EE 213, Microscopic Nanocharacterization of Materials Lecture 8.

other micro-characterization using charged particle beams Class website: https://ee213-winter16-01.courses.soe.ucsc.edu

Mike Isaacson, Baskin 237 Email: <u>msi@soe.ucsc.edu</u> Tele: 831-459-3190 Admin. Asst. Rachel Cordero: <u>rcordero@soe.ucsc.edu</u>, 831-459-2921

Crystal Spectrometer geometry



Henry Augustus Rowland



Henry Augustus Rowland (1848-1901)

Born	November 27, 1848 Honesdale, Pennsylvania, U.S.
Died	April 16, 1901 (aged 52) Baltimore, Maryland, U.S.
Nationality	American
Fields	Physicist
Institutions	University of Wooster Rensselaer Polytechnic Institute Johns Hopkins University

1st chair of physics at Johns Hopkins University



Rowland Circle Arrangement



Generic EMPA/SEM WDS



EDS detector

SE, BSE detectors

Vacuum pumps



Electron gun

Column/ Electron optics

Optical microscope

Scanning coils

WDS spectrometers

Faraday current measurement

WDS Spectrometers

An electron microprobe generally has 3-5 spectrometers, with 1-4 crystals in each. Here, SP4 (spectro #4, LF) with its cover off.



Crystals (2 , pairs)

Proportional Counting Tube (note tubing for gas)

PreAmp

 $n\lambda=2d~sin\theta$

where, n = an integer (1, 2, 3...),

 $\lambda = wavelength$,

d = d-spacing of the crystal,

and θ = incident angle (measured from crystal surface)



Lots of Analyzing Crystals

Over the course of the first 30 years of EPMA, ~50 crystals and pseudocrystals

have been used.



Table 5-Diffracting	Crystals for Use in	Wavelength Disp	ersive Spectrometers	和"你们的是你是"
Crystal designation and/or crystal name	Reflecting	2d spacing	Approximate useful wavelength coveragein_A	Intensity performance relative to Mica
Tir lithium fluoride	420	1.78	down to 0.2	
Tapaz	303	2.712	-2.5	4.3
SiO - cuartz	2023	2.75	-2.2	2.8
JiC_lichium fluoride	220	2.848		
LiF-lithium fluoride	200	4.0267	-3.8	12.8
Al aluminum	100	4.049	0.8-4.3	20.0
Al-aluminum SiO	1120	4.903	0.8-4.7	1.4
NoCl and um ablaride	200	5.640	0.9-5.3	15.1
Caladas	104	6.07	1.0-5.7	4.9
ADB amonium dibydrogen phosphate	112	6.14	1.0-5.7	
ADF-ammonium dinydiogen phosphace	. 111	6.2706		
S1-S111COn .	4. de 4.	6.2	1.1-5.8	4.1
Fluorite	111	6.532	1.1-6.0	- 6.6
Ge-germanium	1011	6.6862	1.1-6.3	5.3
SiU2 ~- quartz	002	6.70	T-2-2-2	
App amonium dibudrogen phosphate	200	7.50	1.2-7.3	8
ADF-ammonium uinydiogen phosphace	1010	8.492	1.4-8.1	1.5
SIU2 ~- quarca	002	8.74	1.4-8.3	. 11.8
TELS pencaelychicitor	020	8 808	1.4-8.3	7.4
ADDI-ELHYTERE diamine d carrière	101	10.642	1.8-10.3	3.5
Abra-ammonium dinydrogen phoophate	110	13.98		
SHA-SOTDILOI NEXAACELALE	020	15.2	2.6-15.0	1.9
Gypsum	020	16.40	-16.0	
Bliltanate		14.2		0.2ª
Ammonium carcrace		14.7		0.2ª
Ammonium citrate	002	19.84	3.3-19.4	1.0
Mica - muscovice	1010	25.9		
nan autidium acid phthalate		26.121	2.0-18.3	
KAP-rubidium actu pitchatate	1010	26.632	4.5-25.4	6.4
RAPA-polassium acid phenalace		28.6		0.2
Chlas alizachlors	0001	28.392	527.4	0.4
Chior-Clinochiore		60	8.3-23.7	
ouv estadeerl hydrogen maleate		63.5		0.4
Und-ocladecyl nydrogen marcace		70	-67	15ª
Llaut-leau lautace		80		
LTD -lead tetradeconate		80.5	18-71	
Lmyr-lead myristate		93.8		
OAO-dioctadecyl adlpate		96.9		
OHS-octadecyl hydrogen succinate		100	17-94	15 ^a
LSD-lead sterate decanoate		100	17-94	10.7
LOD*-lead octadeconate		104		
Fe-Mg layered		126		
LTE -lead tetracosanate		- 130	26-120	4 ^a
Llign*-lead lignocerate		140	31-124	
Lcer-lead cerotate		156		
LTC ⁻ -lead triacontanate		160	35-140	
Lmel-lead melissate		100		

*Indicates the crystals that have been commonly used in electron probe analysis. a) Relative intensity related to mica at 13.3 Å by Henke and Lent (Ref. 56); b) 130 alternating layers each of Fe(14Å) and Mg(16Å); c) pseudo-crystal produced by Biodynamics Research Corp., Rockville, Maryland; d) 100 alternating layers each of Fe(13Å) and Mg(39Å).

$N\lambda = 2dsin\theta$

WDS detector

P10 gas (90% Ar - 10% CH_{a}) is commonly used as an ionization medium. The X-ray enters through the thin window and 3 things can occur: (1) the X-ray may pass thru the gas unabsorbed (esp for high keV X-rays); (2) it may produce a trail of ion pairs (Ar ⁺ + e), with number of pairs proportional to the X-ray energy; and (3) if the X-ray is >3206 eV it can knock out an Ar K electron, with L shell electron falling in its place. There are also 3 possibilities that can result from this new photon:



Figure 5.7. Schematic drawing of a gas flow proportional counter.

(3a) internal conversion of the excess energy with emission of Auger electron (which can produce Ar⁺ + e pairs); (3b) Ar Ka X-ray itself can knock out electron of another Ar molecule, producing Ar⁺ + e pair; or (3c) the Ar Ka X-ray can escape out thru a window, reducing the number of Ar⁺ + e pairs by that amount of energy (2958 eV) & Ray analyns (mt)

2. Li Drifted Si, every dispersive Li arts as down to umpensate for impunty accepta levels (B) - result is intrinsic regime in which e-ht can may be neated by external unique radiation. - bies the detector to drage to I side. xray absorbed in intrinsic layer, Ex #e-ht pairs produced = $\frac{E_X}{E}$ 3.7eV in Si .: change vollected Q = (=>).e detersor has capacitance, so we actually get a voltage pulse $V = \frac{Q}{C} = \frac{e}{C} \left(\frac{Ex}{E} \right)$ pulse ht ~ x ray every -> energy dispersive we count pulses (me at a time) assuming Poissim statistics for Q then stand. dev. of withays pube is the DV = eVEX $\therefore \Delta V = \frac{e_1}{e_1} \frac{e_2}{e_1} = \sqrt{e_1}$ not Pokson $\therefore \frac{\Delta E_{X}}{E_{X}} = \sqrt{\frac{e}{E_{X}}} \Longrightarrow \Delta E_{X} = \sqrt{\frac{e}{E_{X}}}$ exactly AN < IN everyy nes. for a Goussian distrib: FWHM=2.36 SEX /

E

62

(21)

XRay Analysis (1not)
When the effumuus nemating from these vanues layes

$$f_{OET} = [Te^{-(4/2)}(4)] \times [1 - e^{-(4/2)}(4)]$$

ABS, trans
 f_{NOTE} . If $e^{-(4/2)}(4) = 1 - e^{-(4/2)}(4)$
 $ABS, trans
 f_{NOTE} . If $e^{-(4/2)}(4) = 1 - e^{-(4/2)}(4)$
 f_{NOTE} . If $e^{-(4/$$

due cam froms. chan up App. dot. material







Continuum Radiation from charged particles

$$\left(\frac{d\sigma}{dE}\right)_{Br} = \frac{3.2 \times 10^{-27} z^2 z^4}{\beta^2} \left(\frac{M_E}{M}\right)^2 \frac{\ln\left[\frac{1}{E_x}\left(\sqrt{E_0} + \sqrt{E_0 - E_x}\right)^2\right]}{\sum_{x} \frac{1}{E_x} (1 - E_x)^2}$$

where $\beta = v/c$, M_E is the electron mass, Z is the atomic number of the target atom, z is the atomic number of the incident particle. For electrons as the colliding particle, this reduces to:

$$\left(\frac{d\sigma}{dE}\right)_{Br} = \frac{3.2 \times 10^{-11} z^2}{\beta^2} \frac{\ln\left[\frac{1}{E_x}(\sqrt{E_0} + \sqrt{E_0 - E_x})^2\right]}{\sum_{x} \frac{E_x}{x}} \quad \text{in } \mathbb{A}^2/\text{eV} \quad (2)$$

The point to be noted here is that this cross-section (per atom) is orders of magnitude smaller than that of the cross-section for characteristic xray production [for the incident electron energies that we will generally be using for microanalysis]. However, if the number concentration of the atoms giving the characteristic xray signal is small, then this cross-section can be of comparable magnitude to the characteristic cross-section, thus affecting the detectability.

considerations on fundamental bly. in Plearn induced XFay spectra
real spectrum - Jackson, Classical EM - somelassical (1962) dup #15.
evaluate using classically
assume radiation constilled of a dragd
particle in a (automb colloin.
-QM taken into account by
take + taking inc = particle velacity
to be ava betre and after velacity

$$\frac{de}{dt} = \frac{3.2 \times 10^{-11} Z_{12}^{2} L_{12}^{2} \left[\frac{ME}{Ex} + VE_{0} - Ex} \right] / 2m$$

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times what
the backeground for electrons
we can deltat
with XTAYS
 $\frac{de}{dt} = \frac{3.2 \times 10^{-11} Z_{12}^{2} Z_{12}^{2} \left(\frac{ME}{MD} + VE_{0} - Ex} \right)^{2} m h/ev
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Note (for endaux by going to hybrid areas them Merce ,
or by using incident probles of herphan mean them Merce ,$$$$$





θ

0

a°

1. UNSCATTERED $\Delta E = 0, \Delta P = 0$



EELS of Nucleic Acid Bases obtained Using 25keV Incident Electrons



M.Isaacson and D. Johnson, Ultramicroscopy.1 (1975).33-52.



Energy Loss Spectra of Metal Fluorides

M. Scheinfein and M. Isaacson, J.Vac.Sci.Tech.B4(1) 1986. 326 – 332.

How to Measure Energy of Every Loss Electrons. In X Rays, solid state detector evergy deposited => eth pairs creates wetage pubse DE ~ 1.6 VEx revolution resolution ~ 100 eV/ of we used same for electrons, incident energy ~ 10° KeV (in an EM) if everyy lost is ikev, then 99 KeV electron hits detector. .: DEN 1.6 V99 K103 = 500 eV // 0 So we report to E, B fields which deflect the electrons - and separate different energies in space. $\vec{F} = Q(\vec{N} \times \vec{B} + \vec{E})$ more on this later, but can got DE/E ~ 10"6!

$$\frac{Rutherford Scattering}{E_{0}, M_{0}, \overline{P}_{0}, \overline{Z}_{0}} = \frac{E_{0}, \overline{P}_{0}}{D_{0}} = \frac{E_{0}, \overline{P}_{0}}{D_{0}} = \frac{Phil.Mag.21.669(1911)}{Phil.Mag.21.669(1911)}$$

$$\frac{d\sigma}{d\Omega} \propto \frac{(e_{0}^{2})^{2}(e_{0}^{2})^{2}}{E_{0}^{2}} \left[\frac{4(\cos\theta + \sqrt{1-\chi^{2}\sin^{2}\theta})^{2}}{\sin^{4}\theta \sqrt{1-\chi^{2}\sin^{2}\theta}}\right]$$

$$where \chi = M_{0}/M \qquad \text{same for RBS}$$
for electrons $\chi \ll 1$ and $\overline{Z}_{0}=1$

$$\frac{d\sigma}{d\Omega} \propto \frac{e^{4} \overline{Z}^{2}}{E_{0}^{2}} \frac{1}{\sin^{4}(\theta/z)} \qquad \text{OK for larger } \theta$$
for electrons/ Ruth Scatt \sim elastic
ie, virtually no energy loss -
$$\Delta E_{MBX} \cong \frac{4Me}{M} = E_{0} \qquad \text{max. energy that}$$

$$\frac{Me}{M} = 5.4640^{9} \frac{1000 \text{ keV electrons}}{1000 \text{ keV electrons}} \qquad \text{in with unitary}$$

(1)





Single Atom Identification in STEM



From O. Krivanek, et.al. Ultramicroscopy. (2012).in press

STEM Imaging/EELS of individual atoms/defects From Krivanek, et.al. Ultramicroscopy.(2102) In press



Single Atom Detection by EELS



Senga & Suenaga. Nature Communications. 6.7943 (1915)

Figure 3 | Detection of single CI atoms. (a) Atomic model of a CsCI atomic chain inside a DWNT. (b) An ADF image of a CsCI atomic chain. (c,d) EELS chemical maps for the Cs *M*-edge and Cl *L*-edge corresponding to **b**, respectively. (e) An EELS spectrum of the CsCI atomic chain in **b** showing a trace of Cl and Cs, as well as the carbon *K*-edge which corresponds to the DWNT. The ADF image **b** only shows the Cs atomic positions as bright spots which are consistent with the red spots in the EELS chemical map of the Cs *M*-edge **c**. The EELS map for the Cl *L*-edge **d** clearly shows the existence of Cl atoms in between Cs atoms despite of hardly visible ADF contrast in **b**. Scale bar, 0.5 nm.



For ebeam induced Xrays, Concentrations about 0.1-1%

each solid line anexports to the proton energy in MeV.

The detectable weight unientration of a trave element of atomic mumber Z in a 0.1mg/cm² carbon matrix. The conventration calculated assumes peak whento = 2×√2× background whent The solid angle that the Si (Li) deleter subtends is DIC = .003× ATT ster. We assume 100% detector efficiency. The mudent proton charge is 10,00001 and the signal detected is Karxrays.

F. Folkwann, C. Gaarde, T. Huus and K. Kemp. Nuc. Inst. Meth. 116. 487(1974)



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MicroPIXE,

Most of the experimental facilites of our laboratory are based on a 5 MV Van de Graaff (VdG) electrostatic accelerator. VdG provides energetic ions (H+, D+, He+, etc.) for ion beam analysis, physical experiments, as well as for the modification and testing of materials.





Introduction Sample Types Theoretical Background of PIXE **Data Reporting Format Quality Assurance Procedures** System Calibration Analytical Applications Interested in getting a quote from EAI? Request a quote online. PIXE NAA OC/EC XAFS

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Solids – such as plastics, papers or metals, are analyzed "as received," while powdered materials, such as fly ash, activated carbon, catalysts, and corrosion products, are ground to 200 mesh or finer and pressed into pellets for analysis.

Liquid – samples, such as oils, process waters, and solutions, are analyzed using a plastic cup of either 8 mL or 3 mL in capacity with a 0.3 mil Kapton front surface window, and can be analyzed "as received" by this method

without modification. However, some liquids (i.e. highly caustic or highly acidic) may require predilution or neutralization before analysis.

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NIST Home > PML > Physical Reference Data > X-Ray Mass Attenuation Coefficients

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Tables of X-Ray Mass Attenuation Coefficients and Mass Energy-Absorption Coefficients from 1 keV to 20 MeV for Elements Z = 1 to 92 and 48 Additional Substances of Dosimetric Interest*

J. H. Hubbell⁺ and S. M. Seltzer Radiation and Biomolecular Physics Division, PML, NIST

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Abstract

Tables and graphs of the photon mass attenuation coefficient μ/ρ and the mass energyabsorption coefficient $\mu_{\rm CR}/\rho$ are presented for all of the elements Z = 1 to 92, and for 48 compounds and mixtures of radiological interest. The tables cover energies of the photon (x-ray, gamma ray, bremsstrahlung) from 1 keV to 20 MeV. The μ/ρ values are taken from the current photon interaction database at the National Institute of Standards and Technology, and the $\mu_{\rm CR}/\rho$ values are based on the new calculations by Seltzer described in

Radiation Research **136**, 147 (1993). These tables of μ/ρ and μ_{en}/ρ replace and extend the tables given by Hubbell in the International Journal of Applied Radiation and Isotopes **33**, 1269 (1982).

Note on NIST X-ray Attenuation Databases

Table of Contents

- 1. Introduction
- X-Ray Mass Attenuation Coefficients
 - Table 1. Material constants for elemental media.

Table 2. Material constants and composition for compounds and mixtures.

Values of the mass attenuation coefficient and the mass energy-absorption

coefficient as a function of photon energy, for:

- Table 3. [Data] elemental media.
- Table 4. [Data] compounds and mixtures.
- The Mass Energy-Absorption Coefficient
- 4. Summary

3.

5. References

"Work supported by the Standard Reference Data Program of NIST.

*Work carried out for NIST under contract 43NANB412756.

U.S. DEPARTMENT OF COMMERCE - Mickey Kantor, Secretary Technology Administration - Mary L. Good, Under Secretary for Technology National Institute of Standards and Technology - Arati Prabhakar, Director



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Access the Data for:

Elemental Media

or

Compounds & Mixtures

NIST Standard Reference Database 126 Rate our products and services. Online: May 1996 - Last update: July 2004

Contact

Stephen Seltzer Radiation and Biomolecular Physics Division phone: 301-975-5552 fax: 301-869-7682

100 Bureau Drive, M/S 8460 Gaithersburg, MD 20899-8460

http://www.nist.gov/pml/data/ xraycoef/



lttp://www.nist.gov/physlab/data/star/index.cfm



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NIST Home > Physics Laboratory > Physical Reference Data > Stopping-Power & Range Tables: e-, p+, Helium Ions

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Stopping-Power and Range Tables for Electrons, Protons, and Helium Ions

M.J. Berger,¹ J.S. Coursey,² M.A. Zucker² and J. Chang²

¹NIST, Physics Laboratory, Ionizing Radiation Division ²NIST, Physics Laboratory, ECSED



Abstract:

The databases ESTAR, PSTAR, and ASTAR calculate stopping-power and range tables for electrons, protons, or helium ions, according to methods described in ICRU Reports 37 and 49. Stopping-power and range tables can be calculated for electrons in any user-specified material and for protons and helium ions in 74 materials.

Contents:

- 1. Introduction
- 2. ESTAR: Stopping Powers and Ranges for Electrons
- PSTAR and ASTAR: for Protons and Helium Ions (alpha particles) References Appendix: Significance of Calculated Quantities

This work was supported in part by the Department of Energy, Office of Health and Environmental Research, Washington, D.C. 20585; and by NIST's Systems Integration for Manufacturing Applications (SIMA) Program.



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Access the Data

Electrons | Protons | Helium Ions

NIST Standard Reference Database 124 Online: October 1998 - Last update: August 2005

Contact

Stephen Seltzer Ionizing Radiation Division phone: 301-975-5552 fax: 301-869-7682

100 Bureau Drive, M/S 8460 Gaithersburg, MD 20899-8460

RANGE

charged particles: CSDA Range (writimuous string down approx, Bethe Range) total path from beginning to end → depth Range projected Range for electrons, proj. Range < CSOA Range for protons, proj Range ~ CSDA Range XRays: Il absorption refficient energies we are interested & INOREV maurily photo effect - all n nothing $\frac{I(t)}{I(0)} = e^{-\mu t}$ so we can think of a projected Range as R~3/4 1e, I(t)~3×10-2/

PSTAR : Stopping Power and Range Tables for Protons



ESTAR : Stopping Power and Range Tables for Electrons









Introduction Sample Types Theoretical Background of PIXE **Data Reporting Format Quality Assurance Procedures** System Calibration Analytical Applications Interested in getting a quote from EAI? Request a quote online. PIXE NAA OC/EC XAFS

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Sample Types Return to Top

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MicroPIXE,

Most of the experimental facilites of our laboratory are based on a 5 MV Van de Graaff (VdG) electrostatic accelerator. VdG provides energetic ions (H+, D+, He+, etc.) for ion beam analysis, physical experiments, as well as for the modification and testing of materials.



Micro-PIXE analysis of an Egyptian Papyrus

Identification of the pigments used for the "Book of the Dead"

A-M. B. Olsson¹, T. Calligaro², S. Colinart², J.C. Dran², N.E.G. Lövestam³, B. Moignard² and J. Salomon²

¹University of Göteborg, SE-405 30 Göteborg, Sweden

²Centre de recherche et de restauration des musées de France, CNRS UMR 171, Palais du Louvre Paris, France

³Chalmers University of Technology, SE–412 96 Göteborg, Sweden http://www.fastcomtec.com/n-applications/multiparameter-systems/micro-pixe-analysis.html



View of the external microprobe set-up with the papyrus in place

The Papyrus KM 21933 consists of hieroglyphic text ended by a painted vignette. It forms the right end of a "Book of the Dead" from the Theban 19th Dynasty (c.1295–1186 B.C.). This name is given to religious funerary texts and spells for protection and guidance of the deceased entering the afterlife. Probably discovered in the 1820's, it was acquired in Berlin in 1912 by The Kulturen Museum in Lund, Sweden.



HARVARD PIXE SYSTEM



From http://www.mrsec.harvard.edu/cams/PIXE.html

Particle Accelerators in Art & Archaeology

Pier Andrea Mandò

Dipartimento di Fisica and Sezione INFN, Florence, Italy

e-mail mando@fi.infn.it

www.presid.infn.it/er/er04fazio.ppt

Erice, April 17, 2004







External-beam PIXE analysis of the frontispiece of PI.16,22, from Biblioteca Laurenziana in Florence

PIXE analysis of ancient manuscripts

(INFN Firenze, Biblioteca Vaticana, Biblioteca Laurenziana)

Detecting which pigments were employed provides important arthistorical information, both about general trends and specifically about the analysed work.

More or less precious materials ↔ symbolic value of the text. Trade routes of raw material import from countries far away. Added or restored parts.

differential PIXE to discriminate the contributions of different layers



+ simultaneous use of PIGE to detect light elements

<u>external</u> microbeam line

1.0

-

D

0

Final Paper/

- Paper due last day class
 10 page approx.
 (IEEE style references)
- 2. Rough outline due February 26 or earlier

3. Topic should be about a particular microcharacterization technique and comparison with at least one other method. From topics covered in course outline.

4. You must discuss the spatial resolution characteristics and limits.

- 5. Include the abstract of each paper you reference or a one paragraph summary of the url reference.
- 6. Briefly, discuss a particular application.

EE213 Paper Notes

- See IEEE.jour for formatting notes. On class web site.
- Paper should be about 10 pages long including figures.
- Paper should include a 1 paragraph abstract
- Paper should have at least 10 references.
- For each reference, either a summary or the abstract of that reference attached as an Appendix to the paper.

Possible topics for paper SEM imaging Quantitation in the SEM Auger microscopy/spectrosocpy Particle beam induced Xray spectroscopy (electrons, ions, photons, etc.) Xray Microscopy SIMS microscopy **RBS** microanalysis Super resolution optical microscopy Scanned tip microscopy Atom probe microscopy Tomography Other topics (upon approval)