X-ray monochromatization

LEP 5.4.02

Related topics

Bremsstrahlung, characteristic radiation, energy levels, absorption, absorption edges, interference, diffraction, Bragg scattering.

Principle and task

Polychromatic X-rays are energy-analyzed by means of various monocrystals. An adequately selected thin metal foil is applied, the absorption edge of which drastically reduces the intensity of an unwanted characteristic line.

Equipment

X-ray unit, w. recorder output	09056.97	1
Potassium Bromide Crystal, mounted	09056.01	1
Diaphragm tube w. nickel foil	09056.03	1
Counter tube, type A, BNC	09025.11	1
Pulse rate meter	13622.93	1
xyt recorder	11416.97	1
Screened cable, BNC, l 750 mm	07542.11	1
Connecting cord, 1000 mm, red	07363.01	2
Connecting cord, 1000 mm, blue	07363.04	2

Problems

1. Graphically record the intensity of the X-rays emitted from the copper anode as a function of the Bragg angle by means of LiF- and KBr-monocrystals.
2. Determine the energy values of the characteristic copper lines.
3. Using the LiF-monocrystal, filter out a characteristic line. Graphically record the appertaining monochromatization.
4. Repeat the measurements according to step 1, this time using a nickel filter

Set-up and procedure

The experiment is set up as shown in Fig. 1. The aperture of d = 2 mm is introduced into the outlet of X-rays. By pressing the “zero key”, the counter tube and crystal holder device are brought into starting position. The crystal holders are mounted with the crystal surface set horizontally. The counter tube, with horizontal slit aperture, is mounted in such a way that the mid-notch of the counter tube closes onto the back side of the holder. Typical adjustment settings of the peripheral equipment are:

Pulse rate meter:	Couter tube voltage	500 V
	Sensitivity	10^5 Imp/min
	Time constant	0.5 or 1.5 s
x, y-recorder:	x-axis (ϑ-axis)	1 V/cm additionally variable
	y-axis (intensity)	0.1 V/cm, additionally variable

Fig. 1: Experimental set-up for X-ray monochromatization.

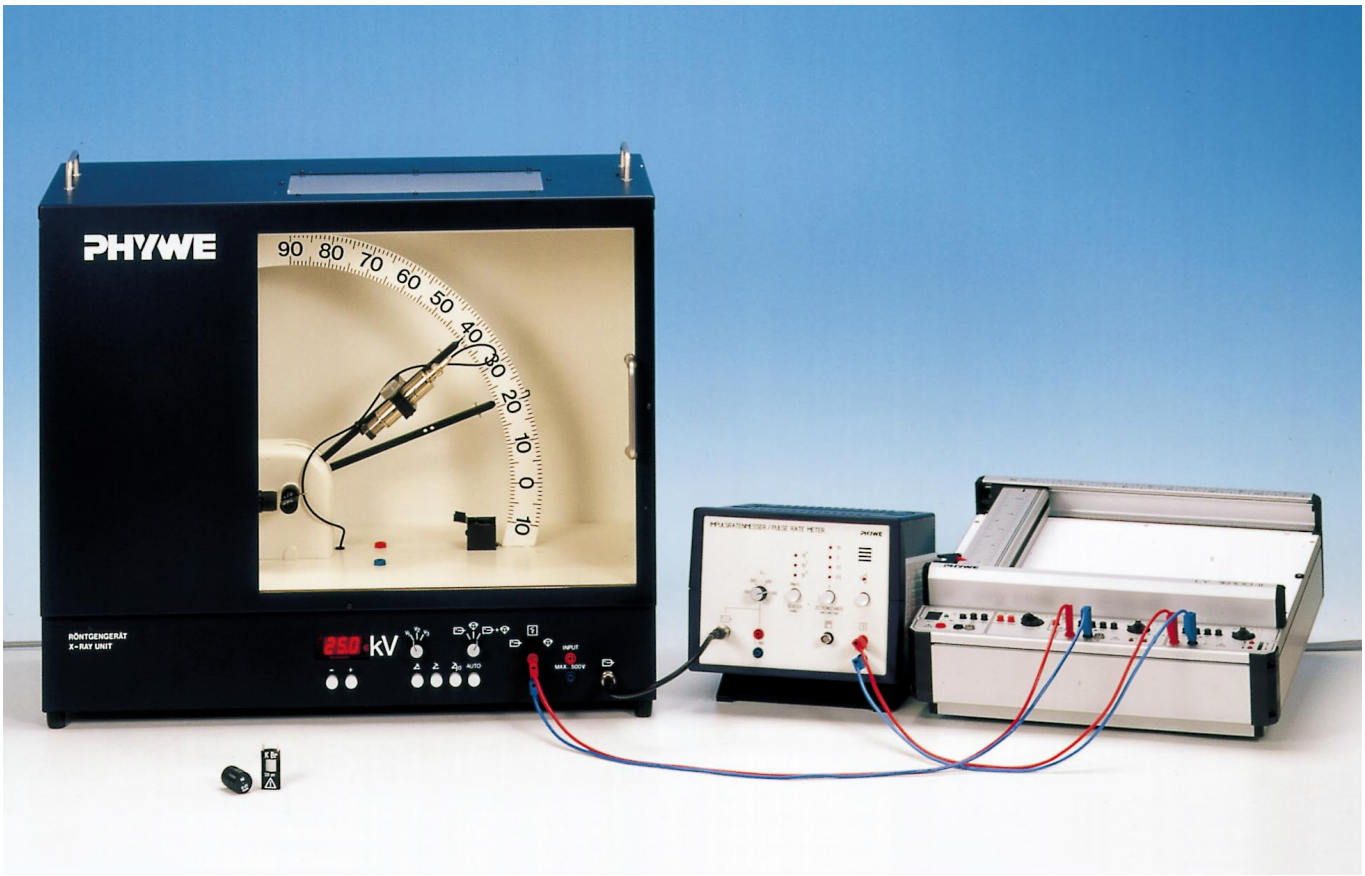
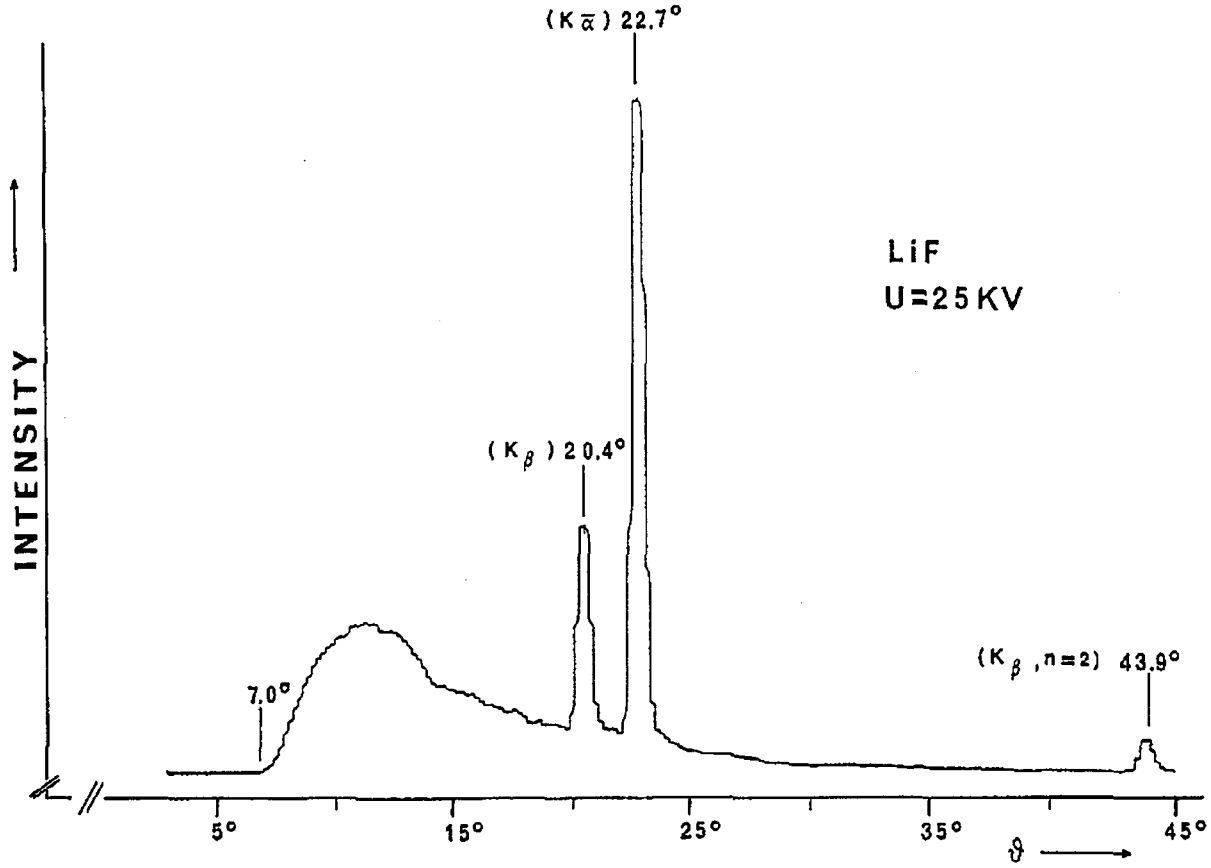


Fig. 2: Copper X-ray intensity as a function of glancing angle ϑ; LiF (100)-monocrystal as analyzer.



The output of the pulse rate meter is connected to the y-input of the recorder. The angle-proportional direct-current voltage (0.1 V/degree) of the X-ray machine lies on the x-input. The plotting of the spectra is performed at a slow velocity of rotation (switch settings "V_1" and "Auto"); crystal and counter tube must rotate in synchronization.

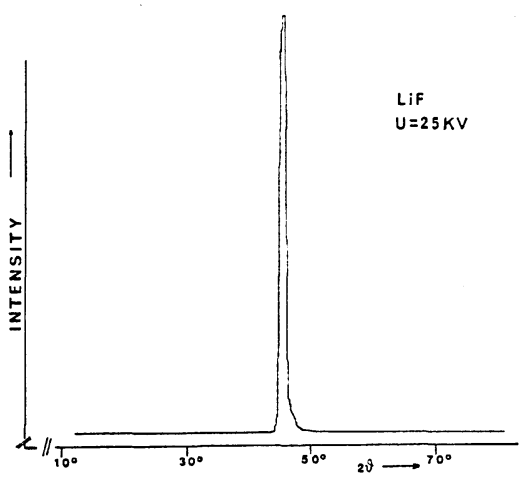


Fig. 3: Copper X-ray monochromatization
Reflected energy interval $\Delta E = E_{K\bar{\alpha}}$.

First, by means of the LiF-crystal, a spectrum is recorded at maximal anode voltage (Fig. 2). Subsequently, the intensive $K_{\bar{\alpha}}$-line must be detected and the synchronized rotation of crystal and counter tube must be decoupled. At an unchanged crystal position, the counter tube must be rotated and the monochromatized energy distribution redcorded (Fig. 3).

For this procedure, the analogue output switch must be set to "counter tube" and the sensitivity of the x-channel of the recorder must be reduced.
The measurement according to Fig. 2 is repeated, this time with the nickel filter d = 0.01 mm (Fig. 4).

Without changing the recording data, the X-ray spectrum analysis is then continued by using the KBr-crystal, both with and without nickel filter (Fig. 5 and 6).

Note:

The counter tube should never be exposed to primary radiation for any longer period of time.

Theory and evaluation

The X-rays emitted from an X-ray tube are polychromatic. Lines characteristic to the anode material whose energy is not dependent upon the anode voltage are superimposed on the continuum of the bremsspectrum. (See also experiment 5.4.1).

For many X-ray oberservations (for example: structure analysis by means of the Debye-Scherrer method), it is necessary to use monoenergetic radiation. Therefore, crystal filters or absorption filters are used.

Monochromatization by crystals

Monocrystals are used in this monochromatization procedure. When X-rays with wavelength λ impinge on the monocrystal under glancing angle ϑ, constructive interference after scattering only appears when the paths of the partial waves on the lattice planes n differ by one or more wavelengths. This situation is explained by the Bragg equation:

$$2 \cdot d \cdot \sin \vartheta = n\lambda \qquad (1)$$

(d = interplanar crystal spacing; n = order of diffraction)

With a known value "d" and measured glancing angle ϑ, it is possible to determine the X-ray energy according to the following relation:

$$E = \frac{n \cdot h \cdot c}{2d \cdot \sin \vartheta} \qquad (2)$$

(h = Planck constant; c = velocity of light)

Fig. 2 shows copper X-ray intensity as a function of the glancing angle ϑ. Here, the LiF-crystal (d = 2.014 · 10^{-12} m) was used as analyzer.

In conjunction with (2), computation yields:

(ϑ = 22.7°, n = 1) $E_{K\bar{\alpha}}$ = 7.98 KeV
(ϑ = 20.4°, n = 1) $E_{K\beta}$ = 8.83 KeV
(ϑ = 43.9°, n = 2) $E_{K\beta}$ = 8.88 KeV

The $K_{\bar{\alpha}}$-line in the 2nd order of diffraction is no longer visible due to the limited angle range. If only a narrow strip-like a portion of the polychromatic spectrum (for example: the $K_{\bar{\alpha}}$-line) is to be used, the analyzer crystal must be brought into the corresponding glancing angle position. The further analysis, made with the independently rotating counter tube detector, indicates the scattered portion consists solely of one intense, sharp line of energy $E_{K\bar{\alpha}}$ (Fig. 3).

Absorption monochromatization

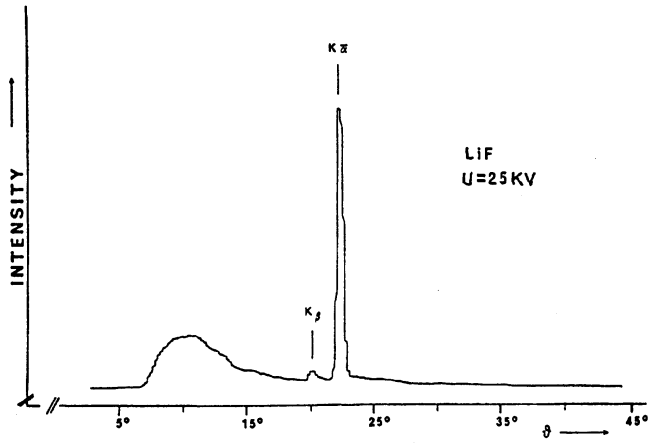
If a thin metal foil of thickness x is brought into an X-ray beam of intensity I_0, the attenuation of the intensity can be described by the absorption law:

$$I_{(E,X)} = I_{(E,O)}^{\ -\mu(E) \cdot x} \qquad (3)$$

(μ [cm^{-1}] = linear absorption coefficient)

Although the absorption coefficient is dependent upon wavelength or energy, it generally shows no dramatic change within an energy interval of several keV. Thus, a very similar attenuation at normal absorption can be expected. An entirely different, discontinuous characteristic absorption appears when the energy of the X-ray quanta barely suffices to ionize the atoms of the absorbing material in the inner shells. This absorption edge is used in order to pinpointedly eliminate certain wavelength ranges from the original spectrum. For example, in order to eliminate the K_β-line from the copper X-ray spectrum, thin nickel foils are used because the energy of the K-level of nickel lies slightly below the energy of the K_β-line.

Fig. 4: Copper X-ray monochromatization with Ni-filter
LiF (100)-monocrystal as analyzer.



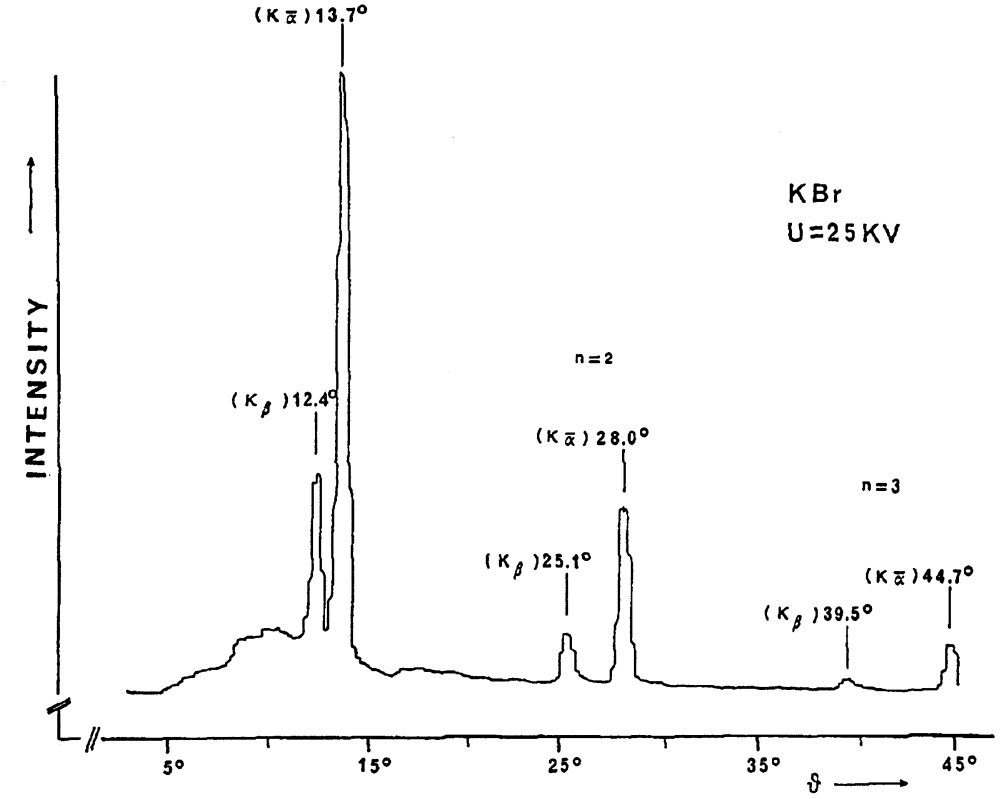
(E_K – Ni = 8.33 keV;
Cu – E_{K_β} = 8.90 keV [Literature value])

The energy of the Cu-$K_{\bar{\alpha}}$-line is already too low to produce ionization in nickel K-shells; thus, due to normal absorption, this line is only slightly attenuated by the Ni-filter.
Fig. 4 shows the result of the energy analysis of the nickel-filtered copper X-rays. The LiF-crystal was used as the analyzer.

A comparison with the unfiltered spectrum (Fig. 2) indicates that the K_β-radiation intensity actually was reduced dramatically and that it can only be indirectly observed in the 1st order of diffraction. If, by approximation, one assumes the intensity proportional to the peak height, the intensity of the Cu-K_β-radiation is attenuated by about 90% by the Ni-filter of thickness d = 0.01 mm due to absorption edge where as the intensity of the Cu-$K_{\bar{\alpha}}$-radiation is attenuated by only 20% due to normal absorption as described by (3).

Using the KBr-crystal as analyzer produces similar results as shown in Fig. 5 and 6. Here, it is also clearly visible that the Cu-K_β-radiation is only indirectly observable in the 1st order of diffraction, where as the Cu-$K_{\bar{\alpha}}$-radiation (despite slight attenuation) is still clearly recordable up to the 3rd order of diffraction.

Fig. 5: Copper X-ray intensity as a function of glancing angle ϑ; KBr (100)-monocrystal as analyzer.



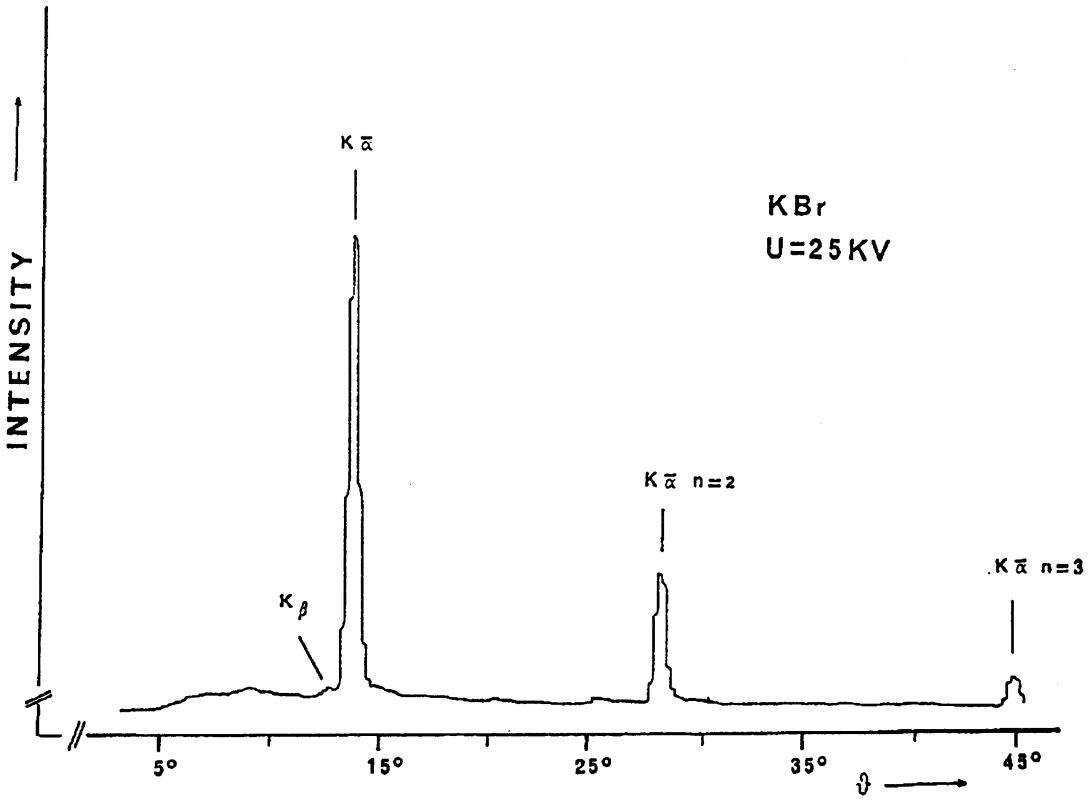


Fig. 6: Copper X-ray monochromatization with Ni-filter KBr (100)-monocrystal as analyzer.