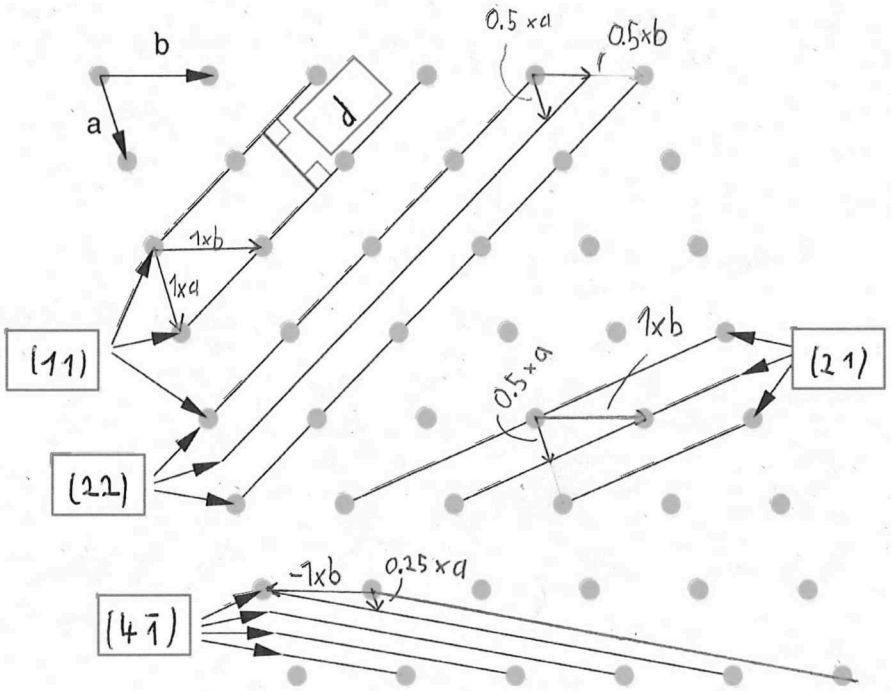
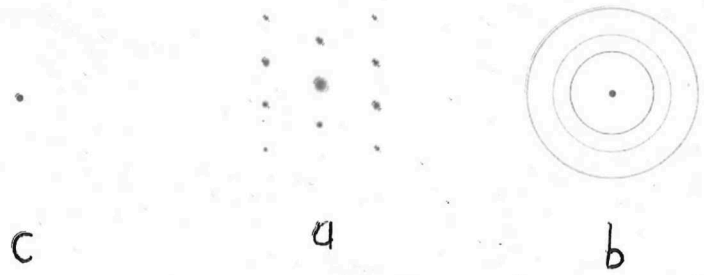
CH314 – Structural Analysis
Part III : X-ray Tools

Exercise 2

1) Identify the lattice planes



2) Which diffraction patter do you attribute to a) single crystal b) powder c) nanocrystalline powder



3) You perform a powder diffraction experiment on your colloidal gold nanoparticles. From the data, figure out if your particles are spherical or if they have a preferred axis. Consider the Debye-Scherrer formalism.

Journal of Colloid and Interface Science 338 (2009) 105–110

Contents lists available at ScienceDirect

Journal of Colloid and Interface Science

www.elsevier.com/locate/jcis

Use of wide-angle X-ray diffraction to measure shape and size of dispersed colloidal particles

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The diffraction experiments were carried out with a Stoe STADI-P X-ray powder diffractometer at University College London with a Ge(111) monochromator before the sample. A Cu $K\alpha_1$ X-ray beam of wavelength 0.154056 nm was used. The samples were measured in 0.35 mm diameter glass capillaries at room temperature. Capillary diameters were checked to be equal with a micrometer. The diffraction patterns were collected using a position-sensitive

Since the particle diameter for various lattice planes is similar the particle appears to be spherical or maybe elliptical.

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 'β' (°)	Instrument resolution (°)	Corrected β (°)	Diameter (nm)
111	38.21	0.580	0.114	0.47	18
200	44.34	0.822	0.115	0.71	12
220	64.69	0.810	0.123	0.68	13
311	77.63	0.905	0.135	0.77	13
420	115.34	1.194	0.119	1.07	15

Example calculation

$2\theta = 38.21°$

$\Rightarrow \theta = 19.105° = 0.333$ rad

Make sure to convert to radians!

Corrected width $= \beta = 0.47° = 0.0082$ rad

$$\text{Diameter} = \frac{k \cdot \bar{\lambda}}{\text{width} \cdot \cos\theta} = \frac{0.9 \cdot 0.154056\ \text{nm}}{0.0082 \cdot \cos(0.333)} = 17.9\ \text{nm}$$