Abstract

X-rays were produced by two bench-top diffractometers using a copper target. Various nickel filters were placed in front of the detector to measure the effect of x-ray absorption. A single crystal of LiF was used to disperse the beam according to wavelength by Bragg diffraction. The wavelength spectrum was then obtained by a $\theta - 2\theta$ scan technique. Analysis of the radiation spectrum confirmed the nature of the target, although several anomalous findings are examined.

1 Introduction

Around the turn of the century, a new type of radiation was discovered by the German physicist Wilhelm Roentgen (Cullity 3). It was soon realized that his so-called x-rays were electromagnetic waves with a wavelength on the order of the atomic spacing in crystals. Further research led to the discovery that shining x-rays on crystalline solids produced discrete patterns that shed light on the internal structure of solids.

Electromagnetic radiation is produced whenever a charged particle undergoes a change in kinetic energy. When high-energy electrons are brought to a sudden stop in a solid material, x-rays are produced. The energies of the resulting x-rays are dependent on several interactions that the electrons may undergo. Electrons simply brought to a stop by collisions with the lattice eject photons equal to the loss in kinetic energy. Others may remove electrons from their orbits around atoms in the target material. A higher energy electron may then transition to the vacated orbital and release a photon of characteristic wavelength.

X-rays may themselves undergo similar interactions with matter, either producing new x-rays or being absorbed by the material. The rate at which x-rays are absorbed therefore depends strongly on the electronic structure of the target material.

2 Experimental Procedures

X-rays were produced using a Rigaku MiniFlex II bench-top diffractometer. The x-ray tube employed an acceleration voltage of 30 kV and a current of 15 mA incident on a copper target. A single crystal of LiF installed in the sample tray in an (002) orientation was used as a dispersive element. Two different diffractometers were used; one equipped with a monochromator and the other with an unobstructed beam.

X-ray intensity was measured by rotating the detector through an angle of $2\theta = 90^\circ$ at a rate of $2^\circ$ per minute. The same scan was then repeated at $3^\circ$ per minute with a Ni filter of unknown thickness installed at the detector.

Four partial scans were then performed on each diffractometer from 40deg-50deg at a rate of $2^\circ$ per minute with Ni filters of varying thicknesses. The experimental parameters are summarized below:

<table>
<thead>
<tr>
<th>Parameters for scans performed with the monochromator</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Scan angle</strong></td>
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<tr>
<td>----------------</td>
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<tr>
<td></td>
</tr>
<tr>
<td>Ni filter thickness [µm]</td>
</tr>
</tbody>
</table>

Parameters for scans performed without the monochromator
3 Results

The measured x-ray intensity, measured in counts per second (although scale is arbitrary) is shown in Figure 1. The results of successive scans over the $K\alpha$ peak with nickel filters of various thickness are shown in Figure 2.

![X-Ray Spectrum with Monochromator](image1)

![X-Ray Spectrum without Monochromator](image2)

Figure 1: $\theta - 2\theta$ Scan

4 Discussion

4.1 Emission

4.1.1 Spectrum Dispersion

The LiF crystal acts as a sort of prism to divide the incident x-ray spectrum into its component wavelengths and cast them over a broad spatial arc. Given that the crystal is oriented with the [001] direction normal to the sample tray, the angle at which each wavelength produces constructive interference is given by manipulation of Braggs Law.

$$\lambda = 2d_{002} \sin \theta$$

The measured spectra for the x-ray source with and without the monochromator are shown in Figure 3.
The single crystal dispersion method is not the most efficient. Only a very small fraction of impinging radiation is scattered by the crystal, while the rest is completely transmitted. It might be possible to create a metallic film that would be sensitive to x-rays. Semiconductor band-gaps can be finely tuned by introduction of impurities. Perhaps a film could be fabricated which receives a gradient of treatments, leaving certain areas susceptible to photoelectric stimulation by certain energies. At least, bands could be constructed on the film which would not detect anything below a certain threshold, and wavelength intensity could be constructed by mathematical methods.

4.1.2 Continuous Spectrum

The short wavelength limit of the x-ray source $\lambda_{swl}$ is the shortest wavelength that the x-ray source is capable of producing. A photon of minimum wavelength (maximum energy) is produced when an electron accelerated through the tube voltage loses all of its kinetic energy in a single collision event. Thus the maximum photon energy is given by the kinetic energy of the electron beam, equal to the accelerating voltage times the charge on the electron.

$$\lambda_{swl} = \frac{12.4}{kV} = 0.413\text{Å}$$

Clearly this condition is not met. X-ray counts continue below this threshold all the way to the 3° cutoff. A voltage of at least 120 kV would be required to produce Bremsstrahlung x-rays at this wavelength. The plausibility of environmental radiation can be effectively ruled out by the inconsequential Geiger counter scan. The possibility of electrical noise or other instrument interference cannot be ruled out.

4.1.3 Characteristic Lines

The characteristic lines are most clearly seen in the unfiltered spectrum. The $K\alpha$ peak is located at 1.53 Å. The $K\beta$ peak is located at 1.39 Å.

<table>
<thead>
<tr>
<th>$2\theta$</th>
<th>$\lambda$ measured</th>
<th>$\lambda$ tabulated</th>
</tr>
</thead>
<tbody>
<tr>
<td>$K\alpha$</td>
<td>44.8</td>
<td>1.534</td>
</tr>
<tr>
<td>$K\beta$</td>
<td>40.26</td>
<td>1.386</td>
</tr>
</tbody>
</table>
The measured peaks are in very close agreement with tabulated values for a copper target (Cullity 509). Moreover, upon close examination the \( K\alpha \) peak actually consists of two separate, overlapping peaks (Figure 5). Although the magnitude is shifted by a few percent, the difference between these peaks matches the difference between the Cu \( K\alpha_1 \) and Cu \( K\alpha_2 \) peaks.

The monochromatic spectrum differs in several ways. The \( K\beta \) peak is noticeably reduced, while the \( K\alpha \) peak is slightly broader and exhibits a shoulder on the short wavelength side. Based on the selective attenuation of the \( K\beta \) peak, the monochromator material must exhibit an absorption edge just under the \( K\alpha \) peak. Nickel matches this condition, with \( K_{\text{edge}} = 1.488\,\text{Å} < \text{Cu}\, K\alpha \).

### 4.1.4 Contamination and Error

Another, smaller peak is present in the monochromatic spectrum at 0.7420 Å (See Figure 3). This matches closely with the Nb \( K\alpha_1 \) peak, or the Mo \( K\alpha \). Since this peak is not present in the unfiltered spectrum, it is possible that it is a result of contamination in the monochromator.

The unfiltered spectrum shows evidence of contamination at \( \lambda = 2.77 \,\text{Å} \). This approximately matches the tabulated value for Sc \( K\beta_1 \) or Ba \( L\alpha_1 \), however neither of these are likely for a number of reasons. First, the measured wavelengths seem to be left-shifted from the tabulated values, thus it is unlikely that an exact match can be made based solely upon a single peak. Secondly, the relative intensity from an \( L\alpha_1 \) peak should be almost undetectable, especially from a small amount of contamination. Secondly, the \( \lambda = 2.77 \,\text{Å} \) peak does not show up in the filtered spectrum. It does not seem to be the case that the peak was simply filtered out by the Ni foil, because the peak shows up just as clearly in the absorption spectrum Figure 6. If the peak had been filtered out, many of the surrounding wavelengths should have been filtered as well, at least on one side if an edge was present.
The monochromatic spectrum and the unfiltered spectrum were produced from different machines. Without obtaining an unfiltered spectrum from the former machine, it must be assumed that all other machine parameters are identical. This is likely a generous approximation, and analysis of the effects of the monochromator will be educated guesswork.

4.2 Absorption

4.2.1 $K$ Absorption Edge

The Cu $K$ edge is the wavelength at which $K$ shell electrons can be ionized leading to $K$ fluorescence. Photons are ejected when an electron from a nearby shell drops into the vacant spot in the $K$ shell and ejects a photon with the energy corresponding to the difference in shell energies. This energy difference is smaller than the energy needed to completely remove a $K$ shell electron, so the $K$ edge is located just below the Cu $K\beta$ peak.

Peaks in the emission spectrum only provide information about the relative energies of neighboring orbitals, not ionization energies. Therefore it is impossible to determine the exact location of the $K$ absorption edge from the emission spectrum alone. However, since the atomic potential drops off dramatically beyond the $K$ shell, it can be surmised that the $K$ edge is close to the $K\beta$ emission peak ($1.386$ Å). This assumption agrees with the tabulated value of $1.381$ Å (Cullity 627).

4.2.2 Nickel Filters

Nickel foils of various thicknesses were installed in the path of the beam. The attenuation factor for a Nickel filter of unknown thickness is shown in Figure 6.
4.2.3 Absorption Coefficient

X-ray absorption occurs through several physical mechanisms. Incident x-rays can be elastically scattered, losing no energy to the material, inelastically scattered, in which there is some energy exchange with the crystal, or absorbed, in which the photon gives up its energy to ionize an electron. Absorption is the cumulative effect of any mechanism which removes photons from the forward propagating beam. Fractional intensity loss is proportional to distance traveled through the material, therefore the intensity decays exponentially.

\[ I_x = I_0 e^{-\frac{\mu x}{\rho}} \] (1)

Taking the natural log of both sides, it is apparent that the log of beam intensity is proportional to \( \mu x \), modulo addition of a constant. The mass absorption coefficient can be obtained by fitting a straight line to experimental data (Figure 7). \( \mu / \rho = 50.1 \) cm\(^2\)/g. This closely matches the tabulated value for Cu K\( \alpha \) radiation in Ni, \( \mu / \rho = 48.83 \) cm\(^2\)/g (Cullity 512).

Since the long scans were performed at a different scan rate than the peak scans, it is not useful to relate \( I_x \) and \( I_0 \). Also, as demonstrated, it is not necessary since addition of a constant will not change the slope.

The thickness required to reduce Cu K\( \alpha \) radiation intensity by half can be found by solving Equation (1).
Figure 6: Effect of a nickel filter of unknown thickness \( \frac{I_x}{I_0} \)

\[
\ln I_x = -\mu \rho x
\]

\[
\frac{1}{2} = e^{-\mu \rho x} \Rightarrow x = \frac{\ln 2}{\mu} = 1.55 \text{ mm}
\]

Given such a trivial requirement, the heavy lead blankets used in medical practice are surely more than sufficient.
5 Conclusions

X-rays are an extremely powerful tool for the practical engineer. The properties of x-rays allow for extremely fine resolution and deep penetration. The emission spectra of common elements exhibit a quiet continuous background spectrum, punctuated by sharp peaks arising from discrete quantum phenomena.

The process of x-ray absorption allows for finely tuned experiments. The utility of characteristic x-rays comes their dominance, many of orders of magnitude over the continuous background. Many experiments require a monochromatic x-ray beam. Through the use of absorption filters, the background noise can be all but eliminated to select a very narrow band of wavelengths.

6 Works Cited