XRD and Raman spectroscopy

F8900 Študentský seminár

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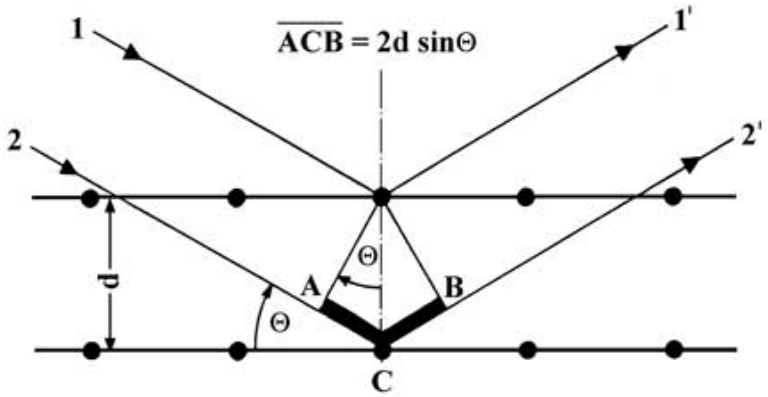
Outline

What is XRD?

- X-Ray Diffraction = X-rays diffracted on lattice of the matter
- constructive interference (Braggs condition):



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

$$2\frac{a}{\sqrt{h^2+k^2+l^2}} \sin\theta = \lambda$$

When do I use XRD?

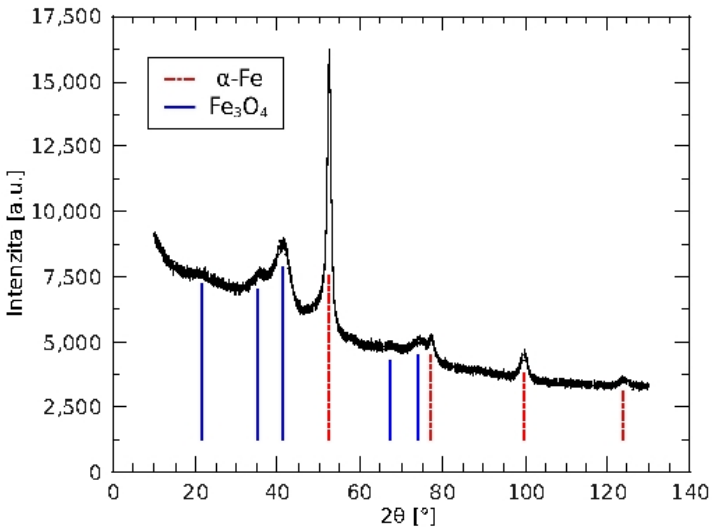
- qualitative and quantitative analysis of crystalline sample
- distinguish between mixture and compound
- size of particles (powder XRD, size $<$ 100 nm)

How mathematics works...

$$F(\vec{Q}) = \sum_s f_s(Q) \overbrace{\exp(-i\vec{Q}\cdot\vec{r}_s)}^{\text{structure factor}} \quad (1)$$

$\vec{r}_s$ direct lattice vector, $\vec{Q}$ reciprocal lattice vector $\Rightarrow$ condition for Miller indeces h, k, l $\Rightarrow$ angle of incidence θ

α-Fe		Fe_3O_4	
hkl	$2\theta[^\circ]$	hkl	$2\theta[^\circ]$
110	52	111	21
200	77	220	35
211	100	311	41
220	124	400	50
310	161	511	67
		440	74



LP#14a - α-Fe/Fe_3O_4: 44:56; 10 nm, 4 nm

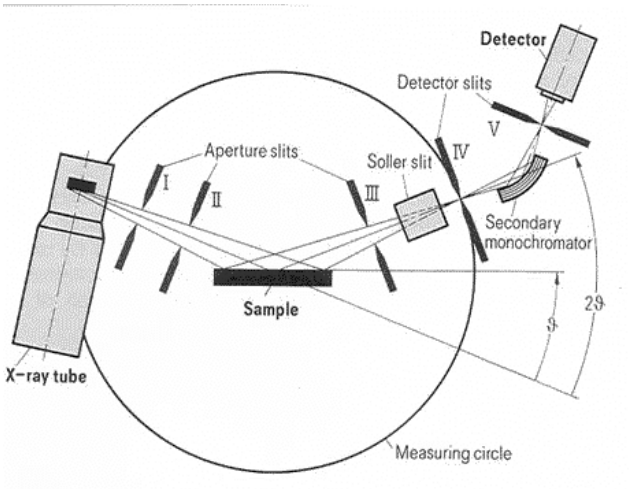
Scherer formula:

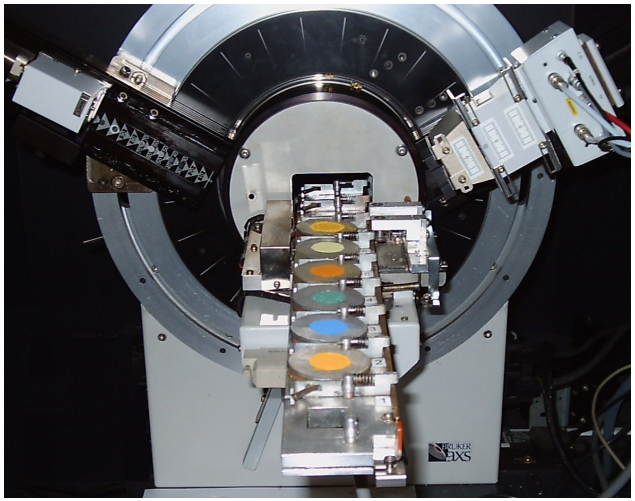
$$\tau = \frac{K\lambda}{\beta \cos\theta}$$

β = FWHM

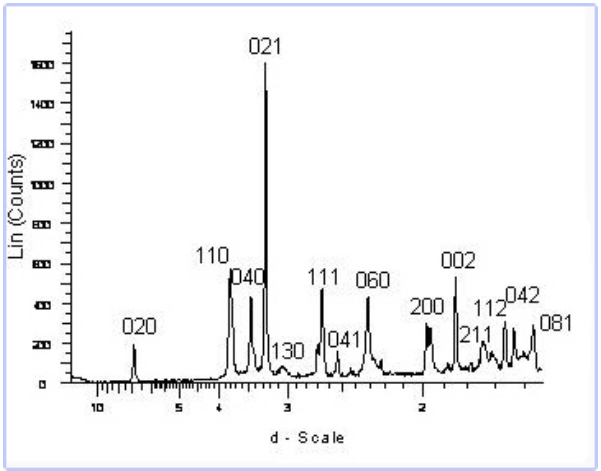
K - shape factor

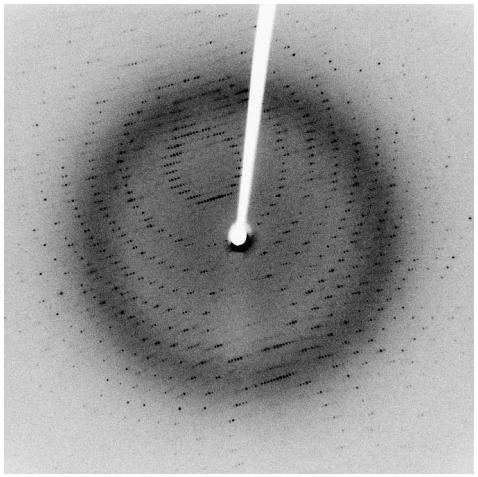
...and physics





- source of X-rays: synchrotron, *bremsstrahlung* (Mo, Cu, Co, Fe, Cr)
- $\lambda \approx 0.06\,\mathrm{nm}\,(\mathrm{Mo}) - 0.23\,\mathrm{nm}\,(\mathrm{Cr})$
- detector: CCD, photopaper



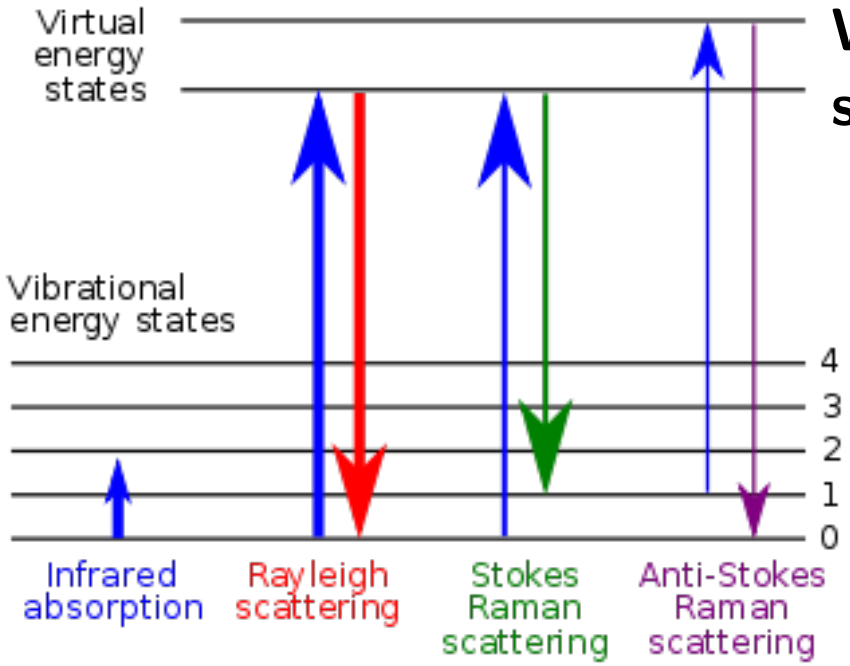


MoO_3 nanowires, `www.nanotul.com`

Raman spectroscopy - Basic theory

- inelastic scattering of monochromatic light
- excitations to virtual energy states
- source: laser (λ_0) - near UV – near IR

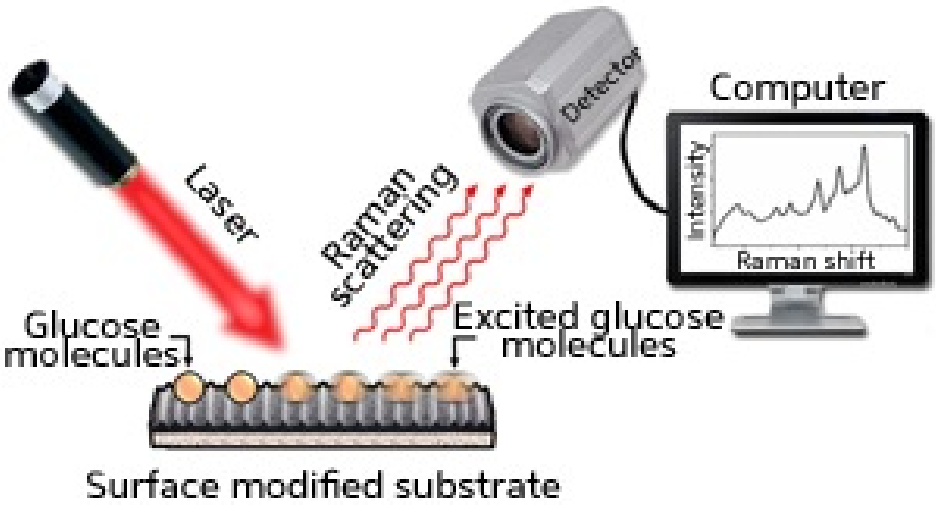
$$\Delta\omega = (\frac{1}{\lambda_0} - \frac{1}{\lambda}) \quad (2)$$



When do I need Raman spectroscopy?

- info about spectra of molecules, chemical bonds
- solids, liquids, samples in water, glass
- NON-destructive, quick, no preparations needed

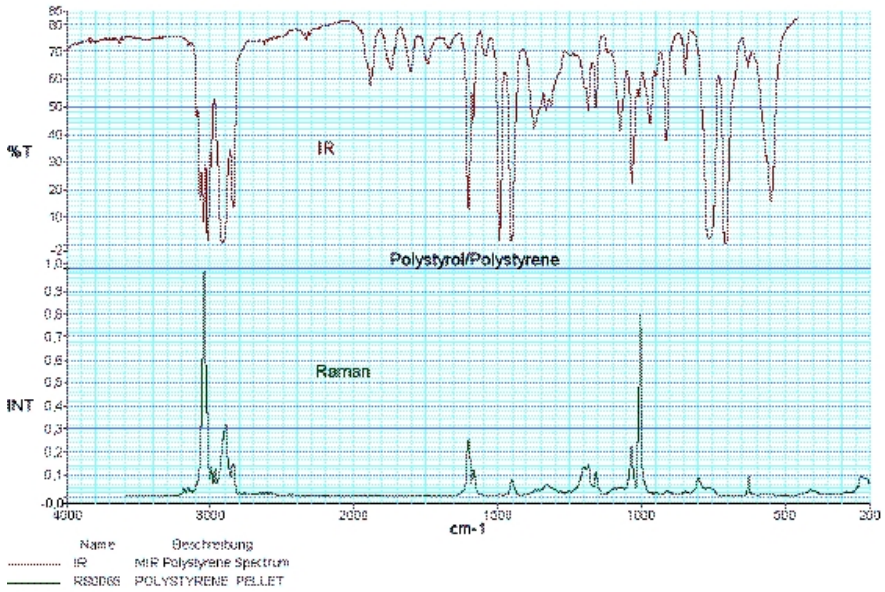
Principle



- light irradiates the sample
- scattered light is detected
- wavelengths $\lambda \approx \lambda_0$ are filtered

Applications

- drugs, explosives detection
- low energy phonons
- complement to IR spectroscopy



To sum up

We use...

XRD if...

- the sample is (poly)crystalline!
- qualitative, quantitative analysis
- nanopowder - size of particles

Raman spectroscopy if...

- info about rotational, vibrational spectra, chemical bonds, phonon and magnon excitations
- complement to IR spectroscopy

Method is useless if...

- the sample is NOT crystalline
- very small objects ($\approx \lambda$)
- similar lattice parameters
- laser changes structure of the sample, heats it
- the sample is metal or alloy

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Thank you for your attention!