Basic Operation and Analysis of Au nanoparticles by TEM

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實習題目：TEM 實驗

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目錄

1. Objectives:

Familiar with analysis technique of C-TEM, including:

Basic structure of electron microscope

Bright field image

Diffraction pattern

Dark field image

Sample:

Au nanoparticle

2. Instrument:

FEI TECNAI F20 Field Emission Gun Transmission Electron Microscope

3. Procedure:

1. Sample on holder
2. Basic structure of electron microscope and essential components of TEM
3. Alignment of TEM for basic observation
4. Taking one picture in bright-field(BF)mode condition with the paper magnifications.
5. turns to diffraction mode to get diffraction pattern of the Au nanoparticle.
6. Taking one picture of the DP
7. turn to diffraction mode to select one diffraction spot by OBJ aperture and take one dark field.

4. Report

4.1 Present the JCPDS file of the Au, indexing the diffraction pattern;

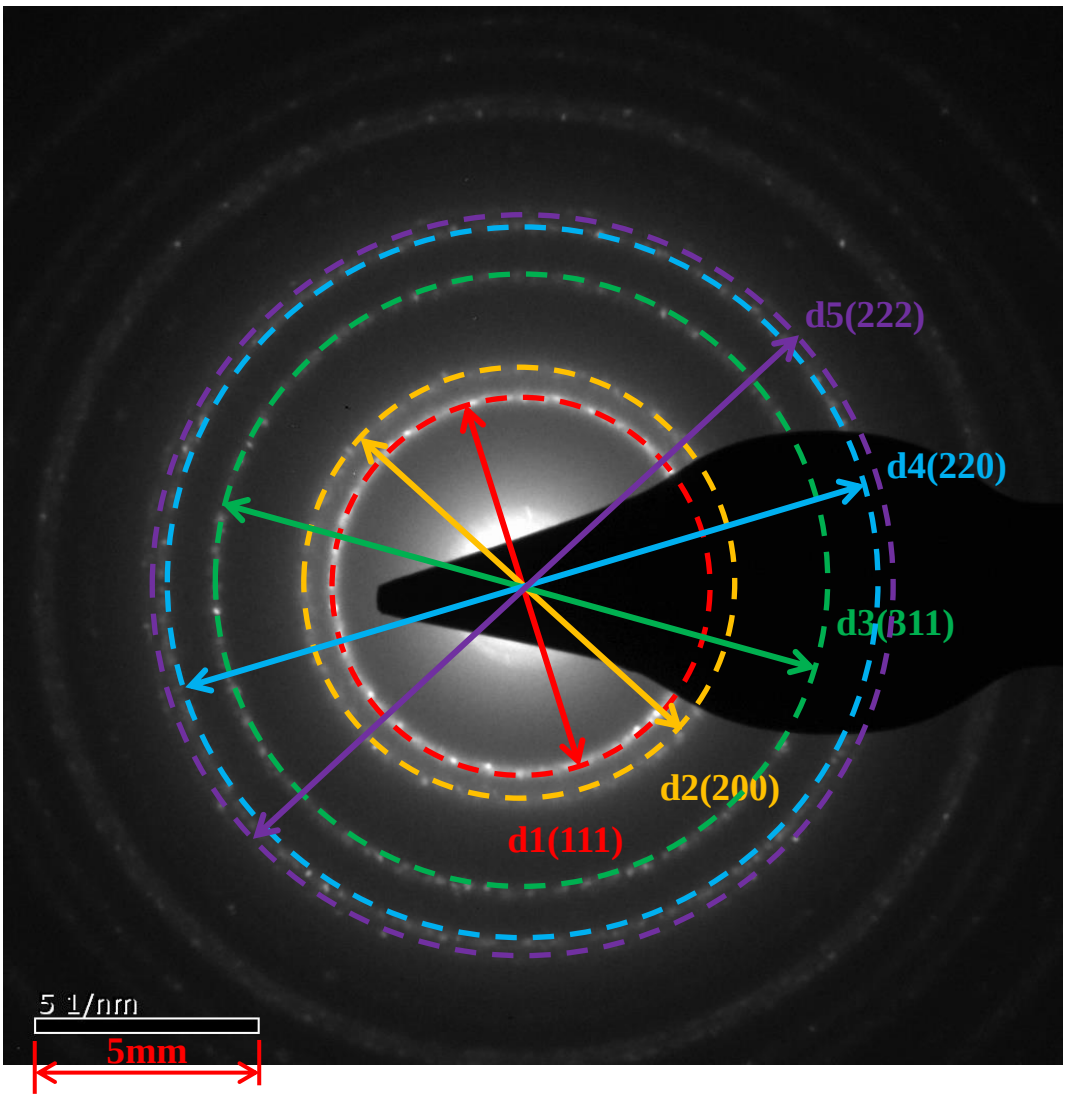

Fig. 1 Diffraction Pattern

經測量後

	D1	D2	D3	D4	D5
數值(mm)	8.428	9.694	13.702	16.117	16.877

$d1:\ d2:\ d3:\ d4:\ d5 = 8.428:\ 9.694:13.702:16.117:16.877$

$\Rightarrow$ r1 : r2 : r3 : r4 : r5 = 4.214 : 4.847 : 6.851 : 8.059 : 8.439

= 1 : 1.15 : 1.626 : 1.912 : 2.002

與 FCC 結構相似 (FCC 1: 1.155 : 1.633 : 1.915)

故可判定金為 FCC 結構。

$$r1 = \frac{4.214\ mm}{5\ mm} = \frac{x1}{5\ (1/nm)} \Rightarrow x1 = 4.214(1/nm)$$

d1=4.214 $(1/\mathrm{nm})$=0.2373 nm = 2.373 Å

$$r2 = \frac{4.847\ mm}{5\ mm} = \frac{x2}{5\ (1/nm)} \Rightarrow x2 = 4.847(1/nm)$$

d2=4.847 $(1/\text{nm})$=0.2063 nm = 2.063 Å

$$r3 = \frac{6.851\ mm}{5\ mm} = \frac{x3}{5\ (1/nm)} \Rightarrow x3 = 6.851(1/nm)$$

d3=6.851 $(1/\text{nm})$=0.1460 nm = 1.460 Å

$$r4 = \frac{8.059\ mm}{5\ mm} = \frac{x4}{5\ (1/nm)} \Rightarrow x4 = 8.059\ (1/nm)$$

d4=8.059 $(1/\text{nm})$=0.1241 nm = 1.241 Å

$$r5 = \frac{8.439\ mm}{5\ mm} = \frac{x5}{5\ (1/nm)} \Rightarrow x5 = 8.439\ (1/nm)$$

d5=8.439 $(1/\text{nm})$=0.1185 nm = 1.185 Å

經轉換後，查表得知

d(A)	Int	h	k	l
2.355	100	1	1	1
2.039	52	2	0	0
1.442	32	2	2	0
1.230	36	3	1	1
1.177	12	2	2	2
1.019	6	4	0	0
.9358	23	3	3	1
.912	22	4	2	0
.8325	23	4	2	2

	d1	d2	d3	d4	d5
實際值(Å)	2.373	2.063	1.460	1.241	1.185
理論值(Å)	2.355	2.039	1.442	1.230	1.177
結晶面	(111)	(200)	(220)	(311)	(222)

4.2 Compare the image conditions of bright field, contrast mechanism.

Bright Field: 調整光圈使中央透射電子束(直射電子)通過，成像清晰。

Dark Field: 調整光圈使旁邊散射電子束(繞射電子)通過，成像分辨率低。

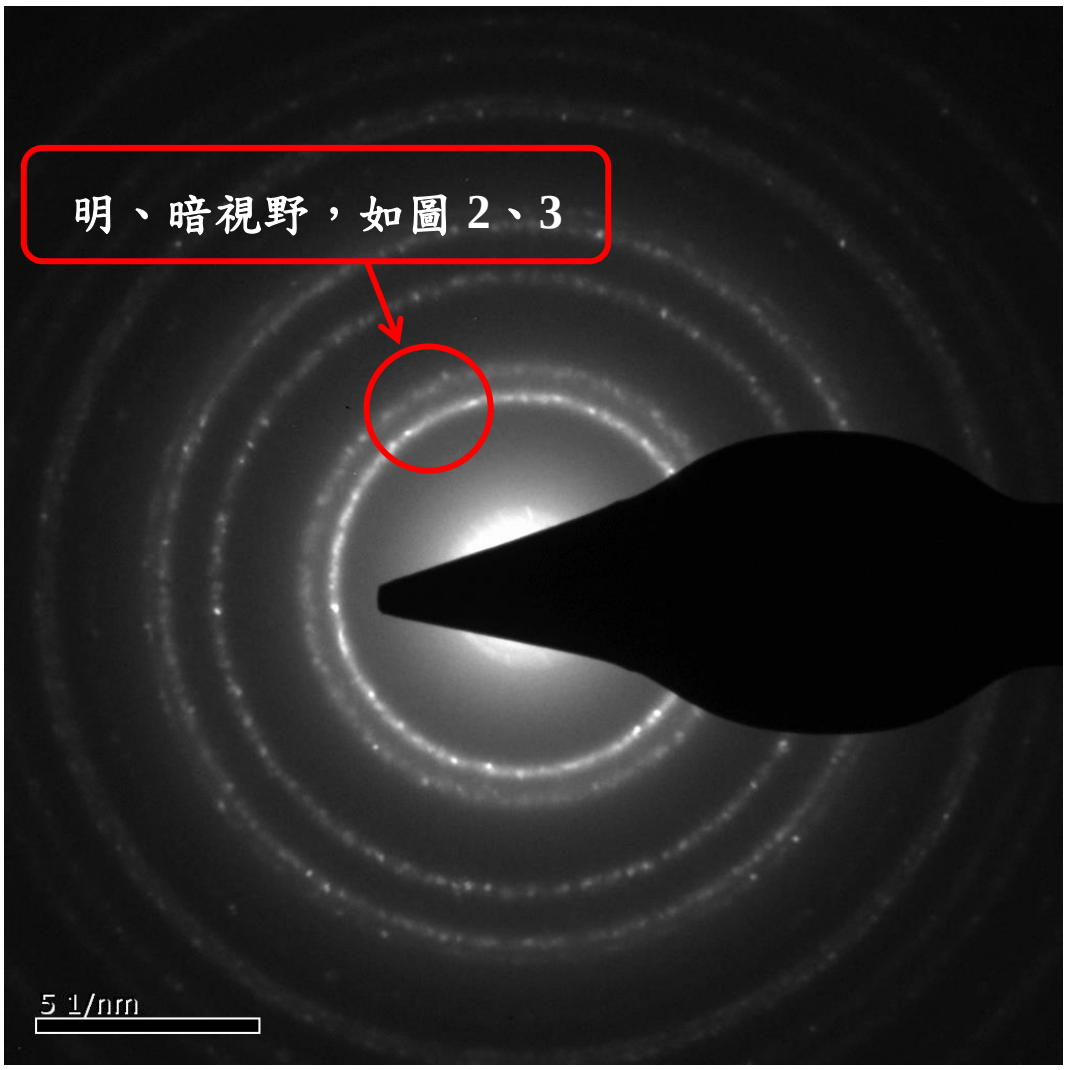

Fig. 2 Diffraction Pattern

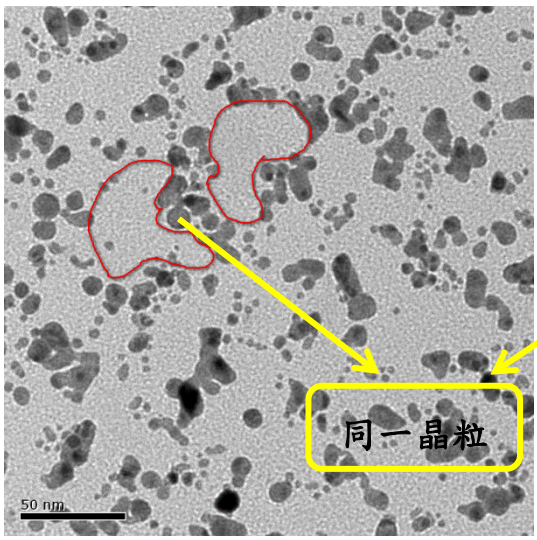

Fig. 2 Bright Field

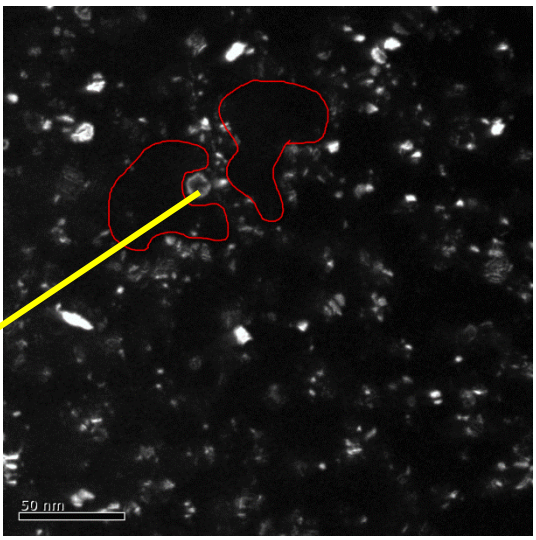

Fig. 3 Dark Field

4.3 Measure the d-spacing and lattice constant of Au and compare with the theoretical value in JCPDS file.

4.3.1 D-spacing calculate

	d1	d2	d3	d4	d5
實際值(Å)	2.373	2.063	1.460	1.241	1.185
理論值(Å)	2.355	2.039	1.442	1.230	1.177
誤差(%)	0.764	1.177	1.248	0.894	0.680
結晶面	(111)	(200)	(220)	(311)	(222)

$$\mathrm{d} = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$

$$\mathrm{d1} = \frac{a1}{\sqrt{1^2 + 1^2 + 1^2}} = 2.373\ \text{Å}$$

$$\Rightarrow \mathrm{a1} = 2.373\ \text{Å} \times \sqrt{3} = 4.110\ \text{Å}$$

$$\mathrm{d2} = \frac{a2}{\sqrt{2^2 + 0^2 + 0^2}} = 2.063\ \text{Å}$$

$$\Rightarrow \mathrm{a2} = 2.063\ \text{Å} \times 2 = 4.126\ \text{Å}$$

$$\mathrm{d3} = \frac{a3}{\sqrt{2^2 + 2^2 + 0^2}} = 1.460\ \text{Å}$$

$$\Rightarrow \mathrm{a3} = 1.460\ \text{Å} \times \sqrt{8} = 4.130\ \text{Å}$$

$$\mathrm{d4} = \frac{a4}{\sqrt{3^2 + 1^2 + 1^2}} = 1.241\ \text{Å}$$

$$\Rightarrow \mathrm{a4} = 1.241\ \text{Å} \times \sqrt{11} = 4.116\ \text{Å}$$

$$\mathrm{d5} = \frac{a5}{\sqrt{2^2 + 2^2 + 2^2}} = 1.185\ \text{Å}$$

$$\Rightarrow \mathrm{a5} = 1.185\ \text{Å} \times \sqrt{12} = 4.105\ \text{Å}$$

4.3.2 Lattice constant calculate

	d1	d2	d3	d4	d5
實際值	4.110	4.126	4.130	4.116	4.105
理論值	4.0789	4.0789	4.0789	4.0789	4.0789
誤差(%)	0.766	1.155	1.241	0.908	0.639

5. Conclusions

根據 D-spacing 跟 Lattice constant 的誤差比率值落在 1~2% 區間，此誤差在可接收範圍，故此次 TEM 校正正常，接下來的實驗數據具有可信度。

6. References

1. Professor Power Pont.
2. W. Wong-Ng, H. F. McMurdie, C. R. Hubbard1, and A. D. Mighell “JCPDS-ICDD Research Associateship(Cooperative Program with NBS/NIST)” J. Res. Natl. Inst. Stand. Technol. 106, 1013–1028 (2001).