LINSEIS

Periodic Laer Heating-

Eine Erweiterung der Laser Flash Methode zum Messen der thermischen Eigenschaften dünner Proben

Agenda

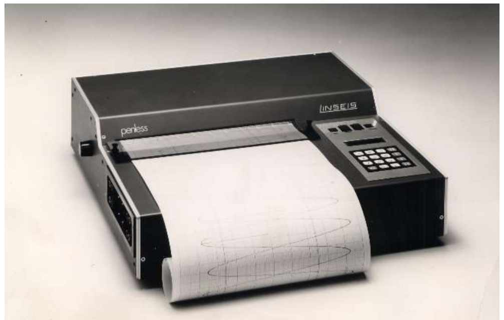
Linseis Messgeräte GmbH in Germany

- **Founded in 1956 by Dr. Maximilian Linseis** in Selb (Bavaria/Germany)
- **Production in Selb/Germany,** subsidiaries in the USA, China, India, more than 65 distributor worldwide

Dr. Maximilian Linseis

Dipl. Phys. Claus Linseis and M.Sc. Florian Linseis

Dr. Ing. Vincent Linseis



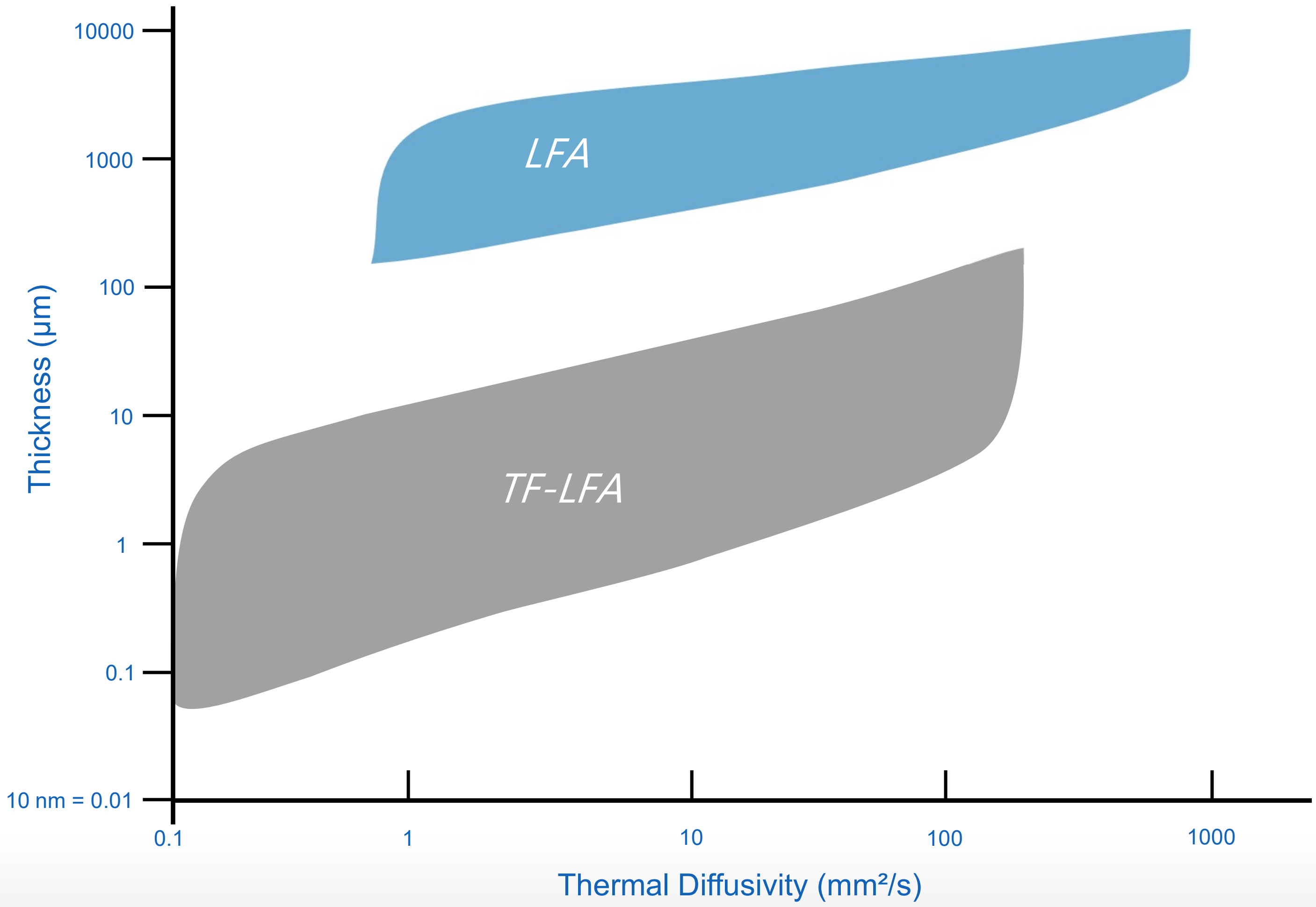
Linseis started up with Data Logger

Dilatometer Lab in Germany

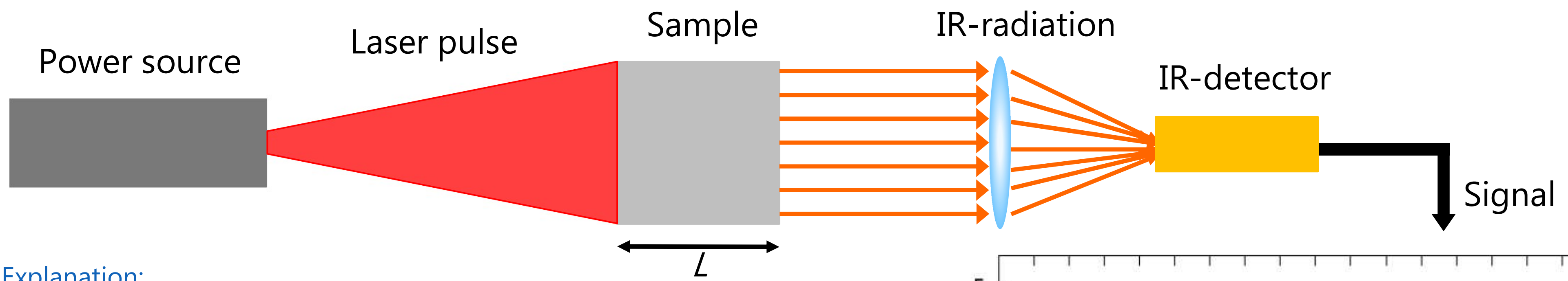
Business area:

- Laboratory instruments for thermal analysis, thermophysics and dilatometry
- Service Lab

Laser Flash Measurement ranges



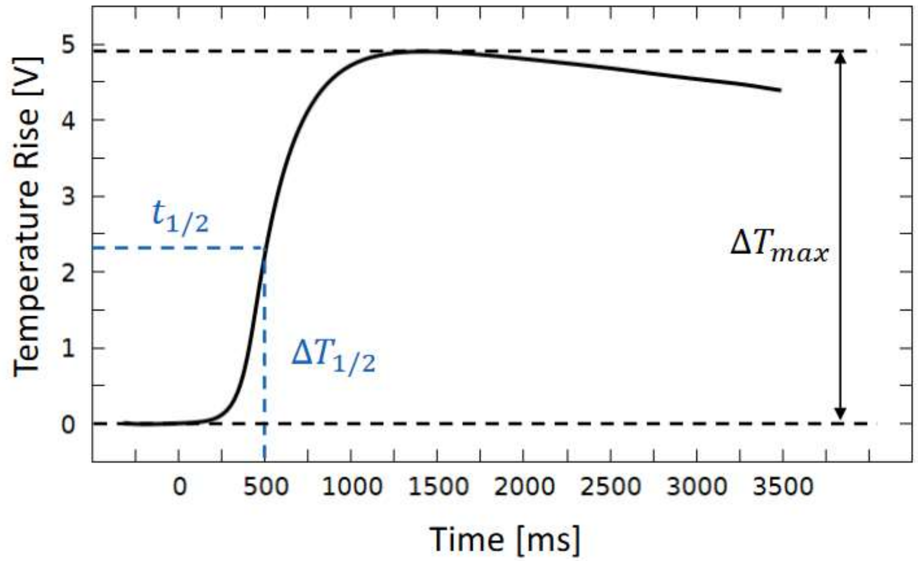
Laser Flash– Working Principal



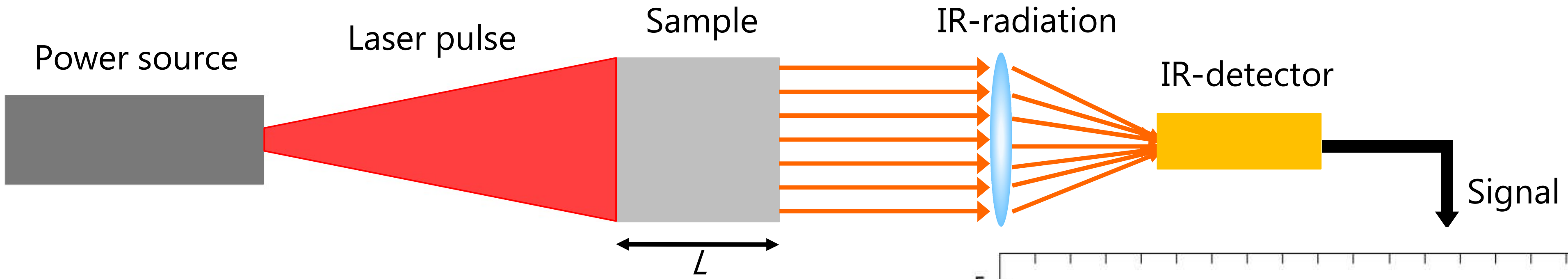
Explanation:

- Short and high intensity Laser pulse excites (heats) the front side of the sample
- Energy is absorbed by the sample
- Thermal wave propagates through the sample to its rear side
- Thermal energy emitted via radiation with a wavelength in the IR-range
- IR-Detector detects the signal as a function of time
- Signal corresponds to the temperature rise at the rear face
- Time required to reach the half of the maximum temperature: $t_{1/2}$

➢ Time $t_{1/2}$ + thickness of the sample L= Thermal diffusivity $\boldsymbol{\alpha}$



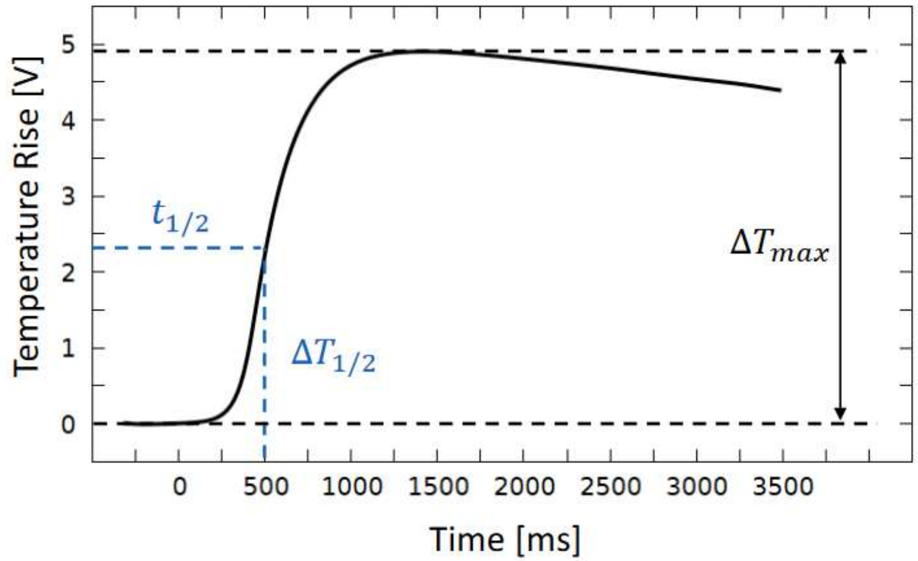
Laser Flash– Working Principal



Determination of the thermal diffusivity with

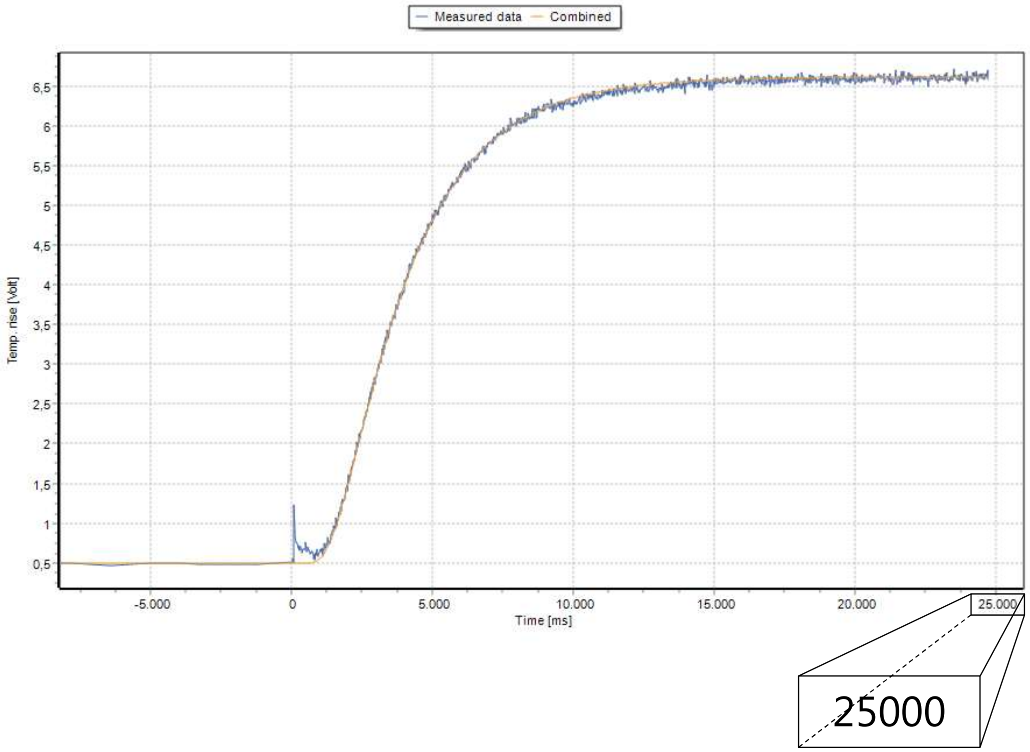
$$\alpha = 0.13879 \cdot \frac{L^2}{t_{1/2}}$$

α = Thermal diffusivity [m^2/s], L = Sample height [m], $t_{1/2}$ = Half time rise [s]

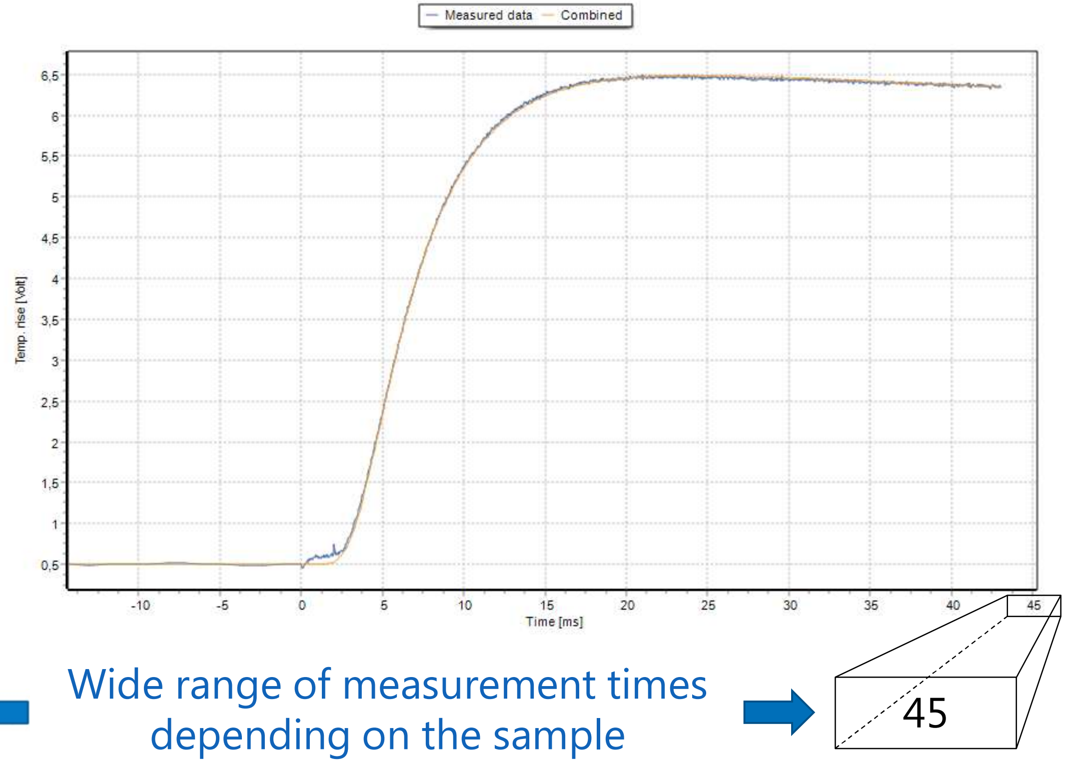


LFA – Time Domain

"Slow" Measurement



"Fast" Measurement



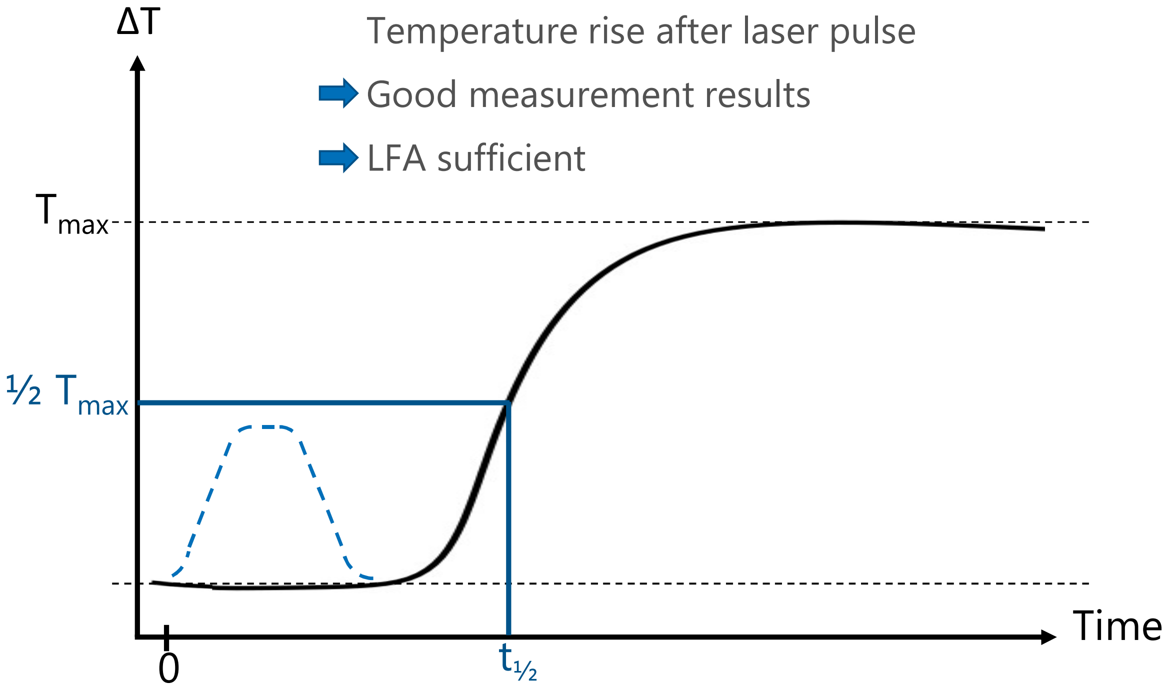
Wide range of measurement times depending on the sample

LFA – Limitations in the Time Domain

Minimal sample thickness depends on:

1. Acquisition rate of the instrument/detector
   (number of measurement points)

2. Duration of the laser pulse
   (overlay of laser pulse and resulting sample temperature rise)



LFA – Limitations in the Time Domain

Minimal sample thickness depends on:

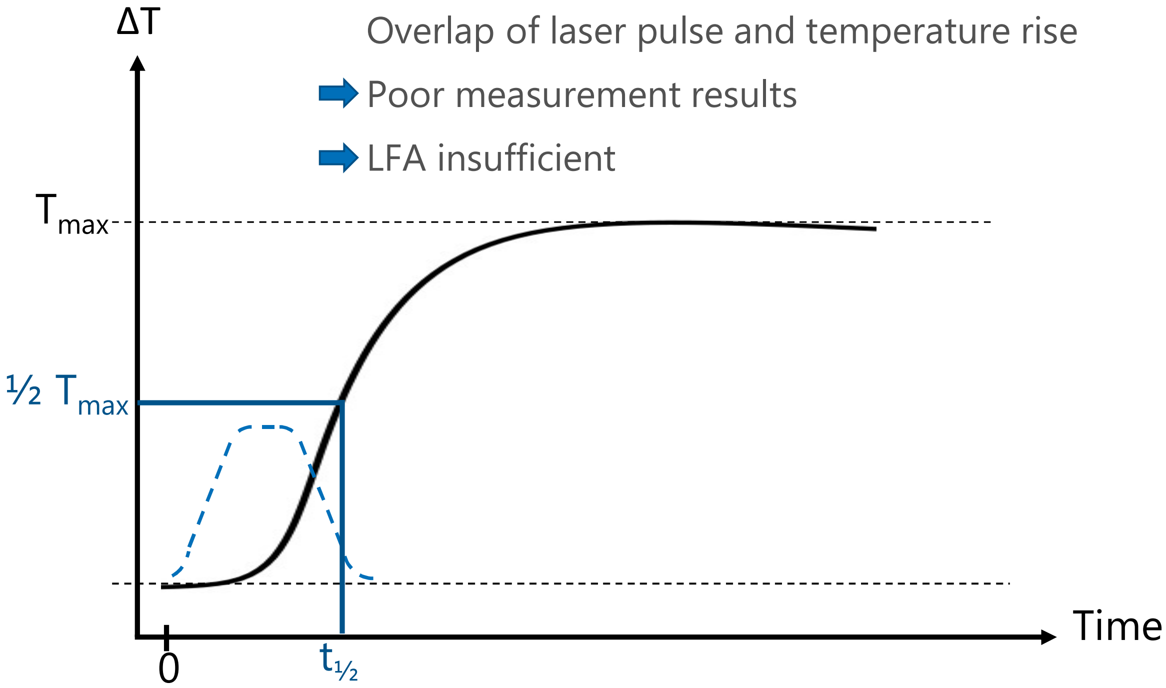
1. Acquisition rate of the instrument/detector

   (number of measurement points)

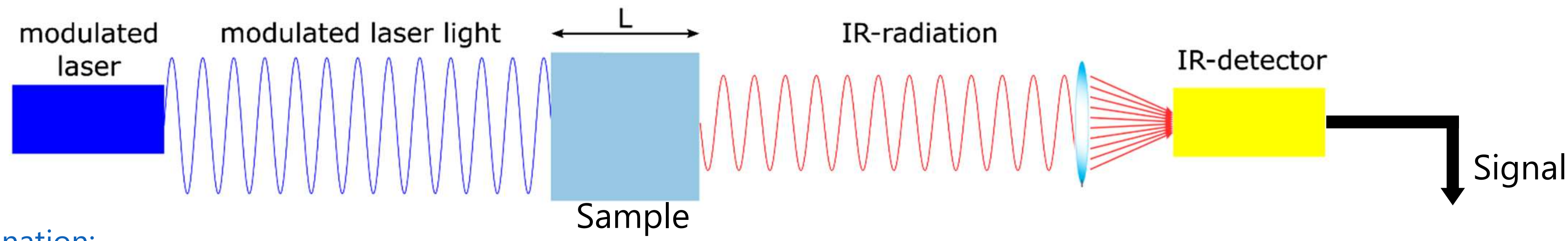
2. Duration of the laser pulse

   (overlay of laser pulse and resulting sample temperature rise)

- ➢ Need for a different approach!
- ➢ Periodic Laser Heating

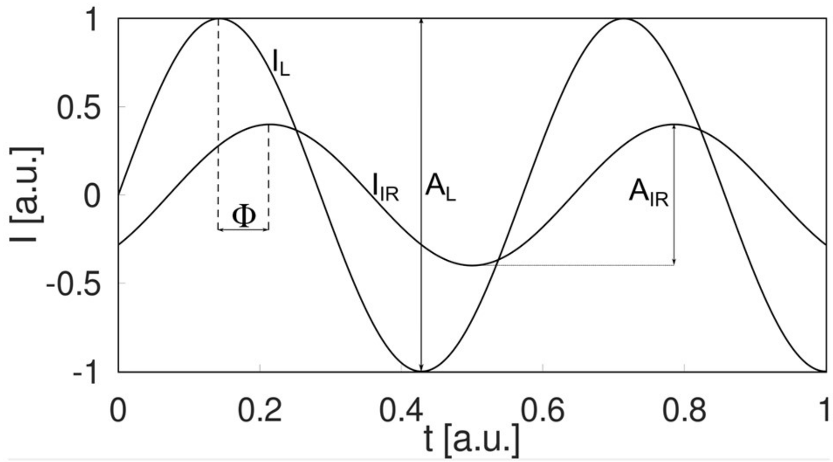


Periodic Laser Heating (PLH) – Working Principal



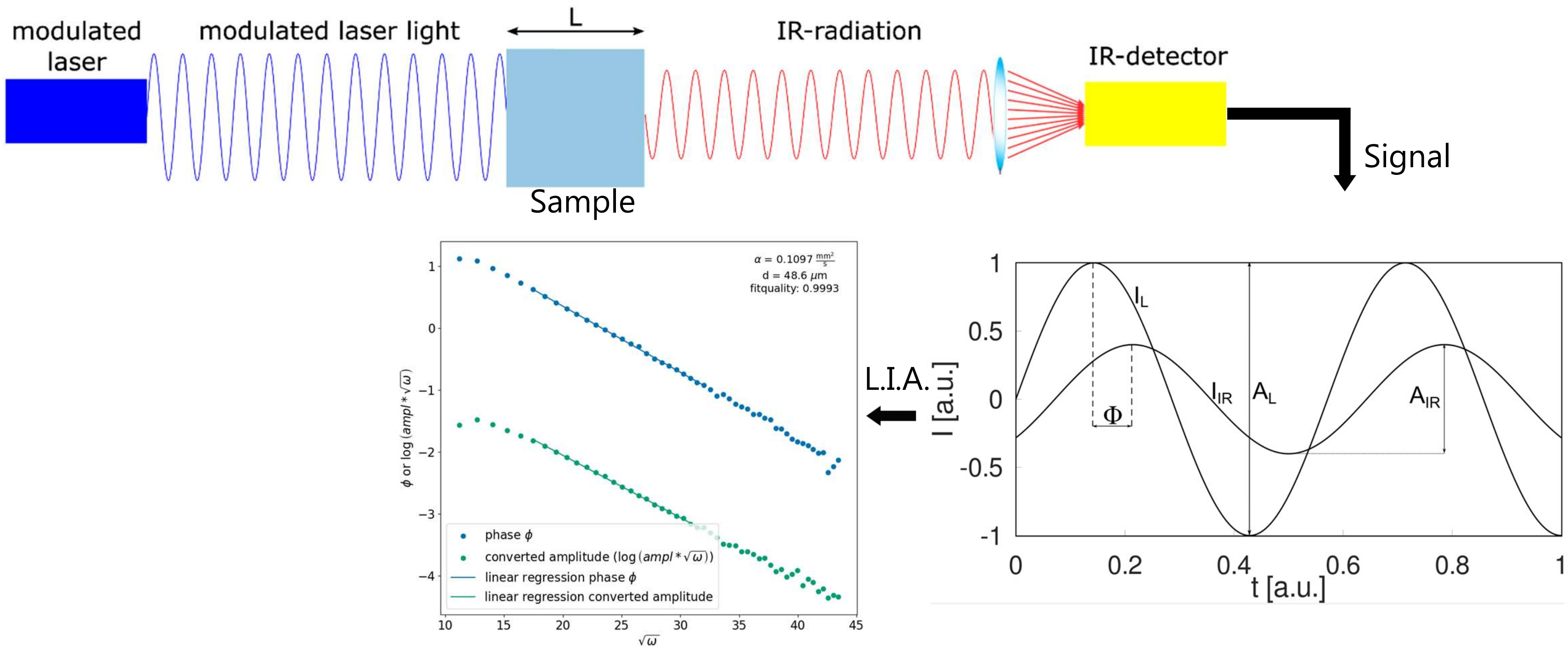
Explanation:

- Periodic modulated laser beam excites (heats) the front side of the sample
- Energy is absorbed by the sample
- Thermal wave propagates through the sample to its rear side
- Thermal energy is emitted via radiation with a wavelength in the IR-range
- IR-Detector detects the signal which is amplified by a Lock-In-Amplifier
- Amplitude and phase shift is monitored
- Frequency of the laser is tuned and Phase and Amplitude is saved

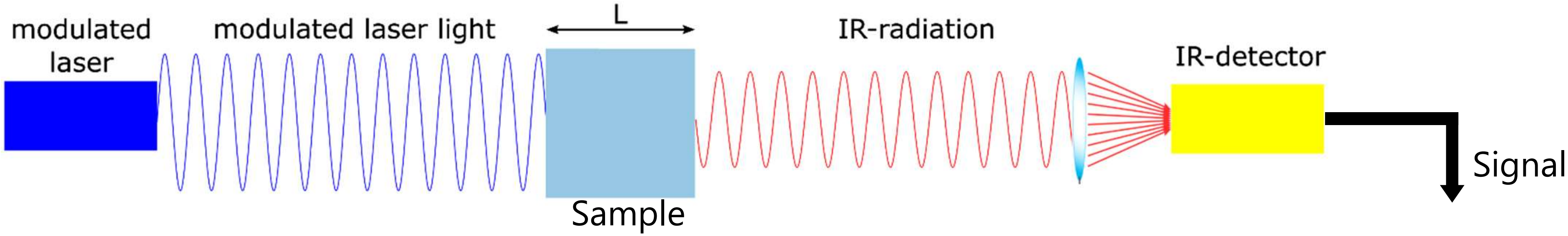


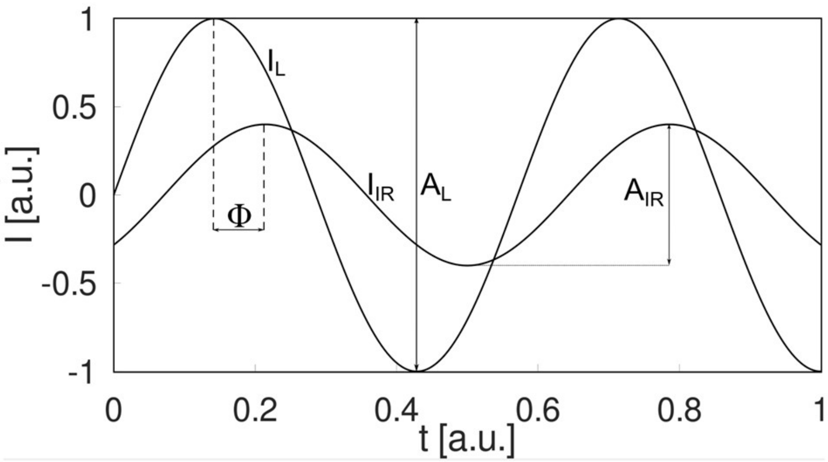
→ Measurement in the frequency domain

Periodic Laser Heating (PLH) – Working Principal

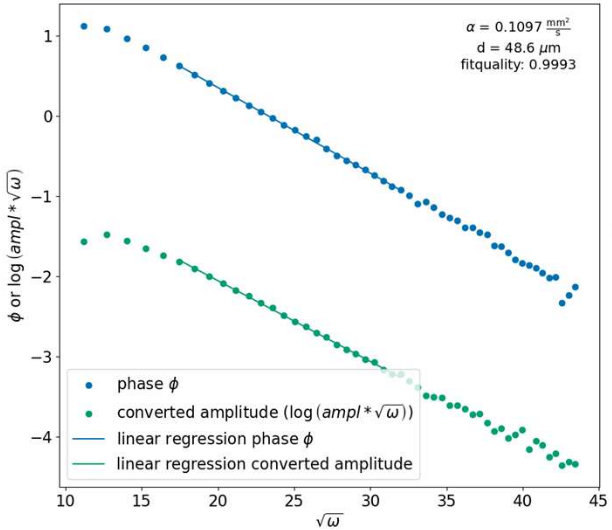


Periodic Laser Heating (PLH) – Working Principal





L.I.A.



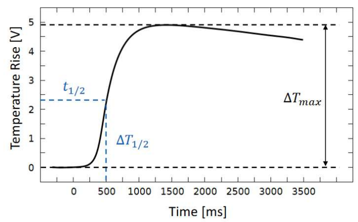
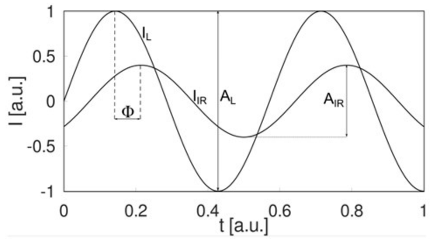
Calculation of the thermal diffusivity with

$$\alpha_{\phi,amp} = \frac{L^2}{2m_{\phi,amp}^2}$$

α = Thermal diffusivity [m^2/s],
L = Sample height [m],
m = Slope of the linear range [$\sqrt{s}$]

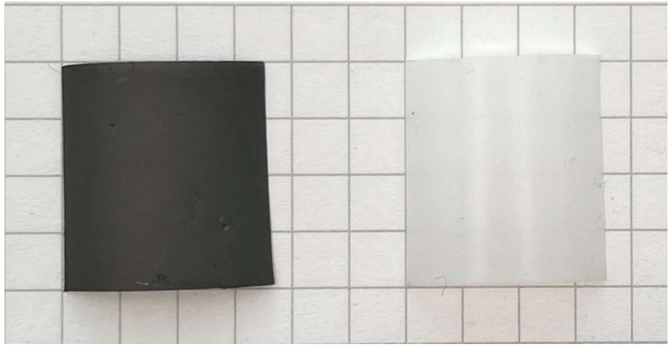
$$\alpha = \sqrt{\alpha_\phi \alpha_{amp}}$$

LFA & PLH Comparison

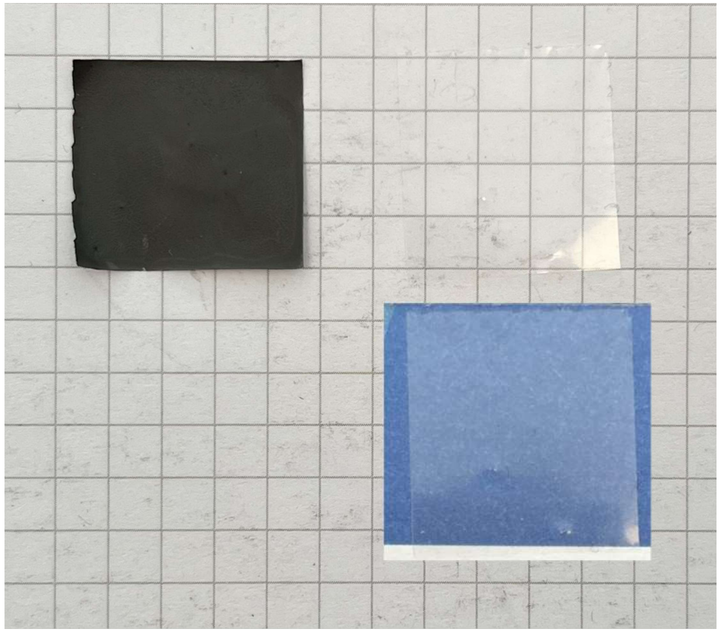
LFA	PLH
• Sample is subjected by thermal disturbance (Pulse) • Disturbance is observed as function of time **(Time Domain)** • Typical measurement range: mm	• Sample is subjected by a periodic thermal disturbance • Disturbance is observed as a function of frequency **(Frequency Domain)** • Typical measurement range: µm
+ Short measurement time + Broad λ-range + Broad temperature range - Complicated theory 	+ Thinner samples + "Model free" evaluation - Little bit longer measurements - Limited temperature range 

Measurement Examples

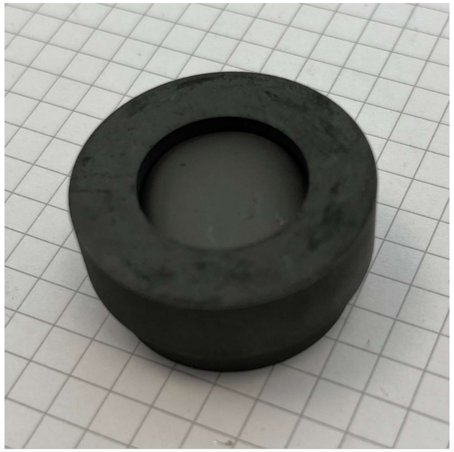
PTFE foil (100 µm)



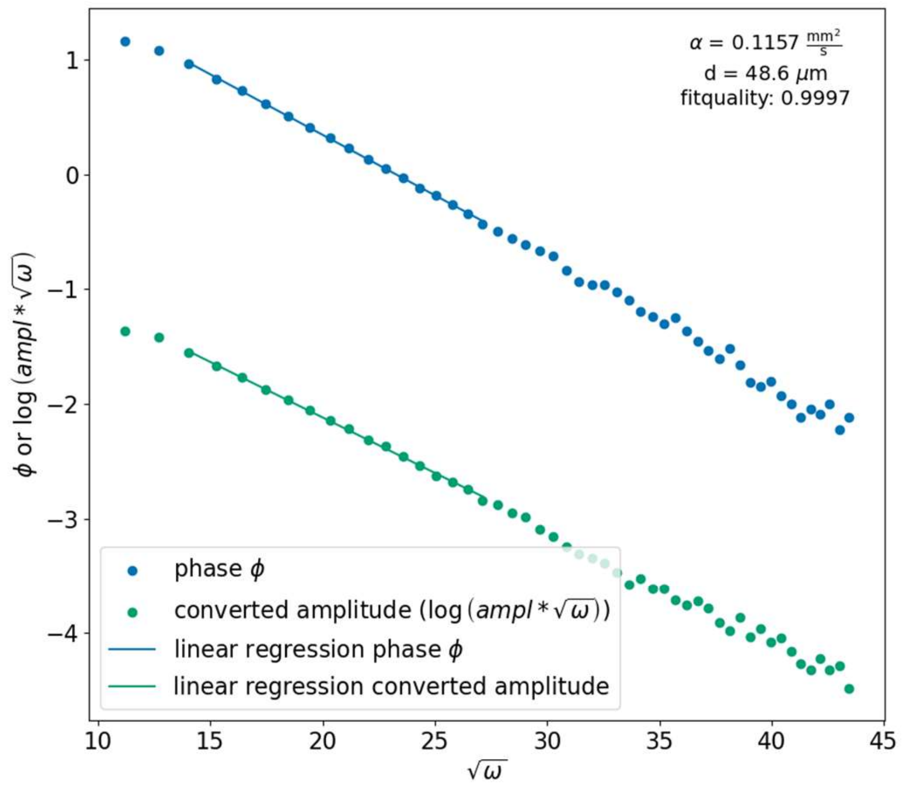
PE foil (25 µm)

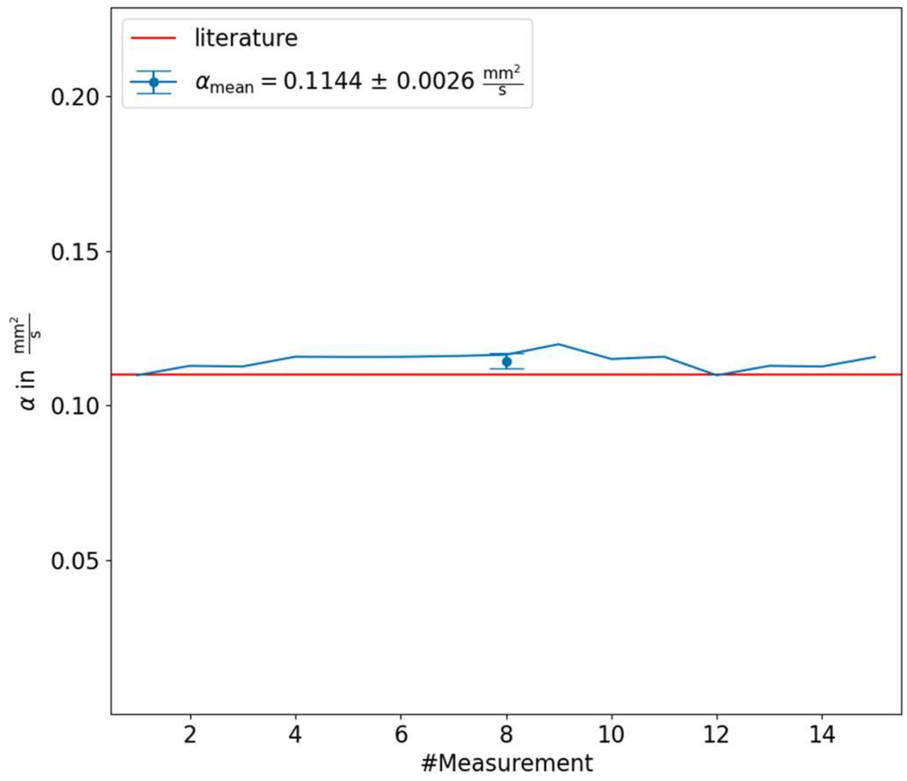


Samples have to be sprayed or coated with carbon

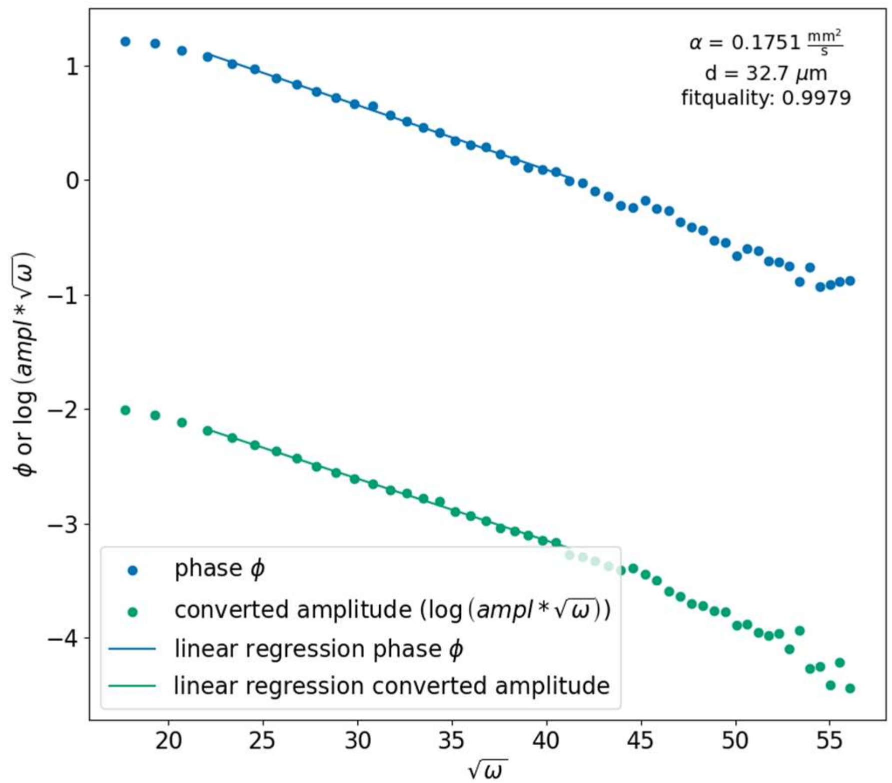


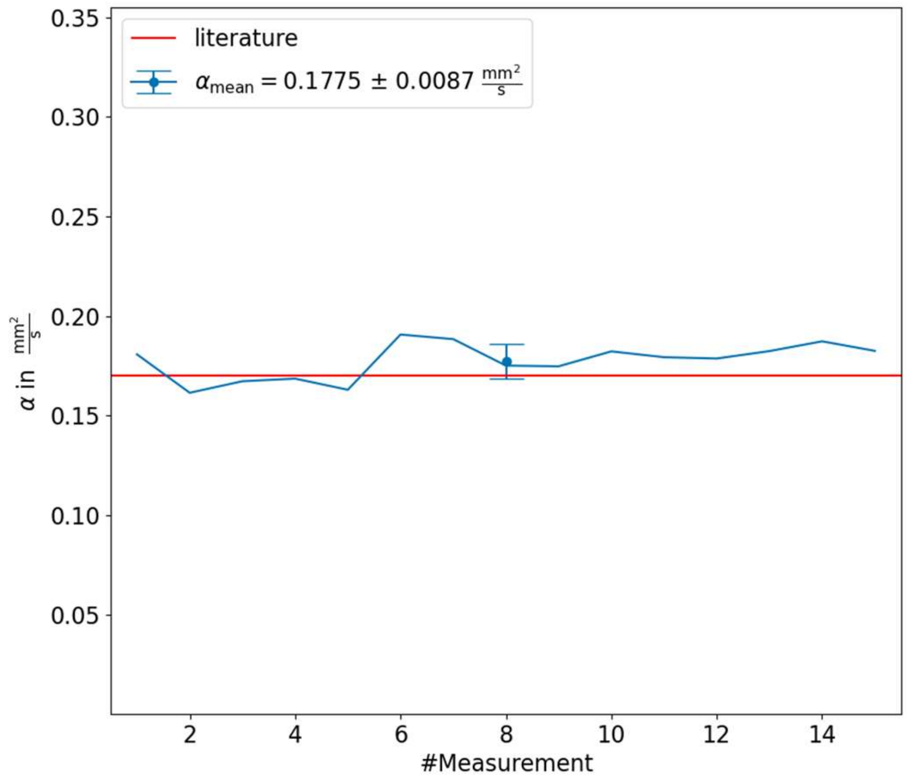
Measurements – Polymers
Polytetrafluoroethylene (PTFE) 50 µm



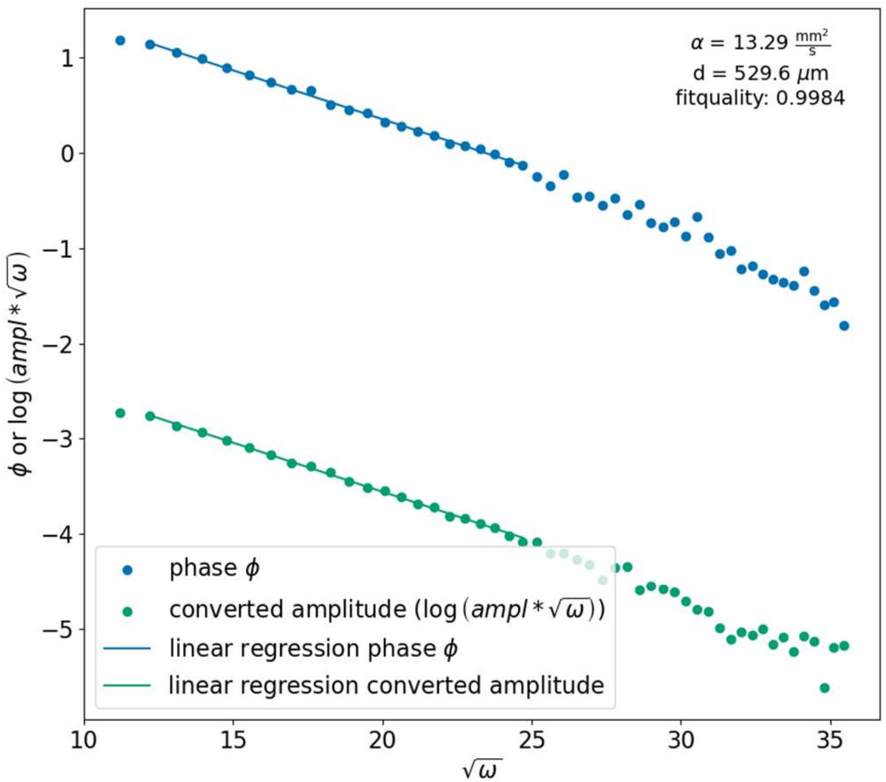


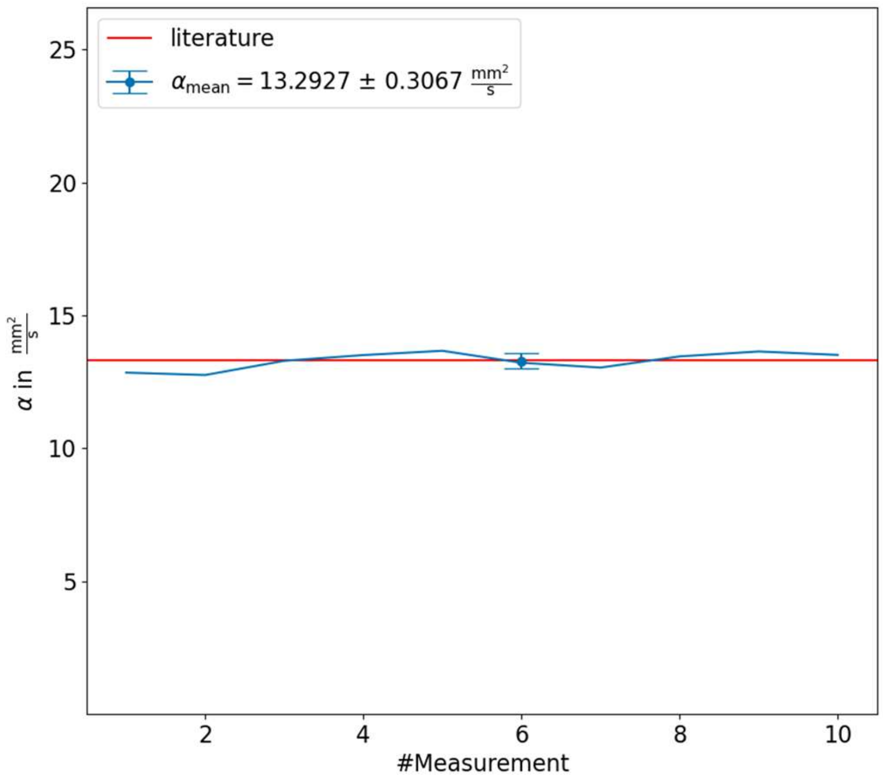
Measurements – Polymers: Polyethylene (PE) 25 µm



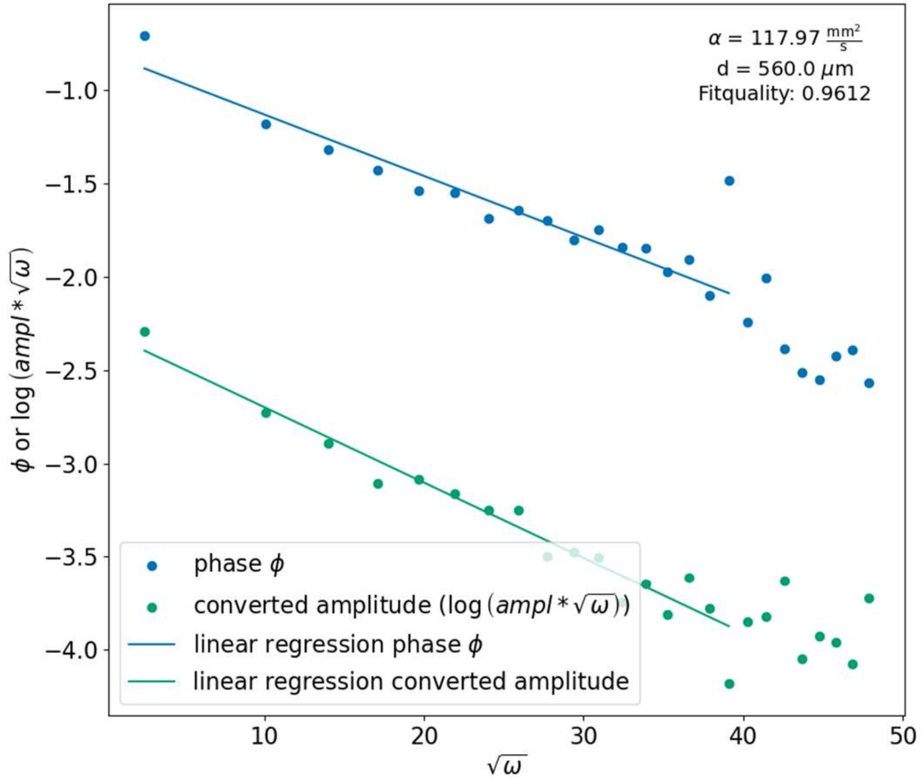


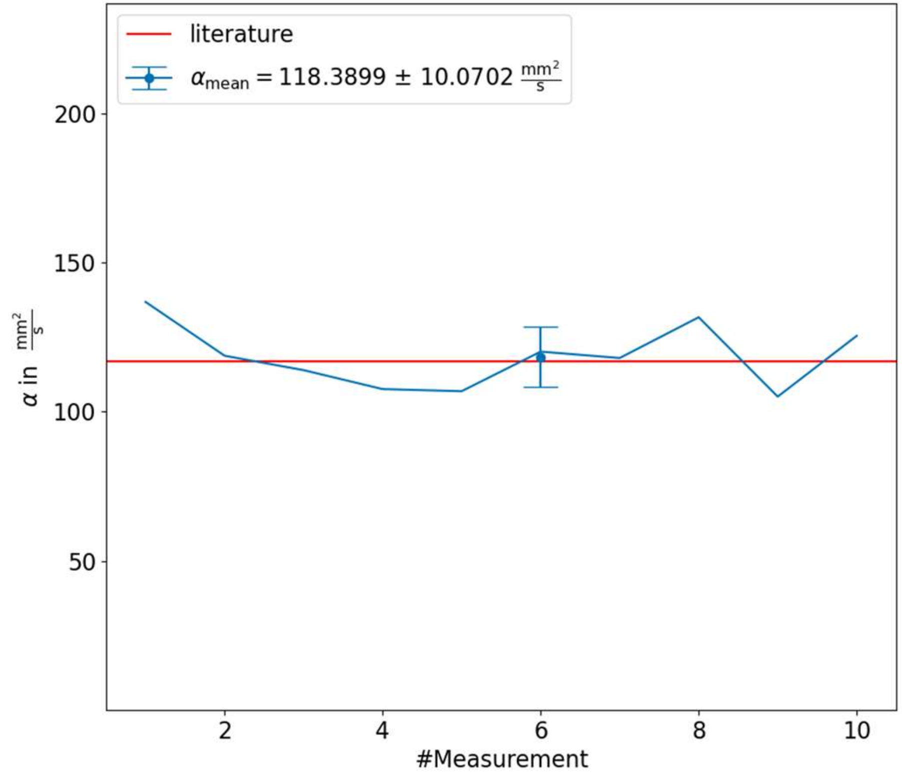
Measurements – Ceramics: Sapphire 500 µm



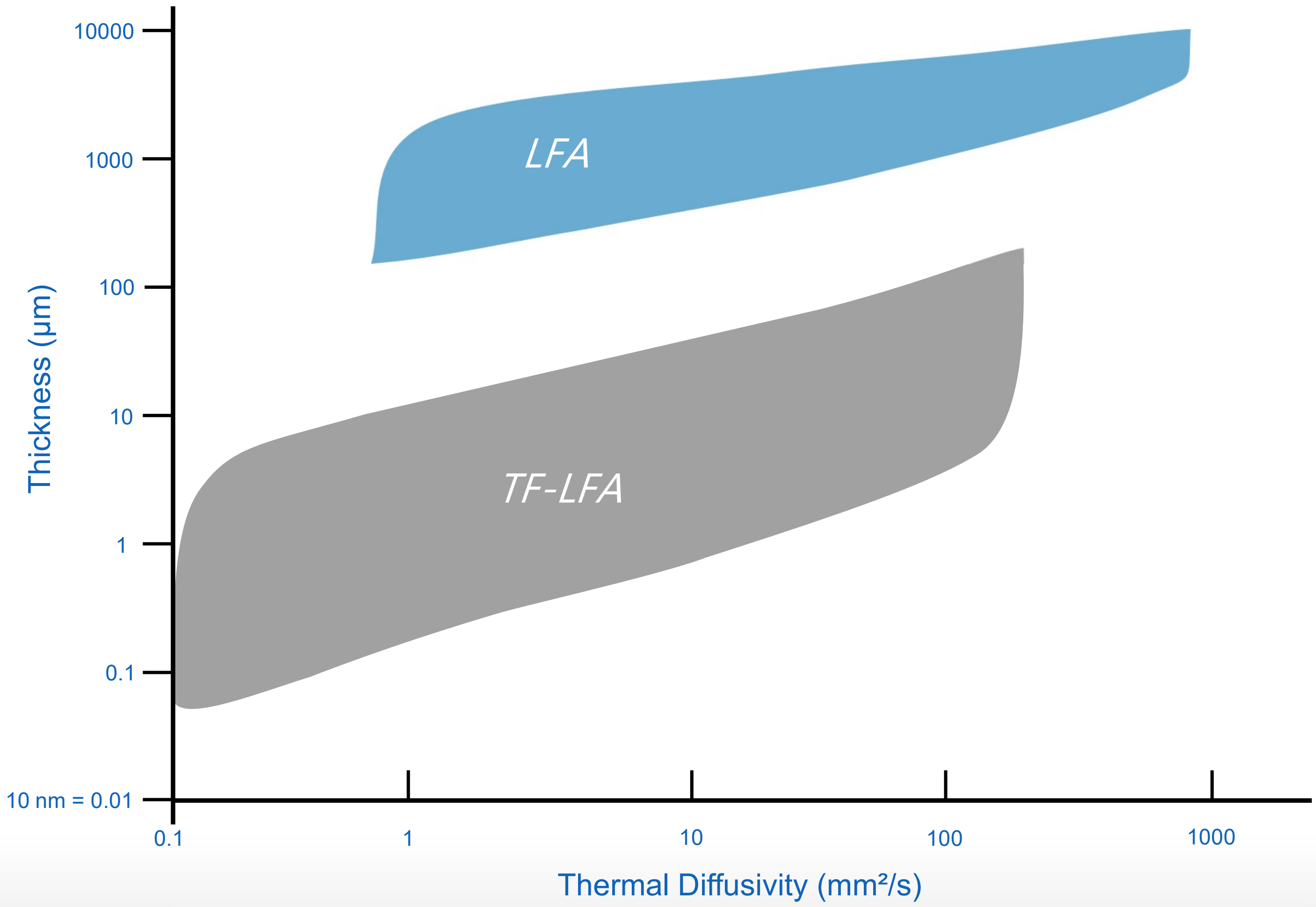


Measurements – Metals: Copper 500 µm





Laser Flash Measurement ranges



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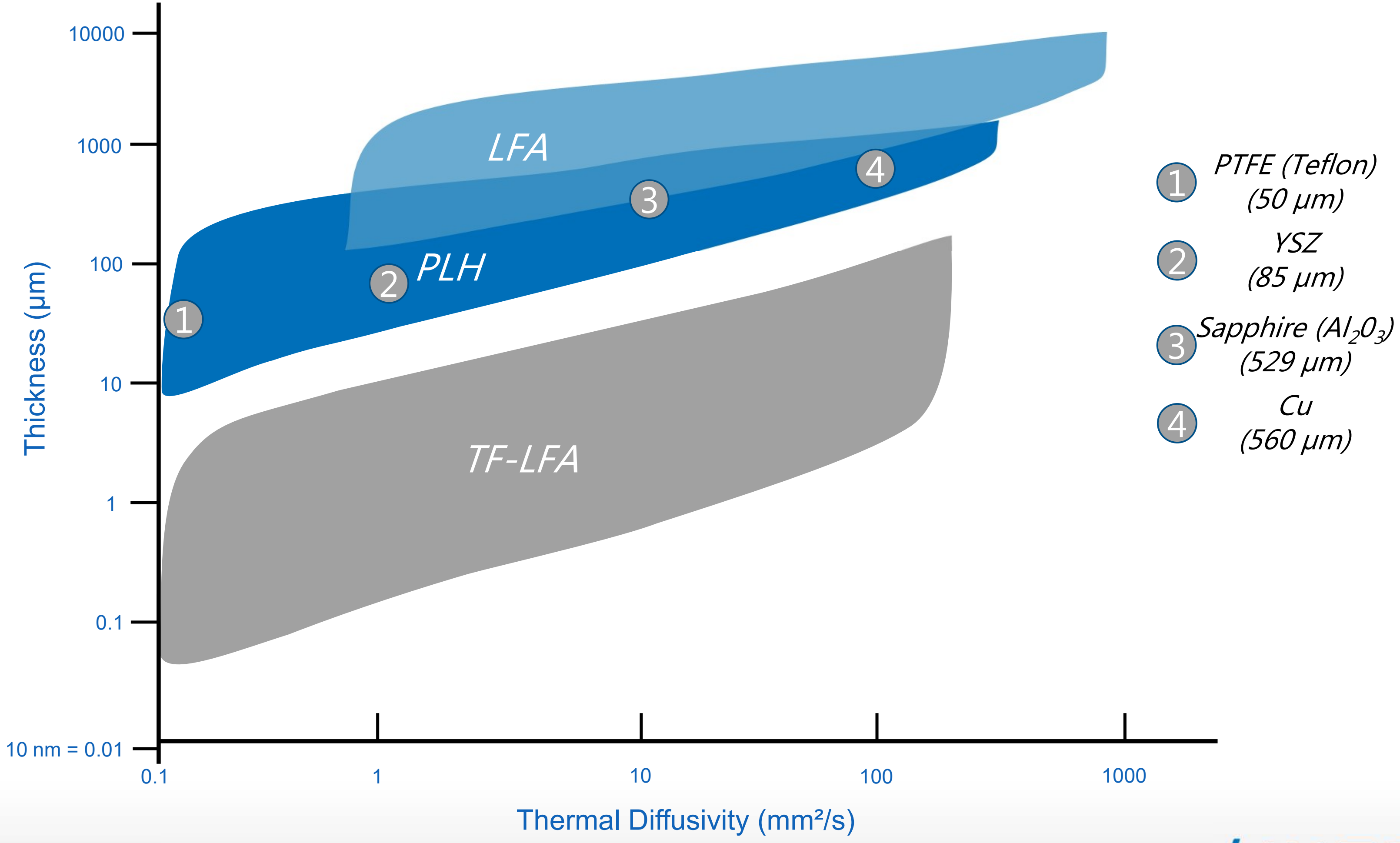
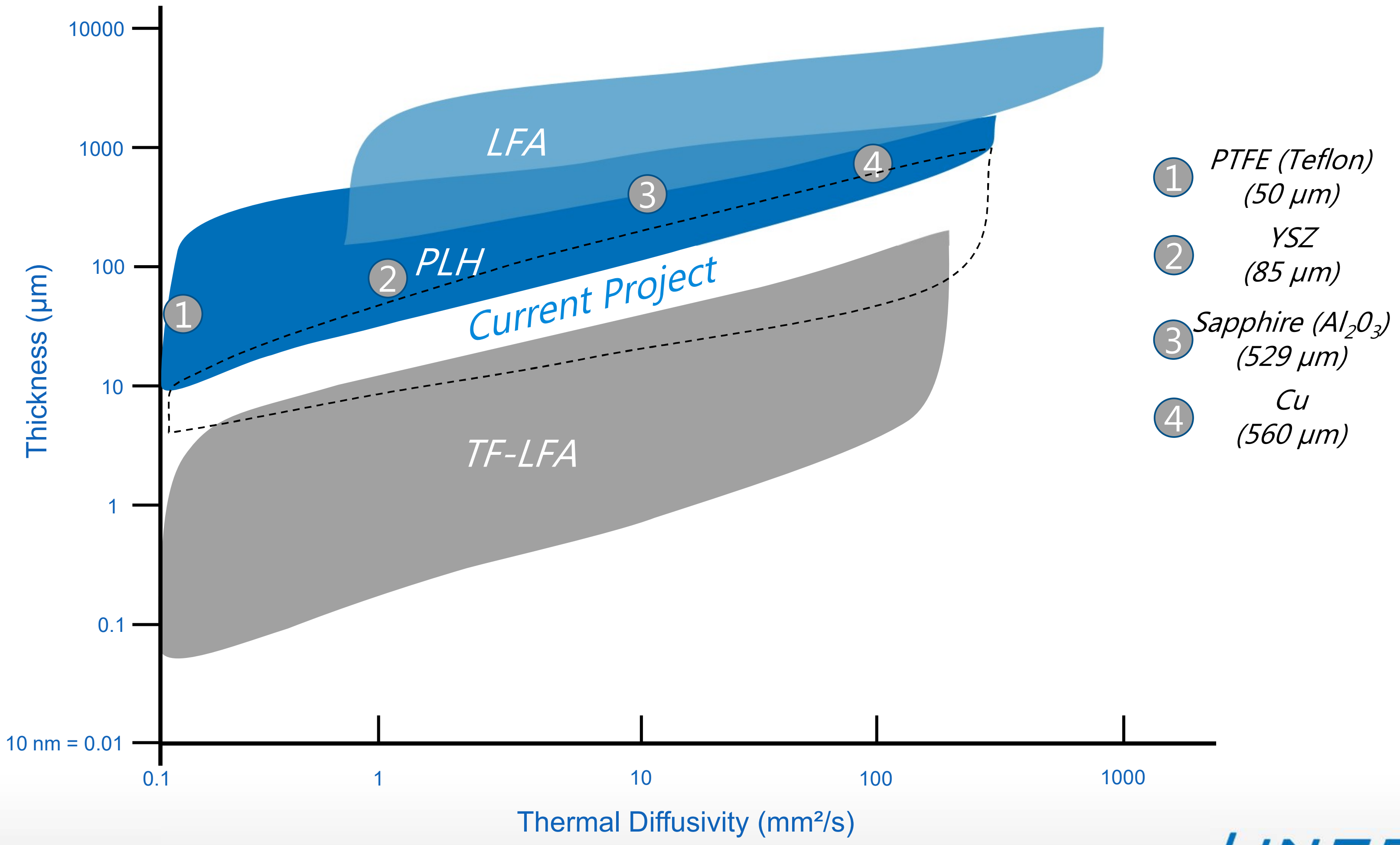
PLH – Extension for µm sample thicknesses



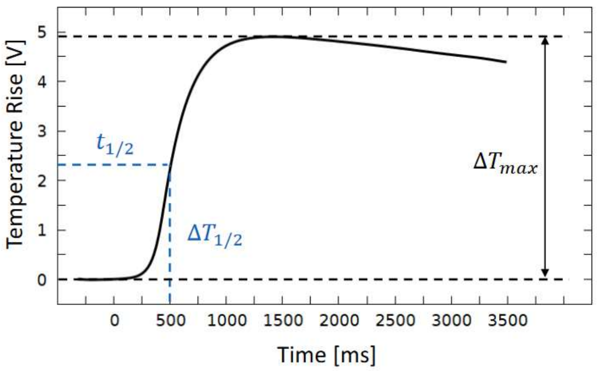
PLH – Extension for µm sample thicknesses



Linseis Product Lineup

Laser Flash Analyzer

- PLH add-on for Laser/Xenon-Flash instruments



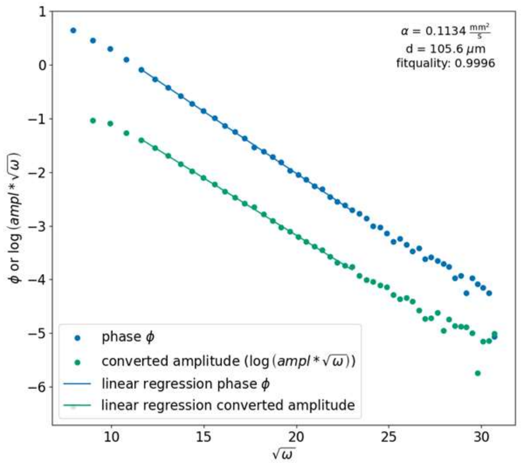
LFA + PLH (combined Version)

- Two measurement techniques combined in one instrument
- Same outer dimensions
- 2 in 1 measurement system

- Worldwide unique combined measurement system
  - ➢ *Combination Patent pending*
- Broadest measurement range

Periodic Laser Heating

- New LFA can be upgraded with PLH option



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Specification PLH

Periodic Laser-Heating | PLH

Most advanced tool
Free standing films, membranes and more
Automatic sample throughput
10 – 500 µm / Up to +300°C
Up to 5 W cw power
Model free evaluation

Specification LFA & PLH

Combined Solution | **LFA & PLH**

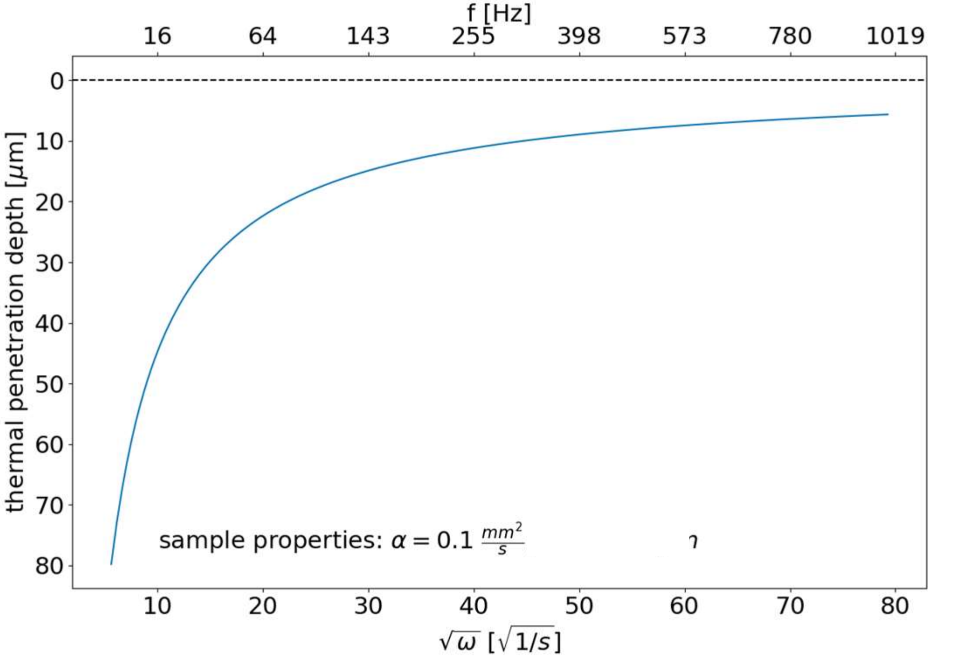
Unique combination
Solid, liquids, pastes, powders, PCM, Free standing films, membranes
Automatic sample throughput
10 – 6000 µm / Up to +2800°C
Up to 25J/puls / 5 W cw power
Advanced Evaluation

PLH – Theoretical Considerations

Thermal Penetration Depth (TPD) μ:

$$\mu = \sqrt{\frac{\alpha}{\pi f}} = \sqrt{\frac{2\alpha}{\omega}}$$

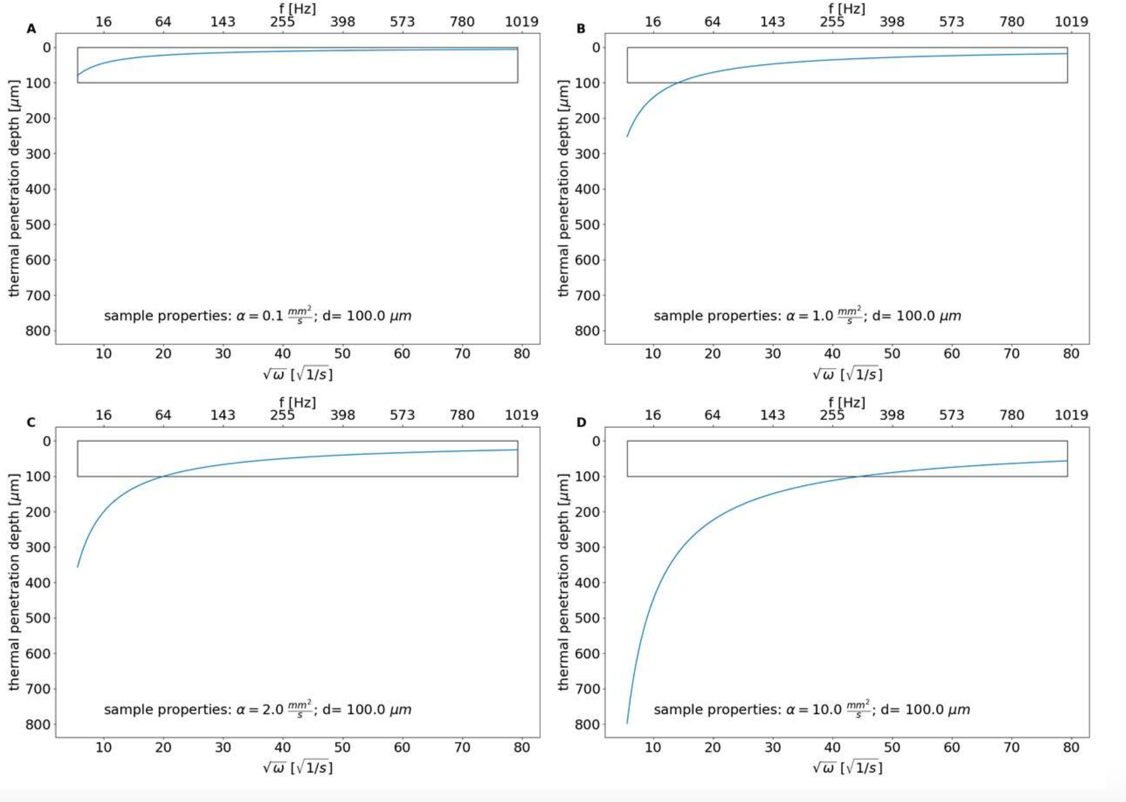
α : thermal diffusivity $\left[\frac{\mathrm{mm}^2}{\mathrm{s}}\right]$
f: frequency [Hz]
ω: angular frequency [rad]



Keep in mind: The higher the modulation frequency gets, the less the laser radiation penetrates the sample

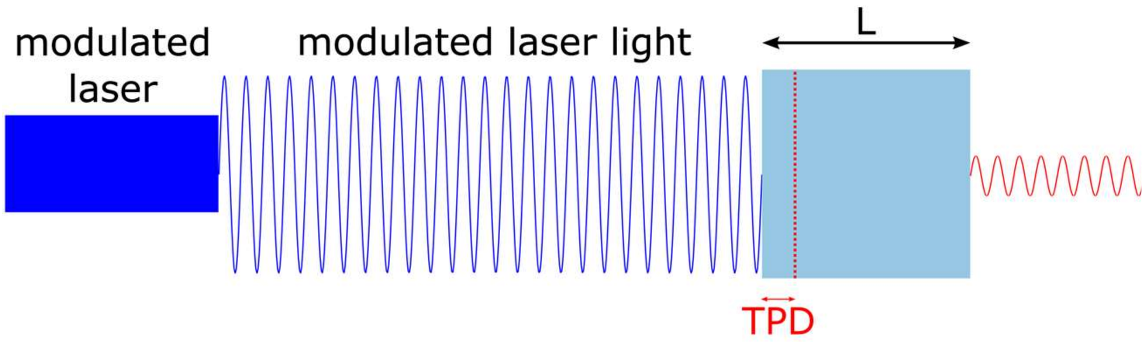
PLH – Theoretical Considerations

Different theoretical sample thermal diffusivities (sample thicknesses are the same):



PLH – Theoretical Considerations

High frequency *f*:



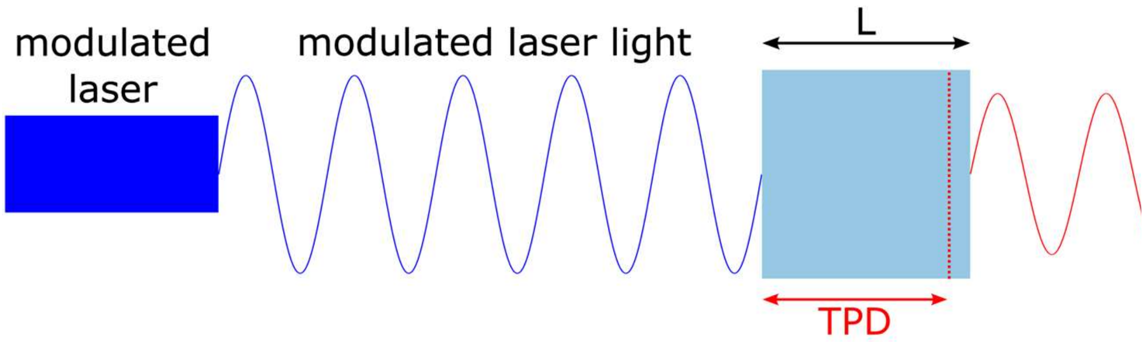
➡ Higher modulation frequency *f*

➡ Lower thermal penetration depth μ

$$\mu \sim \sqrt{\frac{1}{f}}$$

PLH – Theoretical Considerations

Low frequency *f*:



➡ Lower modulation frequency f

➡ Higher thermal penetration depth μ

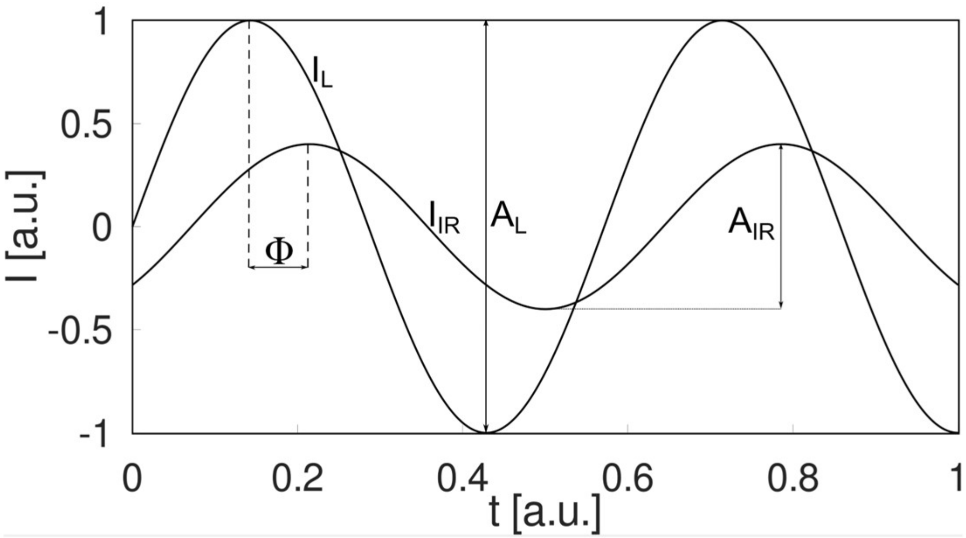
$$\mu \sim \sqrt{\frac{1}{f}}$$

Theory PLH – Data Evaluation

Frequency dependence of phase shift and amplitude:

$$\Delta\phi = -\sqrt{\frac{\omega}{2\alpha}}d - \frac{\pi}{4}$$

$$Amp = A_L \frac{1}{\sqrt{\omega e}} \exp\left(-\frac{d}{\mu}\right);\ \mu = \sqrt{\frac{2\alpha}{\omega}}$$



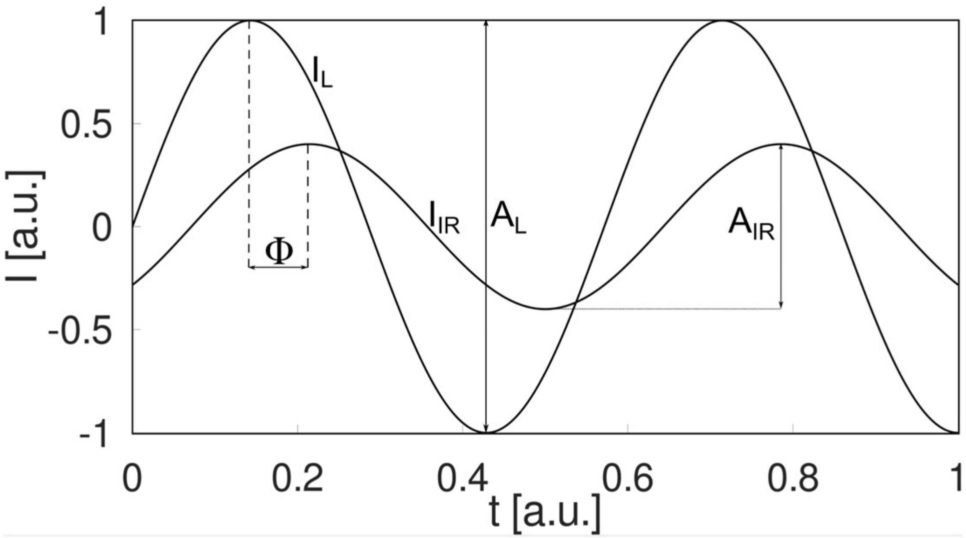
Theory PLH – Data Evaluation

Frequency dependence of phase shift and amplitude :

$$\Delta\phi = -\sqrt{\frac{\omega}{2\alpha}}d - \frac{\pi}{4} = -\frac{d}{\sqrt{2\alpha}}\sqrt{\omega} + c_1$$

$$\Rightarrow Phase\ vs.\sqrt{frequency}$$

$$Amp = A_L \frac{1}{\sqrt{\omega e}} \exp\left(-\frac{d}{\mu}\right)$$



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$$\Rightarrow \ Phase\ vs.\sqrt{frequency}$$

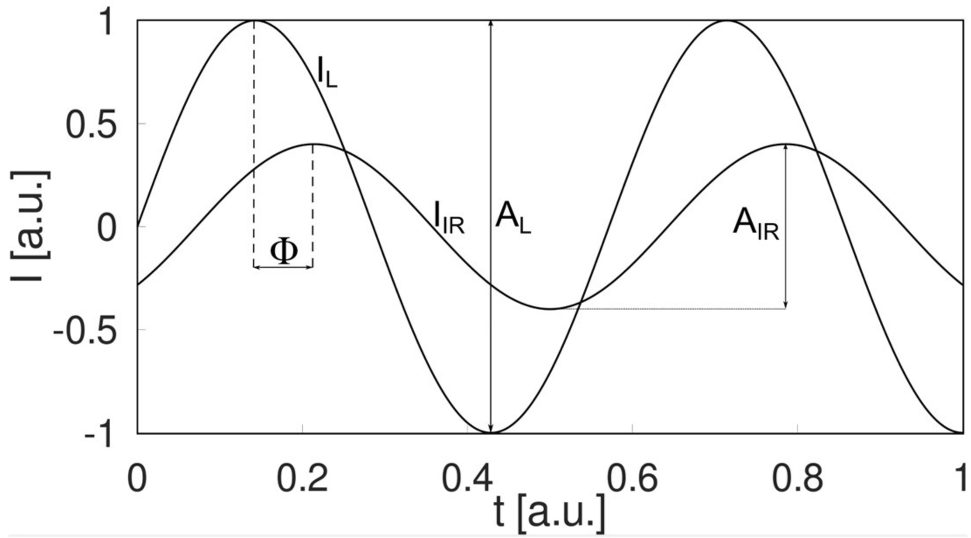
$$Amp = A_L \frac{1}{\sqrt{\omega e}} \exp\left(-\frac{d}{\mu}\right);\ \mu = \sqrt{\frac{2\alpha}{\omega}}$$

$$\vdots$$

$$\log(Amp * \sqrt{\omega}) = -\frac{d}{\sqrt{2\alpha}}\sqrt{\omega} + \log(A_L) - \log(e)$$

$$\log(Amp * \sqrt{\omega}) = \ -\frac{d}{\sqrt{2\alpha}}\sqrt{\omega} + c_2$$

$$\Rightarrow \ converted\ amplitude\ vs.\sqrt{frequency}$$



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Theory PLH – Data Evaluation

Frequency dependence of phase shift and amplitude :

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$$Amp = A_L \frac{1}{\sqrt{\omega e}} \exp\left(-\frac{d}{\mu}\right);\ \mu = \sqrt{\frac{2\alpha}{\omega}}$$

$$\vdots$$

$$\log(Amp * \sqrt{\omega}) = -\frac{d}{\sqrt{2\alpha}}\sqrt{\omega} + \log(A_L) - \log(e)$$

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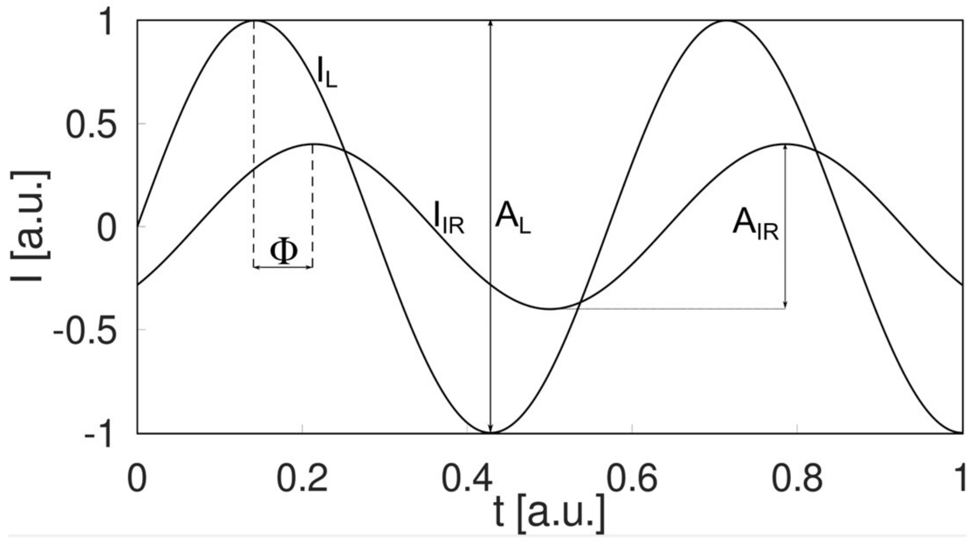
$$\Rightarrow \; converted\ amplitude\ vs.\sqrt{frequency}$$



Theory PLH – Data Evaluation

Slopes are the same and contain the thermal diffusivity:

$$\Delta\phi = -\sqrt{\frac{\omega}{2\alpha}}d - \frac{\pi}{4} = -\frac{d}{\sqrt{2\alpha}}\sqrt{\omega} + c_1$$

$$\Rightarrow \mathit{Phase\ vs.}\sqrt{\mathit{frequency}}$$

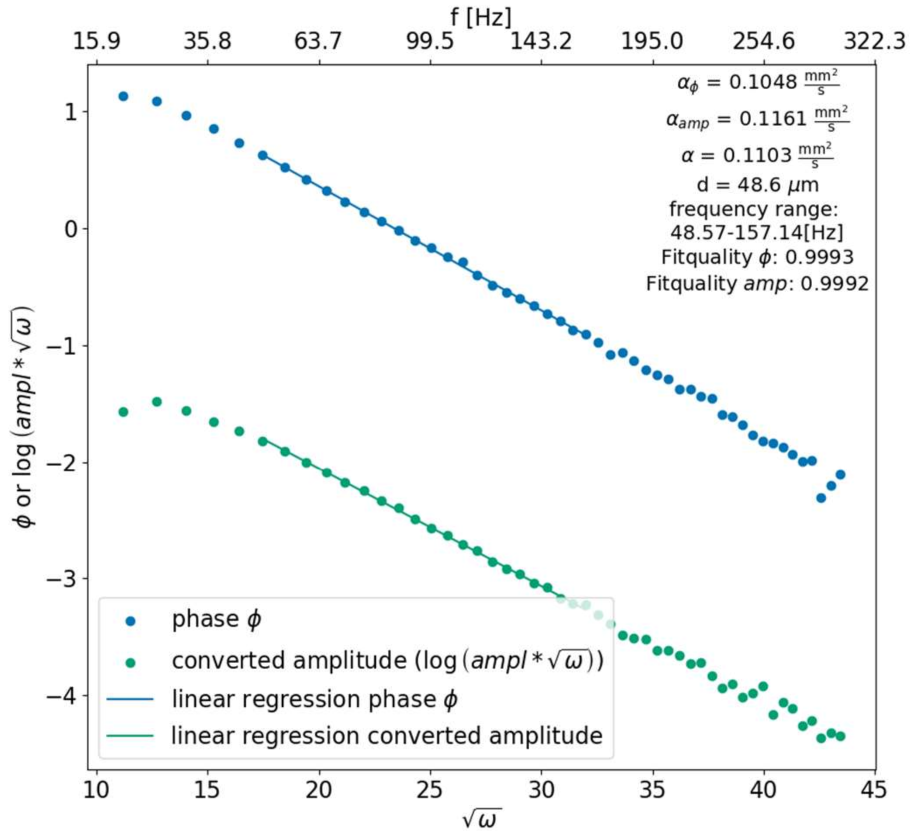
$$Amp = A_L \frac{1}{\sqrt{\omega e}} \exp\left(-\frac{d}{\mu}\right);\ \mu = \sqrt{\frac{2\alpha}{\omega}}$$

$$\vdots$$

$$\log(Amp * \sqrt{\omega}) = -\frac{d}{\sqrt{2\alpha}}\sqrt{\omega} + \log(A_L) - \log(e)$$

$$\log(Amp * \sqrt{\omega}) = -\frac{d}{\sqrt{2\alpha}}\sqrt{\omega} + c_2$$

$$\Rightarrow \mathit{converted\ amplitude\ vs.}\sqrt{\mathit{frequency}}$$



Theory PLH – Data Evaluation

Slopes are the same and contain the thermal diffusivity:

$$\Delta\phi = -\sqrt{\frac{\omega}{2\alpha}}d - \frac{\pi}{4} = -\frac{d}{\sqrt{2\alpha}}\sqrt{\omega} + c_1 = -m_\phi\sqrt{\omega} + c_1$$

$$\Rightarrow \; Phase\ vs.\sqrt{frequency}$$

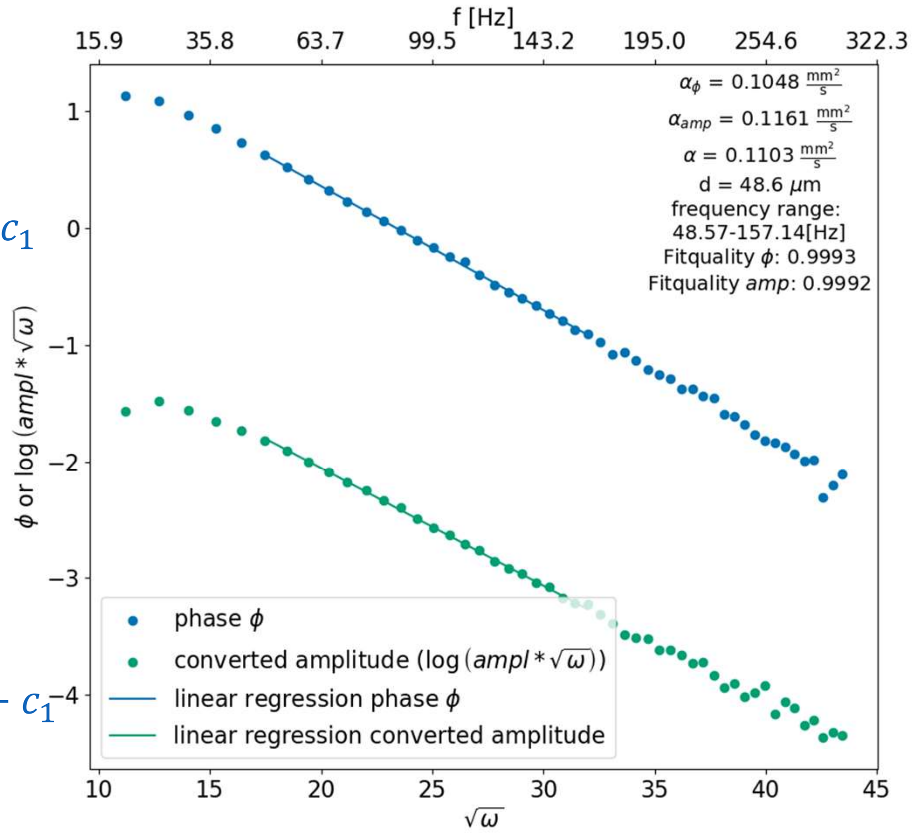
$$Amp = A_L \frac{1}{\sqrt{\omega e}} \exp\left(-\frac{d}{\mu}\right);\ \mu = \sqrt{\frac{2\alpha}{\omega}}$$

$$\vdots$$

$$\log(Amp * \sqrt{\omega}) = -\frac{d}{\sqrt{2\alpha}}\sqrt{\omega} + \log(A_L) - \log(e)$$

$$\log(Amp * \sqrt{\omega}) = -\frac{d}{\sqrt{2\alpha}}\sqrt{\omega} + c_2 = -m_{amp}\sqrt{\omega} + c_1$$

$$\Rightarrow \; converted\ amplitude\ vs.\sqrt{frequency}$$



Theory PLH – Data Evaluation

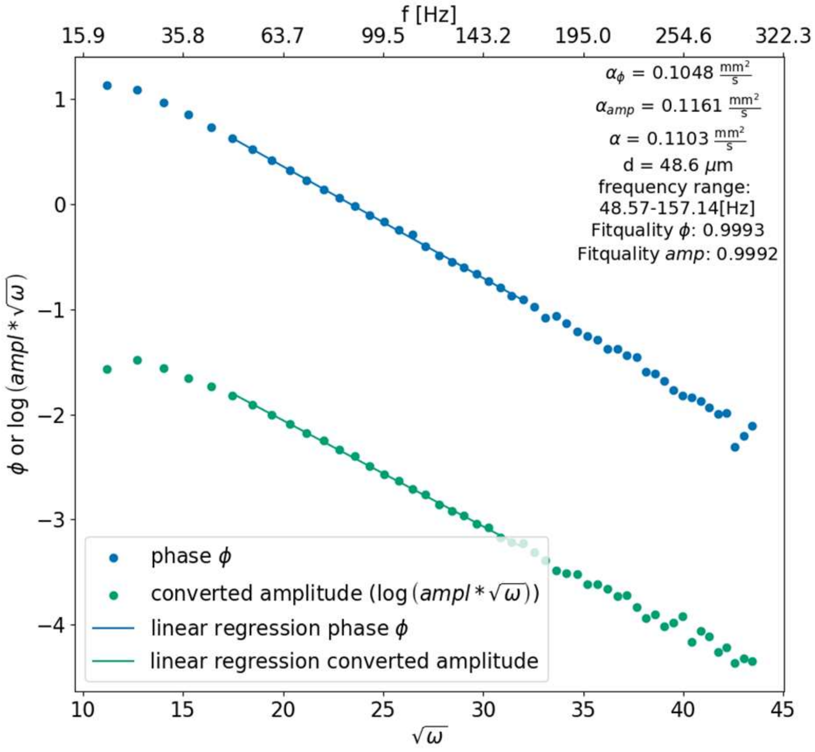
linearized form:

$$\Delta\phi = -\sqrt{\frac{\omega}{2\alpha}}d - \frac{\pi}{4} = -\frac{d}{\sqrt{2\alpha}}\sqrt{\omega} + c_1 = -m_\phi\sqrt{\omega} + c_1$$

$$\Rightarrow \; Phase\ vs.\sqrt{frequency}$$

$$\log(Amp * \sqrt{\omega}) = \; -\frac{d}{\sqrt{2\alpha}}\sqrt{\omega} + c_2 = -m_{amp}\sqrt{\omega} + c_1$$

$$\Rightarrow \; converted\ amplitude\ vs.\sqrt{frequency}$$

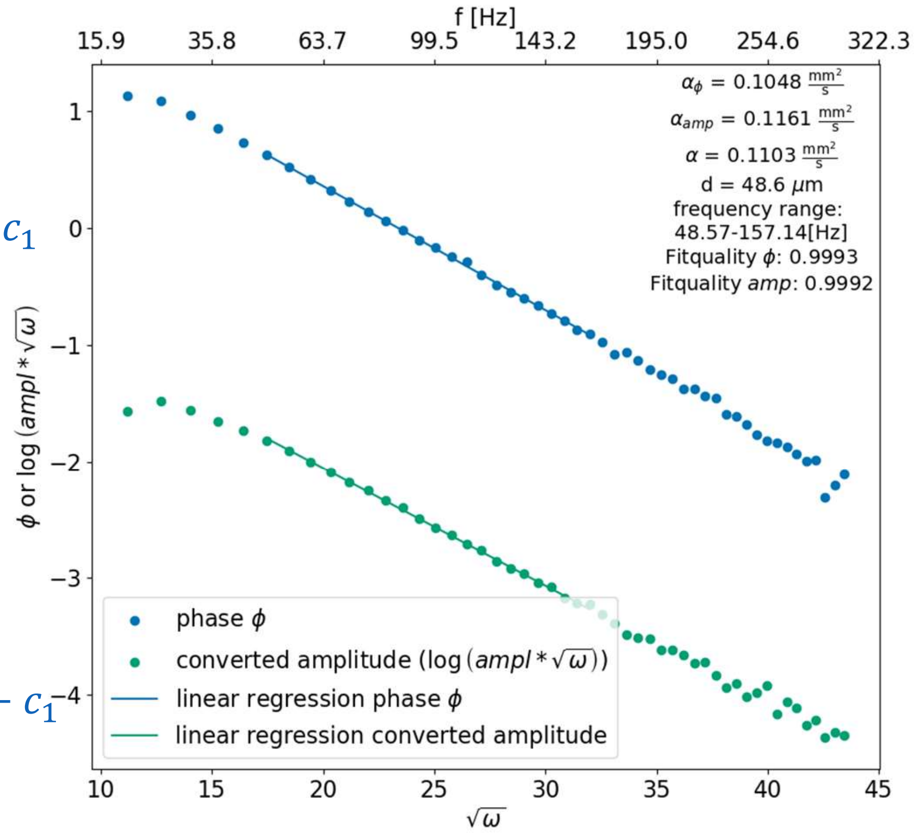


Theory PLH – Data Evaluation

linearized form:

$$\Delta\phi = -\sqrt{\frac{\omega}{2\alpha}}\,d - \frac{\pi}{4} = -\frac{d}{\sqrt{2\alpha}}\sqrt{\omega} + c_1 = -m_\phi\sqrt{\omega} + c_1$$

$$\Rightarrow m_\phi = \frac{d}{\sqrt{2\alpha_\phi}} \Rightarrow \alpha_\phi = \frac{d^2}{2m_\phi^2}$$



$$\log(Amp * \sqrt{\omega}) = -\frac{d}{\sqrt{2\alpha}}\sqrt{\omega} + c_2 = -m_{amp}\sqrt{\omega} + c_1$$

$$\Rightarrow m_{amp} = \frac{d}{\sqrt{2\alpha_{amp}}} \Rightarrow \alpha_{amp} = \frac{d^2}{2m_{amp}^2}$$

$$\Rightarrow \boxed{\boldsymbol{\alpha = \sqrt{\alpha_\phi \alpha_{amp}}}}$$

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THERMAL ANALYSIS

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Thank you for your attention!