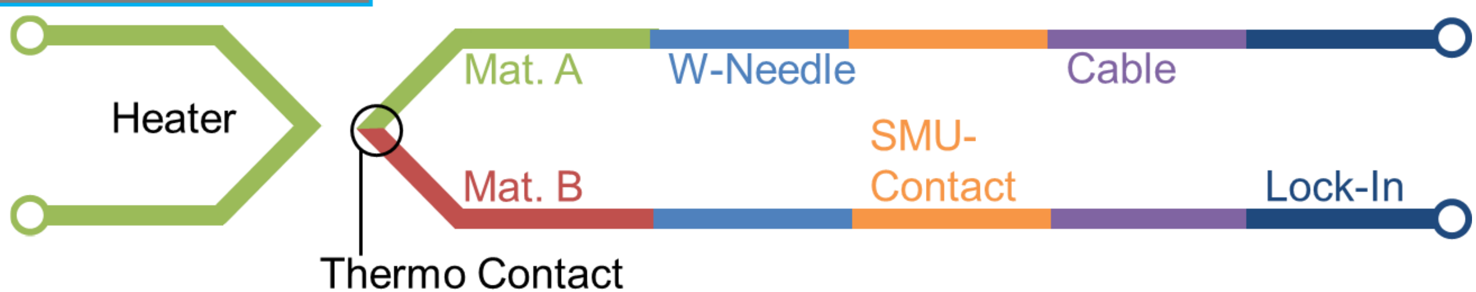


$$w_H = 1\ \mu\text{m}$$
$$r = 2\ \mu\text{m}$$

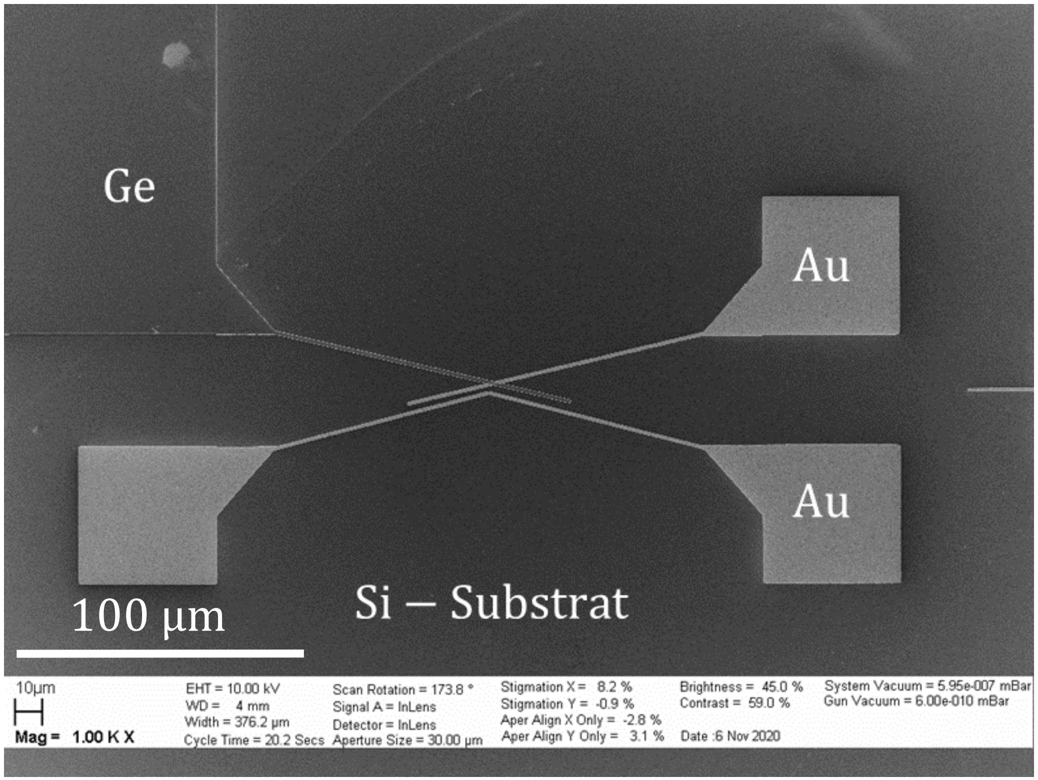
Measurement Setup

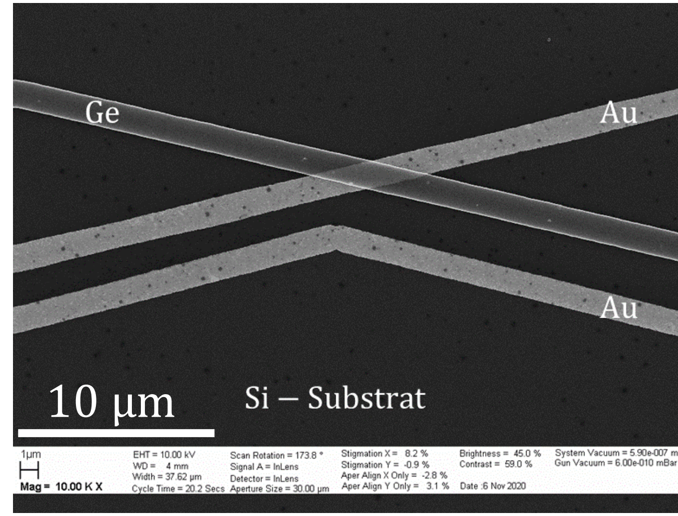


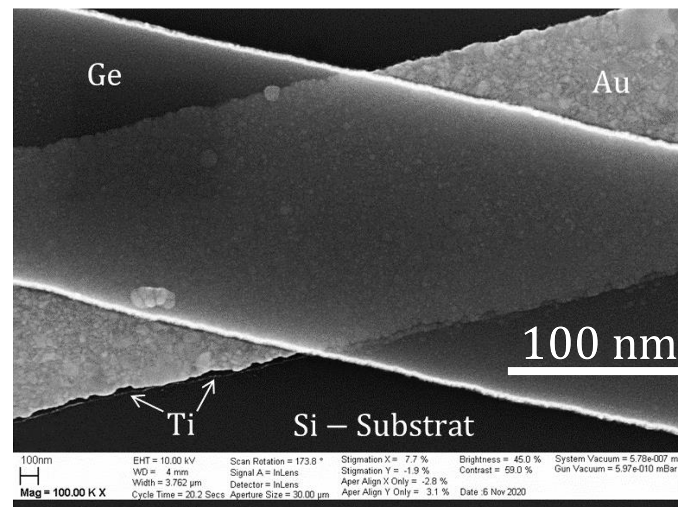
- AC current ($f = 5$ kHz)
- SMU 1 + SMU 2: Heat element
- SMU 3: Thermal contact of material A
- SMU 4: Thermal contact of material B
- Heat propagation through Si-substrate (thermal wave)
- 2ω-Detection: Thermal voltage
  - All four contacts
- 3ω-Detection: 3ω-voltage
  - Heat contacts



Ge/Au Thermocouple

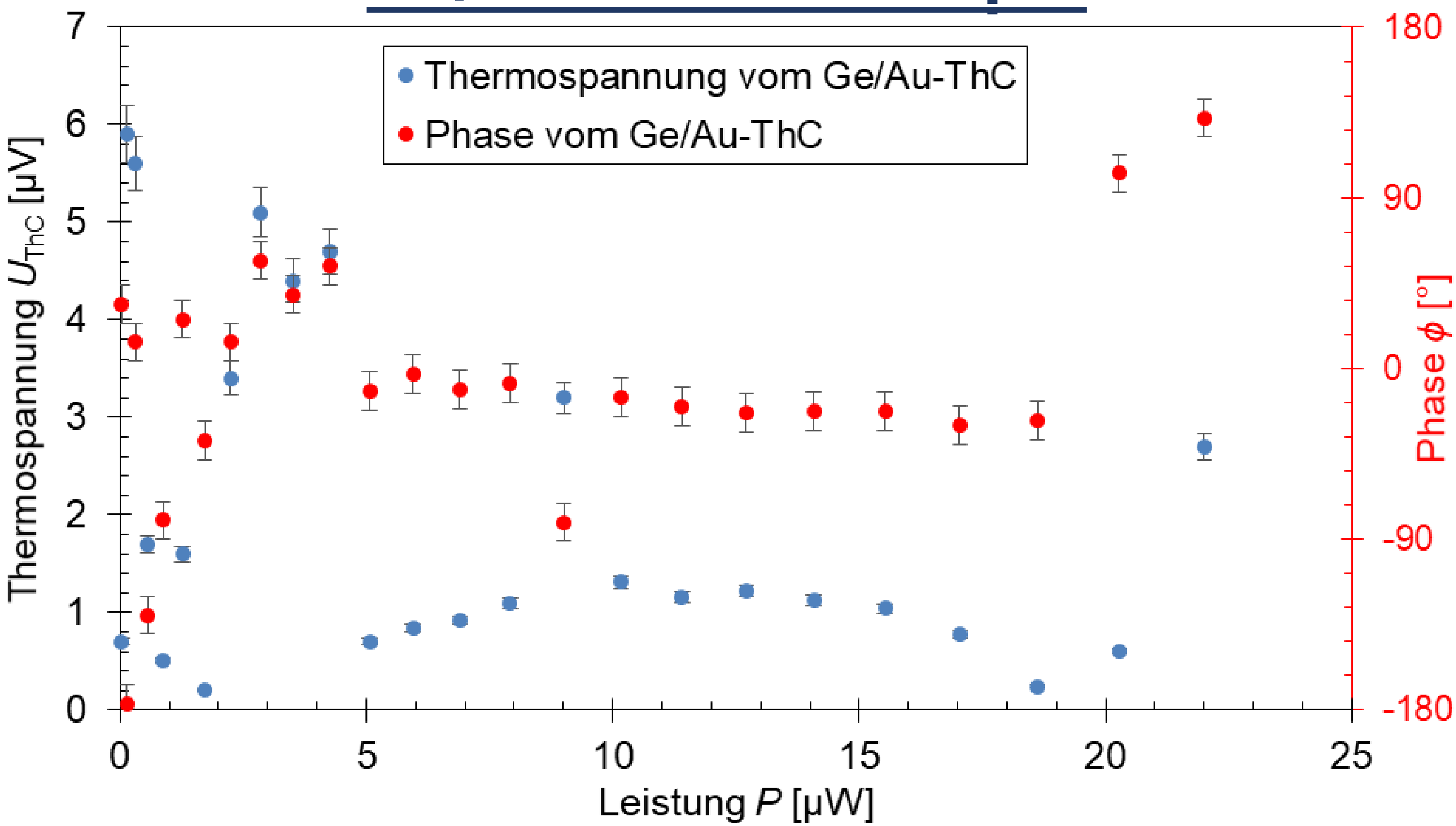






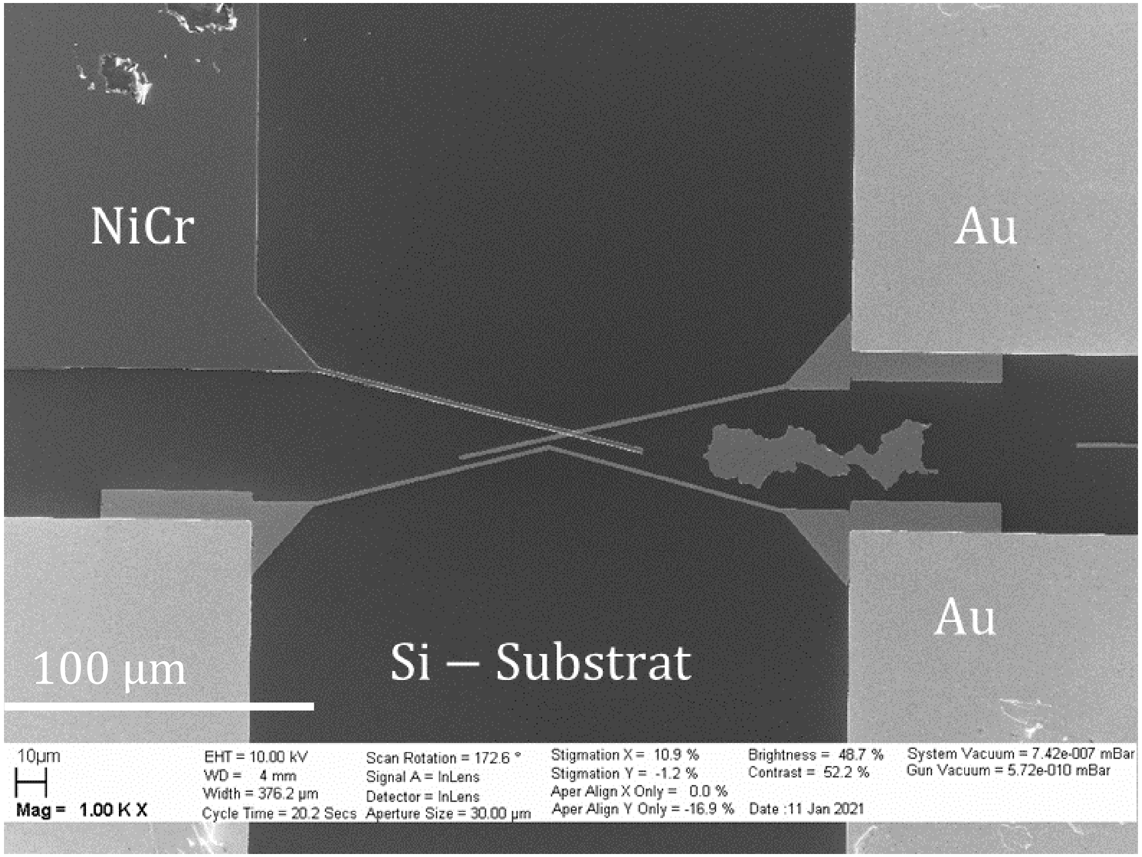
- $S_{\mathrm{Ge,Au}}(273\ \mathrm{K}) = 293{,}5\ \frac{\mu\mathrm{V}}{\mathrm{K}}$
- $r = 2{,}4\ \mu\mathrm{m}$

Ge/Au Thermocouple



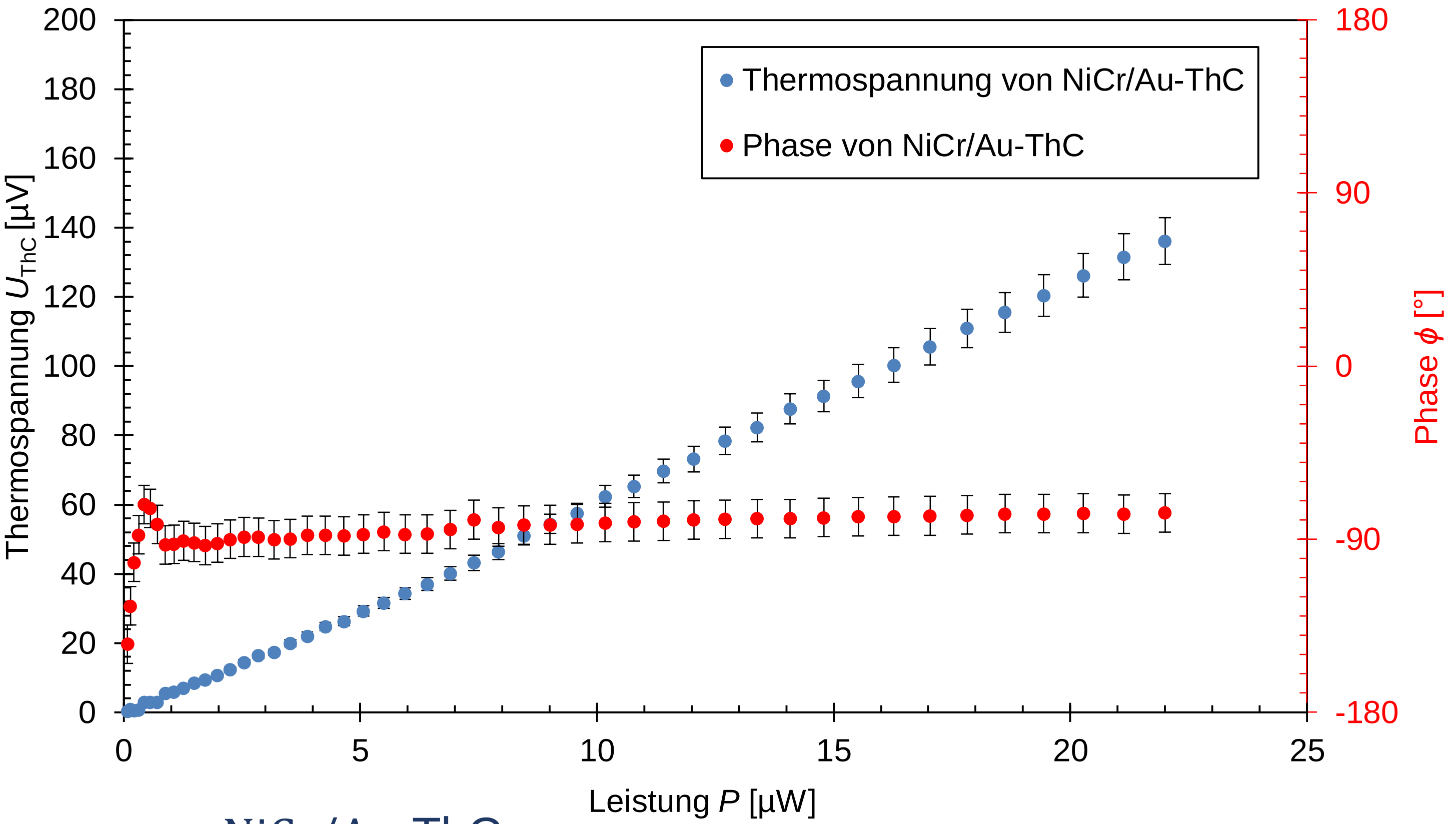
- No constant phase
- Large spread of voltage
- Not measurable

NiCr/Au Thermocouple



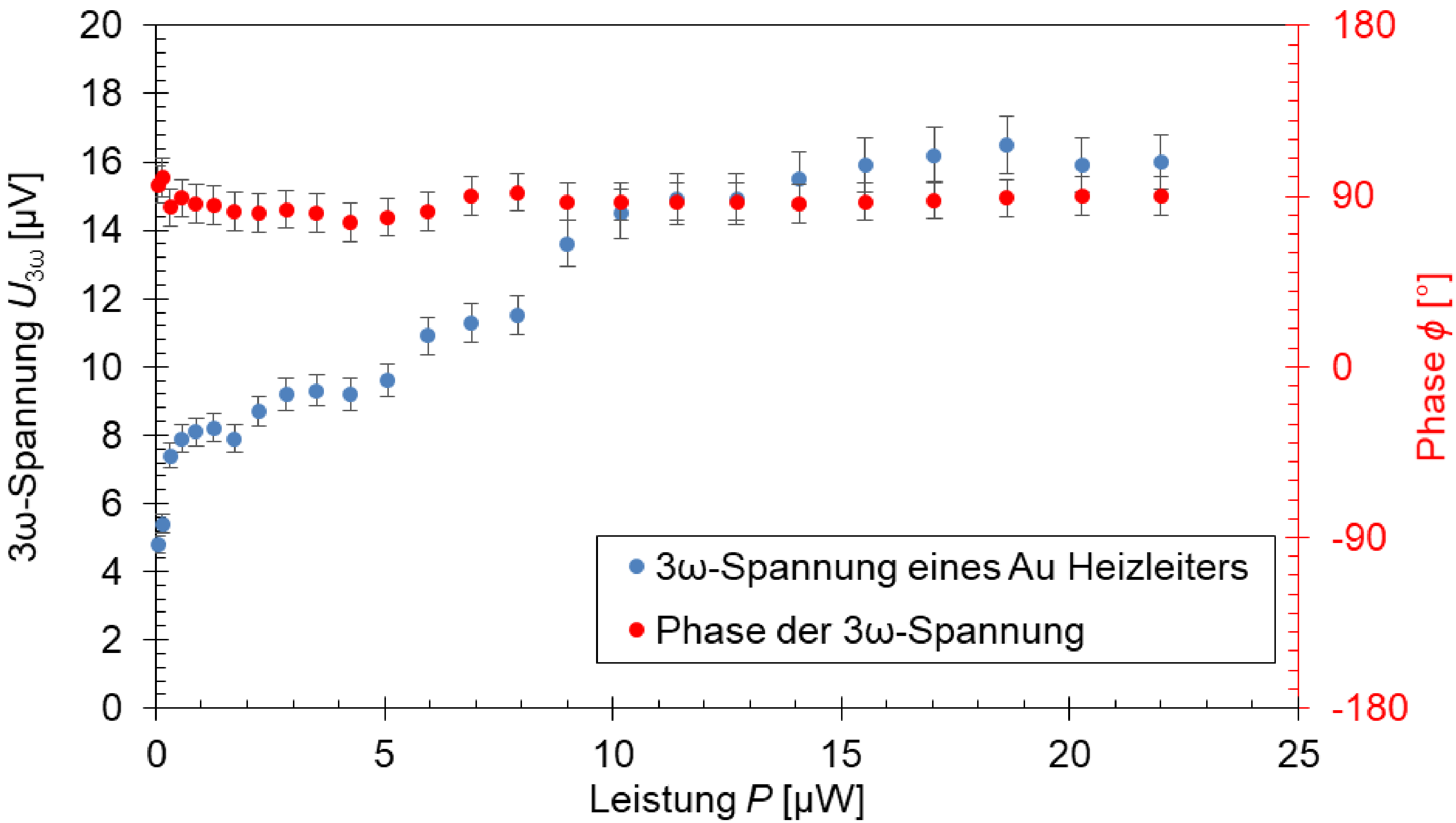
- $\mathrm{NiCr} = 50\%\ \mathrm{Ni}\ + 50\%\ \mathrm{Cr}$
- $S_{\mathrm{NiCr,Au}}(273\ \mathrm{K}) = 18{,}5\,\frac{\mu\mathrm{V}}{\mathrm{K}}$
- $r = 5{,}1\ \mu\mathrm{m}$
- Bigger contacts enable 3ω-measurement
  ➔ 3-step-lithography

NiCr/Au Thermocouple



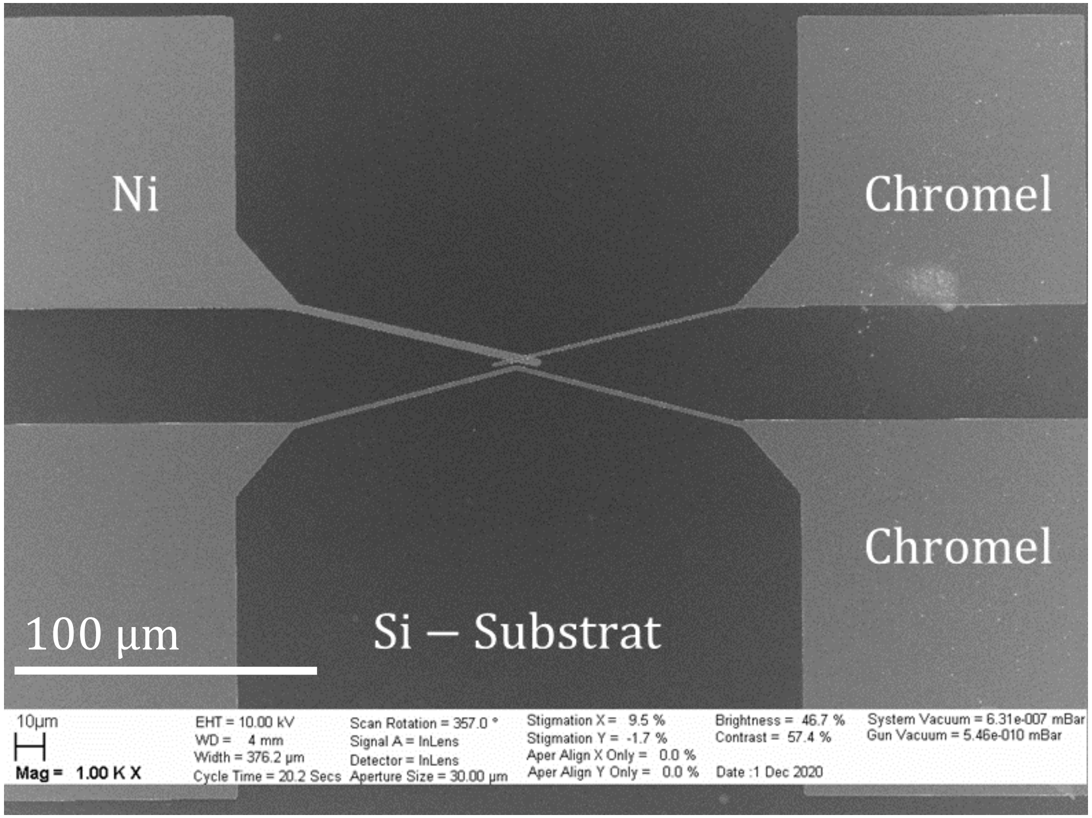
- NiCr/Au-ThC:
  - Phase $\Phi \approx$ const
  - $U_{ThC} \sim P$

NiCr/Au Thermocouple



- Phase $\Phi \approx \mathrm{const}$
- Continuous increase of $U_{3\omega}$

Chromel/Ni Thermocouple

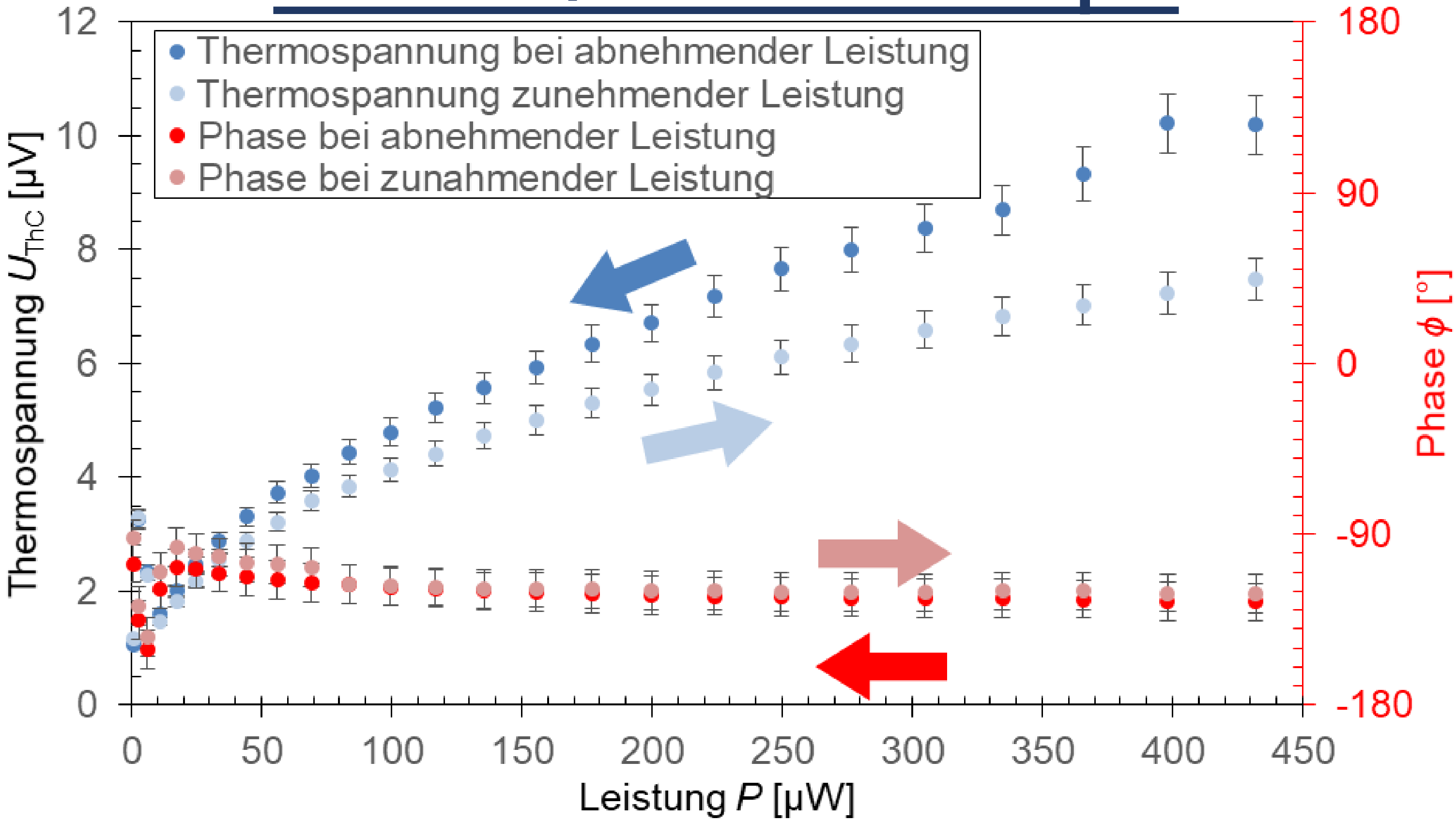


- $S_{\mathrm{NiCr,Au}}(273\ \mathrm{K}) = 40\ \frac{\mu\mathrm{V}}{\mathrm{K}}$
- $r = 1{,}2\ \mu\mathrm{m}$

- Type K = Chromel/Alumel
- Chromel = 89% Ni, 9% Cr, 1% Si, 1% Fe
- Alumel = 95% Ni, 1,5% Si, 1,5% Al, 1,5% Mn, 0,5% Co

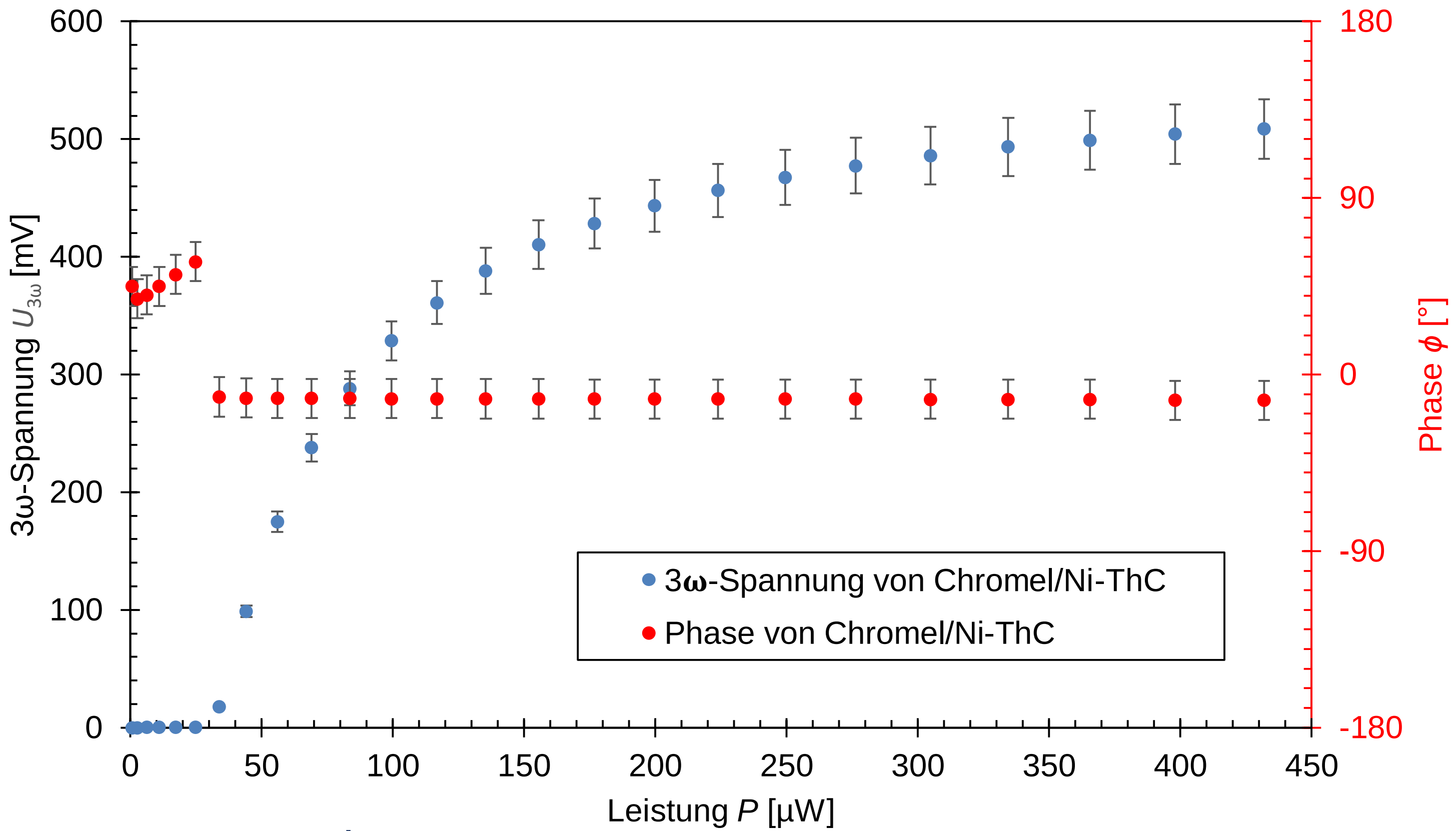
  ➔ Ni instead of Alumel

Chromel/Ni Thermocouple



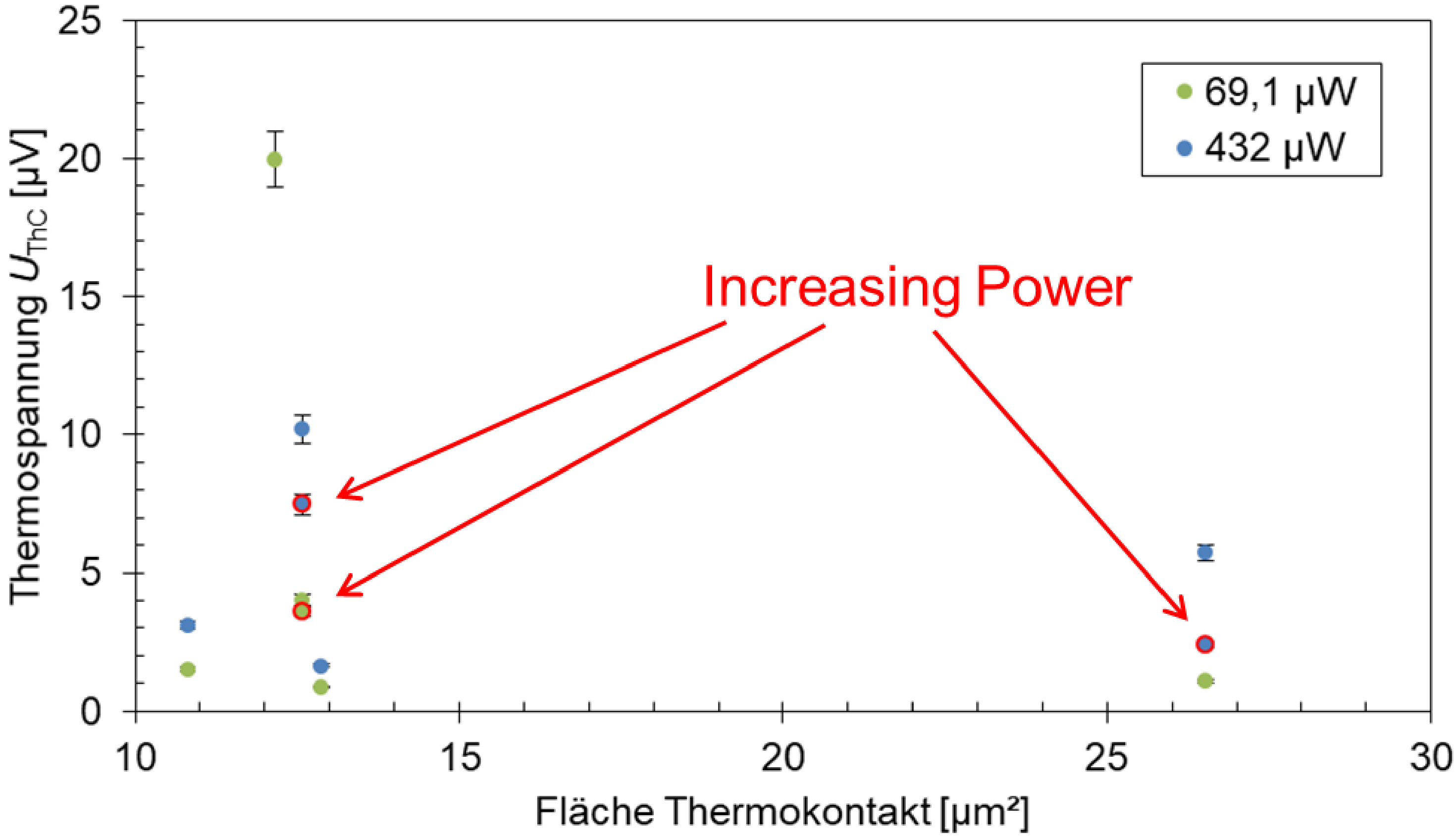
- Measurement with increasing and decreasing power
- Differerent Measurement result
- Phase $\Phi \approx \text{const}$
- Temperature hysteresis

Chromel/Ni Thermocouple



- Phase $\Phi \approx$ const
- Offset at 35 µW
- No linear increase
  ➔ ≈ Root function

Chromel/Ni Thermocouple



- No correlation between thermal voltage and area
- Spread as a result of the production of the structure

Summery

- ThC:

  $U_{\mathrm{A,B}} = S_{\mathrm{A,B}} * (T_1 - T_2)$, independent of number of materials provided all are at the same temperature

  - Seebeck coefficient $S_{\mathrm{A,B}}$ material and temperature dependent
- 3ω-Method = model of temperature determination of planar wires
  - $U_{3\omega} \sim P \sim \Delta T$
- **Ge/Au**, **NiCr/Au**, Chromel/**Ni** with 2- and 3-Step-Lithography
  - Linear temperature and power dependence
  - Measurement two decades larger than model
  - Temperature hysteresis
  - No area dependence: $U_{\mathrm{A,B}} \neq f(A)$

Outlook

- Modifications of the measurement setup and the ThC structures are required
  - Could not be optimized within this work
  - E.g. 3ω-voltage
  - Wafer bonds ➔ No influence of W-Needles
  - Reduction of heat dissipation➔ E.g. membrane-substrate
- New material pairings ➔ Improvement of sensitivity
- Reduction of the structure sizes ➔ Spatial resolution improvement

➔ Measurement in TEM

Thank you for attention!

Questions?