X Ray Fluorescence Spectroscopy

Aleena Tasneem Khan
12100127
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Outline

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  – Bremsstrahlung Rays
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  – Standard Analysis
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  – Experimental Setup
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• Compton Scattering
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  – Experimental Setup
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  – Proposed Hardware
X Ray Fluorescence

- **X-Ray Fluorescence**: *Emission of characteristic secondary X rays from a material that has been excited by bombarding with high energy X-Rays.*
- **Characteristic X-Rays**: *X-Rays emitted from heavy elements when their electrons make transitions from a higher to a lower atomic energy level.*
- **Characteristic X Rays** are unique for each element as energy emitted is proportional to the binding energy of the element.
Bremsstrahlung Radiation: *The electromagnetic radiation produced due to deceleration of a charged particle (electron) when deflected by another charged particle (the atomic nucleus).*

Bremsstrahlung radiations have a continuous spectrum where as intense peaks of the characteristic x rays can be seen on the spectrum.
Bremsstrahlung and Characteristic X Rays - Spectrums
XRF Analysis

- XRF Analysis is of two types
  - Energy Dispersive XRF (EDXRF)
    - *Uses a solid state detector and distinguishes each peak according to its energy*
  - Wave Dispersive XRF (WDXRF)
    - *Uses the scanning crystal as the dispersive element and distinguishes each peak according to its wavelength*
- We have used the EDXRF spectrometer.
EDXRF Spectrometer
Working Principle

1. X-Ray generator
2. X-Ray tube
3. HV supply
4. Vacuum system
5. Pulse processor
6. Sample
7. Si(Li) detector
8. Targets
9. PC, MCA, Printer
Sample Analysis
Sample Analysis

- Obtaining A Spectrum
- Calibration of Curve for Energy
- XRF-FP Corrections
- Sample Analysis
  - Calibration using Standards – Intensities obtained
  - Calculation of Calibration Coefficients – Concentrations Obtained
Obtaining A Spectrum

- We use ADMCA along with the PX4 MCA to obtain the spectrum for the sample.
- To Calibrate: We identify at least two peaks in the spectrum and enter the characteristic energy value for each peak. Using the “Calibrate” function of ADMCA we calibrate the spectrum in terms of energy.
- This Energy calibration is saved in the MCA and loaded automatically each time a new spectrum needs to be obtained.
XRF-FP Analysis

- The XRF-FP software loads the spectrum obtained by ADMCA and using the initial parameters etc, corrects the spectrum.
- Three steps for correction:
  - Corrections for escape peaks, sum peaks, background continuum, background peaks etc.
  - Deconvolution
  - Accounting for attenuation and matrix effects
- Using the corrected spectrum, it then calculates the relative intensities and concentrations of each element in the sample.
XRF-FP Analysis

The following tables show the intensities and concentrations of various elements in a Steel Sample.

<table>
<thead>
<tr>
<th>Element</th>
<th>Concentration (wt (%))</th>
<th>Proposed Concentration (wt(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cr</td>
<td>19.055</td>
<td>18.45</td>
</tr>
<tr>
<td>Mn</td>
<td>1.281</td>
<td>1.63</td>
</tr>
<tr>
<td>Fe</td>
<td>65.939</td>
<td>65.19</td>
</tr>
<tr>
<td>Ni</td>
<td>8.67</td>
<td>12.18</td>
</tr>
<tr>
<td>Cu</td>
<td>0.294</td>
<td>0.169</td>
</tr>
<tr>
<td>Mo</td>
<td>2.902</td>
<td>2.38</td>
</tr>
</tbody>
</table>
Moseley’s Law
Moseley’s Law

- **Moseley's law** is an empirical law concerning the characteristic x-rays that are emitted by atoms.
- Moseley was able to show that the frequencies of characteristic X-rays emitted from chemical elements are proportional to the square of the element's atomic number.

\[
E_{k\alpha} = \frac{3R_\infty(Z - 1)^2}{4}
\]

- A finding which supported Bohr's model of the atom in which the atomic number is the same as the number of positive charges in the nucleus of the atom.
Verification of Results

- Two different samples used for the verification of Moseley’s Law:
  - Steel
  - Silicon-Brass
- A spectrum of the characteristic X Rays was obtained for each sample and calibrated. Using these energies, a graph between square root of energy and atomic number of the element was plotted.
- The slope of the resulting graph gave the Rydberg Constant.
## Silicon – Brass

<table>
<thead>
<tr>
<th>Element</th>
<th>Atomic Number</th>
<th>Energy (keV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chromium</td>
<td>24</td>
<td>5.41</td>
</tr>
<tr>
<td>Manganese</td>
<td>25</td>
<td>5.90</td>
</tr>
<tr>
<td>Iron</td>
<td>26</td>
<td>6.40</td>
</tr>
<tr>
<td>Cobalt</td>
<td>27</td>
<td>6.93</td>
</tr>
<tr>
<td>Nickel</td>
<td>28</td>
<td>7.48</td>
</tr>
<tr>
<td>Copper</td>
<td>29</td>
<td>8.05</td>
</tr>
<tr>
<td>Zinc</td>
<td>30</td>
<td>8.64</td>
</tr>
<tr>
<td>Antimony</td>
<td>51</td>
<td>26.36</td>
</tr>
<tr>
<td>Lead</td>
<td>82</td>
<td>74.95</td>
</tr>
<tr>
<td>Bismuth</td>
<td>83</td>
<td>77.1</td>
</tr>
</tbody>
</table>
Silicon - Brass

\[ E_{k\alpha} = \frac{3R_\infty (Z-1)^2}{4} \]

and thus we get: slope as:

\[ 2.16 \times 10^{-18} J \approx R_\infty \]

\[ R_\infty = 2.11 \times 10^{-18} J \]
# Steel

<table>
<thead>
<tr>
<th>Element</th>
<th>Atomic Number</th>
<th>Energy (keV)</th>
</tr>
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<tbody>
<tr>
<td>Cr</td>
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<tr>
<td>Mo</td>
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<td>17.48</td>
</tr>
</tbody>
</table>
Steel Sample

\[ E_{k\alpha} = \frac{3R_{\infty}(Z - 1)^2}{4} \]

and thus we get: slope as:

\[ 2.253 \times 10^{-18} \text{ J} \approx R_{\infty} \]

\[ R_{\infty} = 2.11 \times 10^{-18} \text{ J} \]
Compton Scattering
Compton Scattering

- Compton scattering is the interaction of a high energy photon with an electron, and the resulting “scattered” photon which has a reduced frequency, and therefore reduced energy introducing a shift in its wavelength.

\[ \lambda' - \lambda = \frac{h}{m_e c} (1 - \cos \theta) \]

\[ = \lambda_c (1 - \cos \theta) \]

\[ \lambda_c = 2.426 \times 10^{-12} \text{ m} \]
Experimental Setup
Experimental Setup

- Scattering of X Rays was observed by varying the angle at which the detector was placed. The distance between x-ray tube and the detector was kept larger than 25cm for the detector’s safety. An appreciable shift in wavelengths was seen when the angle was changed.
Results
Results
Analysis

- A graph between shift in wavelength (delta lambda) and cosine of angle was plotted.
Proposed Assembly
THANKYOU