

# Study of Thermal Diffusivity of Dielectric-Metal Interfaces at Low Temperatures

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06 Sept 2018

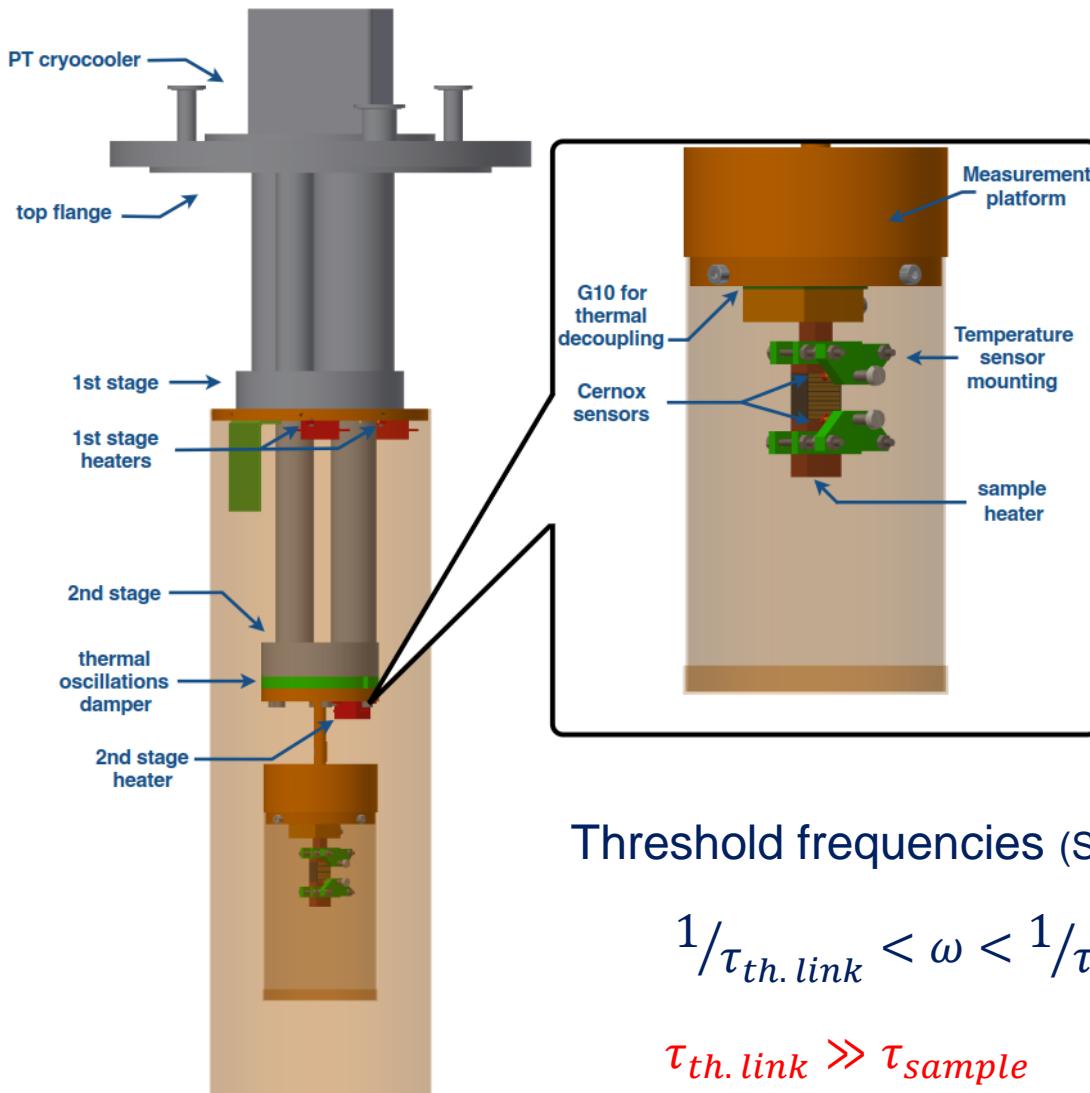
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# Content

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- Introduction to the test set-up
- Method applied and samples
- Measurements and numerical modelling
  - Copper as reference sample
  - Dielectric to metal interfaces
    - Indium or titanium on sapphire
    - SC cable via epoxy impregnation
- Comparison: amplitude vs.  $\tau$ - method
- Conclusion

# Thermal conductivity / diffusivity set-up



## Features:

- 3 temperature sensors calibrated to each other + offset compensation
- Stepwise change of platform temperature 1 K - 2 K
- $\Delta T$  on sample 0.2 K to 0.3 K
- Passive thermal attenuator between cryocooler and thermal platform

Threshold frequencies (Stewart *et al.*, Rev. Sci. Instrum. 54, 1 (1983)):

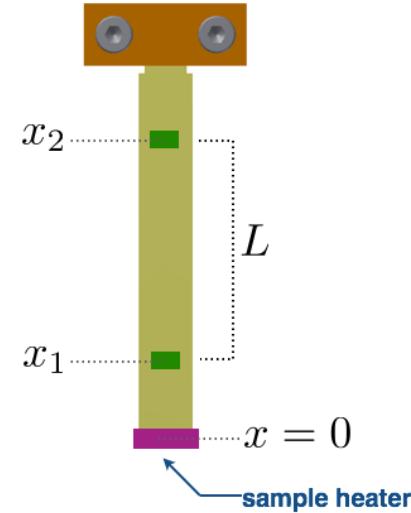
$$1/\tau_{th.\ link} < \omega < 1/\tau_{sample} \quad \text{with} \quad \tau = RC = \frac{C}{\Lambda}$$

$$\tau_{th.\ link} \gg \tau_{sample}$$

# Measurement methodology I

Thermal conductivity:  $\lambda = \frac{\dot{Q} \cdot L}{A \cdot \Delta T}$

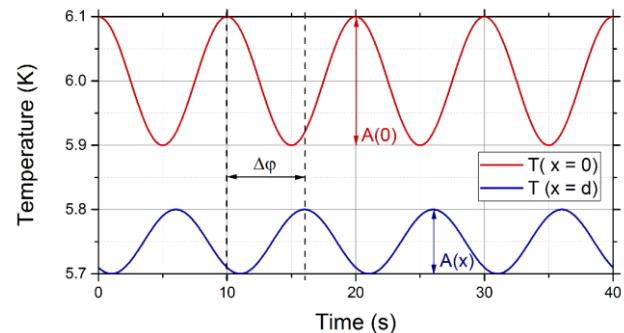
$\lambda$  - thermal conductivity  
 $\dot{Q}$  - heat load  
 $L$  - length along x  
 $A$  - cross section  
 $\Delta T$  - temperature difference



Thermal diffusivity:  $D = \frac{\lambda}{c \cdot \rho}$

$D$  - thermal diffusivity  
 $c$  - heat capacity  
 $\rho$  - density

$$\frac{\delta T}{\delta t} = D \cdot \frac{\delta^2 T}{\delta x^2} \Rightarrow T(L, t) = T_0 \left( 1 + e^{-\frac{L}{\mu} \cos(\omega t - \frac{L}{\mu})} \right)$$



Phase shift method

$$\mu_{phase} = \frac{L}{\phi_0 - \phi_x}$$

$$\phi \text{ (rad)} = \frac{\Delta t \text{ (s)} * 2\pi}{\text{Period time}}$$

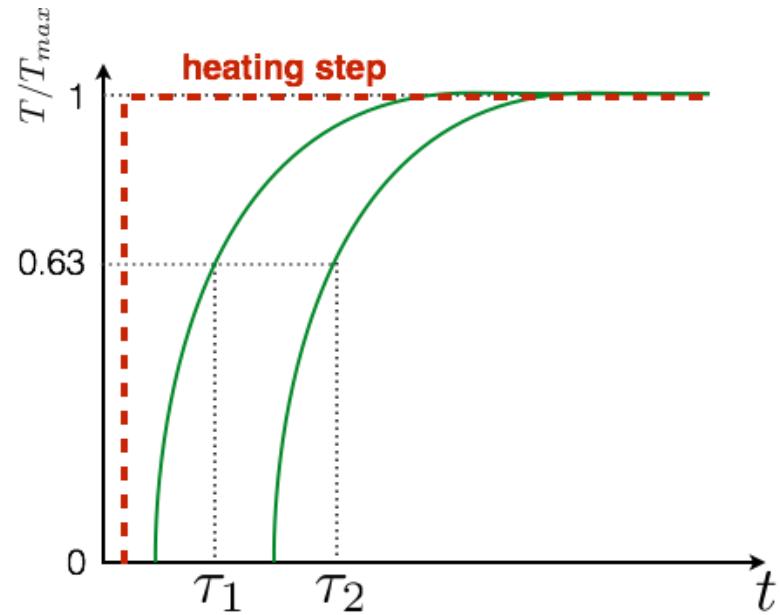
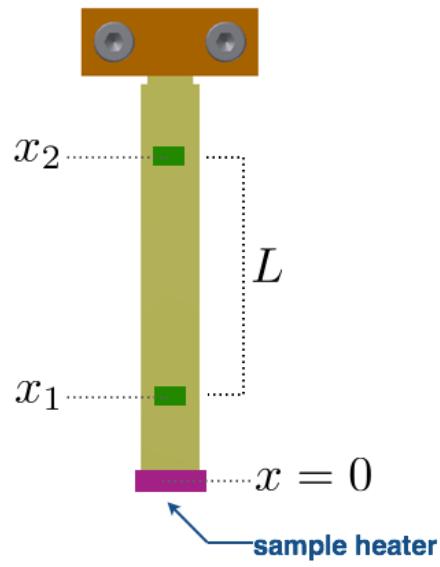
Amplitude method

$$D = \frac{\mu^2 \cdot \omega}{2}$$

$$\mu_{atten.} = \frac{L}{\ln(\frac{A_0}{A_x})}$$

$D$  - thermal diffusivity in  $\text{m}^2/\text{s}$   
 $L$  - length in m  
 $\mu$  - thermal diffusion length in m

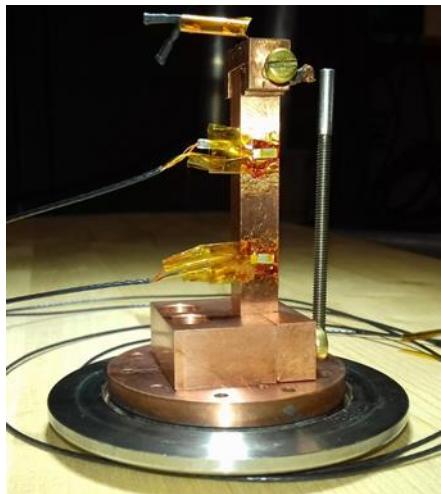
# Measurement methodology II



$$\tau = R \cdot C = \frac{m \cdot c}{A/x \cdot \lambda} = \frac{x \cdot V \cdot \rho \cdot c}{A \cdot \lambda} = \frac{x}{A} \cdot \frac{V}{D} = \frac{x^2}{D}$$

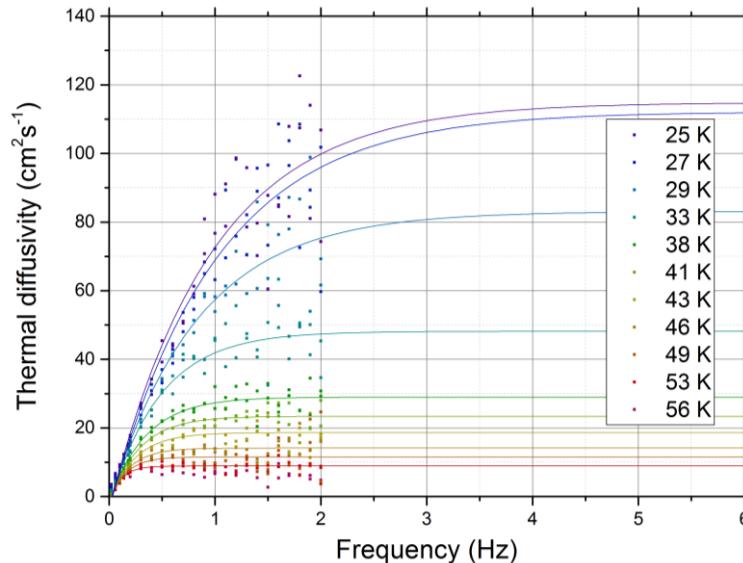
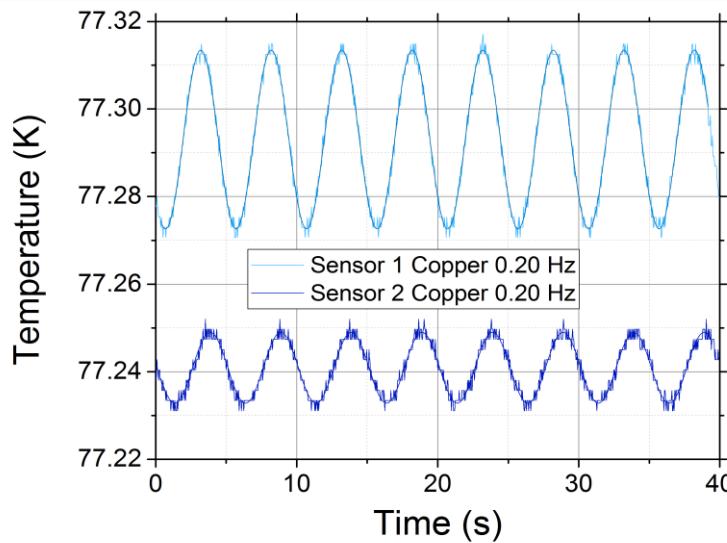
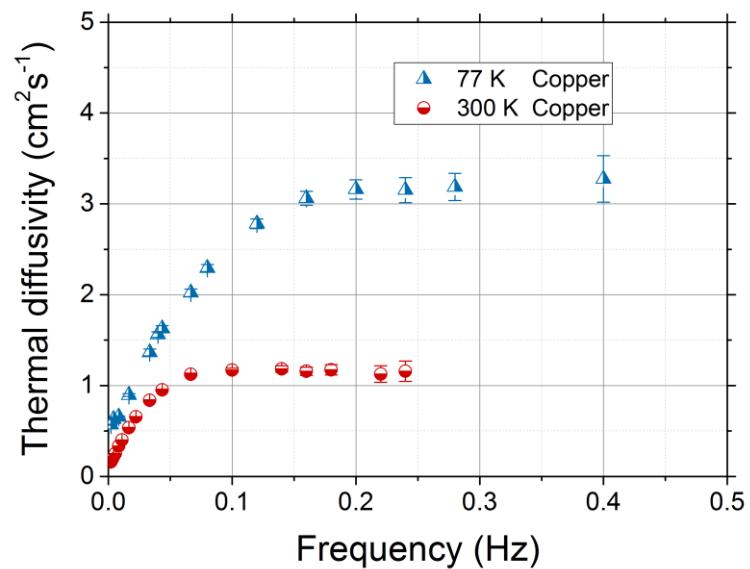
$$D^* = \frac{L^2}{\Delta \tau} \quad \text{--- } \tau - \text{method}$$

# Diffusivity measurements on reference sample Cu-OFHC

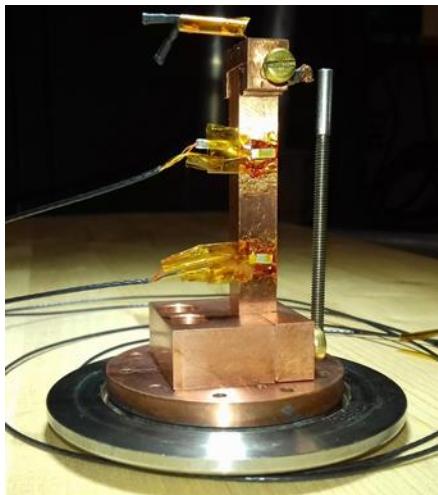


Oxygen-free  
high-conductivity  
(OFHC) copper

Dimensions:  
 $8 \times 8 \times 50 \text{ mm}^3$

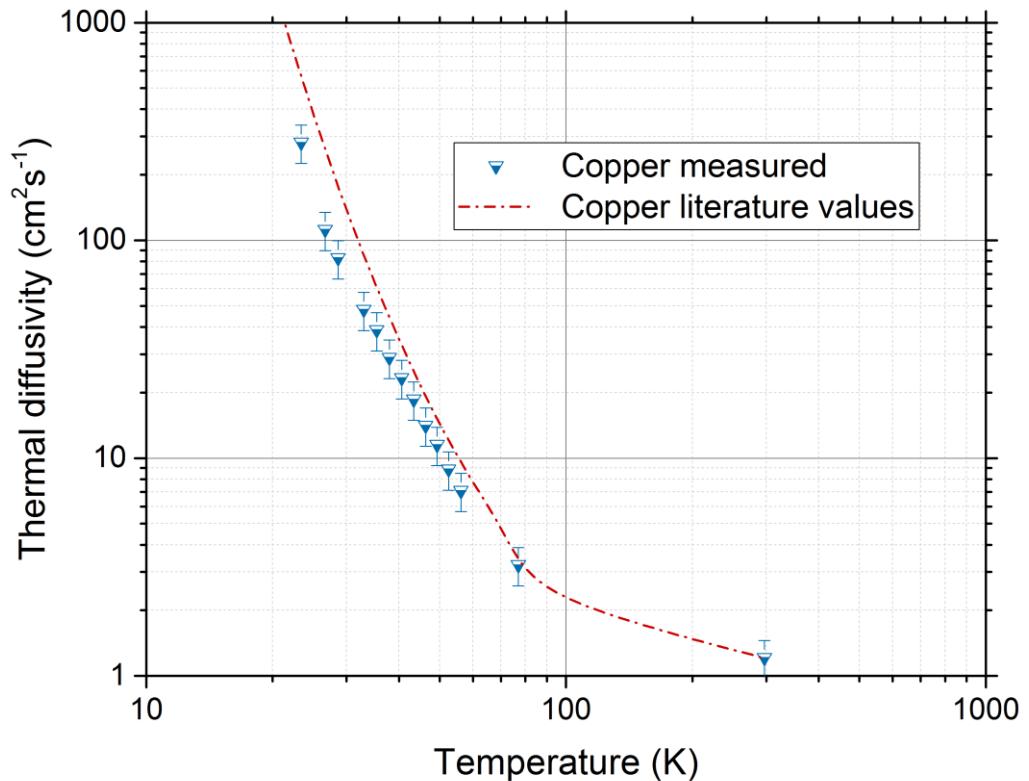


# Diffusivity measurements on reference sample Cu-OFHC



$D(T)$  measured is in good agreement with literature values.

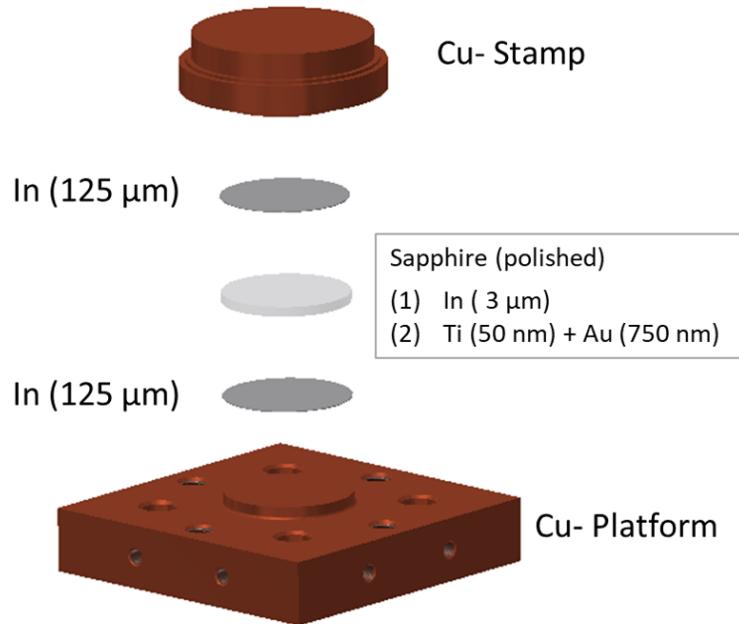
- Amplitude method can be used for more complex samples
- Samples and interfaces need to be adapted



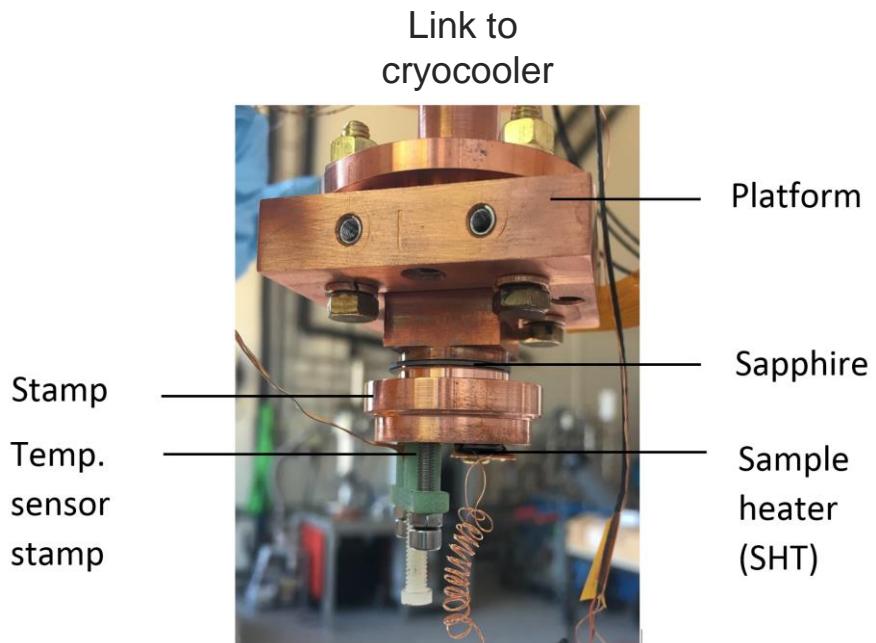
Literature data from: J. E. Jensen *et al.*, Thermal Diffusivity, Brookhaven National Laboratory Selected Cryogenic Data Notebook, Brookhaven National Laboratory (1980).

OFHC copper tested RRR~100 from th. conductivity results

# Thermal diffusivity of a Cu-In- sapphire and Cu-In-Au-Ti-sapphire sandwich



See results presented by J. Liberadzka  
in her talk: E-09: 32

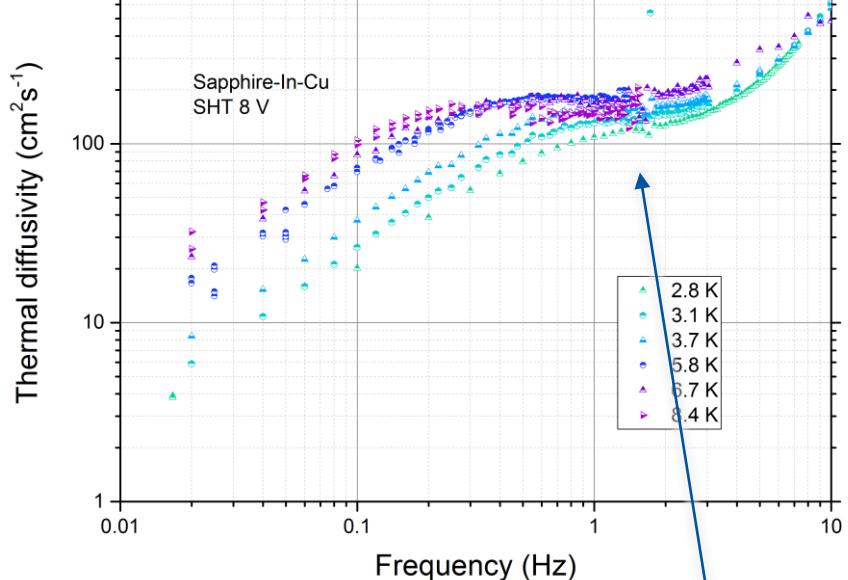


Indium sample :	Cu-In-sapphire-In-Cu
Ti-Au sample:	Cu-In-Au-Ti-sapphire-Ti-Au-In-Cu

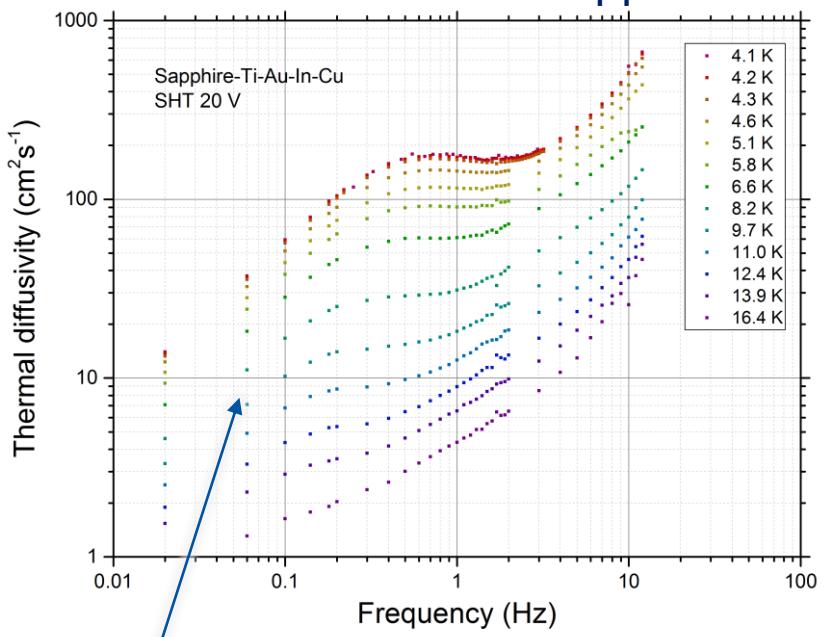
Definition of a diffusivity-like value (due to the presence of interfaces) =>  $D^*$

# Thermal diffusivity of a Cu-In-sapphire and Cu-In-Au-Ti-sapphire sandwich

Cu-In-Sapphire



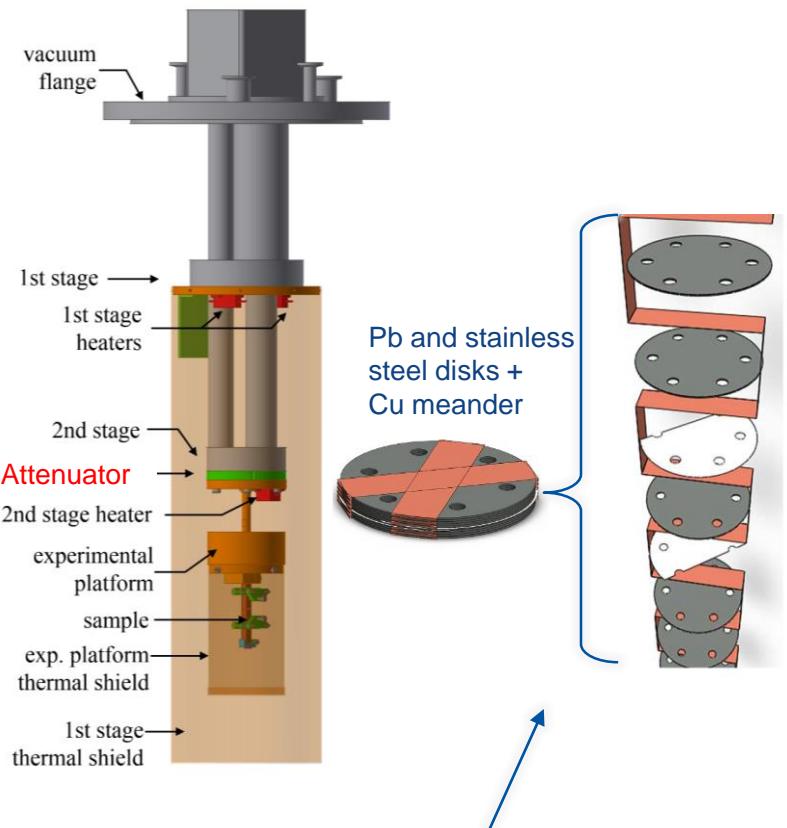
Cu-In-Au-Ti-Sapphire



Influences by the cryocooler frequency and threshold frequency are visible

There is a clever solution to attenuate the cryocooler oscillations and increase  $\tau_{th. link}$

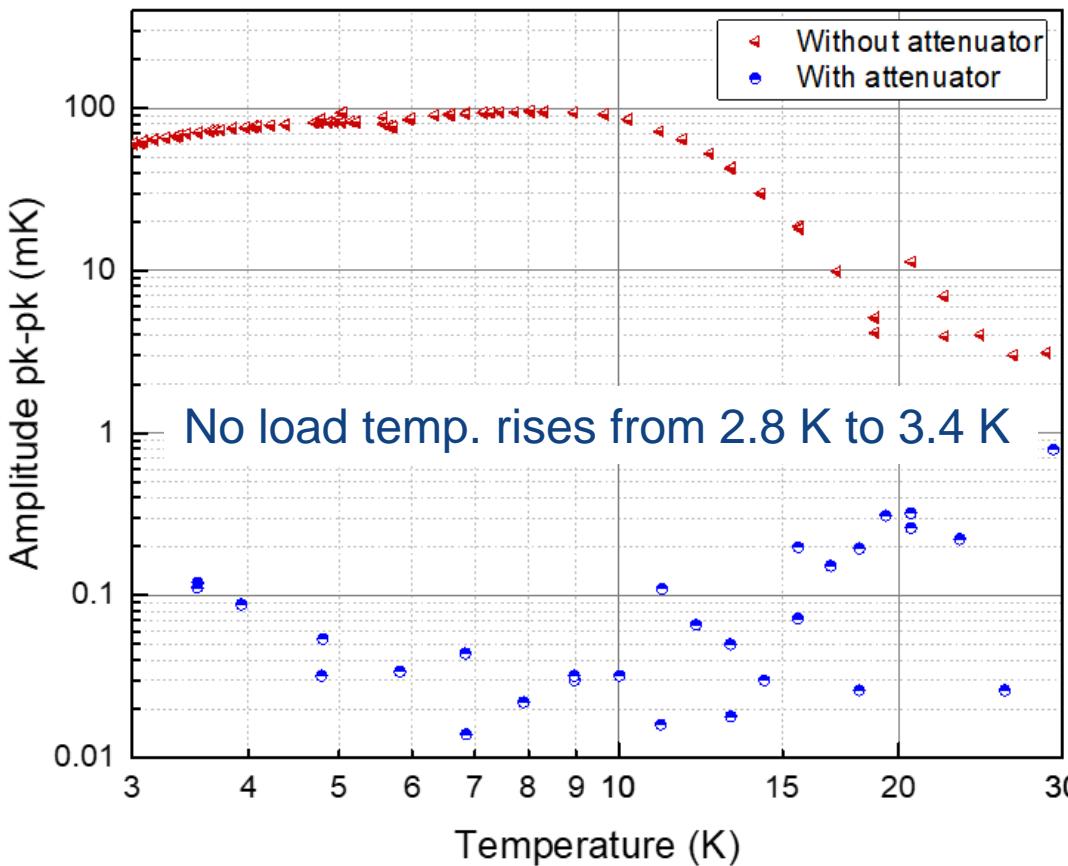
# Passive thermal attenuator (G. Dubuis)



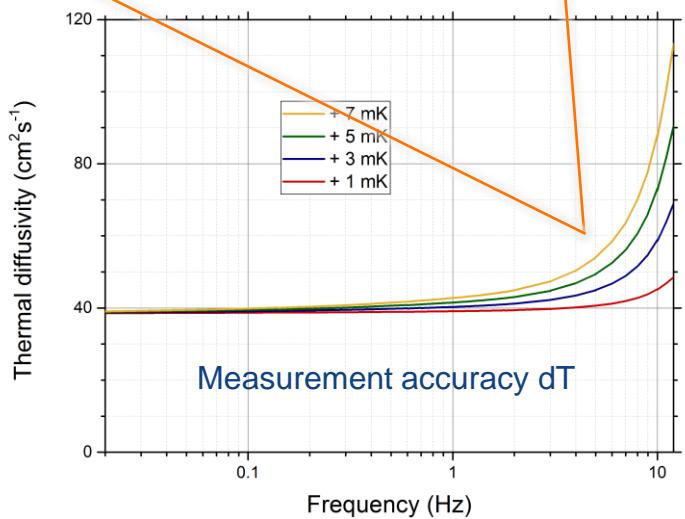
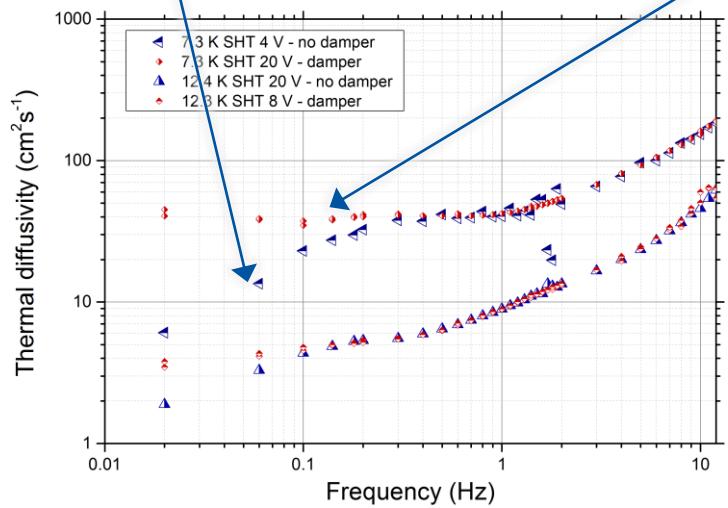
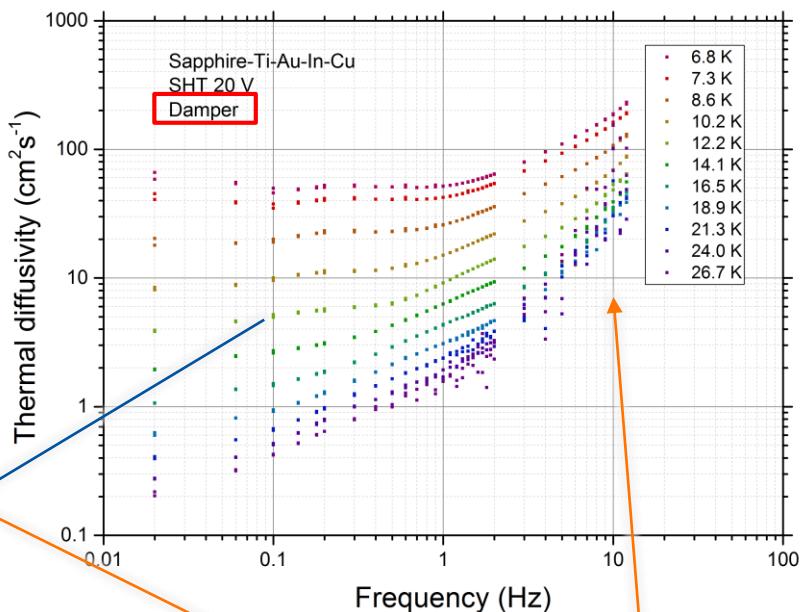
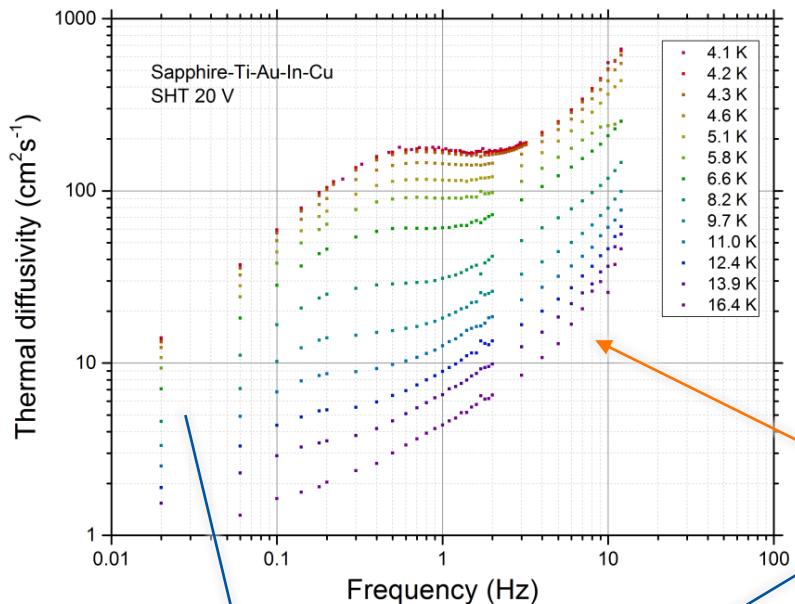
Courtesy: G. Dubuis, X. He, and I. Božovic,  
Sub-milliKelvin stabilization of a closed  
cycle cryocooler, Rev. Sci. Instrum. 85,  
103902 (2014).

Our version CERN Cryolab with:

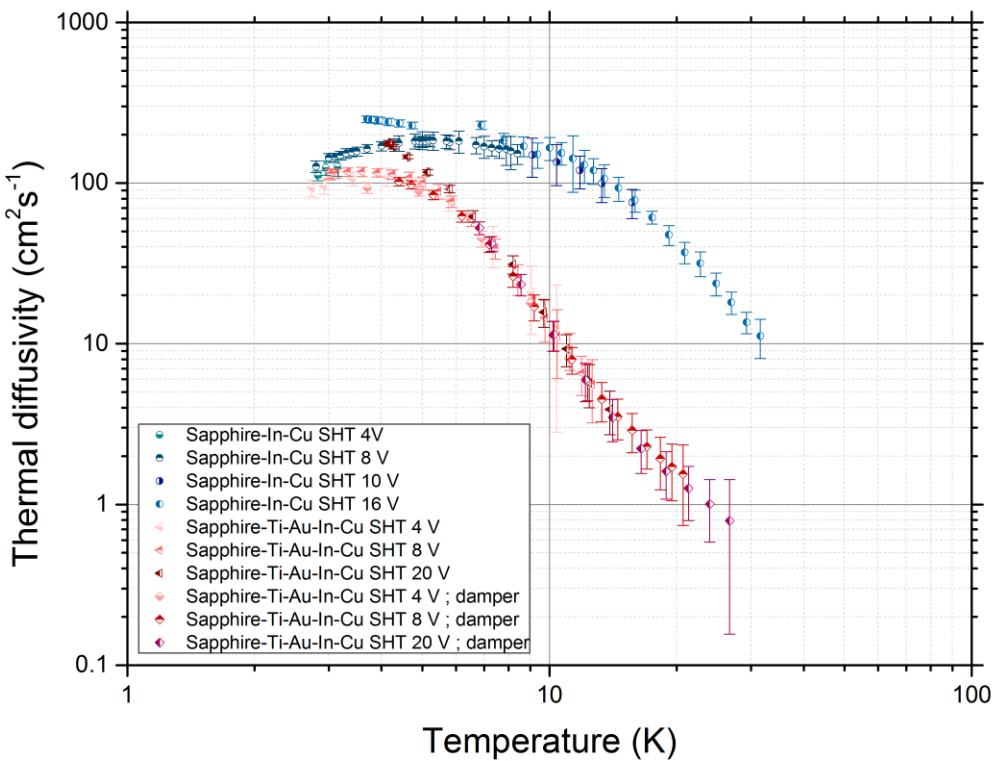
Cryocooler SHI RP-082B2 oscillations at  $f_{op} = 1.725$  Hz



# Thermal diffusivity of a Cu-In-sapphire and Cu-In-Au-Ti-sapphire sandwich



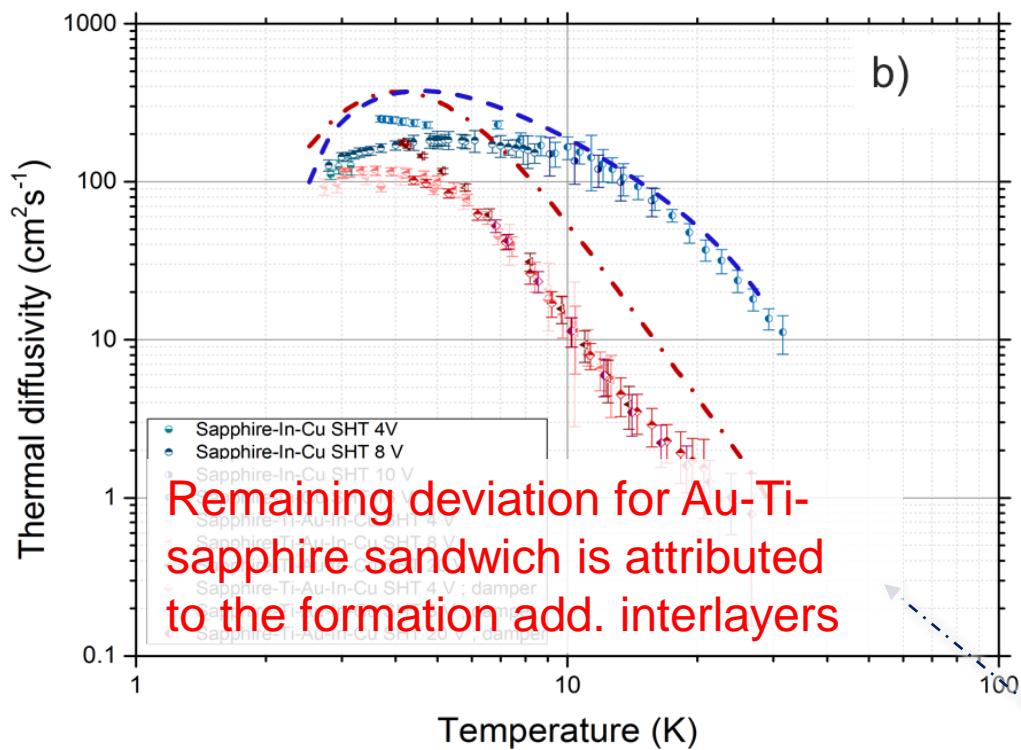
# Thermal diffusivity of a Cu-In- sapphire and Cu-In-Au-Ti-sapphire sandwich



- $D^*_{\text{Cu-In-Sapphire}} > D^*_{\text{Cu-In-Au-Ti-Sapphire}}$
- $D^*_{\max, \text{Cu-In-Au-Ti-Sapphire}}$  is at lower T
- Curves almost merge at about 3 K
- Cu-In-Au-Ti-Sapphire has additional interfaces → additional thermal resistance

Numerical modelling of that structure =>

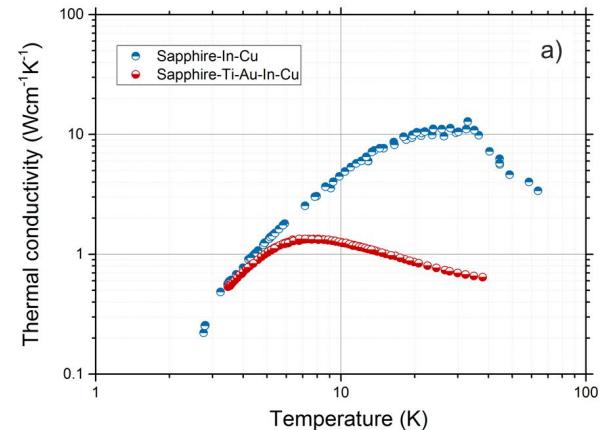
# Thermal diffusivity of a Cu-In-sapphire and Cu-In-Au-Ti-sapphire sandwich



$$\text{Geometry} + c_s \cdot \rho_s = \sum_i \frac{x_i}{d} \cdot c_i \cdot \rho_i \quad \longrightarrow$$

Numerical modelling =>

measured thermal conductivity



$$R_s = R_{sap} + R_{In} + R_{Cu} + R_{InSapIn}$$

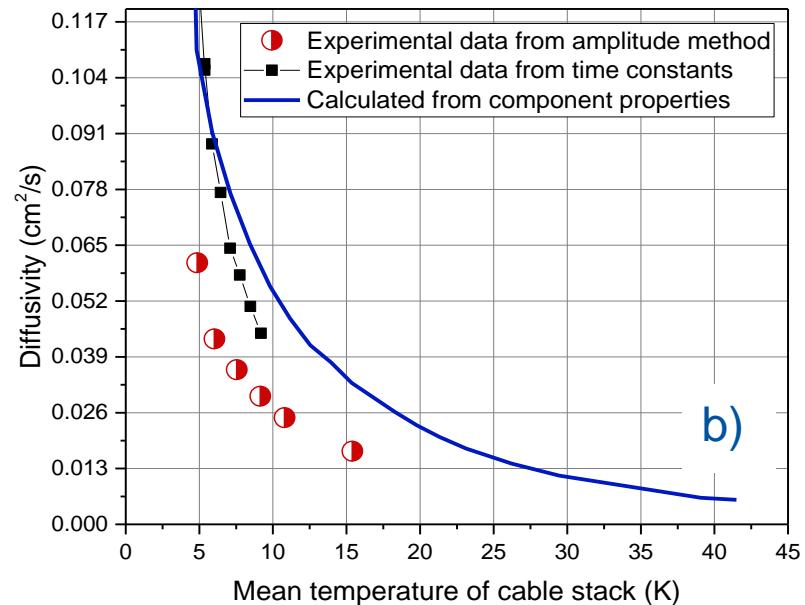
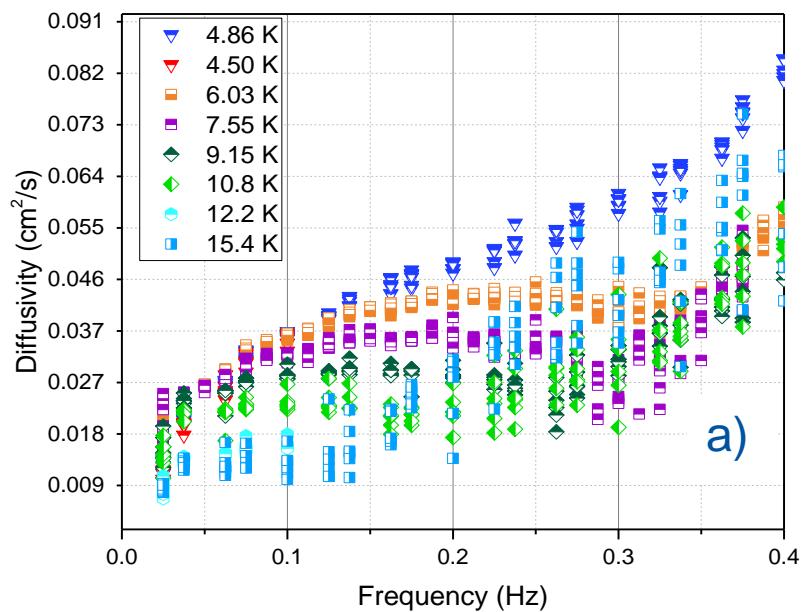
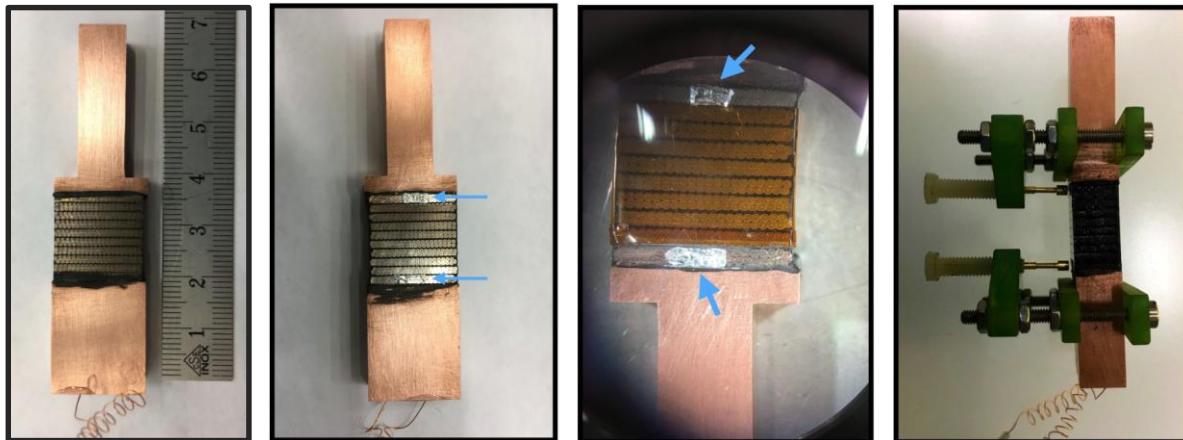
$$R_{InSapIn} = \frac{1}{225} T^{2.5-3.0}$$

$$D^* = \frac{d}{A \cdot R_s \cdot c_s \cdot \rho_s}$$

# Comparing methods: amplitude attenuation vs. step function

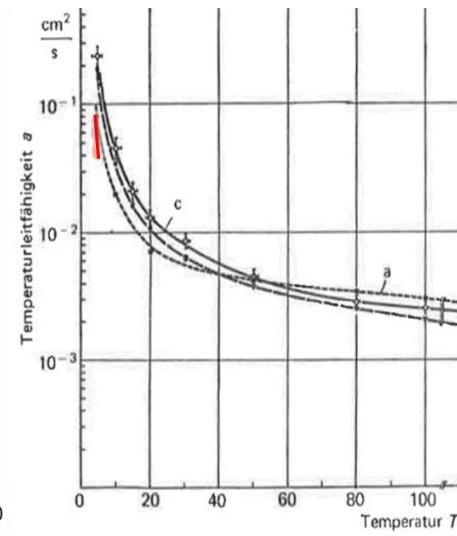
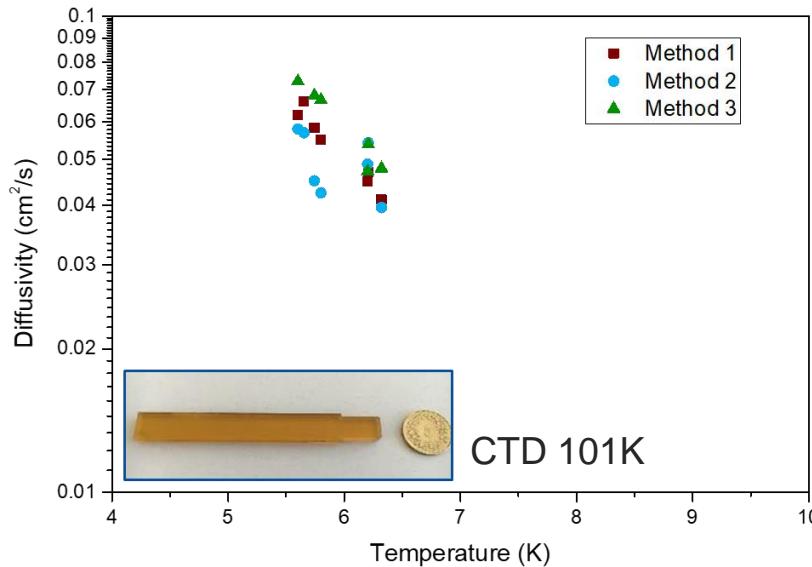
## Sample:

Nb<sub>3</sub>Sn Rutherford type cable stack of 11 T dipole, fully impregnated



# Conclusion

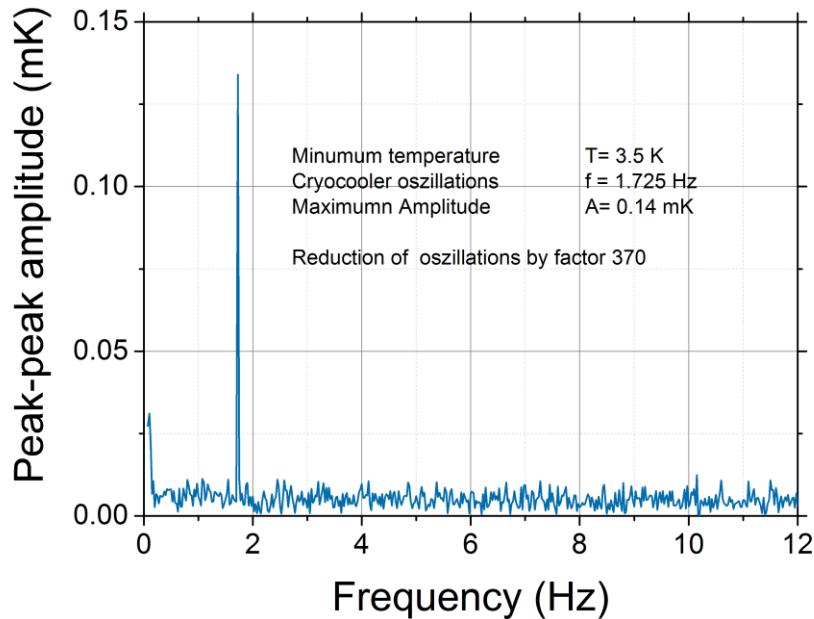
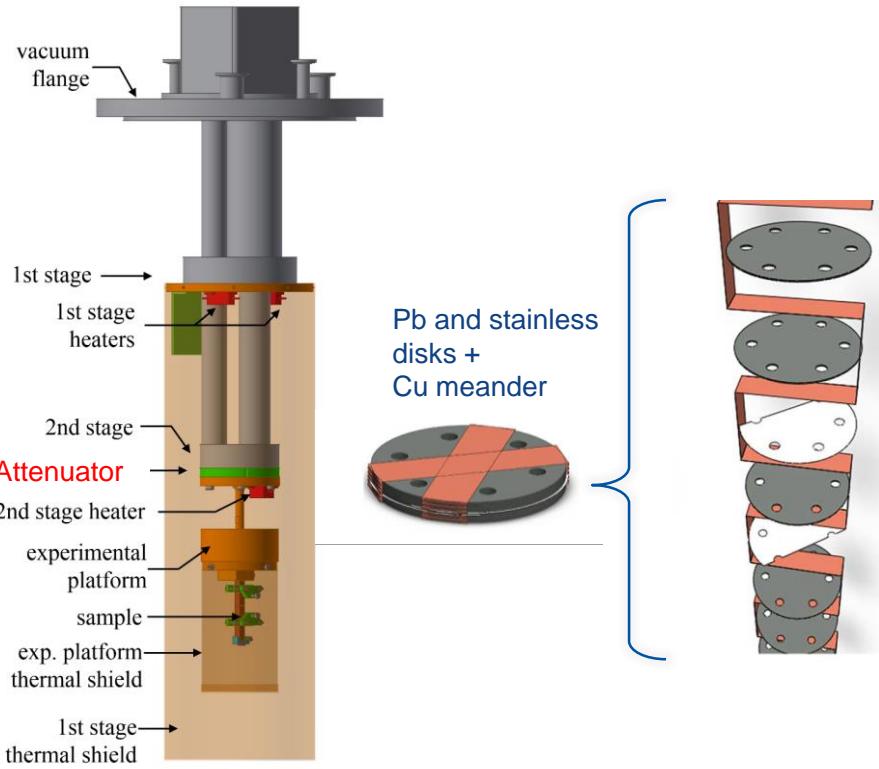
- ✓ Evaluation of measurement methods
- ✓ OFHC-copper measurement and comparison with literature values
- ✓ Inhomogenous stackd with dielectric-metal interfaces
- ✓ Comparison of methods for bulk samples
- ✓ Amplitude attenuation and step methods seem to be complementary





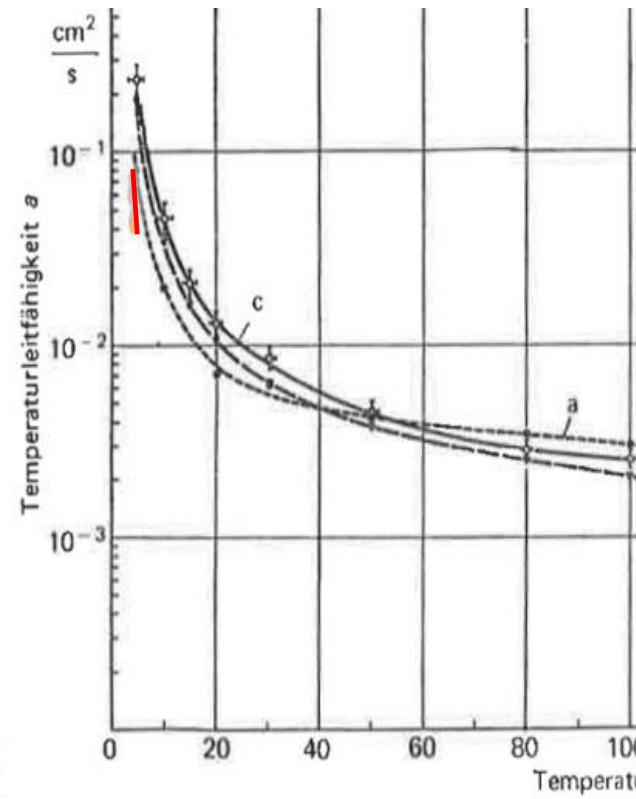
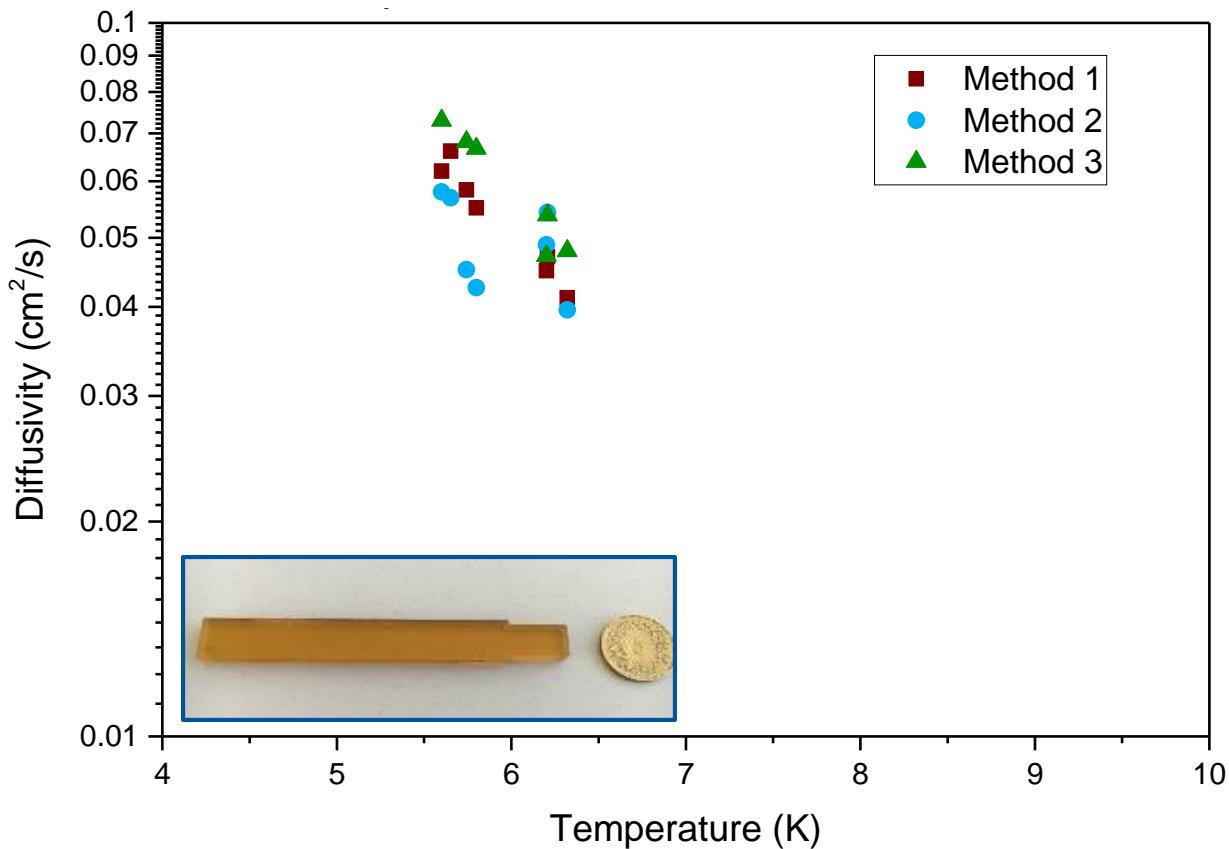
[www.cern.ch](http://www.cern.ch)

# Passive thermal attenuator



Courtesy: G. Dubuis, X. He, and I. Božovic,  
Sub-milliKelvin stabilization of a closed  
cycle cryocooler, Rev. Sci. Instrum. 85,  
103902 (2014).

# HiLumi LHC<sub>11</sub> T Nb<sub>3</sub>Sn Dipole => CTD-101K bulk epoxy sample



From: Frey, H. and Haefer, R. A., *Tieftemperaturtechnologie*



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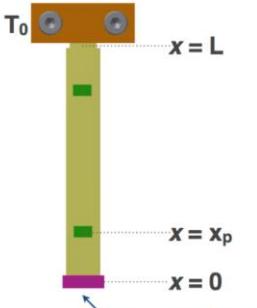
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# Further diffusivity measurement methods

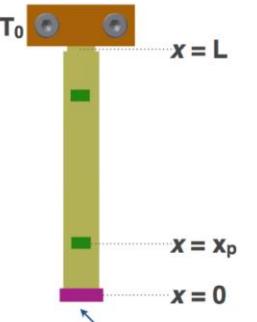
- Pulse:**  $\kappa = \frac{x_p^2}{2t_{max}}$

- $x_p$  is the distance between the heater and the detector
- $t_{max}$  is the time at which the temperature at  $x_p$  reaches its maximum



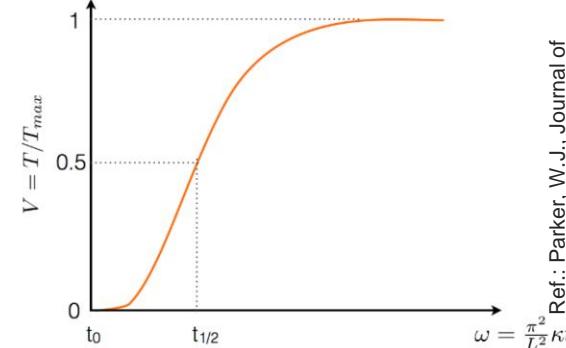
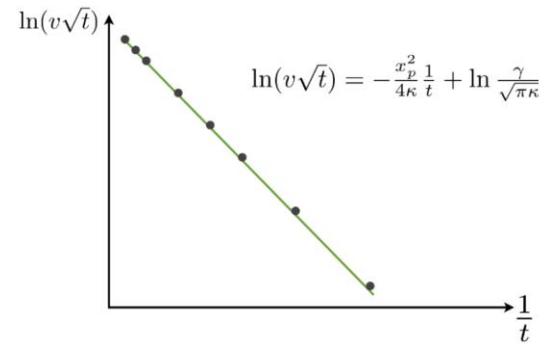
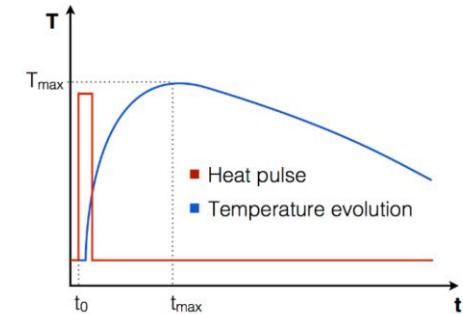
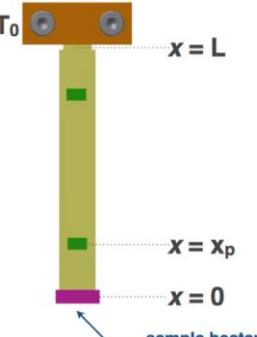
- Pulse 2:**  $\kappa = \frac{x_p^2}{4a}$

- $v = T_1 - T_0$  is the experimental temperature rise
- Plot  $\ln(v\sqrt{t}) = -\frac{x_p^2}{4\kappa t} + \ln\left(\frac{\gamma}{\sqrt{\pi\kappa}}\right) = -a\frac{1}{t} + b$ ,
- diffusivity can be extracted through the slope  $a$

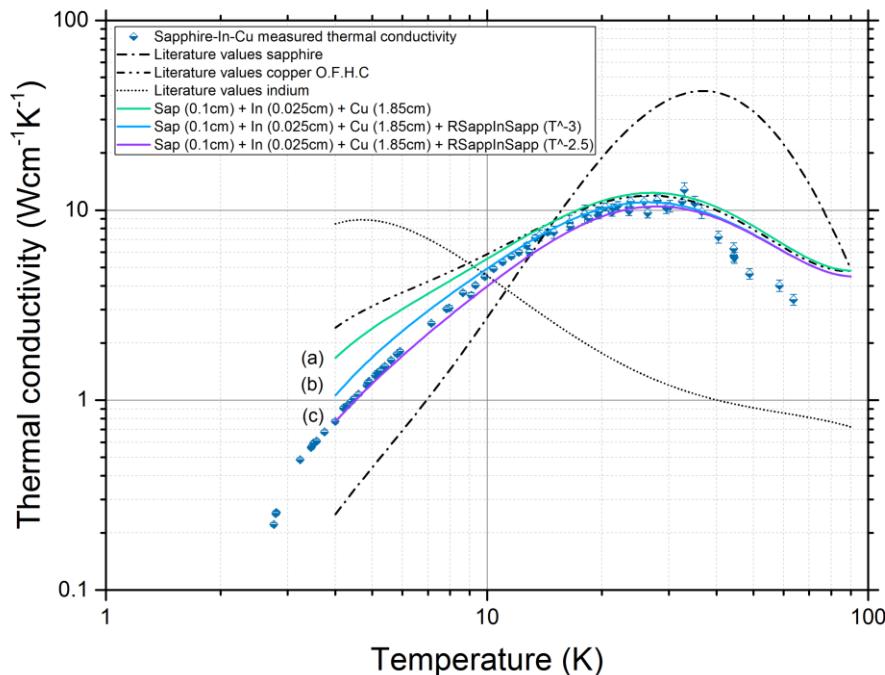
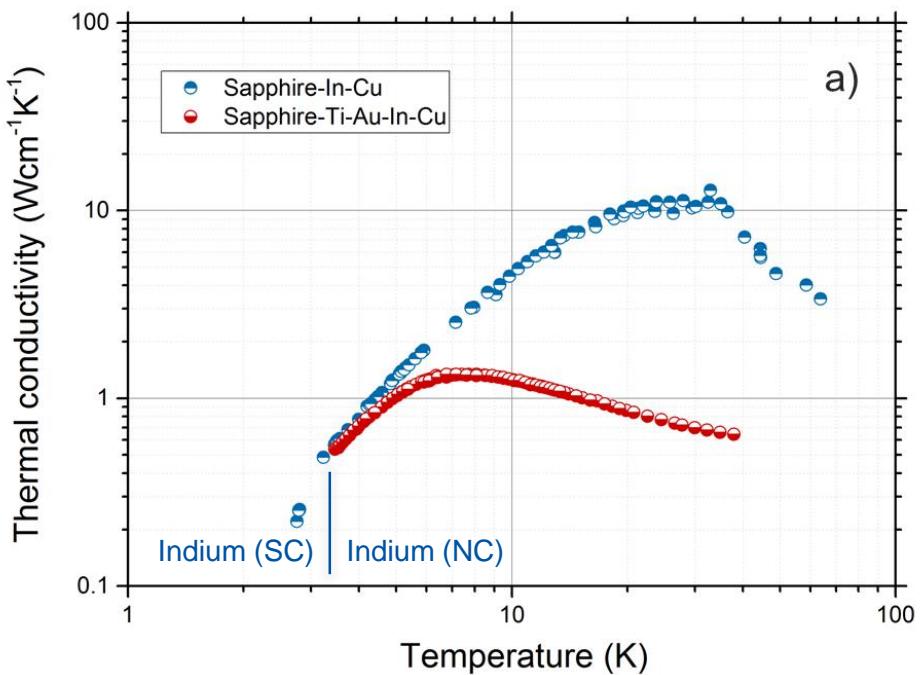


- Step:**  $\kappa = 1.38 x_p^2 / \pi^2 t_{1/2}$

- plot  $V = T(x_p, t)/T_{max}$  as a function of  $\omega = \frac{\pi^2}{L^2} \kappa t$
- $t_{1/2}$  is the time when  $T(x_p)$  reaches half of the maximum value



# $\lambda$ and $\kappa^*$ of a Cu-In-sapphire and Cu-In-Au-Ti-sapphire sandwich



Modelling of thermal conductivity including the boundary resistances e.g. Cu-In-sapphire:

$$R_i = \frac{d}{\lambda \cdot A} \quad R_s = R_{sap} + R_{In} + R_{Cu} + R_{InSapIn}$$

$$\text{with } R_{InSapIn} = \frac{1}{225} T^{2.5-3.0} \quad \text{in } \frac{m^2}{WK}$$

Gmelin et al., J. Phys. D: Appl. Phys. 32 (1999) R19–R43.

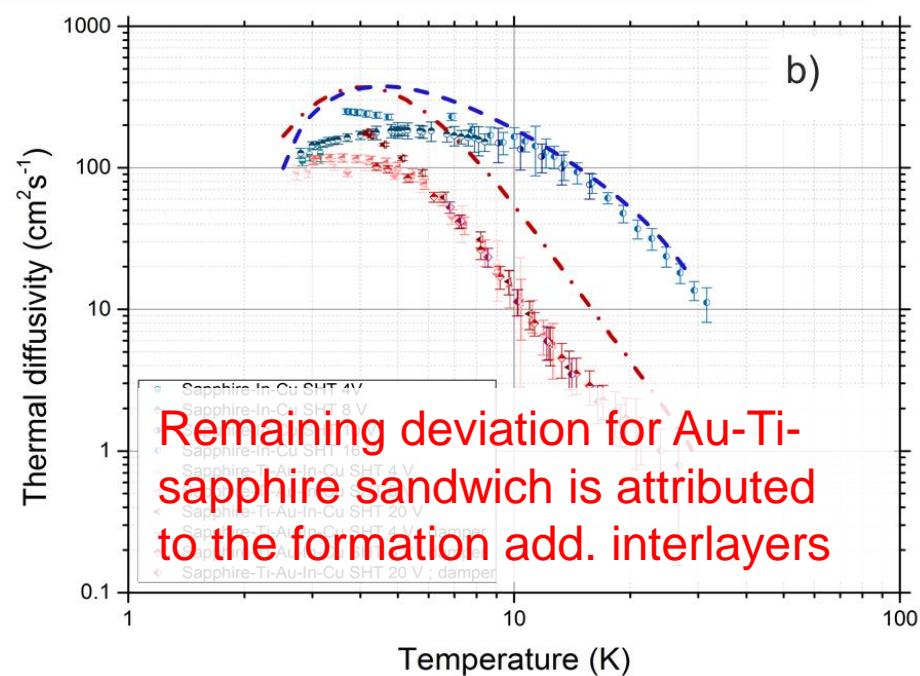
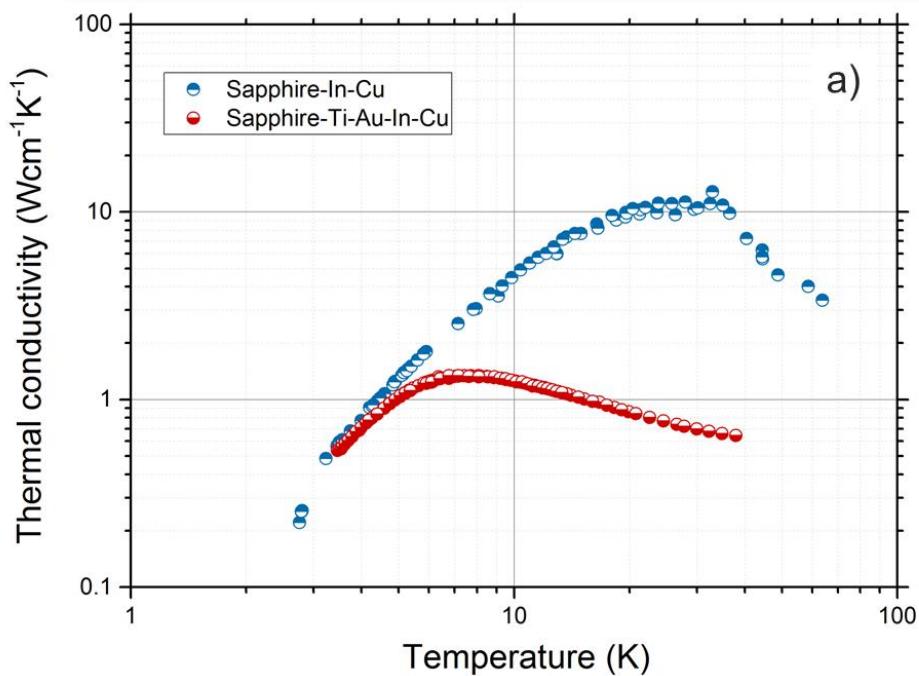
Modelling of diffusivity including  $\lambda$  measured ! of Cu-In-sapphire:

$$c_s \cdot \rho_s = \sum_i \frac{x_i}{d} \cdot c_i \cdot \rho_i$$

$$\kappa^* = \frac{d}{A \cdot R_s \cdot c_s \cdot \rho_s}$$



# $\lambda$ and $\kappa^*$ of a Cu-In-sapphire and Cu-In-Au-Ti-sapphire sandwich



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Gmelin et al., J. Phys. D: Appl. Phys. 32 (1999) R19–R43.

Geometry factor: static vs. transient

$$\frac{V_{\text{total}}}{V_{\text{direct}}} = 3.3$$

Cu-In-Au-Ti-sapphire = (2.7)

The diagram illustrates the geometry factor ratio. On the left, a 'total' assembly is shown as a stack of three layers: Cu (top), In-sapphire-In (middle), and Cu (bottom). On the right, a 'direct' assembly is shown as a single cylindrical layer of In-sapphire-In. The ratio of their volumes is calculated as 3.3, leading to a geometry factor of 2.7 for the Cu-In-Au-Ti-sapphire sandwich.

