Temperature measurement under vacuum: alternatives to thermocouples

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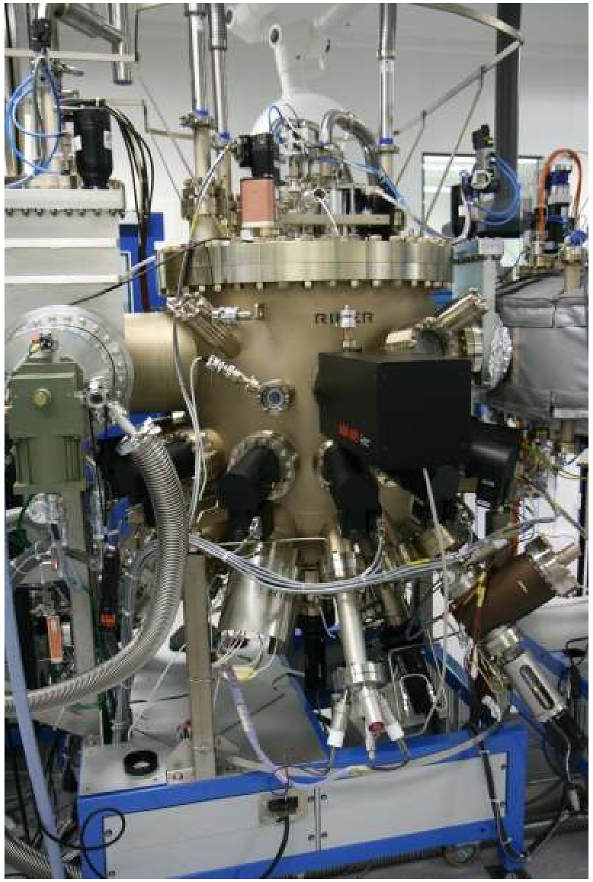
Molecular beam epitaxy with in situ measurements at LAAS

A global approach: **complementary** tools to get a clear picture of growth processes

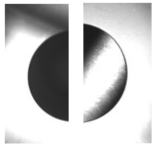


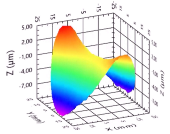
MBE412 - 4” III-V chamber

- **Wafer Temperature**
  - Thermocouples
  - kSA BandiT
  - Pyrometry
- **Spectral reflectivity**
  - White light source
  - CCD sensor
- **Atomic absorption** (OFM)
  - Original tool (Patent FR1856743)
- **RHEED**: synchronised to rotation
  - In-plane lattice parameter, streaks intensity
- **Roughness** (Diffuse Light Scattering)
- **Curvature**
  - MIC : original tool (Patent FR175461)

EpiCentre

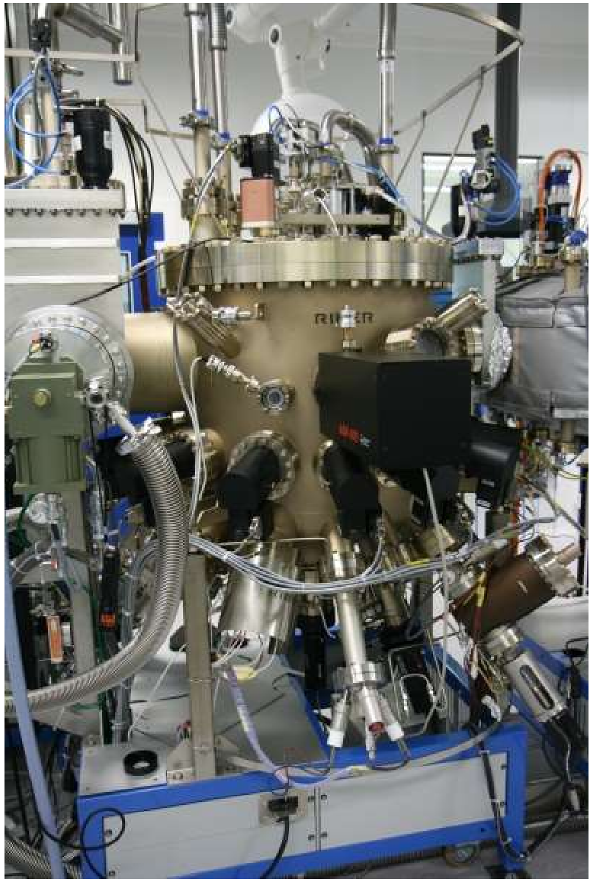
LAAS CNRS - RIBER



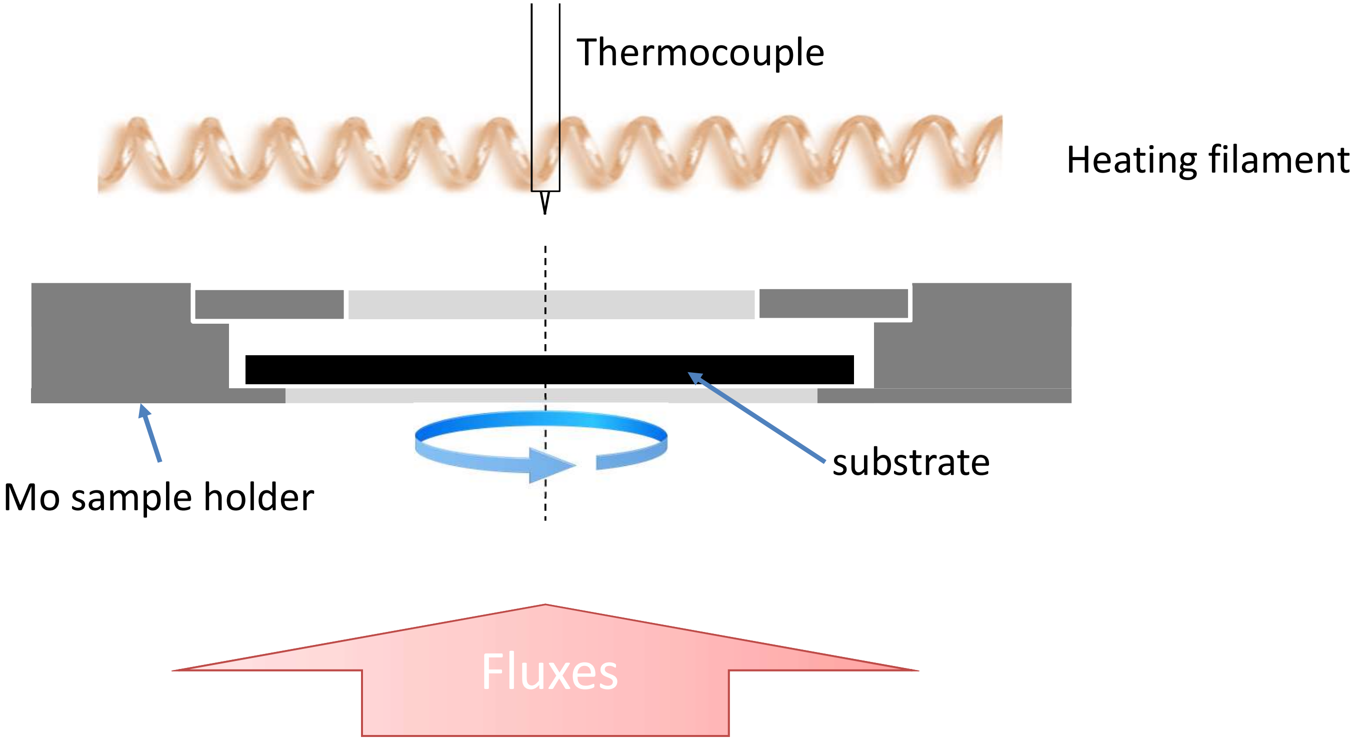


RIBER EZ-CURVE

Molecular beam epitaxy with in situ measurements at LAAS



MBE412 - 4” III-V chamber



4”, 3”, 2”, 3x2” wafers

Outline

- What about thermocouples ?
- Alternatives
  - Pyrometry
  - Emissivity Corrected Pyrometry
  - Band Edge Thermometry
  - Curvature
- Conclusions

Thermocouples

- Thermocouple = thermal measuring device consisting of two wires of different metals joined at each end

Thermocouples

- Thermocouple = thermal measuring device consisting of two wires of different metals joined at each end
  - The Seebeck effect:

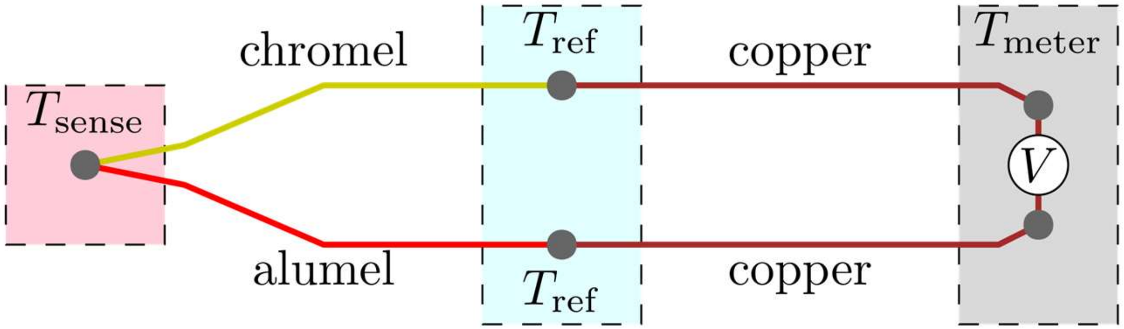
$$\Delta V = -S(T)\Delta T$$

$S(T)$ is the Seebeck coefficient:

- $S(T)$ is temperature dependent
- $S(T)$ is **material** dependent

Thermocouples

> Thermocouple = thermal measuring device consisting of two wires of different metals joined at each end

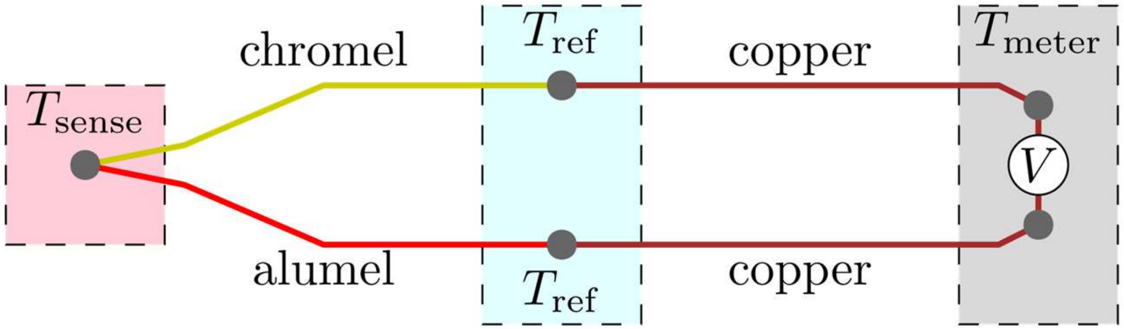


4 voltage contributions :

- T_{meter} to T_{ref}
- T_{ref} to T_{sense}
- T_{sense} to T_{ref}
- T_{ref} to T_{meter}

Thermocouples

- Thermocouple = thermal measuring device consisting of two wires of different metals joined at each end



4 voltage contributions :

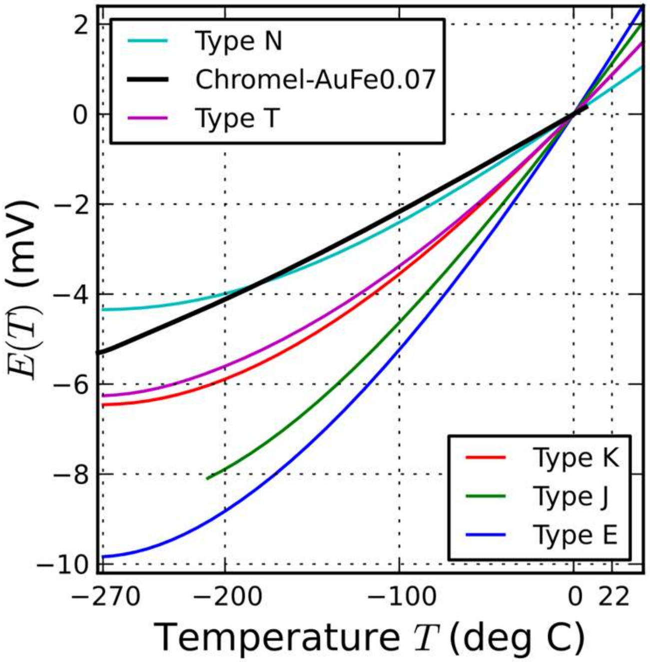
- T_{meter} to T_{ref}
- T_{ref} to T_{sense}
- T_{sense} to T_{ref}
- T_{ref} to T_{meter}

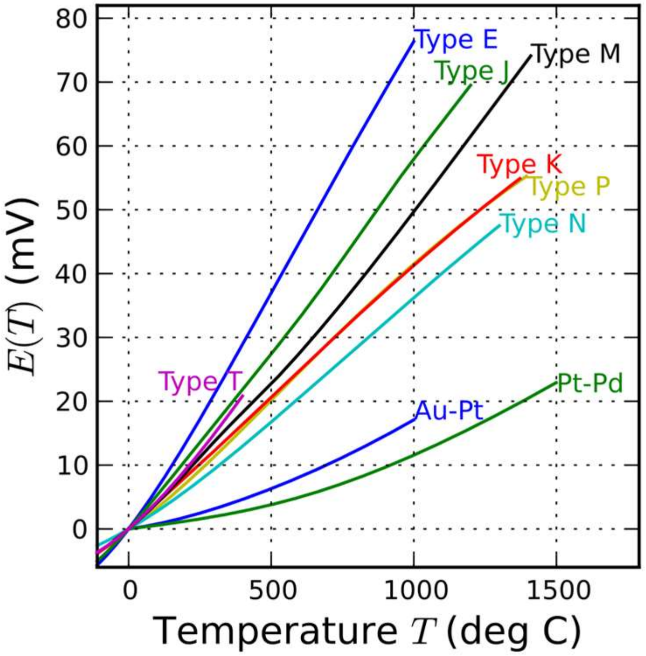
Cancel out exactly (same material and same ΔT)

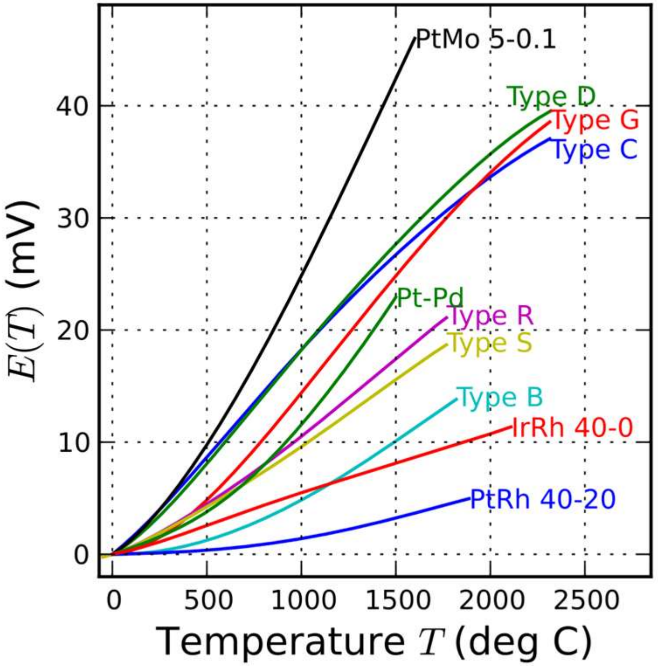
⇨ V = $E(T_{sense}) - E(T_{ref})$

Thermocouples

- Types of thermocouples



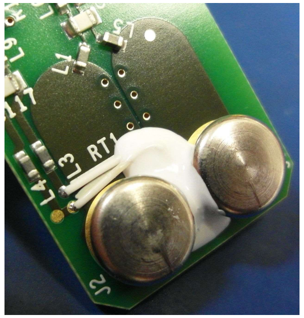




Thermocouples

- Pros
  - Easy to integrate
  - Relatively robust
  - Cost efficient

- Cons
  - Need to compensate for the reference junction temperature
    - “ice bath” method
    - Reference junction sensor (“cold junction compensation”)
  - Alloy manufacturing uncertainties
  - Aging in arch environment
  - Circuit design mistakes
  - …



Reference junction block inside a Fluke CNX 13000 temperature meter

Difficult to achieve better than 1°C accuracy
Physical contact compulsory if accuracy needed

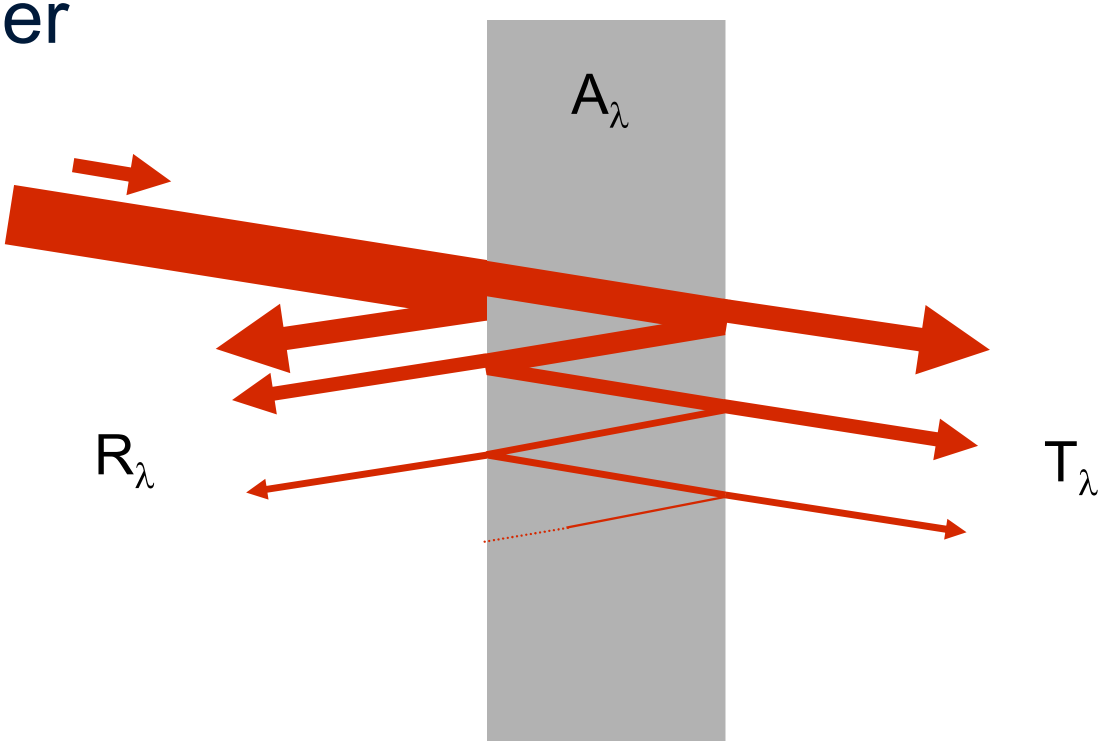
Alternatives

Pyrometry

Alternatives: pyrometry

> A quick reminder

Kirchhoff's studies on heat transfert (1859)



$$R_\lambda + T_\lambda + A_\lambda = 1$$

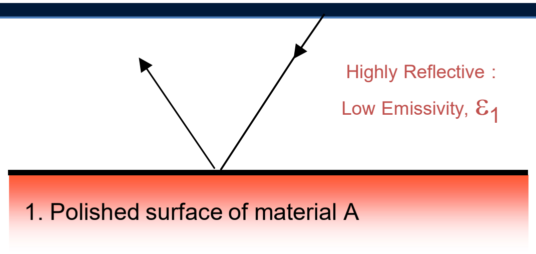
Alternatives: pyrometry

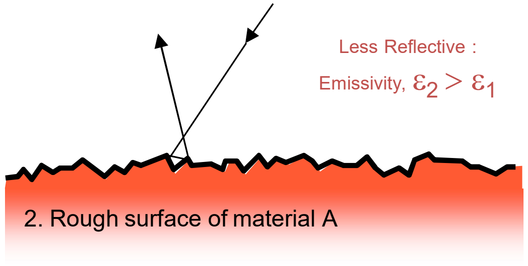
> A quick reminder

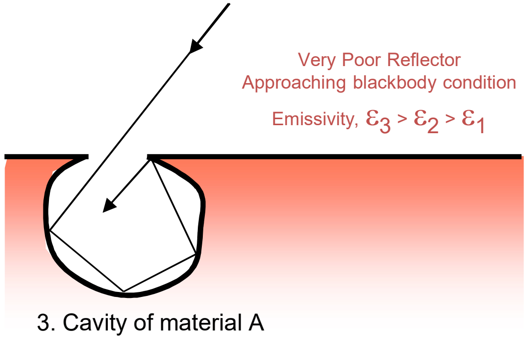
$$\varepsilon_\lambda = \text{Emissivity} = \frac{\text{Emission of real body}}{\text{Emission of black body}}$$

Kirchhoff's law : $A_\lambda = \varepsilon_\lambda$

Kirchhoff, G. (1860). "Ueber das Verhältniss zwischen dem Emissionsvermögen und dem Absorptionsvermögen der Körper für Wärme and Licht". Annalen der Physik und Chemie (Leipzig) 109: 275–301.
Translated by Guthrie, F. as Kirchhoff, G. (1860). "On the relation between the radiating and absorbing powers of different bodies for light and heat". Philosophical Magazine Series 4, volume 20: 1–21







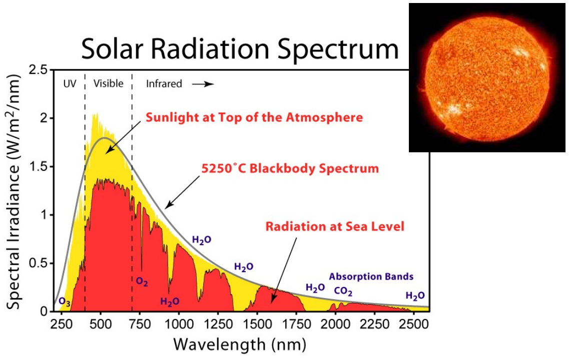
Alternatives: pyrometry

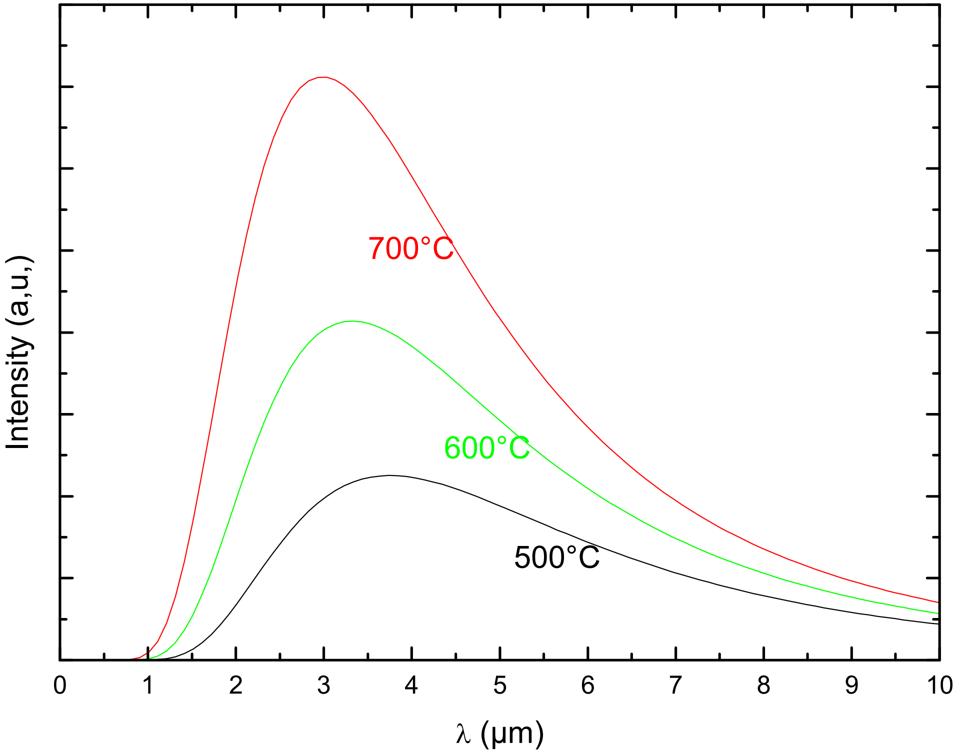
- Blackbody radiation

Planck's law (1900)

$$B_\lambda(T) = \frac{2hc^2}{\lambda^5}\frac{1}{e^{\frac{hc}{\lambda k_B T}} - 1}$$

Spectral radiance



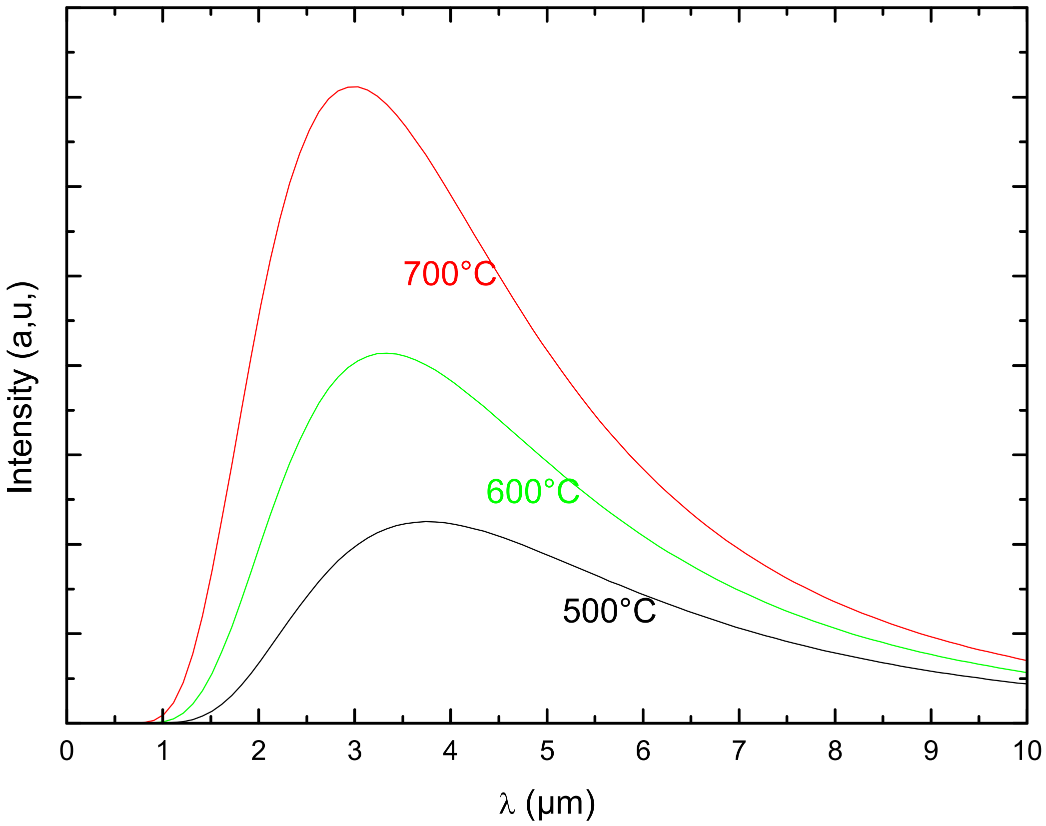


Planck, M. (1914). The Theory of Heat Radiation. Masius, M. (transl.) (2nd ed.). P. Blakiston's Son & Co.

Alternatives: pyrometry

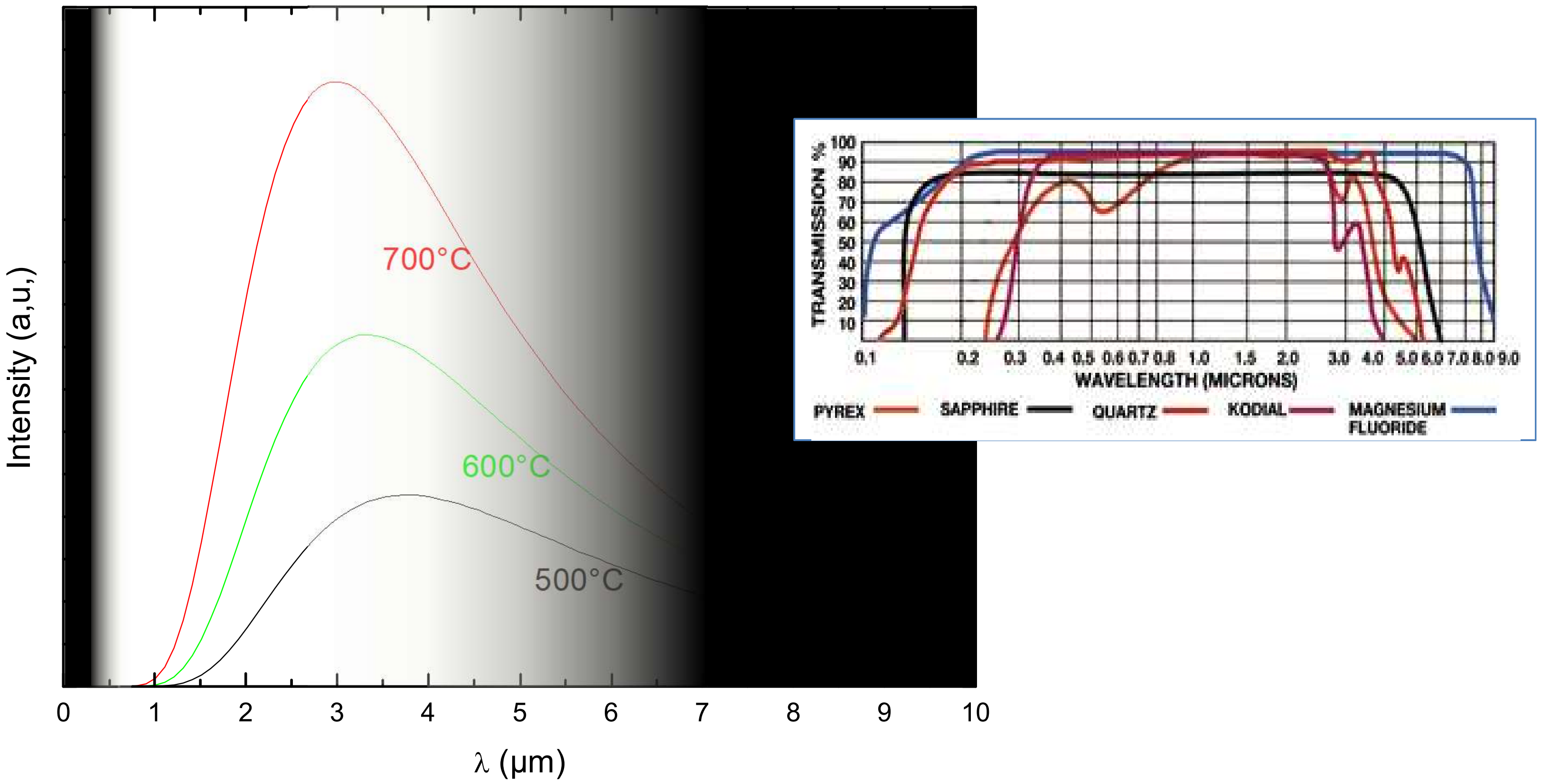
- Pyrometry principle:
  - Measure the blackbody emission at a particular wavelength
    - The temperature is **proportional** to the measured intensity

Alternatives: pyrometry



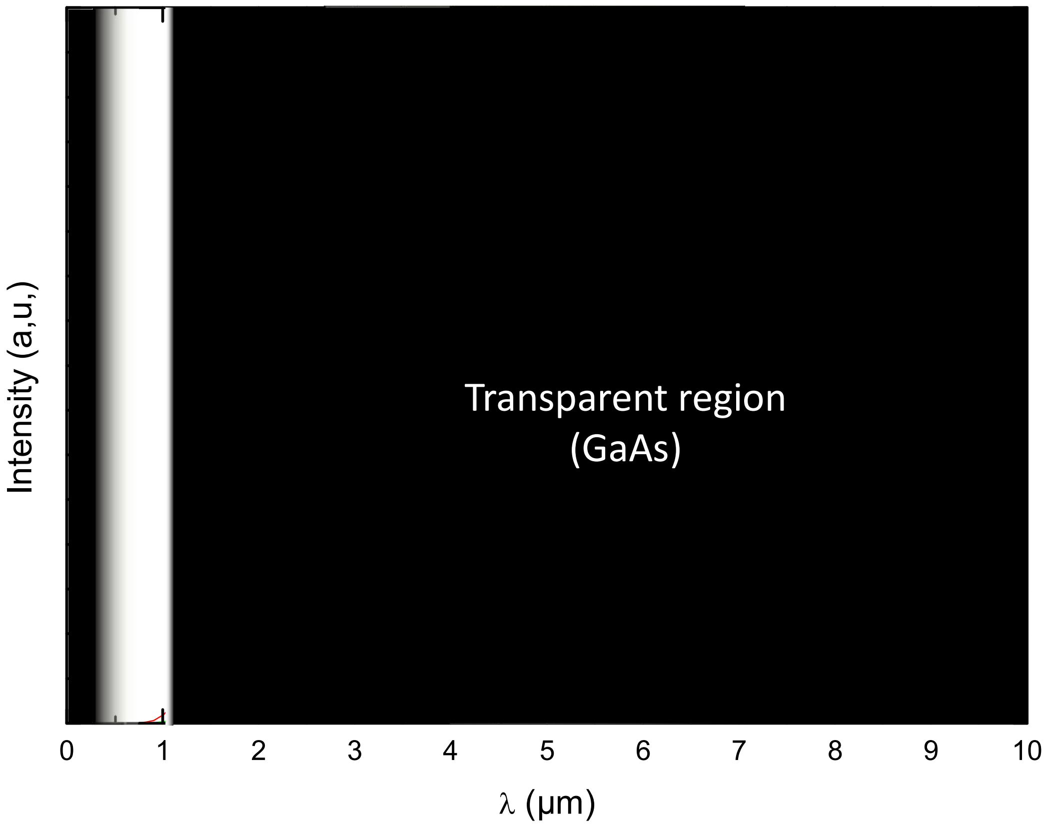
Alternatives: pyrometry

Viewport transmission



Alternatives: pyrometry

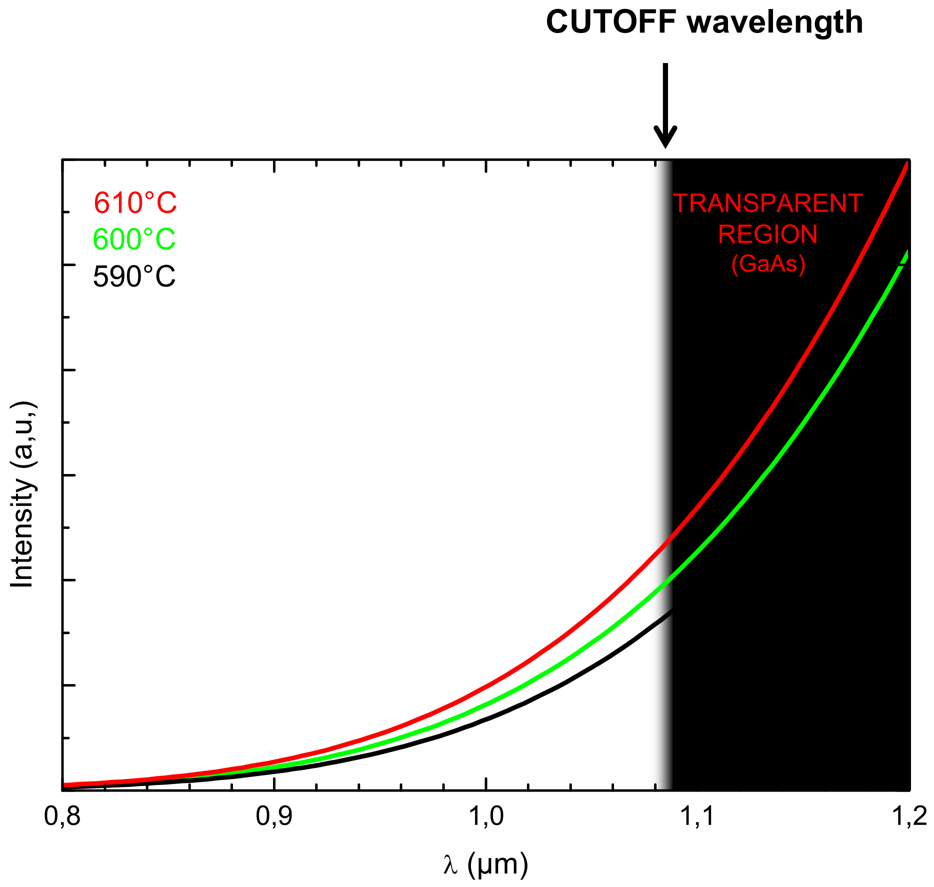
Semiconductor band gap



Alternatives: pyrometry

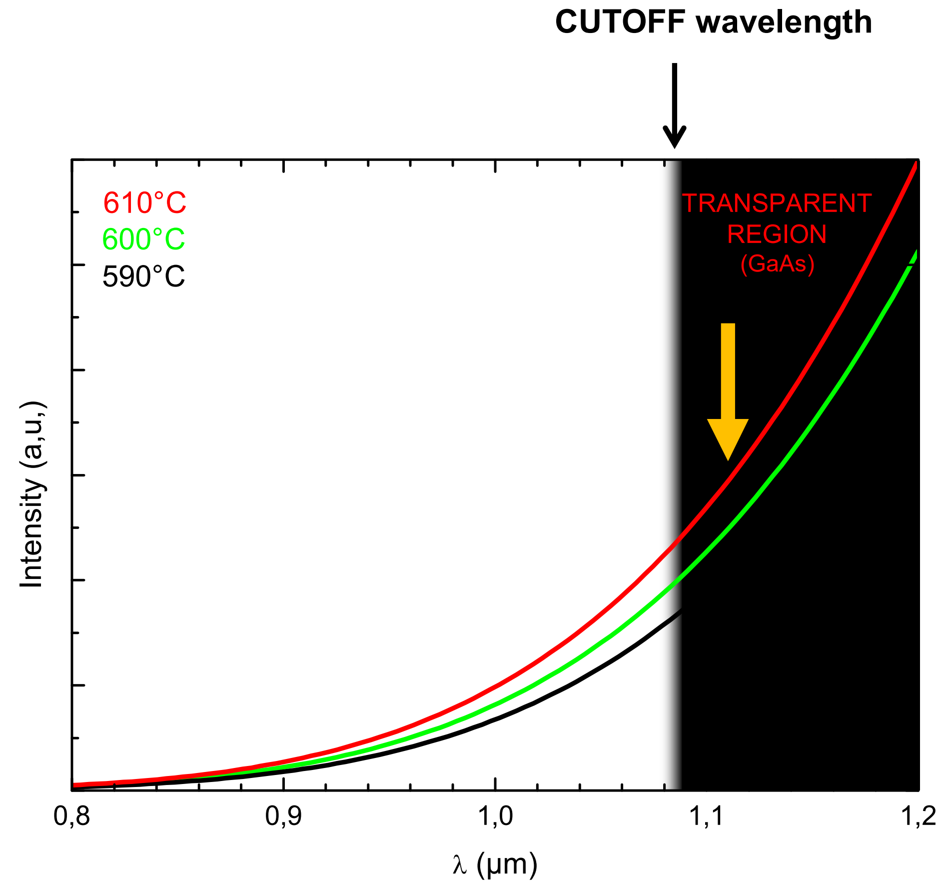
heater

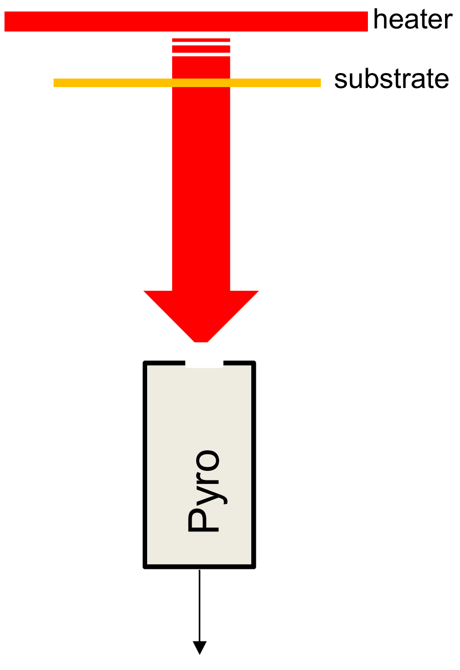
substrate



Pyrometry: record black body emission intensity at a fixed wavelength

Alternatives: pyrometry



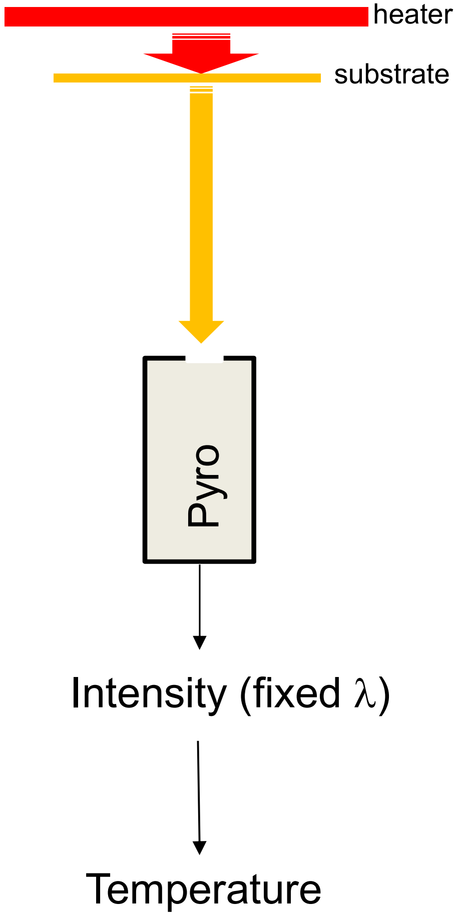


Intensity (fixed λ)

Temperature

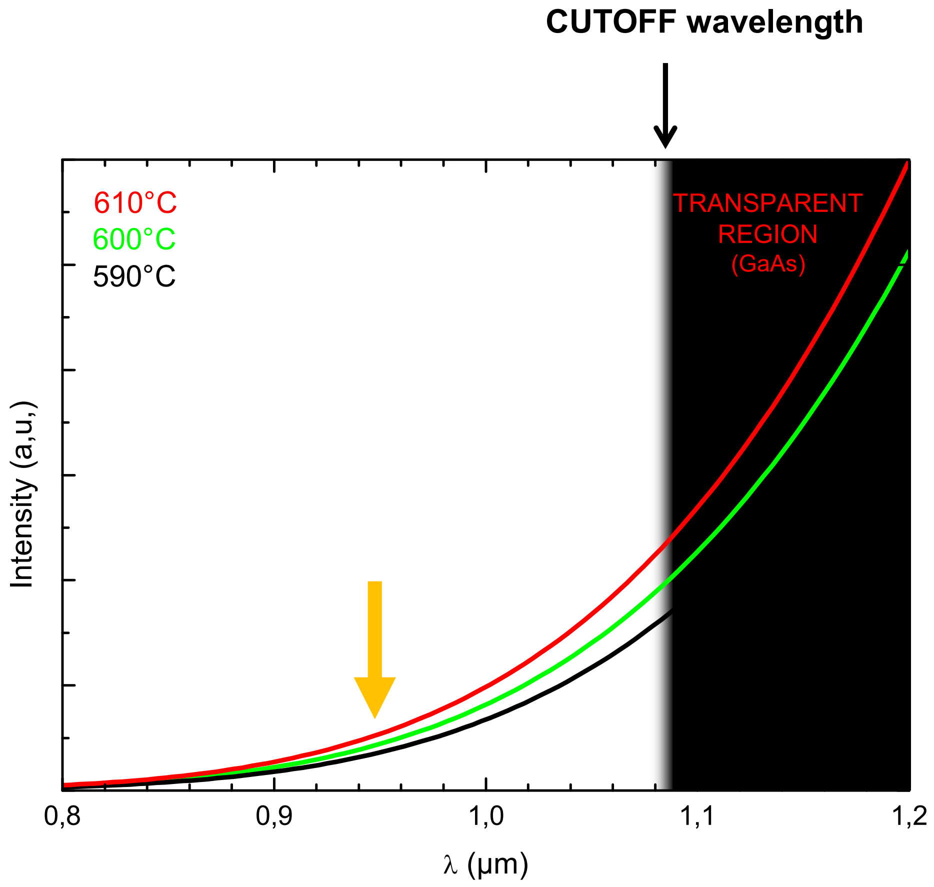
Pyrometry: record black body emission intensity at a fixed wavelength

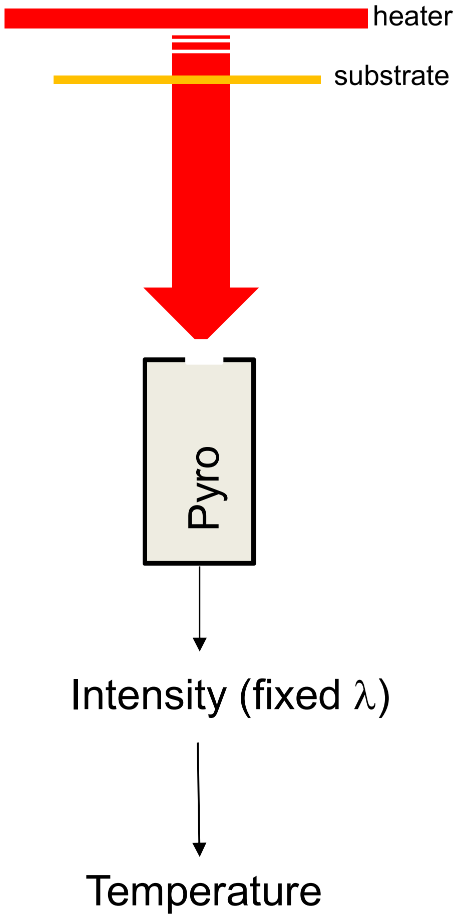
Alternatives: pyrometry



Intensity (fixed λ)

Temperature





Intensity (fixed λ)

Temperature

Pyrometry: record black body emission intensity at a fixed wavelength

Alternatives: pyrometry

Temperature T is deduced from the following equation:

$$\frac{1}{T} = \frac{1}{T_{cal}} - \frac{\lambda}{c_2} \ln\left[\frac{S}{S_{cal}} \frac{\varepsilon_{cal}}{\varepsilon}\right]$$

T = temperature
ε = emissivity

S = radiated power per area per solid angle per wavelength
S_{cal} = calibration radiated power

T_{cal} = calibration temperature
ε_{cal} = calibration emissivity

λ = wavelength
c_2 = second radiation constant

Alternatives: pyrometry

Temperature T is deduced from the following equation:

$$\frac{1}{T} = \frac{1}{T_{cal}} - \frac{\lambda}{c_2} \ln\left[\frac{S}{S_{cal}} \frac{\varepsilon_{cal}}{\varepsilon}\right]$$

What if ε changes ?

S = radiated power per area pe

S_{cal} = calibration radiated power

T = temperature

ε = emissivity

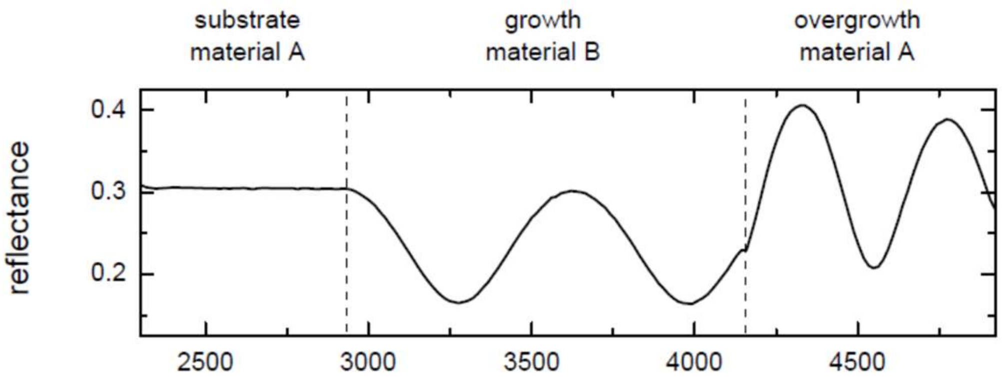
T_{cal} = calibration temperature

ε_{cal} = calibration emissivity

λ = wavelength

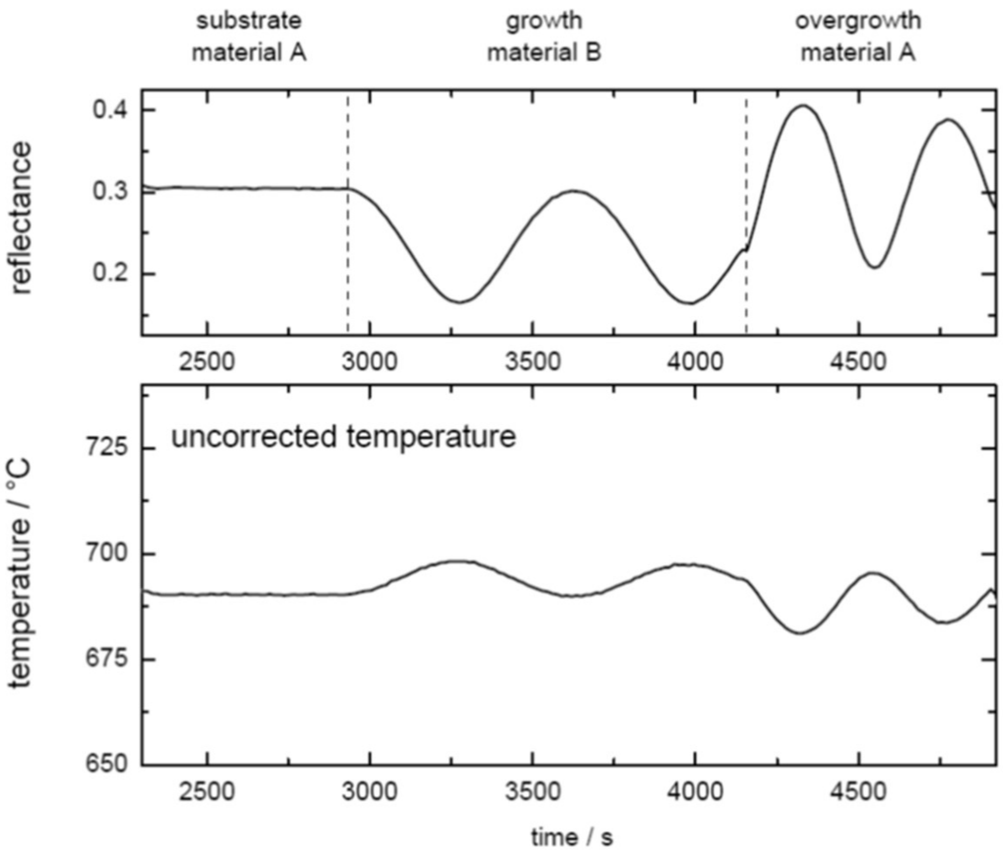
c_2 = second radiation constant

Alternatives: pyrometry

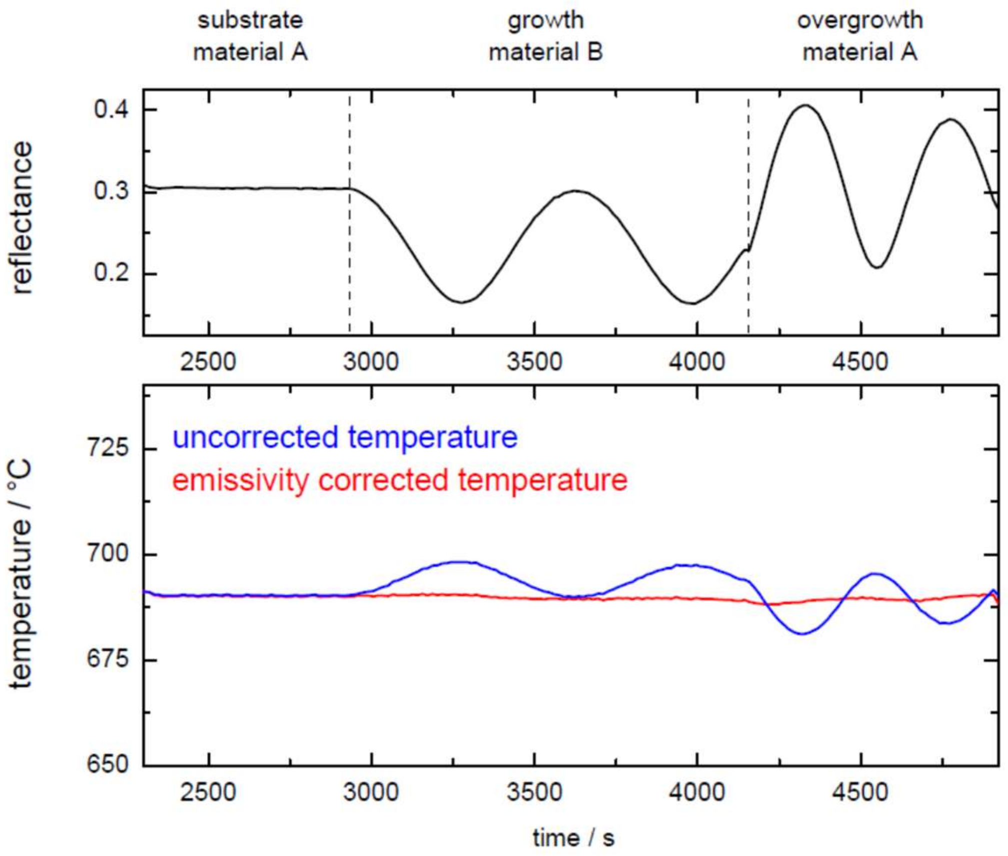


$$\varepsilon_\lambda = 1 - R_\lambda - T_\lambda$$

Alternatives: pyrometry



Alternatives: pyrometry



Alternatives: ECP (Emissivity Corrected Pyrometry)

- Emissivity Corrected Pyrometry

We know from Kirchhoff that $\varepsilon_\lambda = 1 - R_\lambda - T_\lambda$

Now, if $T_\lambda = 0$ ($\lambda < \lambda_{gap}$) i.e. no transmission

Then $\varepsilon_\lambda = 1 - R_\lambda$ →

$$\frac{1}{T} = \frac{1}{T_{cal}} - \frac{\lambda}{c_2} \ln\left[\frac{S}{S_{cal}} \frac{(1 - R_{cal})}{(1 - R)} \right]$$

Only works if

- *Transmission = 0*
- *Surface smooth and flat (light measured for R is fully specular)*
- *R independent of azimuthal angle of the sample*

Alternatives: ECP (Emissivity Corrected Pyrometry)

- Emissivity Corrected Pyrometry: some commercially available devices

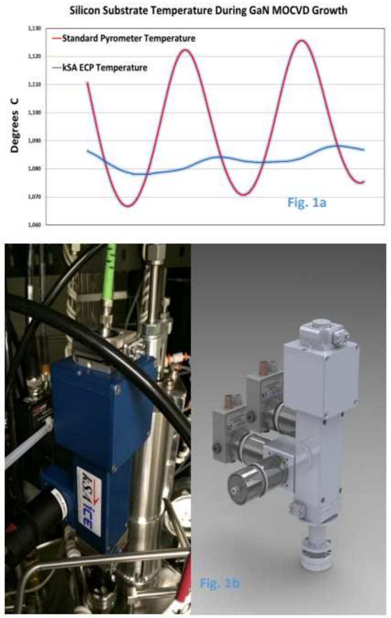


Figure 1: a) Temperature trace for GaN growth on Si comparing kSA ECP Module data (blue) and classical pyrometer data (red). b) Image of kSA ECP module integration with kSA ICE system and its installation on a D-180 MOCVD reactor.

kSA ICE ECP Module

TEMPERATURE MEASUREMENT PRODUCTS

Sekidenko MXE

High-Speed, Non-Contact Optical Temperature Pyrometer with Integrated Reflectance Measurement and Emissivity Compensation

Advanced Energy's Sekidenko MXE measures at ultra-high-speed sampling rates (up to 10 kHz) and offers combined temperature and active reflectance for precise control in dynamic processes.

- Precise temperature measurement enabling closed-loop process control
- Configurable wavelength based on material type and required temperature range
- Both analog and digital communications

Alternatives: Pyrometry and ECP

- **Pyrometry** is a non contact technique able to measure a temperature, but
  - The temperature is RELATIVE to a calibration temperature
  - The measured temperature depends greatly on the optical path (coatings of viewports)
  - Optical access must be available
  - The measured temperature depends on the emissivity of the surface, which changes during thin films growths

- **Emissivity Corrected Pyrometry**
  - Less sensitive to emissivity changes
  - High geometrical requirements

+ no signal below ~350-400°C

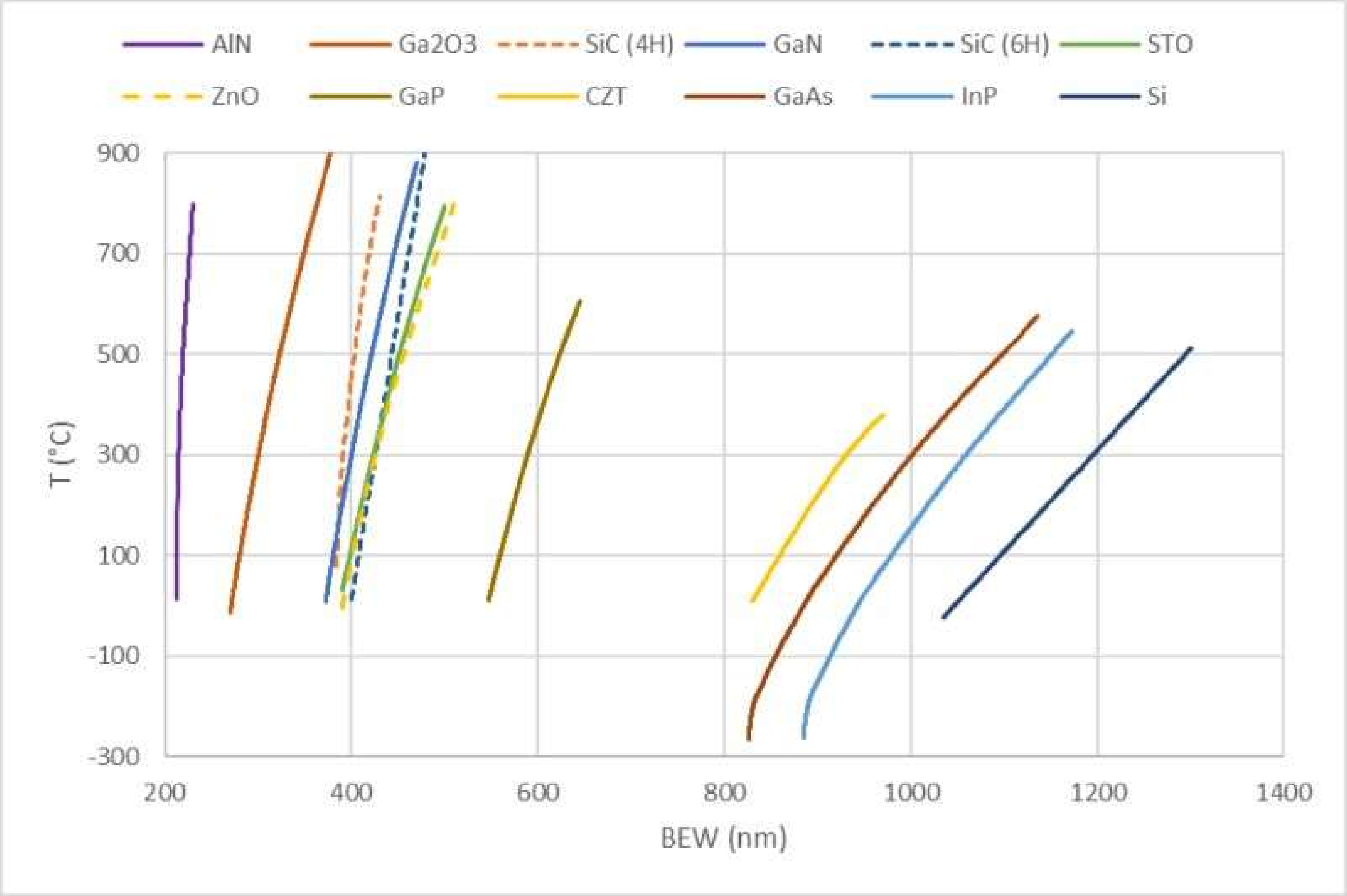
Alternatives

Band Edge Thermometry

Alternatives: Band Edge Thermometry

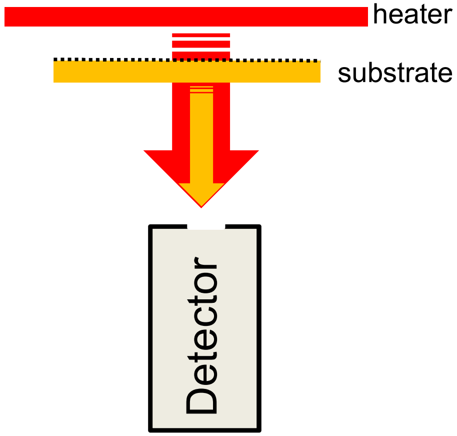
- Band Edge Thermometry principle:
  - Measure the bangap of a semiconductor
    - The temperature vs bangap table gives the temperature

Alternatives: Band Edge Thermometry

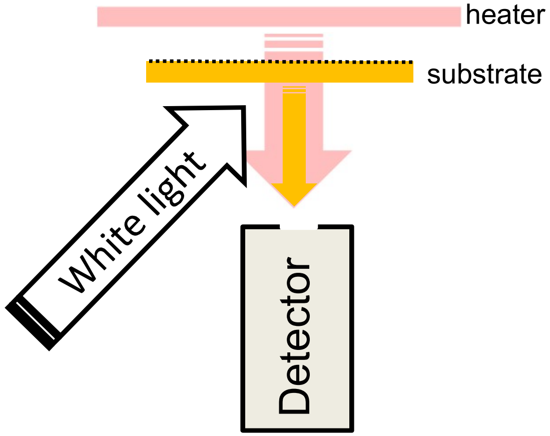


Alternatives: Band Edge Thermometry

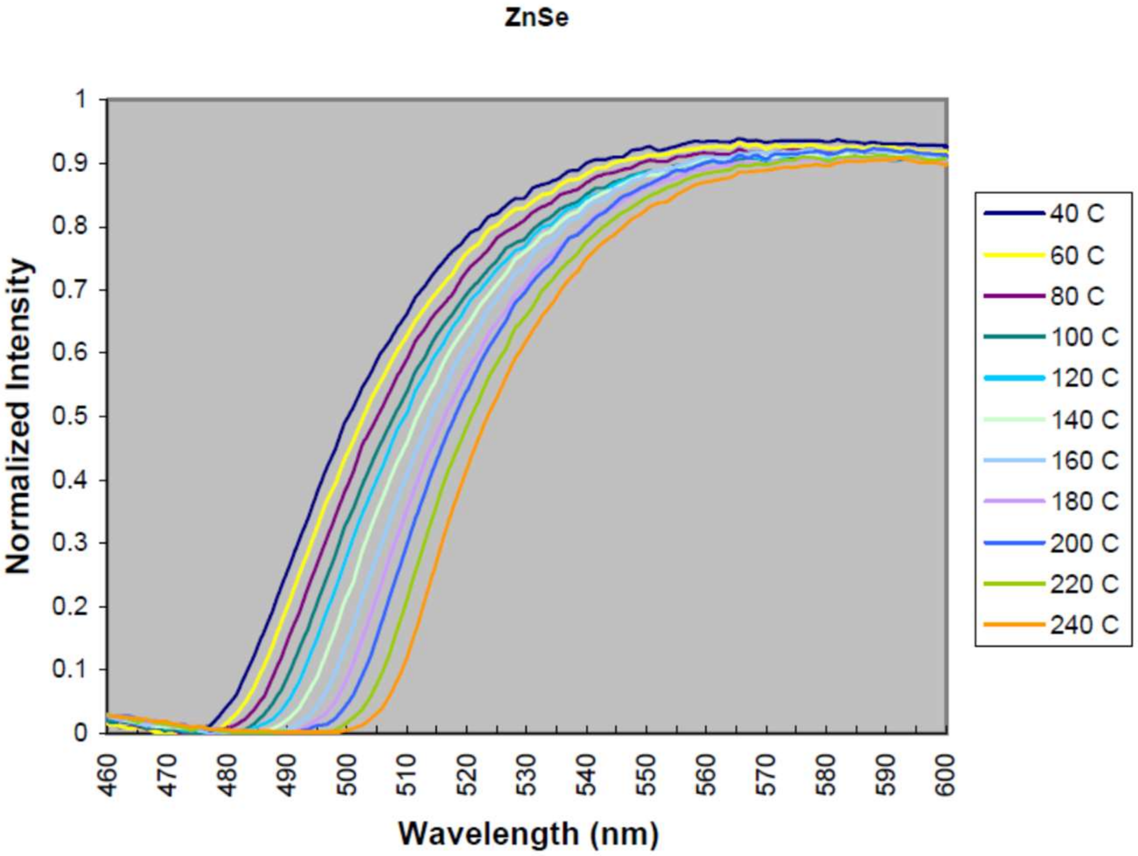
High temperature
transmission mode



Low temperature
Transmission – backside diffusion - transmission mode



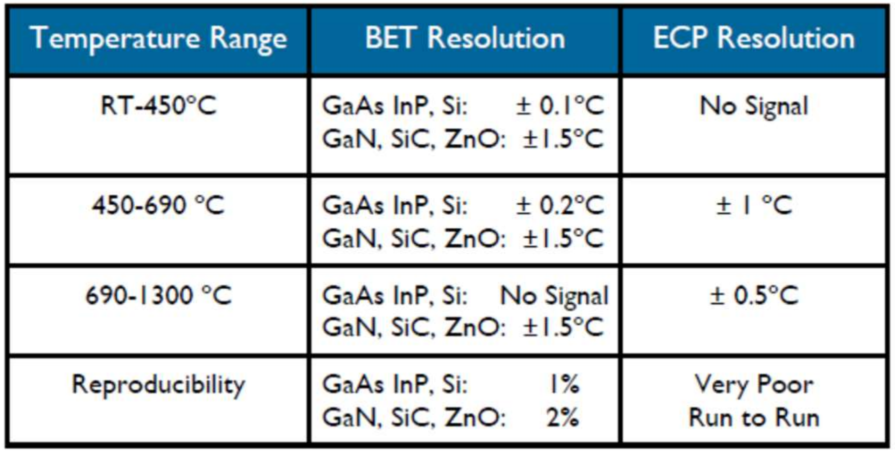
Alternatives: Band Edge Thermometry



\+ The measurement does not depend on the intensity of a signal
This makes it very robust/reproducible

Alternatives: Band Edge Thermometry

- Band Edge Thermometry depends on calibration files but
  - The measured temperature is **absolute** as it measures an intrinsic parameter (bandgap)
    - It does not depend on viewport coating
    - Whatever the position of the optical head, as soon as it “sees” the wafer, the measured temperature is the same
    - It is possible to **exchange** temperature data between systems !



Temperature Range	BET Resolution	ECP Resolution
RT-450°C	GaAs InP, Si: ± 0.1°C GaN, SiC, ZnO: ±1.5°C	No Signal
450-690 °C	GaAs InP, Si: ± 0.2°C GaN, SiC, ZnO: ±1.5°C	± 1 °C
690-1300 °C	GaAs InP, Si: No Signal GaN, SiC, ZnO: ±1.5°C	± 0.5°C
Reproducibility	GaAs InP, Si: 1% GaN, SiC, ZnO: 2%	Very Poor Run to Run

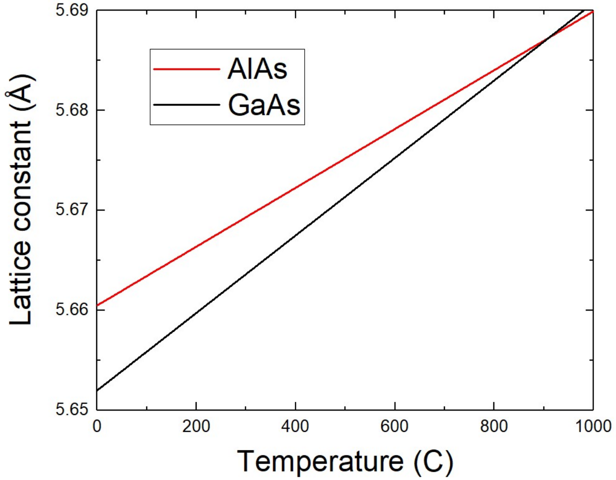
Alternatives

Curvature

Alternatives: Curvature

Thermal stress

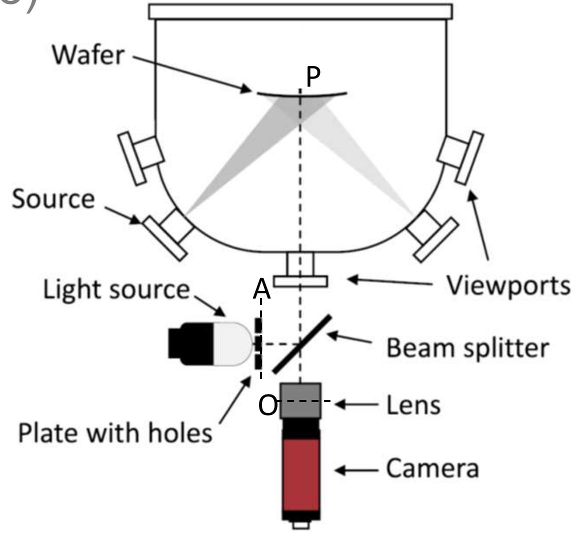
- Because thermal expansion coefficient is material dependent, any change in temperature induces a change in stress/curvature of an heteroepitaxial stack.



*M. Ettenberg, R. J. Paff, J. Appl. Phys., **41**, no.10, pp.3926-3927 (1970)*

Alternatives: Curvature

MIC (Magnification Inferred Curvature)



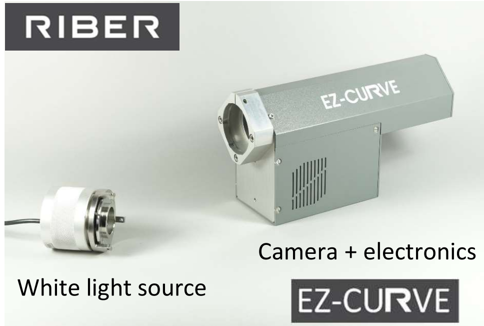
Spherical mirror:

$$\gamma = \frac{\overline{A'B'}}{\overline{AB}} = \frac{1}{1-2\frac{\overline{AP}}{\overline{R}}} \Rightarrow \overline{\kappa_\perp} = \frac{1}{2\overline{AP}}\frac{\gamma-1}{\gamma}$$

γ_c (magnification seen by the camera) ➡

$$\boxed{\overline{\kappa_\perp} = \frac{1}{2\overline{AP}}\frac{\gamma_C-1}{\gamma_C}\times\frac{\overline{AP}+\overline{OP}}{\overline{OP}}}$$

Magnification ➡ Curvature



Arnoult, A., Colin, J. Magnification inferred curvature for real-time curvature monitoring. *Sci Rep* **11,** 9393, 2021
https://doi.org/10.1038/s41598-021-88722-6

Alternatives: Curvature

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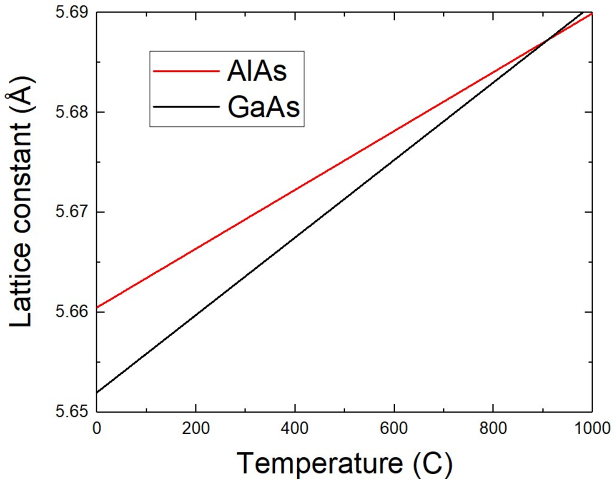

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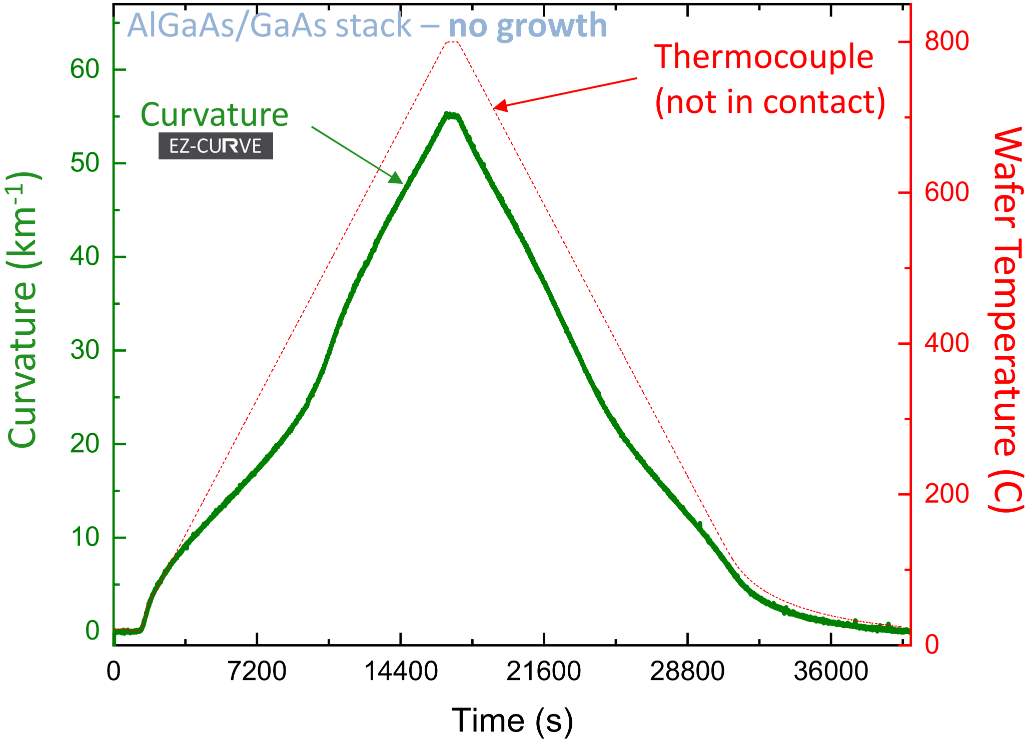
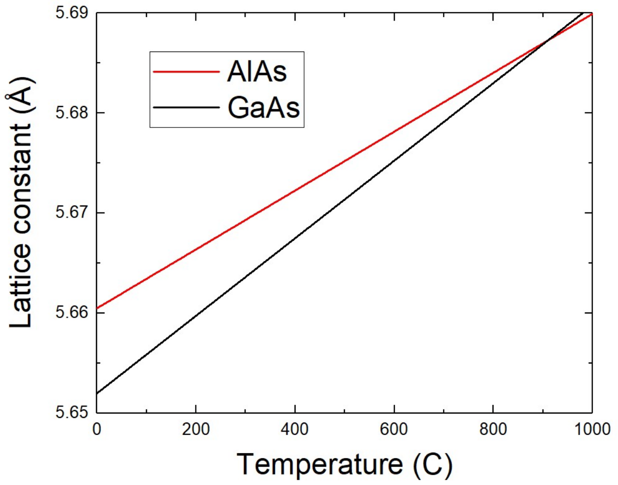


Alternatives: Curvature

Thermal stress



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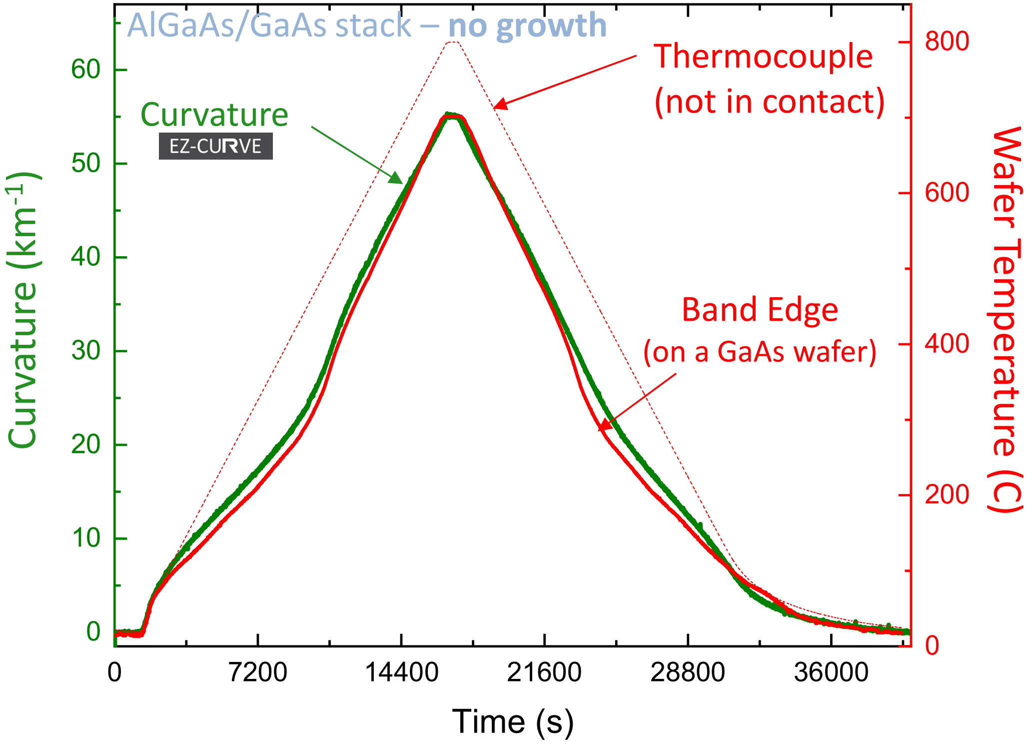


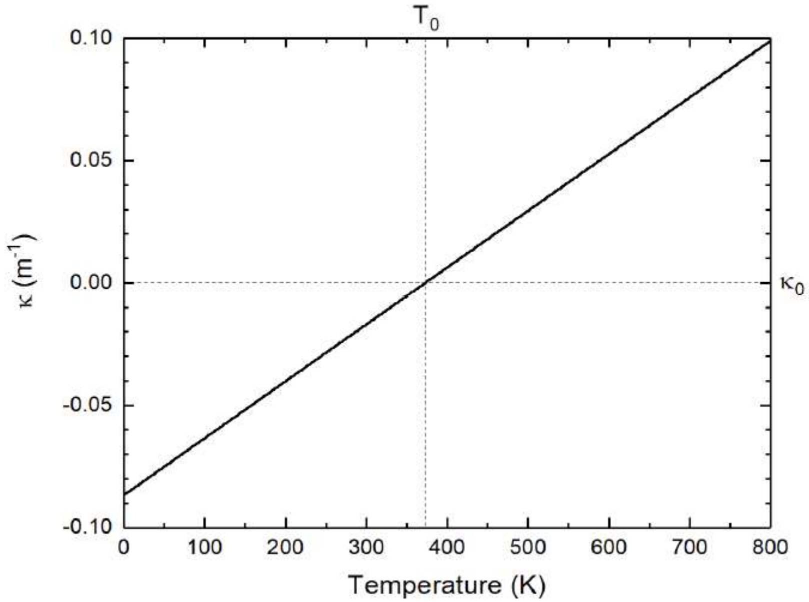
➡ Nice correlation between temperature and curvature

Alternatives: Curvature

Thermal stress

Temperature T is deduced from the following equation:

$$\boldsymbol{T = T_0 + (\kappa - \kappa_0)\frac{1}{\alpha_f(\kappa_0 - A) - \alpha_s(\kappa - A)}}$$



With $A = \frac{6M_f h_f}{M_s h_s^2}\left(\frac{1 + \frac{h_f}{h_s}}{1 + \frac{h_f M_f}{h_s M_s}\left(4 + 6\frac{h_f}{h_s} + 4\left(\frac{h_f}{h_s}\right)^2\right) + \left(\frac{h_f}{h_s}\right)^4\left(\frac{M_f}{M_s}\right)^2}\right)$

And T_0 and κ_0 the reference temperature and curvature

Alternatives

Conclusions

Alternatives

Pyrometry
- High resolution
- Simple measurement set-up
- Insensitive against missalignement, etc…
- Low geometrical requirements
- Accurate temperature measurement during films growth impossible (due to changes in d, T, n, k, etc…)

Emissivity Corrected Pyrometry
- Very high resolution
- Complete compensation of all emissivity changes (d, T, n, k, etc…)
- Sensitive towards misalignment, vibration, angle, distorsion, rotation, etc…
- High geometrical requirements

Band Edge Thermometry
- High resolution
- Insensitive against missalignement, etc…
- bad resolution for indirect gap semiconductors (e.g. Si, Ge)
- Temperature control during Bragg reflector growth problematic

Magnification Inferred Curvature
- High resolution
- Simple measurement set-up
- Insensitive against missalignement, etc…
- Low geometrical requirements
- Need to know thermal expansion coefficients and relative films thicknesses

Alternatives

Temperature range

Pyrometry
- High resolution
- Simple measurement set-up
- Insensitive against missalignement, etc…
- Low geometrical requirements
- Accurate temperature measurement during films growth impossible (due to changes in d, T, n, k, etc…)

T > 350°C

Emissivity Corrected Pyrometry
- Very high resolution
- Complete compensation of all emissivity changes (d, T, n, k, etc…)
- Sensitive towards misalignment, vibration, angle, distorsion, rotation, etc…
- High geometrical requirements

T > 350°C

Band Edge Thermometry
- High resolution
- Insensitive against missalignement, etc…
- bad resolution for indirect gap semiconductors (e.g. Si, Ge)
- Temperature control during Bragg reflector growth problematic

T < 700°C

Magnification Inferred Curvature
- High resolution
- Simple measurement set-up
- Insensitive against missalignement, etc…
- Low geometrical requirements
- Need to know thermal expansion coefficients and relative films thicknesses

Depends on materials
(as long as they are stable)

Temperature measurement in vacuum

Alternatives: pyrometry

> A quick reminder: emissivity

$$R_\lambda + T_\lambda + A_\lambda = 1$$

$$A_\lambda = \varepsilon_\lambda \Rightarrow \boxed{\varepsilon_\lambda = 1 - R_\lambda - T_\lambda}$$