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Title: Fuel Cell Materials Characteriza0on Part 1: X---ray diffrac0on and X---ray fluorescence

Author(s): Kreller, Cortney

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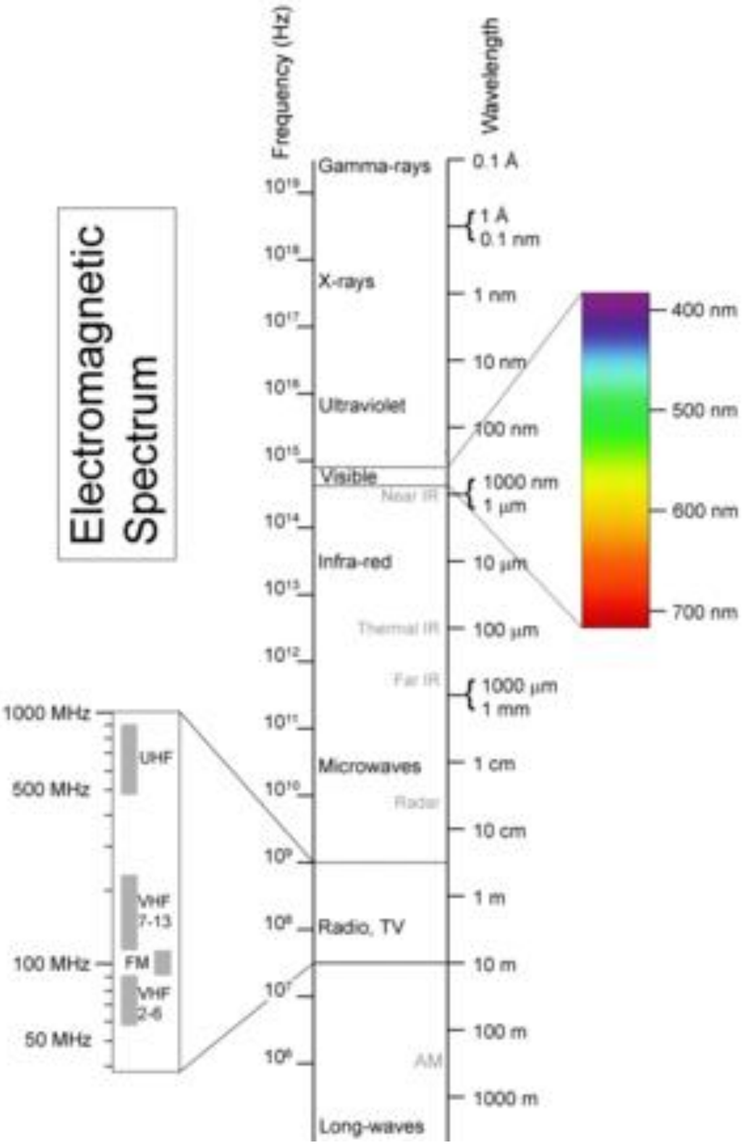
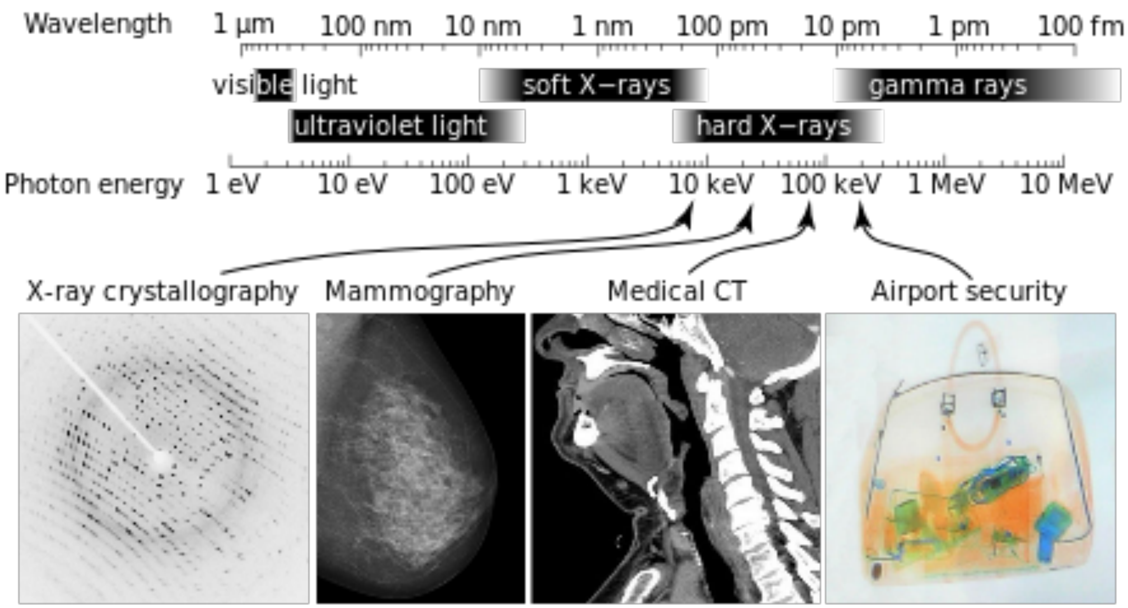
Fuel Cell Materials Characterization Part 1: X-ray diffraction and X-ray fluorescence

LANL Fuel Cell Short Course

Cortney Kreller
August 5,2015

X-Ray Methods





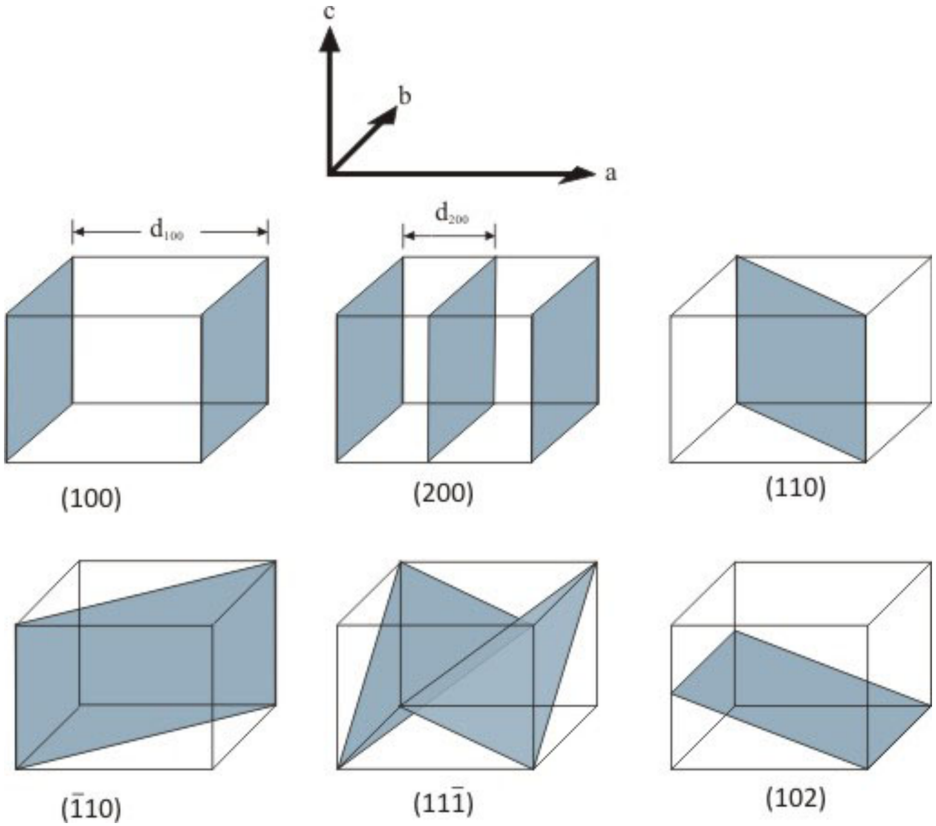
X-rays: short wavelength, high phonon energy:

- Short wavelength scatters off atoms and other small things→ X-ray diffraction (XRD)
- High energy photons ionize inner shell electrons causing elemental fluorescence X-ray→ X-ray fluorescence (XRF)

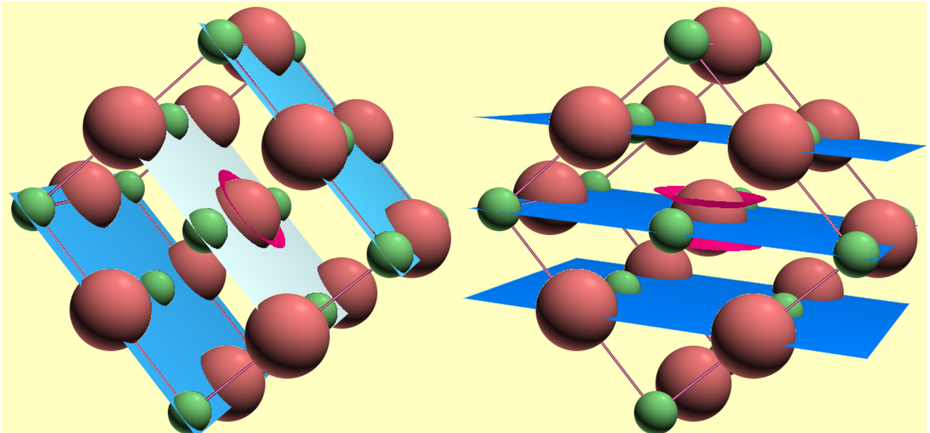
"X-ray applications" by Ulflund *https://en.wikipedia.org/wiki/X-ray*

XRD Basics: Crystalline structure and Miller Indeces

- Any lattice plane can be described by its intersection points with the three unit cell axes
- Miller indices are the reciprocal of the intercepts
  - integers h, k and l refer to reciprocals of intercepts on a, b and c
- Identify planes *adjacent* to origin - planes through the origin cannot be described, as the intercept would be zero
- A plane running parallel to an axis has an intercept of ∞, this corresponds to a Miller index of 0
- Distance *d* is the plane spacing



The (200) planes of atoms in NaCl

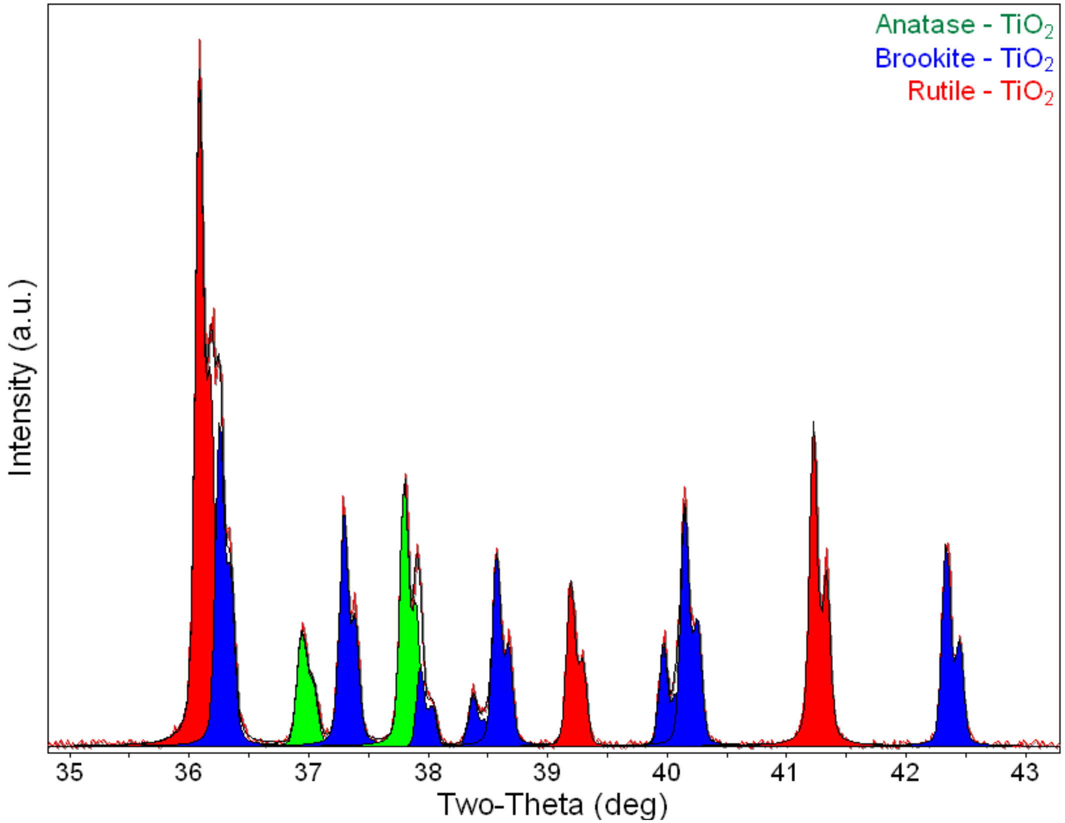


The (220) planes of atoms in NaCl

The atoms in a crystal are a periodic array of coherent scatterers and thus can diffract light.

- Diffraction occurs when each object in a periodic array scatters radiation coherently, producing concerted constructive interference at specific angles.
- The **electrons** in an atom coherently scatter light.
  - The electrons interact with the oscillating electric field of the light wave.
- Atoms in a crystal form a periodic array of coherent scatterers.
  - The wavelength of X rays are similar to the distance between atoms.
  - Diffraction from different planes of atoms produces a diffraction pattern, which contains information about the atomic arrangement within the crystal
- X Rays are also reflected, scattered incoherently, absorbed, refracted, and transmitted when they interact with matter.

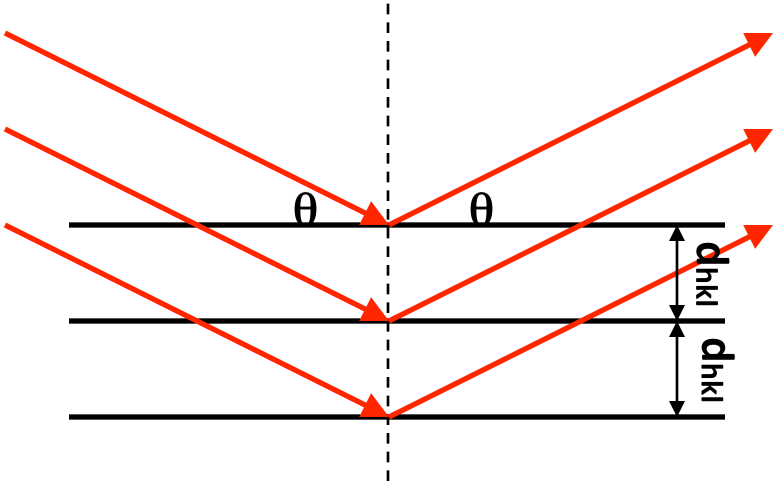
X-Ray Powder Diffraction (XRPD) uses information about the position, intensity, width, and shape of diffraction peaks in a pattern from a polycrystalline sample.



The x-axis, 2theta, corresponds to the angular position of the detector that rotates around the sample.

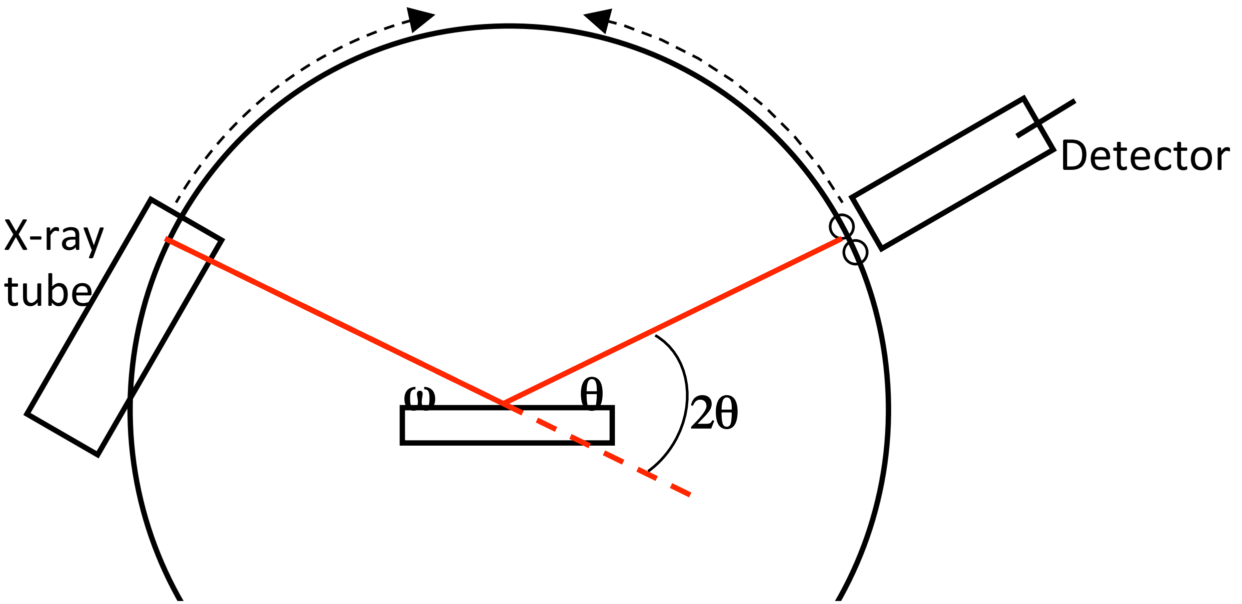
Bragg's law is a simplistic model to understand what conditions are required for diffraction.

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



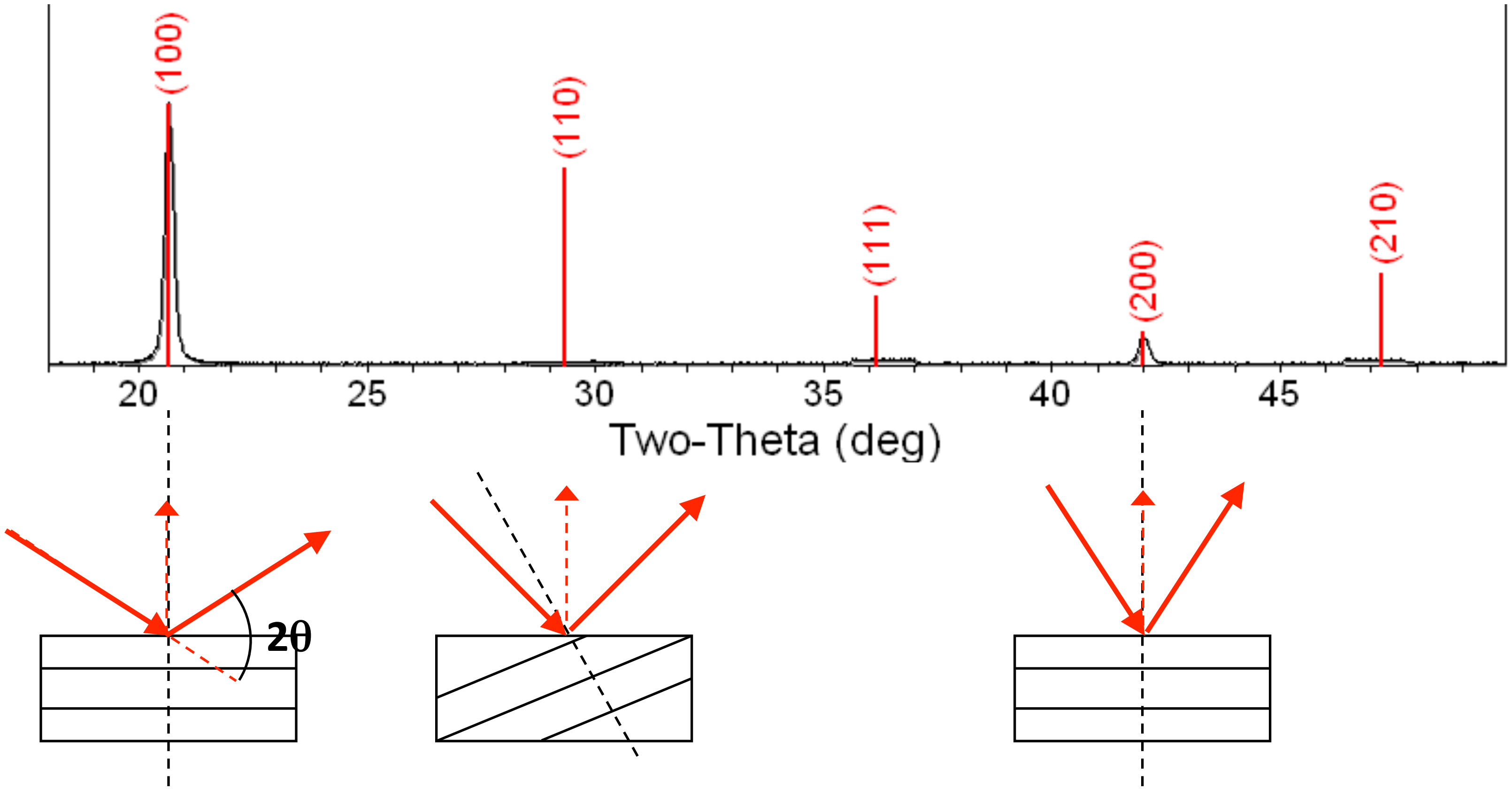
- For parallel planes of atoms, with a space d_{hkl} between the planes, constructive interference only occurs when Bragg's law is satisfied.
  - In our diffractometers, the X-ray wavelength λ is fixed.
  - Consequently, a family of planes produces a diffraction peak only at a specific angle θ.
  - Additionally, the plane normal must be parallel to the diffraction vector
    - Plane normal: the direction perpendicular to a plane of atoms
    - Diffraction vector: the vector that bisects the angle between the incident and diffracted beam
- **The space between diffracting planes of atoms determines peak positions.**
- **The peak intensity is determined by what atoms are in the diffracting plane.**

Powder diffractometers with Bragg-Brentano geometry



- The incident angle, ω, is defined between the X-ray source and the sample.
- The diffracted angle, 2θ, is defined between the incident beam and the detector angle.
- The incident angle ω is always ½ of the detector angle 2θ .
- In a θ:2θ instrument (e.g. Rigaku RU300), the tube is fixed, the sample rotates at θ °/min and the detector rotates at 2θ °/min.
- In a θ:θ instrument (e.g. PANalytical X' Pert Pro), the sample is fixed and the tube rotates at a rate -θ °/min and the detector rotates at a rate of θ °/min.

A single crystal specimen in a Bragg-Brentano diffractometer would produce only one family of peaks in the diffraction pattern.

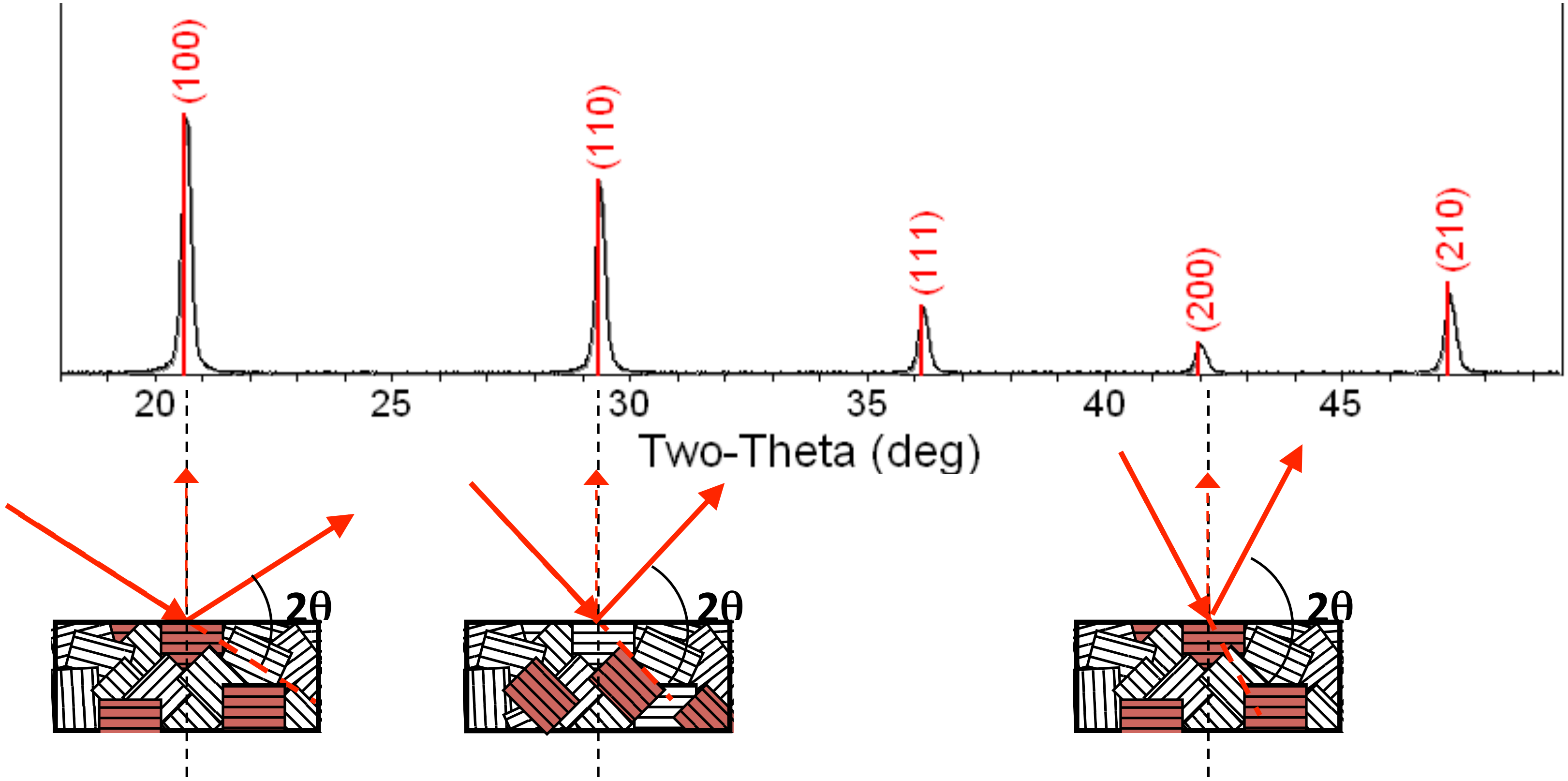


At 20.6 °2θ, Bragg’s law fulfilled for the (100) planes, producing a diffraction peak.

The (110) planes would diffract at 29.3 °2θ; however, they are not properly aligned to produce a diffraction peak (the perpendicular to those planes does not bisect the incident and diffracted beams). Only background is observed.

The (200) planes are parallel to the (100) planes. Therefore, they also diffract for this crystal. Since d_{200} is ½ d_{100}, they appear at 42 °2θ.

A polycrystalline sample should contain thousands of crystallites. Therefore, all possible diffraction peaks should be observed.



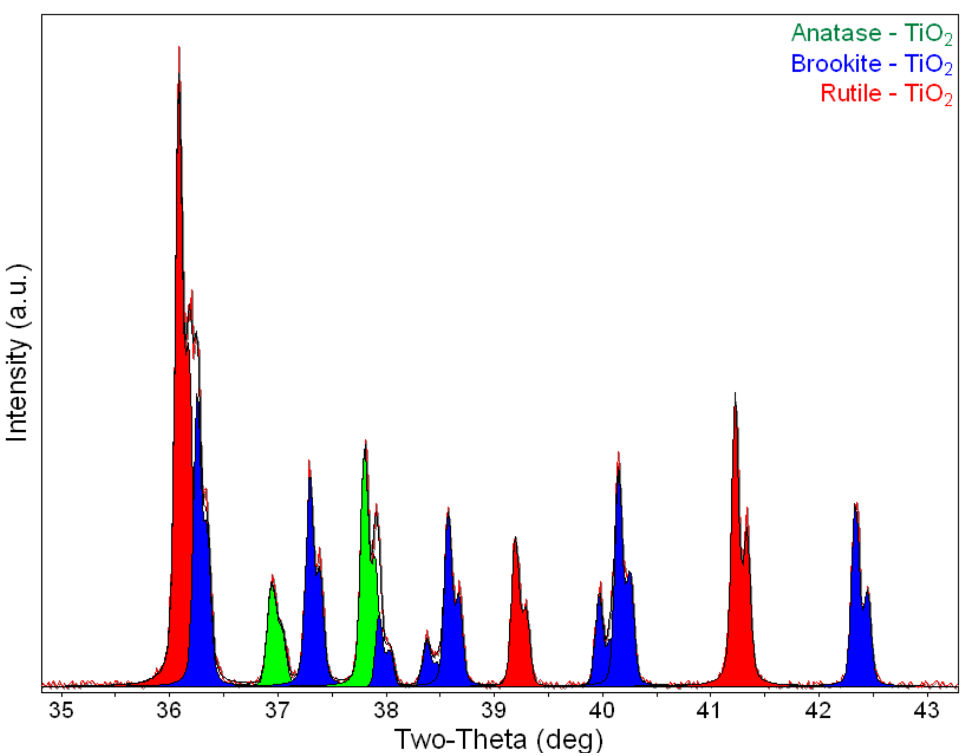
- For every set of planes, there will be a small percentage of crystallites that are properly oriented to diffract (the plane perpendicular bisects the incident and diffracted beams).
- Basic assumptions of powder diffraction are that for every set of planes there is an equal number of crystallites that will diffract and that there is a statistically relevant number of crystallites, not just one or two.

You can use XRD to determine

- Phase Composition of a Sample
  - Quantitative Phase Analysis: determine the relative amounts of phases in a mixture by referencing the relative peak intensities
  - Databases, such as Powder Diffraction File (PDF), contain diffraction pattern information for thousands of crystalline phases
- Unit cell lattice parameters and Bravais lattice symmetry
  - Index peak positions
  - Lattice parameters can vary as a function of, and therefore give you information about, alloying, doping, solid solutions, strains, etc.
- Residual Strain (macrostrain)
- Crystal Structure
  - By Rietveld refinement of the entire diffraction pattern
- Epitaxy/Texture/Orientation
- Crystallite Size and Microstrain
  - Indicated by peak broadening
  - Other defects (stacking faults, etc.) can be measured by analysis of peak shapes and peak width

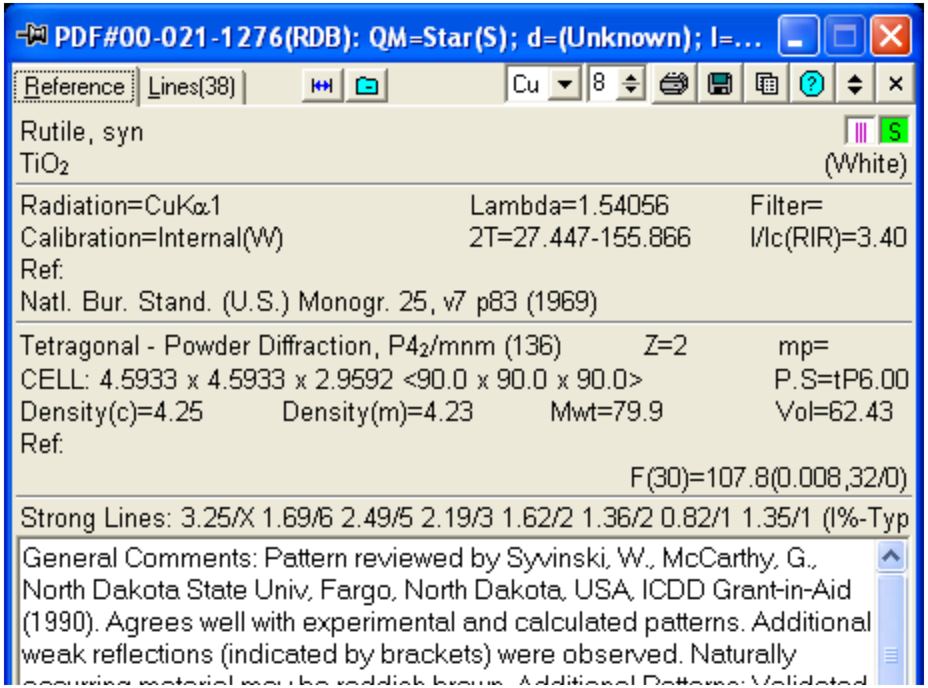
Phase Identification

- The diffraction pattern for every phase is as unique as your fingerprint
  - Phases with the same chemical composition can have drastically different diffraction patterns.
  - Use the position and relative intensity of a series of peaks to match experimental data to the reference patterns in the database



Databases such as the Powder Diffraction File (PDF) contain dI lists for thousands of crystalline phases.

- The PDF contains over 200,000 diffraction patterns.
- Modern computer programs can help you determine what phases are present in your sample by quickly comparing your diffraction data to all of the patterns in the database.
- The PDF card for an entry contains a lot of useful information, including literature references.



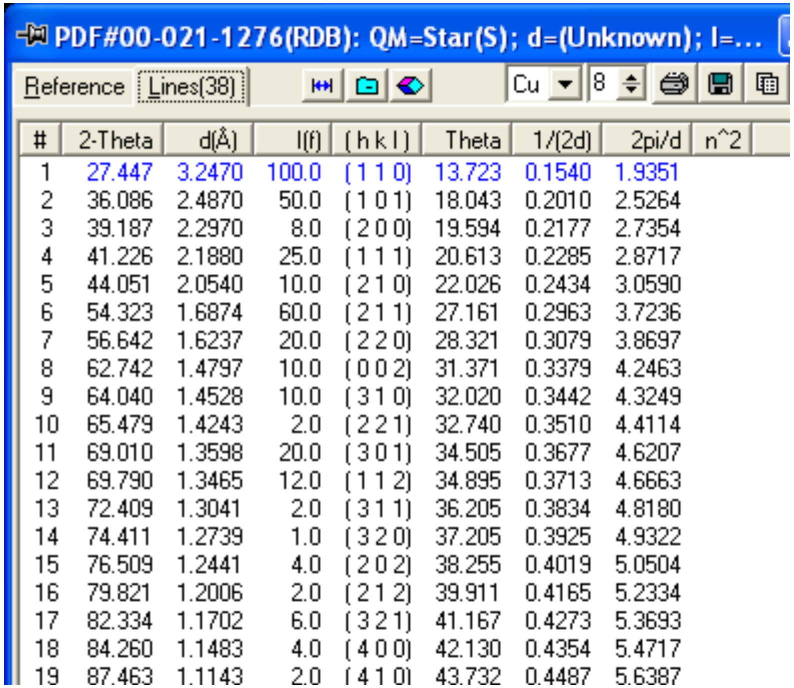
Rutile, syn
TiO_2 (White)

Radiation=CuKα1 Lambda=1.54056 Filter=
Calibration=Internal(W) 2T=27.447-155.866 I/Ic(RIR)=3.40
Ref:
Natl. Bur. Stand. (U.S.) Monogr. 25, v7 p83 (1969)

Tetragonal - Powder Diffraction, $P4_2/mnm$ (136) Z=2 mp=
CELL: 4.5933 x 4.5933 x 2.9592 <90.0 x 90.0 x 90.0> P.S=tP6.00
Density(c)=4.25 Density(m)=4.23 Mwt=79.9 Vol=62.43
Ref:
F(30)=107.8(0.008,32/0)

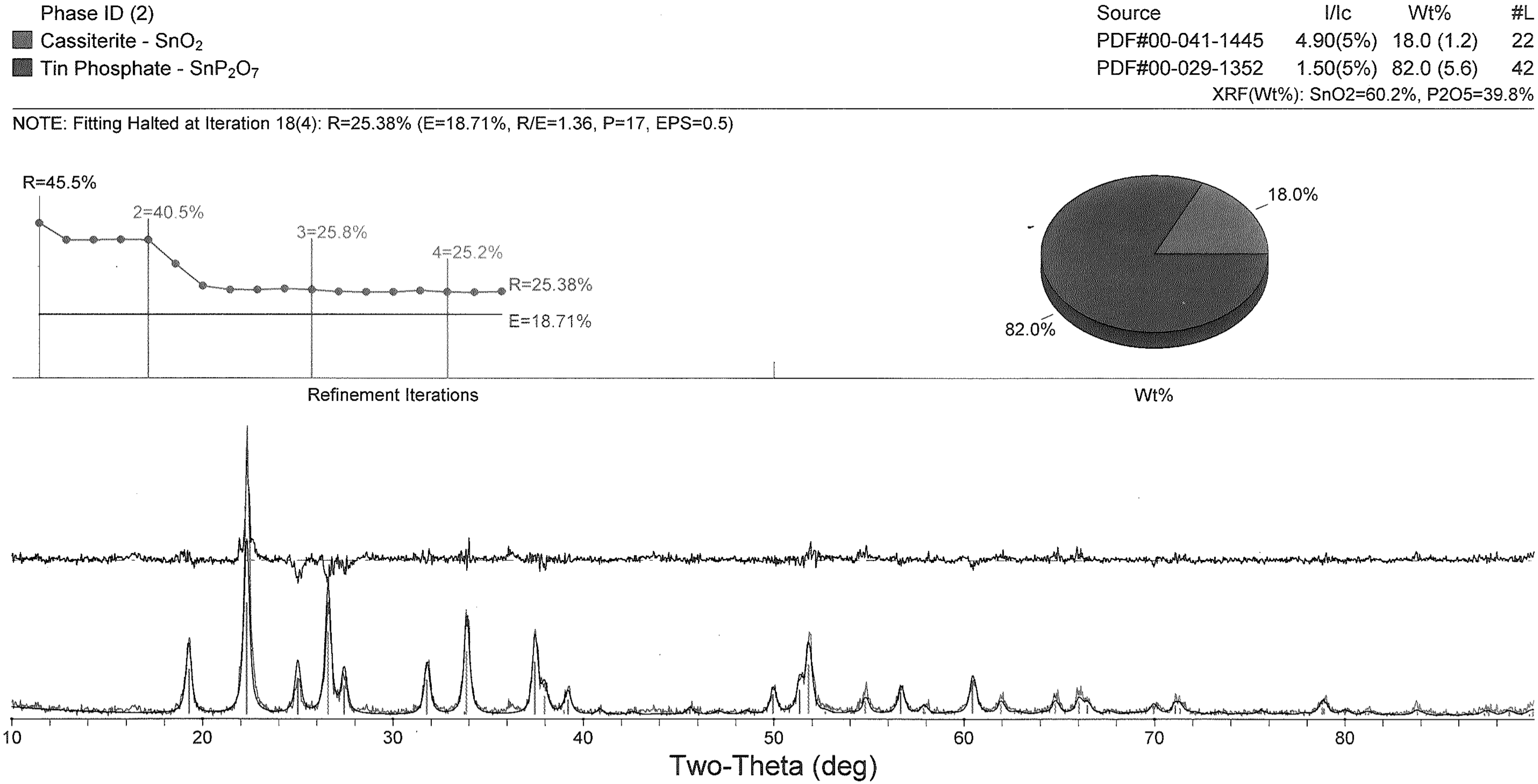
Strong Lines: 3.25/X 1.69/6 2.49/5 2.19/3 1.62/2 1.36/2 0.82/1 1.35/1 (I%-Typ

General Comments: Pattern reviewed by Syvinski, W., McCarthy, G., North Dakota State Univ, Fargo, North Dakota, USA, ICDD Grant-in-Aid (1990). Agrees well with experimental and calculated patterns. Additional weak reflections (indicated by brackets) were observed. Naturally



#	2-Theta	d(Å)	I(f)	(h k l)	Theta	1/(2d)	2pi/d	n^2
1	27.447	3.2470	100.0	(1 1 0)	13.723	0.1540	1.9351	
2	36.086	2.4870	50.0	(1 0 1)	18.043	0.2010	2.5264	
3	39.187	2.2970	8.0	(2 0 0)	19.594	0.2177	2.7354	
4	41.226	2.1880	25.0	(1 1 1)	20.613	0.2285	2.8717	
5	44.051	2.0540	10.0	(2 1 0)	22.026	0.2434	3.0590	
6	54.323	1.6874	60.0	(2 1 1)	27.161	0.2963	3.7236	
7	56.642	1.6237	20.0	(2 2 0)	28.321	0.3079	3.8697	
8	62.742	1.4797	10.0	(0 0 2)	31.371	0.3379	4.2463	
9	64.040	1.4528	10.0	(3 1 0)	32.020	0.3442	4.3249	
10	65.479	1.4243	2.0	(2 2 1)	32.740	0.3510	4.4114	
11	69.010	1.3598	20.0	(3 0 1)	34.505	0.3677	4.6207	
12	69.790	1.3465	12.0	(1 1 2)	34.895	0.3713	4.6663	
13	72.409	1.3041	2.0	(3 1 1)	36.205	0.3834	4.8180	
14	74.411	1.2739	1.0	(3 2 0)	37.205	0.3925	4.9322	
15	76.509	1.2441	4.0	(2 0 2)	38.255	0.4019	5.0504	
16	79.821	1.2006	2.0	(2 1 2)	39.911	0.4165	5.2334	
17	82.334	1.1702	6.0	(3 2 1)	41.167	0.4273	5.3693	
18	84.260	1.1483	4.0	(4 0 0)	42.130	0.4354	5.4717	
19	87.463	1.1143	2.0	(4 1 0)	43.732	0.4487	5.6387	

XRD Basics: Phase Identification/ Whole profile fitting



Phase ID (2)	Source	I/Ic	Wt%	#L
▨ Cassiterite - SnO_2	PDF#00-041-1445	4.90(5%)	18.0 (1.2)	22
▨ Tin Phosphate - SnP_2O_7	PDF#00-029-1352	1.50(5%)	82.0 (5.6)	42

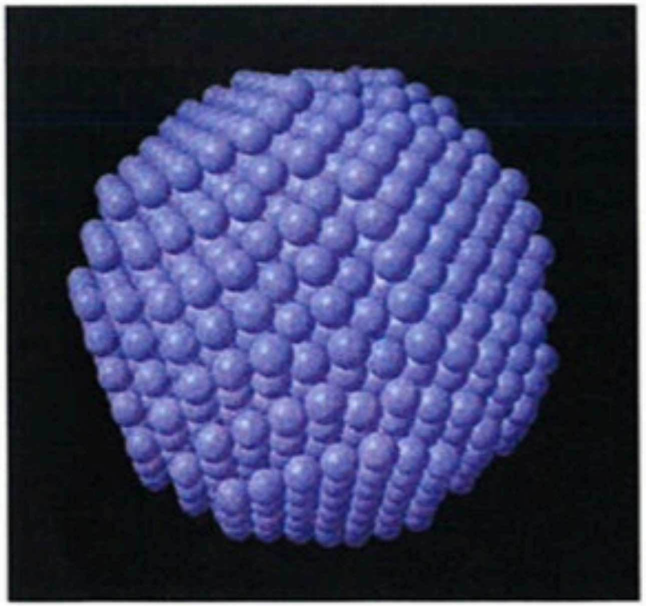
XRF(Wt%): SnO2=60.2%, P2O5=39.8%

NOTE: Fitting Halted at Iteration 18(4): R=25.38% (E=18.71%, R/E=1.36, P=17, EPS=0.5)

- 18% unreacted SnO_2 in synthesis of SnP_2O_7
- Phase composition determined from Relative Intensity Ratio (RIR)
- Primary diffraction peak of “phase” relative to primary diffraction peak of Al_2O_3 in 1:1 mass ratio mixture

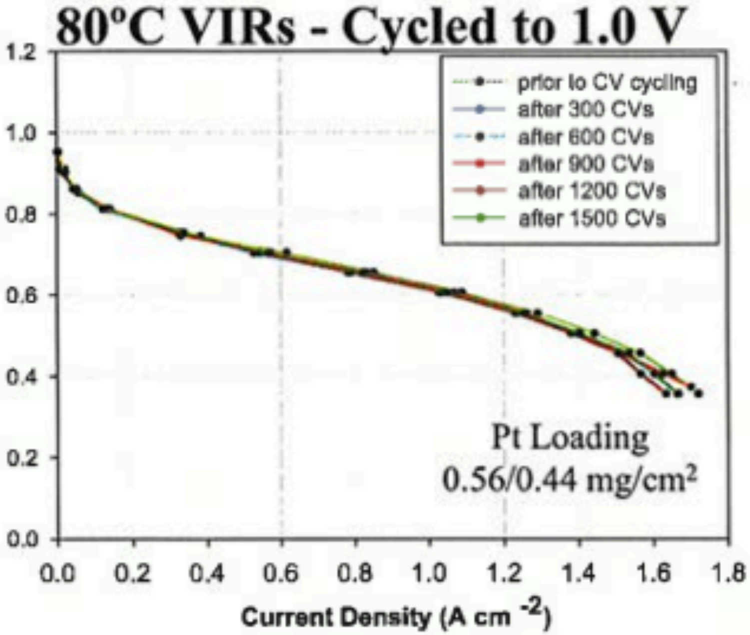
XRD Example: Identifying Pt coarsening

- Catalyst as received from manufacturers in a variety of states:
  - Pt on carbon usually very crystalline
  - Pt alloys e.g. PtRu, Pt_3Cr, PtCo may mixtures of alloys, unalloyed metals, and oxides/hydroxides
- Pt alloy composition and crystallinity may change with fuel cell operation
- Past research indicates Pt catalysts will also coarsen and lose surface area under steady state PEFC operation
  - Wilson et al J. Electrochem. Soc V140 10. 1993

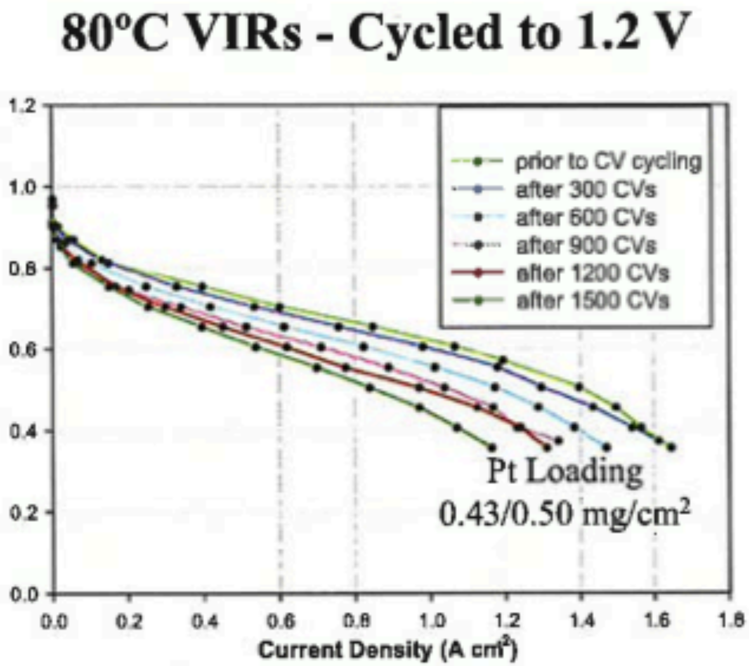


Pt small crystal model

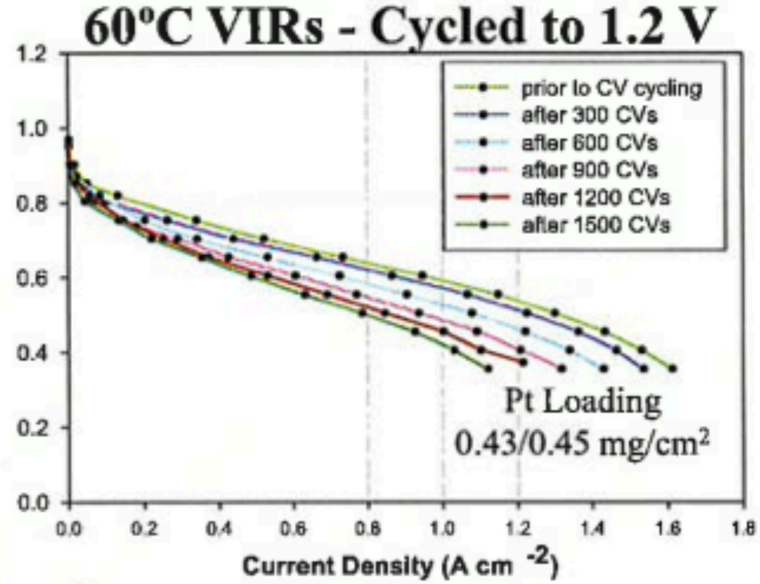
XRD Example: Identifying Pt coarsening



80°C VIRs - Cycled to 1.0 V



80°C VIRs - Cycled to 1.2 V

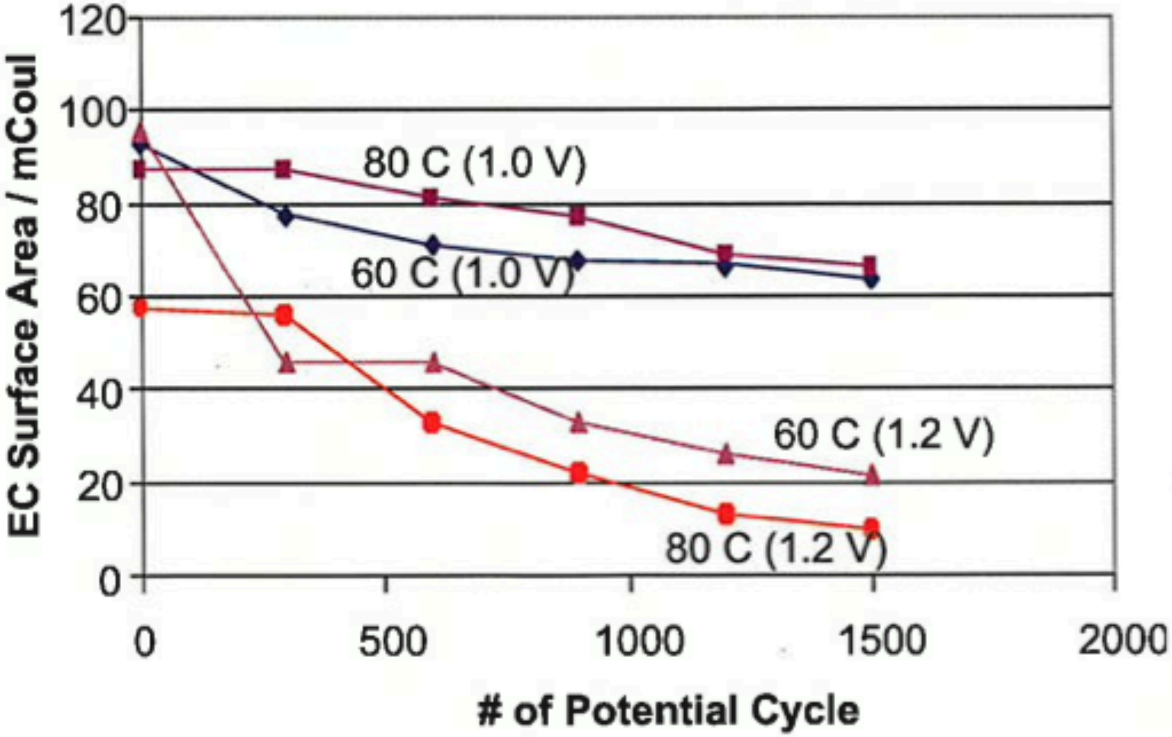


60°C VIRs - Cycled to 1.2 V

- Small degradation at 1.0 V cycling
- Large degradation at 1.2 V cycling
- Only 80 hrs of fuel cell operation

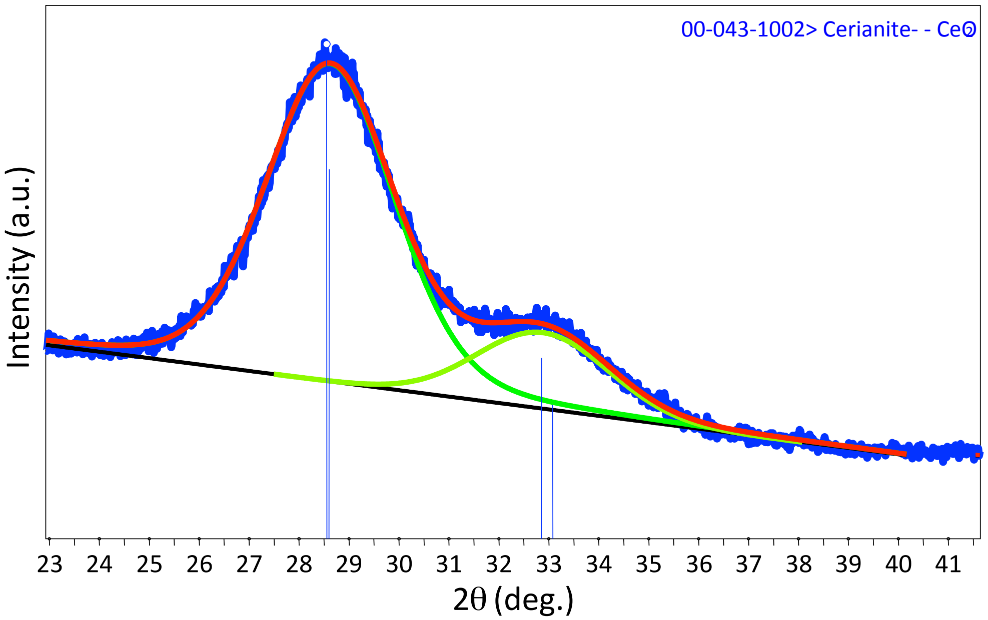
XRD Example: Identifying Pt coarsening

- No net mass loss of catalyst
  - Total loadings the same before and after cycling confirmed by XRF
- Catalyst particle growth?



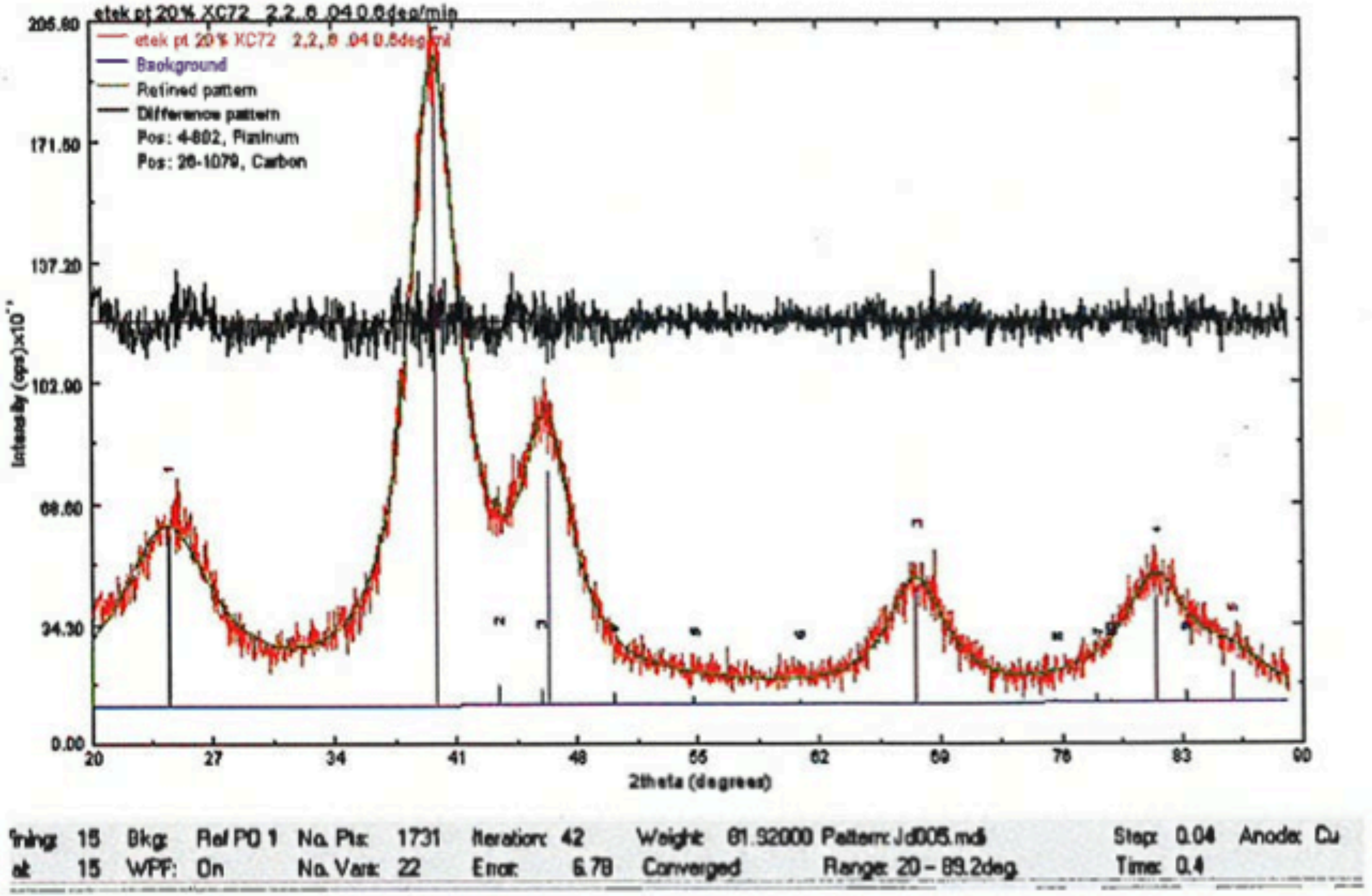
Crystallite Size and Microstrain

- Crystallites smaller than ~120nm create broadening of diffraction peaks
  - this peak broadening can be used to quantify the average crystallite size of nanoparticles using the Scherrer equation
  - must know the contribution of peak width from the instrument by using a calibration curve
- microstrain may also create peak broadening
  - analyzing the peak widths over a long range of 2theta using a Williamson-Hull plot can let you separate microstrain and crystallite size



$$B(2\theta) = \frac{K\lambda}{L\cos\theta}$$

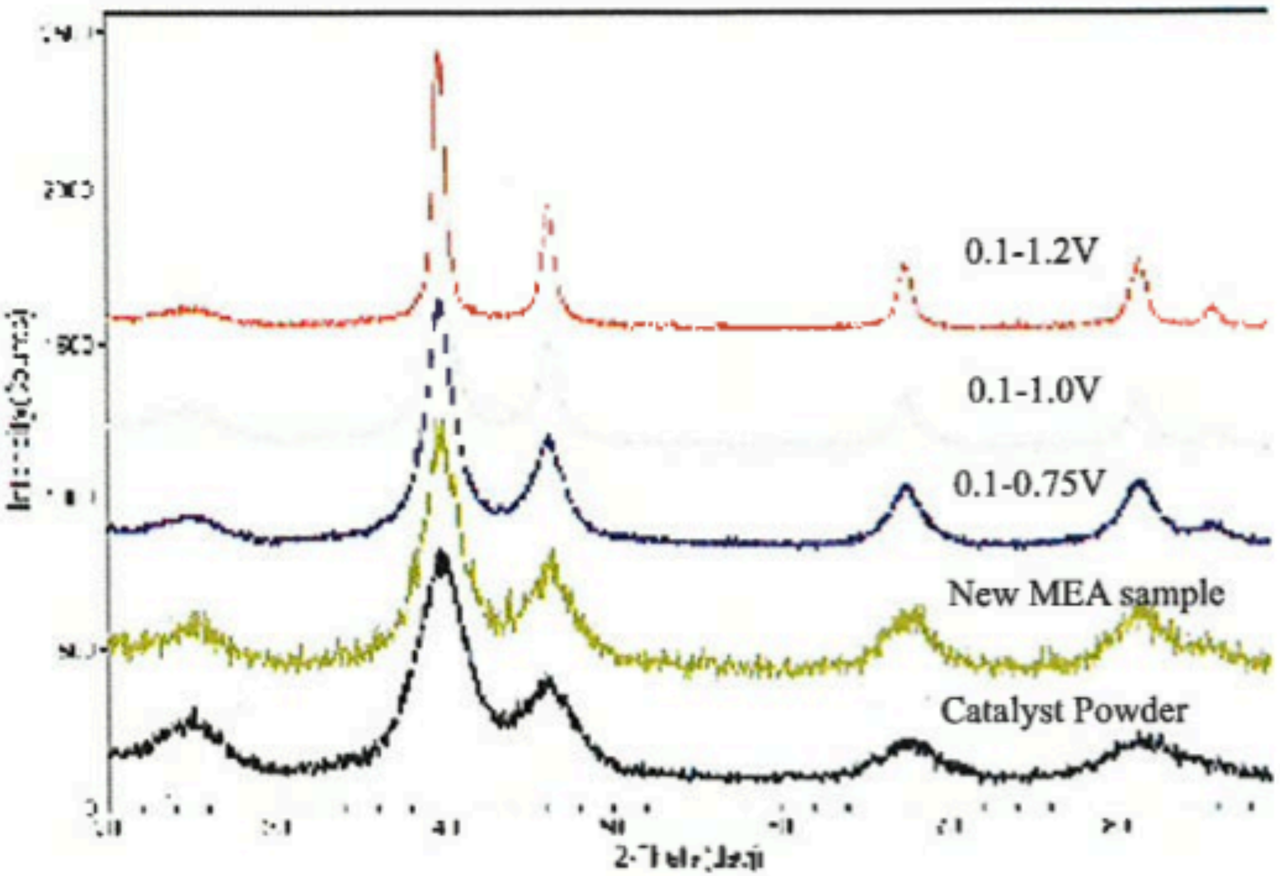
XRD Example: Identifying Pt coarsening



```
hing:  15  Bkg:  Ref PO 1  No. Pts:   1731  Iteration: 42     Weight:  81.92000 Pattern: Jd005.mdi   Step:  0.04  Anode: Cu
at     15  WPF:  On        No. Vars:  22    Error:      6.78  Converged          Range: 20 - 89.2deg   Time:  0.4
```

- ETEK Pt 20% -C sample ~20 Å vol weighted size
- a=3.9261 (0.00068)Å

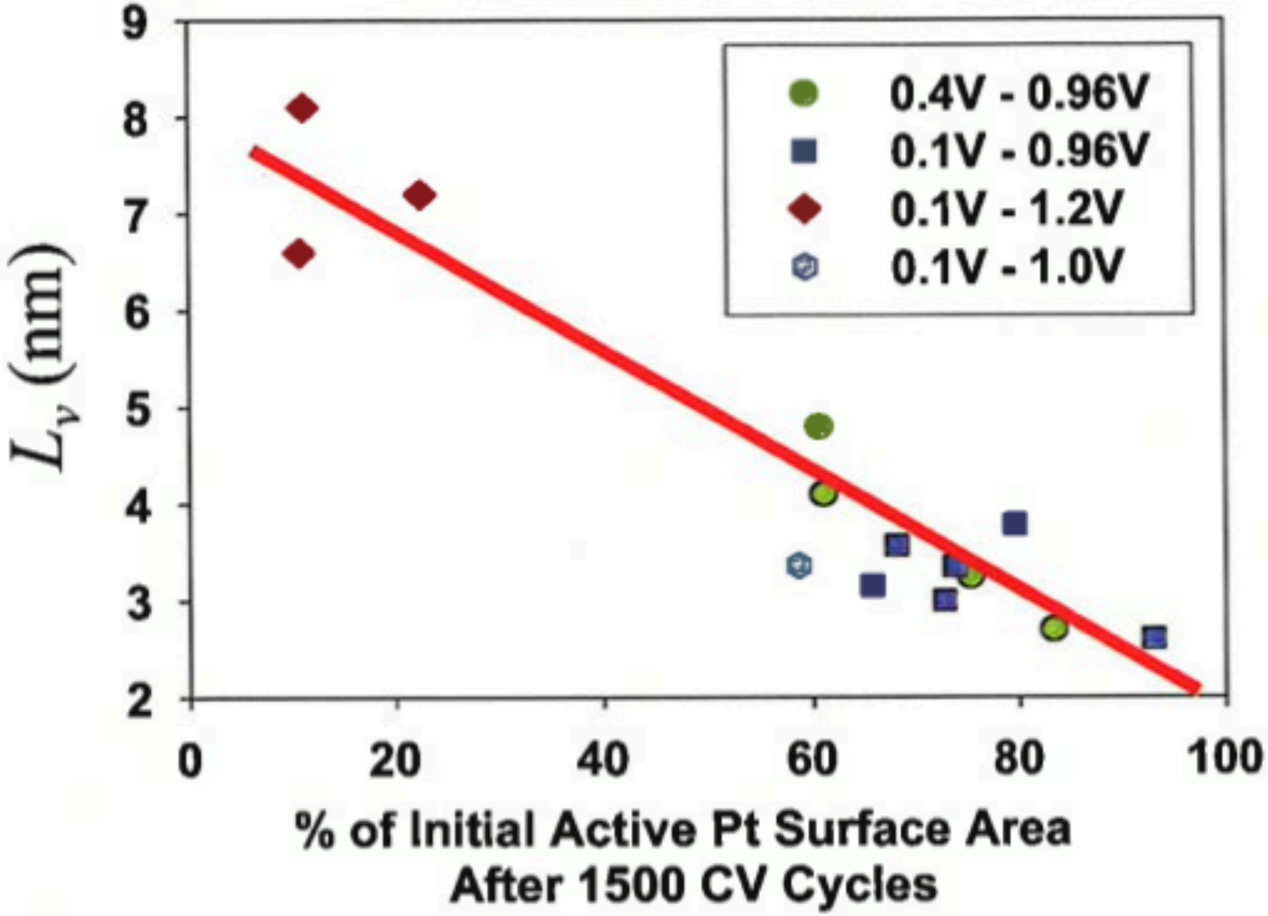
XRD Example: Identifying Pt coarsening



- All cathodes catalysts coarsened
- Particle size growth increases with maximum cycling potentials

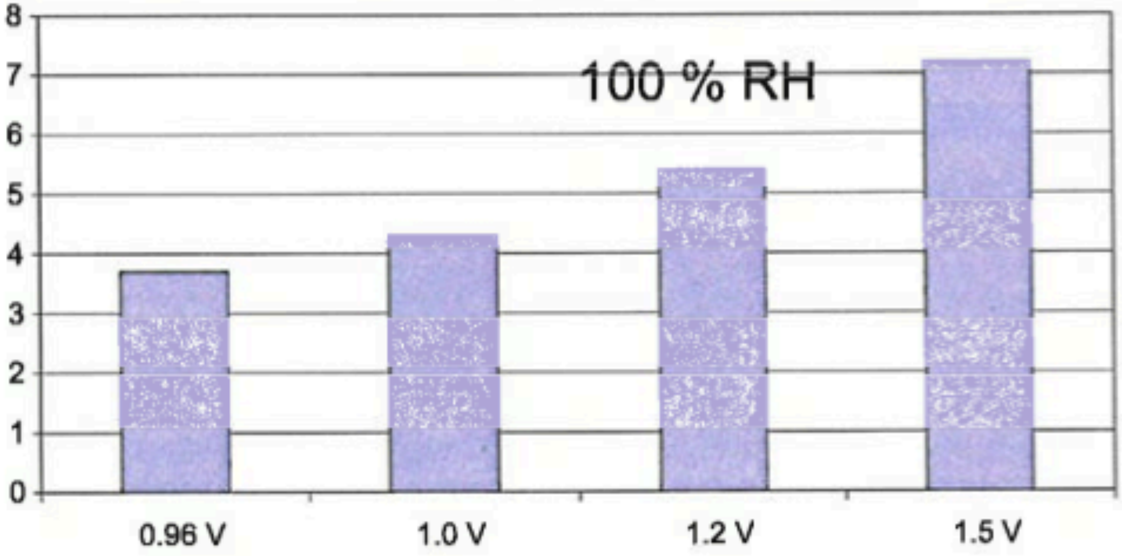
XRD Example: Identifying Pt coarsening

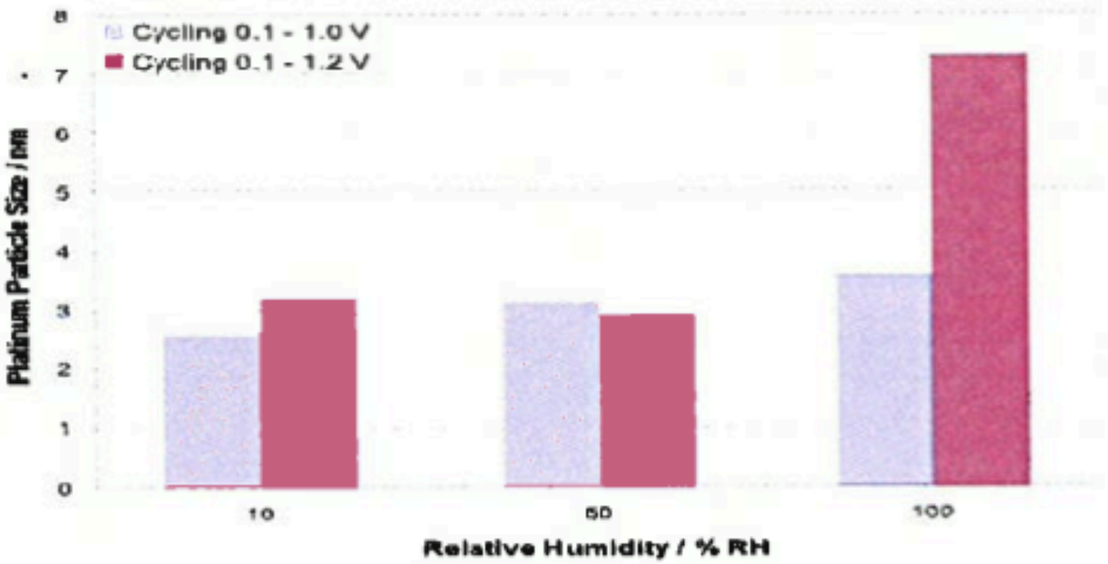
Cell 80 °C
H2 226% RH
Air 100% RH



XRD Example: Identifying Pt coarsening

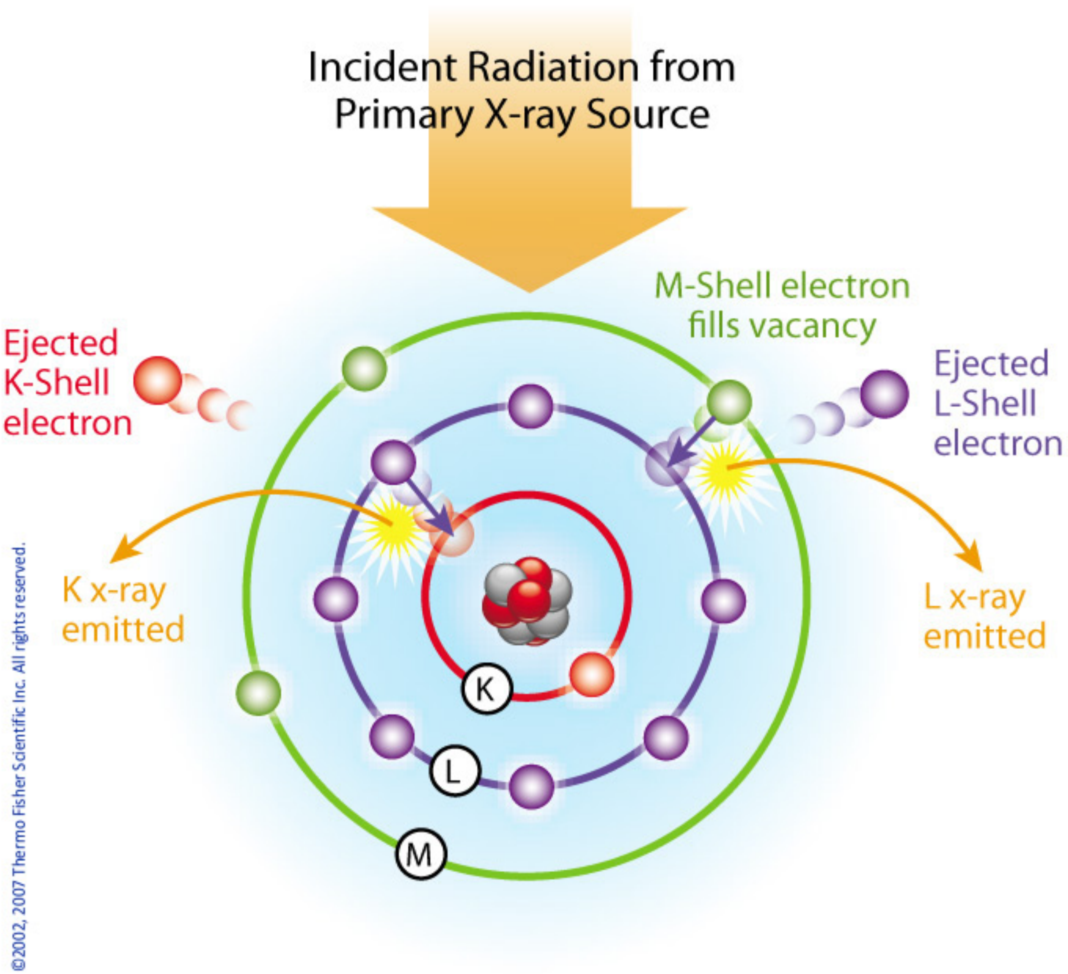
- Strongly correlated to max cycling voltage (80°C)
- Dry cell conditions decrease growth





XRF: Principle of operation

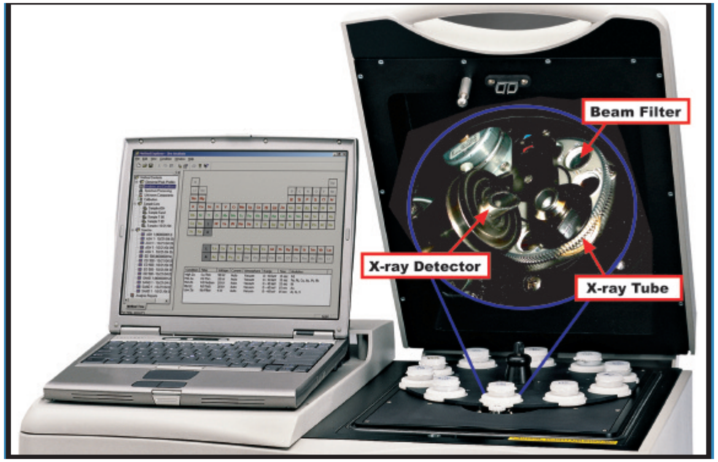
- A fluorescent x-ray is created when an x-ray of sufficient energy strikes an atom in the sample, dislodging an electron from one of the atom's inner orbital shells.
- The atom regains stability, filling the vacancy left in the inner orbital shell with an electron from one of the atom's higher energy orbital shells.
- The electron drops to the lower energy state by releasing a fluorescent x-ray, and the energy of this x-ray is equal to the specific difference in energy between two quantum states of the electron.

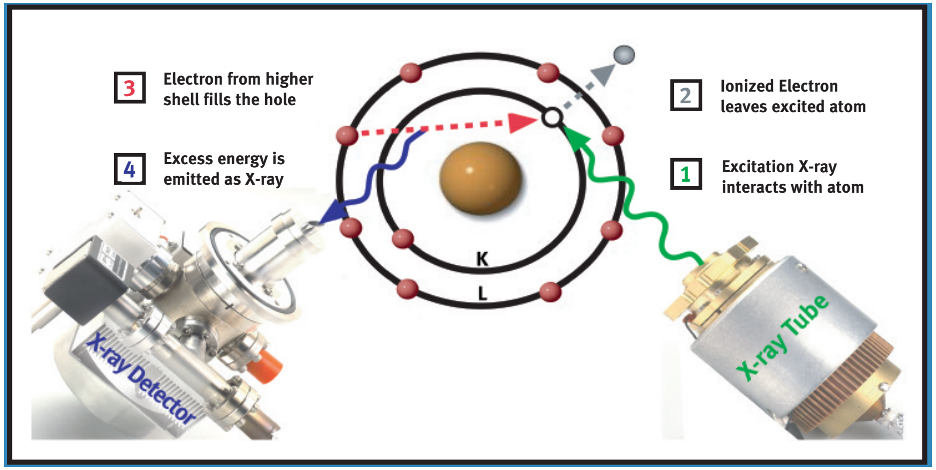


http://www.niton.com/en/portable-xrf-technology/how-xrf-works/x-ray-fluorescence-(edxrf)-overview

XRF: Capabilities and Considerations

- Elemental Identification: The energy difference between two specific orbital shells in a given element is always the same. The photon emitted when an electron moves between these two levels, will always have the same energy.
- Intensity at given energy (counts per second) is proportional to concentration of element in measured sample.
- There must be a significant source peak above absorption edge energy of the element of interest.

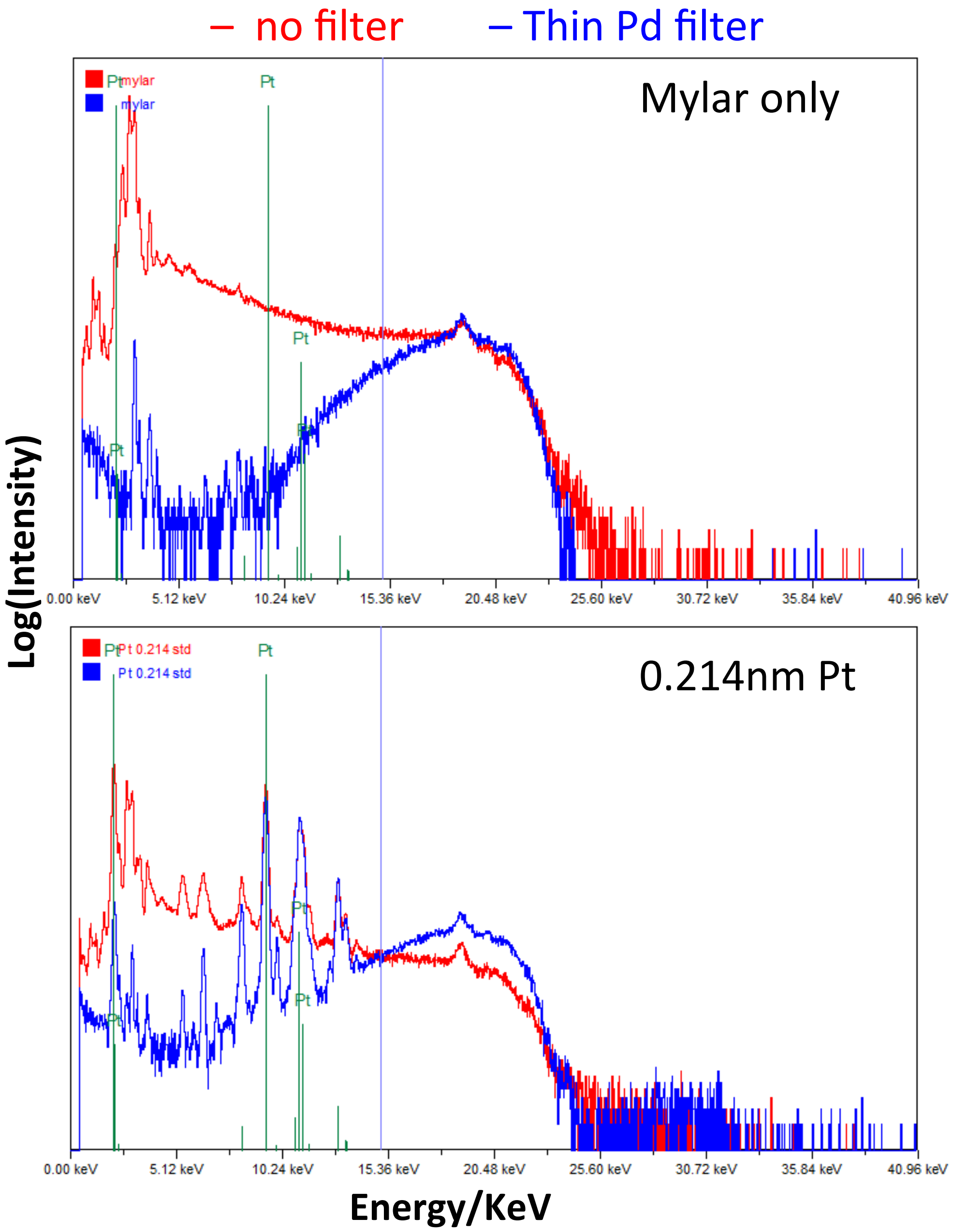




- Nondestructive
- Sensitivity to ppm (μg/cm^2)
- Spatial resolution with mapping stage
- Analysis of unknowns (qual or quant)
- Quantitative analysis through use of Fundamental Parameters Model with standards
  - Composition/loading
  - Multi-layer composition/thickness

www.thermoscientific.com/xray

XRF Example: Defining Acquisition Parameters



User defined settings:

Filter: modifies shape of source spectrum and improves S/N around region of interest

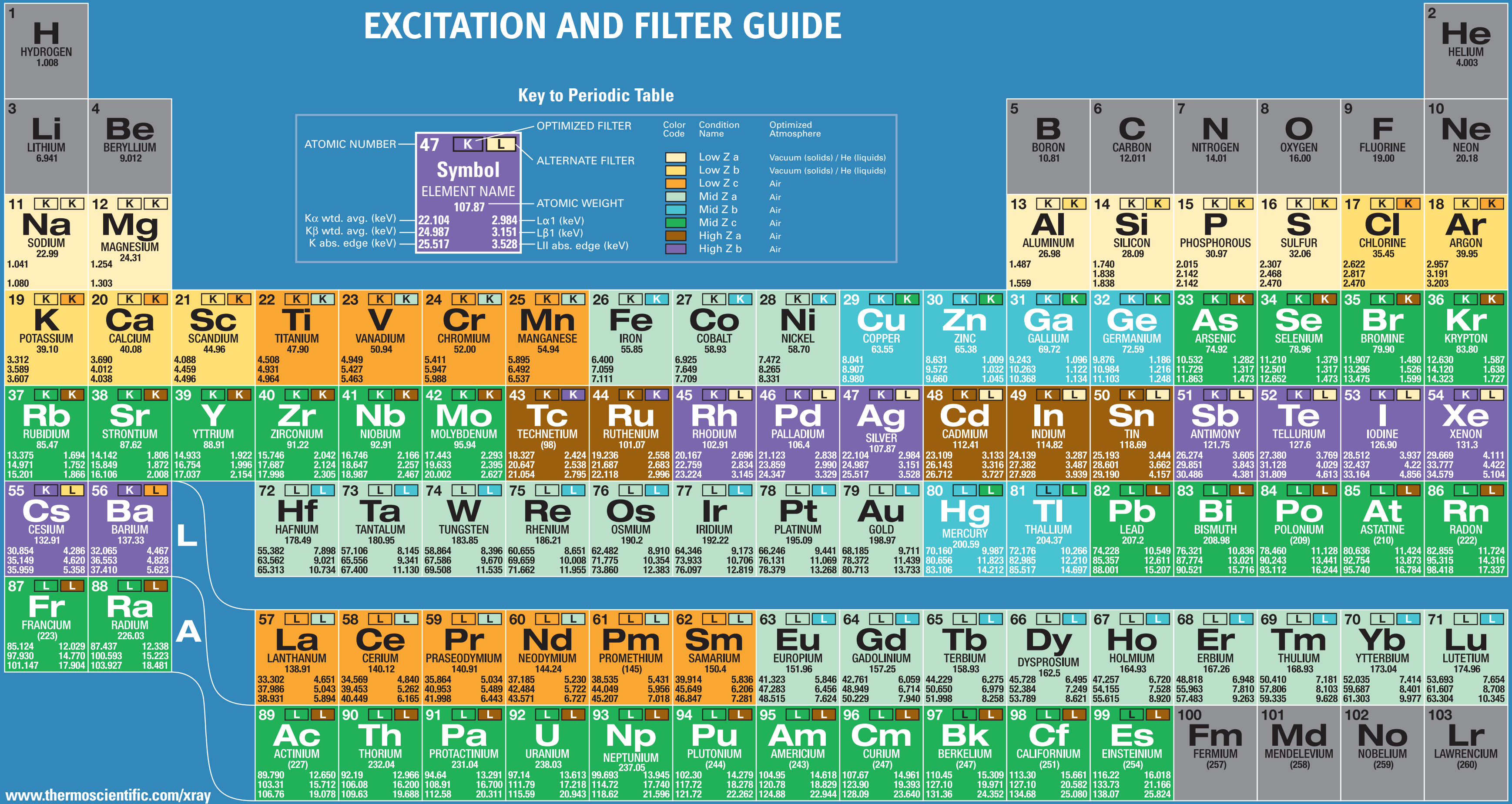
Optimal voltage: usually 1.5–2 times the absorption edge energy of the highest energy element of interest

Current: Maximize count rates relative to detector deadtime (interval during which detector is processing prior signal– subsequent photons entering detector are not counted).

Collection time:
10,000 counts/ROI ≈ 1% error

ENERGY-DISPERSIVE XRF

EXCITATION AND FILTER GUIDE



Key to Periodic Table

ATOMIC NUMBER — 47	K — OPTIMIZED FILTER	L — ALTERNATE FILTER
	Symbol	
	ELEMENT NAME	
	107.87	ATOMIC WEIGHT
Kα wtd. avg. (keV) — 22.104		2.984 — Lα1 (keV)
Kβ wtd. avg. (keV) — 24.987		3.151 — Lβ1 (keV)
K abs. edge (keV) — 25.517		3.528 — LII abs. edge (keV)

Color Code	Condition Name	Optimized Atmosphere
🟨	Low Z a	Vacuum (solids) / He (liquids)
🟨	Low Z b	Vacuum (solids) / He (liquids)
🟧	Low Z c	Air
🟩	Mid Z a	Air
🟦	Mid Z b	Air
🟩	Mid Z c	Air
🟫	High Z a	Air
🟪	High Z b	Air

1 **H** HYDROGEN 1.008																	2 **He** HELIUM 4.003
3 **Li** LITHIUM 6.941	4 **Be** BERYLLIUM 9.012											5 **B** BORON 10.81	6 **C** CARBON 12.011	7 **N** NITROGEN 14.01	8 **O** OXYGEN 16.00	9 **F** FLUORINE 19.00	10 **Ne** NEON 20.18
11 K K **Na** SODIUM 22.99 1.041 1.080	12 K K **Mg** MAGNESIUM 24.31 1.254 1.303											13 K K **Al** ALUMINUM 26.98 1.487 1.559	14 K K **Si** SILICON 28.09 1.740 1.838 1.838	15 K K **P** PHOSPHOROUS 30.97 2.015 2.142 2.142	16 K K **S** SULFUR 32.06 2.307 2.468 2.470	17 K K **Cl** CHLORINE 35.45 2.622 2.817 2.470	18 K K **Ar** ARGON 39.95 2.957 3.191 3.203
19 K K **K** POTASSIUM 39.10 3.312 3.589 3.607	20 K K **Ca** CALCIUM 40.08 3.690 4.012 4.038	21 K K **Sc** SCANDIUM 44.96 4.088 4.459 4.496	22 K K **Ti** TITANIUM 47.90 4.508 4.931 4.964	23 K K **V** VANADIUM 50.94 4.949 5.427 5.463	24 K K **Cr** CHROMIUM 52.00 5.411 5.947 5.988	25 K K **Mn** MANGANESE 54.94 5.895 6.492 6.537	26 K K **Fe** IRON 55.85 6.400 7.059 7.111	27 K K **Co** COBALT 58.93 6.925 7.649 7.709	28 K K **Ni** NICKEL 58.70 7.472 8.265 8.331	29 K K **Cu** COPPER 63.55 8.041 8.907 8.980	30 K K **Zn** ZINC 65.38 8.631 1.009 9.572 1.032 9.660 1.045	31 K K **Ga** GALLIUM 69.72 9.243 1.096 10.263 1.122 10.368 1.134	32 K K **Ge** GERMANIUM 72.59 9.876 1.186 10.984 1.216 11.103 1.248	33 K K **As** ARSENIC 74.92 10.532 1.282 11.729 1.317 11.863 1.473	34 K K **Se** SELENIUM 78.96 11.210 1.379 12.501 1.317 12.652 1.473	35 K K **Br** BROMINE 79.90 11.907 1.480 13.296 1.526 13.475 1.599	36 K K **Kr** KRYPTON 83.80 12.630 1.587 14.120 1.638 14.323 1.727
37 K K **Rb** RUBIDIUM 85.47 13.375 1.694 14.971 1.752 15.201 1.866	38 K K **Sr** STRONTIUM 87.62 14.142 1.806 15.849 1.872 16.106 2.008	39 K K **Y** YTTRIUM 88.91 14.933 1.922 16.754 1.996 17.037 2.154	40 K K **Zr** ZIRCONIUM 91.22 15.746 2.042 17.687 2.124 17.998 2.305	41 K K **Nb** NIOBIUM 92.91 16.746 2.166 18.647 2.257 18.987 2.467	42 K K **Mo** MOLYBDENUM 95.94 17.443 2.293 19.633 2.395 20.002 2.627	43 K K **Tc** TECHNETIUM (98) 18.327 2.424 20.647 2.538 21.054 2.795	44 K K **Ru** RUTHENIUM 101.07 19.236 2.558 21.687 2.683 22.118 2.996	45 K L **Rh** RHODIUM 102.91 20.167 2.696 22.759 2.834 23.224 3.145	46 K L **Pd** PALLADIUM 106.4 21.123 2.838 23.859 2.990 24.347 3.329	47 K L **Ag** SILVER 107.87 22.104 2.984 24.987 3.151 25.517 3.528	48 K L **Cd** CADMIUM 112.41 23.109 3.133 26.143 3.316 26.712 3.727	49 K L **In** INDIUM 114.82 24.139 3.287 27.382 3.487 27.928 3.939	50 K L **Sn** TIN 118.69 25.193 3.444 28.601 3.662 29.190 4.157	51 K L **Sb** ANTIMONY 121.75 26.274 3.605 29.851 3.843 30.486 4.381	52 K L **Te** TELLURIUM 127.6 27.380 3.769 31.128 4.029 31.809 4.613	53 K L **I** IODINE 126.90 28.512 3.937 32.437 4.22 33.164 4.856	54 K L **Xe** XENON 131.3 29.669 4.111 33.777 4.422 34.579 5.104
55 K L **Cs** CESIUM 132.91 30.854 4.286 35.149 4.620 35.959 5.358	56 K L **Ba** BARIUM 137.33 32.065 4.467 36.553 4.828 37.410 5.623	L	72 L L **Hf** HAFNIUM 178.49 55.382 7.898 63.562 9.021 65.313 10.734	73 L L **Ta** TANTALUM 180.95 57.106 8.145 65.556 9.341 67.400 11.130	74 L L **W** TUNGSTEN 183.85 58.864 8.396 67.586 9.670 69.508 11.535	75 L L **Re** RHENIUM 186.21 60.655 8.651 69.659 10.008 71.662 11.955	76 L L **Os** OSMIUM 190.2 62.482 8.910 71.775 10.354 73.860 12.383	77 L L **Ir** IRIDIUM 192.22 64.346 9.173 73.933 10.706 76.097 12.819	78 L L **Pt** PLATINUM 195.09 66.246 9.441 76.131 11.069 78.379 13.268	79 L L **Au** GOLD 198.97 68.185 9.711 78.372 11.439 80.713 13.733	80 L L **Hg** MERCURY 200.59 70.160 9.987 80.656 11.823 83.106 14.212	81 L L **Tl** THALLIUM 204.37 72.176 10.266 82.985 12.210 85.517 14.697	82 L L **Pb** LEAD 207.2 74.228 10.549 85.357 12.611 88.001 15.207	83 L L **Bi** BISMUTH 208.98 76.321 10.836 87.774 13.021 90.521 15.716	84 L L **Po** POLONIUM (209) 78.460 11.128 90.243 13.441 93.112 16.244	85 L L **At** ASTATINE (210) 80.636 11.424 92.754 13.873 95.740 16.784	86 L L **Rn** RADON (222) 82.855 11.724 95.315 14.316 98.418 17.337
87 L L **Fr** FRANCIUM (223) 85.124 12.029 97.930 14.770 101.147 17.904	88 L L **Ra** RADIUM 226.03 87.437 12.338 100.593 15.223 103.927 18.481	A															
			57 L L **La** LANTHANUM 138.91 33.302 4.651 37.986 5.043 38.931 5.894	58 L L **Ce** CERIUM 140.12 34.569 4.840 39.453 5.262 40.449 6.165	59 L L **Pr** PRASEODYMIUM 140.91 35.864 5.034 40.953 5.489 41.998 6.443	60 L L **Nd** NEODYMIUM 144.24 37.185 5.230 42.484 5.722 43.571 6.727	61 L L **Pm** PROMETHIUM (145) 38.535 5.431 44.049 5.956 45.207 7.018	62 L L **Sm** SAMARIUM 150.4 39.914 5.836 45.649 6.206 46.847 7.281	63 L L **Eu** EUROPIUM 151.96 41.323 5.846 47.283 6.456 48.515 7.624	64 L L **Gd** GADOLINIUM 157.25 42.761 6.059 48.949 6.714 50.229 7.940	65 L L **Tb** TERBIUM 158.93 44.229 6.275 50.650 6.979 51.998 8.258	66 L L **Dy** DYSPROSIUM 162.5 45.728 6.495 52.384 7.249 53.789 8.621	67 L L **Ho** HOLMIUM 164.93 47.257 6.720 54.155 7.528 55.615 8.920	68 L L **Er** ERBIUM 167.26 48.818 6.948 55.963 7.810 57.483 9.263	69 L L **Tm** THULIUM 168.93 50.410 7.181 57.806 8.103 59.335 9.628	70 L L **Yb** YTTERBIUM 173.04 52.035 7.414 59.687 8.401 61.303 9.977	71 L L **Lu** LUTETIUM 174.96 53.693 7.654 61.607 8.708 63.304 10.345
			89 L L **Ac** ACTINIUM (227) 89.790 12.650 103.31 15.712 106.76 19.078	90 L L **Th** THORIUM 232.04 92.19 12.966 106.08 16.200 109.63 19.688	91 L L **Pa** PROTACTINIUM 231.04 94.64 13.291 108.91 16.700 112.58 20.311	92 L L **U** URANIUM 238.03 97.14 13.613 111.79 17.218 115.59 20.943	93 L L **Np** NEPTUNIUM 237.05 99.693 13.945 114.72 17.740 118.62 21.596	94 L L **Pu** PLUTONIUM (244) 102.30 14.279 117.72 18.278 121.72 22.262	95 L L **Am** AMERICIUM (243) 104.95 14.618 120.78 18.829 124.88 22.944	96 L L **Cm** CURIUM (247) 107.67 14.961 123.90 19.393 128.09 23.640	97 L L **Bk** BERKELIUM (247) 110.45 15.309 127.10 19.971 131.36 24.352	98 L L **Cf** CALIFORNIUM (251) 113.30 15.661 127.10 20.582 134.68 25.080	99 L L **Es** EINSTEINIUM (254) 116.22 16.018 133.73 21.166 138.07 25.824	100 **Fm** FERMIUM (257)	101 **Md** MENDELEVIUM (258)	102 **No** NOBELIUM (259)	103 **Lr** LAWRENCIUM (260)

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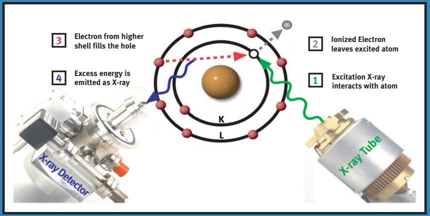
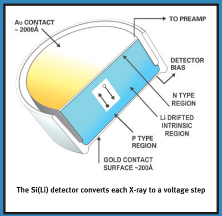
Principles of EDXRF



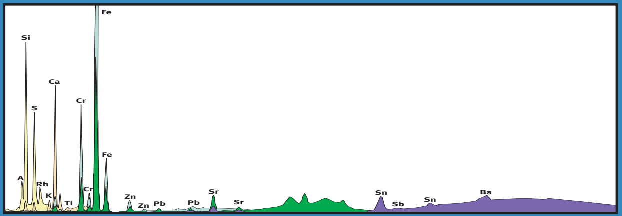
Inside the Spectrometer

Inside the Detector



The Si(Li) detector converts each X-ray to a voltage step

Example of Multi-Filter Analysis of NIST Standard 1645



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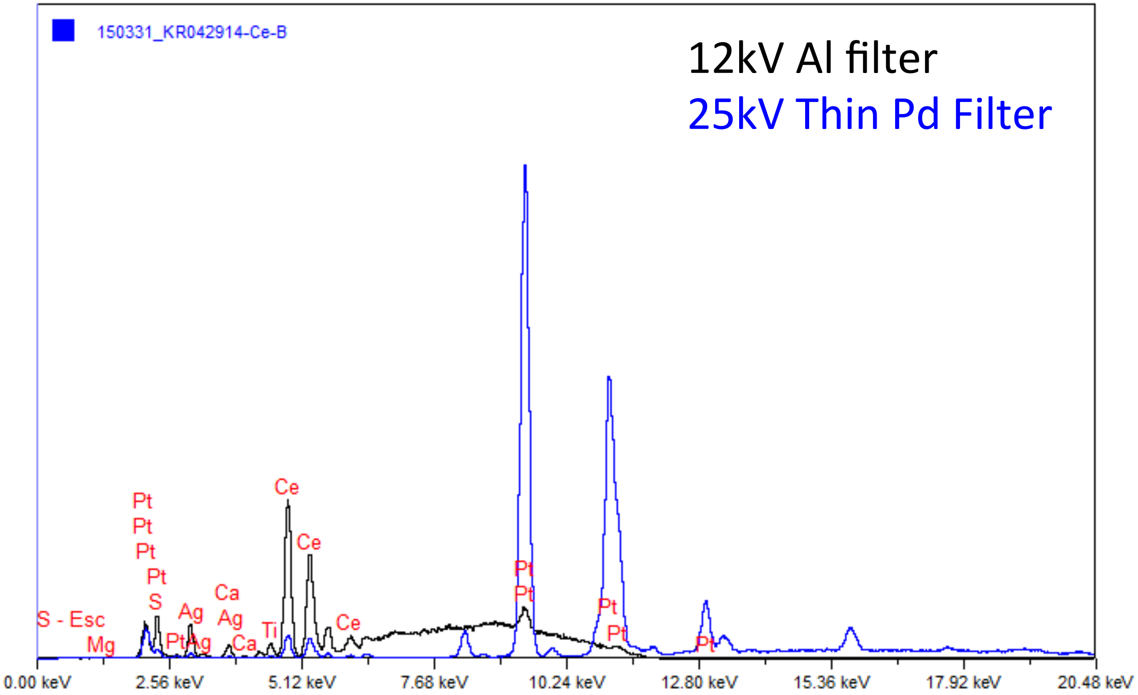
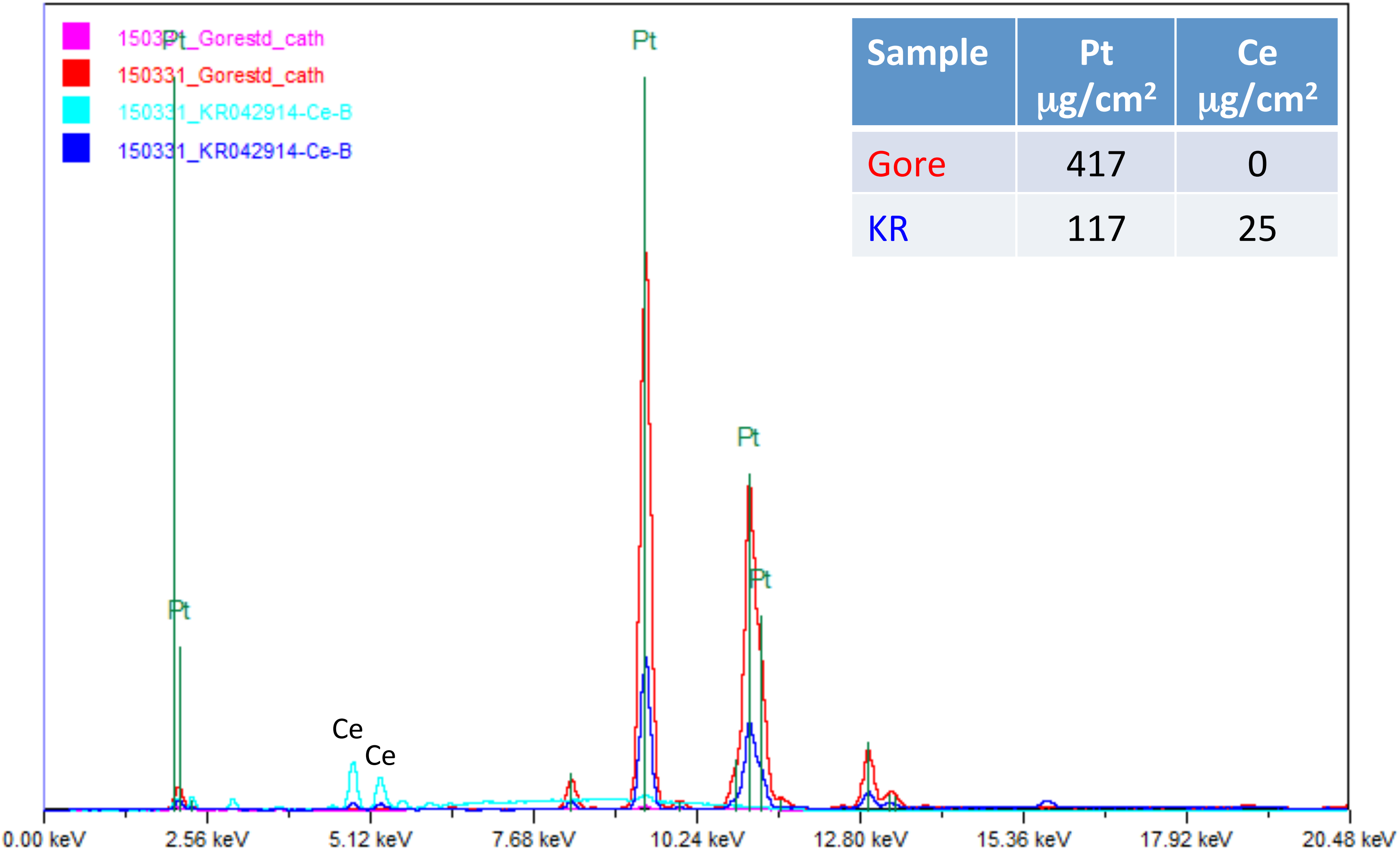
XRF Example: Measuring Pt and Ce loading with fundamental parameters model

1. Create model using known standards
   - CeO_2 bulk powder
   - 0.214nm Pt thin film
2. Record calibration spectra of known standards under Filter/ Voltage setting optimized for each element of interest. This provides relationship between measured intensity and composition of a given element.
3. Record spectra of unknown. FP model will use spectra recorded under optimized conditions for each element.
4. FP model uses iterative regression to calculate composition of unknown based on measured intensities.



XRF Example: Analyzing Pt and Ce loading of two unknowns

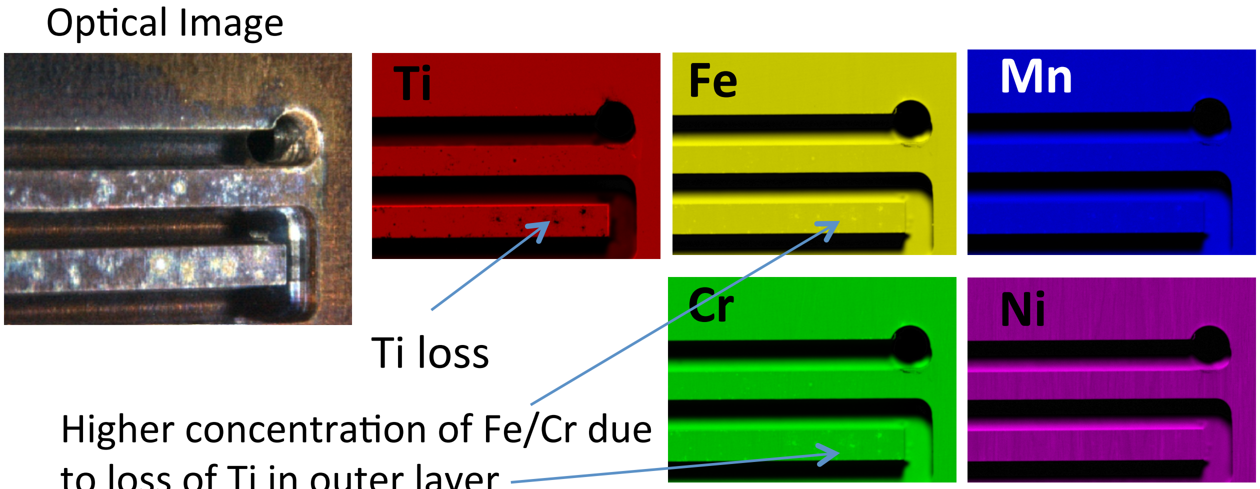


Sample	Pt µg/cm^2	Ce µg/cm^2
Gore	417	0
KR	117	25

XRF Example: Evaluation of novel bipolar plates

- Novel bi-polar plates examined by XRF before and after FC testing
- Analyses of bi-polar plates (post test):
  - Indicates corrosion present on anode plate, typically where large amounts of liquid water is present
  - Minimal corrosion present on cathode plate (but not zero)
- Analysis of MEAs show:
  - Small metal contamination of GDL/ MEA
  - Higher concentrations at outlet where corrosion was evident.

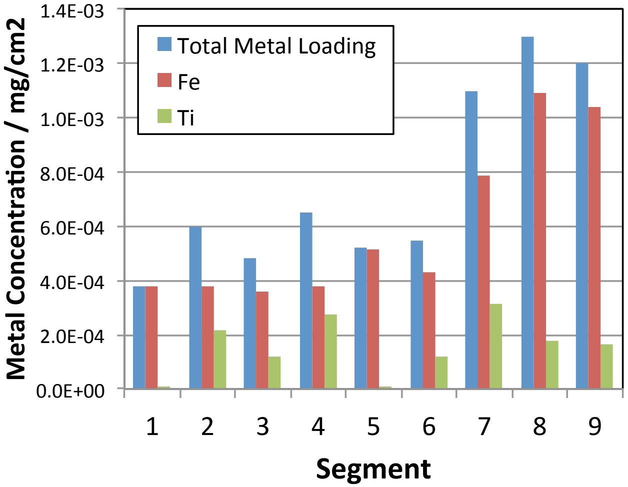
Elemental mapping of anode plate after testing



Ti loss

Higher concentration of Fe/Cr due to loss of Ti in outer layer

Quantitative composition of MEA



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- Data shown in presentation sourced from: F. Garzon, M. Wilson, T. Rockward, and K. Rau

Thank You

Cortney Kreller

ckreller@lanl.gov