KMÜ 396
Materials Science and Tech. I
Presentation

Energy Dispersive X-Ray, EDX
and Wavelength Dispersive X-ray
spectroscopy (WDX)

Prepared and will be presented by:
Ömer Vehbi Karaosmanoğlu
Gökhan Uzun
Osman Turan

KMÜ 396 / Spring 2010 / May 21, 2010, H.Ü. Chemical Engineering Department
Outline

* X-Ray
  * What EDX & WDX is
  * Usage Areas
    * History
    * Instruments
    * How it works
  * EDX-WDX comparison
    * Material Analysis
    * Advantages and Disadvantages
    * Summary

* References

Ömer

Gökhan

Osman
**X-Ray**

* A form of electromagnetic radiation
* Have a wavelength in the range of 10 to 0.01 nanometers
* Largest use is to take images of the inside of objects in diagnostic radiography and crystallography
• Gathering name for several spectroscopic techniques
• Determining the electronic structure of materials by using x-ray excitation.
* Types of X-ray spectroscopy

* X-ray emission spectroscopy or X-ray fluorescence (XRF)**
  * Identification and measurement of concentration of elements

* X-ray absorption spectroscopy
  * A widely-used technique for determining the local geometric and/or electronic structure of matter.

* X-ray magnetic circular dichroism
  * A difference spectrum of two x-ray absorption spectra (XAS) taken in a magnetic field
EDX and WDX

- Variants of X-ray fluorescence (XRF) or X-ray emission spectroscopy
- Chemical analysis methods of this spectroscopy
- Used in conjunction with each other
**What is EDX?**

* Energy dispersive X-ray spectroscopy (EDS or EDX)
* Analytical technique used for the elemental analysis
* Technique used for chemical characterization of a sample
* Investigation of a sample
* Analyzing X-rays emitted by the matter
* Full quantitative analysis showing the sample composition
EDX spectrum of the mineral crust of Rimicaris exoculata (bacteria)
**What is WDX?**

* Wavelength dispersive X-ray spectroscopy (WDXRF or WDS)
* A method used to count the number of X-rays
* Reads or counts only the x-rays of a single wavelength
* Element must be known
* Often used in conjunction with EDS
Usage Areas of EDX

- Materials evaluation and identification
  * Contaminants
  * Elemental diffusion profiles
  * Glassivation phosphorus content
  * Multiple spot analysis of areas from 1 micron to 10 cm in diameter

- Failure analysis
  * Contamination identification
  * Unknowns identification
  * Stringer location and identification

- Quality control screening
  * Material verification
  * Plating specification and certification
* Usage Areas of WDX

Identification of spectrally overlapped elements
* S in the presence of Pb or Mo
* W or Ta in Si, or N in Ti

Detection of low concentration species (10-100 ppm)
* P or S in metals
* Contaminants in precious metal catalysts
* Trace heavy metal contamination
* Performance-degrading impurities in high temperature solder alloys

Analysis of low atomic number elements
* Composition of advanced ceramics and composites
* B in BPSG films (sensitivity to 2000 ppm)
* Oxidation and corrosion of metals
* Characterization of biomedical and organically modified materials
History of X-Ray Techniques

- First discovered by Wilhelm Röntgen (~1875)
- He also named X-Ray: Röntgen rays
- EDX and WDX are new techniques
- EDX since 1950s
- WDX since late 1960s

Wilhelm Röntgen, German Scientist (1845-1923)
Energy Dispersive X-Ray Spectrophotometer (EDX) System Schematic
Wavelength Dispersive X-Ray Spectrophotometer (EDX)
System Schematic
**X-ray Detector**
Detects and converts X-rays into electronic signals

**Pulse Processor**
Measures the electronic signals to determine the energy of each X-ray detected

**Analyzer**
Displays and interprets the X-ray data

<table>
<thead>
<tr>
<th>Element</th>
<th>Mg</th>
<th>Al</th>
<th>Si</th>
<th>Mn</th>
<th>Fe</th>
<th>Ni</th>
<th>Cu</th>
<th>Zn</th>
<th>Totals</th>
</tr>
</thead>
<tbody>
<tr>
<td>Energy</td>
<td>1.26</td>
<td>89.63</td>
<td>0.26</td>
<td>0.96</td>
<td>0.47</td>
<td>1.44</td>
<td>3.25</td>
<td>2.73</td>
<td>100.00</td>
</tr>
<tr>
<td>Counts</td>
<td>1.47</td>
<td>94.20</td>
<td>0.27</td>
<td>0.50</td>
<td>0.24</td>
<td>0.69</td>
<td>1.45</td>
<td>1.18</td>
<td>100.00</td>
</tr>
</tbody>
</table>
Detectors Are Important!

Lithium doped Silicon (SiLi) crystal detector acts as a semiconductor
How it works? - EDX

Obtaining EDX Spectrums

- A high-energy beam of charged particles is focused into the sample
- Ground state (unexcited) electrons in sample are stimulated
- Electrons are excited from lower energy shells to higher energy shell
- The difference in energy between the shells may be released in the form of an X-ray
- The number and energy of the X-rays emitted from a specimen can be measured by an energy dispersive spectrometer
How it works? - WDX

- The WDX operates in much the same way as EDX.
- Unlike the related technique of Energy dispersive X-ray spectroscopy (EDX) WDX reads or counts only the x-rays of a single wavelength, not producing a broad spectrum of wavelengths or energies.
- The crystal structure of sample diffracts the photons in principles of Bragg's law.
- Diffractions are then collected by a detector.
## EDX-WDX comparison

<table>
<thead>
<tr>
<th>Spectral resolution</th>
<th>Higher (160 eV and less)</th>
<th>Lower (2-10 eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Light elements?</td>
<td>With windowless or thin window detector</td>
<td>With synthetic diffractors (&quot;crystals&quot;)</td>
</tr>
<tr>
<td>Detection Limits</td>
<td>~1000-5000 ppm</td>
<td>&lt;100-500 ppm</td>
</tr>
<tr>
<td>Specifications</td>
<td>Cheaper, quicker but some elements are too close together to resolve (eg S Ka, Mo La, Pb Ma)</td>
<td>More expensive, but with much better spectral resolution giving lower detection limits.</td>
</tr>
</tbody>
</table>

Table is adapted from: [www.geology.wisc.edu/~johnf/g777/ppt/00_What_is_777.ppt](http://www.geology.wisc.edu/~johnf/g777/ppt/00_What_is_777.ppt)
* What type of materials can not be tested?

* Elements like H, He, Li, or Be

* The multiple masses of an element (i.e. isotopes)
Analysis of X-Rays

* Point analysis
* Line scanning
* Dot mapping
* Ref: http://www.concrete.cv.ic.ac.uk/durability/research%20techniques%20sem%20edx.htm
*Why EDX?

- Quick
- Versatile
- Inexpensive
- Widely available
* **Why WDX?**

* Analysis for light element
* Higher sensitivity
* Lowered detection limit
* More accurate analysis
* Superior peak resolution
Comparison of EDS (left) and WDS (right)

http://serc.carleton.edu/research_education/geochemsheets/wds.html
Photo: A security guard is running an analysis using X-Rays 😊

Photo ref: http://www.devrimgazetes.com.tr/resim/x%20ray%20cihaz%C4%B1%20devrede%20.JPG
Summary

- Definitions of x-ray and x-ray spectroscopy
- A brief information about edx & wdx
- Definitions of edx & wdx and application areas
- History of edx & wdx
- How edx & wdx work
- Advantages and disavantages of edx & wdx
- Materials that could be tested by edx & wdx
References

http://mee-inc.com/eds.html

http://www.photometrics.net/techniq.html

http://serc.carleton.edu/research_education/geochemsheets/wds.html


http://serc.carleton.edu/research_education/geochemsheets/wds.html
