PROGRAMME TO DETERMINE THE CRYSTAL STRUCTURE

Data Reduction of Diffractometer Experiment

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Contents

1 Introduction

This is a programm written in order to determine the cystal structure after getting the data from a relavent experiment. Along with the structure, other informations like the lattice constant, atomic radius, denisity can be calculated.

The entire programme is written in Java.

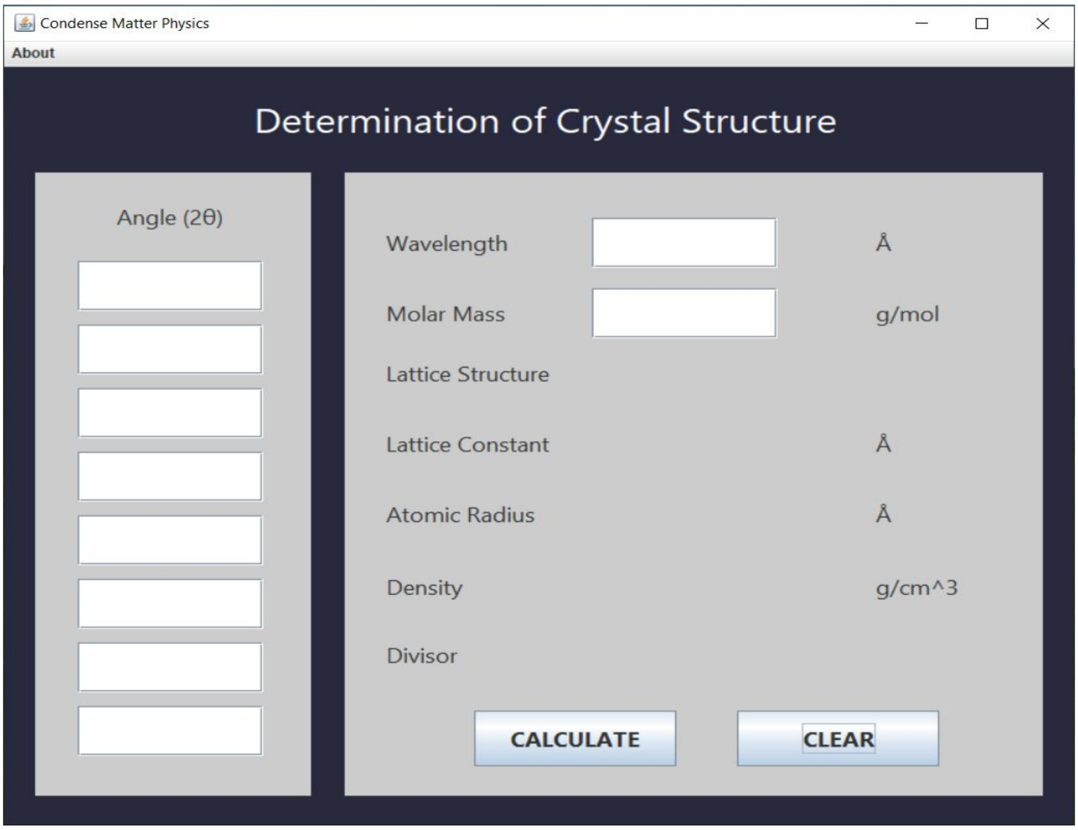

Figure 1: User Interface

2 Interface

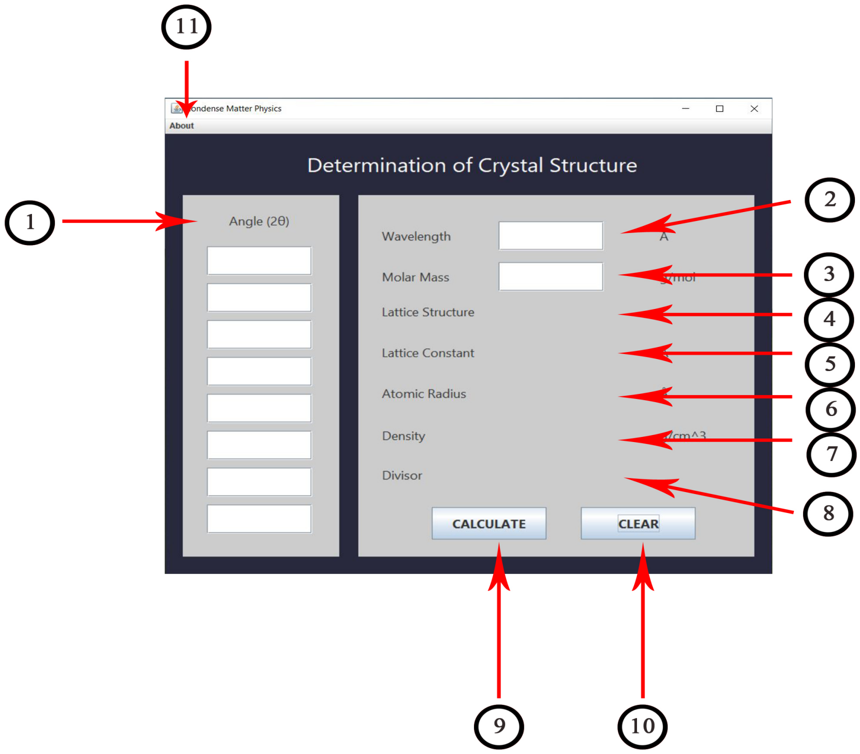

Figure 2: Parts of the interface

1. Textfields to enter the angles.
2. Textfield to enter the value of wavelength used in the experiment.
3. Textfield to enter the molar mass of the crystal.
4. Display the lattice structure.
5. Display the lattice constant.
6. Display the atomic radius.
7. Display the density of the crystal.
8. Display the number which the sine squared value should divide if this is done manually.
9. Carry on the calculations ones clicked.
10. Clear all the textfields and results.
11. Display about the programme.

3 Demonstration

- Case 1:

Consider the angles 38.40, 44.50, 64.85, 77.90, 81.85, 98.40, 111.20 and wavelength 1.5418Å. Molar mass is given 66.6 $gmol^{-1}$ [1]

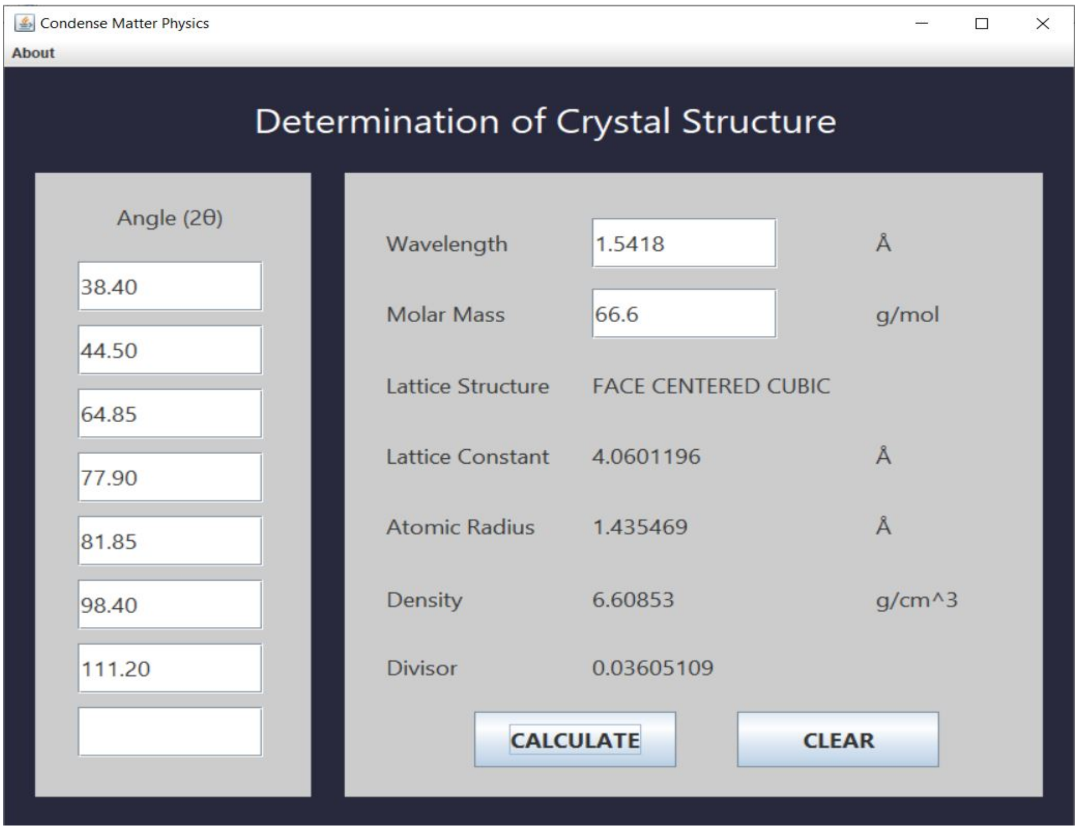

Figure 3: Results for case 1

[1] MIT OCW

Solution

Follow the procedure suggested in lecture:

(1) Start with 2θ values and generate a set of $\sin^2\theta$ values.
(2) Normalize the $\sin^2\theta$ values by generating $\sin^2\theta_n/\sin^2\theta_1$.
(3) Clear fractions from the "normalized" column.
(4) Speculate on the hkl values that would seem as $h^2+k^2+l^2$ to generate the sequence of the "clear fractions" column.
(5) Compute for each θ the value of $\sin^2\theta/(h^2+k^2+l^2)$ on the basis of the assumed hkl values. If each entry in this column is identical, then the entire process is validated.

(a) For the data set in question, it is evident from the hkl column that the crystal structure is FCC (see table below).

(b) $\dfrac{\lambda^2}{4a^2} = \dfrac{\sin^2\theta}{h^2+k^2+l^2} = 0.0358,$

$\lambda_{CuK_\alpha} = 1.5418$ Å, $\therefore$ $a = \dfrac{1.5418}{(4\times 0.0358)^{1/2}} = 4.07$ Å

(c) In FCC, $\sqrt{2}a = 4r$, $\therefore r = \dfrac{\sqrt{2}}{4}\times 4.07$ Å $= 1.44$ Å

(d) Here we'll use atomic mass and atomic volume.

$$\rho = \frac{m}{V};\ \frac{4 \text{ atoms}}{a^3} = \frac{N_A \text{ atoms}}{V_{molar}} \therefore V_{molar} = \frac{6.02\times 10^{23}}{4}\times(4.07\times 10^{-8}\text{ cm})^3 = 10.15\text{ cm}^3$$

$$\therefore \rho = \frac{66.6\text{ g/mol}}{10.15\text{ cm}^3/\text{mol}} = 6.56\text{ g/cm}^3$$

Data Reduction of Debye-Scherrer Experiment:

2θ	$\sin^2\theta$	normalized	clear fractions	(hkl)?	$\frac{\sin^2\theta}{h^2+k^2+l^2}$
38.40	0.108	1.00	3	111	0.0360
44.50	0.143	1.32	4	200	0.0358
64.85	0.288	2.67	8	220	0.0359
77.90	0.395	3.66	11	311	0.0358
81.85	0.429	3.97	12	222	0.0358
98.40	0.573	5.31	16	400	0.0358
111.20	0.681	6.31	19	331	0.0358

Figure 4: Manually worked out solutions for case 1

This tally with the results of the programme.

- Case 2:

Consider the angles 14.10, 19.98, 24.57, 28.41, 31.85, 34.98, 37.89, 40.61 and wavelength 0.574Å. [2]

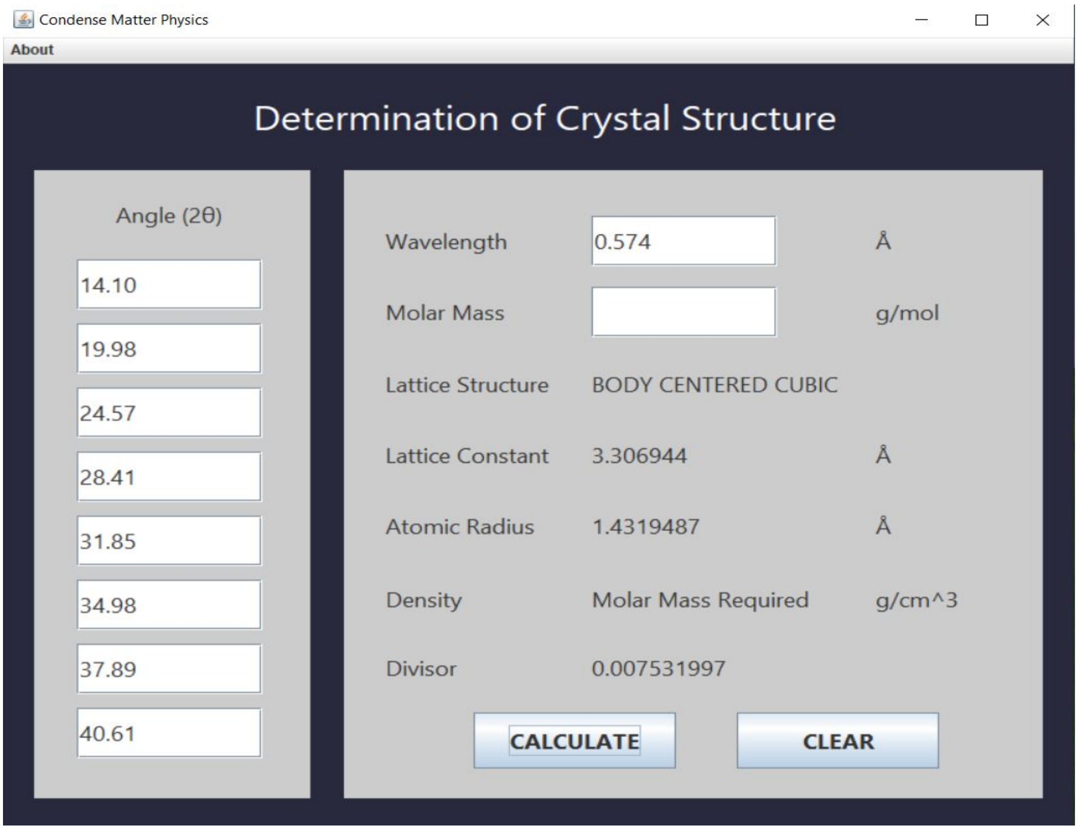

Figure 5: Results for case 2

[2]MIT OCW

Problem #3

The following diffractometer data (expressed as 2θ) were generated from a specimen irradiated with silver (Ag) K_α radiation: 14.10°; 19.98°; 24.57°; 28.41°; 31.85°; 34.98°; 37.89°; 40.61°.

(a) Determine the crystal structure.

(b) Calculate the lattice constant, a.

(c) Assume that the crystal is a pure metal and on the basis of the hard-sphere approximation calculate the atomic radius.

(d) At what angle θ would we find the first reflection if, instead of K_α radiation, we used silver L_α radiation to illuminate the specimen?

Solution

We follow the same approach as described in the answer to Problem 2.

(a) See table below. It is evident that the crystal structure is BCC. Look at the hkl column.

(b) $\dfrac{\lambda^2}{4a^2} = \dfrac{\sin^2\theta}{h^2+k^2+l^2} = 7.53\times10^{-3},\ \lambda_{Ag_{K_\alpha}} = 0.574\ \text{Å}\ \therefore\ a = \dfrac{0.574}{\sqrt{4\times7.53\times10^{-3}}} = 3.31\ \text{Å}$

(c) In BCC, $\sqrt{3}a = 4r\ \therefore\ r = \dfrac{\sqrt{3}}{4}\times3.31\ \text{Å} = 1.43\ \text{Å}$

(d) $\lambda = 2\, d_{hkl}\sin\theta,\ d_{hkl} = \dfrac{a}{\sqrt{h^2+k^2+l^2}} = \dfrac{a}{\sqrt{2}}\ \therefore\ \theta = \sin^{-1}\left(\dfrac{\lambda}{\sqrt{2}a}\right)$

λ_{L_α} given by: $\bar{\nu} = \lambda^{-1} = \frac{5}{36}R(Z-7.4)^2 = \frac{5}{36}\times1.1\times10^7(47-7.4)^2 = 2.40\times10^9\ \text{m}^{-1}$

$\rightarrow \lambda = 4.17\ \text{Å}\ \therefore\ \theta = \sin^{-1}\left(\dfrac{4.17}{\sqrt{2}\times3.31}\right) = 63.0°$

Data Reduction of Diffractometer Experiment: incident x-ray $Ag_{K_\alpha} \rightarrow \lambda = 0.574\ \text{Å}$

2θ	$\sin^2\theta$	normalized	clear fractions	try again	hkl	$10^3 \frac{\sin^2\theta}{h^2+k^2+l^2}$
14.10	0.0151	1.00	1	2	110	7.550
19.98	0.0301	1.99	2	4	200	7.525
24.54	0.0452	2.99	3	6	211	7.533
28.41	0.0602	3.99	4	8	220	7.525
31.85	0.0753	4.99	5	10	310	7.530
34.98	0.0903	5.98	6	12	222	7.525
37.89	0.1054	6.98	7	14	321	7.529
40.61	0.1204	7.97	8	16	400	7.525

Figure 6: Manually worked out solutions for case 2

In this case, the clear fractions have to be multiplied by 2, to eliminate 7. That can be also done by the program and get the correct answer.

4 Other

Programme icon was designed using Adobe Photoshop CS5.1

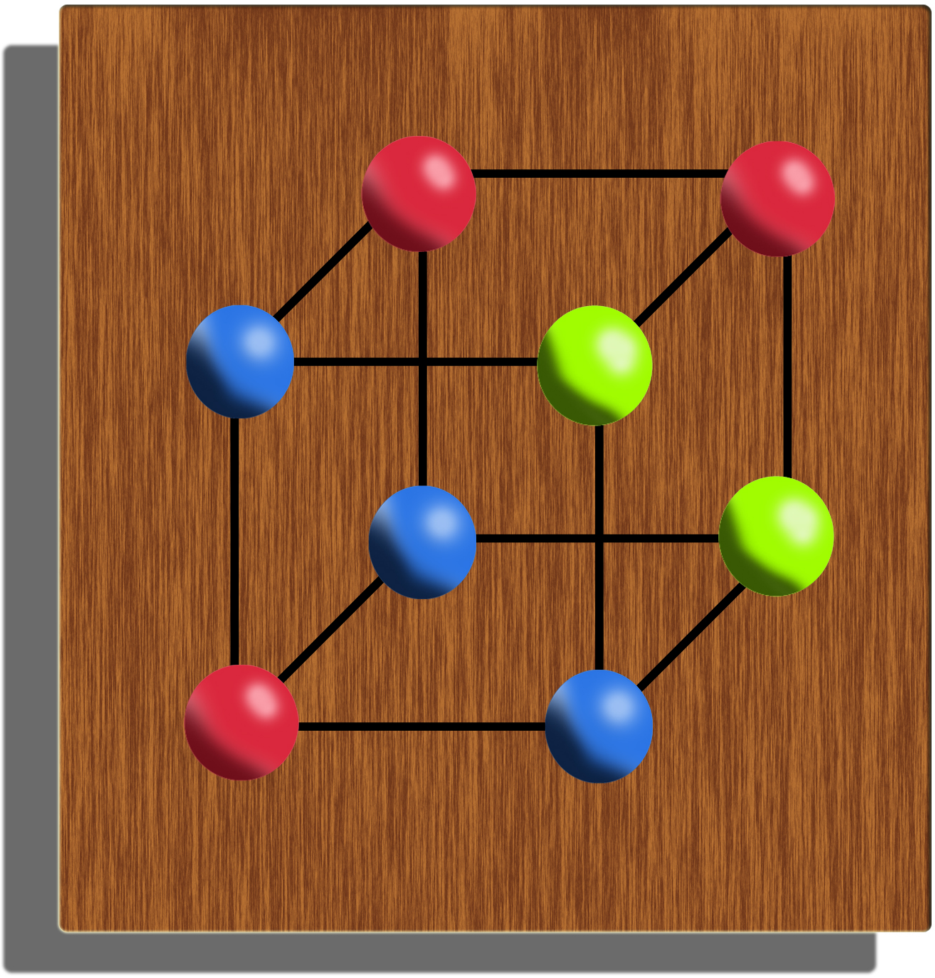

Figure 7: Icon