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Experiment 35 B

Determining the lattice constant for molybdenum using the Debye-Scherrer method

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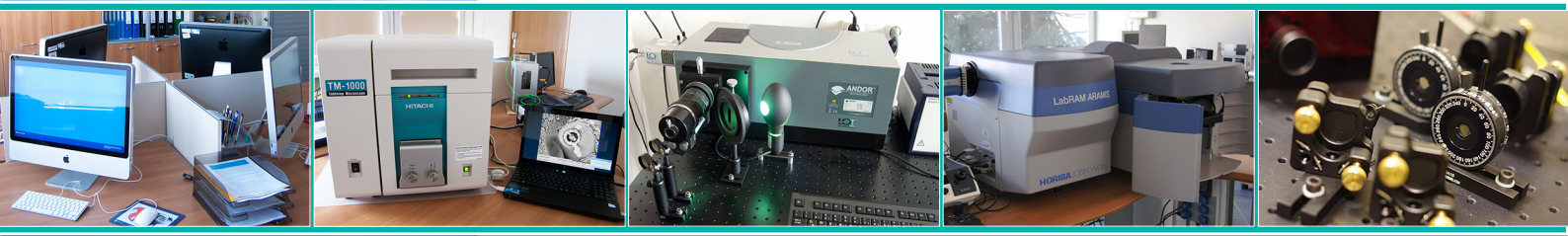

I. Background theory.

1. Production of X-rays.
2. Construction of X-ray tubes.
3. Properties of the X-ray spectrum:
   a) continuous spectrum;
   b) quantum limit;
   c) characteristic spectrum:
      - Moseley's law,
      - absorption edge,
      - atomic energy levels of copper.
4. Fundamentals of crystallography:
   a) point lattice;
   b) crystallographic arrangements;
   c) unit cell;
   d) Bravais lattice;
   e) lattice planes, Miller indices;
   f) crystal structure, monocrystalline solids (for example: Mo);
   g) reciprocal lattice.
5. X-ray diffraction in crystals:
   a) Thomson scattering by electrons, atoms and unit cells;
   b) intensity of the diffracted beam, structure factor;
   c) other factors affecting the intensity of diffraction lines in the powder method (factors: polarization, Lorentz, multiplicity of network planes);
   d) Bragg's law.

II. Experimental tasks.

1. Familiarise yourself with the functioning of the X-ray module shown in *Pictures 1 - 3.*

Picture 1. X-ray module with computer.

2. Measure the intensity of X-ray spectrum as a function of Bragg angle 4ϑ for the polycrystalline Mo sample using a lamp with a Cu anode (see *Appendix A* for instructions).
3. Assign the resulting diffraction lines to their corresponding Miller indices (hkl) for crystallographic planes.
4. Calculate the lattice constant for molybdenum and determine the type of Bravais lattice according to the calculations in *Table 1* in *Appendix B.*
5. Based on formula from equation (3) in *Appendix B,* calculate the number of atoms in the unit cell of molybdenum.

III. Apparatus.

1. X-ray unit with built-in goniometer and replaceable Cu lamp.
2. Computer.

IV. Literature.

1. Ch. Kittel – "*Introduction* to *Solid* State *Physics"*, Wiley & Sons, 2004.
2. G. Burns – "*Solid State Physics"*, Academic Press, Inc. London 1985.
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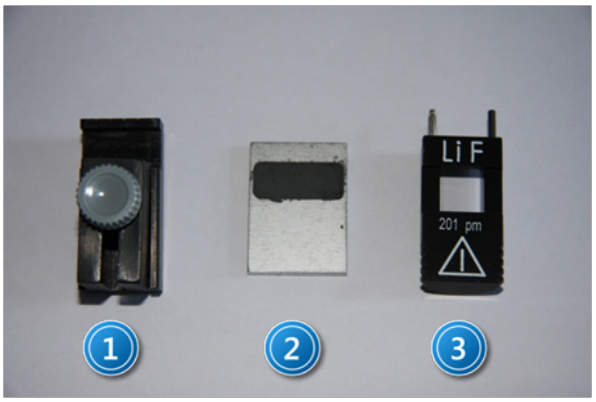
6. H.A. Enge, M.R. Wehr, J.A. Richards – "*Introduction to Atomic Physics*", Wesley, 1981.
7. H. Haken, M.Ch. Wolf – "*The Physics of Atoms and Quanta*", Springer, 2000.
8. V. Acosta, C.L. Cowan, B.J. Graham – "*Essentials of Modern Physics*", New York 1973.
9. D. Halliday, R. Resnick, J. Walker – "*Fundamentals of Physics*", Wiley & Sons, Inc., 2001.
10. Ch. Hammond – "The Basic of Crystallography and Diffraction", Oxford Science Publications, Oxford 2009.

Appendix A

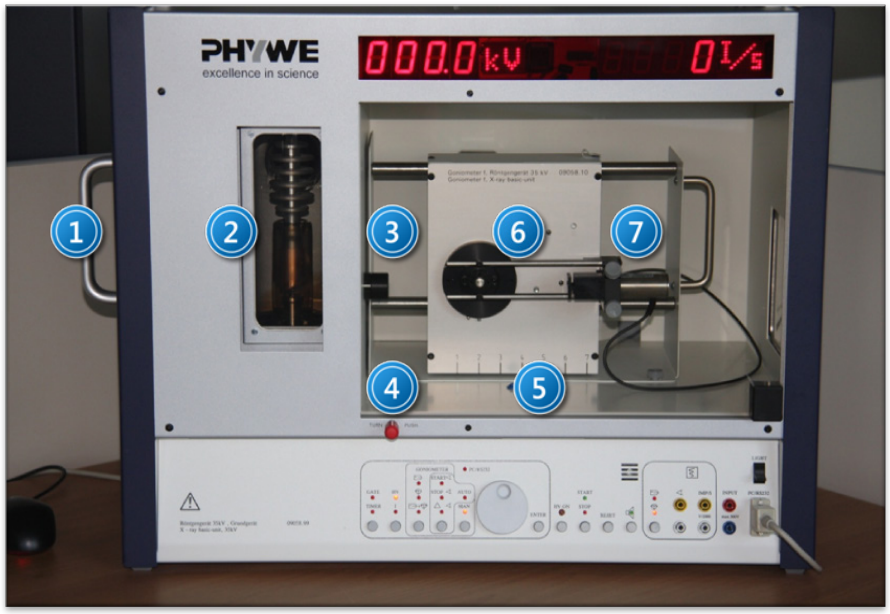
Instructions for the experiment

1. Prepare the sample for measurement.
   Mix an appropriate amount of fine-crystalline molybdenum on a piece of paper with a small amount of Vaseline, to form a uniform mixture. Then place it in the hollow metal plate (2 in *Picture 2*) and level the sample surface with a spatula.
   This is important because surface irregularities have a marked effect on line intensity – uneven surfaces will result in the intensity of low angle reflections being too small.



Picture 2. Additional X-ray equipment: 1 – sample holder; 2 – polycrystalline sample holder; 3 – LiF crystal analyser.

2. Place the plate with the sample in the holder 1 in *Picture 2* and then mount it on the goniometer (6 in *Picture 3*).



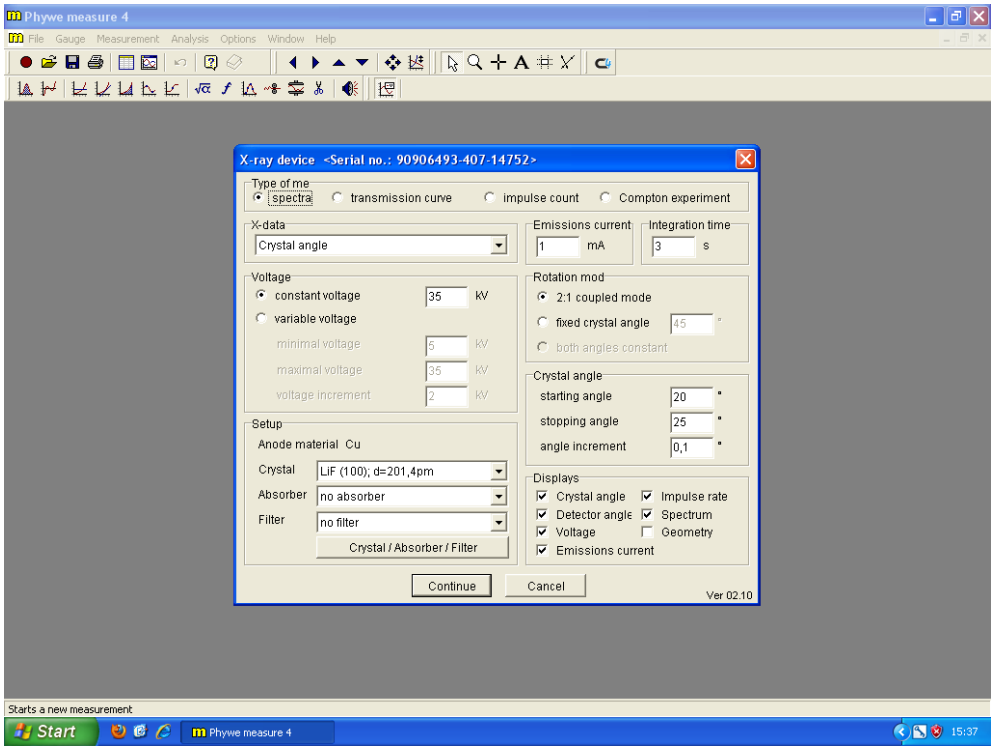
Picture 3. X-ray module: 1 – module with anode; 2 – radiation source; 3 – aperture; 4 – sliding cover locking knob; 5 – goniometer scale; 6 – goniometer; 7 – ionisation detector.

3. Place the aperture with a diameter of Φ = 2mm on the X-ray output with a Ni filter.
4. Set the goniometer to position 4,5 (see *Picture 3*).
5. Turn on the X-ray unit by switching power switch on the back. The display will show the symbol Cu for a few seconds corresponding to the installed anode.
6. Close and lock the sliding glass door in the measuring chamber. To do this, push the red locking knob in and turn it a quarter turn to the left.

ATTENTION!

Operation of the instrument is only possible with the door locked.

7. Turn on your computer and double-click the yellow icon **M** – the shortcut to **Measure.** This will launch the main program window.
8. In order to perform the measurements, select **File** and then **New measurement.** Doing so will open the settings window for the measurement.



Picture 4. Measurement settings window.

9. Using *Picture 4,* set the correct measurement parameters:
   - counting time (*integration time*) – 30 s ;
   - step (*angle increment*) – 0,1° ;
   - scanning range 15° - 50° ;
   - anode voltage $U_A = 35$ *kV*; anode current $I_A = 1$ *mA.*
10. Press **Continue** to accept all the settings and go to the measurement window.
11. Click **START**.

Appendix B

Formulae and other information necessary for the experiment

Bragg formula

$$2dsin\vartheta = n\lambda. \qquad (1)$$

Interplanar distance d_{hkl} for a regular lattice

$$\frac{1}{d_{hkl}^2} = \frac{1}{a^2}(h^2 + k^2 + l^2)\,. \qquad (2)$$

Density ρ

$$\rho = \frac{M}{V} = n \cdot m \cdot \frac{1}{a^3}\,, \qquad (3)$$

where : $m = \frac{m_A}{N}$ $\rightarrow$, $n = \frac{\rho\, N\, a^3}{m_A}$;

n - number of atoms in the unit cell;
m – mass of 1 atom of Mo ;
m_A – atomic weight for Mo : m_A = 10,2 g;
N - Avogadro's number N = 6,022·10^{23} atoms/mol;
a - lattice constant for crystalline Mo;
ρ - density of molybdenum : *ρ* = 10,2 g/cm^3.

Wavelength components K_β and K_α for **Cu anode** : λ (K*β*) = 139,22 pm ; λ (K*α*) = 154,18 pm.

Table 1. Example table to perform the calculations.

Line nr.	$\vartheta°$	$sin\,\vartheta$	$sin^2\vartheta$	$s=h^2+k^2+l^2$	$sin^2\vartheta/s$	a[pm]	hkl	Δa[pm]

The energy level diagram for copper is shown in *Figure 5*.

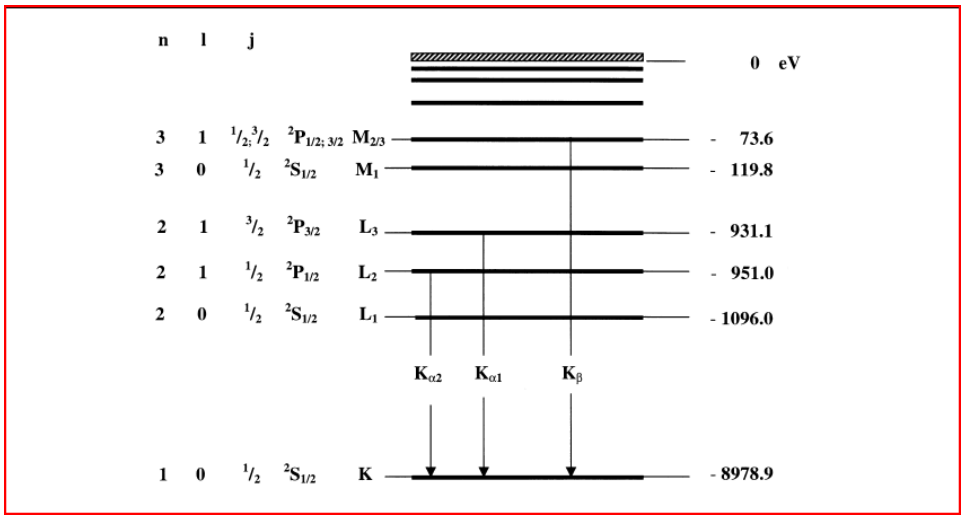


Figure 5. Copper energy level diagram (Z = 29).