Non-contact luminescence lifetime cryothermometry for application in vacuum environment

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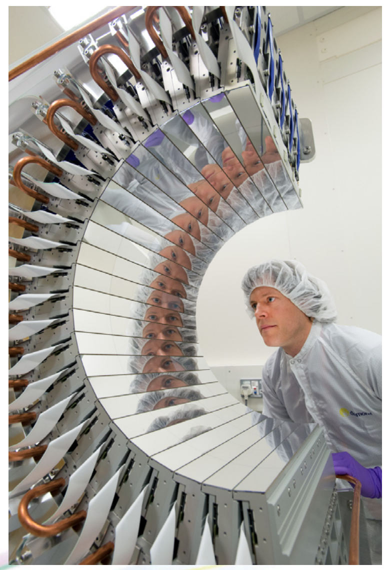
Diamond Light Source

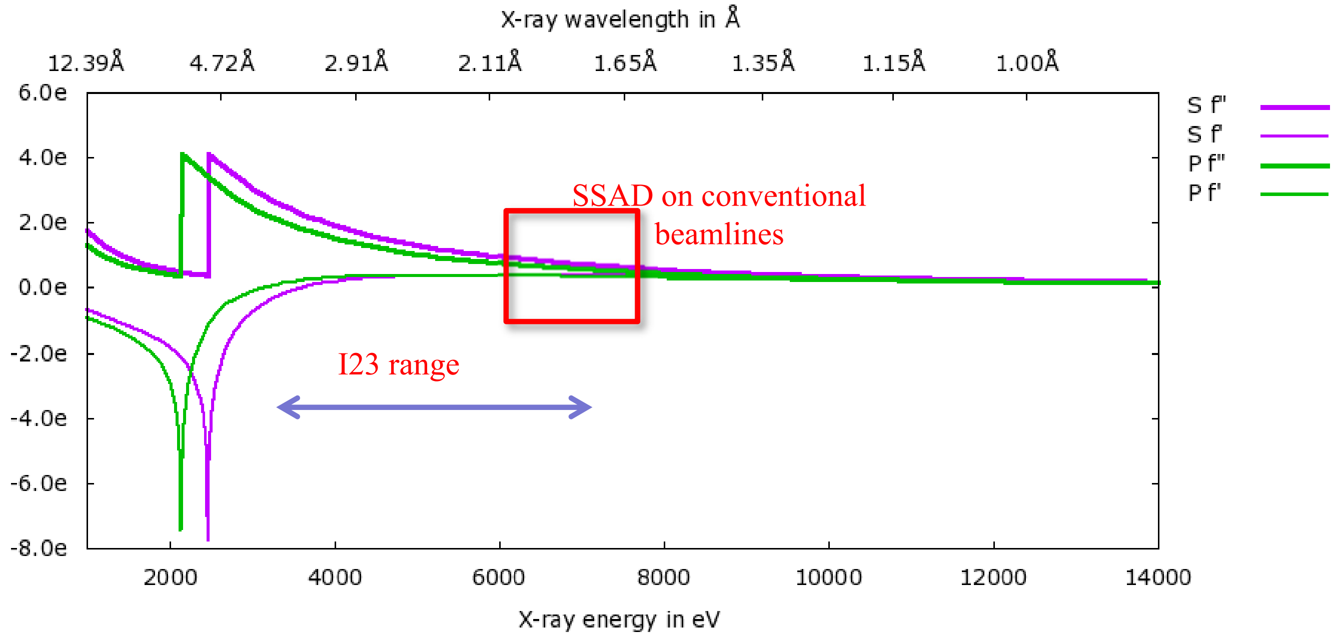
Overview

- Motivation
- Basics of non-contact cryothermometry
- Method of measurements
- Integration of thermometry in the beamline instrumentation
- System calibration, testing and application examples
- Conclusion and outlook

Long-wavelength MX I23 beamline at Diamond

Experimental Phasing from native proteins and DNA/RNA crystals at S-edge





Beamline parameters:

Wavelength range: 1 - 5.9 Å, optimized for 1.5 - 4 Å

Beam size at sample: 100 - 500 µm (unfocused)

Photon flux: 5 x 10^{12} ph/s in 100 x 100 µm @ 4 keV

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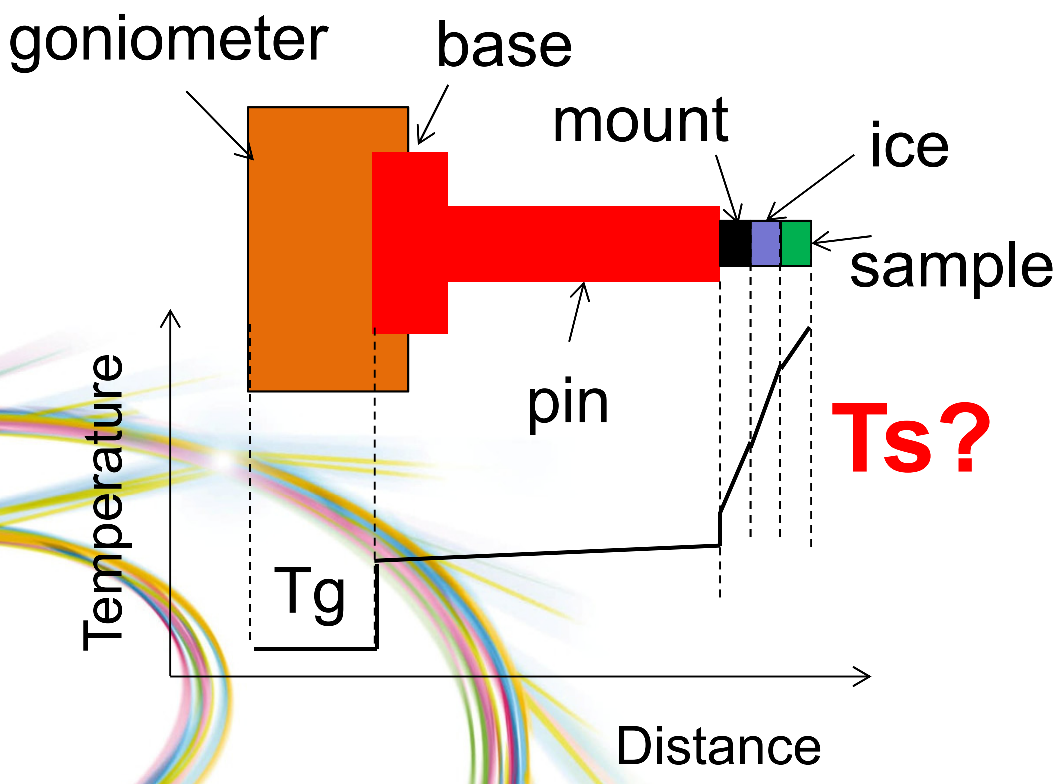
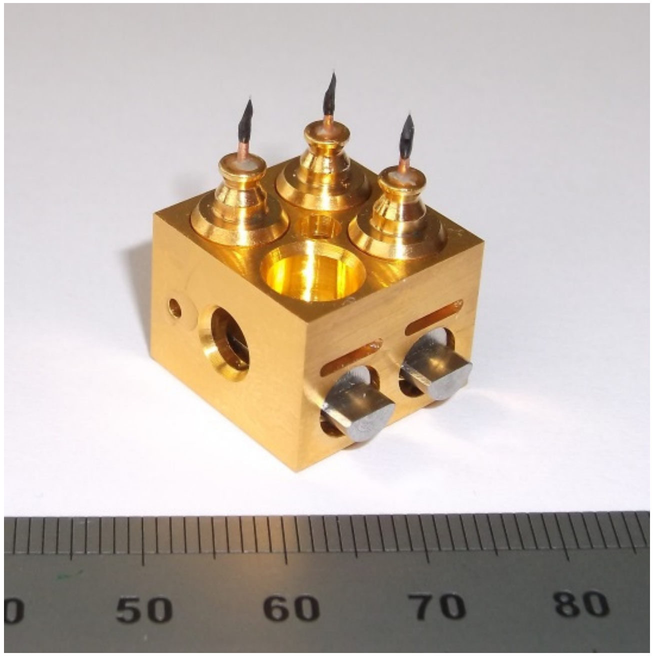
Keeping protein crystal frozen in vacuum

Cooling via thermal conductivity comes *with a few challenges i.e. materials, boundary effects and uncertainty of the sample temperature*

Temperature distribution across sample holder assembly





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How to determine real temperature of my sample in vacuum?

Conventional thermometry with electrical readout ?

i) wiring is not compatible with the need of sample change;

ii) needs tiny sensor (<1 mm^3) comparable with the size of object under test

iii) heat leaks via wiring

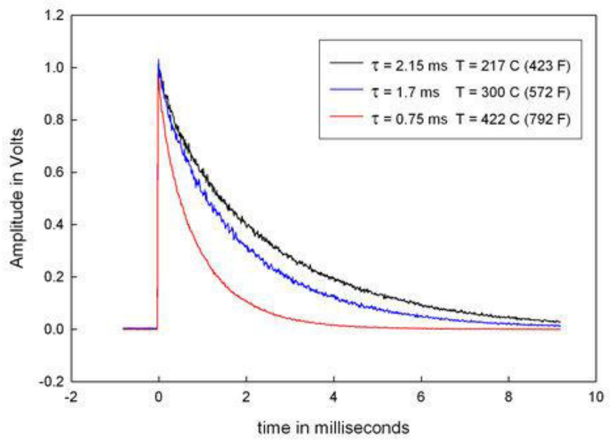
Remote (non-contact) thermometry as a solution?

refractive index, birefringence,

transmission, luminescence light

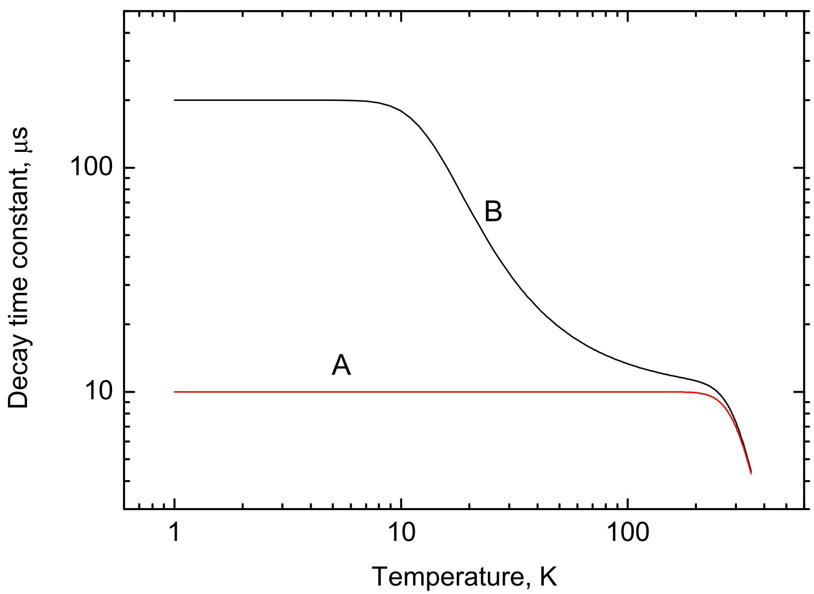
Intensity, wavelength shift,

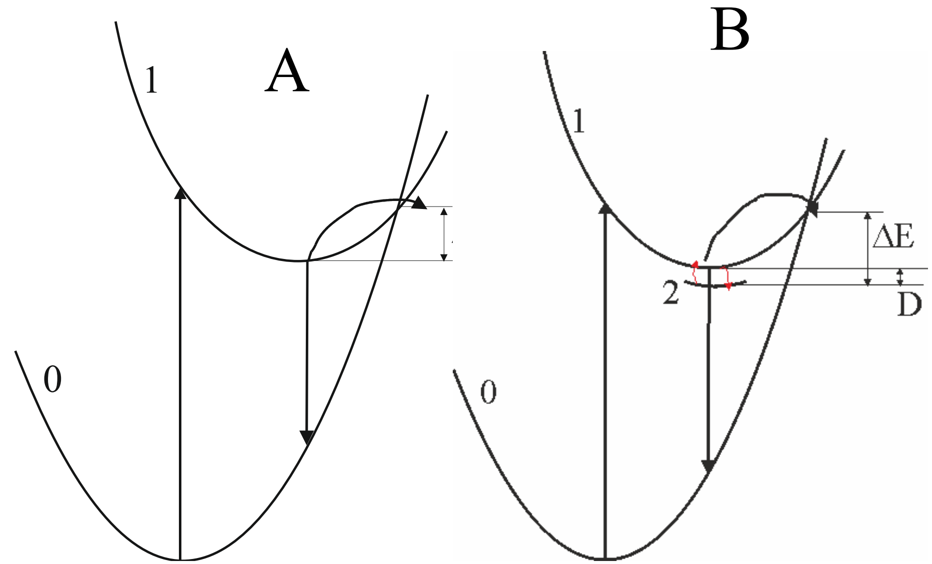
luminescence lifetime (decay time constant)



Decay time constant as a measure of temperature

$$\tau^{-1} = \tau_r^{-1} + K\exp(-\Delta E / kT)$$





$$\tau^{-1} = \frac{w_1 + w_2\exp(-D/kT)}{1 + \exp(-D/kT)} + K\exp(-\Delta E/kT)$$

Features:

- Good responsivity in 1-300 K range (material dependent)
- Low sensitivity to intensity variation (better accuracy)
- Very small sensor size (<1mm^3)
- Measurements technique was available (extensively tested and user ready)

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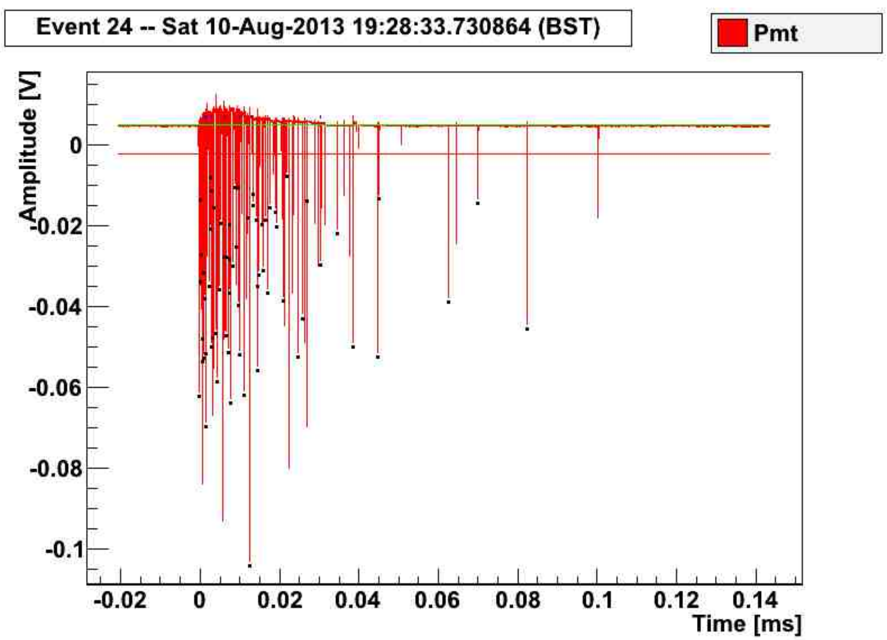
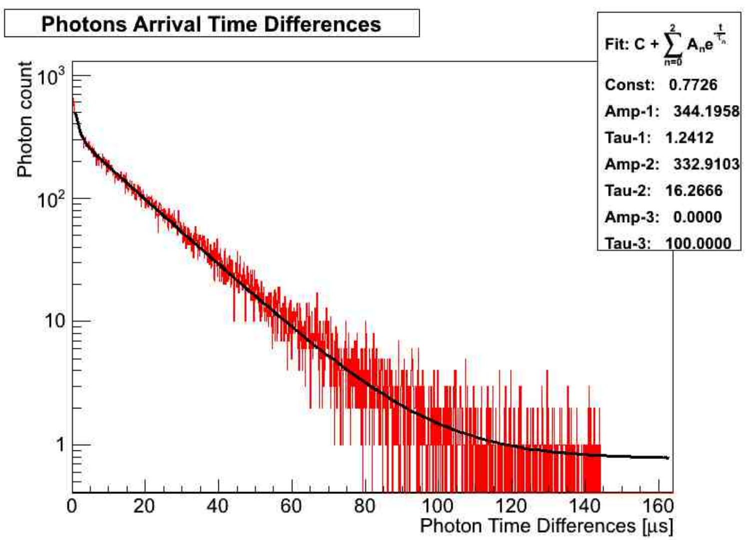
Scintillations recorded and analysed using multiphoton counting technique

STFC funded knowledge transfer project between Oxford University and Diamond

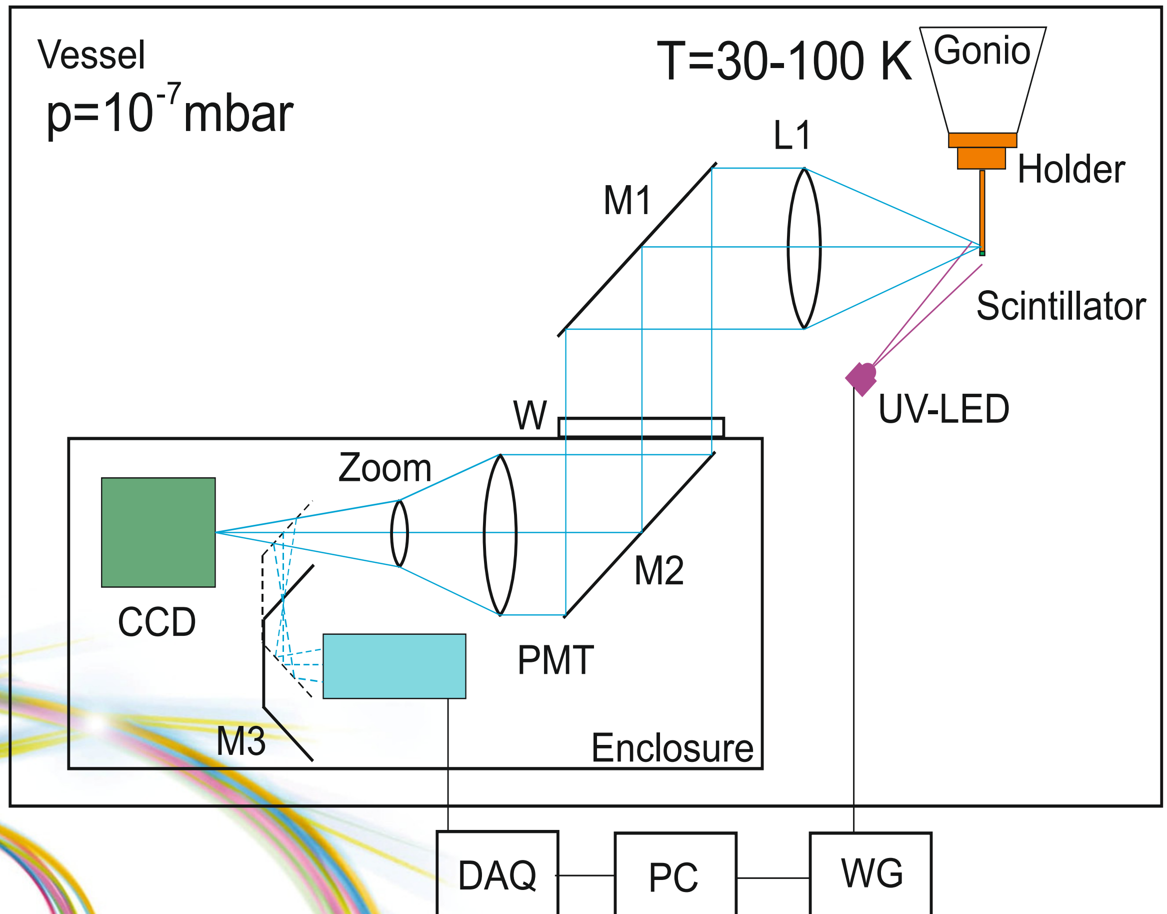
Scintillation event

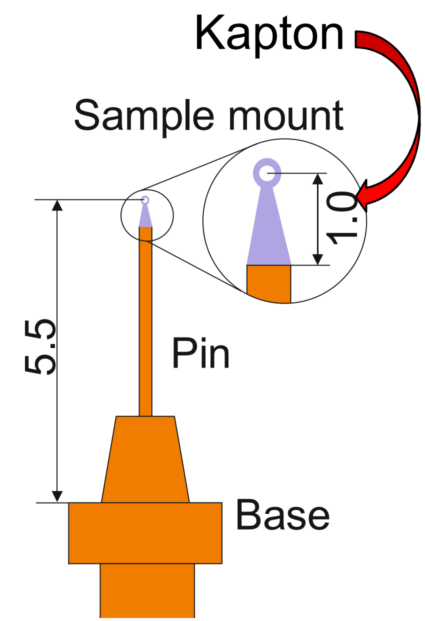


Decay curve

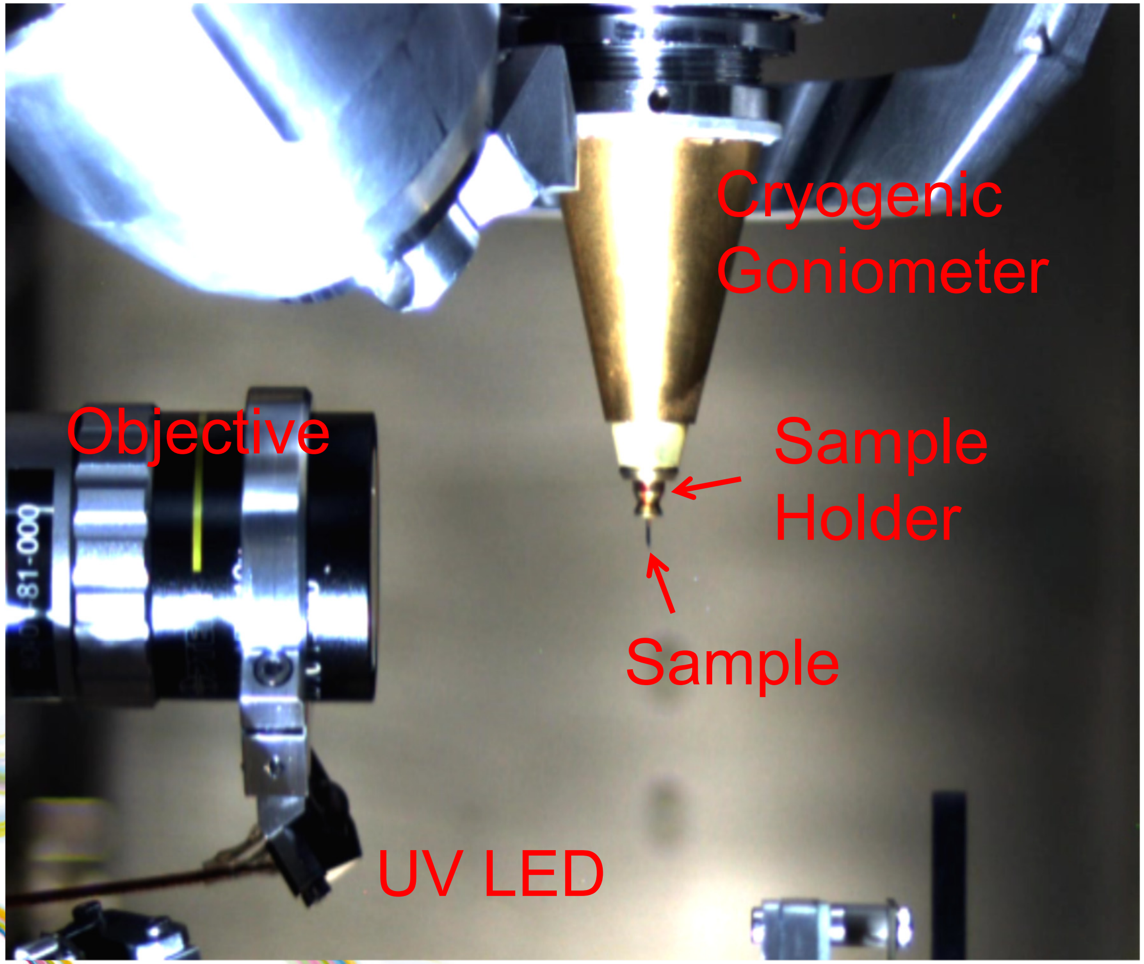


Schematics of temperature monitoring in vacuum at I23 beamline

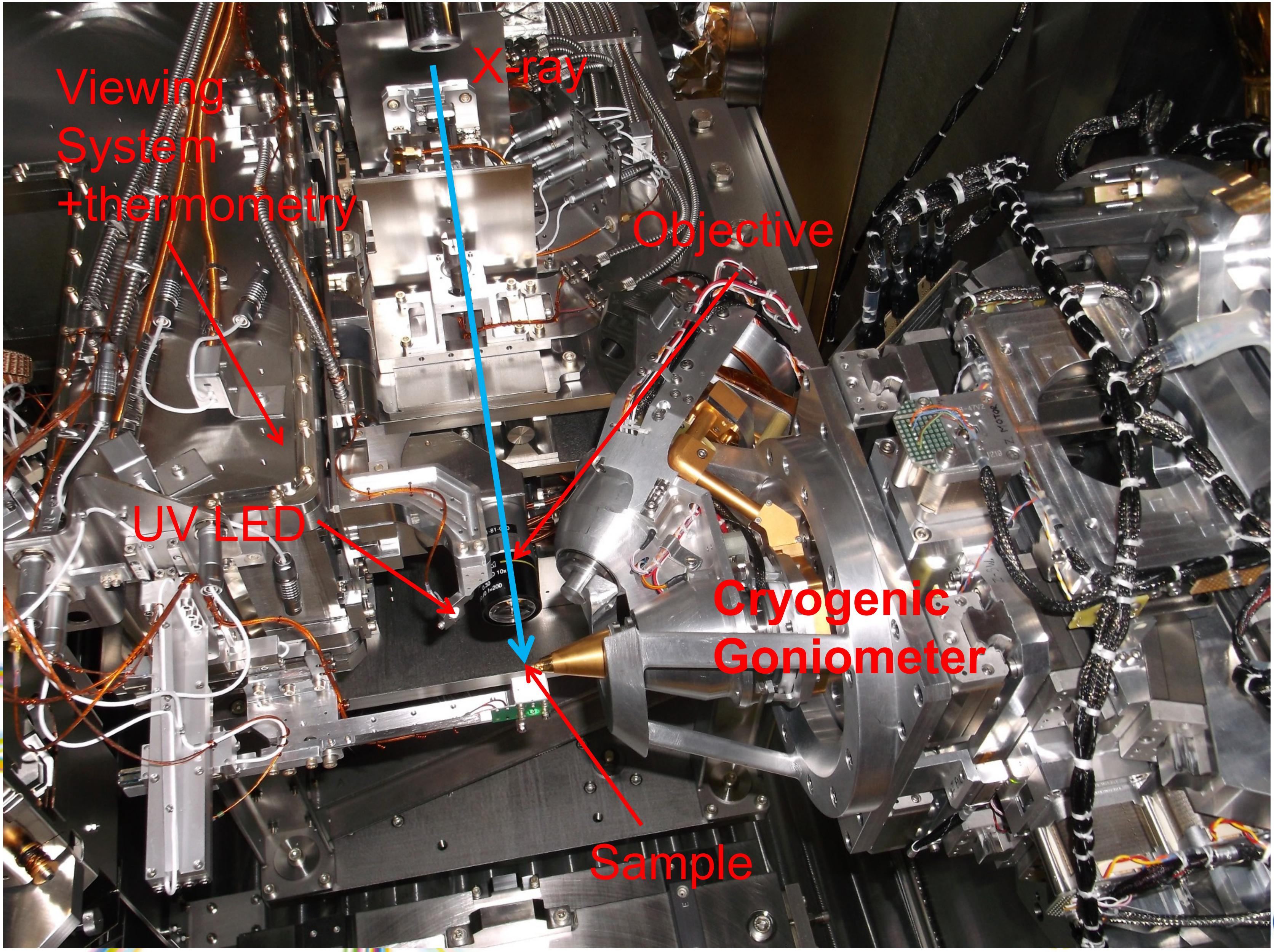




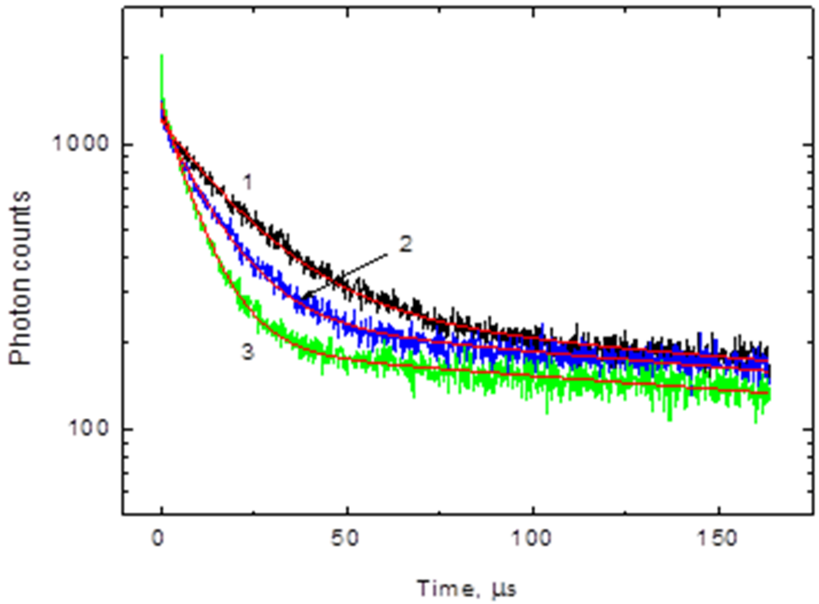
Integration in experimental environment - Top view



Integration in experimental environment - General view



Sensor Calibration

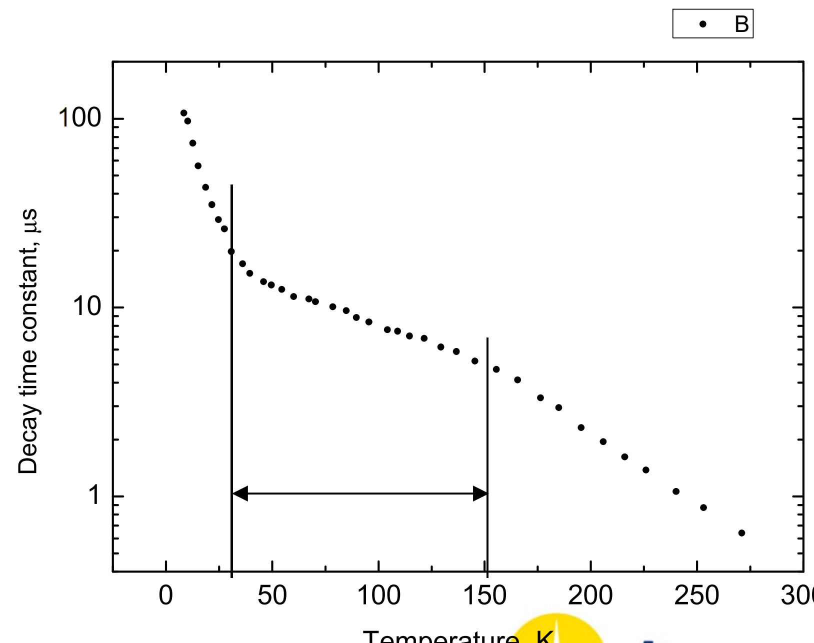


Decay curves of $Bi_4Ge_3O_{12}$ (BGO) scintillator vs T

1- T=31 K

2- T=50 K

3- T=121 K



BGO Decay Time vs T at λ=245 nm UV excitation

Uncertainty of temperature determination ±1.6 K

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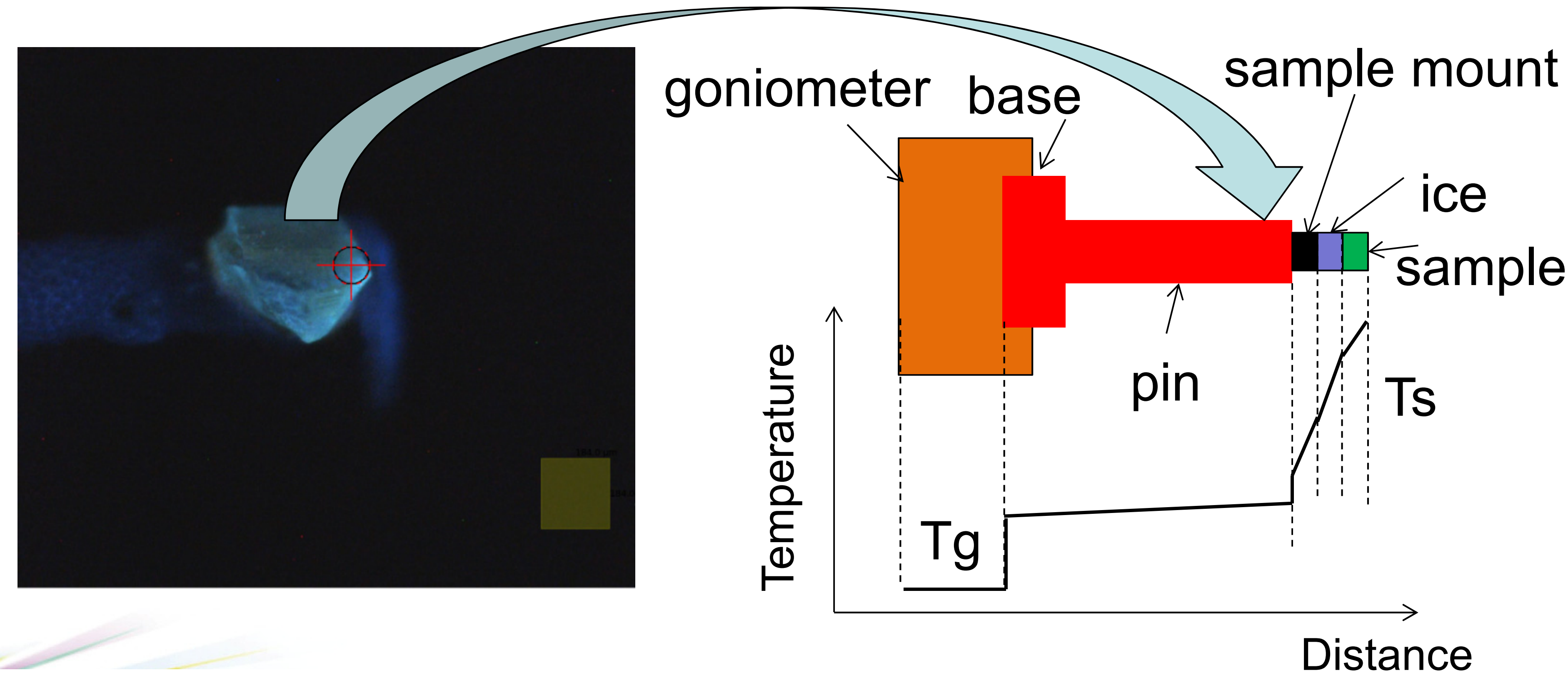
Verification test



BGO sensor mounted on copper pin (square 184x184 μm)

Tgonio=38.6 K DT joint =3 K

Tsensor calc = Tgonio+DT =38.6 K +3 K=41.6 K

Tsensor meas=41.1±0.9 K

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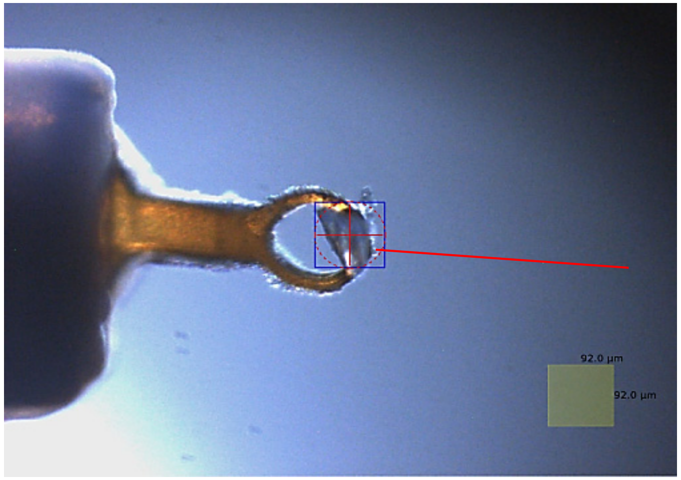
Determination of temperature of the sample mounts



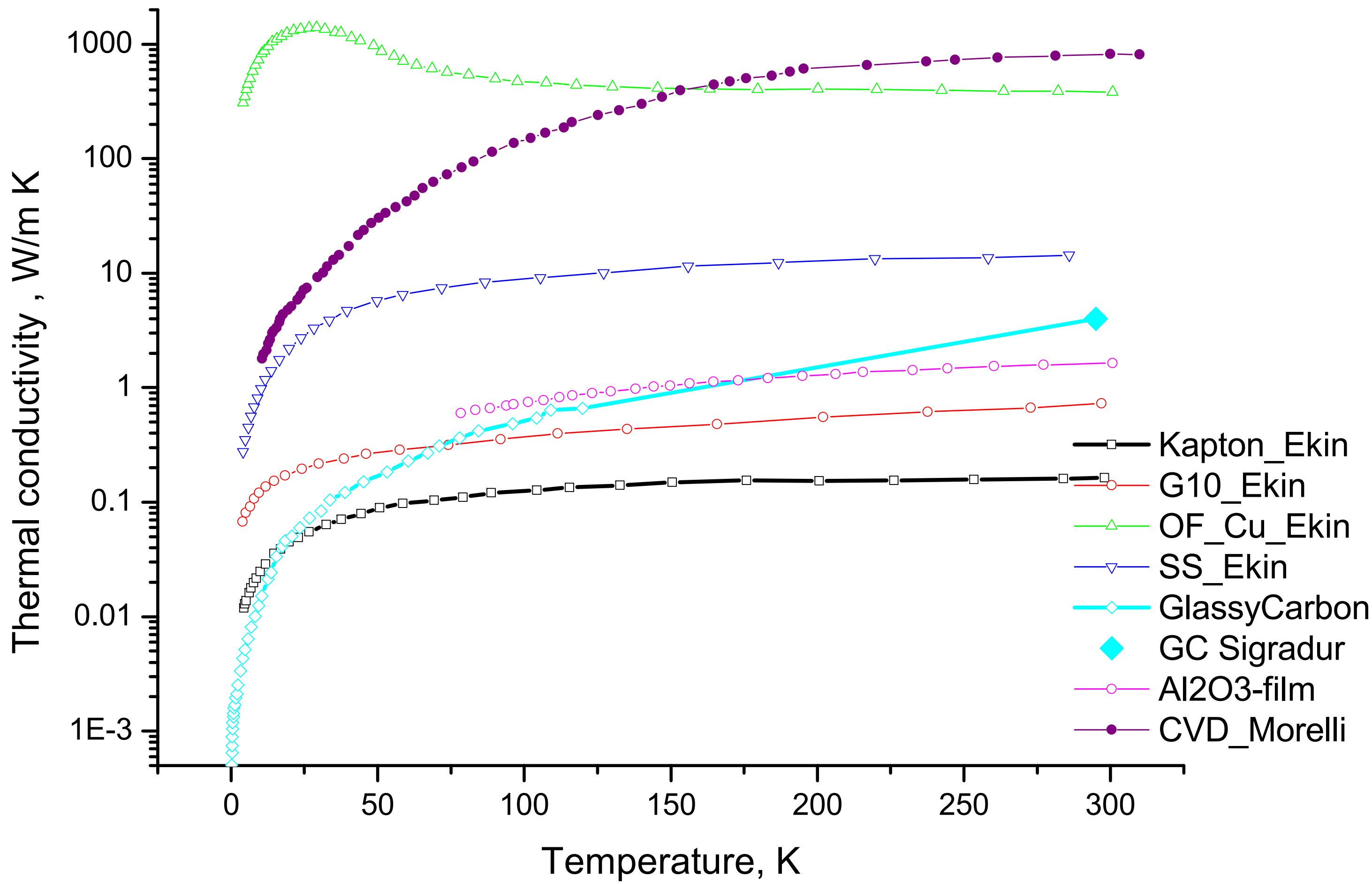
Sensor on the sample mount
Square 92x92µm

Tgonio=40 K

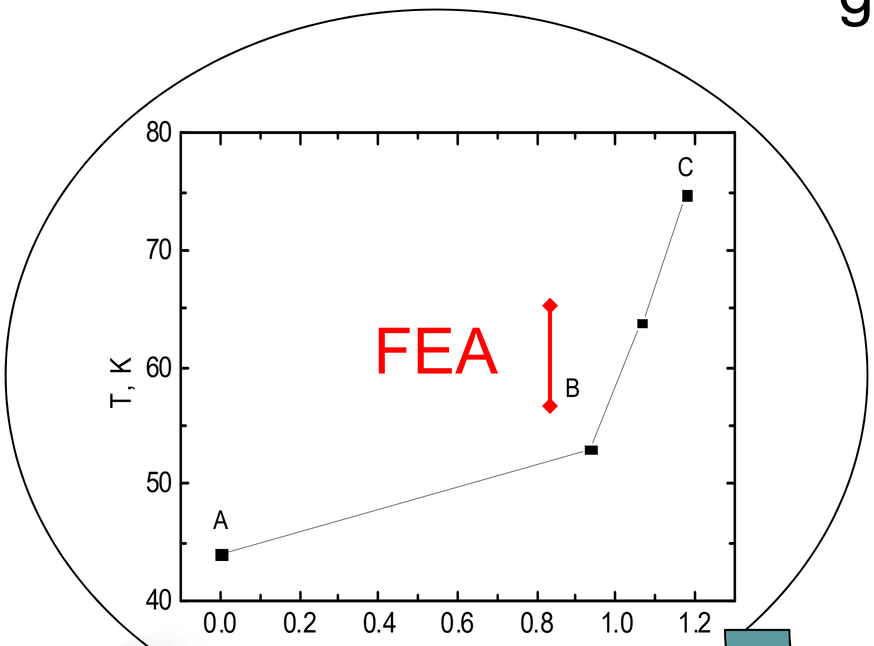
Mount material	Product (Manufacturer)	Thickness, µm	Thermal conductivity, W/mK		Sensor temper., K
			40-100 K	300 K	
Glassy carbon	Sigradur (HTW)	20	0.1-0.5	4.6	**60**
Polyimide	Litholoop (Mol. Dimension)	25	0.2-0.6 0.1-0.2		**68**
Polyimide	Kapton (Dupont)	12	0.2-0.6 0.1-0.2	0.2-0.4	**93**
Polyimide	MicroMount (Mitigen)	10 (loop) 25 (body)	0.2-0.6 0.1-0.2		**110**

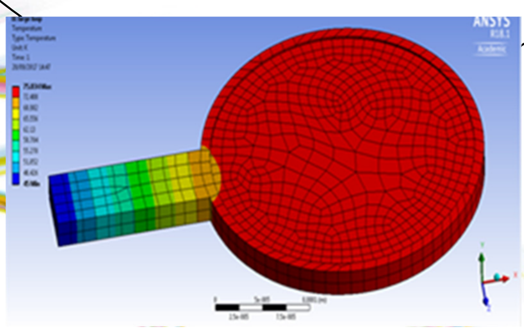
Heat transport and thermal conductivity of different materials vs temperature

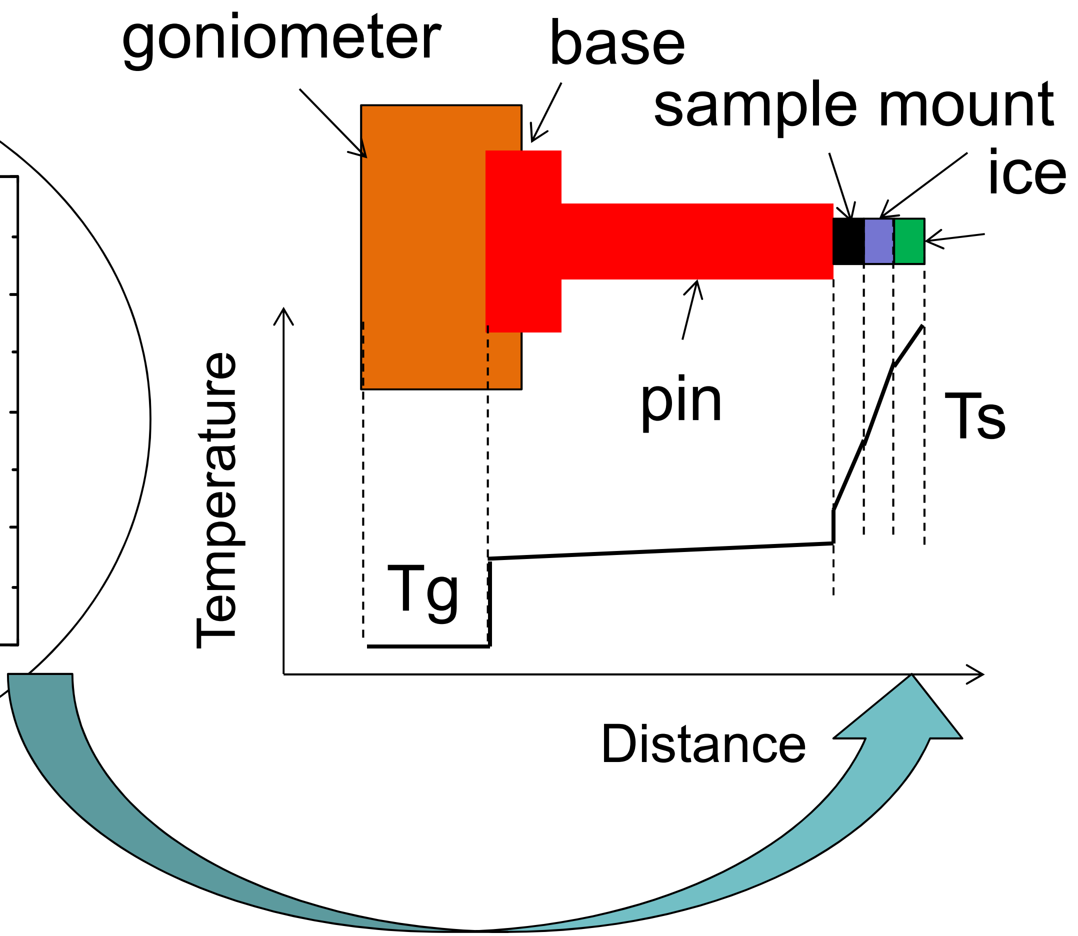
1D steady-state heat transfer $$T2 - T1 = \frac{QL}{Ak}$$



Temperature variation across the sample holder assembly

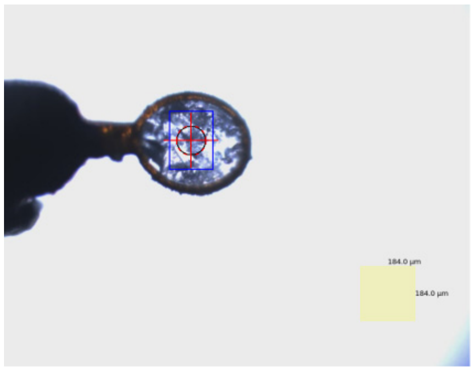


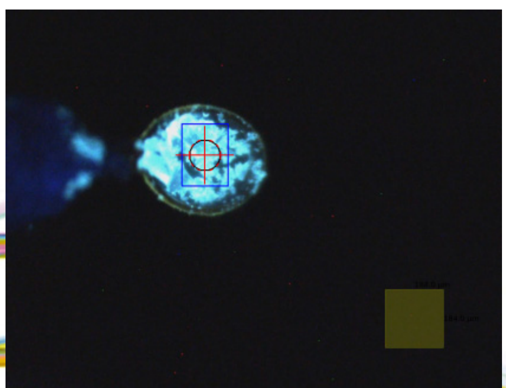




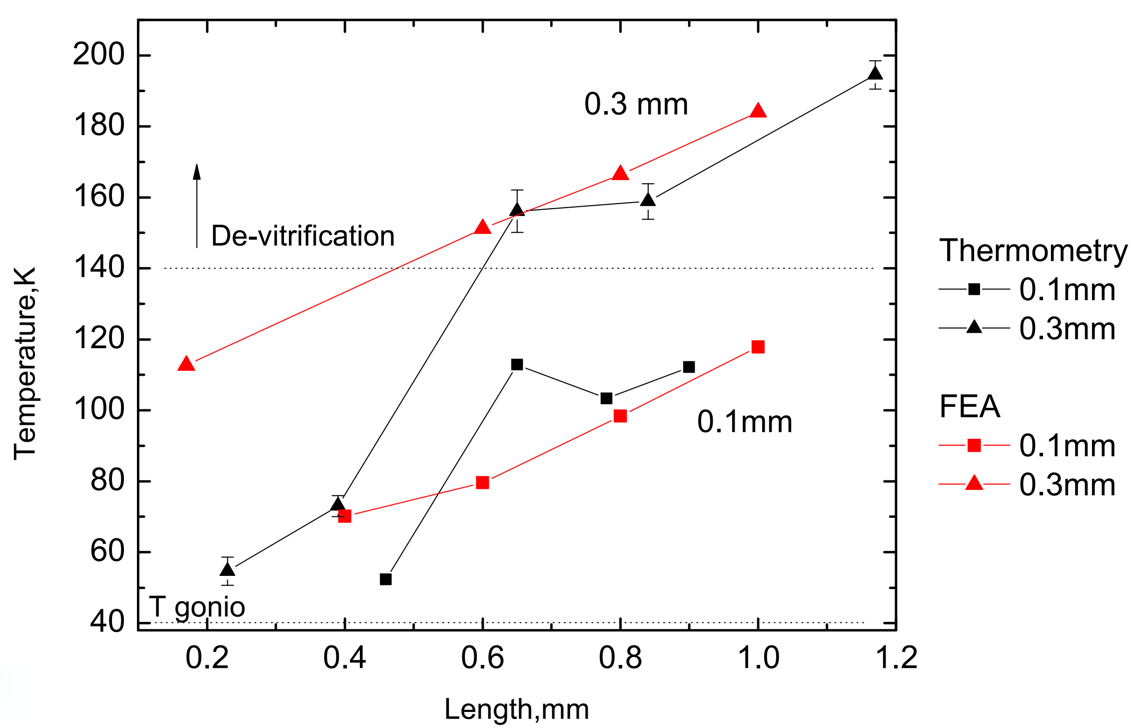
The sample mount is a "bottleneck" for cooling

Temperature variation across the sample mount





The effect of sample mount loop size and length on temperature



Square 92x92µm

Conclusions

Non-contact luminescence cryothermometry at I23

- allows to quantify the process of heat transport in the sample holder assembly
- enables "educated" optimisation of the sample holder assembly as opposed to blind trial-and-error approach.
- offers new experimental possibilities (i.e. studies of temperature induced change in protein crystal during experiment, combined effect of radiation dose & sample heating on structure damages).