

# KMÜ 396

## Materials Science and Tech. I

### Presentation

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

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# Outline

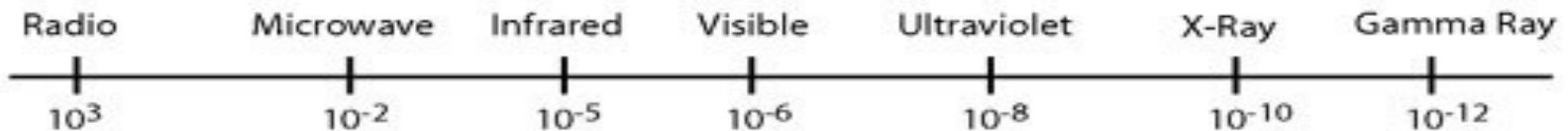
- \* X-Ray
  - \* What EDX & WDX is
  - \* Usage Areas
  - \* History
  - \* Instruments
  - \* How it works
  - \* EDX-WDX comparison
  - \* Material Analysis
  - \* Advantages and Disadvantages
  - \* Summary
  - \* References
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- Ömer
- Gökhan
- Osman

# \*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

THE ELECTRO MAGNETIC SPECTRUM

Wavelength  
(metres)



# \*X-ray Spectroscopy

- Gathering name for several spectroscopic techniques
- Determining the electronic structure of materials by using x-ray excitation.

# \* Types of X-ray spectroscopy

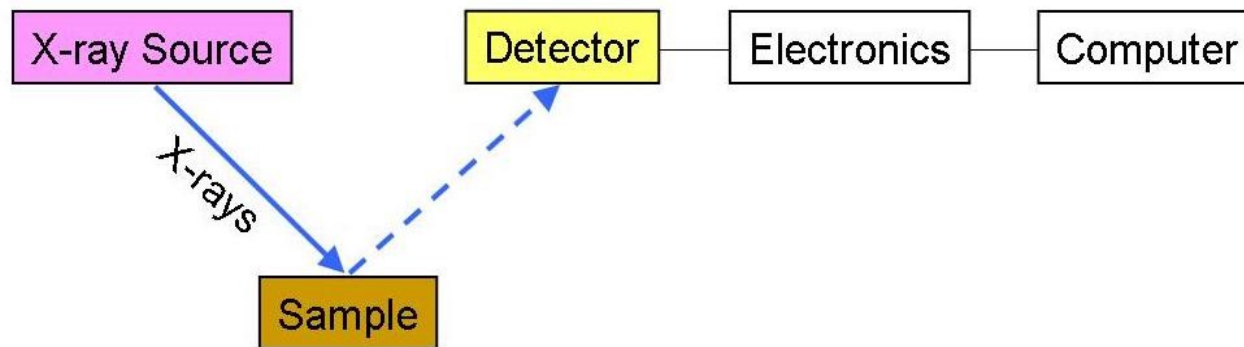
- \* X-ray emission spectroscopy or X-ray fluorescence (XRF)\*\*
  - \* Identification and measurement of concentration of elements
- \* X-ray absorption spectroscopy
  - \* A widely-used technique for determining the local geometric and/or electronic structure of matter.
- \* X-ray magnetic circular dichroism
  - \* A difference spectrum of two x-ray absorption spectra (XAS) taken in a magnetic field

# \* EDX and WDX

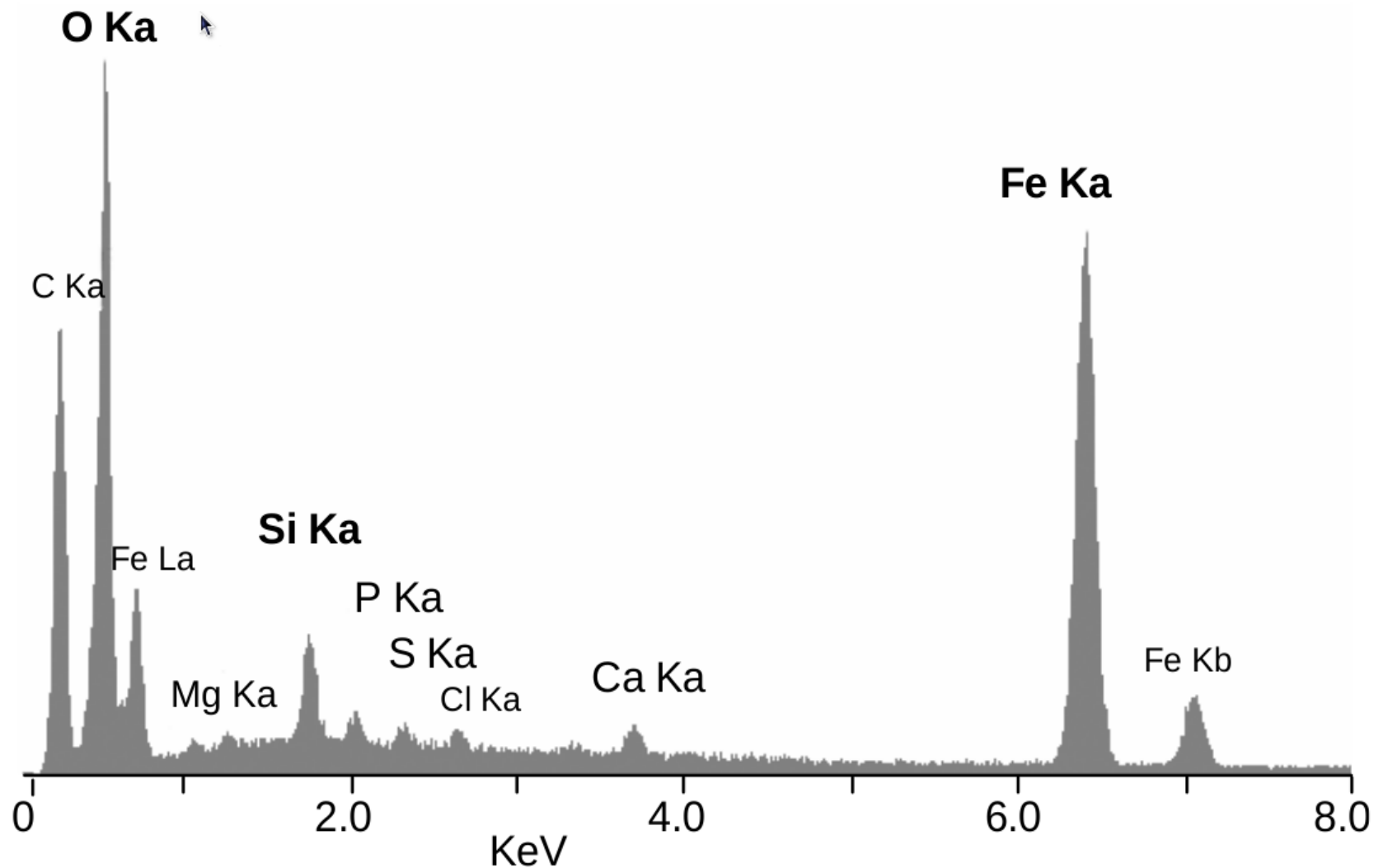
- \* Variants of X-ray fluorescence (XRF) or X-ray emission spectroscopy
- \* Chemical analysis methods of this spectroscopy
- \* Used in conjunction with each other

# \*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



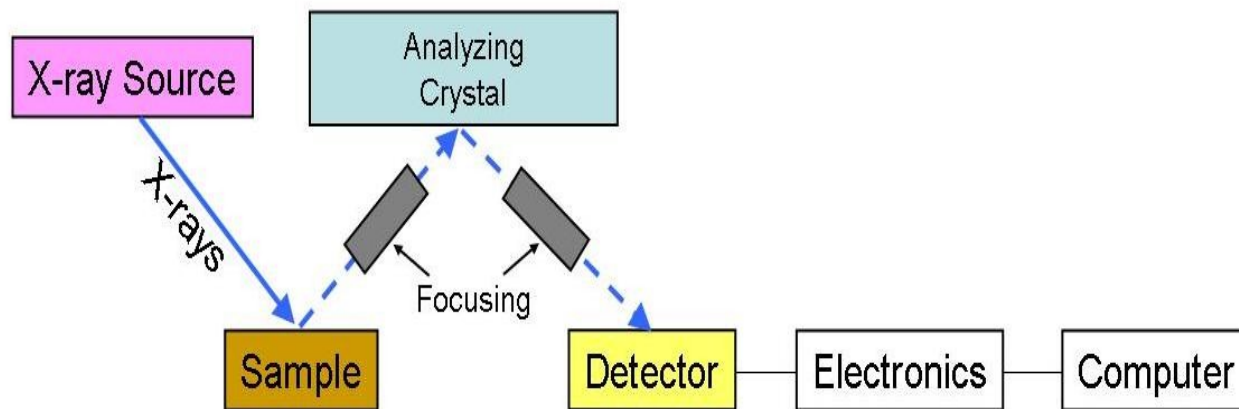
\* EDX spectrum of the mineral crust of *Rimicaris exoculata* (bacteria)





# \*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)

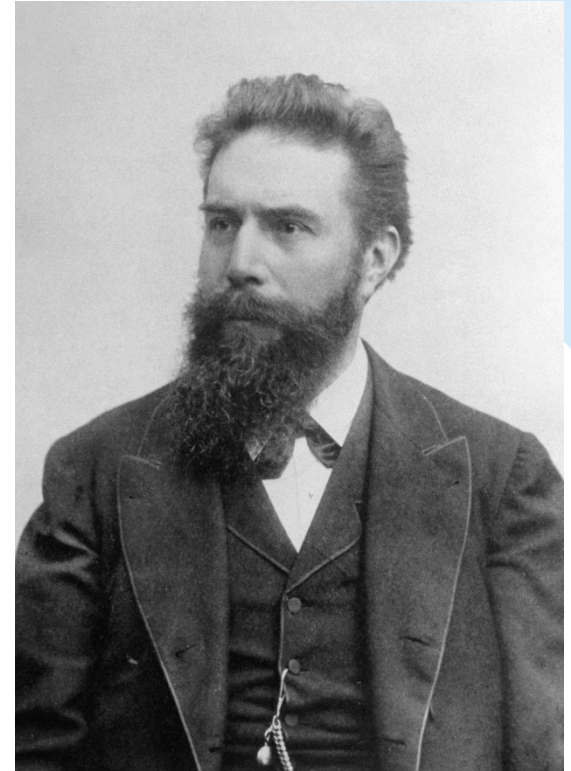
- \* P or S in metals
- \* Contaminants in precious metal catalysts
- \* Trace heavy metal contamination
- \* Performance-degrading impurities in high temperature solder alloys

## 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

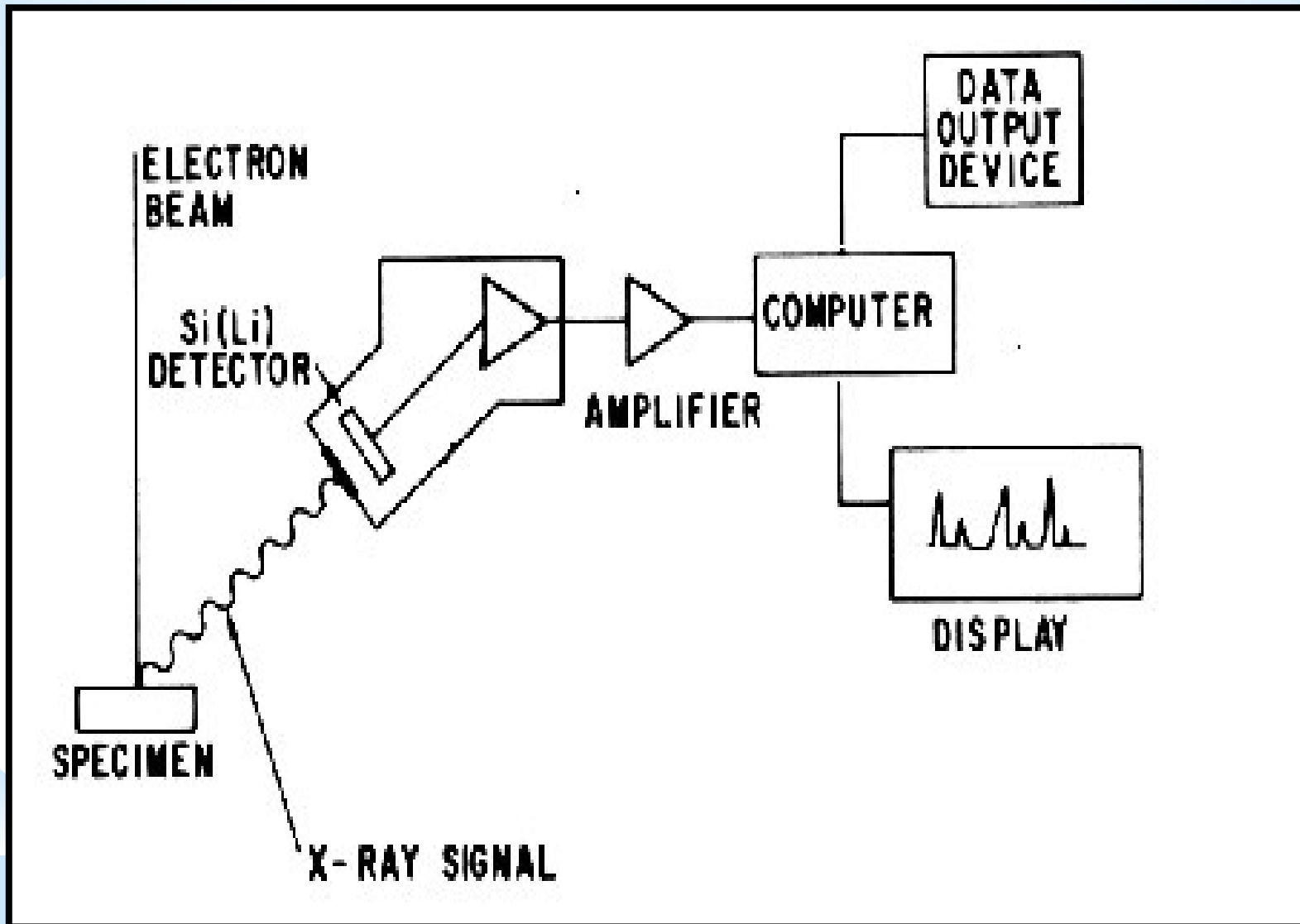
# \* History of X-Ray Techniques

- 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

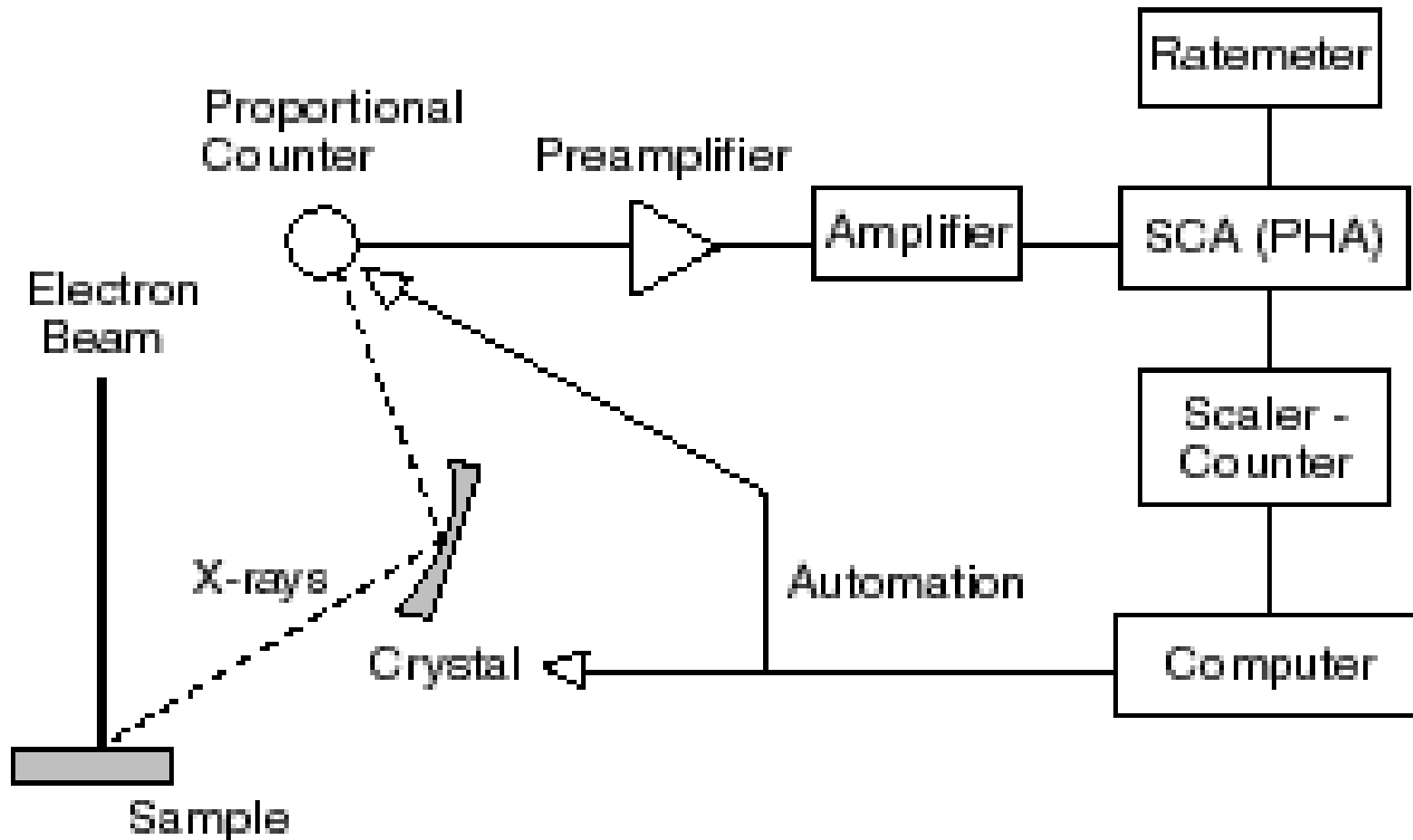


Wilhelm Röntgen, German Scientist  
(1845-1923)

# Energy Dispersive X-Ray Spectrophotometer (EDX) System Schematic



# Wavelength Dispersive X-Ray Spectrophotometer (EDX) System Schematic



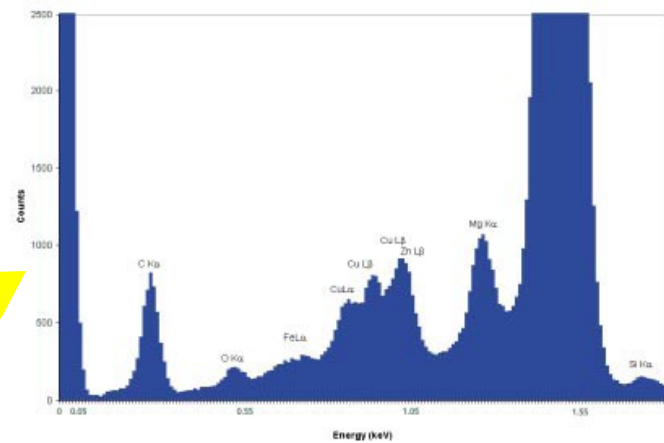


### X-ray Detector

Detects and converts X-rays into electronic signals

### Pulse Processor

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

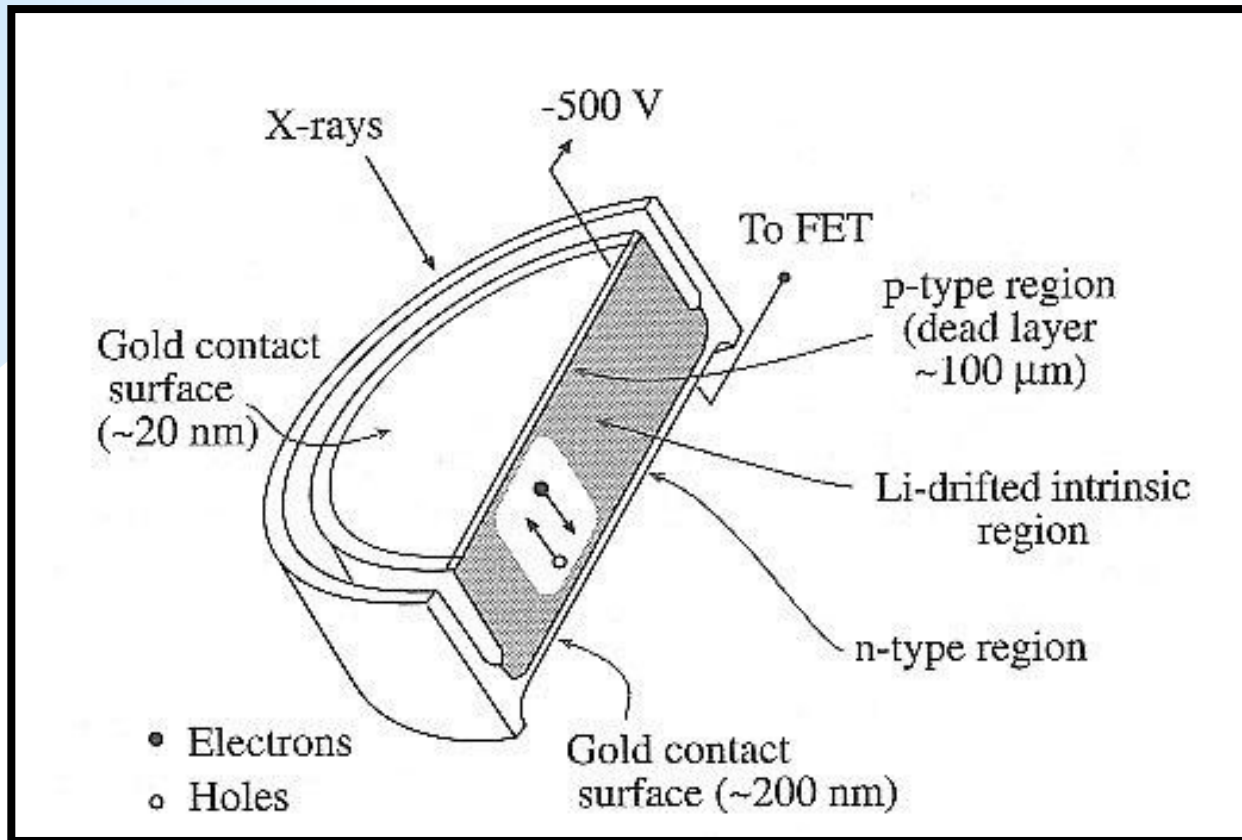


Mg	1.26 +/- 0.05	1.47
Al	89.63 +/- 0.22	94.20
Si	0.26 +/- 0.05	0.27
Mn	0.96 +/- 0.07	0.50
Fe	0.47 +/- 0.07	0.24
Ni	1.44 +/- 0.09	0.69
Cu	3.25 +/- 0.13	1.45
Zn	2.73 +/- 0.14	1.18
Totals	100.00	100.00

### Analyzer

Displays and interprets the X-ray data

# 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

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

# \* What type of materials can not be tested?

\* Elements like H, He, Li, or Be

\* The multiple masses of an element (i.e. isotopes)

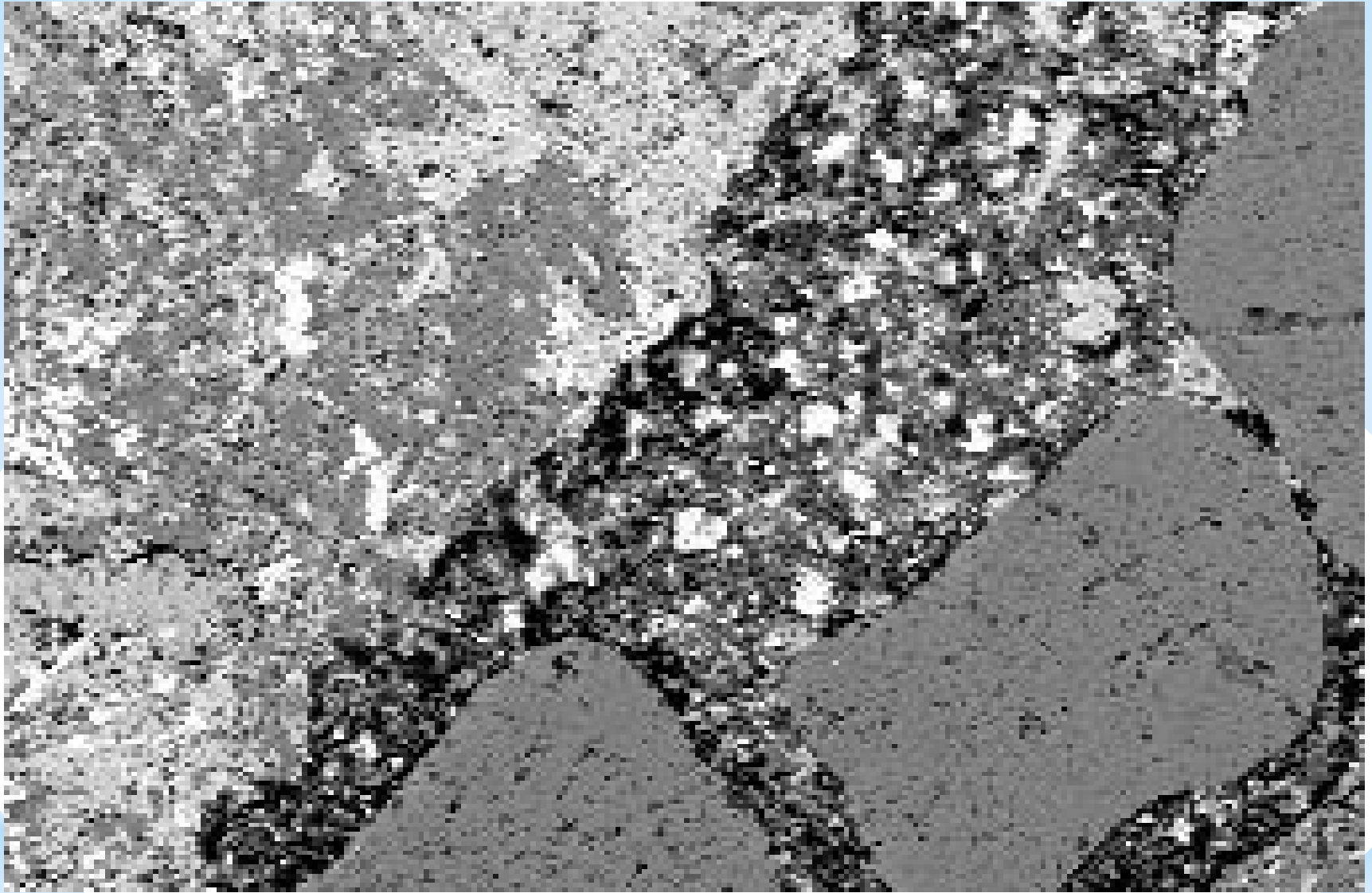
# \* Analysis of X-Rays

\* 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

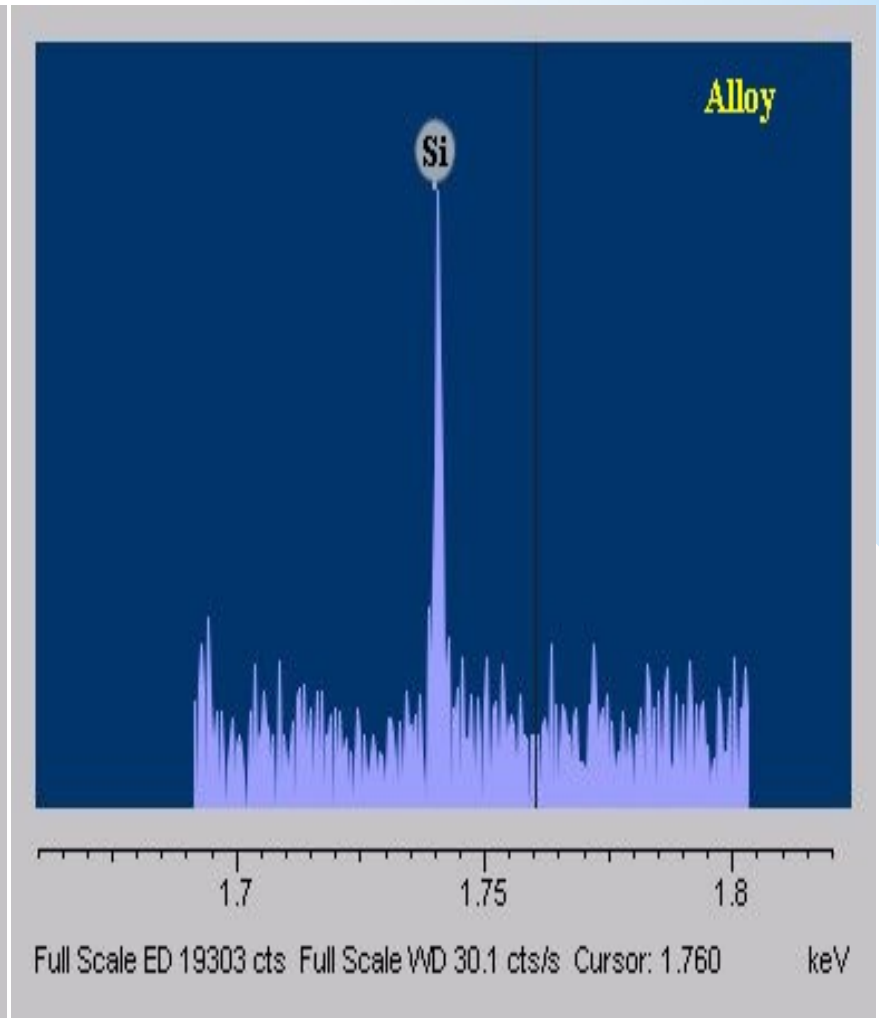
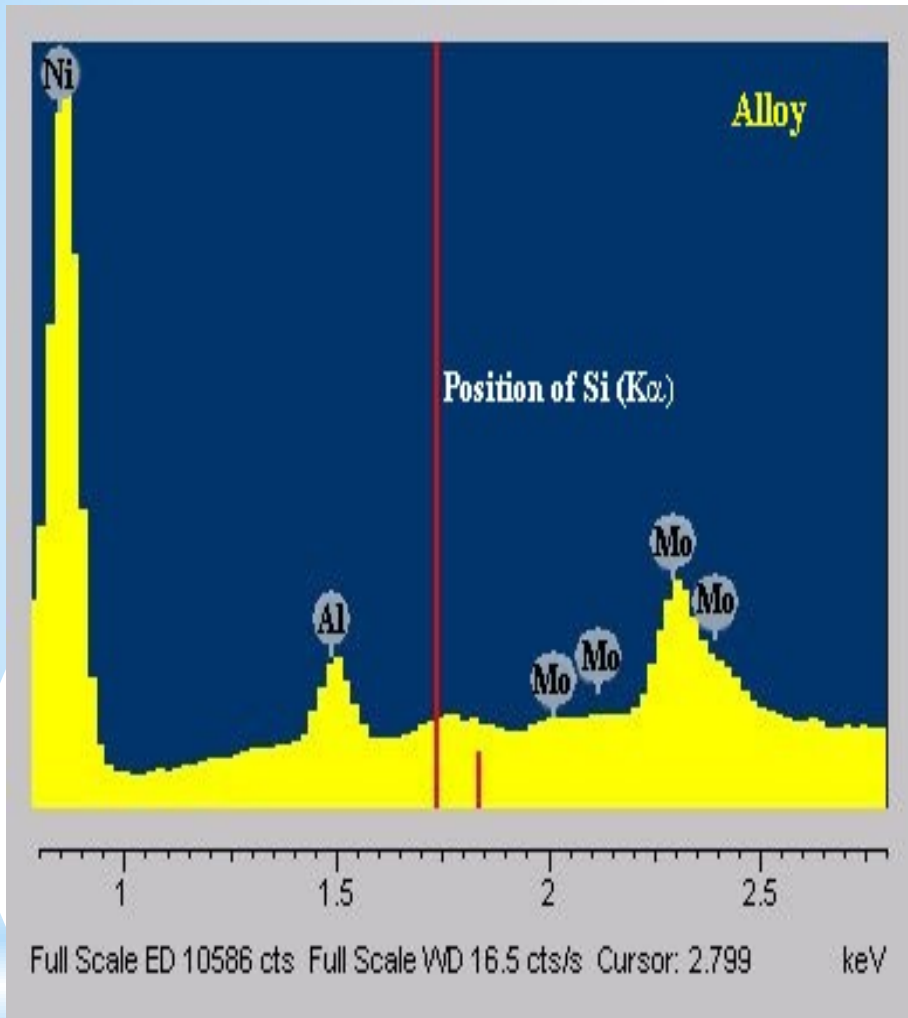


# \*Why WDX?

- \* 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](http://serc.carleton.edu/research_education/geochemsheets/wds.html)



Photo: A security guard is running an analysis using X-Rays 😊

# \*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 disadvantages 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)

Beckhoff, B., Kanngießner, B., Langhoff, N., Wedell, R., Wolff, H., Handbook of Practical X-Ray Fluorescence Analysis, Springer, 2006, ISBN 3-540-28603-9

[http://serc.carleton.edu/research\\_education/geochemsheets/wds.html](http://serc.carleton.edu/research_education/geochemsheets/wds.html)

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Goldstein, J. I. *et al.* (2003). *Scanning Electron Microscopy and X-Ray Microanalysis*. Springer. ISBN 0306472929.

<http://books.google.com/books?id=ruF9DQxCDLQC&printsec=frontcover>.