

Metastable solid helium-4 below its melting pressure

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Le 17 Janvier 2013





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A potential supersolid

A metastable crystal relative to melting

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2. Cryogenic techniques
3. Interferometric measurement of density variations
4. Metastable solid helium-4 and stability limits
6. Conclusion and prospects

Quantum properties of solid helium 4



► Helium has the **most quantum** condensed phase !

$$\sqrt{\langle u^2 \rangle} \sim a/4 \quad \text{for solid helium 4}$$

$$\sqrt{\langle u^2 \rangle} \sim a/10 \quad \text{for classical solids}$$

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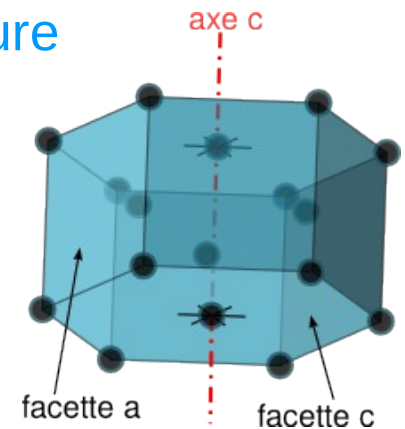
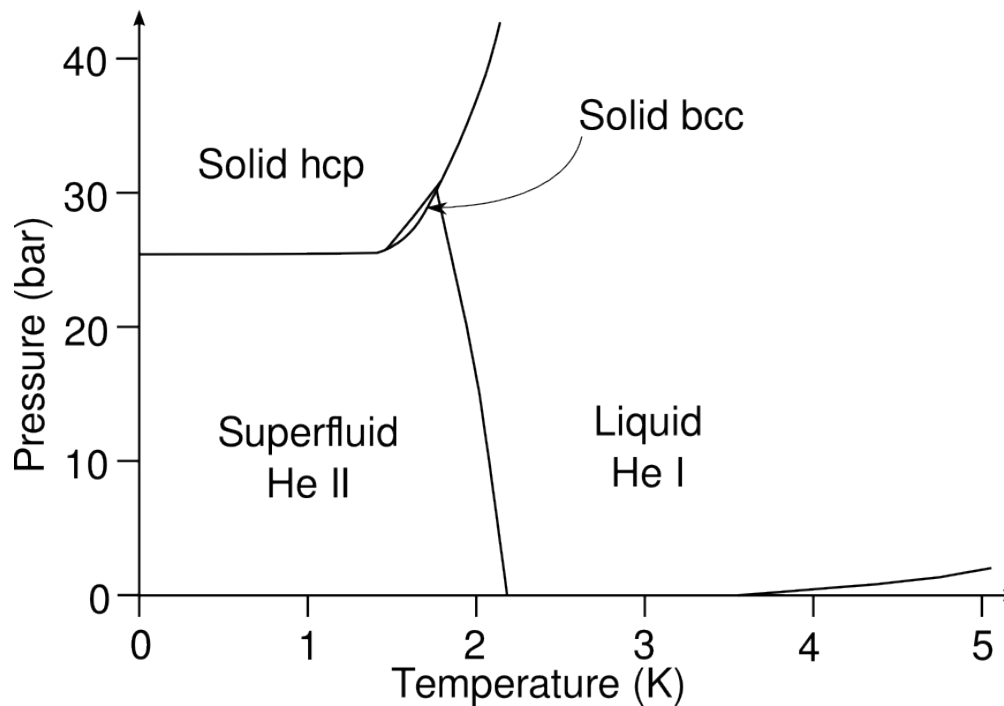
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Crystallize above ~25 bar
hcp structure



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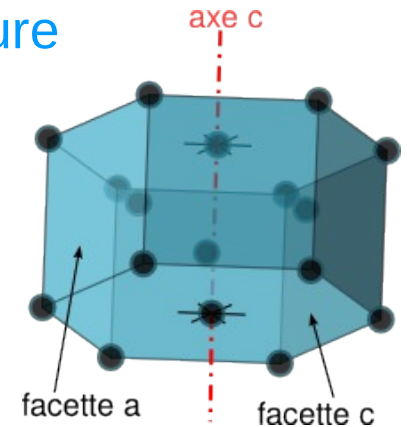
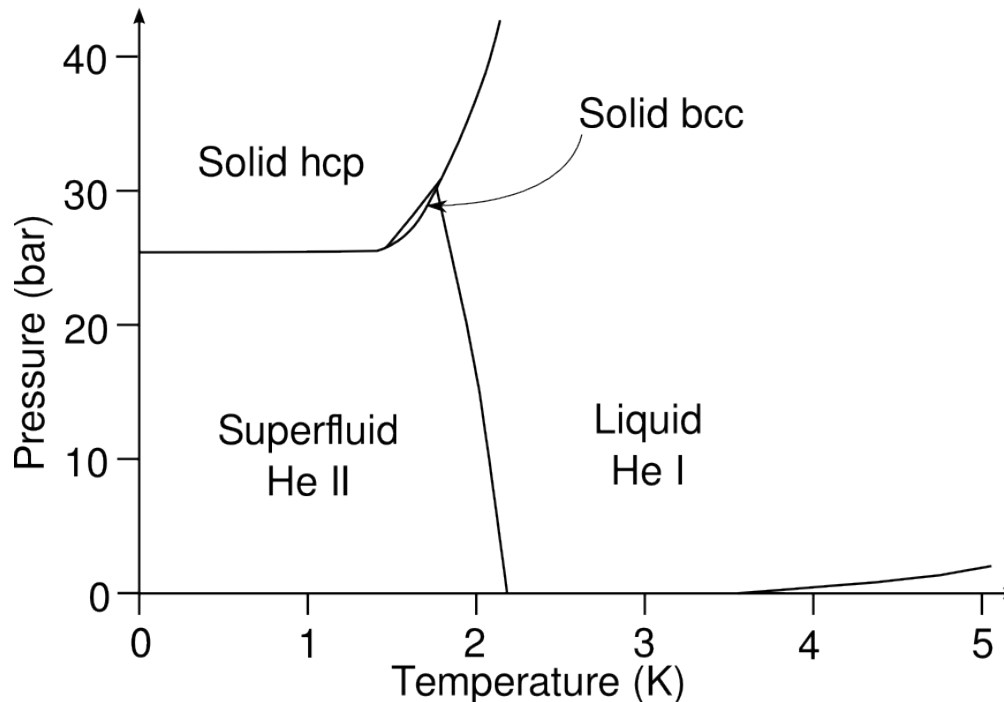
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► These quantum properties make helium a good candidate for the observation of **supersolidity**

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How to combine crystalline and superfluid properties?

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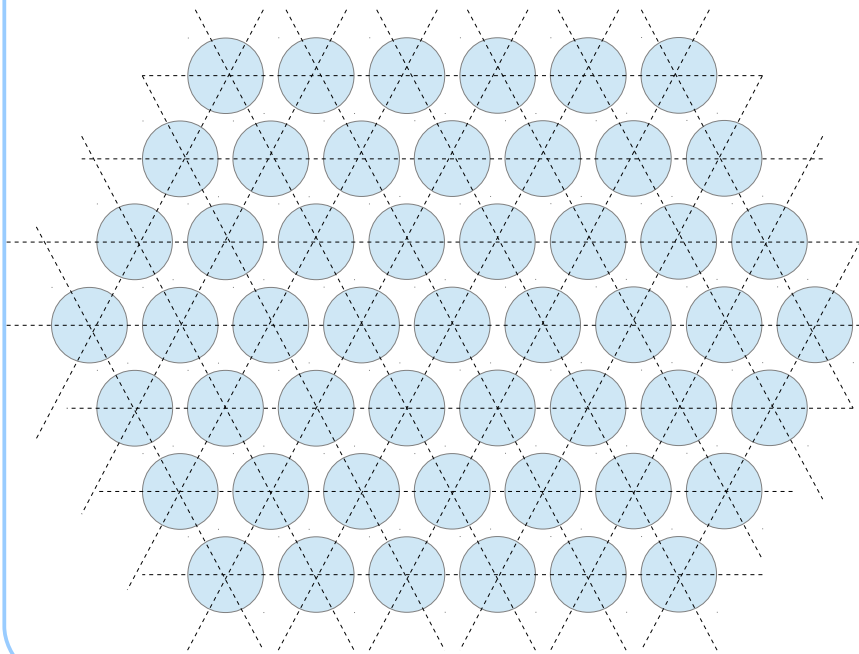
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How to combine crystalline and superfluid properties?

Crystalline properties



► Organized structure of a crystal

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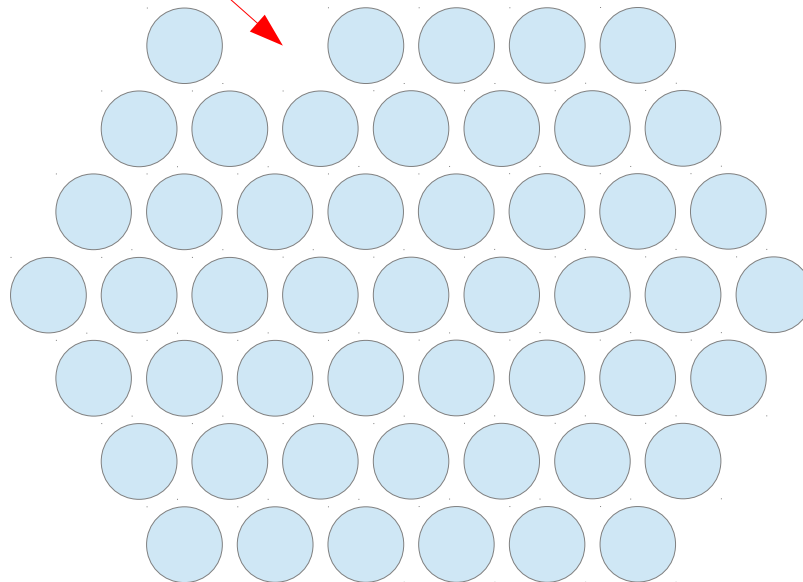
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How to combine crystalline and superfluid properties?

Superfluid properties

vacancy



► Vacancies are missing atoms in the lattice

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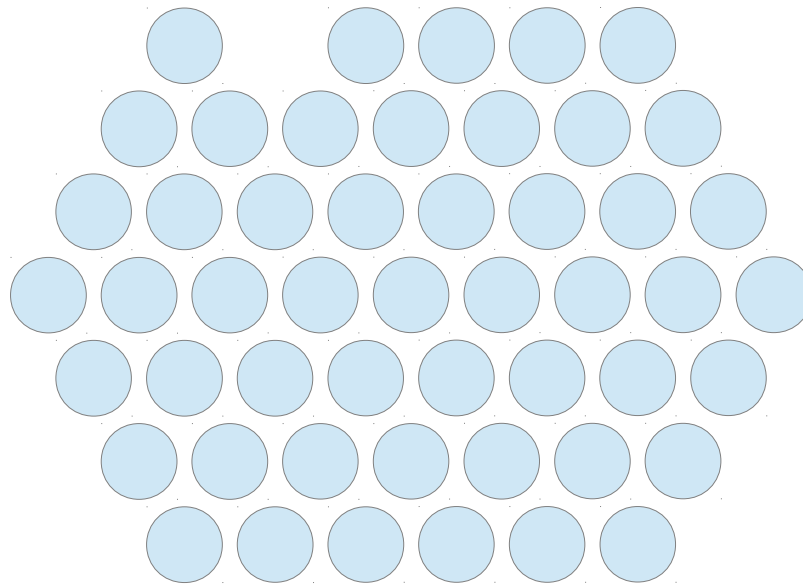
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► Vacancies can **delocalize** inside the crystal

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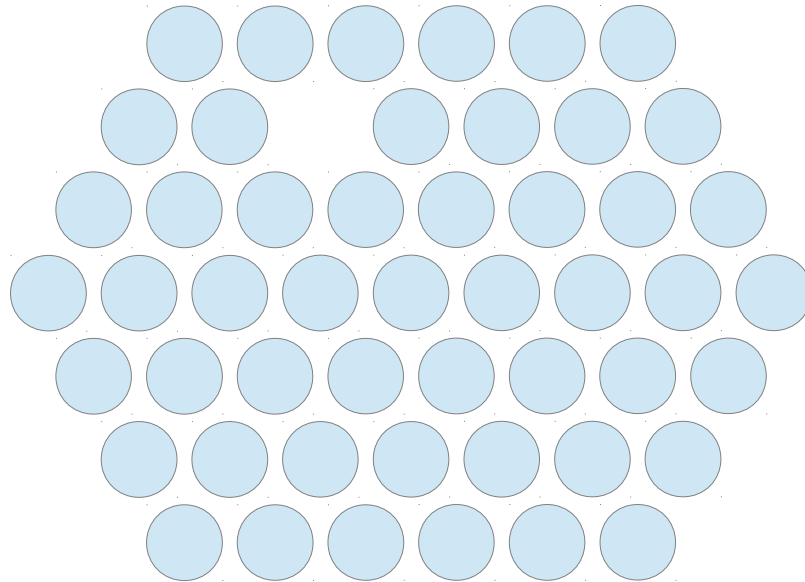
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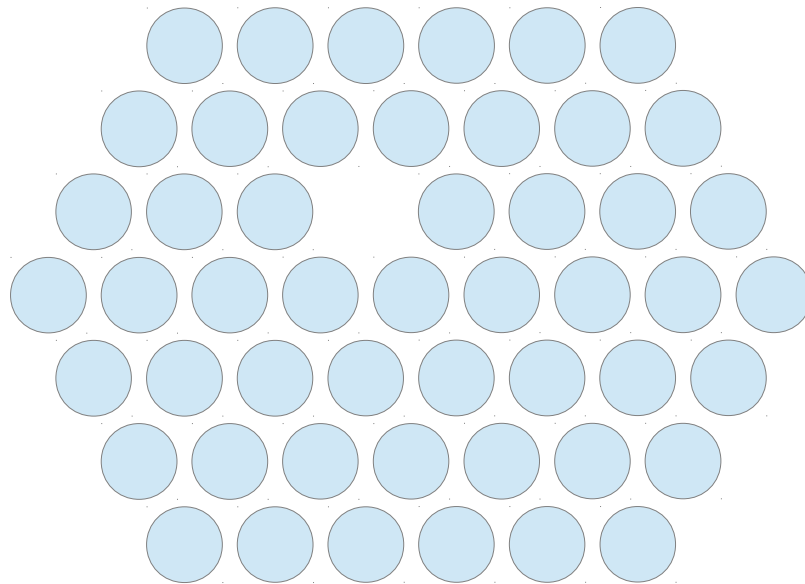
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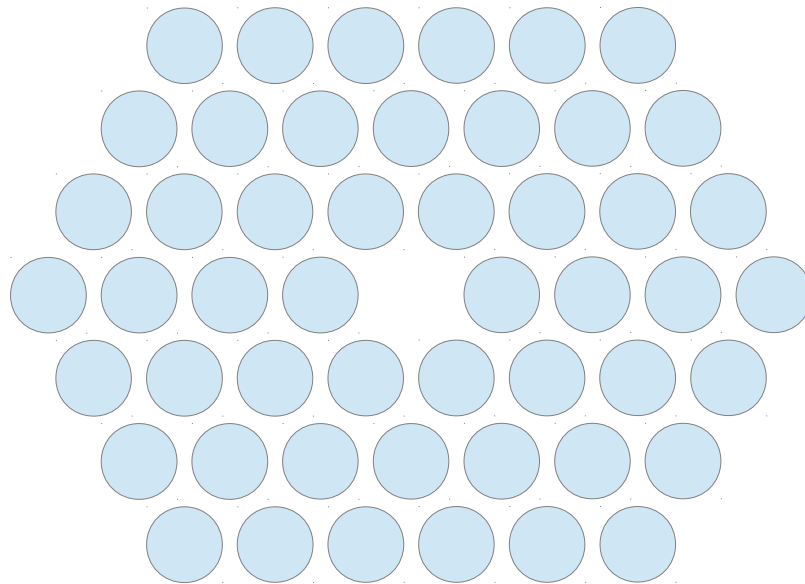
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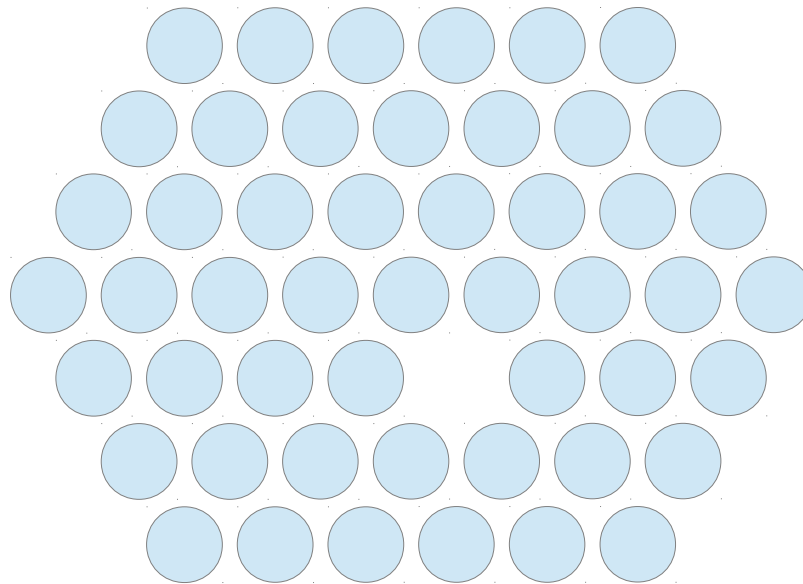
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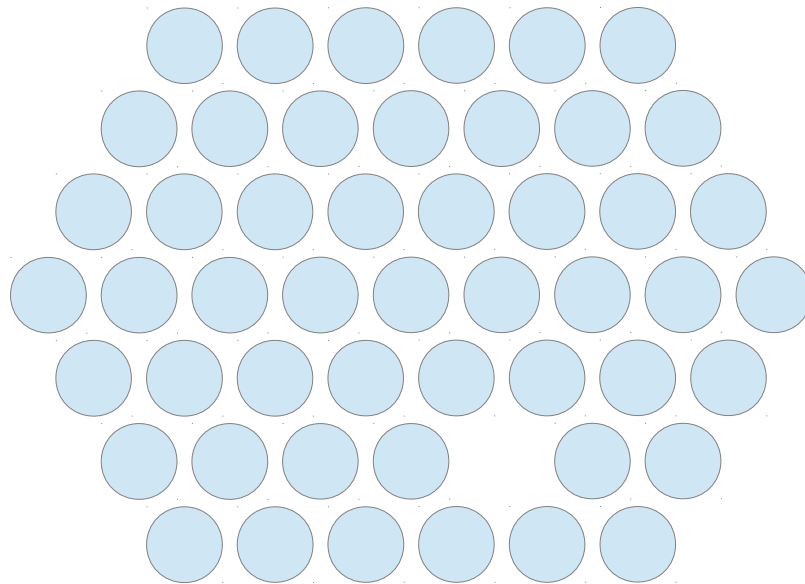
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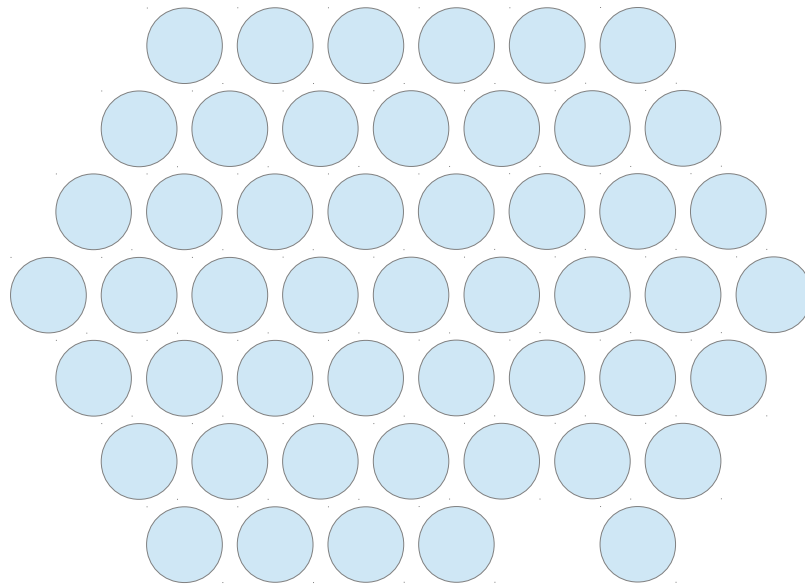
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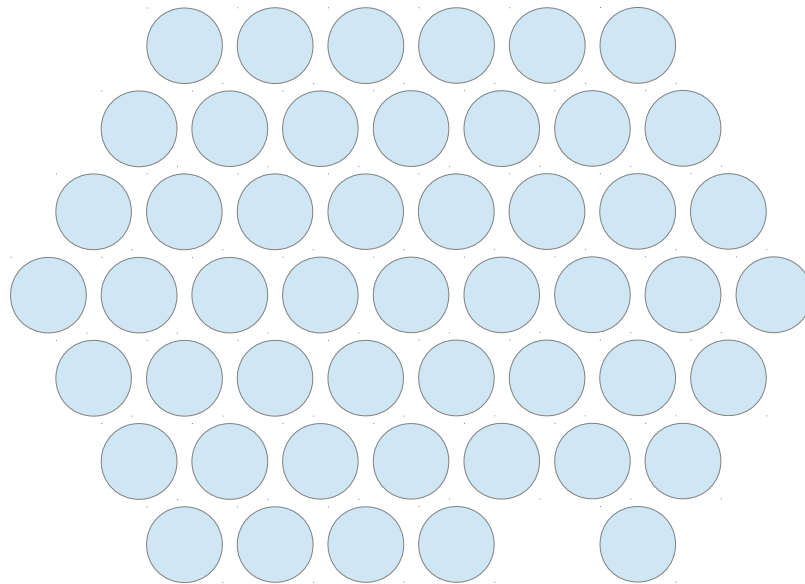
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► Vacancies can **delocalize** inside the crystal

Possibly form Bose condensate
→ **superfluidity of vacancies**



superfluidity of solid

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First experiments

-objects immersed in solid helium-4

-mass flow experiments

-torsionnal oscillators

► No signature of supersolidity [13]

Recent experiments

In 2004 :

Kim and Chan rotation anomaly [14]

In 2007 :

Day and Beamish anomalous stiffening

► Interplay still debated but mechanisms are far from ALC scenario



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Why ?



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Why ? → Vacancy activation energy E_v is too large

Vacancies in solid helium-4



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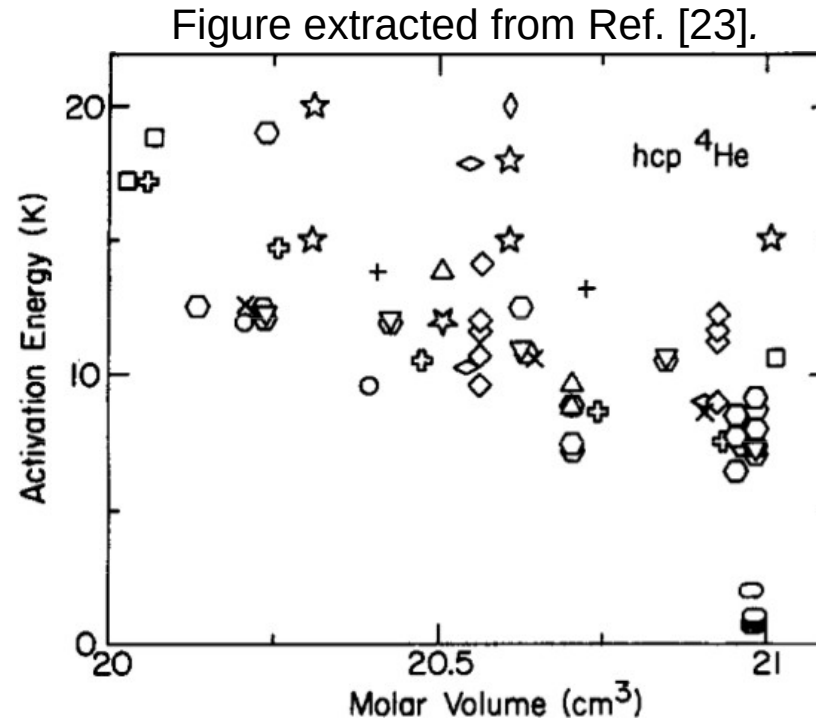
Metastable solid helium-4 and stability limits

Conclusion and prospects

- ▶ Experiments and numerical simulations show [23] that :

$$7\text{K} < E_v < 15\text{K} \quad \text{compared to } T=1\text{K}$$

- ▶ E_v decreases when V_m increases



Vacancies in solid helium-4



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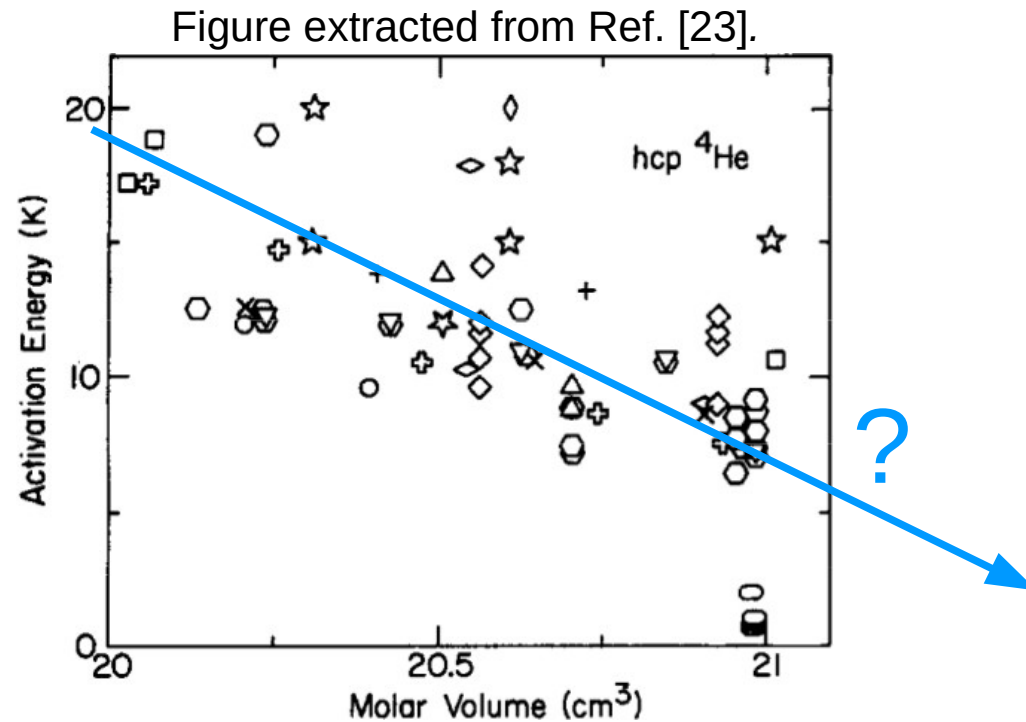
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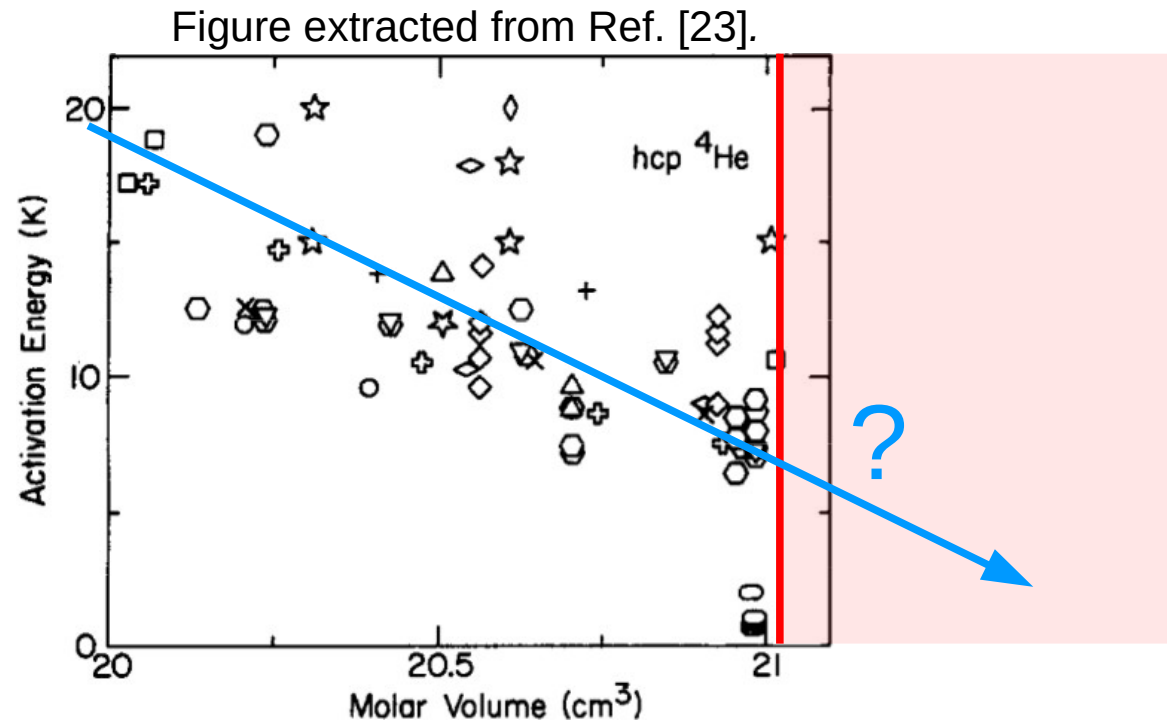
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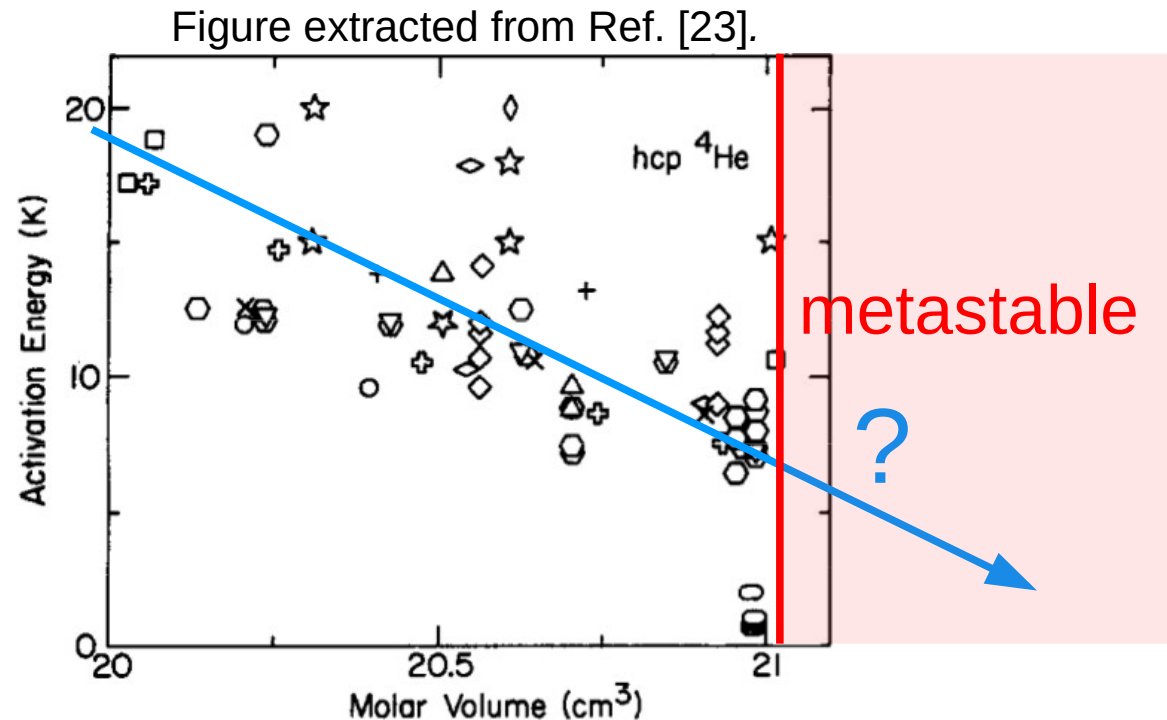
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Could $E_v \simeq 0$ be achieved by stretching the solid up to $V_m = 22 \text{ cm}^3$?

A way to realize ALC scenario?



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Nucleation model of the liquid phase [59]:

► Unstable when nucleation rate too important :

-Happens close to the spinodal point $P_{nucl} \simeq -10$ bar



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Nucleation model of the liquid phase [59]:

- ▶ Unstable when nucleation rate too important :
 - Happens close to the spinodal point $P_{nucl} \simeq -10$ bar

Destabilization by vacancies criterion [40] :

- ▶ Unstable when insulating gap between vacancies/interstitials is closed
 - Obtained by linear extrapolations $P_{clos} \simeq 0$ bar

Stability limits of metastable solid helium-4



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Nucleation model of the liquid phase [59]:

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Destabilization by vacancies criterion [40] :

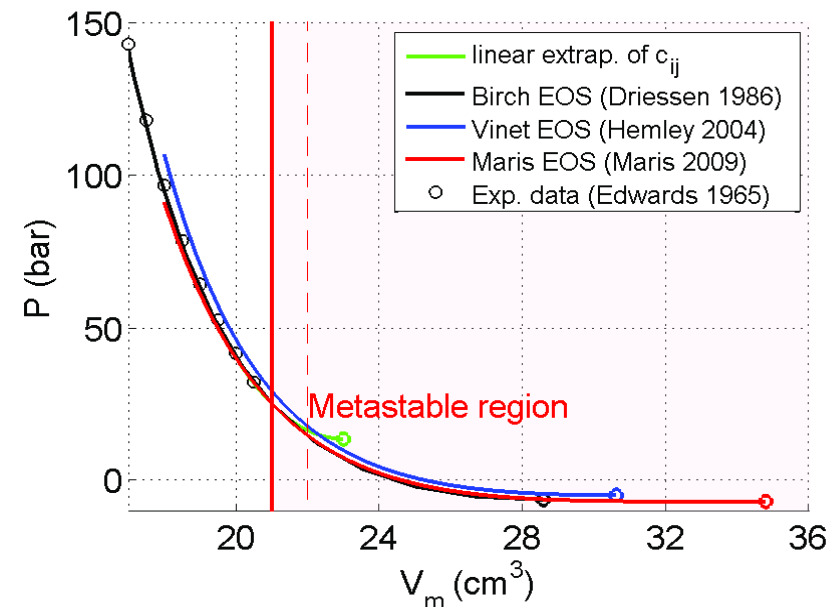
► Unstable when insulating gap between vacancies/interstitials is closed

-Obtained by linear extrapolations $P_{clos} \simeq 0 \text{ bar}$

Conclusion :

Solid helium 4 can be brought down to 0 bar

Extrapolated equation of state gives $V_m = 24 \text{ cm}^3$





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Overcoming sound anisotropy

Numerical simulation of sound propagation

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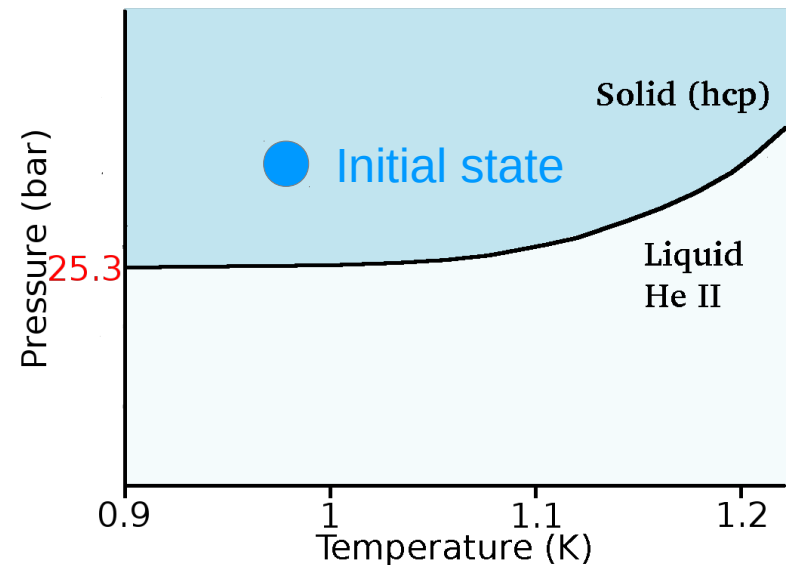
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General principle of the ultrasonic method



- ▶ General principle :
Converging ultrasonic sound wave inside the crystal



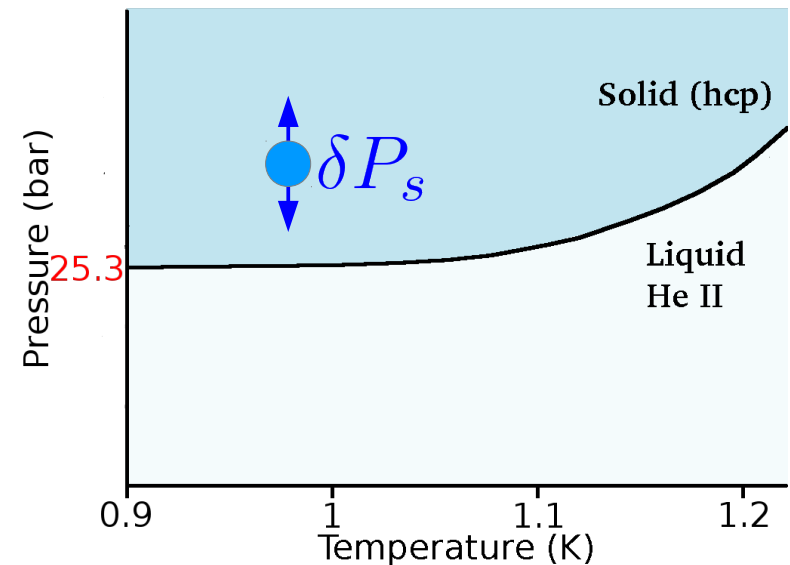
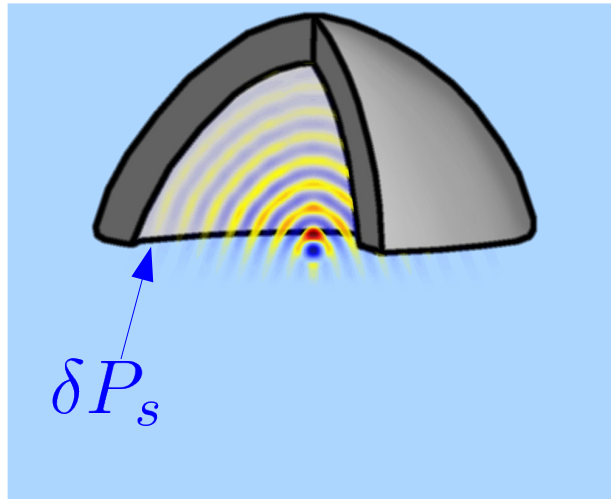
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► General principle :
Converging ultrasonic sound wave inside the crystal

δP_s pressure at the surface : melting line not crossed



General principle of the ultrasonic method

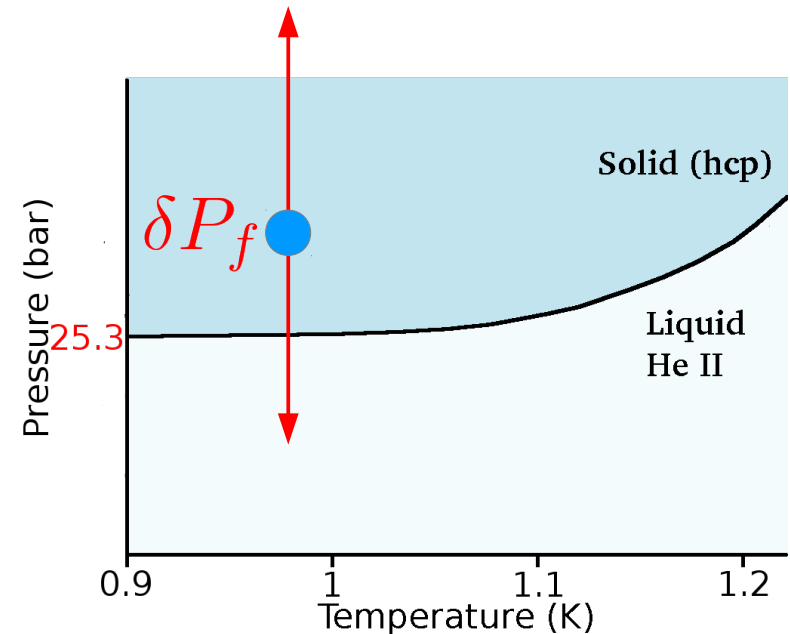
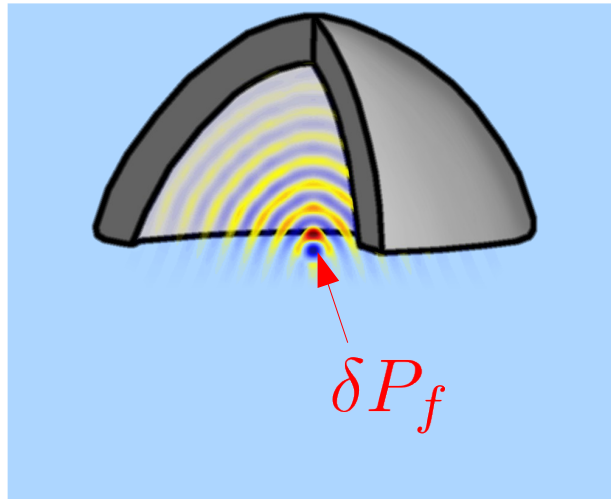


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► General principle :
Converging ultrasonic sound wave inside the crystal

δP_s pressure at the surface : melting line not crossed

δP_f pressure at the focus : pressure swings cross the melting line!



General principle of the ultrasonic method

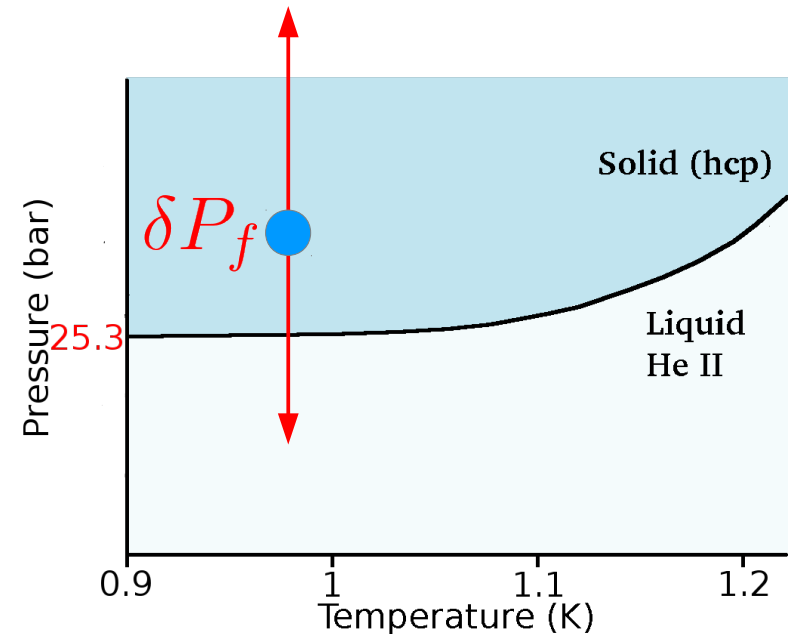
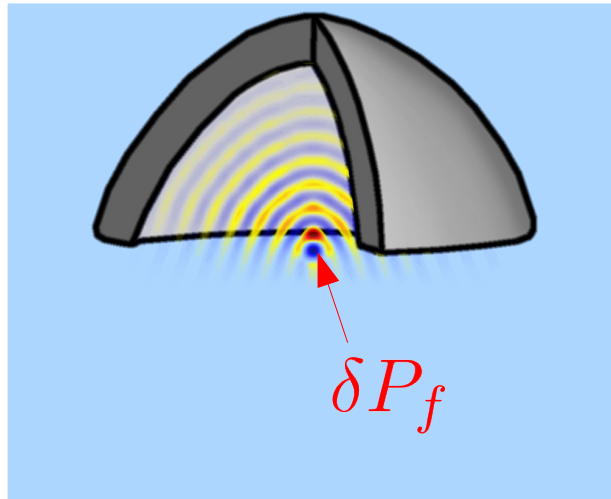


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- ▶ General principle :
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δP_s pressure at the surface : melting line not crossed

δP_f pressure at the focus : pressure swings cross the melting line!



amplification factor $g = \delta P_f / \delta P_s$

- ▶ More complicated in solid because of **sound anisotropy** !

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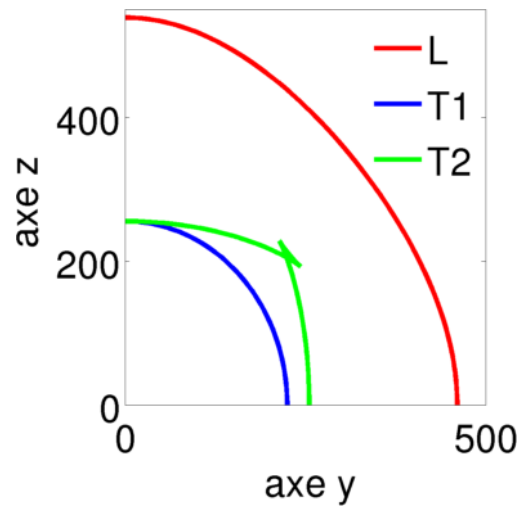
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► Wavesurface to focus sound wave in solid:

-Calculated from elastic constant [82]

-3 propagation **modes**

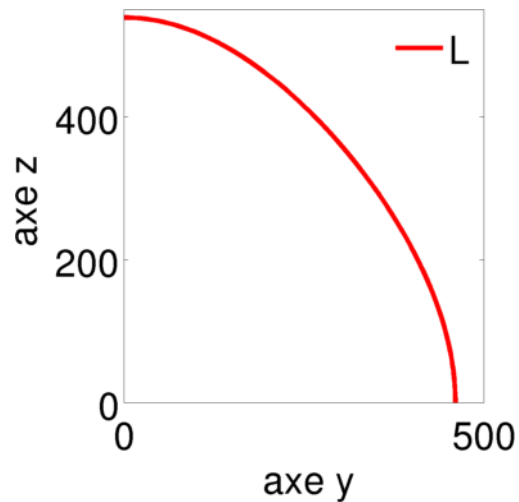




► **Wavesurface** to focus sound wave in solid:

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-3 propagation **modes** : longitudinal mode produce the **largest δP_f**

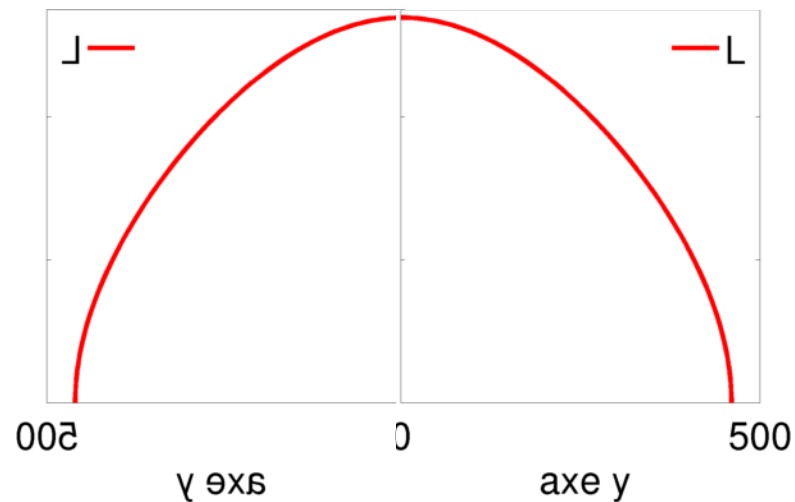




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-3 propagation **modes** : longitudinal mode produce the **largest δP_f**

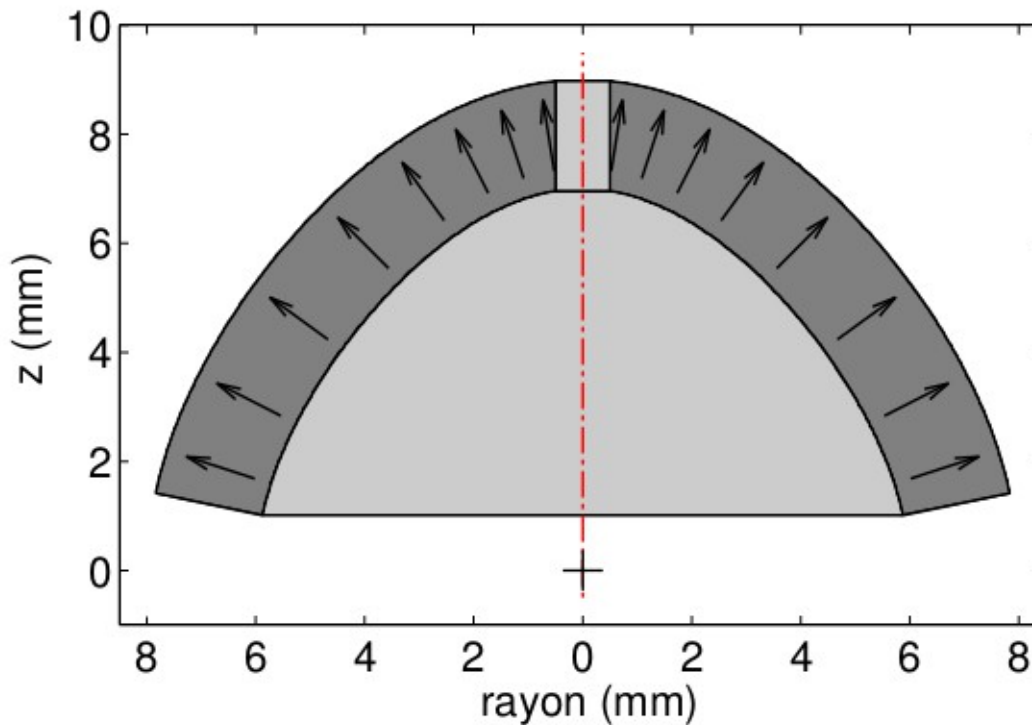




► **Wavesurface** to focus sound wave in solid:

-Calculated from elastic constant [82]

-3 propagation **modes** : longitudinal mode produce the **largest δP_f**





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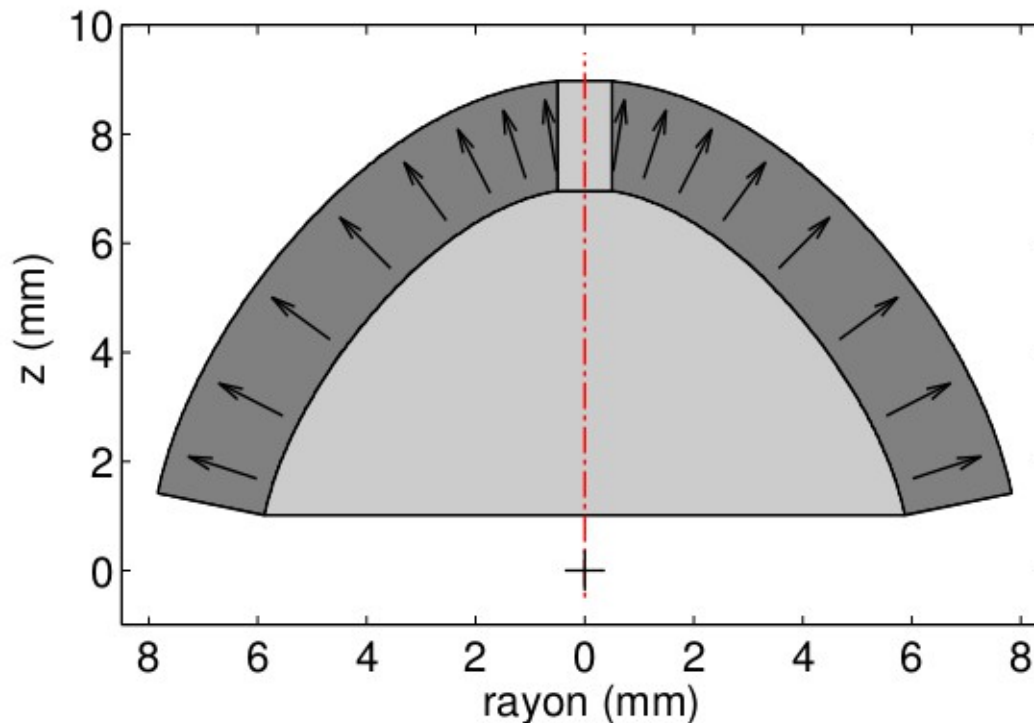
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► **Wavesurface** to focus sound wave in solid:

-Calculated from elastic constant [82]

-3 propagation **modes** : longitudinal mode produce the **largest δP_f**



► Radially polarized

► **Thickness mode** excited :
 $f = 1 \text{ MHz}$



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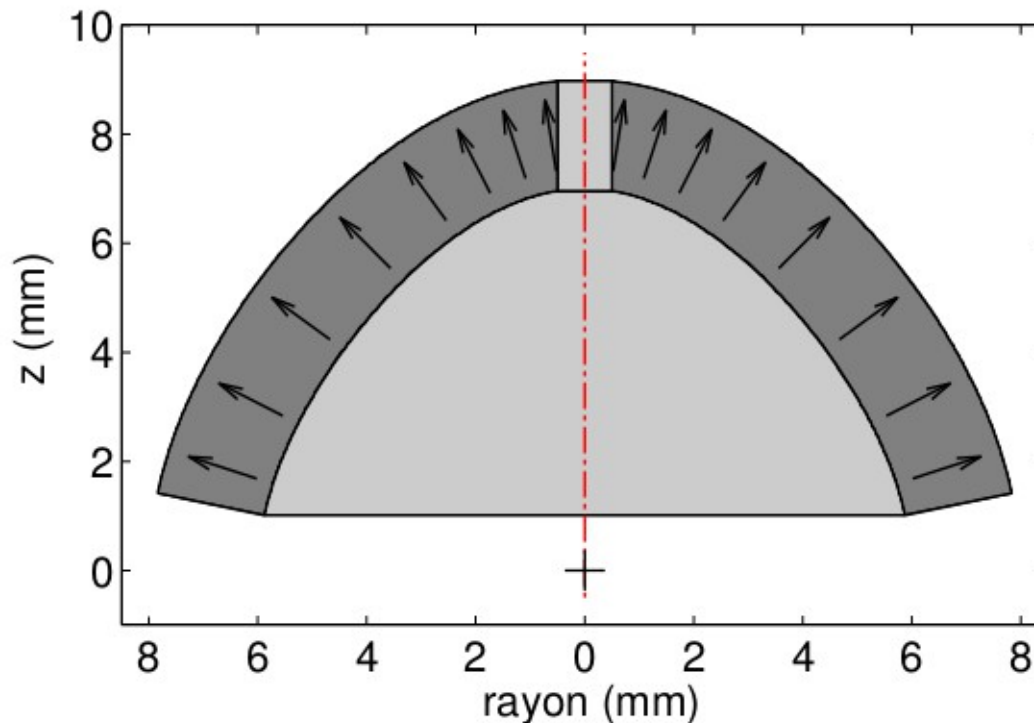
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-Calculated from elastic constant [82]

-3 propagation **modes** : longitudinal mode produce the **largest δP_f**



► Radially polarized

► **Thickness mode** excited :
 $f = 1$ MHz



► The emitter was machined at the **mechanical workshop** with diamond tools by A. Leclerc



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► Numerical simulation developed to **predict sound propagation** :

-Solve the equation of motion for cylindrical symmetry :
2D problem

-A **finite difference** method is used

► **2 computed quantities** : volume variations $\delta V/V$ and shear C

$$\delta V/V = \varepsilon_{rr} + \varepsilon_{\theta\theta} + \varepsilon_{zz}$$

$$C^2 = \frac{1}{3} [(\varepsilon_{rr} - \varepsilon_{\theta\theta})^2 + (\varepsilon_{zz} - \varepsilon_{\theta\theta})^2 + (\varepsilon_{rr} - \varepsilon_{zz})^2] + 2\varepsilon_{rz}^2$$



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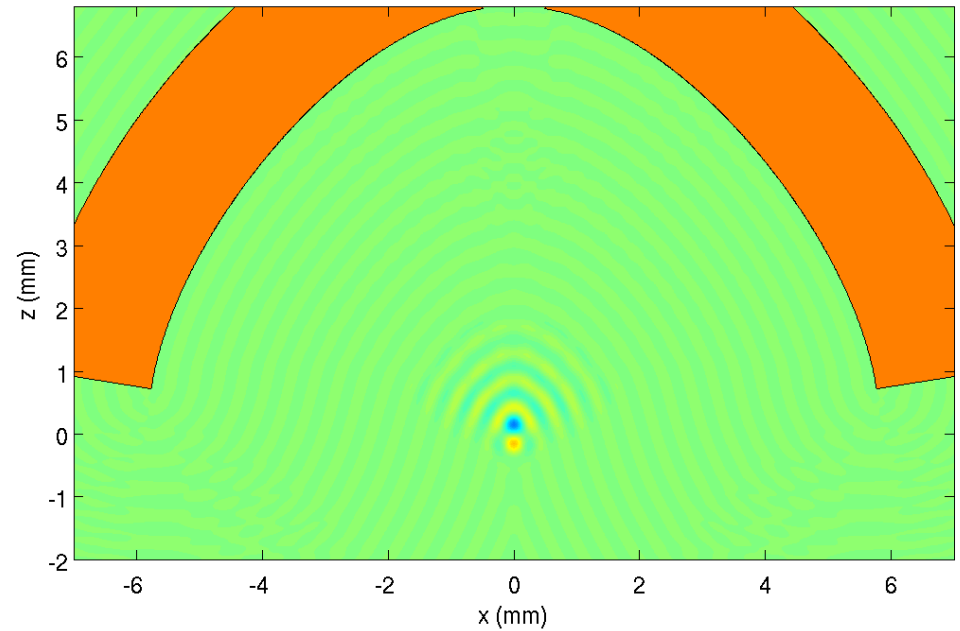
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► One emitter simulation :

-Good focusing

-Amplification factor
 $g = 86$

Champ sonore simulé, $t = 13.80 \mu\text{s}$





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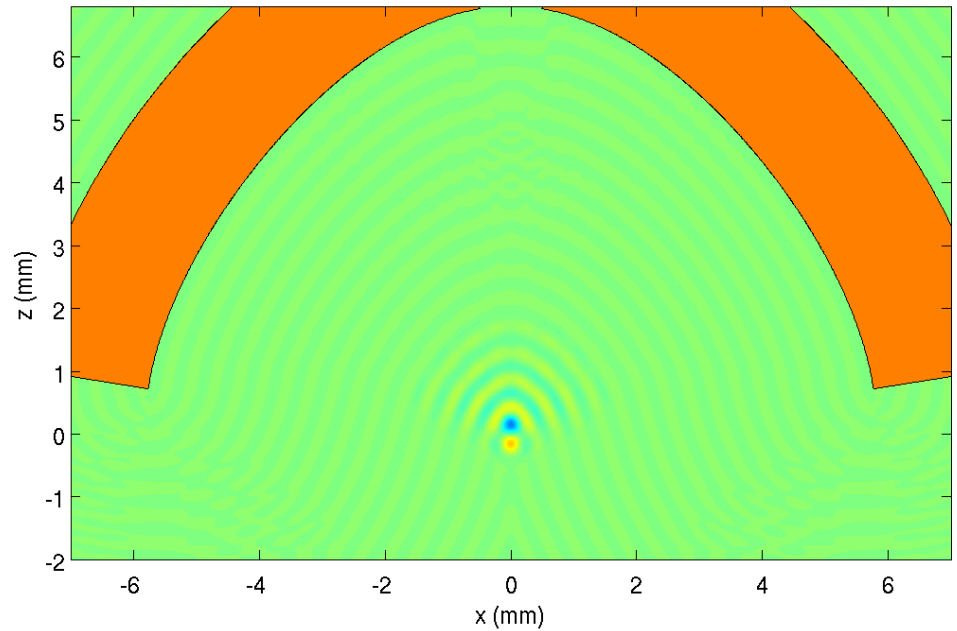
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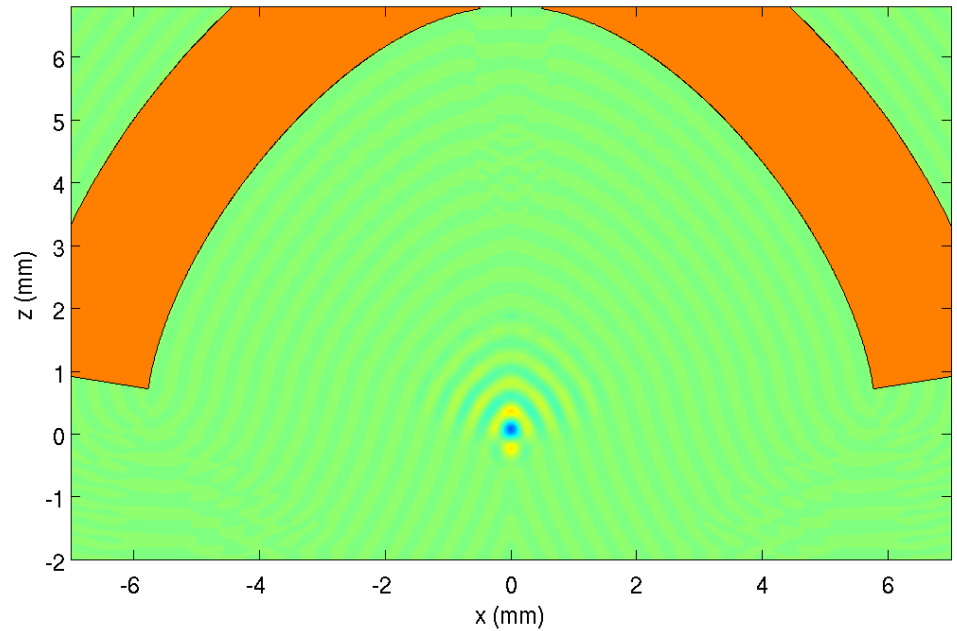
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► One emitter simulation :

-Good focusing

-Amplification factor
 $g = 86$

Champ sonore simulé, $t = 13.90 \mu\text{s}$





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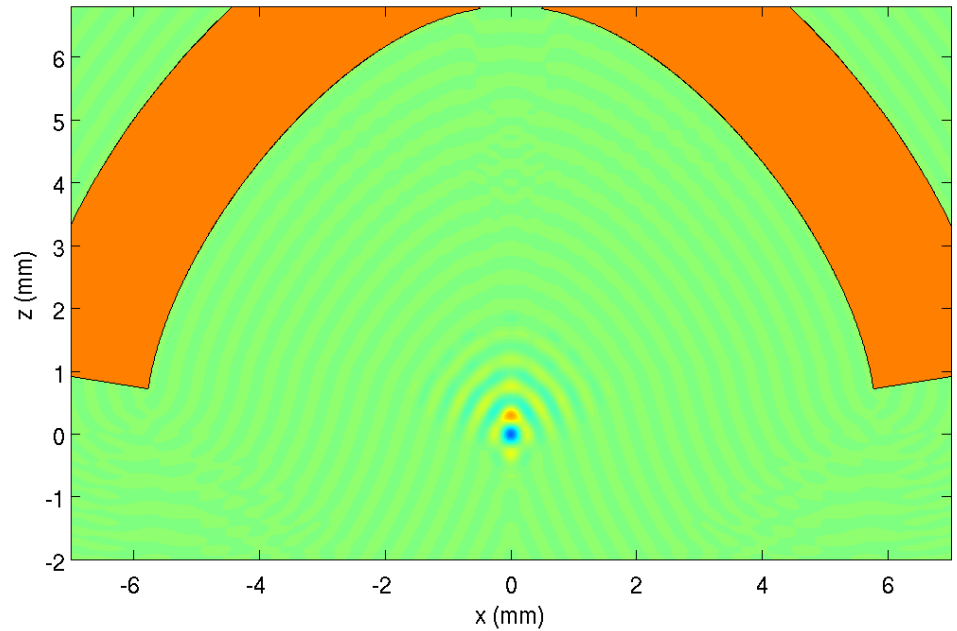
Conclusion and prospects

► One emitter simulation :

-Good focusing

-Amplification factor
 $g = 86$

Champ sonore simulé, $t = 14.00 \mu\text{s}$





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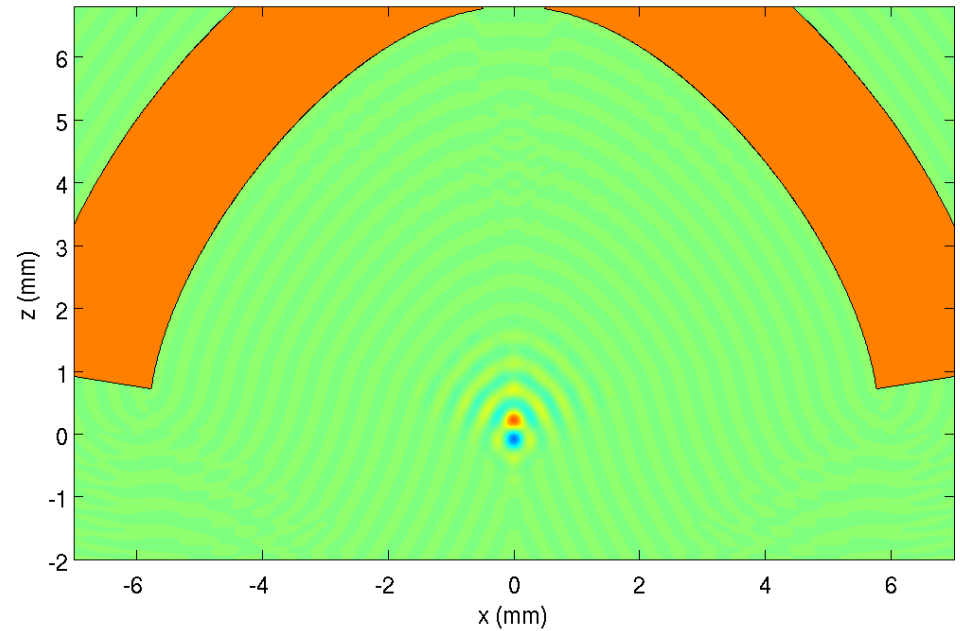
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► One emitter simulation :

-Good focusing

-Amplification factor
 $g = 86$

Champ sonore simulé, $t = 14.10 \mu\text{s}$





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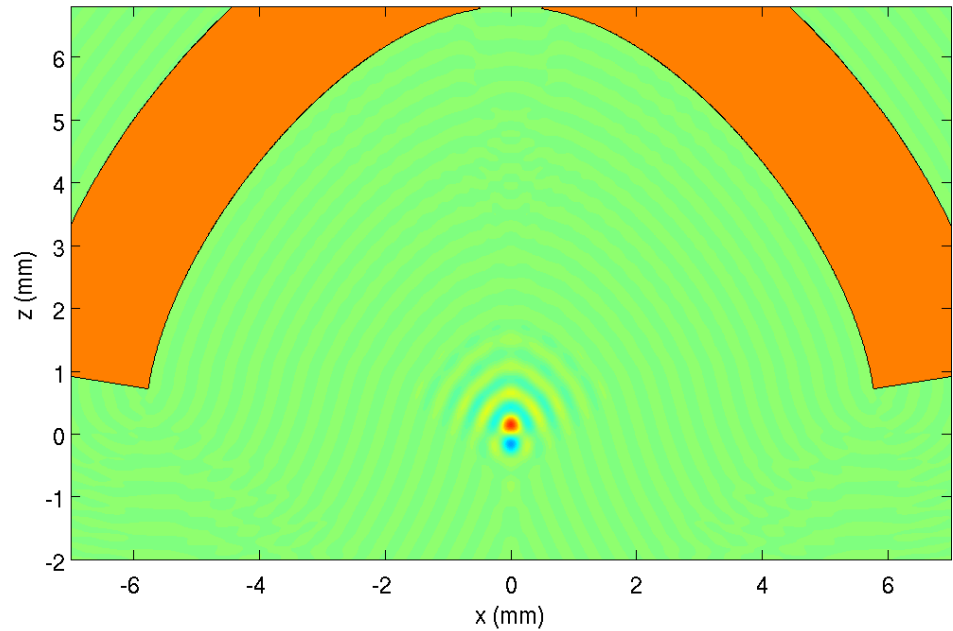
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► One emitter simulation :

-Good focusing

-Amplification factor
 $g = 86$

Champ sonore simulé, $t = 14.20 \mu\text{s}$





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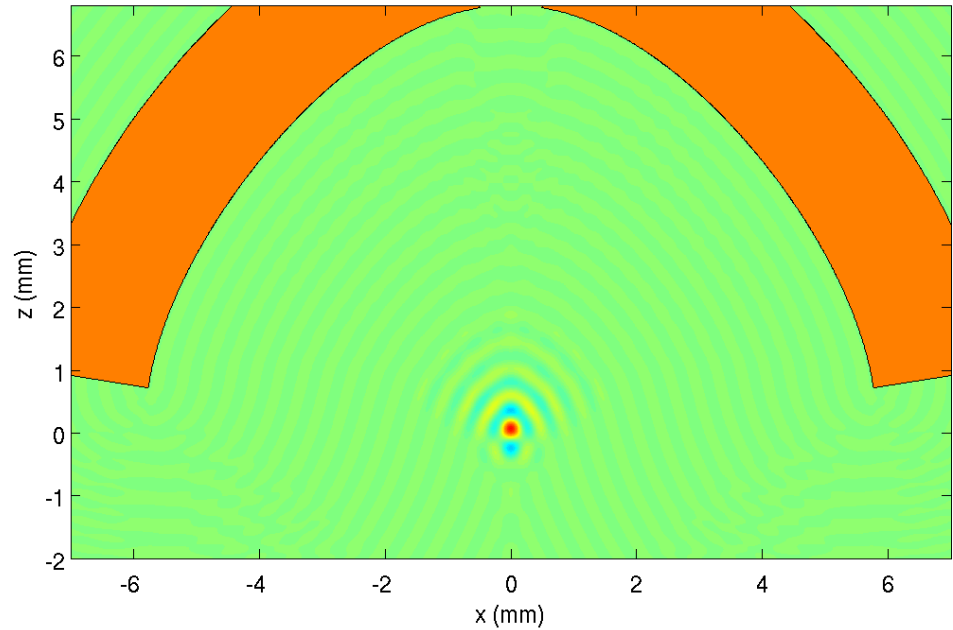
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► One emitter simulation :

-Good focusing

-Amplification factor
 $g = 86$

Champ sonore simulé, $t = 14.30 \mu\text{s}$





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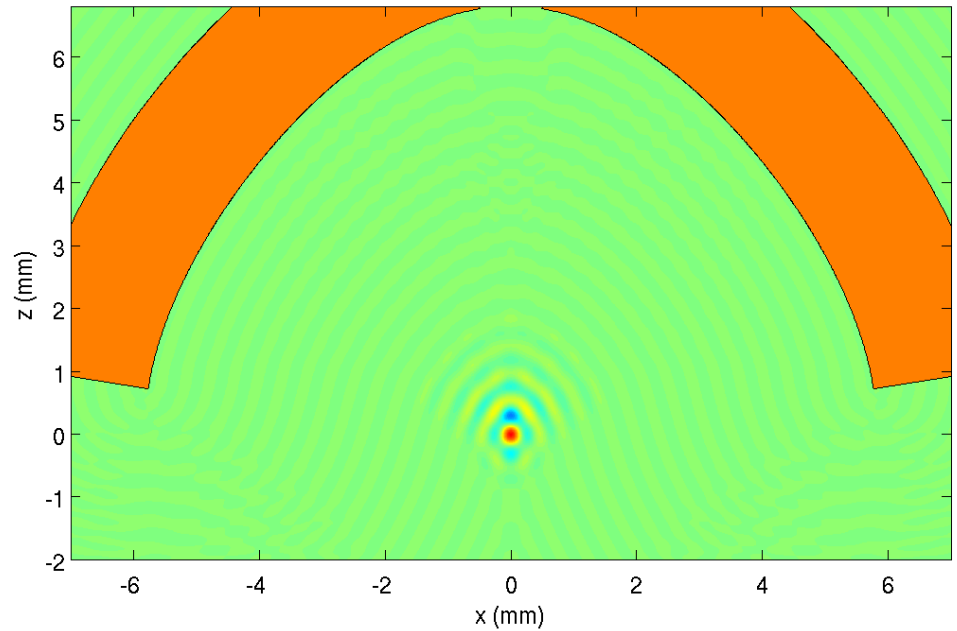
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► One emitter simulation :

-Good focusing

-Amplification factor
 $g = 86$

Champ sonore simulé, $t = 14.40 \mu\text{s}$





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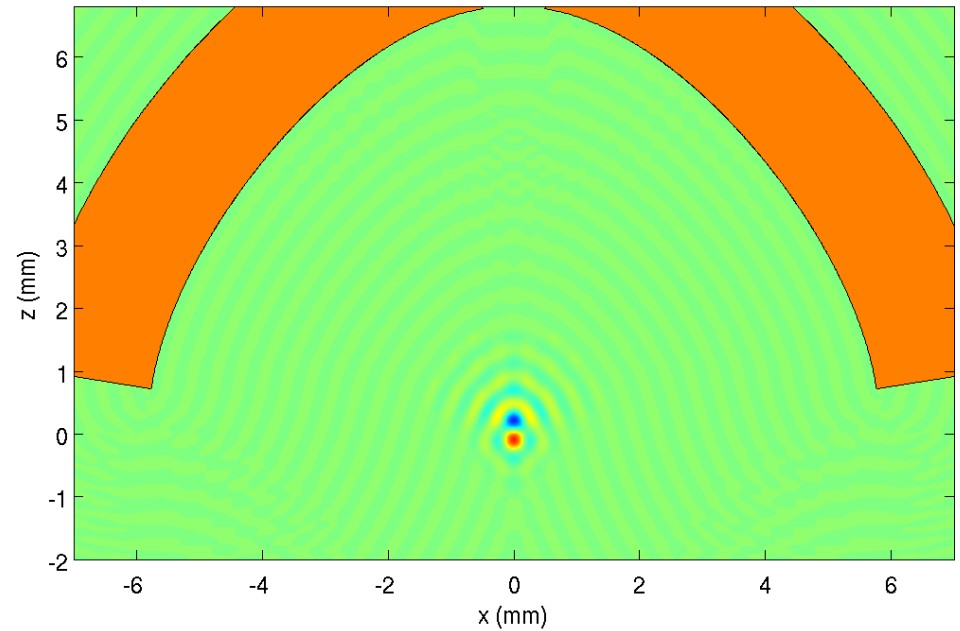
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► One emitter simulation :

-Good focusing

-Amplification factor
 $g = 86$

Champ sonore simulé, $t = 14.50 \mu\text{s}$





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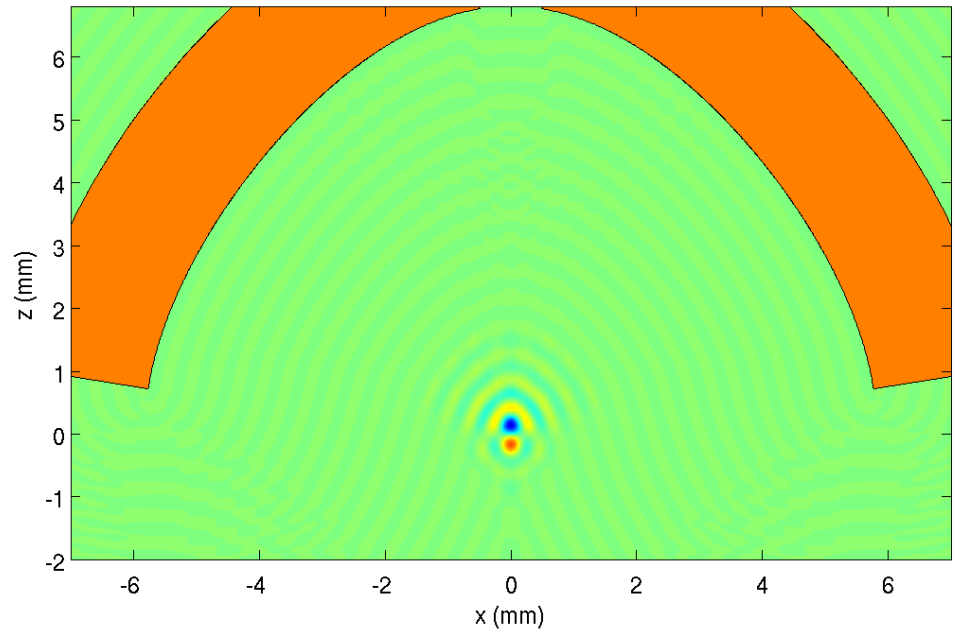
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► One emitter simulation :

-Good focusing

-Amplification factor
 $g = 86$

Champ sonore simulé, $t = 14.60 \mu\text{s}$





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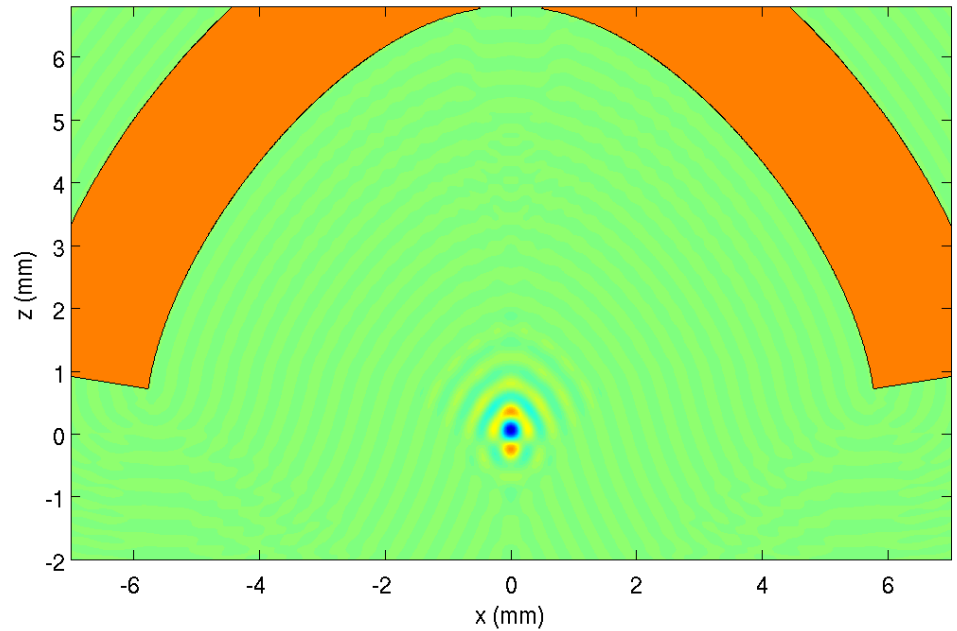
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► One emitter simulation :

-Good focusing

-Amplification factor
 $g = 86$

Champ sonore simulé, $t = 14.70 \mu\text{s}$





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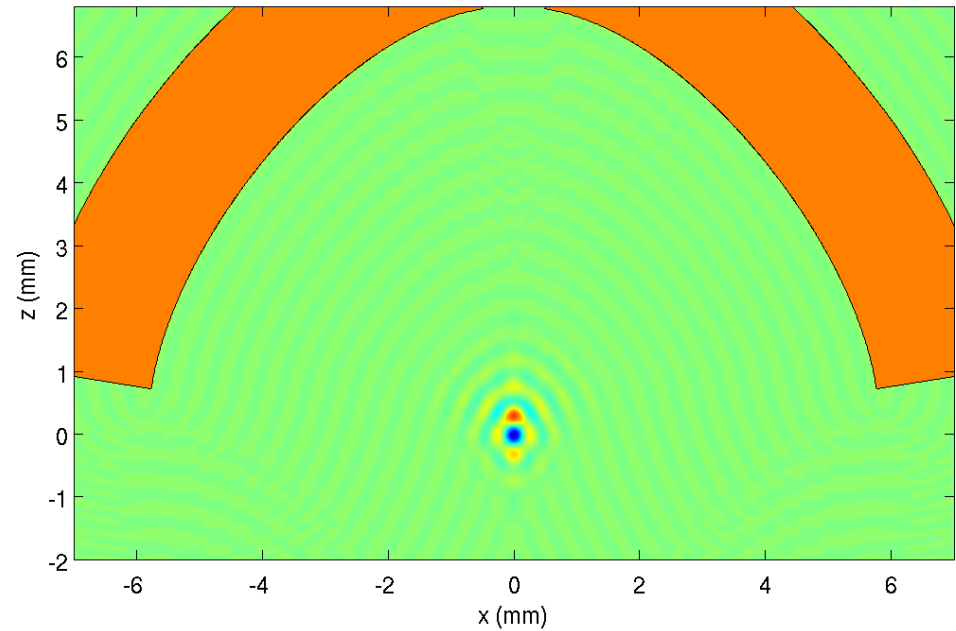
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► One emitter simulation :

-Good focusing

-Amplification factor
 $g = 86$

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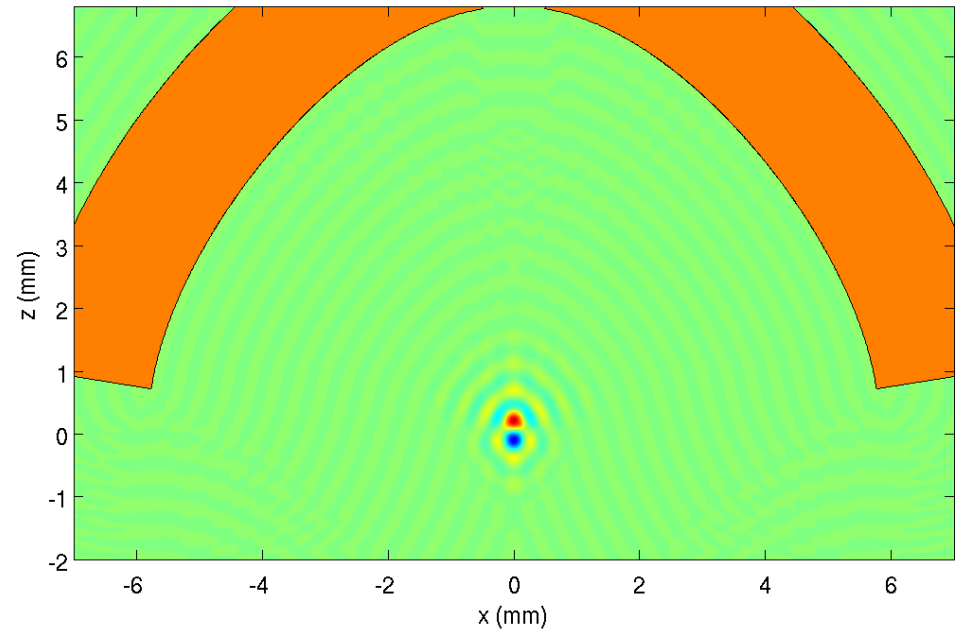
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► One emitter simulation :

-Good focusing

-Amplification factor
 $g = 86$

Champ sonore simulé, $t = 14.90 \mu\text{s}$





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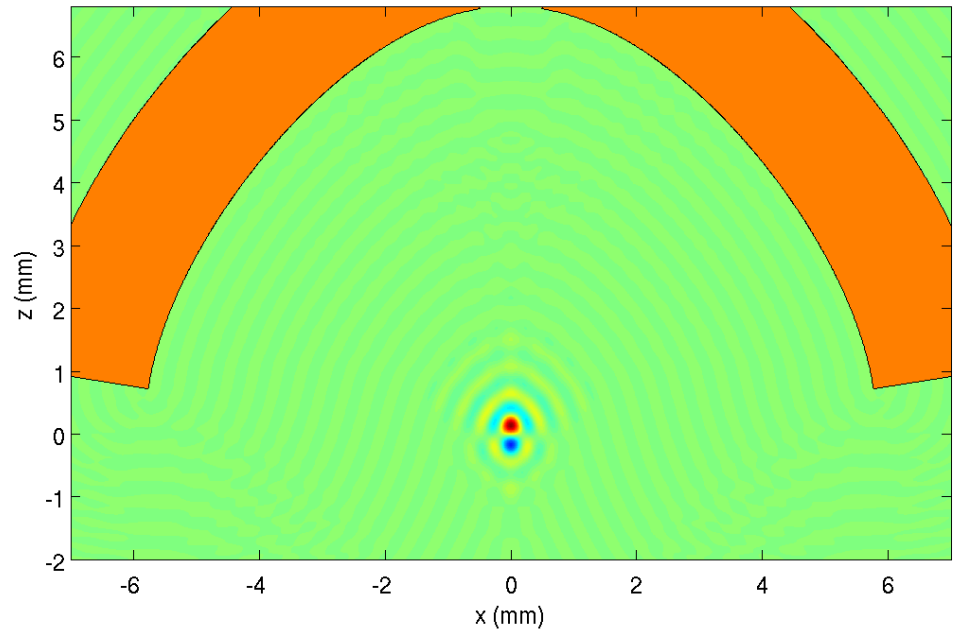
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► One emitter simulation :

-Good focusing

-Amplification factor
 $g = 86$

Champ sonore simulé, $t = 15.00 \mu\text{s}$





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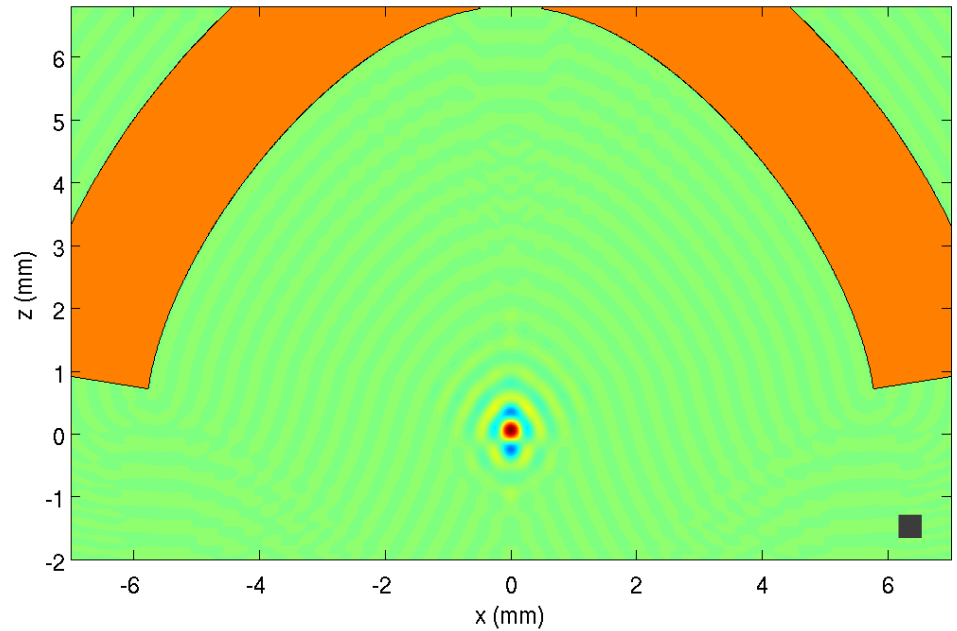
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► One emitter simulation :

-Good focusing

-Amplification factor
 $g = 86$

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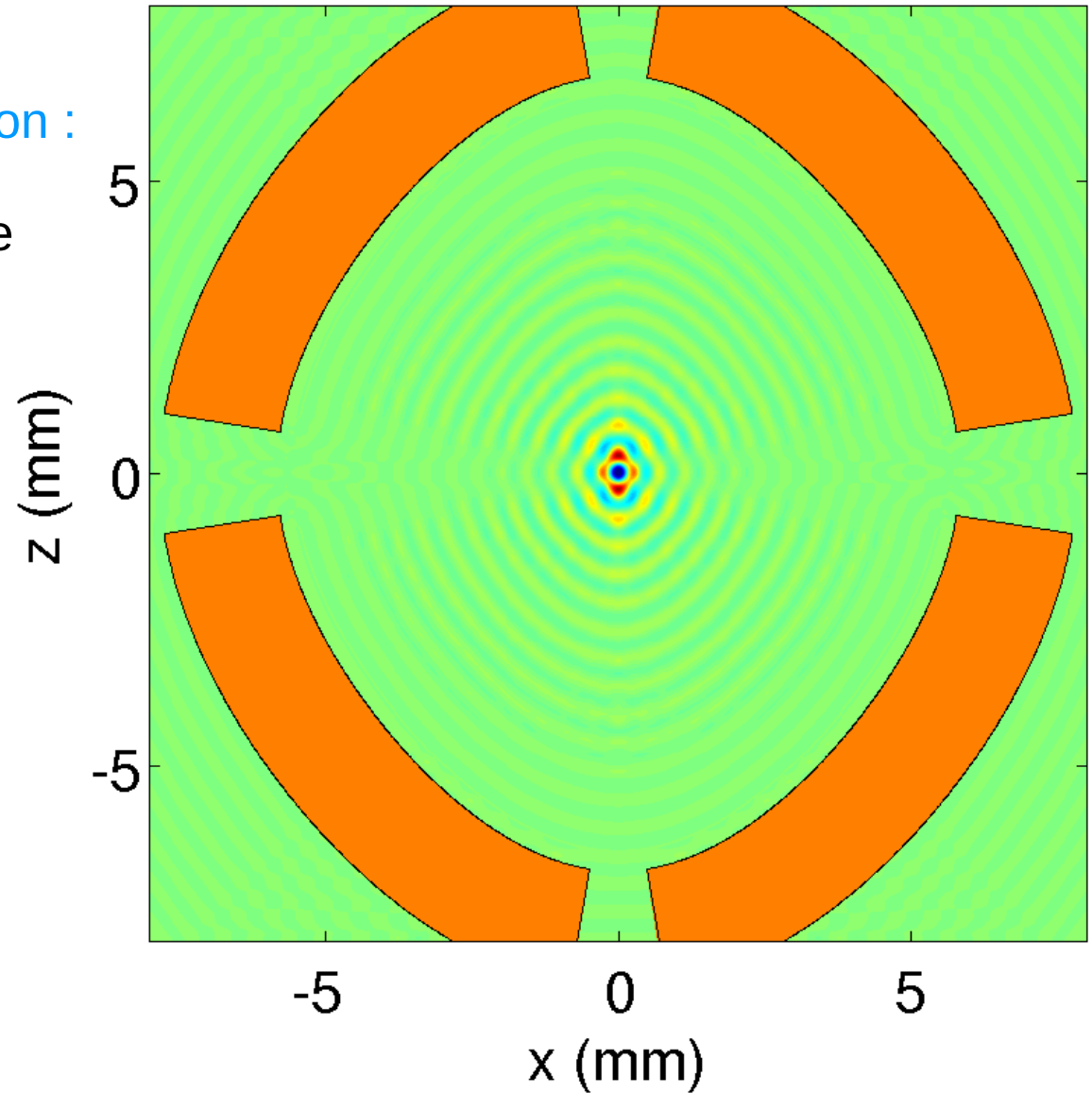
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► Two emitter simulation :

-Stationnary waves are formed

-Amplification factor
 $g = 178$



Conclusion :

Taylor-made sound emitter should be able to focus ultrasound waves in helium 4 crystals



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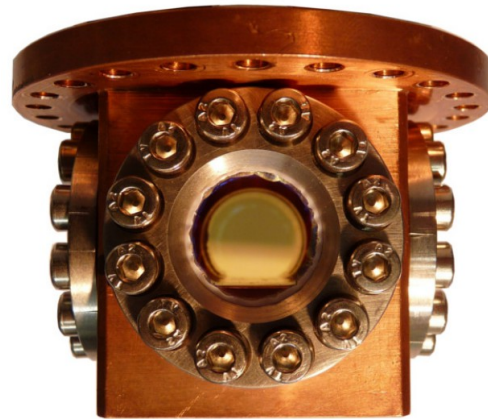
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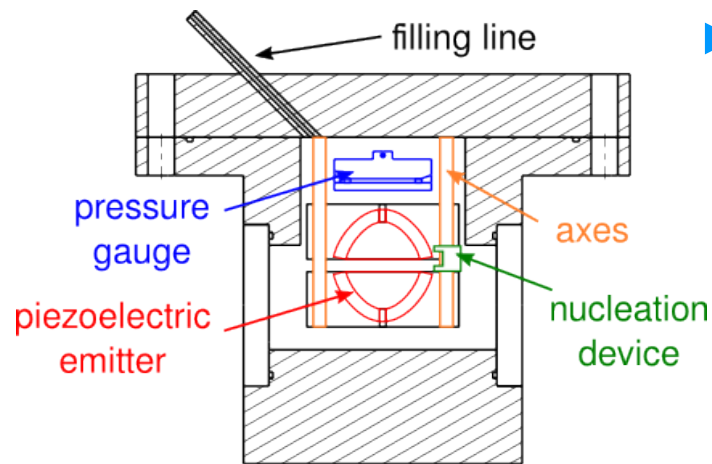
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▶ Experimental cell:

- OFHC copper
- 30 cm³ internal volume
- 4 optical access
- Polished growth surface



▶ Cell equipment:

- Straty-Adams pressure gauge with **mbar resolution**
- 2 piezoelectric emitters **guided** by two axes
- Nucleation device

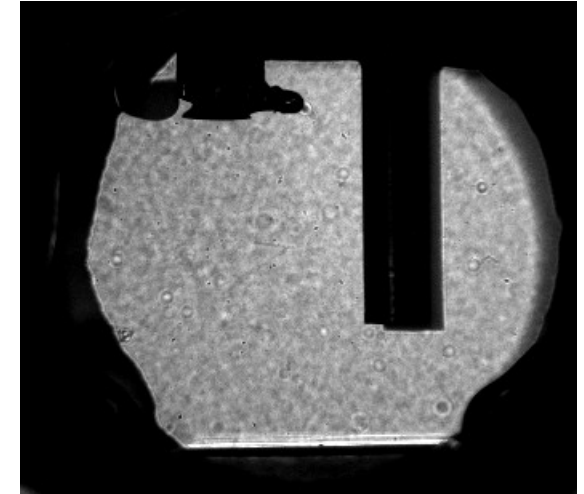


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► Helium 4 crystal seeds :

-Cell filled with liquid helium 4

-Pressure is **slowly increased** until a seed appears on the nucleator



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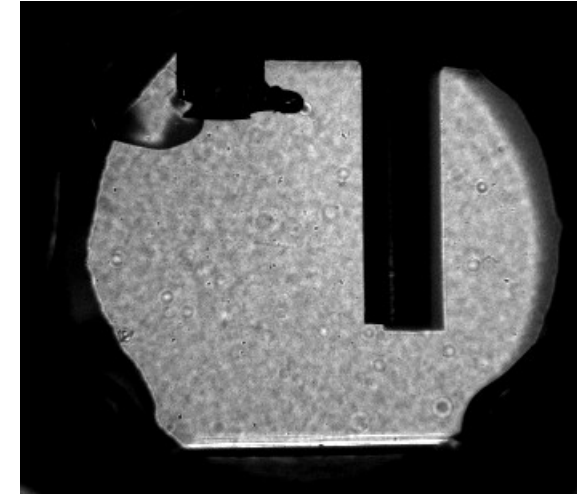


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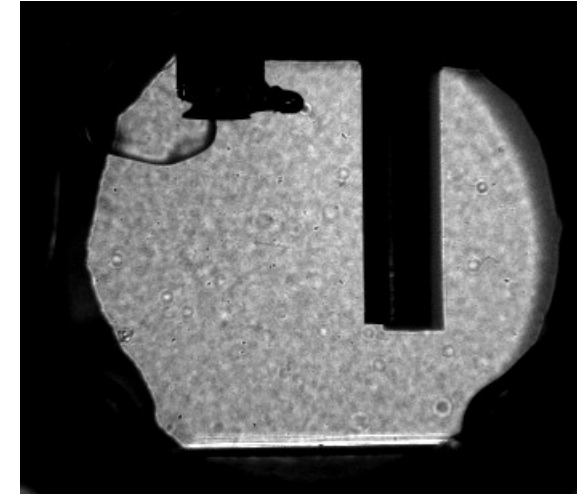
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prospects

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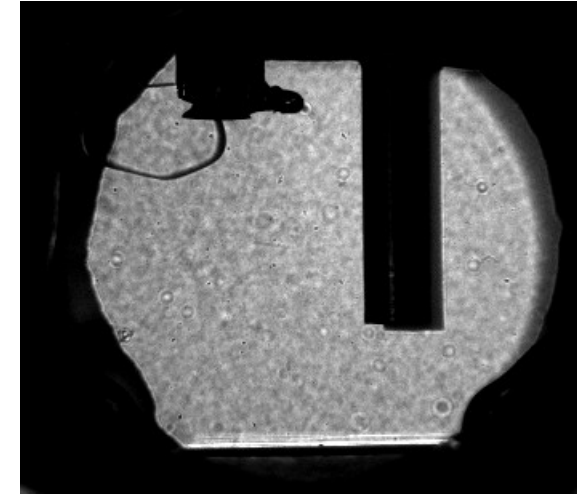




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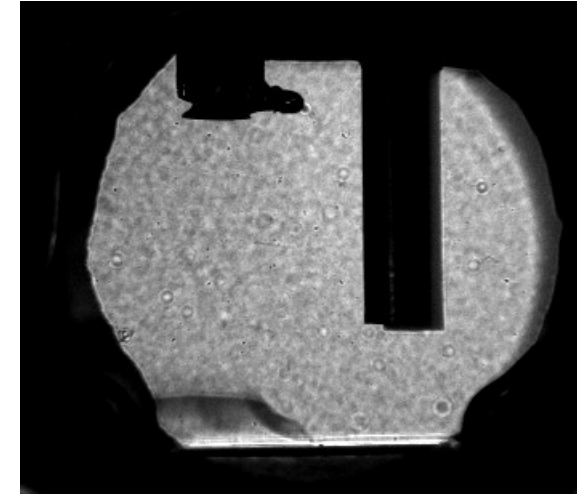
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► Helium 4 crystal seeds :

- Cell filled with liquid helium 4
- Pressure is **slowly increased** until a seed appears on the nucleator
- Seed falls** down on the floor





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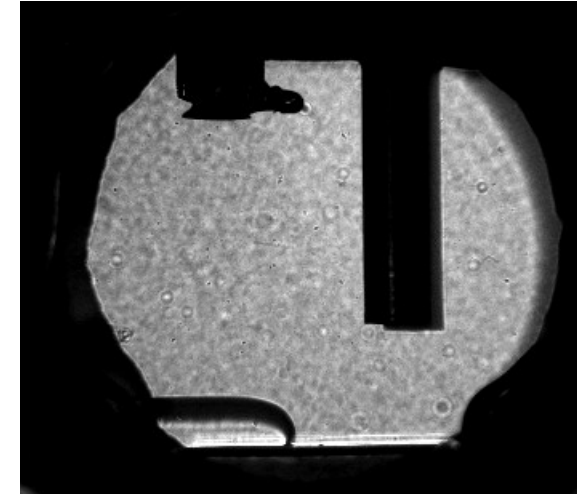
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Nucleation of helium-4 crystals



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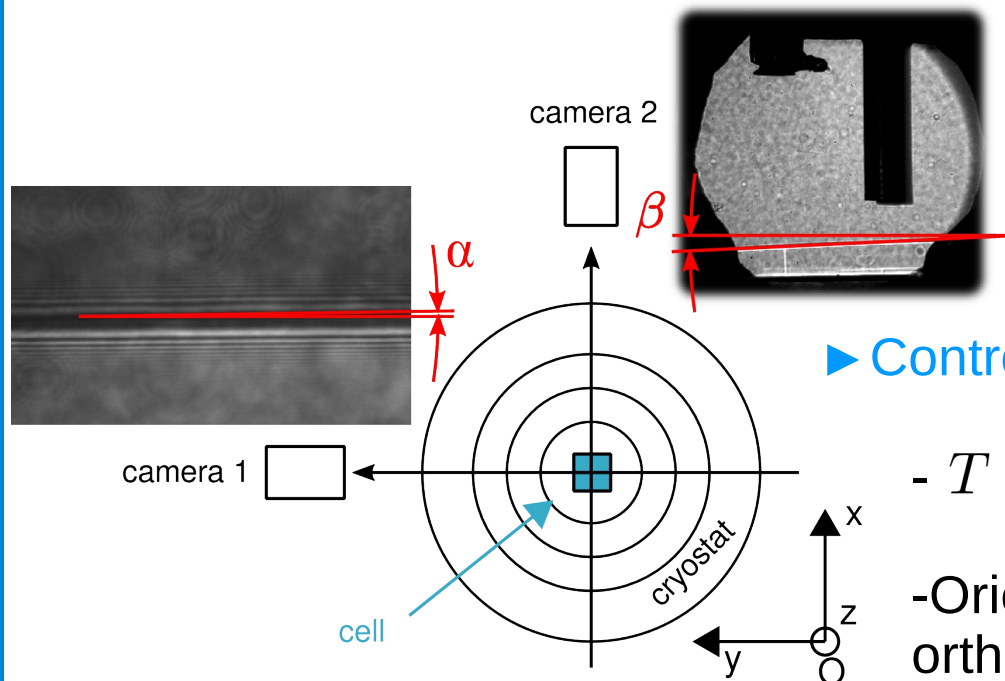
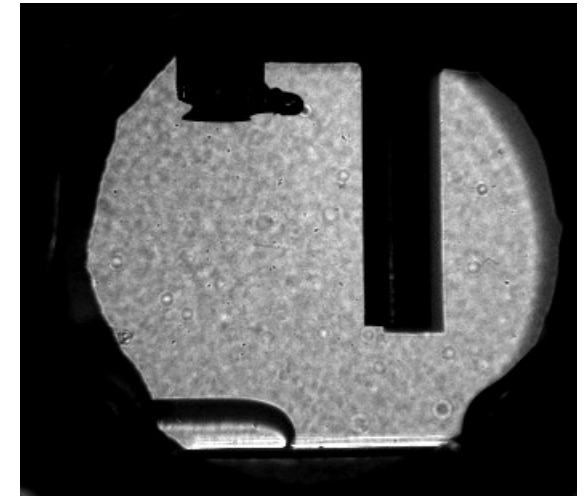
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► Helium 4 crystal seeds :

- Cell filled with liquid helium 4
- Pressure is **slowly increased** until a seed appears on the nucleator
- Seed falls** down on the floor



► Control of crystal orientation :

- $T < T_{rough}$ → **C-facet** visible

-Orientation controlled with two orthogonal directions

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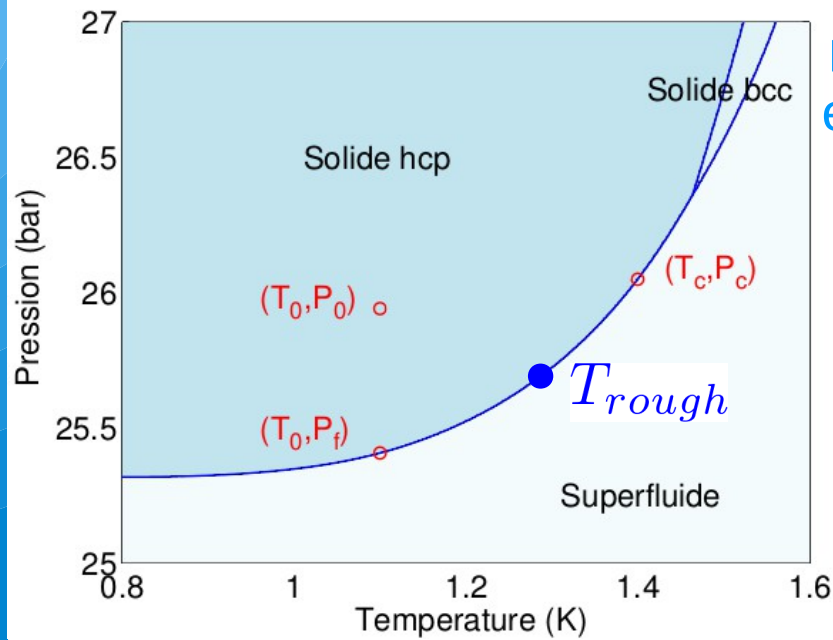
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► Avoid surface melting during sound emission:

-Increase the gap between crystal pressure and melting pressure

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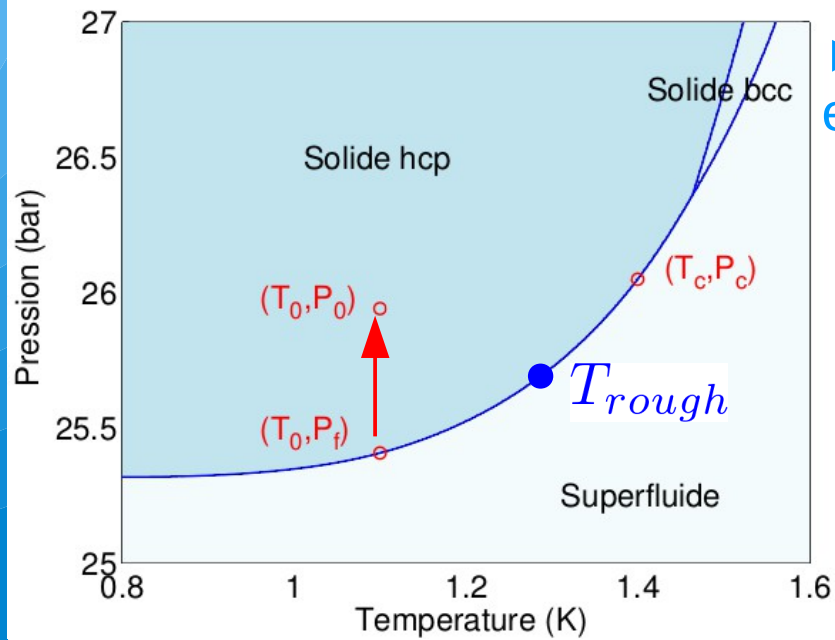
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► Avoid surface melting during sound emission:

-Pressurized at constant T

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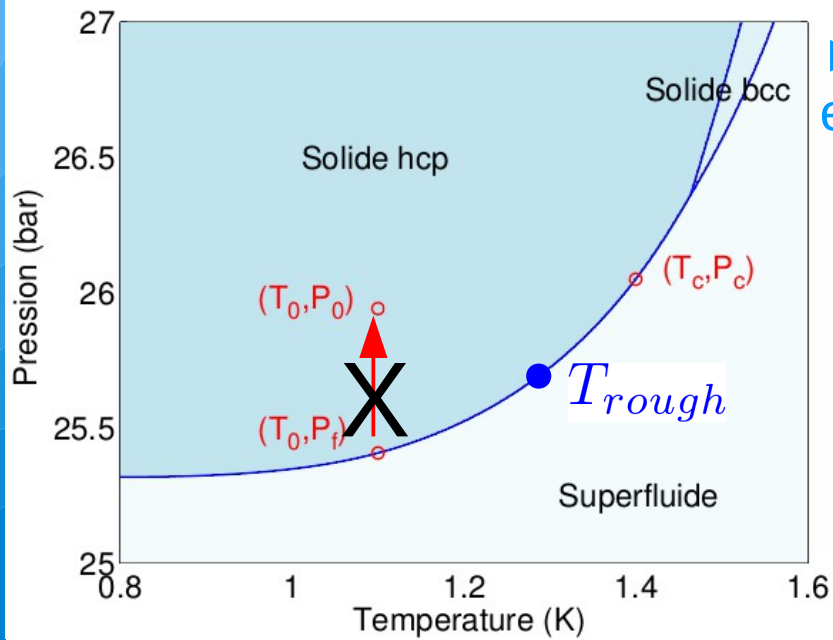
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► Avoid surface melting during sound emission:

~~-Pressurized at constant T~~

Harmful for the cristal !

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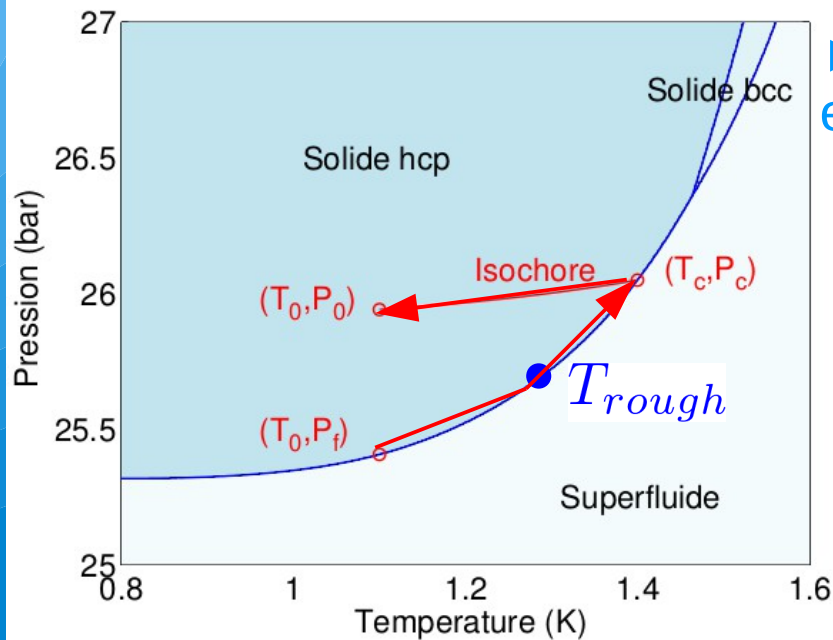
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► Avoid surface melting during sound emission:

- Cristal is **warmed** along the melting curve
- Cell is filled at (T_c, P_c)
- Cristal is **cooled down** to T_0 , it follows a path with **constant** V_m

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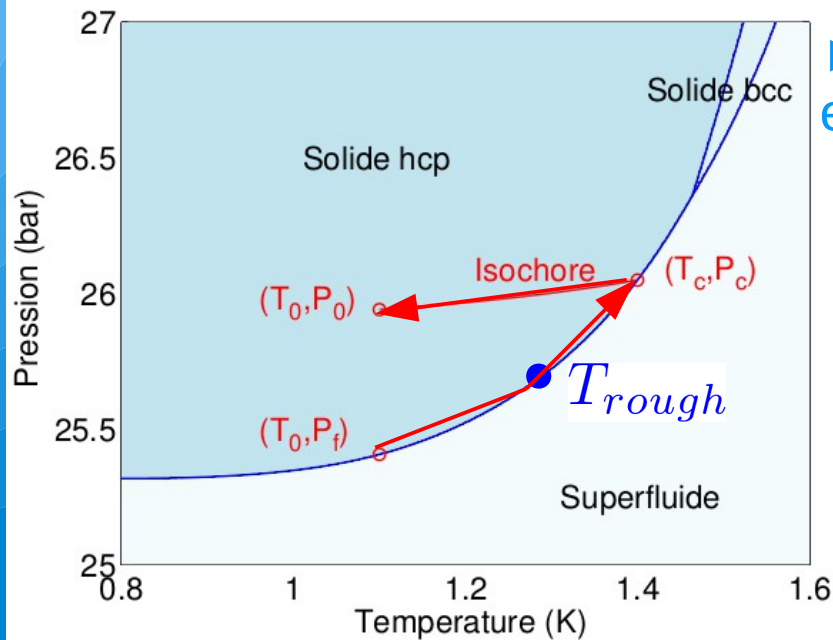
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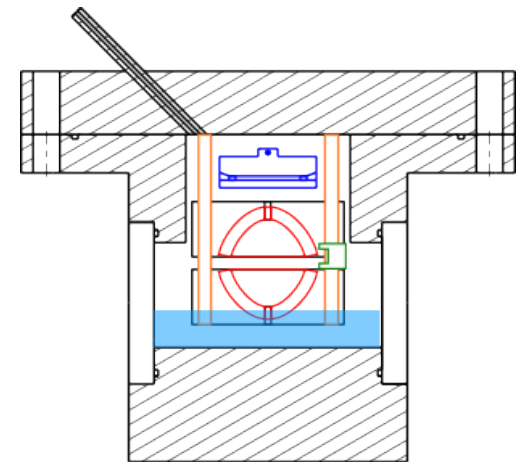
-Cell is filled at (T_c, P_c)

-Cristal is **cooled down** to T_0 , it follows a path with **constant** V_m

► Helium-4 crystal growth :

-Grown from superfluid at (T_c, P_c)

-Cell filled in ~1h



Preparation of helium-4 crystals



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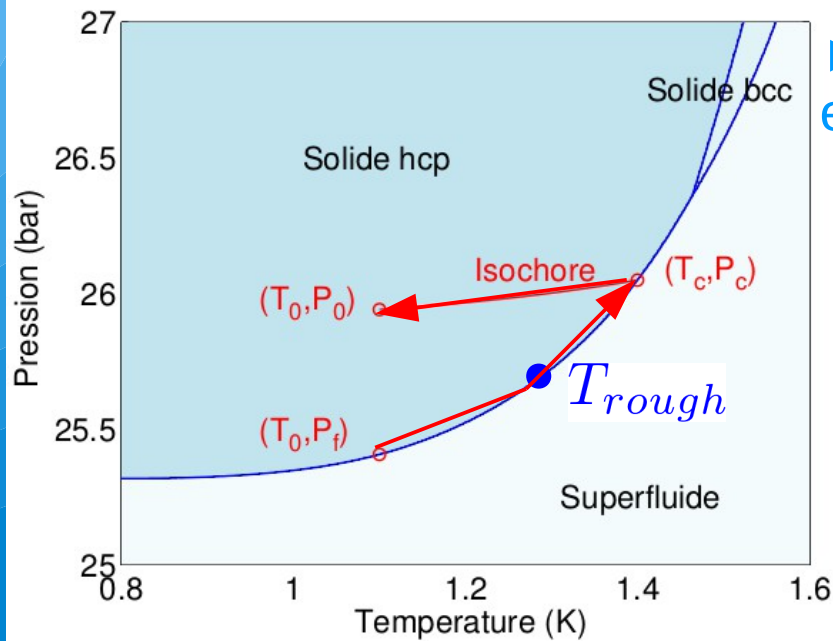
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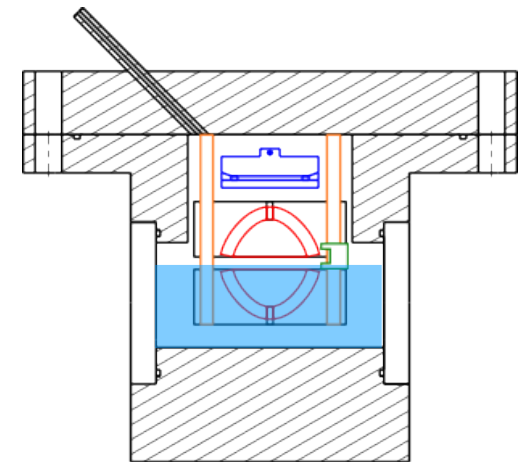
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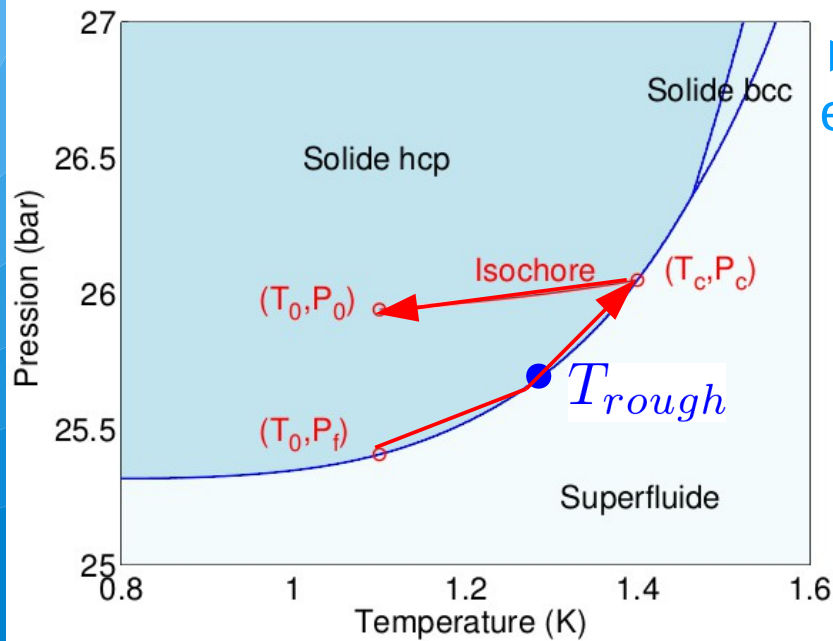
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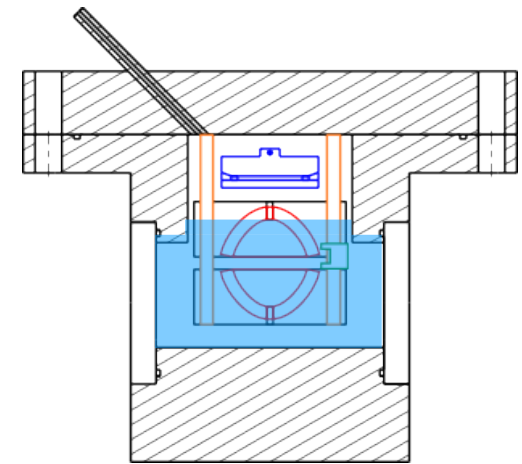
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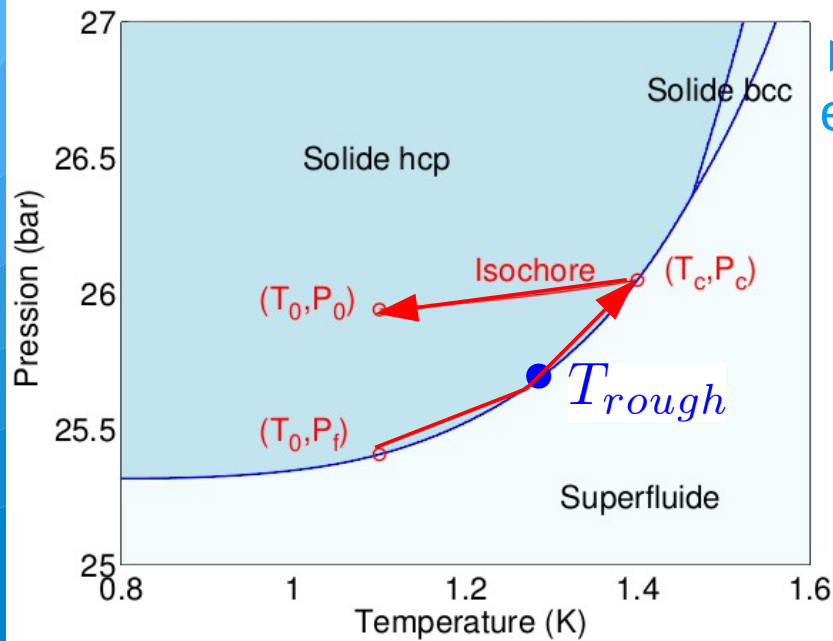
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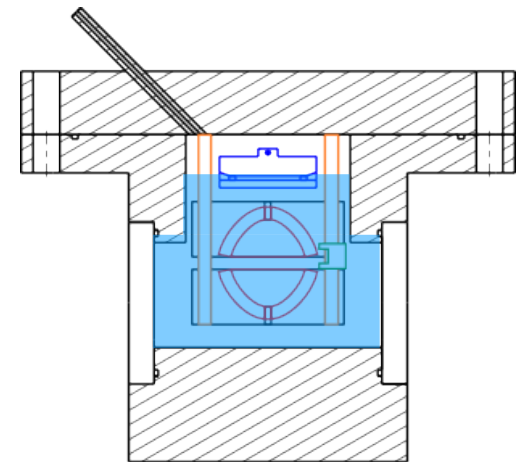
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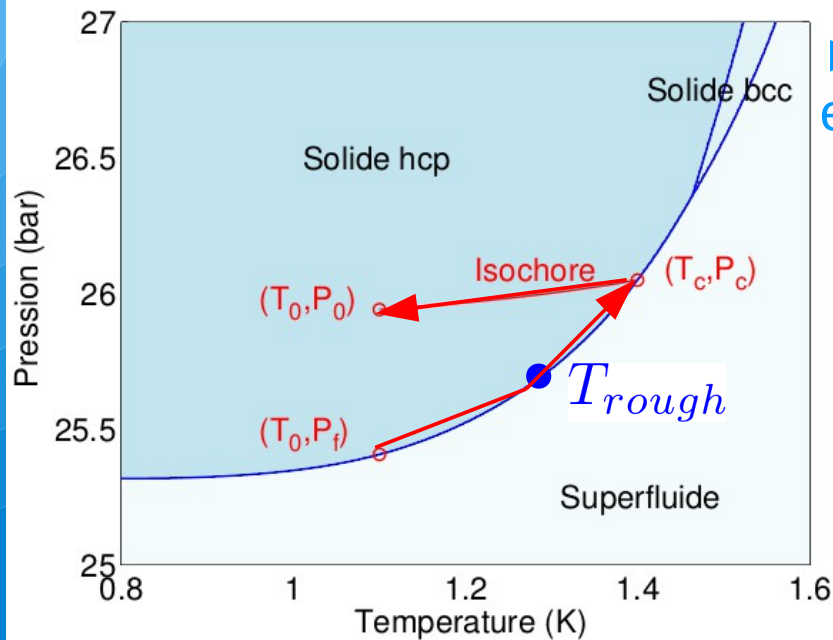
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► Avoid surface melting during sound emission:

-Cristal is **warmed** along the melting curve

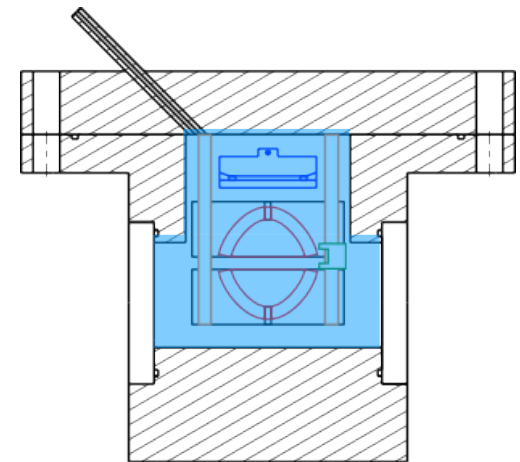
-Cell is filled at (T_c, P_c)

-Cristal is **cooled down** to T_0 , it follows a path with **constant** V_m

► Helium-4 crystal growth :

-Grown from superfluid at (T_c, P_c)

-Cell filled in ~1h



Preparation of helium-4 crystals



^4He

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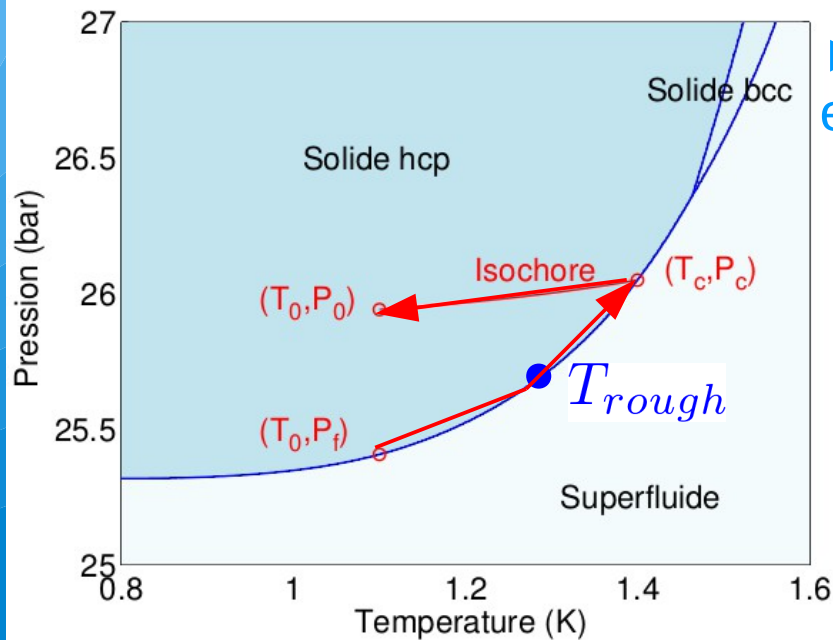
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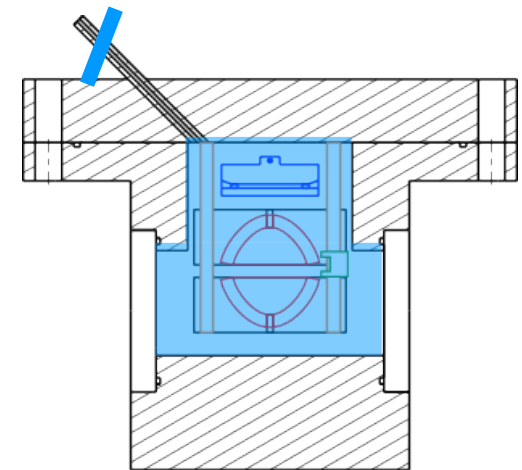
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► Helium-4 crystal growth :

-Grown from superfluid at (T_c, P_c)

-Cell filled in $\sim 1\text{h}$, and **capillary blocked**
→ Volume of the cell is isolated





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2. Focused ultrasound wave in solid helium-4
- 3. Interferometric measurement of density variations**

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4. Metastable solid helium-4 and stability limits

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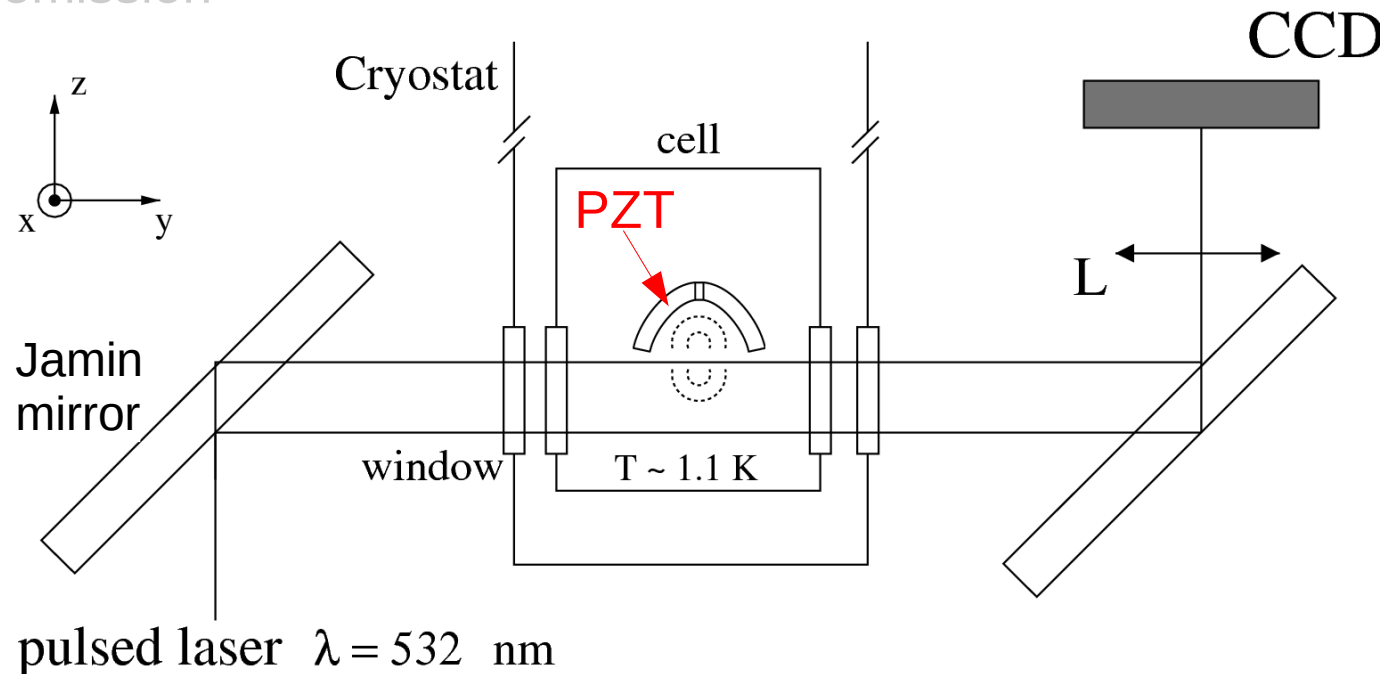
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-Piezoelectric emitter (**PZT**) excited by a **four cycle excitation** at 1 MHz

-**Jamin interferometer** : one arm through acoustic field
other arm in unperturbed region

-Region of interest is imaged onto a **CCD camera** → maps of phase shifts $\delta\phi(x, z)$

-8 ns **pulsed laser** ($\ll 1 \mu s$) takes "snapshots" of the sound field,
time sampling is made by varying the delay between laser pulse and sound emission



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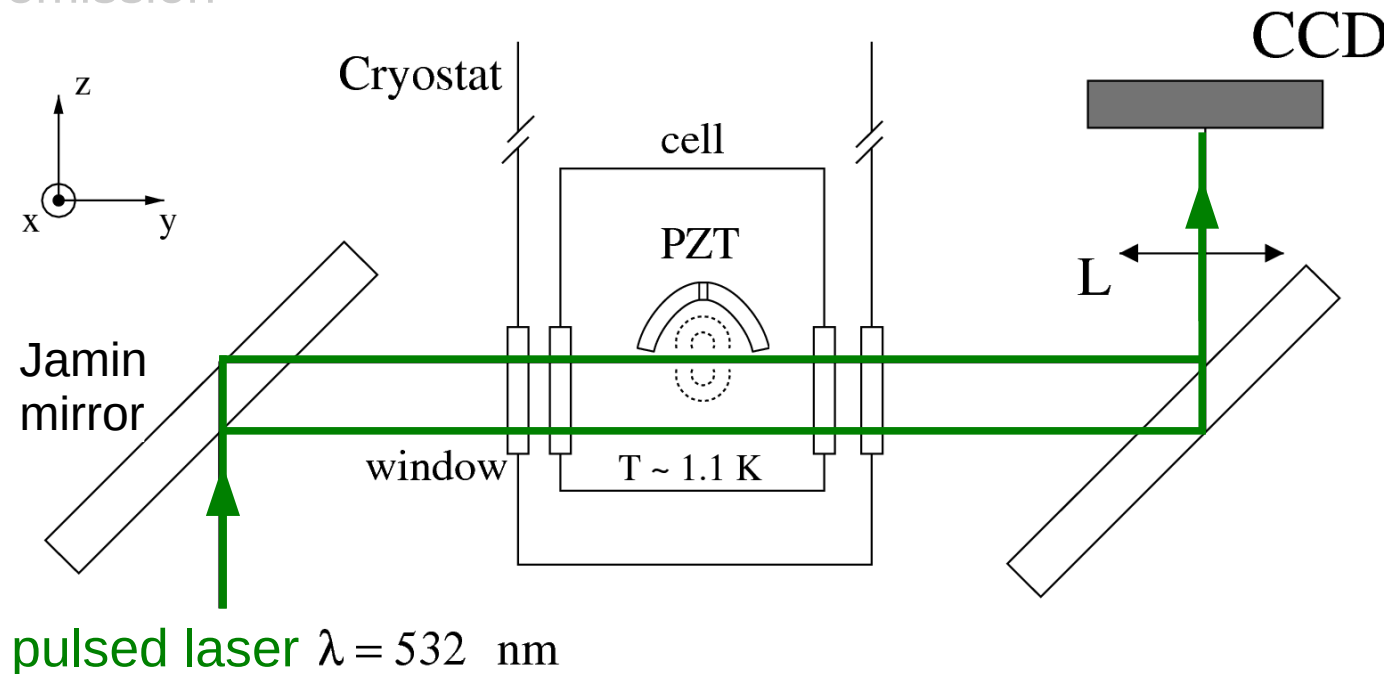
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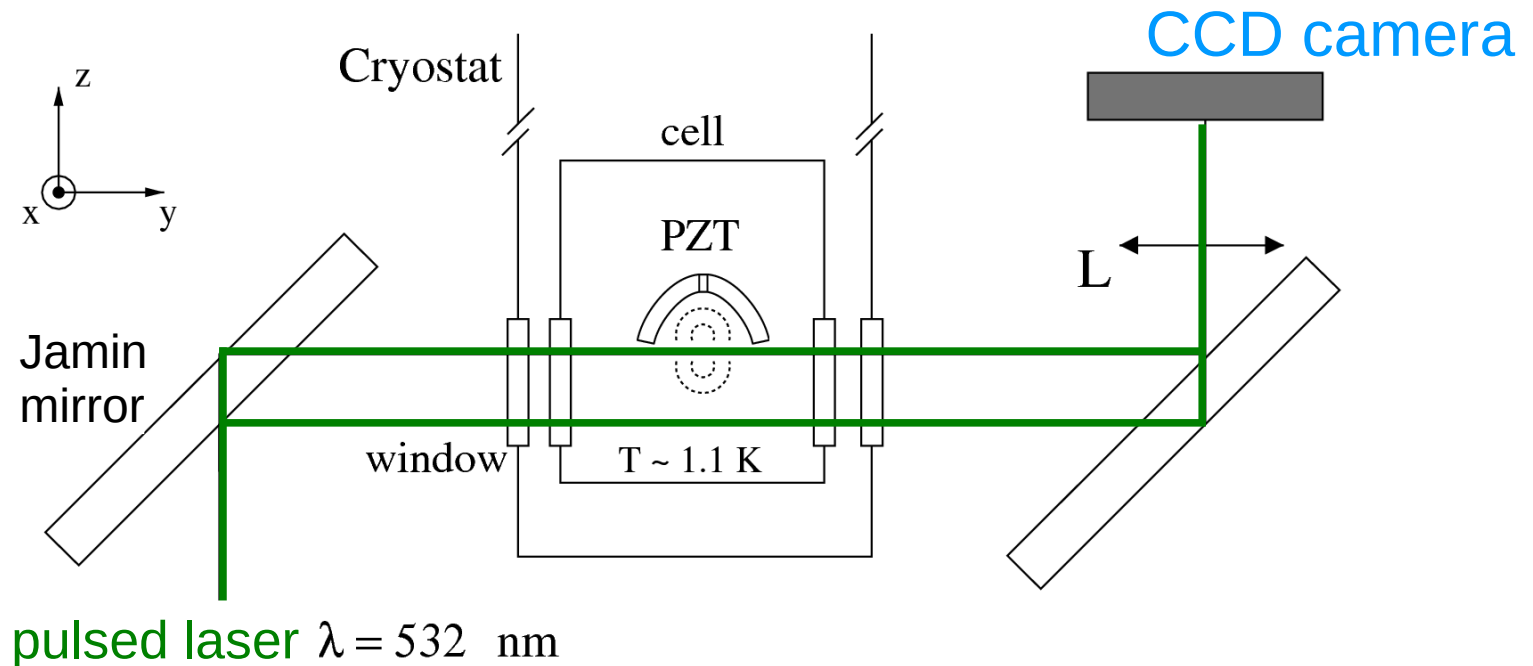
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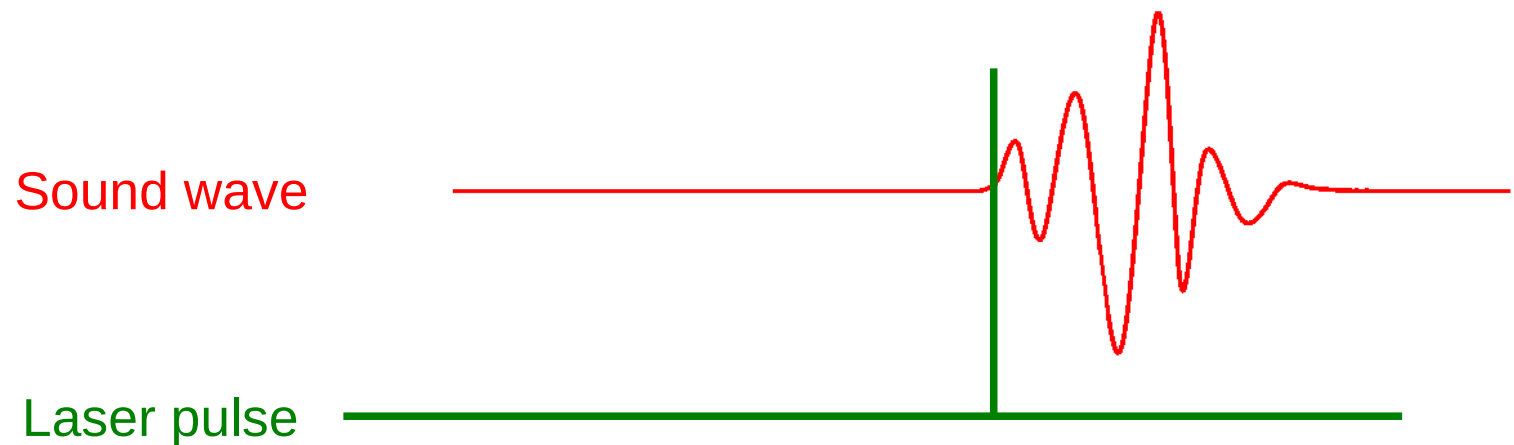


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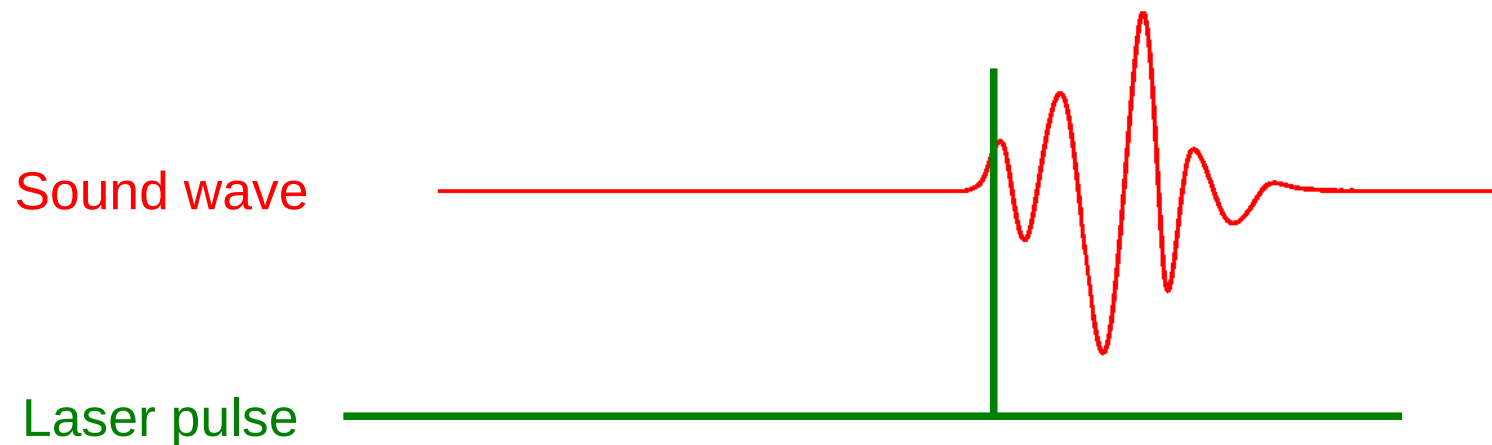
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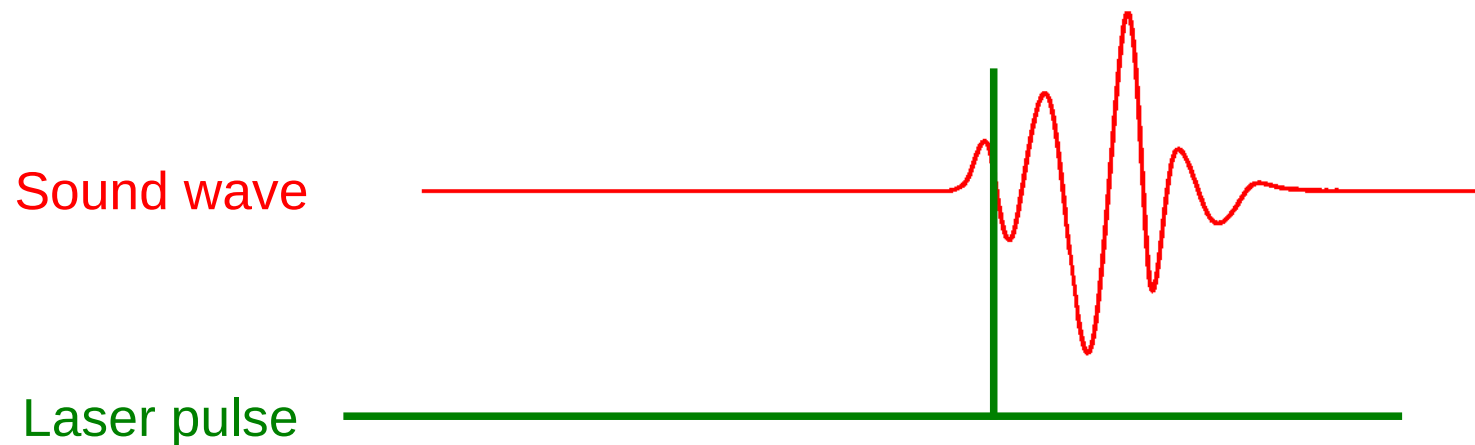
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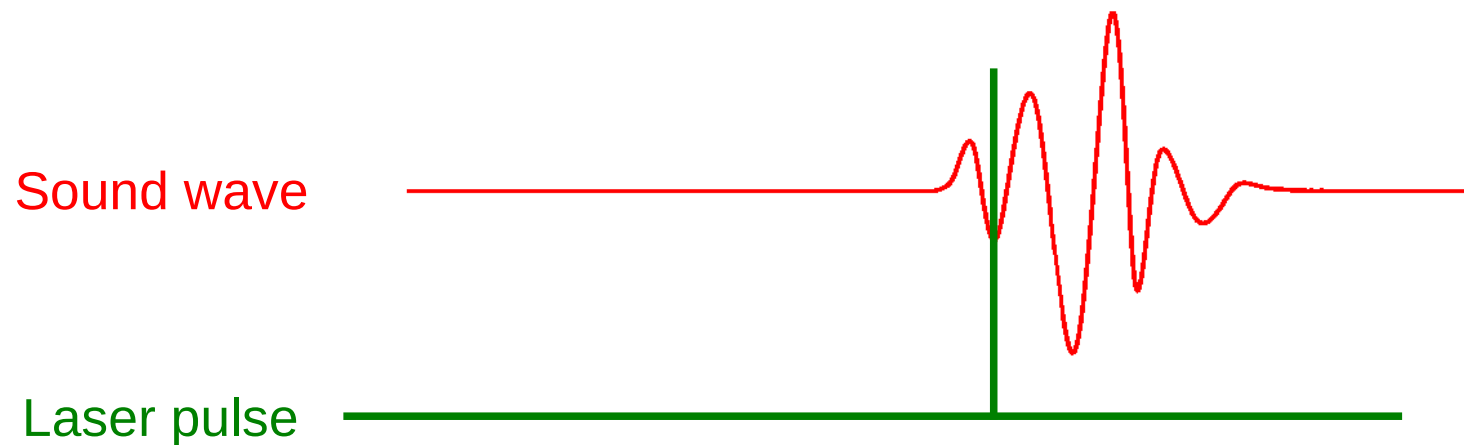
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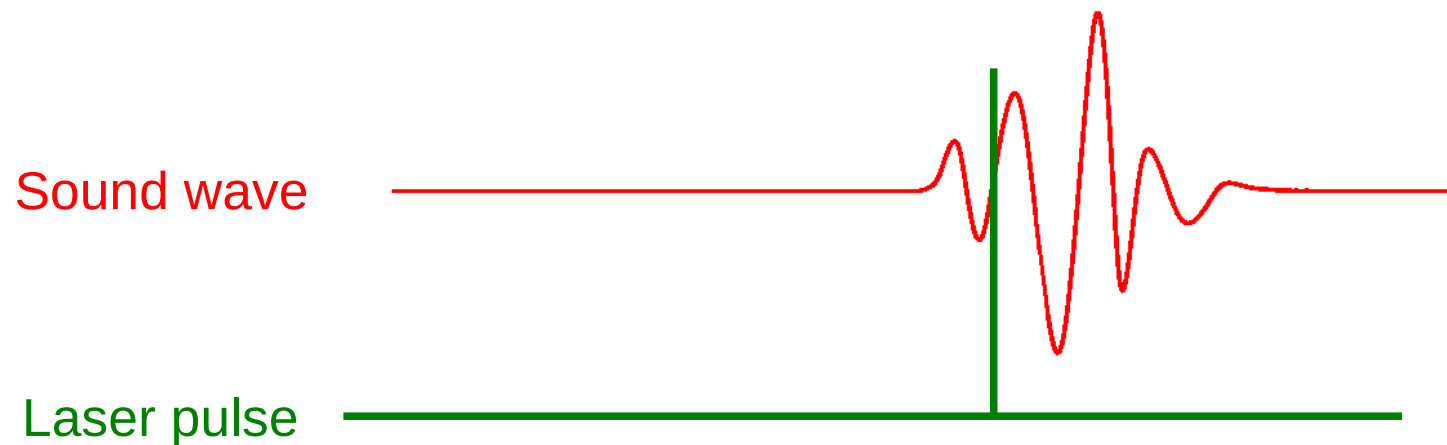


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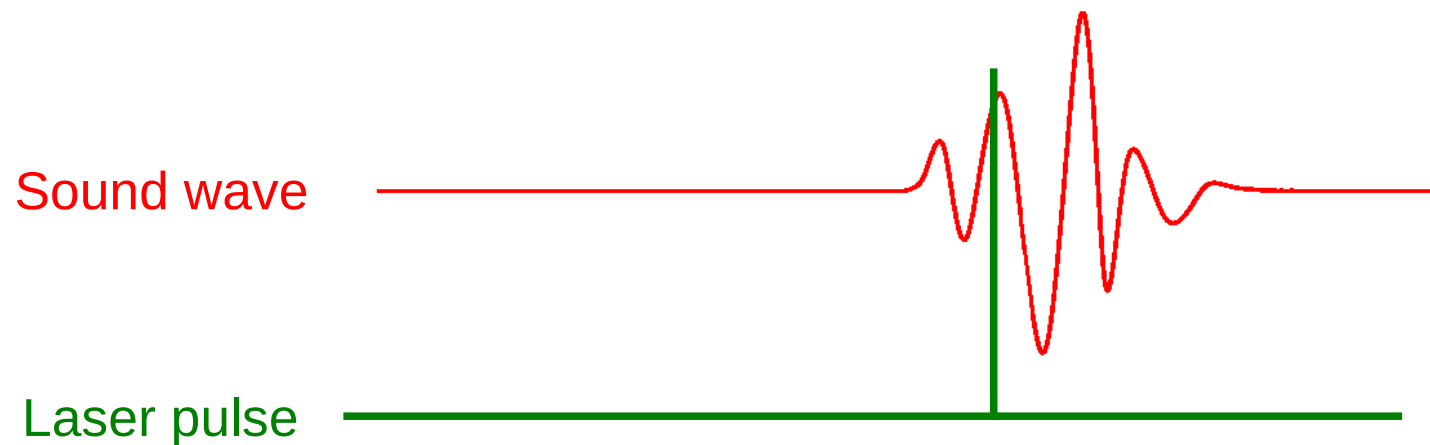
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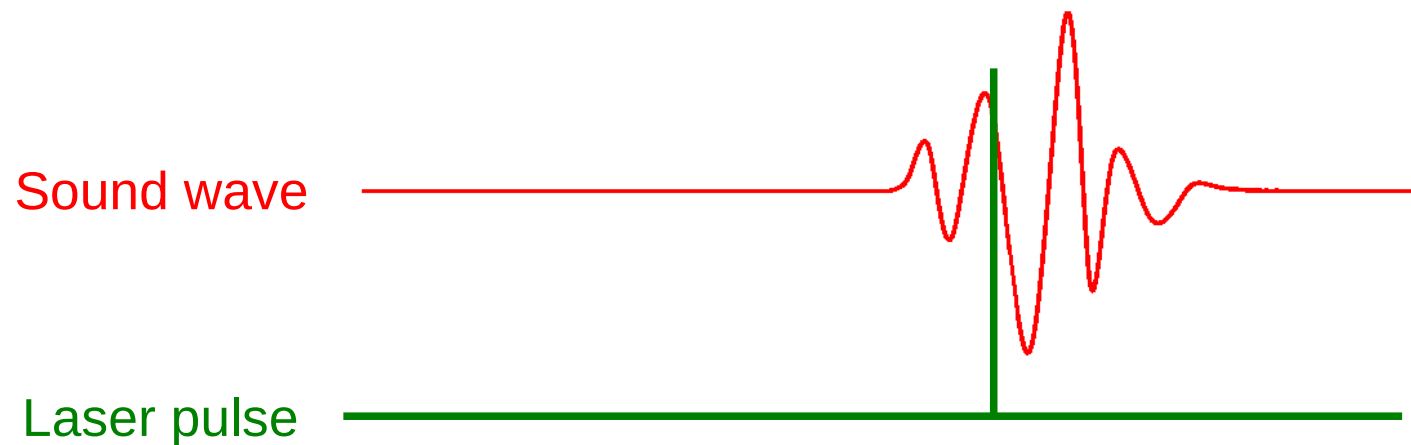
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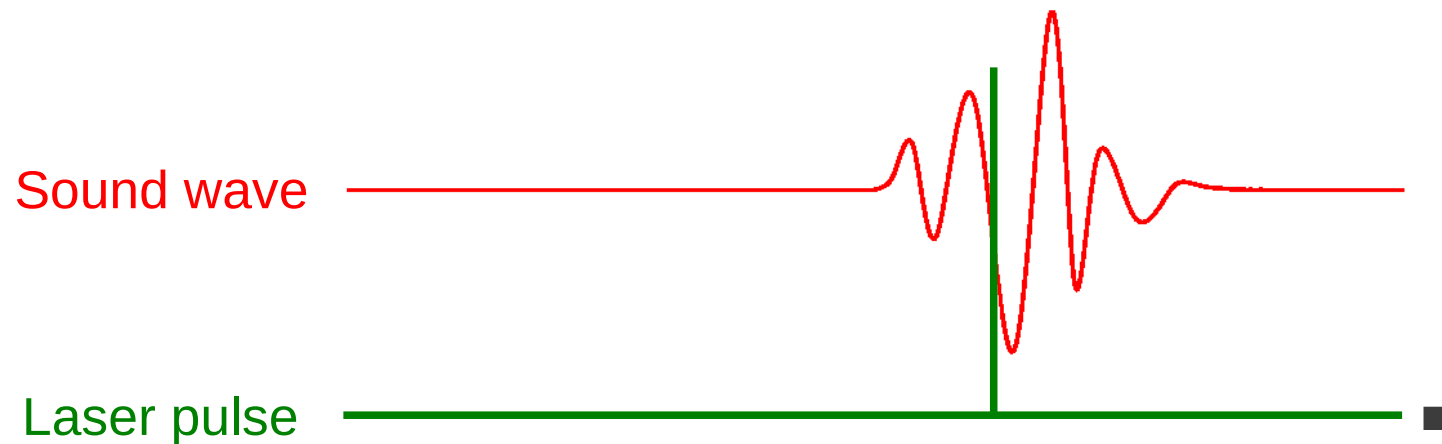
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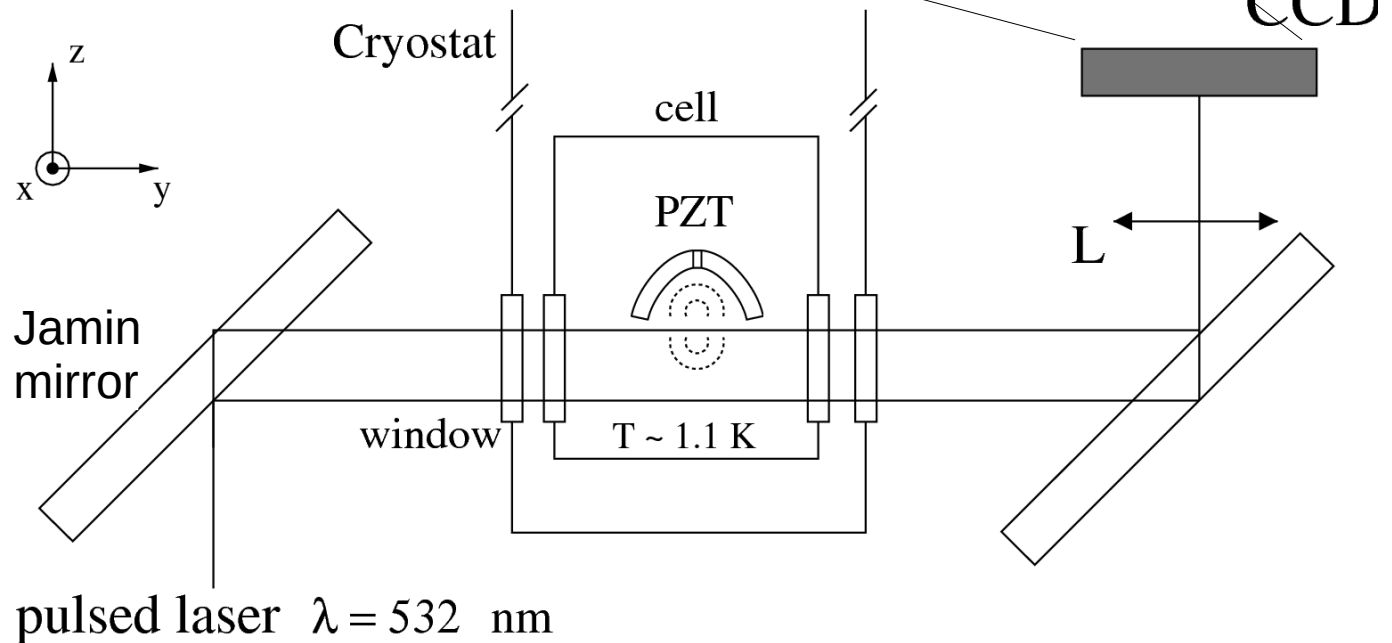
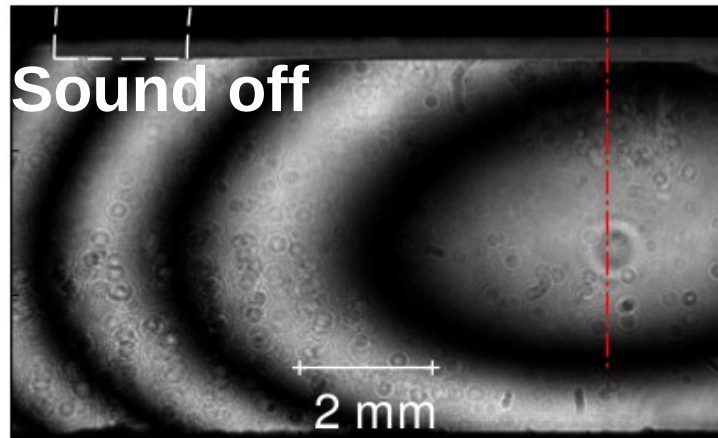
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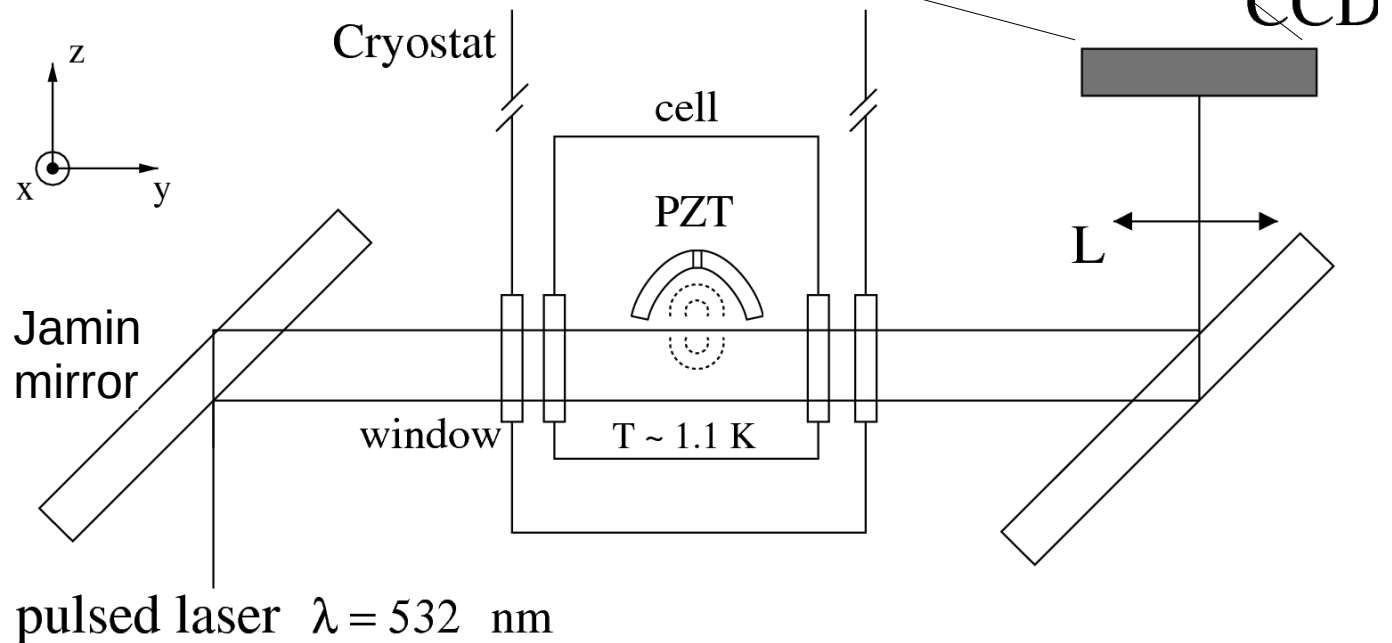
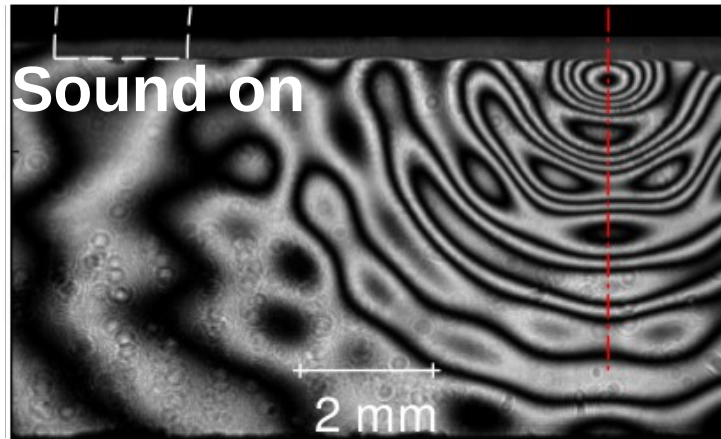
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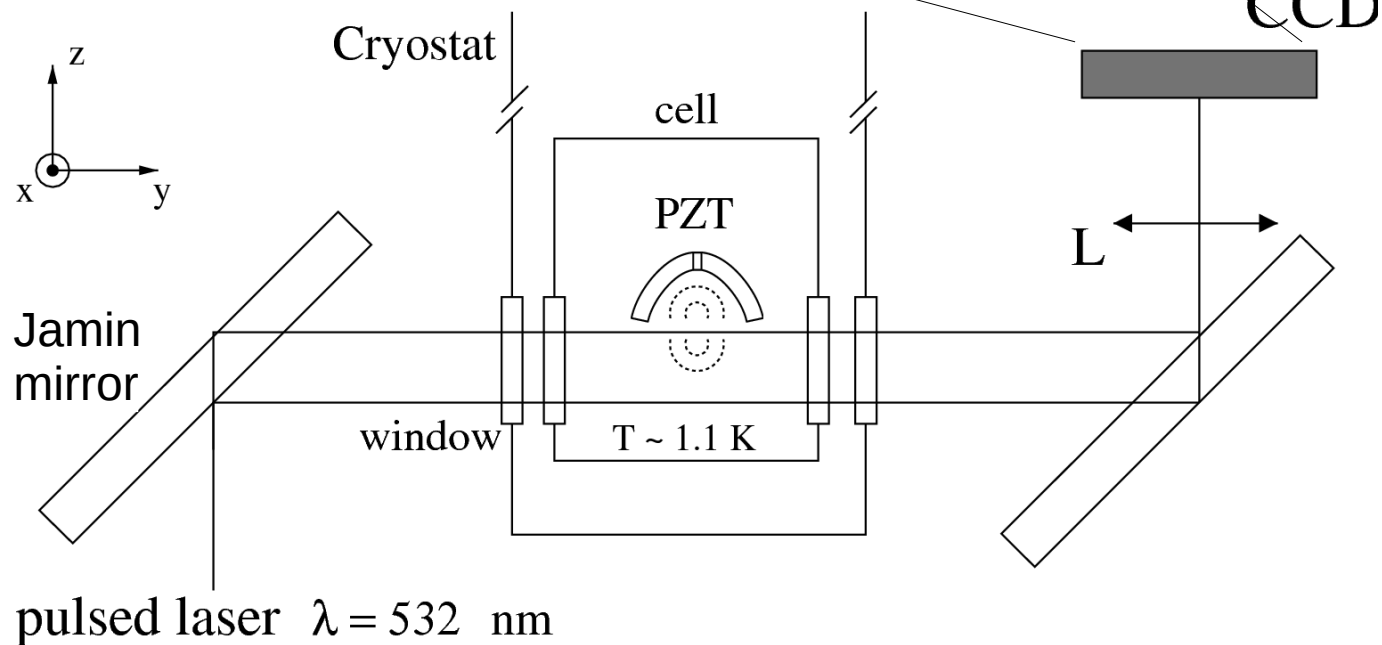
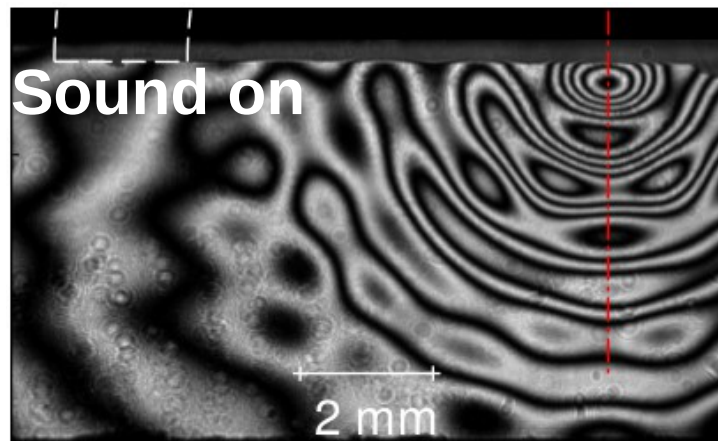
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Interferometric imaging setup



To compute $\delta\phi(x, z)$, pixels are treated independently



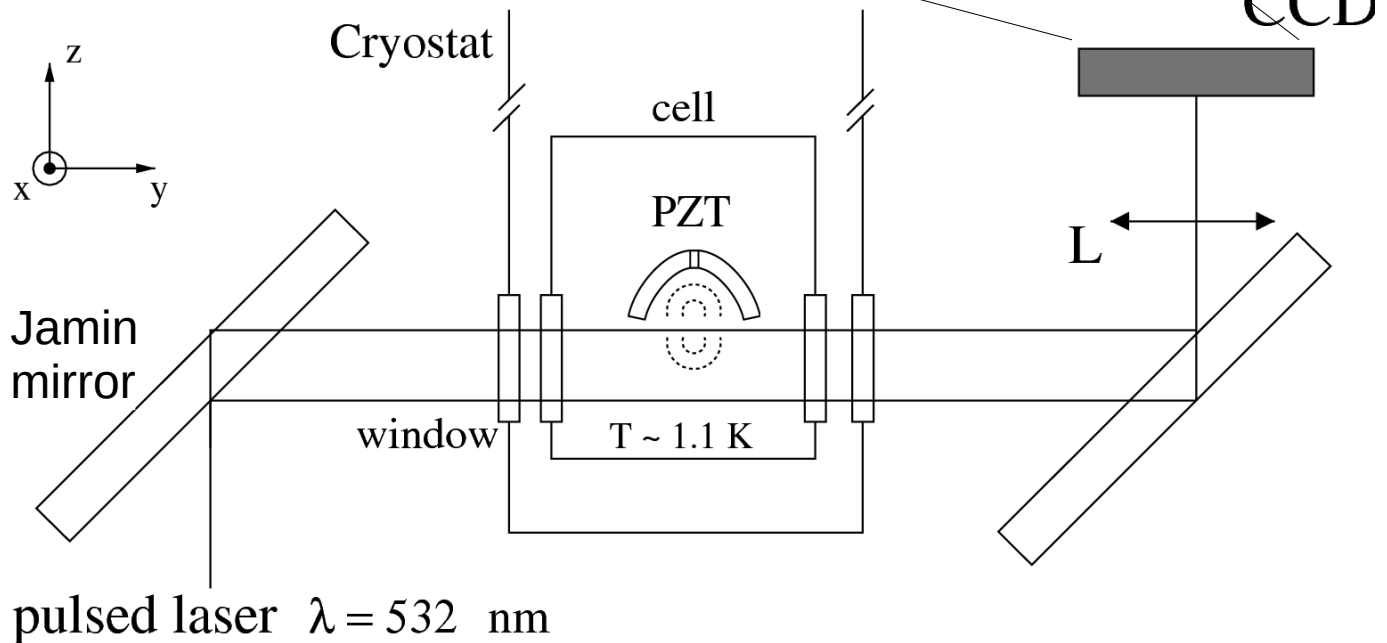
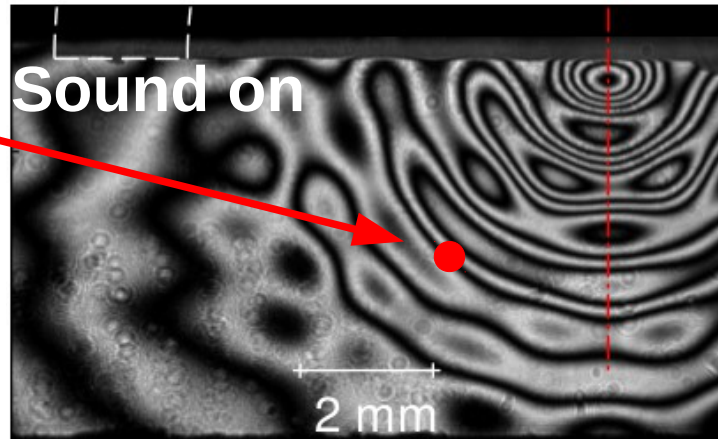
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To compute $\delta\phi(x, z)$, pixels are treated independently

This one



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► Intensity measured
by camera :

$$I = I_{eff} \left[1 + C_{eff} \cos (\delta\phi + \phi_0) \right]$$

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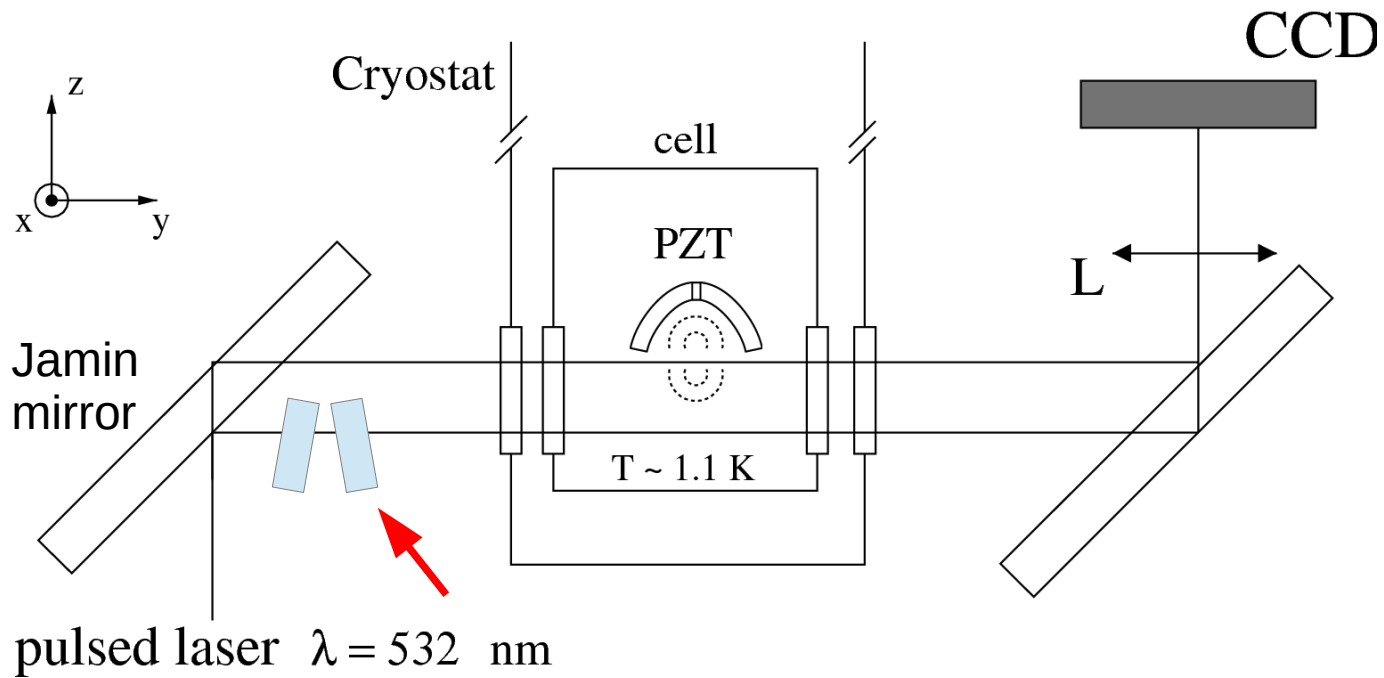
Phase map calculation



⁴He

► Intensity measured by camera :

$$I(p) = I_{eff} \left[1 + C_{eff} \cos (\delta\phi + \phi_0 + \varphi(p)) \right]$$



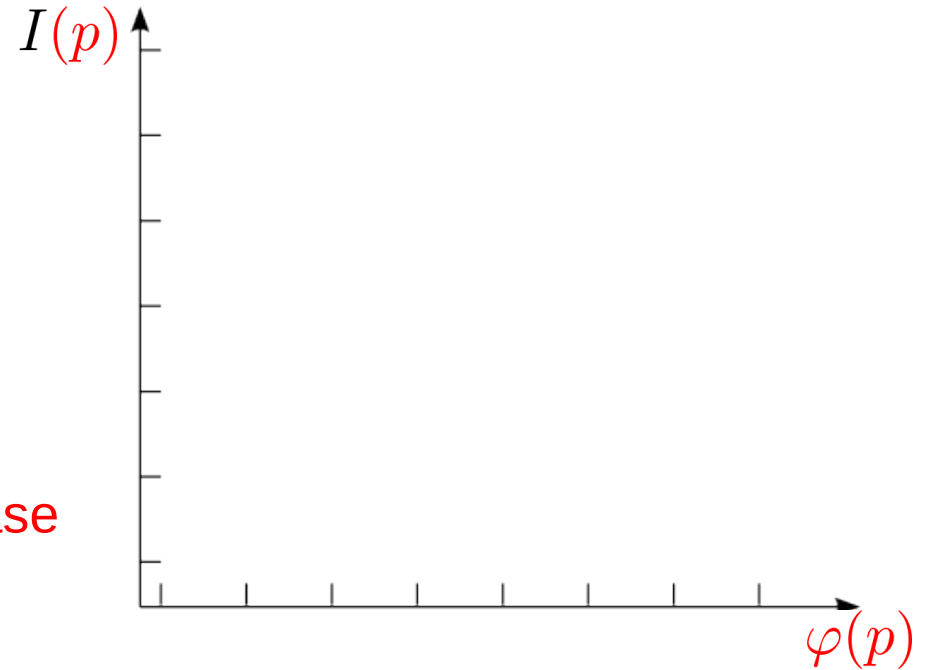
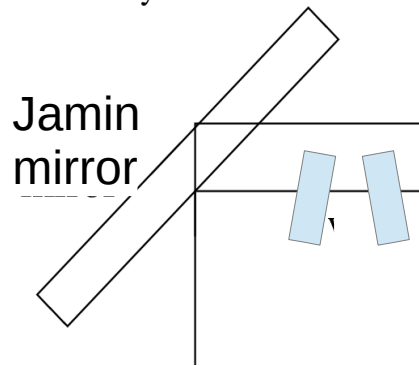
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Plates are tilted to vary the phase

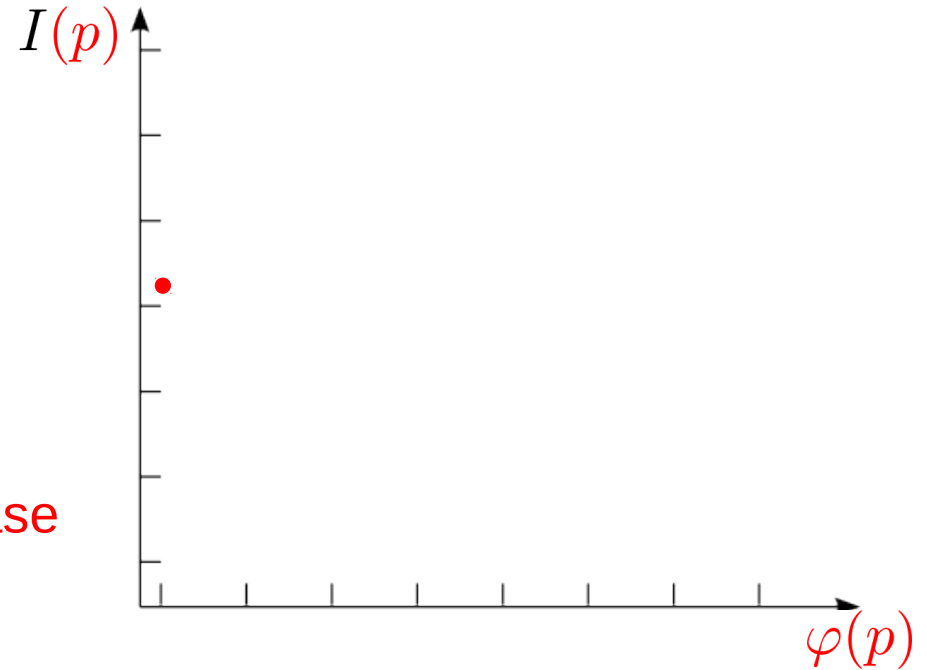
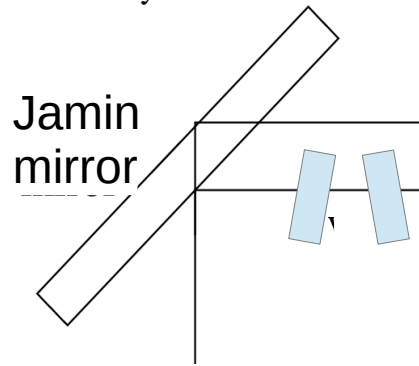
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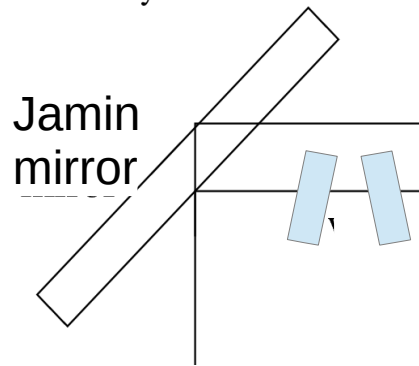
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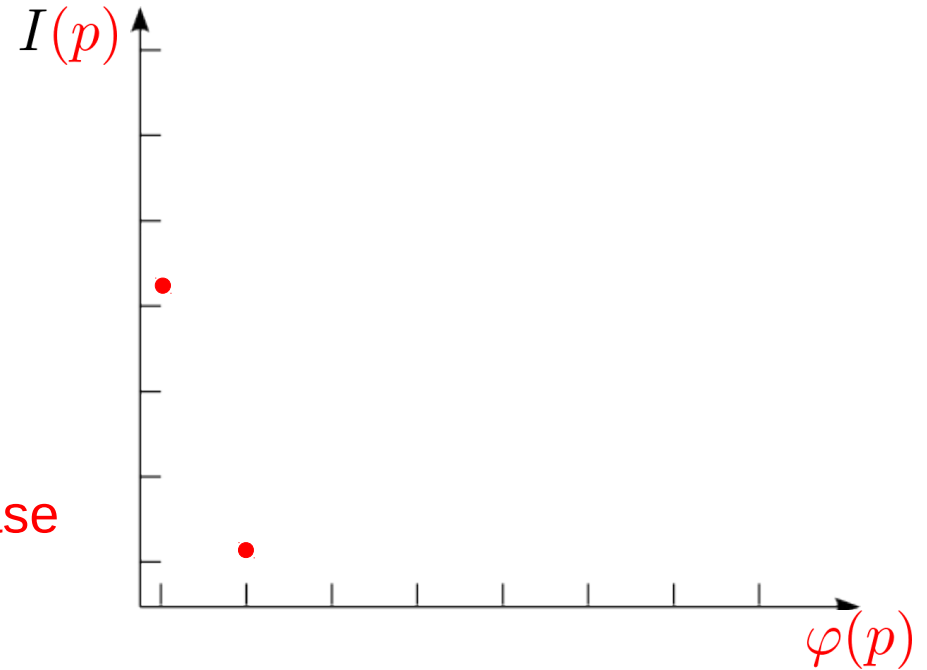
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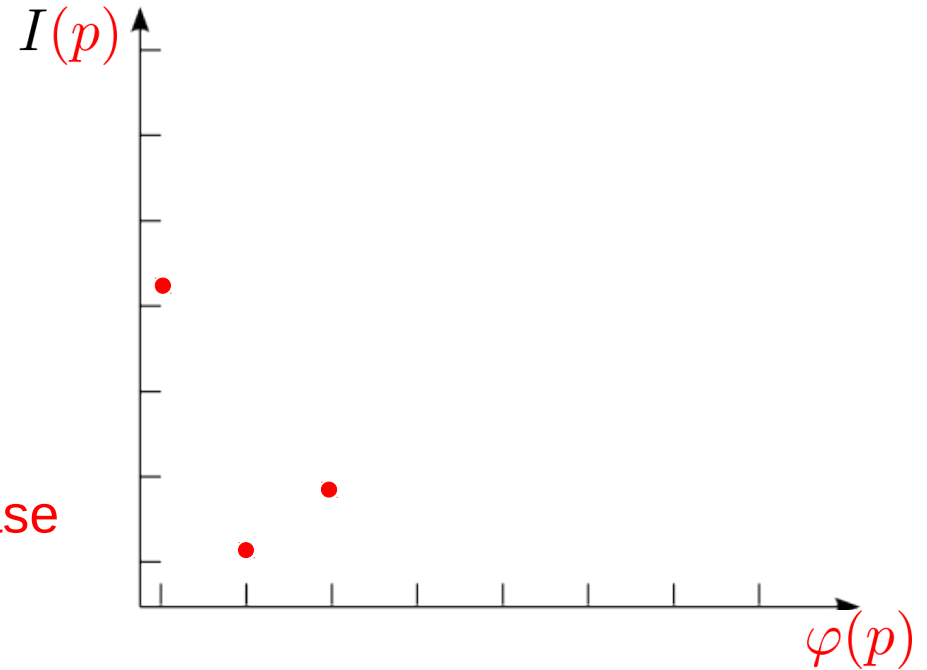
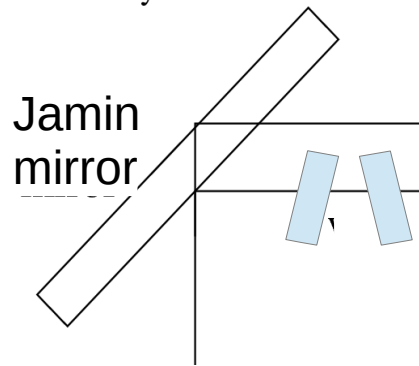
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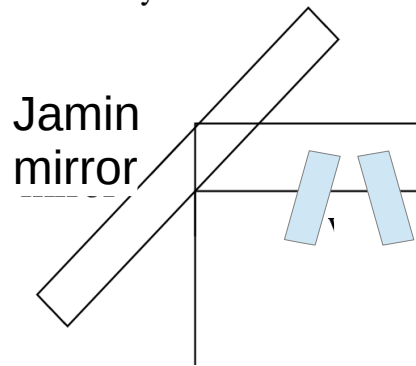
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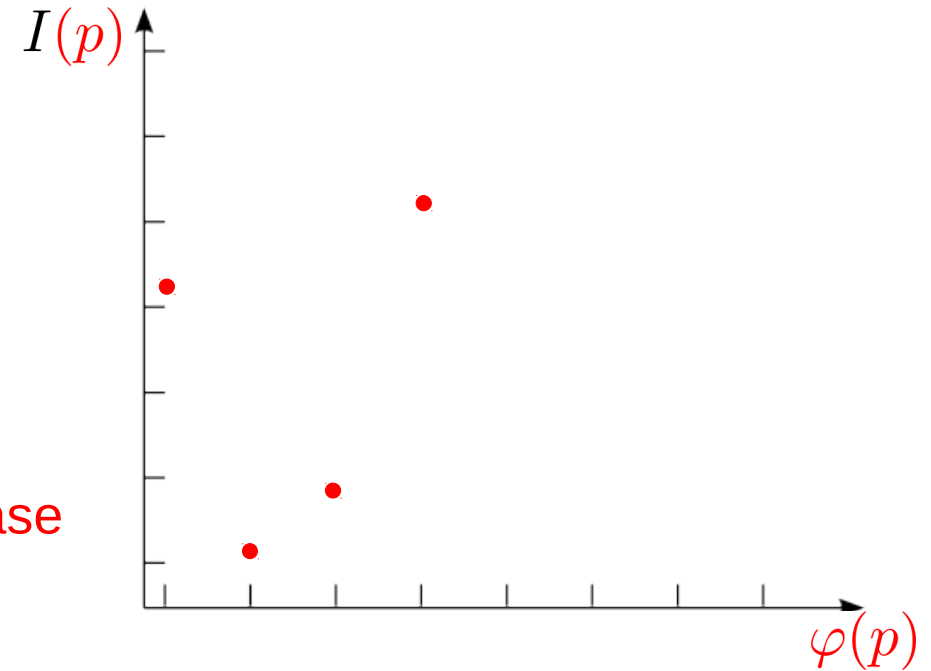
► Intensity measured by camera :

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Jamin mirror

Plates are tilted to vary the phase



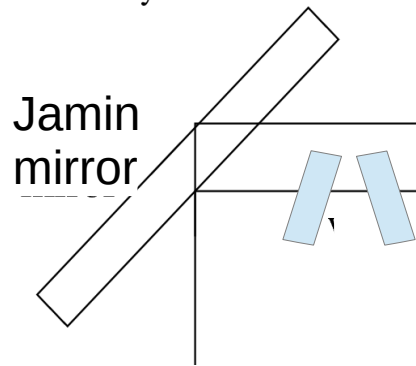
Phase map calculation



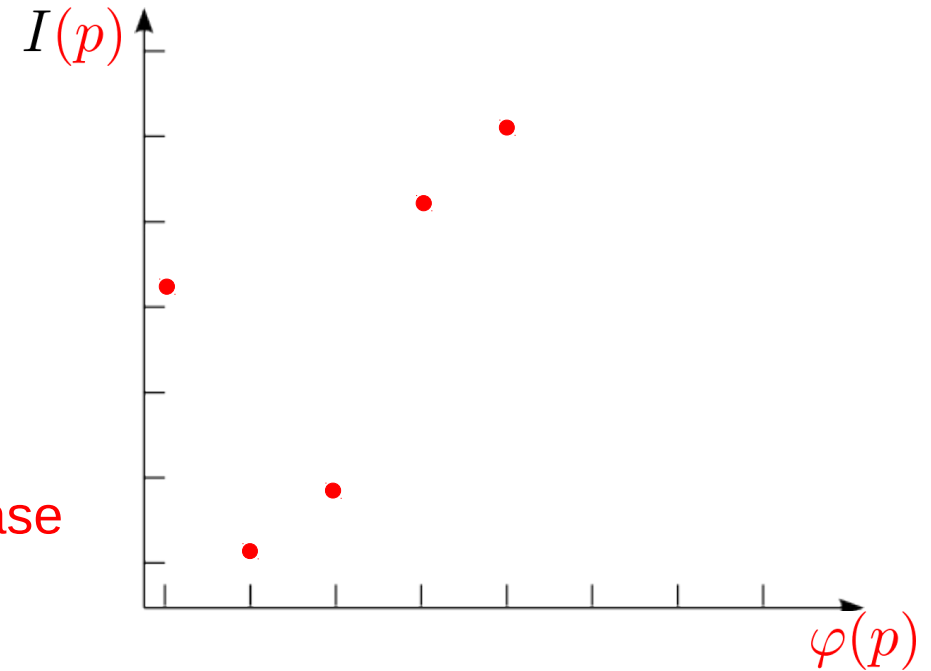
⁴He

► Intensity measured by camera :

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Plates are tilted to vary the phase



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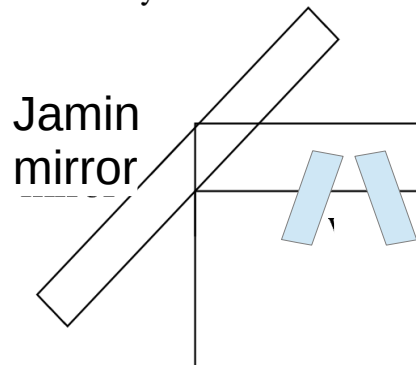
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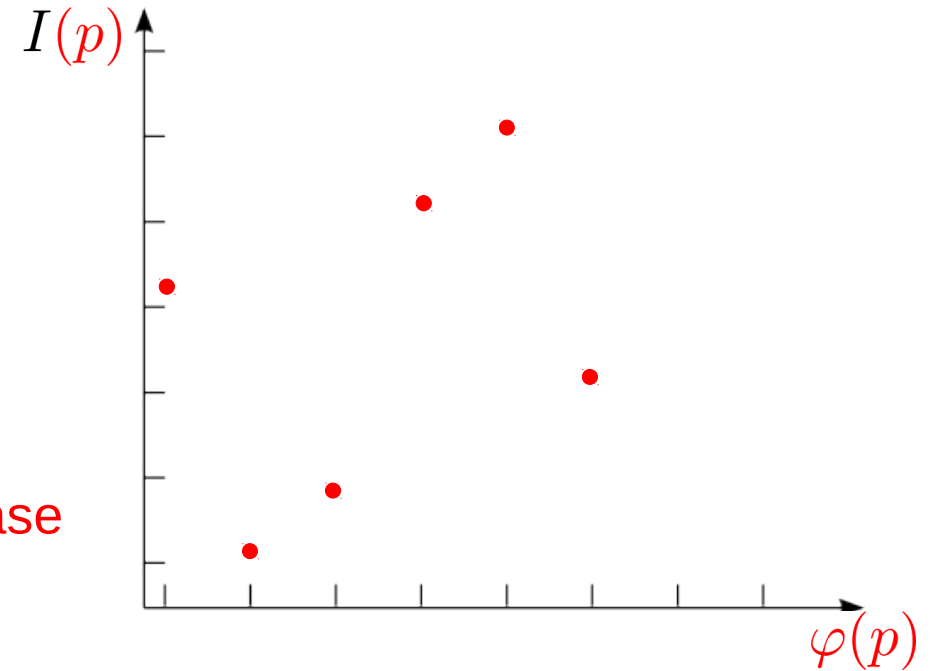
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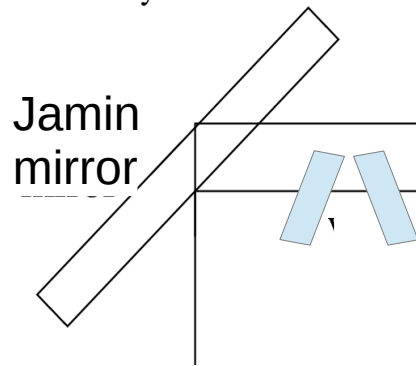
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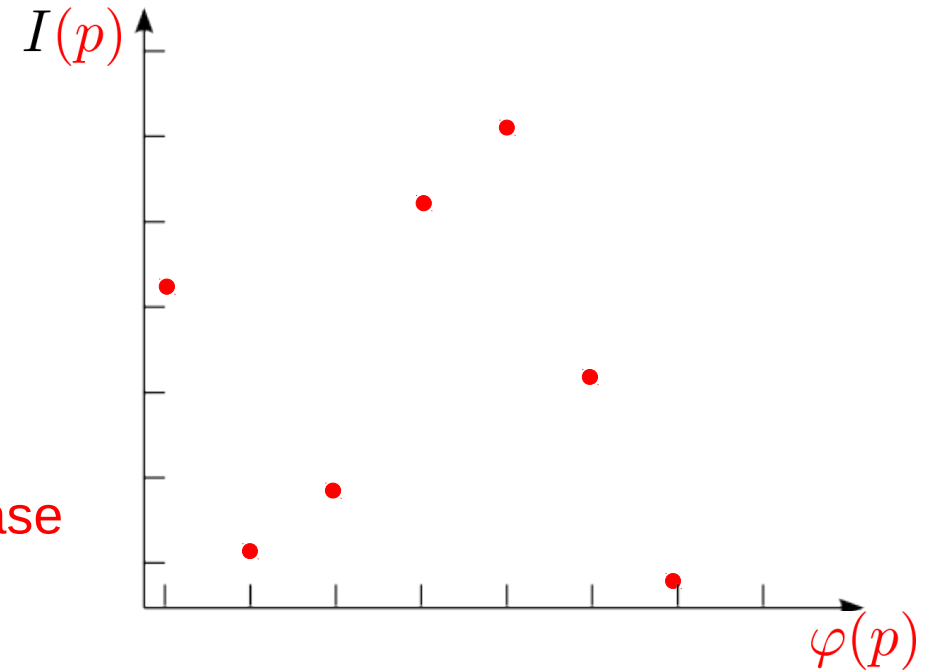
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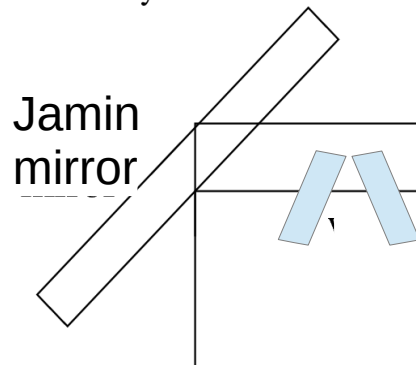
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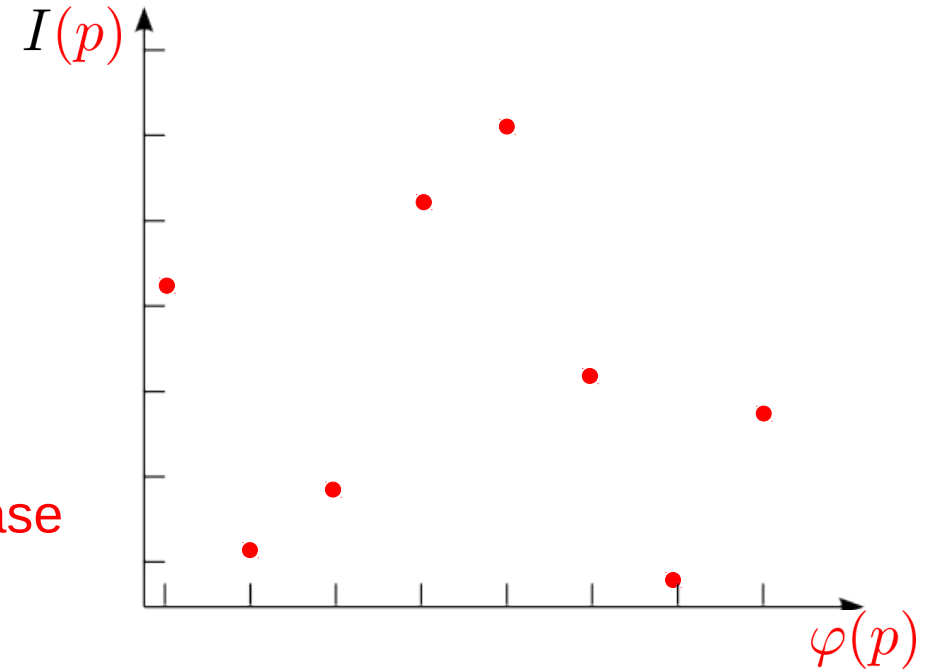
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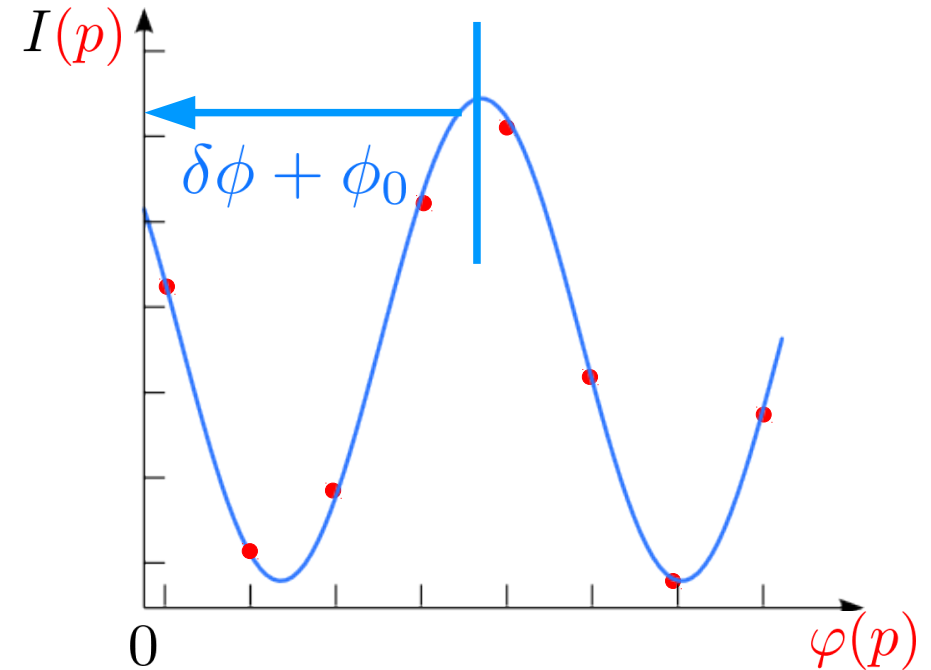
⁴He

► Intensity measured by camera :

$$I(p) = I_{eff} \left[1 + C_{eff} \cos (\delta\phi + \phi_0 + \varphi(p)) \right]$$

► Linear fit with $I(p)$ gives :

- $\delta\phi + \phi_0$
- I_{eff}
- C_{eff}



Phase map calculation



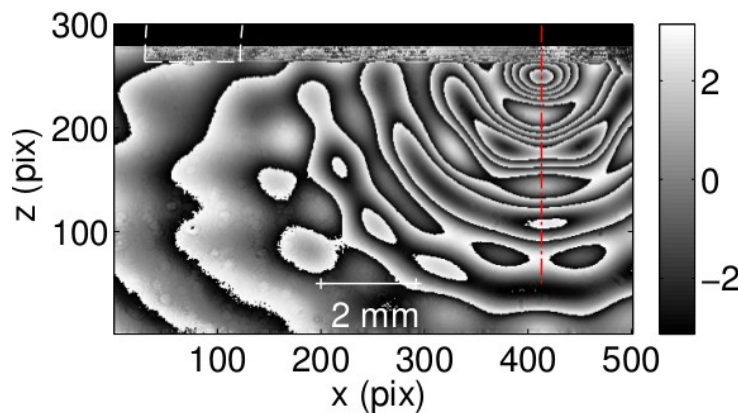
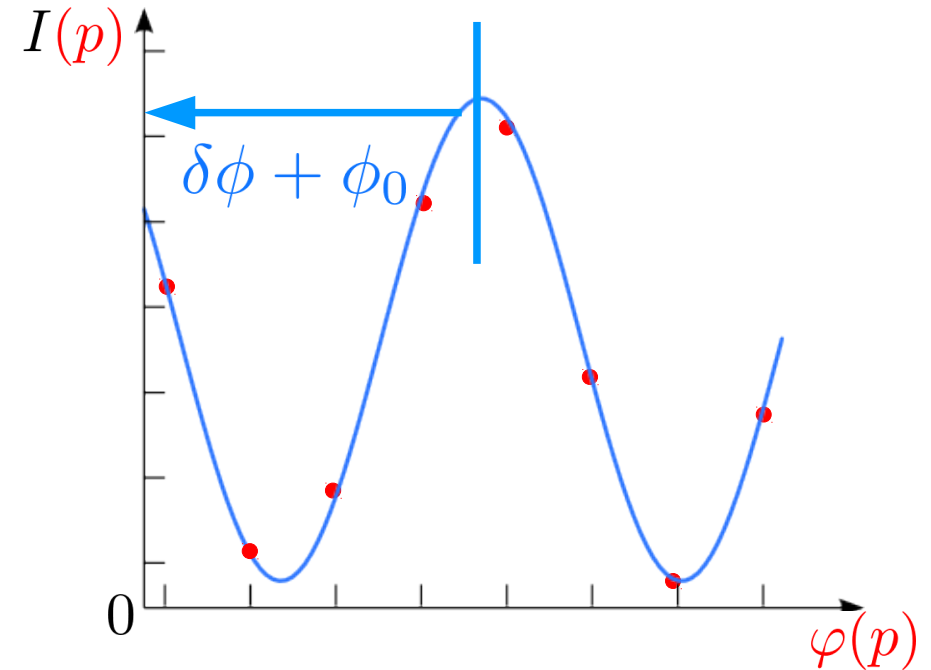
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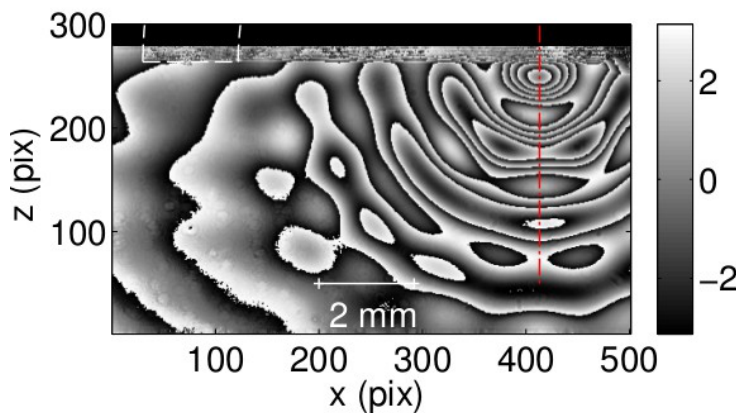
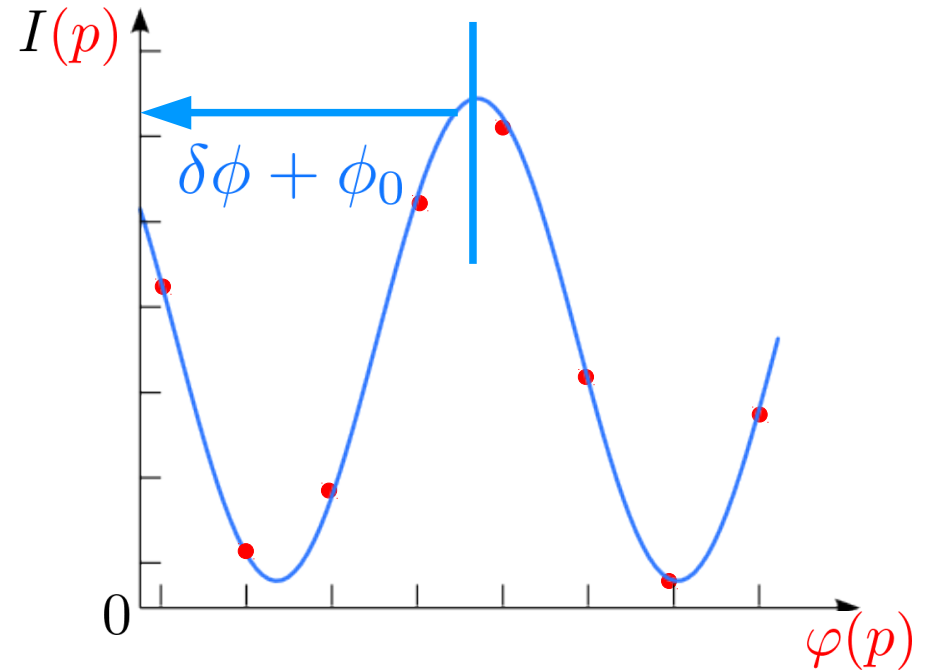
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► Intensity measured by camera :

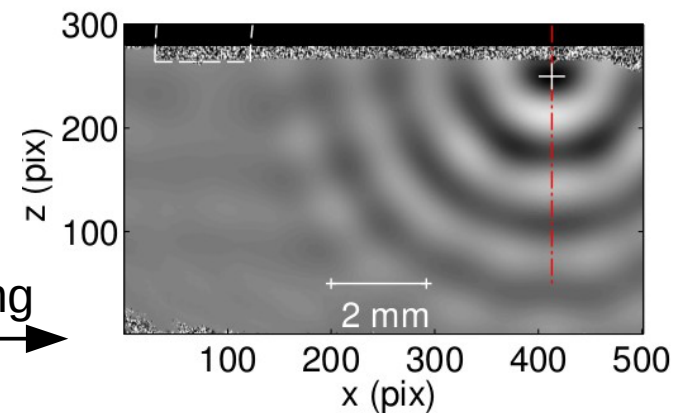
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- $\delta\phi + \phi_0$
- I_{eff}
- C_{eff}



Temporal unwrapping



Refractive index map



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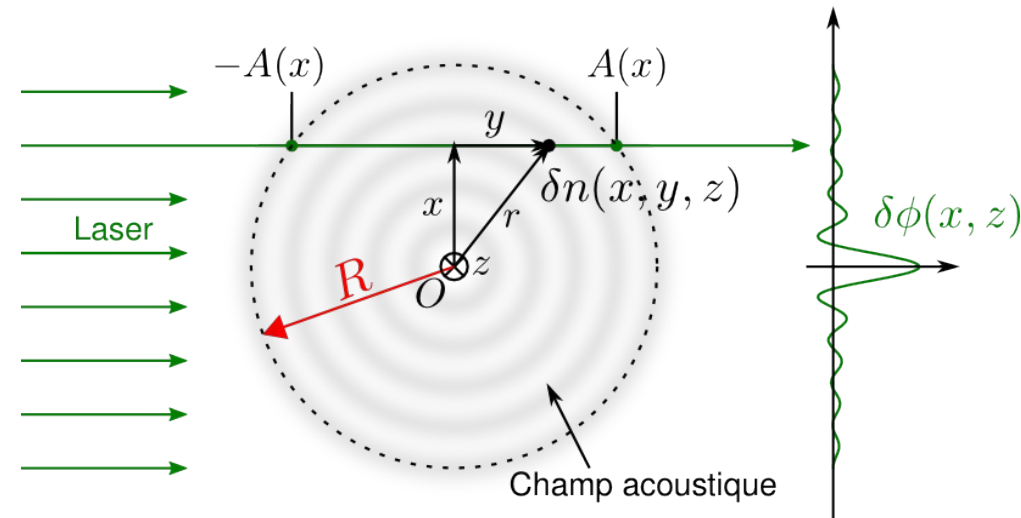
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► Phase $\delta\phi$ is the **integral of refractive index variations** along the beam path, i. e. the Abel transform of $\delta n(r, z)$

$$\delta\phi(x, z) = \frac{2\pi}{\lambda} \int_{-A(x)}^{A(x)} dy \delta n(\sqrt{x^2 + y^2}, z) \quad , \text{ avec } x^2 + A(x)^2 = R^2$$



► An **inverse Abel transform algorithm** has been developed to retrieve radial profile of $\delta n(r, z)$ from the phase maps



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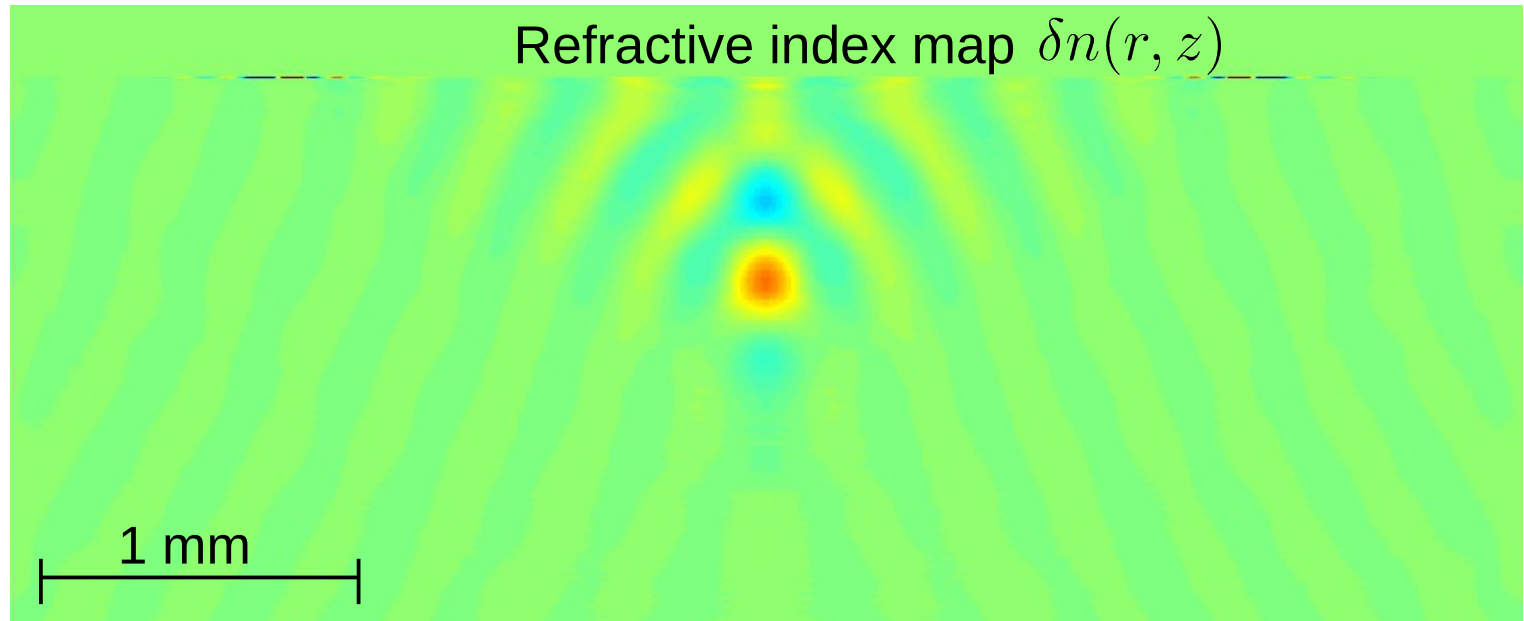
Sound focusing in helium-4 monocrystal



- Does the wavesurface emitter focus the sound wave correctly?

Experiment in helium-4 monocrystal : $T_0 = 1.20$ K and $P_0 = 25.6$ bar

Refractive index map $\delta n(r, z)$

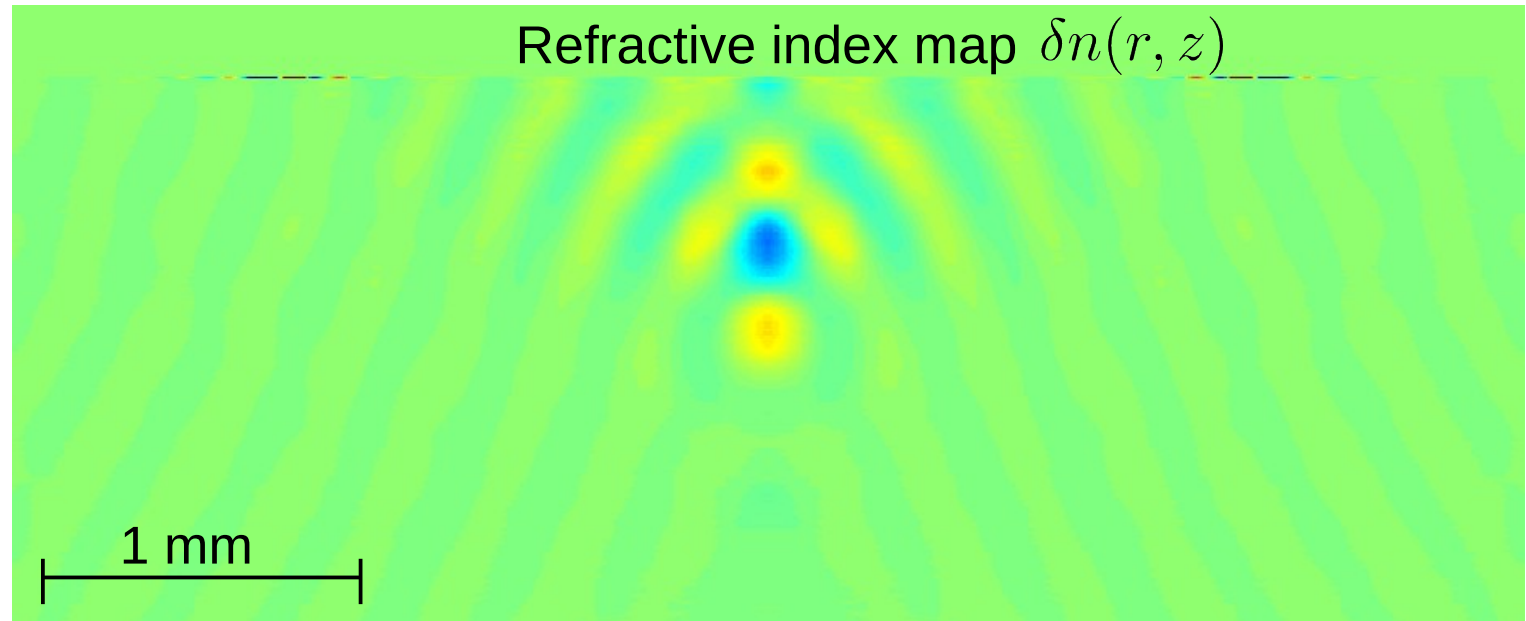


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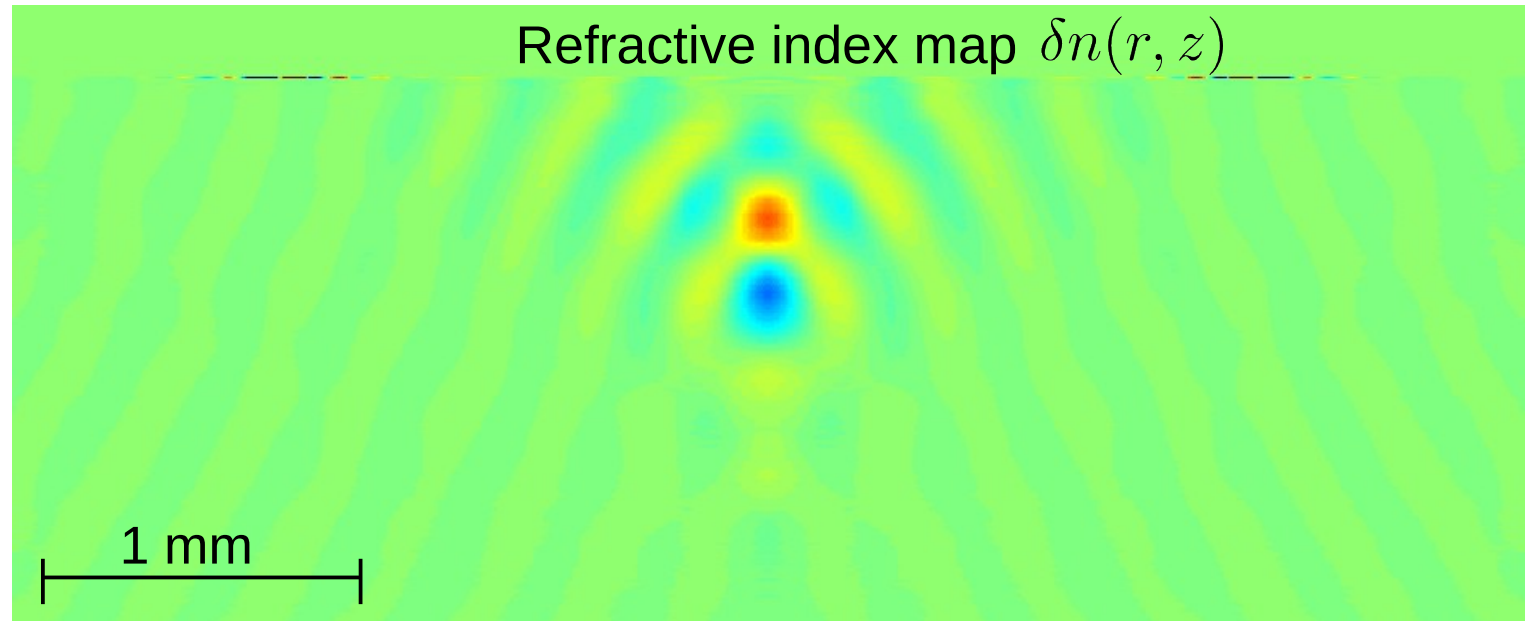


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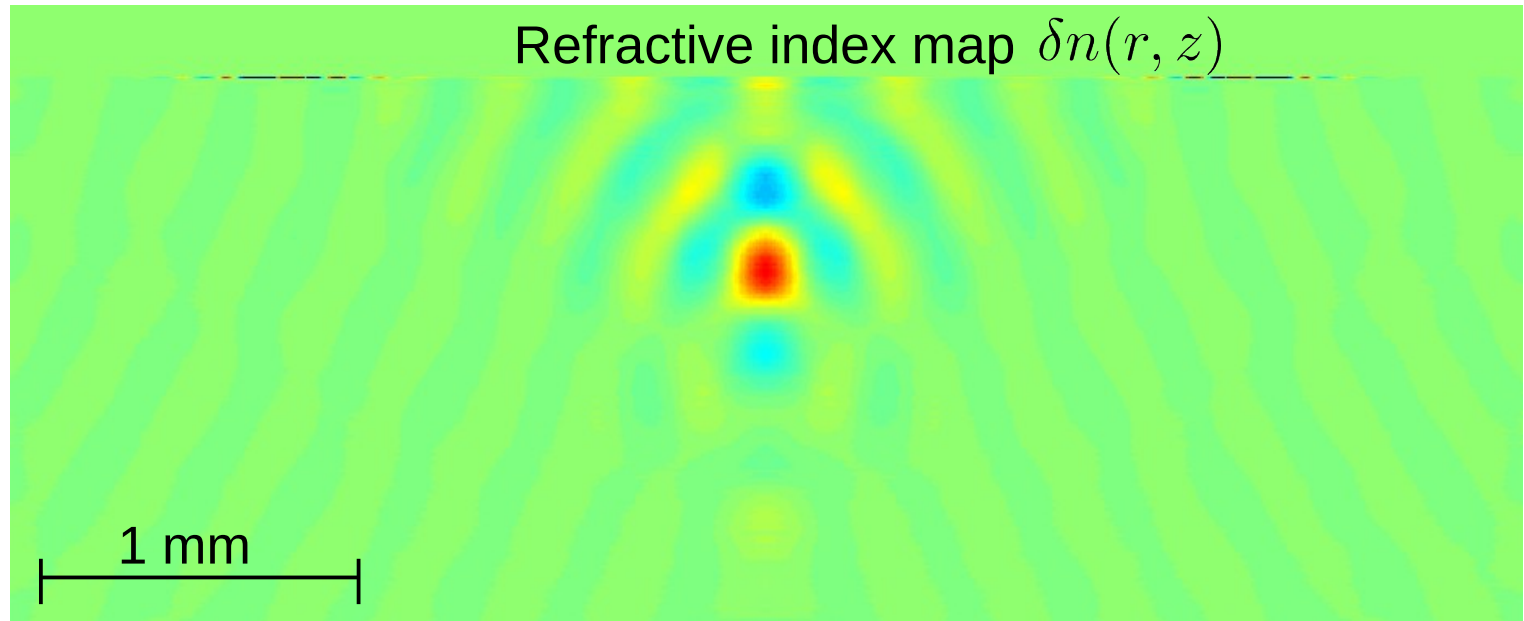
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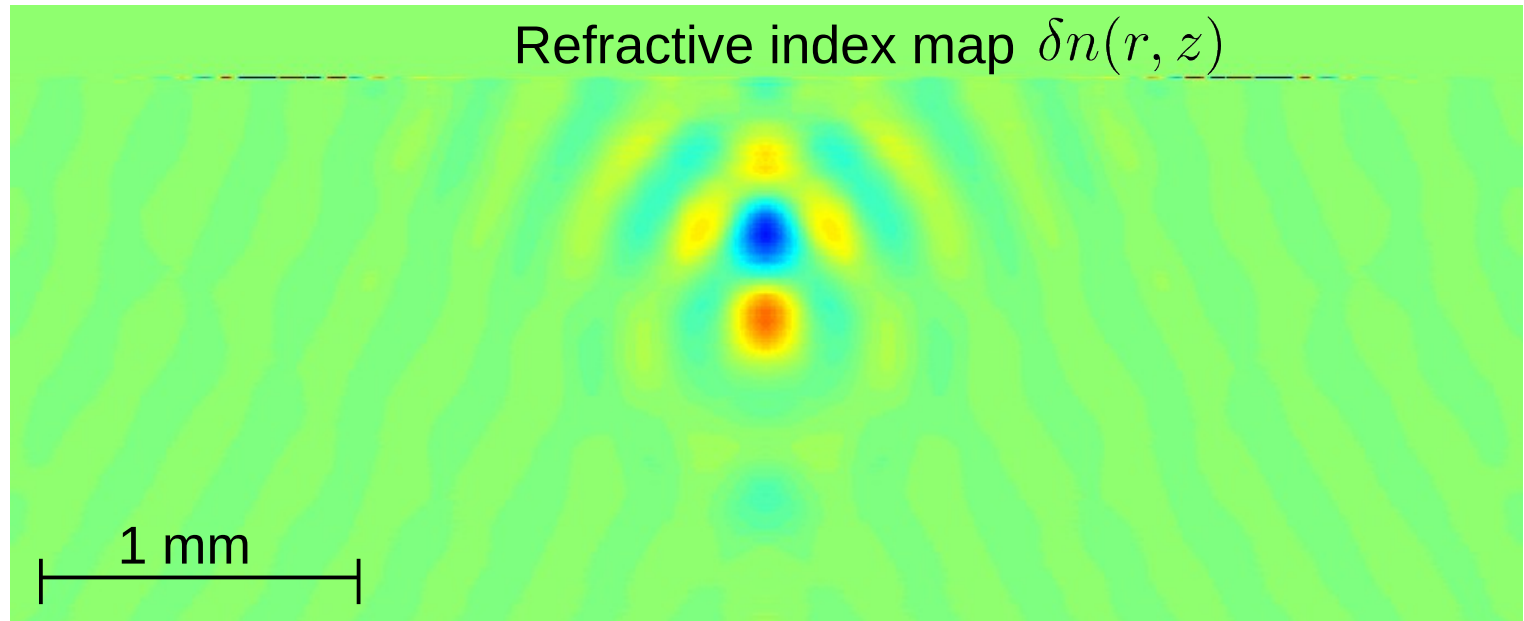
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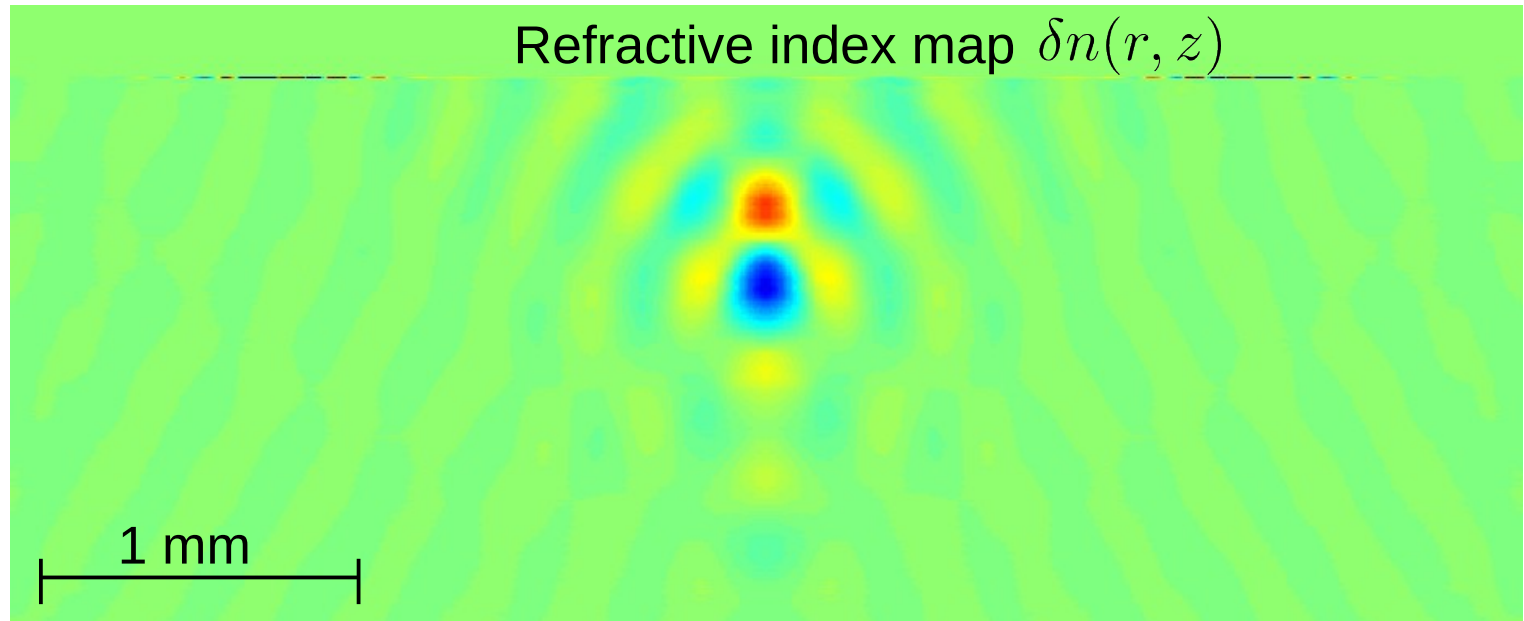
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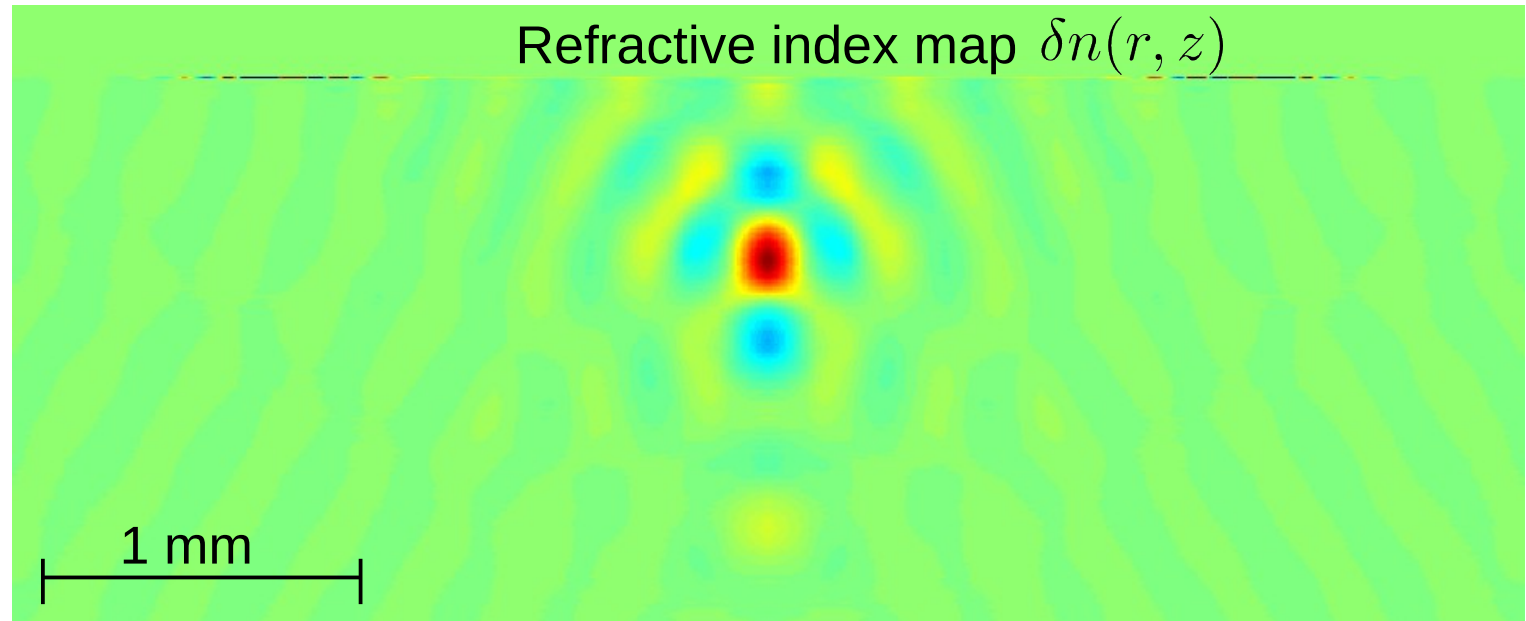


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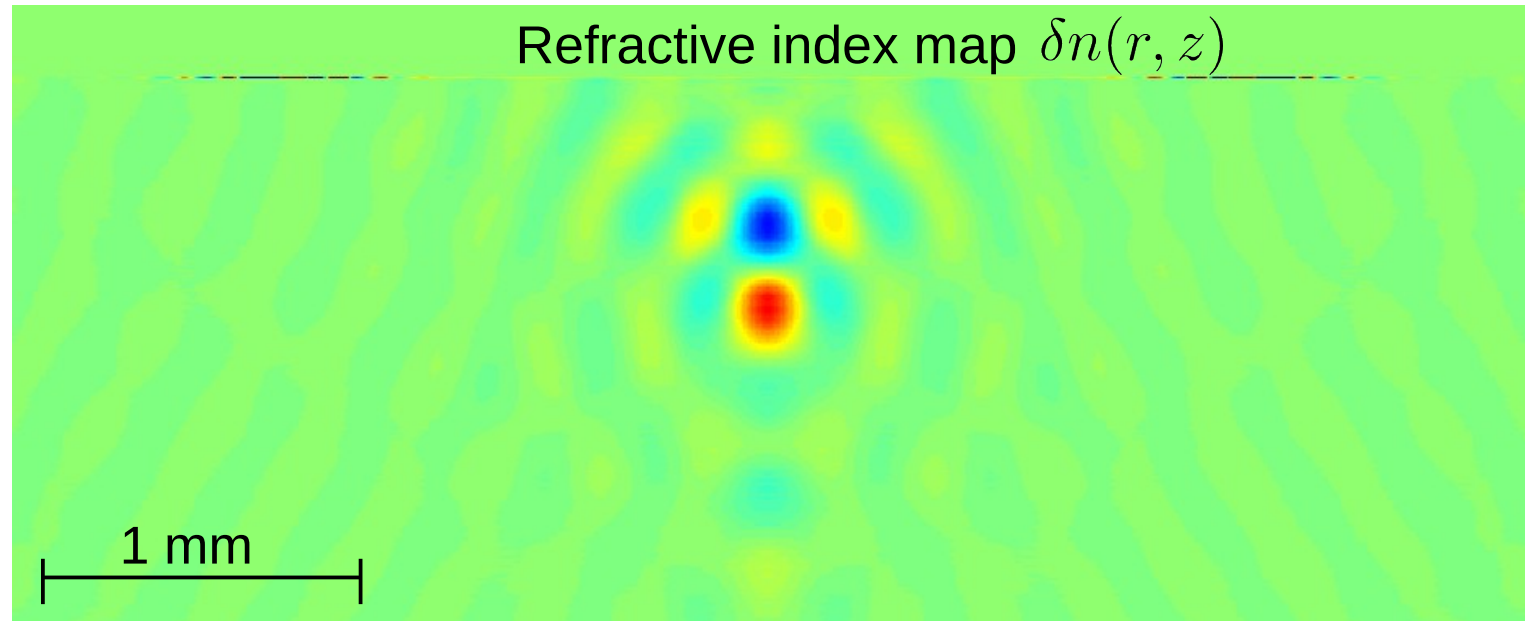


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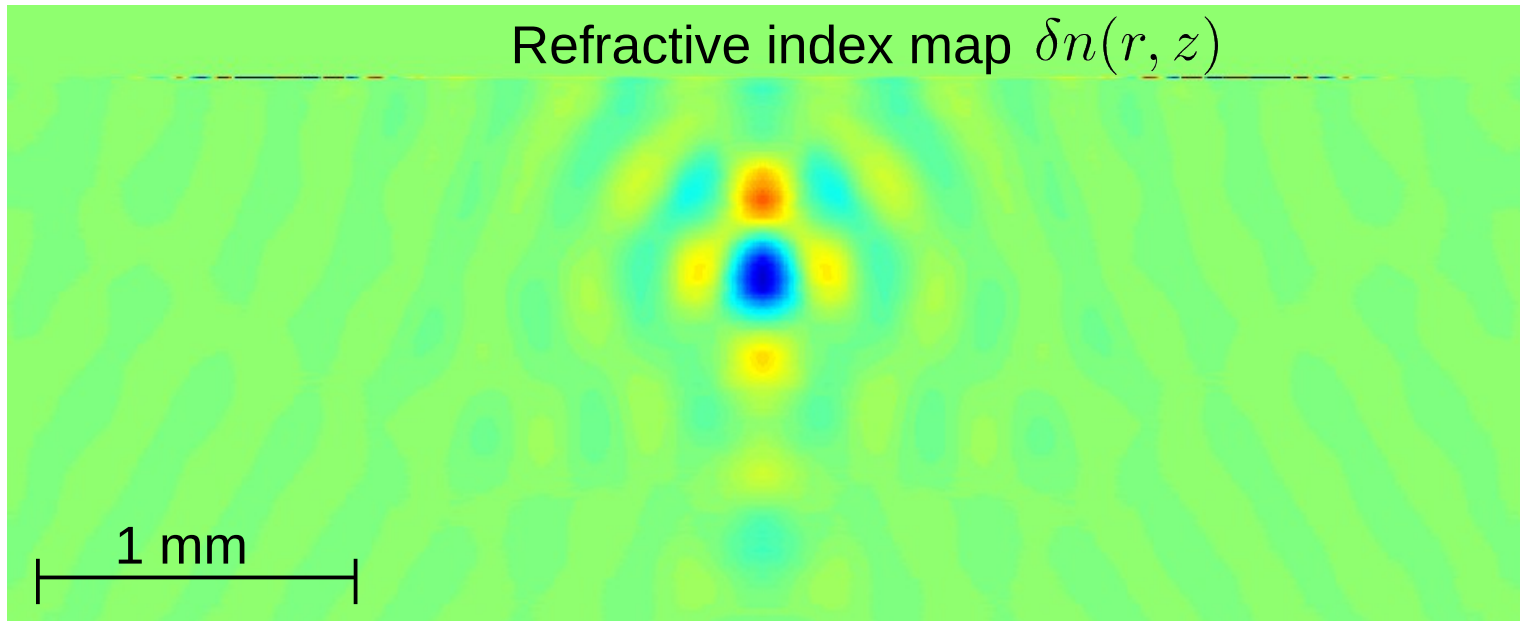
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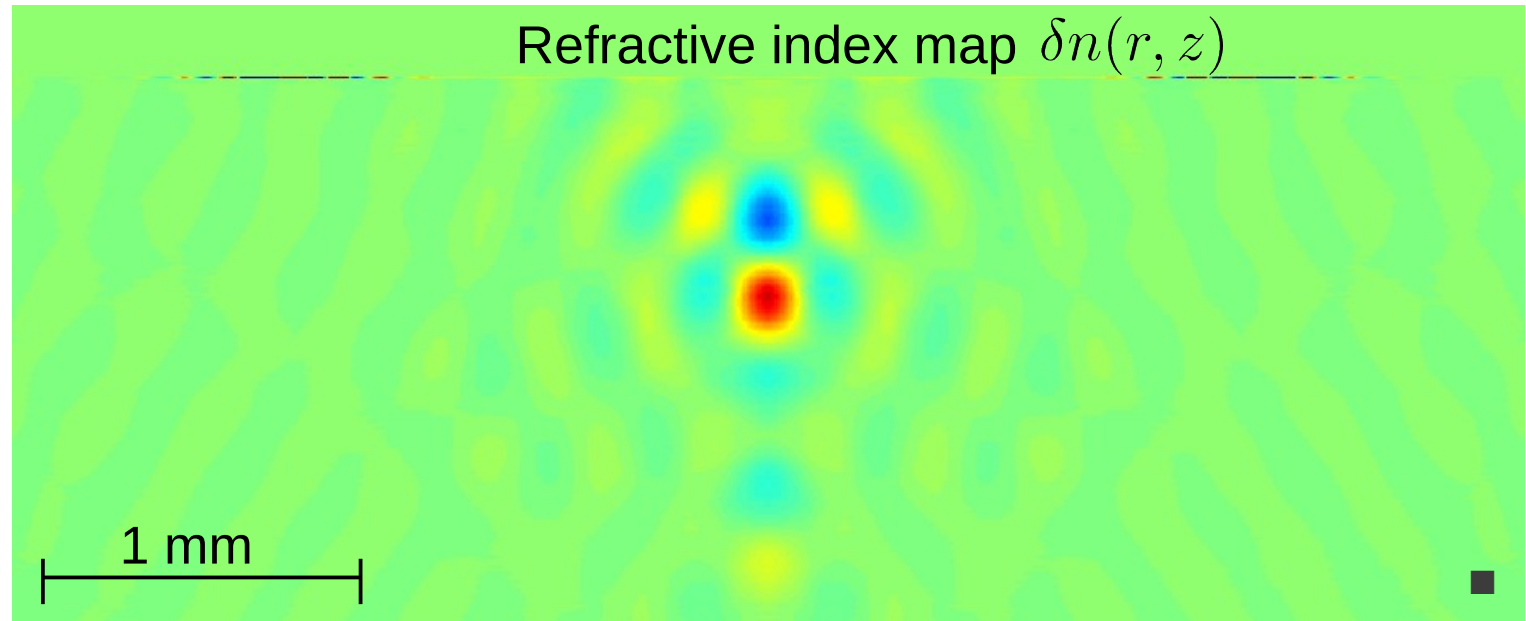


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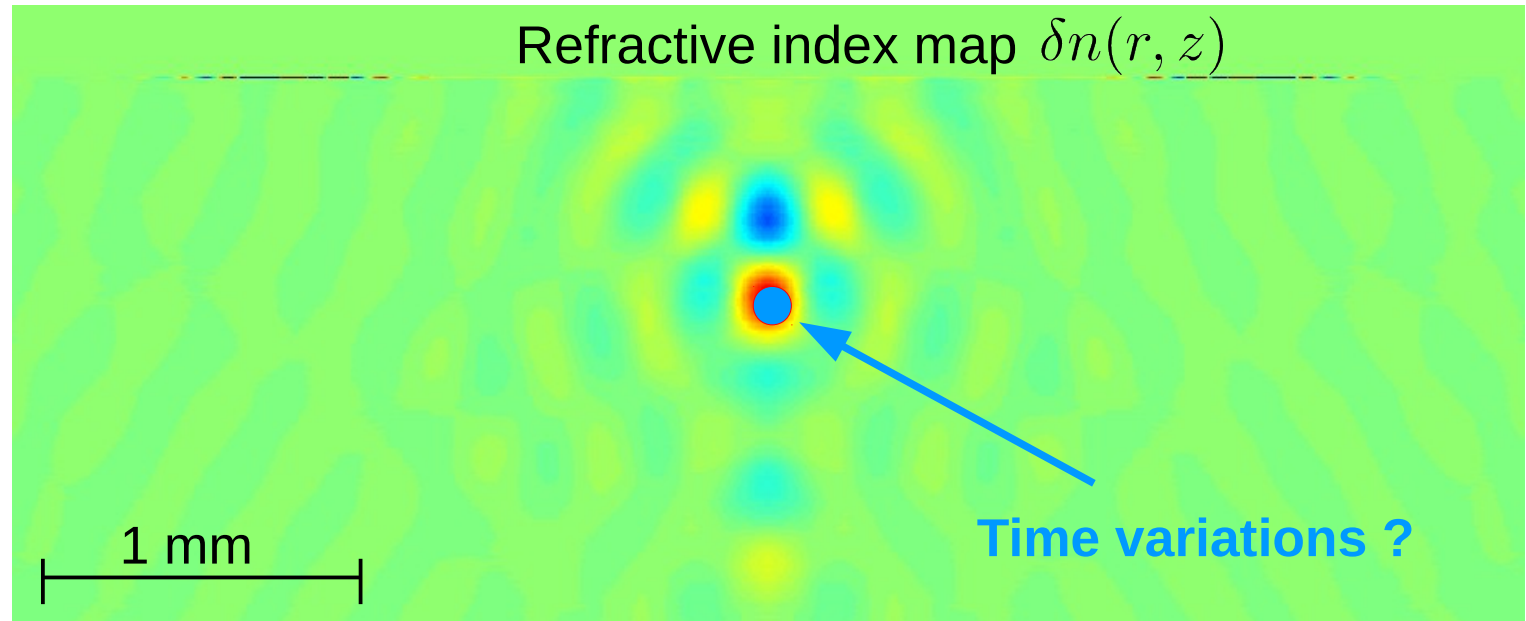


Sound focusing in helium-4 monocrystal



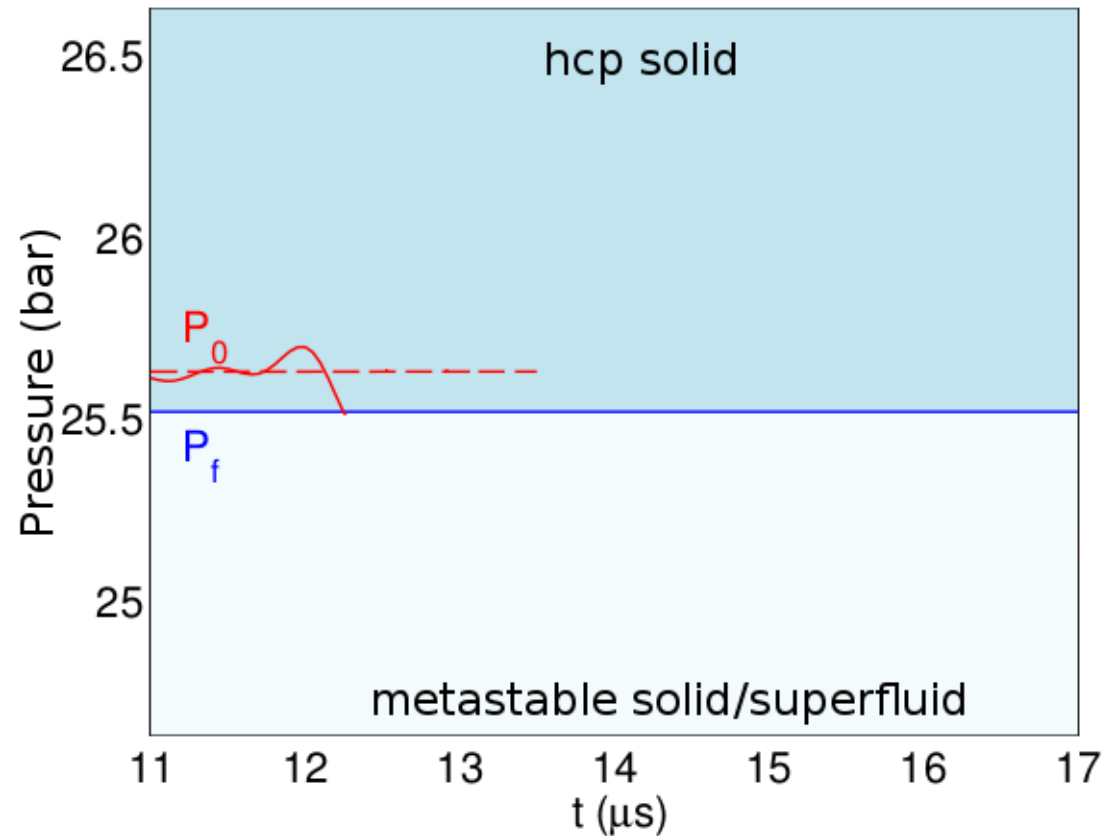
- ▶ Does the wavesurface emitter focus the sound wave correctly?

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► Time evolution at the acoustic focus :



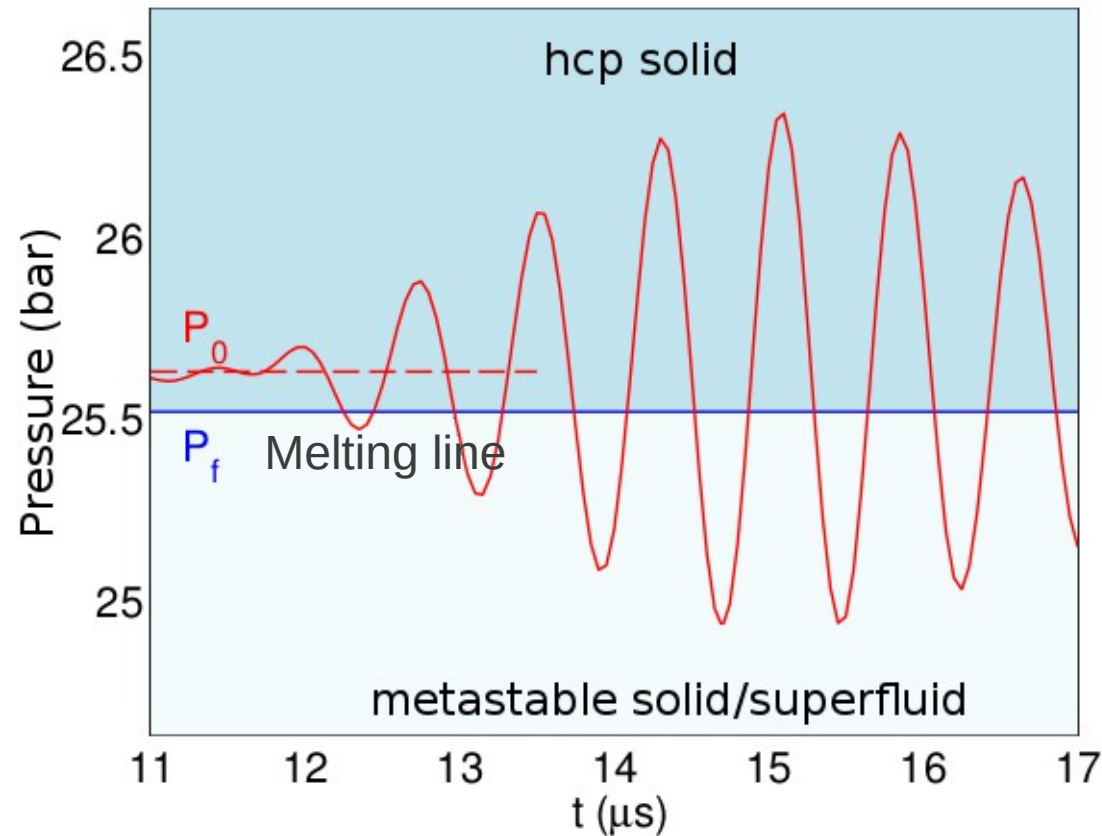
$$\delta\rho = \rho \frac{\delta n}{n-1} \xrightarrow{\text{Constant bulk modulus}} \delta P$$

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Metastable state of solid helium-4



► Time evolution at the acoustic focus :



► Sample pressure goes below the melting line : **the crystal is in a metastable state**



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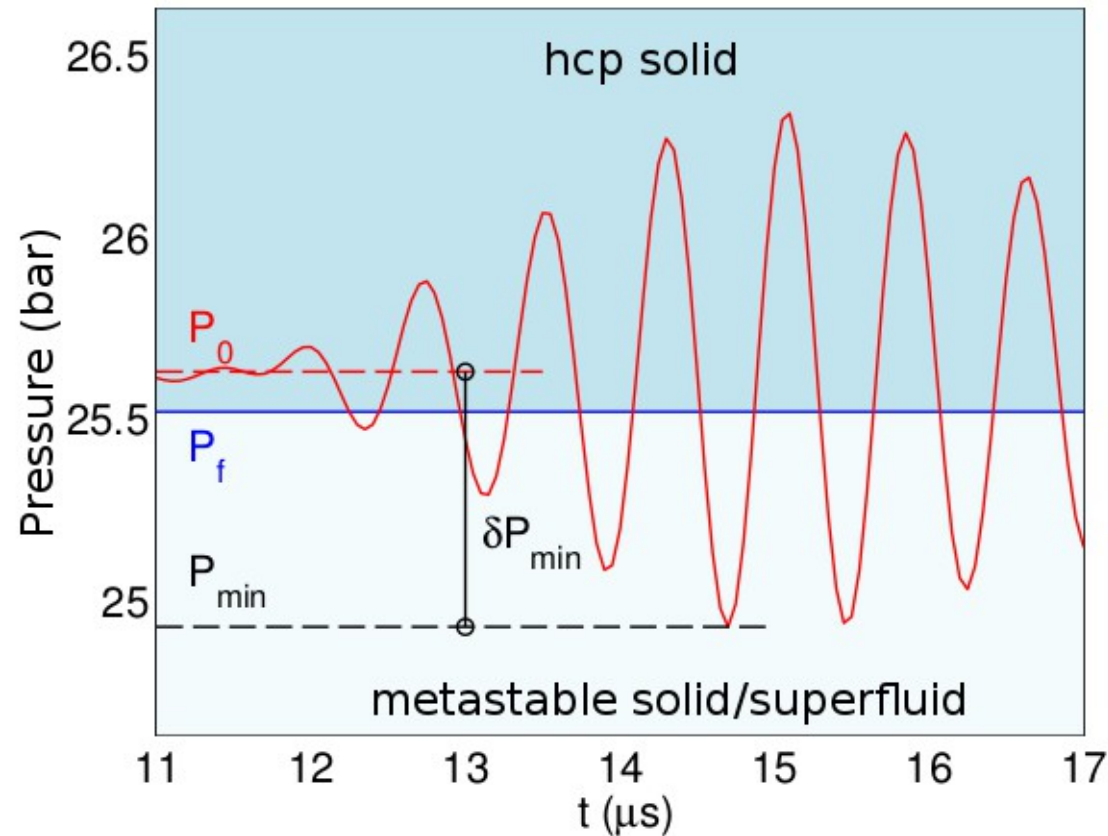
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► Time evolution at the acoustic focus :



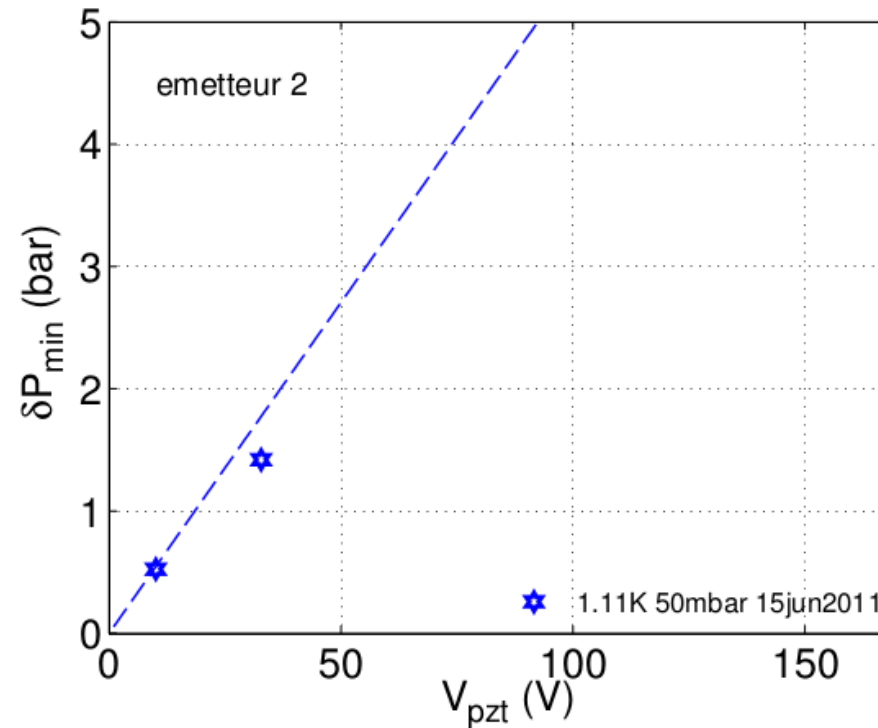
► Sample pressure goes below the melting line : **the crystal is in a metastable state :**

- δP_{min} is the largest pressure variation
- P_{min} the lowest pressure reached

Increasing the metastability



► The voltage V_{pzt} applied to the piezoelectric emitter is varied to increase the metastability :

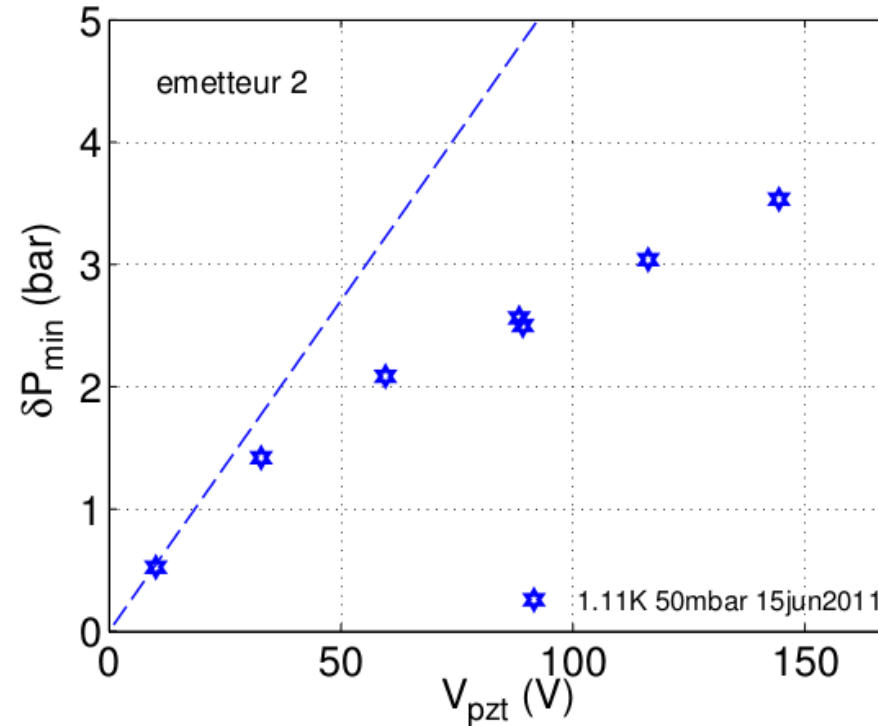


Increasing the metastability



⁴He

► The voltage V_{pzt} applied to the piezoelectric emitter is varied to increase the metastability :



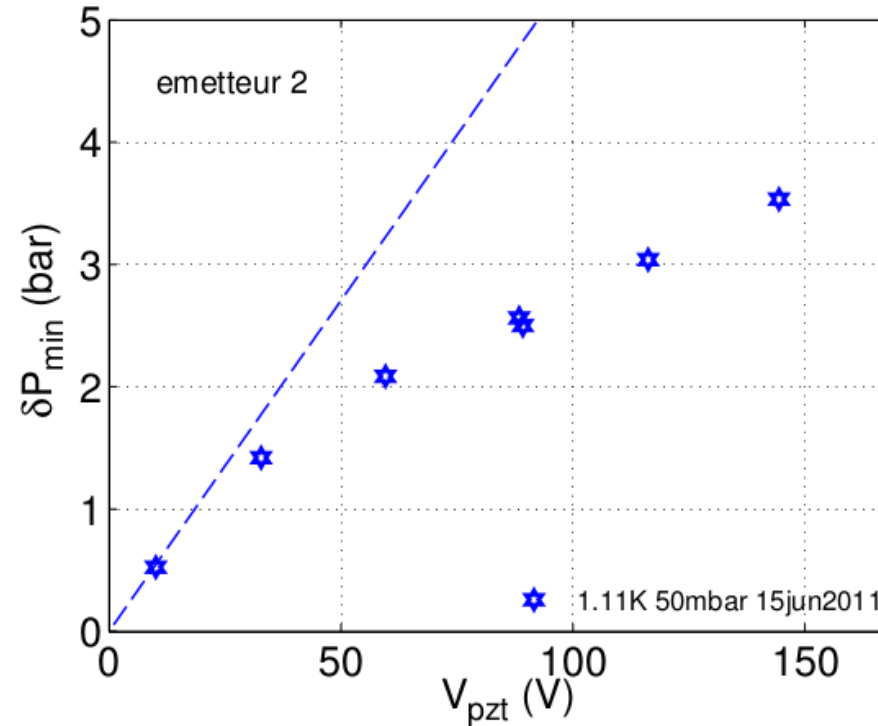
δP_{min} is limited by **nonlinearities during propagation**, probably due to plastic effects

Increasing the metastability



^4He

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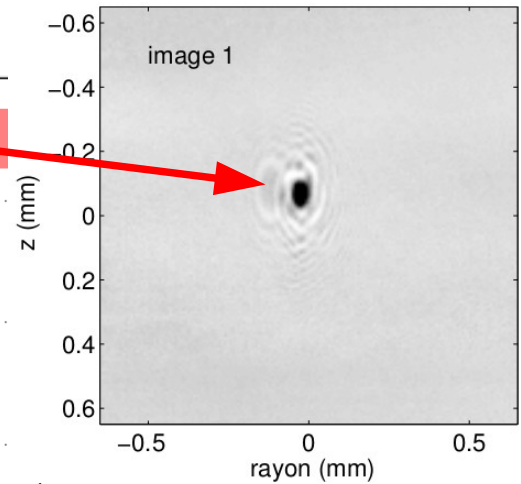
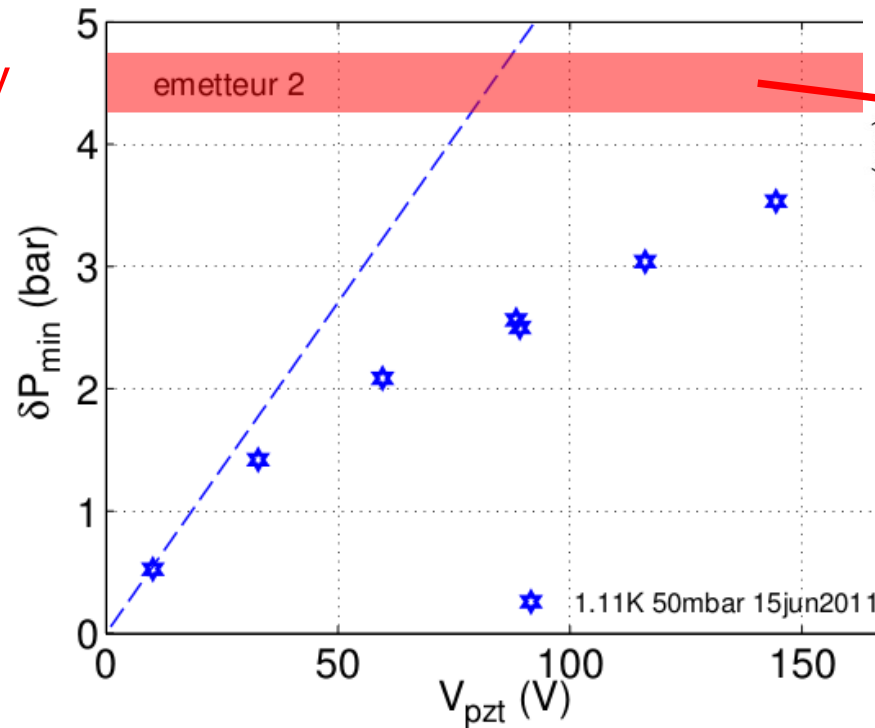
Increasing the metastability



^4He

► The voltage V_{pzt} applied to the piezoelectric emitter is varied to increase the metastability :

Instability
~4.4 bar



δP_{min} is limited by **nonlinearities during propagation**, probably due to plastic effects

If the voltage is increased further, the **crystal becomes unstable**

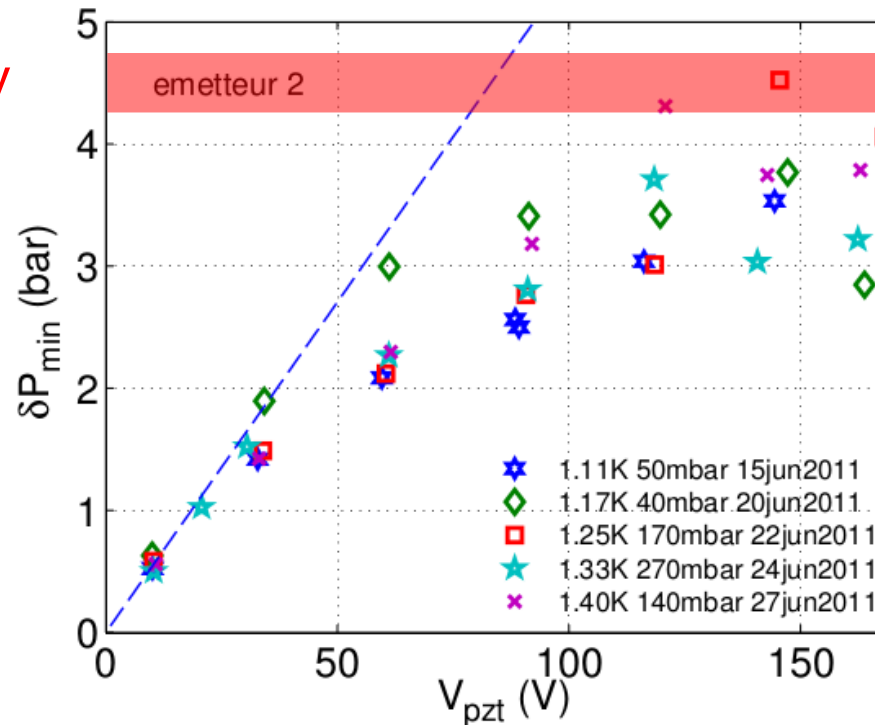
Increasing the metastability



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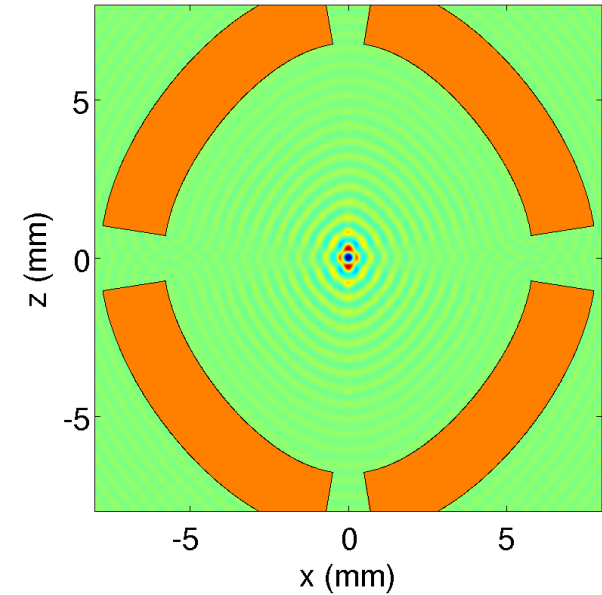
Is the instability a property of helium crystals?

Is it related to non-linearities?



► Two emitters in confocal configuration :

- For the same initial amplitude, the amplitude at the focal point is **doubled**
- Symmetrized** the sound wave



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Reducing nonlinearities

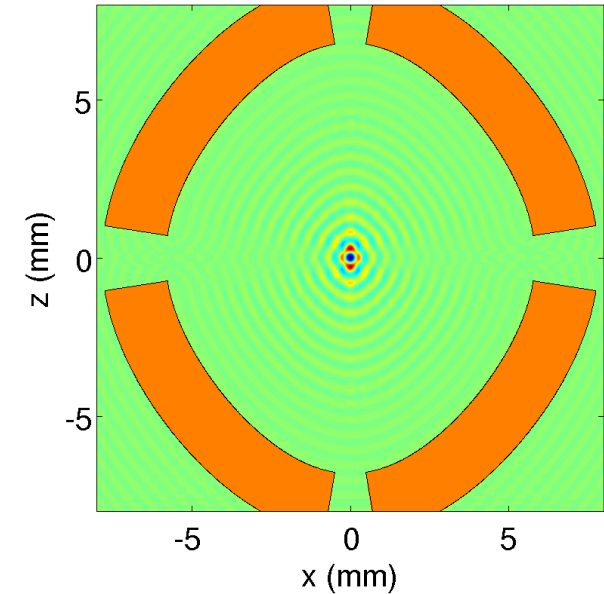


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► Two emitters in confocal configuration :

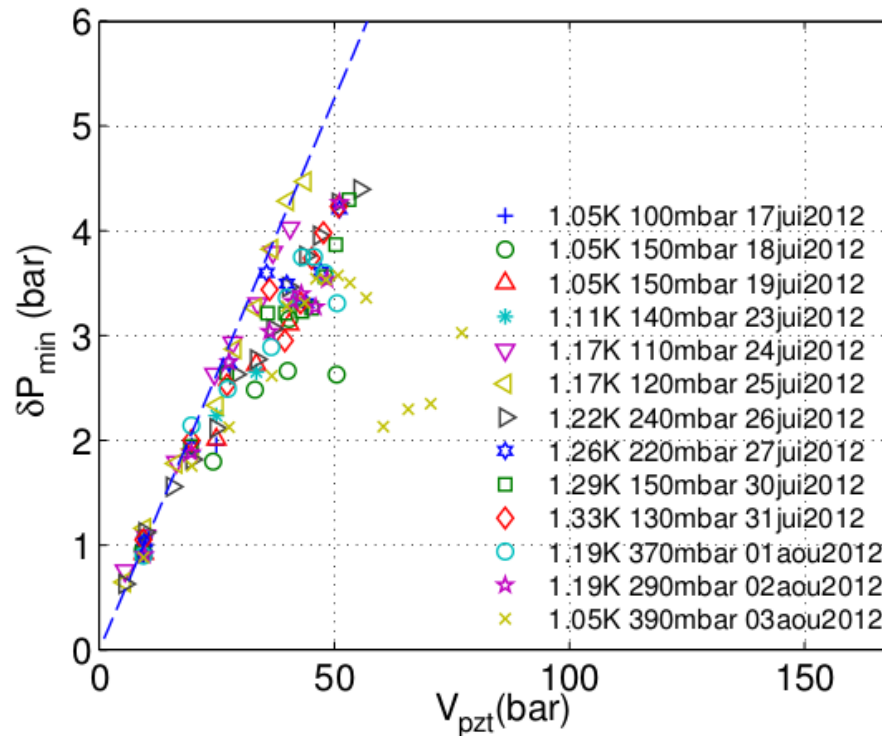
-For the same initial amplitude, the amplitude at the focal point is **doubled**

-**Symmetrized** the sound wave



► Results :

-Much closer to a linear behaviour



Instability NOT related to nonlinearities

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Reducing nonlinearities

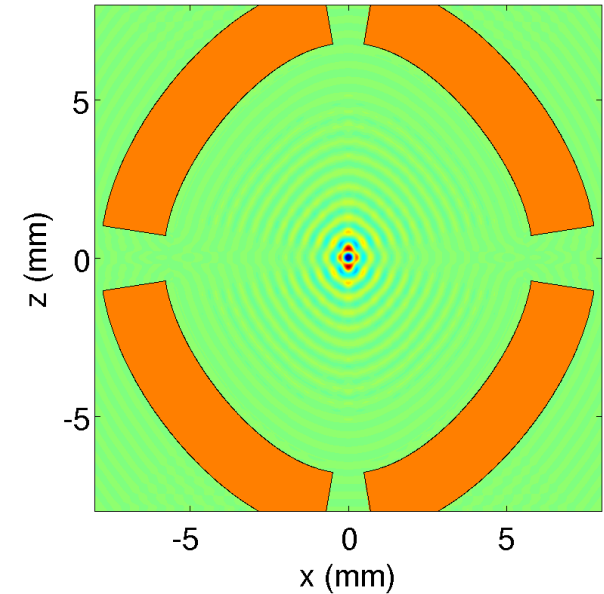


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► Two emitters in confocal configuration :

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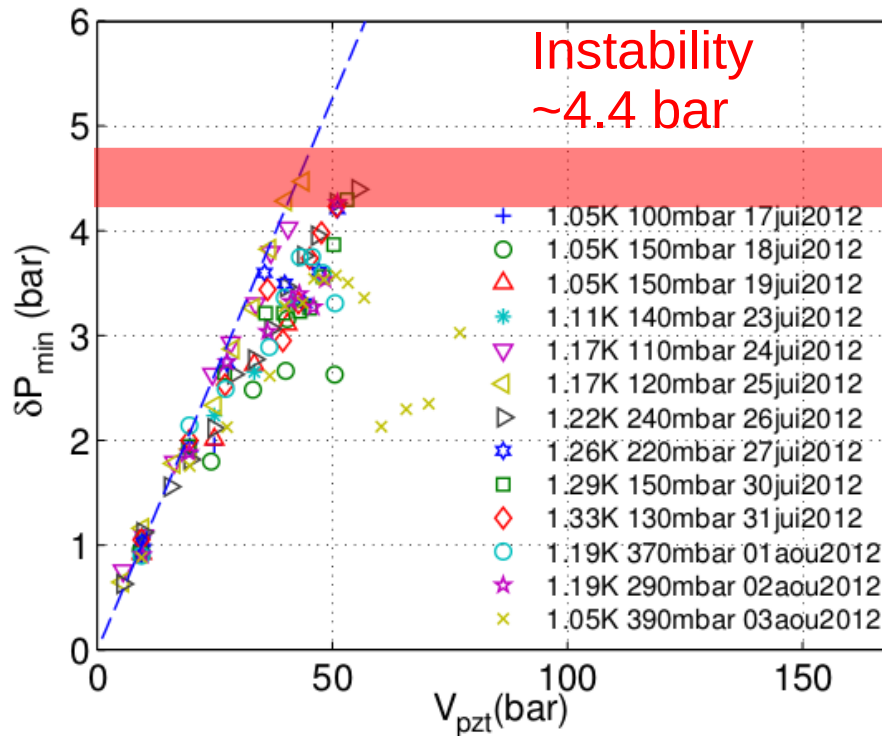
-**Symmetrized** the sound wave



► Results :

-Much closer to a linear behaviour

-Instability still appear for the **same pressure amplitude range**



Instability NOT related to nonlinearities

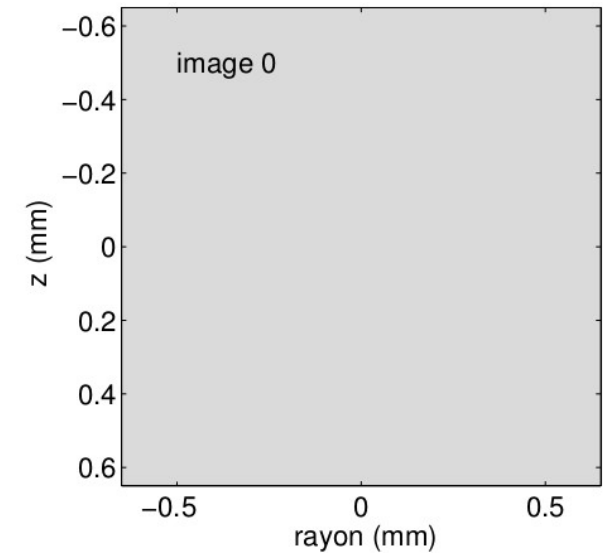


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► Formation of the instability :

-**suddenly** appears on image

-small nucleus (~ 0.1 mm)



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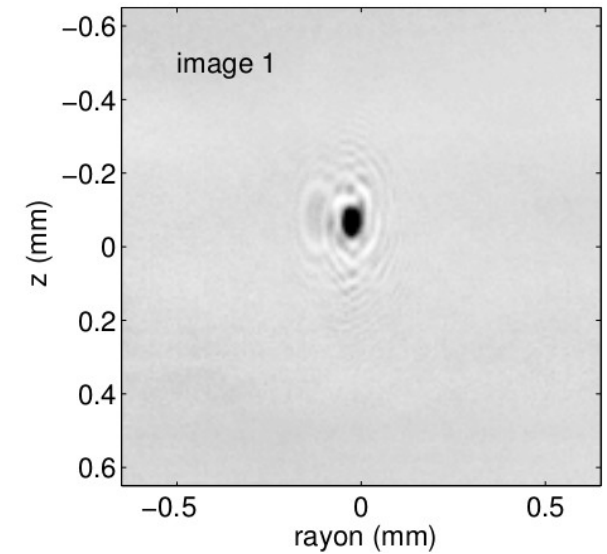


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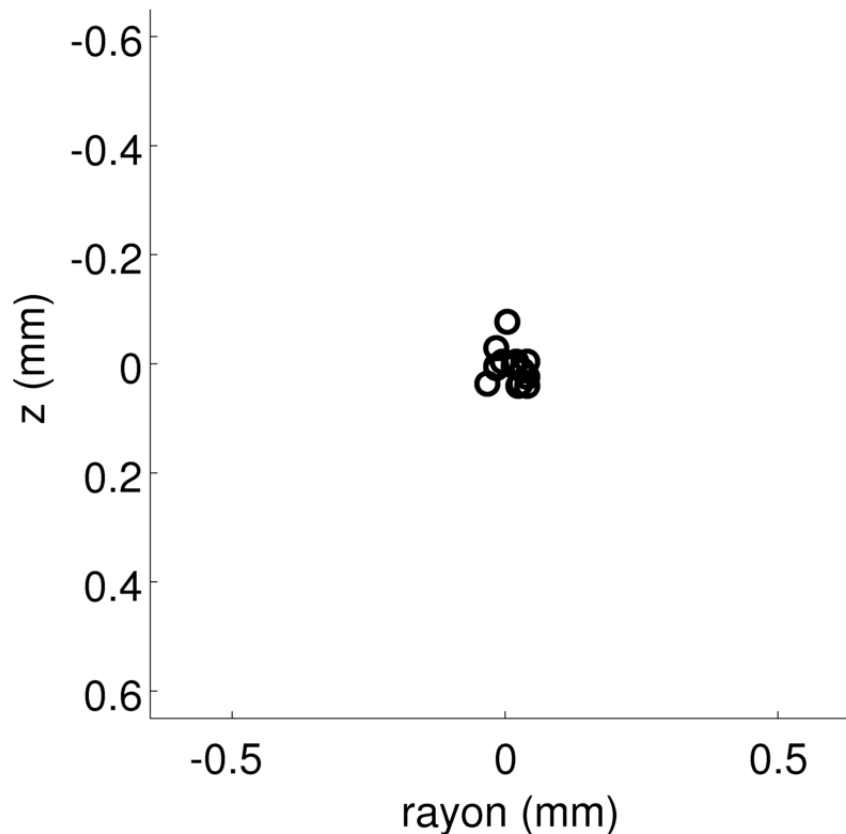
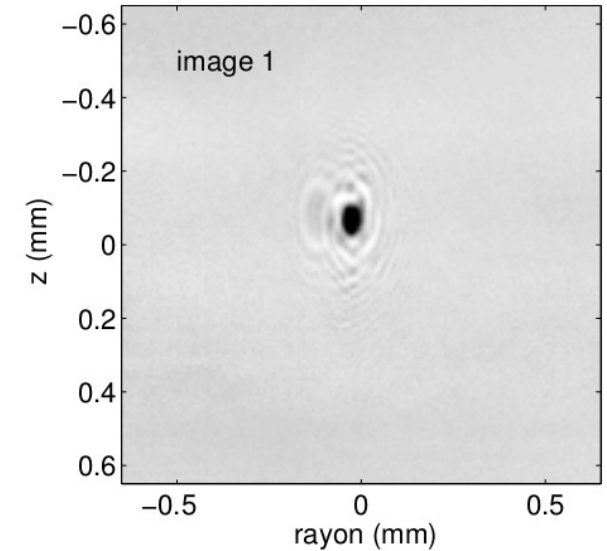


^4He

► Formation of the instability :

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-small nucleus (~ 0.1 mm)



► Localization of the instability :

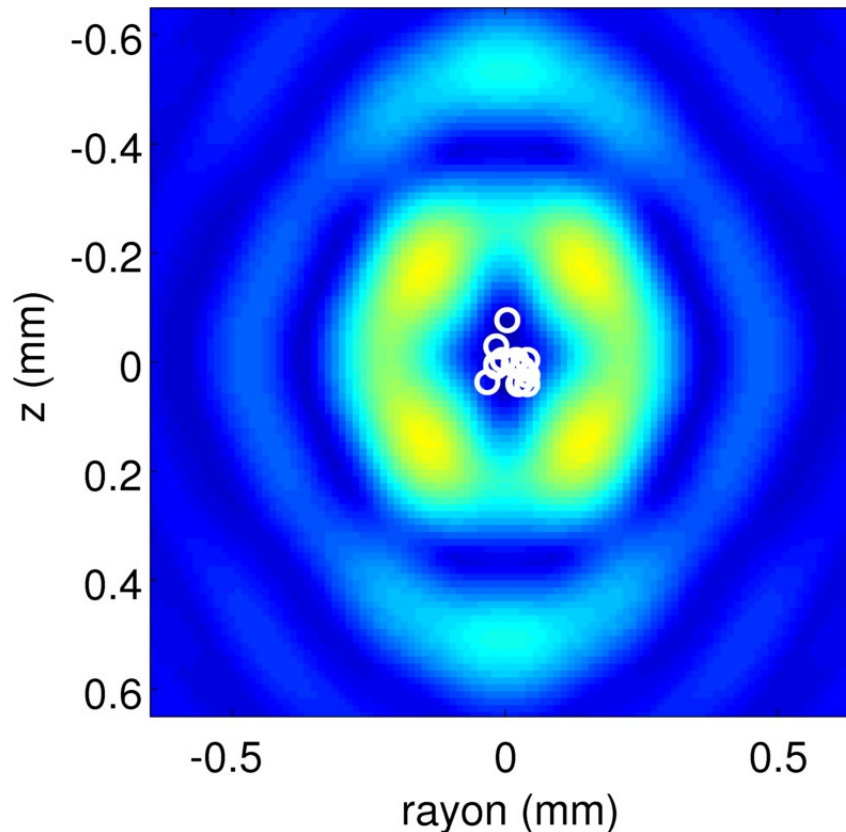
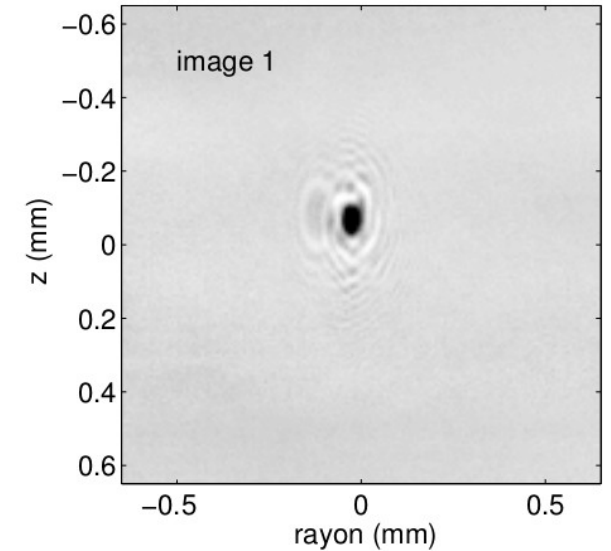
Description of the instability



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► Formation of the instability :

- suddenly appears on image
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► Localization of the instability :

- instability is **NOT** related to **shear**

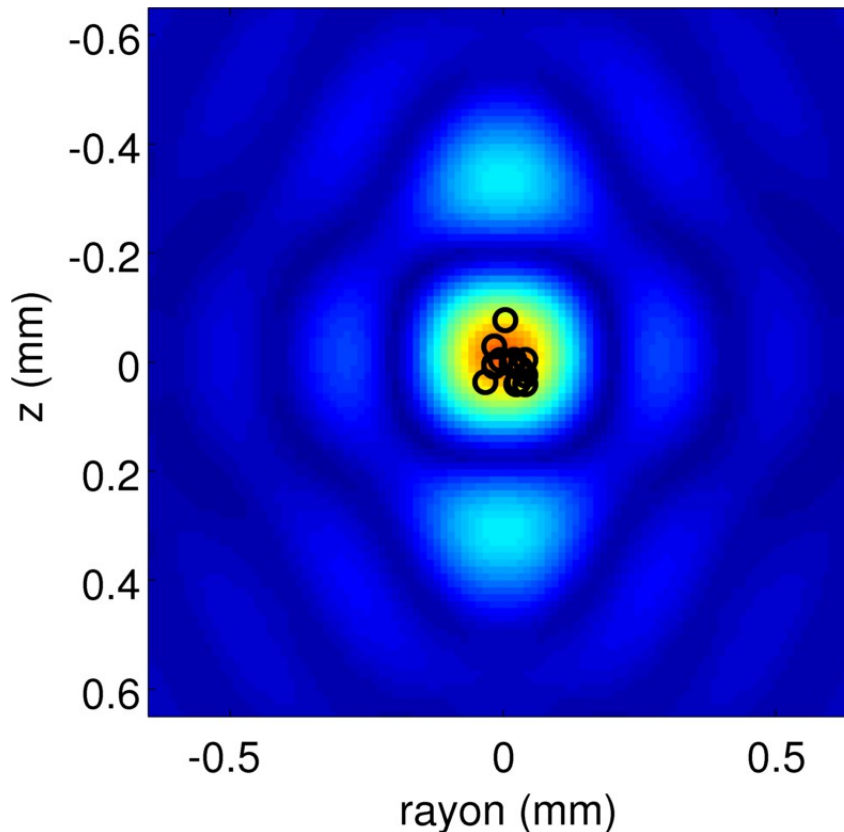
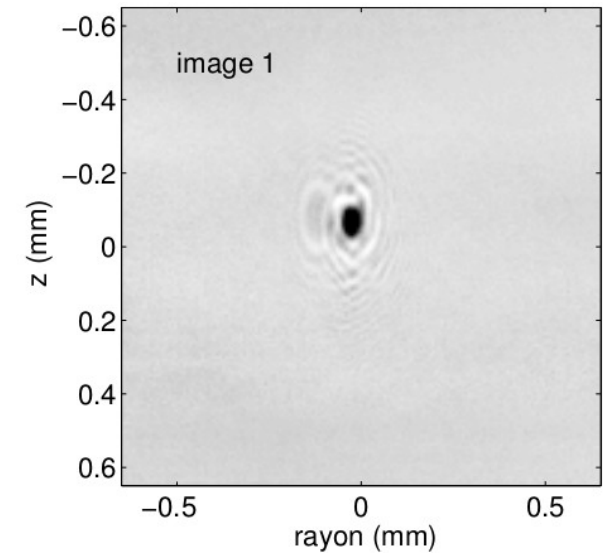
Description of the instability



⁴He

► Formation of the instability :

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► Localization of the instability :

-instability is **NOT** related to **shear**

-localized at the **maximum amplitude** of the sound wave

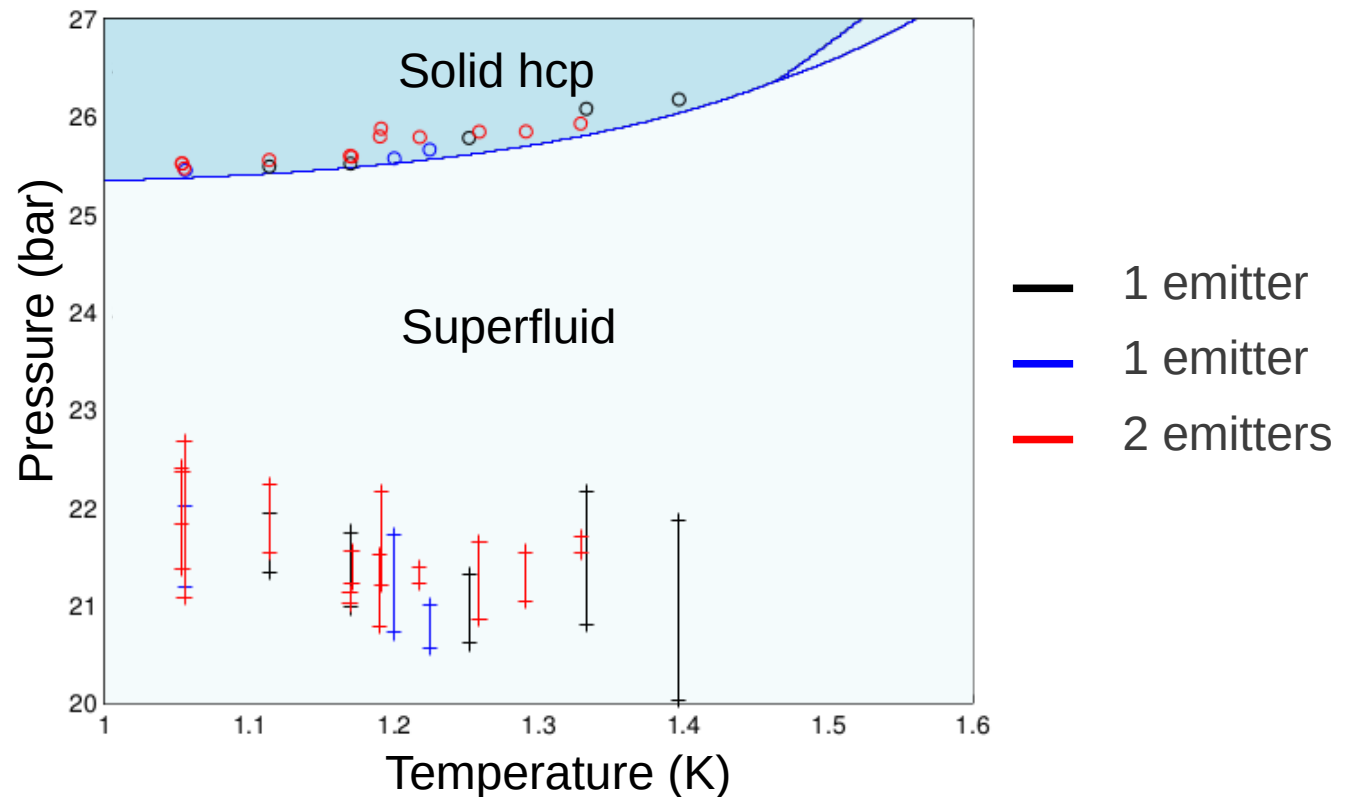
Instability threshold



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► Observations :

- Same results for the two configurations used
- No dependance to the initial pressure
- Small dependance to the temperature



Mean threshold pressure is 21.4 bar ► The stability limit encountered here is clearly different from the one discussed before



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Conclusion and prospects

► The development of two experimental tools made this work possible :

-A **taylor-made** piezoelectric transducer, shaped to a wavesurface to focus ultrasound wave in helium 4 crystals

-A **time resolved multiphase interferometric imaging method** to determine the pressure of the metastable state

► Results on metastable solid helium-4 :

-Metastable phase explored down to **21.4 bar**

-Limiting **non-linear effects** were suppressed by using a two emitters confocal configuration

-Below 21.4 bar solid helium undergoes an **unexpected instability**

-This instability could correspond to the **nucleation of the liquid** phase, but is incompatible with actual model



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► Short and middle term prospects :

- Confirm that instability takes place during **negative swings** of the wave
- Build a nucleation model to take into account **crystal defects**

► Long term prospects :

- Extend the **temperature range** of measurements
- Assess the compressibility in the metastable state by using **Brillouin scattering**



Thank you for your attention



⁴He

Introduction

Focused ultrasound wave in solid helium-4

Overcoming sound anisotropy

Numerical simulation of sound propagation

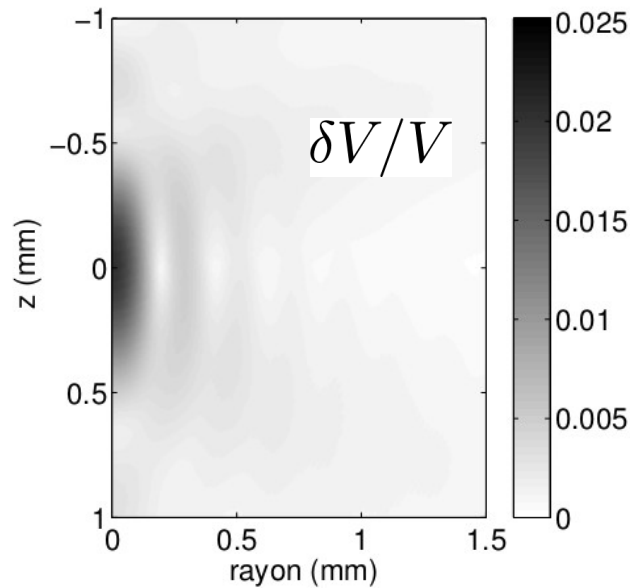
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One ultrasonic emitter



► Maximum of $\delta V/V$ reached :

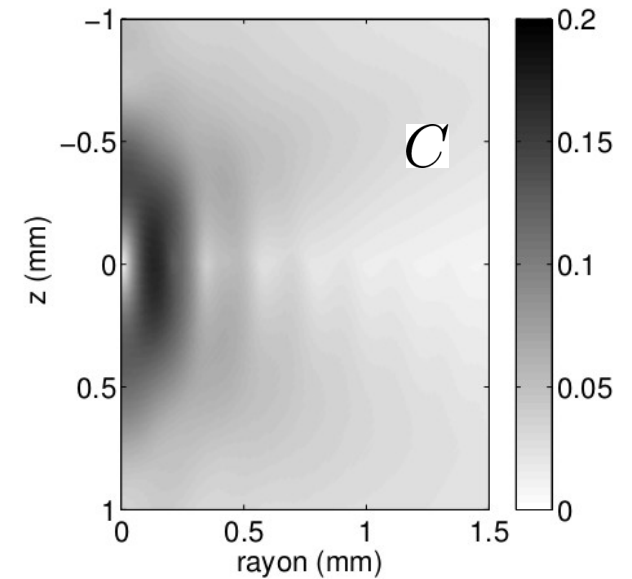
-ovoidal focus (width $\frac{\lambda}{4}$, height λ)

-amplification factor $g = 86$

► Maximum of C reached :

-maximum shear around focus

-no shear at the origin





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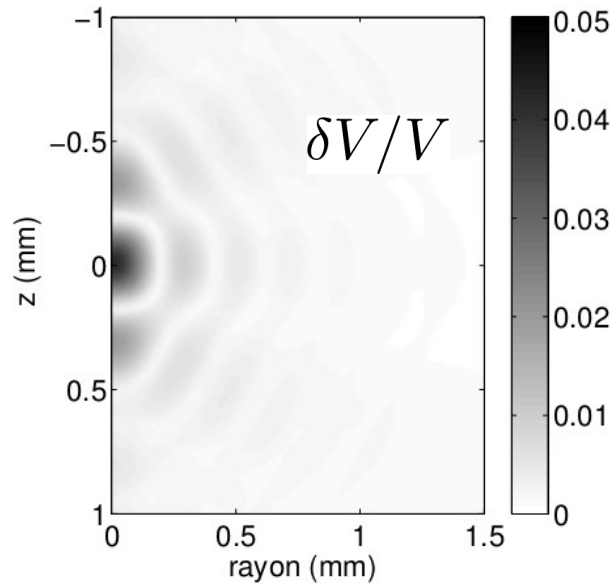
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Two ultrasonic emitters



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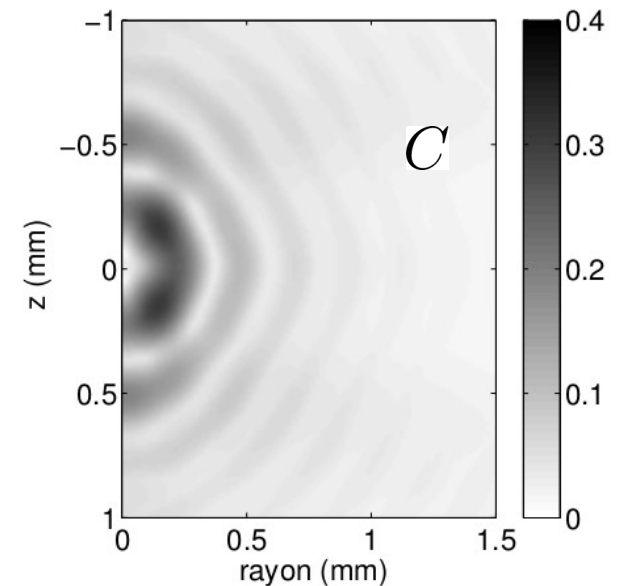
-circular focus (width $\frac{\lambda}{4}$, height $\frac{\lambda}{4}$)

-amplification factor $g = 178$

► Maximum of C reached :

-maximum shear around focus

-no shear at the origin



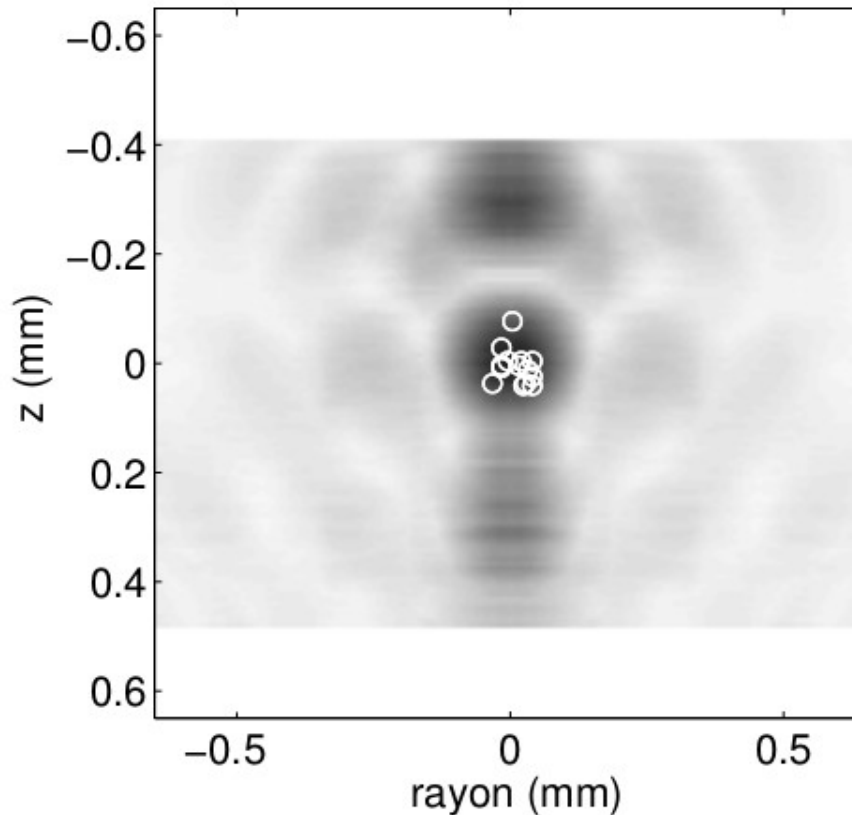
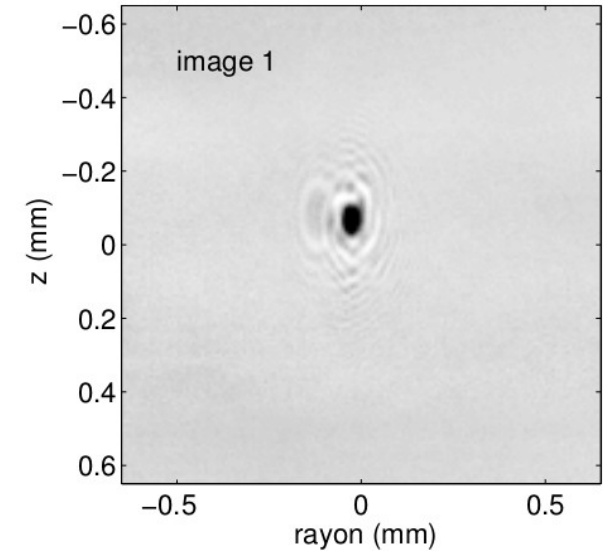
Description of the instability



^4He

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- suddenly appears on image
- small nucleus (~ 0.1 mm)



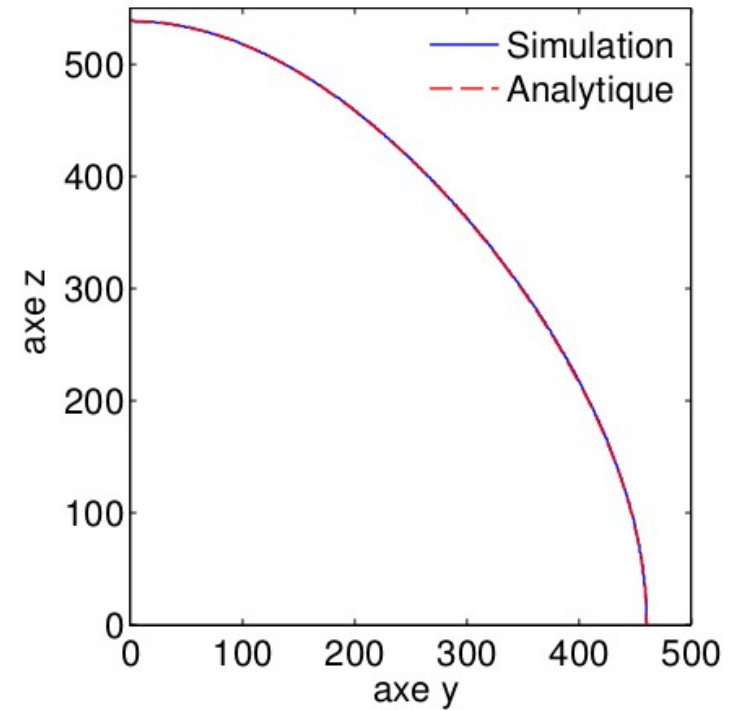
► Localization of the instability :

- instability is **NOT** related to **shear**



► Accuracy checked by creating a wavesurface

Very good agreement with analytical wavesurface



Sound focusing in helium-4 crystals

