The LH-DAC in synchrotrons: general principles and an overview of some important techniques

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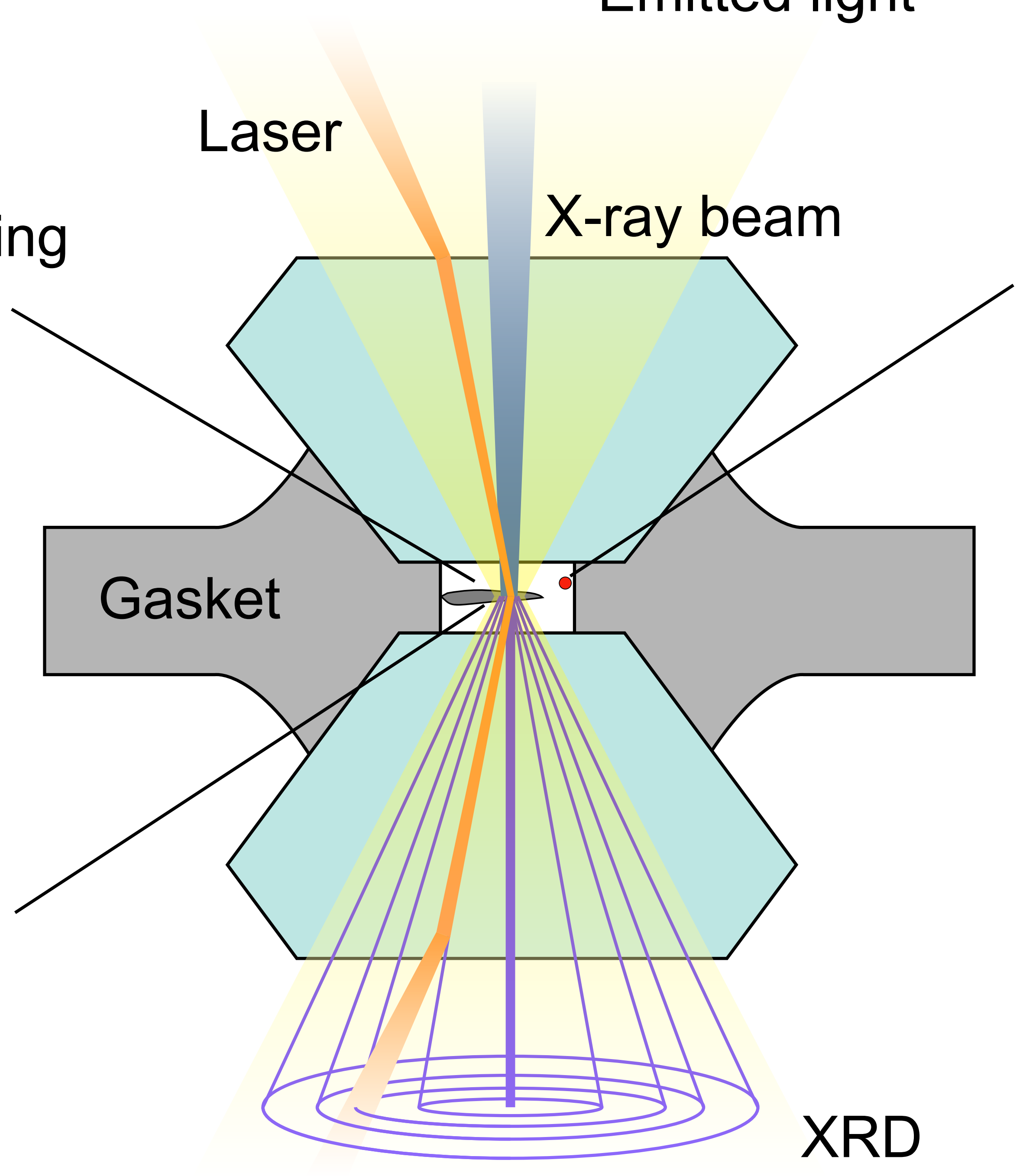
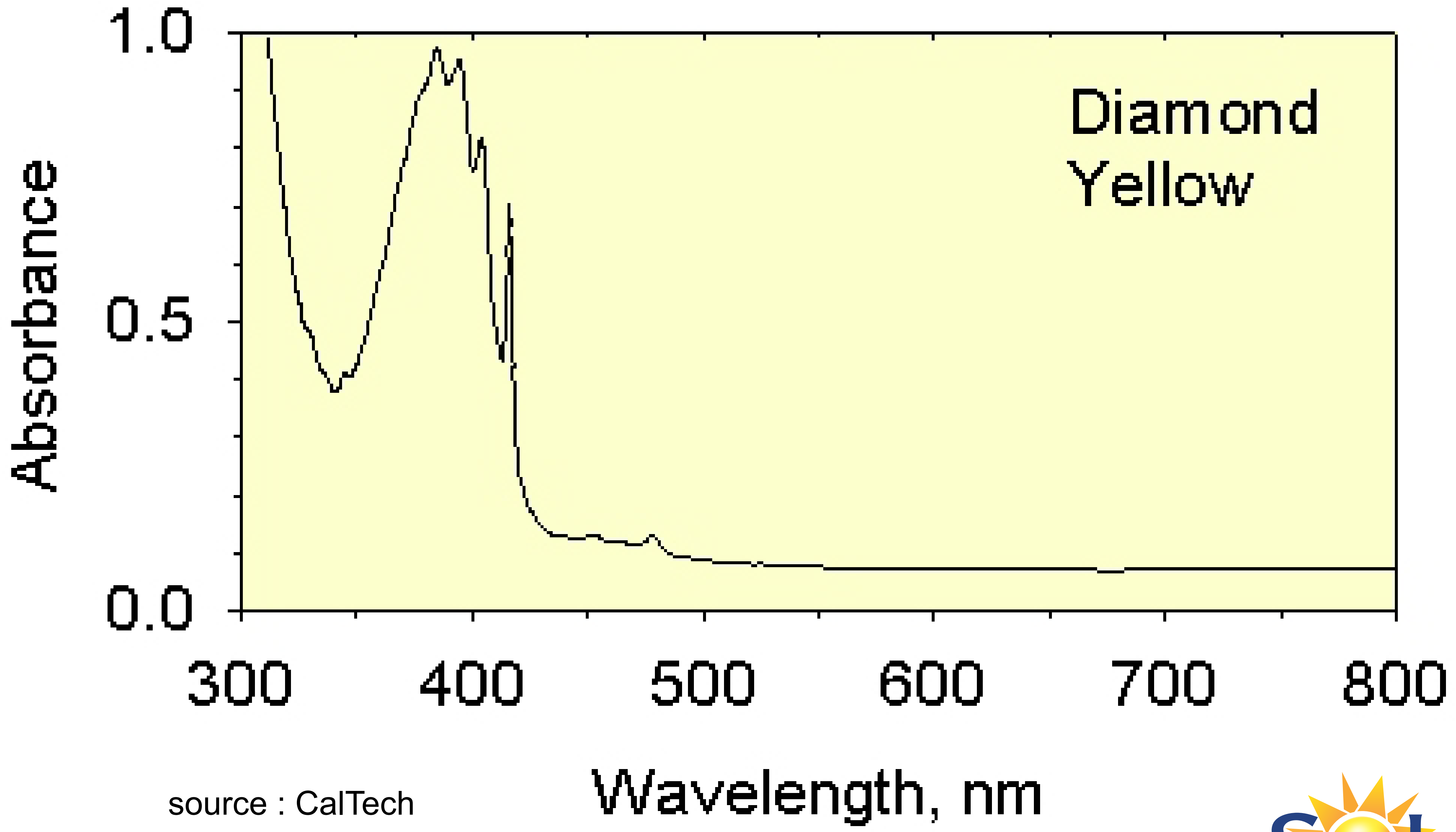
PSICHE beamline, Synchrotron SOLEIL

Why the LH-DAC?

Give access to the highest P-T conditions (static)

Diamonds are transparent in the visible and IR: used for spectroscopy (ex: Raman, IR), but also very useful to focus an IR laser on the sample through the diamonds and to measure the thermal radiation during heating





source : CalTech

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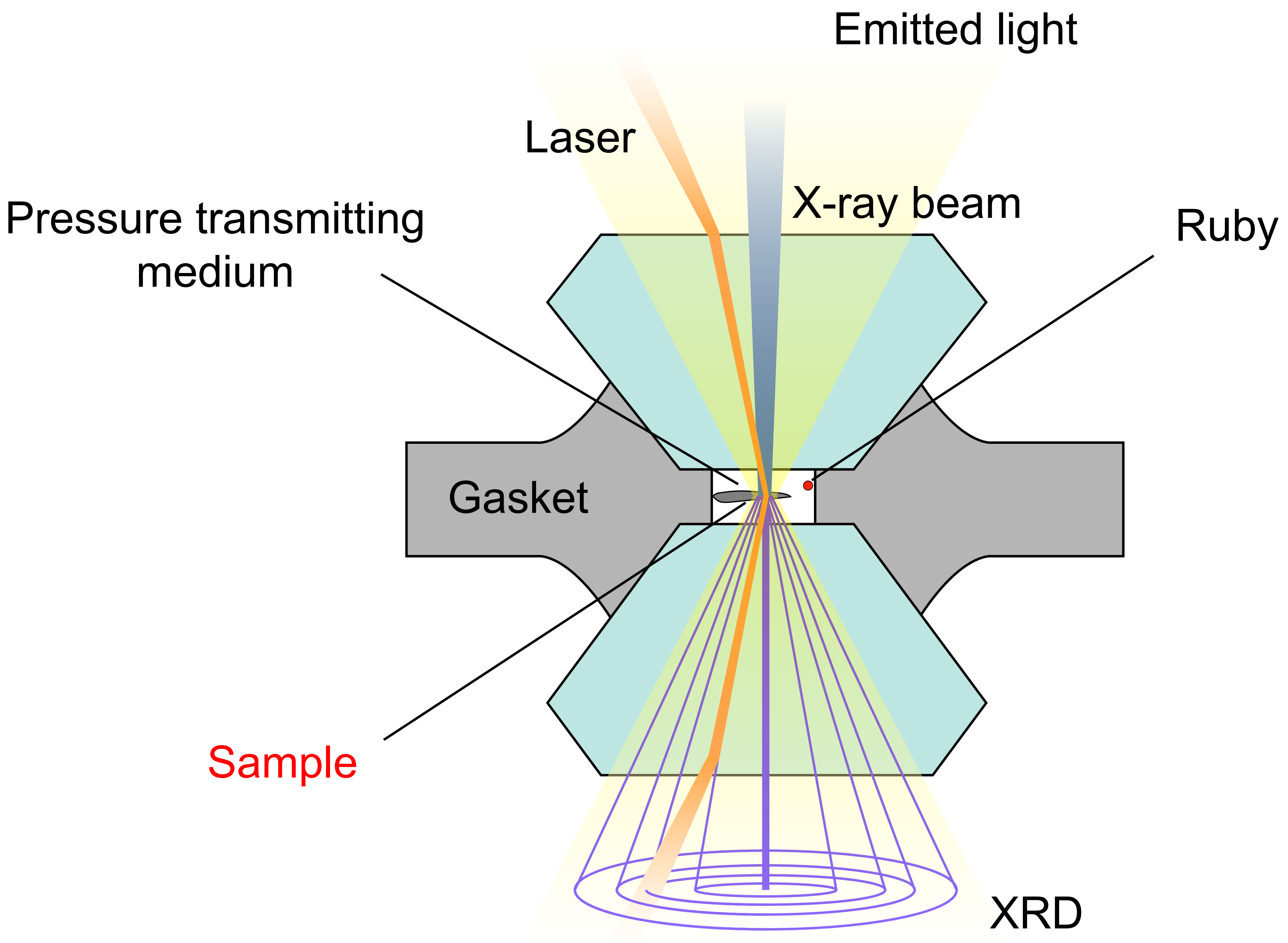

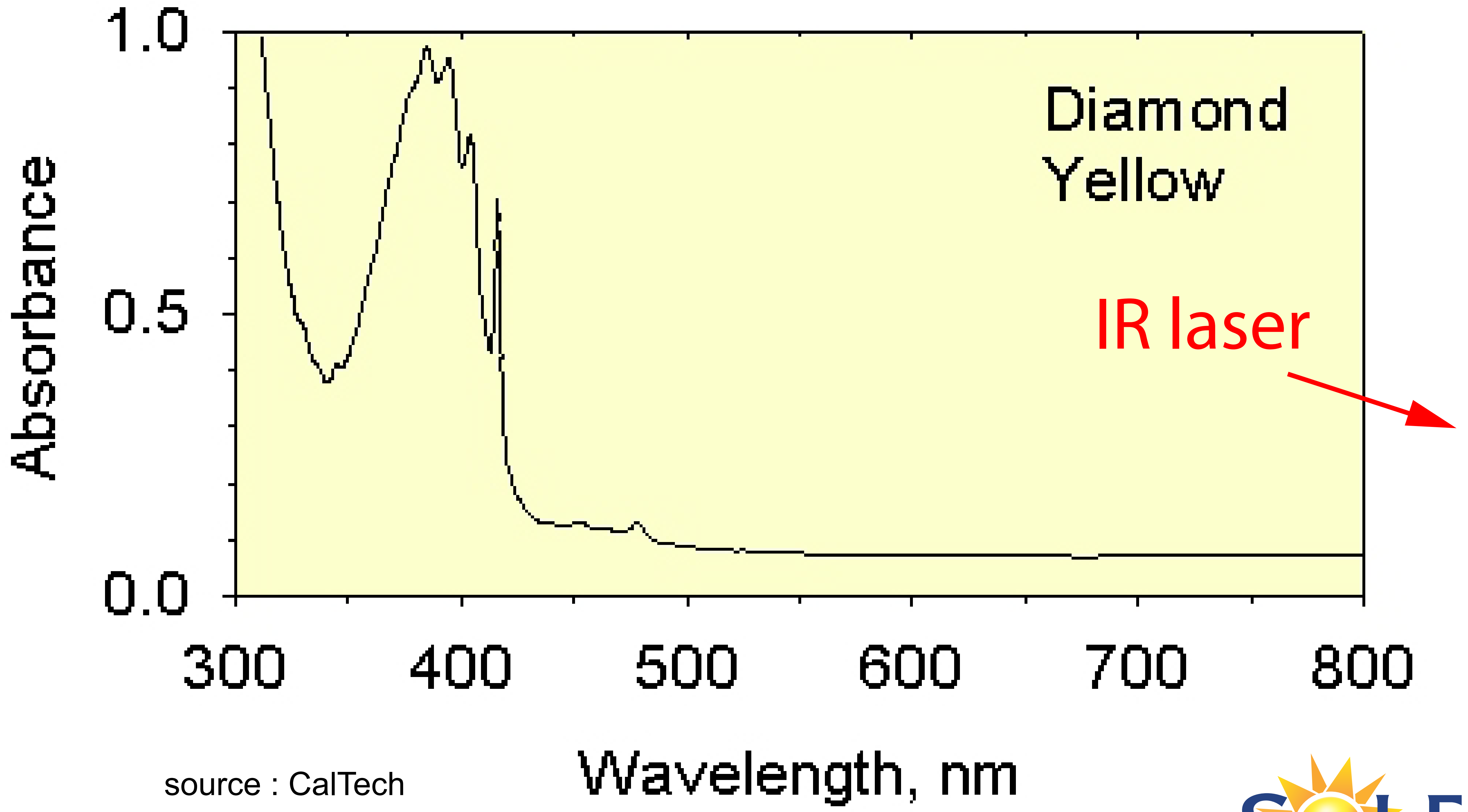

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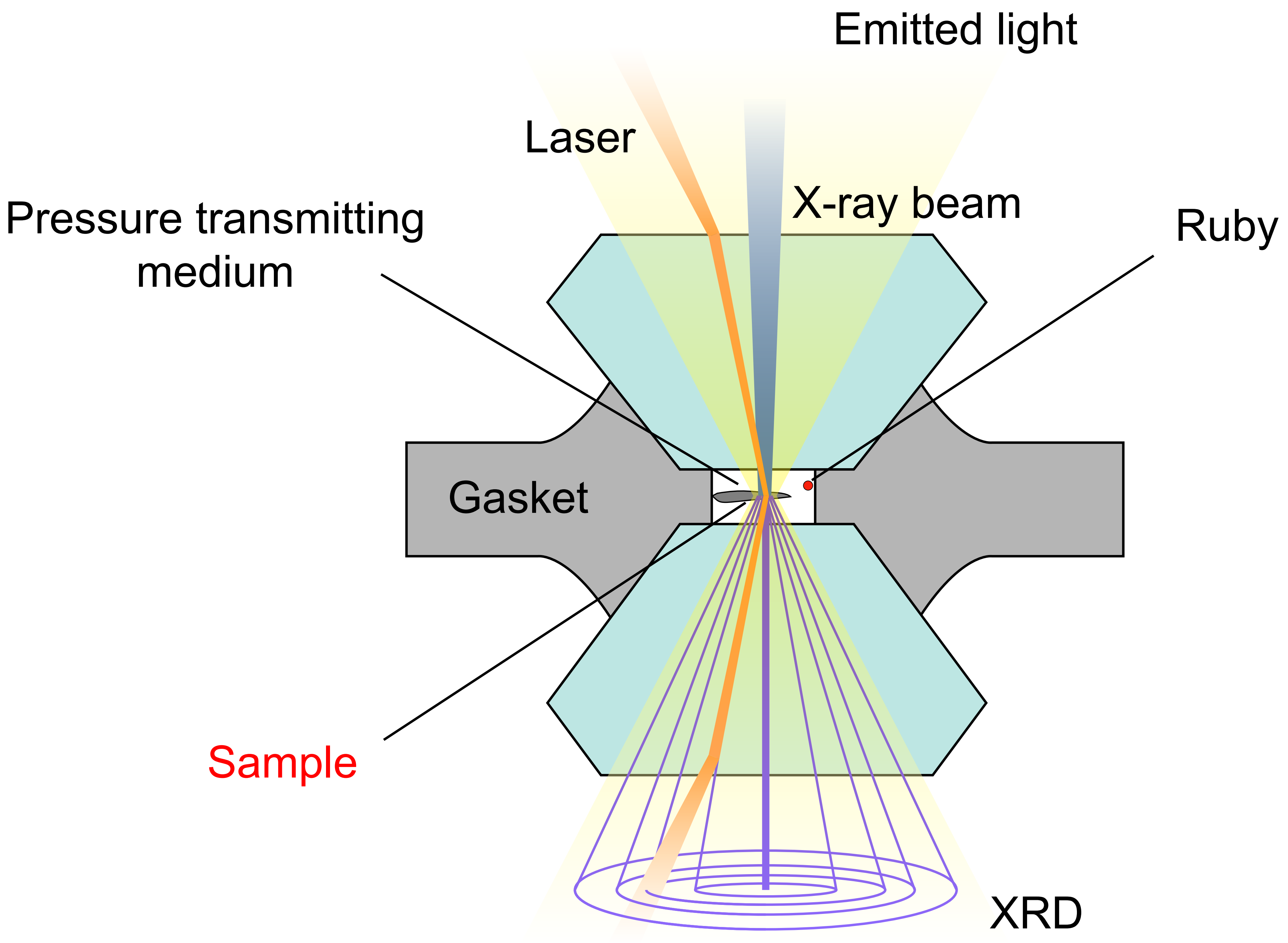

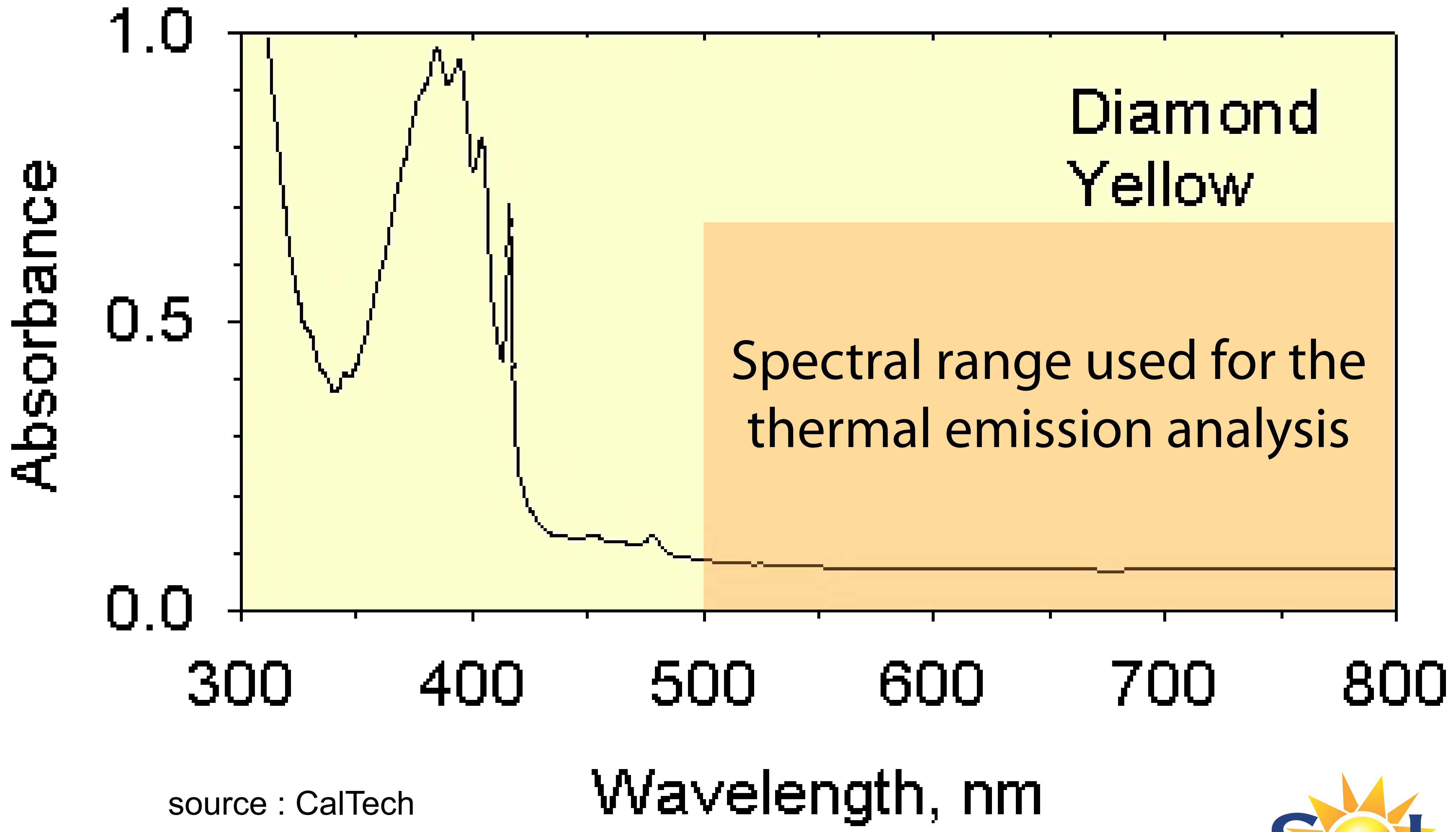

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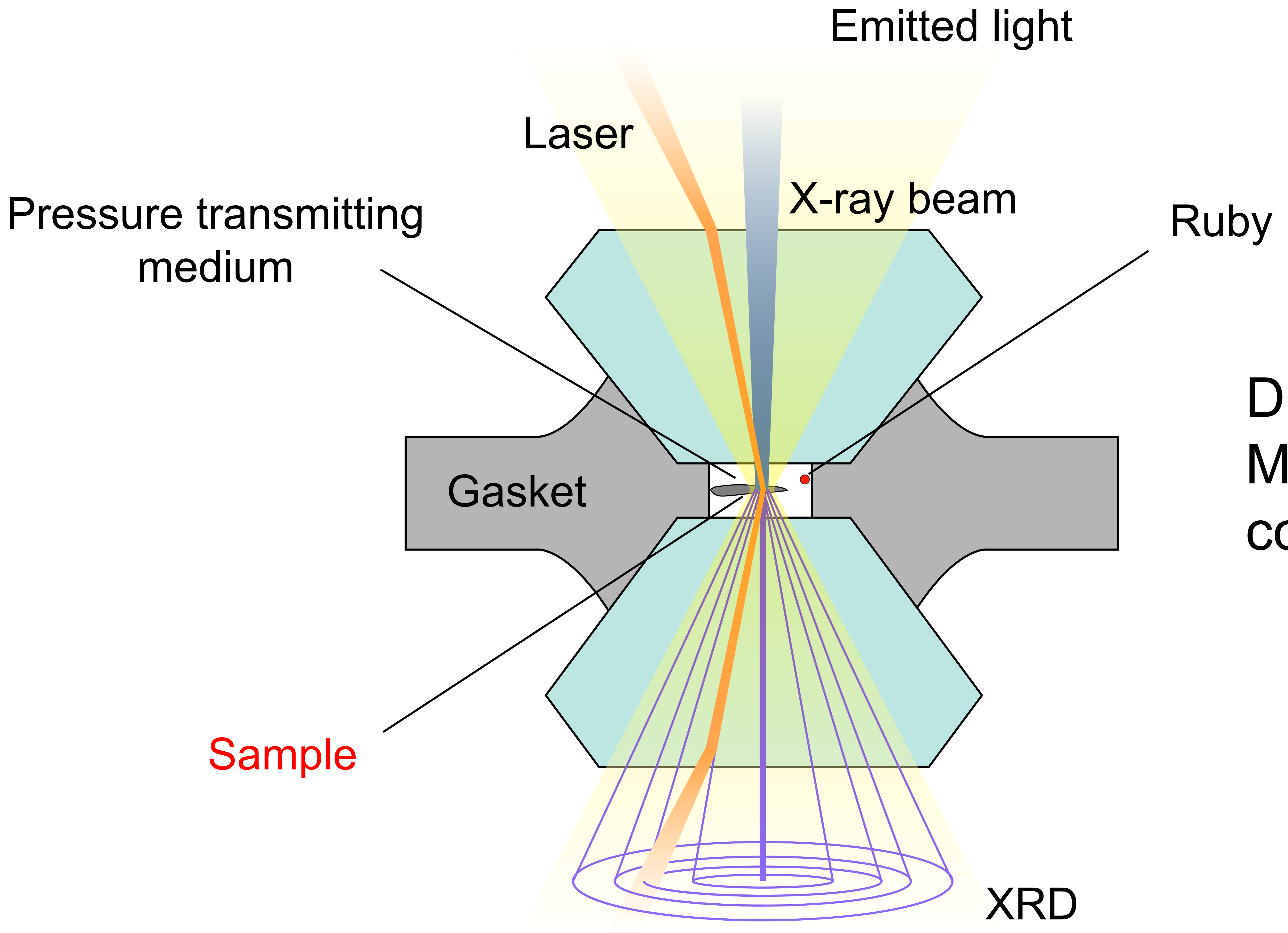


Diamond is also a low Z material (low density). Makes it ideal for in-situ synchrotron studies coupled with LH (XRD, EXAFS)

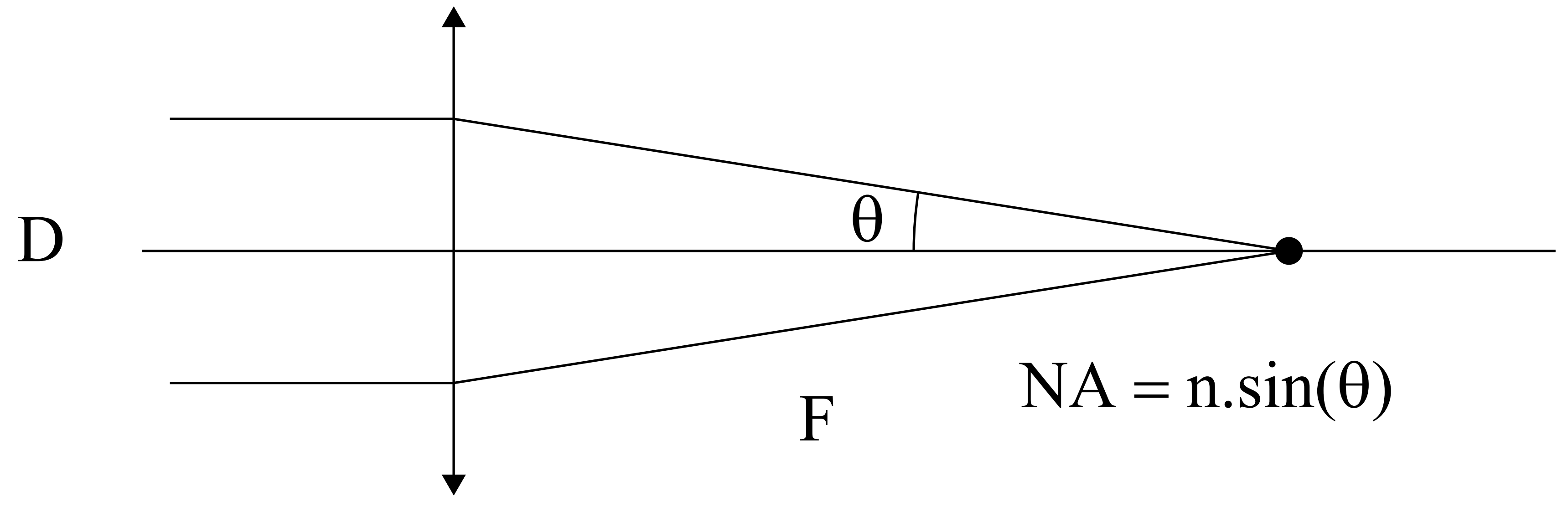
A- Laser focusing

wavelength

lens focal length

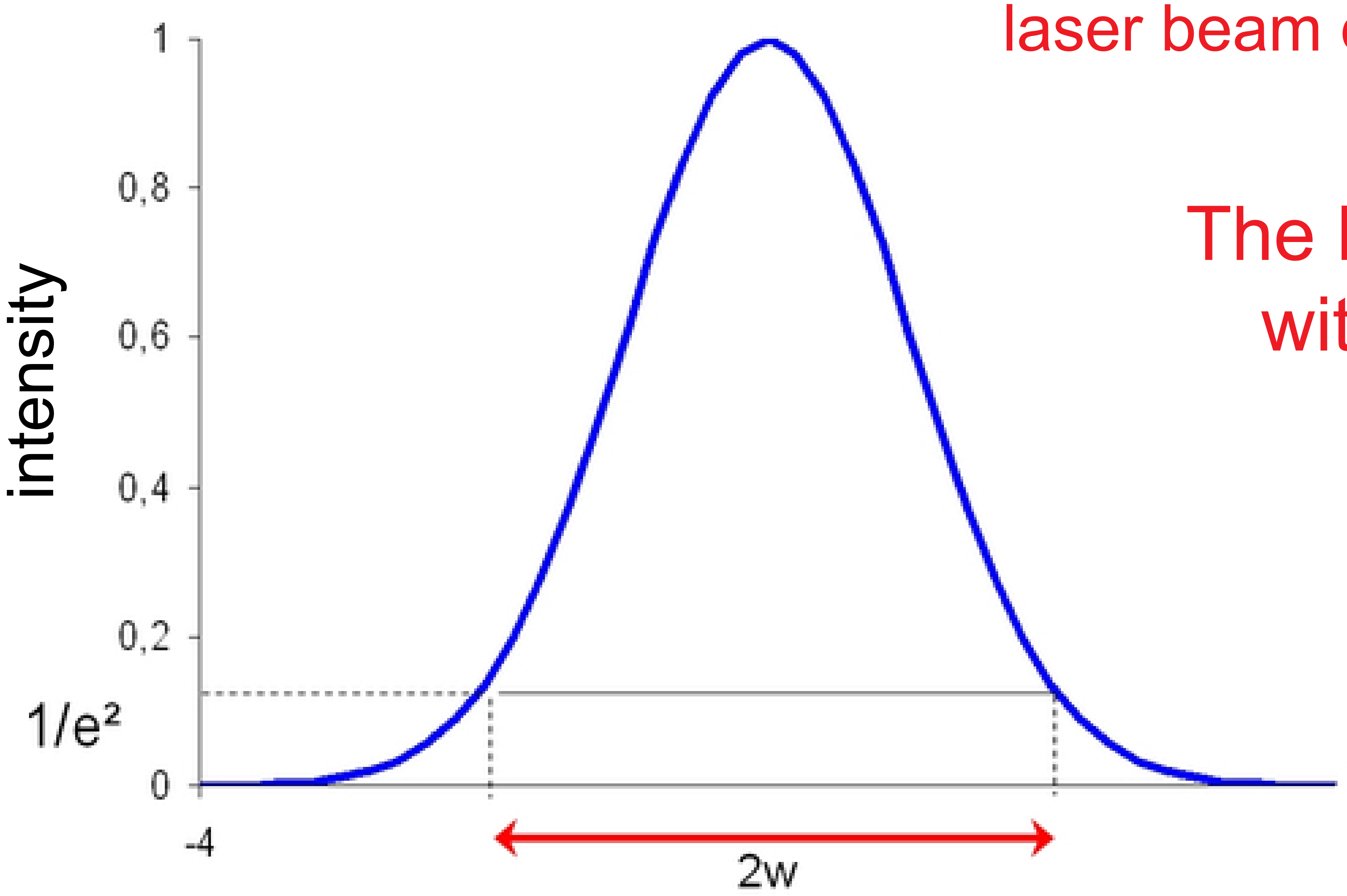
λ YAG = 1,064 µm
λ CO_2 = 10,6 µm

$$2w_0 = \left(\frac{4\lambda}{\pi}\right)\left(\frac{F}{D}\right)$$



laser beam waist

laser beam diameter



The laser beam waist increases with the wavelength and by decreasing the NA

Ex : a 15 µm YAG laser hotspot can be produced with a 5 mm laser beam and a focal length of 55 mm

The hot spot diameter can be adjusted either by (de)focussing or with a beam expander

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A- Laser focusing

wavelength

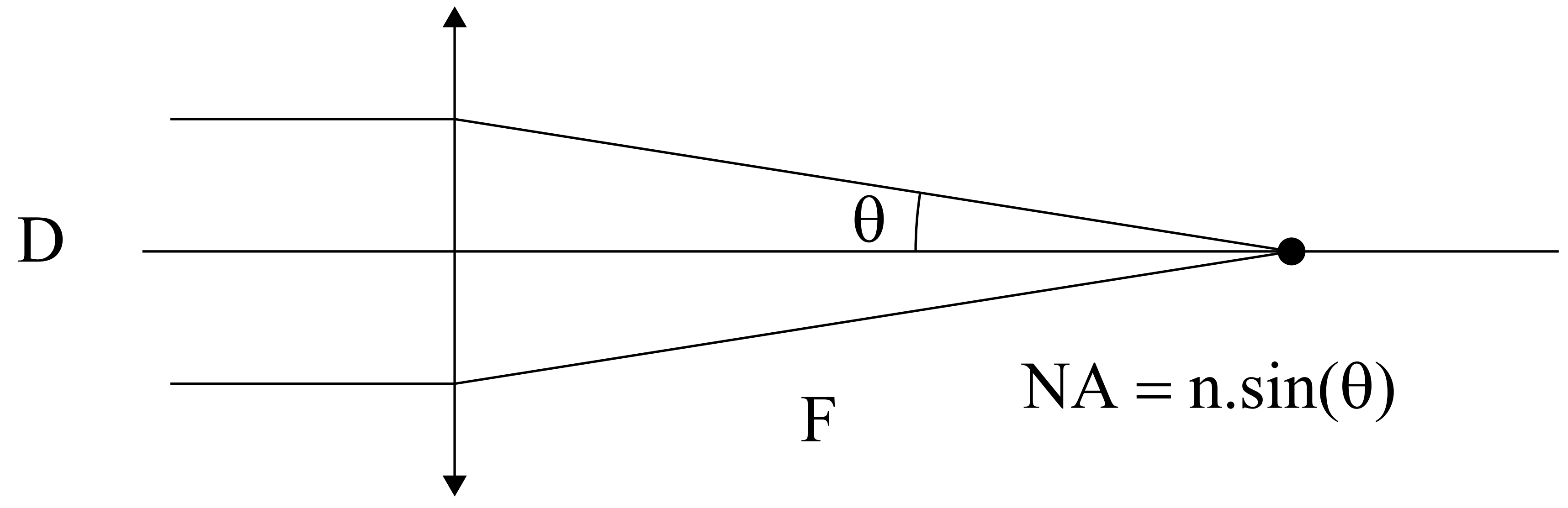
lens focal length

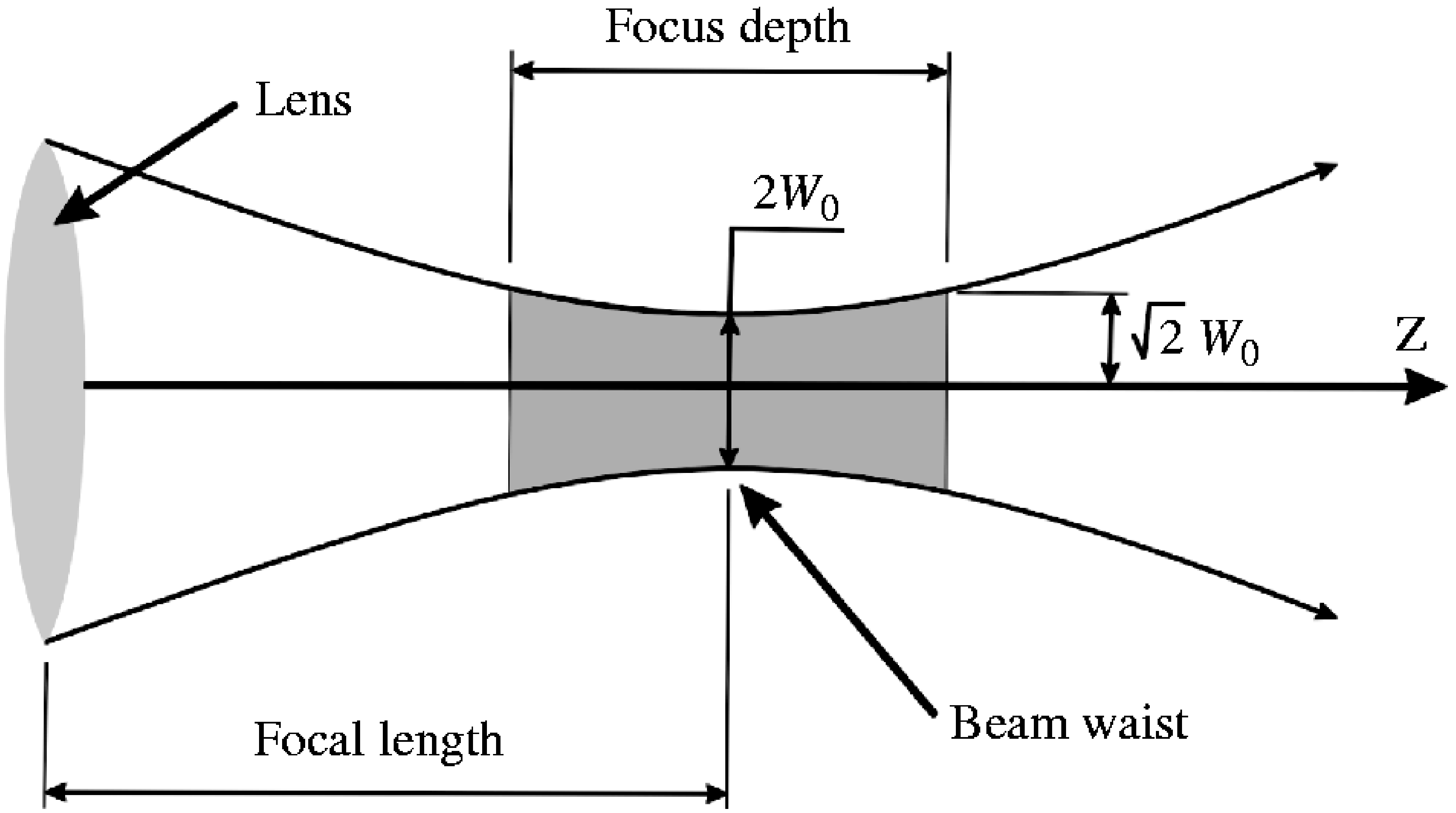
λ YAG = 1,064 μm
λ CO_2 = 10,6 μm

$$DOF = \left(\frac{8\lambda}{\pi}\right)\left(\frac{F}{D}\right)^2$$

laser beam waist

laser beam diameter

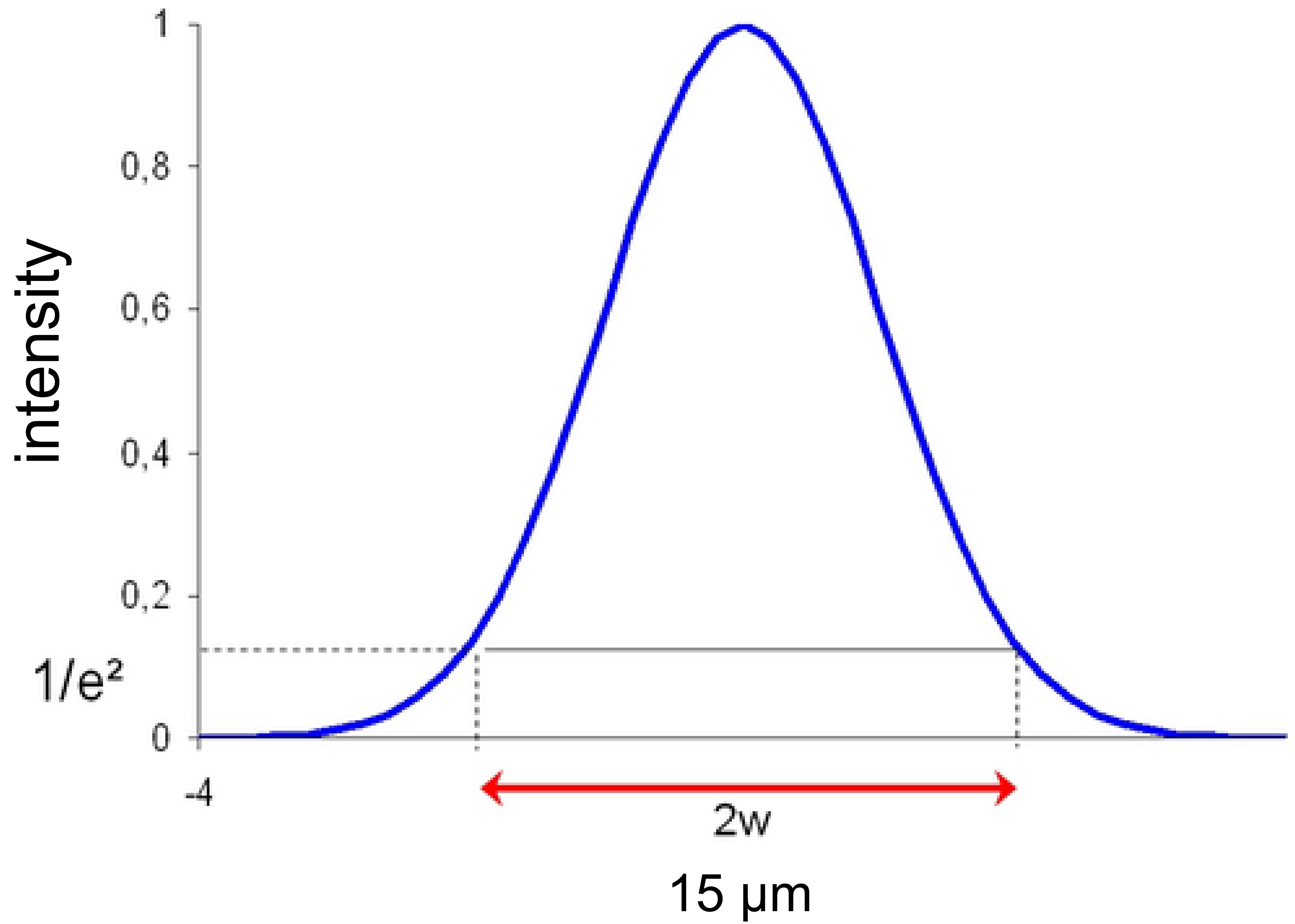




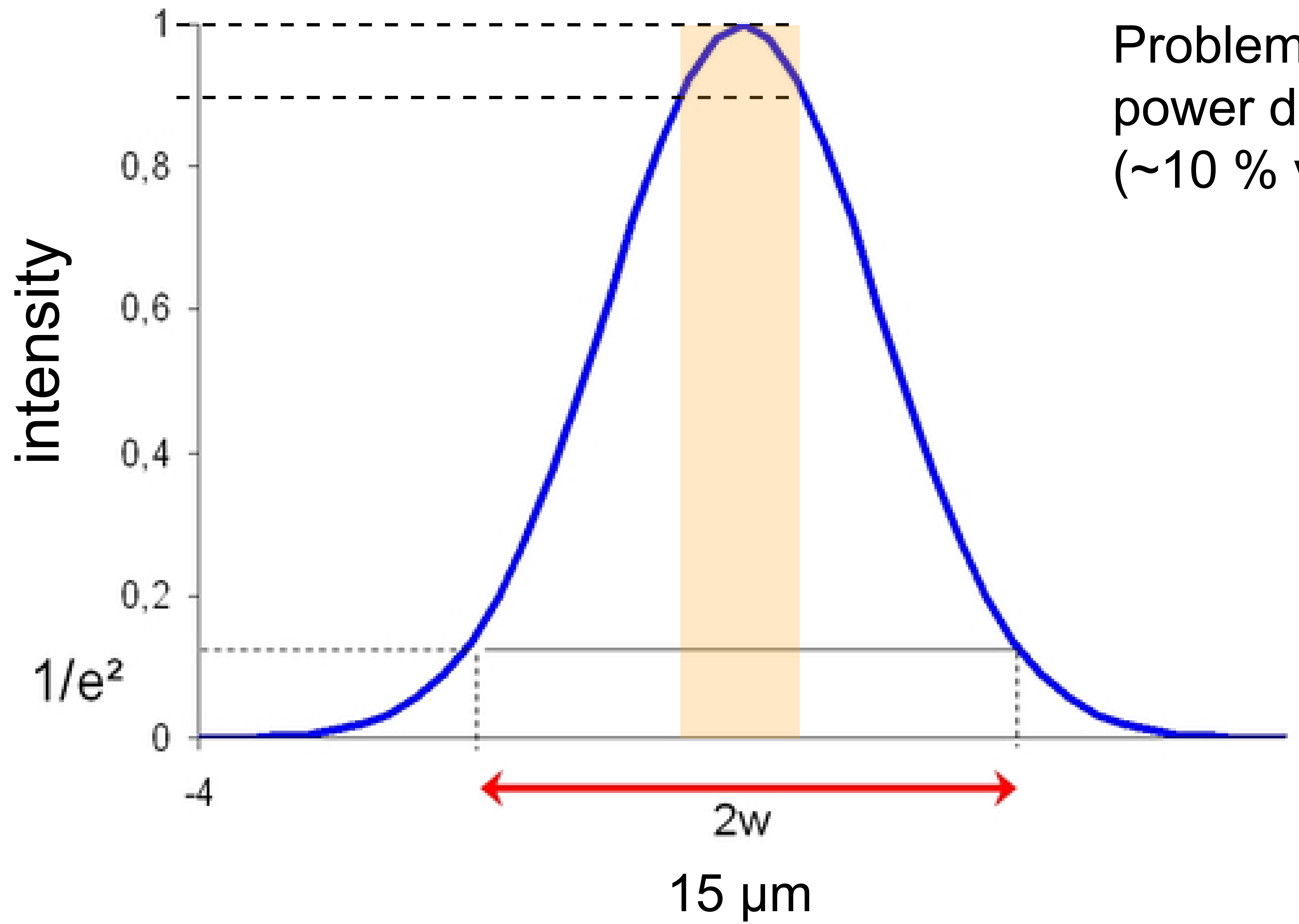
Ex : a 15 μm YAG laser hotspot can be produced with a 5 mm laser beam and a focal length of 55 mm (depth of field: 328 μm)

The hot spot diameter can be adjusted either by (de)focussing or with a beam expander

A- Laser focusing

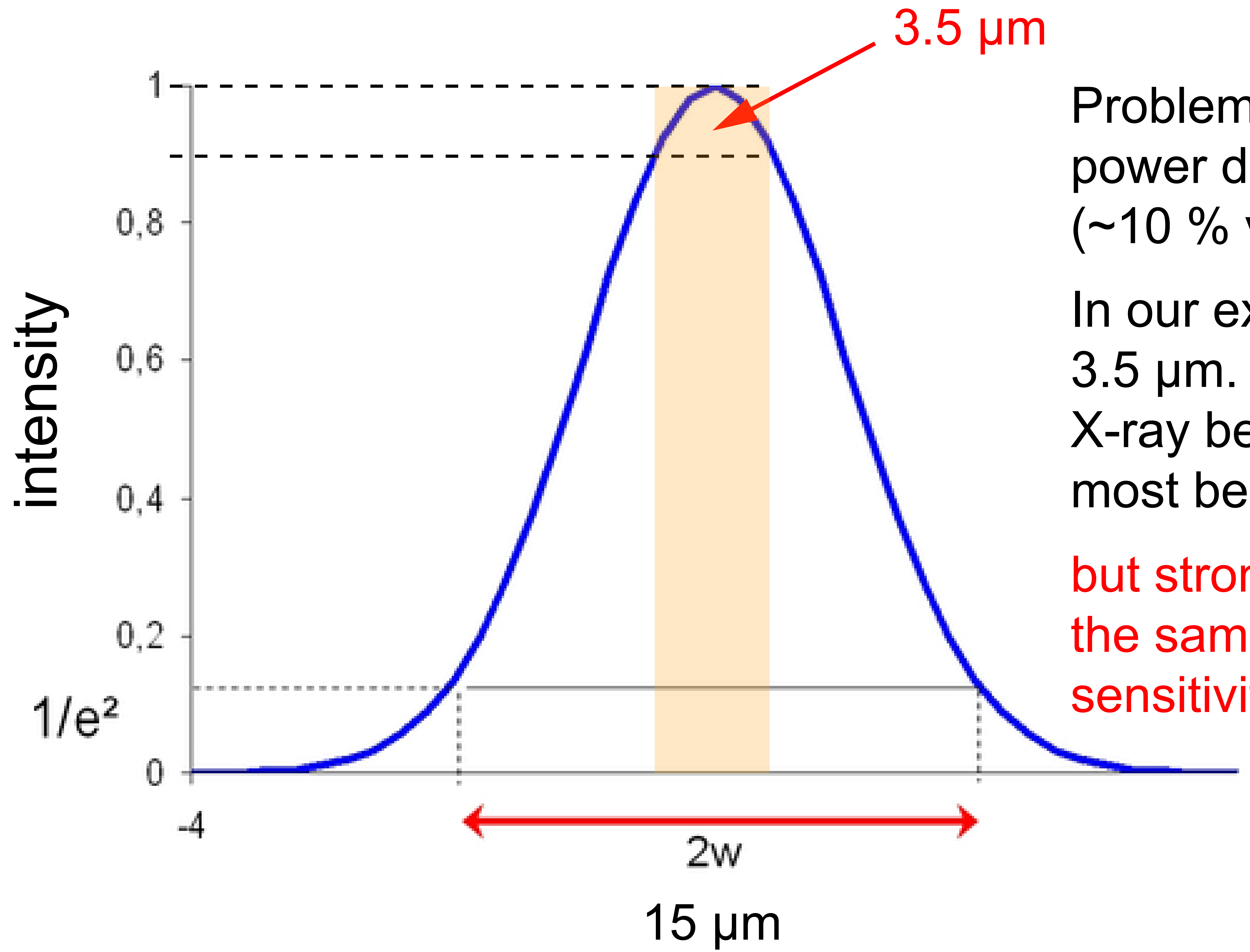


A- Laser focusing



Problem : the region where the power distribution is homogeneous (~10 % variation) is very small

A- Laser focusing



Problem : the region where the power distribution is homogeneous (~10 % variation) is very small

In our example of a 15 µm hot spot: 3.5 µm. This is usually in line with the X-ray beam dimensions available on most beamlines

but strong Gaussian T gradients on the sample surface + increased sensitivity to misalignment

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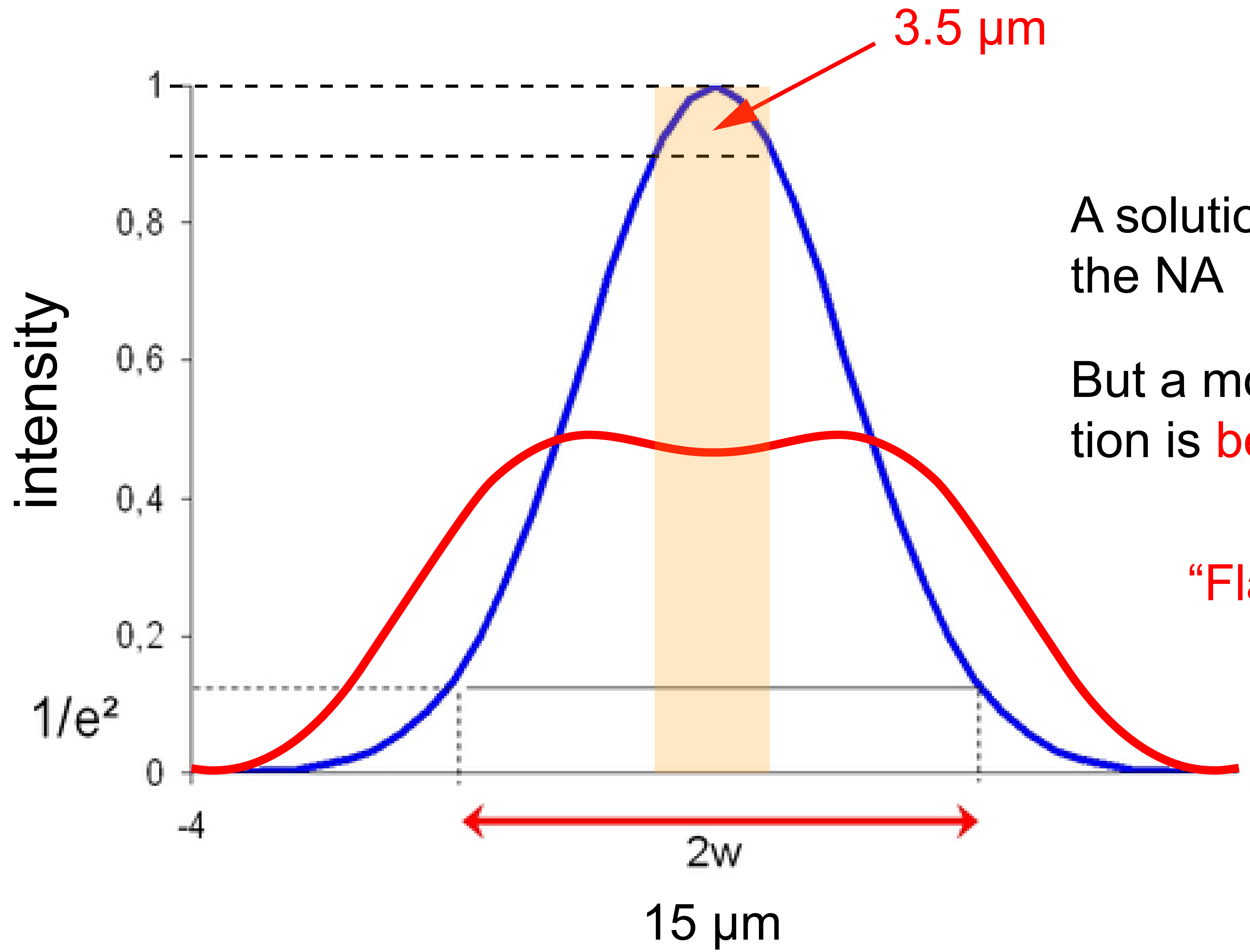
A- Laser focusing



A solution is to defocus or decrease the NA

But a more elegant and efficient solution is beam shaping

"Flat-top", "Top-hat" profiles

(Prakapenka et al. 2008)

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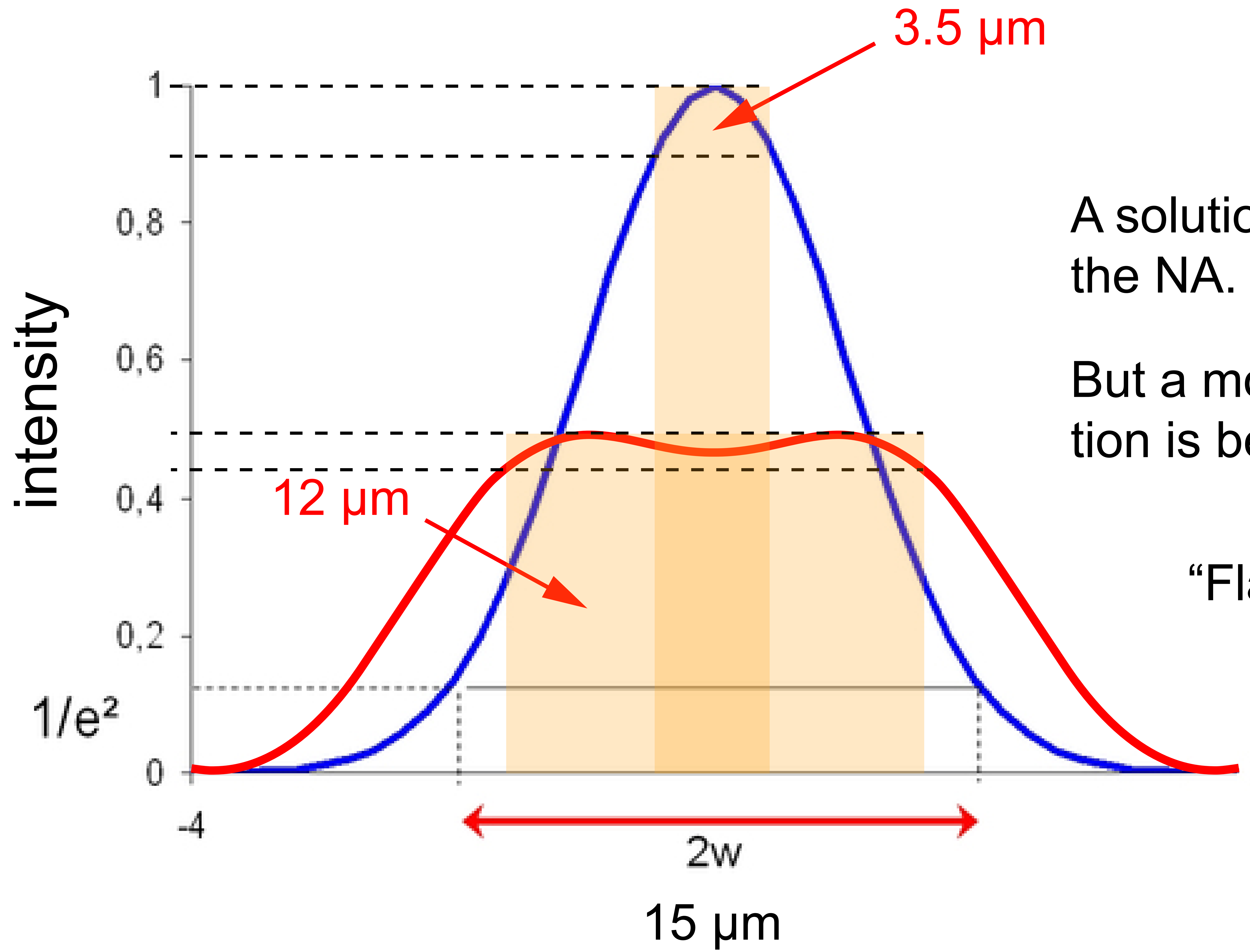
A- Laser focusing



A solution is to defocus or decrease the NA.

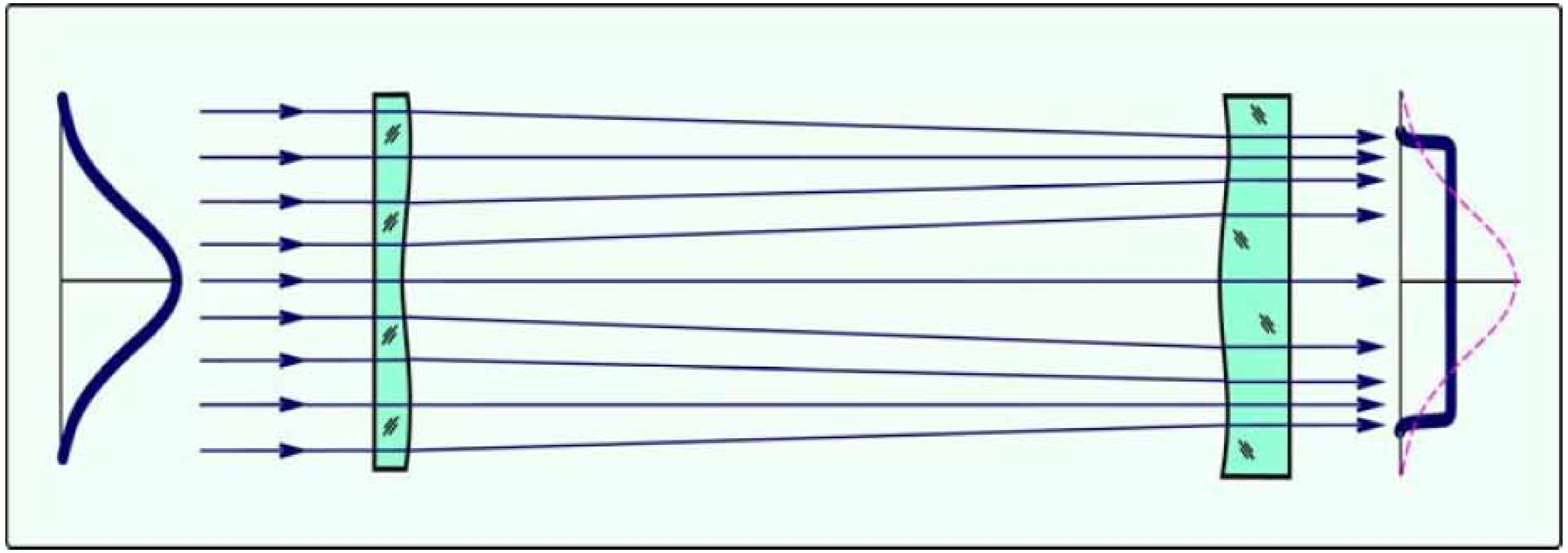
But a more elegant and efficient solution is beam shaping.

"Flat-top", "Top-hat" profiles

A- Laser focusing

source: A. Laskin, AdlOptica

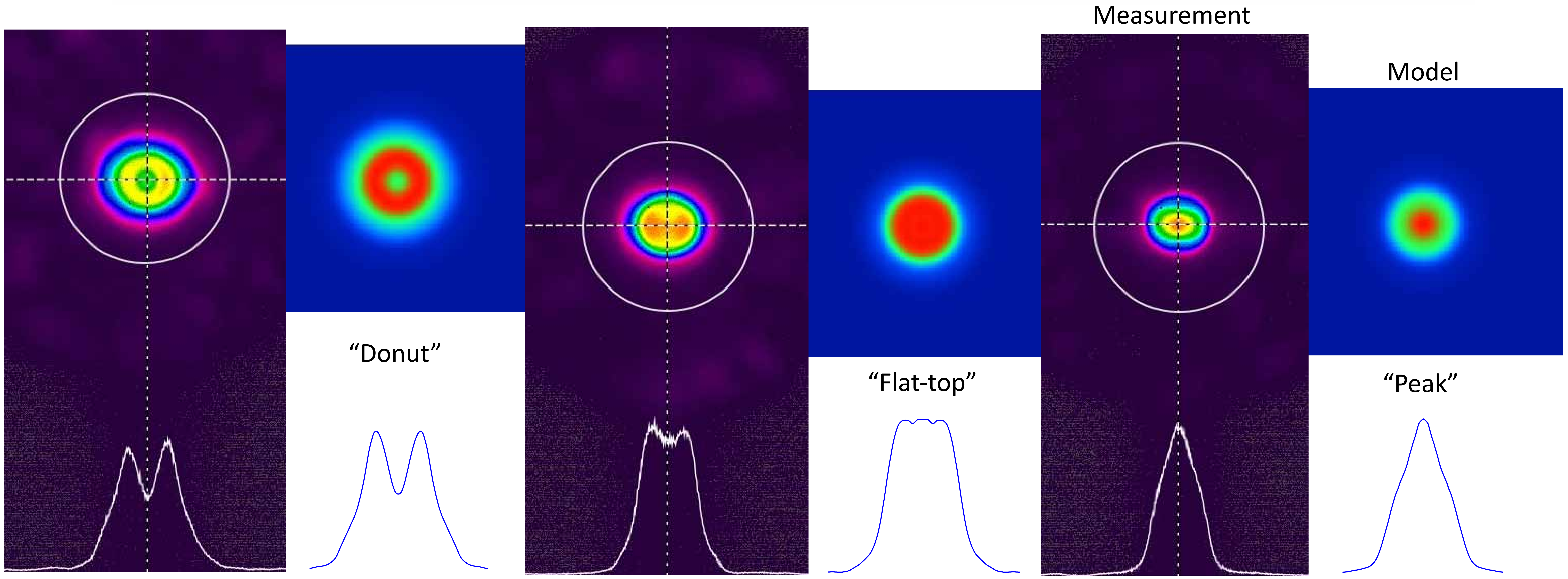
Beam shaping is done by distorsion of the laser beam wavefront inside the optical system (refractive beam shapers)



A- Laser focusing

beam shape (focus)

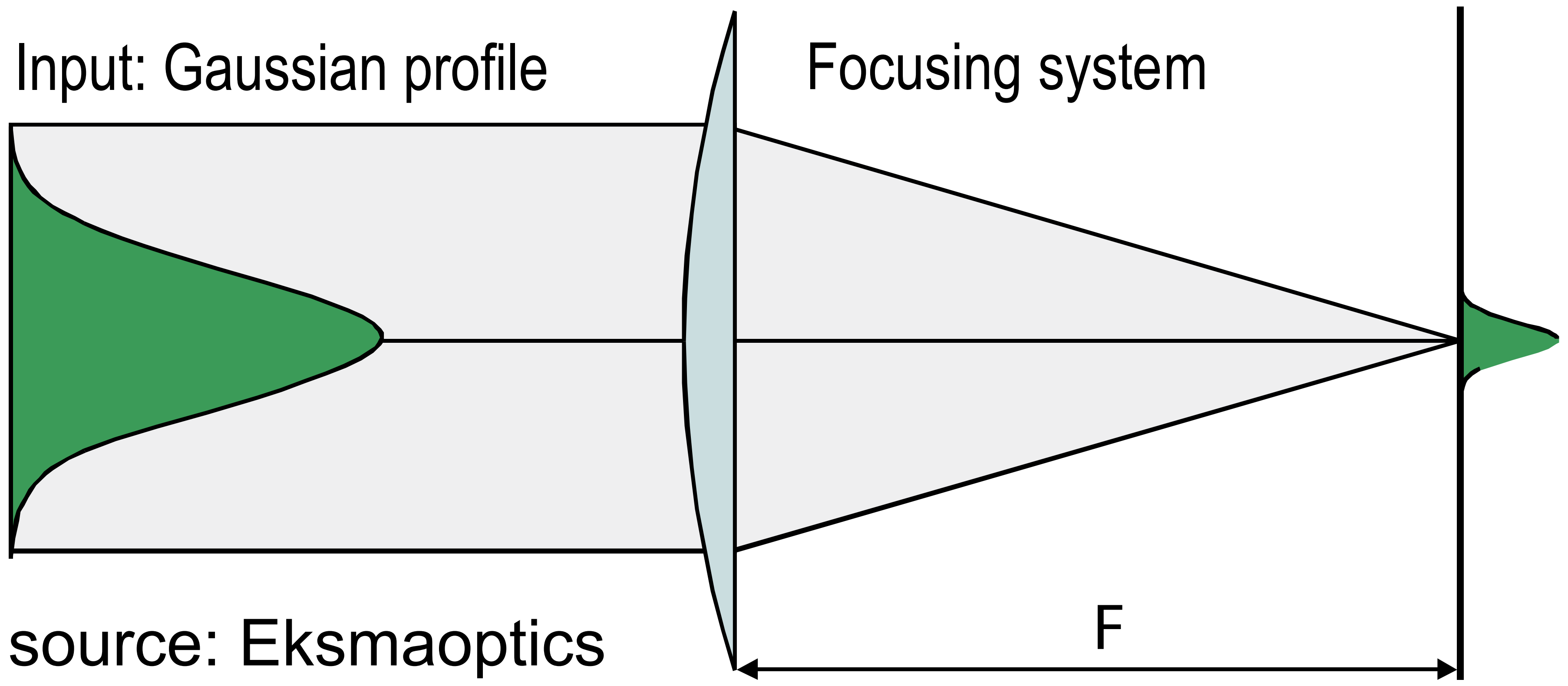
source: A. Laskin, AdlOptica



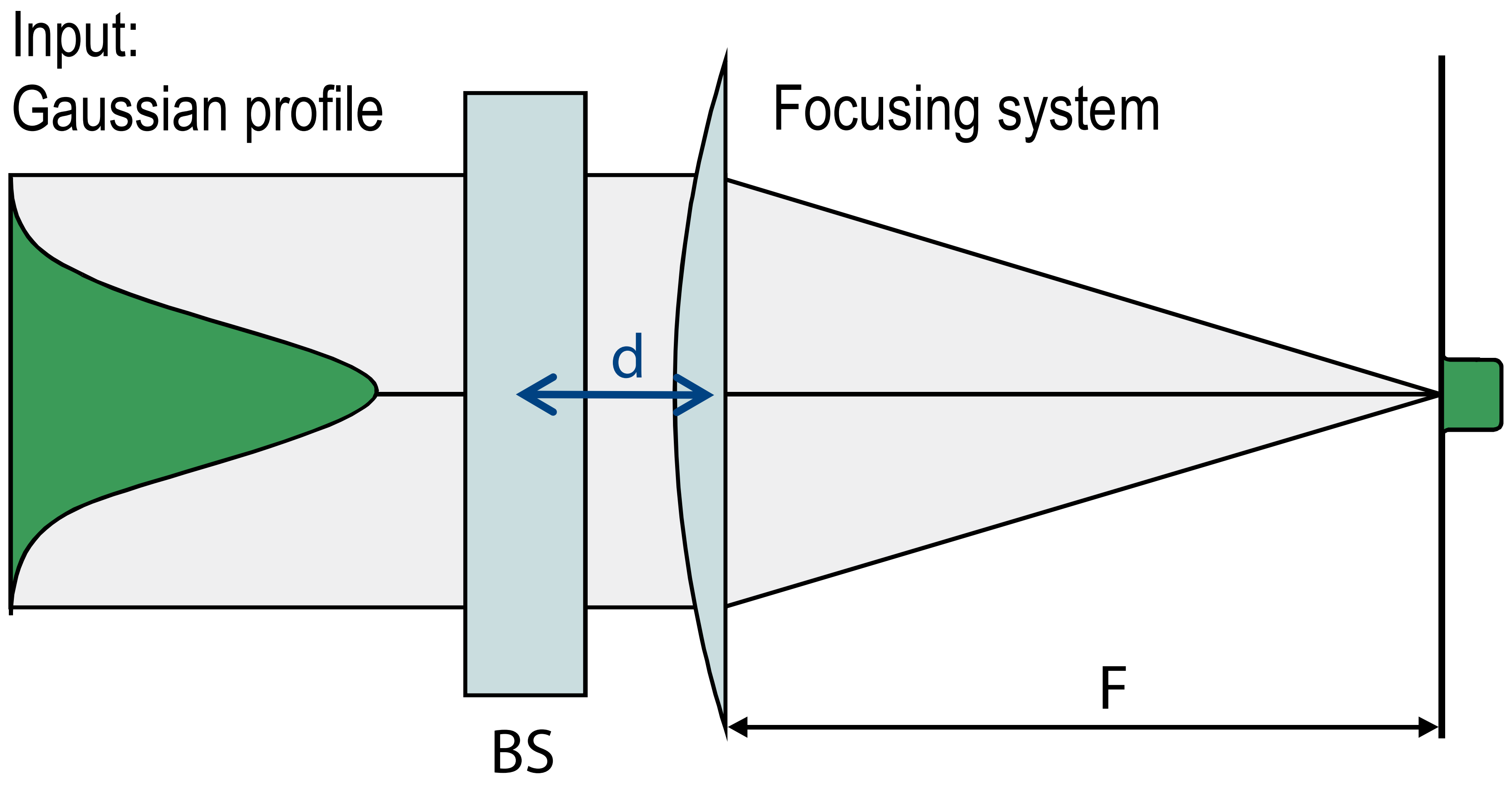
A- Laser focusing

Another type of beam shaper: Diffractive optical element (DOE)

Uses thin micro-structure patterns to alter the phase of the light that is propagated through it



source: Eksmaoptics

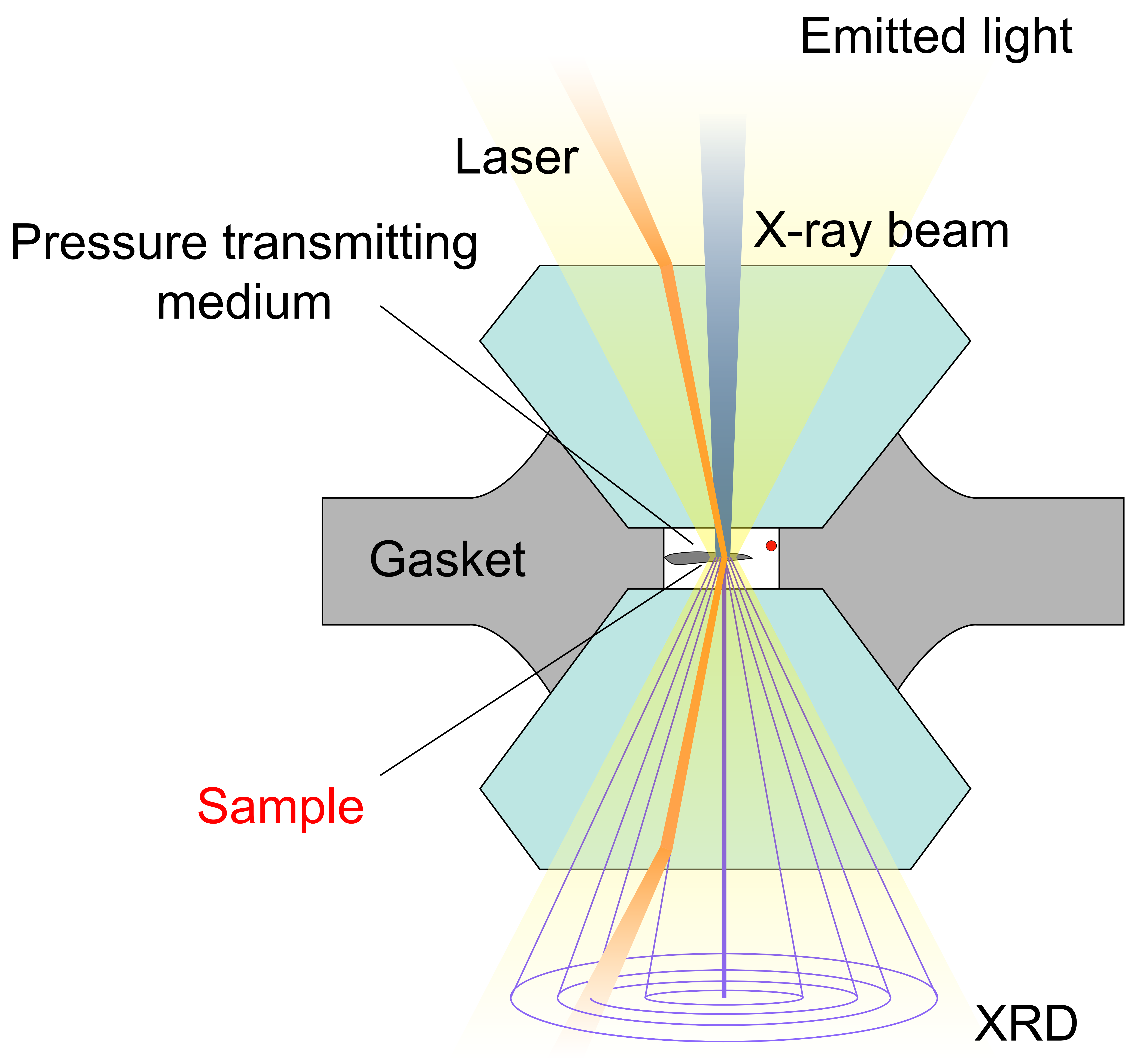


Without beam shaper: Gaussian profile at focal plane

With beam shaper: Top-Hat profile at focal plane

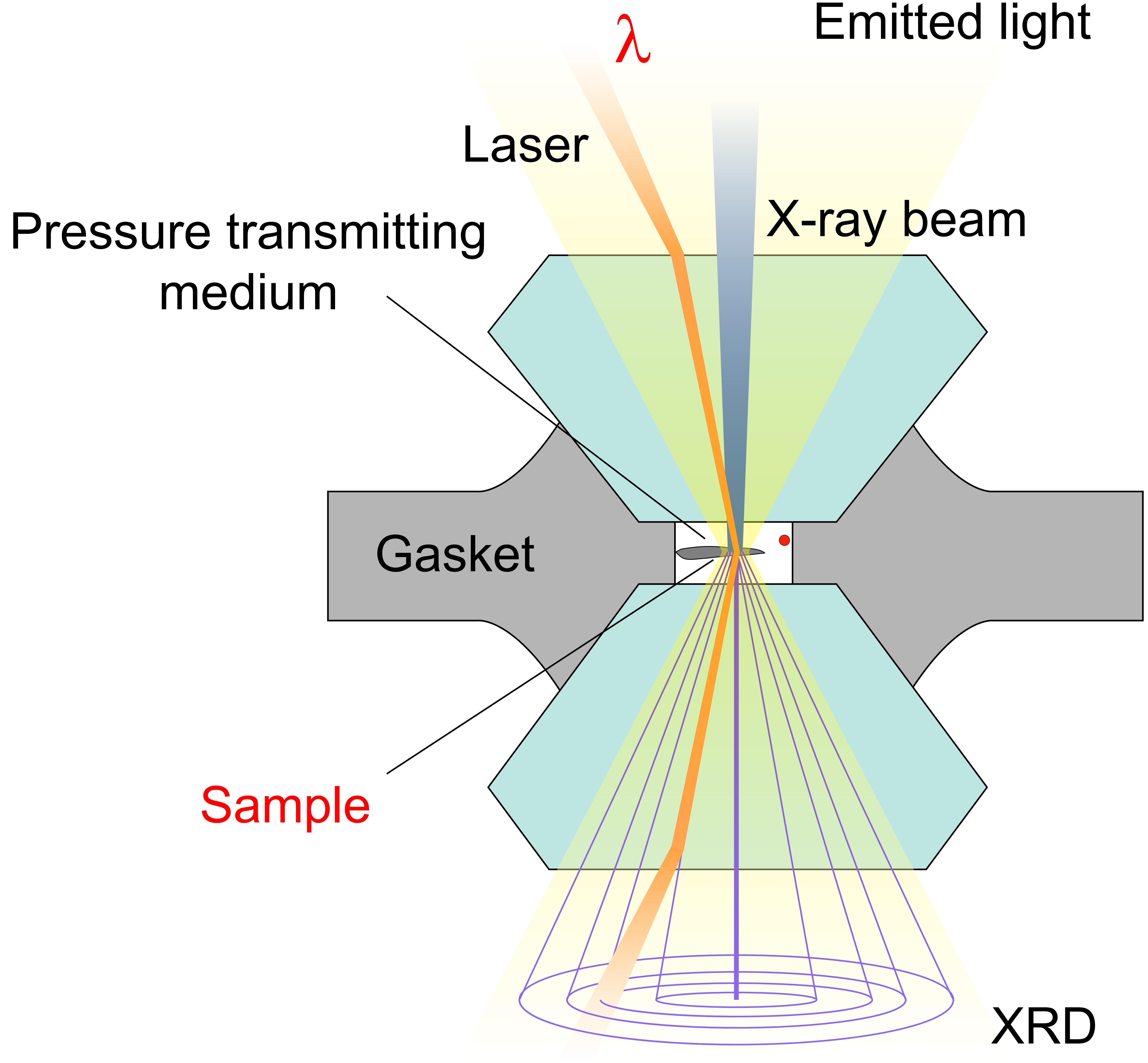
Available shapes: square and round

B- Laser heating



Laser beam absorption depends on:

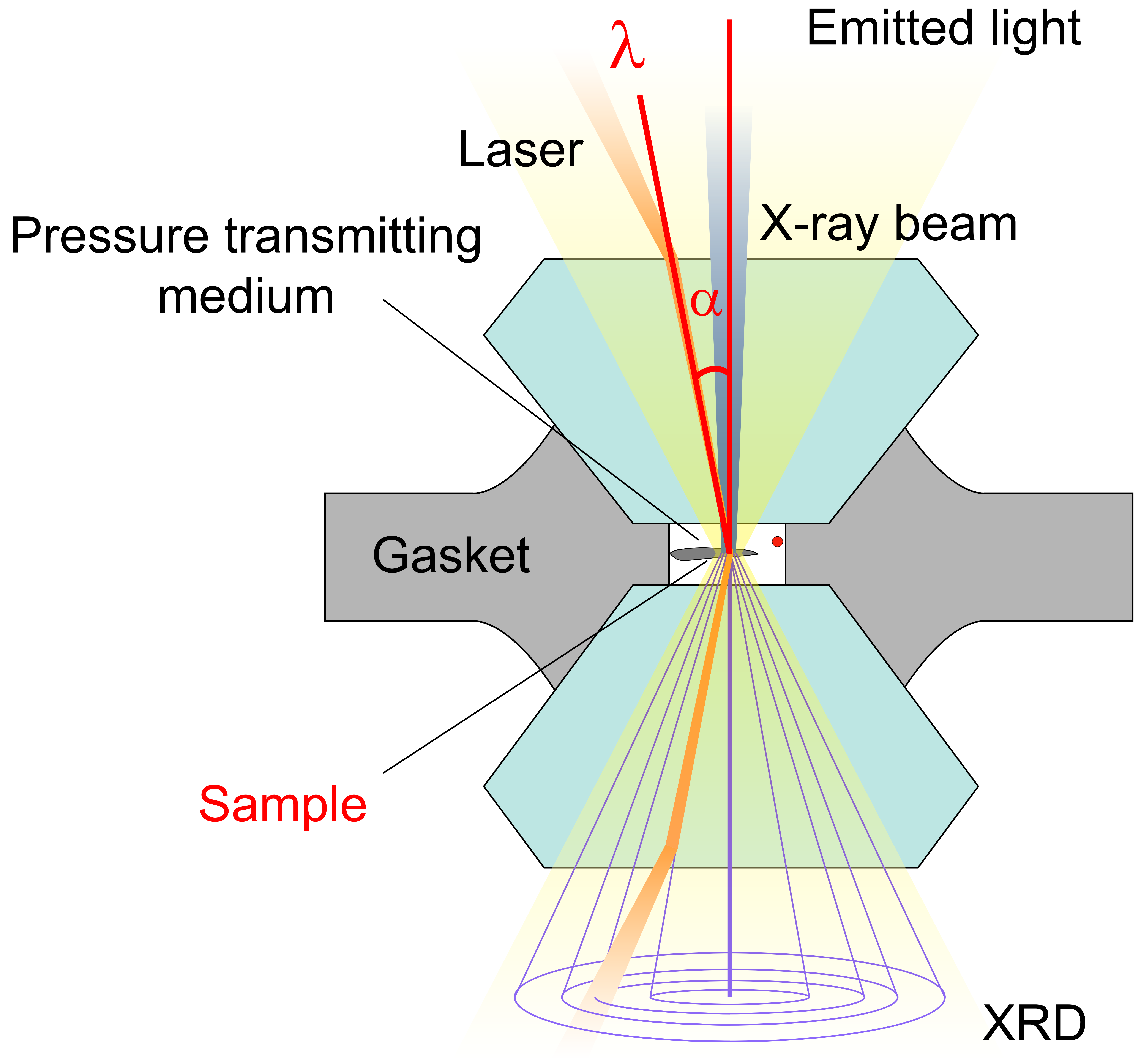
B- Laser heating



Laser beam absorption depends on:

Laser: wavelength

B- Laser heating



Laser beam absorption depends on:

Laser: wavelength, angle of incidence

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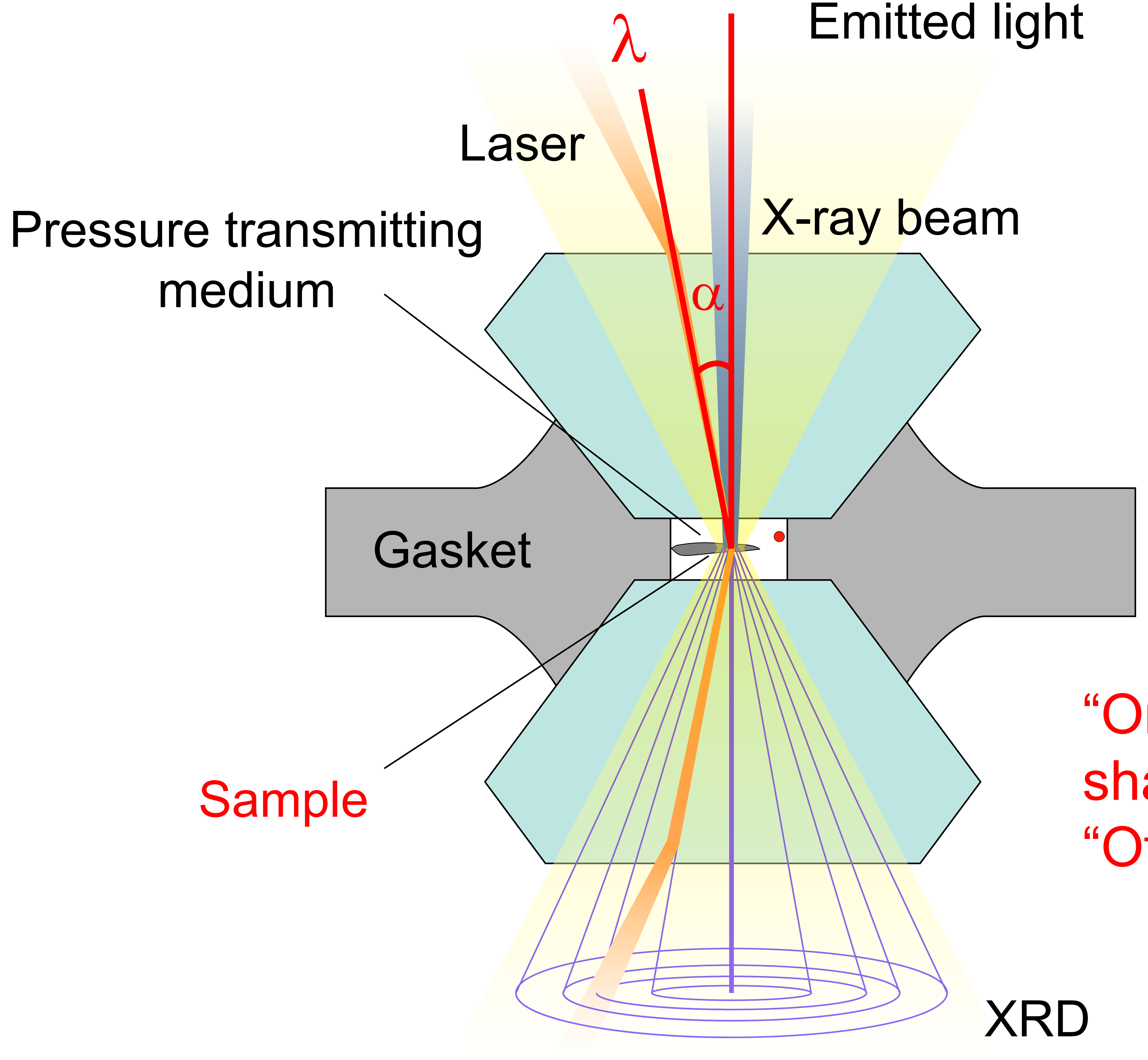
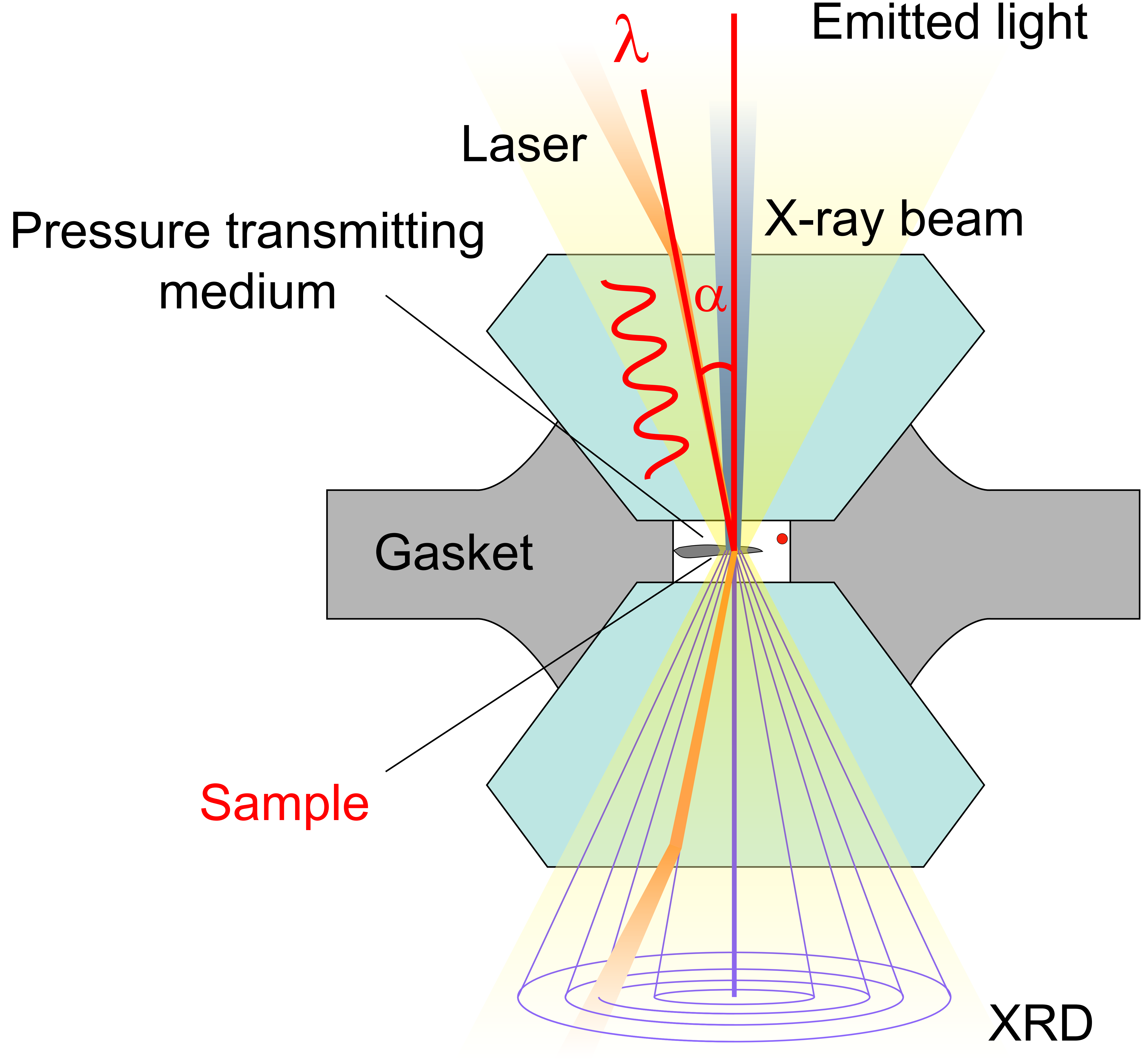


Laser beam absorption depends on:

Laser: wavelength, angle of incidence

"On axis geometry": laser and visualisation share the same optics path, $\alpha = 0$
"Off axis": different paths, $\alpha > 0$

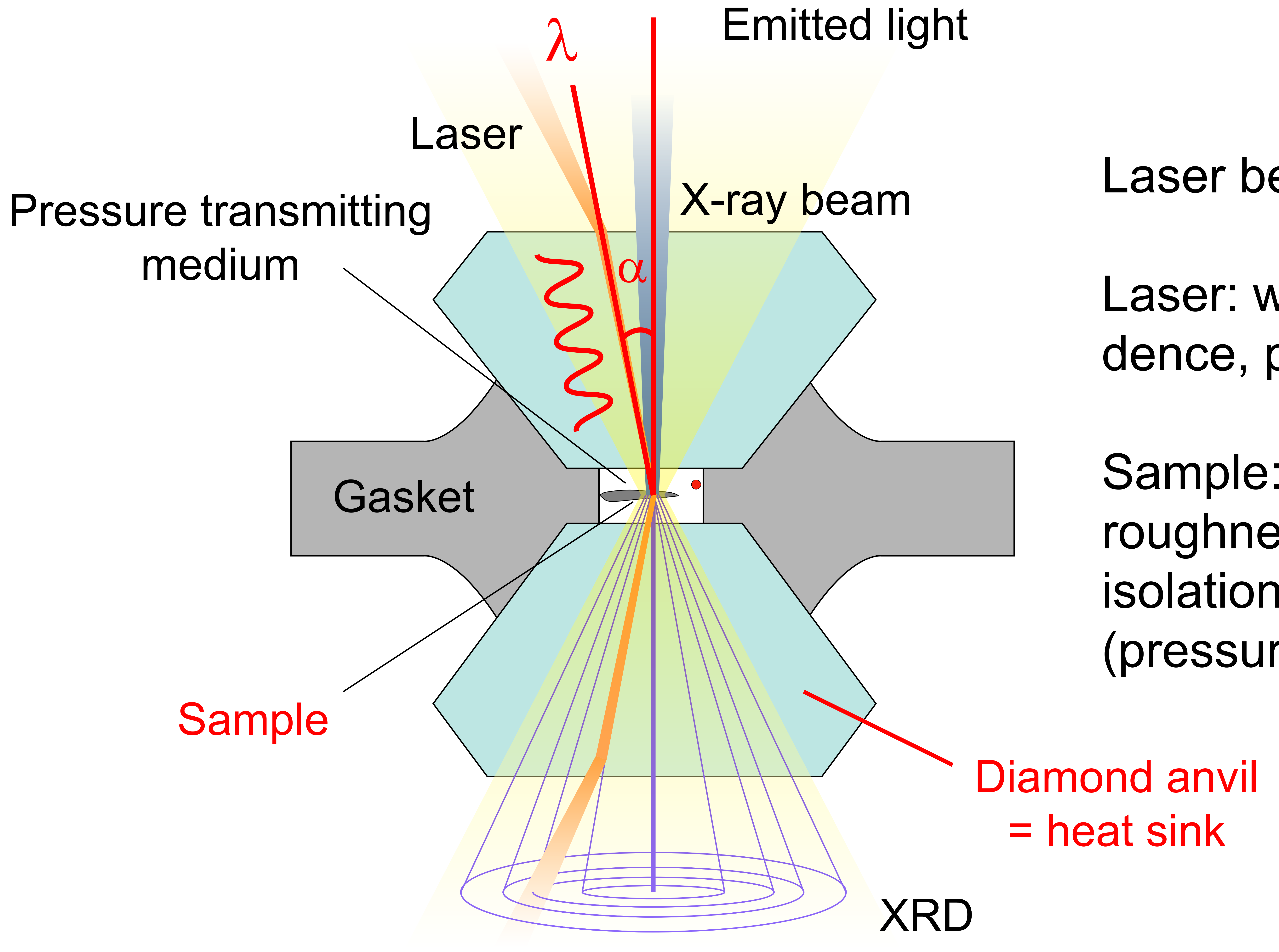
B- Laser heating



Laser beam absorption depends on:

Laser: wavelength, angle of incidence, plane of polarisation

B- Laser heating



Laser beam absorption depends on:

Laser: wavelength, angle of incidence, plane of polarisation

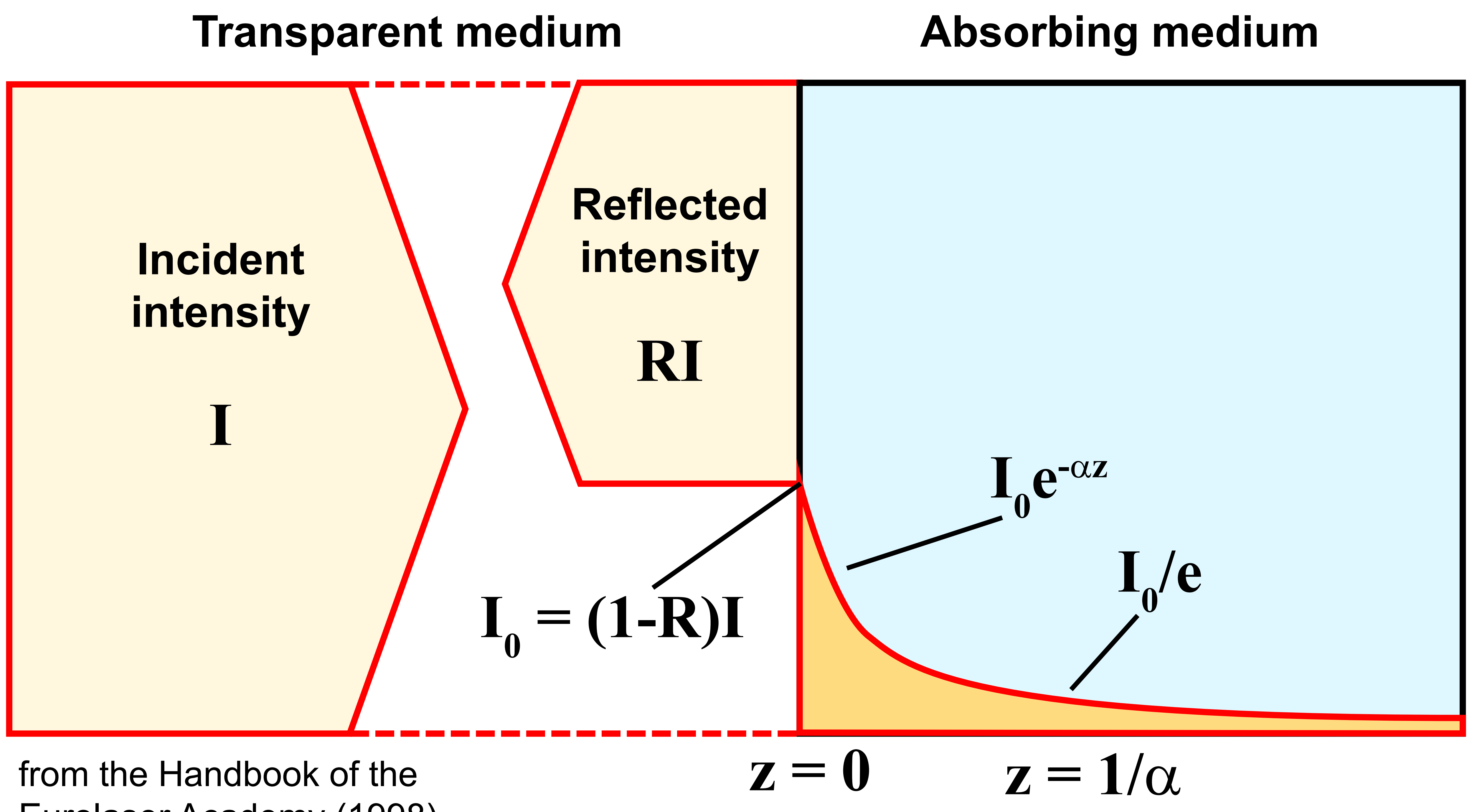
Sample: composition, surface roughness, impurities, temperature, isolation from the diamonds (pressure medium), thickness

B- Laser heating

Laser beam absorption inside the sample follows the Beer-Lambert law:

$$I(z) = I_0 e^{-\alpha z}$$

The intensity of the light decays exponentially with depth at a rate determined by the material's absorption coefficient α.



from the Handbook of the Eurolaser Academy (1998)

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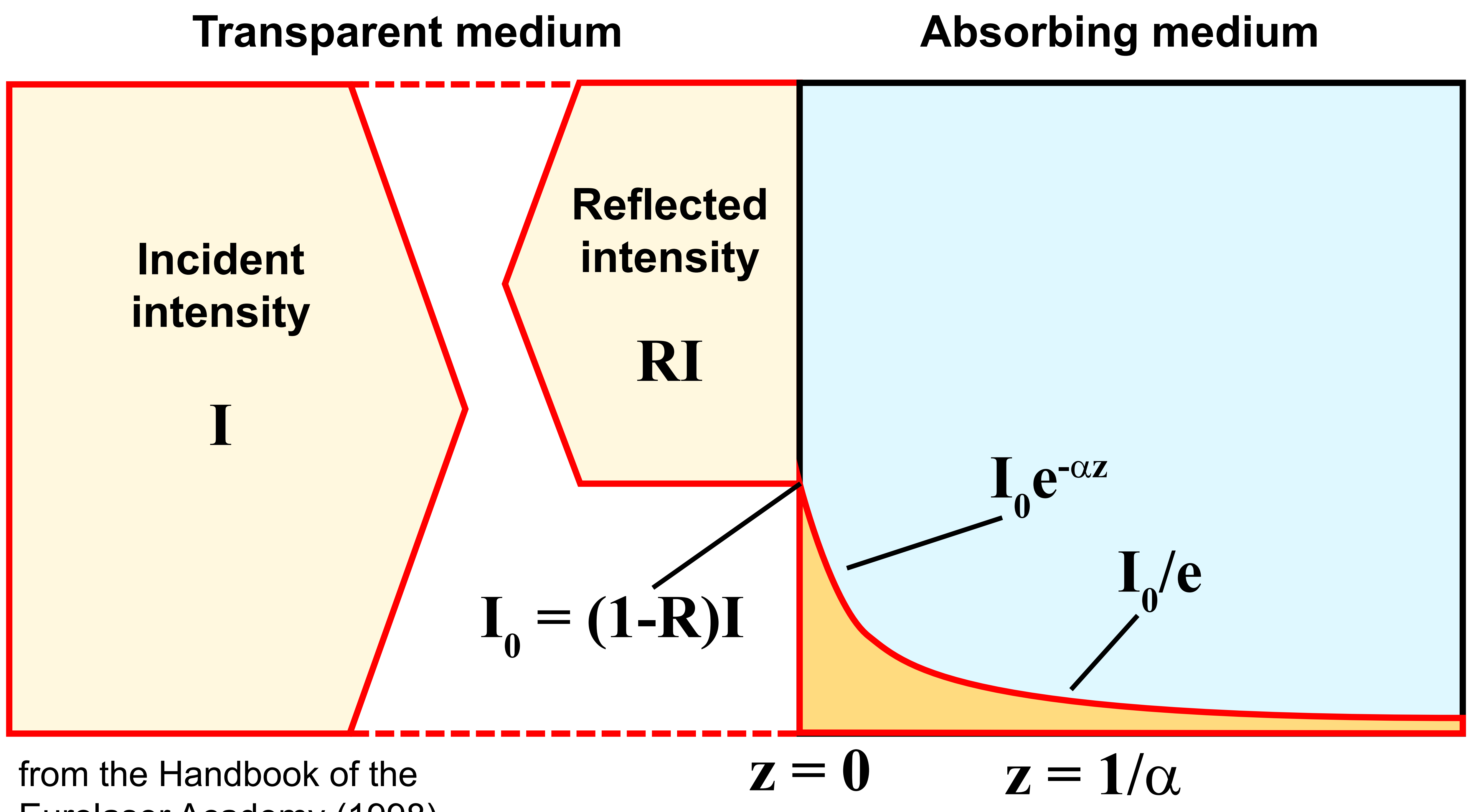

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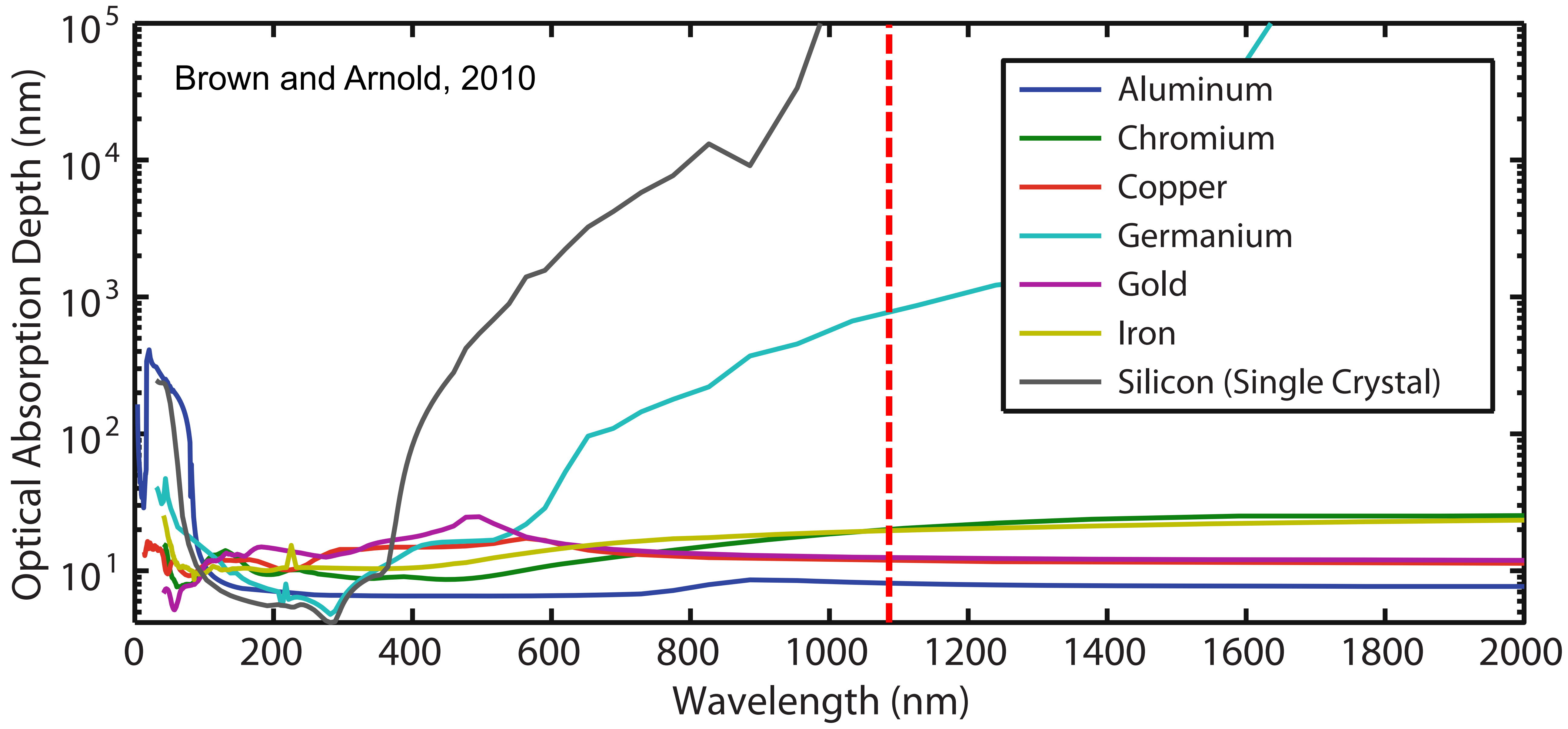


Optical absorption depths for several materials over a range of wavelengths

The optical penetration or absorption depth is defined as $z = 1/\alpha$

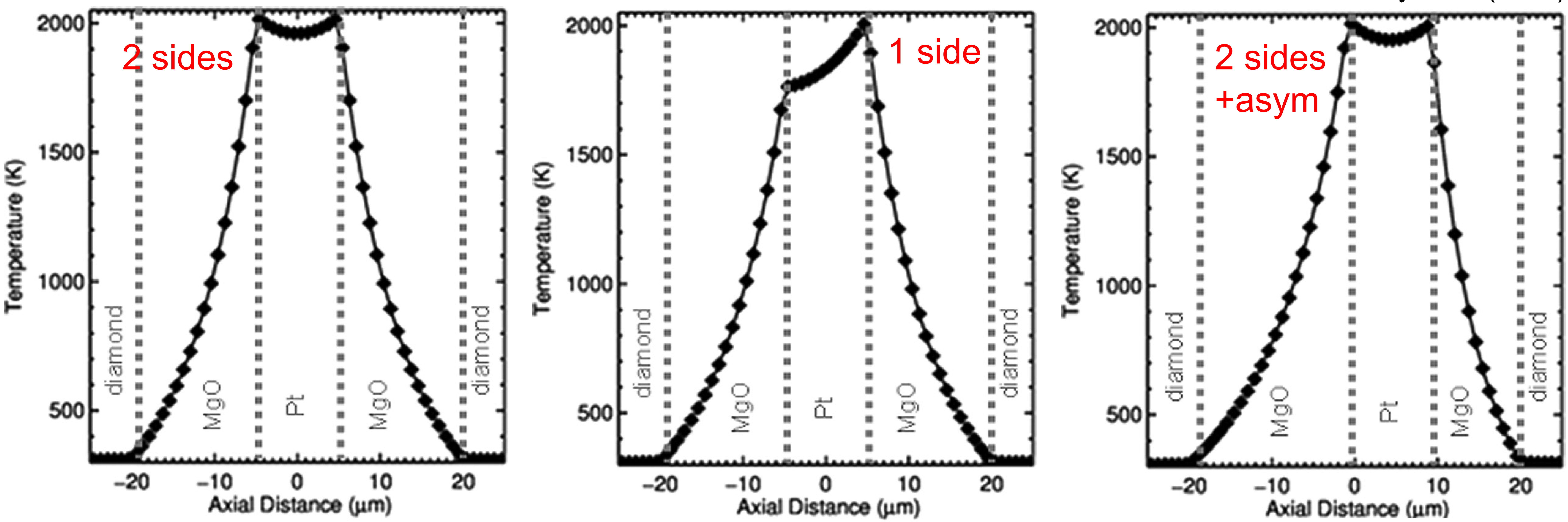
That means that for metals most of the energy of the incoming beam is absorbed in the first 20 nm @1064 nm

B- Laser heating

But the typical thickness of a sample is in the 3-10 µm range. What about the axial gradient, that will affect X-ray diffraction/absorption measurements?

The LH-DAC temperature distributions are dominated by conductive heat transfer in the sample, the pressure medium, the diamonds and the gasket.

Rainey et al. (2013)



Dynamic 3D models show that the axial gradient is actually low, as long as laser absorption by the sample is strong enough, sample thickness low enough and insulation from the diamonds and gasket good enough.

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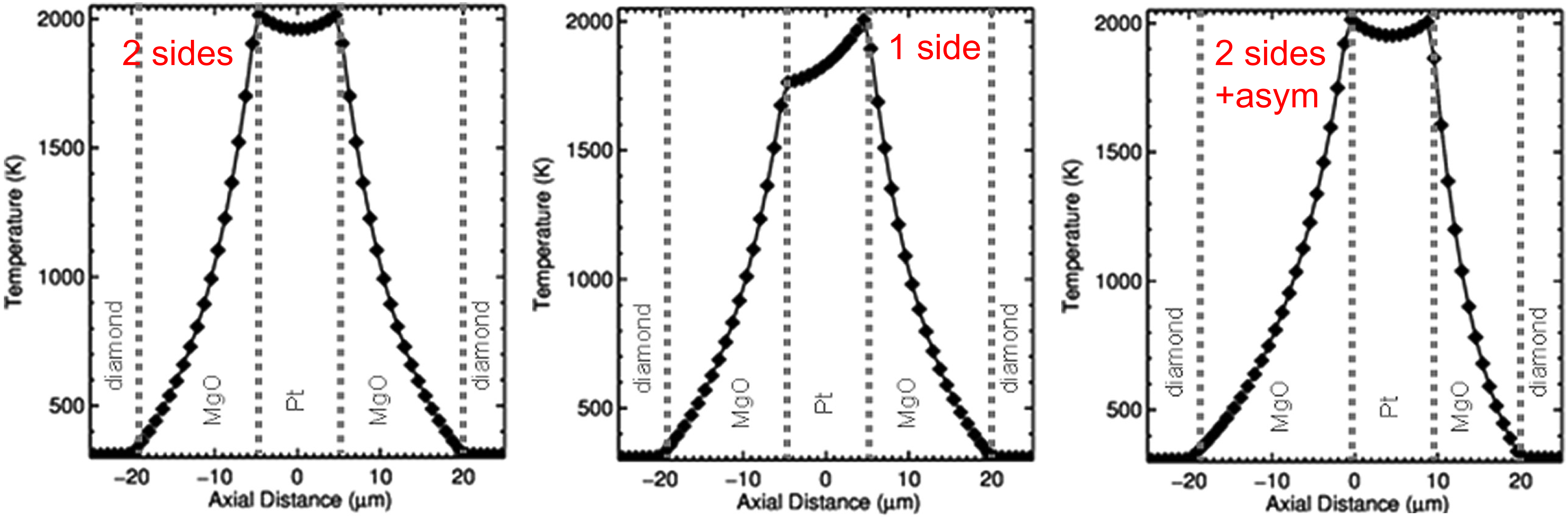


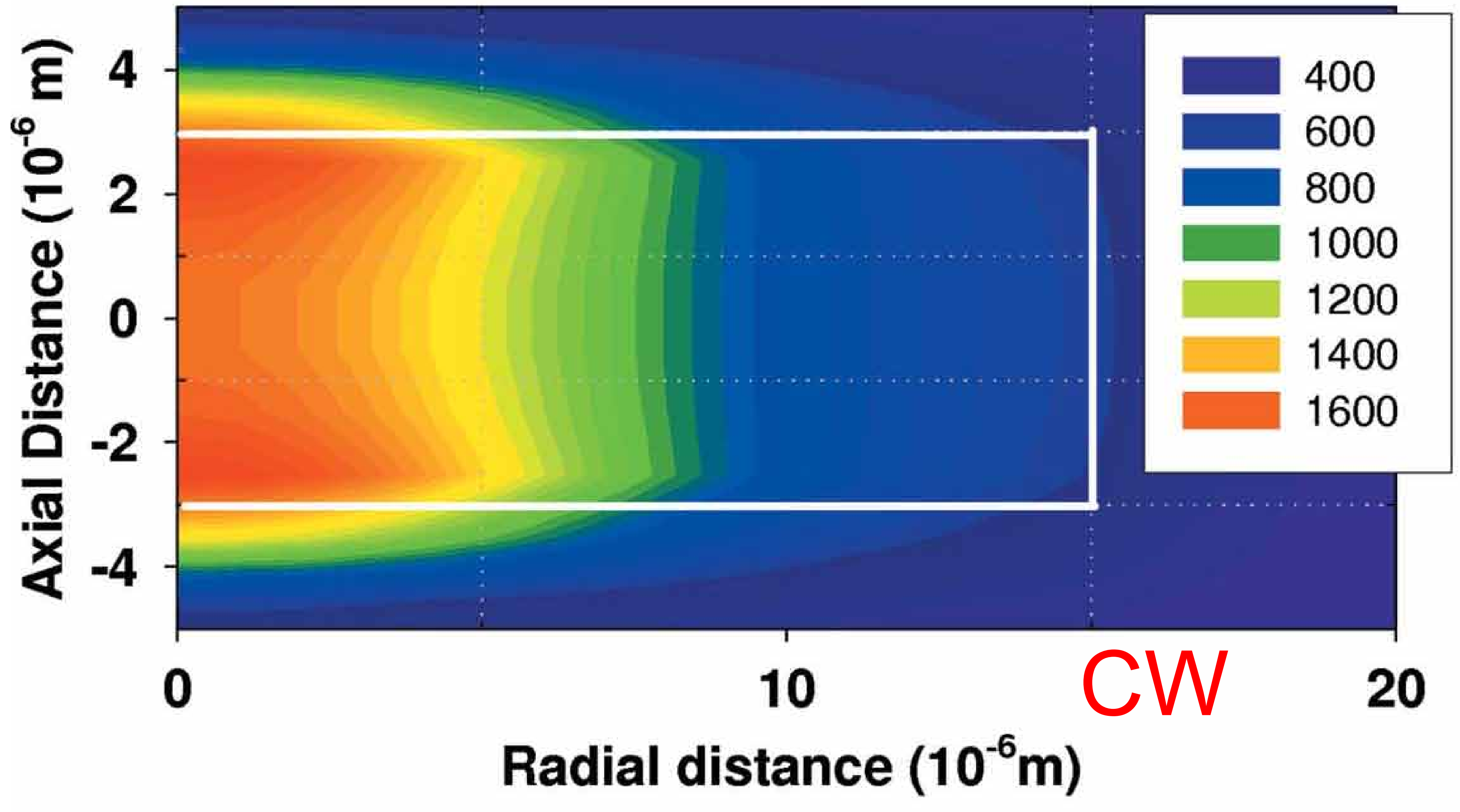
+ thermal equilibrium is achieved very fast (a few µs at most)

B- Laser heating

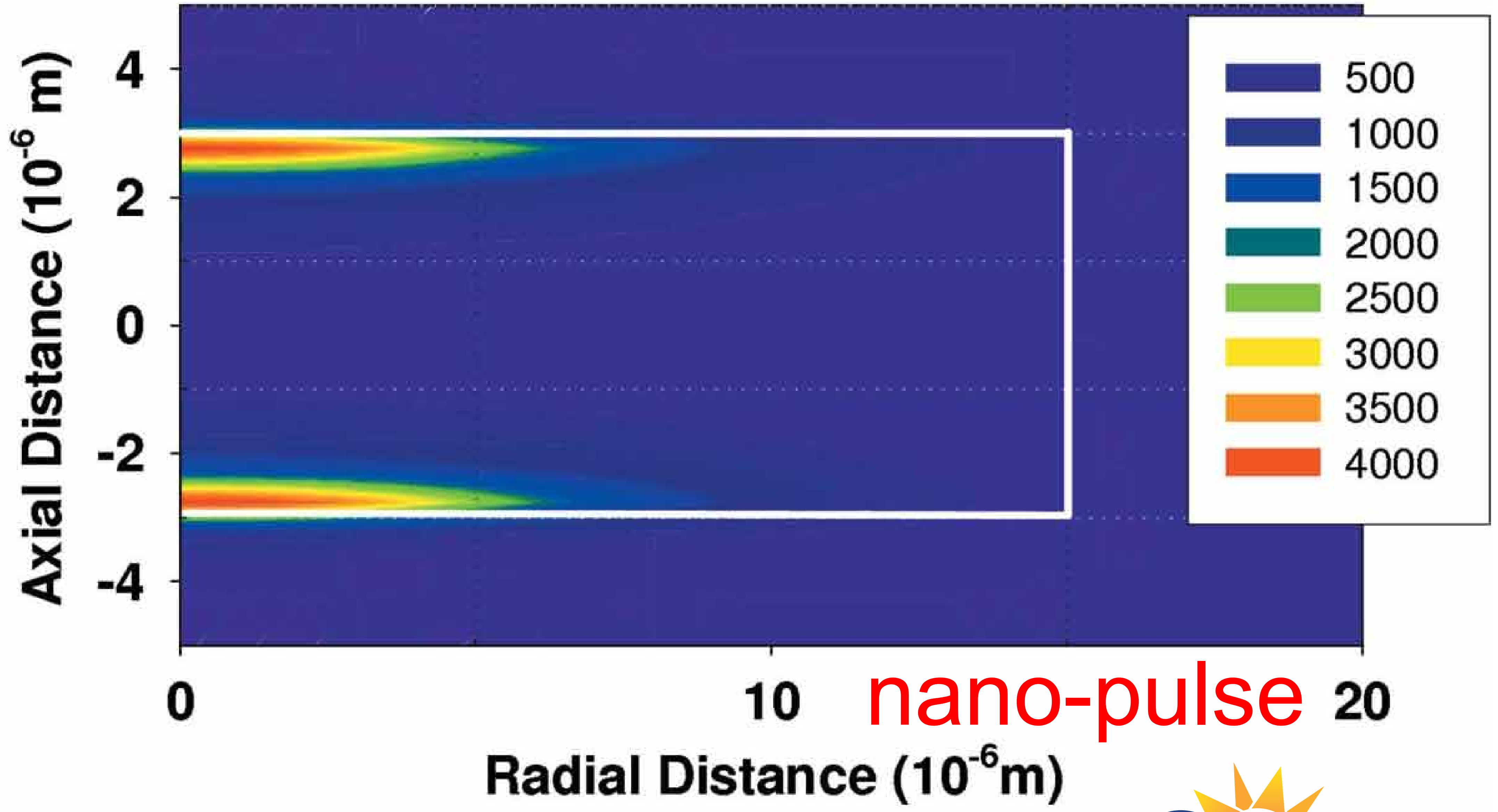
Heating during short period of times can be desirable to reduce chemical reaction, migration of liquid, etc...

If steady state and low axial T gradient is required, pulse durations in the range of a few µs are needed. At the nano scale the amount of material that is being heated is < 1 µm

In conjunction with continuous (CW) laser heating, nano-pulse laser heating is very useful to measure the sample thermal conductivity (e.g. Konopkova et al. 2016)



(a)



(b)

Goncharov et al. (2009)

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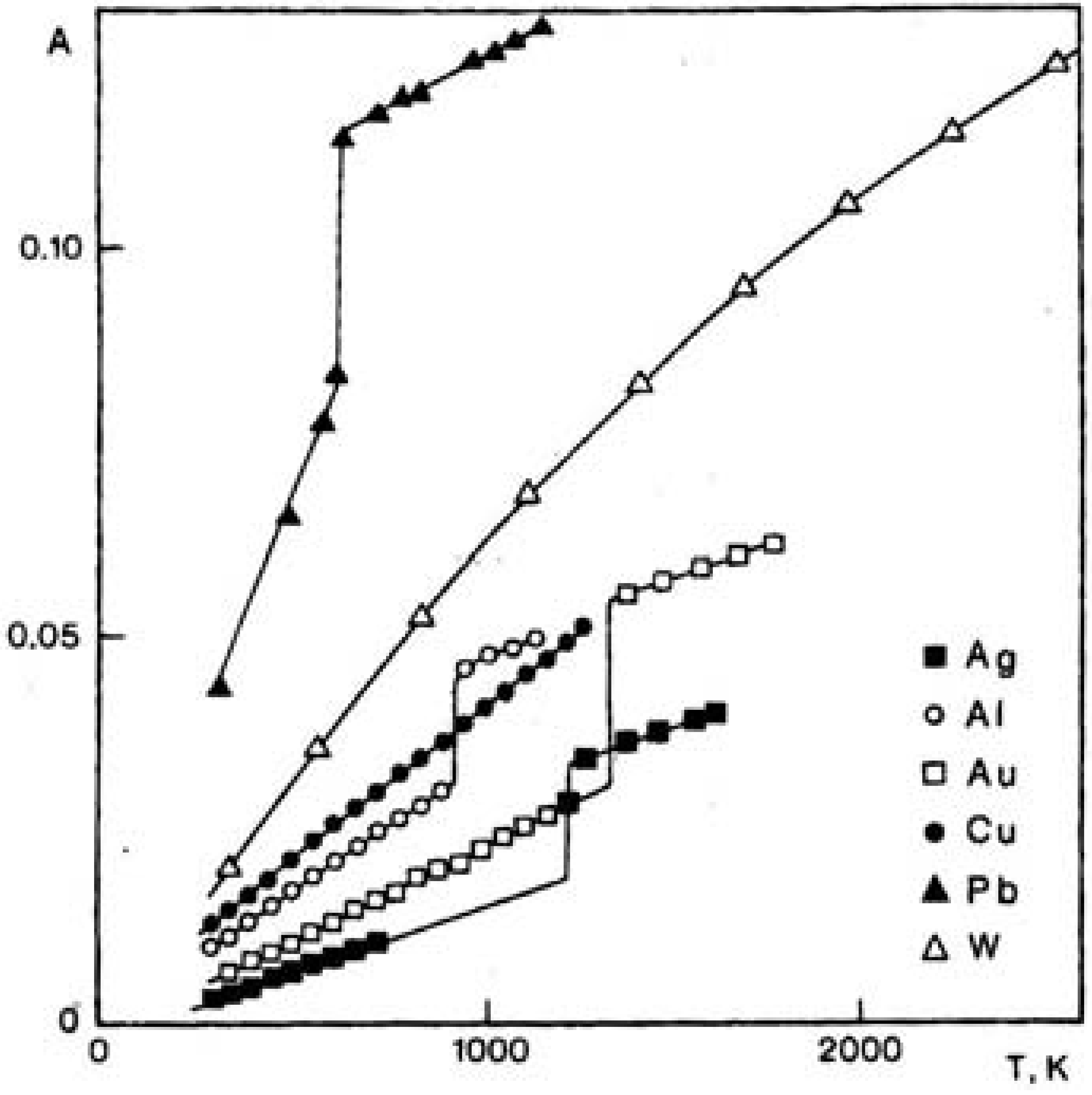
B- Laser heating



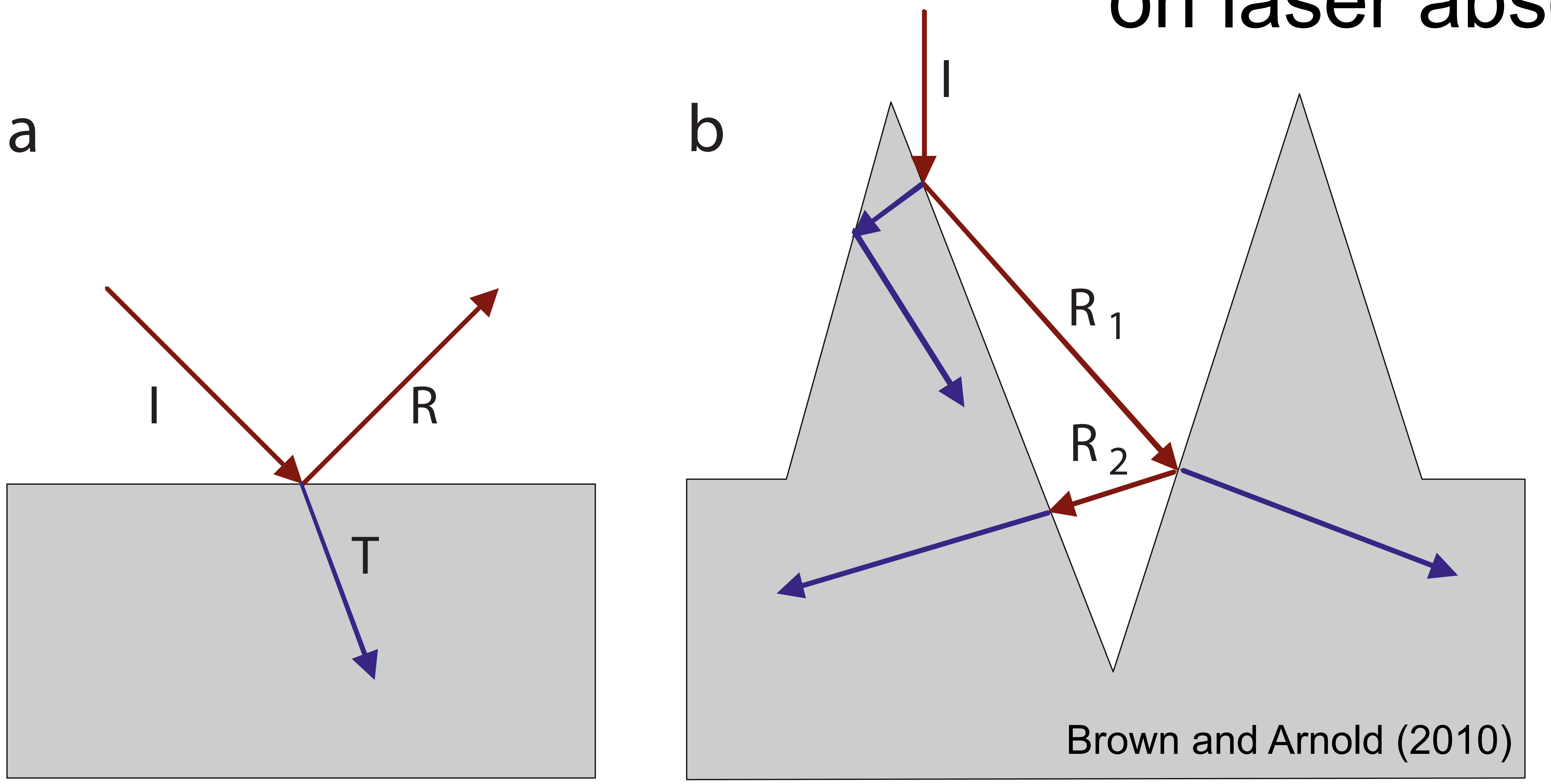
The absorption coefficient depends on T (polynomial law) and a strong jump can be observed during melting

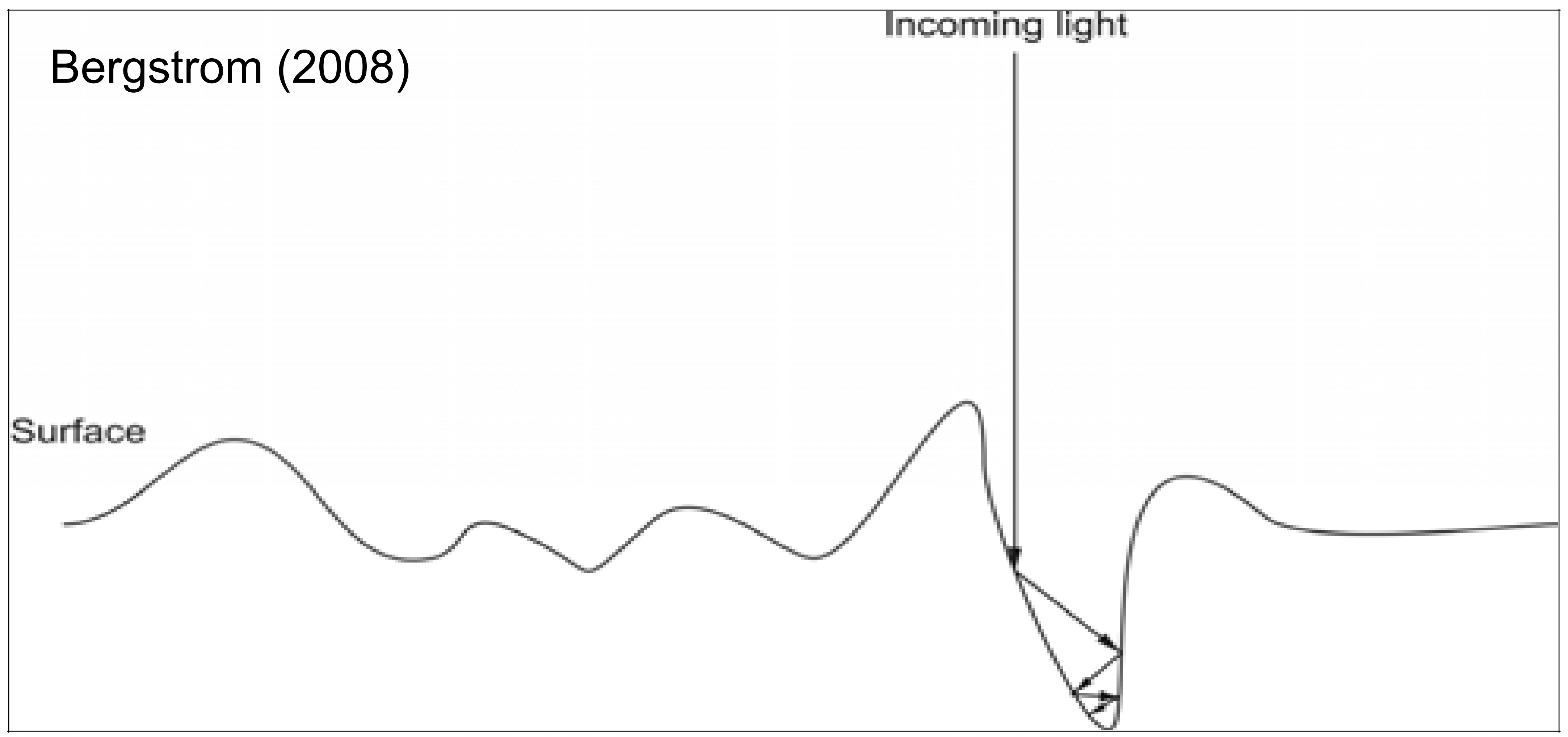
Here are represented models of the evolution of the absorption coefficients for different metals @10.6 µm and ambient P (from Prokhorov et al., 1990)

This can be the source of heating instabilities, especially during melting

B- Laser heating

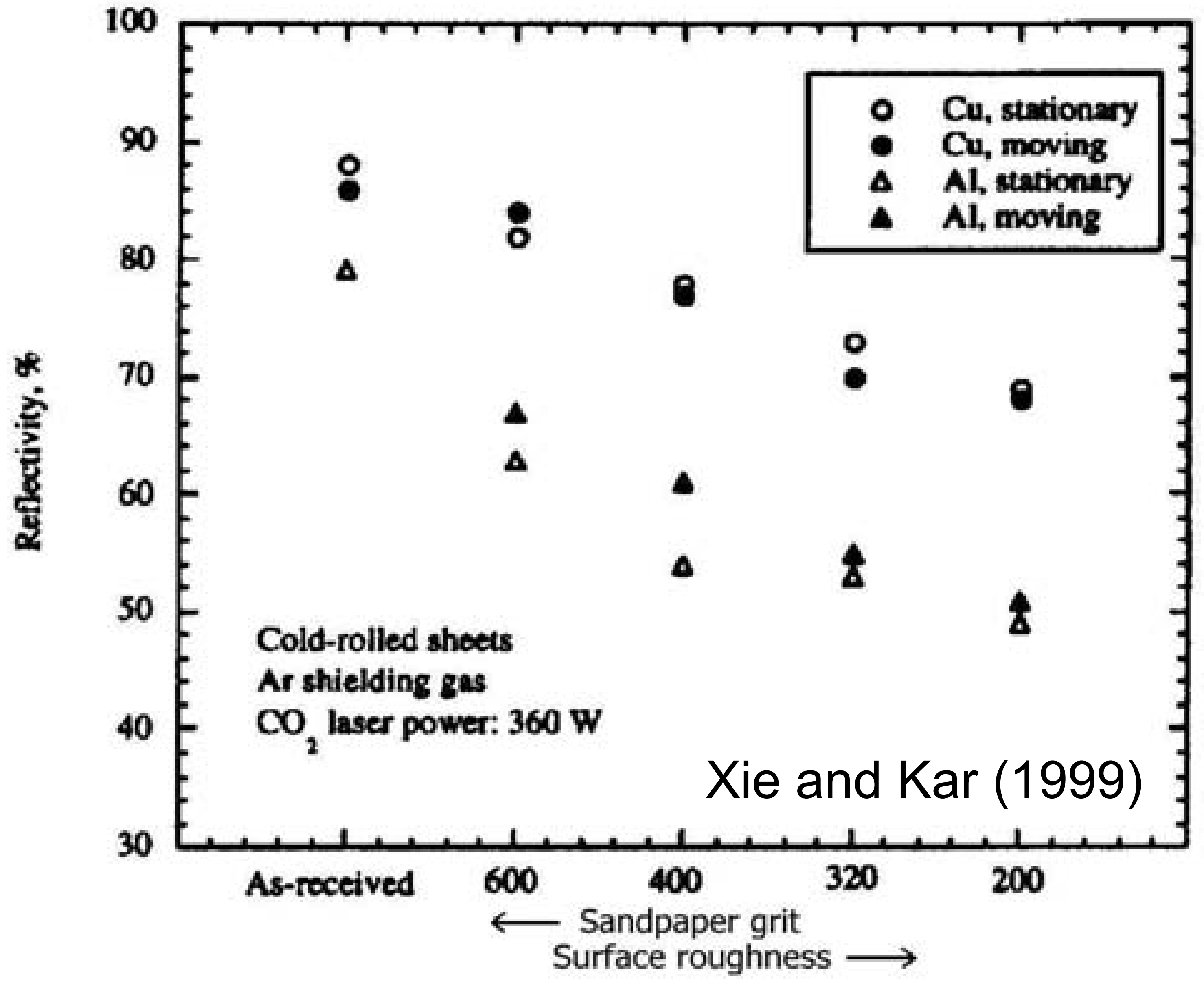
The sample surface roughness has also a strong effect on laser absorption





(a) Light specularly reflecting from a flat surface (b) Multiple reflections from protruding structures enhance coupling into the material

Feature size	Influence on reflectivity
$\gg \lambda$	Light trapping due to multiple reflections enhances coupling into the material. Light refracted at oblique angles increases the effective optical path length
$\approx \lambda$	Small features can successively scatter light, increasing the effective optical path length and enhancing absorption
$\ll \lambda$	Subwavelength structures (SWS) can reduce reflections through the moth-eye effect



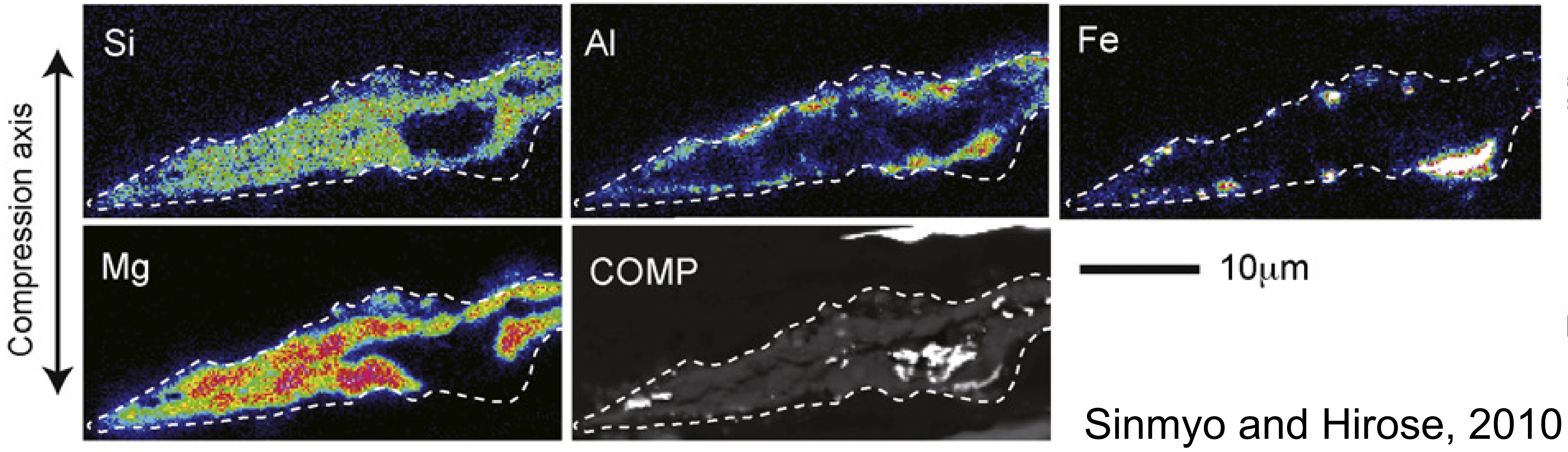
B- Laser heating

In practice the heating heterogeneities that can be observed are due to a combination of subtle variations of all these parameters: surface quality, sample thickness, pressure medium thickness, melting, etc...

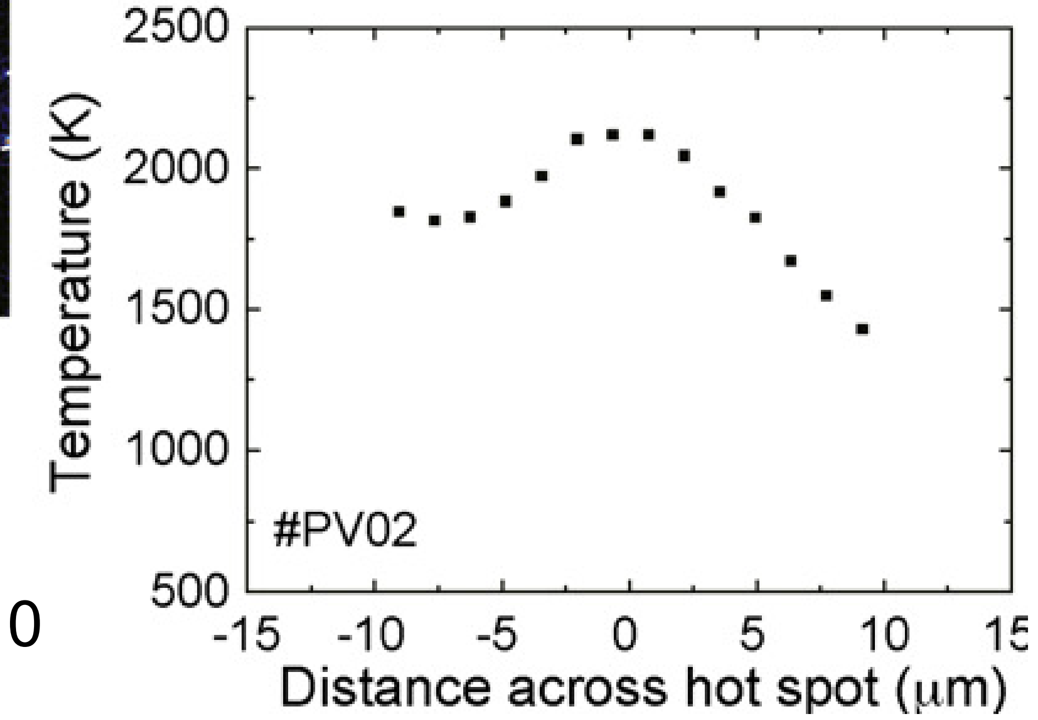
B- Laser heating

Soret effect (also called thermal diffusion) is chemical diffusion driven by a temperature gradient

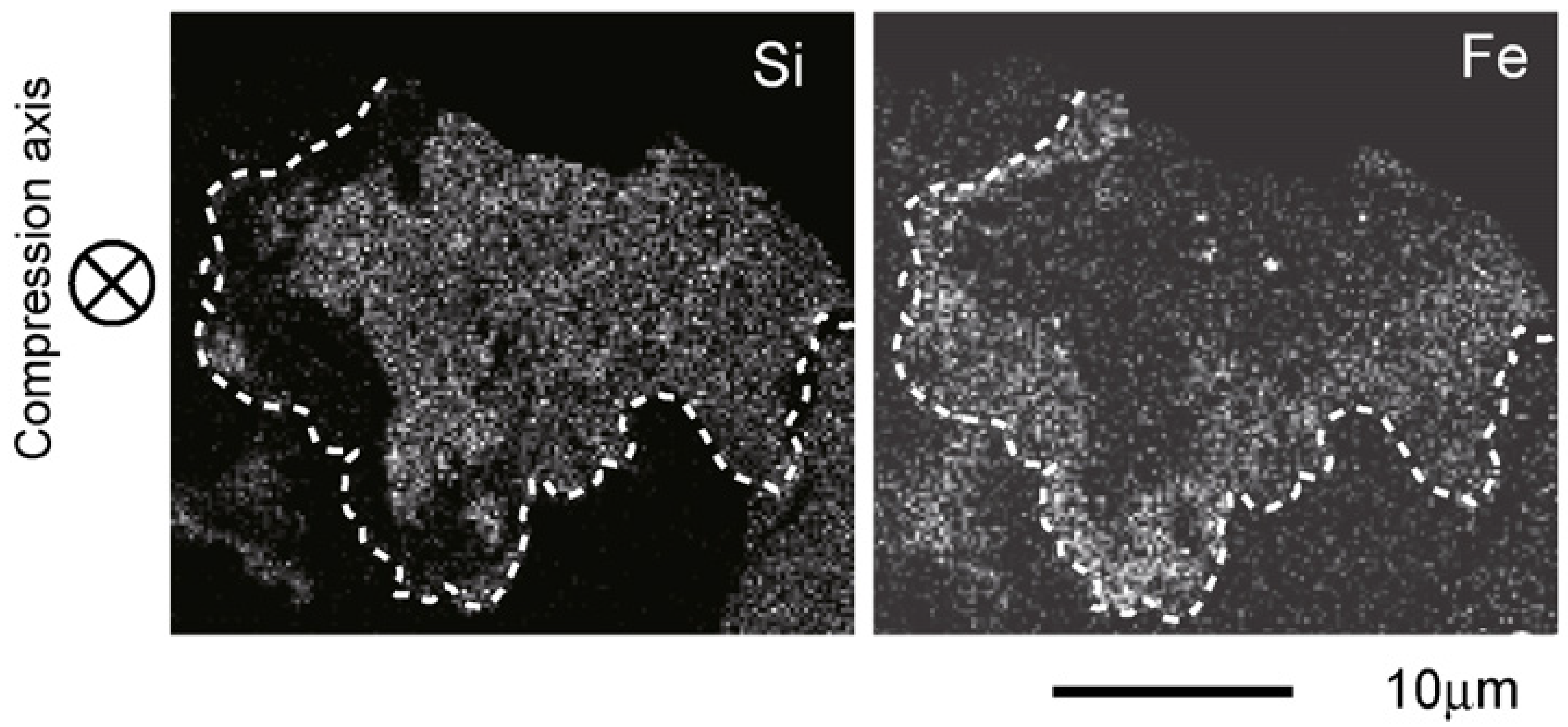
$(Mg,Fe)(Al,Si)O_3$ perovskite, 1900 K, 20 min

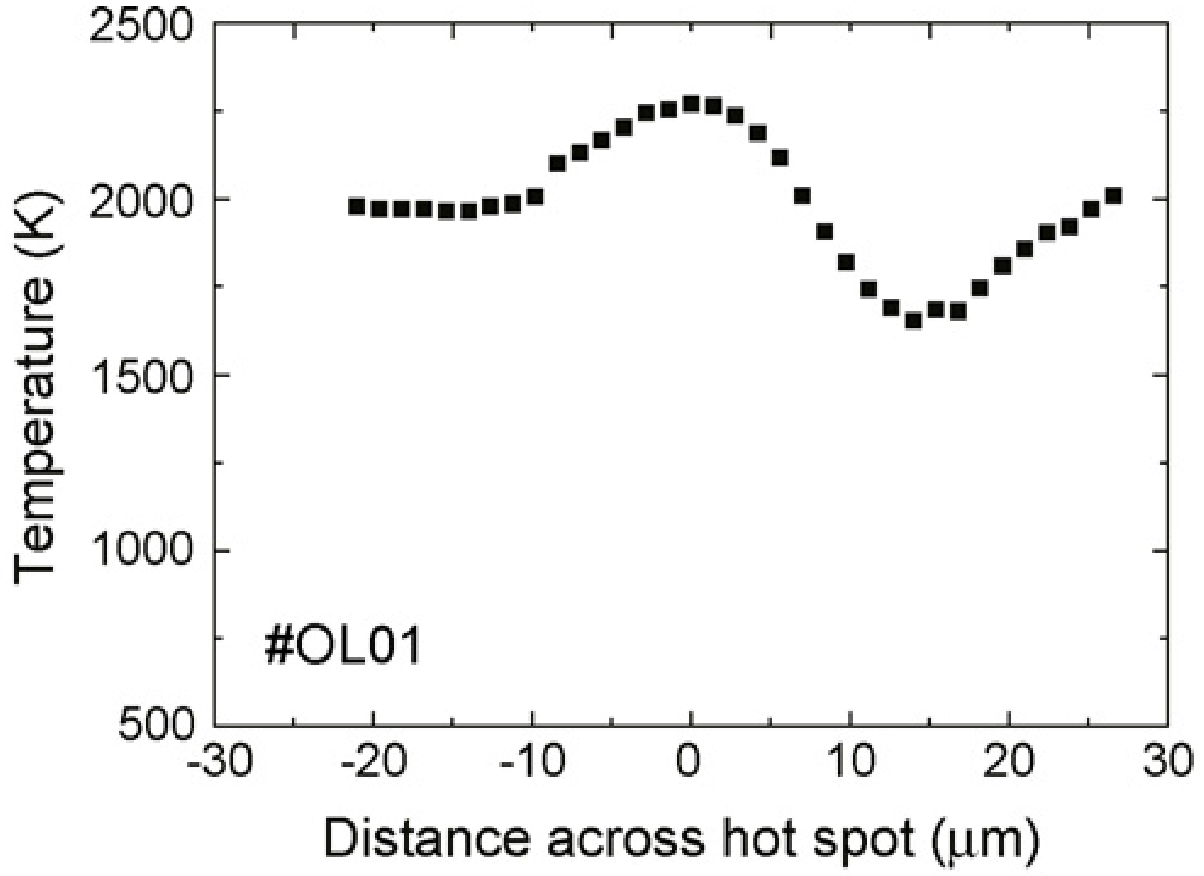


Sinmyo and Hirose, 2010

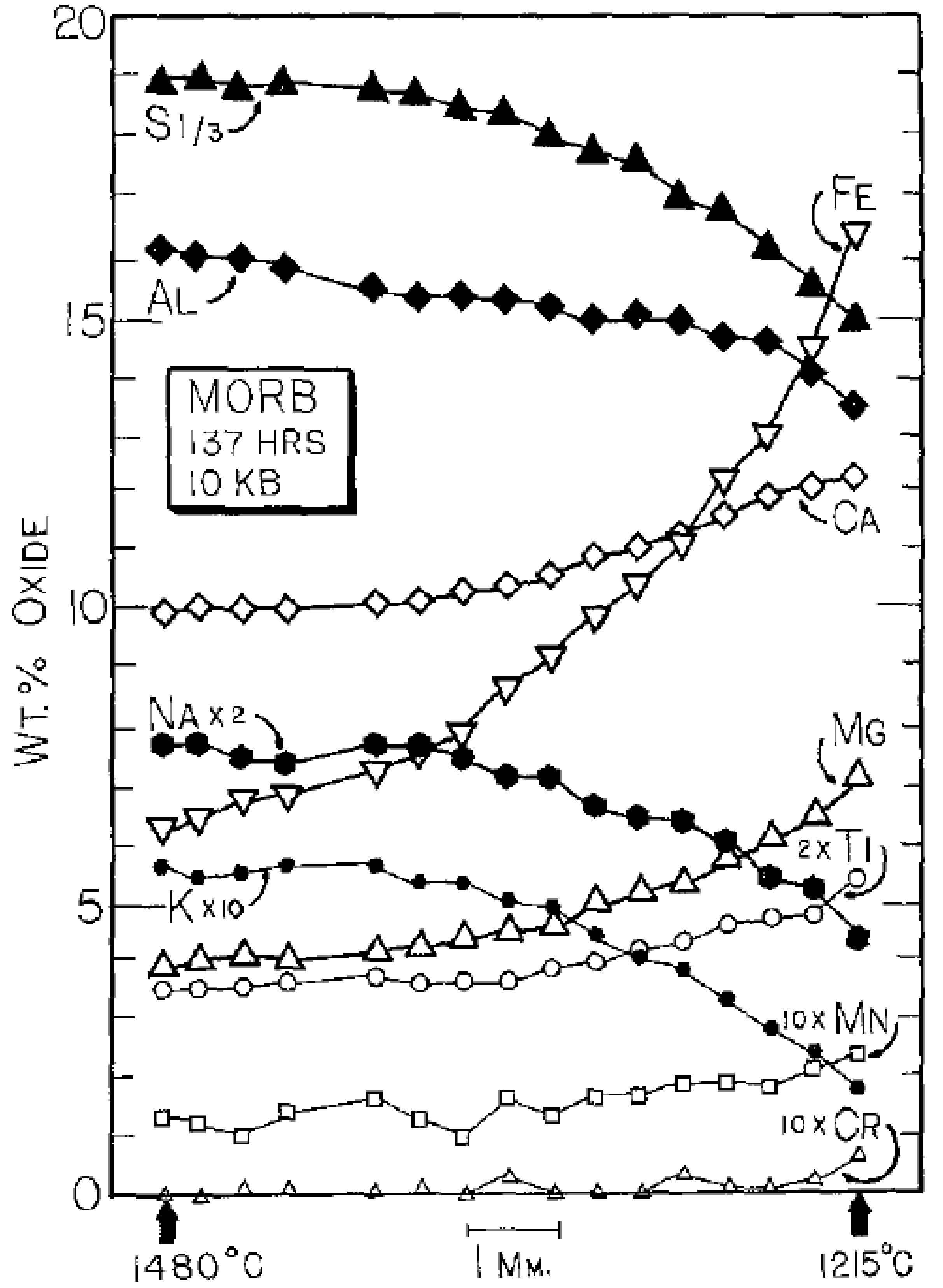


$(Mg,Fe)_2SiO_4$ composition, 2200K, 72min





Walker and DeLong, 1982



Migration of elements along the T gradient: from hot to cold regions, ex : Fe / or from cold to hot regions, ex: Si. Often very difficult to predict, making multi-phases/components samples studies very challenging + source of further laser heating unstabilities

Another good reason to minimize T gradients, regardless of X-ray beam size!

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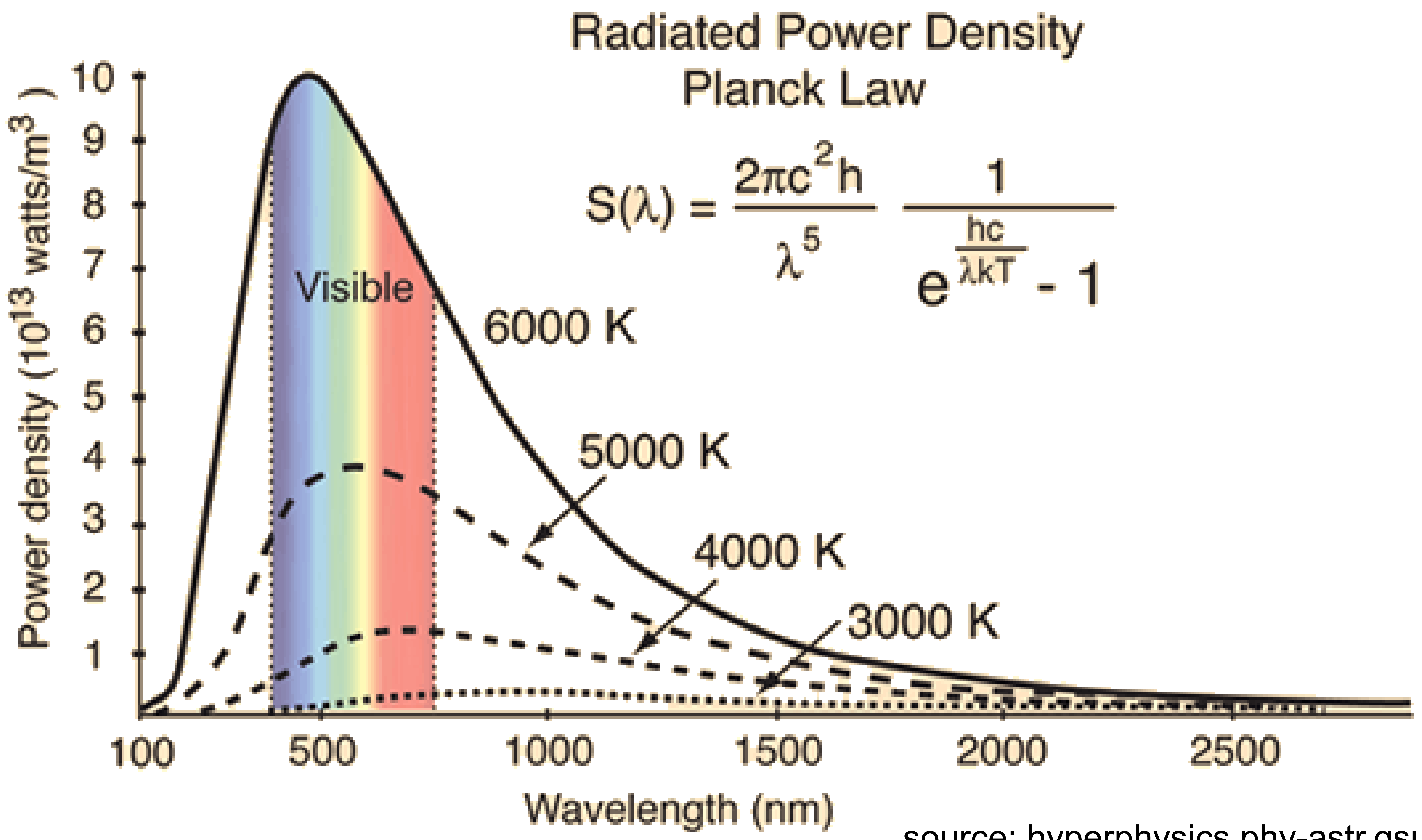
C- T measurement

Temperature is measured via spectroradiometry, using the “gray body” approximation

Black body: body in thermodynamic equilibrium with its environment, emitting light that only depends on its T

Spectral radiance is given by the Planck law:

$$I(\lambda) = \frac{2\pi hc^2}{\lambda^5} \frac{1}{e^{hc/\lambda kT} - 1}$$



source: hyperphysics.phy-astr.gsu.edu

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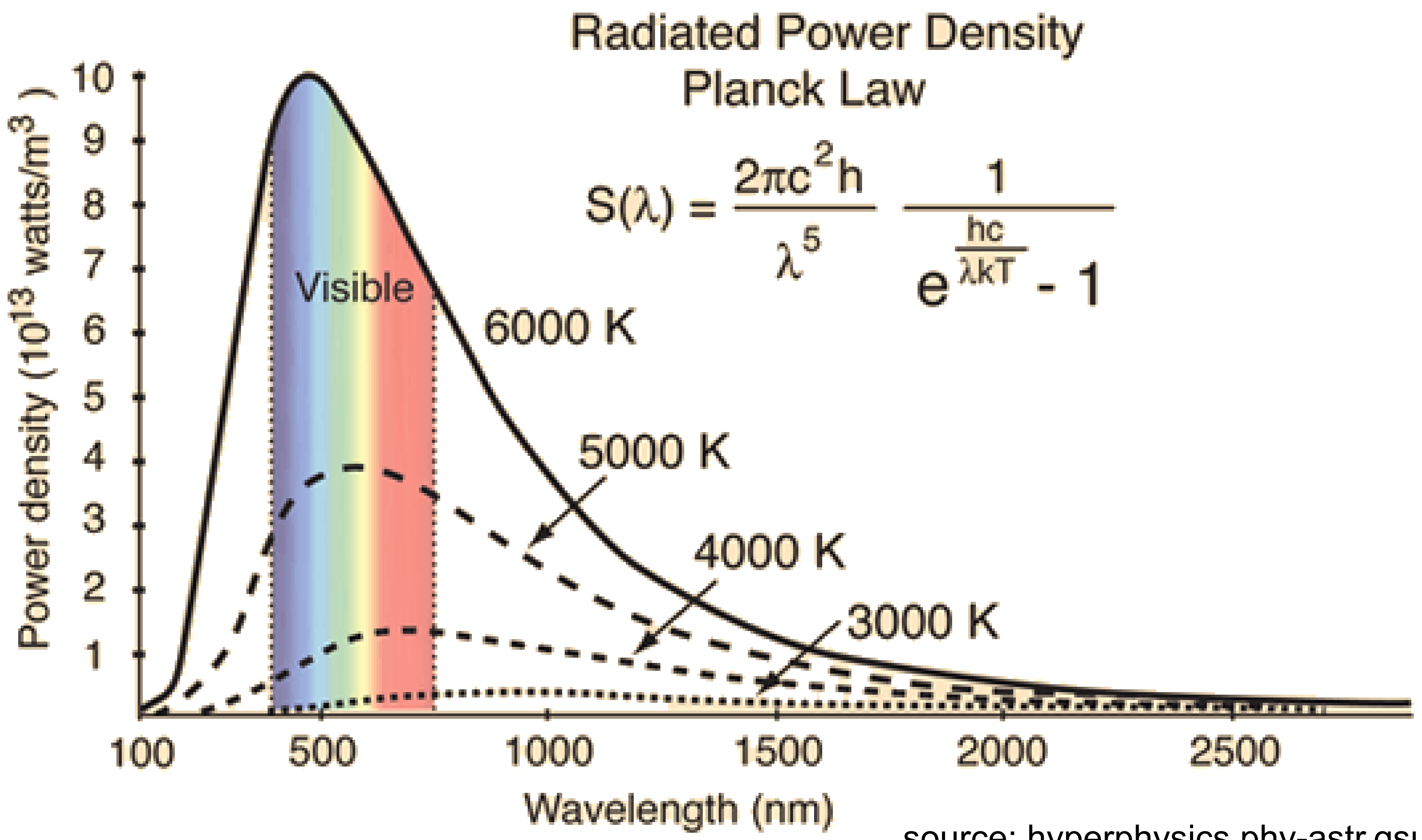
Black body: body in thermodynamic equilibrium with its environment, emitting light that only depends on its T

Spectral radiance is given by the Planck law:

$$I(\lambda) = \varepsilon \frac{2\pi hc^2}{\lambda^5} \frac{1}{e^{hc/\lambda kT} - 1}$$

emissivity, $0 < \varepsilon < 1$

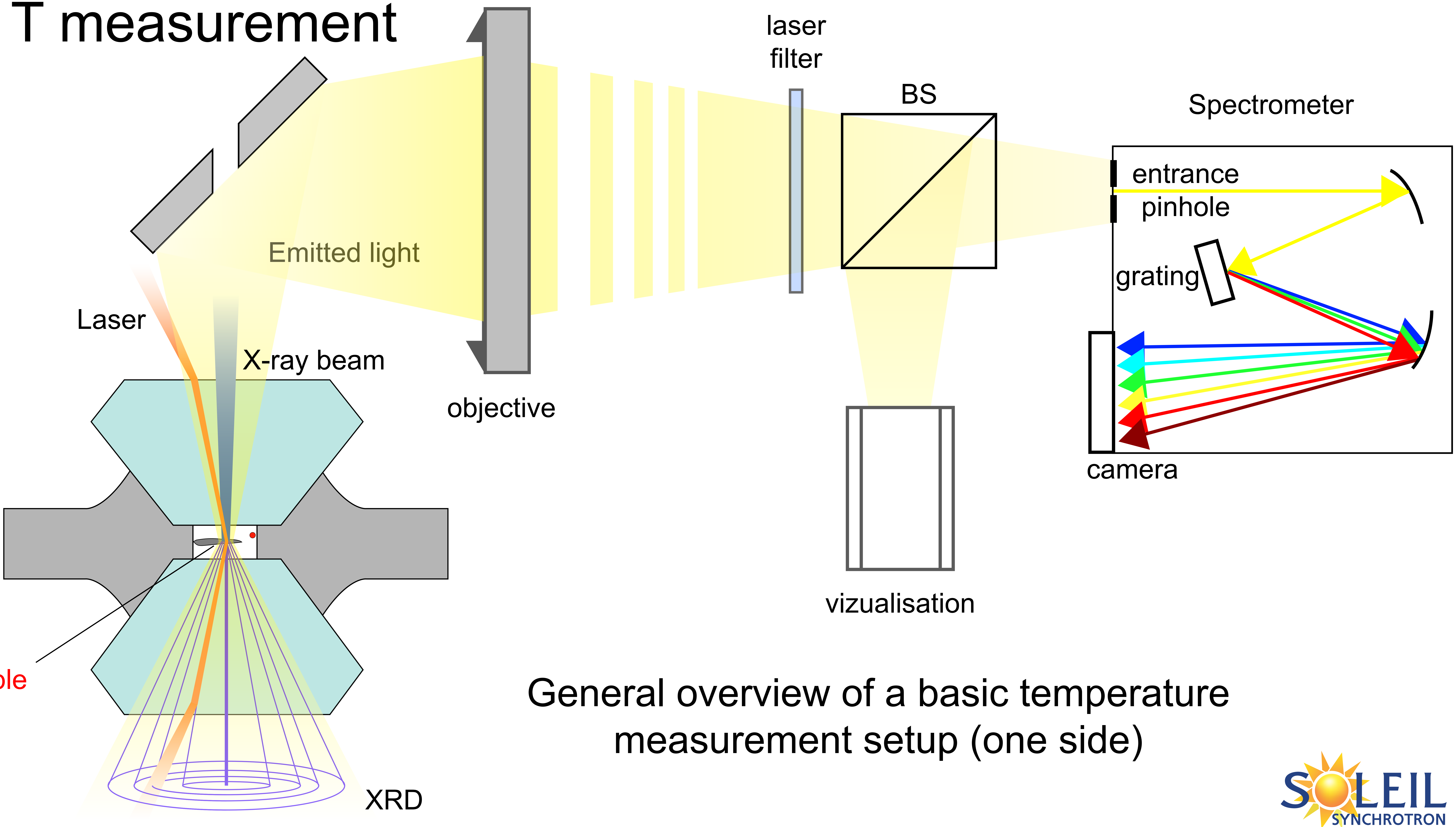


source: hyperphysics.phy-astr.gsu.edu

In practice materials are not ideal black bodies: introduction of emissivity

Emissivity is considered constant (gray body approximation). The thermal emission spectrum can thus be fitted with only 2 parameters : T and ε

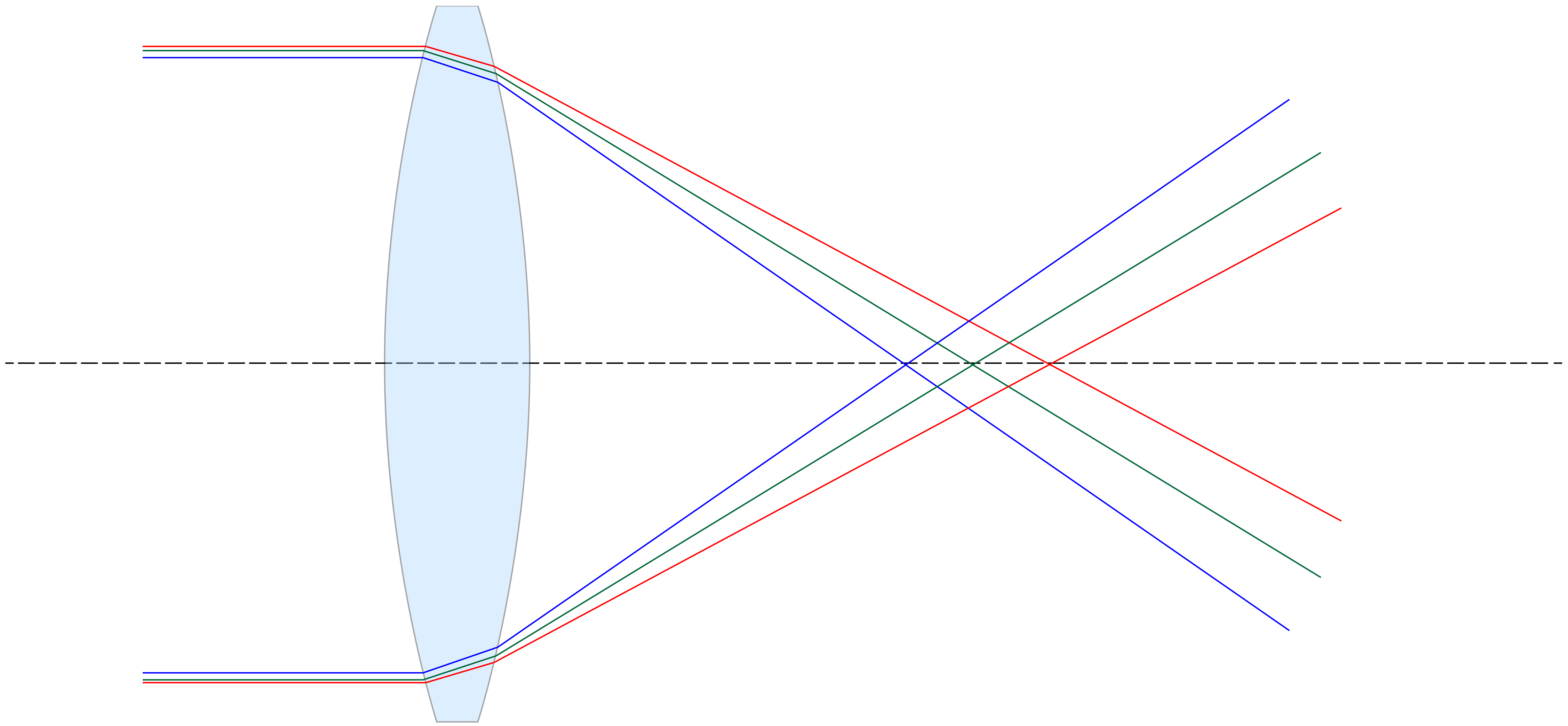
C- T measurement

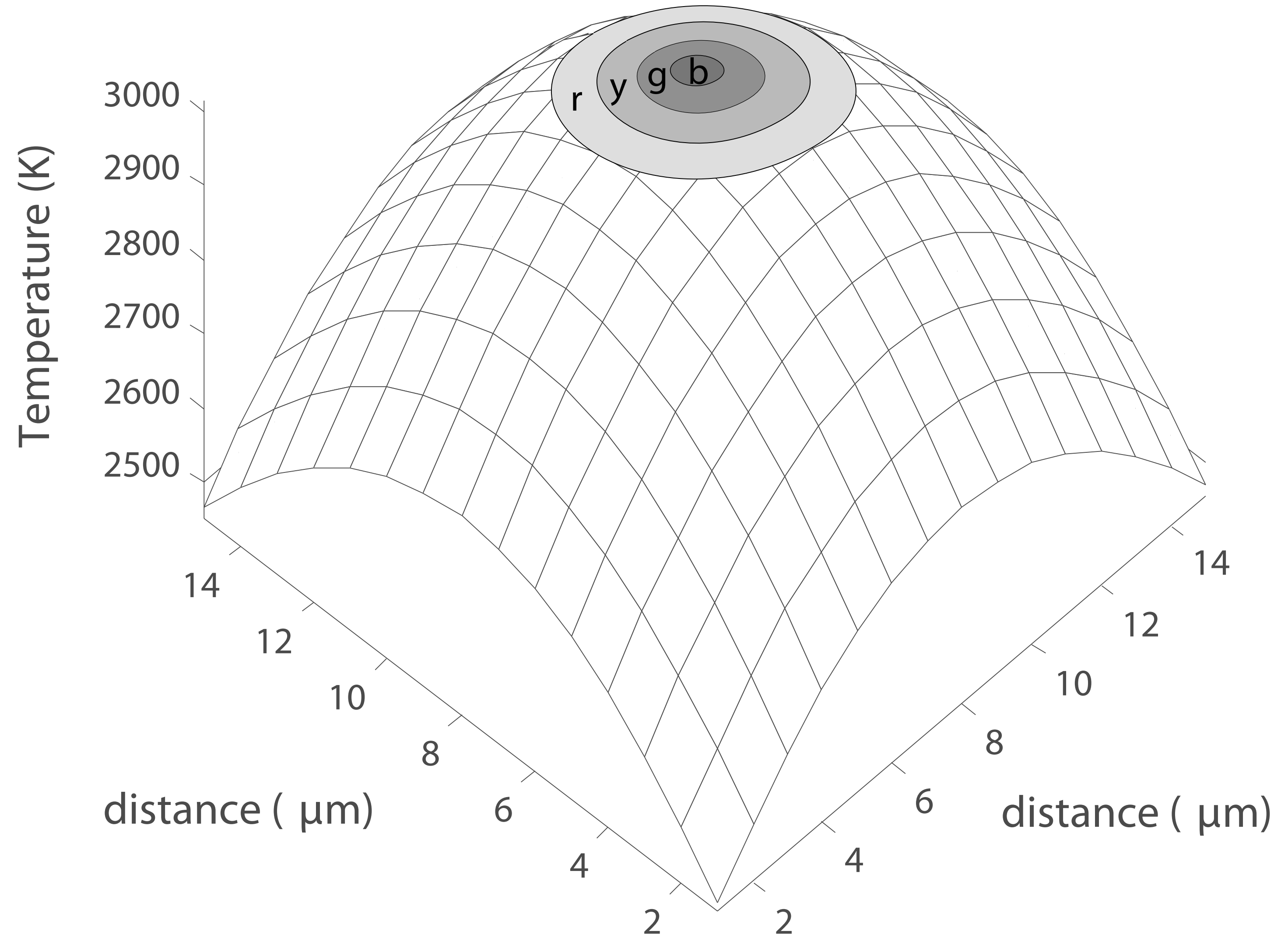


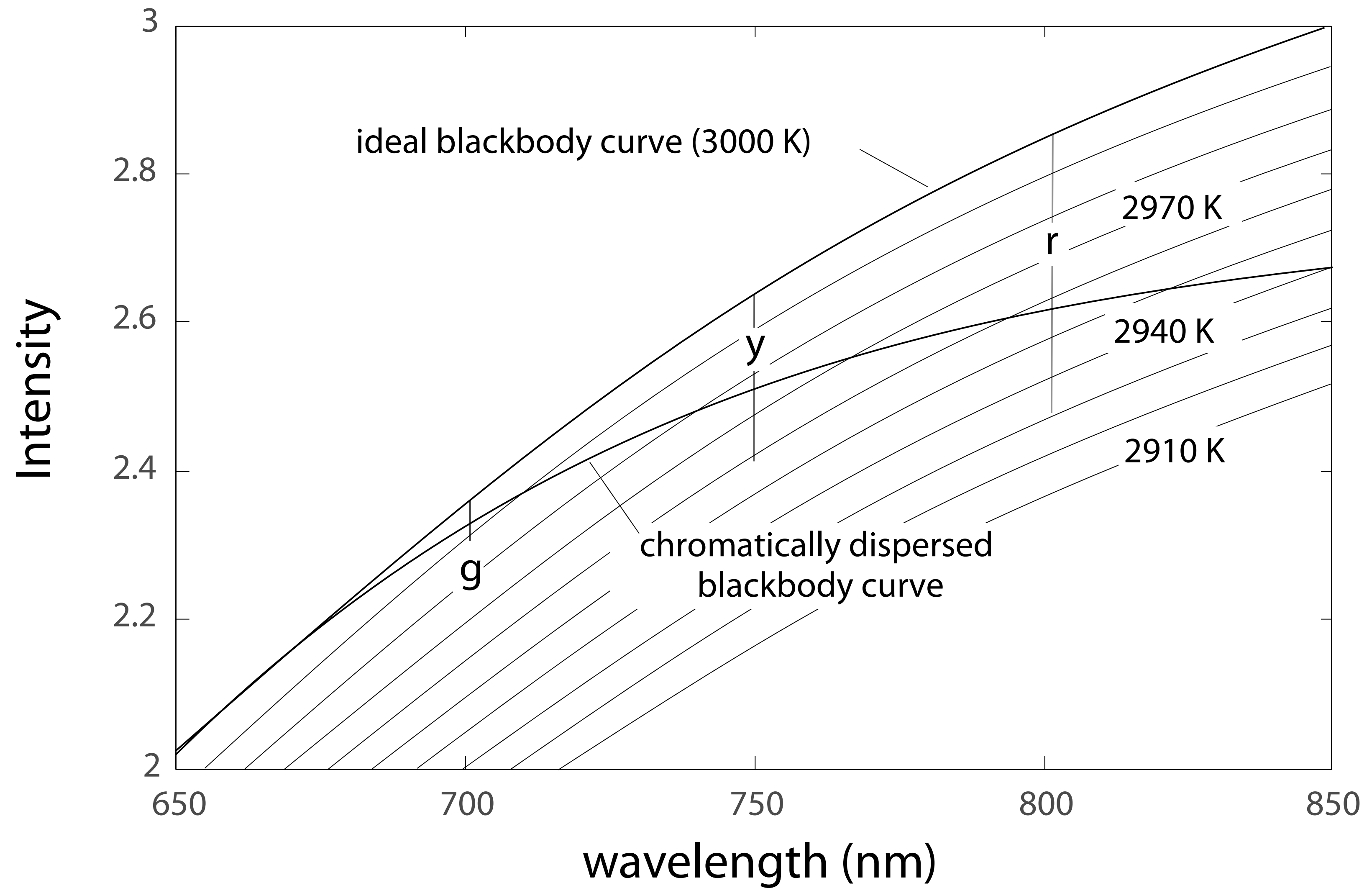
General overview of a basic temperature measurement setup (one side)

C- T measurement

Chromatic aberrations can be problematic in the case of a heterogeneous heating spot (typically: gaussian)







Walter and Koga (2004)

The focal length of a lens or refractive objective is wavelength dependant (different wavelength focused at different positions along the optical axis)

C- T measurement

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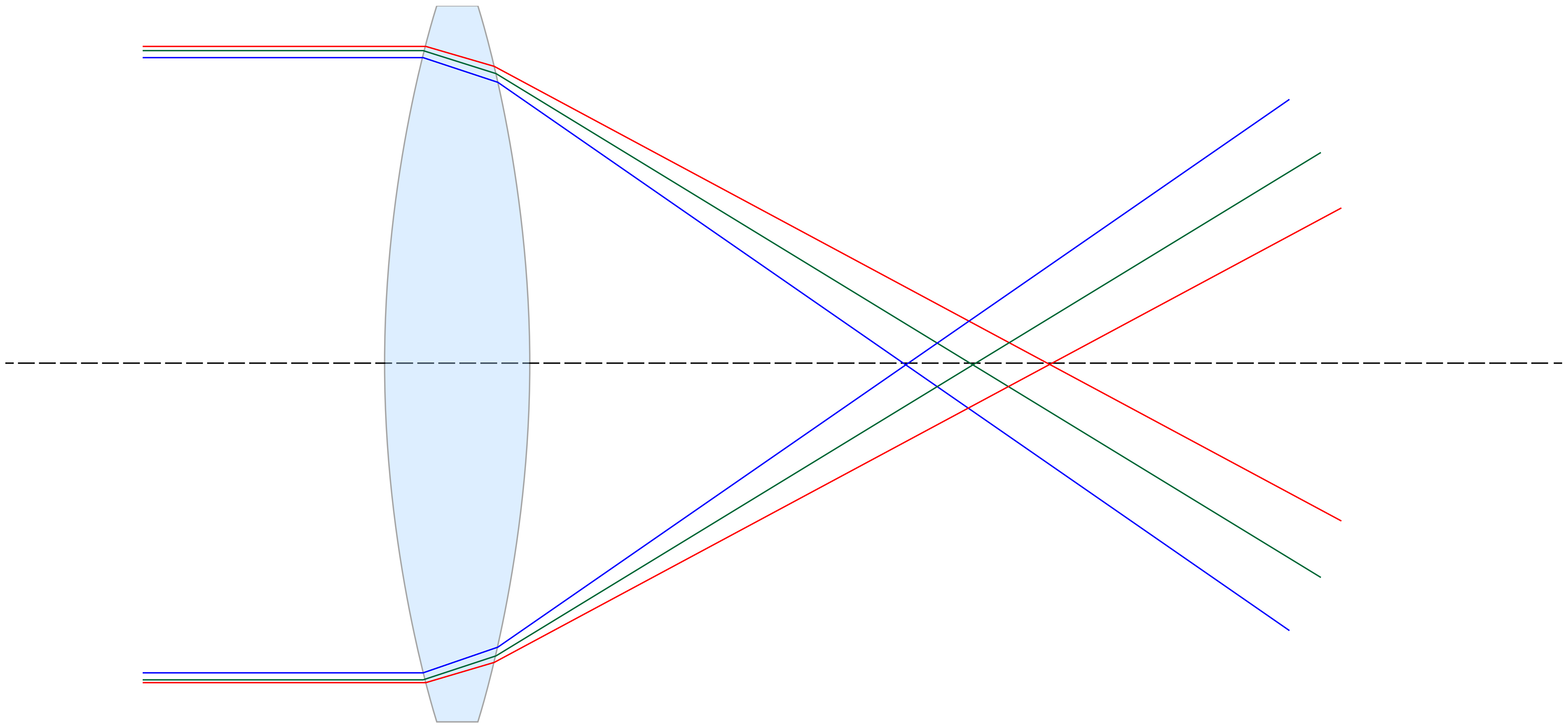

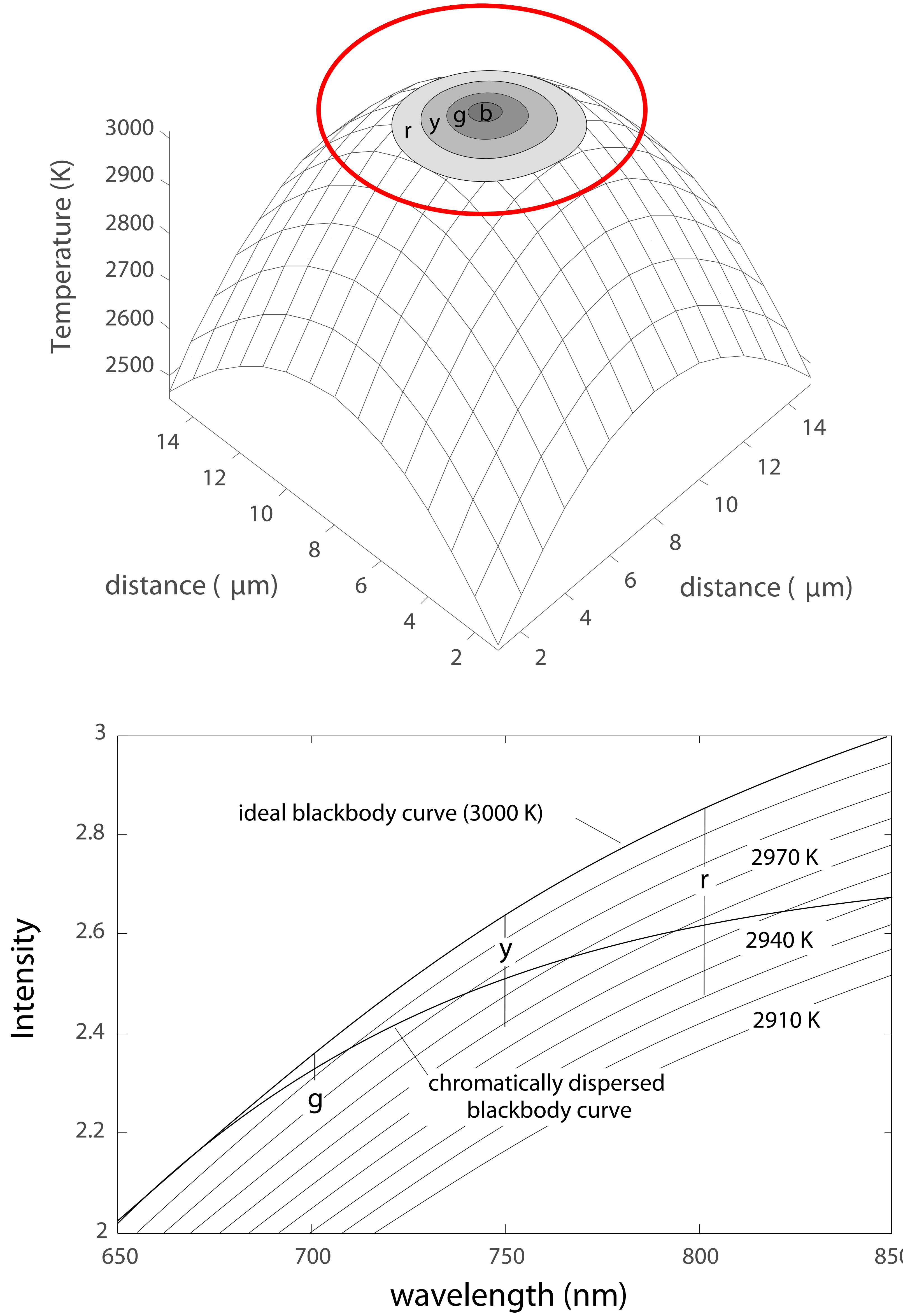

Walter and Koga (2004)

That means that the wavelength focused at a given position (e.g. entrance of the spectrometer) originate from different areas of the hot spot

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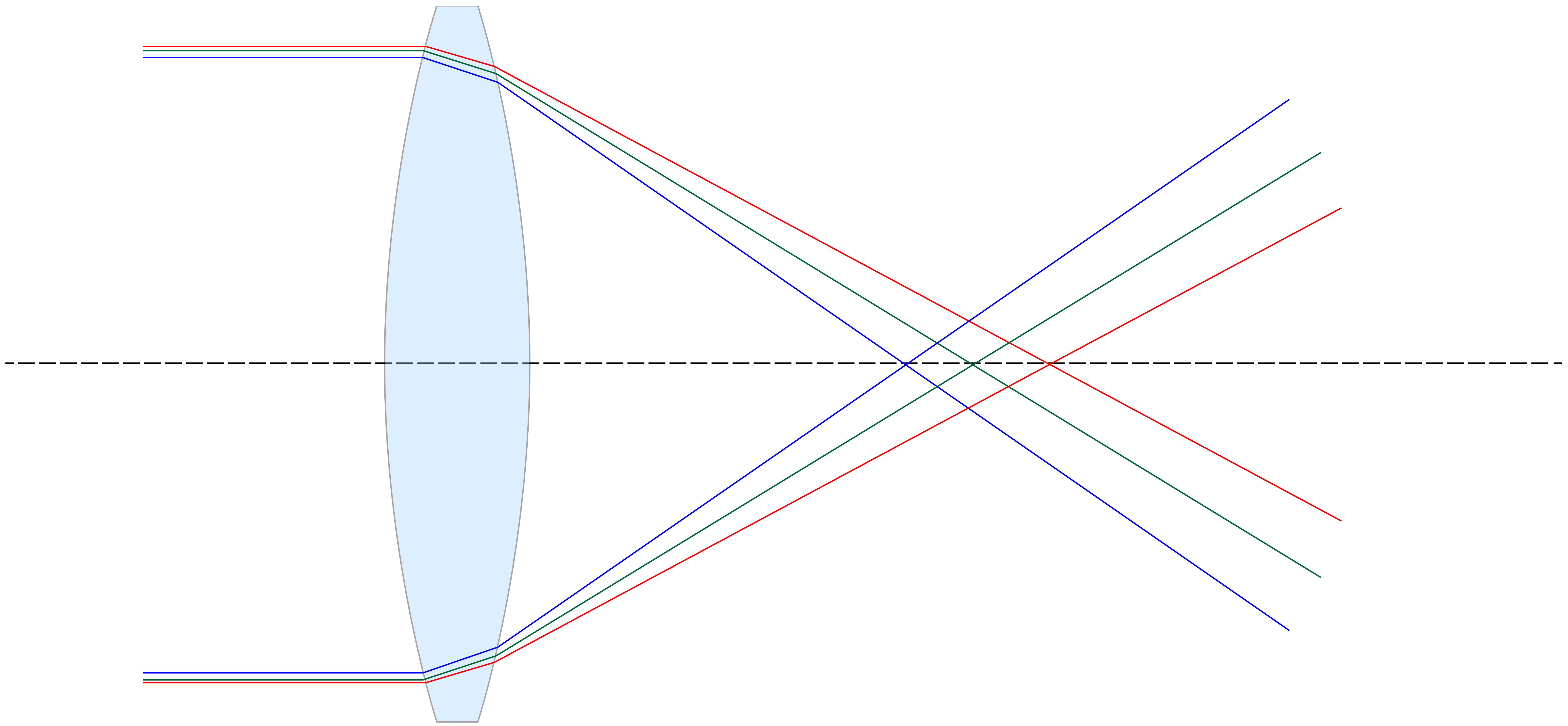
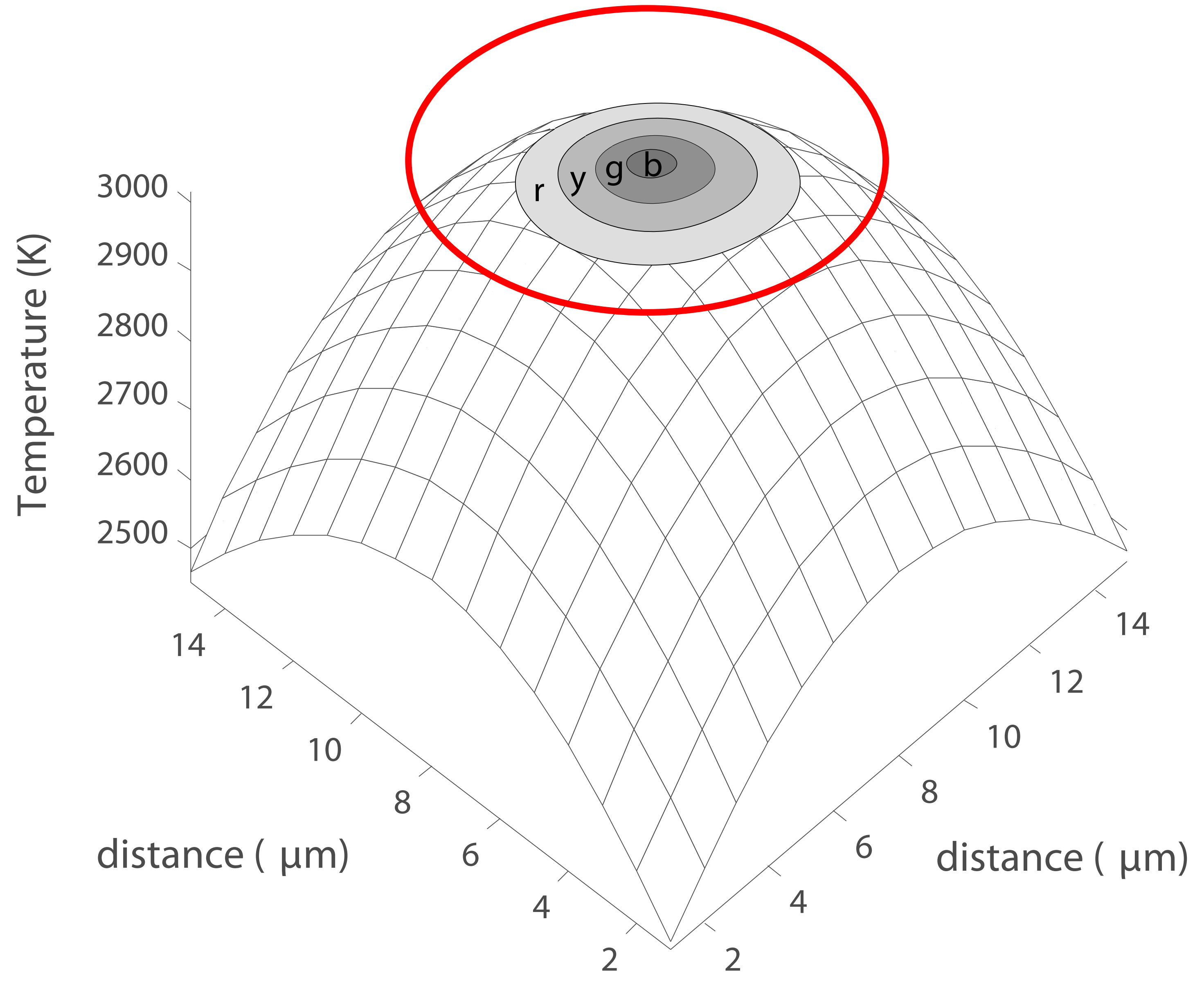
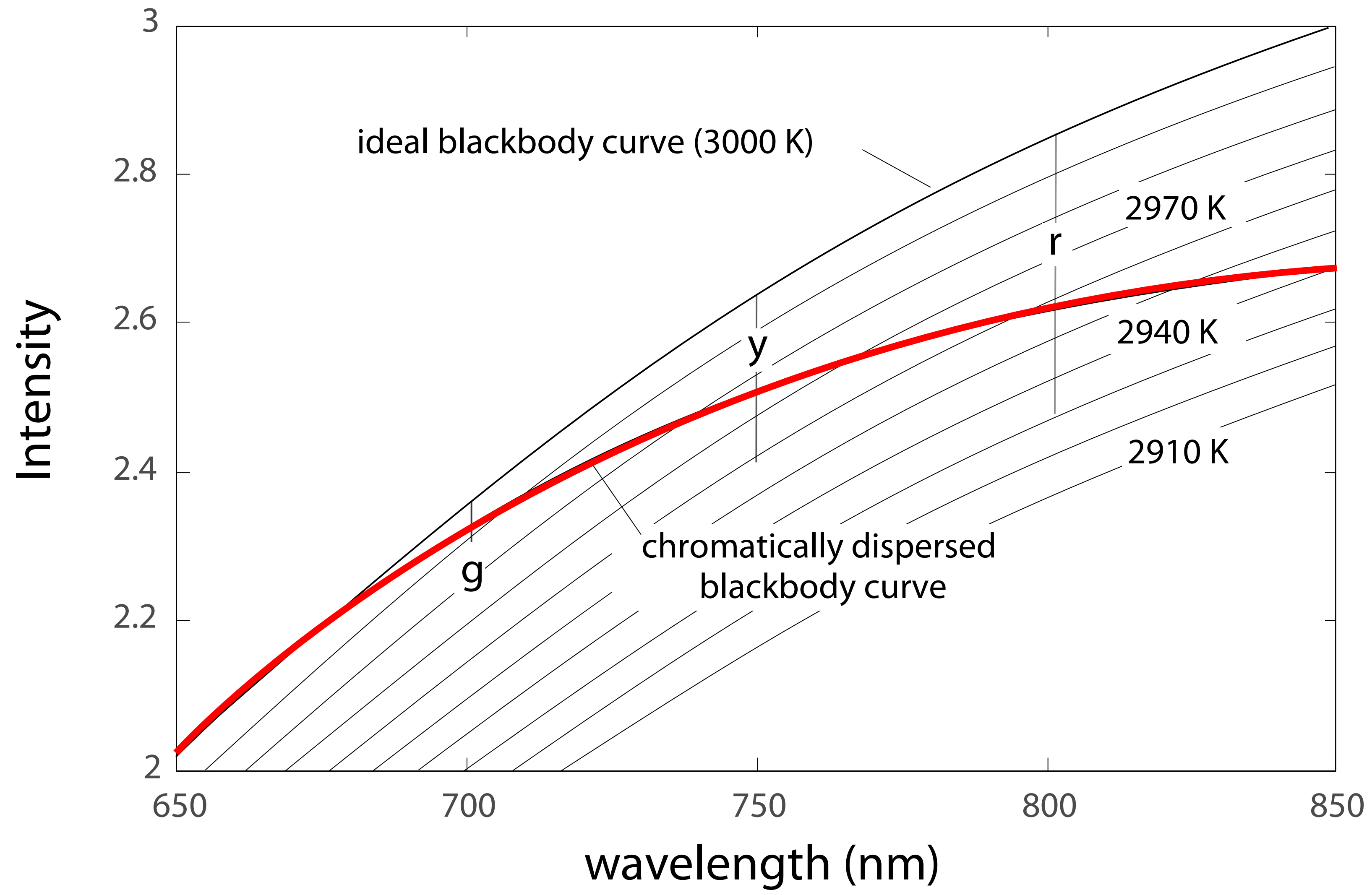






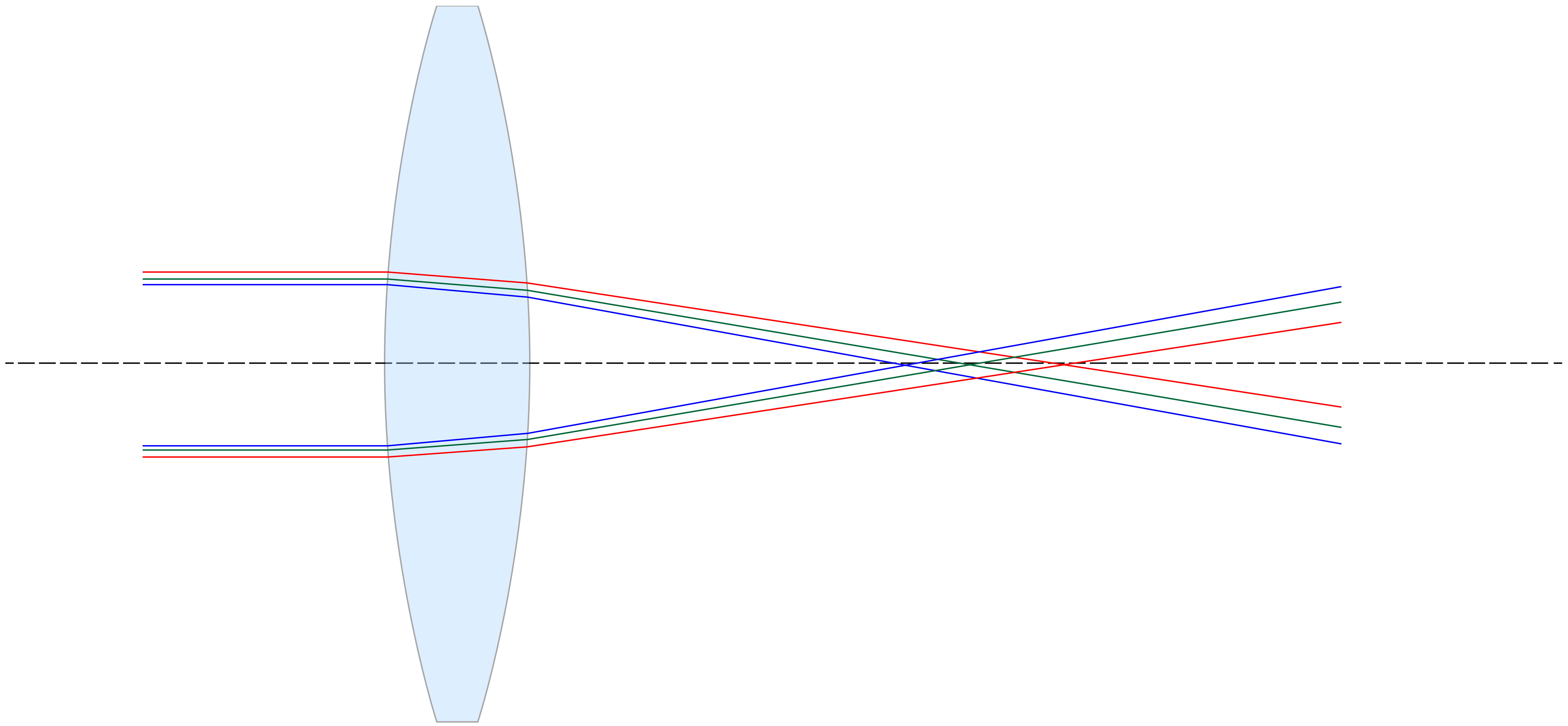
Walter and Koga (2004)

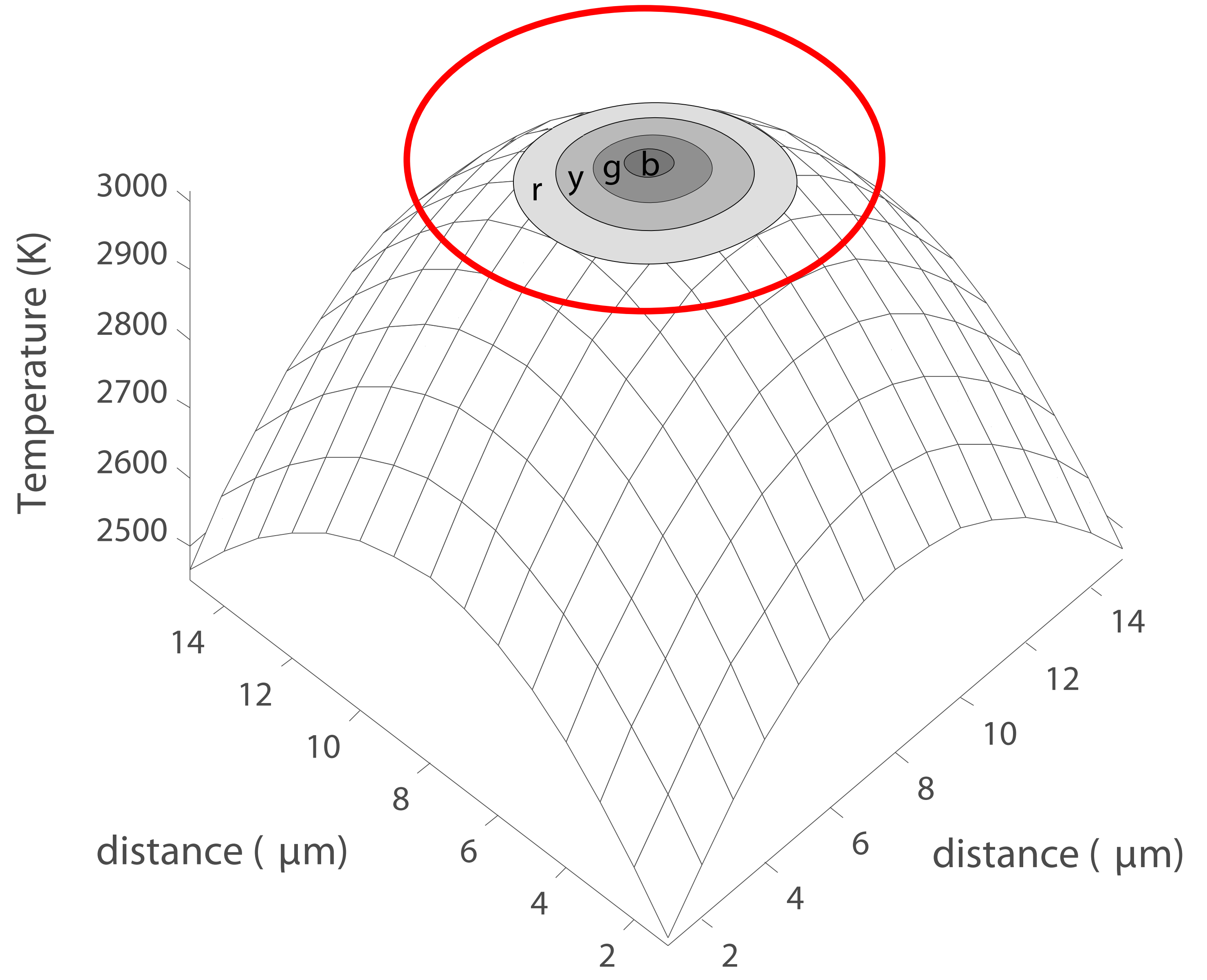
As a consequence the thermal radiation measure-ment is distorted

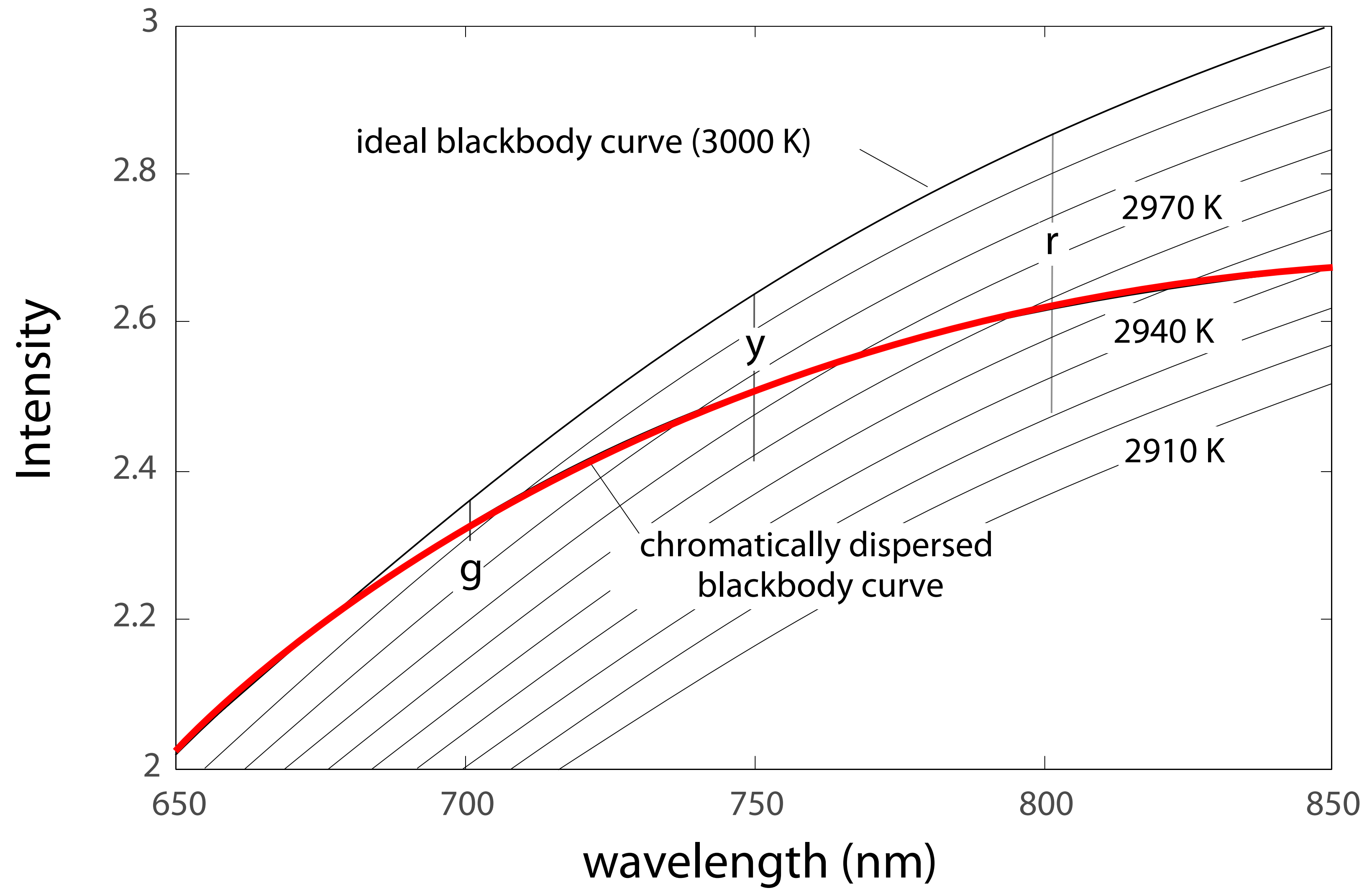
SOLEIL SYNCHROTRON

C- T measurement

Chromatic aberrations can be problematic in the case of a heterogeneous heating spot (typically: gaussian)







Walter and Koga (2004)

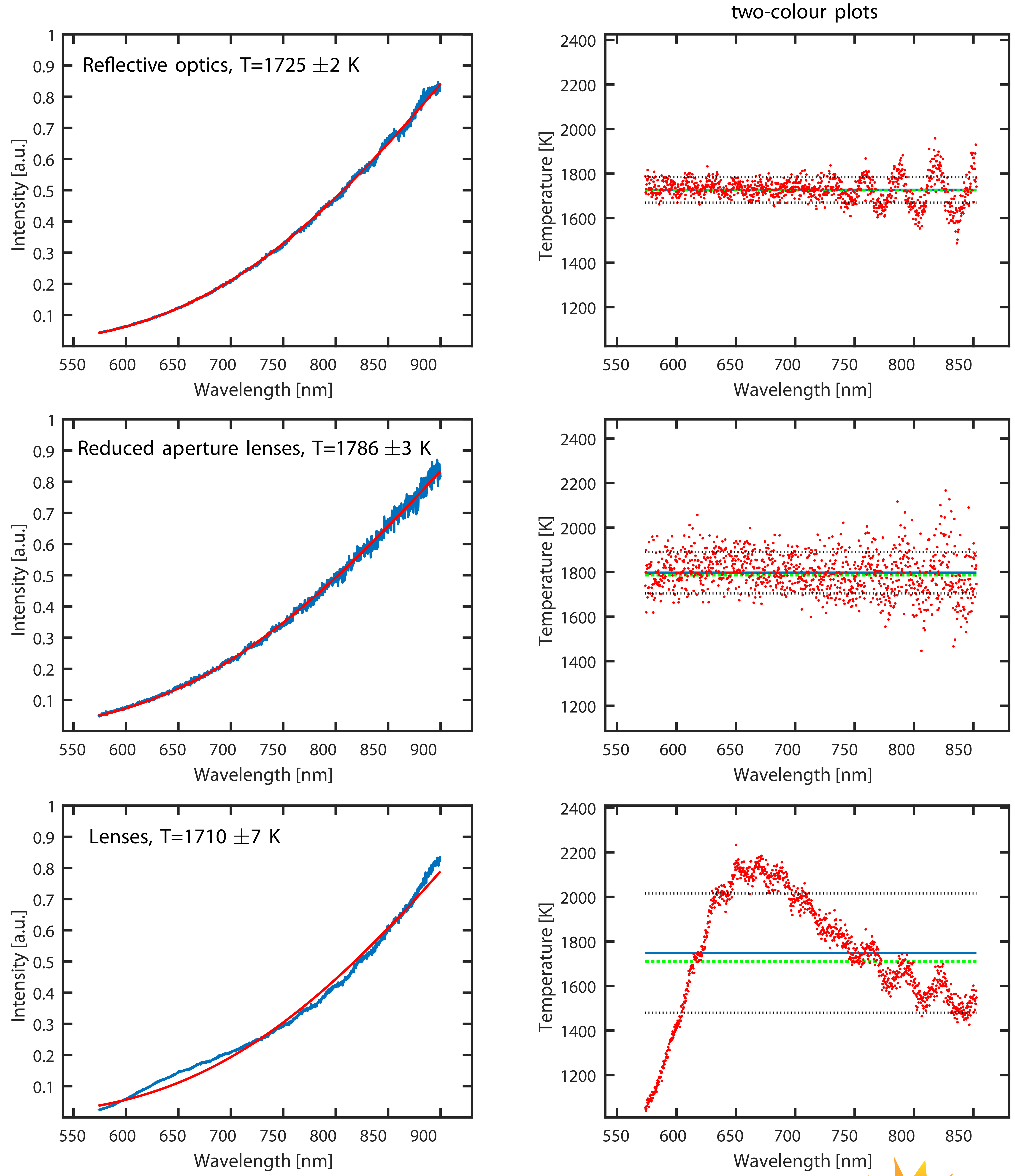
Reducing the NA helps by increasing the DOF, but at the expense of image resolution

C- T measurement

Comparisons between measurements done with reflective objectives, free of chromatic aberrations (Schwarzchild objectives) and refractive objectives show that a good agreement is observed at relatively low T (up to 2600 K) by lowering the NA.

At higher T the difference becomes significative.

Minimizing chromatic aberrations is desirable



Giampaoli et al. 2018

SOLEIL SYNCHROTRON

C- T measurement

The 4-color pyrometry method

Measurement of temperature distributions across laser heated samples by multispectral imaging radiometry

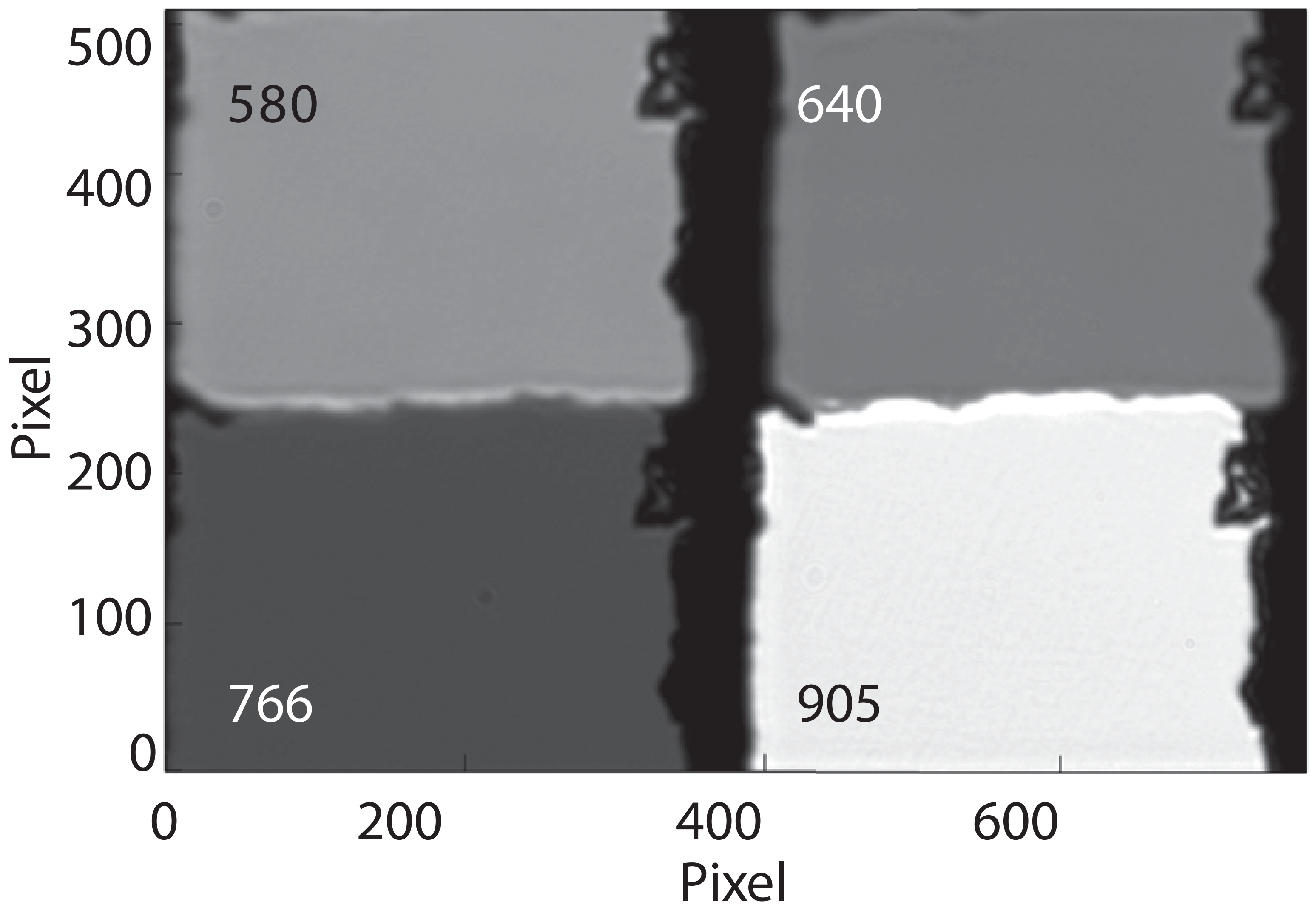
Andrew J. Campbell
Department of Geology, University of Maryland, College Park, Maryland 20742, USA

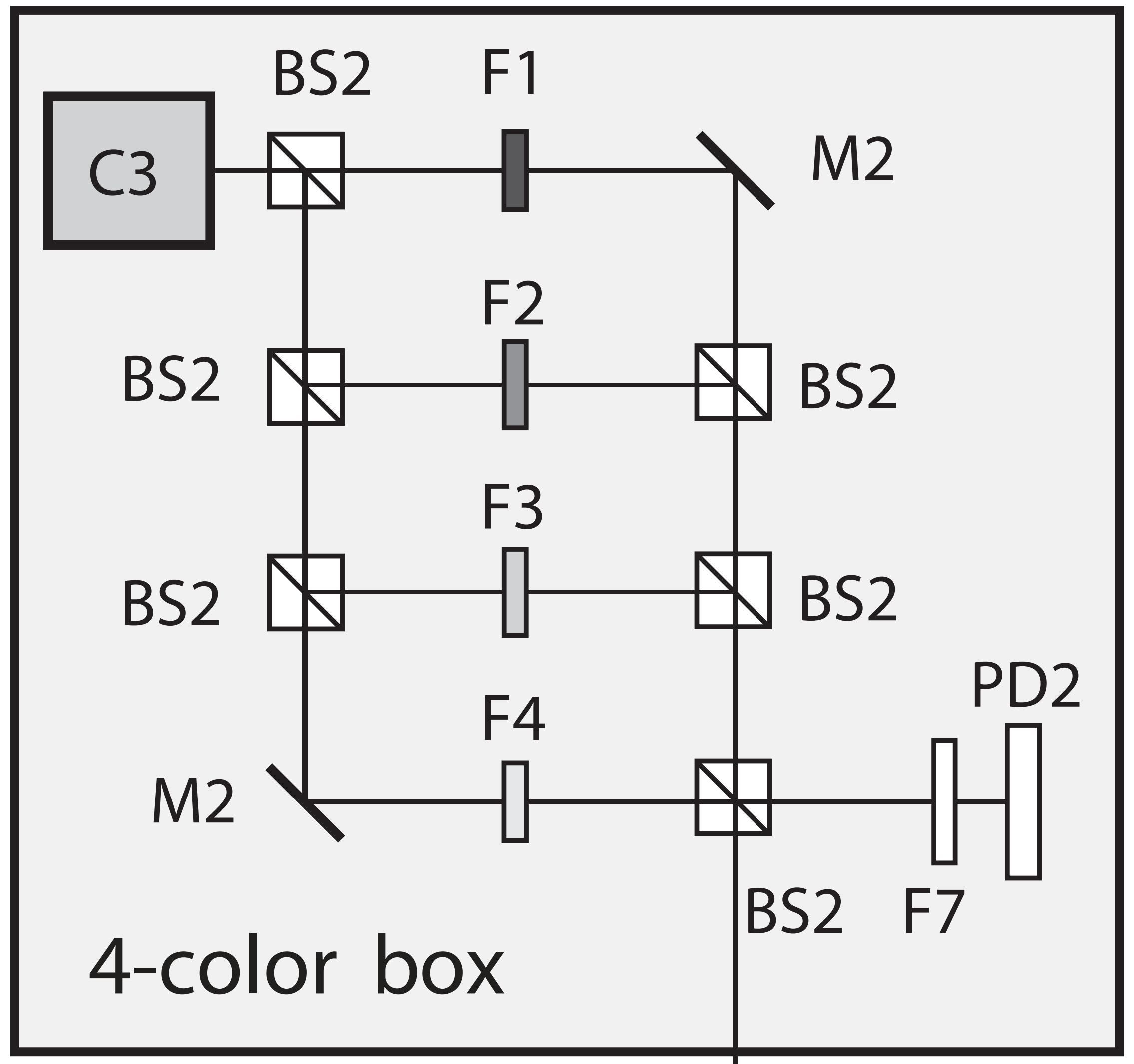
Two-dimensional temperature mapping of laser heated diamond anvil cell samples is performed by processing a set of four simultaneous images of the sample, each obtained at a narrow spectral range in the visible to near infrared.

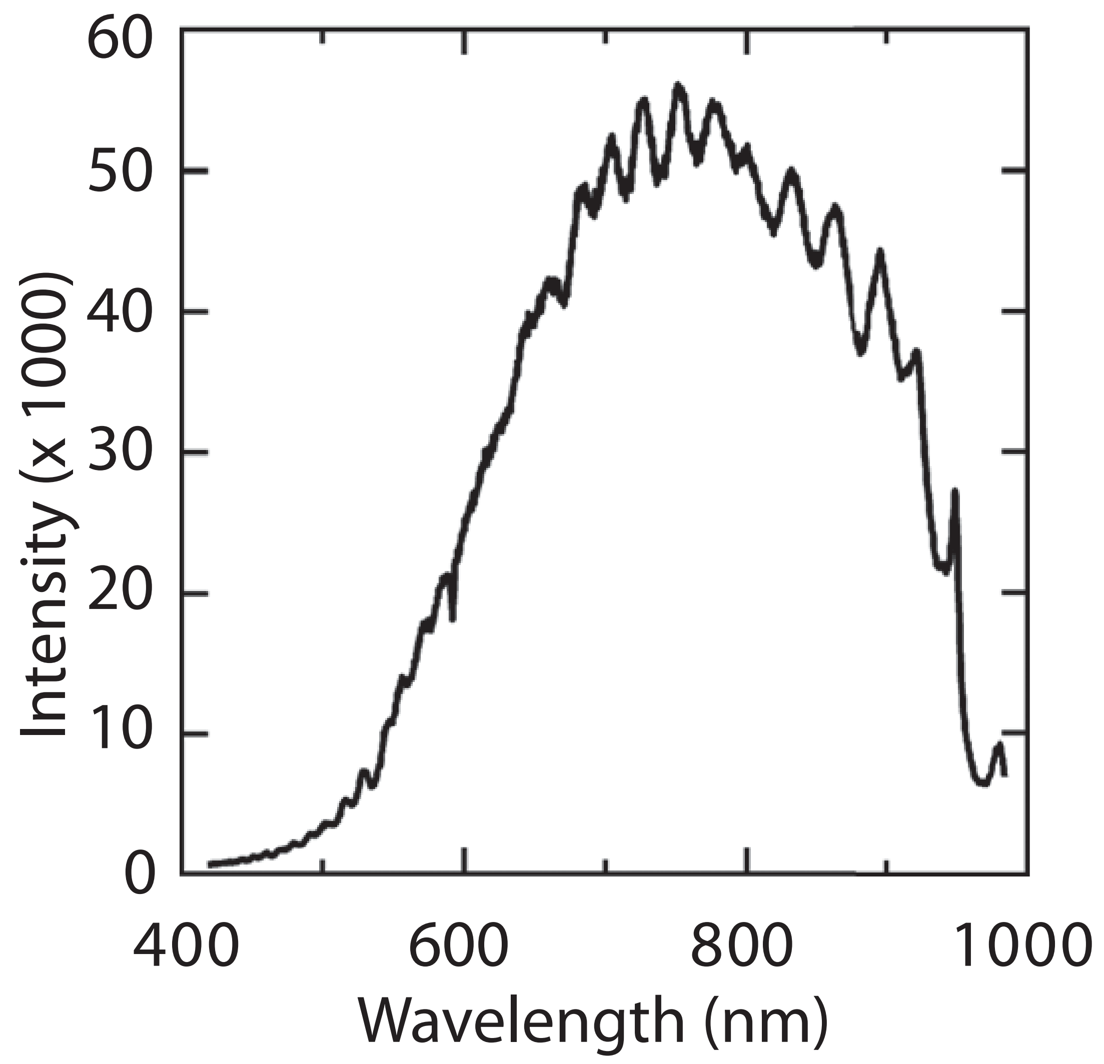
The images are correlated spatially, and each set of four points is fitted to the Planck radiation function to determine the temperature and the emissivity of the sample, using the gray body approximation.

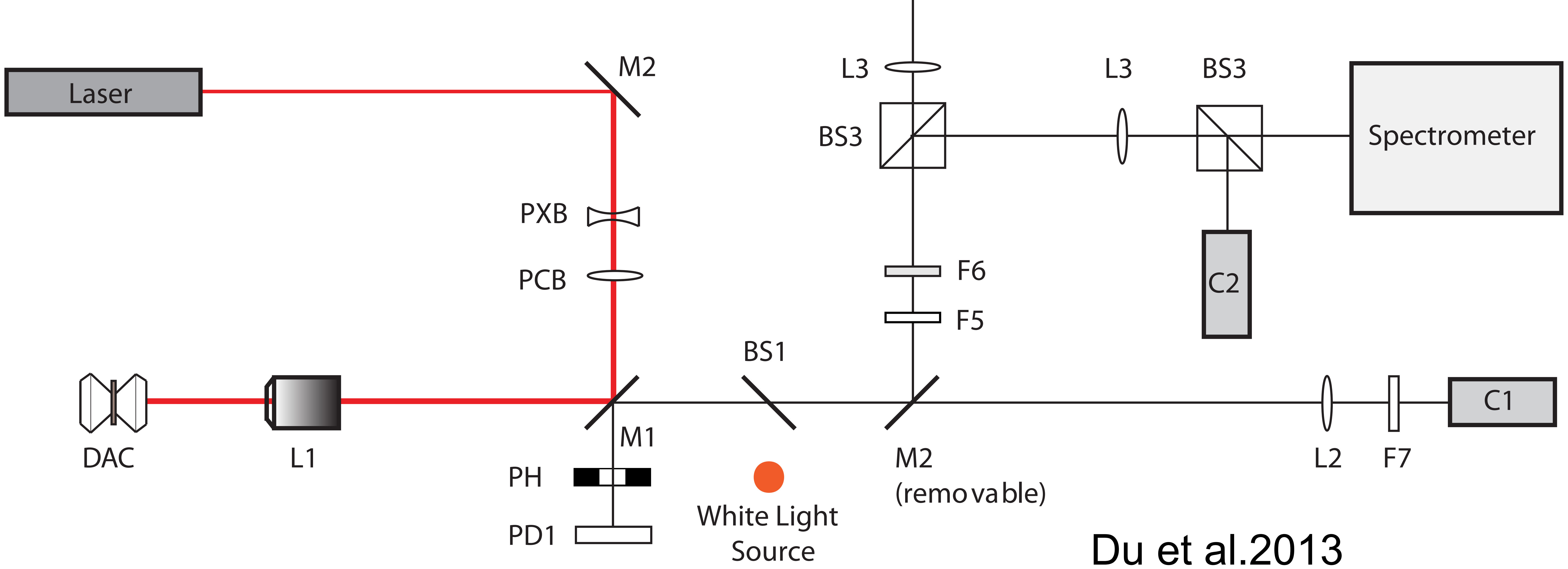
C- T measurement

The 4-color pyrometry method





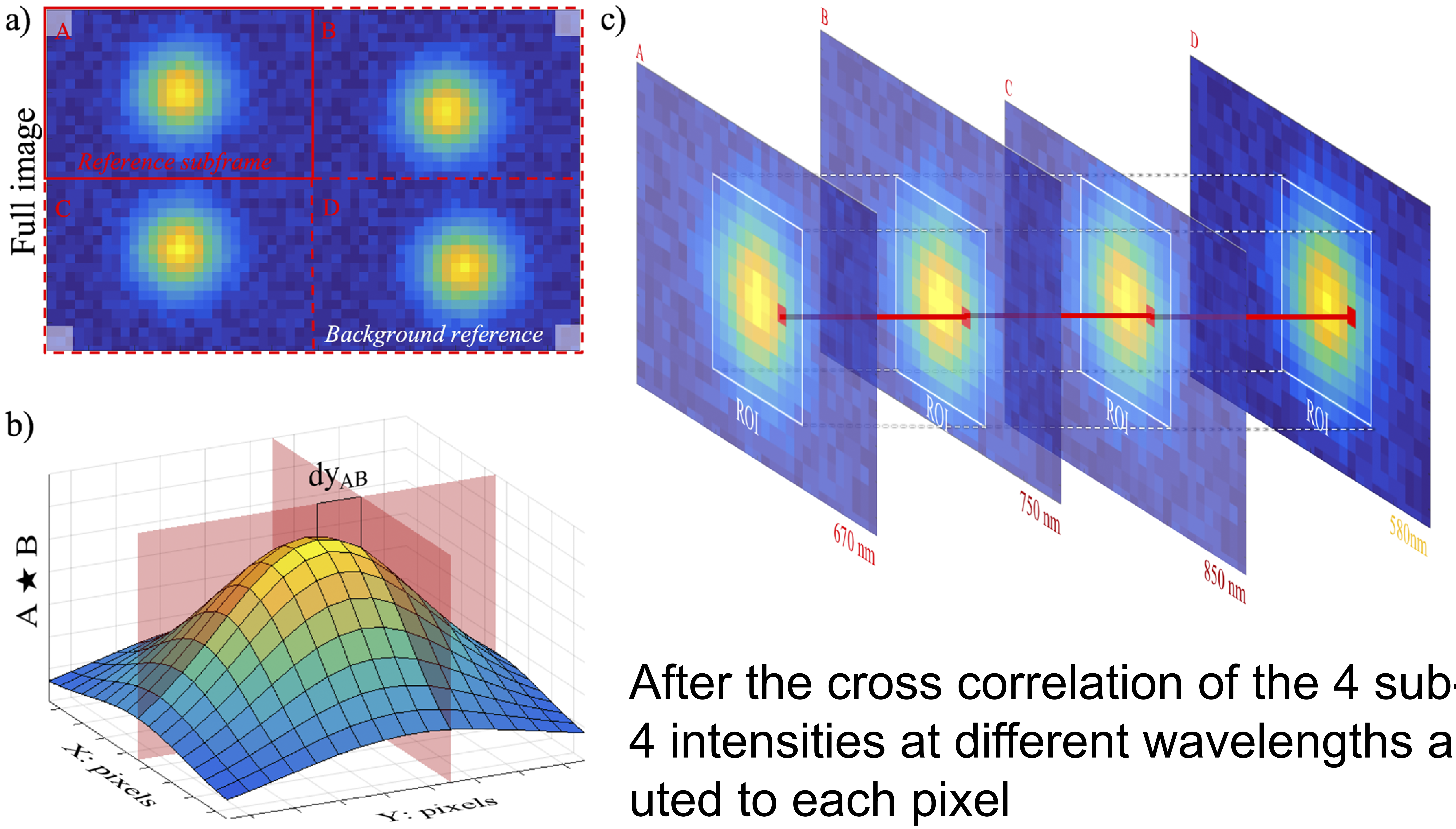




Du et al.2013

C- T measurement

The 4-color pyrometry method



After the cross correlation of the 4 sub-images, 4 intensities at different wavelengths are attributed to each pixel

Lord and Wang 2018 (MIRRORS)

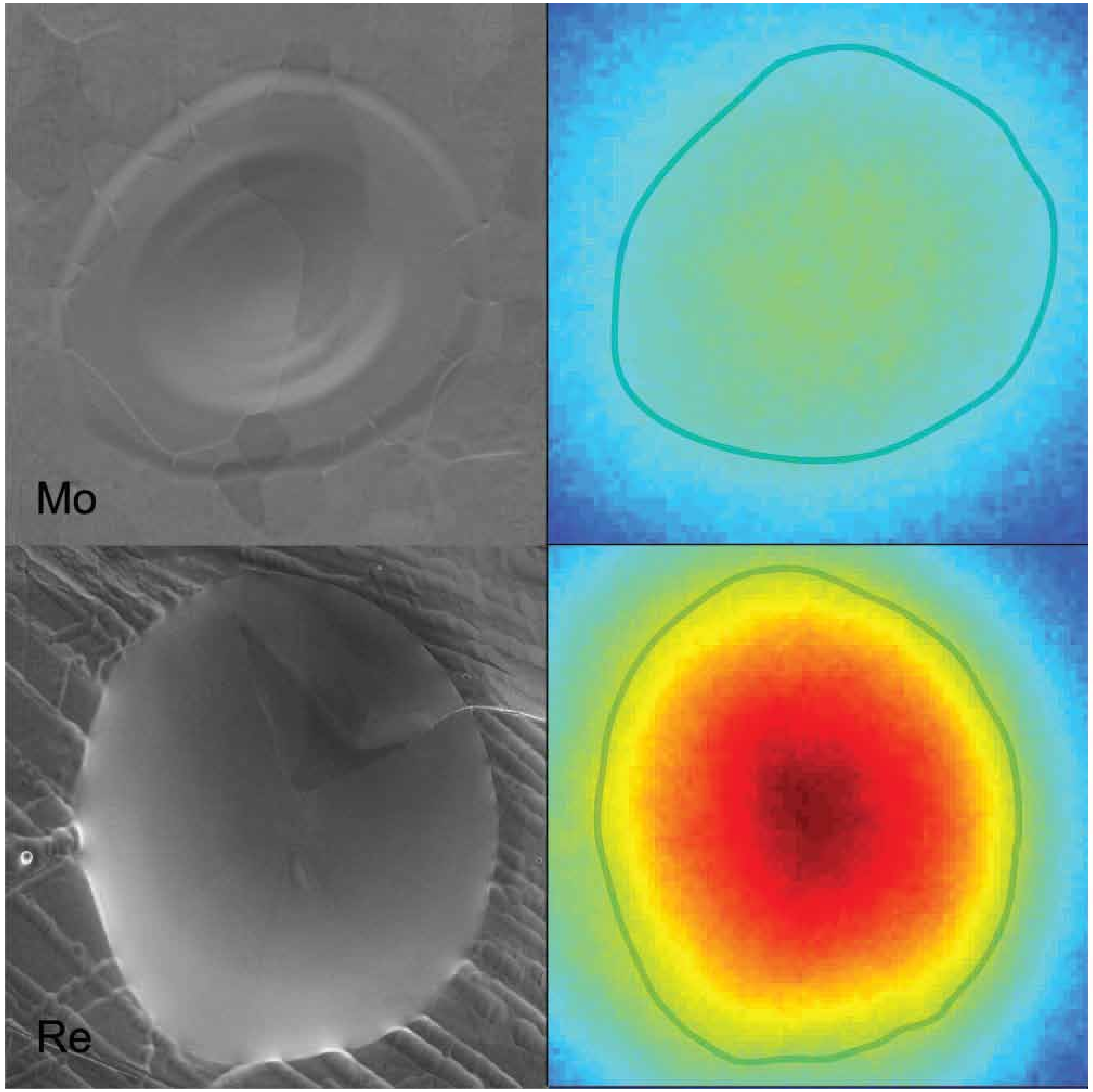
SOLEIL SYNCHROTRON

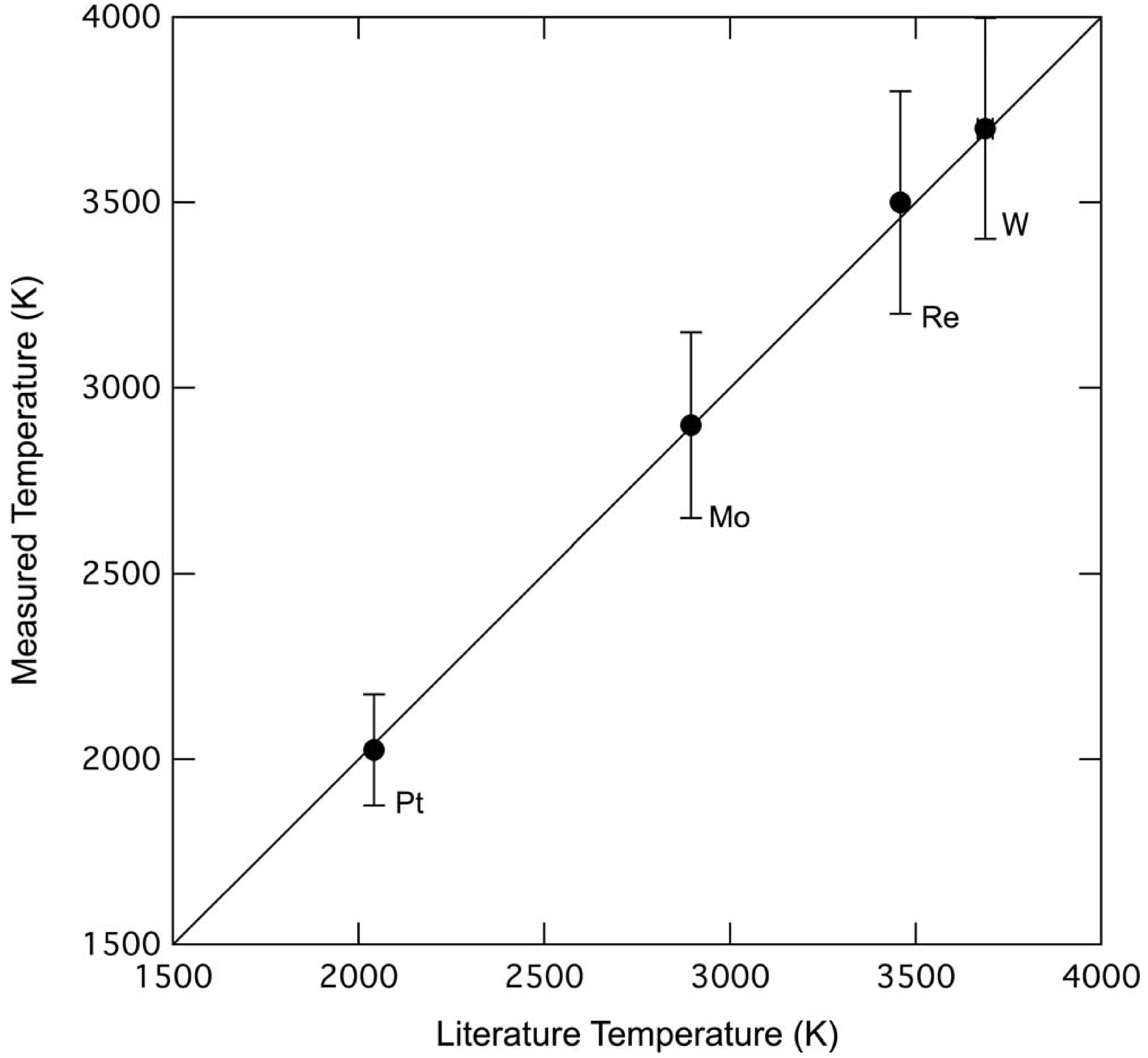
C- T measurement

The 4-color pyrometry method

Du et al. 2013

Surface T mapping





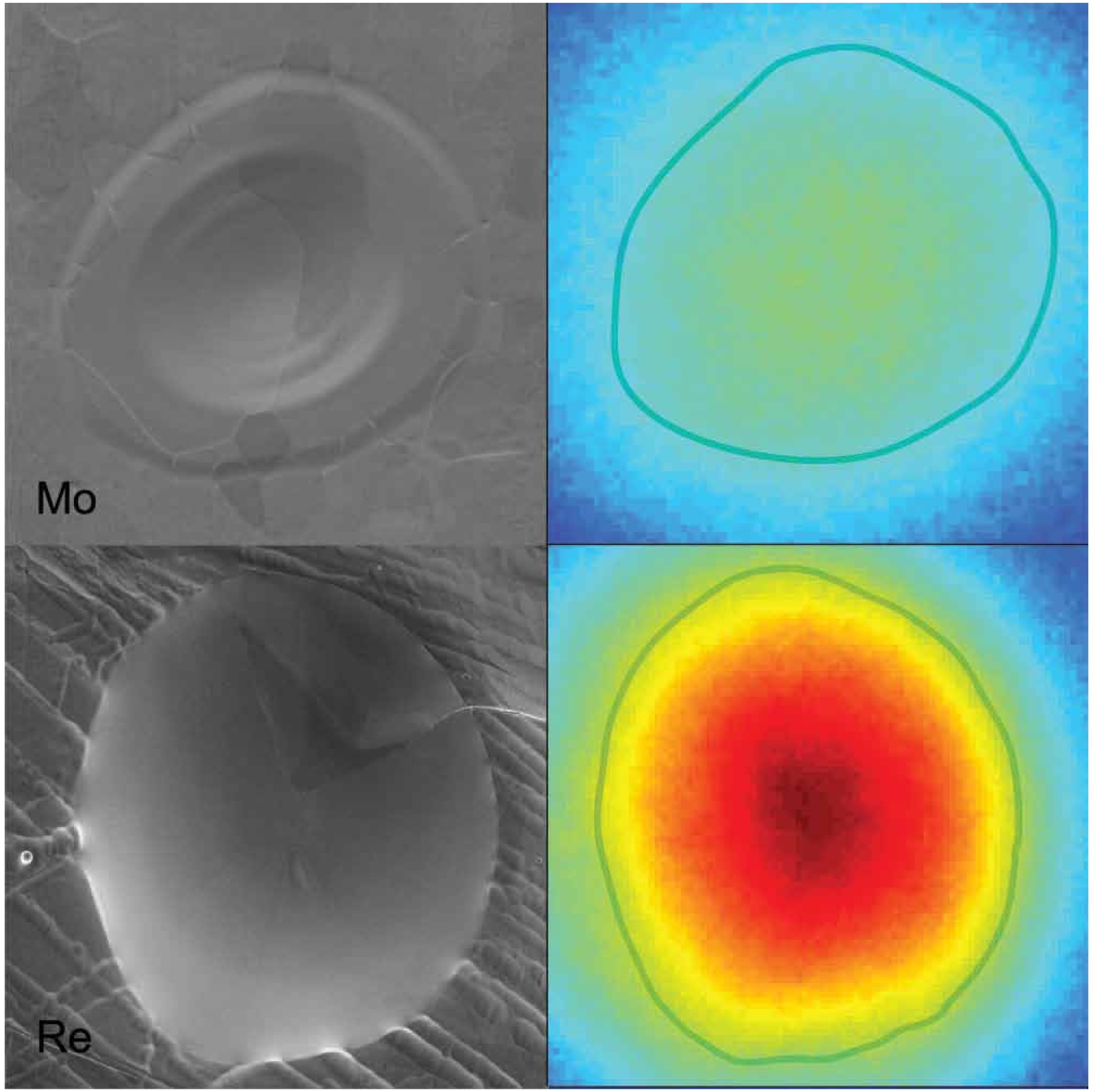
Good correspondance of flash heating T maps and melting structures observed by microscopy

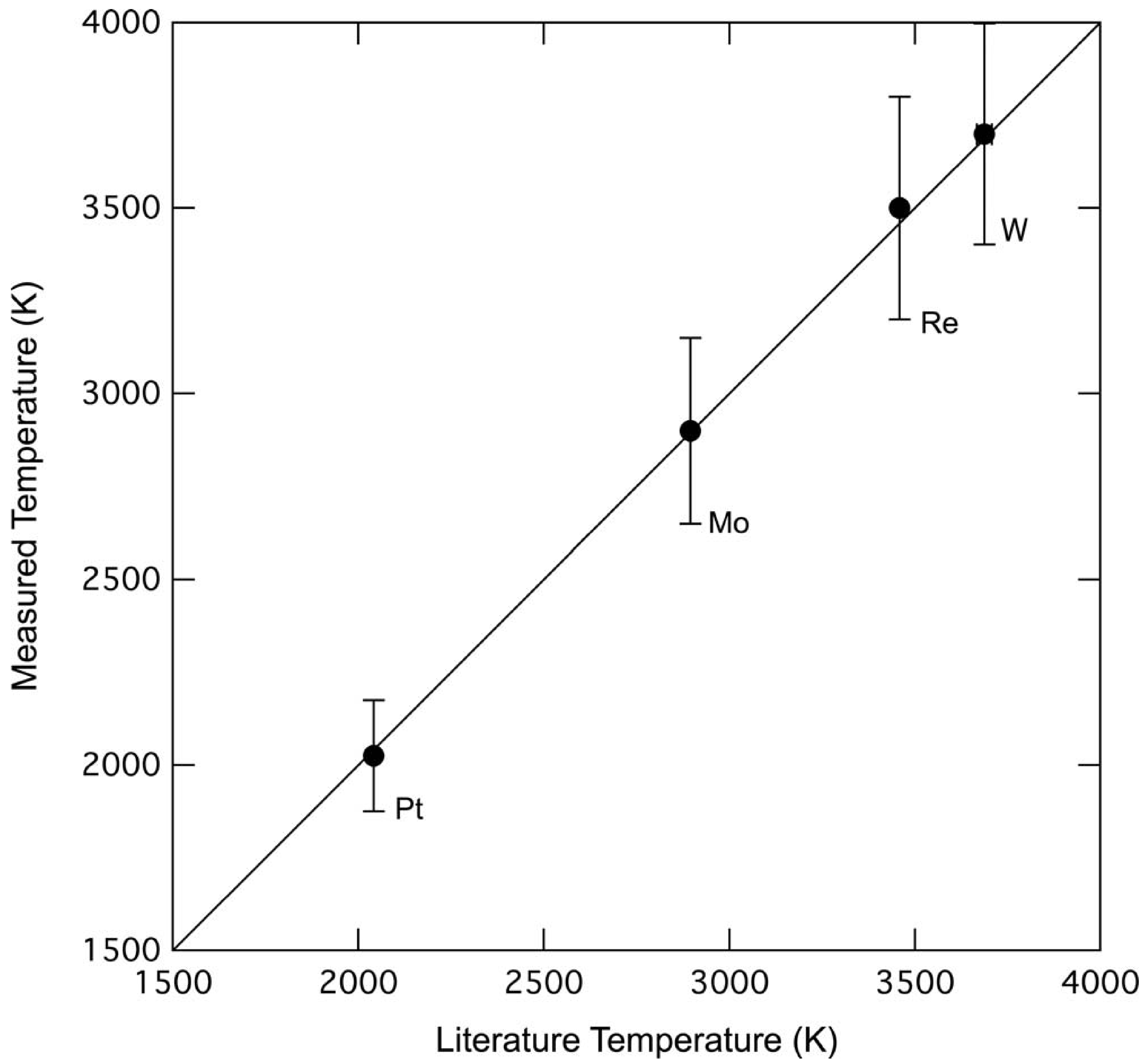
C- T measurement

The 4-color pyrometry method

Du et al. 2013

Surface T mapping

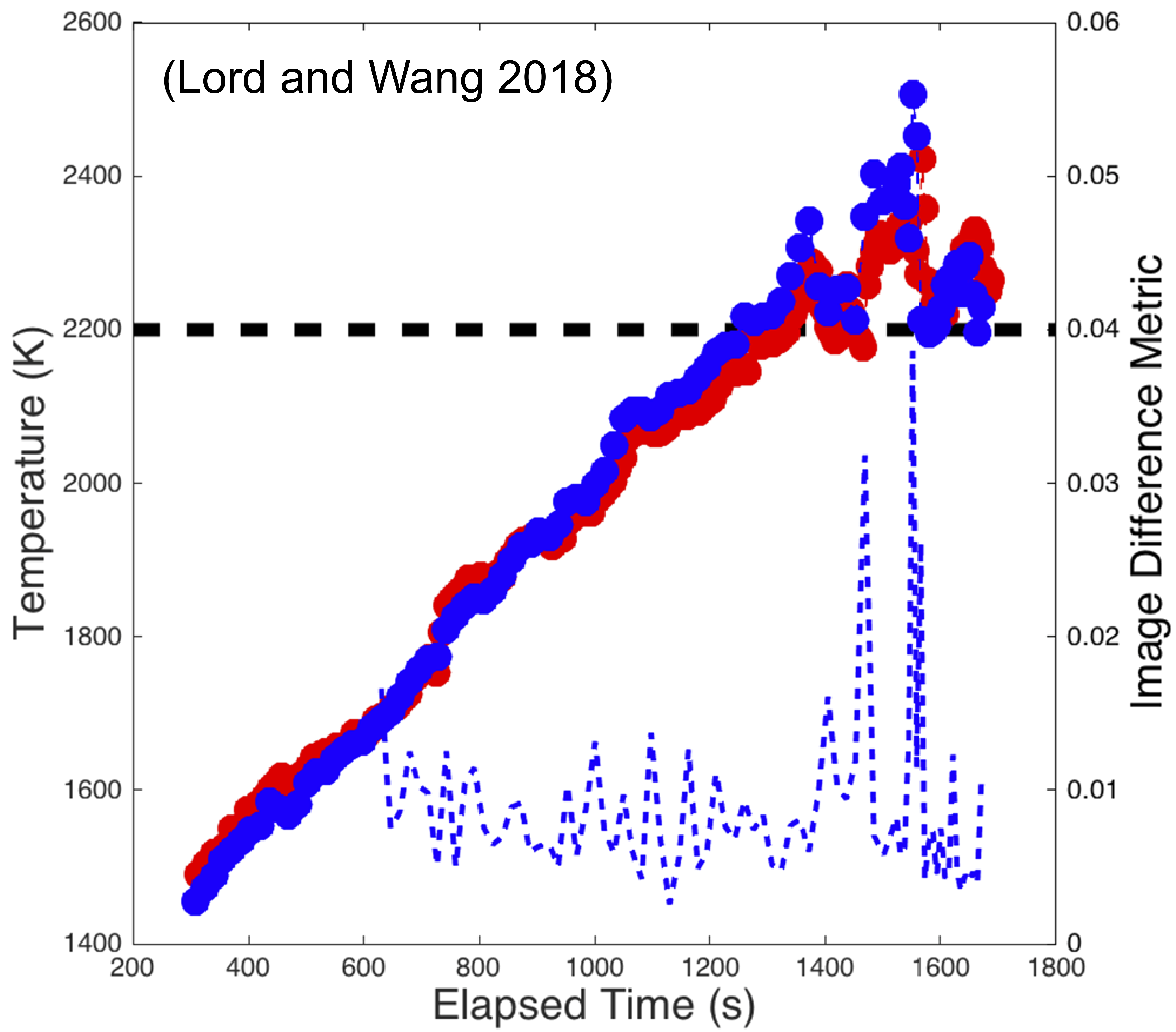




Also a good agreement between measured melting T for different metals and literature values

C- T measurement

The 4-color pyrometry method



In general the agreement between the T measured by spectroradiometry (single point) and 4-color spectrometry looks very good

ex: Heating ramp on a Fe–S–Si ternary alloy at ~50 GPa. Blue dots : peak T measured by the 4-color pyrometry system, red dots: spectroradiometry

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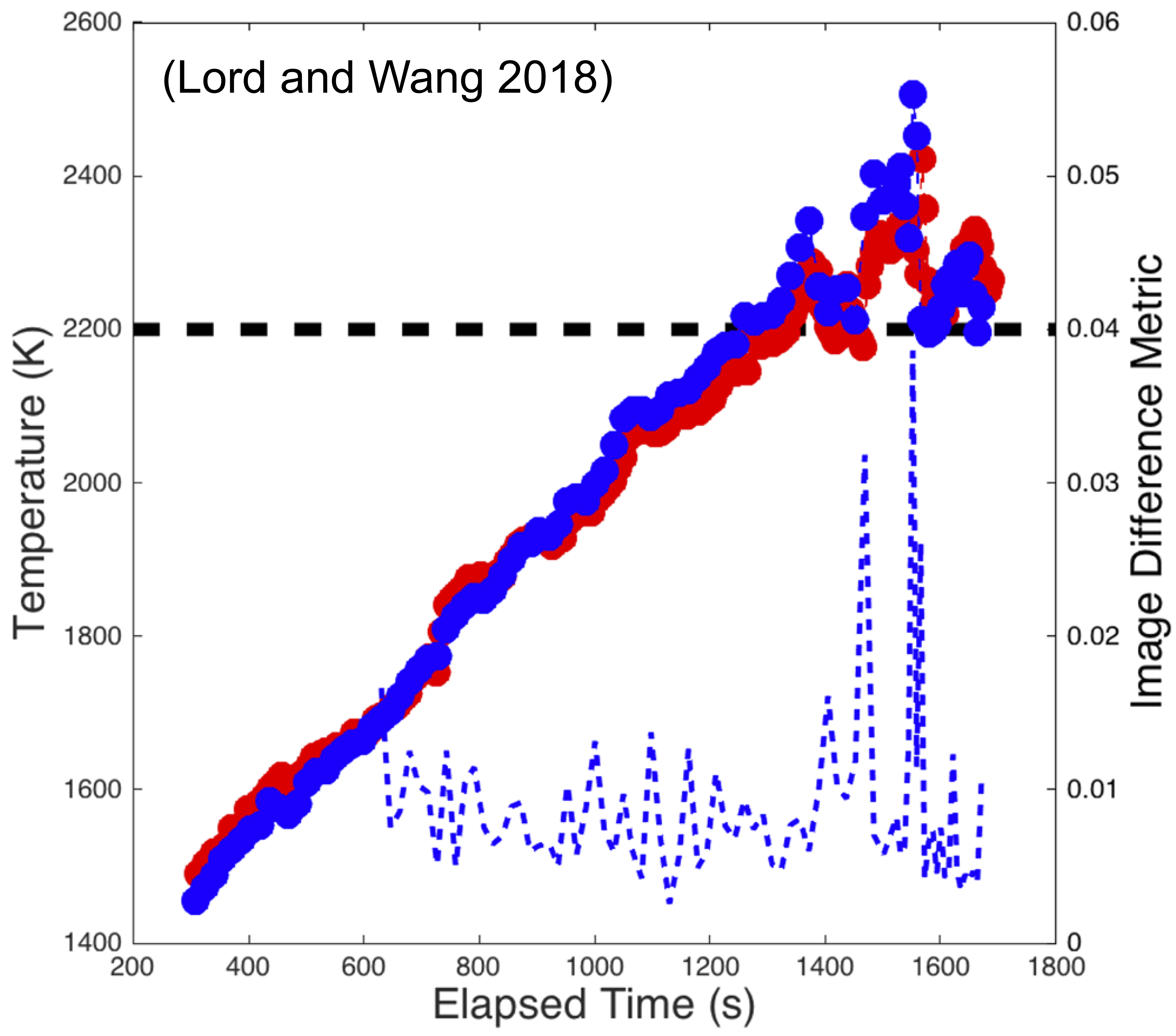
C- T measurement

The 4-color pyrometry method



Advantages of the method:

- truly achromatic (each wavelength focused independantly)
- the peak temperature can always be determined
- insensitive to misalignment (no entrance pinhole)
- observation of the dynamic changes in sample temperature in 2D, especially during melting

Disadvantage:

- larger uncertainties

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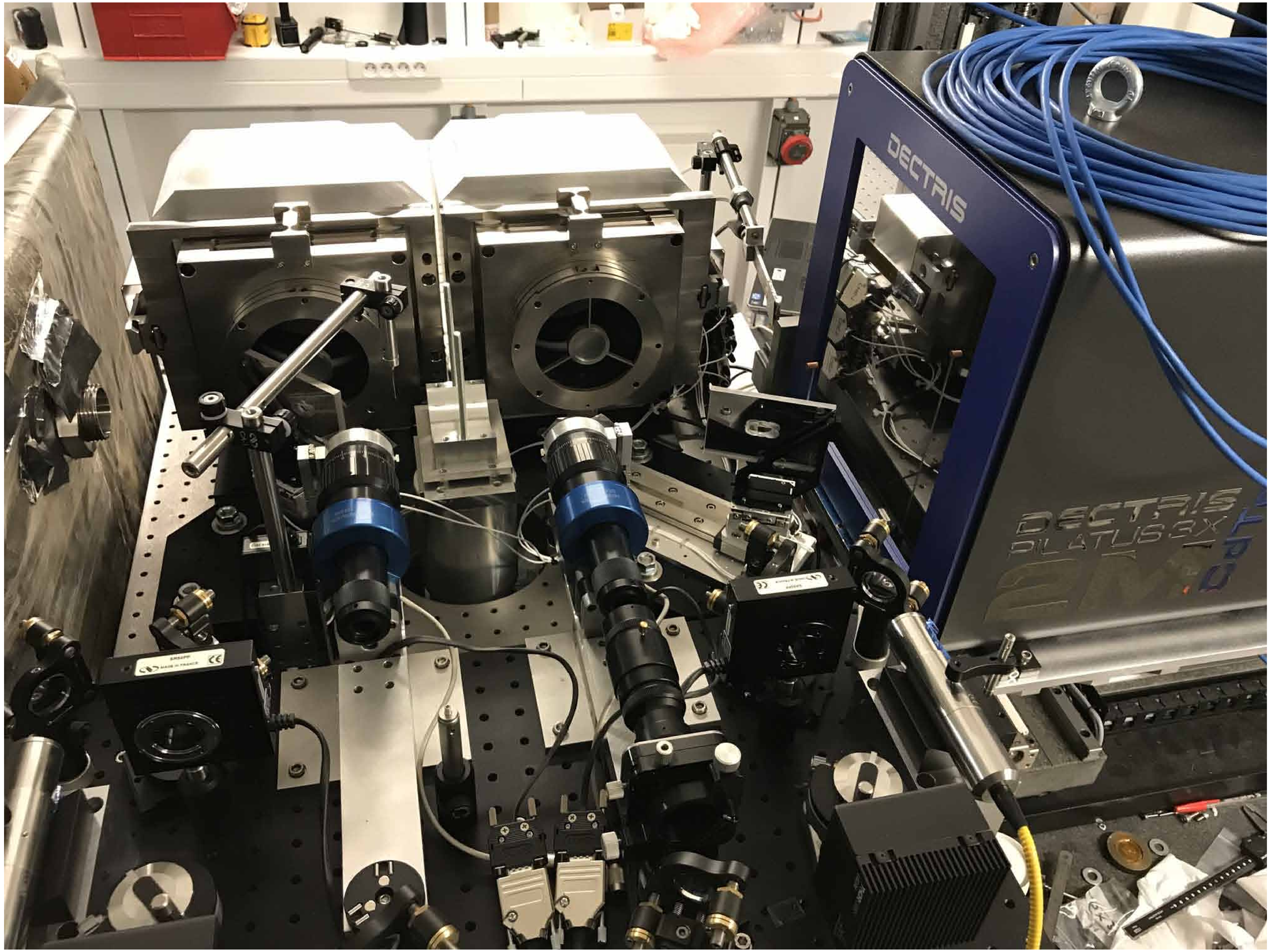
C- T measurement

The PSICHE Laser heating setup



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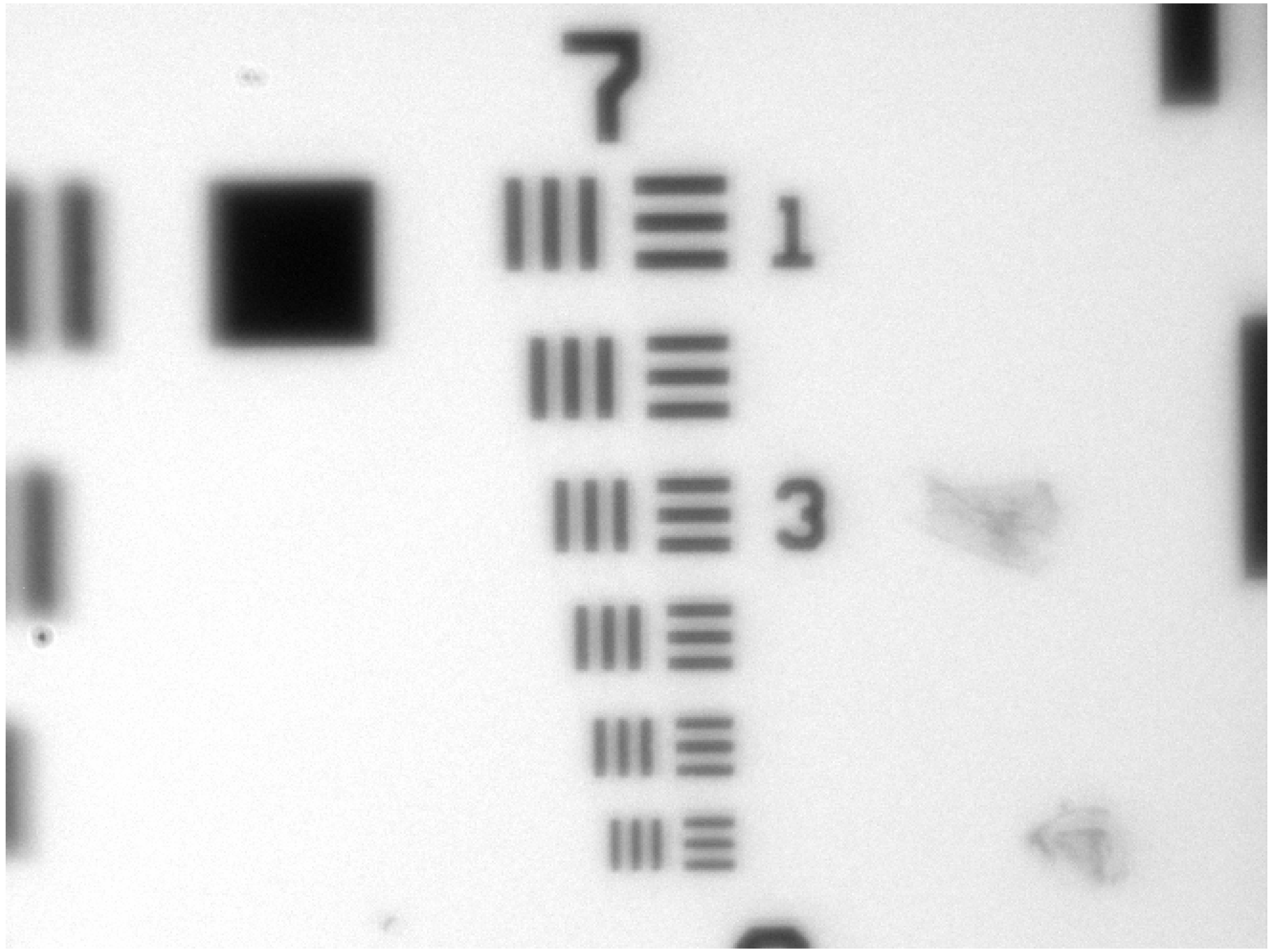
C- T measurement

The Schwarzchild objective is an in-house development

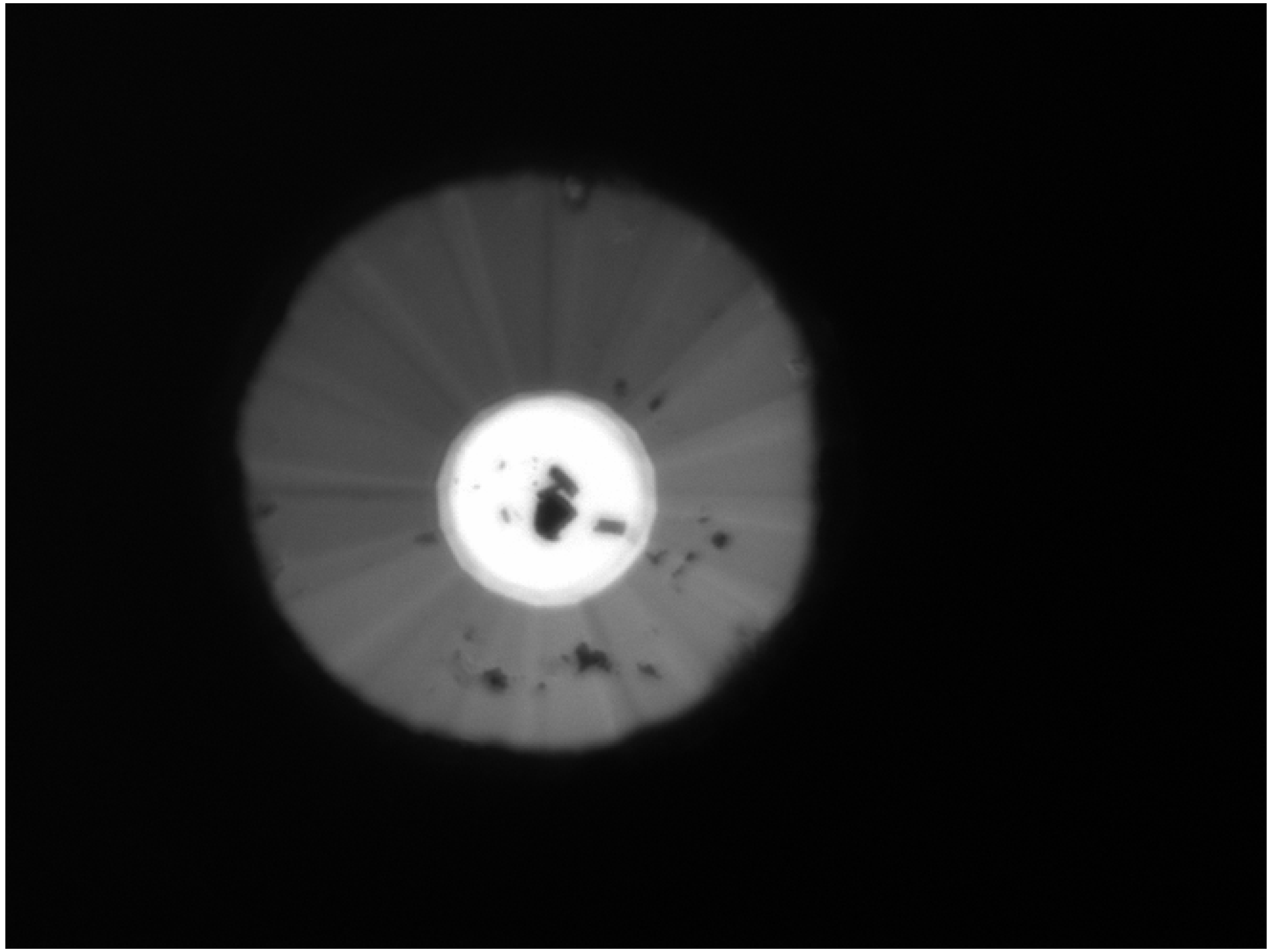
WD: 180 mm, NA 0.24, diffraction limited (1.5 µm resolution)



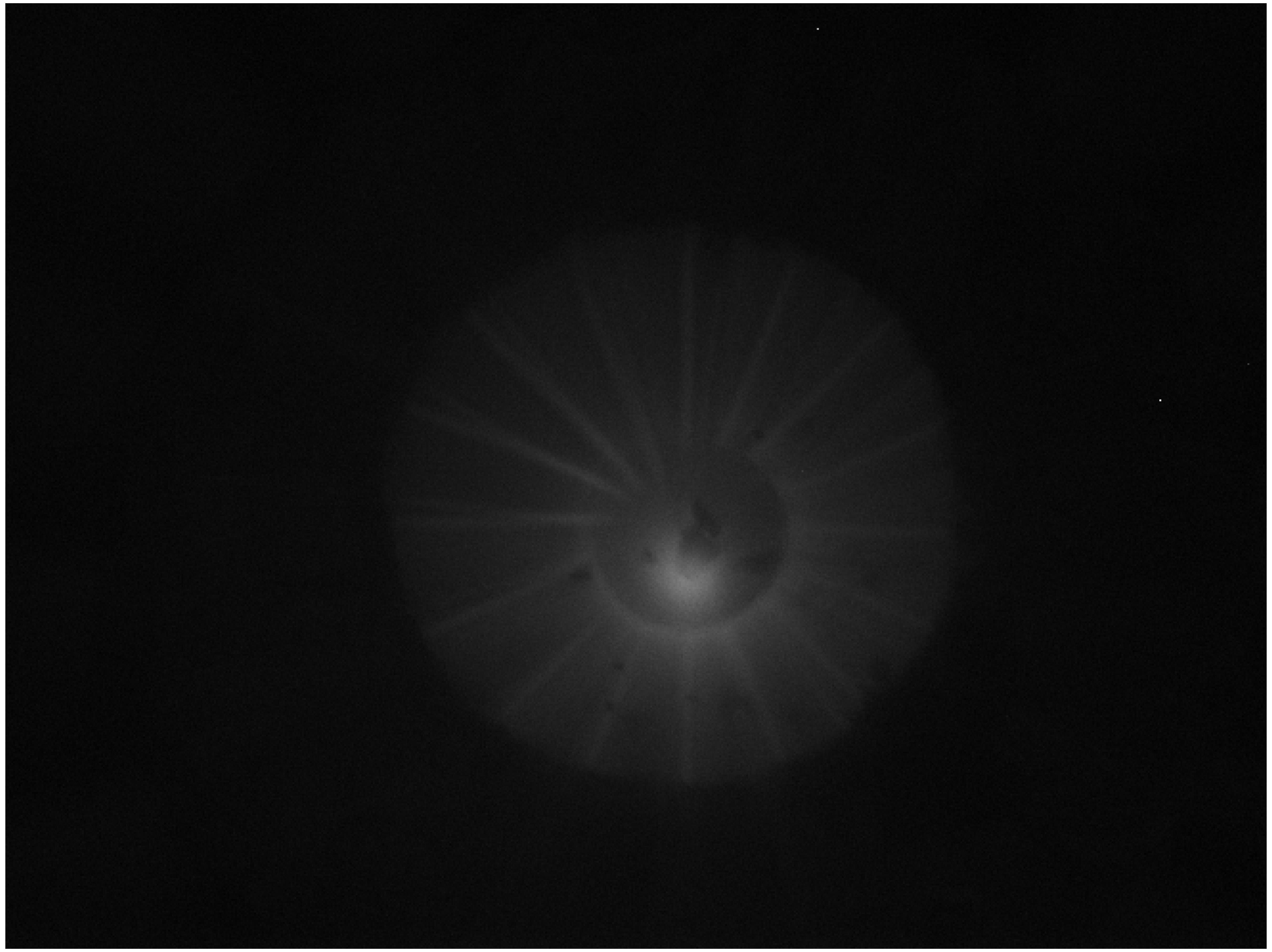
6th element of the group #7 resolved (line width 2.19 µm), USAF 1951

C- T measurement

The Schwarzchild objective is an in-house development



test image of 2 facing 50 µm bevel diamonds + dust



Diamond fluorescence using the X-ray beam

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