3. Experimental Technique

a. Apparatus

The electron diffraction tube is a small cathode ray tube (CRT) similar to that in a small (old-fashioned) TV set. The electrons pass through a target that consists of graphite, only a few molecular layers thick, vapor-deposited on a micromesh nickel grid. The target is crystalline in tiny regions, so a "powder" diffraction pattern results, seen as a pair of rings around a central spot on the luminescent screen. See diagram below. The distance between the target and phosphor screen is L = 13.5 cm.

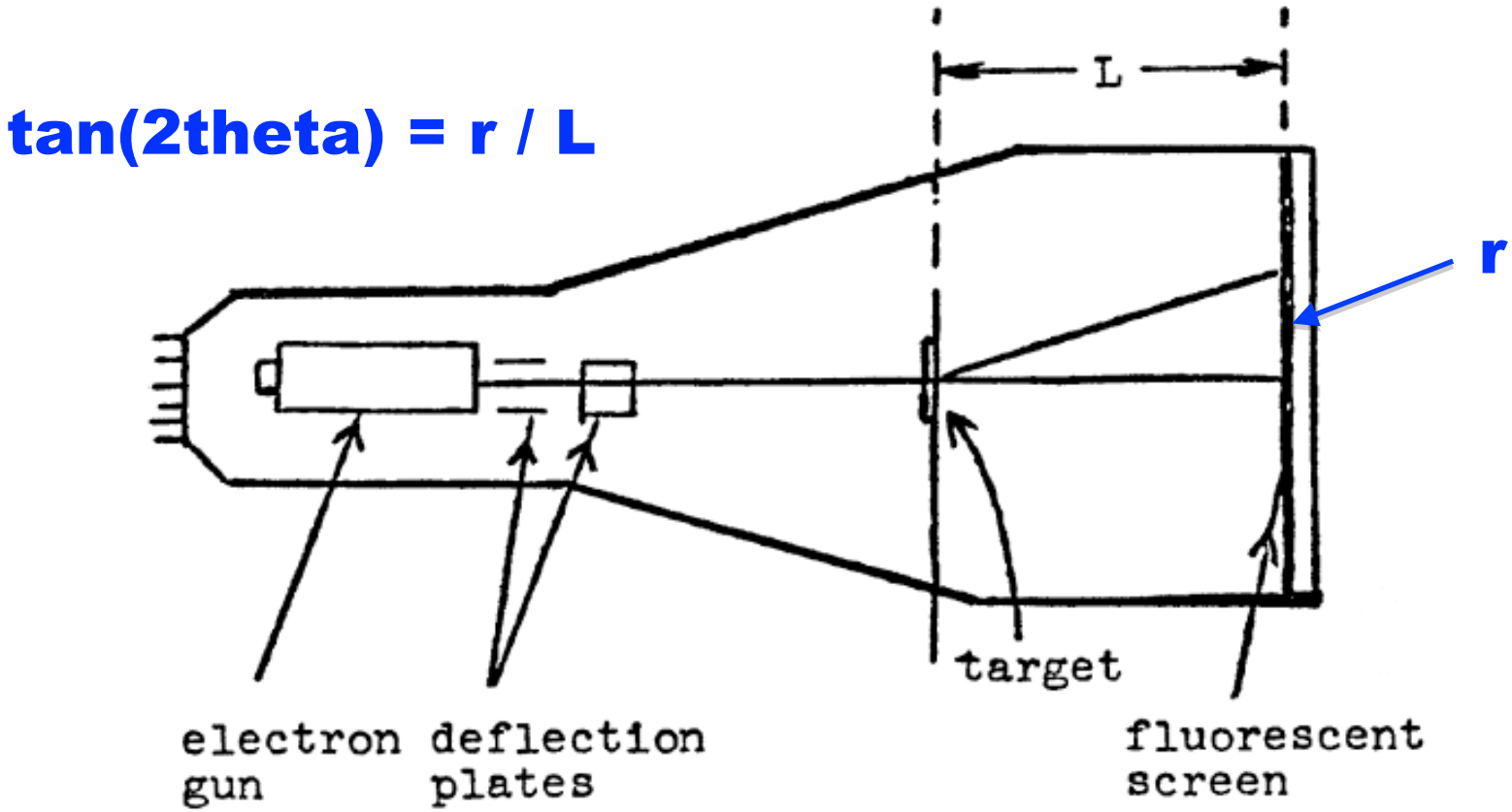


Figure 1 Schematic of the CRT tube

tan(2theta) = r / D

n lambda = 2 d sin(theta)

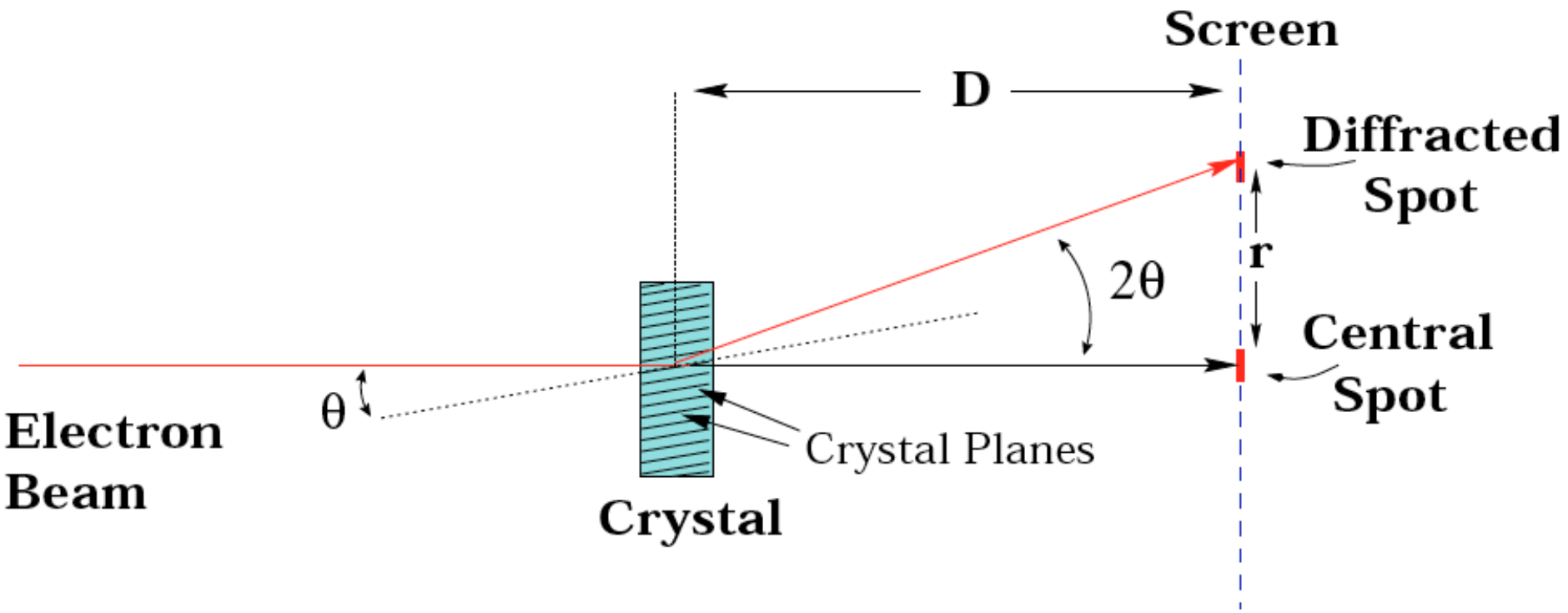
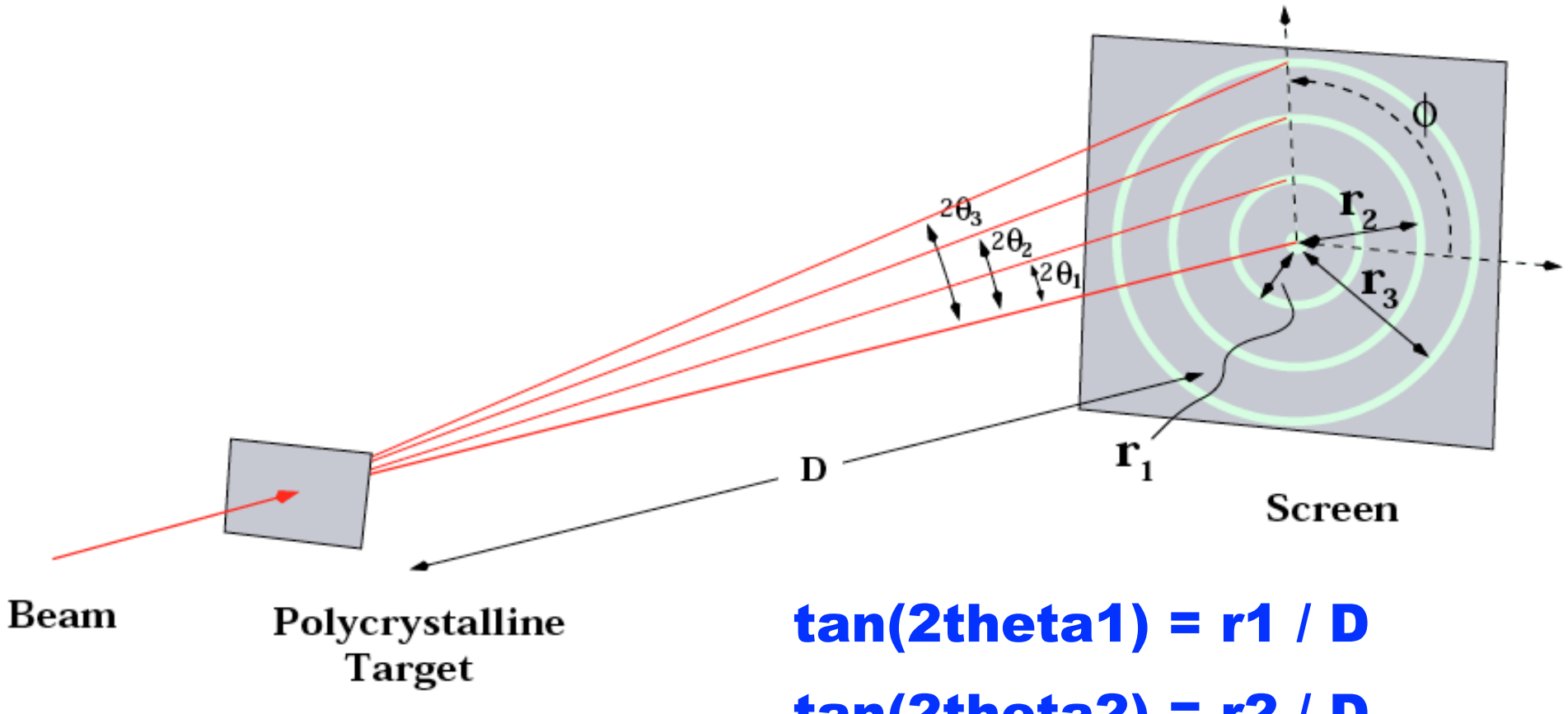


Figure 7 Crystal Diffraction Geometry

The total angle of a diffraction maxima due to reflection off of a particular crystal lattice plane is twice the angle of incidence on that plane. This angle may be approximated as the distance of the diffraction spot from the undiffracted beam over the distance between the imaging screen and the crystal, i.e. $q \approx 1/2\ r/D$.

Multiple diffraction rings



tan(2theta1) = r1 / D

tan(2theta2) = r2 / D

tan(2theta3) = r3 / D

single crystals => diffraction peaks

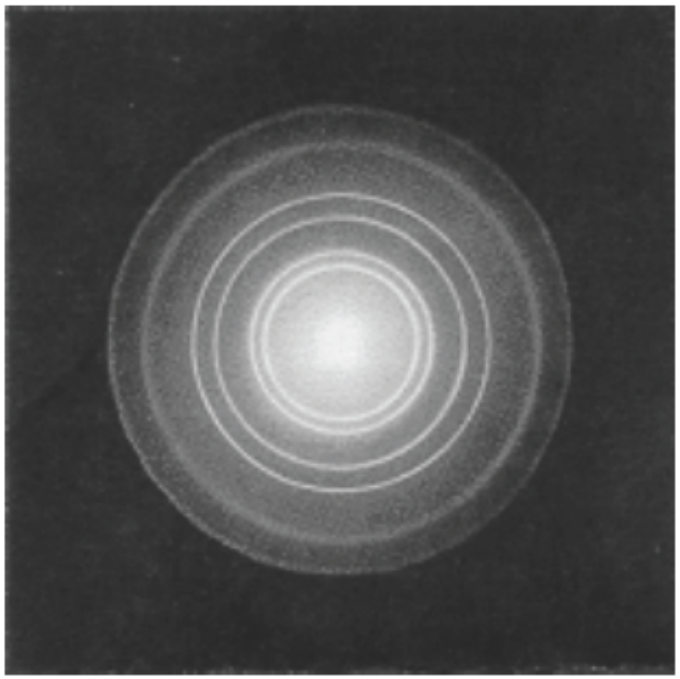
powder => diffraction rings

multiple crystals => spotted rings

graphene

also graphite

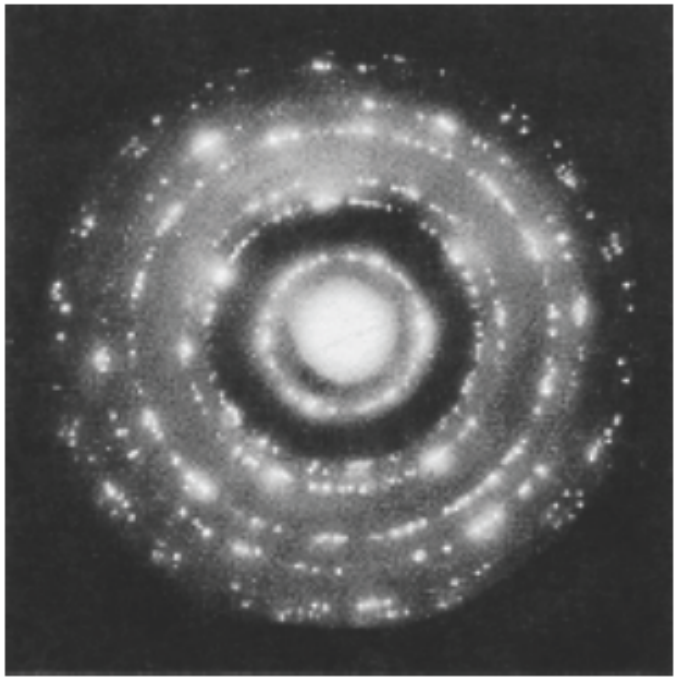
single crystal



fcc aluminum

polycrystalline

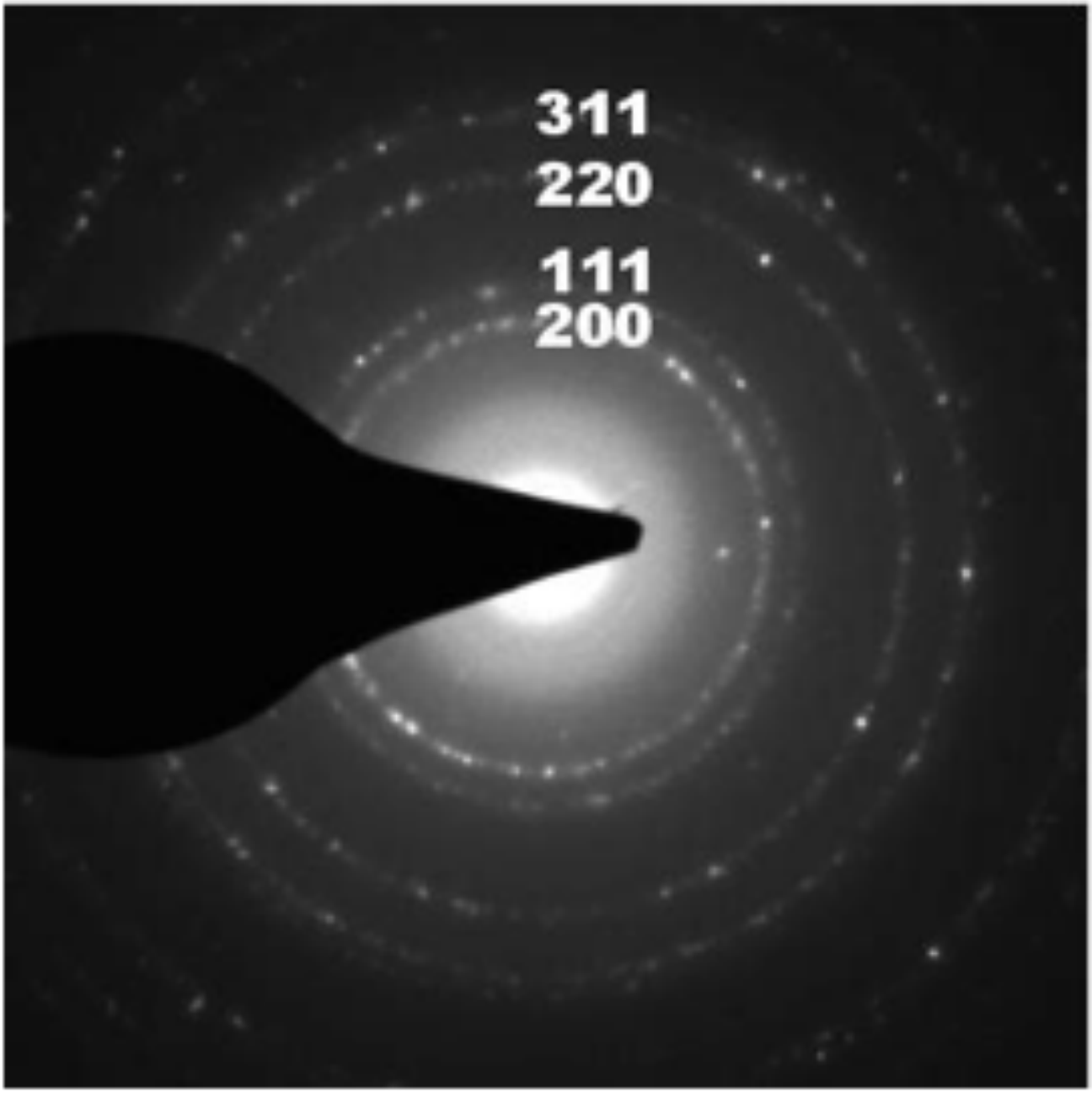
also powder



graphite

pyrolytic

Diffraction pattern for fcc platinum



Wavelength vs Accelerating Voltage

$\frac{U_A}{kv}$	$\frac{\lambda}{pm}$
4.00	19.4
4.50	18.3
5.00	17.3
5.50	16.5
6.50	15.2
7.00	14.7
7.40	14.3

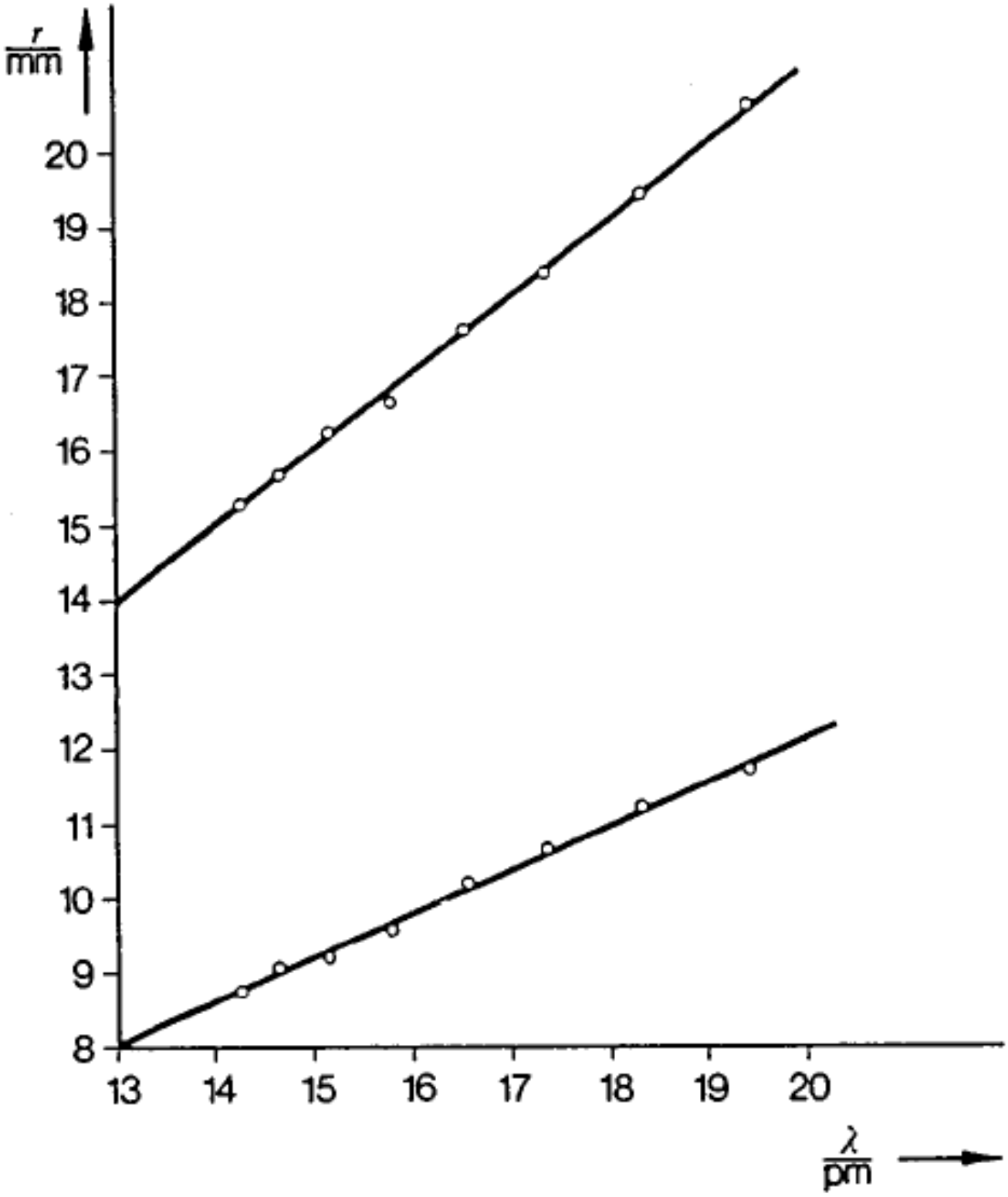


Fig. 5: Radii of the first two interference rings as a function of the wavelength of the electrons.

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Direct Lattice	**Simple Cubic**		**Body-Centred Cubic**	**Face-Centred Cubic**
Reciprocal Lattice (cube lattice parameter)	Simple Cubic ($2\pi/a$)		Face-Centred Cubic ($4\pi/a$)	Body-Centred Cubic ($4\pi/a$)
Planes, spacing	(100)	a	No	No
	(110)	$a/\sqrt{2}$	Yes	No
	(111)	$a/\sqrt{3}$	No	Yes
	(200)	a/2	Yes	Yes
	(210)	$a/\sqrt{5}$	No	No
	(211)	$a/\sqrt{6}$	Yes	No
	(220)	$a/\sqrt{8}$	Yes	Yes
	(221)	a/3	No	No
	(300)	a/3	No	No
	(310)	$a/\sqrt{10}$	Yes	No
	(311)	$a/\sqrt{11}$	No	Yes
	(222)	$a/\sqrt{12}$	Yes	Yes
	(320)	$a/\sqrt{13}$	No	No
	(321)	$a/\sqrt{14}$	Yes	No
	(400)	$a/\sqrt{16}$	Yes	Yes
Rule:	All h, k, l		Sum (h + k + l) even	h, k, l all odd or all even
Ratio of sines of Bragg angles	$1:\sqrt{2}:\sqrt{3}:2:\sqrt{5}:\sqrt{6}:\sqrt{8}:...$		$1:\sqrt{2}:\sqrt{3}:2:\sqrt{5}:\sqrt{6}:\sqrt{7}:\sqrt{8}:...$	$1:2/\sqrt{3}:\sqrt{8}/\sqrt{3}:...$

allowed reflections

APPENDIX 6

QUADRATIC FORMS OF MILLER INDICES

Cubic					Hexagonal	
	hkl					
$h^2 + k^2 + l^2$	Simple	Face-centered	Body-centered	Diamond	$h^2 + hk + k^2$	hk
1	100				1	10
2	110	. . .	110		2	
3	111	111	. . .	111	3	11
4	200	200	200		4	20
5	210				5	
6	211	. . .	211		6	
7					7	21
8	220	220	220	220	8	
9	300, 221				9	30
10	310	. . .	310		10	
11	311	311	. . .	311	11	
12	222	222	222		12	22
13	320				13	31
14	321	. . .	321		14	
15					15	
16	400	400	400	400	16	40
17	410, 322				17	
18	411, 330	. . .	411, 330		18	
19	331	331	. . .	331	19	32
20	420	420	420		20	
21	421				21	41
22	332	. . .	332		22	
23					23	
24	422	422	422	422	24	
25	500, 430				25	50
26	510, 431	. . .	510, 431		26	
27	511, 333	511, 333	. . .	511, 333	27	33
28					28	42
29	520, 432				29	
30	521	. . .	521		30	
31					31	51
32	440	440	440	440	32	
33	522, 441				33	
34	530, 433	. . .	530, 433		34	
35	531	531	. . .	531	35	
36	600, 442	600, 442	600, 442		36	60
37	610				37	43
38	611, 532	. . .	611, 532		38	
39					39	52
40	620	620	620	620	40	
41	621, 540, 443				41	
42	541	. . .	541		42	
43	533	533	. . .	533	43	61
44	622	622	622		44	
45	630, 542				45	
46	631	. . .	631		46	
47					47	
48	444	444	444	444	48	44
49	700, 632				49	70, 53

(*cont.*)

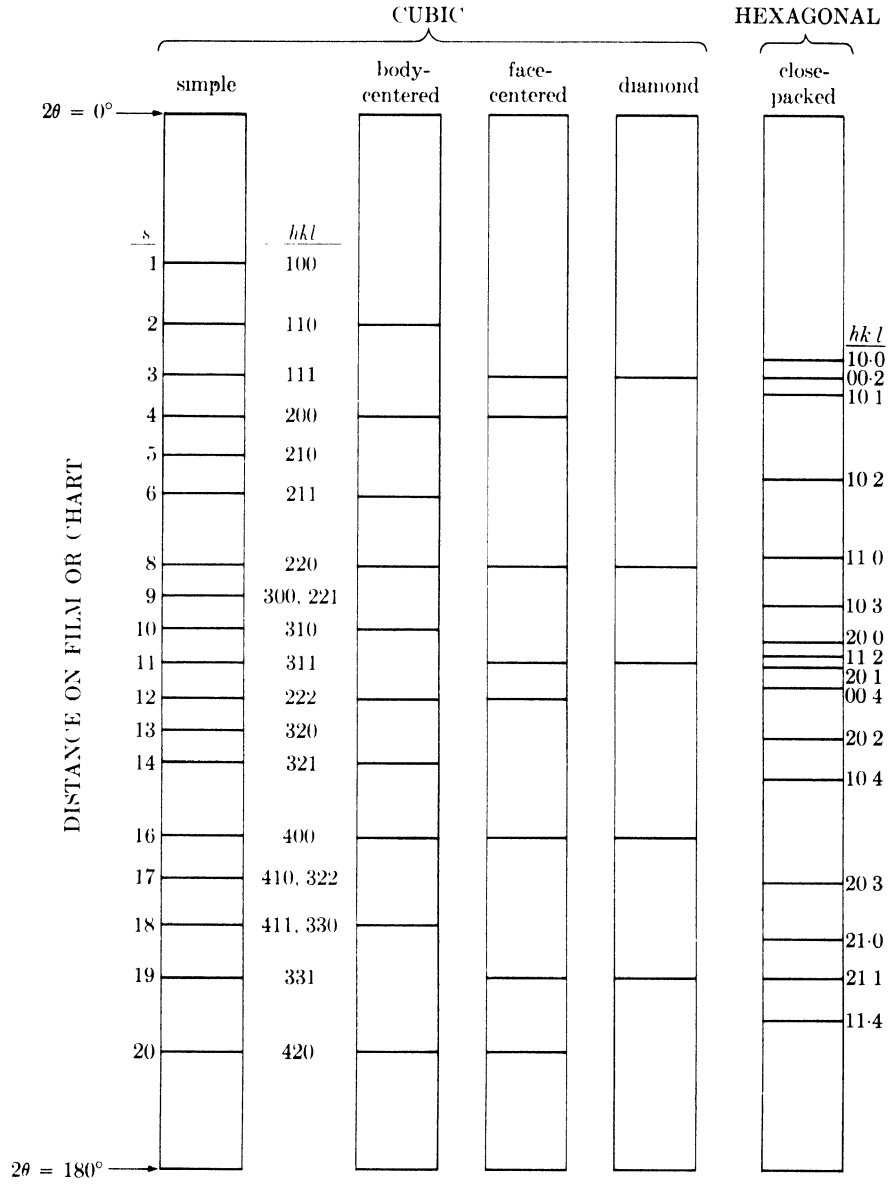
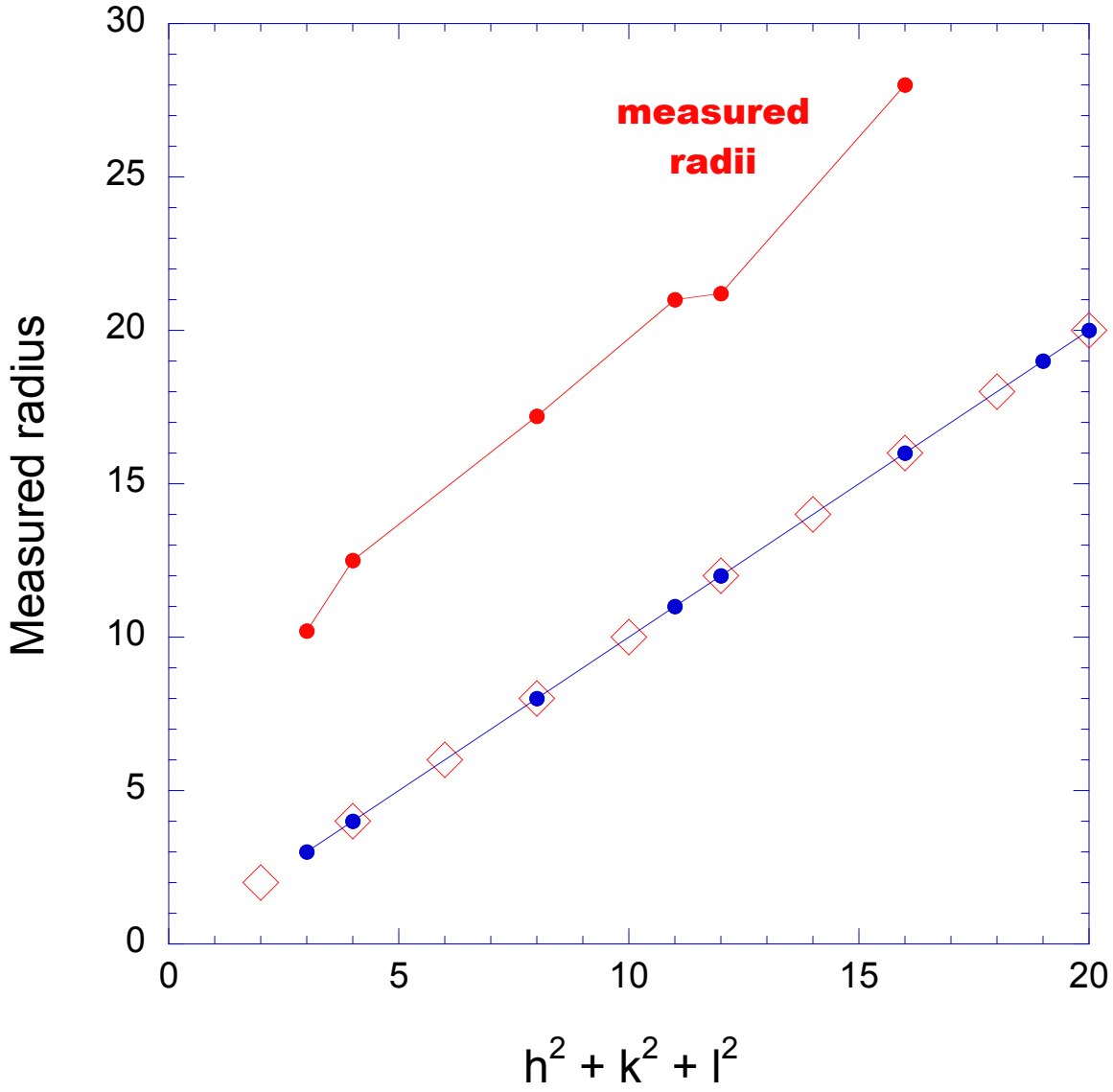


FIG. 10-2. Calculated diffraction patterns for various lattices. $s = h^2 + k^2 + l^2$.

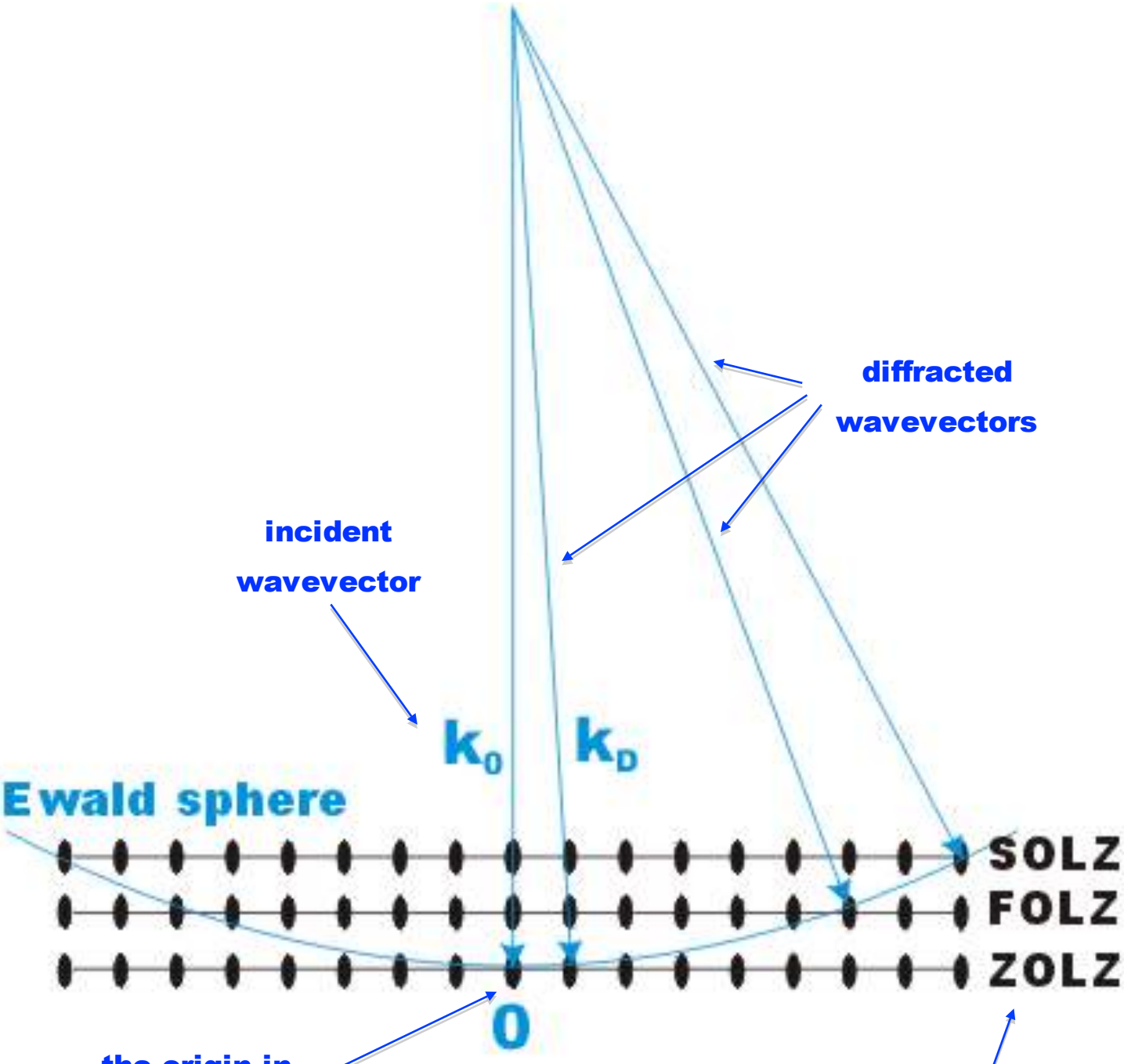
encountered among metals and alloys. The line positions are calculated for Cu $K\alpha$ radiation, $a = 2.50$A, and $c/a = 1.633$, which corresponds to the close packing of spheres.)

Powder patterns of cubic substances can usually be distinguished at a glance from those of noncubic substances, since the latter patterns nor-

allowed FCC reflections = blue dots
allowed BCC reflections = red diamonds
measured reflections = red dots

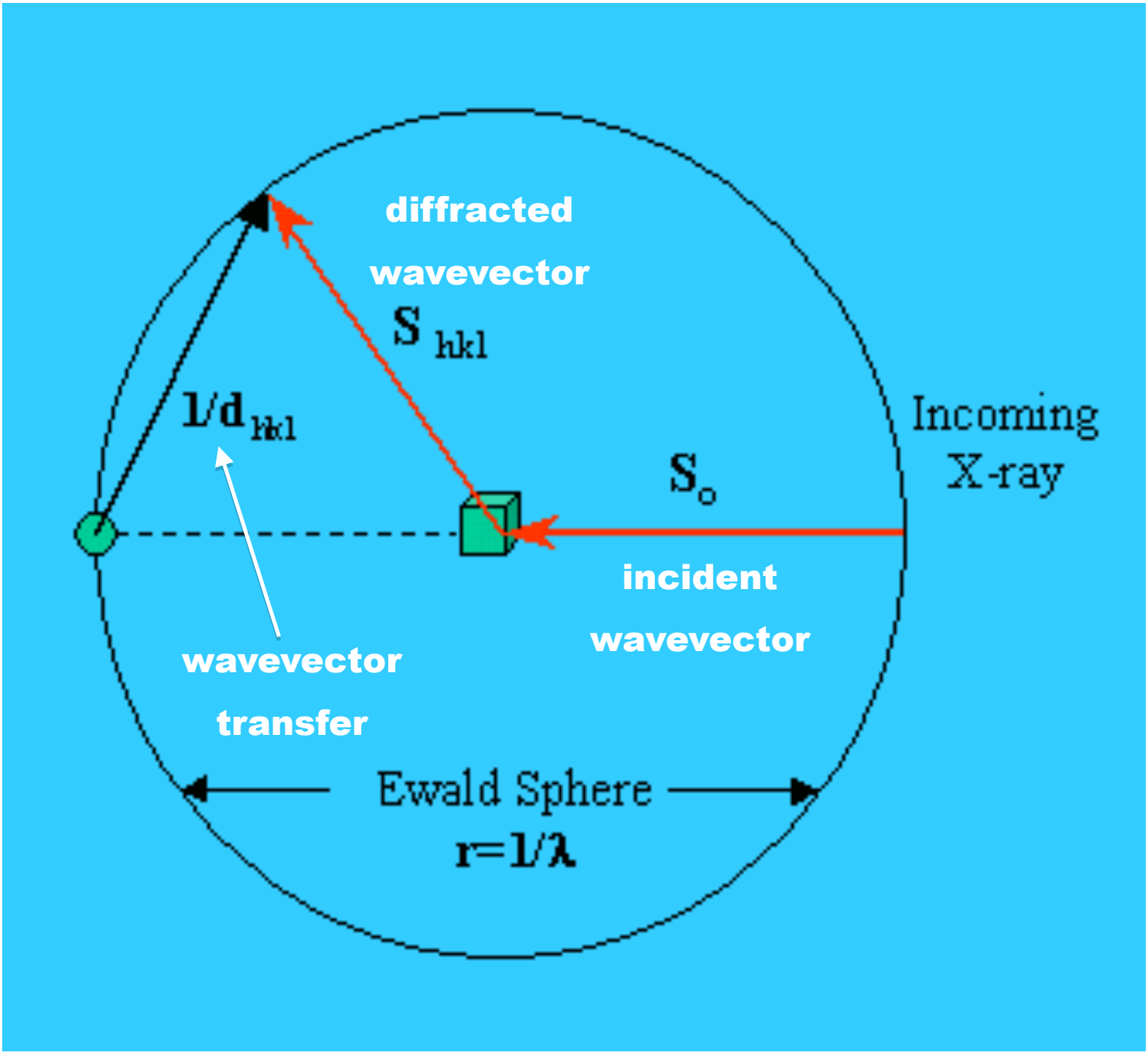


For our electron and optical diffraction experiments the wavelength is small compared to the lattice constant. Consequently, the wavevector k = 2 pi / lambda is much longer than the reciprocal lattice spacing 2 pi / a and the Ewald Sphere cuts through many reciprocal lattice points



In our experiments, we only see ZOLZ peaks

Ewald sphere geometry for x-ray diffraction

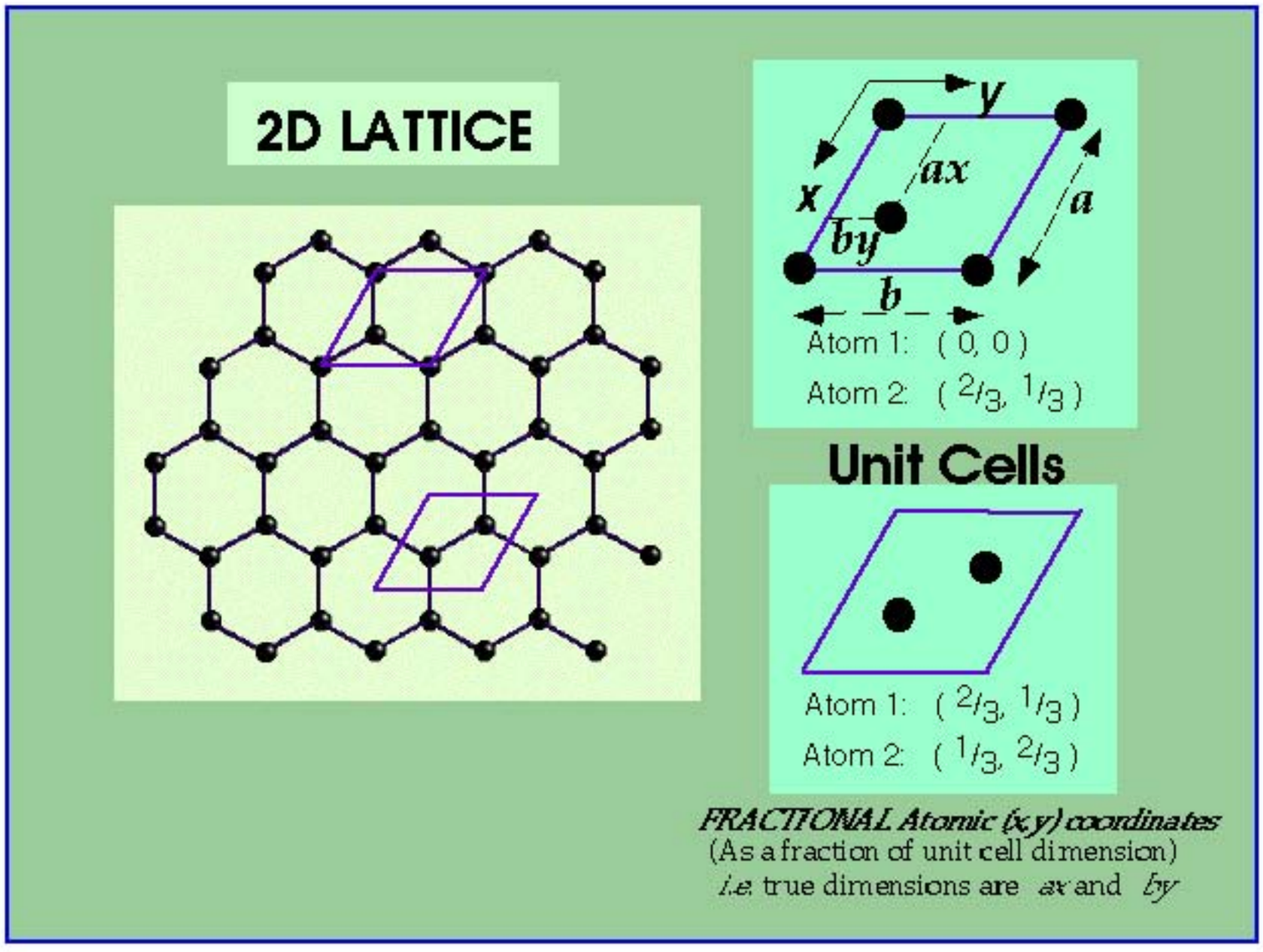


In contrast to electron and optical diffraction where there are many Bragg peaks on the Ewald sphere, in x-ray diffraction there is usually only one Bragg peak on the Ewald sphere

2D LATTICES

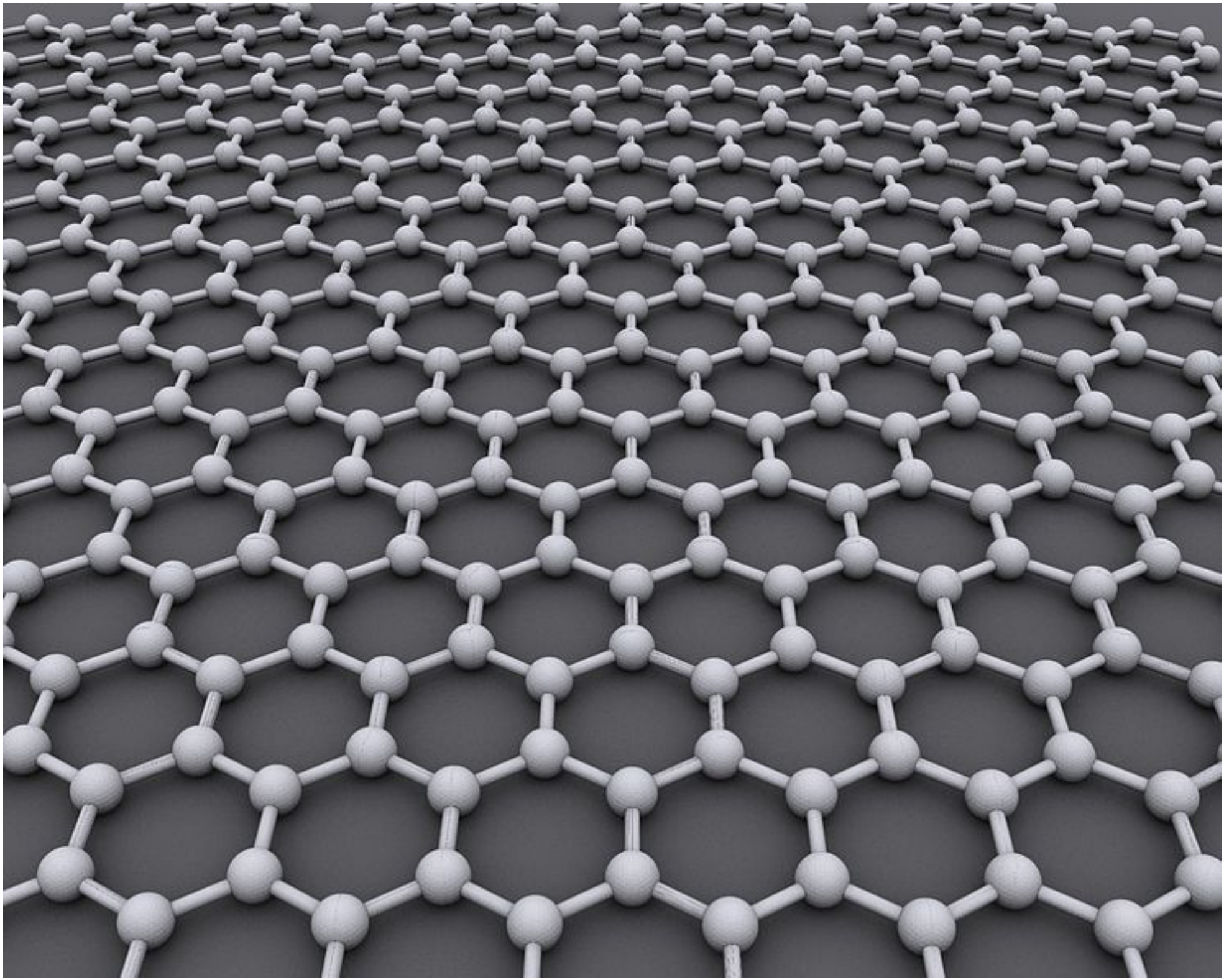
graphene = 1 layer of graphite

e.g. the fused hexagonal pattern of a **single layer of GRAPHITE**



Counting Lattice Points/Atoms in 2D Lattices

- Unit cell is **Primitive (1 lattice point)** but contains **TWO atoms** in the Motif
- Atoms at the **corner** of the 2D unit cell contribute only $^1/_4$ to unit cell count
- Atoms at the **edge** of the 2D unit cell contribute only $^1/_2$ to unit cell count
- Atoms **within** the 2D unit cell contribute **1** (*i.e.* uniquely) to that unit cell



d = a cos(30 degrees)

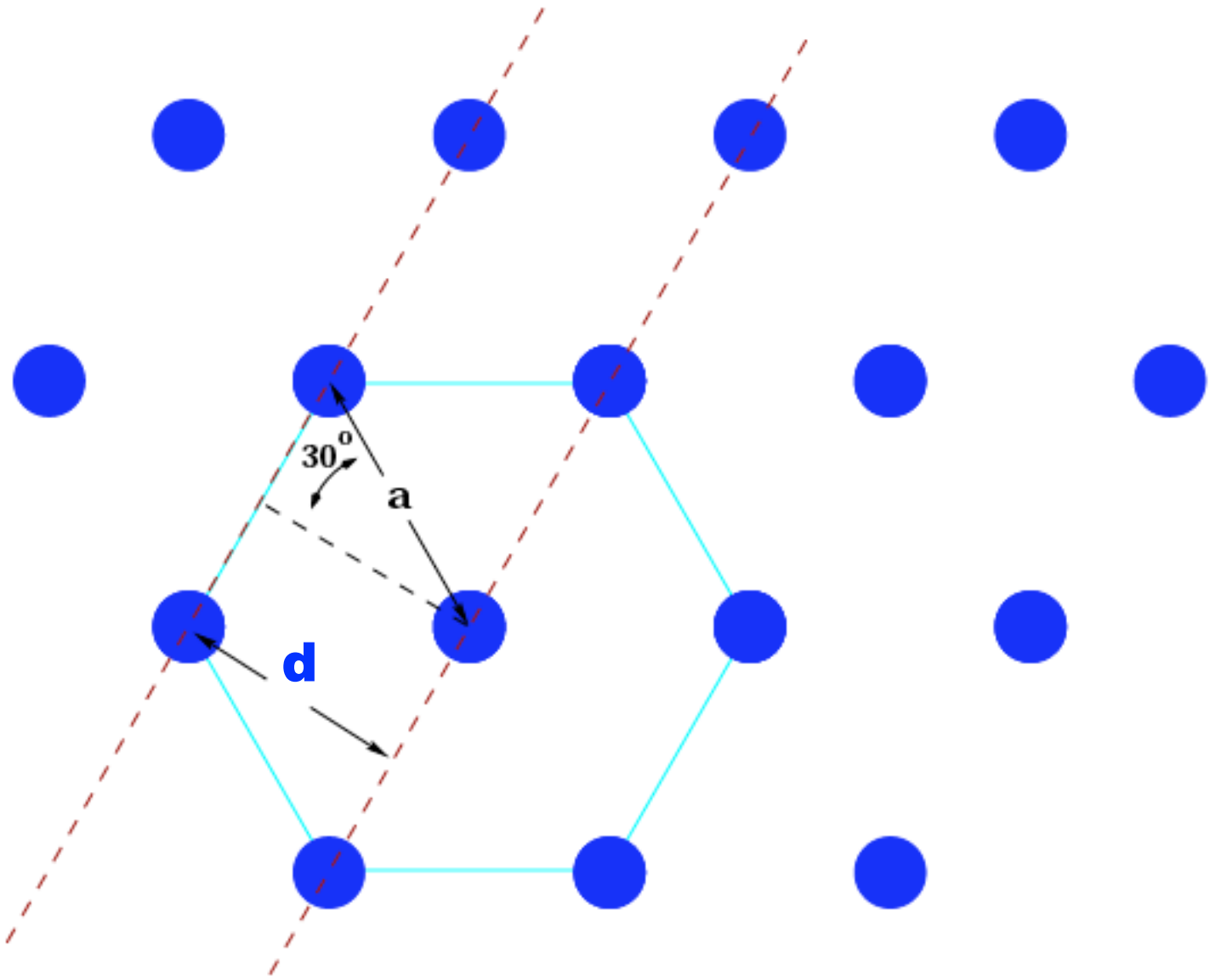


Figure 9 Lattice Plane Spacing of the Fundamental Lattice Planes with the Widest Spacing for Hexagonal Crystal Structures

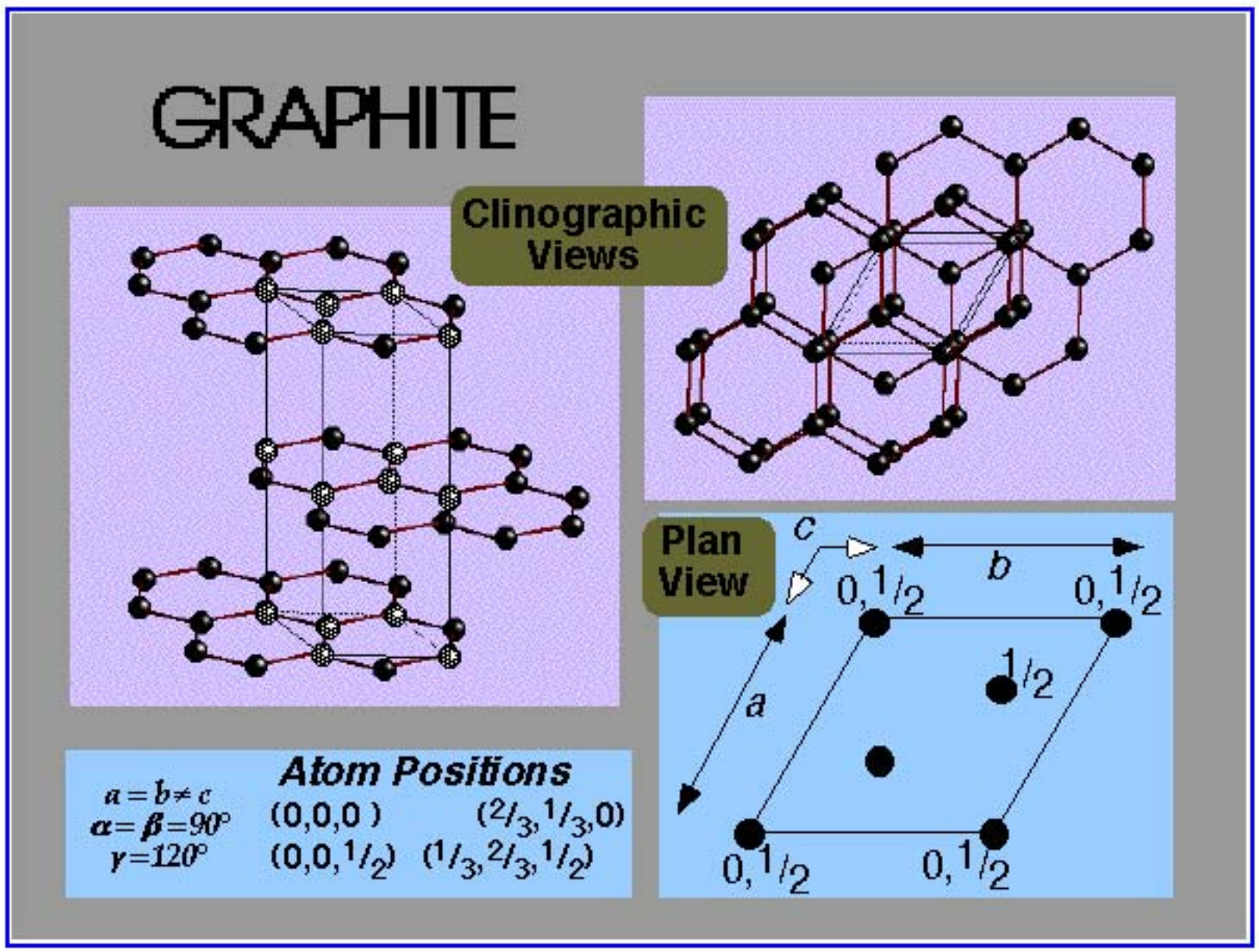
Analysing a 3D solid

e.g. **Graphite** = a staggered arrangement of stacked hexagonal layers

Perspective: Clinographic views of solids

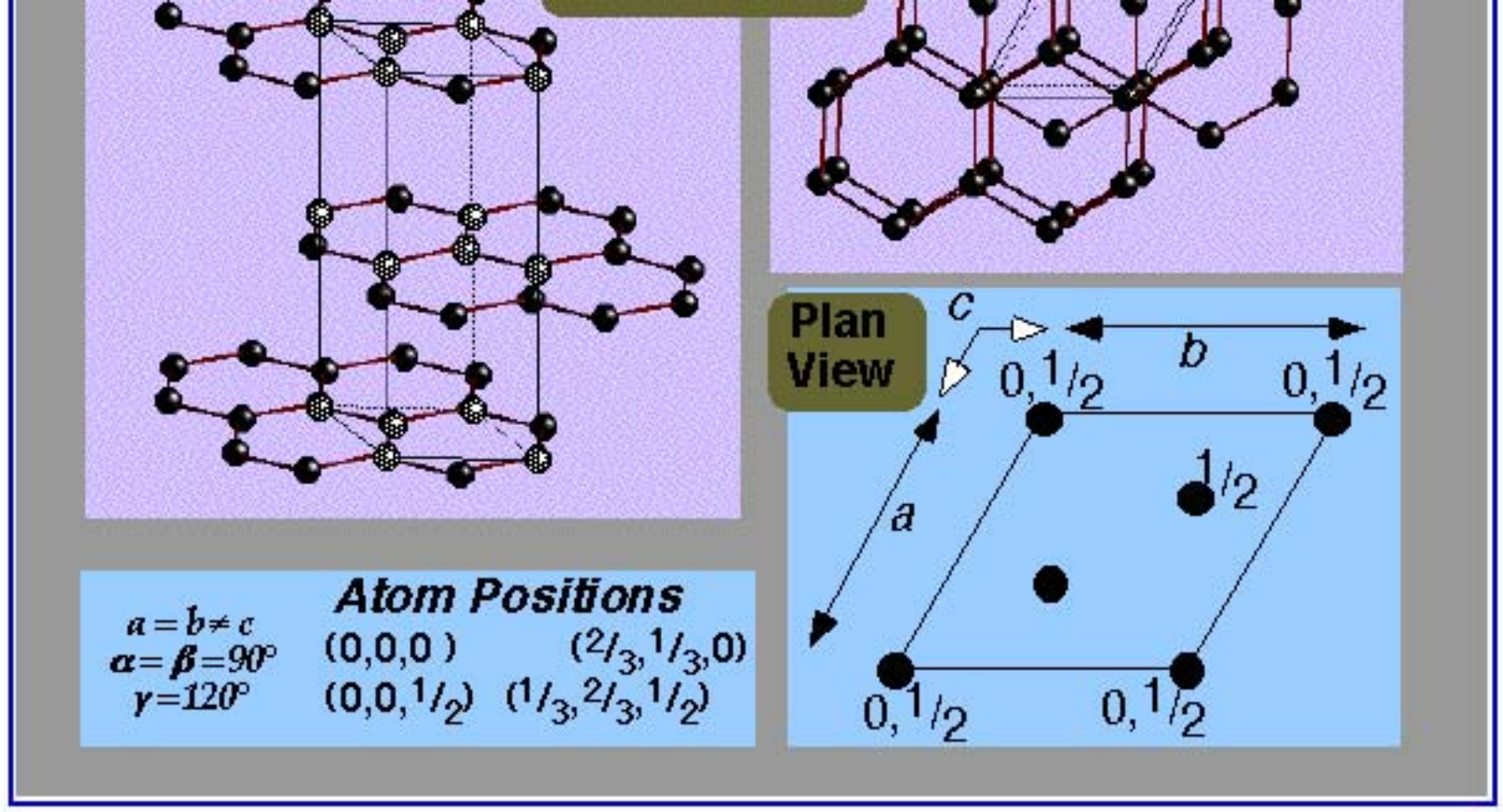
Projection onto a Plane: Plan views of solids

GRAPHITE



Unit Cell Dimensions

- ***a***, ***b*** and ***c*** are the unit cell edge lengths



Graphite continued

Unit Cell Dimensions

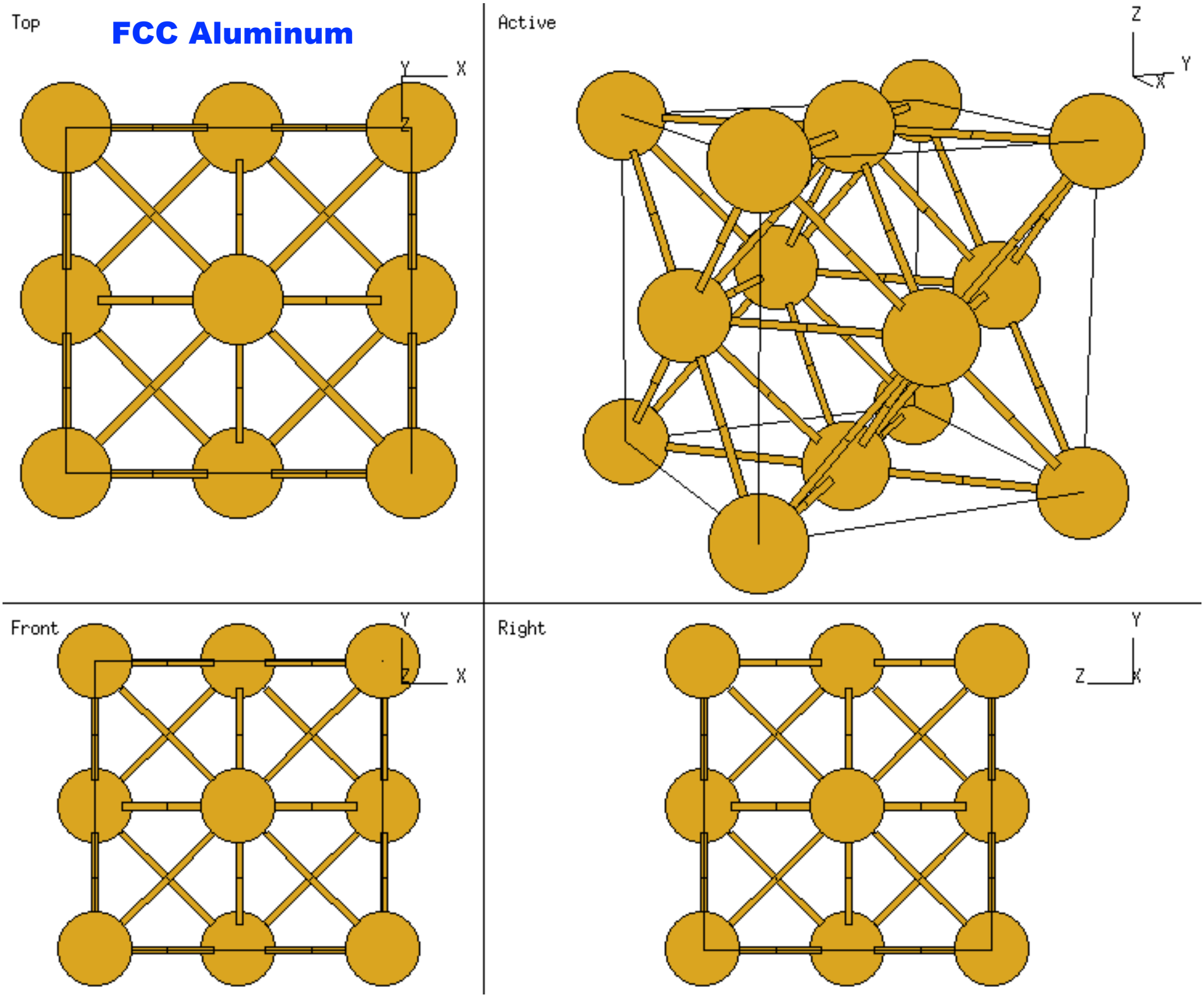
- ***a***, ***b*** and ***c*** are the unit cell edge lengths
- **a**, **b** and **g** are the angles (a between *b* and *c*, etc....)

Counting Atoms in 3D Cells

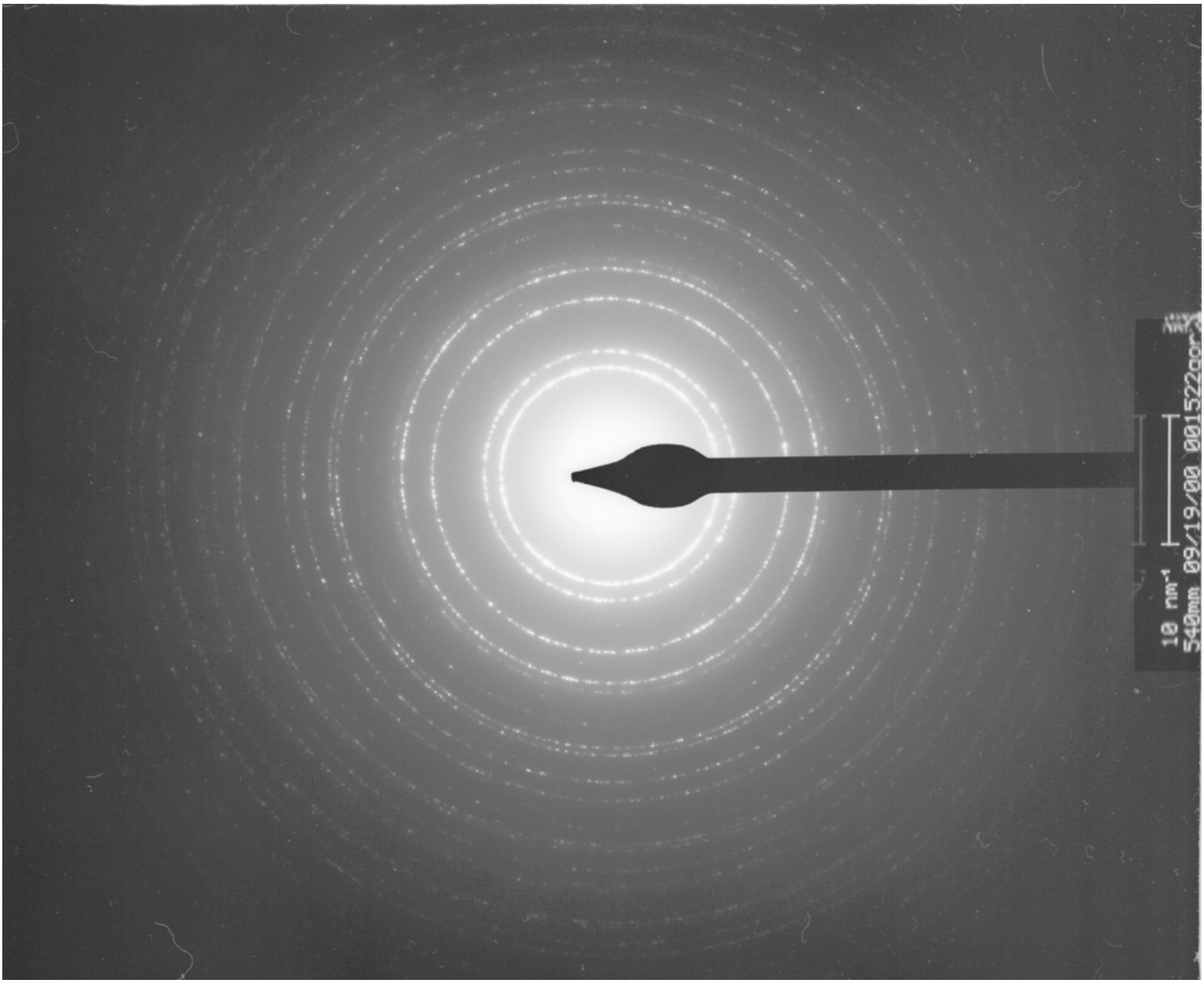
Atoms in different positions in a cell are shared by differing numbers of unit cells

- Vertex atom shared by **8** cells Þ $^{1}/_{8}$ atom per cell
- Edge atom shared by **4** cells Þ $^{1}/_{4}$ atom per cell
- Face atom shared by **2** cells Þ $^{1}/_{2}$ atom per cell
- Body unique to **1** cell Þ **1** atom per cell

FCC Aluminum



Aluminum diffraction rings



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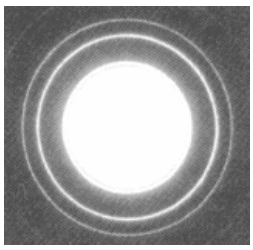

Aluminum Diffraction Pattern

In order to use a standard in electron diffraction properly, several conditions are necessary:

1) After obtaining the pattern of an unknown, it is necessary to expose the known standard to the same electrical and magnetic conditions, specifically the same lens current or high voltage.
(2) The specimen must be in the same position as the unknown had been. Small movements of the stage are permissible in order to obtain a clearer pattern of the standard.

After developing the diffraction plates, the standard is measured first. The indices are assigned per the attached ASTM "d" spacings. After assigning the spacings, calculate "K" using the formula K = Sd; where "K" is a constant that represents wave length of the beam, camera length and associated variable crystallographic data, "S" is the diameter of the ring in centimeters and "d" is the interplanar spacing in angstroms. The values for "K" for the first five lines should be within 1% of each other. Use the mean value of "K".

Next, the pattern of the unknown is measured, and using "K", determined from the standard, the "d" spacings of the unknown are calculated. It is then necessary to establish identity of the unknown from the ASTM published data.

Aluminum: ASTM "d" spacings

Miller Indices hk1	Lattice Spacing d (Å)	Intensity I	Lattice Constant a (Å)
111	2.338	100	4.050
200	2.024	47	4.048
220	1.431	22	4.047
311	1.221	24	4.0489
222	1.1690	7	4.0495
400	1.0124	2	4.0496
331	0.9289	8	4.0490
420	.9055	8	4.0495
422	.8266	8	4.0495

Average unit cell from last five lines.....4.0494

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