

Qualitative X-ray fluorescence analysis of alloyed materials



Physics

Modern Physics

Production & use of X-rays



Difficulty level

hard



Group size

2



Preparation time

45+ minutes



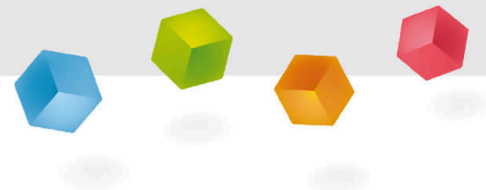
Execution time

45+ minutes

This content can also be found online at:

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General information

Application

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Setup

Most applications of X rays are based on their ability to pass through matter. Since this ability is dependent on the density of the matter, imaging of the interior of objects and even people becomes possible. This has wide usage in fields such as medicine or security.

Other information (1/2)

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**Prior****knowledge****Main****principle**

The prior knowledge required for this experiment is found in the Theory section.

The composition of various alloys is analysed with the aid of polychromatic X-rays. The energy of the characteristic fluorescence lines of the alloy constituents is analysed with the aid of a semiconductor detector and a multichannel analyser. The alloy constituents are identified by comparing the line energies with the corresponding table values.

Other information (2/2)

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**Learning
objective****Tasks**

The goal of this experiment is to get to investigate the spectra of fluorescence radiation.

1. Calibrate the semiconductor energy detector with the aid of the characteristic radiation of the molybdenum X-ray tube.
2. Record the spectra of the fluorescence radiation that is generated by the samples.
3. Determine the energy values of the corresponding fluorescence lines and identify the alloy constituents with the aid of table values.

Theory

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When X-rays interact with matter, they lose energy due to Compton scattering, pair production, and photoelectric effects. In the range of energy that is available during this experiment, the photoelectric effect plays the most important role. This means that in the atom on one of the lower shells an electron is ejected due to the absorbed photon energy.

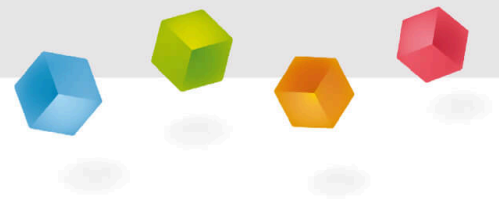
The now free space is taken by an electron from one of the higher shells. The energy that is produced during this process can be used for the ejection of another electron from one of the higher shells (Auger effect) or for the generation of a photon (fluorescence radiation). Since the energy of the energy levels that are involved in this process is atom-specific, the type of the emitting atom can be determined based on the energy of the fluorescence radiation. In order to determine the type of atom, the experimental energy values are then compared to the corresponding table values (e.g. "Handbook of Chemistry and Physics"-CRC-Press, Inc. USA). During the assignment of the fluorescence lines, it must be taken into consideration that the relaxations that follow the primary ionisation process can only take place if they fulfil the quantum-mechanical selection rules $\Delta j = 0, \pm 1$ and $\Delta l = \pm 1$ (j = total angular momentum, l = orbital angular momentum).

Equipment

Position	Material	Item No.	Quantity
1	XR 4.0 expert unit, 35 kV	09057-99	1
2	XR 4.0 X-ray goniometer	09057-10	1
3	XR4 X-ray Plug-in Cu tube	09057-51	1
4	XR 4.0 X-ray material upgrade set	09165-88	1

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Setup and Procedure



Setup (1/2)

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- Screw the adapter ring onto the inlet tube of the energy detector and connect the signal and supply cables to the corresponding ports of the detector with the aid of the right-angle plugs.
- Connect the signal and supply cables to the corresponding ports in the experiment chamber of the X-ray unit. In Figure 1, the port for the signal cable is marked in red and the port for the supply cable is marked in green. Connect the external X RED ports of the x-ray unit (see Fig. 2) to the multi-channel analyser (MCA). Connect the signal cable to the "Input" port and the supply cable to the "X-Ray Energy Det." port of the MCA.

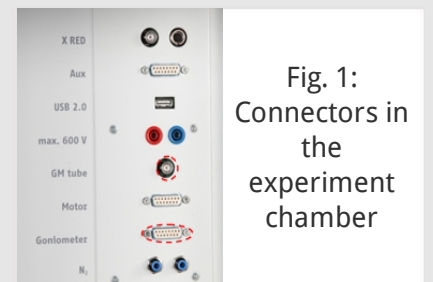


Fig. 1:
Connectors in
the
experiment
chamber



Fig. 2: Connection of the
multi-channel analyser

Setup (2/2)

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- Secure the energy detector in the holder of the swivel arm of the goniometer. Lay the two cables with sufficient length so that the goniometer can be swivelled freely over the entire range.
- Connect the multi-channel analyser and computer with the aid of the USB cable.

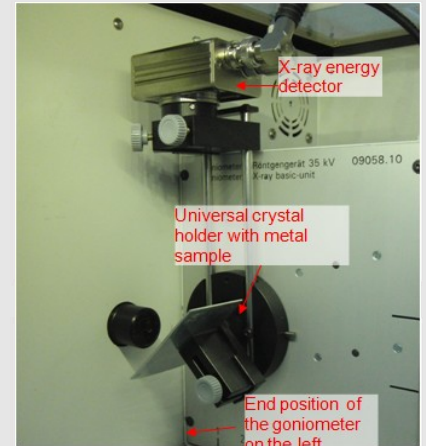


Fig. 3: Goniometer set-up

Procedure (1/4)

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- Bring the goniometer block and the detector to their respective end positions on the right.
- Insert the tube with the 1mm-aperture into the exit tube of the X-ray tube.
- With the X-ray unit switched on and the door locked, bring the detector to the 0° position. Then, shift the detector by some tenths degree out of the zero position in order to reduce the total rate.
- Operating data of the tungsten X-ray tube: Select an anode voltage $U_A = 25$ kV and an anode current $I_A = 0.02$ mA and confirm these values by pressing the "Enter" button.
- Switch on the X-radiation

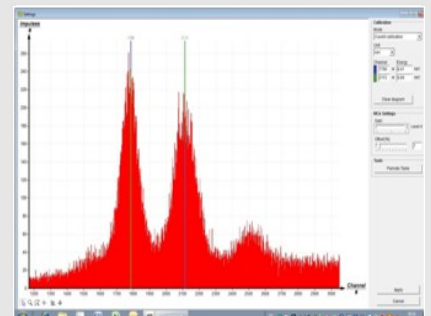


Fig. 4: calibration of the multi-channel analyser

Procedure (2/4)

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- In the MEASURE program, select "Multi channel analyser" under "Gauge". Then, select "Settings and calibration". After the "Calibrate" button has been clicked, a spectrum can be measured. The counting rate should be < 300 c/s. Energy calibration settings: - 2-point calibration, - Unit = keV, Gain = 2 – Set the offset so that low-energy noise signals will be suppressed (usually a few per cent are sufficient), See Fig 4.
- Measuring time: 5 minutes. Use the timer of the X-ray unit.
- Make the two coloured calibration lines congruent with the line centres of the two characteristic X-ray lines. The corresponding energy values (see e.g. P2544705) $E(L_3M_5/L_3M_4) = 8,41\text{keV}$ and $E(L_2N_4) = 9,69\text{keV}$ are entered into the corresponding fields, depending on the colour. (Note: Since a separation of the lines L_3M_4 and L_3M_5 Lines is not possible, the mean value of both lines is entered as the energy of the line).
- Name and save the calibration.

Procedure (3/4)

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

Spectrum recording

- Insert the tube with the 2-mm-aperture.
- Bring the goniometer block and the detector to their respective end positions on the left. Bring the detector to the 90° position in the 1:2 coupling mode.
- Insert the metal sample with the universal crystal holder (sample at 45°).
- Operating data of the molybdenum X-ray tube: Adjust an anode voltage $U_A = 35\text{ kV}$ and an anode current so that the counting rate is ≤ 300 c/s.
- Measuring time: 3 minutes (use the timer of the X-ray unit).

Procedure (4/4)

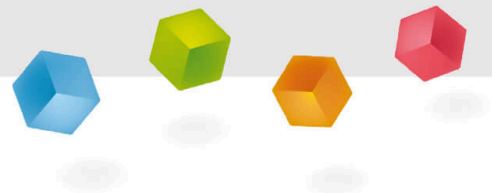
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Evaluation of the measurement curves

- In order to determine the line energy, switch from the bar display to the curve display. To do so, click "Display options" and then "Interpolation and straight lines".
- Extend the relevant line section with the aid of the zoom function .
- Then, select the curve section with  Open the window "Function fitting"  Then, select "Scaled normal distribution" and confirm.
- Find the line centroid of the normal distribution with "Peak analysis"  or determine it with the function "Survey" .

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Evaluation



Evaluation (1/7)

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Evaluation of the fluorescence spectra of two different magnetic materials

Figure 5 and 6 show the fluorescence spectra of two different magnetic materials. Table 1 shows the corresponding evaluation.

NdFeB magnet: The element boron with a K_{α} -line of an energy level of ~ 0.2 keV cannot be detected by the energy detector. Line 4, which is located at the high-energy side of the Fe- K_{α} -line, includes the two lines Fe- K_{β} and Nd- L_{γ} that cannot be separated under the present conditions. Table 1 shows the evaluation of the lines.

Fig. 5: Fluorescence spectrum of an NdFeB magnet

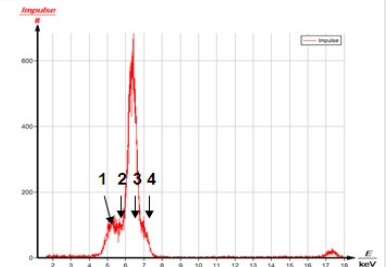
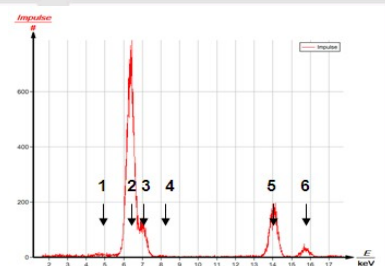


Fig. 6: Fluorescence spectrum of magnetic film



Evaluation (2/7)

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Magnetic film: Table 2 shows the evaluation of the lines. The magnetic component of the adhesive film consists of a strontium-ferrite powder that is bound in a thermoplastic substrate. The low-intensity line 1 can be assigned to caesium, although the K_{α} transition ($\text{Cs-E}_K \sim 36$ keV) cannot be stimulated under the present conditions.

Line	E_{exp} [keV]	E_{it} [keV]	Transition	Element
1	~ 5.29	5.21-5.23	L_{α}	Nd
2	5.66	5.72	L_{β}	Nd
3	6.36	6.40	K_{α}	Fe
4	7.01	7.06	K_{β}	Fe
		7.11	L_{γ}	Nd

Table 1:
NdFeB
magnet

Line	E_{exp} [keV]	E_{it} [keV]	Transition	Element
1	~ 4.75	4.72-4.78	L_{β}	Cs
2	6.36	6.40	K_{α}	Fe
3	7.01	7.06	K_{β}	Fe
4	8.02	8.04	K_{α}	Cu
5	14.06	14.16	K_{α}	Se
6	15.74	15.83	K_{β}	Se

Table 2: Magnetic film (curve b)

Evaluation (3/7)

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Comparison of the fluorescence spectra of iron and steel

Figure 7 shows the spectrum of iron and Figure 8 shows the spectrum of alloyed steel for comparison. Table 3 shows the evaluation.

Line	E_{exp} [keV]	E_{it} [keV]	Transition	Element
1	6.36	6.40	K_{α}	Fe
2	7.01	7.06	K_{β}	Fe

Table 3: Iron

Fig. 7: Fluorescence spectrum of iron

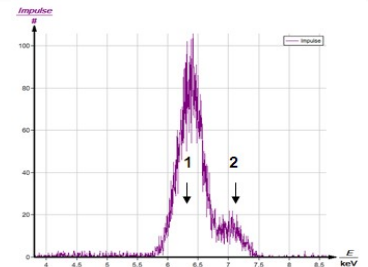
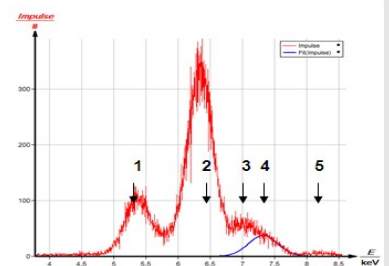


Fig. 8: Fluorescence spectrum of alloyed steel



Evaluation (4/7)

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Iron: There are only the K_{α} and K_{β} lines of iron. Table 3 shows the evaluation of the lines.

Steel (X5CrNi18-10): Table 4 shows the evaluation of the lines. In order to make line 4 clearer, it is highlighted by a fitted normal distribution. At $E = 5.95$ keV, the Cr- K_{β} line can just be seen in the original curve. The fluorescence lines of the spectrum are all part of the same $K_{\alpha,\beta}$ transition of elements that are contiguous in the periodic table of elements. Since, as a result of this, a comparable fluorescent yield can be assumed, the line maxima roughly indicate the corresponding constituents of the alloy. The alloy includes twice as much chromium as nickel.

Line	E_{exp} [keV]	E_{it} [keV]	Transition	Element
1	5.41	5.41	K_{α}	Cr
2	6.36	6.40	K_{α}	Fe
3	7.01	7.06	K_{β}	Fe
4	7.41	7.46	K_{α}	Ni
5	8.25	8.26	K_{β}	Ni

Table 4: Steel

Evaluation (5/7)

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Fluorescence spectrum of soldering tin for electronic applications

Figure 9 shows the fluorescence spectra of soldering tin (information provided by the manufacturer: $\text{Sn}_{60}\text{Pb}_{38}\text{Cu}_2$). Table 5 shows the evaluation of the lines. Tin, which amounts to 60% of the alloy, provides only low-intensity K lines (10 and 11), since the energy of the exciting radiation is only slightly higher than the energy of the Sn-K level ($E_K \sim 29$ keV). The intensity of the soft L radiation (line 1) of the tin is partly reduced by absorption in the sample (self-absorption) and by absorption in the air. The iron line (2) may originate from the material of the detector housing, while the molybdenum lines (8 and 9) are caused by the primary radiation that is scattered at the sample.

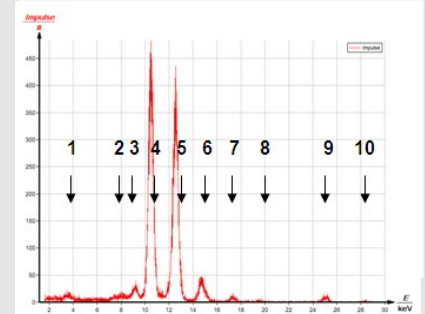


Fig. 9: Fluorescence spectrum of soldering tin

Evaluation (6/7)

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Line	E_{exp} [keV]	E_{it} [keV]	Transition	Element
1	3.57	3.66	L_{β_1}	Sn
2	8.01	8.04	K_{α}	Cu
3	9.16	9.18	L_I	Pb
4	10.48	10.44/10.55	$L_{\alpha_{1,2}}$	Pb
5	12.54	12.61	L_{β_1}	Pb
6	14.73	14.76	L_{γ_1}	Pb
7	17.34	17.48	K_{α}	Mo
8	19.54	19.61	K_{β}	Mo
9	25.10	25.27	K_{α}	Sn
10	28.48	28.48	K_{β}	Sn

Table 5: Elements of the soldering tin

Evaluation (7/7)

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Fluorescence spectrum of a superconductor (YBaCu-O):

Figure 10 shows the fluorescence spectrum of a superconductor. Line 1 includes numerous L transitions of the element barium. $K_{\alpha,\beta}$ transitions cannot be produced, since the energy of the Ba-K level ($E_K \sim 37.5$ keV) is higher than the primary energy that is available during the experiment. The line pairs 2,3 and 4,5 are caused by copper and yttrium.

Line	E_{exp} [keV]	E_{it} [keV]	Transition	Element
1	5.41	5.41	K_{α}	Cr
2	6.36	6.40	K_{α}	Fe
3	7.01	7.06	K_{β}	Fe
4	7.41	7.46	K_{α}	Ni
5	8.25	8.26	K_{β}	Ni

Table 6:
Elements of the
superconductor

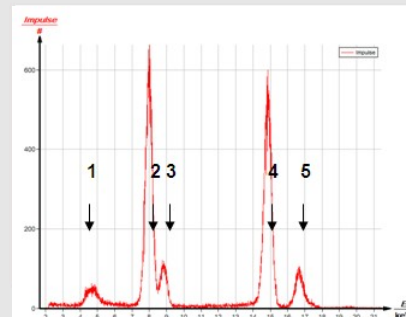


Fig. 10: Fluorescence
spectrum of a
superconductor (YBaCu-O)