CH314 – Structural Analysis
Part III : X-ray Tools

Exercise 3, Dec 7

1) Lets pick up the problem from last week again. The data below shows diffraction patterns from a solution of colloidal nanoparticles taken with an x-ray tube using the Cu 1Kα line with a wavelength of 1.54 Angstrom

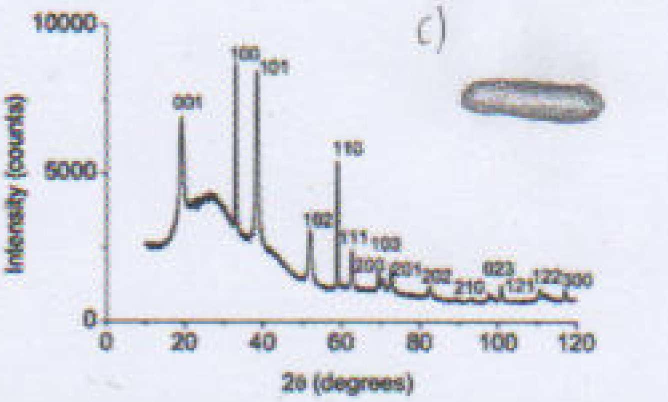


Fig. 1. X-ray diffraction pattern from $Ni(OH)_2$ dispersions ($a = 3.1273(2)$ Å, $c = 4.610(3)$ Å) with diffraction peaks labeled with *hkl* indices. The 001 peak is

Table 1
Results from the pseudo-Voigt function fit to the $Ni(OH)_2$ sample. 2θ is the peak position for the Cu $K\alpha_1$ radiation wavelength (0.154056 nm).

hkl	Bragg angle 2θ (°)	Peak width ${}^{T}\beta$ (°)	Instrument resolution (°)	Corrected β (°)	Diameter (nm)
001	19.25	1.140	0.121	1.02	[redacted]
100	33.06	0.197	0.115	0.08	
101	38.52	0.593	0.114	0.48	
102	52.06	0.876	0.117	0.76	
110	59.05	0.248	0.120	0.13	
111	62.67	0.550	0.122	0.43	

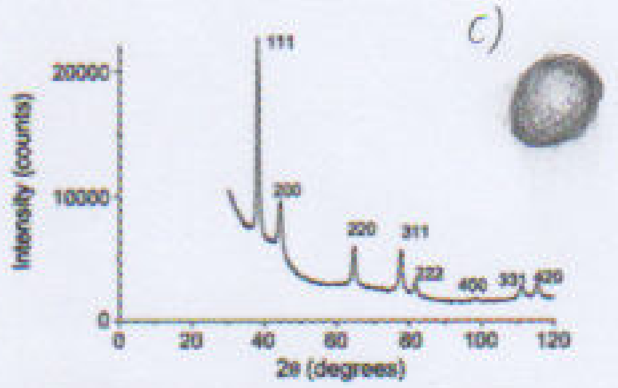


Fig. 2. X-ray diffraction patterns from dispersed Au particles ($a = 4.067(3)$ Å) with diffraction peaks are labeled with *hkl* indices.

Table 2
Results from the pseudo-Voigt fit to the Au particles. 2θ is the peak position for the Cu $K\alpha_1$ radiation wavelength (0.154056 nm).

hkl	Bragg angle 2θ (°)	Peak width ${}^{T}\beta$ (°)	Instrument resolution (°)	Corrected β (°)	Diameter (nm)
111	38.21	0.580	0.114	0.47	[redacted]
200	44.34	0.822	0.115	0.71	
220	64.69	0.810	0.123	0.68	
311	77.63	0.905	0.135	0.77	
420	115.34	1.194	0.119	1.07	

a) What can you tell about the $Ni(OH)_2$ and the Au nanoparticles by just looking at the data?
b) Calculate the nanoparticle dimensions for the 001 and 100 reflections for the $Ni(OH)_2$ and 111, 200 reflections for the Au particles.
c) Sketch the dimensionality of each of the particles.
d) Which lattice do the particles exhibit? Work on the Au nanoparticles first and guess for the $Ni(OH)_2$ particles.

b) $Size = \dfrac{k \cdot \lambda}{width \cdot \cos\theta}$ (see exercise 2)

	$Ni(OH)_2$		Gold
001	7,9 nm	111	17,9 nm
100	103,5 nm	200	12,1
101	17,5	220	13,8
102	11,6	311	13,2
110	70,2	420	13,9
111	27,6		

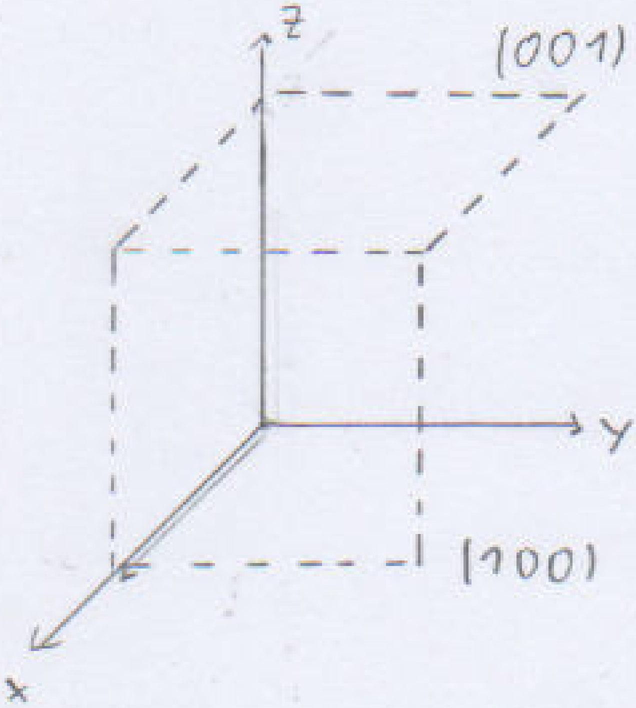
a) For Gold the peaks have a somewhat similar width → almost spherical particle
For the $Ni(OH)_2$ particles the peaks on perpendicular planes (100 and 001) differ severely → cylindrical particle

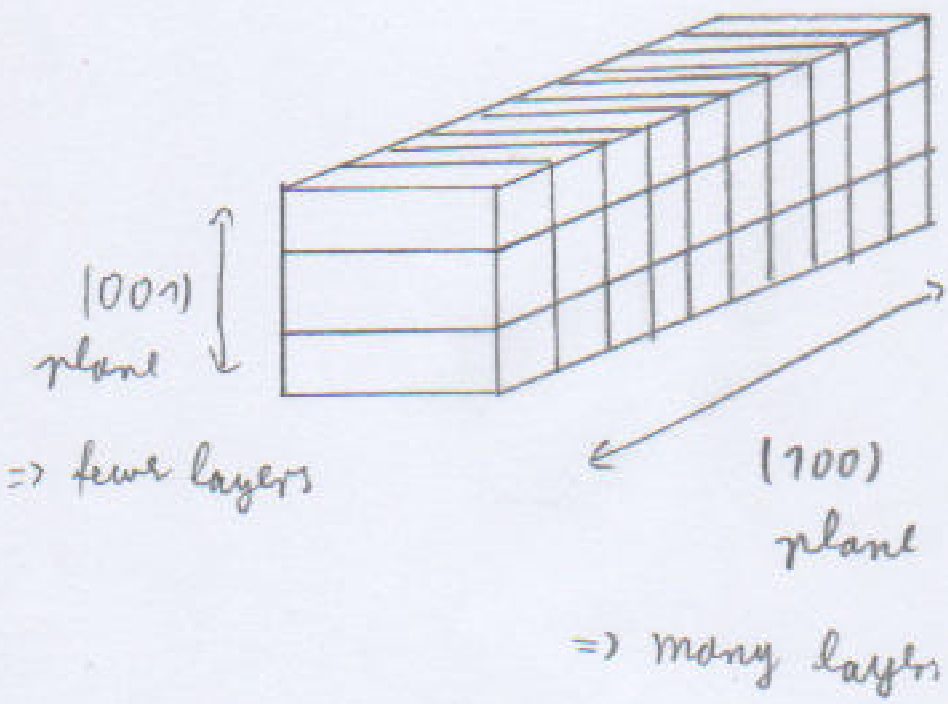
d) Gold : fcc
$Ni(OH)_2$: SC

$Ni(OH)_2$

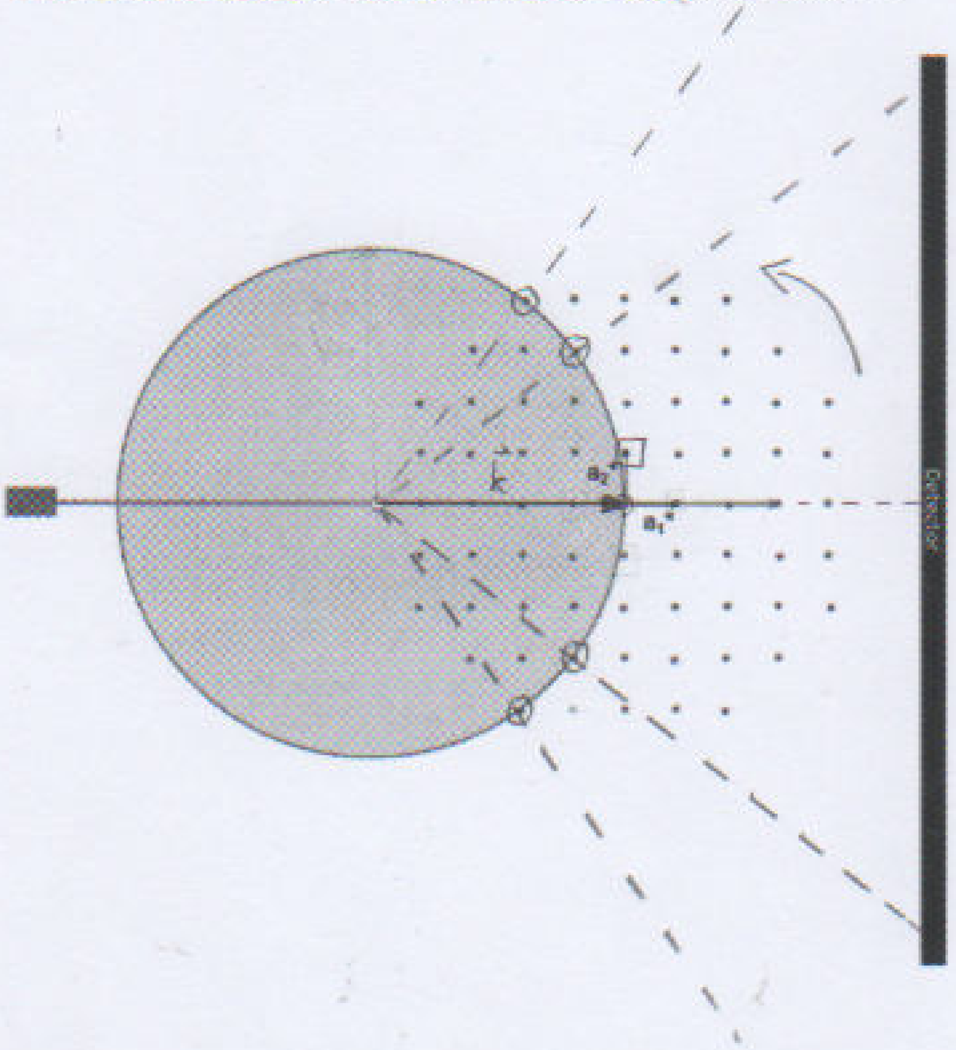
Plane	2θ	β	Diameter
001	19	1.02	8 nm
100	33	0.08	104 nm

=> Broader peak indicates fewer layers





2) The figure below shows a x-ray diffraction experiment in an Ewald sphere construction. The inverse lattice vector **a** has a length of $4 nm^{-1}$.



a) Which points will yield a diffraction peak?
b) Which Bragg peaks will be observable on the detector?
c) What do you need to do to observe the (01) peak)?
d) What is the photon energy in this experiment? Remember that 1.24 keV corresponds to a wavelength of 1 nm.

a) The ones on the Ewald sphere

(-1 3) (-2 4)

(-1 -3) (-2 -4)

b) (-1 3) (-1 -3)

c) Slightly rotate the crystal counter clockwise

d) $|\vec{k}| = 5 \cdot 4 nm^{-1} = 20 \ nm^{-1}$

$\Rightarrow h\nu = |\vec{k}|^{-1} = (20 \ nm^{-1})^{-1} = 24{,}8 \ keV$