



Neutron Spectroscopy 2: high resolution spectrometers

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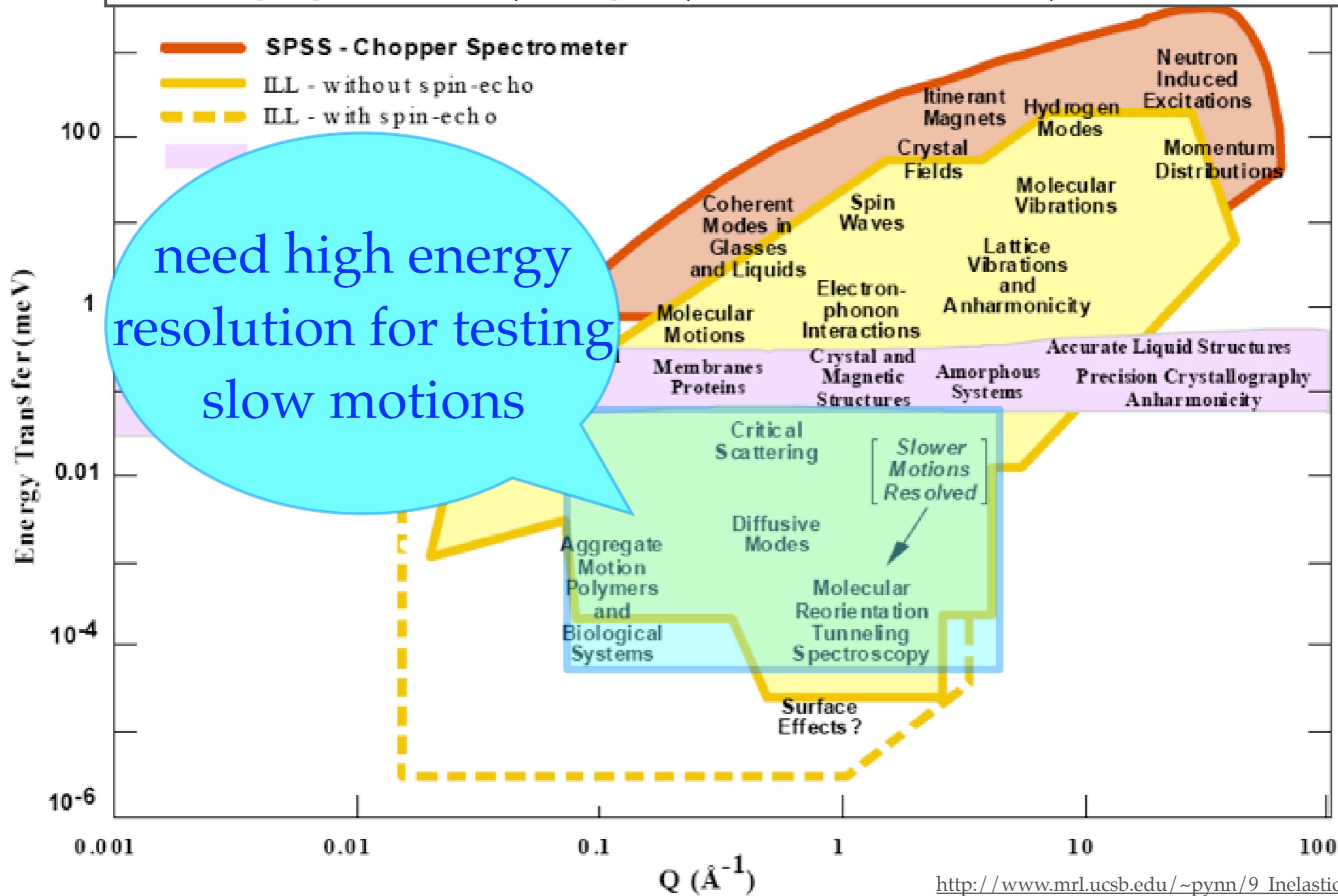


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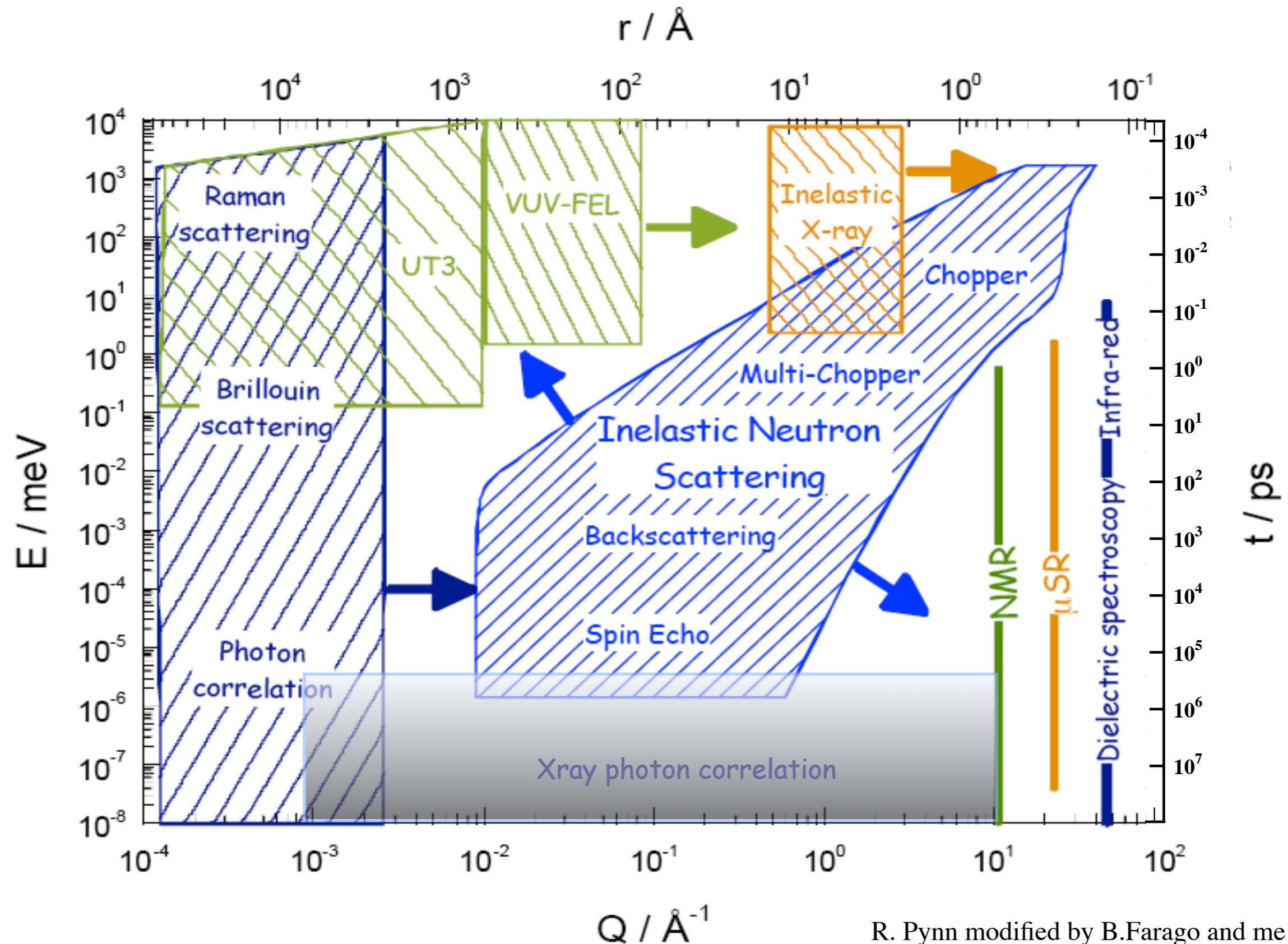
- why do we need high resolution neutron spectroscopy?
- how to achieve the best energy resolution
 - in time-of-flight spectroscopy
 - in crystal spectroscopy
 - in neutron spin echo
- some applications
 - slow diffusion processes
 - translational-, jump-, rotational diffusion
 - spectroscopy of local motions
 - EISF
 - quantum tunnelling

SCIENTIFIC APPLICATIONS

IN CONDENSED MATTER MAP

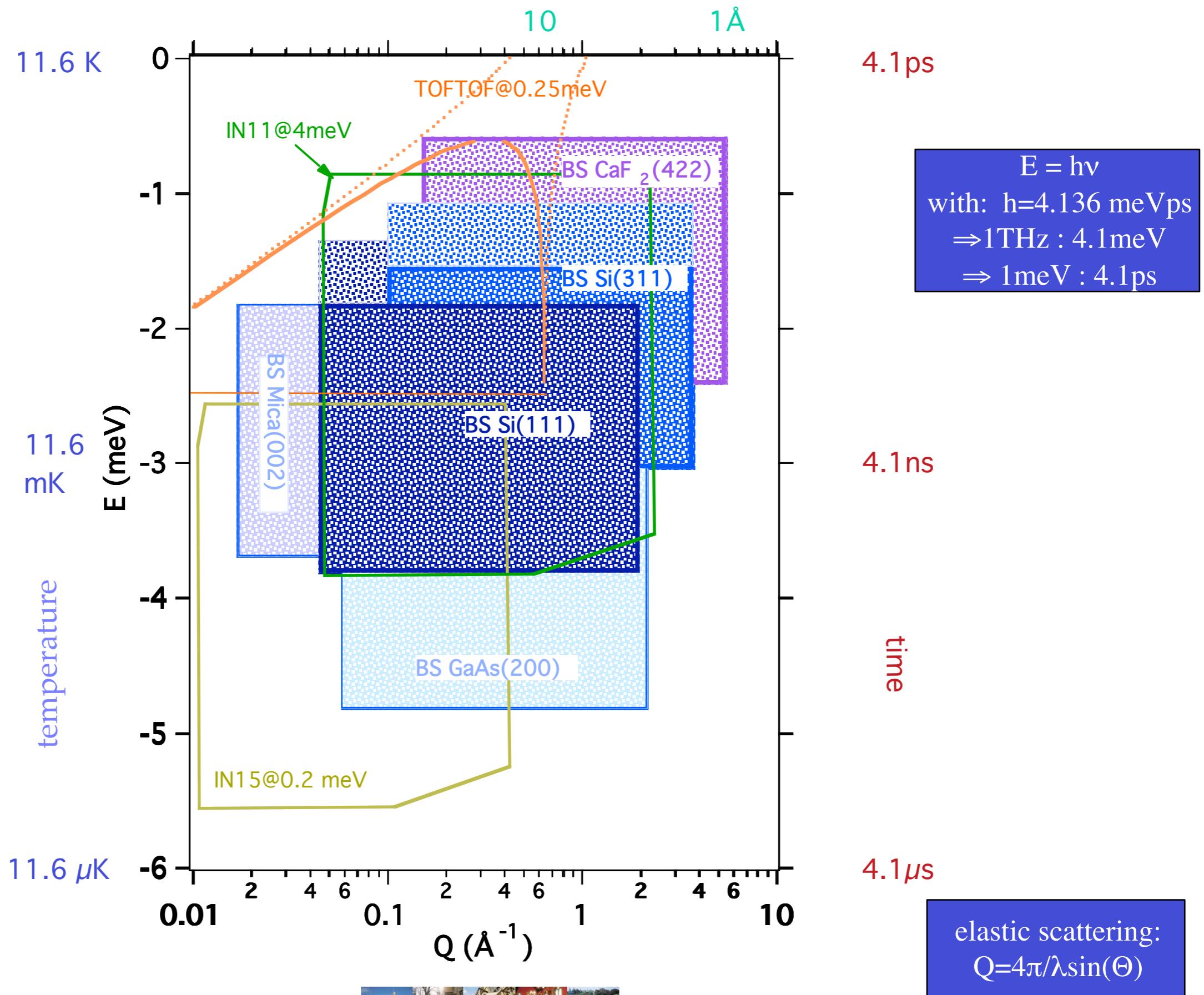


SPECTROSCOPY MAP



R. Pynn modified by B.Farago and me

HIGH ENERGY RESOLUTION MAP



density correlation functions - pair correlation function

T. Springer, 'Quasielastic Neutron Scattering for the Investigation of Diffusive Motions in Solids and Liquids', Springer Tracts in Modern Physics 64, 1972

for a classical system in terms of a microscopic particle density function:

$$G(\mathbf{r}, t) = \frac{1}{N} \int <\rho(\mathbf{r}' - \mathbf{r}, 0)\rho(\mathbf{r}', t)> d\mathbf{r}'$$

i.e. auto-correlation function of the particle density

asymptotic behaviour:

$$G^\infty(\mathbf{r}) = \lim_{\mathbf{r}, t \rightarrow \infty} G(\mathbf{r}, t) = \frac{1}{N} \int \bar{\rho}(\mathbf{r}' - \mathbf{r}) \bar{\rho}(\mathbf{r}') d\mathbf{r}'$$

liquid:

$$G^\infty(\mathbf{r}) = \bar{\rho} = \frac{N}{V}$$

density correlation functions - self correlation function

T. Springer, 'Quasielastic Neutron Scattering for the Investigation of Diffusive Motions in Solids and Liquids', Springer Tracts in Modern Physics 64, 1972

define average probability per volume to find particle i at \mathbf{r}' :

$$p_i(\mathbf{r}') = \langle \delta[\mathbf{r}' - \mathbf{r}_i(t)] \rangle$$

asymptotic behaviour:

$$G_s^\infty(\mathbf{r}) = \frac{1}{N} \sum_{i=1}^N \int p_i(\mathbf{r}' - \mathbf{r}) p_i(\mathbf{r}') d\mathbf{r}'$$

liquid:

$$\lim_{t \rightarrow \infty} G_s^\infty(\mathbf{r}, t) = \frac{1}{V}$$

thus: $G_s^\infty(\mathbf{r}) \rightarrow 0 \quad \text{for} \quad V \rightarrow \infty$
but not if V is finite: EISF!

assume identical scattering particles (drop index i)

$$G_s^\infty(\mathbf{r}) = \int p(\mathbf{r}' - \mathbf{r}) p(\mathbf{r}') d\mathbf{r}'$$

and separate \mathbf{G}_s in decaying and stationary part:

$$G_s'(\mathbf{r}, t) = \underline{G_s(\mathbf{r}, t)} - \underline{G_s^\infty(\mathbf{r})}$$

elastic incoherent structure factor:

$$S_{inc}^{el}(\mathbf{Q}) = \int \exp\{-i(\mathbf{Q}\mathbf{r})\} G_s^\infty(\mathbf{r}) d\mathbf{r} = |\int \exp\{-i(\mathbf{Q}\mathbf{r})\} p(\mathbf{r}) d\mathbf{r}|^2$$

density correlation functions - self correlation function

T. Springer, 'Quasielastic Neutron Scattering for the Investigation of Diffusive Motions in Solids and Liquids', Springer Tracts in Modern Physics 64, 1972

elastic **coherent** structure factor:

$$S_{coh}^{el}(\mathbf{Q}) = |\int \exp\{-i(\mathbf{Q}\mathbf{r})\} \rho(\mathbf{r}) d\mathbf{r}|^2$$

“... diffraction from different particles distributed in space...”

elastic **incoherent** structure factor:

$$S_{inc}^{el}(\mathbf{Q}) = |\int \exp\{-i(\mathbf{Q}\mathbf{r})\} p(\mathbf{r}) d\mathbf{r}|^2$$

“... diffraction of the neutron wave on the probability distribution in space of an individual particle spread over a finite volume...”

(thermal cloud due to vibrations, rotations, tunneling,)

thus we need high energy resolution in neutron spectroscopy, because

- ➊ of its suitable time resolution for some dynamic phenomena at the proper length scale
- ➋ it is in some cases the unique method, often it is complementary to other spectroscopic techniques (time & spatial probe)

content

 why do we need high resolution neutron spectroscopy?

• how to achieve the best energy resolution

- in time-of-flight spectroscopy (Toby Perring)
- in crystal spectroscopy
- in neutron spin echo (Ross Stewart)

• some applications

- slow diffusion processes
- translational-, jump-, rotational diffusion
- spectroscopy of local motions
 - EISF
 - quantum tunnelling

TOF-resolution

- fore more see talk by Toby Perring

- the art is to keep $\Delta t/t_{\text{elastic}}$ small
- short pulses: (fast choppers with narrow slits; multi-choppers, counter rotating...)
- long flight path
-
- long wavelength / slow neutrons - but then Q is limited (elastic $Q: 4\pi/\lambda \sin(\Theta)$)use of modern multi-chopper

content

why do we need high resolution neutron spectroscopy?

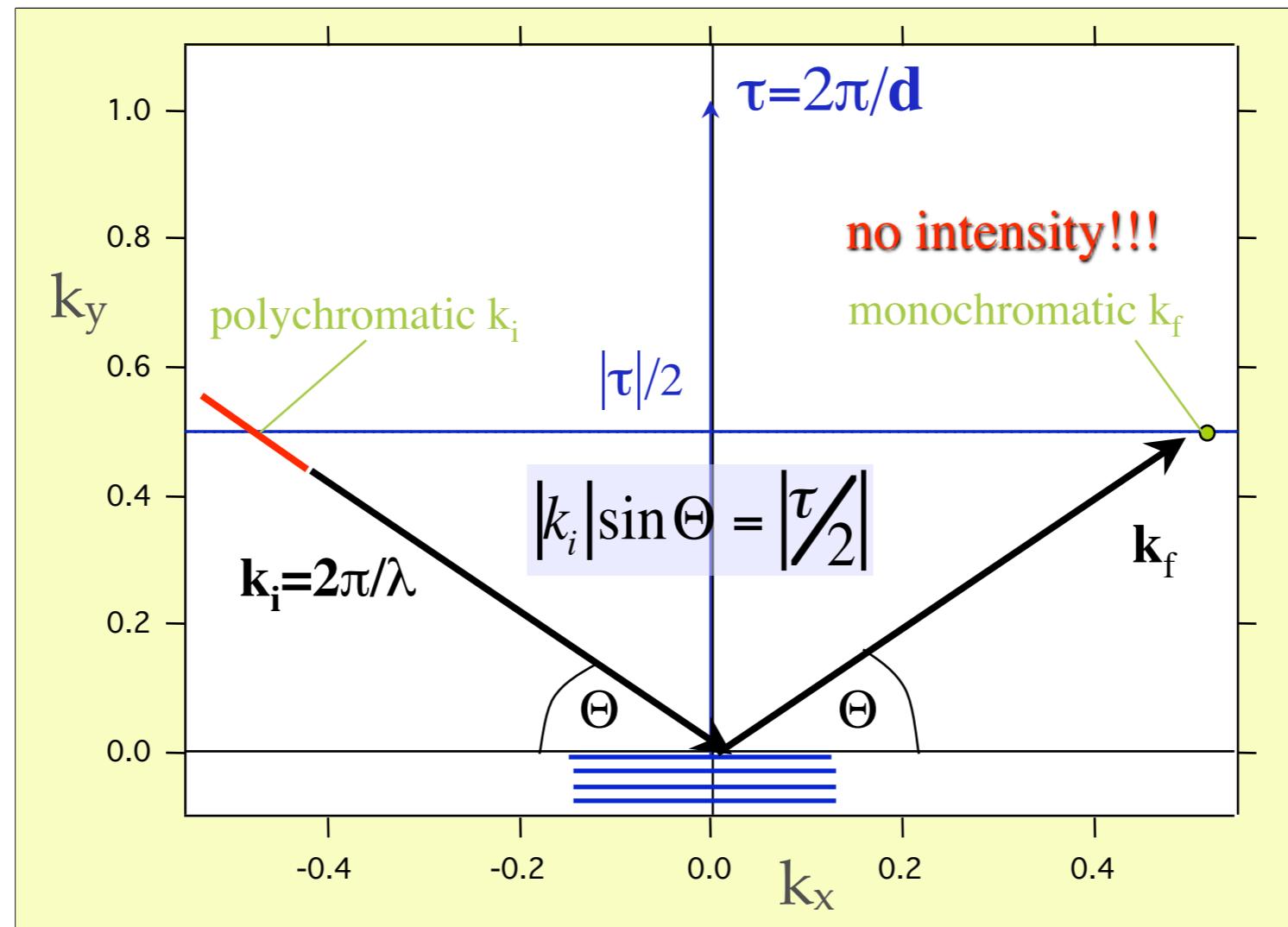
• how to achieve the best energy resolution

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• some applications

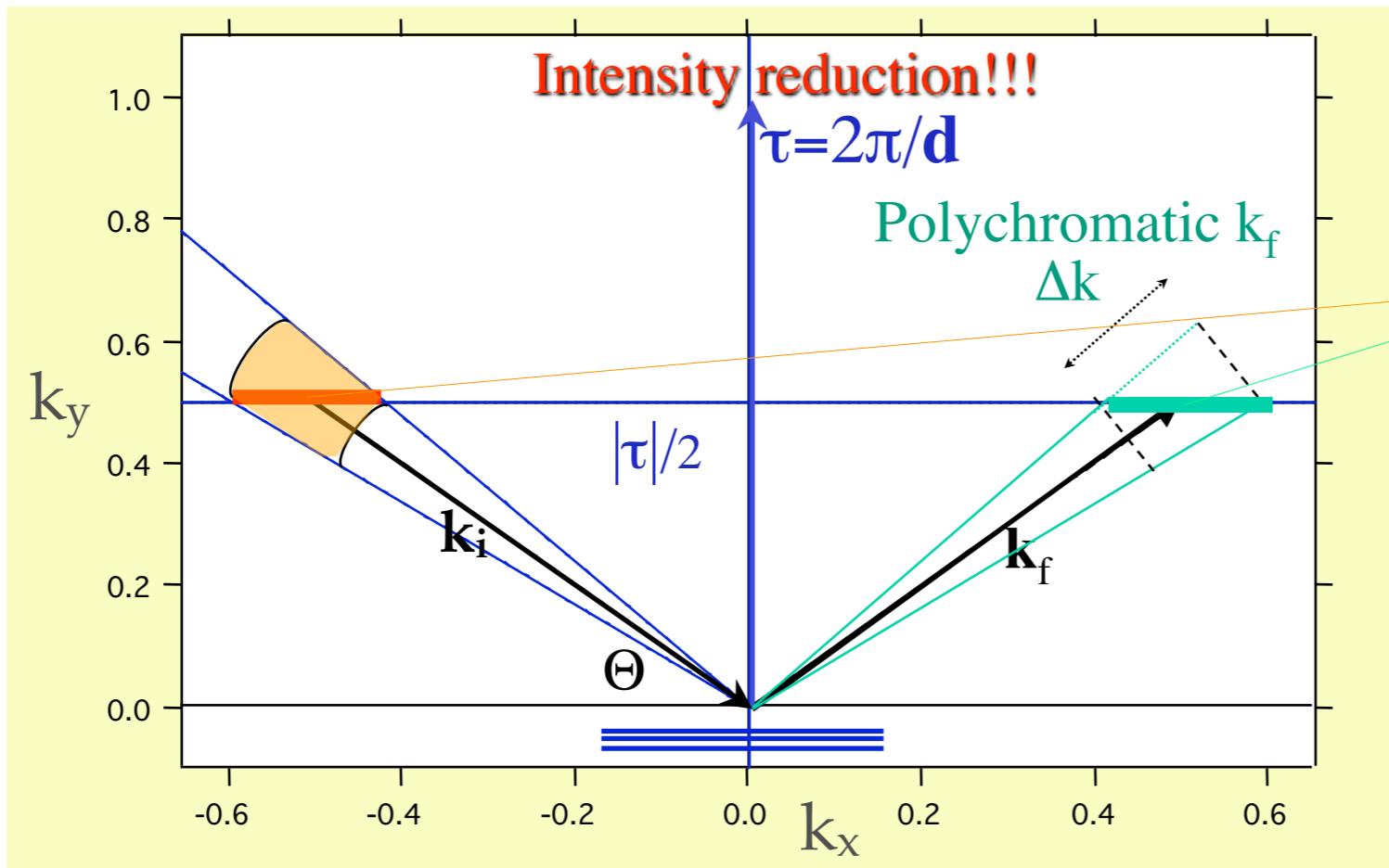
- slow diffusion processes
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ENERGY RESOLUTION FROM A PERFECT CRYSTAL



*reciprocal space representation
- perfectly collimated white beam*

ENERGY RESOLUTION FROM A PERFECT CRYSTAL AND DIVERGENT BEAM

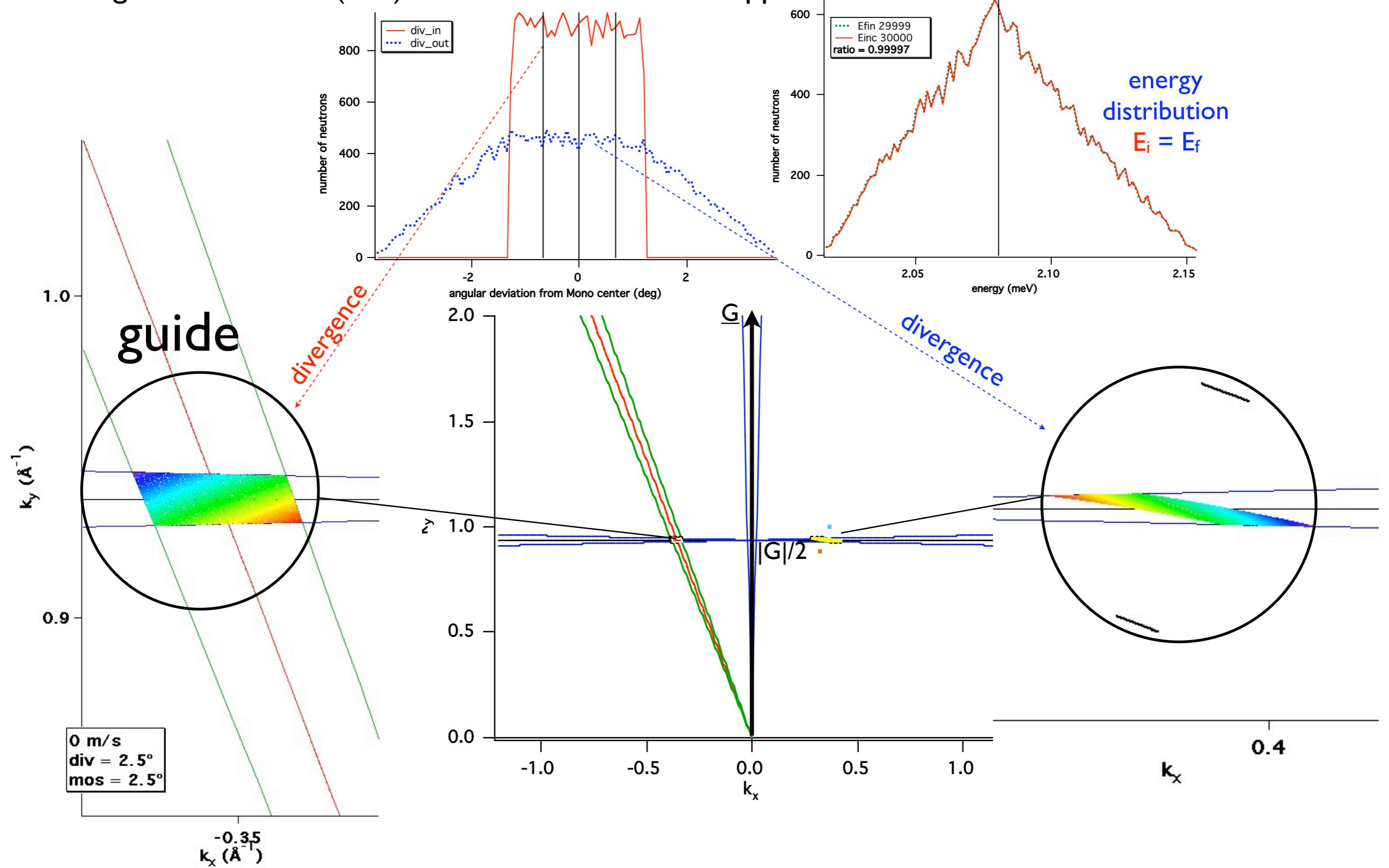


reciprocal space presentation
- open collimation and nearly white beam

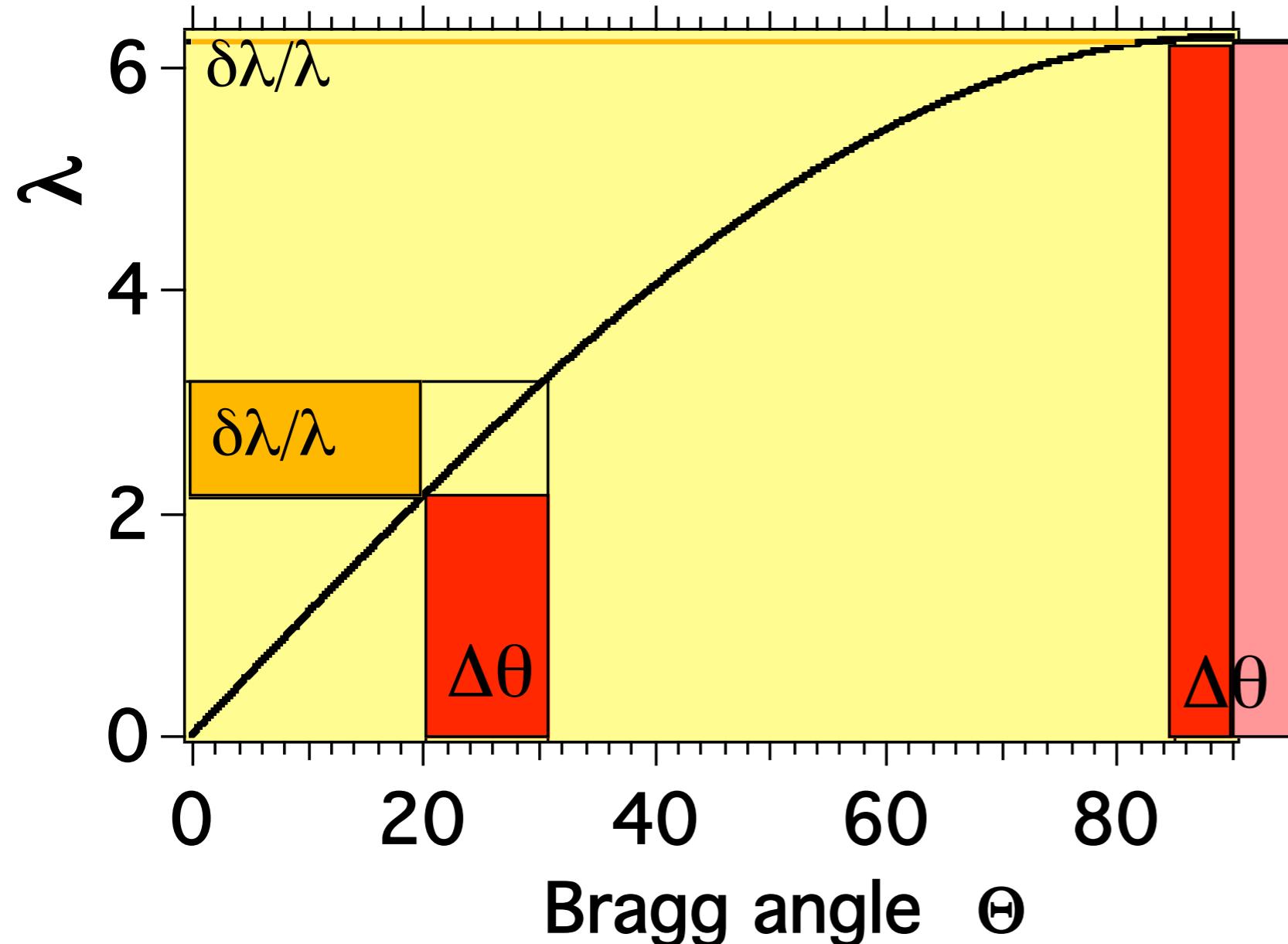
- intensity proportional phase space element
- the better the energy resolution, the lower the intensity
- energy resolution & divergence are coupled and depend on Θ

mosaic crystal

guide: $m=2.0$; PG(002) mosaic 2.5° ; distance chopper $d_{\text{DI-ch}}=2140\text{mm}$; focus width 50mm



ENERGY RESOLUTION AND DIVERGENCE



Bragg's law :

$$2d \sin\Theta = n\lambda$$

differentiate :

$$2\Delta d \sin\Theta + 2d \cos\Theta \Delta\theta = \Delta\lambda$$

or :

$$\frac{\Delta\lambda}{\lambda} = \frac{\Delta d}{d} + \cot\Theta \Delta\theta$$

$$\frac{\Delta\lambda}{\lambda} = \frac{\Delta k}{k} = \frac{\Delta E}{2E} \quad \text{and} \quad \frac{\Delta d}{d} = \frac{\Delta \tau}{\tau}$$

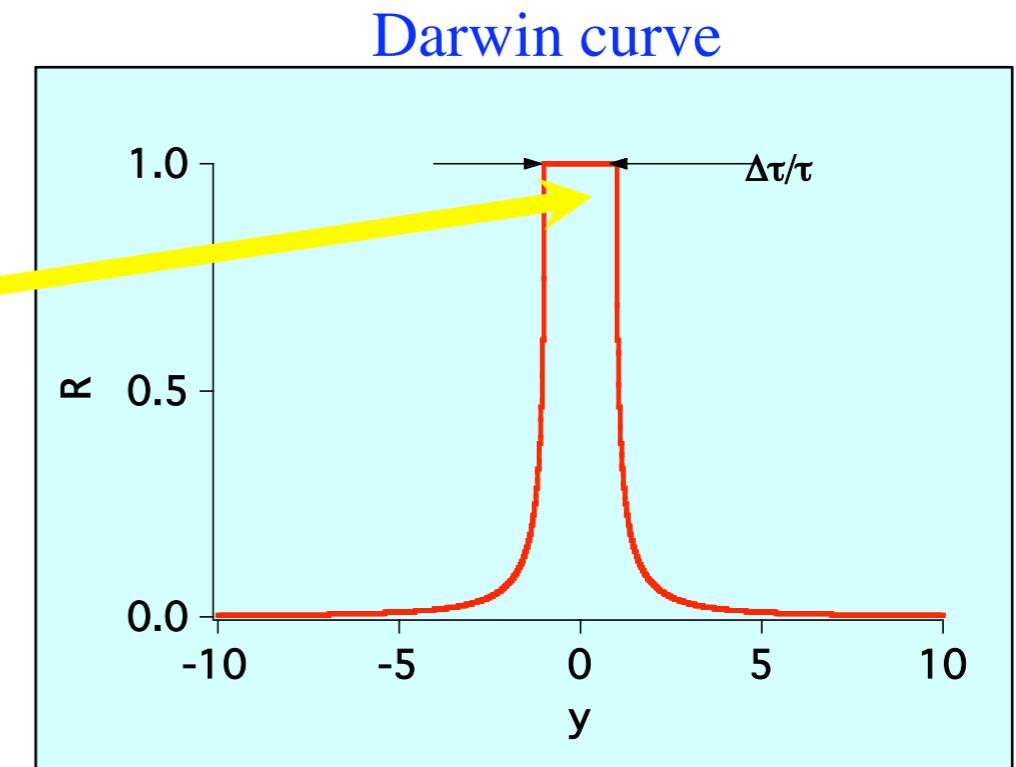
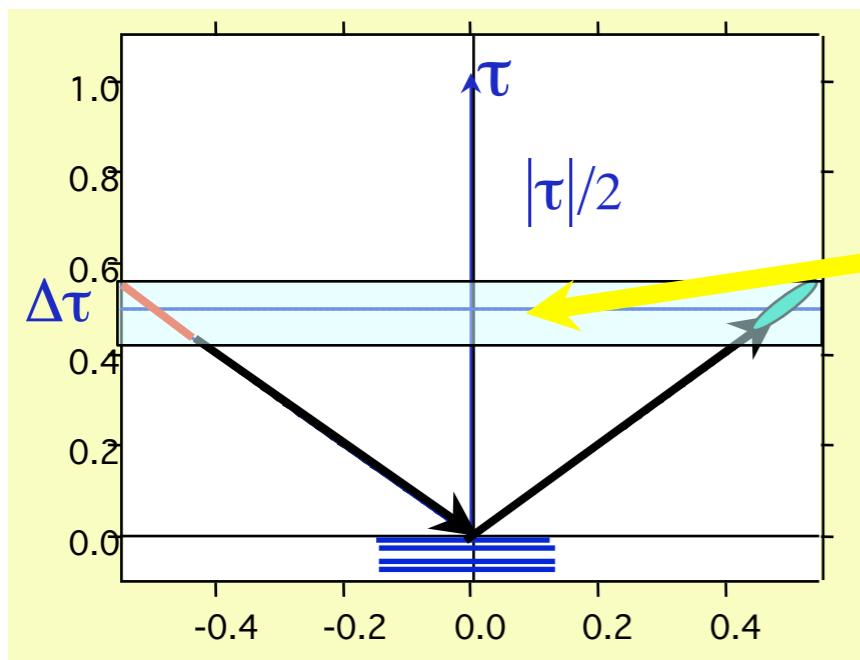
$$\frac{\Delta\lambda}{\lambda} = \frac{\Delta\tau}{\tau} + \frac{\Delta\theta^2}{8} + \frac{\Delta\varepsilon^2}{4}$$

ε = deviation from BS

$\Delta\theta$ = beam divergence

- energy resolution improves towards backscattering
- decoupling of divergence and energy resolution

REFLECTIVITY OF CRYSTALS IN DYNAMIC SCATTERING THEORY



- **primary extinction** leads to finite energy resolution for perfect crystals

$$R = \begin{cases} 1 & \text{for } |y| \leq 1 \\ \left(|y| - \sqrt{y^2 - 1} \right)^2 & \text{for } |y| > 1 \end{cases}$$

HOW LARGE IS THE PRIMARY EXTINCTION FOR A BS-CRYSTAL?

$$\frac{\Delta\tau}{\tau} = \frac{16\pi F_\tau N}{\tau^2}$$

N=number density of unit cells

F= structure factor

$$F_\tau = \sum_{sites\ i\ in\ cell} b_{coh}^i \cdot DWF_i \cdot \exp(-i\vec{Q}\vec{R}_i)$$

for neutrons:

$$\Delta E = 2E \frac{\Delta\tau}{\tau}$$

$$\Delta E = 2 \frac{\hbar^2}{2m} \left(\frac{\tau}{2}\right)^2 \frac{16\pi F_\tau N}{\tau^2} = \frac{\hbar^2}{m} 4\pi F_\tau N$$

Typical backscattering monochromators:

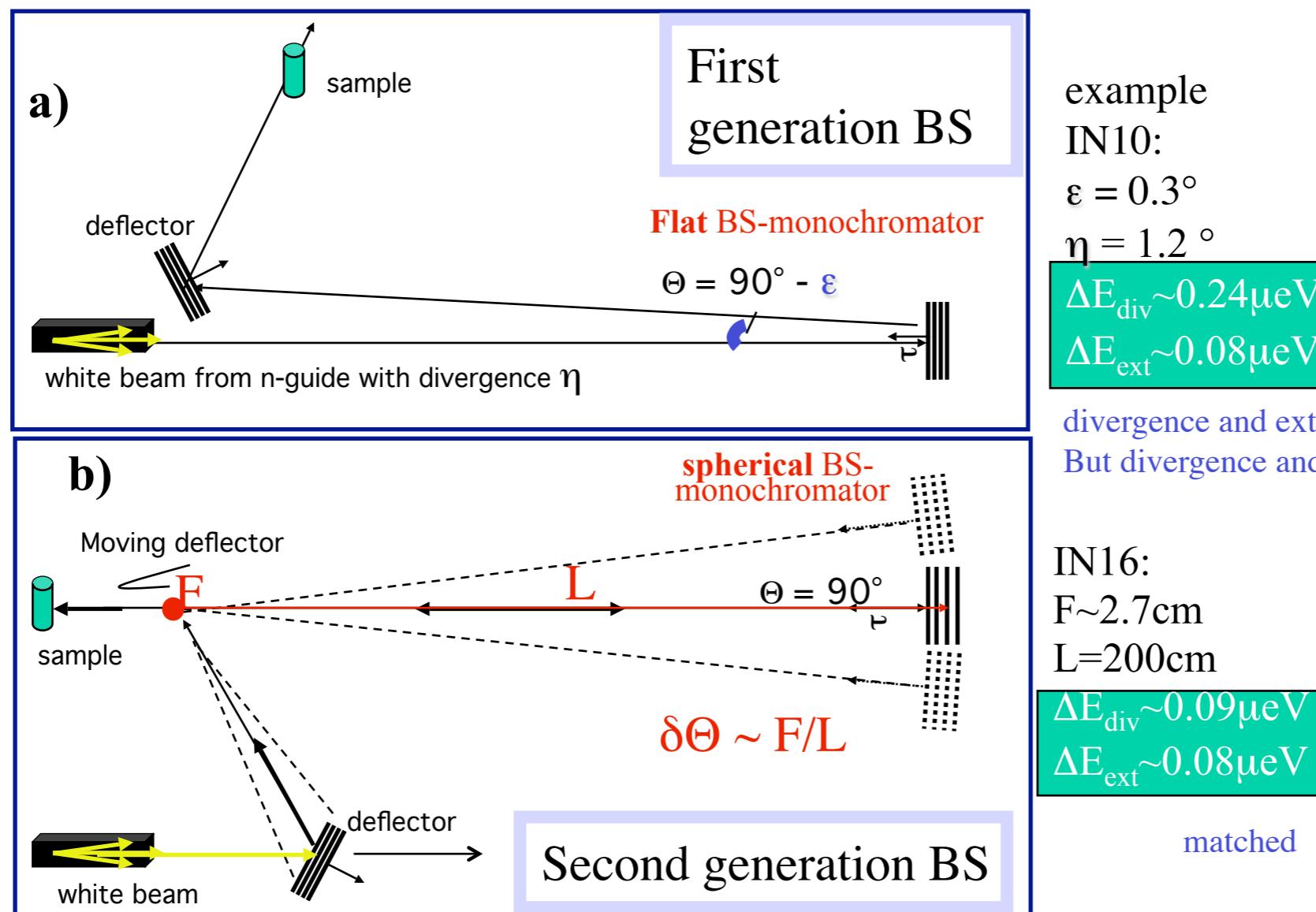
Crystal plane	$\frac{\Delta\tau}{\tau}$	$\Delta E_{ext} (\mu eV)$	$\lambda (\text{\AA})$ for $\Theta = 90^\circ$
Si (111)	$1.86 \cdot 10^{-5}$	0.077	6.2708
Si (311)	$0.51 \cdot 10^{-5}$	0.077	3.2748
Ca F ₂ (111)	$1.52 \cdot 10^{-5}$	0.063	6.307
Ca F ₂ (422)	$0.54 \cdot 10^{-5}$	0.177	2.23
Ga As (400)	$0.75 \cdot 10^{-5}$	0.153	2.8269
Ga As (200)	$0.157 \cdot 10^{-5}$	0.008	5.6537
Graphite (002)	$12 \cdot 10^{-5}$	0.44	6.70

<http://www.ill.fr/YellowBook/IN16/BS-review/index.htm>

- energy resolution independent of order of reflection (for same F)
- this is different for X-rays
- extremely small crystal contribution (other resolution contributions for TOF or 3X-spectrometers much larger)

HOW TO GET THE SAMPLE AND DETECTOR OFF FROM PRIMARY BEAM?

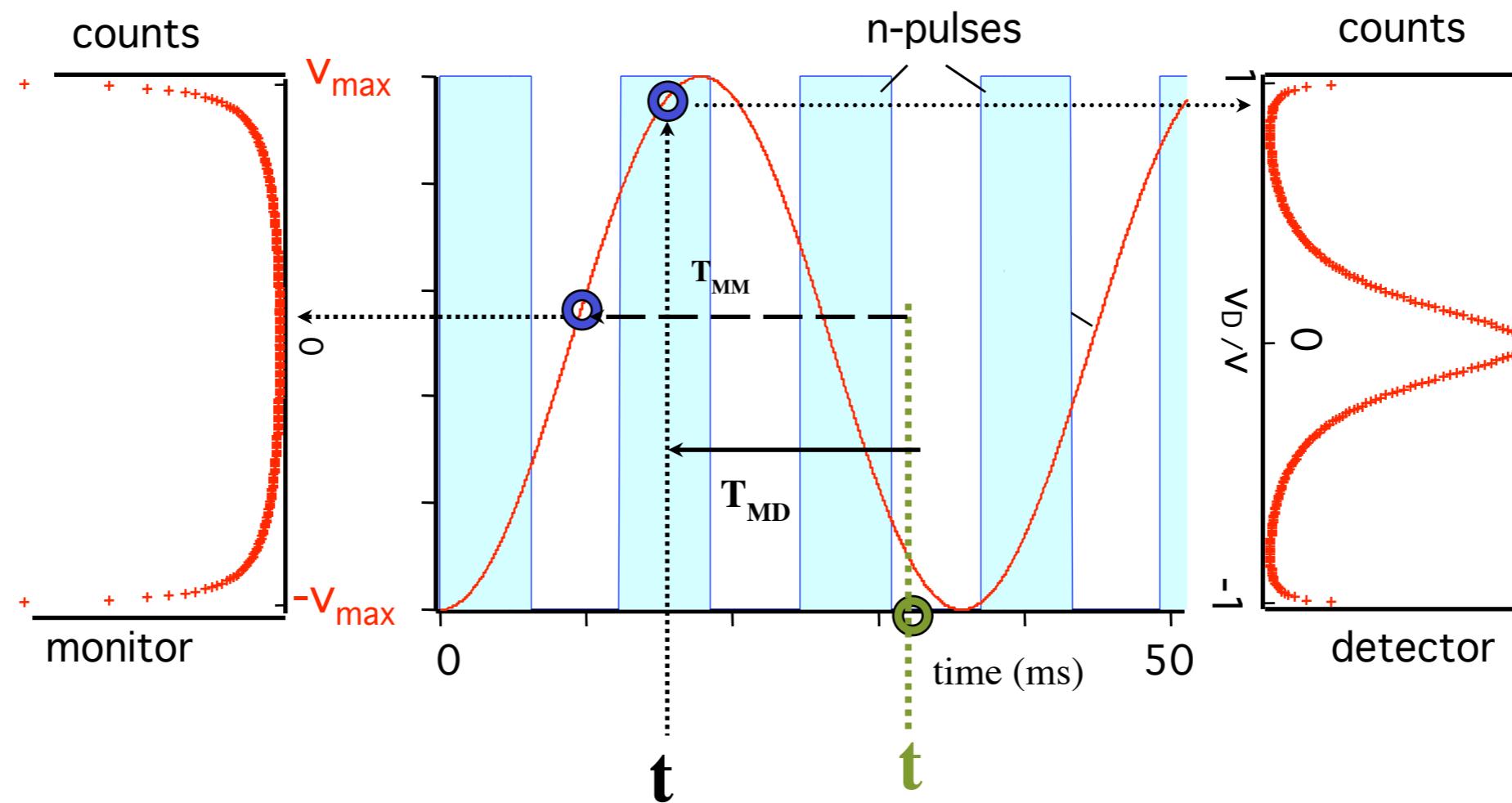
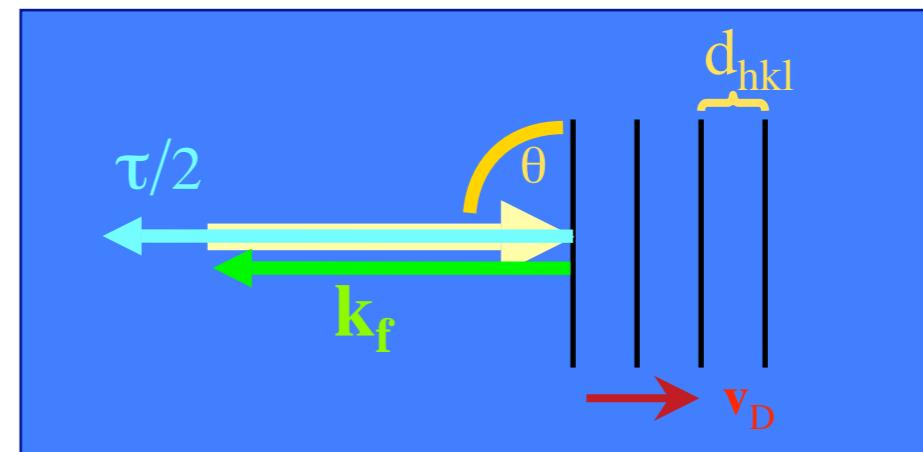
generic primary spectrometer concepts:



HOW TO SCAN THE ENERGY AND REMAIN IN BS?

1) Doppler motion of the monochromator:

$$\frac{\delta E_D}{E_i} \approx 2 \frac{v_D}{v_i}$$

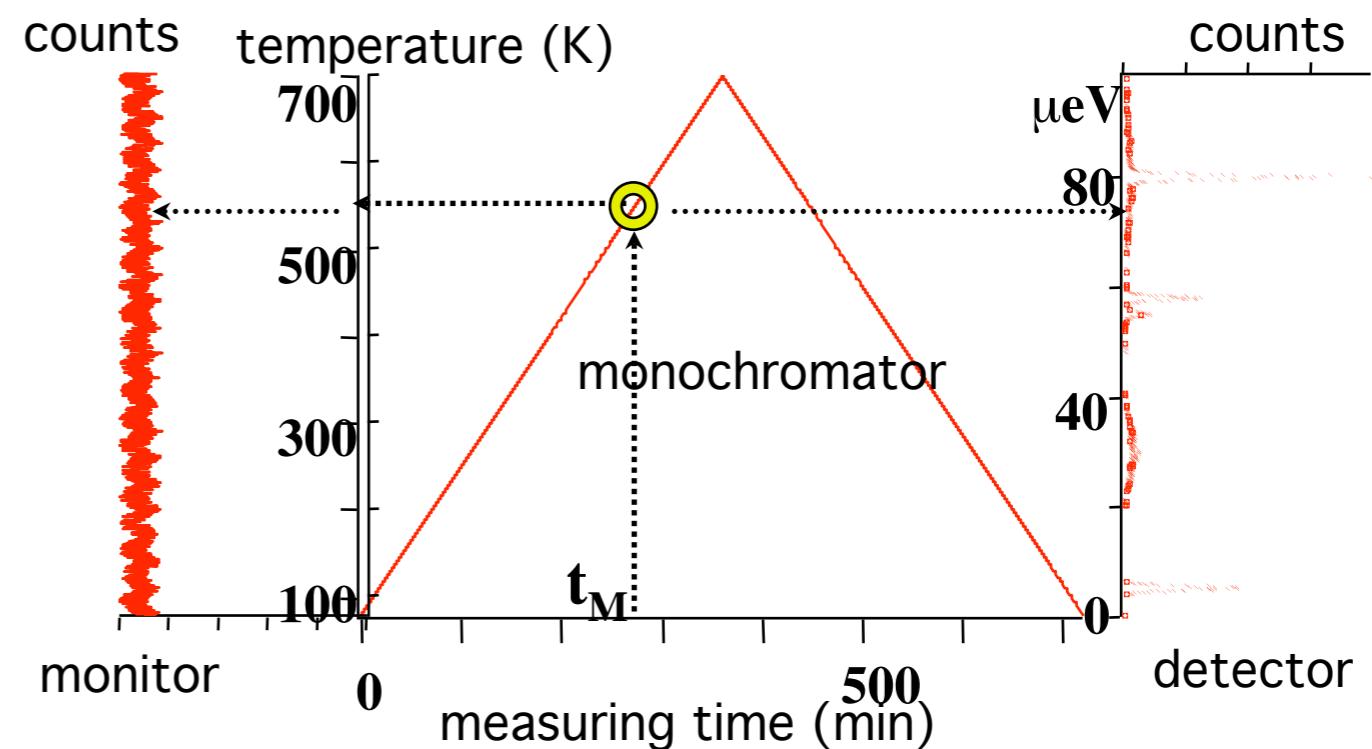
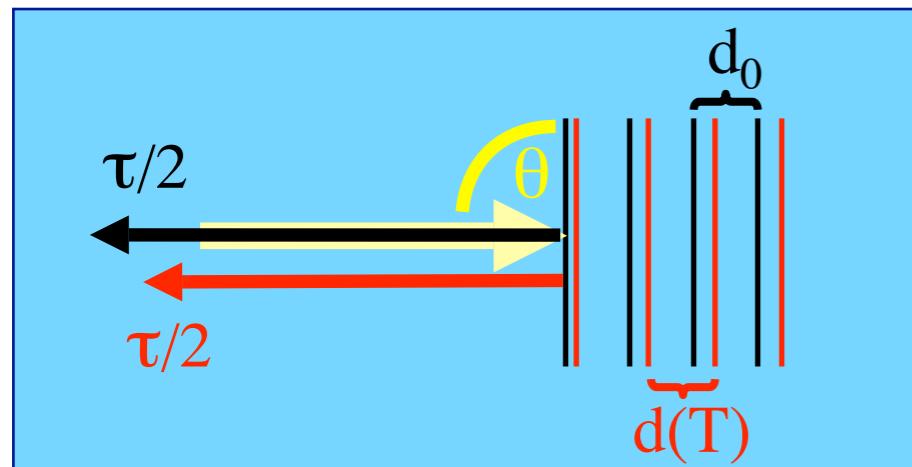


HOW TO SCAN THE ENERGY AND REMAIN IN BS? (2)

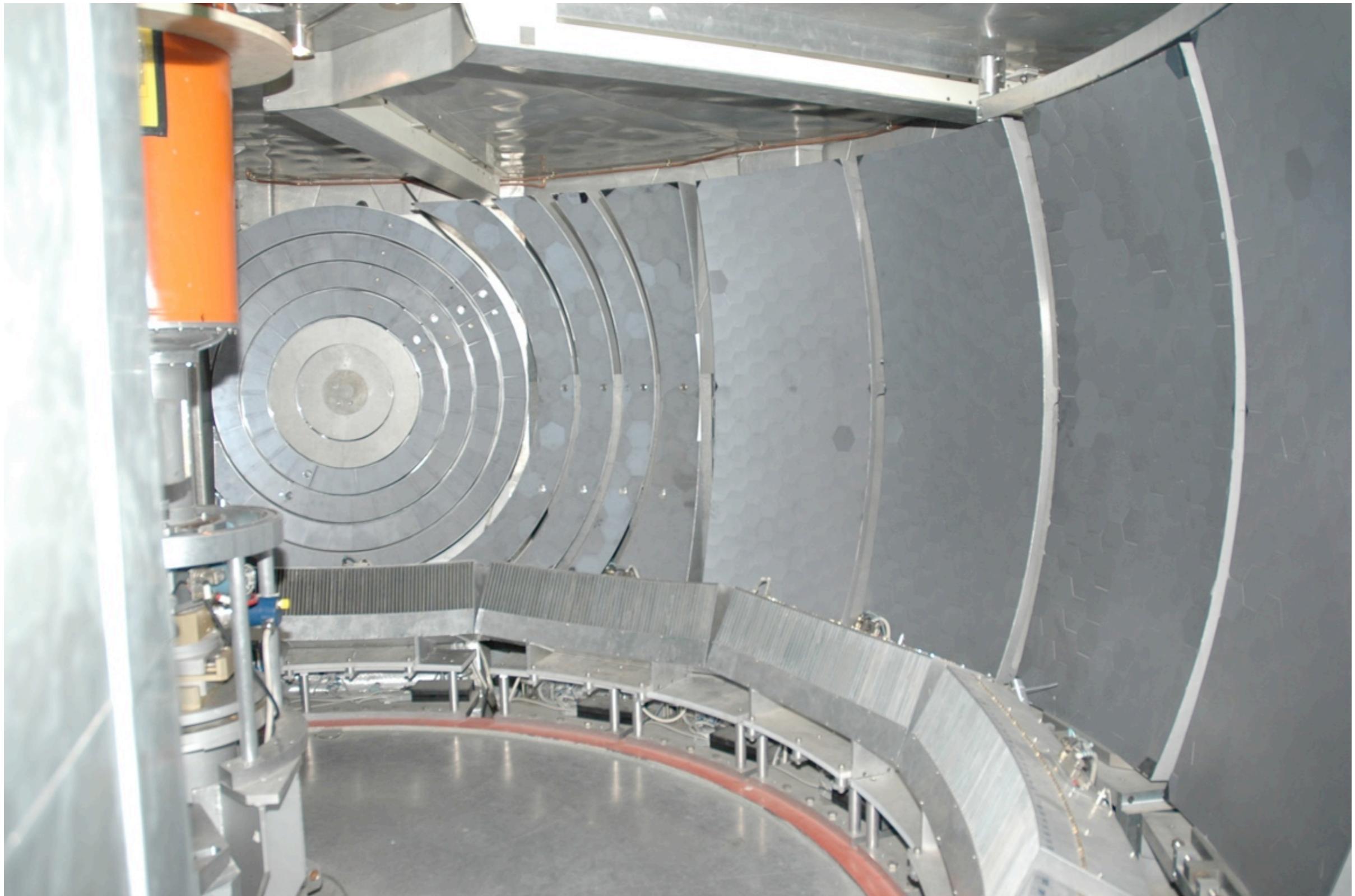
2) Change of the *monochromator temperature* = change of lattice distance :

$$d(T) = d_0 (1 + \alpha T + O(2\dots))$$

$$\frac{\delta E}{E_i} = \frac{\delta d(T)}{d_0}$$

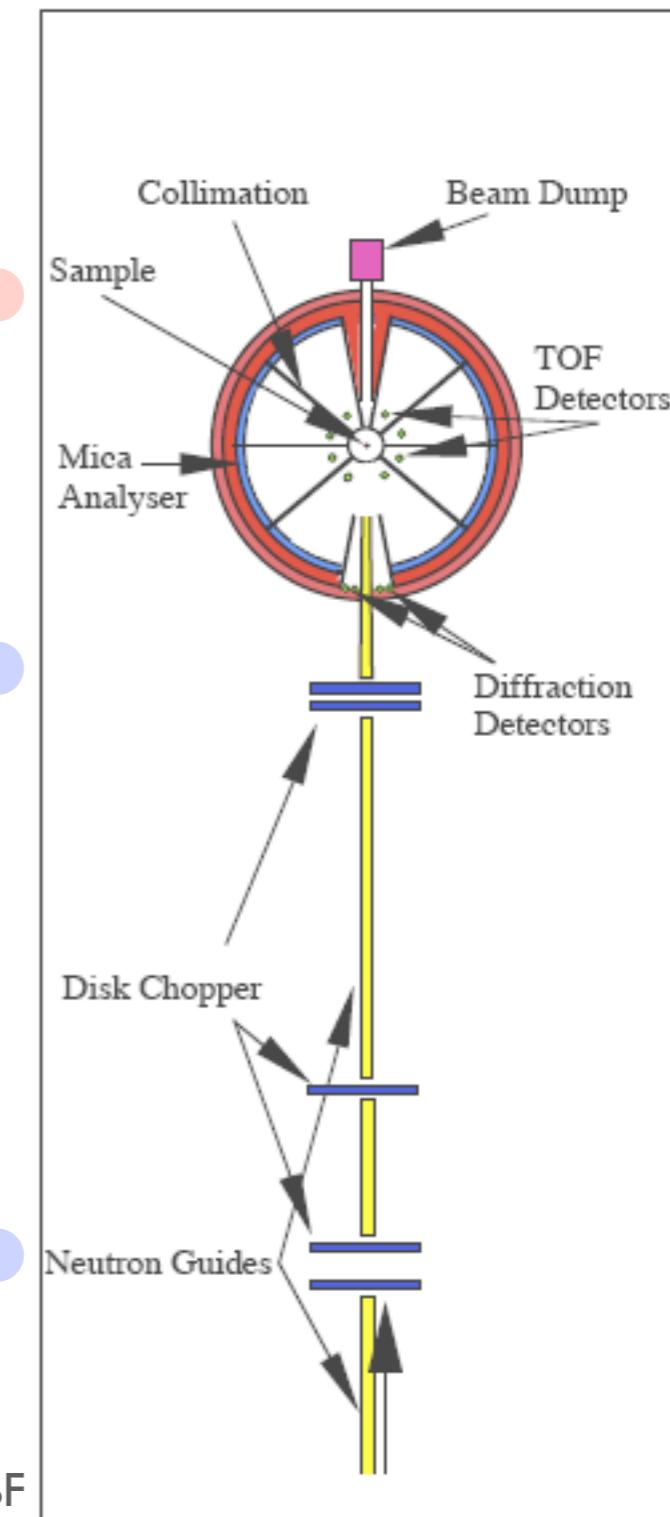
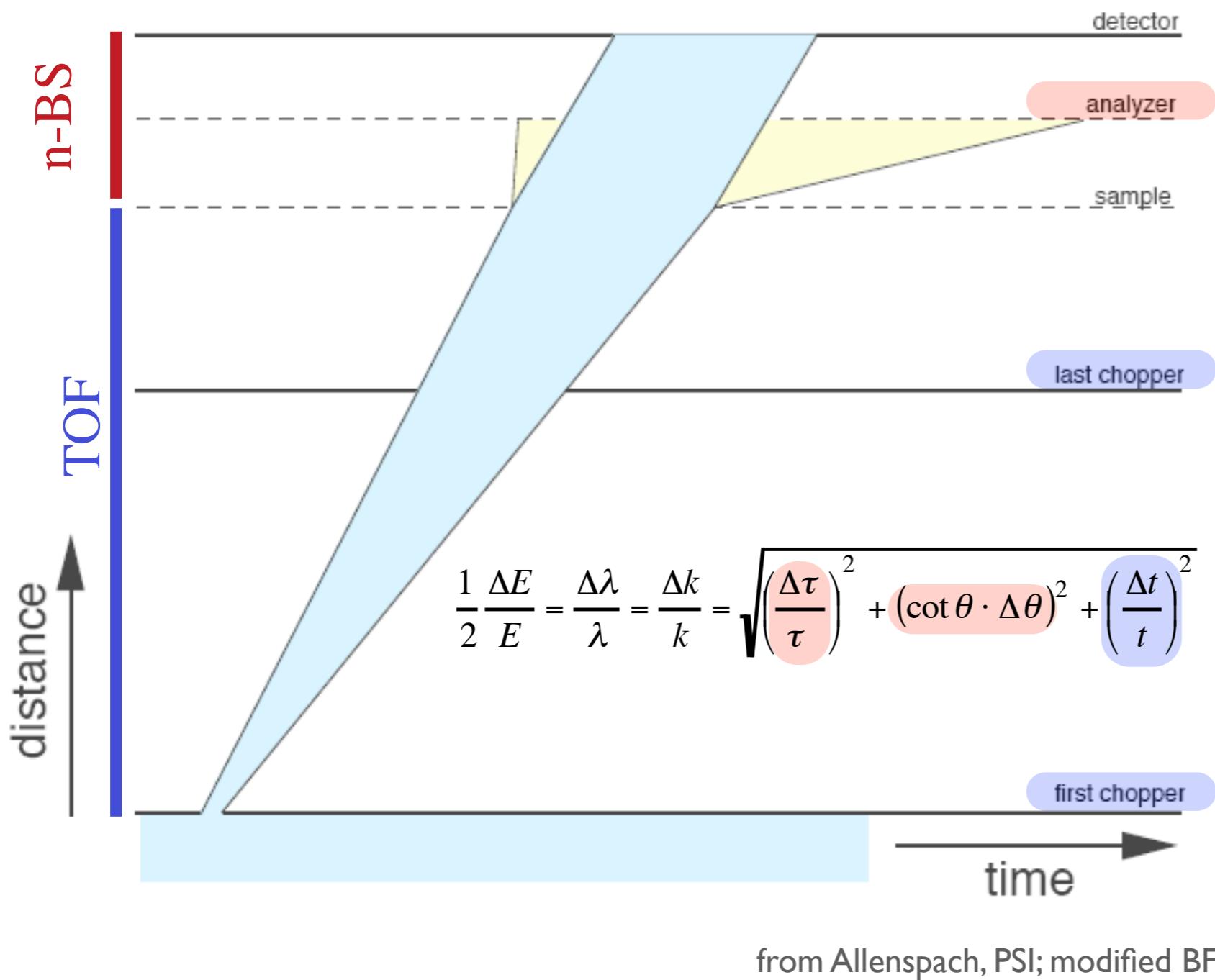


IN16 - reactor cold neutron backscattering instrument



Inverted TOF spectrometers near backscattering

examples: IRIS-IRIS, MARS-PSI, BASIS-SNS



content

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-  in neutron spin echo (Ross Stewart)

• some applications

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Neutron Spinecho

fore more see talk by Ross Stewart

Echo condition:

$$\int_{\pi/2}^{\pi} B_1 d\ell = \int_{\pi}^{\pi/2} B_2 d\ell$$

The measured quantity is: $S(q,t)/S(q,0)$
where

$$t \propto \lambda^3 \int B d\ell$$

For elastic scattering:

$$\varphi_{tot} = \frac{\gamma B_1 l_1}{v_1} - \frac{\gamma B_2 l_2}{v_2} = 0$$



For omega energy exchange:

$$\varphi_{tot} = \frac{\hbar \gamma B l}{m v^3} \omega + o \left(\left(\frac{\omega}{1/2 m v^2} \right)^2 \right)$$



The probability of omega energy exchange:

$$S(q, \omega)$$

The final polarization: $\langle \cos \varphi \rangle = \frac{\int \cos(\frac{\hbar \gamma B l}{m v^3} \omega) S(q, \omega) d\omega}{\int S(q, \omega) d\omega} = S(q, t)$

from Bela Farago, ILL

Fourier time (in nsec):

8Å		10Å		16Å		20Å	
t min	t max						
0.12	26.3	0.34	51.5	0.8	174	1.4	300



content

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how to achieve a good energy resolution

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some applications

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applications of high resolution spectroscopy

- low frequency inelastic spectroscopy
 - tunneling spectroscopy
 - hyperfine interaction
 - ^4He excitation spectrum (roton)
 - aerogels & fractons
- quasielastic spectroscopy
 - Diffusion in metals, alloys, intercalation compounds, hydrogen in metals, fuel cells, membranes
 - Dynamics in polymers and biological systems
 - Dynamics of the phase transition and glass transition in molecular glasses and spin glasses
 - Atomic and molecular motion on surfaces and in confinement
 - Critical scattering near phase transitions
 - Dynamics of liquid crystals and liquids
 - Dynamics of hydrogen bonds
 - rotations of molecules, confinement

density correlation functions - self correlation function

T. Springer, 'Quasielastic Neutron Scattering for the Investigation of Diffusive Motions in Solids and Liquids', Springer Tracts in Modern Physics 64, 1972

define average probability per volume to find particle i at \mathbf{r}' :

$$p_i(\mathbf{r}') = \langle \delta[\mathbf{r}' - \mathbf{r}_i(t)] \rangle$$

asymptotic behaviour:

$$G_s^\infty(\mathbf{r}) = \frac{1}{N} \sum_{i=1}^N \int p_i(\mathbf{r}' - \mathbf{r}) p_i(\mathbf{r}') d\mathbf{r}'$$

liquid:

$$\lim_{t \rightarrow \infty} G_s^\infty(\mathbf{r}, t) = \frac{1}{V}$$

assume identical scattering particles (drop index i)

$$G_s^\infty(\mathbf{r}) = \int p(\mathbf{r}' - \mathbf{r}) p(\mathbf{r}') d\mathbf{r}'$$

elastic incoherent structure factor:

$$S_{inc}^{el}(\mathbf{Q}) = \int \exp\{-i(\mathbf{Q}\mathbf{r})\} G_s^\infty(\mathbf{r}) d\mathbf{r} = |\int \exp\{-i(\mathbf{Q}\mathbf{r})\} p(\mathbf{r}) d\mathbf{r}|^2$$

high energy/time resolution needed!

typical incoherent scattering law

$$S_{inc}(\mathbf{Q}, \omega) = A_0(\mathbf{Q})\delta(\omega) + (1 - A_0(\mathbf{Q}))\mathbf{L}(\mathbf{Q}, \omega)$$

elastic, EISF
stationary part

quasi-elastic
decaying part

for a given \mathbf{Q} :

$$\int_{-\infty}^{\infty} S_{inc}(\mathbf{Q}, \omega) d\omega = 1$$

$$EISF = \frac{S_{inc}^{el}(\mathbf{Q})}{S_{inc}^{el}(\mathbf{Q}) + S_{inc}^{qel}(\mathbf{Q})}$$

simple incoherent scattering law: isotropic translational diffusion

no stationary part -> no elastic scattering,

time space

$$S_{inc}(\mathbf{Q}, t) = \exp(-DQ^2 t)$$

relaxation rate $|\tau| = 1/(DQ^2)$

energy space

$$S_{inc}(Q, \omega) = \frac{1}{\pi} \frac{DQ^2}{\omega^2 + (DQ^2)^2}$$

Lorentzian with energy width
 $HWHM = \Gamma/2 = \hbar DQ^2$

D= self diffusion constant [m²/s]

$D \sim \exp(-E_a/kT)$

$$Q = 1 \text{ \AA}^{-1} : D = \frac{\Gamma \pi}{h Q^2} = \frac{\Gamma [\mu eV] \pi}{4.136 [\mu eV ns] 10^{16} [cm^2]} \approx \frac{3}{4} \Gamma [\mu eV] \cdot 10^{-7} \frac{cm^2}{s}$$

example:

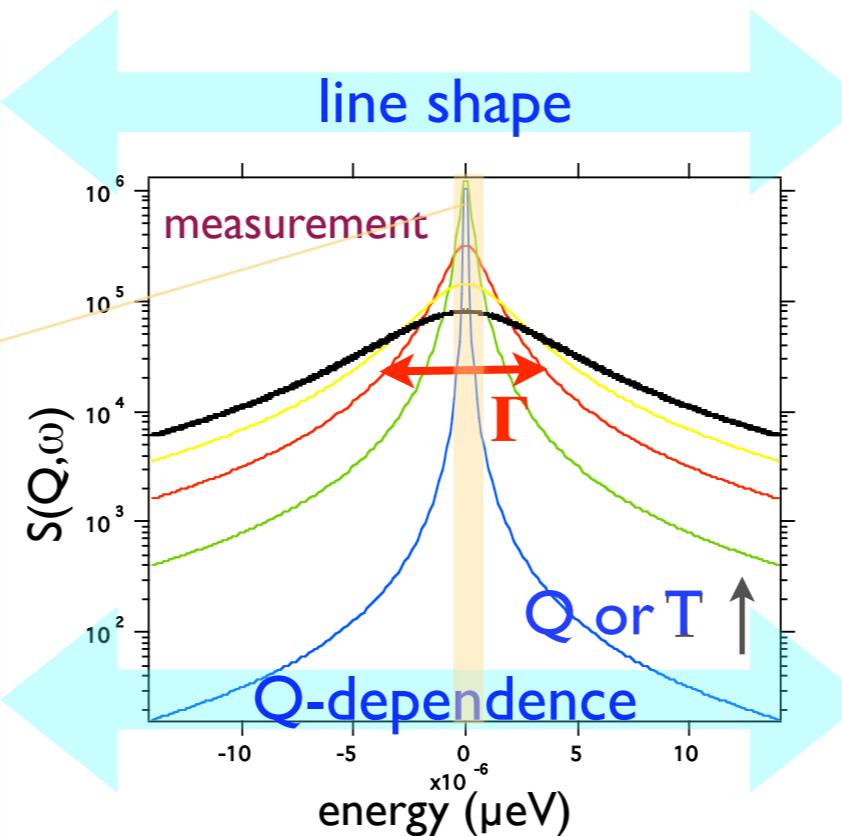
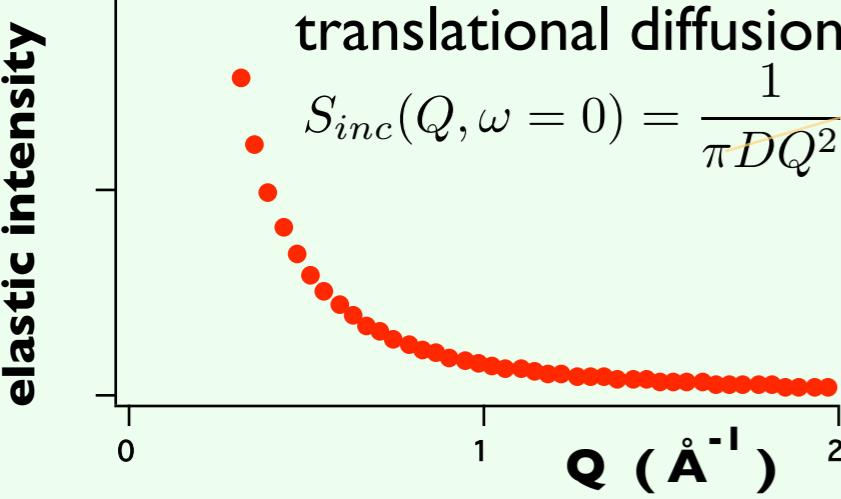
$D(\text{H}_2\text{O} @ RT) \sim 2 \cdot 10^{-5} \text{ cm}^2/\text{s}$
 $\text{FWHM} \sim 270 \text{ } \mu\text{eV} @ 1 \text{ \AA}^{-1}; \tau = 5 \text{ ps}$
 $\text{FWHM} \sim 2.7 \text{ } \mu\text{eV} @ 0.1 \text{ \AA}^{-1}; \tau = 50 \text{ ps}$
 $\Rightarrow \text{TOF dynamic range}$

simple incoherent scattering law:

isotropic translational diffusion

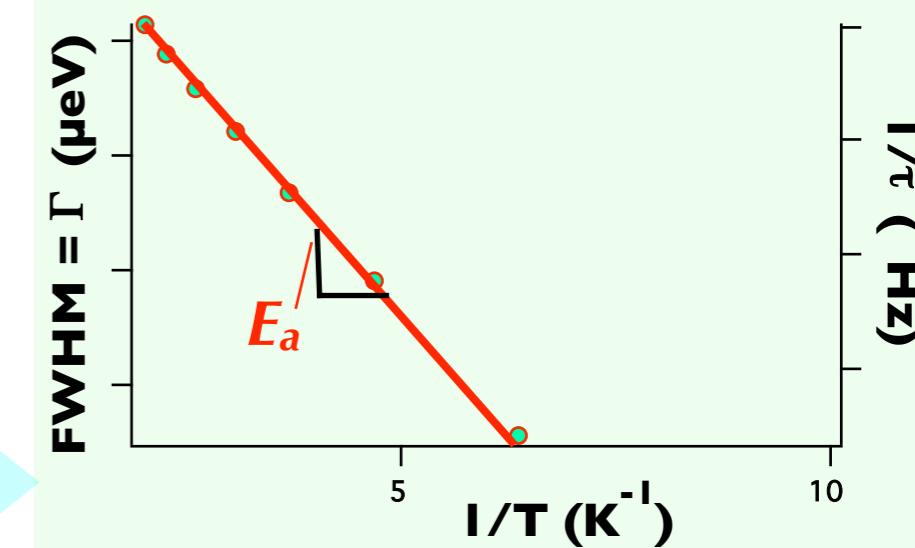
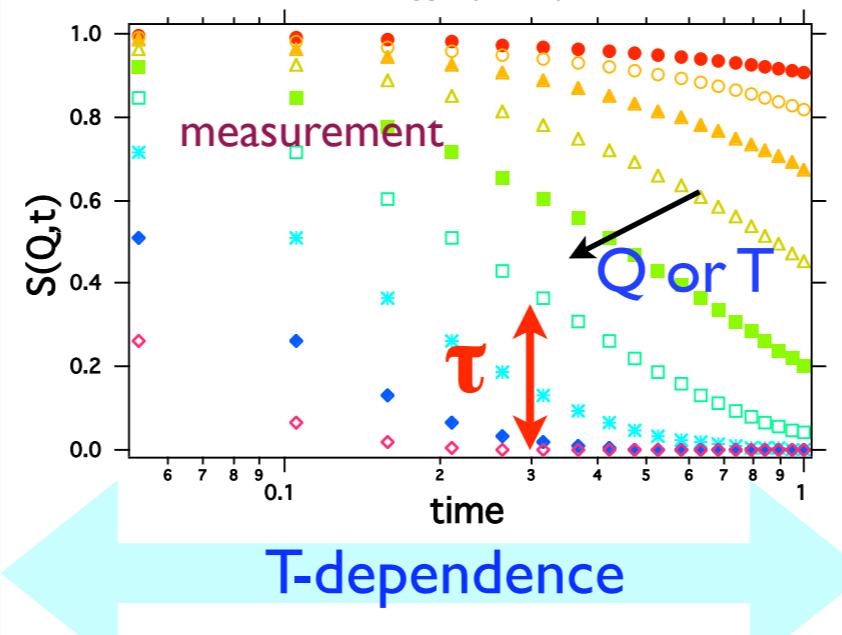
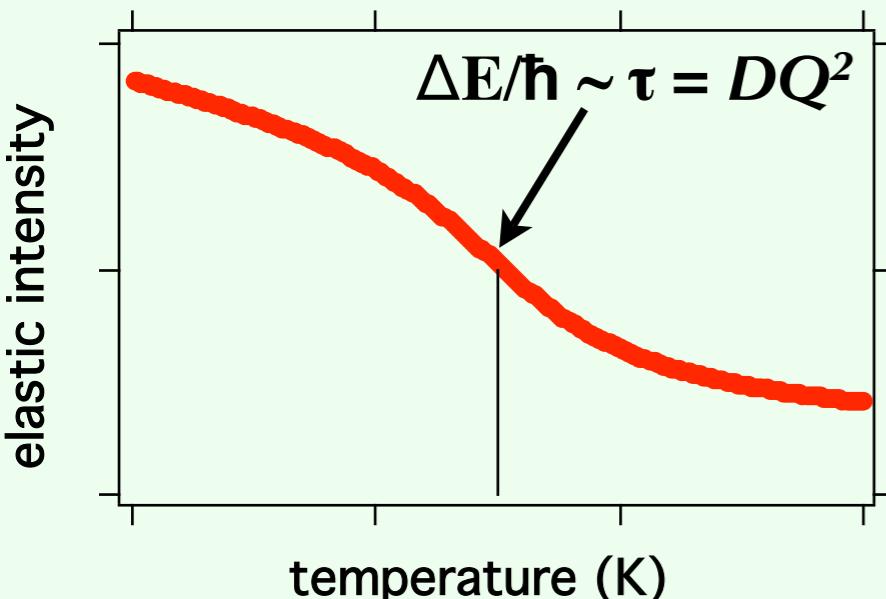
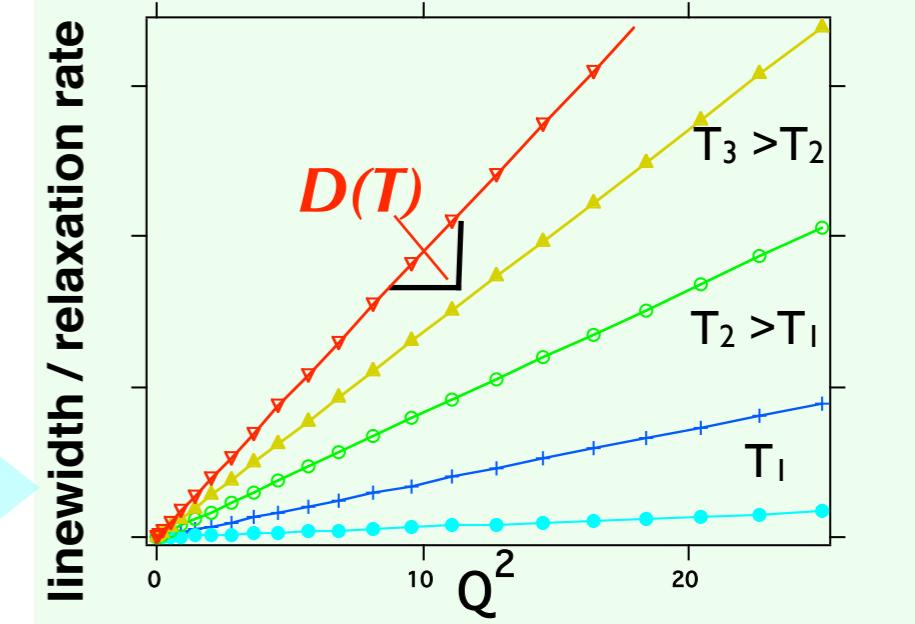
no stationary part -> no elastic scattering

e.g. elastic scans on BS



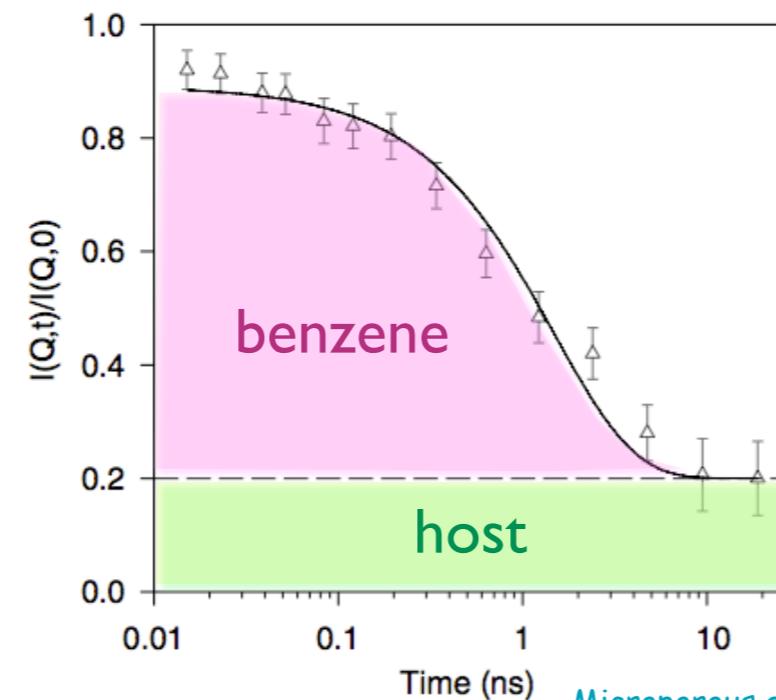
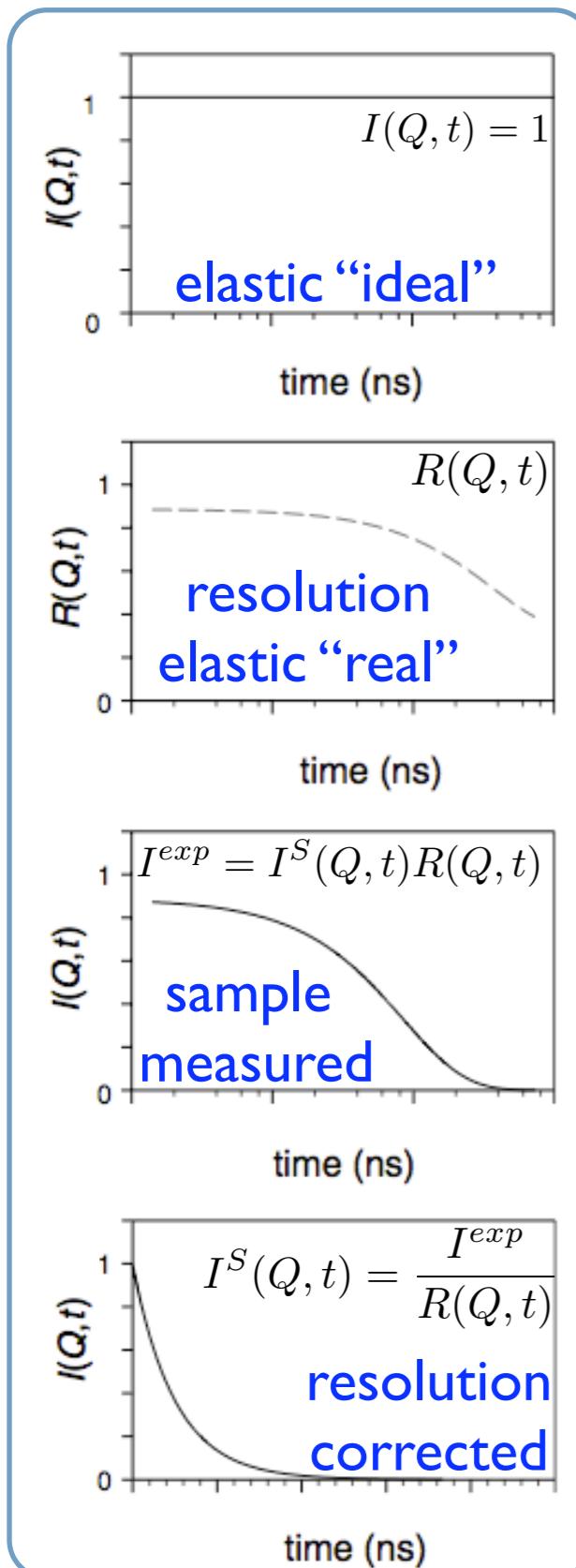
t- or E-spectroscopy on NSE or TOF, BS

Lorentzian with $\Gamma/2 = \hbar D Q^2 = \hbar/\tau$



simple coherent scattering law: transport diffusion - example experiment

neutron spin echo



H. Jobic, Theodorou
Microporous and Mesoporous Materials 102 (2007) 21

Fig. 6. Normalized intermediate scattering function obtained for benzene in NaY zeolite (1 molecule per supercage, on average, $T = 475$ K, $Q = 0.3 \text{ \AA}^{-1}$). The fraction of the intensity due to the zeolite is represented as a dashed line.

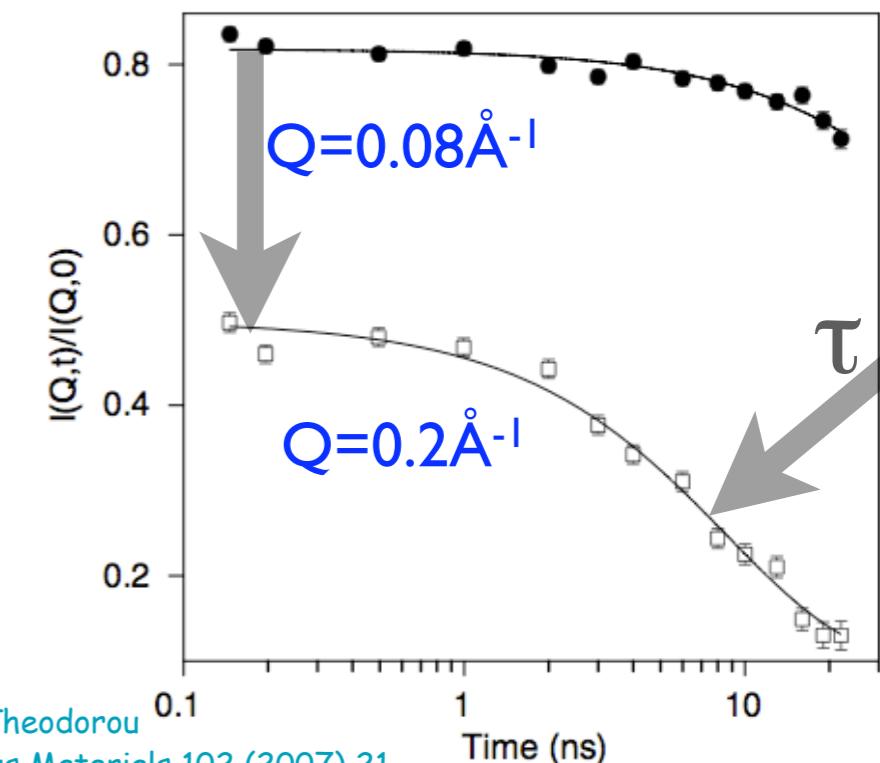
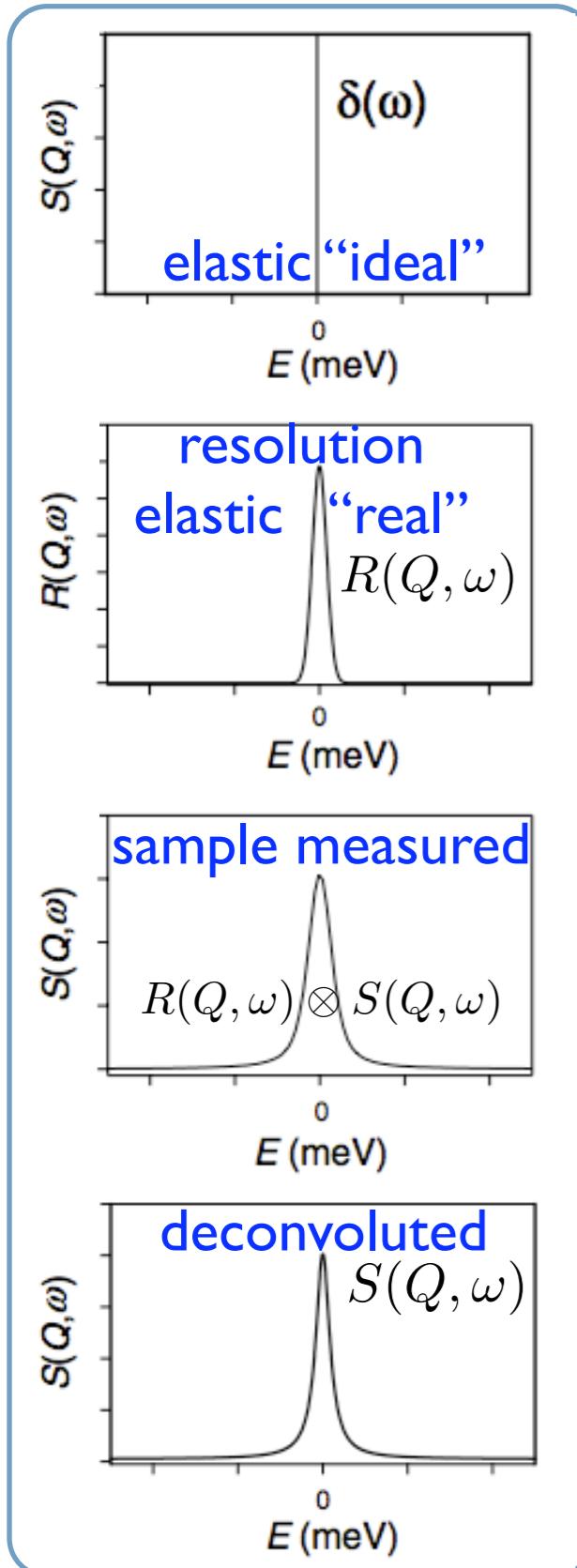


Fig. 7. Normalized intermediate scattering functions obtained for isobutane in silicalite-1 at 550 K, for two different Q values: (●) 0.08 \AA^{-1} , (□) 0.2 \AA^{-1} .

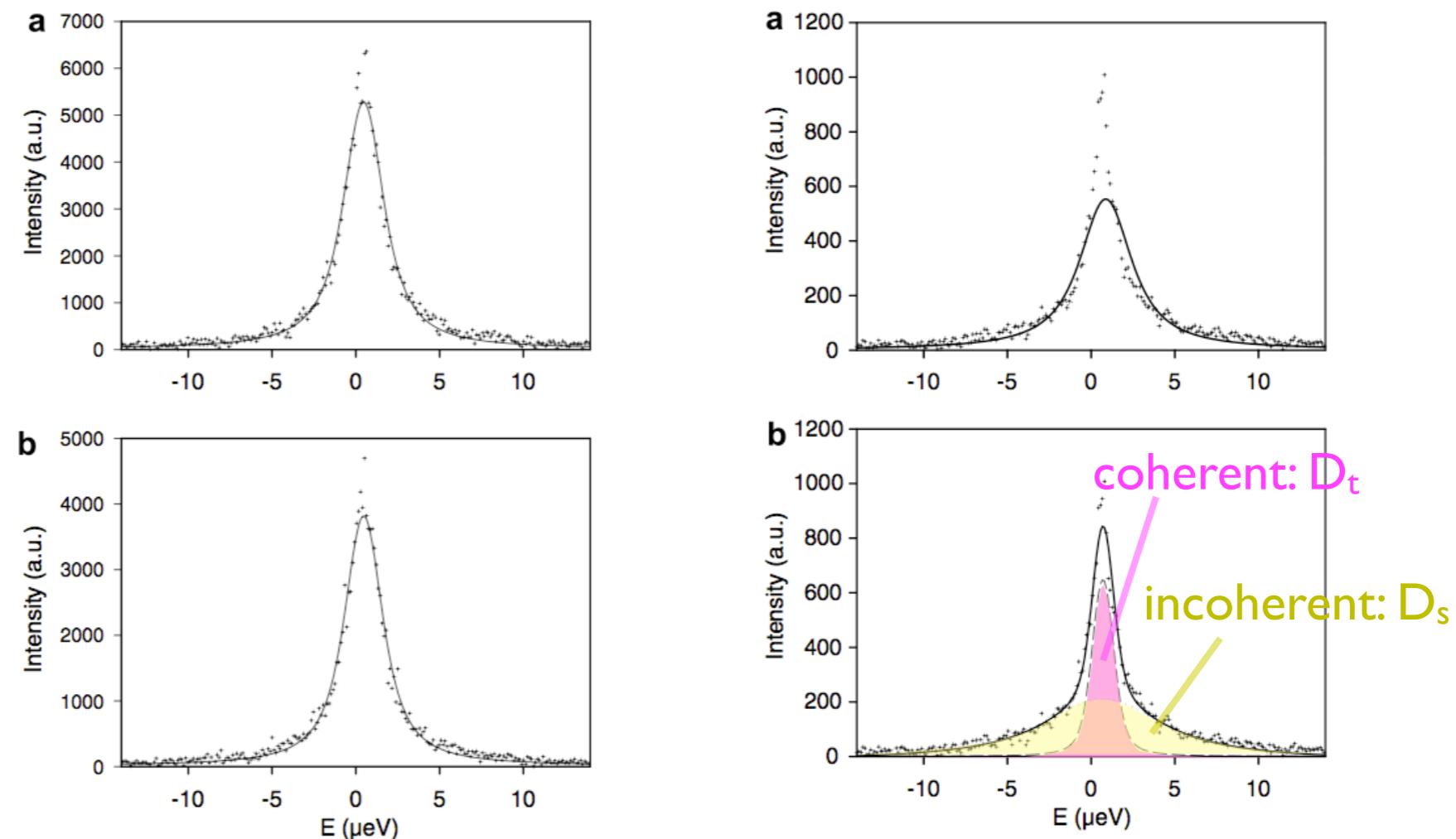
NSE best for coherent scattering
incoherent: 1/3 ‘signal’ on 2/3 background
wide dynamic range: important for line-shape studies
best time resolution
difficult for partially deuterated samples with similar relaxation times

simple incoherent scattering law: translational diffusion - D_s and D_t



example from neutron backscattering at reactors

H. Jobic, D.N. Theodorou / Microporous and Mesoporous Materials 102 (2007) 21–50



best for very low frequency inelastic excitations; good for quasielastic
 best for incoherent scattering, but possible for partially deuterated
 (knowledge of $S_i(Q)$ needed)
 narrow dynamic range, but combined by FT with TOF as wide as NSE
 best energy resolution

diffusion models

deviations from (Fick's law) translational diffusion at larger Q

example from TOF instruments

continuous / translational diffusion

jump diffusion models:

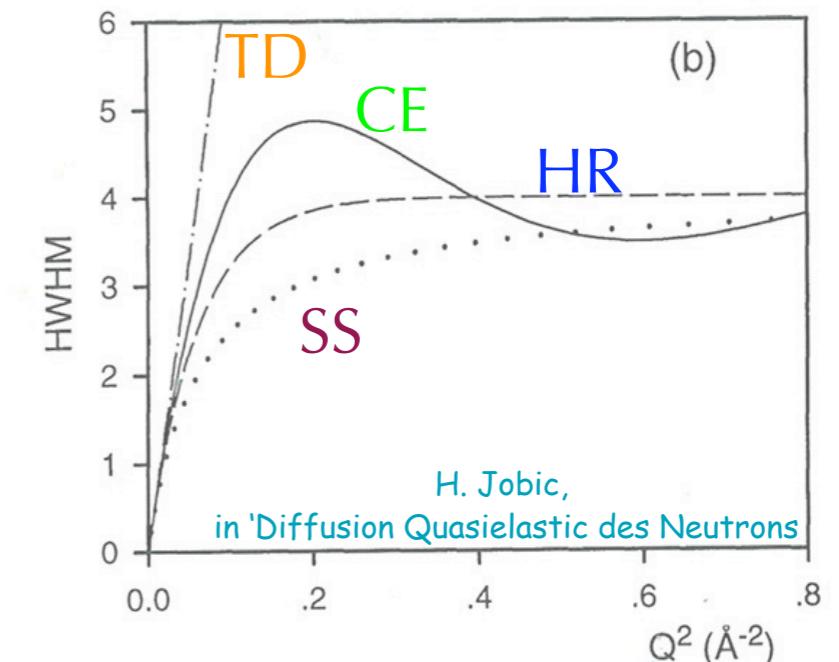
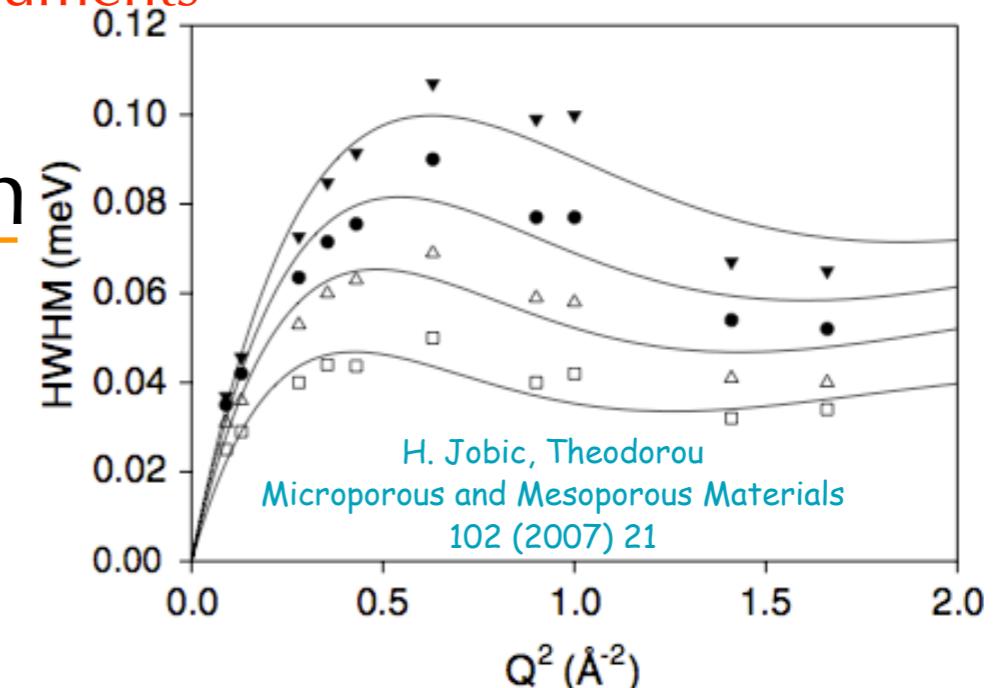
Chudley-Elliott: HWHM is Q-dependent

Singwi-Sjölander

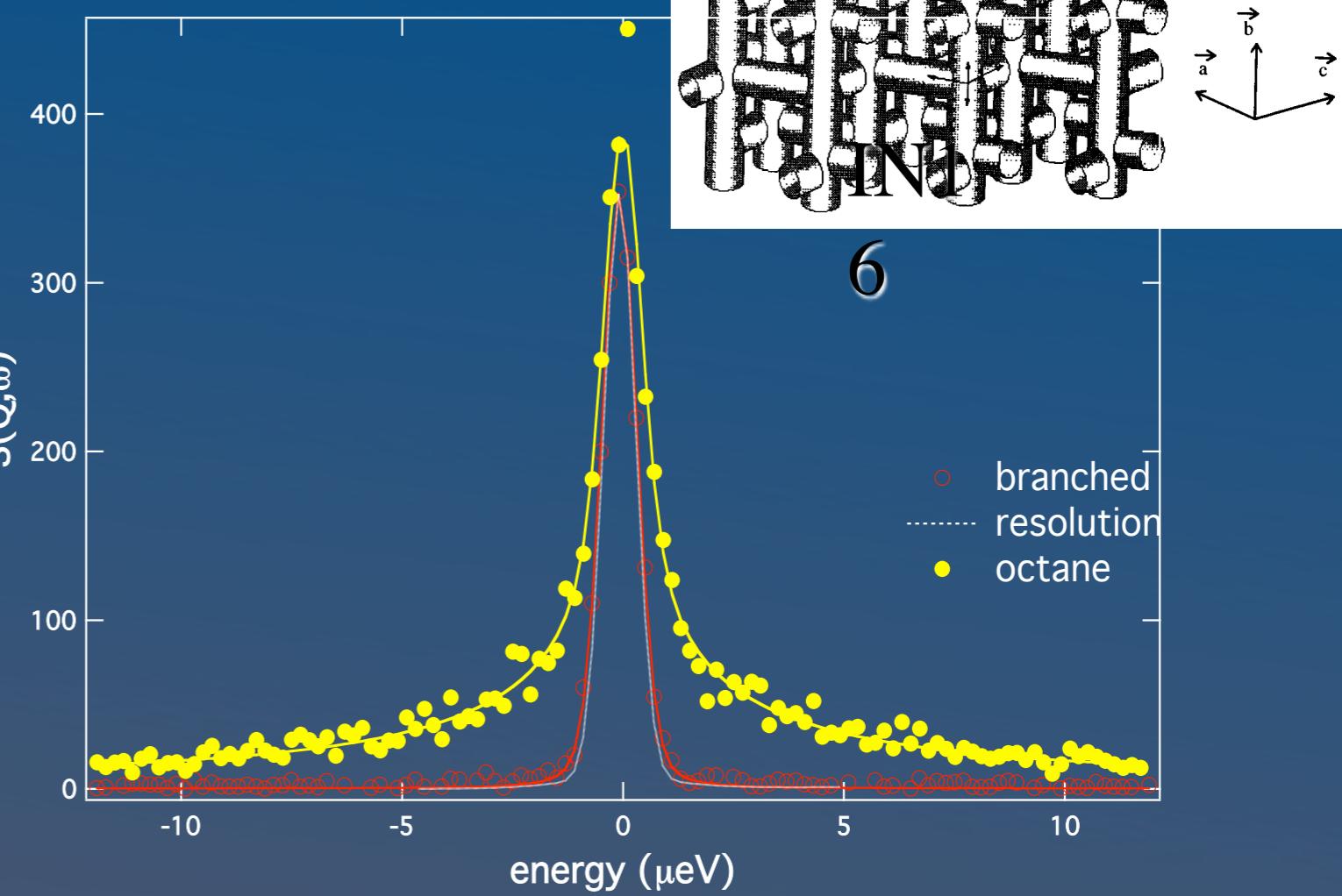
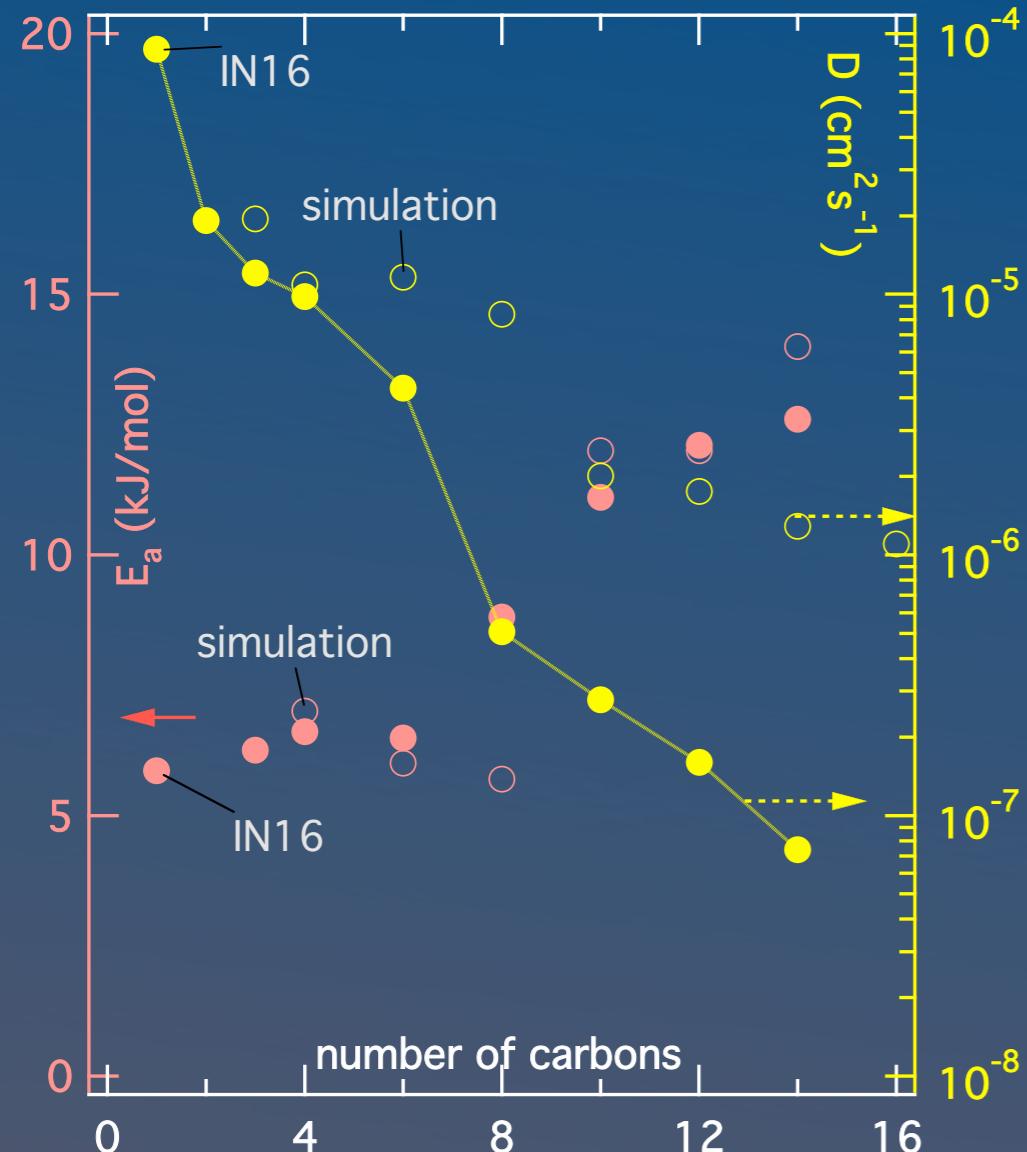
Hall-Ross

anisotropic diffusion models

diffusion in lower dimensions



diffusion of alkanes in zeolites



See e.g. Hervé Jobic, J.Mol.Catalysis A:Chemical 158,(2000),135

- studies as a function of chain length, branching, loading of alkanes
- ⇒ fit of Chuddley-Elliott type jump models; wide Q-range needed

simple incoherent scattering law: - jump models

see e.g.: - M. Bée, 'Quasielastic Neutron Scattering'

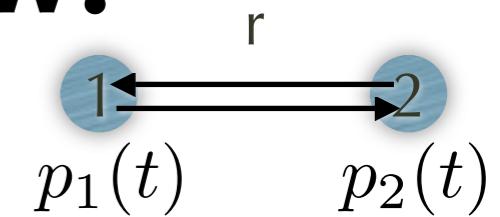
local motions -> Q-independent linewidth & EISF

- jumps between 2,3,4,...n (non-)equivalent sites
 - rotational diffusion on a circle
 - diffusion on a sphere
- diffusion inside a sphere, cylinder,...

simple incoherent scattering law:

- two site jump among equivalent sites

stationary part -> elastic scattering



$$I(\mathbf{Q}, t) = A_0(\mathbf{Q}) + A_1(\mathbf{Q}) \exp\left(-\frac{2t}{\tau}\right)$$

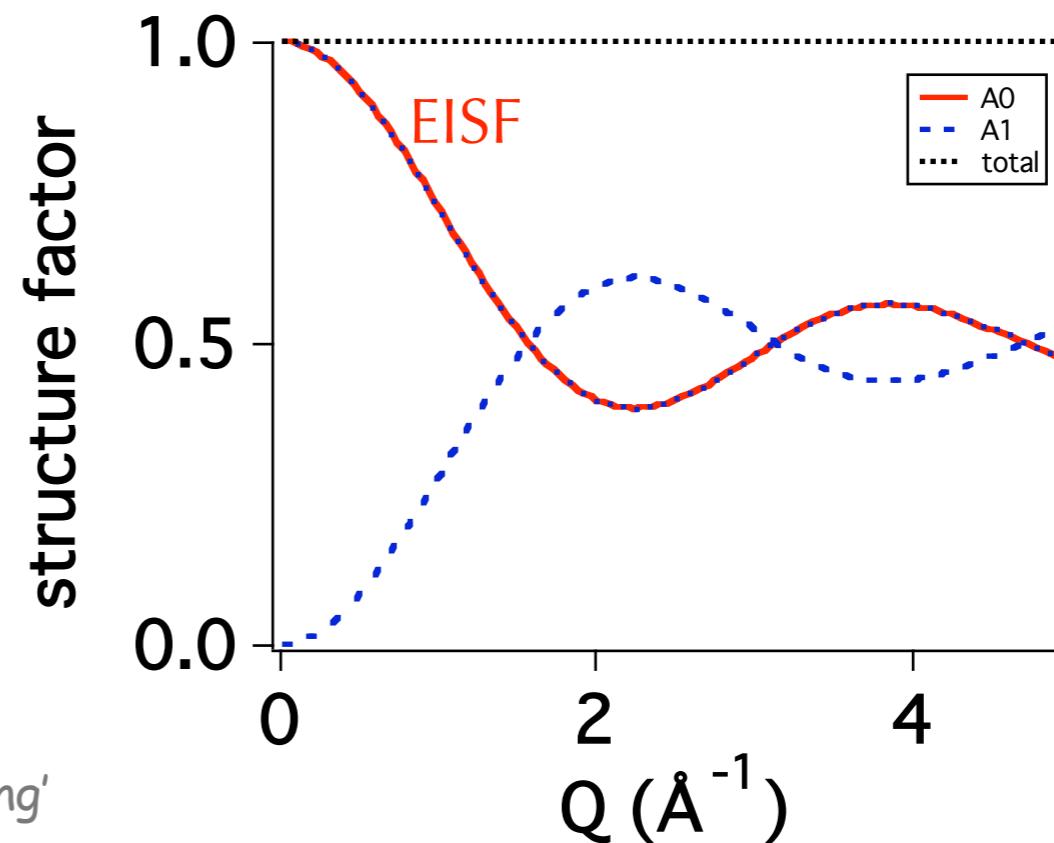
$$S_{inc}(\mathbf{Q}, \omega) = A_0(\mathbf{Q})\delta(\omega) + A_1(\mathbf{Q}) \frac{1}{\pi} \frac{2\tau}{4 + \omega^2\tau^2}$$

structure factors after powder averaging:

$$\text{EISF: } A_0(Q) = [1 + j_0(Qr)]/2$$

$$A_1(Q) = [1 - j_0(Qr)]/2$$

j_0 = 0-th order spherical Besselfct.



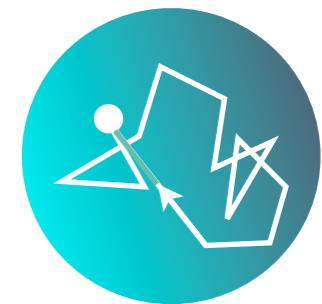
see e.g. M. Bée, 'Quasielastic Neutron Scattering'

simple incoherent scattering law:

- free diffusion inside a sphere

stationary part -> elastic scattering

Volino F., Dianoux A.J. , Mol. Phys. 41, 271-279 (1980)



$$S(q, \omega) = A_0^0(q) \delta(\omega) + \frac{1}{\pi} \sum_{\ell, n} (2\ell + 1) A_n^\ell(q) \frac{(x_n^\ell)^2 D/R^2}{[(x_n^\ell)^2 D/R^2]^2 + \omega^2}$$

$$A_0(Q) = \left(\frac{3j_1(Qa)}{Qa} \right)^2 \text{ and } j_1(x) = \frac{\sin(x)}{x^2} - \frac{\cos(x)}{x} \text{ spherical Besselct. of 1st order}$$

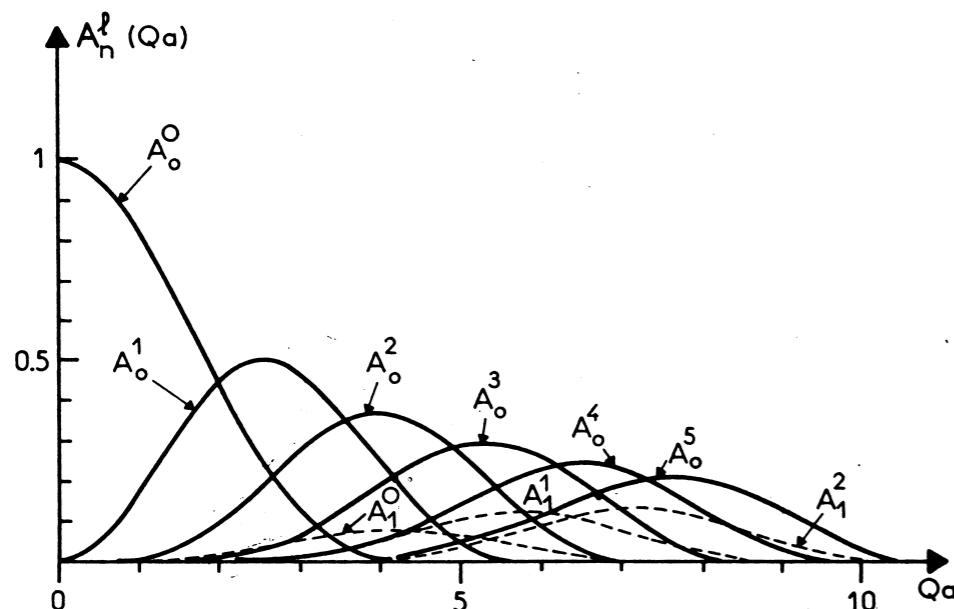


Figure 1. Variation of the first incoherent structure factors $A_n^l(Q)$ versus Qa (31-32) for diffusion inside an impermeable sphere of radius a . The elastic incoherent structure factor (EISF) is $A_0^0(Q)$.

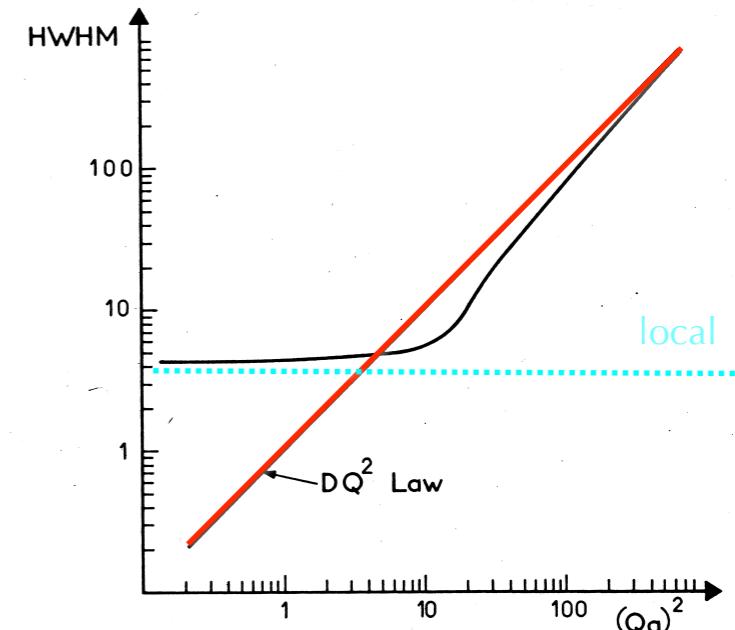
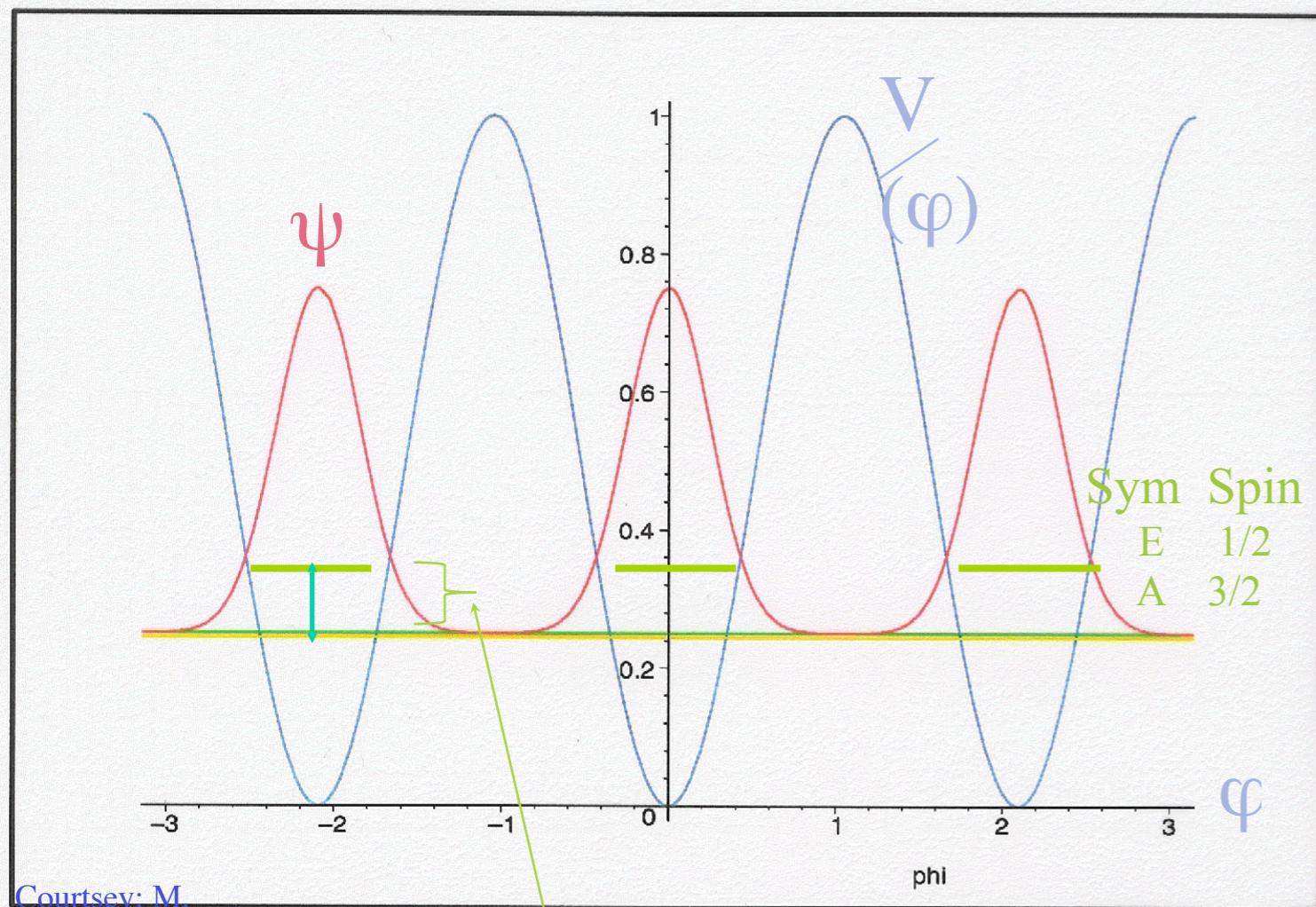


Figure 2. Variation of the half width at half maximum (HWHM) of the broadened component of the scattering law for diffusion inside a sphere of radius a , (33), expressed in D/a^2 energy units, versus $(Qa)^2$. The HWHM of the DQ^2 law is also represented. In the present units, its value is $(Qa)^2$.

Rotational tunneling

Single particle model: environment=potential $V(\varphi)$



Courtesy: M.
Prager

$|123\rangle$ $|231\rangle$ $|312\rangle$

Extreme sensitivity: $\hbar\omega_t \sim e^{-\alpha V(\varphi)}$

Rotational Tunneling and Neutron Spectroscopy: A Compilation

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1d rotor e.g.: CH_3

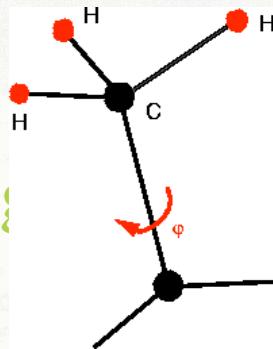
1 tunnel transition; doublet state (A,E)

3d rotors:

CH_4, NH_4

5 sub-states (A,3T,E)

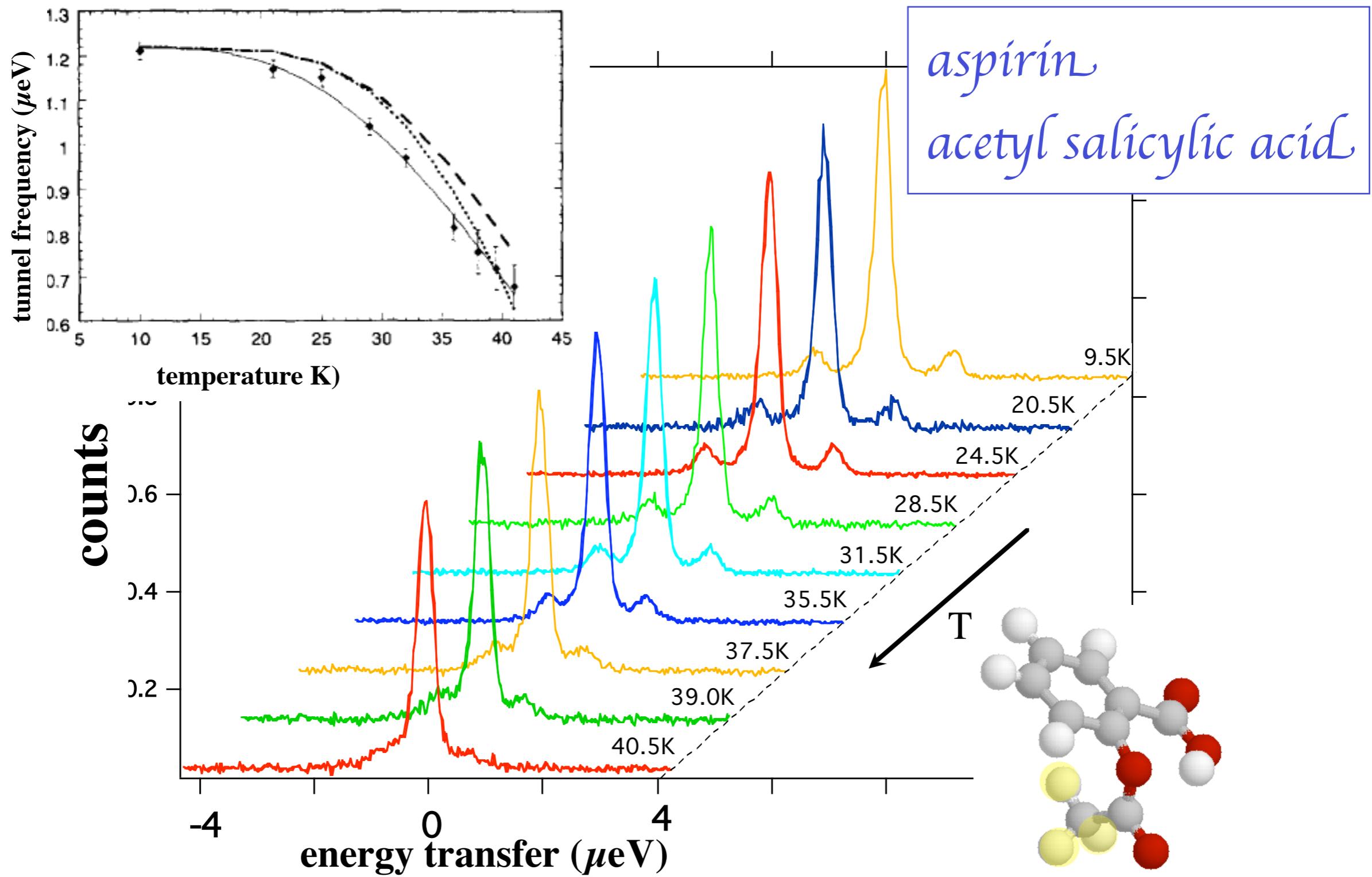
more complex tunneling spectra



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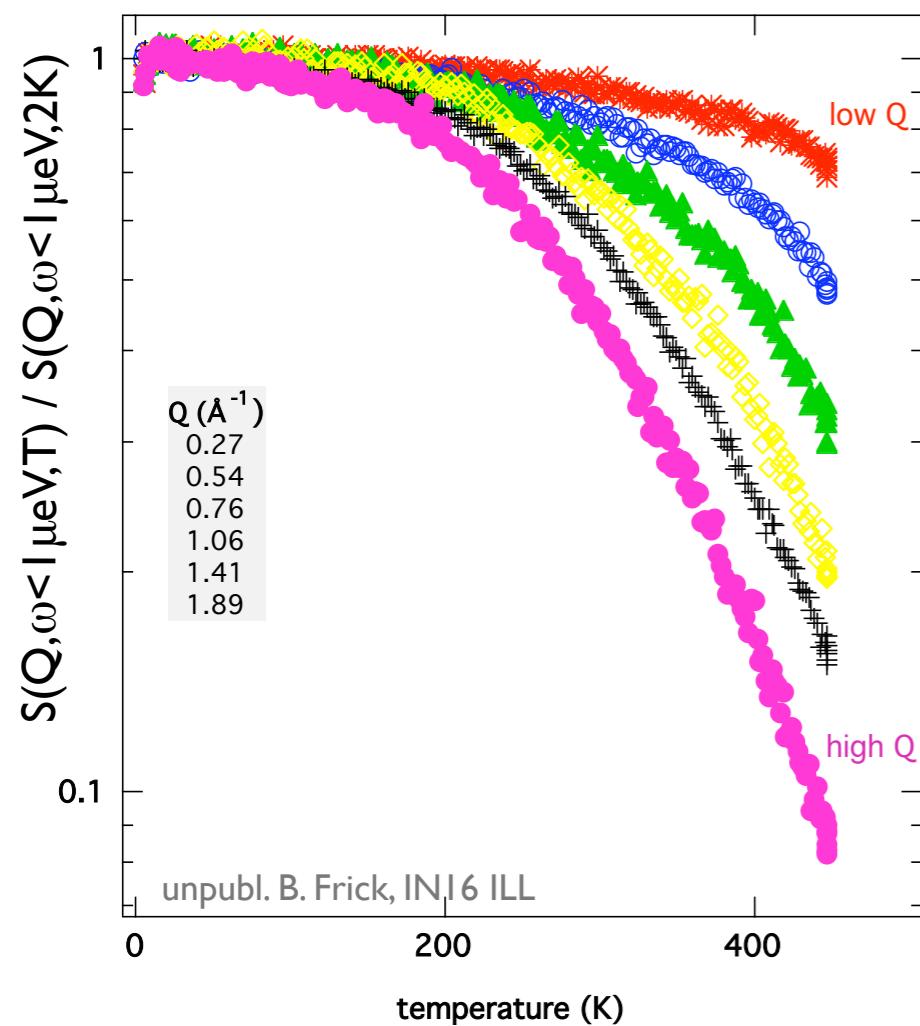
Reprinted from
Volume 97, Number 8, Pages 2933–2966

methyl group tunneling

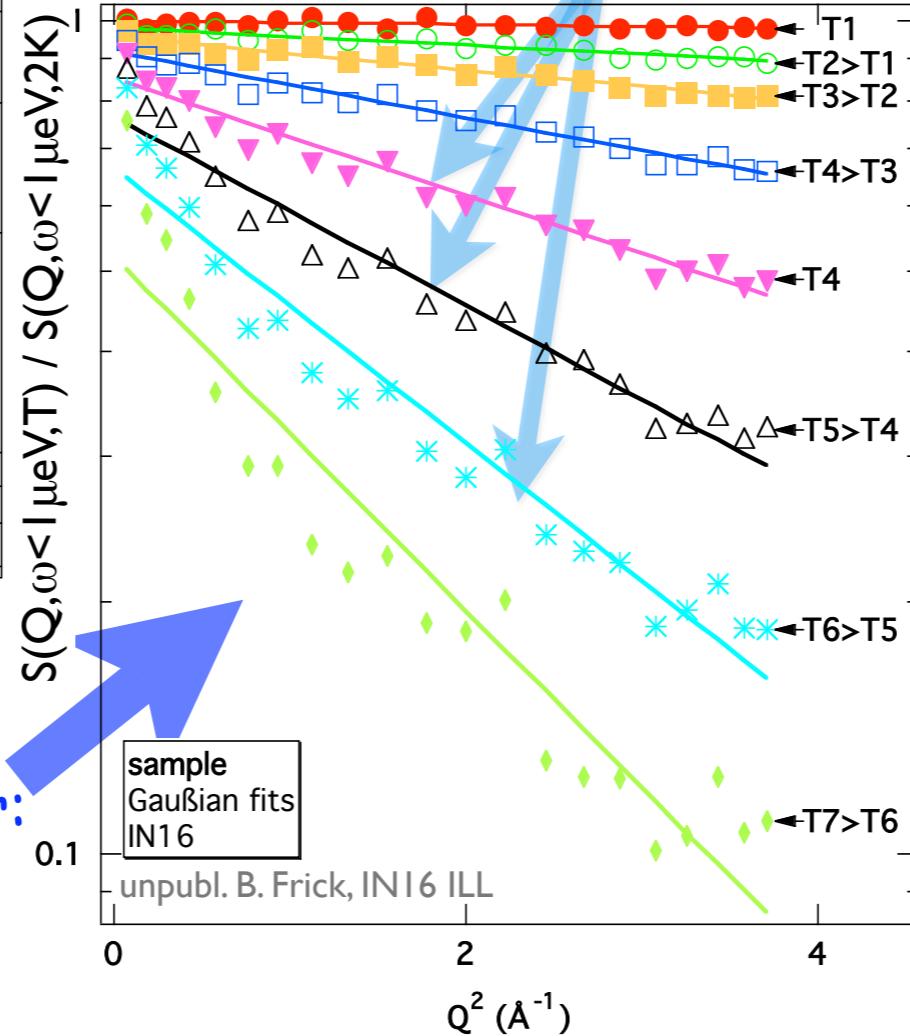


elastic scans

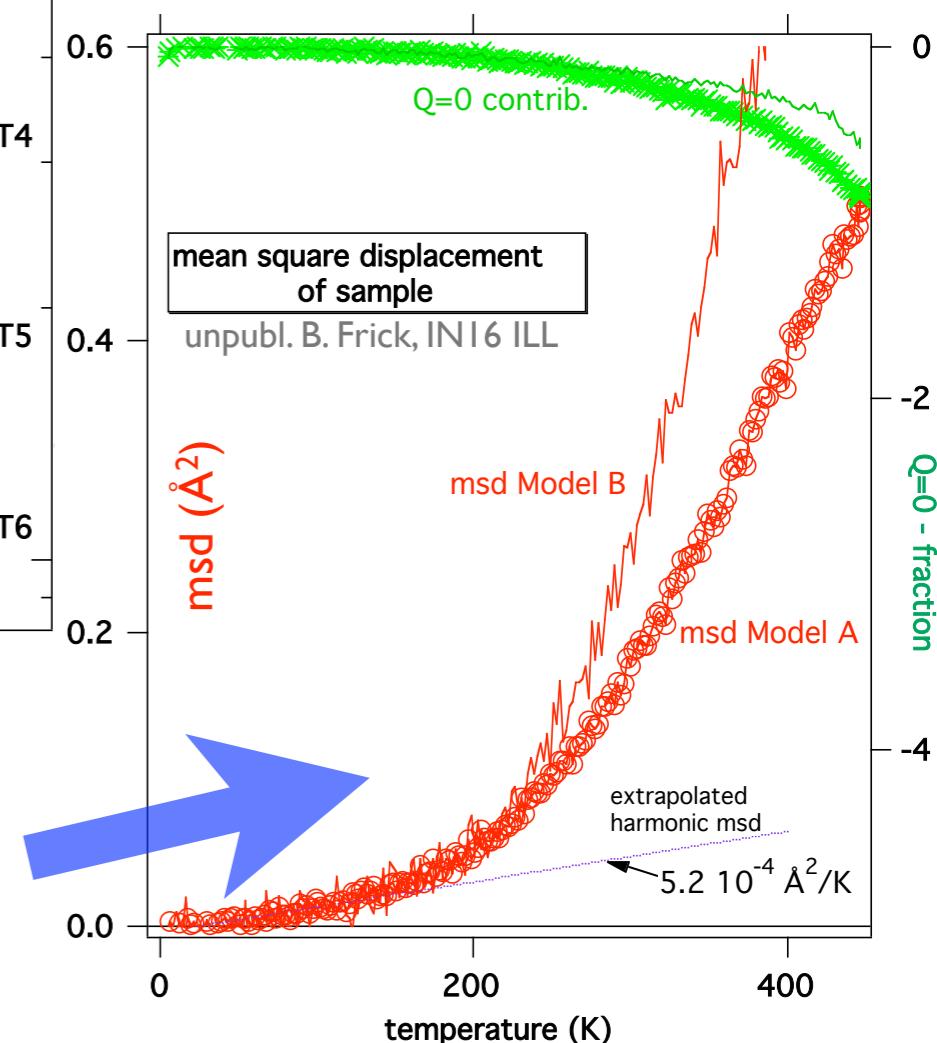
- and “Gaussian” behaviour



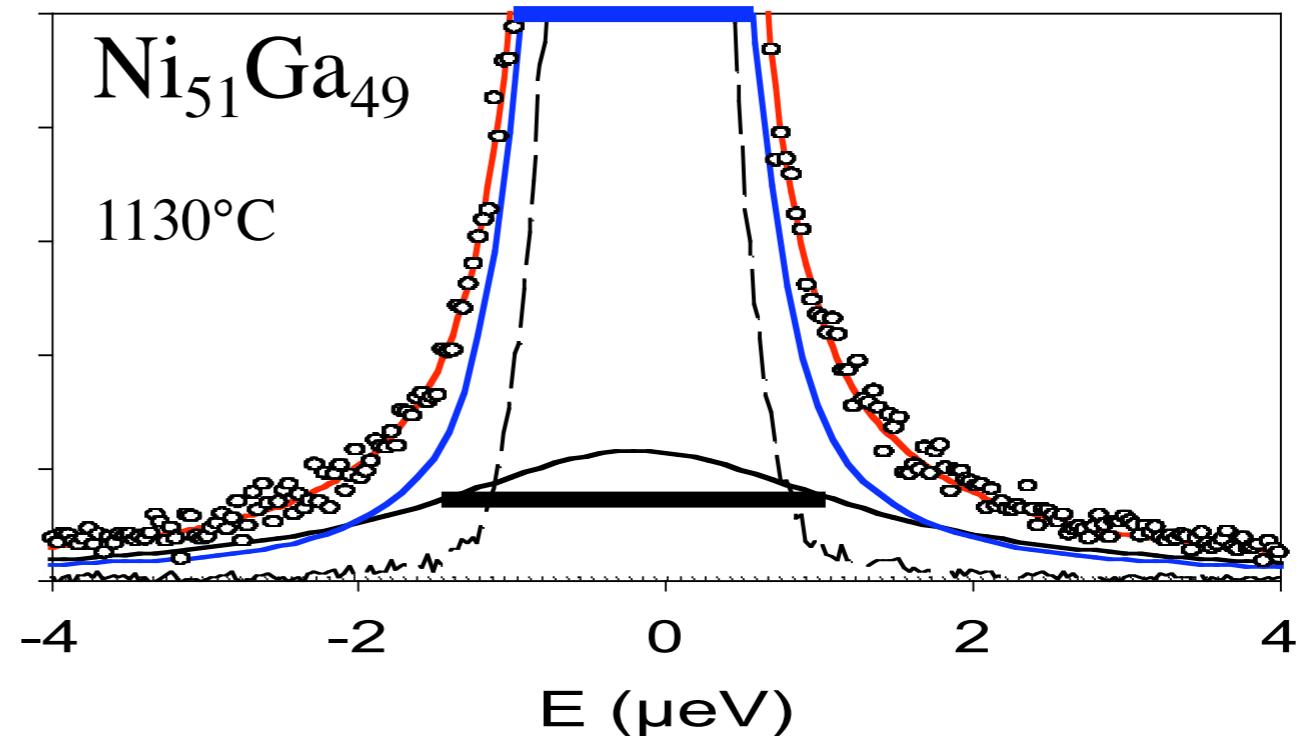
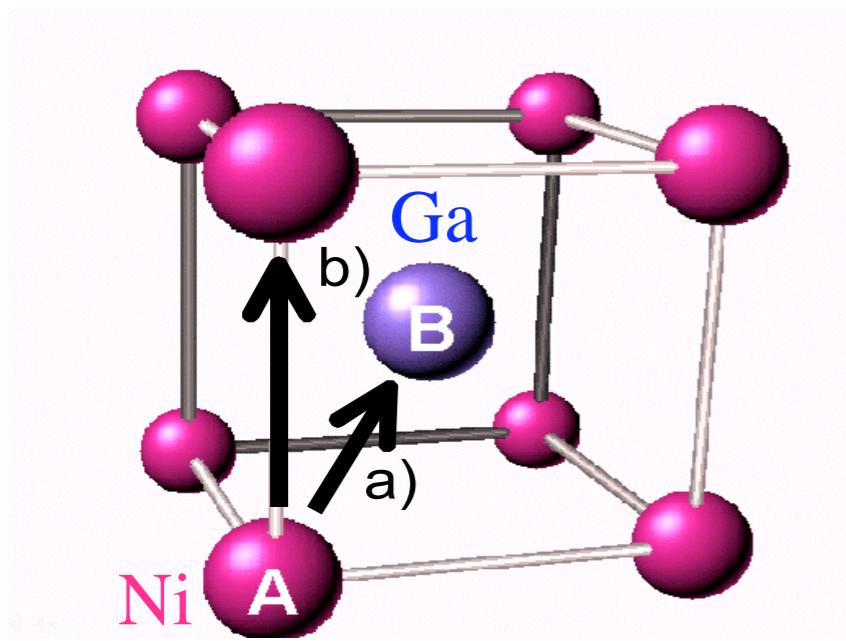
check for Gaussian behaviour:
linear in $\ln(I/I_0)$ vs Q^2



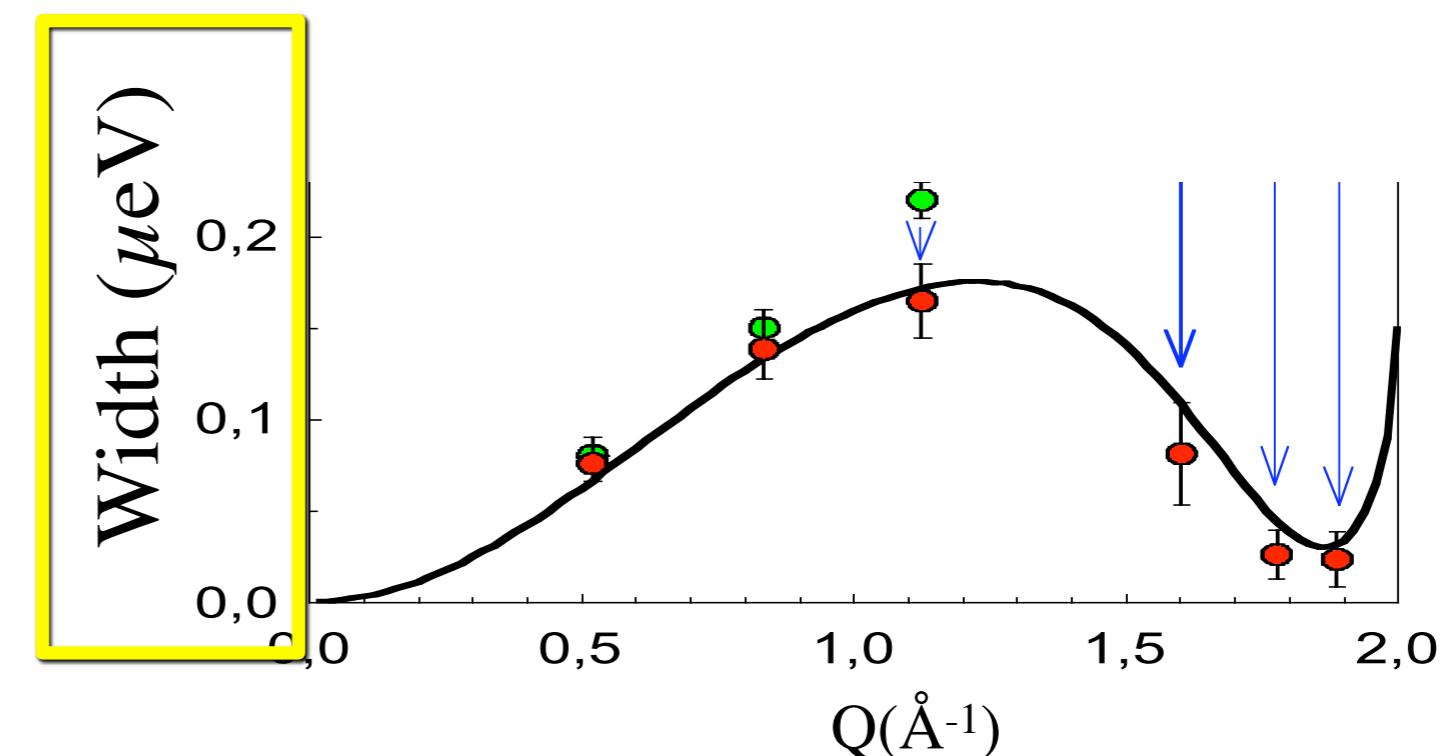
calculate mean squared
displacement:



jump diffusion in single crystals



Using single crystals
diffusion constants
and jump vector can
be determined.



Kaisermayr et al.; ILL Annual report 2000
Kaisermayr et al., PRB, 63 (2001)

IRIS-example

Nature of the Bound States of Molecular Hydrogen in Carbon Nanohorns

F. Fernandez-Alonso,^{1,*} F. J. Bermejo,^{2,†} C. Cabrillo,²

PG(002) $\delta E = 8.8 \mu\text{eV}$

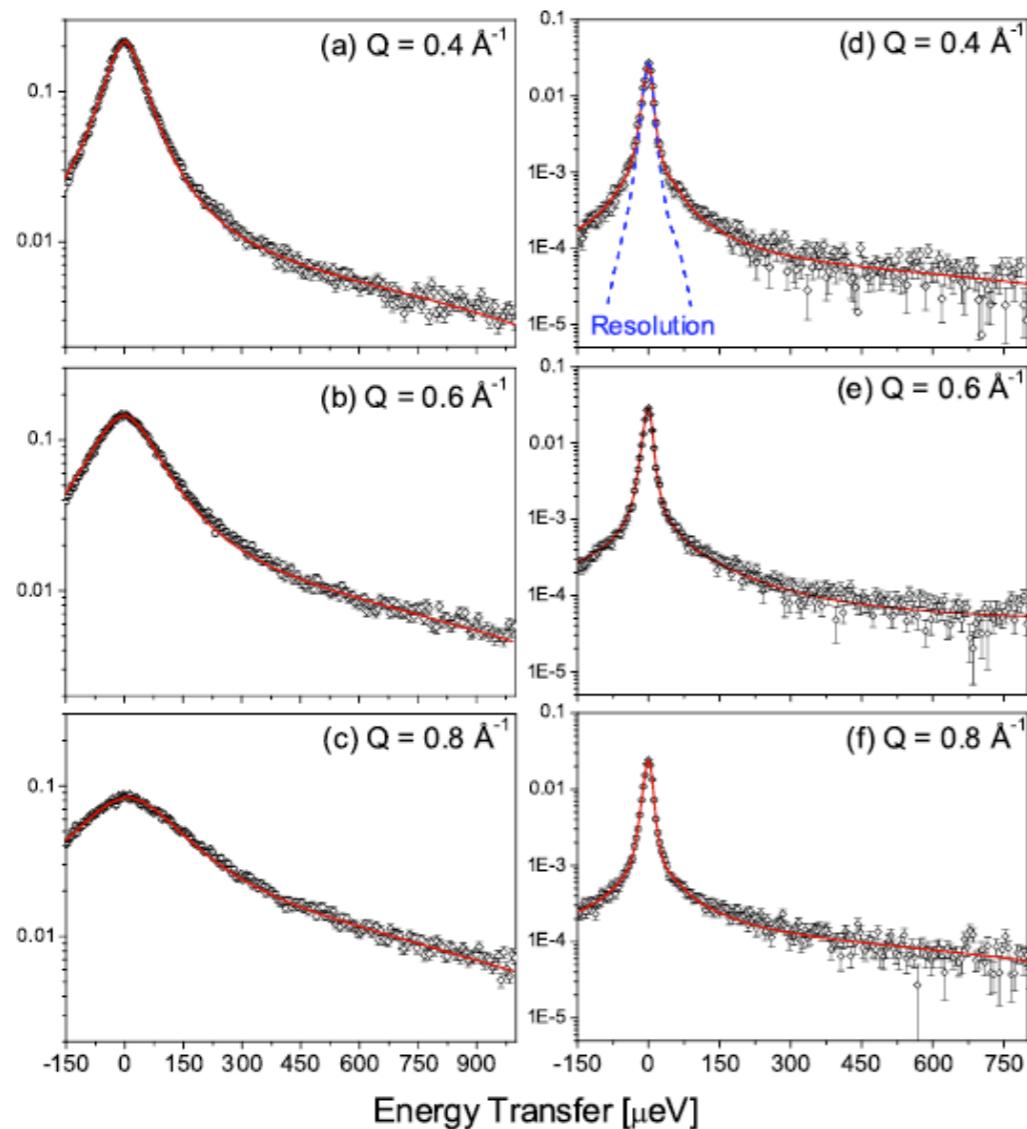
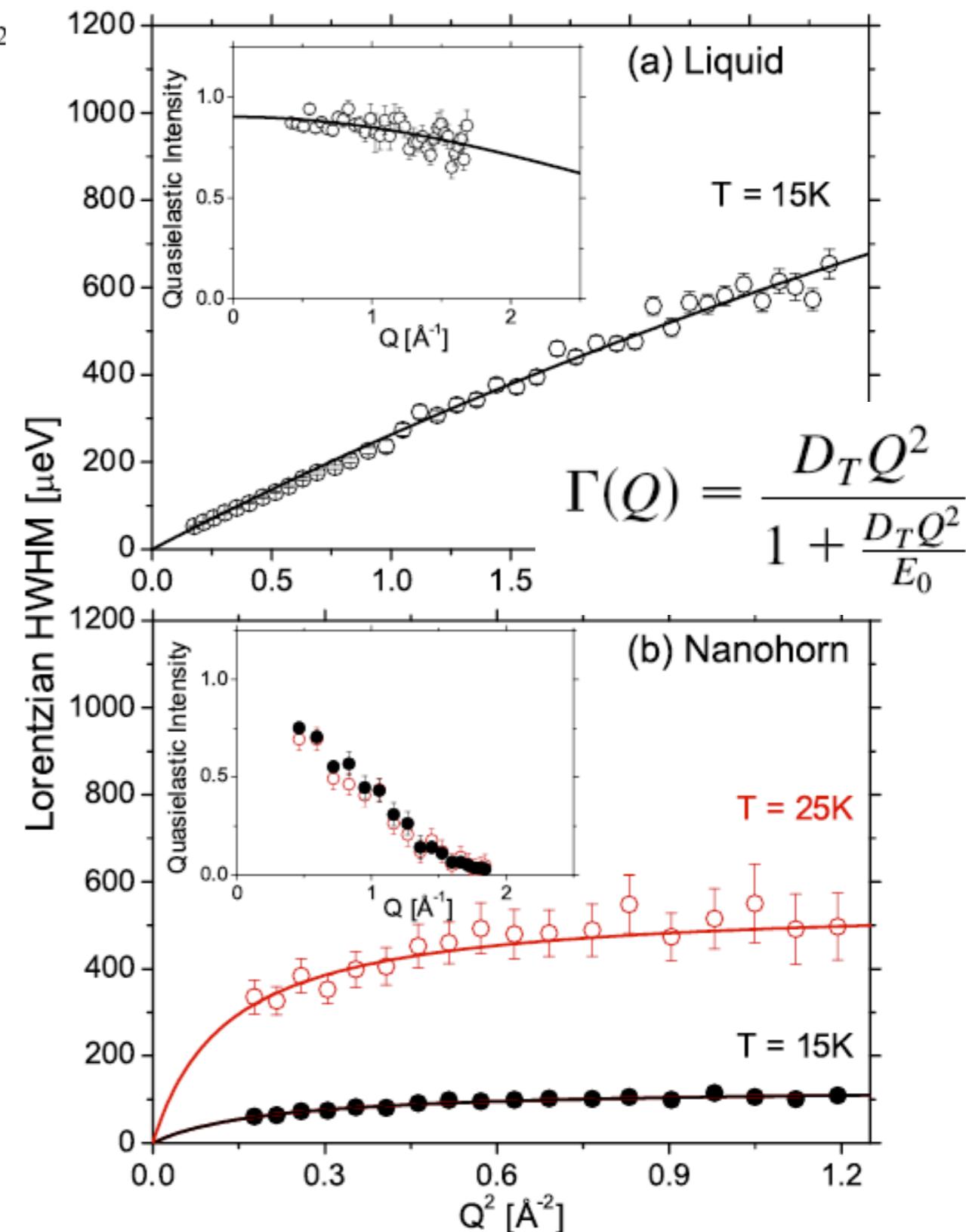
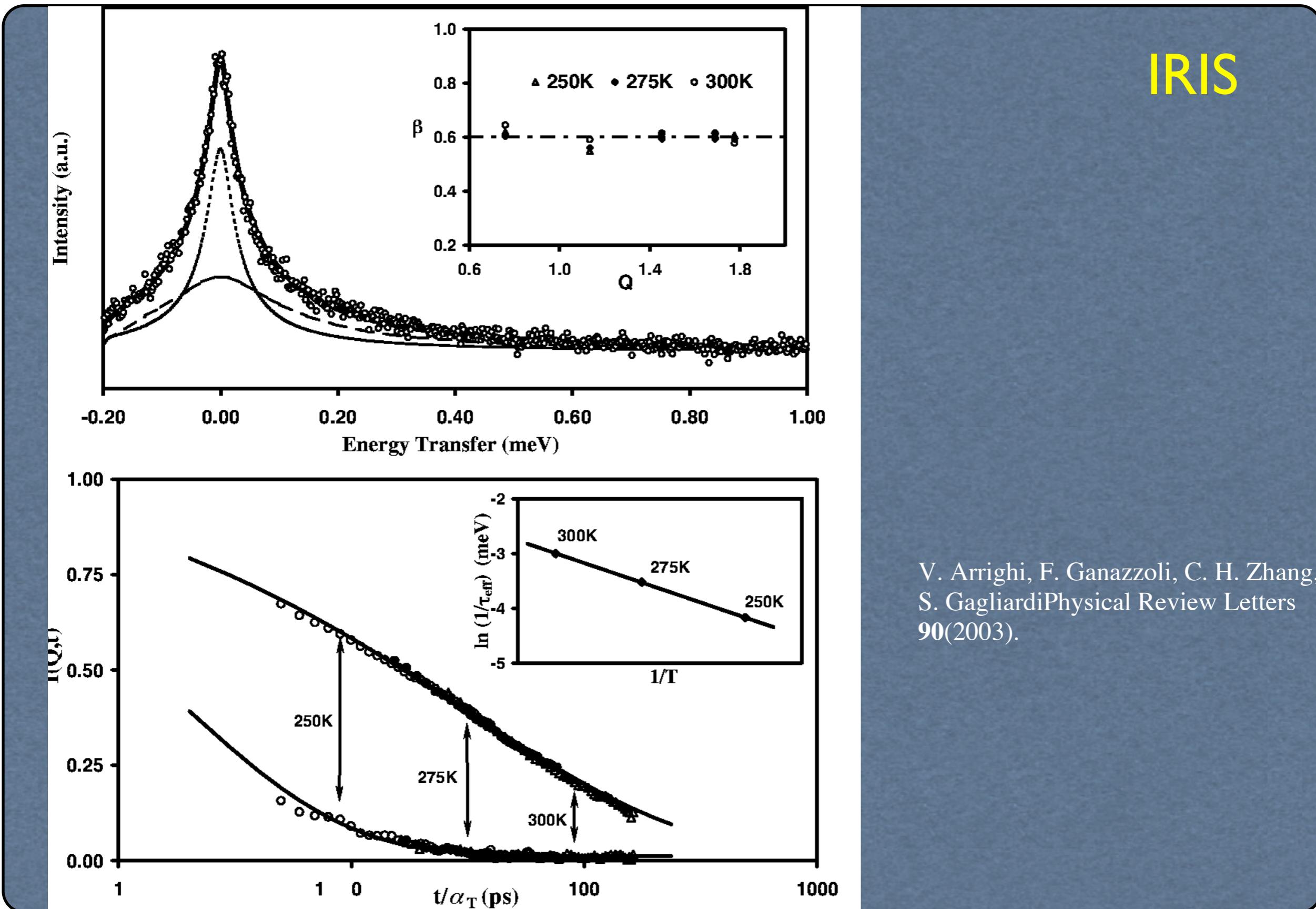


FIG. 1 (color online). QENS spectra at various wave vectors Q for the bulk liquid (a)–(c) and H_2 -NH samples (d)–(f) at $T = 15 \text{ K}$. The solid lines are model fits to the data. The narrow line in (d) represents the instrumental energy resolution.



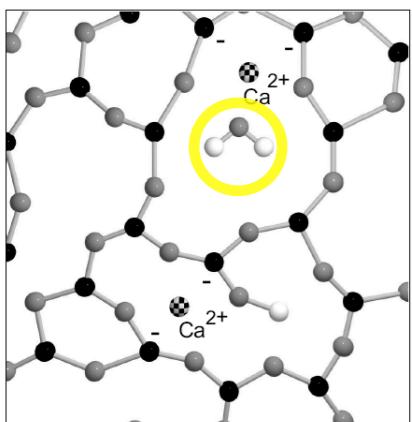
more complex relaxation patterns: e.g. polymers



combination of TOF and BS

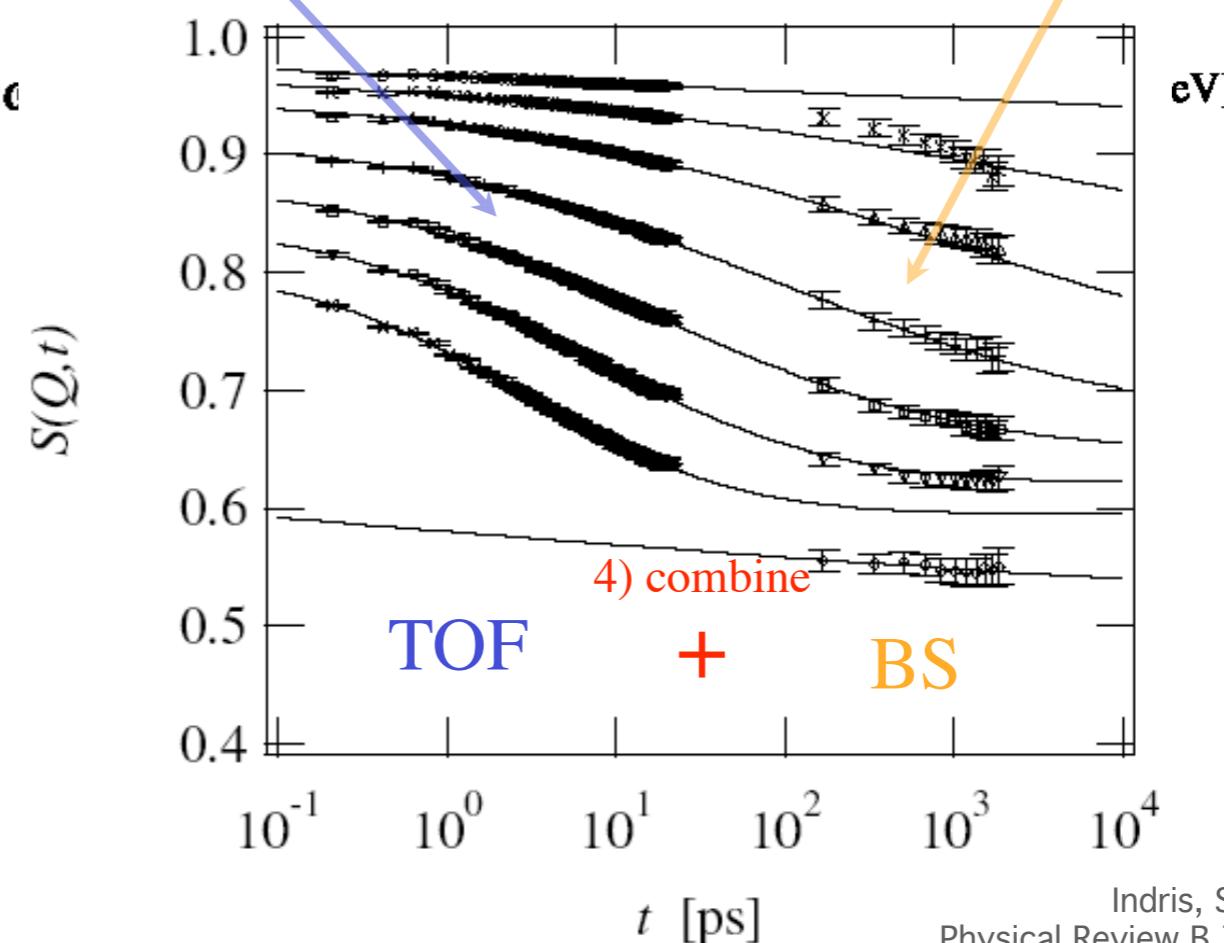
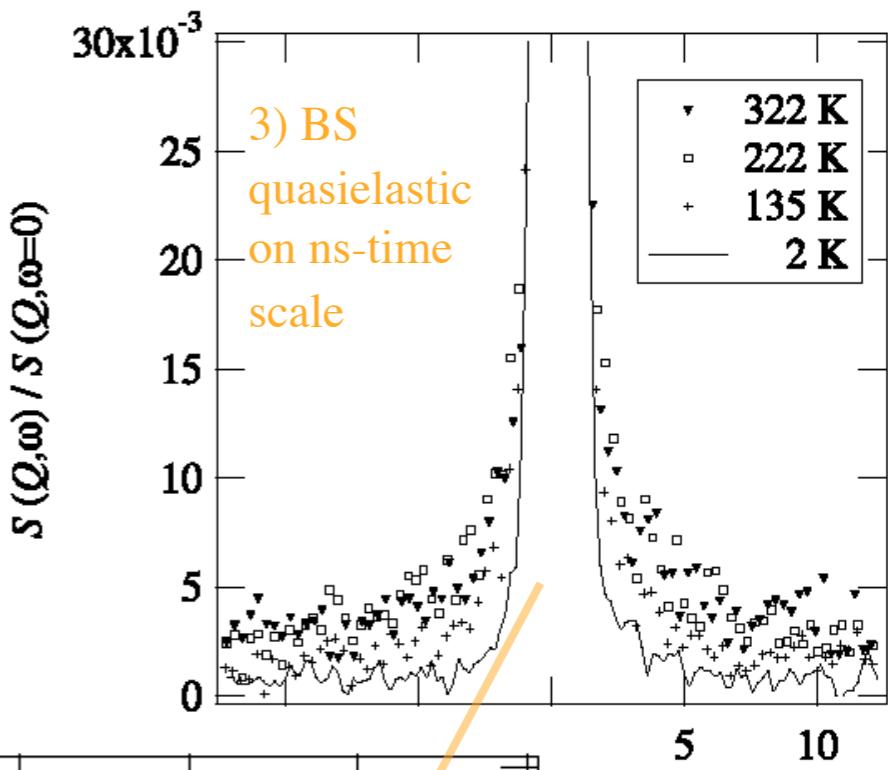
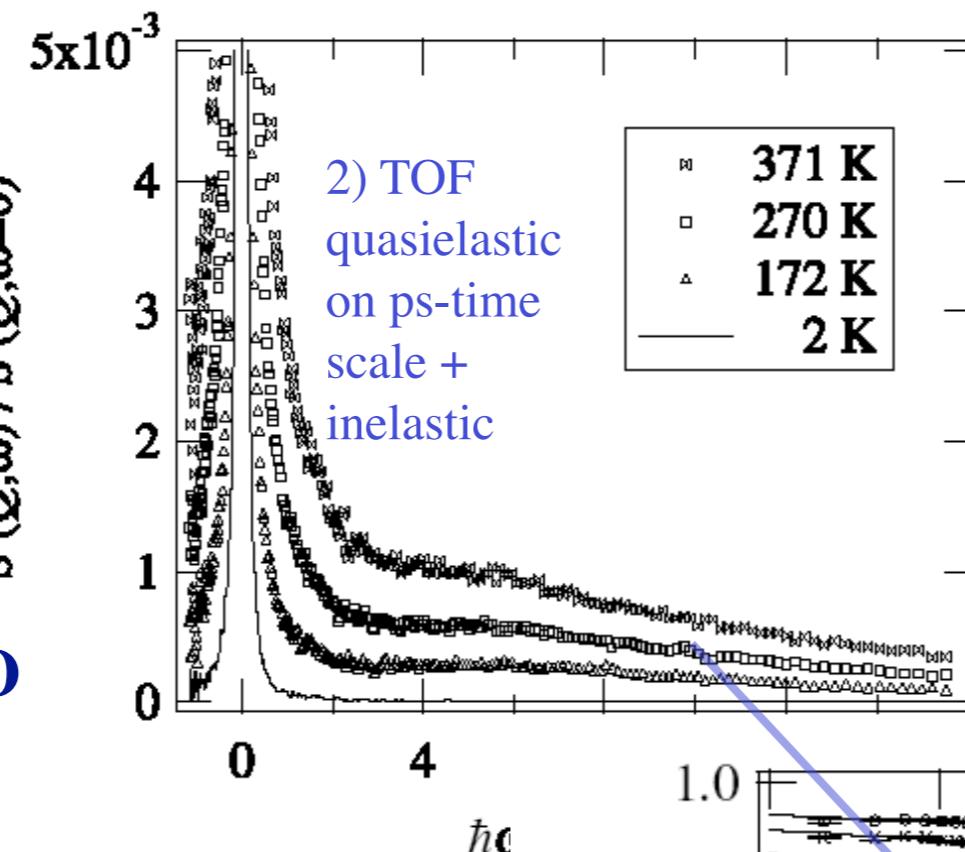
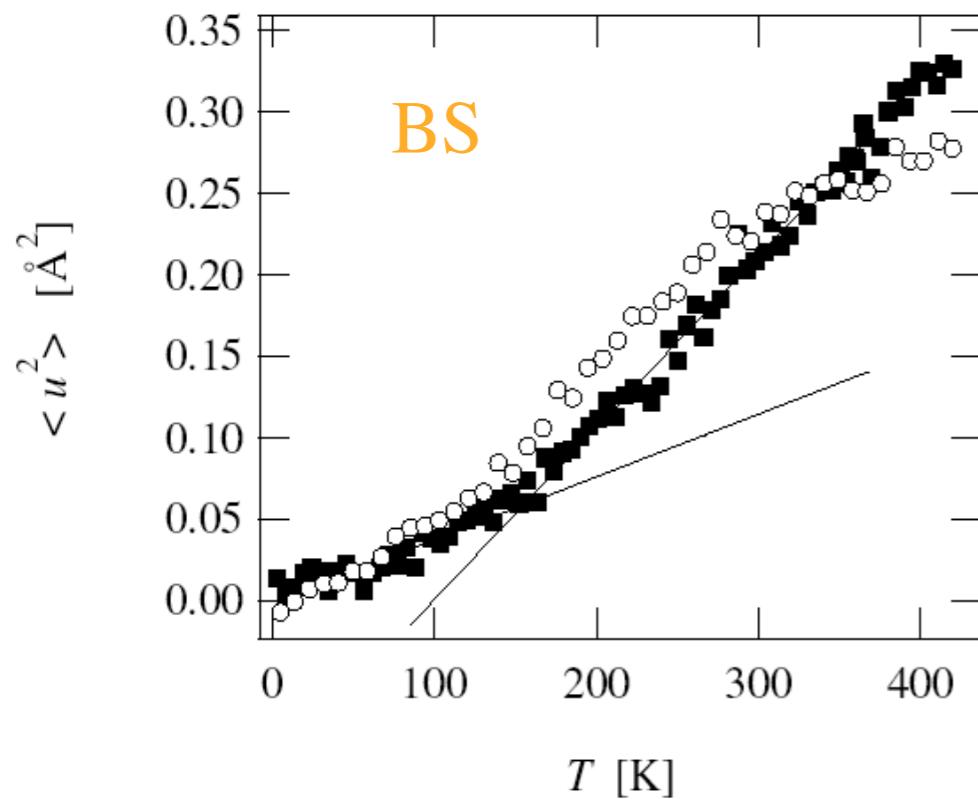
IN6 / INI6

- an example from geoscience



**H₂O dynamics in
Ca_{0.5}AlSi₃O₈+8%H₂O**

1) overview from elas



Indris, S. et al.,
Physical Review B 71, 64205 (2005).

thank you for your attention

some literature:

books:

T. Springer, 'Quasielastic Neutron Scattering for the Investigation of Diffusive Motions in Solids and Liquids',
Springer Tracts in Modern Physics 64, 197

M. Bée, 'Quasielastic Neutron Scattering Principles and Applications in Solid State Chemistry, Biology and Material Science',
Adam Hilger, Bristol 1988

recent reviews:

techniques: NSE: F. Mezei, C. Pappas, T. Gutberlet , 'Neutron Spin Echo Spectroscopy: Basics, Trends and Applications'
Springer 2003 ; BS: A. Heidemann, B.F., <http://www.ill.fr/YellowBook/IN16/BS-review/index.htm>

diffusion in zeolites: H. Jobic, Theodorou, Microporous and Mesoporous Materials 102 (2007) 212

confinement studies: e.g. 'Int. Workshop on Dynamics in Confinement', Eur. Phys. J. - ST 141 (2007)

D. Richter, M. Monkenbusch, A. Arbe, J. Colmenero, Neutron Spin Echo in Polymer Systems, Advances in Polymer Science 174