Qualitative X-ray fluorescence analysis of alloyed materials (Item No.: P2544601)

Curricular Relevance

| Area of Expertise: ILIAS | Education Level: Physik | Topic: Hochschule | Subtopic: Moderne Physik | Experiment: Röntgenphysik |
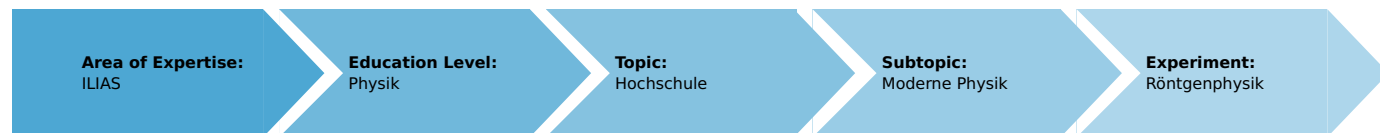
Difficulty	Preparation Time	Execution Time	Recommended Group Size
Difficult	1 Hour	2 Hours	2 Students

Additional Requirements:

- PC

Experiment Variations:

Keywords:

Bremsstrahlung, characteristic X-radiation, energy levels, fluorescent yield, semiconductor energy detectors, multichannel analysers

Overview

Short description

Principle

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.

Fig. 1: P2544601

This experiment is included in the upgrade package: XRM 4.0 X-ray material analysis.

Equipment

Position No.	Material	Order No.	Quantity
1	XR 4.0 expert unit, X-ray unit, 35 kV	09057-99	1
2	XR 4.0 X-ray goniometer	09057-10	1
3	XR 4.0 X-ray plug-in unit W tube	09057-81	1
4	Multichannel analyser	13727-99	1
5	XR 4.0 X-ray energy detector (XRED)	09058-30	1
6	XR 4.0 X-ray Specimen set alloys for fluorescence, set of 5	09058-33	1
7	XR 4.0 X-ray Universal crystal holder for X-ray unit	09058-02	1
8	Crucible tongs, 200 mm, stainless steel	33600-00	1
9	Porcelain dish 25ml, d 50mm	32514-00	1
10	measure Software multi channel analyser	14452-61	1
11	XR 4.0 XRED cable, 2 m	09058-35	1
12	XR 4.0 X-ray Diaphragm tube d = 1 mm	09057-01	1
13	XR 4.0 X-ray Diaphragm tube d = 2 mm	09057-02	1
14	Screened cable, BNC, l = 750 mm	07542-11	1

Tasks

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.

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Robert-Bosch-Breite 10
D - 37079 Göttingen

Tel: +49 551 604 - 0
Fax: +49 551 604 - 107

info@phywe.de
www.phywe.com

Setup and Procedure

Setup

- Screw the adapter ring onto the inlet tube of the energy detector.

- Connect the signal and supply cables to the corresponding ports of the detector with the aid of the right-angle plugs. In Figure 2, the port for the signal cable is marked in red and the port for the supply cable is marked in green.

- Connect the signal and supply cables to the corresponding ports in the experiment chamber of the X-ray unit. Connect the external X RED ports of the X-ray unit (see Fig. 3) 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.

- 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 swivelling range.

- Connect the multi-channel analyser and computer with the aid of the USB cable.

Fig. 2: Connectors in the experiment chamber

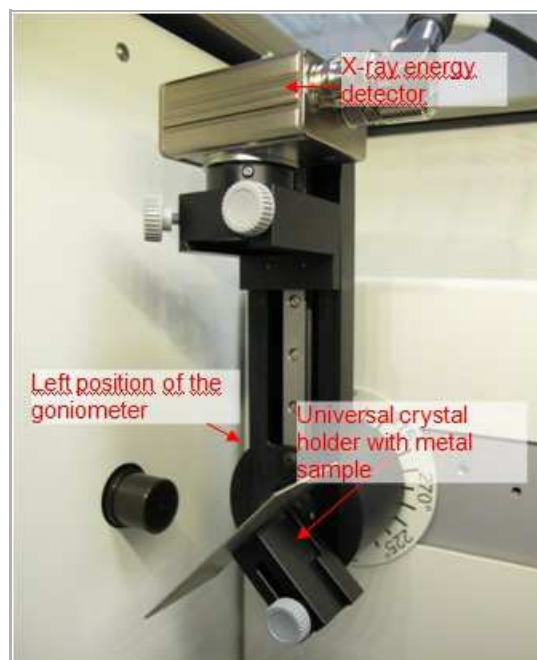Fig. 3: Connection of the multi-channel analyser

Fig. 4: Set-up at the goniometer

Procedure

Calibration of the multi-channel analyser
(if there is no other already existing calibration that can be used)

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D - 37079 Göttingen

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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

- 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 5.

- 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. P2544701) $E(L_3M_5/L_3M_4)$ = 8,41keV and $E(L_2N_4)$ = 9,69 keV are entered into the corresponding fields, depending on the colour. (Note: Since a separation of the lines L_3M_5 and L_3M_4 Lines is not possible, the mean value of both lines is entered as the energy of the line).

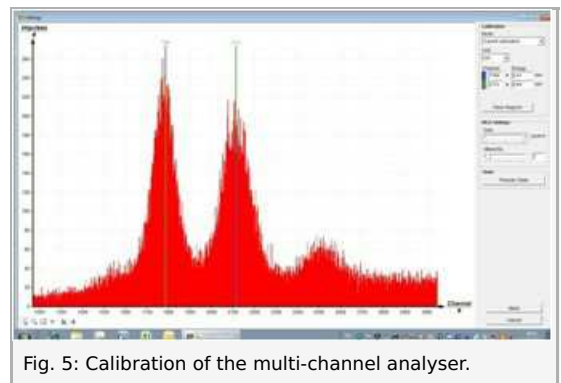- Name and save the calibration.

Fig. 5: Calibration of the multi-channel analyser.

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 2:1 coupling mode.
- Insert the sample with the universal crystal holder (sample at 45°).
- Operating data of the tungsten X-ray tube: Adjust an anode voltage Ua = 35 kV and an anode current so that the counting rate is ≤ 200 c/s.
- Measuring time: 3 minutes (use the timer of the X-ray unit).

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"

Sample preparation

Soldering tin (commercially available soldering tin for electronic applications): Melt a small bead of tin off the tin wire with the aid of a soldering iron and let it drop to the floor. The small "pancake" thus produced can be used as the sample.

Theory and Evaluation

Theory

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).

Evaluation

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.
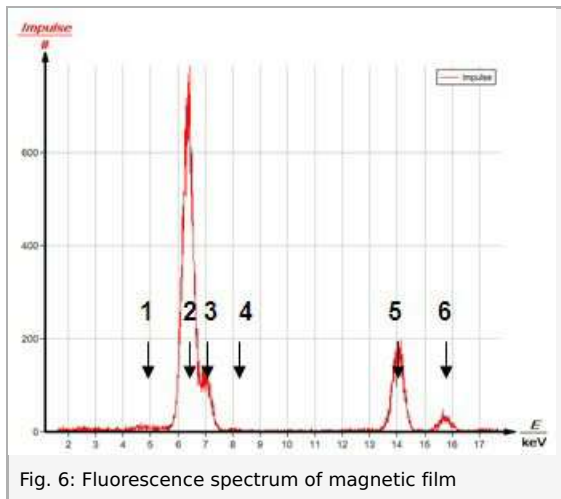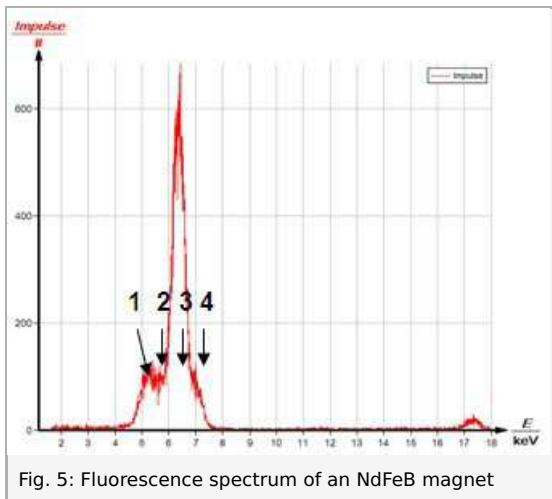

Fig. 5: Fluorescence spectrum of an NdFeB magnet

Fig. 6: Fluorescence spectrum of magnetic film

Table 1: NdFeB magnet

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

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 (Cs-E_K ~36 keV) cannot be stimulated under the present conditions.

Table 2: Magnetic film (curve b)

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	Sr
6	15.74	15.83	K_β	Sr

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.

Fig. 7: Fluorescence spectrum of iron

Fig. 8: Fluorescence spectrum of alloyed steel
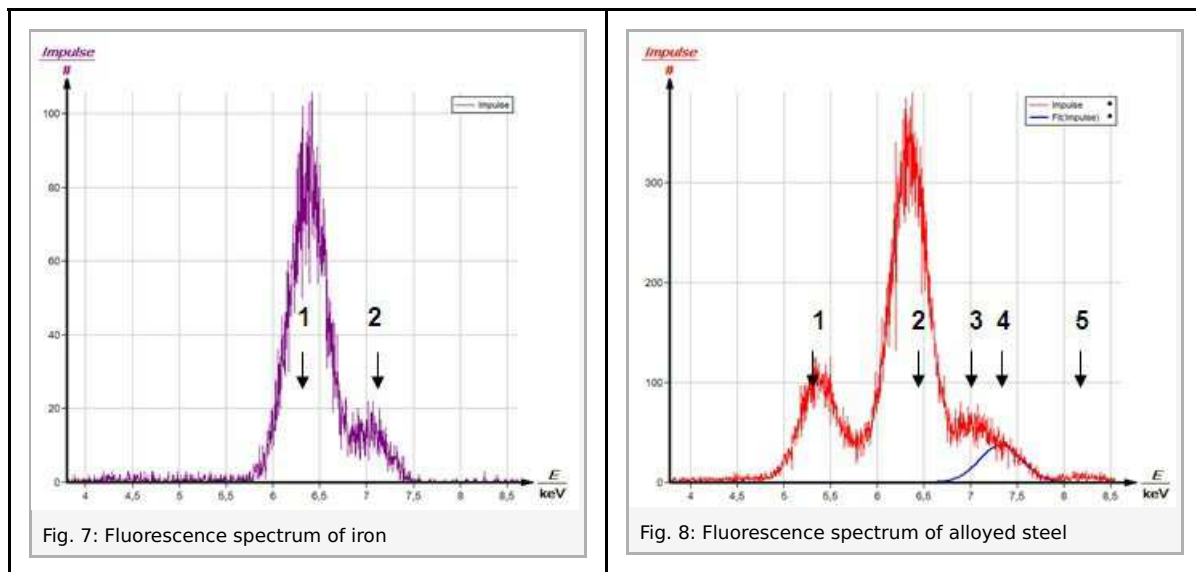
Iron: There are only the Kα and Kβ lines of iron. Table 3 shows the evaluation of the lines.

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.

Table 3: Iron

1	6.36	6.40	K_α	Fe
2	7.01	7.06	K_β	Fe

Table 4: Steel

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

Fluorescence spectrum of soldering tin for electronic applications

Figure 9 shows the fluorescence spectra of soldering tin (information provided by the manufacturer: $Sn_{60}Pb_{38}Cu_2$). 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 (line1) 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.
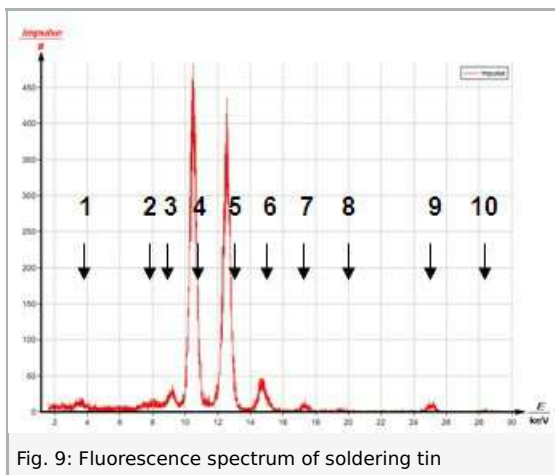

Fig. 9: Fluorescence spectrum of soldering tin

Table 5: Elements of the soldering tin

1	3.57	3.66	$L_{\beta 1}$	Sn
2	8.01	8.04	K_α	Cu
3	9.16	9.18	L_l	Pb
4	10.48	10.44/10.55	$L_{\alpha 1,2}$	Pb
5	12.54	12.61	$L_{\beta 1}$	Pb
6	14.73	14.76	$L_{\gamma 1}$	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

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.
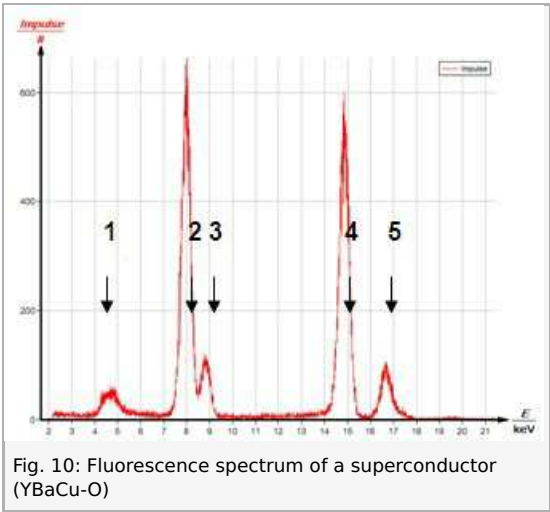

Fig. 10: Fluorescence spectrum of a superconductor (YBaCu-O)

Table 6: Elements of the superconductor

1	4.74	4.45-4.93	L_α	Ba
2	8.02	8.04	K_α	Cu
3	8.86	8.90	K_β	Cu
4	14.87	14.96	K_α	Y
5	16.71	16.73	K_β	Y

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Robert-Bosch-Breite 10
D - 37079 Göttingen

Tel: +49 551 604 - 0
Fax: +49 551 604 - 107

info@phywe.de
www.phywe.com