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Experiment 33

X-ray diffraction study of copper (Cu) and molybdenum (Mo)

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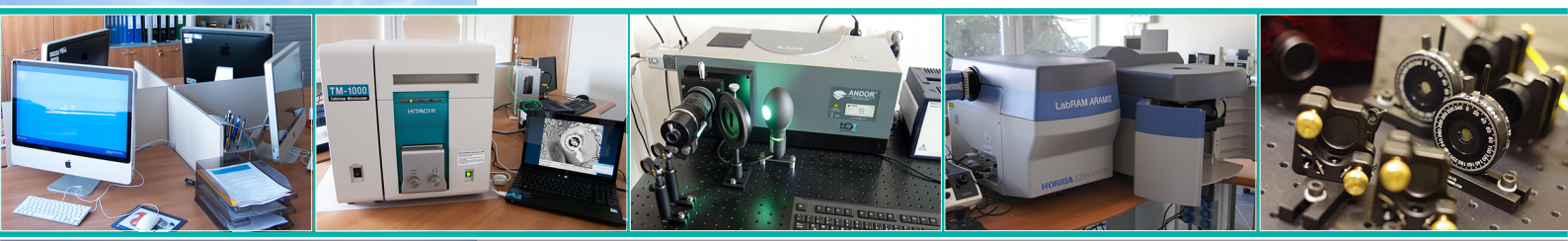


I. Background theory.

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,
      - energy levels of copper and molybdenum..
4. Fundamentals of crystallography:
   a) point lattice;
   b) crystallographic arrangements;
   c) unit cell;
   d) lattice planes, Miller indices;
   e) crystal structure, monocrystalline solids (for example: LiF and KBr);
   f) 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) 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-rays as a function of Bragg angle $\measuredangle\vartheta$ For a lamp with a Cu (or Mo) anode for the given angles ϑ (see *Appendix A* for instructions) for both monocrystalline analysers: LiF and KBr.
3. Based on the relationship I(ϑ), determine the location of the lines K_α and K_β for all orders of diffraction for a Cu (or Mo) anode for both crystal analysers: LiF and KBr.
4. Use Equation (1) in *Appendix B* to calculate the energies of the lines K_α and K_β. Calculate their average values and include uncertainties.
5. Compare the experimental results obtained with the values calculated from *Figures 5* and *6.*

III. Apparatus.

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

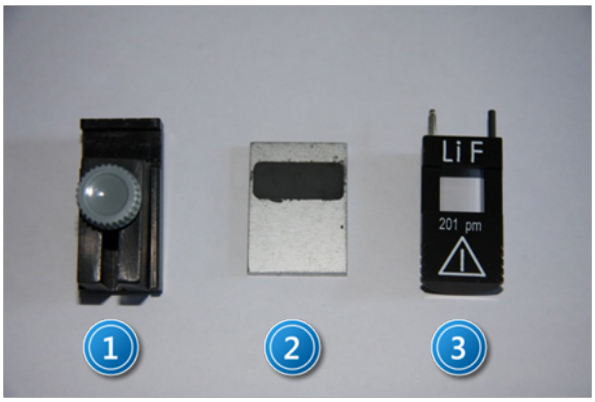
IV. Literature.

1. Ch. Kittel – "*Introduction* to *Solid* State *Physics*", Wiley 2004.
2. G. Burns – "*Solid State Physics*", Academic Press, Inc. London 1985.
3. R. Steadman – "*Crystallography*", Van Nostrand Reinhold (UK) Co.Ltd., 1982.
4. M. F. Ladd, R.A. Palmer – "*Structure Determination by X-ray Crystallography*", Plenum Press. New York and London 1985.
5. K. Hermbecker - Handbook "*Physics X-ray Experiments*", PHYWE-Series of Publication, 2010.
6. H. Haken, M.Ch. Wolf – "*The Physics of Atoms and Quanta*", Springer, Berlin, Heidelberg 2000.
7. V. Acosta, C.L. Cowan, B.J. Graham – "*Essentials of Modern Physics*", Harper & Row, Publishers, New York 1973.
8. D. Halliday, R. Resnick, J. Walker – "*Fundamentals of Physics*", Wiley & Sons, Inc., 2001.
9. Ch. Hammond – *"The Basic of Crystallography and Diffraction",* Oxford Science Publications, Oxford 2009.

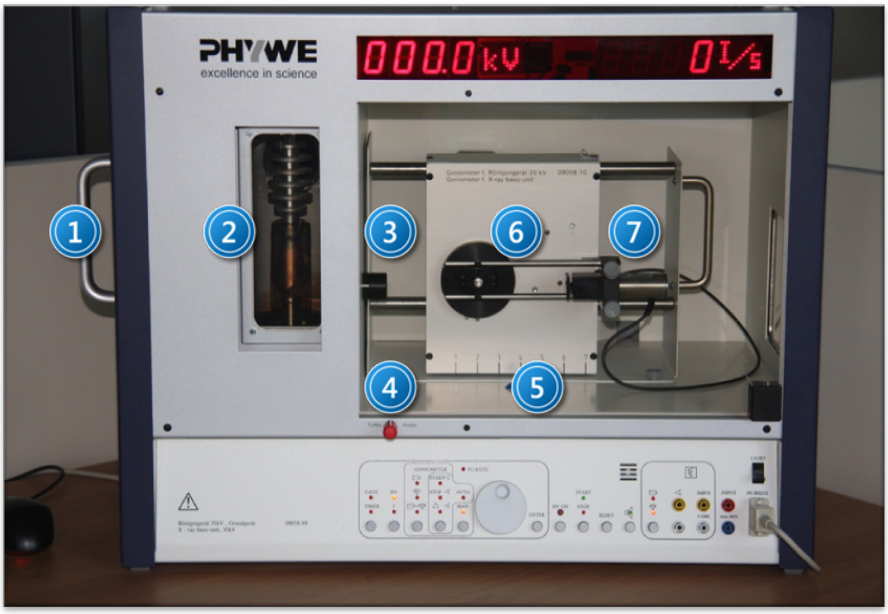
Appendix A

Instructions for the experiment

1. Use *Pictures 2* and *3* mount a crystal –analyser on the goniometer: LiF or KBr.



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



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

2. Place the appropriate aperture on the X-ray output:

   a. **for Cu anode**:
   for LiF – diameter 1 mm ,
   for KBr – diameter 2 mm ;

   b. **for Mo anode**:
   for both crystal analysers – diameter – 2 mm.

3. Set the optimum location of the goniometer (graduations 1 – 7, *Picture 3*).
   The following are recommended settings:

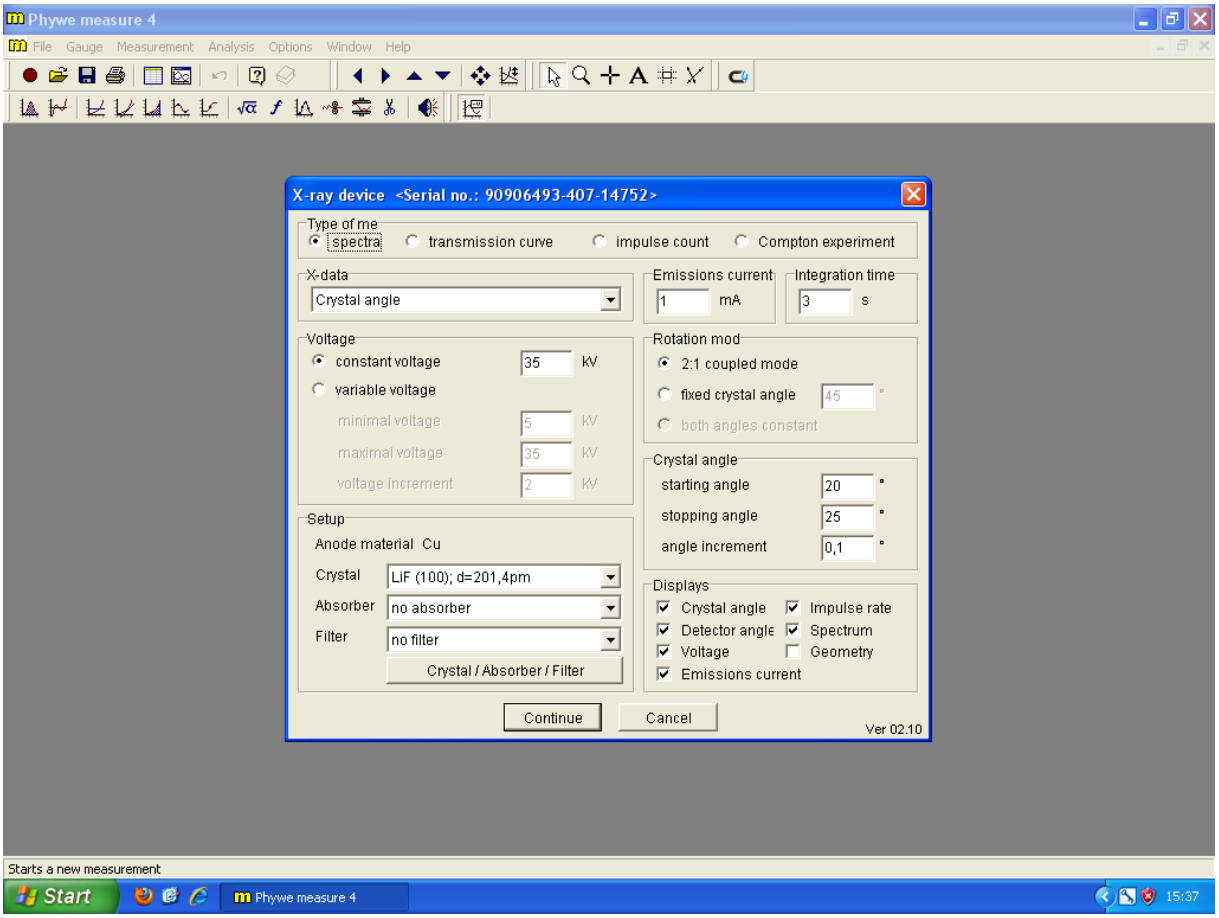
cathode	analyser	position
Cu	KBr	1
Cu	LiF	4
Mo	KBr	7
Mo	LiF	7

4. Turn on the X-ray unit by switching power switch on the back.
   The display will show the symbol Cu or Mo for a few seconds depending on the lamp used.
5. 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.

6. Turn on your computer and double-click the yellow icon **M** – the shortcut to **Measure.** This will launch the main program window.
7. In order to perform the measurements, select **File** and then **New measurement.** Doing so will open the settings window for the measurement (*Picture 4*).



Picture 4. Measurement settings window.

8. Adjust the X-ray measurement settings. The following are the recommended settings:

for Cu anode:

- counting time (*integration time*) – 2 s ,
- step (*angle increment*) – 0,1 ^{o},
- scan range for LiF: 3 ^{o}- 55 ^{o}, for KBr: 3 ^{o} - 75 ^{o},
- anode voltage $U_A = 35\ kV$, anode current $I_A = 1\ mA$.

for Mo anode:

- counting time – 2 s ,
- step – 0,1 ^{o},
- scan range for LiF: 4 ^{o} - 65 ^{o}, for KBr: 3 ^{o}- 30 ^{o},
- anode voltage U_A = 35 kV , anode current I_A = 1 mA.

9. Press **Continue** to accept all the settings and go to the measurement window.
10. Click **START**.

Appendix B

Formulae and other information necessary for the experiment.

Transformed Bragg formula:

$$E(\vartheta) = (n \cdot h \cdot c)/(2 \cdot d \cdot \sin\vartheta) \quad , \quad (1)$$

where:

d- interplanar distance:	for LiF (200)	$d = 2{,}014 \cdot 10^{-10}$ m
	for KBr (200)	$d = 3{,}290 \cdot 10^{-10}$ m
h – Planck constant;		$h = 6{,}6256 \cdot 10^{-34}$ Js
c – speed of light;		$c = 2{,}9979 \cdot 10^{8}$ m/s
ϑ - Bragg angle.		

Wavelength for component Kβ - **anode Cu**	$\lambda(K_\beta)$ = 139,22 pm
Wavelength for component Kα - **anode Cu**	$\lambda(K_\alpha)$ = 154,18 pm
Wavelength for component Kβ - **anode Mo**	$\lambda(K_\beta)$ = 63,26 pm
Wavelength for component Kα - **anode Mo**	$\lambda(K_\alpha)$ = 71,14 pm

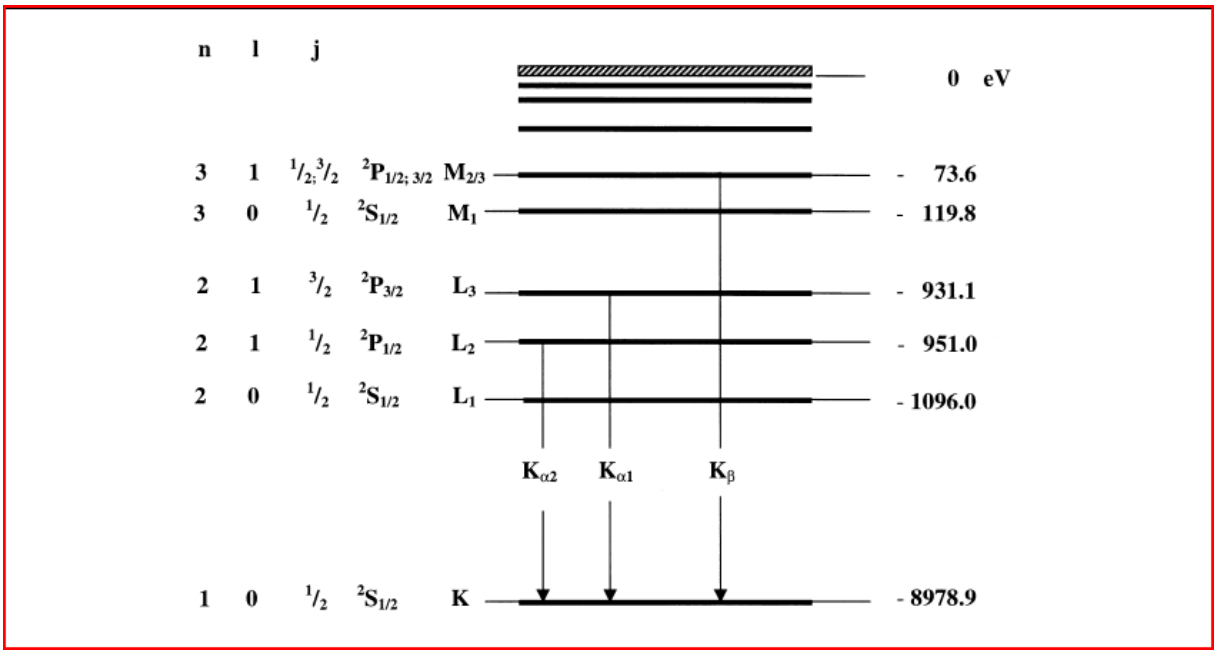


Figure 5. Energy level diagram for copper (Z = 29).

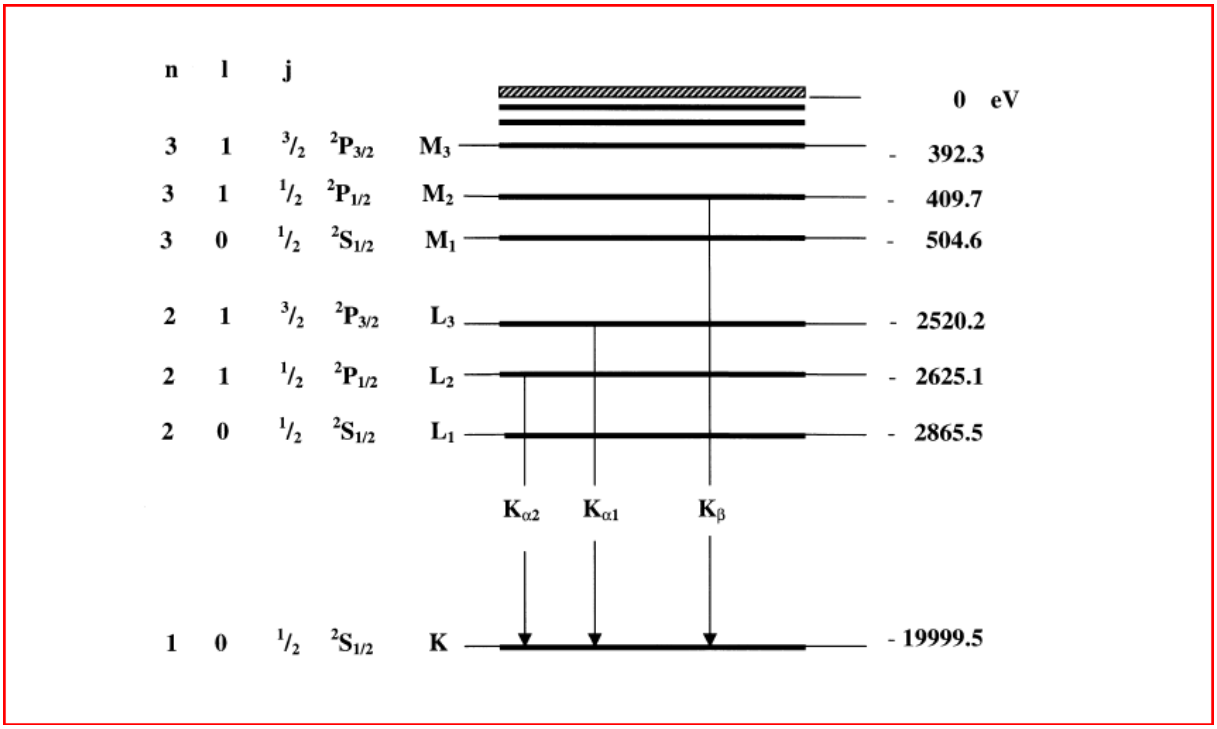


Figure 6. Energy level diagram for molybdenum (Z = 42).