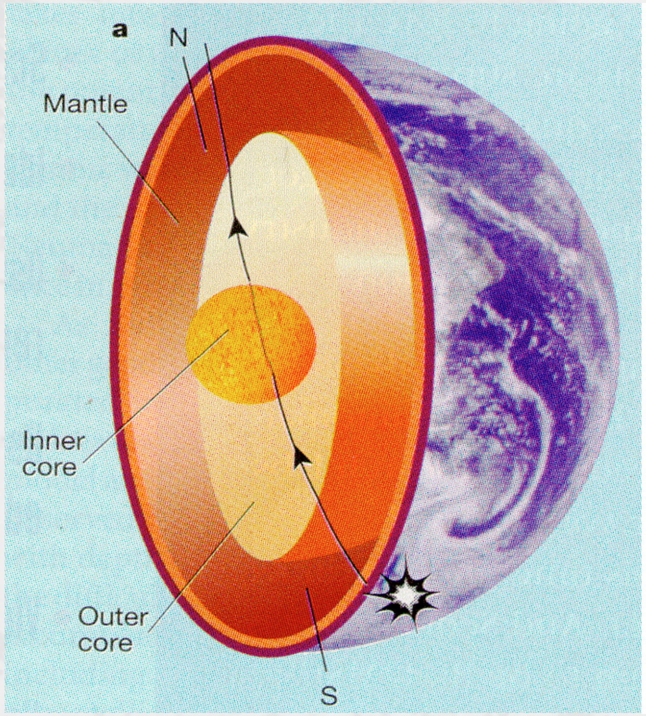
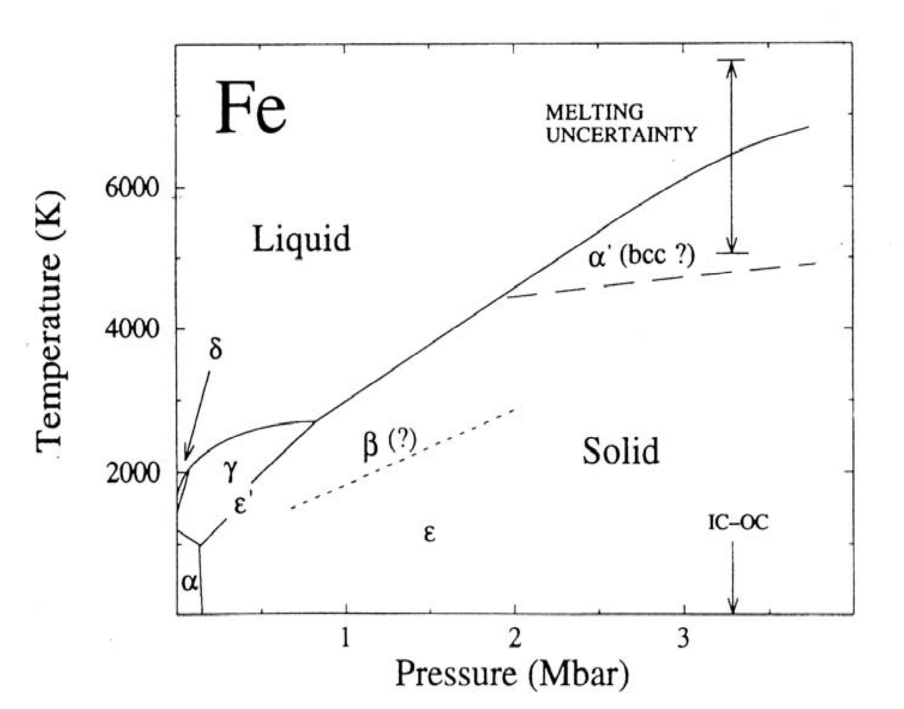
# IXS from polycrystalline samples

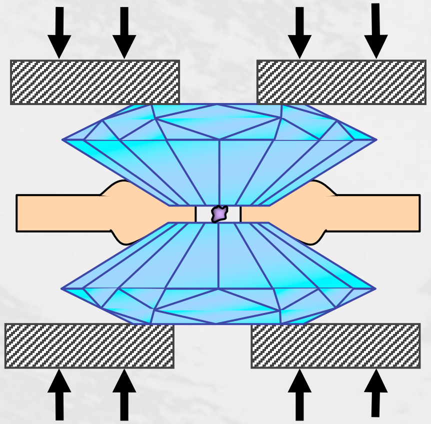
**Determination of orientation averaged properties**

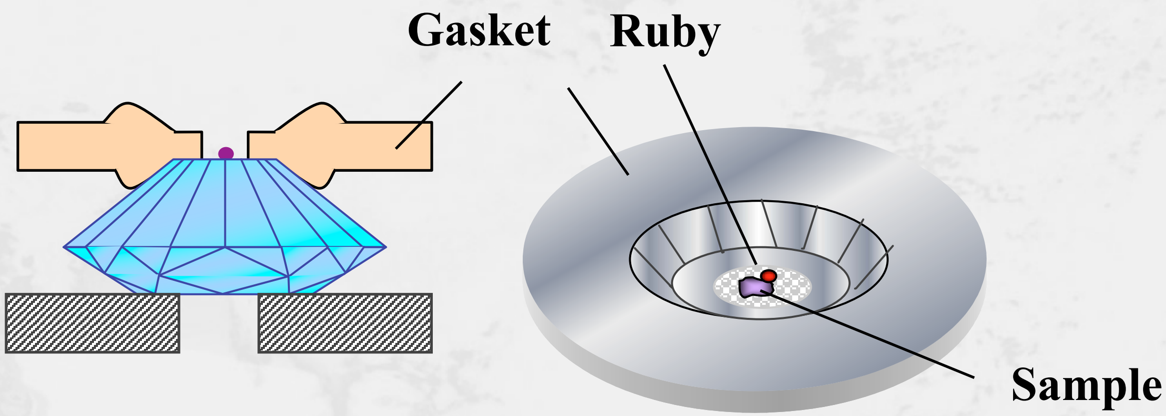
- aggregate sound velocities: $V_L$, ($V_T$)
- phonon density of states: $V_D$, $C_V$, $\Theta_D$, …



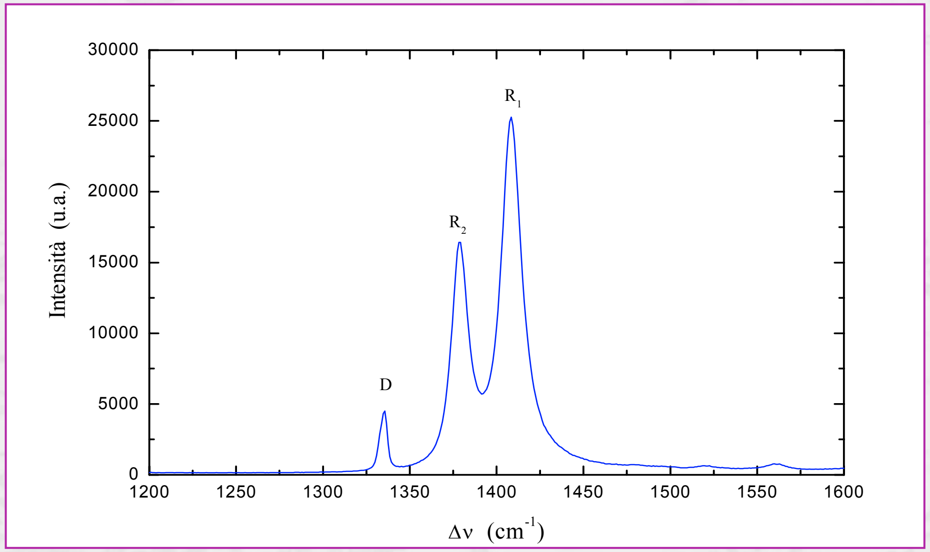


# Diamond anvil cell (DAC) techniques



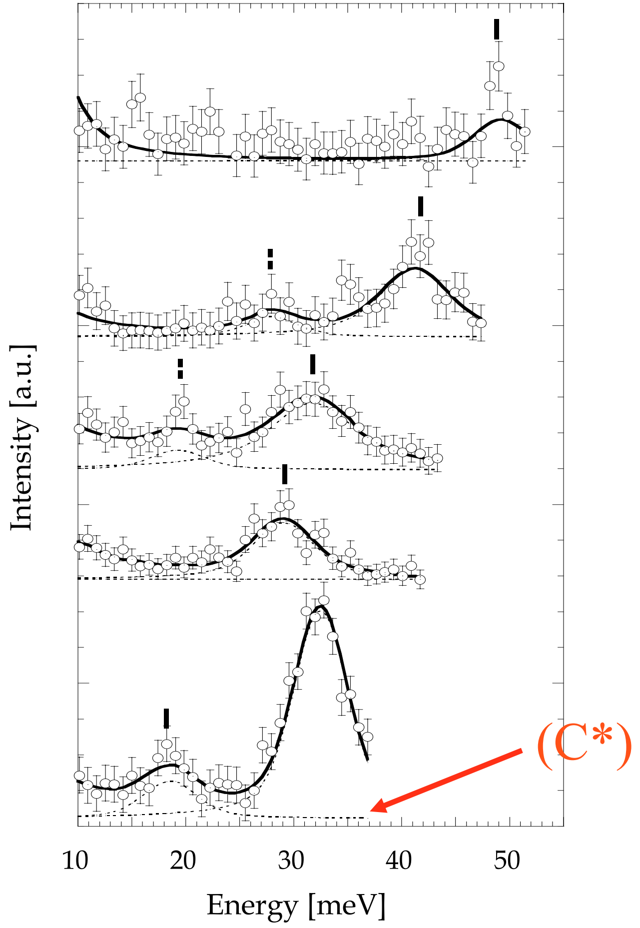


Pressure measurement by frequency shift of ruby fluorescence

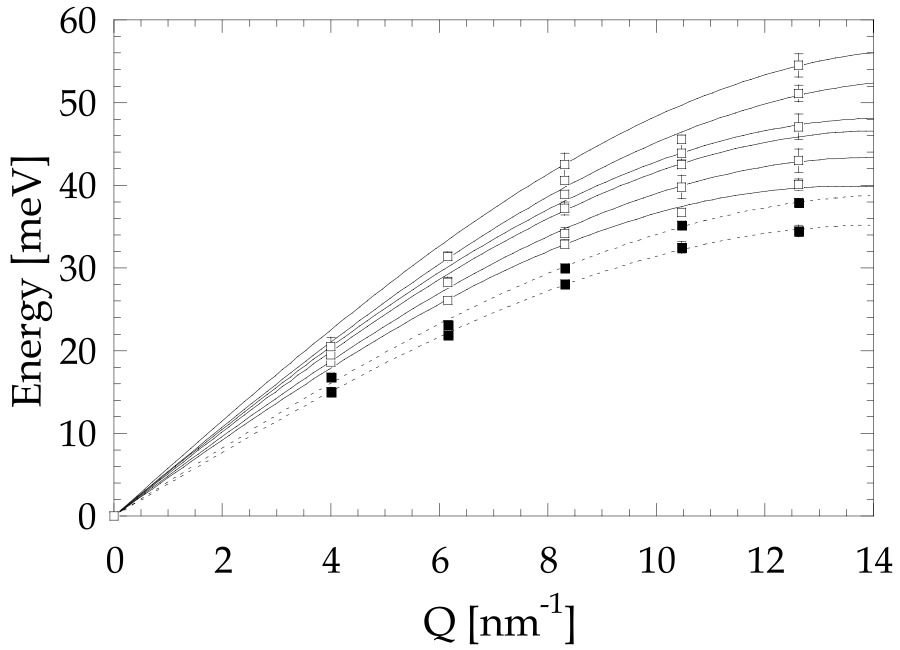


# Polycrystalline ε (hcp)-iron

**P = 28 GPa**



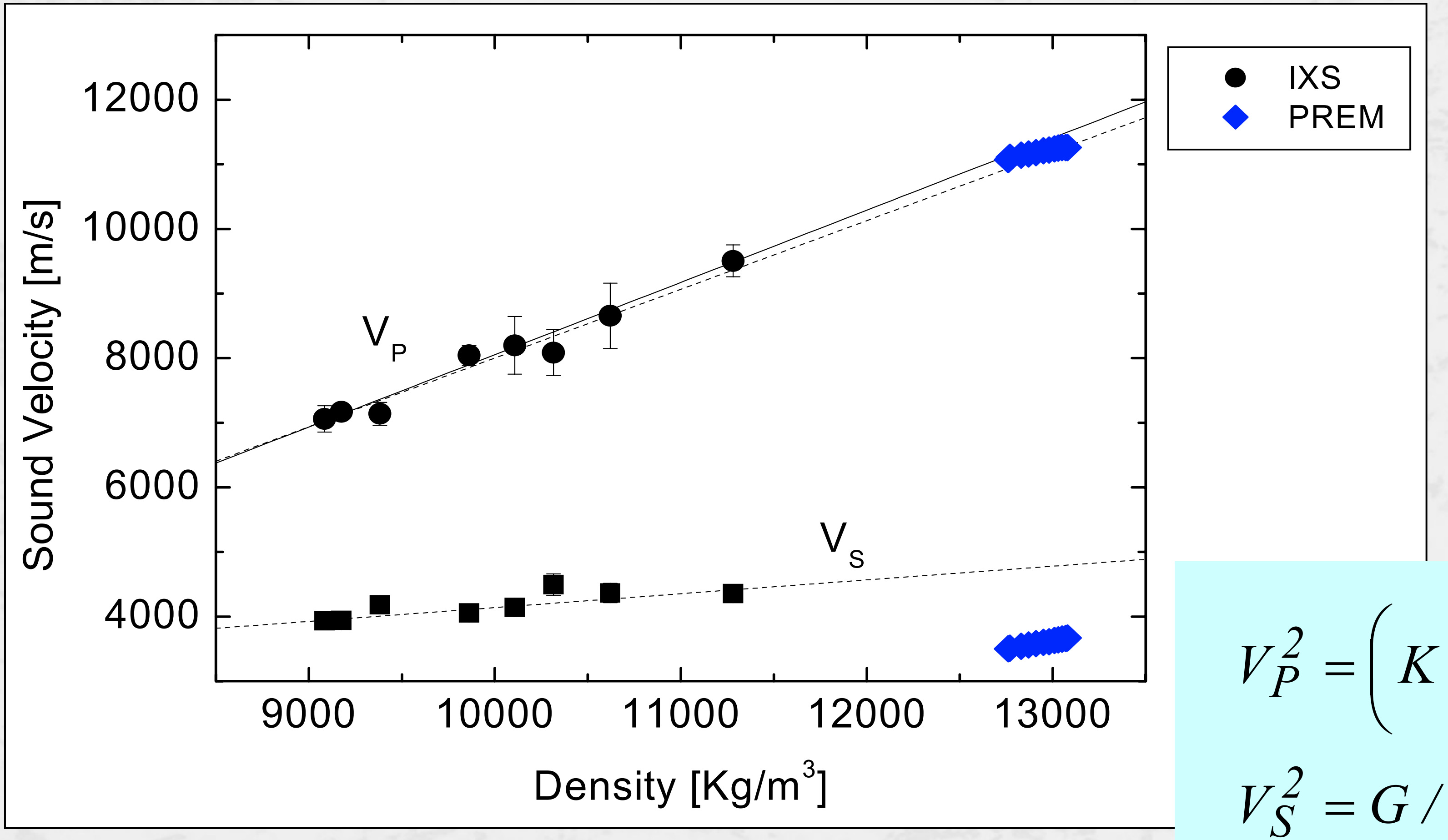
- $\Delta E = 5.5$ meV @ 15816 eV



$$E(Q) = I \cdot \left[ \sum_{n} \left( 1 - \cos\left( \frac{n\pi Q}{Q_{\max}} \right) \right) \right]^{1/2}$$

*G. Fiquet, J. Badro, F. Guyot, H. Requardt and M. Krisch; Science 291, 468 (2001)*

# Density dependence of $V_P$ and $V_S$

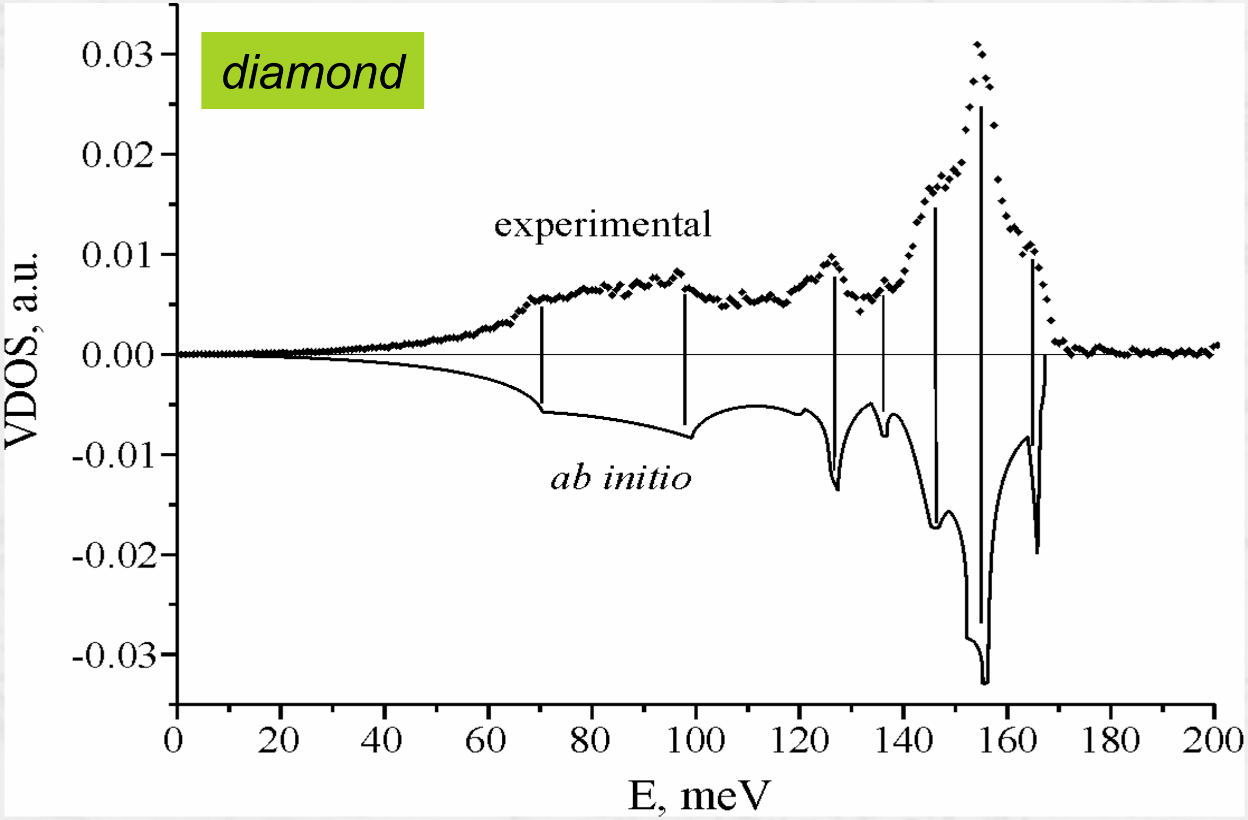


$$V_P^2 = \left( K + \frac{4}{3} G \right) / \rho$$

$$V_S^2 = G / \rho$$

*D. Antonangeli et al; Earth and Planetary Science Letters 225, 243 (2004)*

# Determination of the phonon density of states

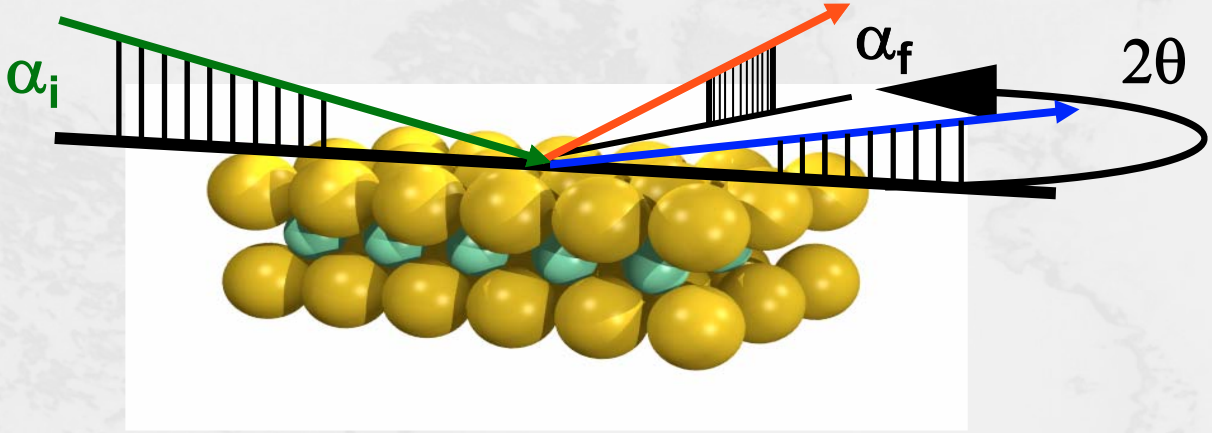


- ΔE = 3 meV
- Sum of 10 IXS spectra (45 $nm^{-1}$<Q<60 $nm^{-1}$

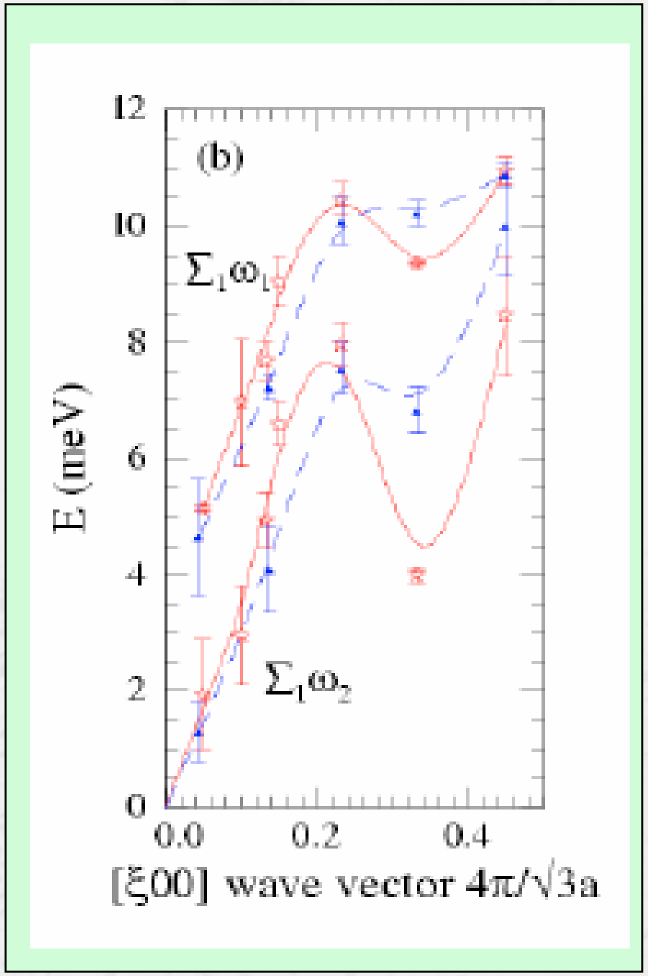
*A. Bosak and M. Krisch; Phys. Rev. B 72, 224305 (2005)*

# IXS in surface sensitive geometry

*B. Murphy et al.; Phys. Rev. Lett. 95, 256104 (2005)*



**2H-NbSe$_2$**



**$\alpha_i$ = 0.18°– 0.03°, penetration depth: ~30 Å**
**Energy resolution: 3 meV**

# Sample environment

**high pressure**

Paris-Edinburgh press
diamond anvil cells

cooled
high-pressure cells

resistive heating
laser heating (ID27)

**low temperature**

cryostats

**high temperature**

ovens

# Conclusions I

## IXS complements INS

**Single Crystals:**

- Determination of $C_{ij}$’s with a few percent precision.
- Full dispersion scheme of simple systems in reasonable time (4-6 days).
- Maximum pressure limited by single crystal quality/thickness.

# Conclusions II

**Powders:**

- Orientation averaged $V_P$ and LA acoustic branch.
- Phonon density of states.

**Disordered systems:**

- longitudinal sound velocities and damping.
- structural relaxation time and strength, thermal diffusivity, viscosity

# ADDITIONAL MATERIAL

# IXS versus INS: scattering kinematics

**Energy Transfer:**

Neutrons:

$\lambda_1$ = 1 Å => $E_1$ = 82 meV E = some meV $E_1 \neq E_2$

=> moderate energy resolution: $E/E_1 = 0.05$

X-rays:

$\lambda_1$ = 1 Å => $E_1$ = 12398 eV E = some meV $E_1 \approx E_2$

=> extremely high energy resolution: $E/E_1 = 10^{-7}$

# IXS versus INS: scattering kinematics

**MomentumTransfer:**

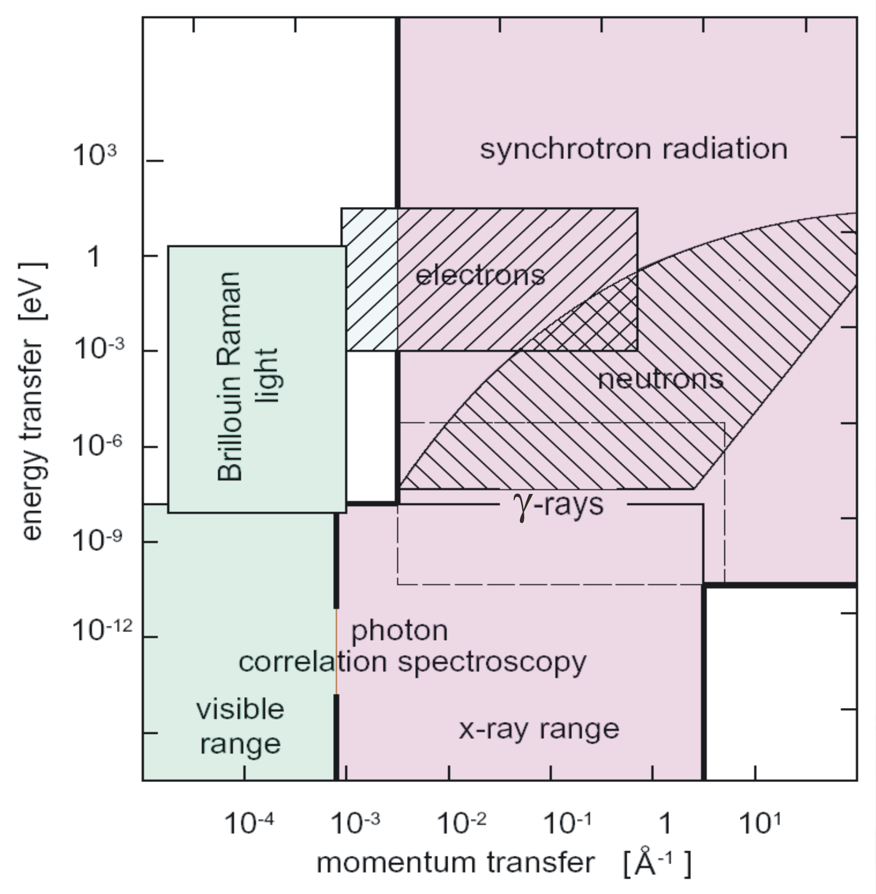
Neutrons: $Q = \sqrt{k_1^2 + k_2^2 - 2k_1 k_2 \cos(\vartheta)}$

=> strong coupling between E and Q
inaccessible E-Q region

X-rays: $Q = 2k_1 \sin\left(\vartheta/2\right)$

=> Q only controlled by scattering angle $\vartheta$

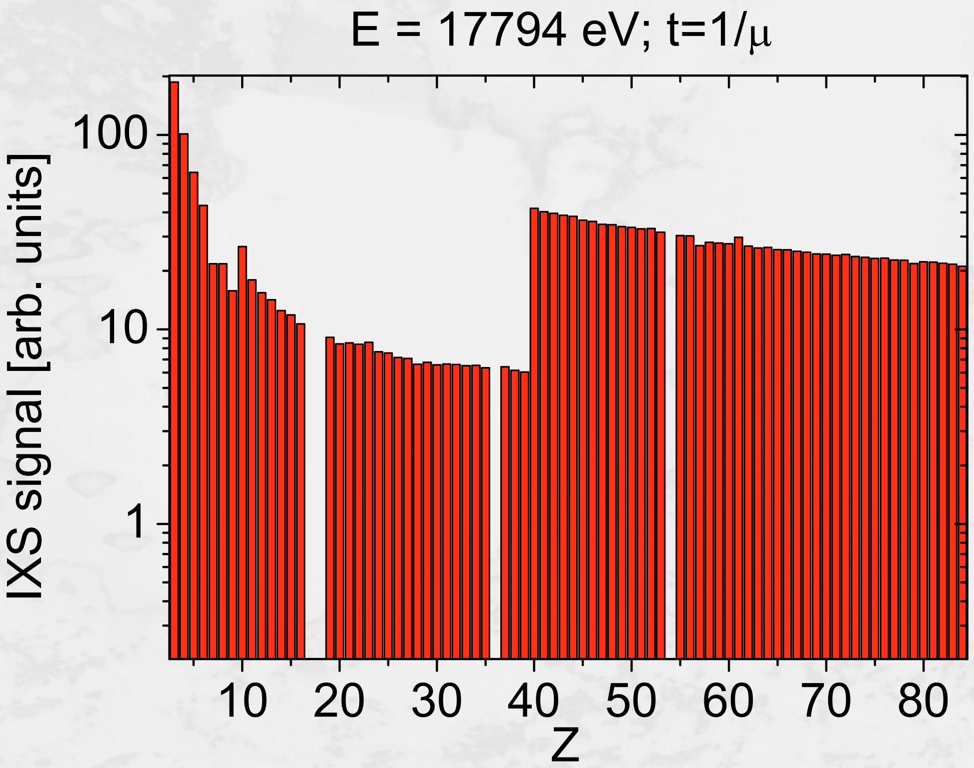
# Q-E range and experimental techniques



**E. Burkel,** Rep. Prog. Phys. **63** (2000) 171–232

# Efficiency of the IXS technique

- IXS signal ~ $n/\mu = n\, t_\mu$



$n$ = concentration of scatterers
$\mu$ = photoelectric absorption

# IXS from phonons: the central approximations

(i) Adiabatic approximation: $\left|S\right\rangle = \left|S_e\right\rangle \left|S_n\right\rangle$

(ii) $\left|I\right\rangle = \left|I_e\right\rangle \left|I_n\right\rangle$ $\left|F\right\rangle = \left|I_e\right\rangle \left|F_n\right\rangle$

## For a mono-atomic system:

$$\frac{\partial^2\sigma}{\partial\Omega\partial E} = \underbrace{r_0^2\,(\vec{\varepsilon}_1\cdot\vec{\varepsilon}_2)^2\,\frac{k_1}{k_2}}_{\text{Thomson scattering cross section}}\,\underbrace{\left|f(Q)\right|^2}_{\text{Atomic form factor}}\;\underbrace{S\left(\vec{Q},E\right)}_{\text{Dynamical structure factor}}$$

# X-ray inelastic cross section

$$\frac{d^2\sigma}{d\Omega dE_f} = \underbrace{r_0^2(\hat{e}_i \cdot \hat{e}_f)}_{\text{Thomson scattering cross-section}}\underbrace{S(Q,E)}_{\textbf{dynamical structure factor}}$$

**Single crystals:** $S(\vec{Q},E,T) = \sum_j G(\vec{Q},j)F(E,T,\vec{Q},j)$

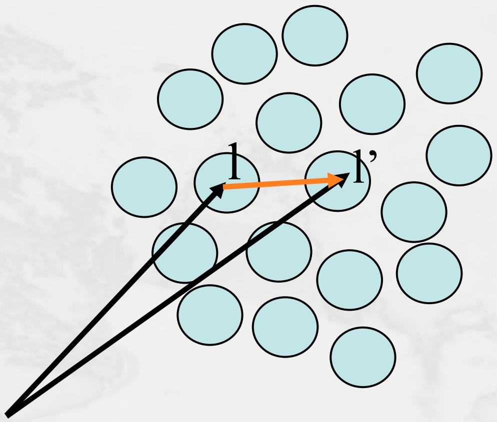
$$F(E,T,\vec{Q},j) = \frac{\left(\left(\exp\left(\frac{E_{\vec{Q},j}}{kT}\right)-1\right)^{-1} + 1/2 \pm 1/2\right)}{E_{\vec{Q},j}} \cdot \delta(E \mp E_{\vec{Q},j})$$

linked to temperature

$$\boxed{G(\vec{Q},j) = \left|\sum_n f_n(\vec{Q})e^{i\vec{Q}\vec{r}_n - W_n}(\vec{Q}\cdot\hat{\sigma}_n(\vec{Q},j))M_n^{-1/2}\right|^2}$$

defines selection rules

# Dynamical structure factor

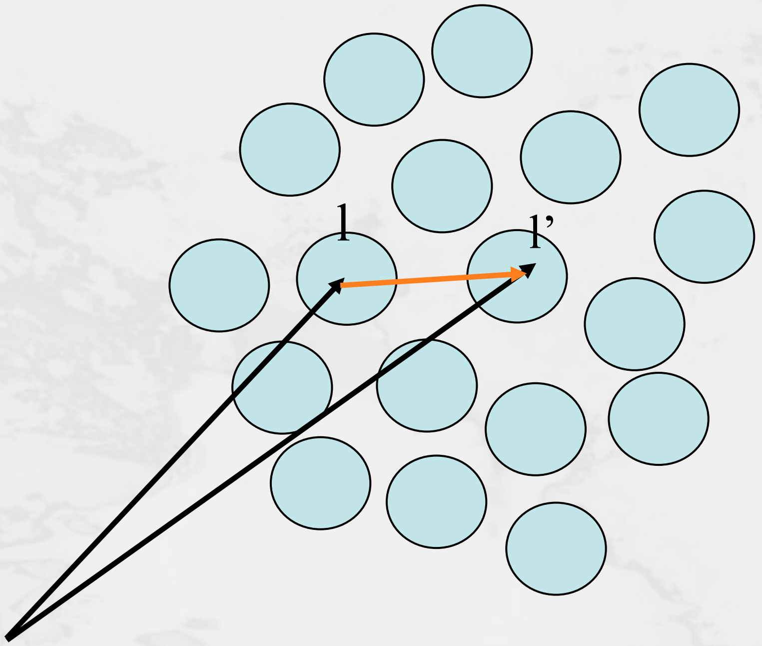


**Dynamical structure factor** $S(\mathbf{Q},\omega)$**:**

**Space** and **time** Fourier transform of $G_P(\mathbf{r},t)$.

**Pair correlation function** $G_P(\mathbf{r},t)$**:**

$G_P(\mathbf{r},t)$ is the probability to find two different particles at positions $\mathbf{R}_{l'}(t=0)$ and $\mathbf{R}_{l'}(t)$, separated by the **distance r** and the **time interval t.**

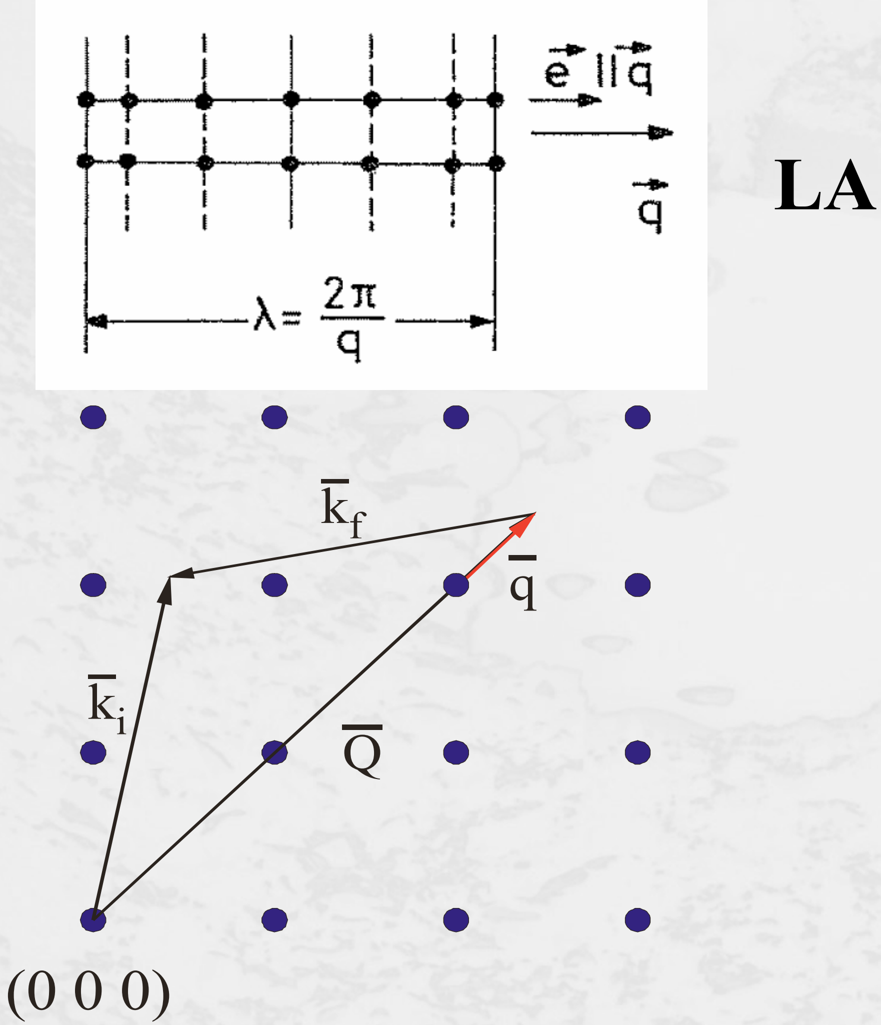


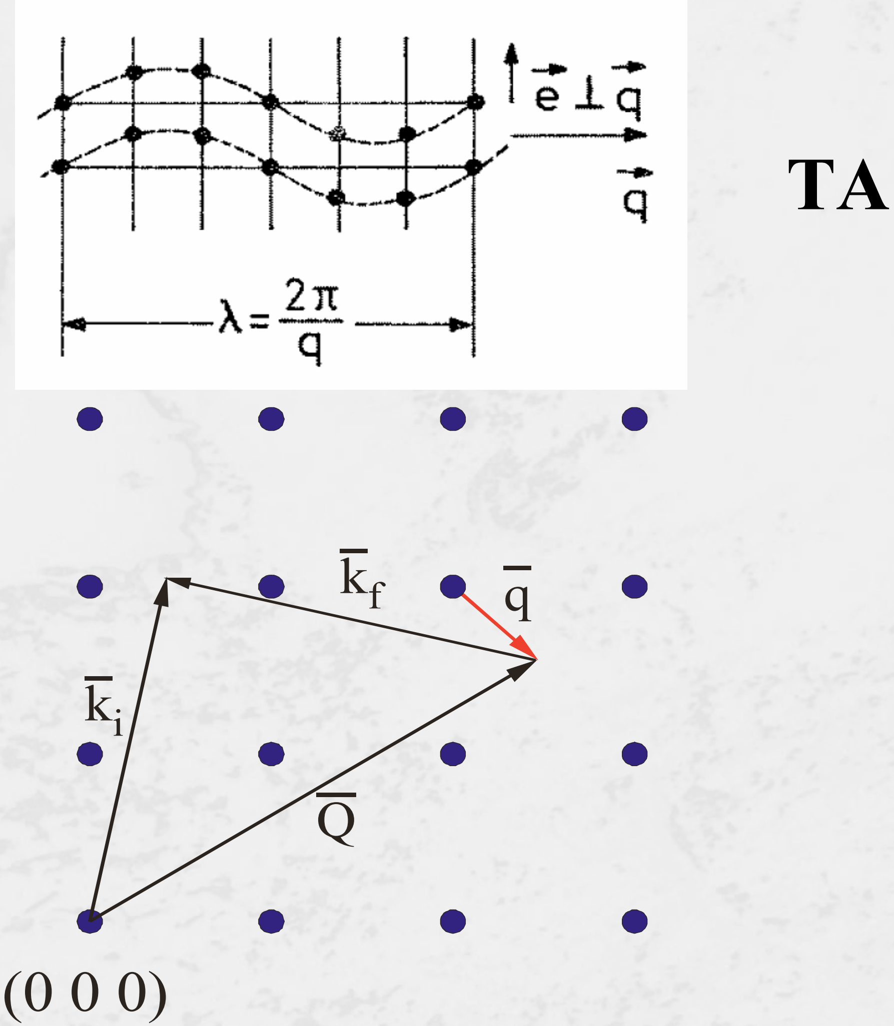
$Q \approx 2\pi/\xi$ $\xi$ is a characteristic length

$\omega = E/h \approx 2\pi/\tau$ $\tau$ is a characteristic (relaxation) time

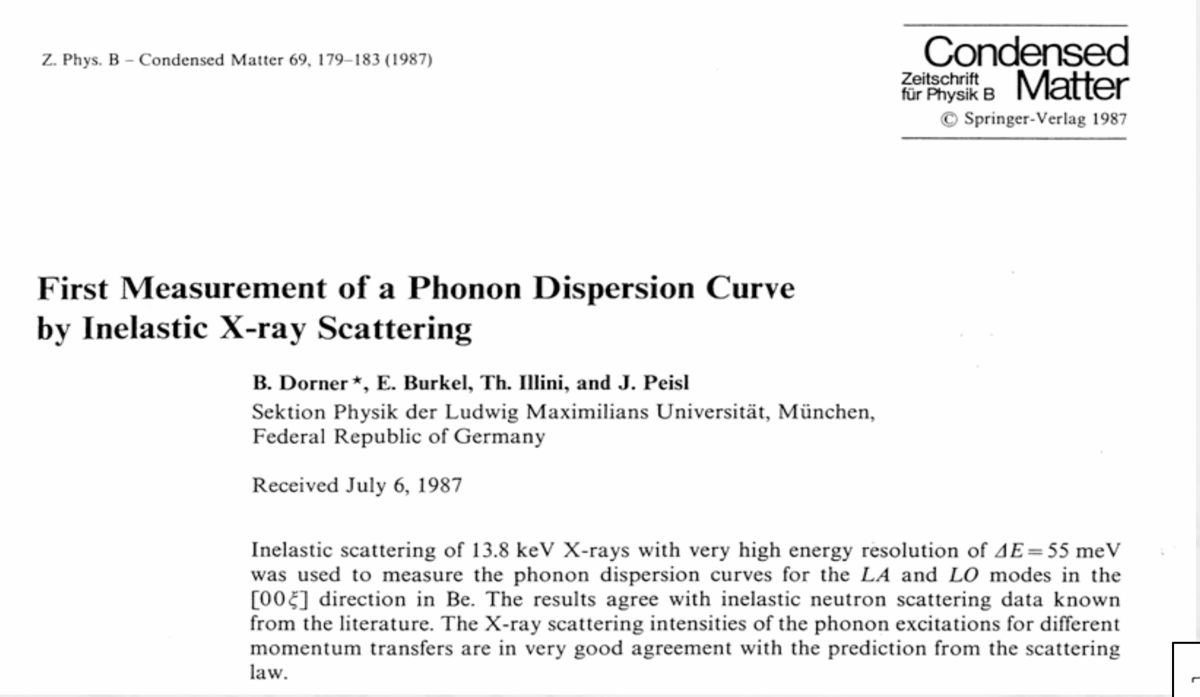
# Selecting phonons

$$G(\vec{Q}, j) \sim \left| \sum_d f_d(\vec{Q})(\vec{Q} \cdot \hat{e}_d(\vec{Q}, j)) M_d^{-1/2} \right|^2$$





# 1987 - first IXS measurements



Z. Phys. B – Condensed Matter 69, 179–183 (1987)

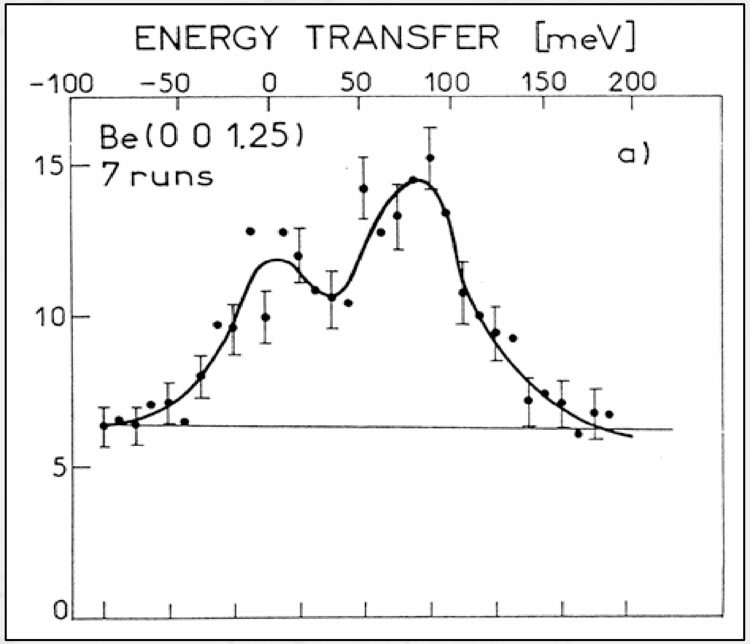
Condensed Matter
Zeitschrift für Physik B
© Springer-Verlag 1987

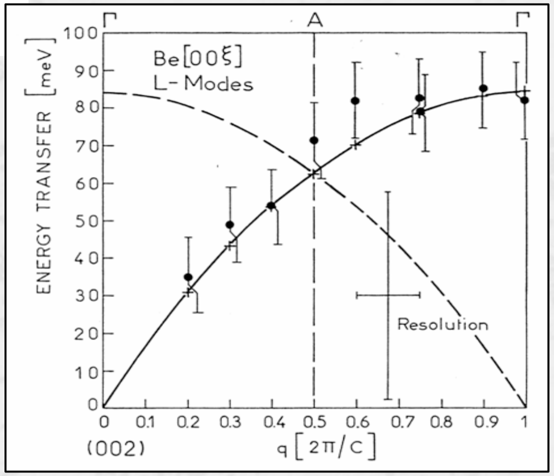
## First Measurement of a Phonon Dispersion Curve by Inelastic X-ray Scattering

B. Dorner *, E. Burkel, Th. Illini, and J. Peisl
Sektion Physik der Ludwig Maximilians Universität, München,
Federal Republic of Germany

Received July 6, 1987

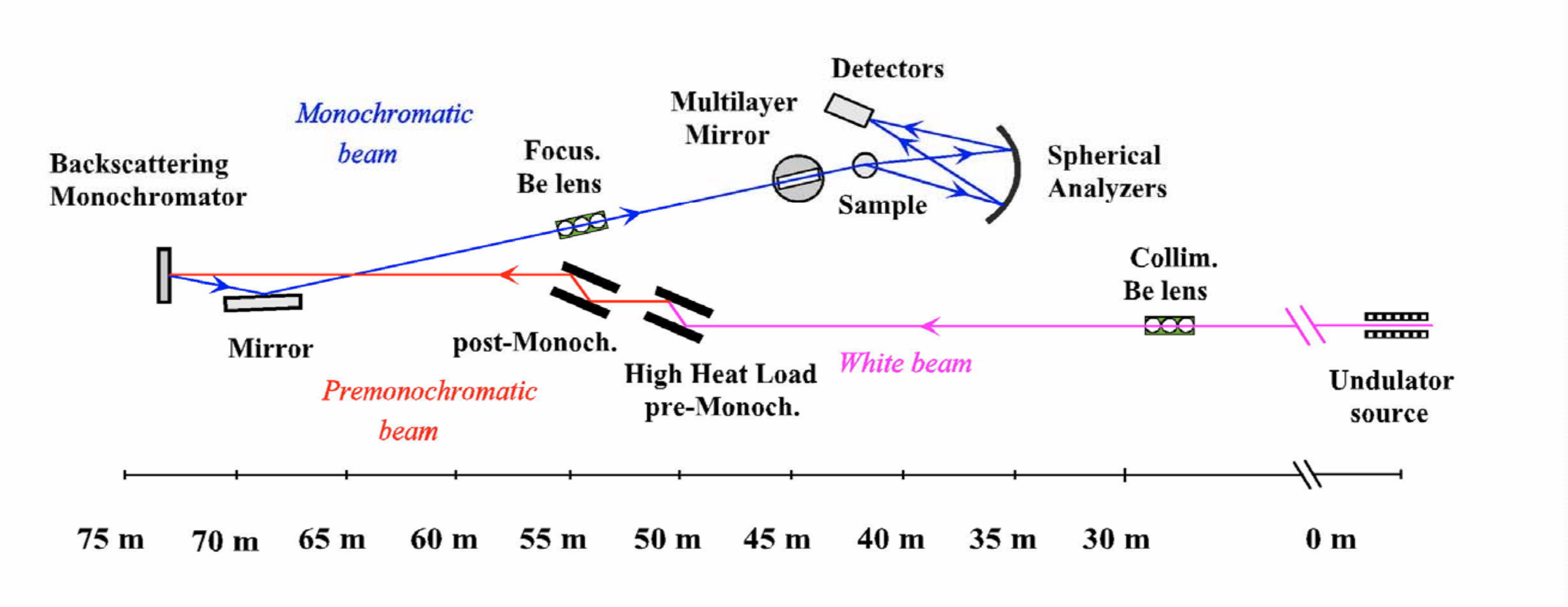
Inelastic scattering of 13.8 keV X-rays with very high energy resolution of $\Delta E = 55$ meV was used to measure the phonon dispersion curves for the *LA* and *LO* modes in the $[00\xi]$ direction in Be. The results agree with inelastic neutron scattering data known from the literature. The X-ray scattering intensities of the phonon excitations for different momentum transfers are in very good agreement with the prediction from the scattering law.





**HASYLAB**

# The IXS spectrometer on ID28



| Reflection | ΔE [meV] | $Q_{max}(7)$ [$nm^{-1}$] |
|---|---|---|
| (7 7 7) | 7 | 64 |
| (8 8 8 ) | 5.5 | 73 |
| (9 9 9) | 3.0 | 82 |
| (11 11 11) | 1.7 | 100 |
| (13 13 13) | 0.9 | 119 |